CAMECA
SCIENCE & METROLOGY SOLUTIONS

NanoSIMS 50L users guide

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One-page Table of Contents (2 levels)

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1 Introduction

In this user's guide, the operator can find the main information allowing him to use a CAMECA NanoSIMS 50L with best results. This instrument is based on Secondary Ion Mass Spectrometry (SIMS) and designed to provide benchmark capabilities for SIMS analysis at high spatial resolution.

After introduction to SIMS and NanoSIMS in this chapter 1 the first part of this document (chapters 2 and 3) describes the physical principle, the optics, the hardware and the software of the CAMECA NanoSIMS 50L to have an overview of the instrument. It is mandatory to understand at least the main optical concepts in order to make sensible use of the following, more practical chapters.

In the second part (chapters 3 to 8) the operator will have access to an extensive software description and operation interface. One can come back to this part at any time to find details of individual functions.

The third part (chapter 9) is a practical tutorial. There are three levels of operation: basic, advanced and expert. It is our opinion that it is dangerous (for the instrument and analytical results!) to dig directly into expert operation without a clear mental image of the optics and the mastering of lower level operation!

The last part (chapter 10) is dedicated to maintenance operations.

1.1 Introduction to Secondary Ion Mass Spectrometry (SIMS)

Secondary Ion Mass Spectrometry (SIMS) is one of the most sensitive surface analysis techniques. It is based on the mass over charge analysis of secondary ions, emitted from the surface of a sample submitted to a primary ion bombardment. This analysis is performed with a mass spectrometer, the sample and the instrument being under high vacuum to reduce sample contamination and ion/electron beam attenuation or scattering.

SIMS analyses deliver information about the elemental, isotopic and molecular composition of the uppermost atomic monolayers of a sample. It allows the acquisition of mass spectra from the surface, 2D maps or 1D line-scans of the lateral distribution of elements or isotopes, 1D depth profiles or 3D images.

Classically, one divides SIMS technique into dynamic SIMS, where the focus of the analysis is on elements and isotopic ratios, and molecular or static SIMS which is mostly used for molecular characterization of the surface.

The NanoSIMS falls in the dynamic SIMS category. In this mode, relatively energetic (4-16keV for the NS50L) *reactive* primary ions (here Cs^+ and O^- mostly) are used to bombard the surface of the material to analyze. These primary ions will implant in the material and mix the atomic layers through a collision cascade of ~10-20nm depth (Figure 1). As the primary ion dose increases, the *surface concentration* of projectile atoms grows (in the transient regime) then stabilizes (in the steady state regime). In parallel, the sample is sputtered: a small crater forms at the bombarded area. SIMS is thus a *destructive* technique allowing depth profiling.

Quantitative information will always require the use of known reference sample(s).

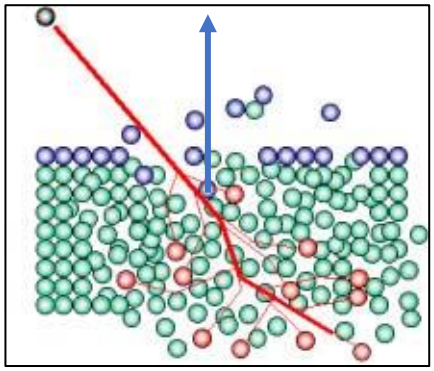


Figure 1: Illustration of the ion collision cascade

The main strengths of SIMS are:

- Surface technique (a few atomic layers).
- Excellent spatial resolution (50nm with the NS50L)
- High sensitivity for most elements (concentrations at ppm level with the NanoSIMS, ppb level with high current instruments).
- Depth profiling capability with excellent depth resolution.
- Access to hydrogen, lithium and other light elements.
- Isotopic ratio information
- Analysis of electrically insulating samples
- Survey and navigation over millimetric to centimetric samples.
- Quantification is very linear and reliable for atomic concentration below say [1at%] and works with a single standard over a concentration range of many decades.

The main challenges of SIMS are:

- A few elements are quasi-inaccessible (rare gases on the NS, mercury)
- A few have moderate sensitivity (zinc, cadmium).
- Secondary ion yield varies greatly with chemical environment and sputtering conditions (ion, energy, angle). This can add complexity to the quantitative aspect of the technique (e.g. the response/sensitivity of silicon will vary strongly between Si, SiO_2, Si_3N_4, SiC,…).
- Quantification requires standard(s) with a similar matrix as the sample.
- Quantification of major elements (above 1at%) requires a calibration curve with several standards of different concentrations.
- Finally, SIMS is a destructive technique, which can be a limitation for samples of very limited availability.

The first thing one does when analyzing the composition of a sample with dynamic SIMS is changing its surface composition by implanting Cs or O. But this is for a good cause: to increase the ionization yield of sputtered atoms by *several* decades. The ionization yield (sensitivity) will be determined by the electron affinity of the element (for negative ions) and by the work function (for the positive ions).

There are many informative literatures in the SIMS domain. A possible start would be to read the small booklet “Dynamic Secondary Ion Mass Spectrometry” Essential Knowledge Briefings (EKB) published by John Wiley & Sons Ltd, downloadable from Cameca web site. The booklet also lists additional references if you wish to go further.

In summary, SIMS is recognized as the most sensitive elemental and isotopic surface analysis technique. This technique is "destructive" by nature (sputtering of material). It can be applied to any type of solid material (insulators, semiconductors, metals) that can sustain high vacuum.

1.2 Introduction to the NanoSIMS

The NanoSIMS is a SIMS instrument optimized for high spatial resolution. Thus, its concept follows a logical analysis of the following problems:

1- As SIMS is destructive and there is a limited number of atoms in small objects (e.g. only 50 atoms in 1 nm^3 of silicon), a first requirement to produce images is the optimization of the **signal level** (number of detected particles) through several parameters: break large molecular ions into more numerous atomic ions, maximize the ionization yield of sputtered atoms, the collection and transfer of the secondary ions, the transmission of the mass analyzer, the detection efficiency and the reduction of noise and background signals (which requires fast analysis under ultra-high vacuum).

2- As SIMS is destructive the analysis of small objects requires a **parallel detection** of as many isotopes or elements as possible. Indeed, for very small objects, with a monocollection SIMS one could record the distribution image of a first element and the small object could have been already completely sputtered: too late for other elements or isotopes!

3- As SIMS is destructive the analysis of small objects requires **high mass resolution** (without losing on the useful yield !) because there won't be more material available from small objects to re-run the analysis and elucidate the numerous potential mass interferences present at each single mass (e.g. $^{12}C^{15}N$, $^{13}C^{14}N$, ^{27}Al, $^{12}C^{14}NH$,… all at 27 amu).

Instrumental choices are derived from these constraints:

- The **use of reactive primary ions**: cesium for electronegative secondary elements and oxygen for electropositive elements are the two first choices. Cesium is heavy so the collision cascade is very shallow, resulting in a good depth resolution. Cs concentration is high at the surface, increasing the ionization. Oxygen ions are excellent for enhancing the ionization yield of electropositive elements. However, O is light, so the sputtering yield is lower (lower speed of analysis) and the mixing thickness is larger degrading the depth resolution compared to Cs. One solution can be to use small molecular oxygen ions: O_2 or O_3. Iodine or bromine could be other choices providing ion sources presented high enough brightness.

  Forming a small primary beam requires a bright ion source (in $A/sr*cm^2$), a transport optics and an objective lens with low aberrations, not to blur the transported image of the source. Theoretically, the brightness of the source is the maximum value obtainable on the object using a perfect transport and focusing optics. But brightness is not the only goal: e.g. liquid metal ion sources (Ga, Au, Bi…) are very bright and used in all focused ion beam (FIB) instruments to cut materials. But their induced ionization yields are very low: most atoms are sputtered as neutral, which are lost for SIMS analysis. For SIMS we need high brightness of *reactive* ions. For the NanoSIMS, a new cesium source was developed. For oxygen, a duoplasmatron oxygen ion source was first used, replaced now by a brighter RF-plasma ion source.

- The obtention of intense small spots relies on low aberrations coming from the objective lens: the shorter the working distance, the better the lens properties. Hence the **shortest possible working distance** is necessary. This brings a new constraint: one needs also to position an **extraction optics as close to the sample as possible,** in order to collect the few emitted secondary ions as early and as strongly as possible. This problem is solved on the NanoSIMS by the adoption and optimization of a normal incidence, single co-axial objective and extraction lens.

- In order to maximize secondary ion collection, an early and strong electrostatic extraction field is required. We then need a transfer optics and a mass analyzer optimized for high energy. A quadrupole, an ion trap or an orthogonal time-of-flight are excluded as they require (presently) low

ion energy. Slowing down the ions would defocus the secondary ion beam and reduce the transmission (=sensitivity). We are thus left with magnetic sectors and time-of-flight mass analyzers with pulsed primary beam. As far as magnetic sector transmission is concerned the optimization of transmission from small area implies to give up on stigmatic analyzer (SIMS microscope) for a pure *microprobe* analyzer: the NanoSIMS is thus based on a highly focused primary beam, scanning pixel by pixel over the sample. The secondary ion extraction/collection solid angle is maximized, over a reduced area. A *dynamic emittance matching* optics scans this small collection area in synchronism with the scanning of the primary beam, in order to form homogeneous acceptance from a larger area. A transfer optics then *shapes* the secondary ion beam into the entrance slit in order to minimize aberrations and optimize transmission of the following high mass resolution mass spectrometer.

- A small ion probe size leads to small primary current (Ip prop. to $diam^2$) thus to a small signal: acquisition will be slow, leading to associated problems (recontamination, mandatory single ion detection with low background noise, high mass resolution). Also, recording useful isotopic ratios means at the minimum having a good enough statistics (counts) on the minor isotope(s) within a reasonable time. These two points require maximizing the duty cycle (time spent for measurement/ time of analysis) and maximizing the useful yield. This in turn excludes TOF-SIMS with pulsed beam (most of the time is spent waiting for ions to reach the detector, not sputtering) and dual beam geometry (a good portion of the rare secondary ions from tiny objects are not collected during the reactive ion sputtering phase). We are now logically left with a **continuous (DC), magnetic sector design**.

- As SIMS is a destructive technique, we need a **parallel detection** to optimize the collection of all the sputtered material.

We thus come to a **high mass resolution, high transmission, continuous, magnetic sector SIMS, with multicollection.** A Mattauch-Herzog-like (double focusing) geometry was adopted on the NanoSIMS, well-suited for a large mass range rather than a pure isotopic multicollection with smaller mass range.

For a small beam size, associated with low current and low sputtering speed, especially for light element analysis, an Ultra High Vacuum technology is necessary to reduce contamination from the residual gas of the analysis chamber.

The NanoSIMS instrument is now defined in its main lines: bright reactive primary ion sources (Cs and O), near lens, coaxial geometry, high transmission, high mass resolution, magnetic sector with multi-elemental multicollection, high vacuum.
See below a simplified synoptic in Figure 2: NanoSIMS 50L simplified schematics (from: M. Steinhauser et al., Nature, 2012) and a picture of the NanoSIMS 50L instrument in Figure 3: The CAMECA NanoSIMS 50L

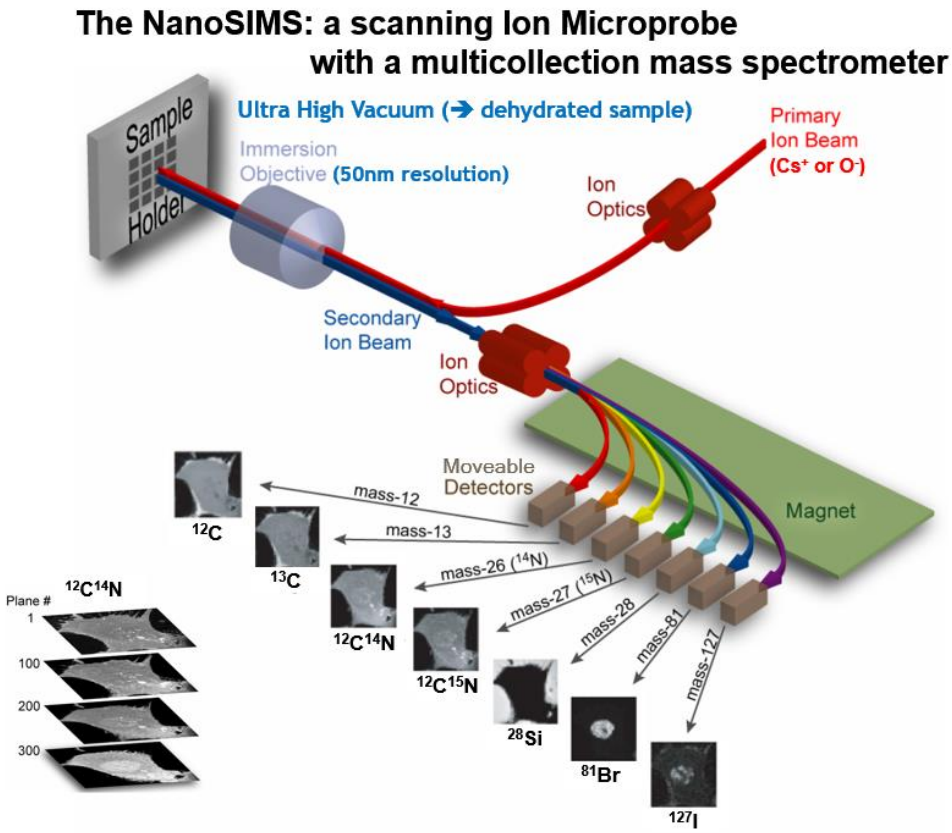


Figure 2: NanoSIMS 50L simplified schematics (from: M. Steinhauser et al., Nature, 2012)

Figure 3: The CAMECA NanoSIMS 50L

Chapter 2 of this user's guide describes in more details the optical solutions adopted to make the whole concept work with high performance.

The chosen instrument design induces certain physical constraints:

- Opposite polarity between primary and secondary ions:
  - This precludes the use of O_2^+ primary ions to detect positive secondary ions. O^- (or O_2^-or O_3^-) ions are used instead, despite a lower source brightness compared to O_2^+.
  - This also precludes the MCs^+ semi-quantitative SIMS mode.

    - It renders charge compensation with electrons impossible in O^-/positive secondary mode. Sample coating and use of O_2^-/O_3^- primary ions are usual solutions.

- Oxygen (or ozone) flooding used in other SIMS designs to further enhance the positive ionization yield has not being implemented (yet) by fear of electrical discharge in the high field lens.

- The best lateral resolution is obtained at 16keV leading to depth resolution of ~10-20nm. This depth resolution can be improved by reducing the impact energy down to 4keV on the NS50L but it is still far from sub-nm depth resolution obtained on other CAMECA SIMS using ultra low impact energy (down to 100eV), but at larger spot sizes.

The NanoSIMS has been a joint development of nearly 10 years between three French institutions: the University of Paris Sud (UPS) with mainly Prof. Georges Slodzian, original designer, the ONERA (space and industrial research) with mainly research engineers Bernard Daigne and François Girard, and CAMECA with mainly François Hillion, physicist.

Two early papers can be a useful introduction to the design of the NanoSIMS:

Hillion F., Daigne B., Girard F. and Slodzian G. (1993). A new high performance SIMS instrument: The Cameca “Nanosims 50”. In: Benninghoven A., Nihei Y., Shimizu R. and Werner H.W. (eds), Secondary Ion Mass Spectrometry SIMS IX. Wiley (Chichester), 254–257.

Slodzian G., Daigne B., Girard F. and Hillion F. (1993). Ion optics for a high resolution scanning ion microscope and spectrometer: Transmission evaluations. In: Benninghoven A., Nihei Y., Shimizu R. and Werner H.W. (eds), Secondary Ion Mass Spectrometry SIMS IX. Wiley (Chichester), 294–297.

The launch of a first commercial product, NS50, has been allowed in 1999 by two first orders based on prototype evaluations in cosmochemistry by: Prof. Robert M. Walker and Prof. Ernst Zinner from Washington University in Saint Louis, MI, USA and Dr. Peter Hoppe from MPI Mainz, Germany.

The major evolutions have been since then:
- The development of a larger multicollection, NS50L, with seven detectors instead of five, a larger mass range and smaller minimum mass interval, and motorized switch between EM and FC detectors.
- Automation of apertures, slits, hexapole, and development of software functions (e.g. beam alignment, EM drift correction…)
- Replacement of the duoplasmatron by a brighter RF-plasma oxygen ion source.

The CAMECA NanoSIMS is constituted of three main parts: the physical part, the electronical part and the computing system detailed in the following chapters.

2 NS50L Optics & general hardware

2.1 Physical and Optical description

Note: A minimal physics background (electrical or magnetic fields, charged particles, optics…) would be helpful to understand this part of the manual and get the best results using a NanoSIMS.

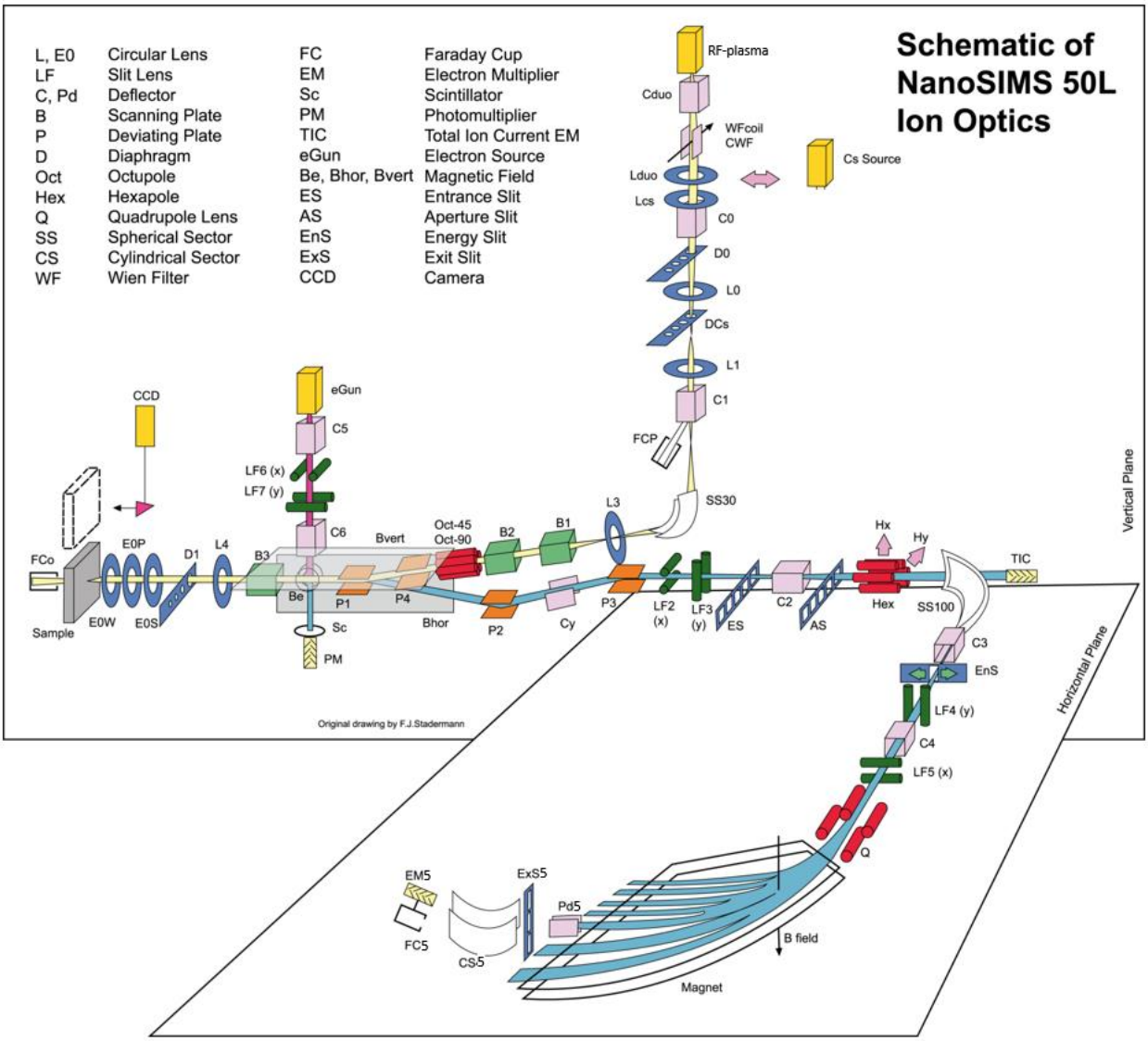


Figure 4: Overall NS50L optical schematic

The above optical schematic will be detailed progressively in the following chapters.

2.1.1 The primary column

The primary ion optics is made of four different sections: (1) the Cs/RF-Plasma switch, (2) the lower primary column section, (3) the central column and finally (4) the coaxial lens which allows to focus the primary beam on the sample.

The first two sections (Figure 5) are exclusively used by the primary ion beam while the last two are shared with the secondary ion beam (Figure 6). Either Cs^+ or O^- can be used as primary ions, depending on the source used and the polarity configuration required for a given analysis. O_2^-, O_3^- and O_2^+ can also be used but do not change anything to the principle and explanations. Indeed, if the primary beam is positive, the secondary beam will be negative while if the primary beam is negative, the secondary ion beam will be

positive. The whole primary column is used to accelerate the primary ions (Cs+ or O- depending on the used source) to a specific energy to strike the surface of the sample.

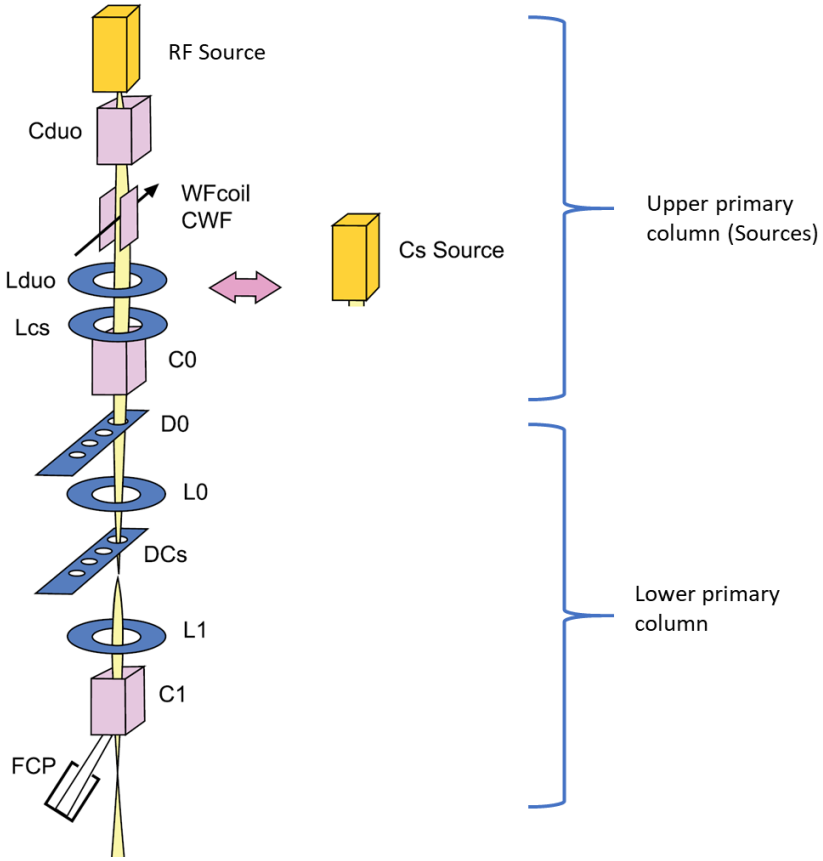


Figure 5: Illustration of the Cs/RF-Plasma switch and the lower primary column

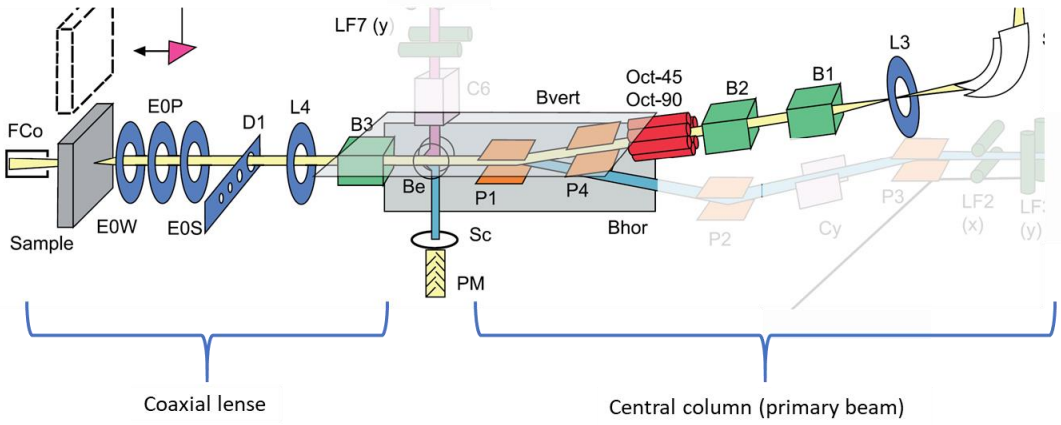


Figure 6: illustration of the central column and the coaxial lens

2.1.1.1 Upper part: the Cs/RF-Plasma source switch

2.1.1.1.1 Overview

The Cs/RF-Plasma source switch is made of two ion sources: the CAMECA Cs+ microbeam ion source and the RF-Plasma O- gas source (Figure 8). In addition, when the RF-Plasma source is used, a Wien Filter is available.

The source chamber is isolated from the rest of the primary ion column by a gate valve and is equipped with a turbo pump. This allows maintenance of the sources without venting the whole primary column. The

source interchange mechanism also allows switching between ion sources without venting: a trolley supporting the Cesium source and the Lduo lens can be moved under vacuum between two different positions, using a manual goniometer.
As shown in
Figure 7 below, in Position 1 (around 1.0 mm on the goniometer), the Cesium source is set on the axis of the primary column and Lduo is pushed away. In position 2 (around 3.4 mm on the goniometer) the Lduo lens is set on the axis of the primary column allowing oxygen ion focusing and the Cs source is pulled back.

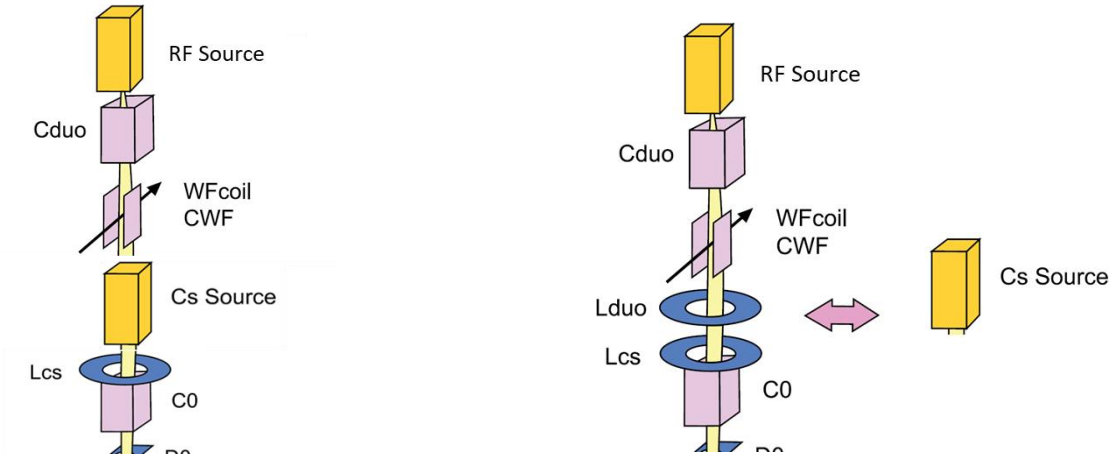


Figure 7: Cs+/RF-Plasma switch mechanism and upper part of the primary column. Left: Cs+ mode; Right: oxygen mode.

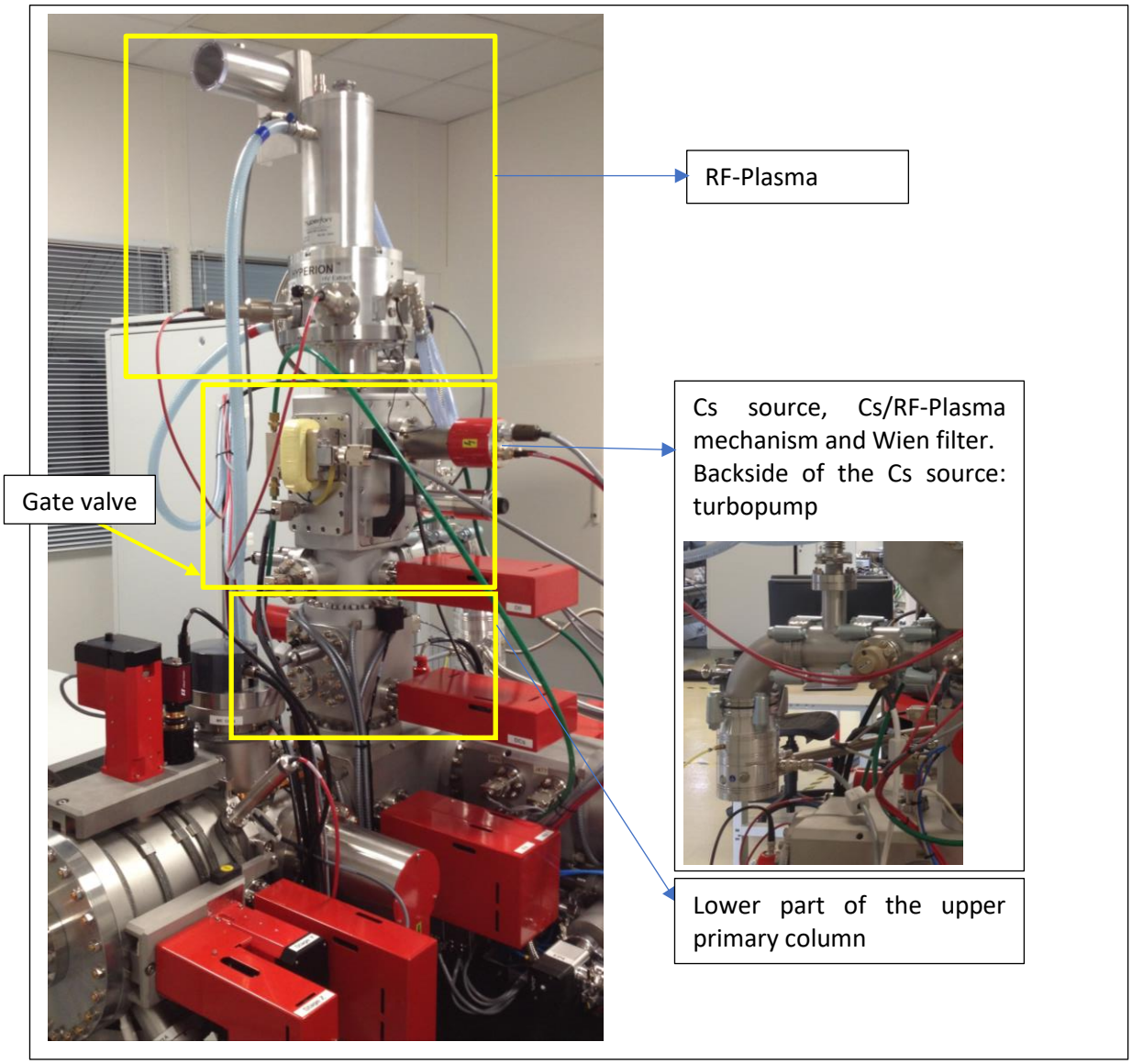
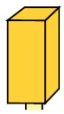
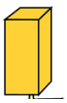
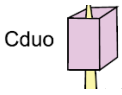


Figure 8: Main parts of the upper primary column

2.1.1.1.2 Component description

Device	Label	Description and functionality
 Cs Source	Cs Source	The Cs^+ source is used to make analyses with cesium primary ions. This source emits Cs^+ after having heated an ionizer and a reservoir of Cs_2CO_3. Cs^+ ions are extracted and accelerated by applying a high voltage.
 RF source	RF Plasma	The RF-Plasma source is used to make analyses with O^- primary ions. A RF antenna is used to create an oxygen plasma. O^- ions are extracted and accelerated by applying a high voltage.
Cduo corrector  Cduo	Cduo	A 4-plate deflector is used to center the RF-Plasma ion beam before it enters the Wien filter.

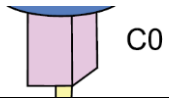
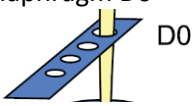
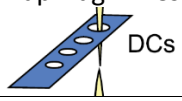
Wien Filter 	CWF WFcoil	The Wien Filter is a mass filter consisting of 2 electrostatic deviators (CWF) and a magnetic deviator (WF Coil).
Lens Lduo 	Lduo	Lens used to focus the RF-Plasma ion beam on D0 at the exit of the Wien filter.
Lens LCs 	LCs	Lens to focus the Cs ion beam at the exit of Cs source. Usually not used anymore but still present in the column.
Corrector C0 	C0	A 4 plates deflector used to center the primary ion beam at the exit of the Cs/duo switch.
Diaphragm D0 	D0	D0 is a diaphragm limiting the angular aperture of the Cs ion beam or acts as a mass selection diaphragm for the Wien filter. 5 different diameters are available: D0-1: 200µm, D0-2: 150µm, D0-3: 100µm, D0-4: 100µm, D0-5: 50µm.
Diaphragm DCs 		Diaphragm previously used in association with the Cs beam. Not used anymore.

Figure 9: Table of the elements of the upper part primary column

2.1.1.1.3 The CAMECA Microbeam primary Cesium ion Source

For more detail refer to: G. Slodzian, B. Daigne, F. Girard, F. Boust, F. Hillion: A thermal ionization source for a Cs+ ion probe. Proceedings of the 8th SIMS Conference, Amsterdam sept. 91.
For practical operation refer to chapters 5.4 and 9.1.2 and 9.1.3.

2.1.1.1.3.1 Overview

A cesium vapor is generated from a cesium carbonate (Cs_2CO_3) pellet contained in a reservoir raised to a temperature of 400°C. This temperature is required to release the cesium vapor.
The cesium vapor encounters a tungsten plate enclosed in the ionizer head, heated to 1100°C. The hot tungsten plate ionizes the Cs vapor into positive ions (Cs^+). The reservoir and ionizer are set to a voltage adjustable between +2 and +8 kV and heated independently by electron bombardment. Those electrons are emitted by applying a current to two annular filaments set at 0 Volt.
The extraction electrode, placed in front of the ionizer at ground potential, generates an electric field to extract and accelerate the Cs^+ ions.

A constant emission of cesium ions is obtained by regulating the electron current flowing between the ionizer and its associated filament (I_{ION}) and between the reservoir and its associated filament (I_{res}).
The total current delivered by the high voltage power supply (I_{TOTAL}) is the sum of the ionizer, reservoir electron currents and a leak current (Figure 10). The leak current is mainly due to secondary electrons produced by low density plasma surrounding the source and secondary electrons re-emitted by the extractor toward the source (especially when there is too much cesium deposited on the surfaces, leading to the source stop, venting and extractor cleaning) .

$$I_{TOTAL} = I_{RES} + I_{ION} + I_{Leakage}$$

Figure 10: Total current expression

Leakage, negligible during normal operating conditions, may be important during a runaway (first usage of a source); however, a security is designed to limit the over-heating of the source.

Figure 11 shows the layout of the various parts of the source.

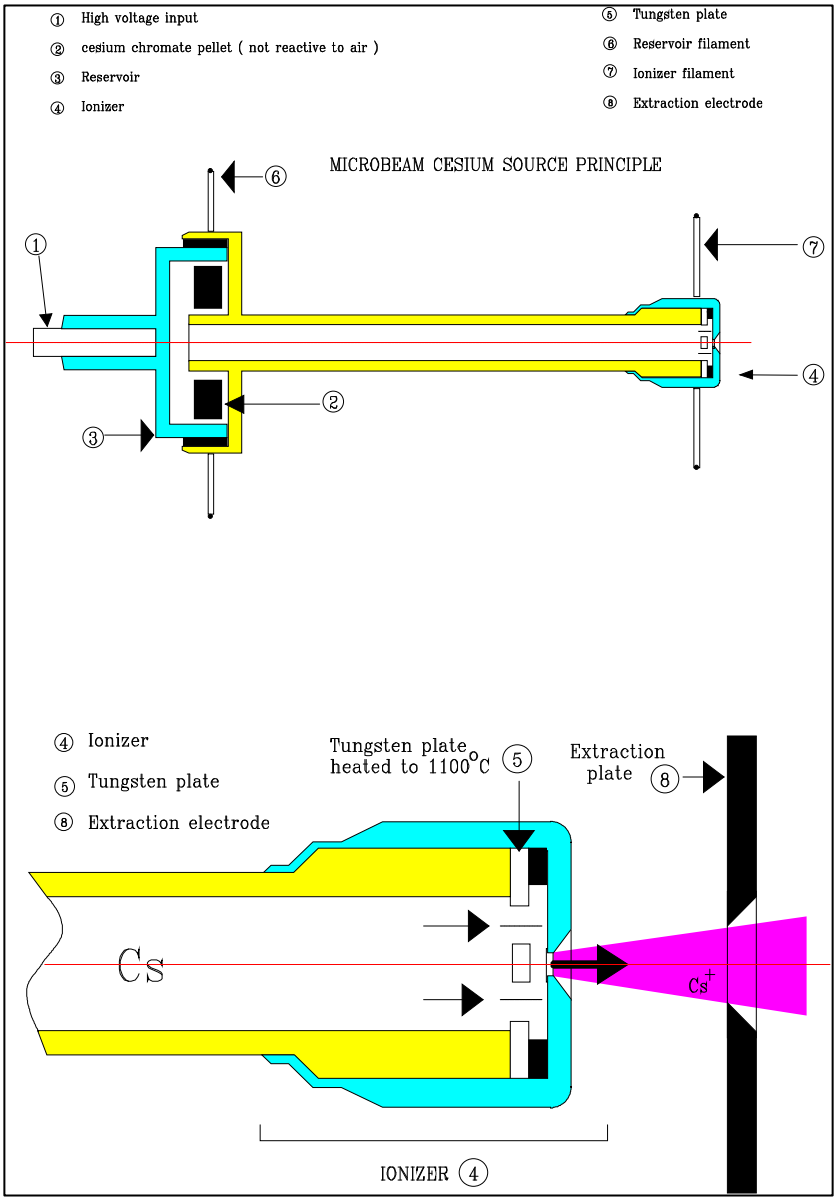


Figure 11: The Cs Microbeam source

2.1.1.1.3.2 Tuning and aging issue

The carbonate source (Cs_2CO_3) is generally employed with the following values (Figure 12):

Type	Ionizer at 8kV	Reservoir at 8kV
Carbonate (Cs_2CO_3)	1.8 mA	0.35 mA

Figure 12: Usual ionizer and reservoir current values

The Cs^+ current emitted by the ionizer varies dramatically with the reservoir current, and slightly with the ionizer current. The following data (Figure 13) have been recorded with a Cs carbonate source:

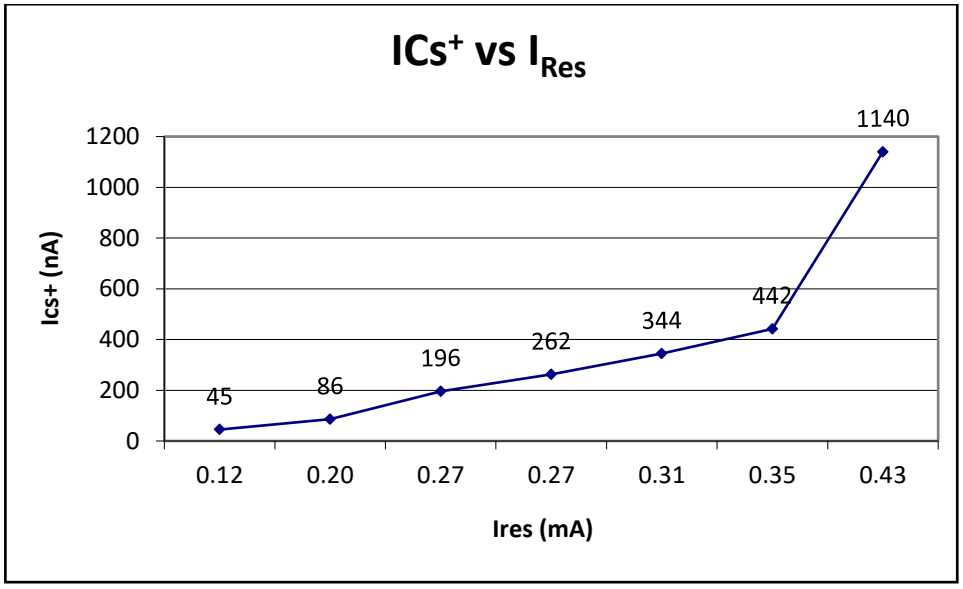


Figure 13: Evolution of the Cs+ beam current with the reservoir current

After using the Cs^+ source for a long period of time (of the order of hundred hours), the beam current in FCp will start decreasing. This must be compensated by increasing the reservoir current (usually by 0.05 to 0.1 mA at a time, up to 0.6 mA max).
It is standard and good practice to check and note down regularly (e.g. every day at the beginning of a session) the FCp primary beam current. This allows the monitoring of the aging of the cesium source.

Using and adjusting the source are detailed in chapter 9.1.2.1.
Servicing the cesium source is detailed in chapter 10.7.

2.1.1.1.4 The RF-Plasma primary ion source

The NanoSIMS has been historically developed around a single, fixed cesium source. For its commercialization a Cameca duoplasmatron ion source was added to work in O^- mode with a Wien mass filter. The cesium source was made moveable. And a third step has been the replacement of the duoplasmatron by the Hyperion RF-plasma ion source from Oregon Physics. It offers two main advantages over the duoplasmatron:

- A higher brightness in O^- mode leading to smaller final spot size and higher beam intensity. Typically, with O^-, the duoplasmatron has a source size of ~300µm, a brightness of 10-20A cm sr^{-1} and dE < 15eV. The RF-plasma will have a source size of ~70-80µm, a brightness of 100A cm sr^{-1} and dE < 5eV.
- A largely reduced need for cleaning as the RF-plasma source does not sputter internally. The maintenance can be annual whereas a duoplasmatron, especially used in negative primary ions and in vertical mode, had to be dismounted every one-two weeks for cleaning the sputtered material and particles.

In addition, the source is very stable over long periods (few % over several hours), ultimately limited by the temperature regulation of the laboratory (causing variations of gas flow through the leak valve).
The drawbacks are a lower maximum beam current, which is not a problem on the NanoSIMS dealing with small beams and currents anyway, and a higher cost compared to a duoplasmatron.

For practical operation refer to chapters 5.4 , 9.1.2 and 9.1.3.

2.1.1.1.4.1 Photos of RF-plasma Oxygen source and cabinet

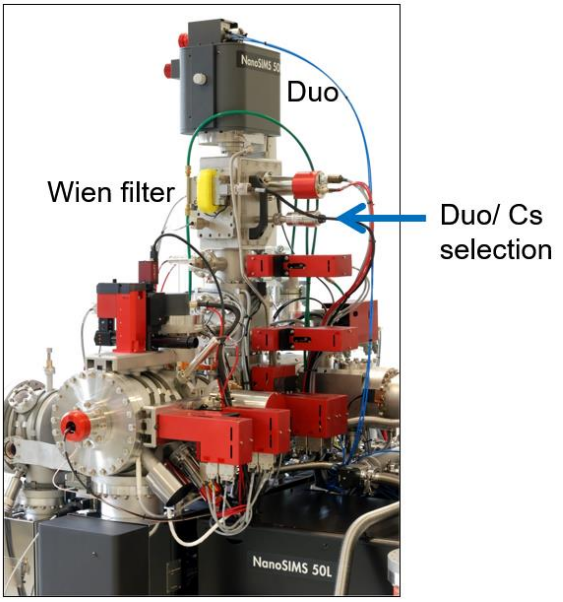


Figure 14: NanoSIMS fitted with duoplasmatron O- source

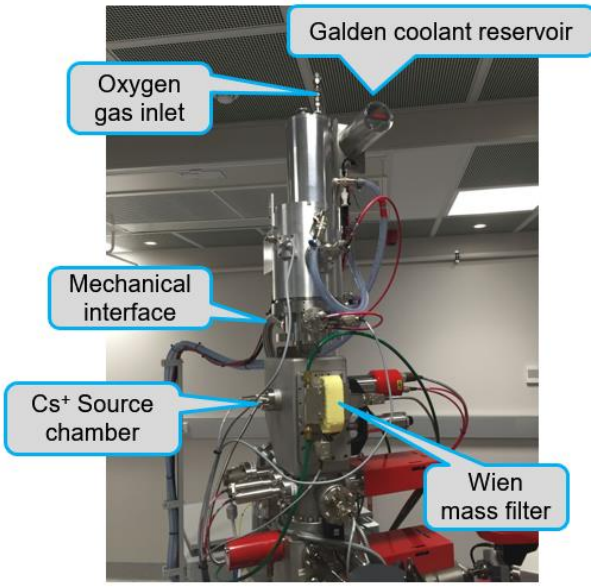
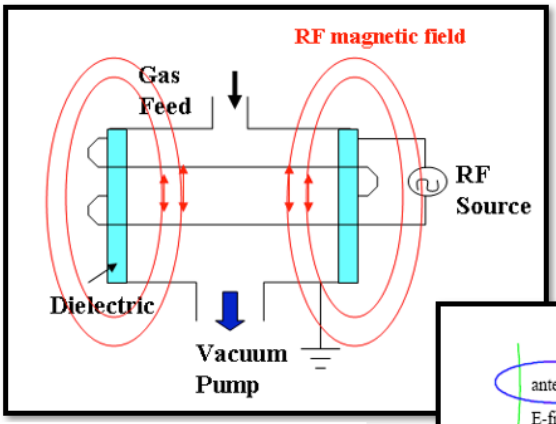


Figure 15:NanoSIMS fitted with a RF-plasma ion source

Figure 16: RF-plasma electronics and cooler rack

2.1.1.1.4.2 RF-plasma ion source principle

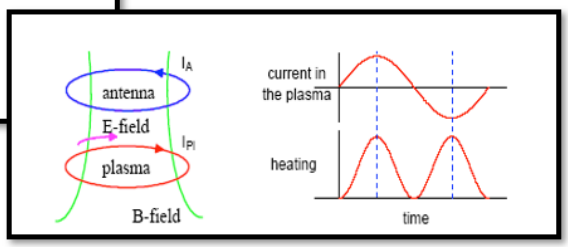
The principle is to create a cold plasma with inductive power coupling:



Time varying current in an external coil creates a time varying B field, which induces an azimuthal E field.

$$\nabla \times \underline{E} = -\partial \underline{B}/\partial t$$

Induction E field accelerates electrons to ionize resident gas.



the electron plasma frequency

$$\omega_{pe} \triangleq \left(\frac{n_0 e^2}{\varepsilon_0 m_e} \right)^{1/2}$$ ~9GHz

the ion plasma frequency

$$\omega_{pi} \triangleq \left(\frac{n_0 e^2}{\varepsilon_0 m_i} \right)^{1/2}$$ ~18MHz

(when Xe plasma density, $n_o = 1x10^{12} cm^{-3}$)

Above slide from Oregon Physics

2.1.1.1.4.3 Plasma properties (inert gases)

Mean thermal electron energy, $(kT_e/2) \sim 3$ eV

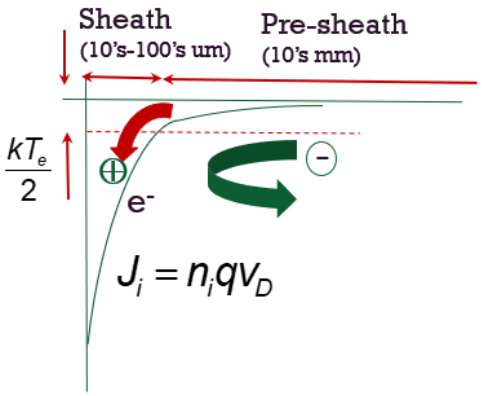
Mean thermal ion energy, $(kT_i/2) < 0.1$eV

$\therefore \; v_e >> v_i$

and the walls charge up, until $\Gamma_i = \Gamma_e$

Creates a plasma potential, V_p

$$V_p = \frac{-kT_e}{2} \ln(m_i / m_e)$$



Above slide from Oregon Physics

2.1.1.1.4.4 Extracting negative ions

The NanoSIMS is used mostly with O^- primary ions. Note though that it is possible to use O_2^- and O_3^- as well (to optimize depth resolution or surface oxidation at the cost of a lower beam current) or O_2^+ (superior mode in term of brightness but the NanoSIMS works with opposite polarity between primaries and secondaries and not many negative ions have high yield under oxygen (some oxides can be useful in some cases e.g. BO_2^-,…).

For **negative ion** generation, the key point is to have two plasma zones isolated by a magnetic field region. The upper plasma region is rich with fast electrons and positive ions (and less negative ions). The magnetic region protects the lower plasma region against too many fast electrons that can neutralize the produced ions.

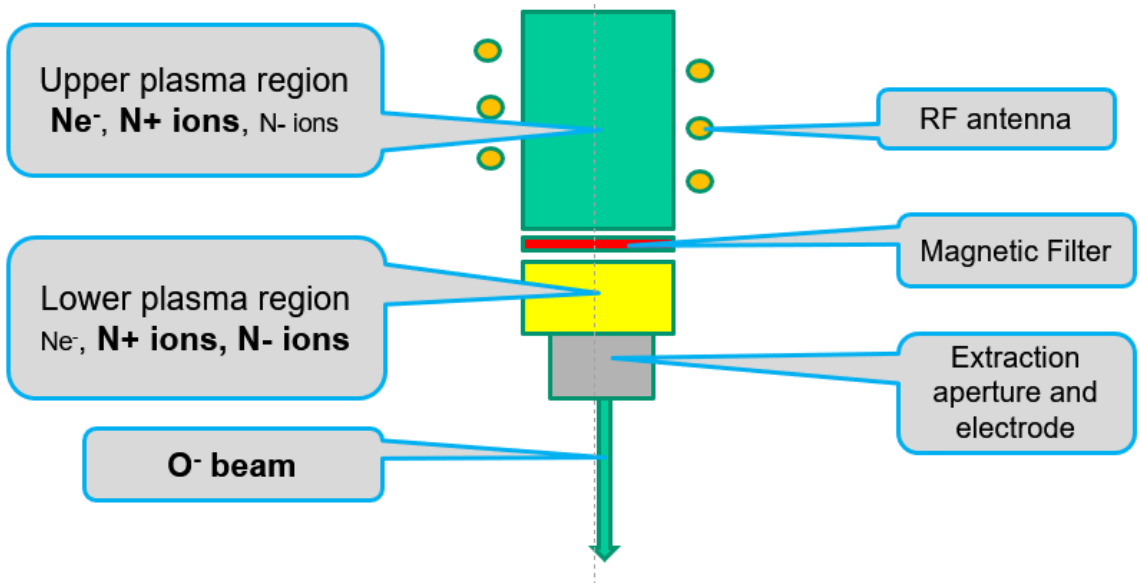


Figure 17: RF plasma schematic with its two plasma regions

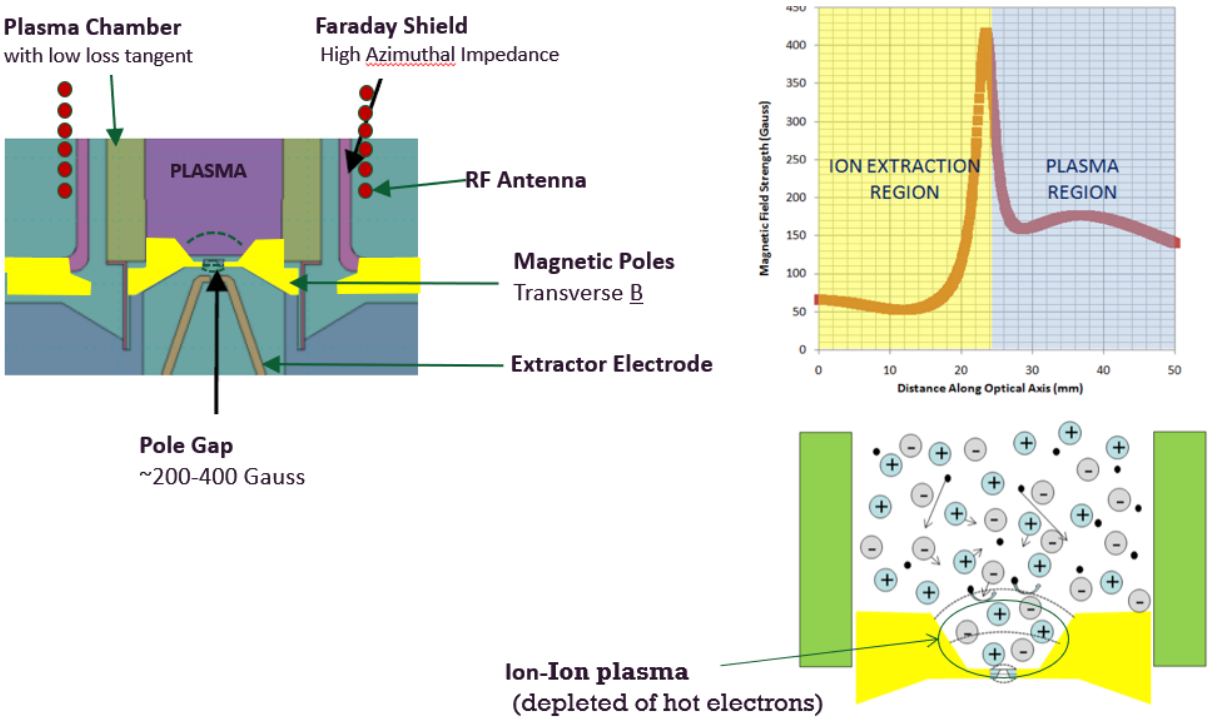


Figure 18: plasma region principle

Above slide from Oregon Physics

2.1.1.1.5 The Wien Filter

2.1.1.1.5.1 Physical principle

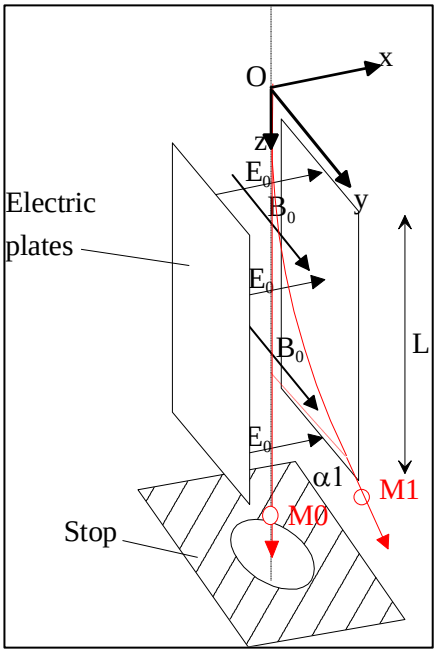


Figure 19: Illustration of the Wien filter

A *Wien Filter* is basically formed by superimposing a homogeneous electrostatic field and a magnetic field. As shown in the figure above (Figure 19), the magnetic field B is parallel to Oy axis, while the electric field $\vec{E}$ is parallel to Ox axis. Thus, a charged particle moving along Oz axis is submitted to two forces parallel to the Ox axis.
The charged particle is not deflected if the electrostatic force and the Lorentz force balance along the z axis.

$$\frac{e\,V_0}{M_0} = 4.9\ 10^{-3}\left(\frac{E_0}{B_0}\right)^2 \qquad \text{[eV/amu]}$$

Where E_0 and B_0 are respectively expressed in V/cm and gauss. M_0, eV_0 and e are respectively the mass, the energy and the electric charge of the particle. M_0 is expressed in amu.
As eV_0/M_0 is proportional to the particle velocity, it can be said that the Wien filter is a velocity filter. As it can be assumed that the particle energy eV_0 is constant, a Wien filter is in fact a mass filter. The main advantage of the Wien filter is to be straight-line.

For achieving a mass filter, a diaphragm must be included in the system, downward the combined electric and magnetic fields, so that the selected M_0 trajectories can pass through the diaphragm while the deflected mass trajectories are stopped.
When using a Wien filter in an ion optical system where the charged ions are accelerated at V_0, the ratio E_0/B_0 is determined by the mass M_0 which must be kept on axis. E_0 and B_0 intensities are determined by the closest mass M_1 minimum deflection α_1 required to be rejected by the diaphragm defined as follow:

$$\alpha_1 = \frac{B_0(gauss)\,L(cm)\,e^{1/2}}{204\,V_0^{1/2}}\left(\frac{1}{M_1^{1/2}} - \frac{1}{M_0^{1/2}}\right)$$

Where L is the combined field length.

The electric field $\vec{E}$ is controlled by the voltage V applied to the electric plates, and the magnetic field B is controlled by a current I supplied by the coils of a magnetic circuit.

The CAMECA NanoSIMS 50L Wien filter consists of a pair of 76 mm height plates, adjusted by a voltage supply ranging within ±250 Volts and a magnetic circuit. This circuit both inside and outside the vacuum chamber is excited by a coil supplied by a 7A source. Both the plate voltage and the coil current are controlled from the tuning user interface.

The Cs+ primary beam is mainly composed of Cs+ ions and does not require a mass purification for CAMECA NanoSIMS 50L application. However, the Wien filter is used as a mass filter to eliminate various ions which can be generated with the RF-Plasma source, even when using purified oxygen.
In the negative polarity, there are three major peaks as shown on Figure 20: $^{16}O^-$, $^{16}O_2^-$, $^{16}O_3^-$. This graph is obtained by scanning with a magnet (and so, separating) all the masses emitted from the source. The signal is collected on a Faraday Cup detector.

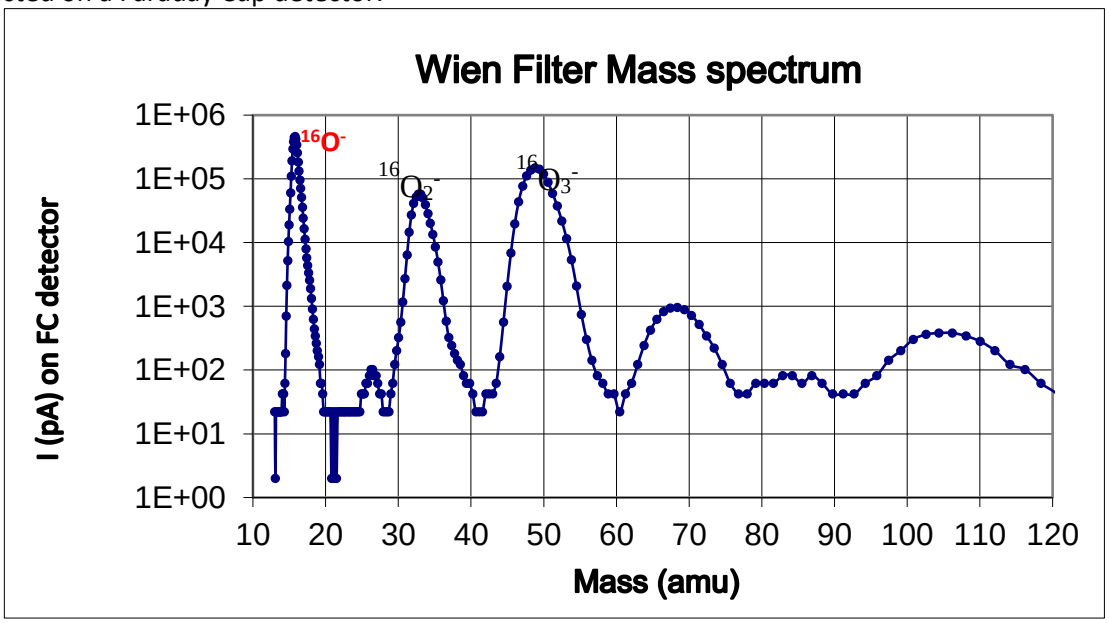


Figure 20: Wien filter mass spectrum in logarithmic scale

Figure 21 displays the experimental relationship between CWF and WF coil for $^{16}O^-$ ions. It can be checked that the plate voltage is proportional to the magnetic field.

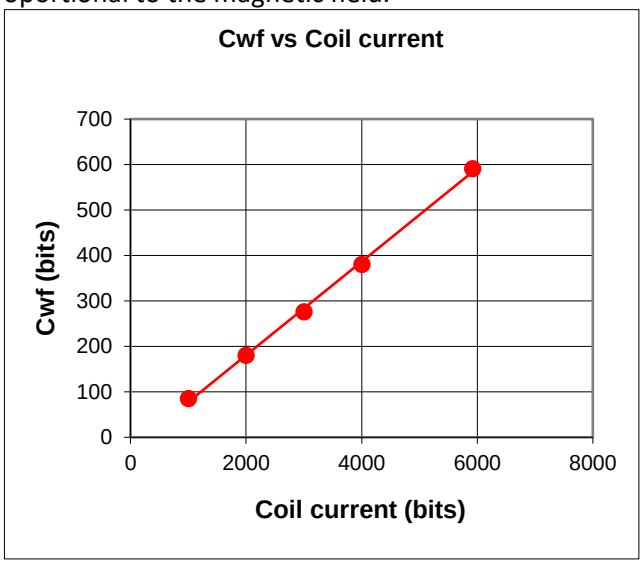


Figure 21: Experimental relationship between C_{wf} and the coil current

This graph is obtained by applying a Coil current and looking for the Cwf value allowing the maximum primary current. When increasing both the magnetic field and the plate voltage, it can be useful to re-adjust D0. Most of the time, D0-2 is used.

The usual value for the coil current is 2000 bits and the Cwf value is around 200.

In most applications O^- primary ions are used in the NanoSIMS because of the higher source brightness. In geology, when ultimate lateral resolution is not mandatory (e.g. for dating in geochronology where a "large" volume of material must be sputtered anyway in order to get enough statistics on the minor lead isotopes) O_2^- or O_3^- primary ions can be interesting. Sputtering yield is higher, charging is lower and for some elements the ionization can be also higher. In materials, depth resolution can be improved using O_2^- , O_3^- or O_2^+ and/or through the use of a lower impact energy.

2.1.1.2 Lower primary column

2.1.1.2.1 Overview

This section of the NS50L is either used to increase the primary ion beam current or to decrease the size of (i.e. demagnify) the cross-over of the source. In addition, a Faraday cup called "FCp" allows to measure the beam current entering in the central column.

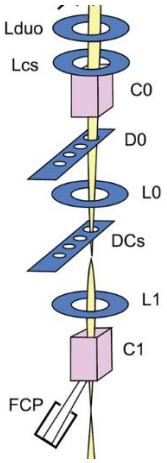
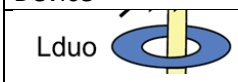
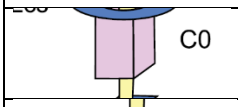
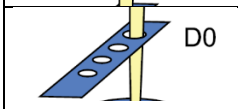
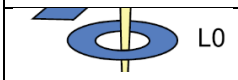
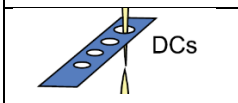
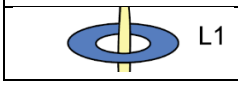


Figure 22: Illustration of the lower part of the primary column

2.1.1.2.2 Description of the optical components

Device	Label	Description and functionality
	Lduo	Lens used to focus the O- beam on the D0 plane. Usually set at a constant value.
	LCs	Lens used to adjust the demagnification of the source image for the Cs+ source only. Usually not used.
	C0X, C0Y	A 4 plates deflector used to center the primary ion through L0
	D0	Aperture stop which limits the angular aperture of the primary ion beam and controls the primary beam current and the spot size. Only used in oxygen mode.
	L0	Lens used to adjust the demagnification of the source image and adjust the probe current for both sources.
	DCs	Aperture stop which limits the angular aperture of the primary ion beam and controls the primary beam current and the spot size. Usually not used.
	L1	Lens used to adjust the demagnification of the source image and adjust the probe current for both sources.

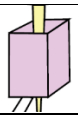
	C1x C1y	A 4 plates deflector used to center the primary ion beam into SS30 (see below) or the FCp, depending whether the beam is ON or OFF.
Secondary electrons Suppressor (not represented)	SE FC	Tube at –30 volts located at the entrance of FCp, used to prevent secondary electrons from escaping the primary Faraday Cup FCp.
	FCp	The Faraday Cup used to measure the primary ion beam current at the exit of the lower primary column. When FCp is used, the primary ion beam (OFF state) is focused with L1 and deflected with C1 in the entrance of the FCp, thanks to pre-set values.

Figure 23: Table of the elements of the lower part of the primary column

Note that certain version of the NS50L also show a lens LCs and a diaphragm DCs on the primary column. These were made to try and reduce the primary beam size in Cs^+. However the use of additional lens and diaphragm make the tuning really difficult and while DCs (DCs : 200 um, 200 um, 150 um, 100 um et 100 um.) and LCs are still present, they are generally not used.

2.1.1.2.3 Effect of L0 and L1 on the primary beam

L1 can be used to adjust the degree of demagnification of the source image. L1 produces a real reduced image which will be seen by the subsequent sections of the primary column as a real object. This reduced image is located between L1 and SS30.

Figure 24 shows the variation of the Gaussian probe size versus L1, with a Cs^+ source set at 8kV. This theoretical graph has been computed with a 40 microns source size at the exit of the Cs source.

While reducing the probe size, the probe current will decrease. Figure 25 shows the theoretical and experimental variations of the probe current versus L1 for the same D1 diameter.

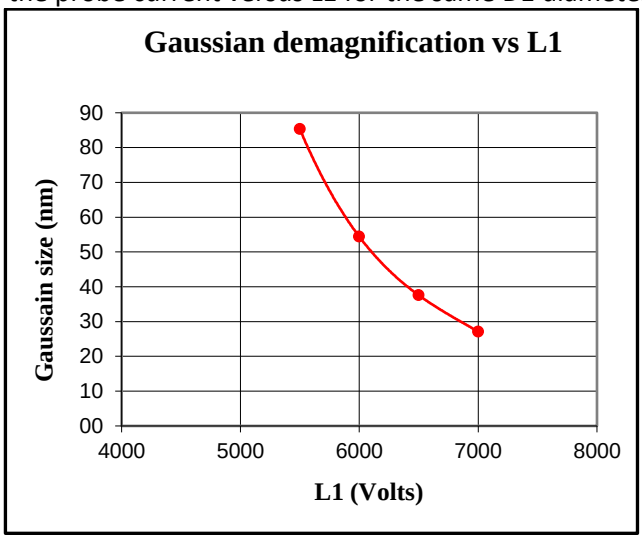


Figure 24: Gaussian de-magnification vs L1

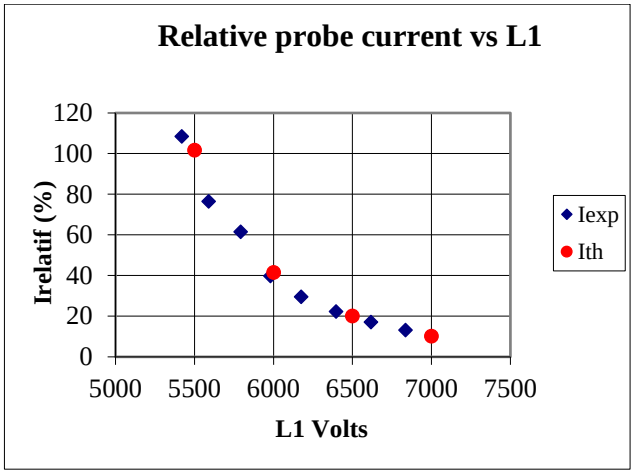


Figure 25: Relative probe current vs L1

L0 or L0 + L1 are also commonly used to increase the probe current. Of course, while increasing the probe current, the probe size increases.
Figure 26 shows the variation of the probe current vs L0. L1 is kept at 0 volt and the source species is Cs+. Probe current limitations are not only due to D1 but also to the small differential pumping tube located between the source chamber and the central column. A comparison has been made between theoretical and experimental values showing the effect of the pumping tube which limits the current at high L0 values (Figure 26).

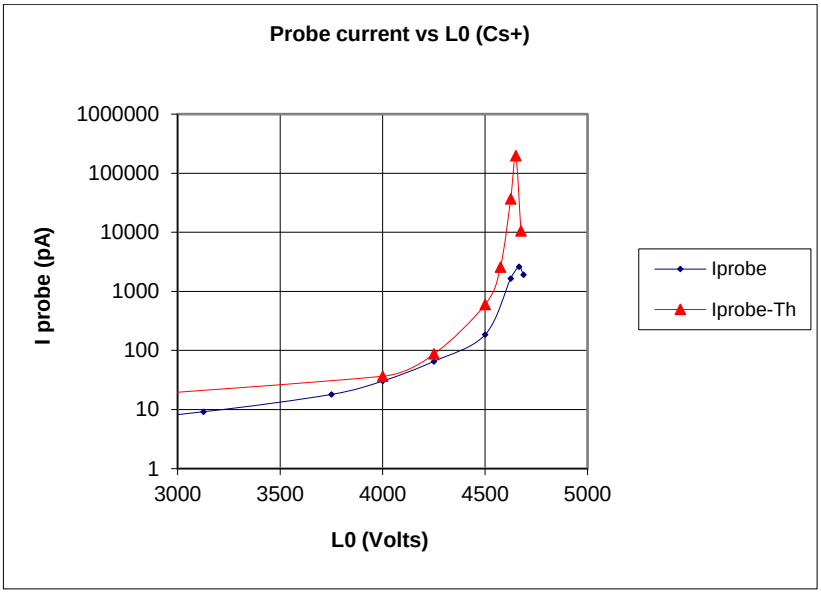


Figure 26: Probe current vs L0 (Cs+)

One can find different L0-L1 combinations giving a maximum for the probe current as shown on Figure 27 and Figure 28 while using L0 and L1 for Cs+.

As an example, 29 nA of Cs+ were measured on the sample for the following settings: FCp = 50 nA, D1-1 = 750 microns, L0 = 4250 V, L1 = 3100 V

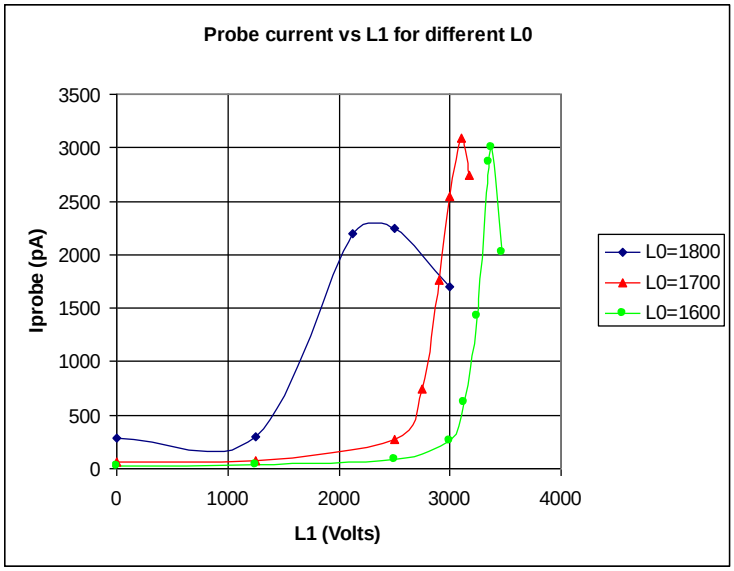


Figure 27: probe current vs. L1 for different L0. L0 values are here given in bit units for a NS50 instrument with 12 bit cards. For NS50L instruments L0 is in 16 bits. Multiply the L0 values by 16 to obtain the NS50L equivalents.

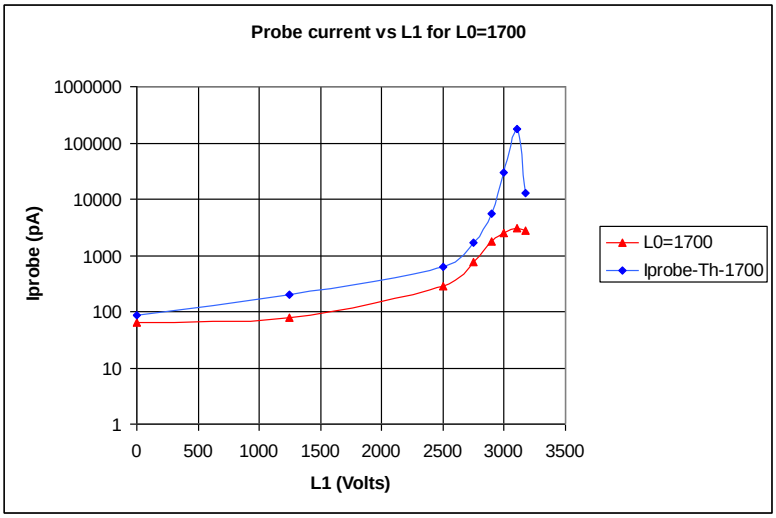


Figure 28: probe current vs L1 for L0=1700

Note that in these extreme conditions of very high primary beam current, the probe size is “huge”, and aberrations dominate the probe shape, leading to very long tails.

2.1.1.3 Central column

2.1.1.3.1 Overview

This section of the primary column (Figure 29) is used to send the primary ion beam on the axis of the coaxial column and to raster the beam on the sample surface. An octupole is available to correct the astigmatism. A part of this section, the P1 deflector plates, is common for the primary and the secondary ion beam.

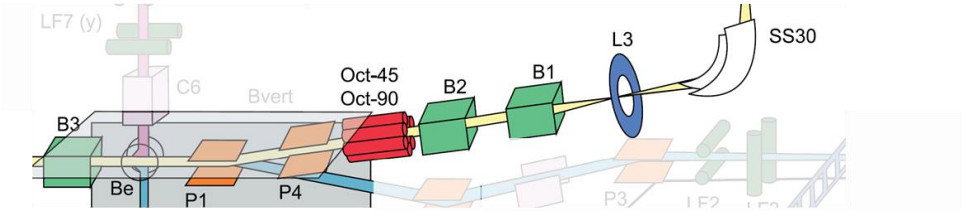
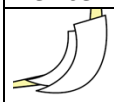
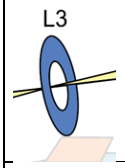
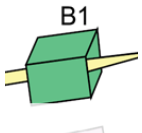
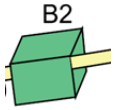
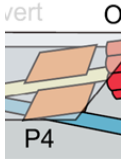


Figure 29: Illustration of the central column for the primary beam

2.1.1.3.2 Description of the optical components

Device	Label	Description and functionality
 78° ESA	SS30	30 mm radius spherical electrostatic sector used to rotate the primary ion beam by 78°.
	L3	Lens used to couple SS30, P1 and P4 in order to provide an achromatic deviation of the primary ion beam.
 Scanning plates B1	B1	A 4 plates deflector used to raster the primary ion beam on the sample surface.
 Scanning plates B2	B2x B2y	A 4 plates deflector used to raster the primary ion beam on the sample surface.
 Stigmator	Oct-90 Oct-45	Octopole used as a stigmator which acts like two quadrupole at 45°.
 Plates P4	P4h P4b	Deviating plates used to rotate the primary ion beam by 6°.

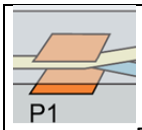
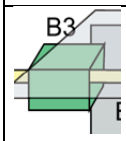
 Plates P1	P1h P1b	Deviating plates used to rotate the primary ion beam by 6° and to rotate the secondary ion beam by -6°.
 Scanning plates B3	B3	A 4 plates deflector used to raster the primary ion beam on the sample surface and which also act as a dynamic transfer system for the secondary ion beam.

Figure 30: Table of the central column elements

2.1.1.3.3 Detail of the achromatic deviation

L3 lens is used to couple electrostatic sector SS30 with plates P1 and P4 in order to provide an achromatic deviation of the primary ion beam. Indeed, the primary ions issued from the source have a certain energy dispersion and deflecting them with a simple electrostatic deflection would introduce a lot of chromatic aberrations (Figures Figure 31, Figure 32 and Figure 33).

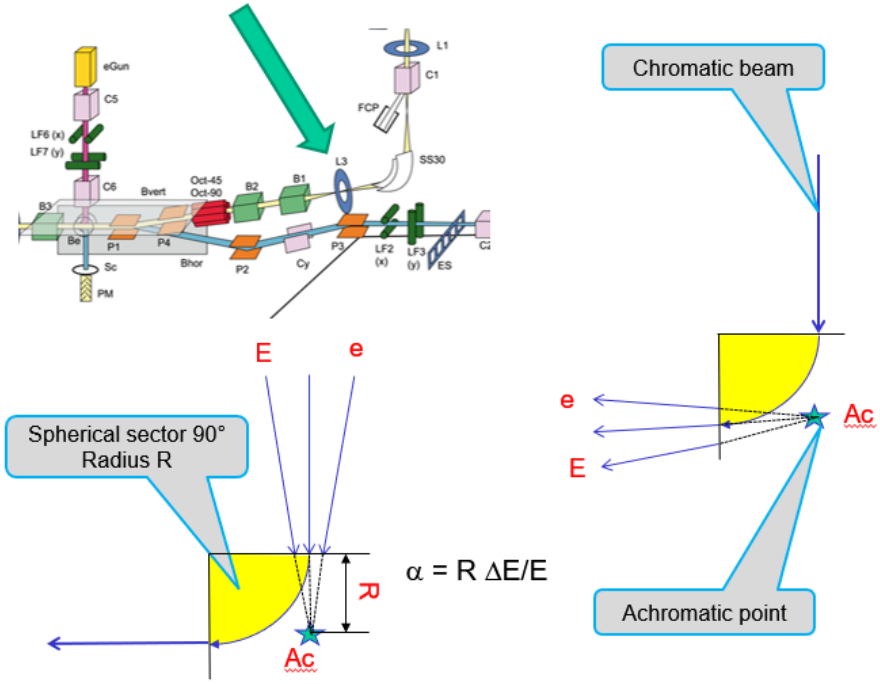


Figure 31: Principle of the effect of a 90° spherical sector on a polychromatic ion beam

- Two spherical electrostatic sectors, radius = R
- Chromatic compensation of a polychromatic beam.
- Ac2 is the image of Ac1.

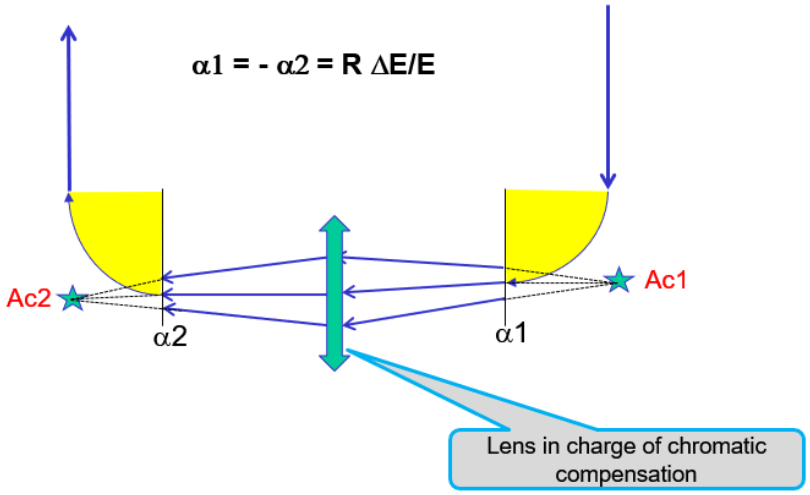


Figure 32: Principle of coupling two 90° sectors with a lens for an achromatic deviation

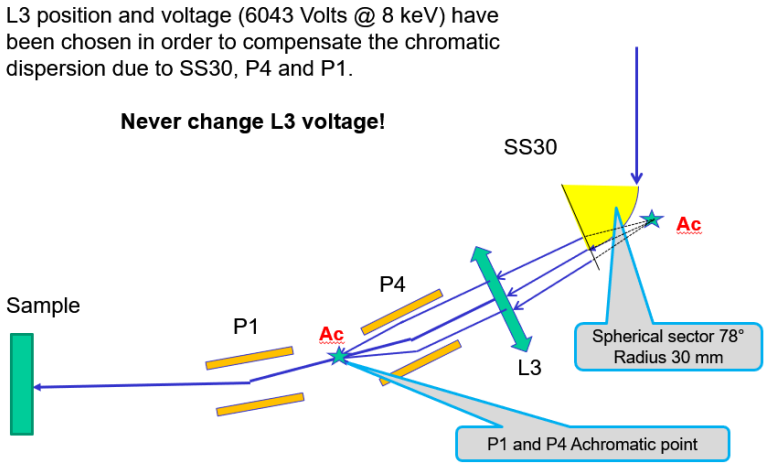


Figure 33: NS50L specific achromatic deviation optical scheme

2.1.1.3.4 Rastering the primary ion beam

The primary ion beam is scanned over the sample surface by the action of a set of three pairs of parallel plates B1, B2 and B3. Plates B3 are powered in synchronism with the two others scanning plates to cancel the motion of the secondary ion beam (dynamic transfer) at the entrance slit of the mass analyzer. The maximum practical field of view that can be scanned by the primary beam is 200*200 microns, with a number of pixels ranging from 64x64 to 1024x1024. However, note that increasing the field of view beyond 50 microns leads to defocusing and aberration effects on the primary ion probe.

As an optical image of the sample surface image is located near D1, **D1 acts also as a field aperture diaphragm** and thus limits the maximum field of view (FOV) visible by the mass analyzer. This FOV is defined by the following equation:

FOV = 0.6 * diameter of D1.

Consequently, the maximum field of view when using the smallest D1 diaph of 100µm is around 60 microns. While it is physically possible to scan a larger area with the primary beam, the ion image will display a dark area surrounding the center image, introduced by a small D1 aperture.

The tuning of B1, B2 and B3 is mainly linked to the dynamic transfer. B3 and B1 are set at their theoretical values: 4096 and 3700 bits, respectively. B2 is the free parameter and can be tuned independently in X and Y. The theoretical values for B2 are: B2X = 3170 and B2Y = 3480 but can differ depending on the instrument.

2.1.1.3.5 Dynamic Transfer

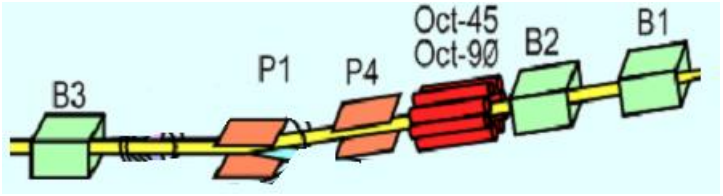


Figure 34: Illustration of the dynamic transfer elements

The dynamic transfer is in charge of keeping the secondary ion beam motionless at the analyzer entrance slit while the primary ion beam is scanned over the sample surface. The plates B3 are powered in synchronism with the two others scanning plates B1 and B2 (Figure 34), in order to cancel the motion of the secondary ion beam (dynamic transfer) at the entrance slit.

In the schematics below (Figure 35):

- B1, B2 and B3 rotate the primary ion beam around the center of D1,
- B3, in addition to its action on the primary ion beam, is in charge of the “Dynamic transfer”. B3 is powered so as to cancel the secondary ion beam motion at the slit level,
- B2X and B2Y can be set to different values to take into account the difference between the horizontal and vertical planes

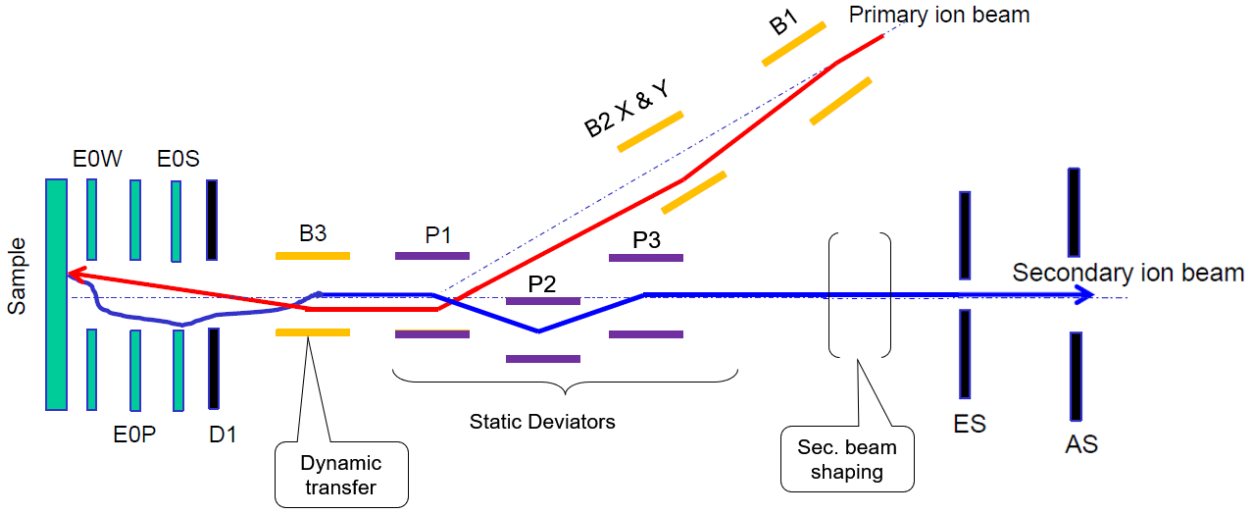


Figure 35: simplified primary scanning and dynamic transfer

Figure 36 shows the beam position at the entrance slit and the angle value at the cross-over position (roughly B3 center) while the beam is scanned over 30x30 microns *without any dynamic transfer*. As the entrance width is ranging from 10 to 30 microns it is obvious that without dynamic transfer the transmission would be dramatically reduced and that for field of view larger than a few microns there would be nearly no beam going through the entrance slit.

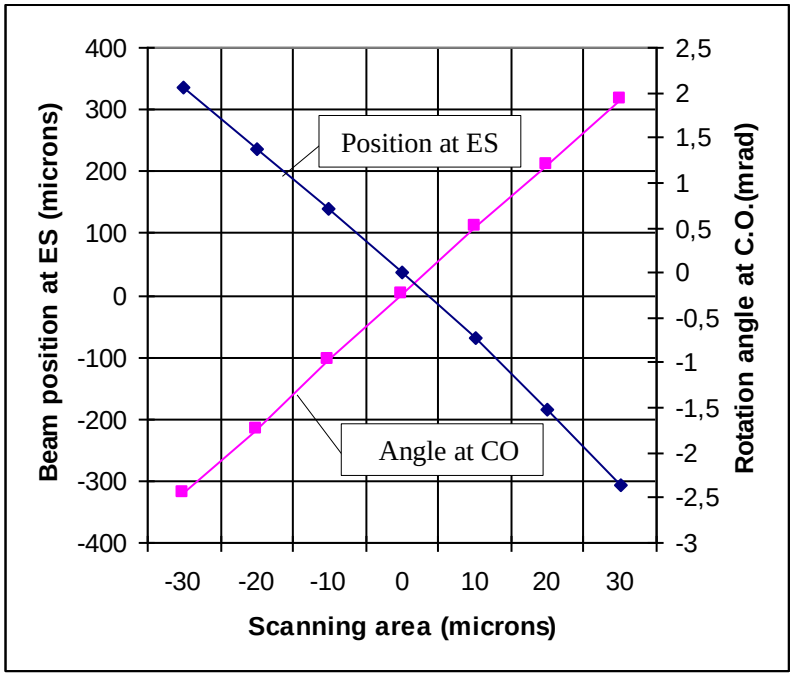


Figure 36: Theoretical variation of beam position at ES and rotation angle at CO vs scanning area .

These simulations have been made with the followings conditions: B3 = 0 Volts, Sec. Ions emitted at different position on the sample

2.1.1.4 Coaxial column

2.1.1.4.1 Overview

The same optical system is used to focus the primary ion beam and to collect secondary ions. The objective column is the communal path for primary ions, secondary ions, as well as primary and secondary electrons (Figure 37).

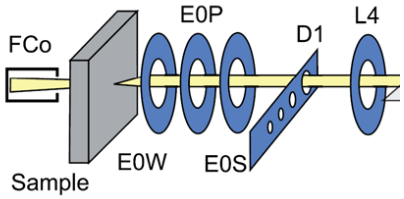


Figure 37: Illustration of the coaxial and central column

Compared to other SIMS instruments where the primary ion beam is introduced obliquely, this arrangement has the great advantage of considerably shortening the distance between the sample and the probe-forming lens. Thus, focal length and aberrations of the objective lens are minimized, which leads to a smaller probe diameter for a given ion current.

A second advantage of this experimental setup is that the secondary ions experience a strong electric field as they leave the sample, leading to a higher useful yield, and to a dramatic reduction of the broadening of the secondary ion beam at the exit of the probe forming system, due to the initial angular and energy distribution. In addition, the normal incidence of the primary ions minimizes shadowing effects on rough samples compared to oblique incidence in other SIMS.

The diaphragm D1 controls the angular aperture of the primary ion beam and acts as a field diaphragm for secondary ions.

2.1.1.4.2 Description

In the following table, all the elements (lenses, deflectors, sources, etc…) are described.

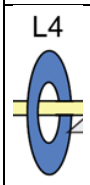
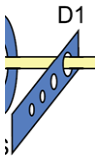
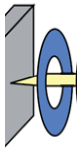
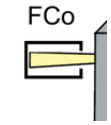
Device	Label	Description and functionality
 Lens L4	L4	Fourth electrode of the immersion lens. It acts mainly on the secondary ion beam.
 Diaphragm D1	D1	Aperture stop which limits the angular aperture of the primary ion beam, and controls the primary beam current and the spot size. But D1 also limits the field of view.
 Electrode E0S	E0S	Third electrode of the immersion lens E0 which acts mainly on the secondary ion beam.
 Lens E0P	E0P	Second electrode of the immersion lens E0 which focus the primary ion beam on the sample.
 Electrode E0W	E0W	First electrode of the immersion lens E0 which acts mainly on the secondary ion beam.
 Faraday Cup	FCo	Faraday Cup used to measure the probe current.

Figure 38: Coaxial column elements description

2.1.1.4.3 Probe diameter

Probe size can be theoretically determined by means of the following relationship:

$$(\text{Probe size})^2 = (\text{Gaussian size})^2 + \Sigma\,(\text{aberrations})^2$$

The main aberrations for this kind of optical system are:

- Aperture aberration: ½ Cs α^3
- Chromatic aberration: Cc α ΔE/E

α is the half-aperture at the sample. E and ΔE are the nominal energy and the energy spread of the primary ion beam, respectively. Cs and Cc are the aperture and chromatic aberration coefficients, respectively. Cs and Cc are linked to the optical properties of the immersion lens. The shapes of the electrodes have been designed to minimize these two coefficients; practical values for the N50 are Cs = 66 mm and Cc = 16 mm.

For a given probe size (d) one can theoretically determine an optimum value for D1 (or α) which maximizes the probe current. In a first approximation, by neglecting chromatic aberrations, one can determine this optimum:

$$\alpha_{opt} = ½ (d/Cs)^{1/3}$$

And $$I_{opt} = (3\pi^2/16) B (1/Cs)^{2/3} d^{8/3}$$

A complete simulation with chromatic aberrations gives for a probe size of 100 nm, D1 = 240 microns and I_{opt} = 2-3pA.
Above simulations have been made with the following hypothesis at 8keV with Cs+ primary ions and the followings hypothesis: Source size 40 microns, ΔE = 1 eV, Cs = 66 mm, Cc = 16 mm.

As D1 is not continuously adjustable, a compromise needs to be found for each probe size, Figure 39 is a summary of practical D1 vs probe size for Cs+ and RF source.

Probe size	D1	L1
Not significant	D1-1	Not significant
100 – 120 nm	D1-2 or D1-3	0
70 – 100 nm	D1-3 or D1-4	6000 V < L1 < 7000 V
< 70 nm	D1-4 or D1-5	> 7000 V

Figure 39: Practical rules for small probe diameters with Cs+

Figure 40 shows the standard D1 apertures used for analyses.

D1 #	1	2	3	4	5
D1 diameter (microns)	750	300	200	150	100

Figure 40: D1 Standard aperture diameter

2.1.1.4.4 Probe size vs E0P

In most analyses, the primary ion beam cannot be used focused as the beam will drill a very deep and narrow hole. Decreasing E0P will increase the probe size as shown on Figure 41. Experimental conditions were as following: Cs+ at 8 kV, D1 = 150 microns. In this measurement, Delta E0P was in fact negative. Practical value is 52V per micron for D1 = 150 microns.

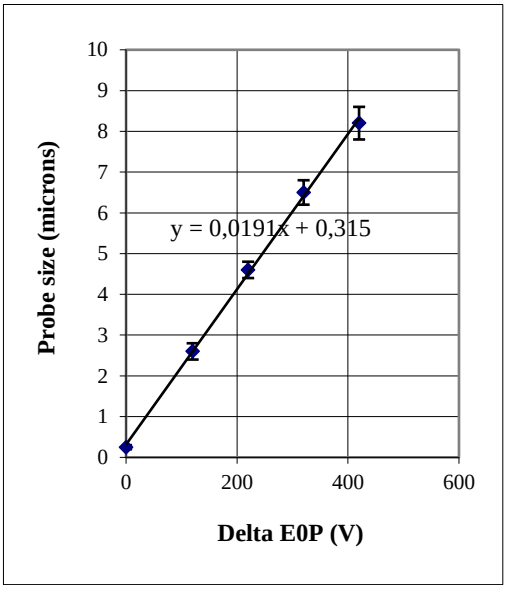


Figure 41: Relation between the probe size and the EOP delta value

2.1.1.4.5 Influence of Z

The CAMECA NanoSIMS 50L has been designed to work with a distance between the immersion lens E0W and the sample set to 400 microns. Any change of this value will affect the focusing value of E0P and thus the focal length. The practical rules for E0P is this following link: 60 Volts = 100 microns in Z.
Any z variation will not have any effect on the lateral resolution. Figure 42 and Figure 43 show the typical relationship between aberration coefficients Cs and Cc and the focal length.

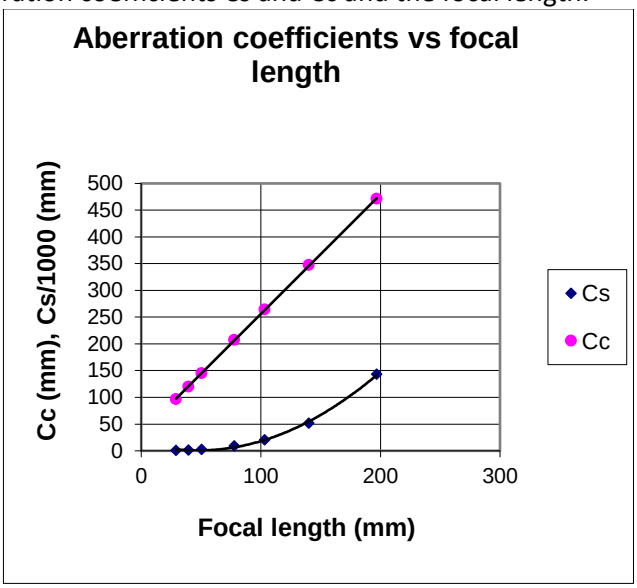


Figure 42: Aberration coefficients vs focal length

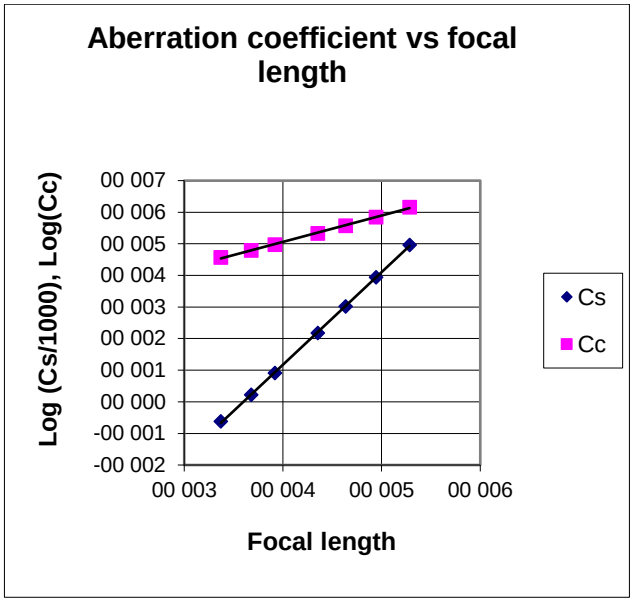


Figure 43: Aberration coefficient vs focal length

As shown on Figure 43 above, Cc is proportional to f $^{0.83}$ and Cs is proportional to f $^{2.93}$, f is the E0 focal length (roughly 6 mm). Changing Z is equivalent to changing the focal length of E0 leading to dz/z = df/f. In addition, relative variation of α will be also equal to relative variation of f: da/a = -df/f.
Assuming aberrations are expressed by:

$$Ab = \frac{1}{2} Cs\ \alpha^3$$

Thus:

$$dAb/Ab = dCs/Cs + 3\ d\alpha/\alpha$$
$$dAb/Ab = 2.93\ df/f - 3\ df/f$$
$$dAb/Ab = -0{,}07\ df/f$$

As f = 6mm and Delta z = 100 microns: df/f = 0,015 leading to dAb/Ab = 0,001. This result shows obviously that even a z variation of 100 microns will have no real effect on the aberration.
As the Gaussian reduction factor is directly proportional to the focal length, it will also be negligible.

2.1.2 The secondary ion column

The secondary ion optic is composed of four different sections: the coaxial column, the matching optics, the mass spectrometer and the multicollection system. The three last sections are exclusively used by the secondary ion beam while the first one is shared by the secondary and the primary ion beams as well as the electron beam.

Once this user's guide is mastered, users with instrumentation and optics background can fruitfully read the much more detailed article: *Dynamic transfer applied to secondary ion imaging over large scanned fields with the nanoSIMS 50 at high mass resolution*. Georges Slodzian, Ting-Di Wu, Jean Duprat, Cécile Engrand, Jean-Luc Guerquin-Kern. Nuclear Instruments and Methods in Physics Research B 412 (2017) 123–173. https://doi.org/10.1016/j.nimb.2017.06.019

2.1.2.1 The Coaxial column

2.1.2.2 Crossover and image plane notions

To understand how the immersion lens, coaxial column and spectrometer work, it is useful to remember two basic optical notions: **images** and **crossovers**. In the schematic below, the lens produces an **image** of the object in an image plane with a given magnification (A'B'/AB). In the NanoSIMS one can replace "object" by "sample", "lens" by "E0S" and "image plane" by "ES entrance slit plane" for example. Note that optical objects can be real (like the sample) but also virtual (the apparent origin of particle trajectories). The **Crossover** plane is at a position where a distance at the object side is converted into an angle: the secondary beams are **rotating** around this point.

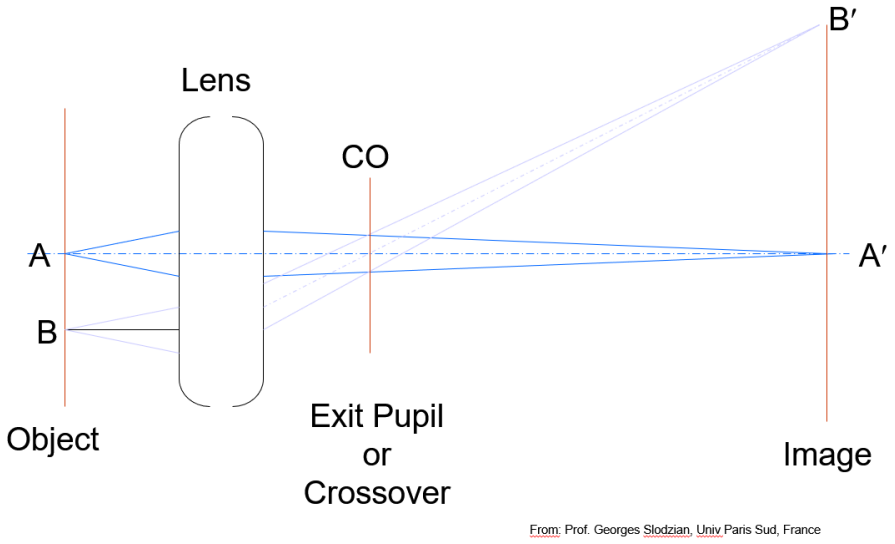


Figure 44: notions of object, crossover and image planes

2.1.2.2.1 Overview

The same optical system is used to focus the primary ion beam and to collect secondary ions (Figure 45). The objective column is the common path for primary ions (2.1.1.4), secondary ions, primary and secondary electrons.

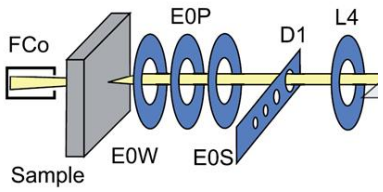


Figure 45: Illustration of the coaxial column

One can summarize in one slide the main points of the immersion lens of a CAMECA SIMS:

- High Acceleration Voltage, High Extraction Field
  - Reduction of angular divergence and relative energy dispersion of emitted ions
- Direct ion images of sample surface
- Diaphragm on the Crossover controls:
  - Collection solid angle
  - Aberration on image points
- The sample surface is the first "electrode". Any local geometry or potential change may influence optical properties.

Figure 46: general immersion lens highlights

Note the last point, especially important for the NanoSIMS: the **sample, integral part of the immersion lens,** must be flat (hence embedding/polishing for rocks), parallel to the immersion lens electrode (careful mounting in a sample holder in good shape, analysis at least 1mm from the hole edges) and its electrical potential must be as well defined and homogeneous as possible (hence metal or carbon coating, good electrical contact and/or electron flooding for electrically insulating samples).

Note also point 2: the direct ion image capability is not used in the NanoSIMS. Indeed when one wants the immersion lens to collect secondary ions with the largest solid angle as possible, the angular aberration of this lens limit its lateral resolution. Hence the NanoSIMS is instead working uniquely in a **microprobe** mode, not in ion microscope like the Cameca IMS series.
The **immersion lens** collecting the secondary ions will be followed by a **transfer optical system** in charge of **adapting** the beam characteristics (energy, angle, position) to enter in the mass spectrometer in the most favorable conditions for a good (transmission/mass resolution) couple. In the NanoSIMS a large solid angle is effectively collected and the originally circular secondary ion beam is shaped into a beam of rectangular section with optimized energy-position-angle distribution at the spectrometer entrance, minimizing the mass spectrometer aberrations. Note that such shape optimization results in a beam striction or beam waist rather than a perfectly defined shape at the entrance slit level.

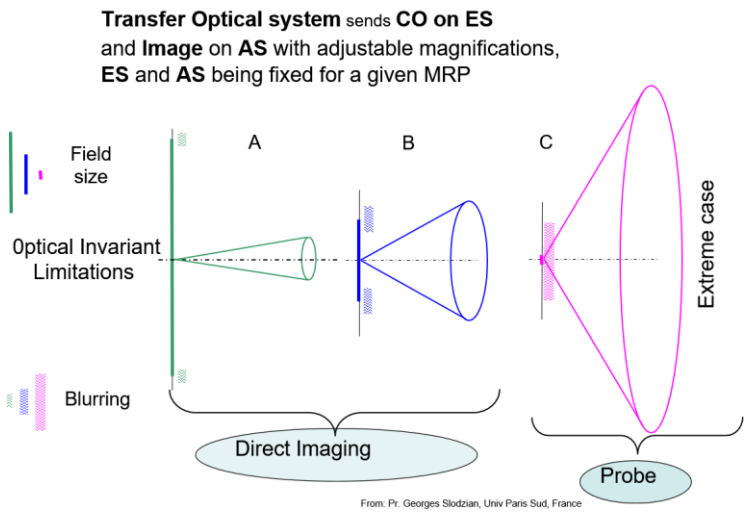


Figure 47: from an ion microscope to an ion microprobe

2.1.2.2.2 Crossover and image planes in the NanoSIMS

The NanoSIMS secondary line includes several crossovers and image planes:
The wehnelt electrode EOW will produce a first crossover of secondary ions very near the sample.
A second crossover is located in the middle of the dynamic emittance matching system. B3 will thus contribute to the scanning of the primary ion beam, but also of the "de-scanning" of the secondary ion beam to cancel its movement at the entrance slit ES level.
A third crossover is located between entrance slit ES and aperture slit AS.
Concerning image planes, the first image of the surface is positioned after E0S. The D1 diaphragm is positioned just here, allowing two simultaneous actions: a) controlling the beam size & current, and b) limiting the field of view on the sample.
This image of the sample is then re-imaged at the level of the entrance slit. Hence using very small ES size will limit the FOV in the horizontal plane.

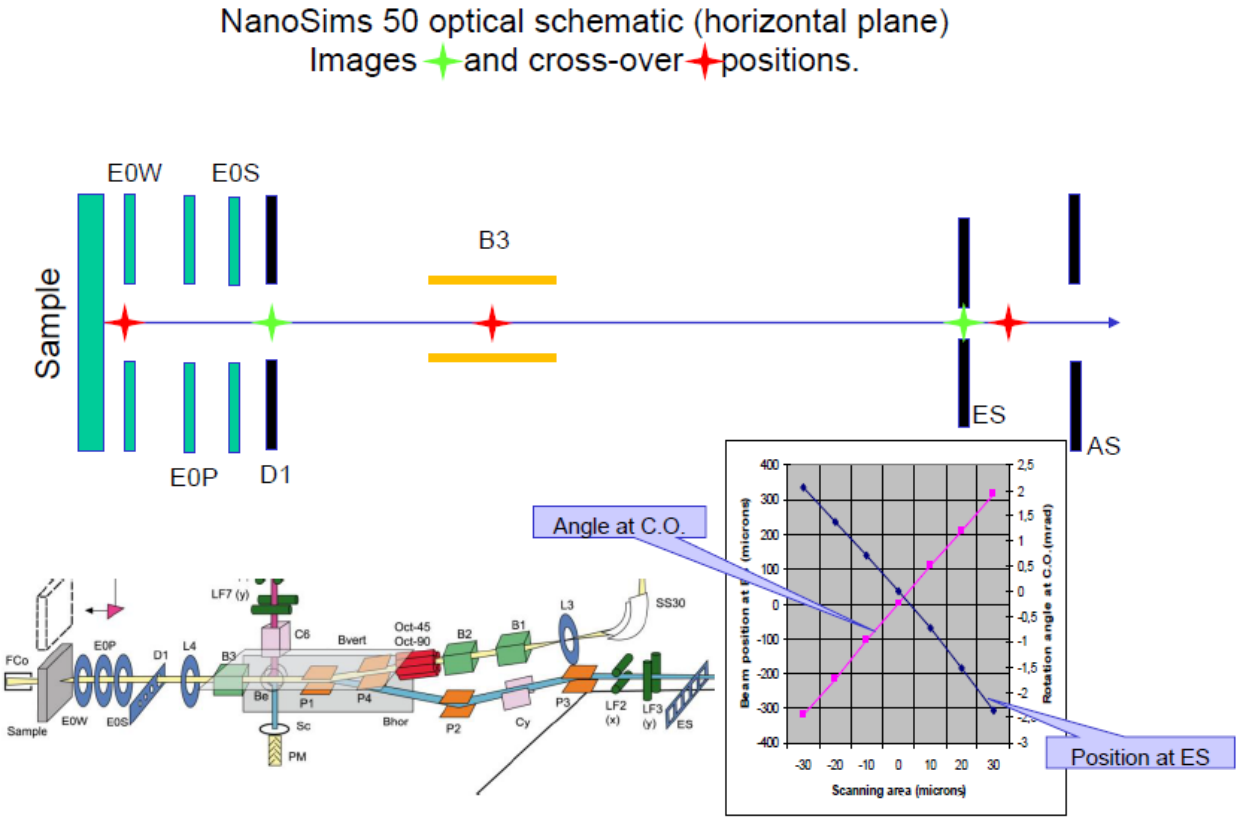
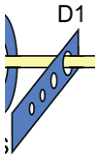


*Figure 48:transfer optic: Crossovers and image positions in the **horizontal** plane*

2.1.2.2.3 E0S Description

In the following table (Figure 49), all the elements (lenses, deflectors, sources, etc…) are described.

Device	Label	Description and functionality
 Electrode E0W	E0W	First electrode of the immersion lens E0 which acts mainly on the secondary ion beam. W stands for “wehnelt”.
E0P  Lens E0P	E0P	Second electrode of the immersion lens E0 which focus the primary ion beam on the sample. P stands for “primary”
 Electrode E0S	E0S	Third electrode of the immersion lens E0 which acts mainly on the secondary ion beam. S stands for “secondary”
 Diaphragm D1	D1	Aperture stop which limits the angular aperture of the primary ion beam and limits the field of view.

L4  Lens L4	L4	Fourth electrode which acts mainly on the secondary ion beam.

Figure 49: Table of the elements of the coaxial column

2.1.2.2.4 Considerations on E0S

The electrode E0S which mainly acts on the secondary ion beam, **focuses the secondary ion beam in the entrance slit ES** (Figure 37). Assuming that the primary ion beam is already properly tuned, the first tuning to perform in the secondary ion column is E0S.

The optimum E0S value changes with the ion species and the distance Z between the sample and E0S. One bit of E0S is roughly equivalent to 1.5 micron of sample Z translation. Hence for best reproducibility an important starting point will always be to precisely adjust the sample surface to the same Z-position. It is much more sensitive than on other Cameca SIMS which use an extraction gap of several mm. This is achieved by obtaining the in-situ optical microscope image in focus while varying Z sample position.

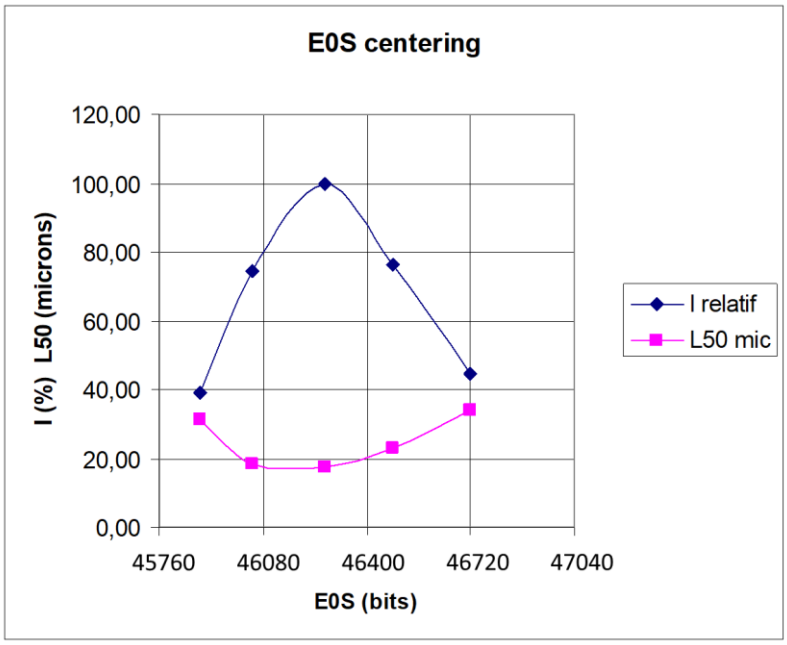


Figure 50: Variation of beam intensity (I) and width (L50) with E0S.

In addition to E0S, L4, LF2 and LF3 which are also acting on the secondary ion beam focusing but these last three lenses are at preset fixed potentials for a source potential of 8kV (Figure 51).

Lens	E0S	L4	LF2	LF3
Voltage (in bits)	44800 +/- 1600	44800	20000	28960

Figure 51: typical values for E0S, L4, LF2 and LF3

Check Operations (9.1.12.3, Optimizing Transmission) for details on E0S Tuning.

Remark: Changing E0S leads to a variation of the secondary ion beam cross-over position at the exit of the coaxial column. In addition to this position variation there is also a variation of the exit angle (Figure 52):

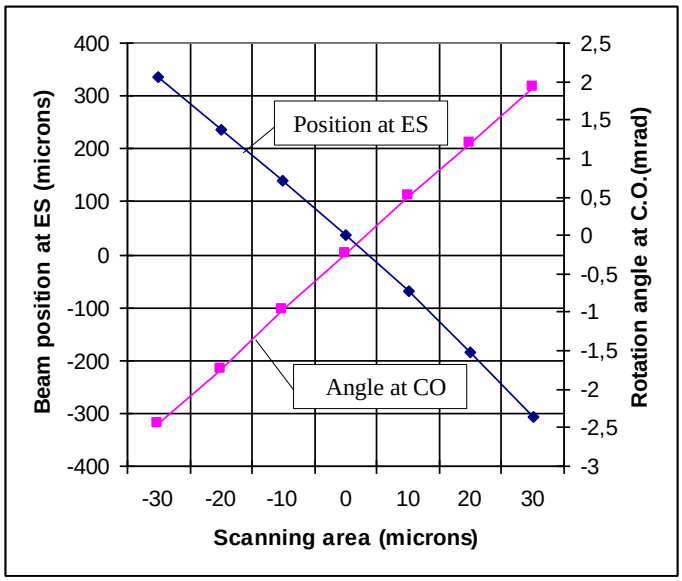


Figure 52: Theoretical variation of the crossover (CO) position (simulations with B3 = 0 Volts and SI emitted from the sample).

As a consequence, for each E0S value corresponds a particular setting of the dynamic transfer (especially B3) (Figure 54). It could then be useful to check the dynamic transfer tuning (see procedure in expert operation 9.3.1.3.1), especially if the mass spectrometer has to be used at high mass resolving power and if E0S has been largely modified.

While the sample is moving in Z, E0S has to be modified to keep the beam waist position at the ES plane, leading to a slight movement of the crossover in B3.

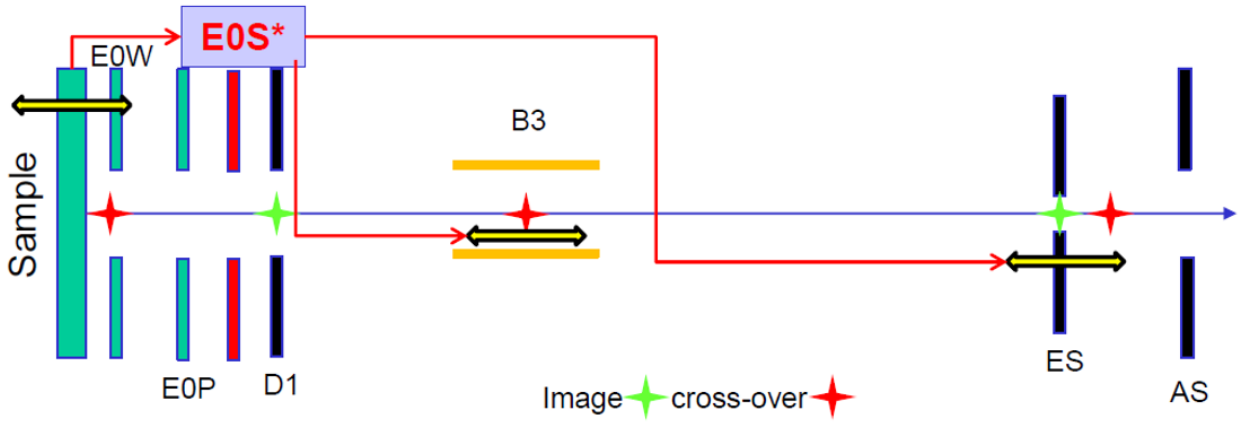


Figure 53: E0S, sample, cross-over and beam waist positions

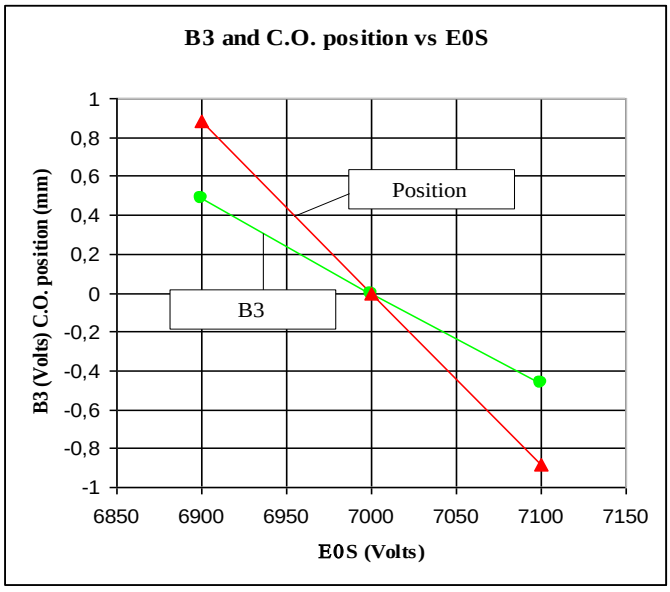


Figure 54: Theoretical variations of B3 vs E0S

These simulations have been made with the following conditions: B3 = 10 Volt; Sec. Ions emitted at 30 microns; Standard value for E0S: 7000 Volts. Depending on each particular instrument the standard E0S value can vary a lot.

In conclusion, for the best reproducibility, E0S should be maintained as constant as possible, hence the sample surface distance from E0W and the surface potential should be also kept as stable as possible.
For practical tuning details refer to the operation chapter 9 but as an order of idea, at 16keV impact energy (=E0W at 8000 volts) :

- for elemental imaging E0S must be kept stable within +/-75 V
- for Low reproducibility Isotopic ratios (1s > 2-3 permil): E0S must be kept within +/-50 V.
- for High reproducibility Isotopic ratios (1s < 1 permil): E0S must be kept stable within +/-25 V.

These ranges of value for E0S are proportional to Ep and varying from instrument to instrument depending on the relationship between E0S and Cy:

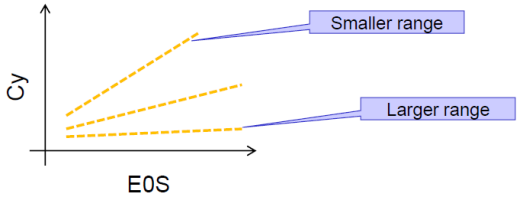


Figure 55: instruments with different Cy/EOS relation

2.1.2.3 Matching optics

2.1.2.3.1 Overview

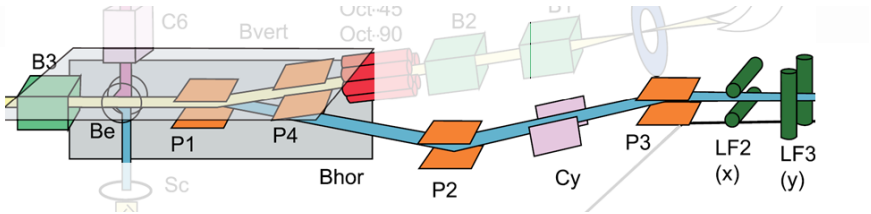


Figure 56: Illustration of the matching optics

At the exit of the coaxial column the secondary ion beam travels through a set of parallel plates P1 which is in fact an electrostatic separator for primary and secondary ions of opposite signs. This symmetric design keeps the secondary and primary ion beam energies equal. These energies can be tuned from a few keV to 10 keV.
Because of its high degree of dispersion in aperture and energy, the secondary ion beam must travel as straight as possible before it enters in the mass spectrometer. Otherwise its trace diagram (angular – space graph) will show very intricate folds leading to a dramatic reduction of the cutting efficiency of the slits in the mass spectrometer leading to a reduction of the transmission for a given mass resolving power. Thus, to minimize these effects, small deviations have been chosen: 6° in P1 and P3 and 12° in P2. In addition, P1, P2 and P3 have been set to ensure an achromatic deviation of the secondary ions.

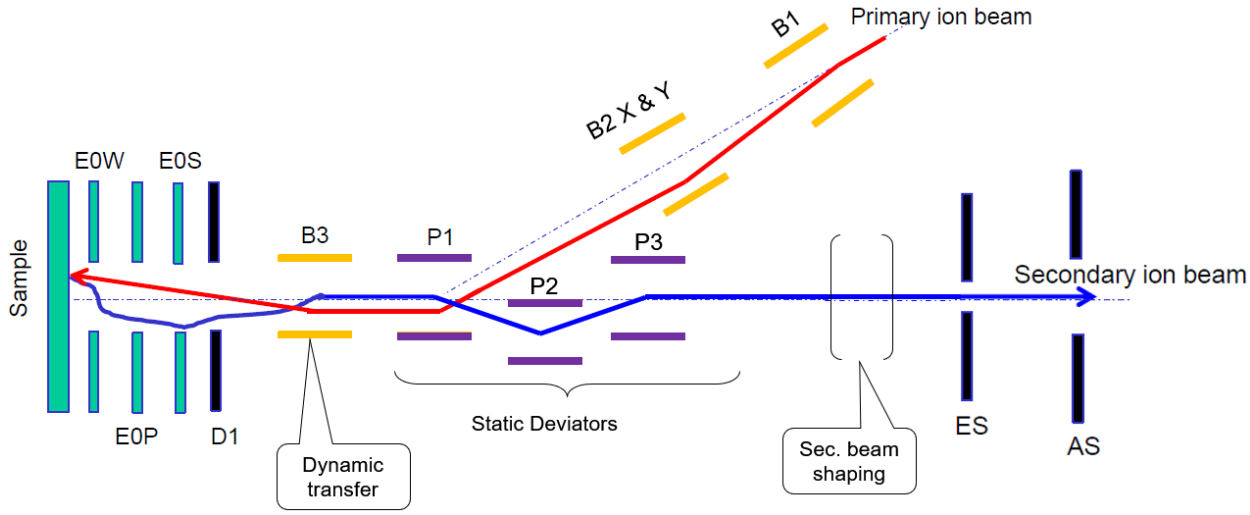


Figure 57: simplified dynamic transfer, static deviation & sec. beam shaping

After these three parallel plates the beam enters in the matching section of the mass spectrometer. A set of two slit lenses (unidirectional focusing lens), LF2 and LF3, are necessary to adapt the secondary ion beam in terms of angular aperture and spatial dimensions in the horizontal (radial) and in the vertical (transverse) plane. As the mass spectrometer is corrected for second order aperture aberration in the radial plane, the angular aperture in this plane can be relatively large; this possibility has been used as the beam is focused on the entrance slit in the horizontal plane. In the vertical plane, the aperture is kept as small as possible and the beam is not focused in the entrance slit.

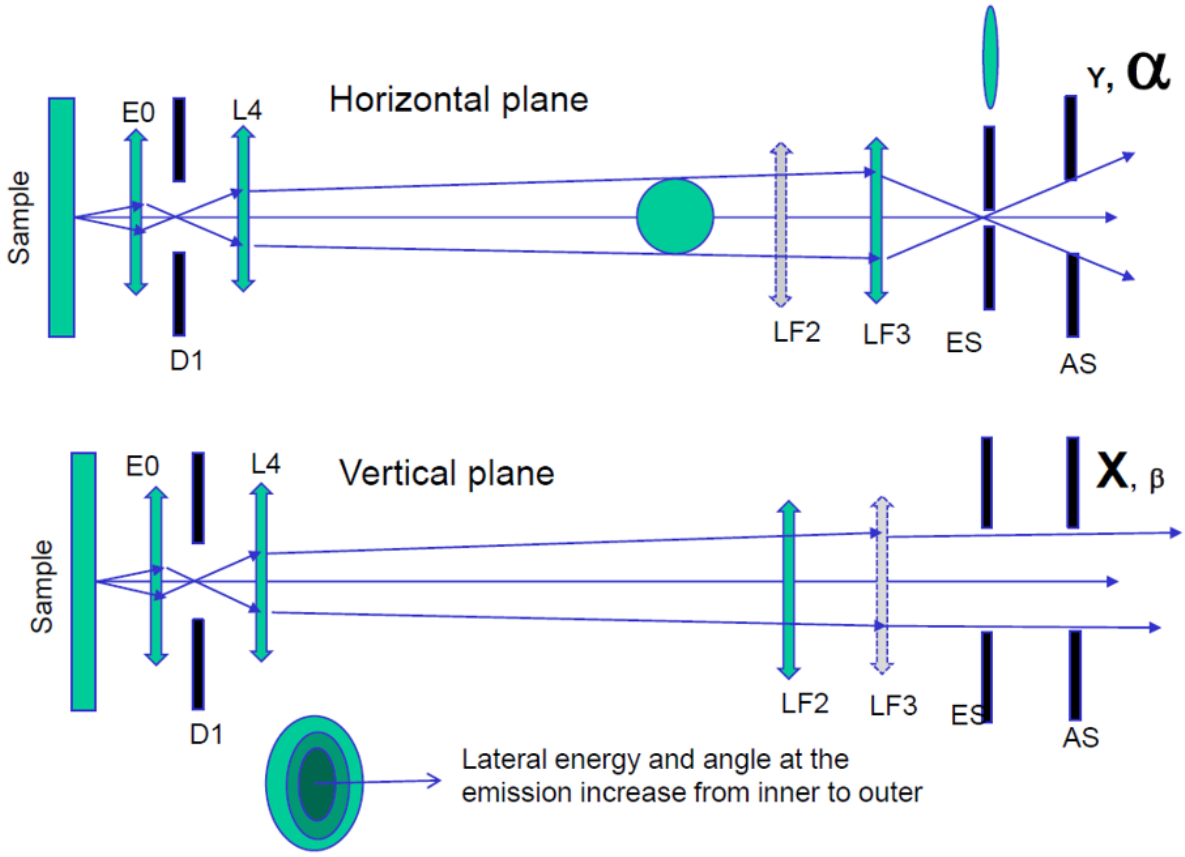
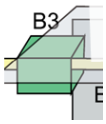
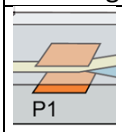
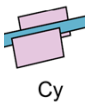
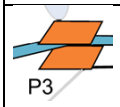


Figure 58: Focusing of the matching optics in the horizontal and vertical planes

2.1.2.3.2 Description

In the following table (Figure 59), all the elements of the matching optic are detailed.

Device	Label	Description and functionality
 Scanning plates B3	B3	A set of 4 plates used to scan the primary ion beam on the sample surface and which also act as a dynamic transfer system for the secondary ion beam.
 Plates P1	P1h P1b	Deviating plate used to rotate the primary ion beam by 6° and to rotate the secondary ion beam by -6°.
 Plates P2	P2h P2b	Deviating plate used to rotate the secondary ion beam by 12°.
 Corrector Cy	Cy	A 2 plate deflector used to center the secondary ion beam in the horizontal plane.
 Plates P3	P3h P3b	Deviating plate used to rotate the secondary ion beam by 6°.

 Lens LF2	LF2	Slit lens used to control the height of the secondary ion beam in the vertical plane.
 Lens LF3	LF3	Slit lens used to focus the secondary ion beam in the entrance slit (horizontal plane).

Figure 59: table of the elements of the matching optics

P1, P2 and P3 allow a centering of the secondary ion beam in LF2 and in the entrance slit (ES) in the vertical plane. Cy allows a centering of the secondary ion beam in LF3 and ES in the horizontal plane. To maintain the mass spectrometer settings unchanged, it is recommended to re-center the secondary ion beam in ES with CY and P2-P3 (see Operation chapter 9.1.12.3).

Then, in order to minimize aberrations along the secondary ion beam path it is necessary to center the beam in LF2 in the vertical plane, and in LF3 in the horizontal plane. As LF2 is a slit lens acting only in the vertical plane, a reasonable misalignment in the horizontal plane will have no effect on the secondary ion beam quality. Similarly, LF3 is a slit lens acting only in the horizontal plane, a reasonable misalignment in the vertical plane will have no effect on the secondary ion beam quality. See chapter 9.3.1.3.1 for details on how to center the beam in LF2 and LF3.

2.1.3 The mass spectrometer

2.1.3.1 Overview

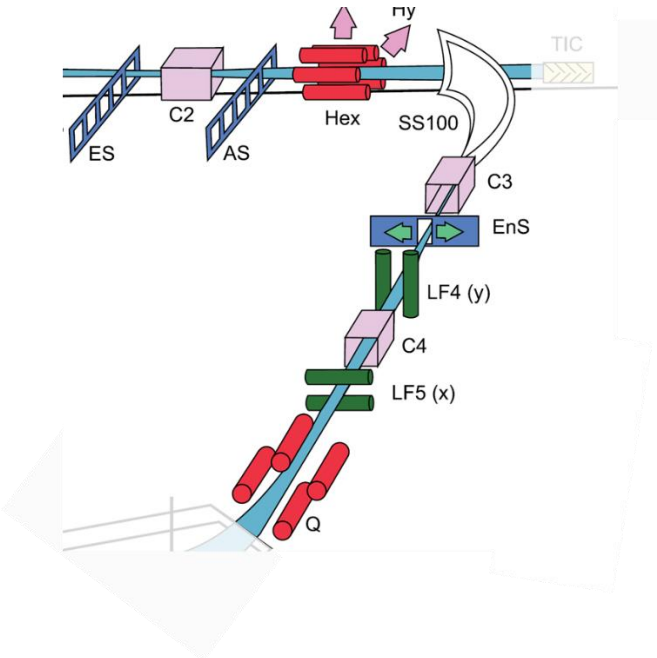


Figure 60: Illustration of the mass spectrometer

The mass spectrometer is a double focusing system with a focal plane. In order to achieve angular and energy focusing along the whole focal plane, the magnetic prism and the electrostatic sector are coupled by a quadrupole lens (Q) and two slits lenses (LF4 and LF5).
Three different slit systems limit the beam extensions:

- The Entrance slit (ES) limits the spatial extension and the lateral energy of the secondary ion beam. It has a rectangular shape.
- The Aperture slit (AS) limits the angular extension of the beam. It has a rectangular shape.
- The Energy slit (EnS) limits the energy bandwidth of the beam.

Lens LF4 acts in parallel with Q on the energy and angular focusing. Lens LF5 controls the height of the secondary ion beam in the magnet. The Hexapole (H) corrects second order aperture aberrations in the horizontal plane.
A proper choice of the matching section and of the three limiting slits leads to a good compromise between mass resolving power and transmission.
The following figure shows the main optical elements that will help to explain later in the text the two different focusing in the horizontal and the vertical planes.

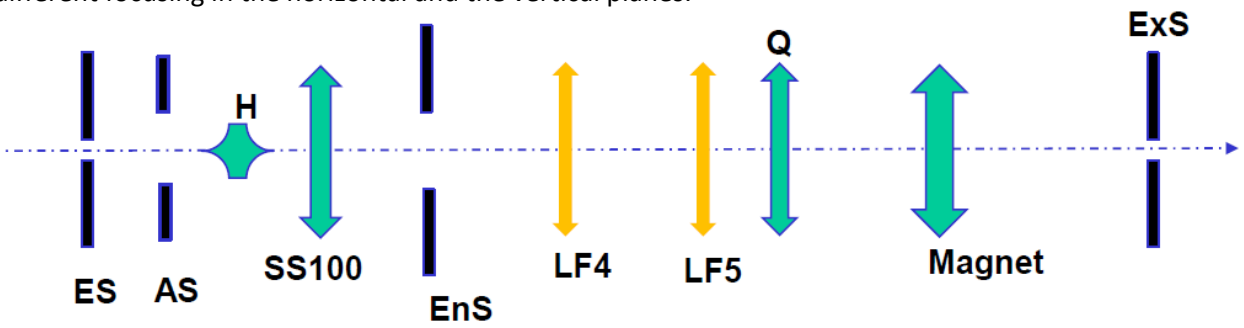
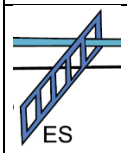
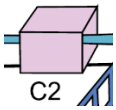
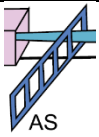
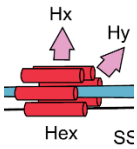
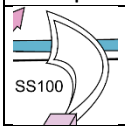


Figure 61: mass spectrometer main optical elements

2.1.3.2 Description

In the following table (Figure 62), all the elements of the mass spectrometer are detailed.

Device	Label	Description and functionality
 ES Entrance slit	ES Esx Esy	Entrance slit of the mass spectrometer – It has 5 different positions corresponding to 5 different slit width x height: **ES1: 30** X 180 µm, **ES2: 25** X 160 µm, **ES3: 20** X 140 µm, **ES4: 15** X 120 µm and **ES5: 10** X 100 µm
 C2 Corrector C2	C2x C2y	A 4 plate deflector used to center the secondary ion beam.
 AS Aperture slit	AS	Aperture slit of the mass spectrometer – It has 5 different positions corresponding to 5 different slit widths and heights. The **width** x height of aperture slit sizes are usually: **AS1: 350** x 350µm, **AS2: 200** x 200µm, **AS3: 150** x 150µm, **AS4: 80** x 80µm and **AS5: 40** x 40µm.
 Hx Hy Hex SS Hexapole	H Hx Hy	Hexapole used to correct second order aperture aberration.
 SS100	SS100	100 mm radius spherical electrostatic sector used as an energy analyzer.

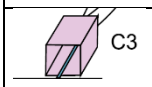
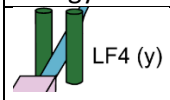
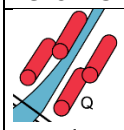
90° ESA		
 Corrector C3	C3x C3y	A 4 plates deflector used to center the secondary ion beam.
 Energy slit	EnS pos EnS width	Continuously adjustable slit used to control the energy bandwidth of the secondary ion beam.
 Lens LF4	LF4	Slit lens used to assure the chromatic focalization (horizontal plane).
 Corrector C4	C4x C4y	A 4 plates deflector used to center the secondary ion beam.
 Lens LF5	LF5	Slit lens used to control the height of the secondary ion beam in the vertical plane.
 Quadrupole	Q	Quadrupole lens used to focalize the secondary ion beam on the magnet focal plane

Figure 62: table of the elements of the mass spectrometer

2.1.3.3 Mass Fractionation at the entrance slit

Due to the presence of leaking B_{field} along the secondary ion trajectories, the secondary ion beam at the entrance slit is mass fractionated. These fringing fields are mainly produced by the two ion pumps in charge of pumping the analysis and the central chambers. This effect leads to severe mass fractionation at the entrance slits in both planes: ion intensities are attenuated or cut differently depending on their mass (proportionally to the square root of their mass if it is purely magnetic). Although isotopic measurements are usually corrected by the ratio of a standard of known value this can be a problem for precise and reproducible measurements.

Two external coils have been added to generate a compensating B-field when flowing a DC electrical current through them. This cancels the dispersion effect, resulting in having secondary ions of different mass over charge ratio centered at the same position in the entrance slit for one specific value of P3 and Cy.
Two optimum values of Bhor and Bver which depend on each instrument are adjusted to limit the mass fractionation. These values will then stay constant for this primary energy. The propagation software will automatically change this value (prop. to sqrt(Ep)) when the primary energy is changed.

See chapter 9.3.1.3.2 on how to practically tune the B-field coils.

As an illustration see below Figure 63 and Figure 64: on a SiC sample for a few values of Bhor or Bver we record the values of P3 or Cy voltage positioning $12C^-$ and $28Si^-$ peaks in the central position in the entrance slit. For one given value of the compensating Bfield, the two peaks are centered in the slit for the same P3 or Cy voltage: this is the optimum value to apply and save.

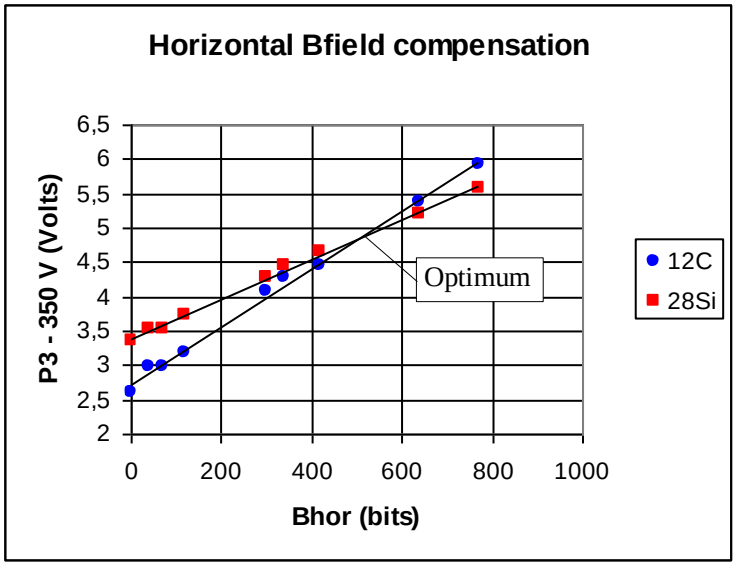


Figure 63: Horizontal coil compensation

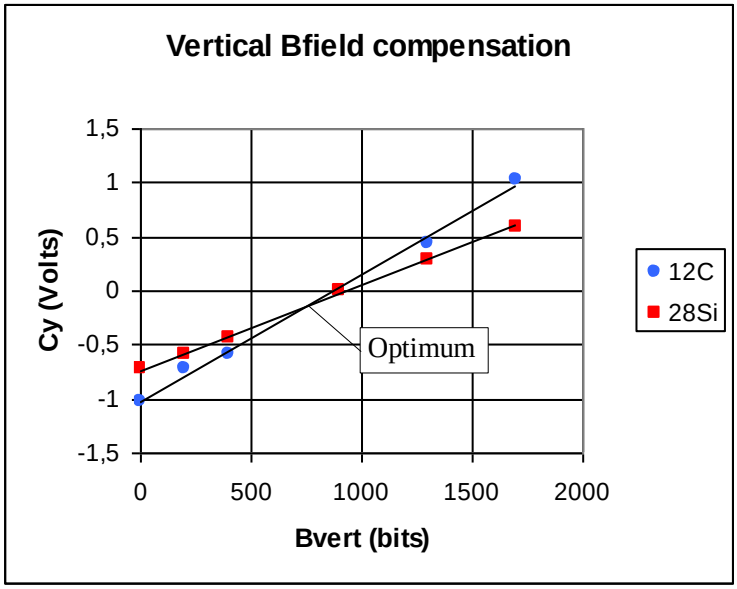


Figure 64: Vertical coil compensation

The following measurements (Figure 65), have been made on a Si wafer. Three different secondary ion beams ($^{16}0$, ^{30}Si and $^{28}Si_3$) have been scanned across the entrance slit (slit #3, 30 microns) in the horizontal plane. Corrector Cy has been used to scan these beams. Without vertical B_{field}, the distance between $^{16}0$ and $^{84}Si_3$ is roughly 0.44 Volts which can be estimated to be 5 microns. This would be huge for entrance slit size of 10 to 30 microns width.

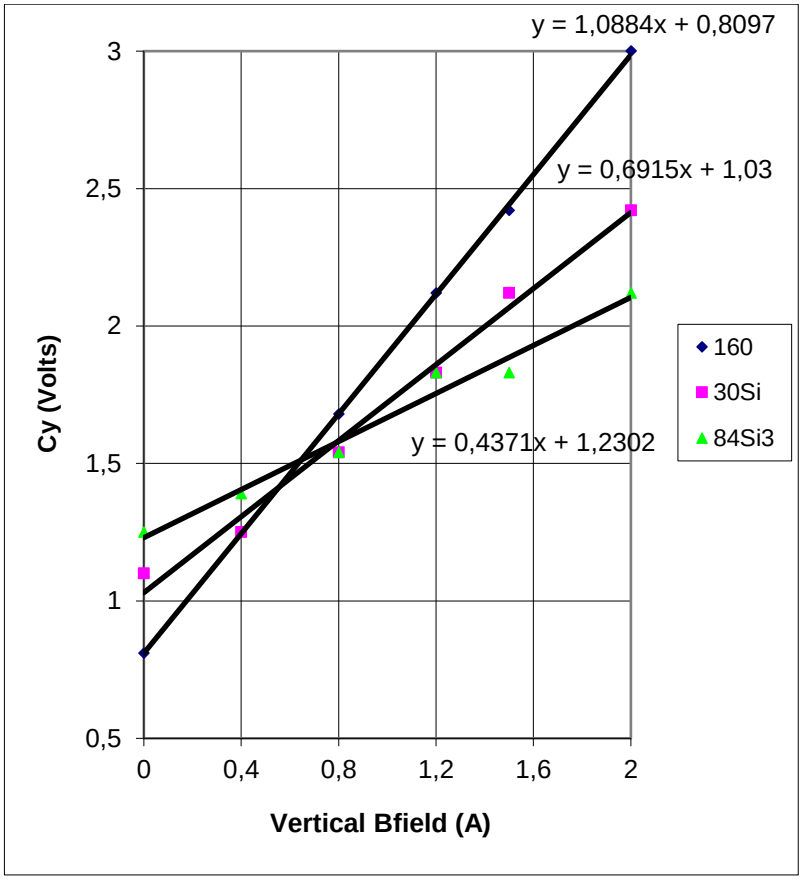


Figure 65: Example of Horizontal coil tuning

Figure 66 shows that the slope of the linear fit is proportional to 1/sqrt(M), demonstrating that this effect is a pure magnetic field effect.

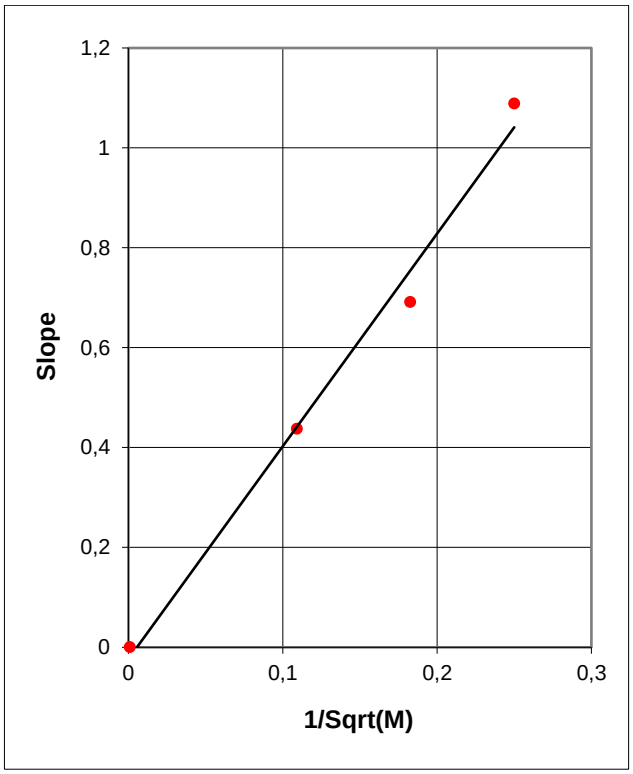


Figure 66: Slope vs $1/M^{1/2}$

2.1.3.4 Mass spectrometer tuning

To reach high mass resolution without loss of transmission, the mass spectrometer has been designed with low aperture and chromatic aberration coefficients. Due to a special design of the magnet, it is free of second order aperture aberrations in the radial plane; thus, it can accept secondary ion beam with large angular aperture.
The mass resolving power M/ΔM is dependent of numerous terms and this dependence is not only linear but also of higher order. The following formula describes the effect of the main ion beam parameters on the inverse of the mass resolving power.

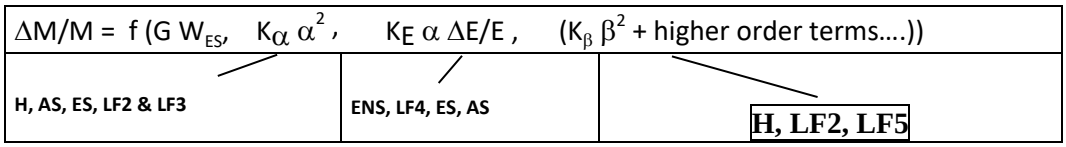


Figure 67: main ion beam parameters of the mass resolving power

- W_{ES}: Entrance slit width (or beam waist),
- G: magnification of the spectrometer. G = R/537; as the mass dispersion is R/2, **the MRP is independent of R**.
- $K\alpha\ \alpha^2$: second order aperture aberration term,
- $K_E\ \alpha\ \Delta E/E$: main chromatic aberration term,

with

- α the half aperture angle in the radial (horizontal) plane,
- β the half aperture angle in the vertical plane,
- K_α, K_β and K_E second order coefficients for the radial aperture, vertical aperture and chromatic aberrations respectively,
- $\Delta E/E$ the relative energy spread of the secondary beam.

The bottom part of Figure 67 shows the optical elements influencing the aberration terms.

2.1.3.4.1 Angular focusing in the Horizontal (radial) plane

As shown on figure 66 and Figure 69, the electrostatic sector SS100 produces an image of the entrance slit located on the energy slit plane. The location and the size of this image are not adjustable. Then **Q and LF4** produce a **parallel beam at the entrance of the magnet** leading to a focus point located on the focal plan. The location of this focus point depends directly on the mass and charge of the incoming ion beam.
Both **Q and LF4 act on angular focusing** but due to the location of LF4 –as close as possible to EnS – **Q is the essential lens to be tuned in order to reach the optimum focus**.
To tune Q, check the procedure in chapter 9.1.13

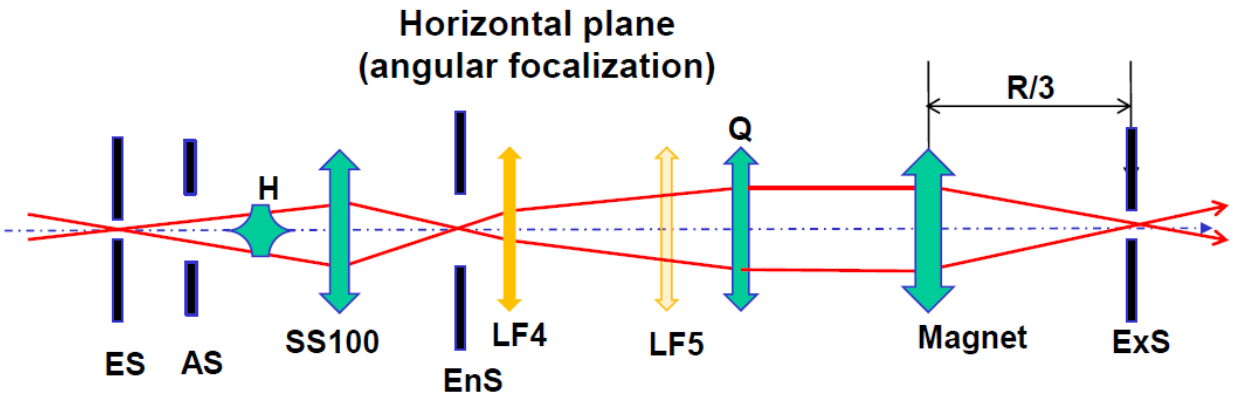


Figure 68: angular focusing in the horizontal plane

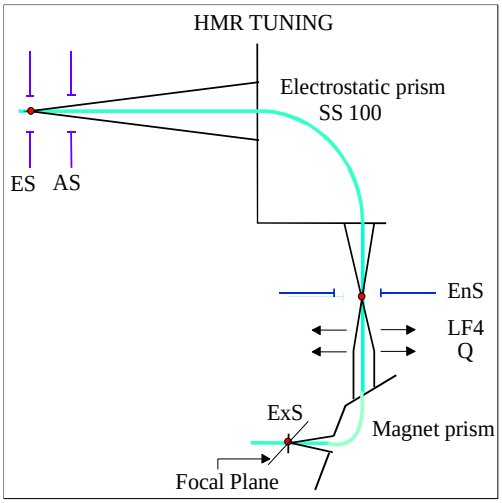


Figure 69: Angular focusing

2.1.3.4.2 Angular focusing in the Vertical plane

In the vertical direction the obtention of a parallel beam to enter the magnetic sector is achieved through the combination of LF5 and Q.
Refer to chapter 9 (operation) for practical tuning.

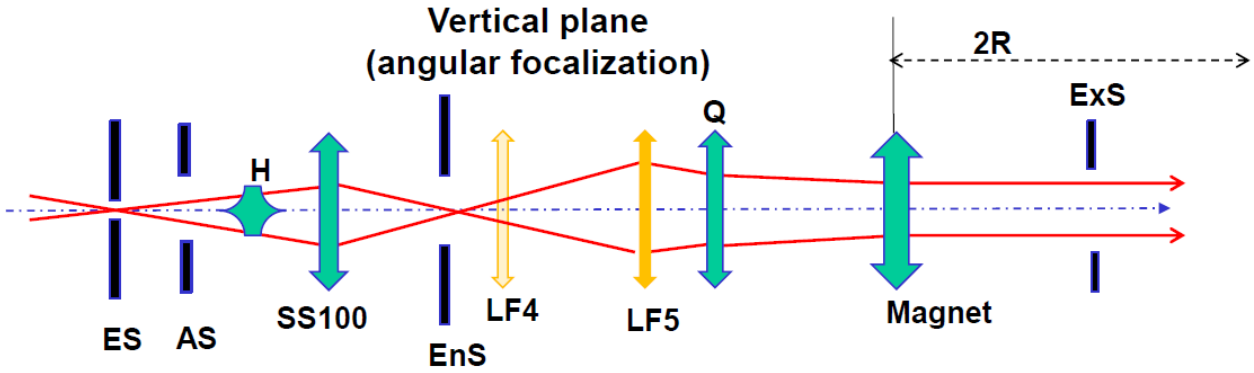


Figure 70: angular focusing in the vertical plane

2.1.3.5 Mass Resolving Power

Different definitions of the mass resolving power (MRP) are conceivable, each being suited to a special situation. The purpose of the MRP definition being referred to as the "CAMECA definition", is to characterize the mass line width in relation to mass dispersion. Here is a general approach to MRP definitions.

The width labelled $L_{10\text{-}90}$ is the width inside which one finds 80% of the line intensity and with 10% of intensity left on each side. If intensity is assumed to be uniformly distributed across the line and if aberration effects on the wings of the line are neglected (sharp line assumption), the total width of the line h_L would be,

$$h_L = L_{10\text{-}90} / 0.8 = 1.25\, L_{10\text{-}90} \qquad (1)$$

$L_{5\text{-}95}$ is also available in the HMR spectrum and is the width inside which one finds 90% of the line intensity To simplify, we consider situations where two adjacent mass lines are of equal intensity.

The dispersion in the plane of the exit slit is given by the following relation:

$$h_M \cong k\, R \frac{\Delta m}{m} \qquad (2)$$

h_M is the distance between the center of two mass lines differing by a mass difference Δm and k is a numerical factor which depends upon the design of the spectrometer. R is the radius of the circular path in the uniform magnetic field and "m" is the ion mass. In the CAMECA NanoSIMS 50L setup, k = 0.5. Thus, the general expression is obtained with atomic masses M,

$$h_M \cong \frac{R}{2} \frac{\Delta M}{M} \qquad (3)$$

Now let us select a slit width h_S and make the sharp line assumption. To define an MRP we usually have to introduce additional conditions: either the percentage of peak reduction, P% of full intensity, due a narrow exit slit if $h_S < h_L$ or the distance h_T over which the mass line may move inside the exit slit while keeping its full intensity if $h_S > h_L$ (flat top peak). The other condition regards the valley to peak ratio being accepted as a separation for two adjacent lines.

If one makes the choice of having a full intensity line and a point of zero intensity between two adjacent lines, MRP can be defined with the relation,

$$h_M \cong h_S + h_L \qquad (4)$$

That is,

$$h_S + h_L = \frac{R}{2} \frac{\Delta M}{M} \quad \rightarrow \quad \frac{M}{\Delta M} = (MRP)_0 \cong \frac{R}{2\left(h_S + h_L\right)} \qquad (5)$$

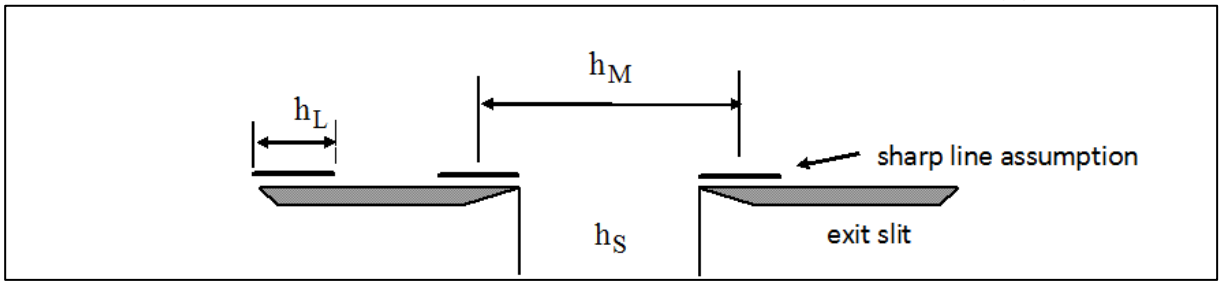


Figure 71: Illustration of the Exit slit for the MRP calculation

If one wishes to characterize the mass resolving limit capabilities, the conditions may be written as:

- P arbitrarily fixed at 80% of the full intensity
- The valley to peak ratio arbitrarily fixed at 25%.

These conditions lead to:

$$h_s = L_{10\text{-}90} \text{ and } h_M = 2\ L_{10\text{-}90} \qquad (6)$$

and result in CAMECA's definition:

$$\mathbf{MRP_{Cam} = M/\Delta M = R / 4\ L_{10\text{-}90}} \quad (7)$$

This value of MRP indicates the possible performance of the instrument consistent with the specified conditions, regardless of the actual width of the exit slit. But it should not be understood as a definition of MRP being suitable for any situation.

It is worthwhile noting that the width $L_{10\text{-}90}$ is a parameter which takes its full meaning in relation with the mass dispersion h_M for a given mass difference Δm at each radius R. Instead of considering the variations of $L_{10\text{-}90}$ and h_M with the radius R separately, it is convenient to use MRP_{Cam}.

From a practical point of view, the procedure to follow is:

- $L_{10\text{-}90}$ is measured experimentally (or any other L(a%-b%) width....).
- For each specific problem, the width of the exit slit has to be chosen according to some criteria: P% of the full intensity or the "length" h_T of the flat top.
- The valley to peak ratio between two adjacent lines.
- Then the MRP is determined with the help of equation (3).

To provide users with information upon the intrinsic performances of the instrument, it is essential to determine the relations between the signal intensity and the mass line width at different radius R. But, considering the great variety of situations, it is left to users to determine which MRP definition they should use according to their specific problem (taking into account the intensity of interfering lines, the precision of the peak top flatness, ...) and which decision they have to make concerning the width of the exit slit.
Figure 72 shows a typical example of data obtained on CN^- ions. MRP has been computed according to the CAMECA definition. The relative transmission refers to the ratio (Intensity with slits) / (intensity without any slit). The different points correspond to different combinations of slits. There are no specific rules to determine which set of slits has to be used to reach a given mass resolving power. However, one can set for the slits an order of efficiency as following: firstly ES, then EnS and lastly AS.

It is good practice to check regularly (every week and before sessions) and keep record in a long term stability spreadsheet of the transmission in the same multicollection set-up (for instance: slits fully opened and always on the same sample standard).
For example, using 16keV Cs+ on silicon one should measure FCo and $^{28}Si^-$ count rate and obtain roughly the following:
With I = n Q/t , using 1 pA there are 1 E-12/ 1.6 E-19 = 6.25 E6 PI/s.

- 625 000 $^{28}Si^-$ c/s per pA of Cs+ with no slit. Or SI/PI = 10%
- 250 000 $^{28}Si^-$ c/s per pA of Cs+ with ES-3, AS-2 (MRP > 6000). Or SI/PI = 4%.

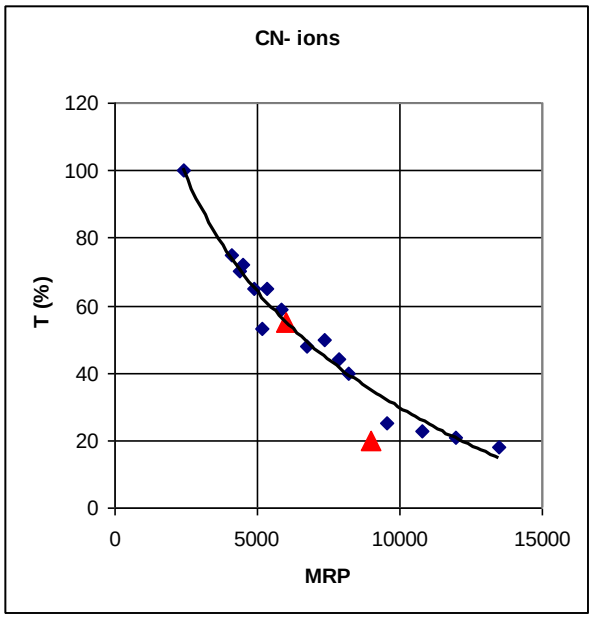


Figure 72: Relative transmission versus Mass resolving power. 100 % = slits fully opened

2.1.3.6 Chromatic (=energy) compensation

As shown on Figure 74, two secondary ion beams with two different energies are emerging from the entrance slit on the axis. The electrostatic sector SS100 will disperse them according to their energy and the two emerging trajectories will appear as coming from a single point named achromatic point (Ac.). This specific point is located at one radius from the SS100 exit face.
The magnet has also achromatic points. Let's suppose that we send two trajectories focus on the magnet achromatic point Ac', they will emerge with the same angle from the magnet.
Q and LF4 have been design to conjugate Ac and Ac' in order to compensate chromatic dispersion at the exit of the magnet.
Q and LF4 act both on angular focusing and on chromatic compensation**. Q is the main lens for angular focus *and* chromatic compensation**. Due to its location - as close as possible from EnS – **LF4 acts mainly on chromatic compensation (fine energy focusing)**.
For the LF4 tuning procedure inside the overall secondary tuning, read chapter 9.3.1.3.2

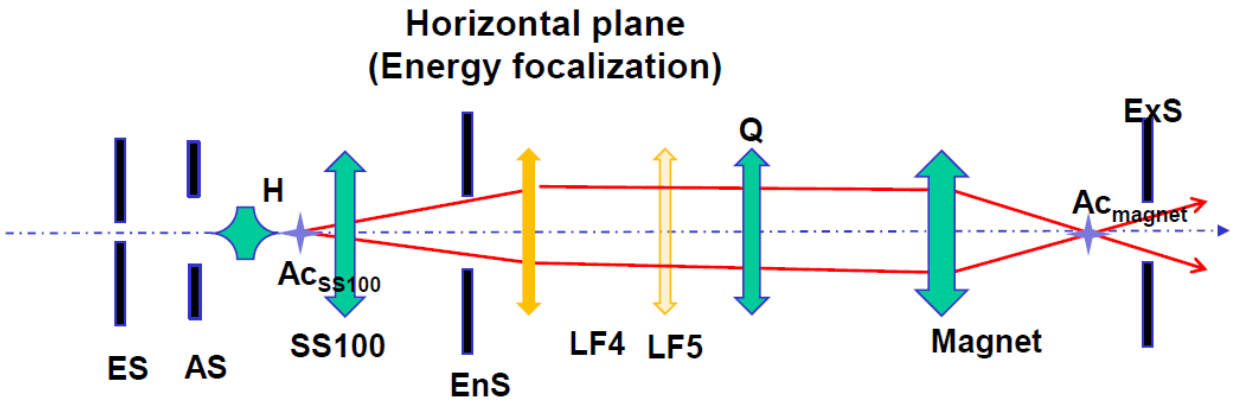


Figure 73: energy focusing in the horizontal plane

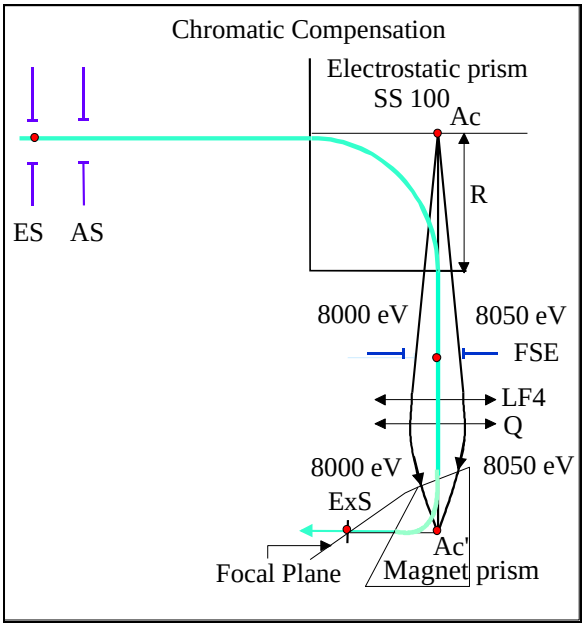


Figure 74: Energy focusing

As Q acts on Chromatic compensation, LF4 cannot remain unchanged as Q varies. Thus, for each value of the Quadrupole Q one can determine an optimum value for LF4 (Figure 75).

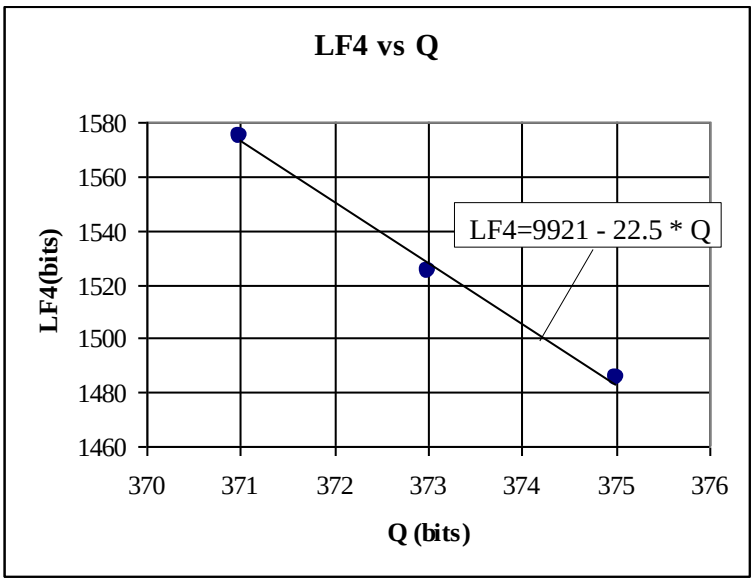


Figure 75: LF4 vs Q

2.1.3.7 Second order aperture aberration

These aberrations are mainly due to the electrostatic sector SS100. Let suppose we have a mono energetic secondary ion beam emerging from the entrance slit. This beam has a very small aperture in the horizontal plane in a mean direction doing a (radial) angle α with respect to the axis in the horizontal plane.
This beam will be focused in the energy slit plane but not on the axis. The distance z from the axis is proportional to the power 2 of α (Figure 76)

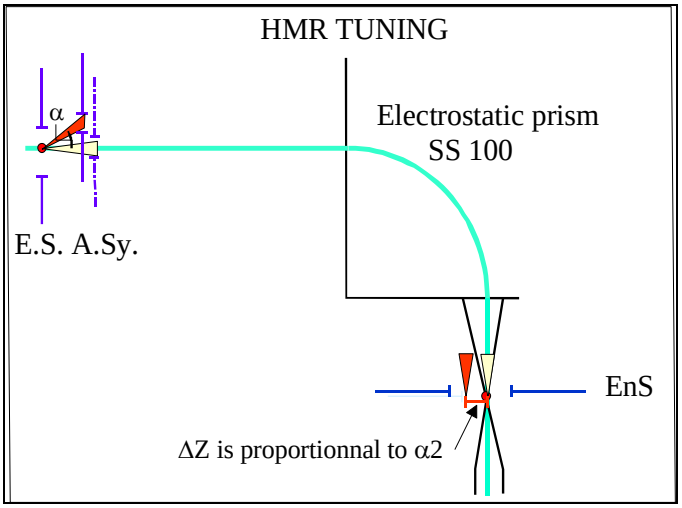


Figure 76: α^2 aberrations

Let suppose we have another mono-energetic secondary ion beam emerging from the entrance slit. This beam has a very small aperture in the vertical plane in a mean direction having an angle β in the vertical direction with respect to the axis.
This beam will be focused in the horizontal plane at the energy slit but not on the axis. The distance z from the axis is proportional to the power 2 of β (Figure 77)

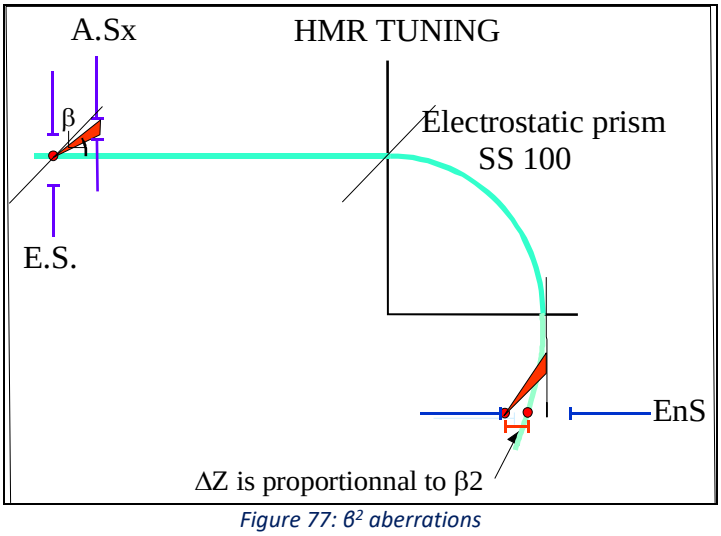


Figure 77: β^2 aberrations

Figure 78 and Figure 79 demonstrate the existence of these aberrations. These measurements have been done with ES5 and AS5 on a SiC sample. AS5 position has been changed either in the horizontal plane ($As5_y$ for α^2 aberrations) or in the vertical plane ($AS5_x$ for β^2 aberrations).

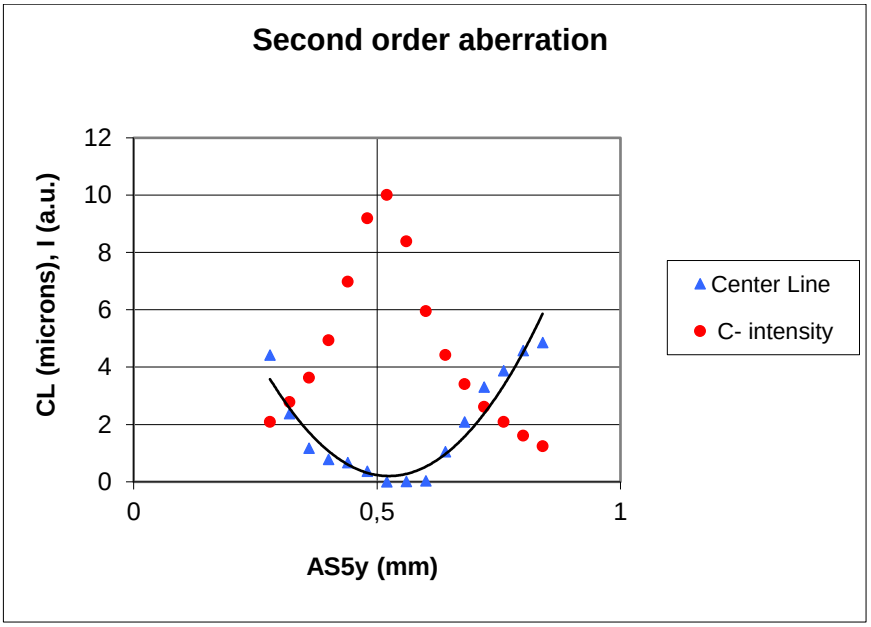


Figure 78: aberrations in the radial plane (α^2)

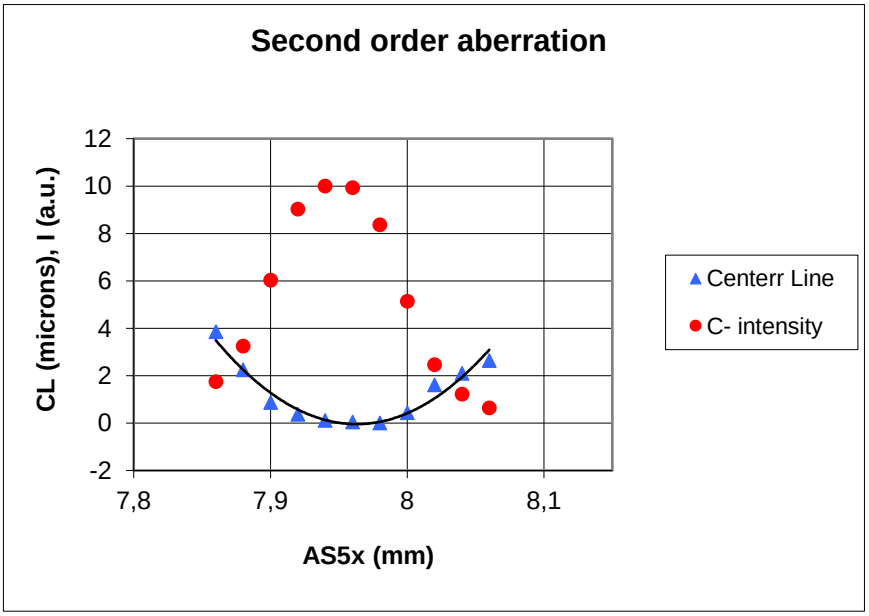


Figure 79: aberrations in the radial plane (β^2)

These two last measurements are extremely difficult to achieve as the secondary ion beam has to perfectly focus in the exit slit. If not, one will see a linear dependence instead of a parabolic one.

2.1.3.8 Hexapole tuning

The mass spectrometer is corrected for second order aperture aberrations in the radial plane by the Hexapole H. In the vertical plane, the beam shape has been transformed from a circular one to a slit one, leading to a dramatic reduction of angular aperture and thus of aberration effects.
The followings figures (Figure 80, Figure 81 and Figure 82) show typical variations of the mass resolving power while the positions and values of H are changed. Experimental conditions were : ES-3, without AS, EnS-2 (intensity reduction of 20%).

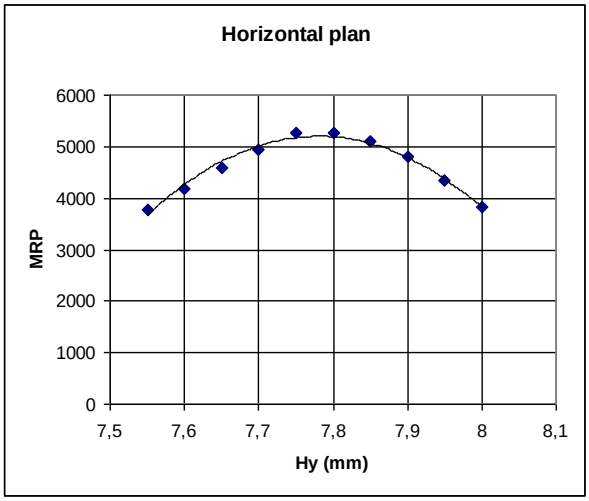


Figure 80: Mass Resolving Power vs H position in the horizontal plane

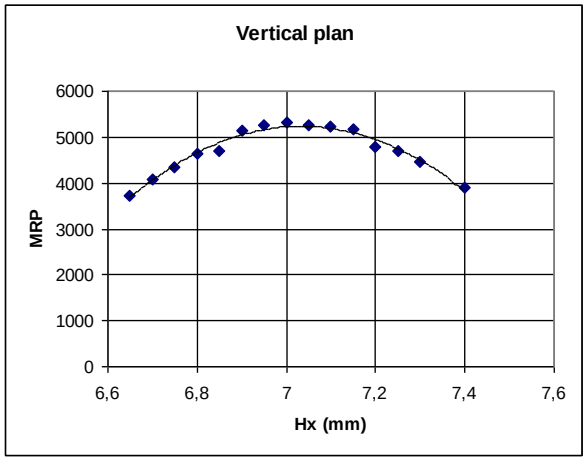


Figure 81: Mass Resolving Power vs H position in the vertical plane

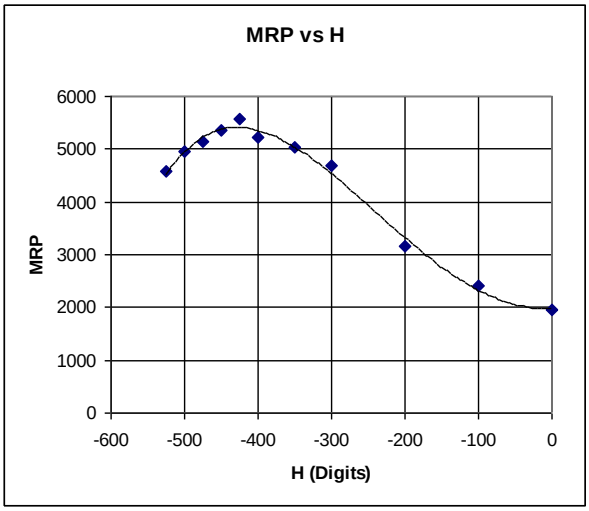


Figure 82: Mass Resolving Power vs Hexapole value

It is thus critical to properly tune the Hexapole to optimize the Mass Resolving Power. The hexapole tuning procedure inside the general secondary tuning is described in chapter 9.3.1.3.2.

2.1.4 The multicollection system

2.1.4.1 Overview

The magnetic sector, derived and optimized from a Mattauch-Herzog configuration, permits to obtain a straight focal plane along which seven detectors (more precisely their exit slit) can be moved and positioned precisely. The ions travel in a flat tube between the pole pieces where the magnetic field is very homogeneous. They follow a circular path but note that the shape and angles of the poles and the field shape leaking outside near the entrance and exit gaps play an important role too in the overall optics. As an illustration, the electron multipliers after the exit slits must be magnetically shielded otherwise the electrons cannot travel from one dynode to the next under the B-field!
The multicollection system follows the magnetic sector. It is one mechanical ensemble inserted (and removable for service) inside the multicollection chamber through a rectangular flange.
The multicollection incorporates:

- Six *moveable* (computer controlled) trolleys, each equipped with a miniature discrete dynode Electron Multiplier, a Faraday Cup with repeller and a slit assembly (3 widths, commutable under vacuum): detectors # 1, 2, 3, 4, 5 and 6. For fine mass adjustment and scanning, each trolley is equipped with an electrostatic sector preceded by deflection plates, before each exit slit. This set-up allows the recording of mass spectrum (called HMR as high mass resolution) around a single unit mass, with a range of mass line deflection of +/- 500 µm.
Each trolley is mechanically equipped with one FC and one EM. All FCs are connected to the flange but in the basis configuration only the FC of trolley #1 is equipped with a preamplifier and electrometer.
The "MULTICO SEVEN FARADAY" accessory permits to have all seven FCs preamplifiers/electrometers.
The FC/EM switches are automated and can be performed under vacuum. A window port allows the observation of the slit exchange mechanism.

- One *fixed* trolley delivered in standard with one EM and one FC (no preamp supplied), and a slit assembly (3 widths, commutable multicollection opened): detector # 7. It is mounted at FIXED radius R= 680 mm. It is also equipped with deflection plates before the slit for fine mass adjustment and scanning.
The FC/EM switch is manual and thus require a venting of the multicollection chamber.

Four main factors will determine the capabilities of the NS50L multicollection:

1) The **mechanical-physical limits** of the multicollection:
The extreme radiuses of ion trajectories are R_{min} = 145mm and R_{max} = 680mm. Within this range the mass resolution and transmission are approximatively constant. Using lower or higher radiuses would lead to a loss of performance.

2) The **magnetic field** obtained by flowing a given stabilized current in the coils around the laminated iron pole pieces. Inside the magnetic field, the radius R of circular ion trajectory is R = sqrt (2mU/q) / B so it is proportional to the square root of the mass M of the ion: R = a * sqrt(M) or R1/R2 = sqrt(M1/M2). *So the unit masses becomes nearer and nearer as we move up along the focal plane.*

3) **The Mass Range** is a **ratio D = 22** between minimum mass and maximum mass (e.g. from 1 amu to max 22amu or from 10 amu to max 220 amu). $D= (R_{max}/R_{min})^2$

4) **The minimum Mass interval between neighbor detectors:** once the mass M_{max} of detector 7 is defined through a chosen B-field, the minimum mass separation between two adjacent small detectors at the highest radius is given by: **M_{max}/58**.

So up to 58amu, single amu interval is always possible; between 58 and 116amu the highest masses can be limited to 2 amu interval ; between 116 and 174amu the mini interval can be limited to 3amu, etc.
Numerically, as R_{max} = 680mm the radius of ions of mass M will be: r = 680/ sqrt (M_{max}) *sqrt (M),
so δM_{min} (mini mass interval between 2 adjacent det.) = sqrt (M_{max}) * sqrt (M) * δr_{min}./ 340.
The mini NS50L radius interval between detectors (δr_{min}) is 5.8mm so δM_{min} ~ sqrt (M_{max} * M) * 0.017.

For example, selecting Mmax = 127 amu (iodine) on the 7th detector will give approximately:
δMmin = 0.66amu at 12amu (nearest mass: 13 amu),
δMmin = 0.98 amu at 26amu (nearest mass: 27 amu),
δMmin = 1.08 amu at 32amu (nearest mass: 34 amu),
δMmin = 1.72 amu at 81amu (nearest mass: 83 amu).
δMmin = 2.13 amu at 124 amu (nearest mass: 127amu).

The NanoSIMS control software will simulate and inform the operator of the exact possibilities or conflicts.

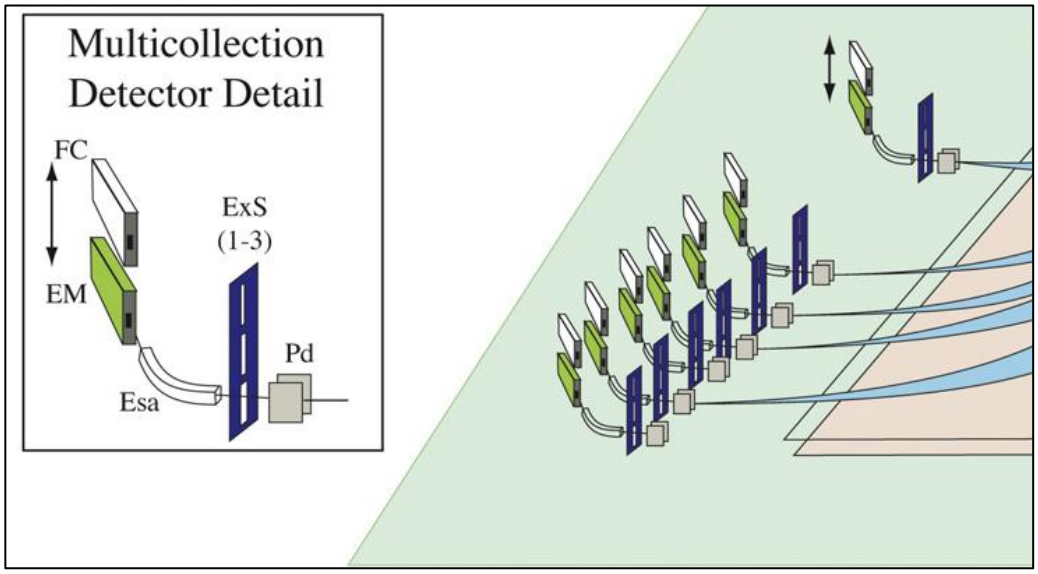
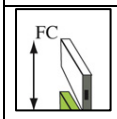
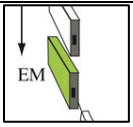
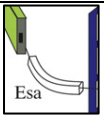


Figure 83: Illustration of the multicollection detection system

The multicollection system (Figure 83) is essentially made of two parts: a mechanical system which allows the operator to move detectors under vacuum and 7 detectors. These detectors can be either Faraday Cup (FC) or Electron Multiplier (EM).

2.1.4.2 Description

Device	Label	Description and functionality
 Trolley Faraday Cup	FC	Each trolley is equipped with a FC detector which is exchangeable with an Electron Multiplier detector depending on the analysis. This switch is motorized for the trolleys 1 to 6.
 EM	EM	Each trolley is equipped with a miniature electron multiplier (EM) exchangeable with a FC detector if needed. This switch is motorized for the trolleys 1 to 6.
 Esa	Esa	Each trolley is equipped with an electrostatic sector called Esa allowing to deflect the beam into the detector. Esa is shared by EM and FC detectors.

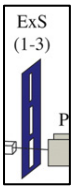
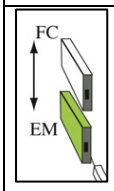
 Exit slits	ExS	Each trolley is equipped with three exit slits moveable depending on the analysis. ExS1 : 100 X 2400 µm, ExS2 : 70 X 2400 µm and ExS3: 40 X 2400 µm. These exit slits are used to separate the masses at the entrance of the detector. They can be used in conjunction with the other slits to define and optimize peak shape (flat peak top or gaussian shape, etc…). Exit slits are shared by EM and FC detectors
 Pd	Pd+ and Pd-	This is a pair of parallel plates (with opposite potentials) which allow to scan the mass line across ExS for recording a HMR mass spectrum, and to adjust a mass line position in the exit slit.. Pd is shared by EM and FC detectors
 Trolley 7		Detector 7 is a fixed detector. The radius cannot be changed and the EM/FC switch or the Exit slits switch needs to be made manually (see chapter 10.4.2)

Figure 84: Table of the elements of the multicollection

In front of each detector is a pair of parallel plates (Pd) allowing scanning of the mass line across the exit slit. The deflection coefficient is roughly 10 microns per Volt. These plates are also used to focus the secondary ion beam in each exit slit by applying the same voltage to both plates. 50 Volts is roughly equivalent to one bit of the quadrupole lens used to conjugate the electrostatic sector SS100 to the magnet. One can select independently, under vacuum, the exit slit size for each detector (EM or FC) mounted on a trolley. There are three different widths, the height remaining the same (2400 microns).
Each trolley can be moved along the focal plane, driven by a step by step motor under computer control with a minimum step of 1.2 micron (one external motor rotation of 1000 steps is equivalent to 1.2mm).
The trolley positioning reproducibility (1s) is demonstrated during installation to be better than 5 µm.
The minimum distance between trolleys is 5.8 mm. It is defined by electrical safety contacts on each trolley, in order to avoid crashes between trolleys.

2.1.4.3 Electron Multipliers (EMs)

The NanoSIMS 50L can simultaneously detect ions of seven different masses with a multicollection composed of one fixed and six movable EMs. The detectors must be as thin as possible to allow the simultaneous detection of heavy mass isotopes. The CAMECA NanoSIMS 50L multicollection trolleys are equipped with Hamamatsu® R4146 electron multipliers, customized especially for CAMECA. The R4146 width is smaller than 7mm.

The EMs of the NanoSIMS 50L are always working in a direct pulse counting mode where the secondary ions are counted one per one. A secondary **ion** striking the first dynode (conversion dynode) of the EM induces a secondary **electron** emission (Figure 85). These electrons are accelerated through the successive dynode stages in order to amplify the secondary electron current. A *gain* (mean number of electrons per secondary ions) of about 10^8 is obtained. For most of the secondary ions reaching the detector, a charge pulse is produced at the last dynode output. The charge amplitude is converted in voltage, and the pulse amplitude, in Volt is proportional to the EM gain.

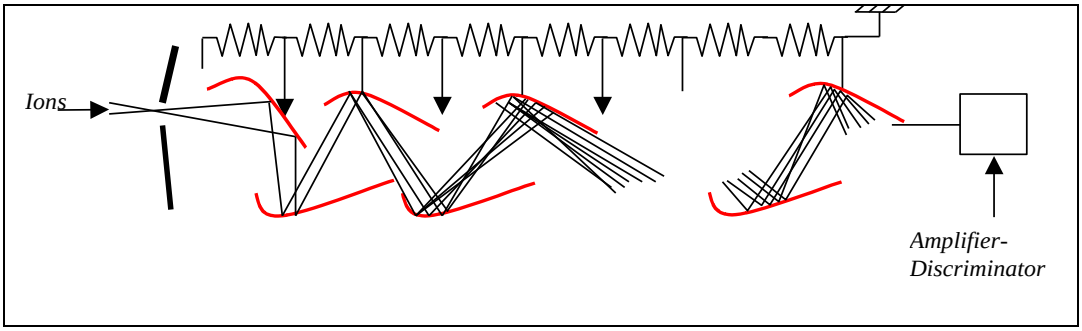


Figure 85: Illustration of an Electron multiplier

Due to the internal capacitance, it will take a certain “dead-time" for the EM & electronics to restore its gain and be ready to detect a next pulse, well separated from the previous pulse. It may occur that two or more ions impinge the EM first dynode within a time interval small enough to be detected as a single ion. The measured count rate is then lower than reality. This EM dead time effect can be corrected numerically as long as the dead time is known precisely. To ensure this on the NanoSIMS, the EM channel detection is by construction electronically “paralyzed” for a precisely fixed duration after a pulse is detected. This electronic deadtime is stored in the setup (see chapter 5.9.9.1).

2.1.4.3.1 EM output and discriminator threshold

The following definitions are useful to understand the electron multiplier language:

- The *ion/electron conversion efficiency* (*Np*)corresponds to the response of the first dynode. It is derived from the *P(k)* distribution law which gives the probability for one ion to produce *k* electrons. It is reasonable to assume that *P(k)* is a *Poisson* law where *Np* is the mean. *Np* depends on the first dynode local chemical surface composition and the incident ion characteristics: mass, velocity and nature (single or molecular).
- The *EM gain* is the ratio between the electron output current and the ion input current. It involves both the first dynode *ion/electron conversion efficiency* and the other dynodes amplification effect. This last amplification depends on both the *EM HV* and the EM age (chemical composition of the local surface).
- The pulse height distribution (PHD) is the curve showing the probability *P(V)* for an EM output pulse to have a given voltage amplitude *V*. Like the EM gain, it depends on both the first dynode *ion/electron conversion efficiency* and the other dynode amplification effect.
- The *EM Yield* is the ratio between the number of output pulses counted after the EM discriminator (see below) and the number of incident ions.
- The EM detection channel. The first electrons produced by the first dynode when impinged by an ion are amplified by the successive stages within the electron multiplier with a gain in the range of 10^8 (EM gain). As it is displayed on a PHD distribution curve, the pulses detected at the EM output do not have all the same amplitude (Figure 86). A preamplifier converts the charge pulses into voltage pulses and amplifies them. Then, a discriminator selects the pulses larger than a given *threshold*.

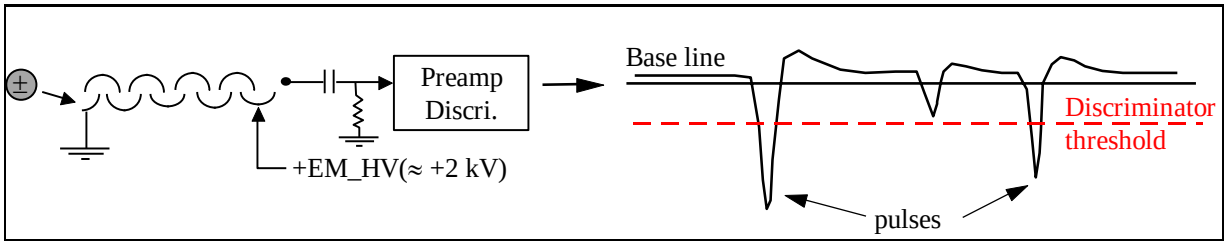


Figure 86: Pulses detected at the EM output

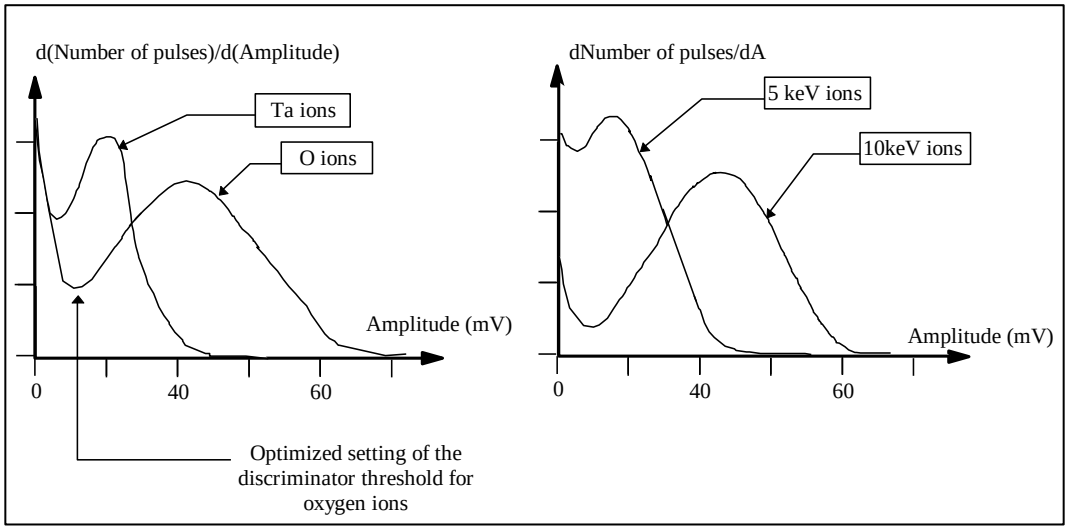


Figure 87: Typical PHD distribution curve

The large number of pulses with a small amplitude (first part of the pulse amplitude distribution) are due to the system noise. These pulses are therefore eliminated by using a discriminator with an adjustable threshold. The setting of the threshold is the result of an optimization which minimizes the *EM background* (typically < 5 counts/min) and optimizes EM detection efficiency (number of counted pulses per secondary ion).

2.1.4.3.2 EM aging

When an EM is getting older (meaning the EM has been used to detect ions), its gain (*output electrons per ion*) decreases (Figure 88), leading to a Y_{EM} decrease if the EM HV is kept constant. For recovering the original gain and yield, the EM HV must be increased.

The lifetime of an electron multiplier depends on the gain and the total number of ions counted (total integrated charge). Frequent high intensity measurements shorten the EM lifetime! Hence it is recommended to blank the mass analyzer during high current pre-implantation and to use minor isotopes when the signal of the major one is above a million c/s, especially for long depth profiles. Alternatively, if not in image mode, one can switch to FC detection.

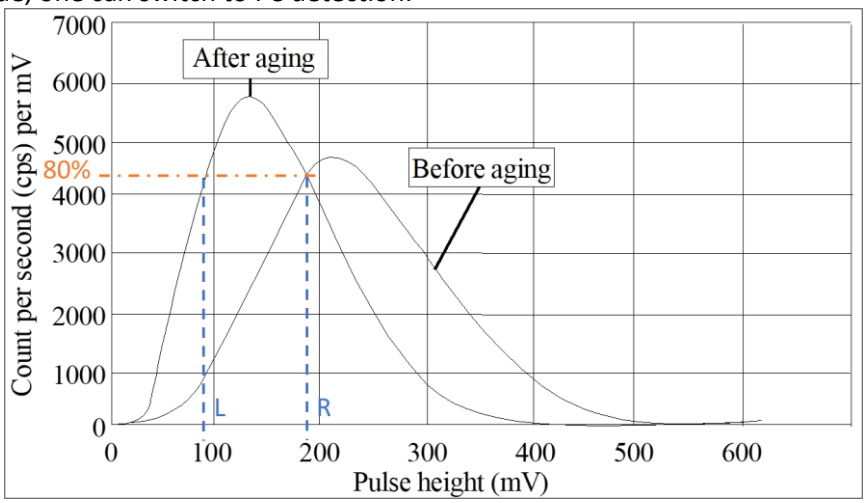


Figure 88: Discriminator threshold

Figure 88 shows the evolution of the pulse height distribution curve with use. It can be characterized by two parameters:

- the maximum of the PHD (Max_D, in mV here)

- The curve width, which can be translated by the ratio (R/L) between the pulse height at 80% on the right flank and the on the left flank of the curve as shown in Figure 88.

One can see that the gain decreases (the max of the distribution is moving to smaller amplitudes) and the width is changing. Overall, this is not good as more pulses corresponding to real secondary ions will fall under the threshold and not be counted. More quantitatively, Figure 89 shows the evolution of these two PHD parameters with time (= integral of received secondary ions) for $^{32}S^-$ ions. Secondary ion beam intensity was 1.4 x10^6 cps over 3 hours.

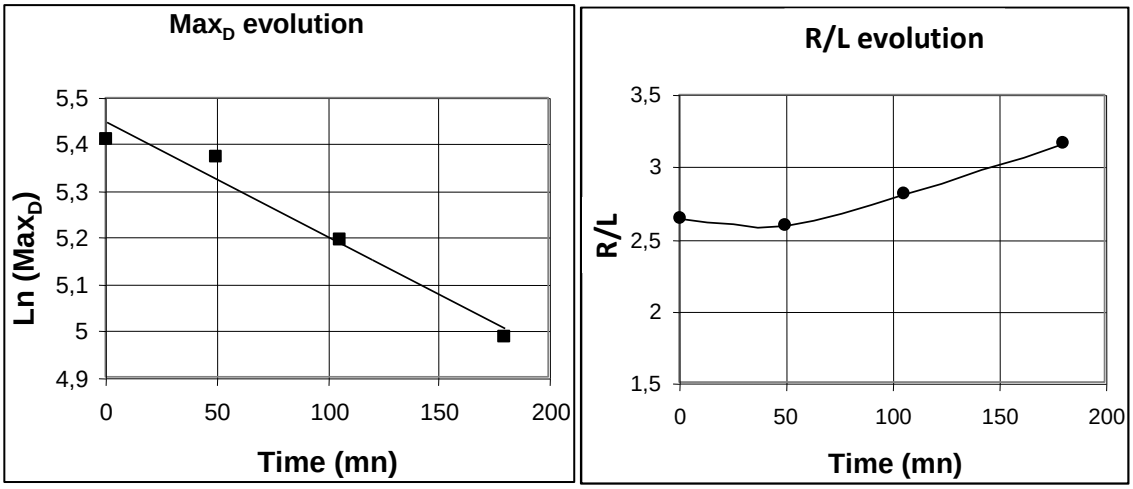


Figure 89: evolution of Maximum of the PHD and the R/L ratio parameters versus time for 32S- ions

As shown on Figure 90, the evolution of Max$_D$ with time can be expressed as:

$$Max_D \propto \exp\left(-t/\tau_D\right)$$

Figure 90: Max$_D$ equation

With t being the exposure time and τ_D the fitting parameter.

Figure 91 shows a comparison between two types of miniature electron multipliers. These two types of EM differ by the size of the dynode; the large one has a dynode surface larger by a factor 3. These measurements obviously show that:

- $1/\tau_D$ is proportional to the ion beam intensity.
- The aging effect has been reduced by a factor ranging from 5 to 22 thanks to the larger EM version

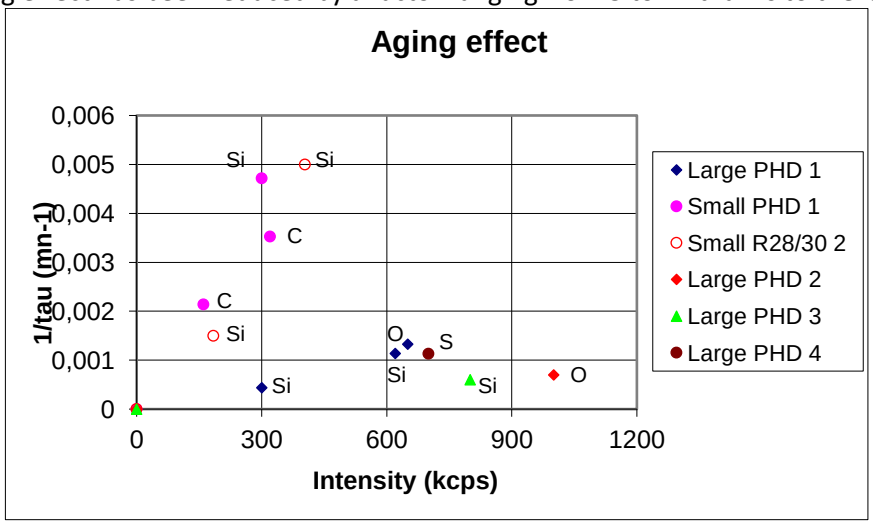


Figure 91: 1/τ Comparison between two EMs version

This aging effect will lead to a dramatic decrease of the detection efficiency with time and especially for high count rate. One way to estimate this effect is to measure an isotopic ratio with one abundant isotope and a very weak one. The two isotopes have to be recorded simultaneously with two different EMs.
The EM detecting the abundant isotope exhibits a change of its detection efficiency due to aging effect while the others remain unchanged.

The relative variation of isotopic ratio R can be expressed as:

$$\Delta R/R \propto \exp\left(-t/\tau_R\right)$$

Figure 92: The relative variation of isotopic ratio R expression

An empirical relationship between τ_D and τ_R has been established as following: $\tau_R = 1/20\ \tau_D$

Figure 93 summarizes the change in Silicon isotopic ratios before and after an EM has been aged. These experimental values are in good agreement with the above empirical formulae.

	Before aging	After aging
High voltage	1640 V	1670 V
MaxPHD	220 mV	175 mV
$^{29}Si/^{28}Si$	5,070 10^{-2}	5,025 10^{-2}
$^{30}Si/^{28}Si$	3,376 10^{-2}	3,339 10^{-2}
Change of Max PHD		-20,5 %
Change of $^{29}Si/^{28}Si$		-0,88 %
Change of $^{30}Si/^{28}Si$		-1,09 %

Figure 93: Change in Silicon isotopic ratios before and after an EM has been aged

In summary the Electron Multiplier gain decreases with the received ion dose. The loss of gain visualized by the reduction of PHD_{max} can be restored by increasing the HV. Before any precise measurement and regularly (each week), PHDs and discriminator levels must be checked.
For ultimate isotopic measurements with high count rate (5 x10^5 c/s) on the major isotope the drift of PHD_{MAX} will limit the long-term reproducibility. An automated PHD_{MAX} control with EM HV automatic adjustment routine is available (refer to 9.3.3.4) for cancelling this drift. It is generally necessary for maintaining isotopic ratio reproducibility better than the permil level when using EM with high count rate (above 1 E5 c/s).

The progressive **carbonation of the last dynode** (which sees the highest current flow) is a crucial factor for aging. This is why the multicollection vacuum is an important factor.

The local heterogeneity of the first dynode (original and sputtered areas) can contribute to a limitation of reproducibility, especially when using small slits (a very small secondary beam line moving between a damaged area to a fresh area of the 1rst dynode would change the overall detection probability).

For high precision measurements, before **using a new EM freshly replaced, pre-aging is necessary** to stabilize its gain (e.g. a few tens minutes at 5 x10^5 c/s).

2.1.4.3.3 The ion to electron conversion issue

The ion to electron device is the EM first dynode. Although the first dynode can become *locally* inhomogeneous after long exposure to focused mass line of high ion intensity, aging does not occur at the first dynode but at the last one, probably caused by contamination problems (poor local vacuum simultaneously with high electron current bombardment induces carbonation of this dynode). However, it

can be demonstrated that an improvement of the first dynode electron/ion rate should make the EM insensitive to the last dynode aging.

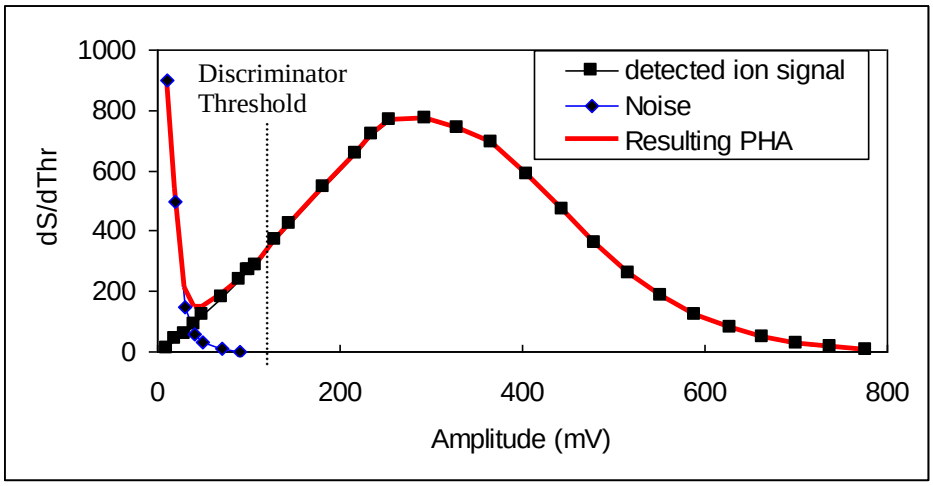


Figure 94: PHD curve

On Figure 94, the red curve is the PHD distribution as it can be displayed with our instruments. It results from the sum of the amplified ion signal and a noise consisting of short pulses. Slodzian et al. (2001)[1] has shown that from such a curve shape, just by assuming that the electron emission at each dynode was following a Poisson law, it can be deduced, for CuBe dynode EM, an ion/electron conversion efficiency of 9 and an electron/electron yield of some 2.5.

The issue is that for eliminating noise pulses, it is required to set the discriminator threshold at a level such as it cuts also several per cent of the useful signal, leading to a yield of some 93%, which is not constant if the EM gain varies because of aging. It should be highly desirable to have the hereunder PHD distribution curve (Figure 95).

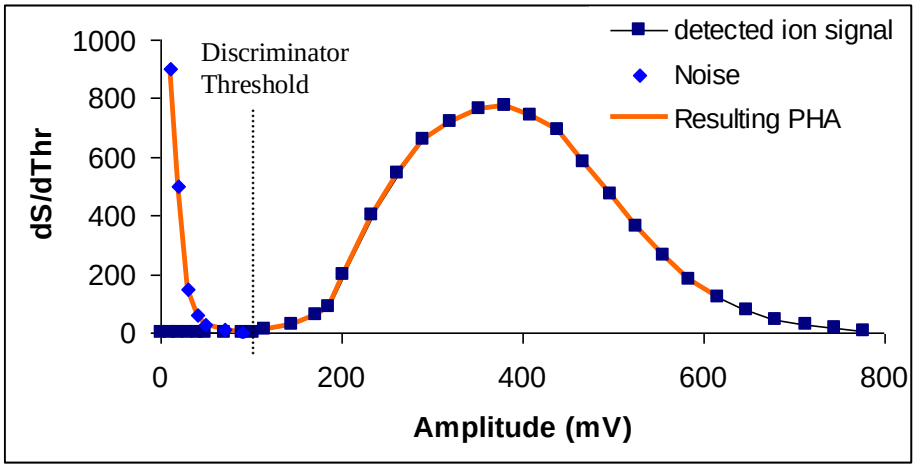
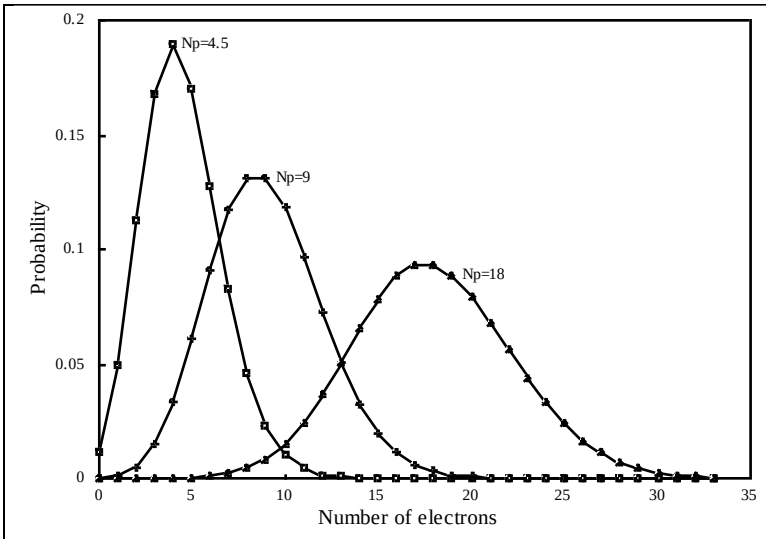


Figure 95: "ideal" PHD curve

Obtaining such an improved PHD from an EM would require increasing the ion to electron conversion efficiency probably through dynode material and geometrical optimizations.

[1] Precise in situ measurements of isotopic abundances with pulse counting of sputtered ions, G. Slodzian et al., EPJ, Appl. phys., 2001, vol. 14, no 3, pp. 199 - 231



Ion to electron Conversion:
The POISSON law Model

The curves on the left display the probability of electrons expected from the Poisson law for a mean efficiency of respectively 4.5, 9 and 18.

It can be expected that an improvement of the first dynode conversion efficiency improves dramatically the overall PHD curve

Figure 96: Poisson law model

The PHD distribution curve is dominated by the first dynode conversion efficiency but depends also on the next dynodes (mainly the next 2 dynodes with a mean yield of 2.5 each). Because the electronic discriminator cannot accept pulses larger than 1.5 Volts, let's assume that the pulse mean amplitude is always tuned at 300 mV and that the threshold level to eliminate all the noise pulses is 100 mV. From the simulation of the first 3 dynodes, it is possible to draw the probability that an incident ion leads to a pulse smaller than 0.33 of the mean amplitude. Hence increasing the ion/electron efficiency would lead to more ions detected.

1st dynode ion to electron conversion efficiency (Np)	Mean number of electrons emitted by the 3rd dynode (Np x 6.25)	Probability (number of electrons < 0.33 Mean)
4.5	28.125	12%
9	56.25	3.8%
18	112.5	0.38%

2.1.4.3.4 QSA Effects on Isotopic ratio measurements

QSA stands for quasi-simultaneous arrivals.
Secondary ions are often considered to be only a small fraction of the bunch of sputtered particles resulting from the impact of primary ions. However, the average number K of secondary ions ejected per primary ions may reach values as high as 20% for some elements. In such conditions, the probability to get more than one secondary ion emitted *per primary impact* is not negligible and those ions may arrive at nearly the same time on the conversion dynode of the electron multiplier *for high transmission instruments like the NanoSIMS*. QSA are registered as single pulses so that the registered number of counts is slightly lower than the true number of incoming ions.
Assuming a Poisson statistics, the correction factor is given in a first order approximation by:

$N_{cor} = N_{exp} (1 + K/2)(1)$

Where Ncor is the real number of ions reaching the first dynode and Nexp the number of pulses counted with a given threshold and K is the ratio secondary over primary.

In order to show the effect of QSA on isotopic ratio measurements, the ratio $^{34}S/^{32}S$ (figure 31) has been measured for different K. As K for ^{34}S is roughly 22 times lower than for ^{32}S, the effect of QSA on 34S can be neglected. Thus, the experimental Sulfur isotopic ratio R_{exp} must vary with K according to:

$K_{cor} = K_{exp} / (1 - K_{exp}/2)$

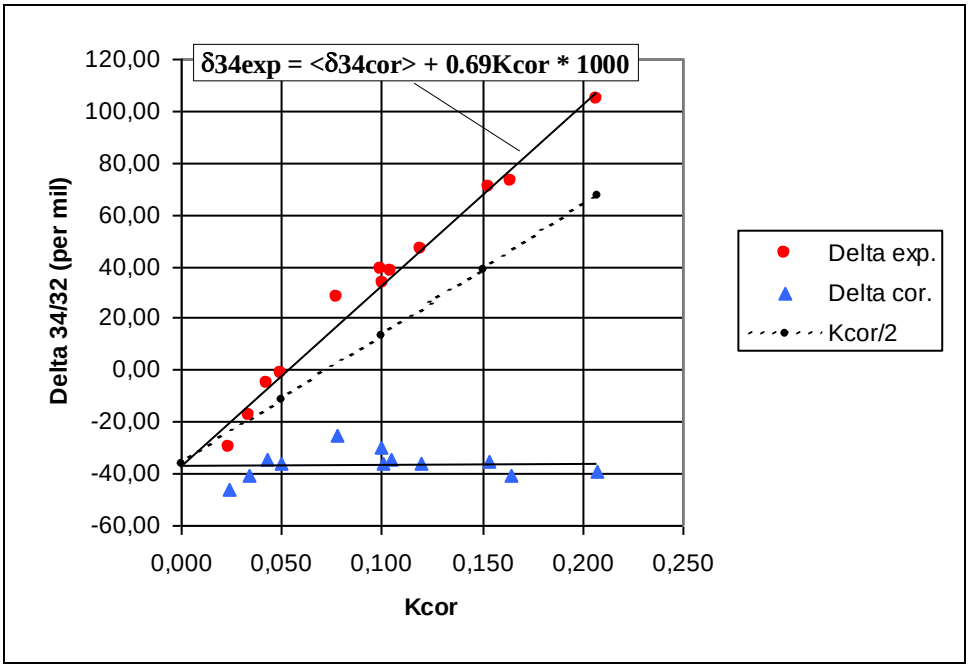


Figure 31: QSA on Pyrite (Primary ion Cs^+, 1 pA)

If <δ34cor> represents the corrected relative deviation of 34S/32S ratio, the linear relation (Figure 31) writes down:
δ34exp = <δ34cor> + 0.69 Kcor * 1000

This experimental coefficient, 0.69 instead of being 0.5, is obviously different from the value given by relation (2) obtained from Poisson statistics. It might be due to the inadequacy of Poisson statistics to describe the phenomenon or to other effects such as fractionations due to differences in ion selection generated by the change of K. Further investigation needs to be done with measurements coupling Faraday cup and EM and on different elements.

For further details see: G. Slodzian et al. / Applied Surface Science 231–232 (2004) 874–877

2.1.4.4 Faraday cups (FCs)

As explained above electron multipliers are aging proportionally to their total integrated counts. To preserve their lifetime, one should keep the EM count rates below a few millions c/s. At such count rate, a dead-time correction is mandatory. For the highest reproducibility of isotopic measurements, the count rate should be about 5 $x10^5$ c/s maximum and at this rate, one should use the continuous PHD automated adjustment routine to handle EM aging over long measurements (chapter 9.3.3.4).

For higher count rates and/or better isotopic reproducibility, it is best to switch the detectors to **Faraday Cups and electron current pre-amplifiers**. Note that these positive aspects are balanced by their long response time precluding fast scanning imaging or electronic gating window, accessible only with EMs.

On the NanoSIMS, it is necessary to mechanically switch between EM and FC on each trolley. On recent NS50L versions, the switch can be automated while the multicollection chamber stays under vacuum. The only exception is the 7th detector, which is fixed and still requires a manual switch. The multicollection chamber must then be opened to atmospheric pressure (See chapter 10.4.2 for switching detector #7 or for NS50/L not equipped with switch automation).

On an EM used in pulse counting mode the statistical uncertainty will be given by Poisson statistics:
Uncert = 1/ sqrt (N). e.g. at 5 $x10^5$ c/s with 80s integration the uncertainty will be: 1/ sqrt(5 $x10^5$ * 80) = 0.35 $x10^{-3}$ (= 0.35 permil 1s). Of course, other factors will play on the final isotopic reproducibility.

On a FC one let the secondary ion current flow through a huge resistance (10^{11} ohm on the NS50L). The current is measured as a voltage across the resistance, converted in frequency (V/F conversion) which is measured. Any minor variation of the resistance through temperature or surface contamination will lead to variation of current and background noise. The resistances and the pre-amplifiers are located in a thermostated container above the multicollection flange.

On the NS50L, the background noise of a FC with a 10^{11} ohm resistance is given as 5 $x10^{-16}$ A, measured over 5s. Noise, count rate and integration time will determine the incertitude for short term measurements (long term baseline drift must be controlled also): the basic rules applies: I = n*q / t e.g the equivalent count rate of the FC background noise is: n = 5 $x10^{-16}$ / 1.6 $x10^{-19}$ = 3 100 c/s.
This noise will be reduced when integrated over a longer time *t* instead of 5s, following sqrt(*t*/5).

- Case 1: with 5 $x10^5$ c/s over 80s, the relative incertitude will be: [3100/ sqrt(80/5)] / (5 $x10^5$) = 1.5 $x10^{-3}$ = 1.5 permil. It is poorer than with EM as above.
- Case 2: with 1 $x10^7$ c/s (not possible with EM) over 80s, the incertitude will be: 0.75 $x10^{-4}$ = 0.07 permil, which is much better than with EM (other factors will limit the overall isotopic reproducibility to a few tenth permil on the NS50L).

2.1.5 The optional Normal Incidence Electron Gun (NEG)

2.1.5.1 Electrical Charging Effects

During SIMS analyses, positive, negative or neutral primary particles impinge the sample surface and either positive secondary ions or negative secondary ions, and secondary electrons, leave the sample surface.
As the “secondary ions/primary ions” and “secondary electrons/primary ions” ratios are not equal to 1, an excess of charge will occur over the sputtering area of insulating samples. If the sample has an intrinsic conductivity, or at least a surface conductivity (through a metallic coating for example), the excess electrical charges can flow towards the conductive sample holder and the potential of the surface will be kept constant. On the other hand, if the sample is an insulator, the electrical charges accumulate on the sample surface and the potential surface is modified positively or negatively, depending on the sign of charges appearing over the sputtering area.

POSITIVE SECONDARY ION MODE: In order to collect positive secondary ions, the sample is brought at a positive potential (e.g. +8 kV). In that case, only positive secondary ions can escape from the sample surface and leave behind (via surface and depth collision cascade) negative charges (secondary electrons and negative ions being trapped on the sample surface by the strong extraction field). The number of charges Q+ appearing on the sputtered area per incoming primary particle is therefore given by the relationship:

$$Q+ = q_p + q_s * Y^+$$

where:
q_p is equal to 0 or neutral, +1 for positive and -1 for negative primary particle (only single charged primary ions are considered).
q_s is the sign of the charges left behind by the secondary ions.

Y^+ Is the yield for positive secondary ions/primary particles. This yield is always less than 1.

This equation shows that for the use of **positive primary ions** (not available on the NS50)**, a positive charge** is always building up on an insulating material and for **negative primary ions** (on the NS: O^-, O_2^-, O_3^-) or neutral (FAB), it is a **negative charge**. In fact, to give a complete description of the phenomena occurring in negative mode, secondary electrons and ions coming back from the front plate of the immersion lens should be also considered, but, in first approximation, they can be neglected. It should be noted that the use of polyatomic primary ions (e.g. O_3^-) is favorable in term of charging (at the cost of a lower ion source brightness i.d. reduced beam density and ultimate spatial resolution).

NEGATIVE SECONDARY ION MODE: In order to collect negative secondary ions, the sample is brought at a negative potential (e.g. - 8 kV). In that case, negative secondary ions and secondary electrons can escape from the sample and leave behind positive charges where the positive secondary ions are trapped on the sample surface and in the collision cascade volume by the extraction field. In positive mode, equation 1 therefore becomes:

$$Q- = q_p + q_s (Y- + Y^e)$$

where:
Y- Is the yield for negative secondary ions/primary particles, which is always less than 1.
Y^e Is the yield for secondary electrons/primary particles, which is always more than 1.

This equation shows that for **negative secondary** ions whatever the primary particles are (on the NS50: Cs^+, O_2^+), a **positive charge** is building up over the sputtered area. The following table summarizes the different cases which can occur for an insulator analysis:

	Secondary Ions	Primary Ions	Charging Up
1	+	Neutral	< 0
2	+	—	< 0
3	+	+	< 0
4	—	Neutral	> 0
5	—	—	> 0
6	—	+	> 0

The above shows that depending on the experimental conditions (polarity of secondary ions), the sign of electrical charging can be either positive or negative. If no charge compensation is carried out, the potential of the sputtered area will exceed the nominal value of 8kV and, therefore, no secondary ions will be collected since the mass spectrometer is adjusted to analyze ions accelerated under 8 kV.

In order to keep the surface potential of the analyzed area constant when a positive charge occurs, a flood of low energy electrons (secondary electron yield < 1) may be used for charge compensation. But, for negative charging effects, no charge compensation is possible by using an electron gun.

However, it must be noted that the use of neutral or negative primary particles for positive secondary ion analysis enables one to perform SIMS analysis of insulators, even if negative charge builds up. As a matter of fact, low density of neutral or negative primary beam allows one to reach **a steady state** with negative charging corresponding to **a few** tens of volts, which may be compensated by applying an **offset on the sample holder**.

IDENTIFICATION OF CHARGING EFFECTS: When an analysis is performed on an insulating material, it is important to know if the charge compensation is optimum. Of course, a high voltage sample breakdown is

an obvious indication of incorrect charge compensation. However, sometimes there is no HV breakdown but charging effects are present.
One way to verify if there is charging effects is to check the energy slit position and to compare it with the standard position on a conductive sample.
Another criterion to identify charging effects is the shape of the secondary ion energy distribution. On a conductive sample when a narrow energy slit (a few eV), centered on the peak intensity of the energy distribution, is mechanically pulled towards the low energy ion side, the secondary ion intensity decreases to zero for a small shift (corresponding to 5-10eV). On an insulating material, when there are charging effects, the energy distribution is deformed, and even by pulling the energy slit over the complete range, no sharp decrease of the secondary ion intensity can be reached.

Scanning EOW can help to see the seriousness of charging. For moderate charging on homogeneous samples adjusting slightly EOW can solve the problem.

Using O_2^- or O_3^- instead of O^- can help reduce charging for positive secondary ion mode but will reduce the primary beam current; it is effective for “large” spot size but for very small spot size it might become unusable.

Solving other charging will generally require **coating the sample** (***before mounting*** in the hole to ensure good contact with it !) with a film of 10-20nm of e.g. platinum, carbon or others and/or the use of electron flooding.
Controlling the electrical conductivity (< 1ohm) between the coating and the sample holder, using an ohmmeter, is mandatory after sample mounting in the sample holder.

For homogenizing the charging and easier charge compensation, **scanning the beam with a high scanning rate (tens of µs/pix) and acquire many cycles will always be favorable compared to a slow scanning (e.g. 50ms/pix).** The drawback is, in imaging mode, to result in files containing hundreds of cycles but they can be accumulated in WinImage.

2.1.5.2 Normal incidence Electron flood Gun (NEG) description

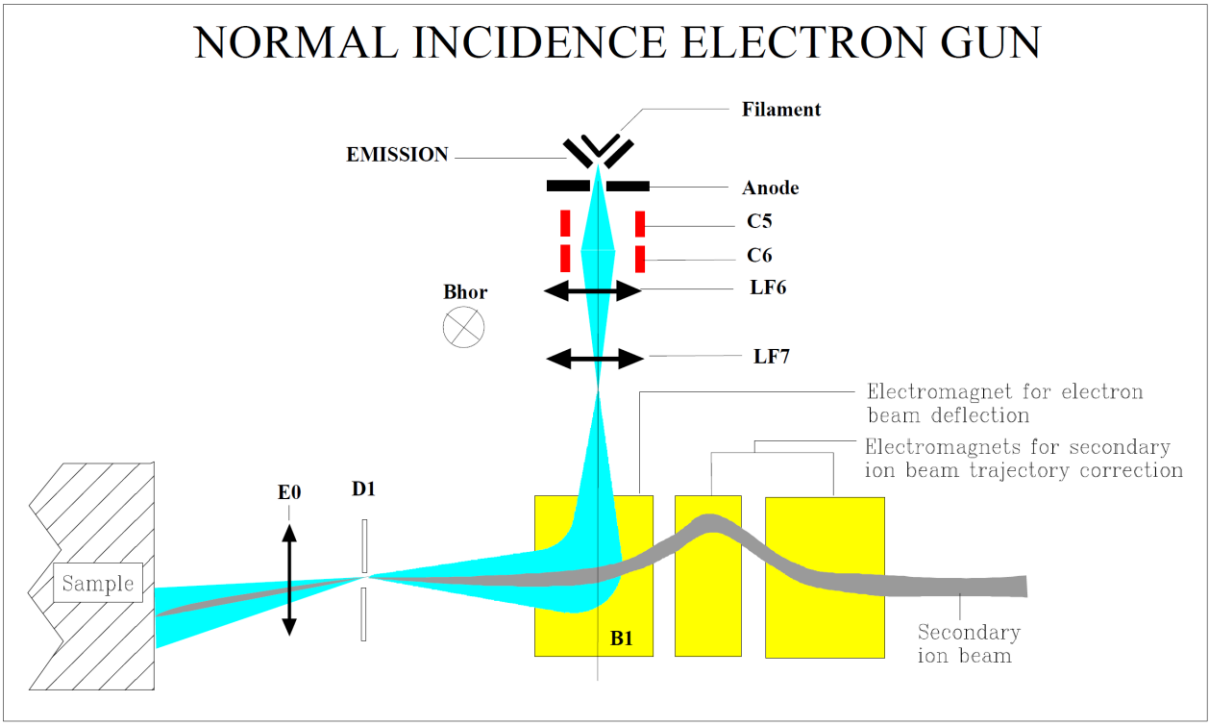


Figure 97: description of the Normal incidence Electron Gun (NEG)

Figure 97 shows a schematic drawing of the normal incidence electron gun. The electron source is a tungsten filament which can be brought to a potential adjustable between 0 and -10kV and a Wehnelt. The optical column of the electron gun is composed of two slit lenses, two sets of deflectors (in X and Y) and a magnetic sector (B1). This optical system is adjusted to form an **image of the e-gun cross-over in the plane of D1.**

Two other magnetic sectors are required to compensate the deviation of **secondary** ion beam undergone in the B1 sector. The shape and the size of these two sectors have been determined in order to compensate the deviation in B1 to the first order. A fine tuning of this compensation is available by using Bhor (vertical coils).

In positive primary ion mode, making use of the reversibility principle, it is clear that incoming e- will follow the same trajectories than the secondary ions. And, if electrons go through the crossover plane on the secondary optical axis, they will arrive on the sample surface with a normal incidence and energy close to zero since the potentials of the filament and the sample are the same.

The practical use of the NEG and its tuning are described in chapter 9.3.2

2.1.6 The Total Ion Current detector

At the end of the matching optics (Figure 98), the beam can be directed toward an additional detector, the Total Ion Current detector (TIC). This detector is an electron multiplier (EM) identical to the EMs in the multicollection chamber (see chapter 2.1.4.3 for details).

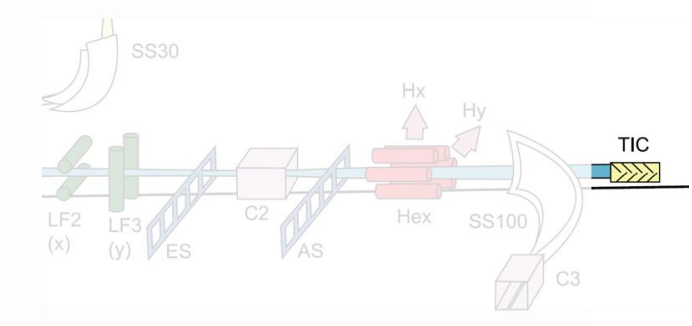


Figure 98: position of the Total Ion Current detector (TIC)

In TIC mode, the secondary beam is directed toward the TIC detector by turning LF2 and LF3 lenses as well as electrostatic sector SS100 to zero. Since this EM is located before the mass spectrometer, it receives the entirety of the secondary beam and it is much less dependent on the sample composition compared to mass-filtered images. It is therefore very useful during tuning and troubleshooting, and the signal is usually quite strong. It might also be used for some normalization process but by definition it is not possible to acquire a TIC image simultaneously with mass-filtered images.
For details on how to use the TIC detector see chapter 9.1.8.

2.1.7 The optional Secondary electron detector

Under primary ion bombardment there is an emission of secondary electrons. If the sample holder is negatively polarized (= when using **Cs^+** or O_2^+ primary ions), these electrons escape from the sample surface and can therefore be collected, similarly to negative ions. A scanning electron image induced by the primary ion bombardment can therefore be formed. The ratio e-/primary ions being much higher than the ratio secondary ions/primary ions, scanning electron images often offer excellent signal to noise images.
Note, though, that the topographical and shadowing contrasts, so useful in SEMs, are reduced on the NanoSIMS because of its normal beam incidence and strong co-axial, normal SE extraction. Nevertheless, ion-induced SE images on the NanoSIMS are extremely useful to localize quickly the smallest objects like oxide particles with high SE yield, cracks, grain boundaries, cells grown or filtered on a flat surface, etc.
Secondary electrons are accelerated in the secondary ion extraction space, through the co-axial lens. After dynamic transfer plates B3, the Be coil used for the e-gun can also be used to deflect in the opposite direction the secondary electron beam towards the electron detector fitted underneath the collection optics of the instrument.
This detector is an Everhart-Thornley detector, made of a scintillator to convert electrons in photon in combination with a photo multiplier (Figure 99). Working in pulse counting mode similarly to the EMs of the mass spectrometer, the obtained image can be displayed via the real time imaging or acquired as an image, just like any ion images of the multicollection.

➔ ***Note that SE detection is only possible without the electron flood gun (NEG).*** Otherwise the strong light emitted by the NEG filament in front would saturate the photomultiplier (PM) and a security on its max count rate will switch off the PM HV above 1 E7 count/sec.

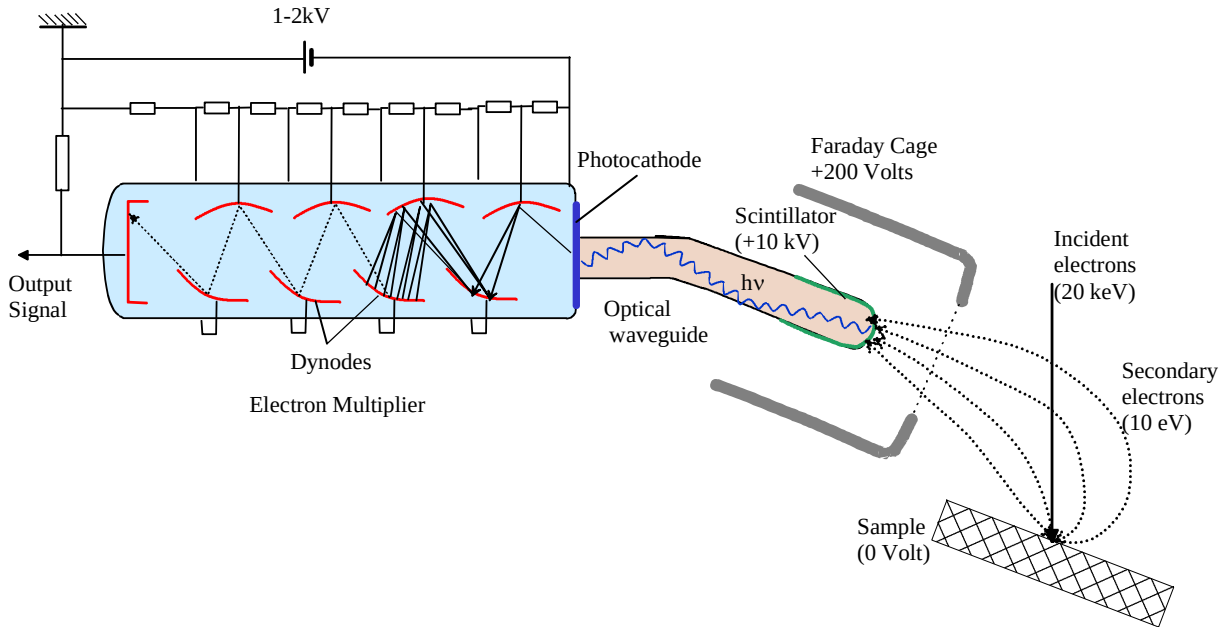


Figure 99: schematic of a generic Everhart-Thornley secondary electron detector in a SEM

2.2 Electronics

The CAMECA NanoSIMS 50L electronic is divided in four different parts:

- Integrated electronics inside the physics chassis which controls the lens high voltages and the communication between the computer and the instrument.
- The “cabinet A”, which controls the power supply, the vacuum (automation, pumps and a part of low voltages.
- The “cabinet B”, which controls motorizations, the Cs+ and electron source supplies, the rest of low voltages, the NMR, the magnetic field supply, the keyboard and the vacuum gauge displays.
- The RF-Plasma electronics.

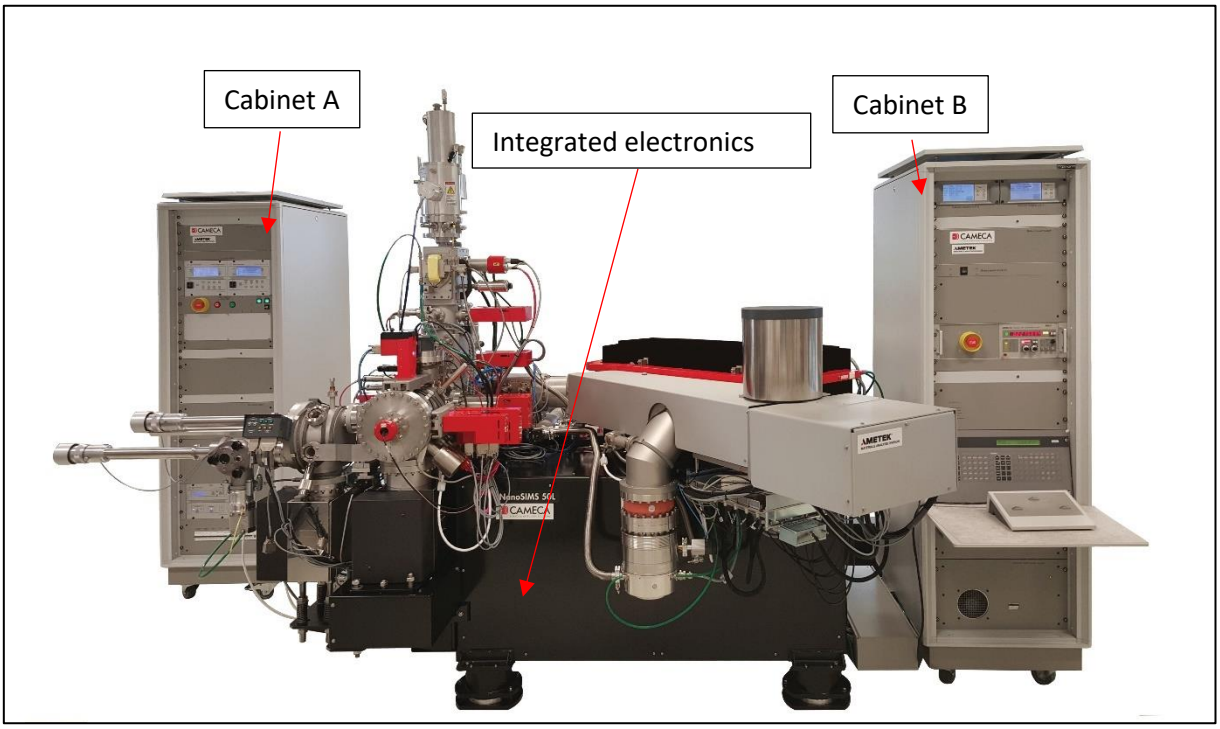


Figure 100: Three main parts of the electronics

2.2.1 Integrated electronics

The integrated electronics includes:

- the real-time unit which is the link between the computer and the NanoSIMS instrument. Furthermore, all the signal acquisition, scanning and keyboard values go through this unit.
- The lens high voltages supplies are dedicated to the following elements: P1, P2, P3, P4, EOS, EOP, EOW, L4, LF2, LF3, LF4, LF5, LF6, LF7, L0, L1,, LCs, L3, SS30, EMs, L_{duo}, SS100, ESAs.

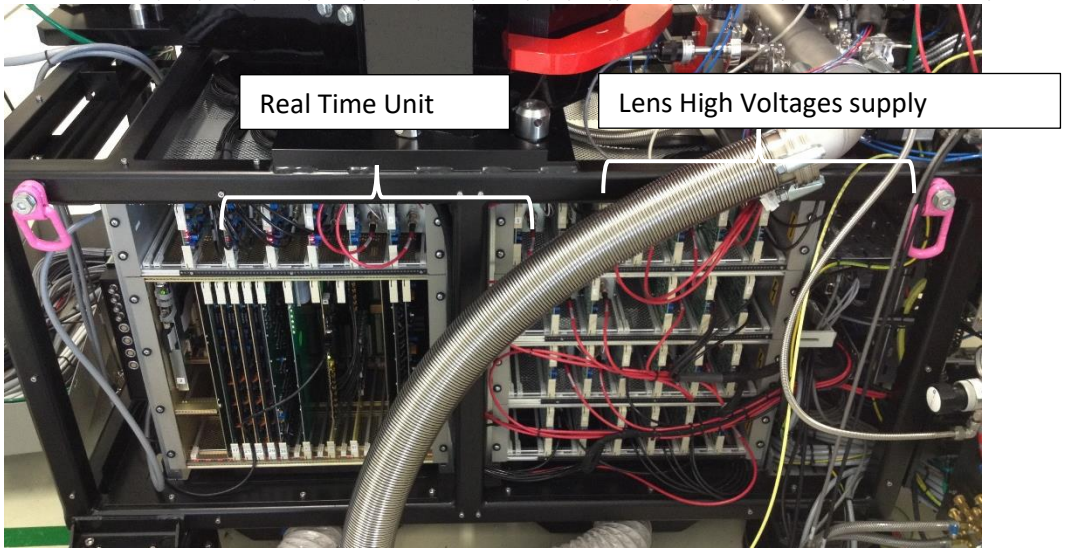


Figure 101: rear view of the integrated electronic inside the physics chassis

2.2.2 Cabinet A

The “cabinet A” (Figure 102) controls the power supply, the vacuum (automation and pumps), a part of low voltages supply and the coils supply.

Vacuum automation

Ion pump controllers (and turbo pumps controllers at the rear)

Electronics and EMO supply

Coil supply

Low voltage power supply 3 (HV unit supply, camera light power supply)

Low voltage supply 1 (Real time unit)

Ti sublimation

General power

Figure 102: Description of the cabinet A

2.2.3 Cabinet B

The “cabinet B” (Figure 103) controls motorizations, the Cs+ and electron source supplies, the other part of low voltages, the NMR, the magnetic field supply, the keyboard and the gauge displays.

Figure 103: description of the cabinet B

2.2.4 The RF-Plasma electronic unit

The RF-Plasma source electronics (Figure 104) is located at the rear of the instrument or under the transfer rods. This small cabinet is composed of a command system which allows a communication between the source and the computer, a RF generator and a cooling unit. The RF generator unit is, generally, not controlled from its front panel as it is controlled from the computer.

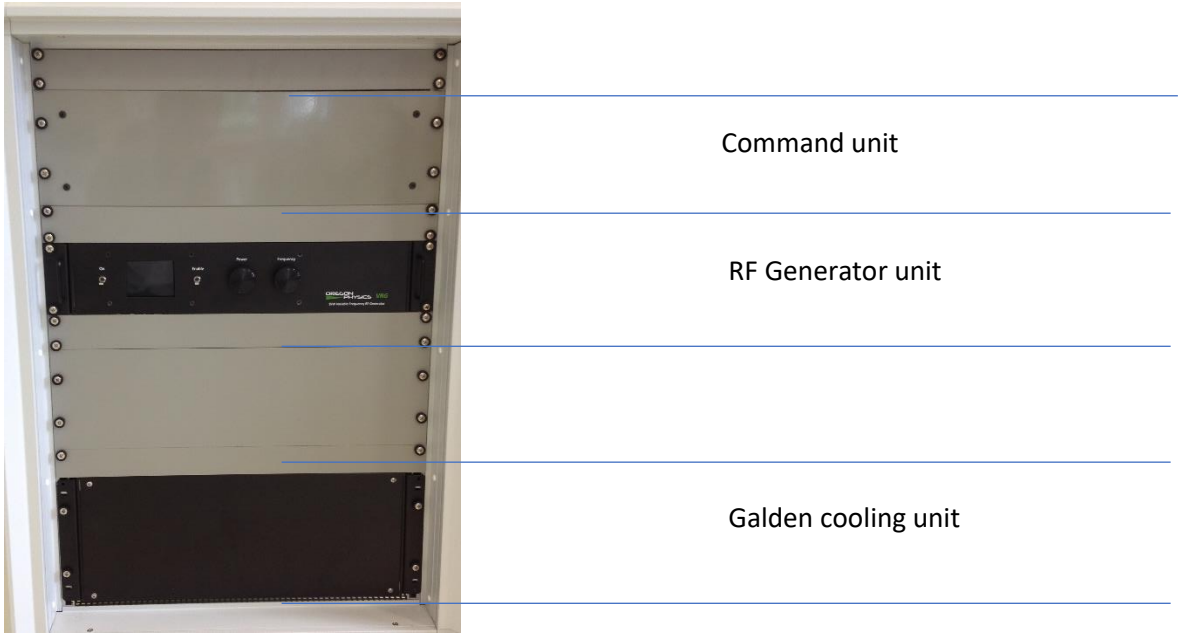
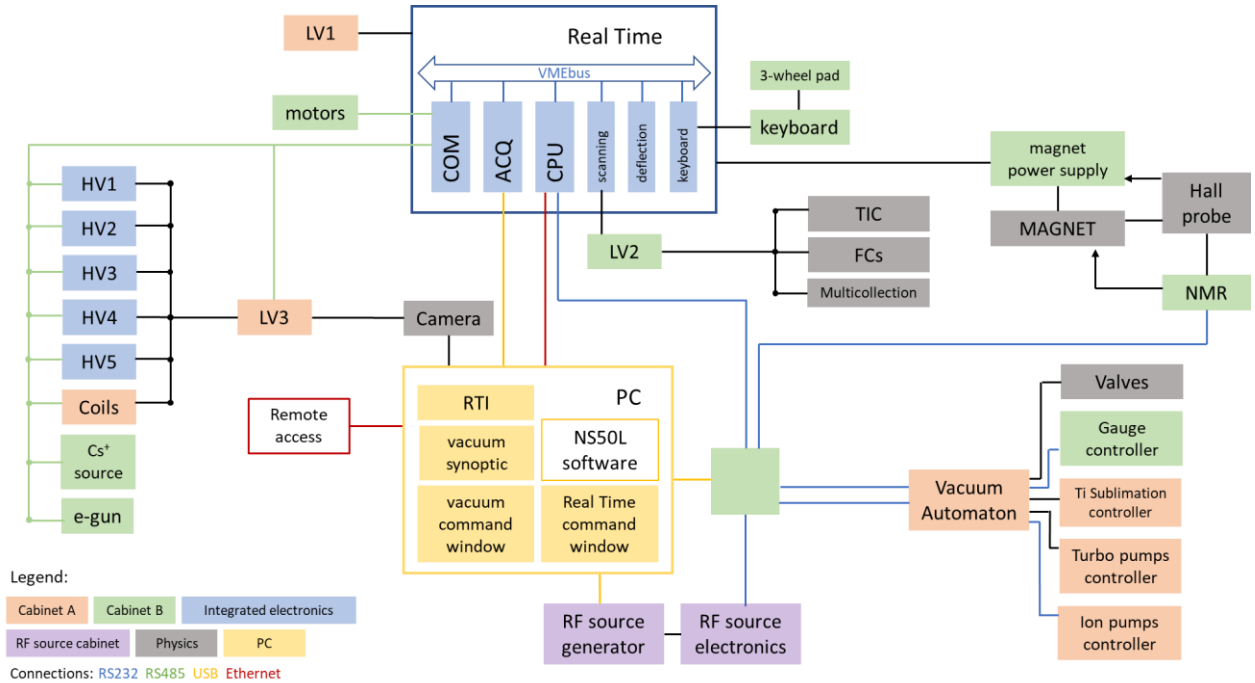


Figure 104: RF-Plasma cabinet configuration

2.2.5 Electronics interconnection schematics



Acronyms: HV: high voltage; LV: low voltage; TIC: total ion current detector; FCs: Faraday cup detectors; NMR: nuclear magnetic resonance B-field probe; RTI: real time interface; PC: personal computer.

Figure 105: electronics interconnections

This schematic shows that the PC can be turned off, as well as all electronics, while the vacuum system can stay on independently, as long as the vacuum automaton and vacuum devices are powered.

2.3 Fluids and Vacuum

2.3.1 Gas interconnexions (compressed air, dry N2, pure O2)

The Figure 106 below shows the gas interconnexion (compressed air, high purity oxygen, dry nitrogen) with valves, turbomolecular and primary pumping.
Ion pumps and Ti sublimator pump are not represented. The compressed air blue pipes show their numbered connection to the pneumatic panel and their length.

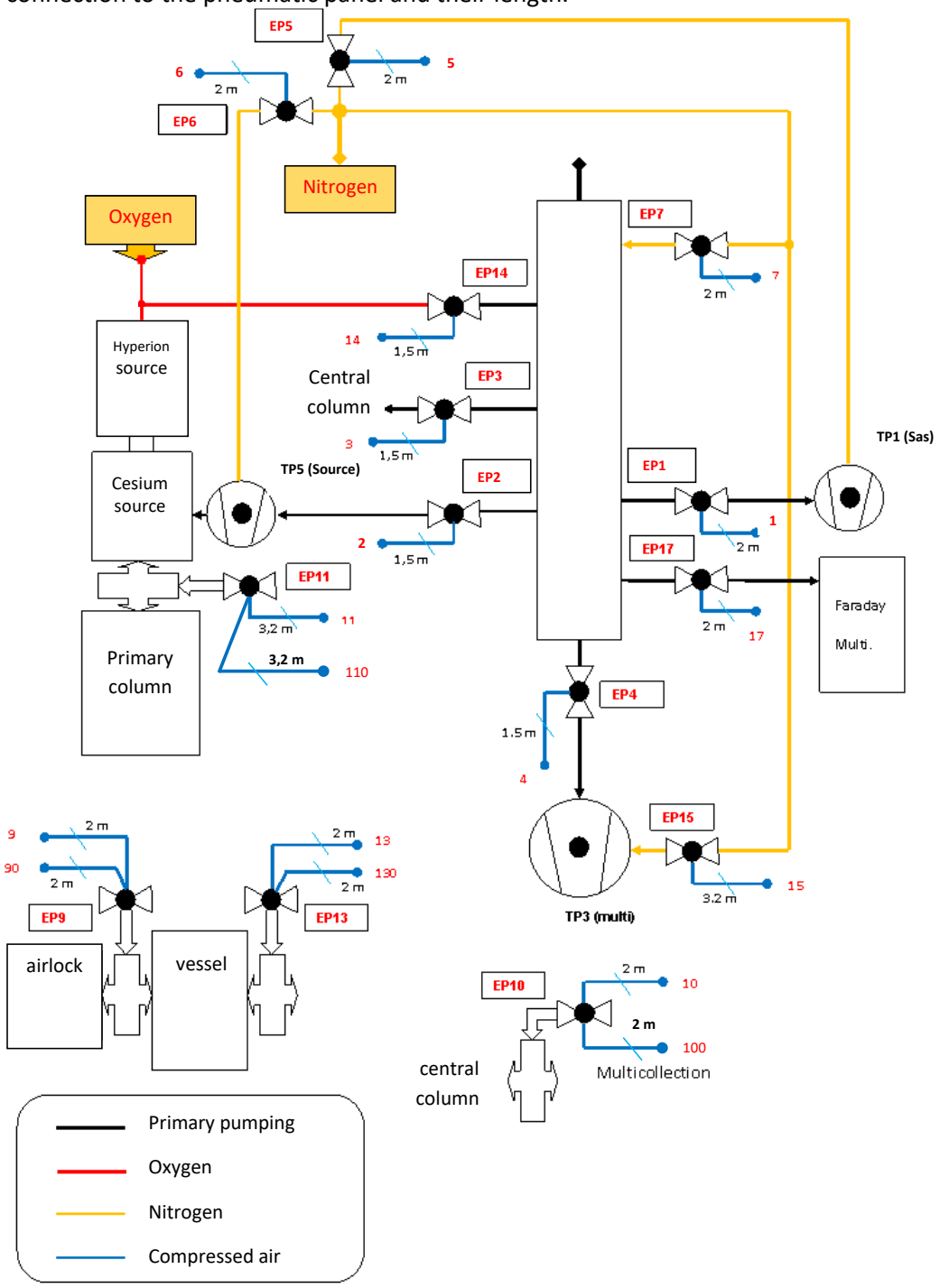


Figure 106: Interconnection of rough pumping, valves, compressed air, O2 and N2 gases.

2.3.2 Pumping & vacuum system

Pumping of the instrument is insured by several types of pumps (Figure 107). The reference names of pumps and vacuum gauges are the ones used in the vacuum synoptic software (Figure 108 and see chapter 5.10).

Chamber	Pump	Pump type	Vacuum Gauge	Vacuum Gauge
Primary line	PM	Agilent TPS Compact combining a TwisTorr 74 FS turbomolecular pump, backed by the Agilent 60 L/min IDP-3 dry scroll primary pump	TC 1A	Thermocouple, range : 10^{-3} mbar to atmosphere.
Load-lock	TP1	Turbomolecular Agilent TwisTorr 84 FS turbomolecular pumps (67 l/s N_2)	UHV 1B	UHV24 Bayard-Alpert range : 10^{-10} to 10^{-3} mbar
Storage vessel	IP3	Agilent diode ion pump Vaclon Plus 150 (150 l/s)	UHV 3B	
Analysis chamber	IP1	Agilent diode ion Vaclon Plus 300 (300 l/s)	UHV 1A	
		Titanium sublimator, 3 filaments		
Central column	IP2	Agilent diode ion Vaclon Plus 300 (300 l/s)	UHV 3A	UHV24 Bayard-Alpert range : 10^{-10} to 10^{-3} mbar
Ion Source chamber	TP5	Turbomolecular Agilent TwisTorr 84 FS turbomolecular pumps (67 l/s N_2)	UHV 2B	
Multicollection chamber	TP3	One TV551 SEM turbomolecular pump (550 l/s N_2)	UHV 2A	

Figure 107: summary table of all pumping and vacuum elements.

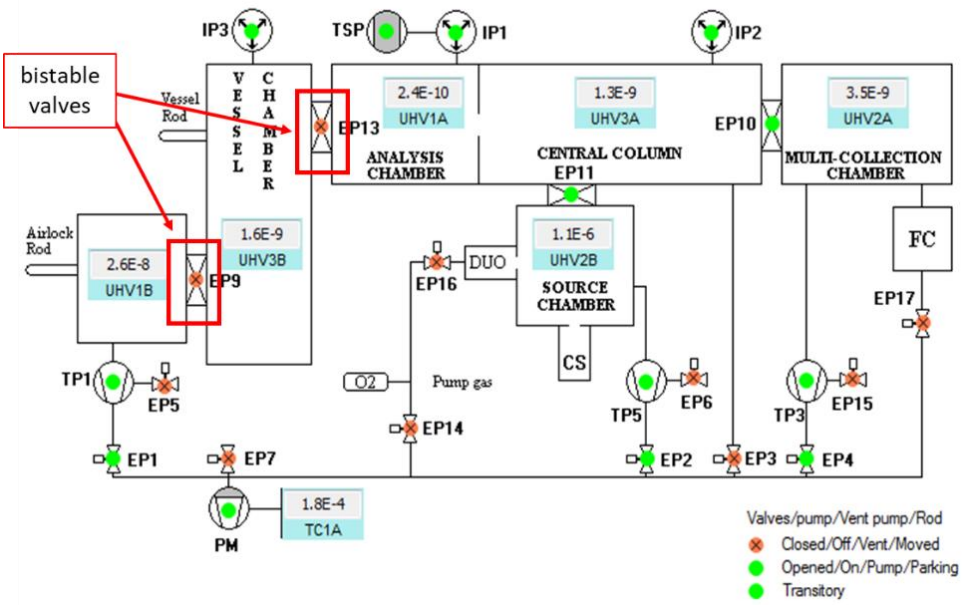


Figure 108: vacuum synoptic showing location of all the pumps, valves and gauges.

2.3.3 Compressed air

Compressed air is used to control all the pneumatic valves of the NanoSIMS 50L. The COMPRESSED AIR circuit is recognized by its **BLUE** plastic tubes. All information goes from the vacuum unit to the valves *via*

the pneumatic valve panel (Figure 109) sending commands to all valves. **All valves are monostable, except for EP9 and EP13** (between airlock and vessel chamber, and vessel chamber and analysis chamber, respectively) which are bistable. **In case of a drop in compressed air pressure, the monostable valves will automatically close**, keeping the system under vacuum, while the bistable valves will remain in their position, avoiding them to potentially close on the transfer rods (Figure 110).

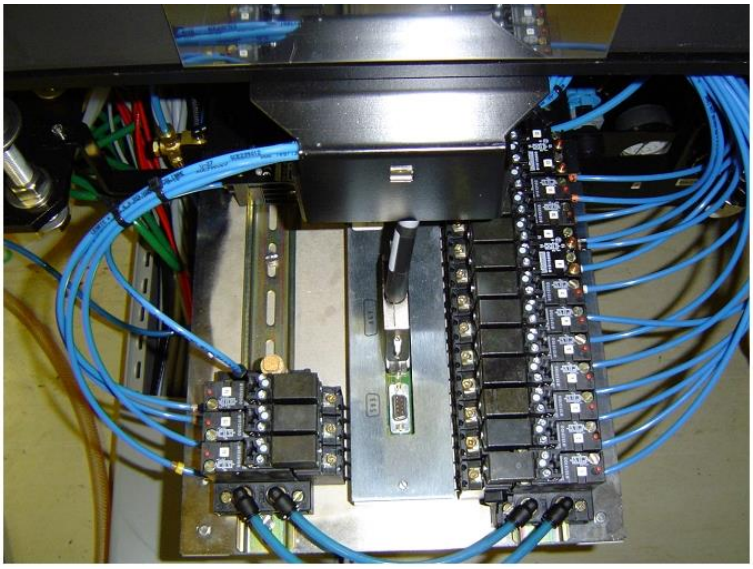


Figure 109: Pneumatic valve controller panel. Compressed air is distributed via the blue pipes.

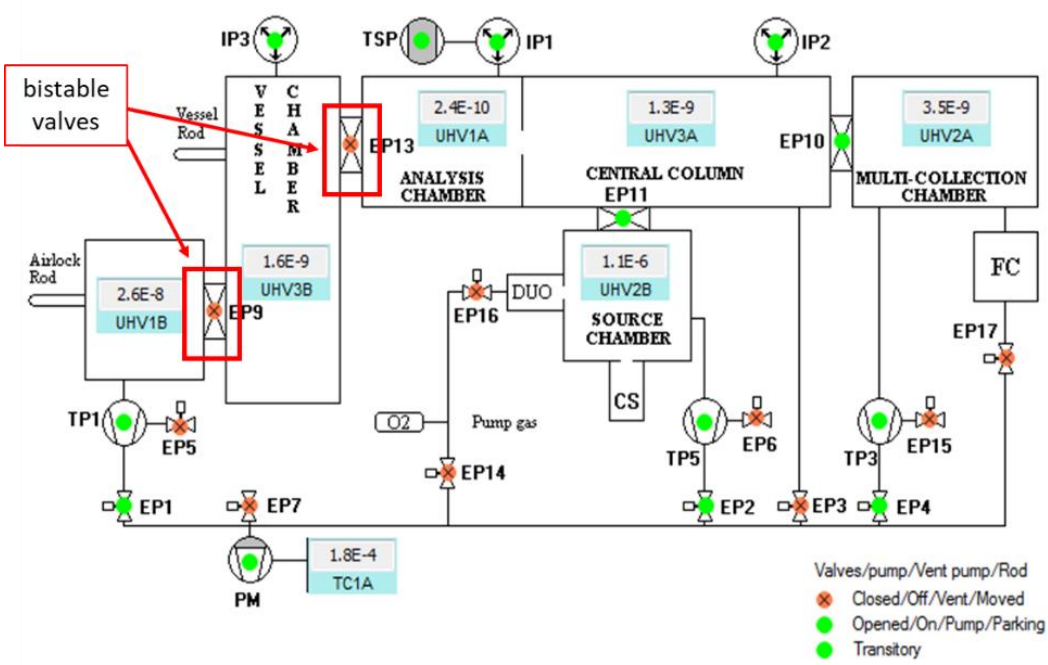


Figure 110: vacuum synoptic showing the position of all the valves.

All valves of the above synoptic are noted EP (for electro-pneumatic). They all are monostable, except for EP9 and EP13, which are bistable.

Compressed air is also used for the antivibration suspension system below the feet of the instrument. Refer to maintenance chapter 10.11 on how to adjust the of the antivibration system.
The required compressed air pressure at the entrance of the CAMECA NanoSIMS 50L is 7 bars. This high-pressure level is especially crucial for the antivibration suspension system below the feet. A limitator on the pneumatic valve control panel keeps the compressed air for the valves at 5.5 bars.

CAMECA offers an air compressor in the list of NanoSIMS accessories. Note that it is wise to power this compressor on the UPS if it is available.

2.3.4 Purified Nitrogen gas

The circuit of purified NITROGEN, used for venting the chambers, is based on **YELLOW** pipes. They are connected on the three turbo pumps and the roughing pump in order to vent the instrument.
Purity of nitrogen must be of at least 99.998%.

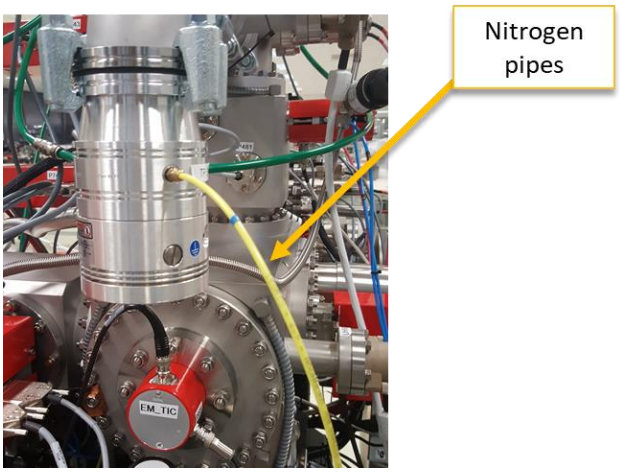


Figure 111: nitrogen pipes are yellow

2.3.5 Purified Oxygen gas

The oxygen is only used for the RF-Plasma source. Purity of oxygen must be of at least 99.998%. A Wien filter will additionally select the primary ions of choice between the ions generated by the source.

The RF-Plasma leak valve is directly connected to the oxygen bottle with a stainless-steel flexible pipe. A manometer attached to the Oxygen bottle lets 0.5 mbar into the flexible pipe. This pipe can be pumped/purged by the primary pump through valve EP14.
A manual leak valve (grey knob, EP16) located at the top of the RF-plasma source allows the user to adjust the Oxygen pressure in the source (it should be of the order of 10^{-5} mbar, when open).
See 2.1.1.1.4 and 9.1.2.2 for details on the RF-plasma source and how to operate it.

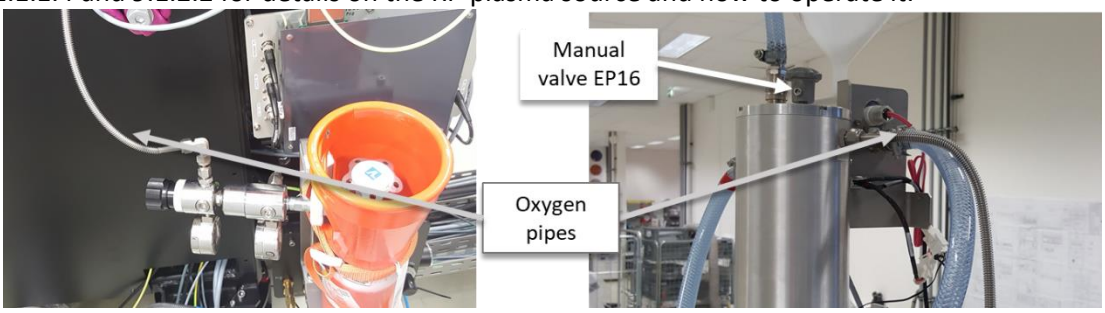


Figure 112: the oxygen pipe is a stainless-steel flexible pipe.

2.3.6 Cooling fluid circuit (water, Galden)

The NanoSIMS requires a circuit of cold water for cooling. It is generally coupled with a water chiller, in charge of evacuating calories whether in the air (water/air) or in another water circuit (water/water).
If the chiller is located in a separate service room, this room must be air-conditioned to evacuate the calories.

The temperature stability of the cold water generated by the chiller is **crucial** for the performance of the instrument, especially for long measurements at high mass resolution and/or high lateral resolution. The water chiller should provide cold water adjusted between 17°C and 19°C with a permanent stability better than 1°C.
As there is a single chiller, it is important to wait for thermal stabilization after changes of power consumption (e.g. turning on/off the NEG, an ion source or changing the magnet B-field), before starting very high mass resolution or lateral resolution measurements.

The chiller feeds 2 lines: one line cools the physics parts (in blue in Figure 113) while the other line cools the electronics (in red). Both lines are equipped with taps from and to ("in" and "out") the chiller, in order to stop the water flow during maintenance operations. In addition, there are shunt junctions (black dots in Figure 113) that allows the user to short circuit the Cs Source or the electronics cabinet during maintenance.

In total, there are three distinct cooling circuits on the NanoSIMS 50L:

- One water circuit (blue line) is used to cool all the electronics,
- another water circuit (red line) is used to cool various optics elements (magnet, Cs^+ source, Wien filter and multicollection turbo pump).
- The Hyperion source (green line) is cooled separately by galden.

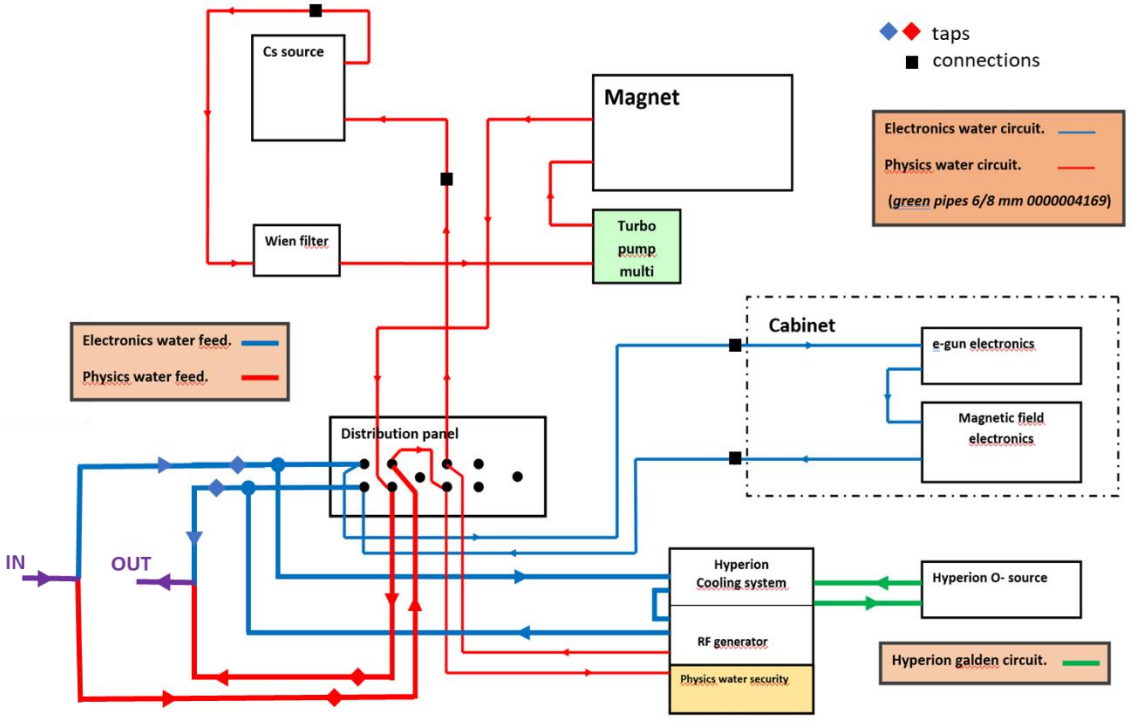


Figure 113: cooling fluid synoptic

3 NS50L Interface: Keyboard and Thumbwheel

3.1 Getting started

The keyboard and thumbwheels allow the user to access a number of lenses, diaphragms and other components of the instrument and adjust their voltage or position.
Each button (for example, L1) is associated with a series of parameters, grouped by 3. When clicking on a button, the small screen above the keyboard will display the first 3 parameters associated with this button (Here: L1, Co X and Co Y). By default, the 3 parameters displayed are the ones associated with Def1. To display the next groups of 3, click on Def2 or Def3 in the dark grey group of buttons in the middle (in the case of L1, Def2 will show L1, C1 X and C1 Y).

Figure 114: keyboard and thumbwheel system used for tuning the NanoSIMS NL50

The parameters displayed on the screen are the ones that can be adjusted via the thumbwheel board. The parameter on the first line can be adjusted with the top wheel. The parameter displayed on the bottom left can be adjusted with the bottom left wheel, and the parameter displayed on the bottom right of the screen is adjusted with the bottom right wheel.
Note that a digital version of this keyboard is accessible via the software (see below). This allows remote control and tuning, through network or the internet.

3.2 Keyboard parameter table

The table below lists all the parameters accessible via the keyboard. The left column indicates the key label. X, Y, Z indicates which wheel allows the user to change the parameter. Grey boxes indicates the default parameters showing when one presses on a key.

Table 1: complete description of the keyboard

Label	DEF1		DEF2		DEF3		SSXX		STIG		COIL		THD		LENS
	X	Y	X	Y	X	Y	X	Y	X	Y	X	Y	X	Y	Z
L0	C0x	C0y													L0
L1	C0x	C0y	C1x	C1y											L1
L2	C0x	C0y	C1x	C1y		SE FC									LCs
SS30	C0x	C0y	C1x	C1y		SE FC	SS30 Int	SS30 Ext							
L3	C0x	C0y	C1x	C1y		SE FC	SS30 Int	SS30 Ext							L3
P1P4	P1b	P1h	P4b	P4h											
L4	P1b	P1h	P4b	P4h		Cy									L4
E0S					P2P3	Cy									E0S
E0P	C0x	C0y	C1x	C1y					Oct 90	Oct 45					E0P
E0W	E0W Offset	Cy													E0W
P2P3	P2b	P2h	P3b	P3h											P2P3
LF2		Cy	C2x	C2y											LF2
LF3		Cy	C2x	C2y											LF3
HEX		Cy	C2x	C2y	HEX X	HEX Y									HEX
SS100		Cy	C2x	C2y			SS100 Int	SS100 Ext							
LF4	C4x	C4y	C2x	C2y	C3x	C3y	SS100 Int	SS100 Ext							LF4
LF5	C4x	C4y	C2x	C2y	C3x	C3y	SS100 Int	SS100 Ext							LF5
Q	C2x	C2y	C3x	C3y	C4x	C4y	SS100 Int	SS100 Ext							Q
EM1	C4x	C4y	Def-1	Foc-1	ESA Int	ESA 1							THD 1	FC Rep	EM1
EM2	C4x	C4y	Def-2	Foc-2	ESA Int	ESA 2							THD 2	FC Rep	EM2
EM3	C4x	C4y	Def-3	Foc-3	ESA Int	ESA 3							THD 3	FC Rep	EM3
EM4	C4x	C4y	Def-4	Foc-4	ESA Int	ESA 4							THD 4	FC Rep	EM4
EM5	C4x	C4y	Def-5	Foc-5	ESA Int	ESA 5							THD 5	FC Rep	EM5

EM6			Def-6	Foc-6	ESA Int	ESA 6							THD 6	FC Rep	EM6
EM7			Def-7	Foc-7	ESA Int	ESA 7							THD 7	FC Rep	EM7
EMTIC		Cy	C2x	C2y									Thd TIC		EM TIC
PM	PM Offset		Bf-Hor	Bf-Vert							EGun Be				PM
BFIELD			C4x	C4y											BField
Raster	Xlow RTI	Ylow RTI													Raster
Sample	X Axis	Y Axis													Z Axis
LDUO	CDuoX	CDuoY	COminx	COminY											LDUO
WF	CWF	WF Coil	CDuoX	CDuoY											
EMLD	C4x	C4y	C7x	C7y									Thd EM		EM LD
SC60	C7x	C7y					SC60 int.	SC60 ext.							
EMTIC		Cy	C2x	C2y									Thd TIC		EM TIC
PM	PM offset		Bhor	Bvert							eGun Be				PM
LF6	C5x	C5y									eGun Be				LF6
LF7	C5x	C5y	C6x	C6y							eGun Be				LF7
D0	D0 X	D0 Y													
D1	D1 X	D1 Y													
ES	ES X	ES Y													
AS	AS X	AS Y													
ENS	ENS X	ENS W													

Note: On most keyboards, “Sample” is not noted, though it does work. When selecting the “Sample” key, X, Y and Z thumbweels are allocated to X, Y and Z motions of the stage (Figure 115).

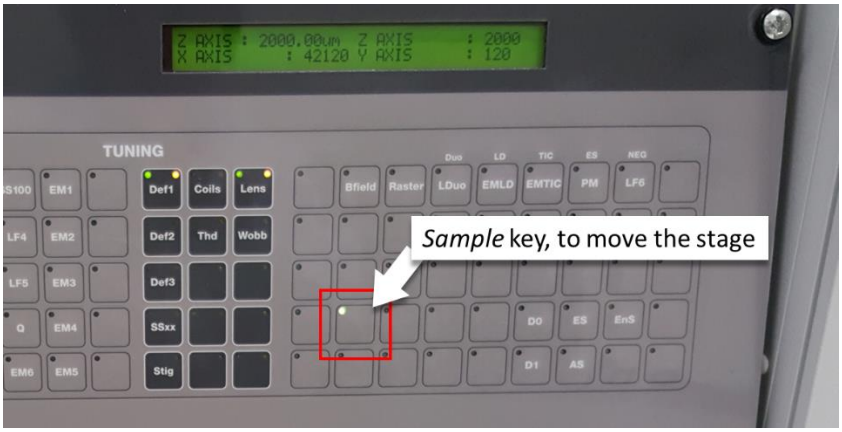


Figure 115: Unlabelled "sample" key on the keyboard, used to move the stage in the X, Y and Z directions.

4 NS50L Software: the "BOARD" interface

The board is the main interface of the software (Figure 116) and is usually located on the right side of the desktop screen. It is launched with along all the software component at each start of the instrument (see maintenance operation 10.1 for start procedure and how to launch the board) and must be kept on at all times. From the board, the operator can access all the programs necessary to tune and control the instrument, move the sample and run analyses.
The board It is composed basically of **three tabs: MAIN, TOOL and OTHER**, giving access to different programs.

Each icon on the board represents one specific program. To **launch** a program, the operator must click on the corresponding icon. A **green light turns on** aside the icon when the program is running (Figure 117). To **open** the running program window, click a second time on the icon. To **hide** this window while keeping the program running, click once more on the icon, or minimize the window.

If you **close** the window – either by clicking on the top-right corner cross or the exit button – it will **stop** the associated program and the **green light** on the associated icon will **turn off**.

Alternatively, all programs can be stopped via the ***control* button** at the bottom of the board. When clicking on control, a new window opens. Select the program you want to close in the pulldown menu and click on abort to close the program (Figure 118). The green light on the associated icon will turn off.

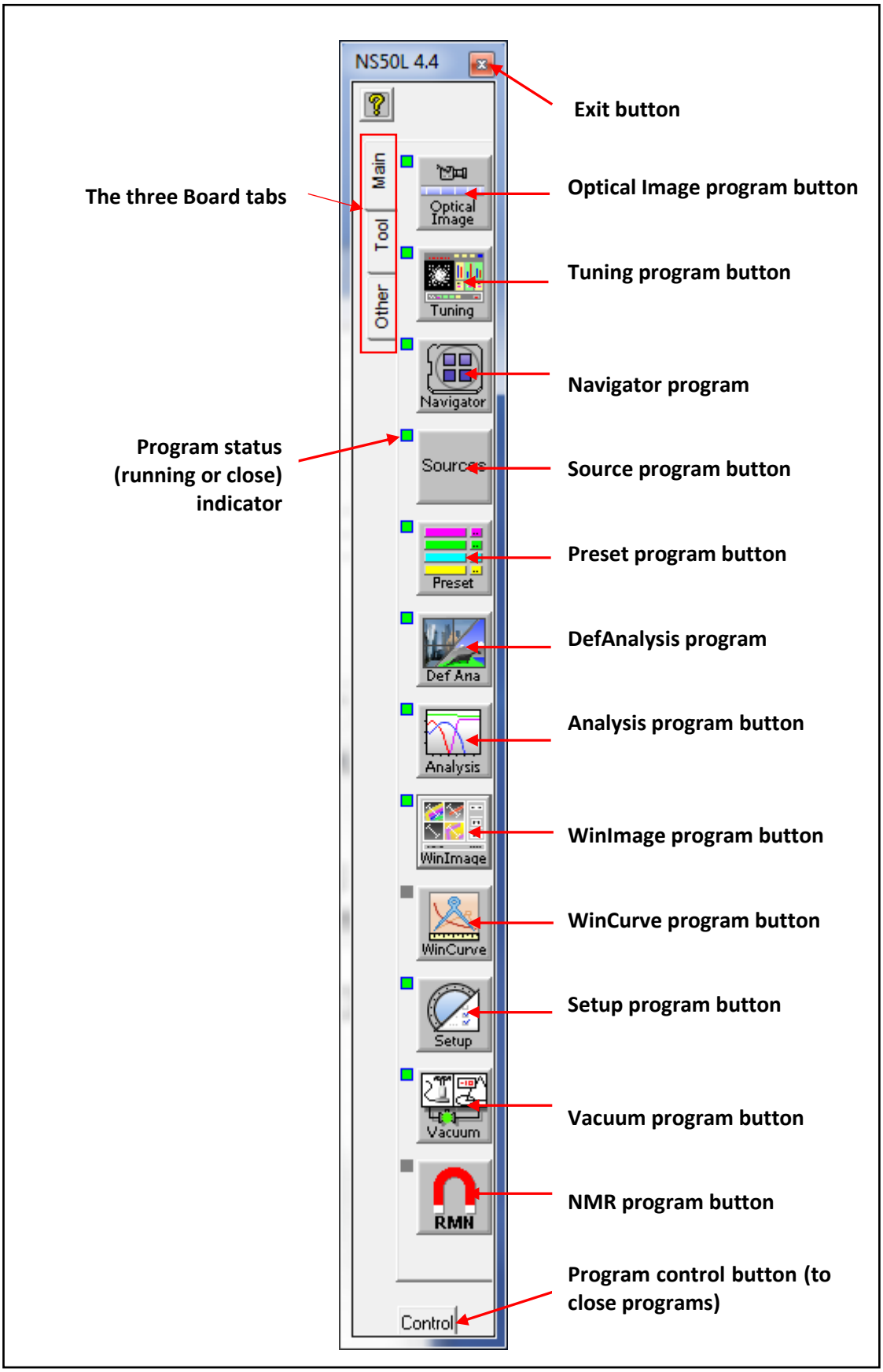


Figure 116: "Board" task bar

Running:  Closed: 

Figure 117: icon of the Tuning program displayed in the board.

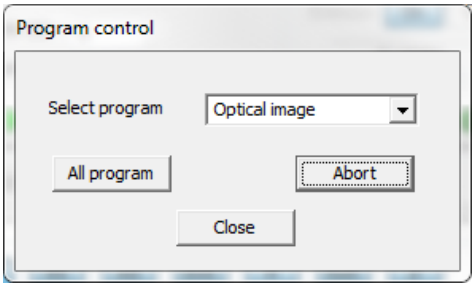


Figure 118: Board program control window.

In the following sections, we will describe the programs, tab by tab (Main, Tool, Others).

4.1 Principle/architecture of the NS50 software

Figure 119 shows the architecture of the different programs of the NS50 software. All those programs (in blue in the figure) are accessible via the BOARD. Additional programs (in green) allow the communication with the instrument Real Time and other hardware.

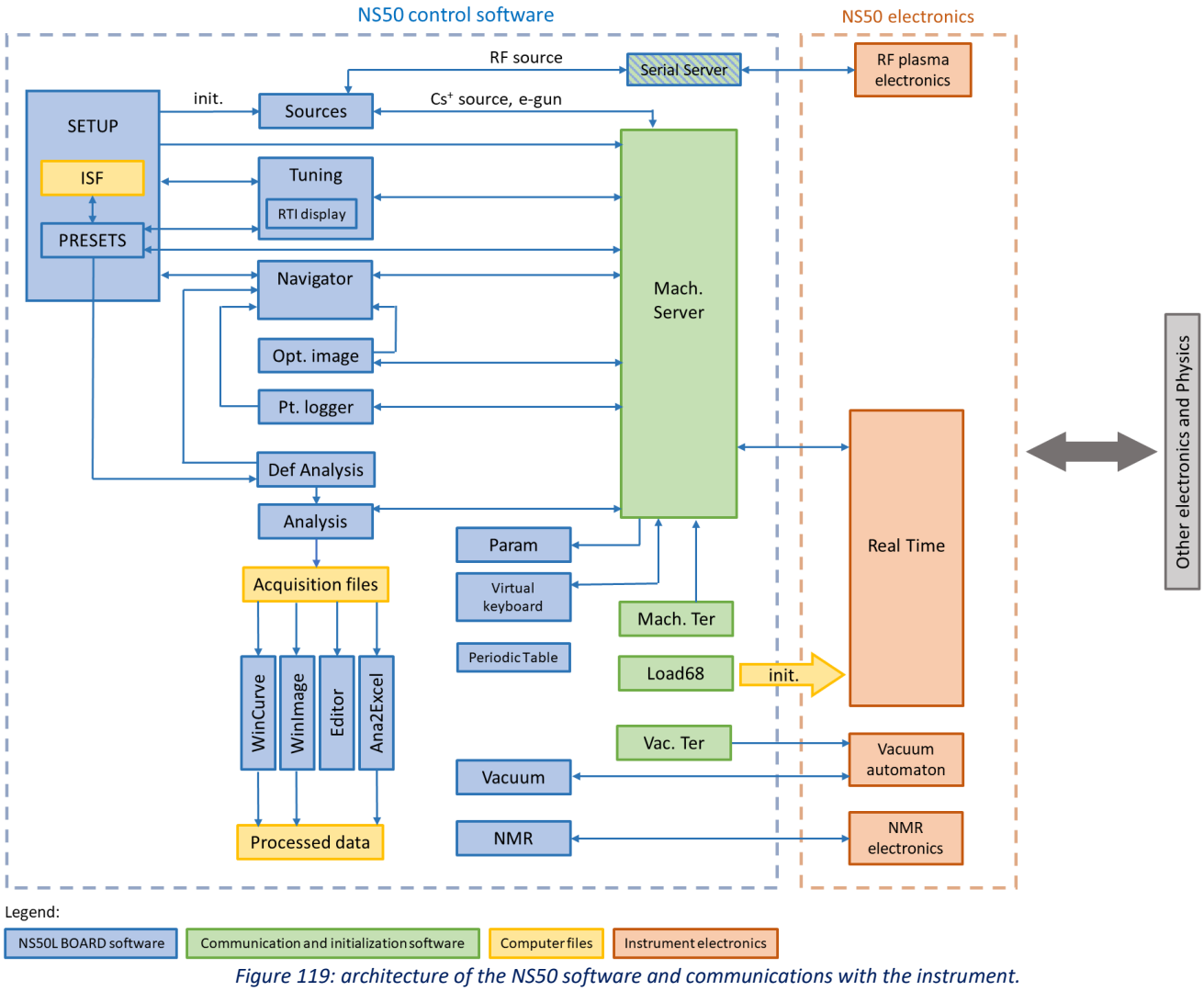


Figure 119: architecture of the NS50 software and communications with the instrument.

5 NS50L Software: The “MAIN” Taskbar

5.1 Optical image

Due to the short working distance between the sample and the immersion lens, the NanoSIMS has two sample positions: the SIMS position (“SIMS”) and the optical microscope position (“CCD”). The user can switch between the two positions with one click in the *Navigator* program (see 0 for details). The CCD position moves the sample under a CCD camera and the *optical image* program allows the user to view the sample via this camera (Figure 120) in order to navigate on the sample.

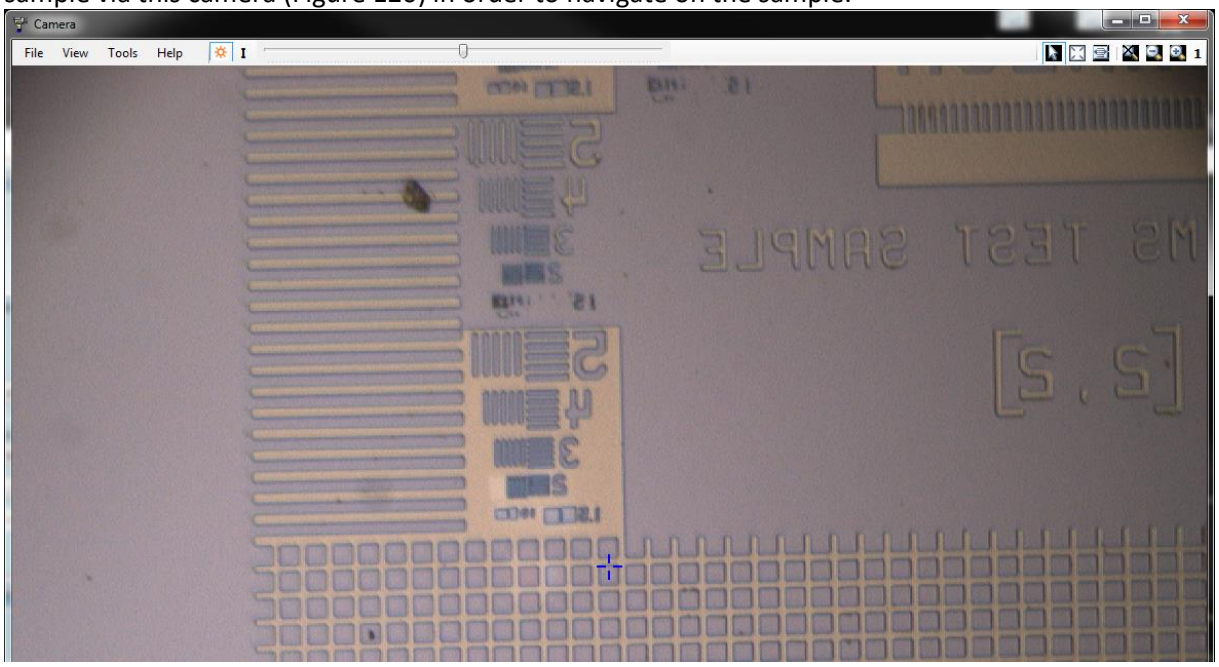


Figure 120: image of the optical image window, showing the surface of the sample

5.1.1 Getting started

Navigate on the sample: double click on point of the sample to move the stage and put the beam on this point.

Beam position: the blue cross (beam position) at the center of the image shows the reference position of the ion beam. For better accuracy of positioning while navigating the whole sample holder, the user can also display a working SIMS position (white cross) and a user position (green cross), via the menu “View”.

See 9.1.5 for details on how to use the optical image as well as 9.2.9 for adjustment of the beam position.

Save an image: The user can easily save a capture of the image (.jpg) of the sample on display via the menu File > Save > image.

5.1.2 Menu

File	Load:	allows the user to load an image previously saved.
	Save:	Image: save the image shown on the screen.
		Window: save the image as well as the marks (crosses, scale, etc…)
	Copy:	Image: copy the image (to be pasted directly in another software).
		Window: copy the image as well as the marks (crosses, scale, etc…)

Print: Image: to print the image.
Window: print the image as well as the marks (crosses, scale, etc…)
Print Setup: Image: change the print settings to print an image.
Window: change the print settings to print the image as well as the marks (crosses, scale, etc…).

View Camera
Working Position: when checked, shows the working position as a white cross.
Beam Position: when checked, shows the working position as a blue cross.
User Position: when checked, shows the users position as a green cross.
Field of View: when checked, shows the scale bar.
Line scan measurement: click and drag a line between two points to measure the distance
Labels: when checked, show the labels (see below)
Image freeze: stop the live feed
Zoom reset: reset the zoom to 1/1

Tools Label Properties: allows to add labels to features on the image
Image control: contrast and light control of the camera
Camera configuration: allows horizontal and vertical image and stage flips
Linescan measurement: click and drag a line between two points to measure the distance
Working Position Adjustment: when checked, allows to modify the “Working position”
Beam Position Adjustment: when checked, allows to modify the “beam position”
Help Software Version

See chapter 9.1.5 for details on how to use the CCD camera functions.

5.1.3 Icons

 When selected, the cursor is an arrow.

 When selected, a click on the image will zoom in the image.

 When selected, the zoom is controlled by the scroll button of the mouse.

 Zoom tools. Respectively, reset the zoom to zero, reduce the zoom, increase the zoom. The number indicates the level of zoom.

5.2 Tuning

This program is used to tune the instrument. It is the main program to set up the instrument before an analysis. From this window you can read the signal received by all available detectors, manage the multicollection trolleys, select slits and diaphragms, launch a real time imaging, as well as various automated tuning functions.

5.2.1 Getting started

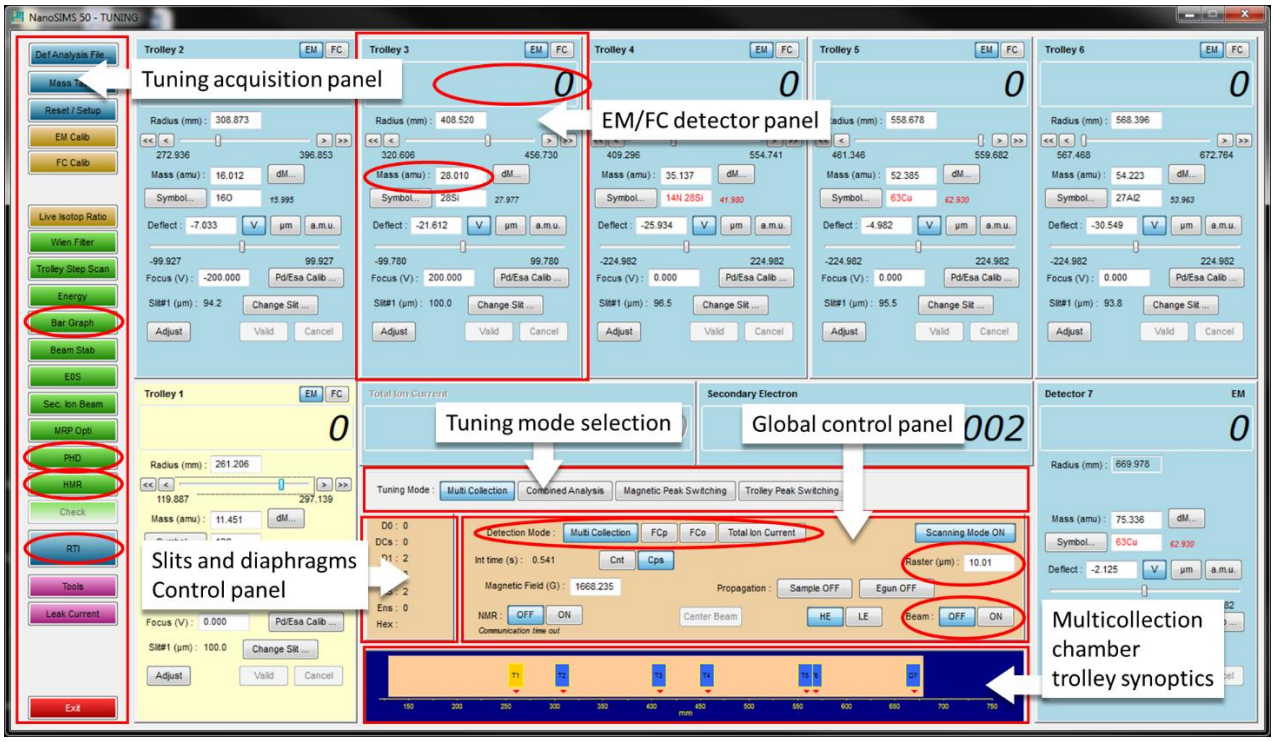


Figure 121: the TUNING window

The TUNING window (Figure 121) can be divided into 6 main sections:

- **The Global control panel**: This panel allows the user some basic actions, such as turning the beam on/off, setting the size of the raster, and selecting the detection mode so that the signal will be directed toward the multi-collection detectors, FCp, FCo, or the TIC.
- **The EM/FC detector panels:** There are 7 identical panels, representing all 7 multicollection detectors, which can be either electron multiplier (EM) or Faraday cup (FC). Via those panels, the user can set the detectors at the desired mass, as well as read the signal counted by each detector.
- **The multi-collection chamber trolley synoptics:** shows the positions of the trolleys inside the chamber.
- **The tuning mode selection:** this will determine how a detector will be set at the right mass.
    - In "**multi-collection**" mode the B-field is fixed: when a mass is entered on a detector panel, the detector will move to reach the position associated with the mass.
    - In **Magnetic Peak Switching** mode, it is the Magnetic Field that is adjusted to set the mass on the detector. The detector doesn't move.
    - The case of **detector 7** is special: it cannot move. Whatever the mode its mass will be adjusted by changing the magnetic field (hence when setting up a multicollection configuration with seven masses one should start by the heaviest mass on the 7th detector).
- **The tuning acquisition panel** on the side of the screen**:** it allows the user to access various sub-programs necessary for the tuning. Among those major functions:
    - The **bar graph**: by scanning the magnetic field on a given detector, the user can identify the masses (and thus the elements) composing the sample.
    - The **PHD** tool: allows to adjust the voltage and noise threshold of each EM.

- The **HMR** (High Mass Resolution): records a small mass spectrum at high resolution on a given detector by scanning the deflection plates before its exit slit. It is used to properly tune the peak shape, center and select the desired peak, detect mass interferences.
- The "**real time** imaging" (**RTI**) that allows the user to see live ion images of the sample allowing, for example, the tuning of the primary beam.

- **The slit and diaphragm** control panel: When hovering the mouse over this section, the panel deploys (Figure 122).

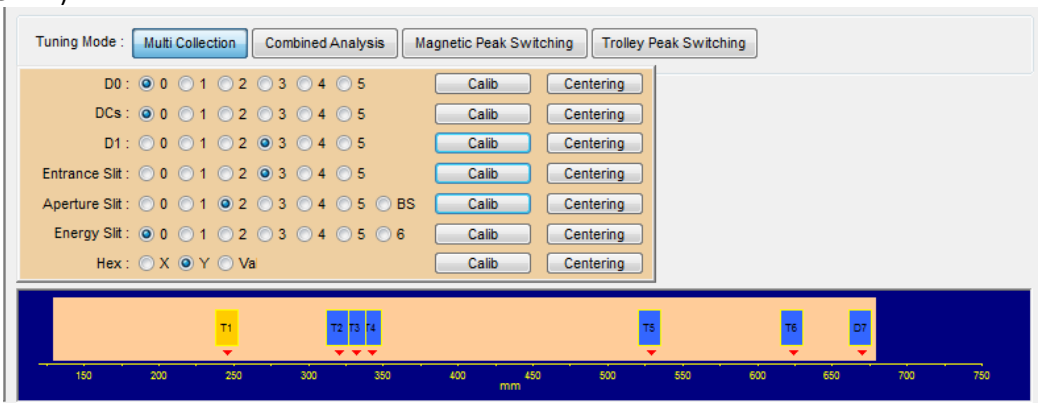


Figure 122: Slit and diaphragm control panel (appears when hovering the mouse over this TUNING section).

5.2.2 EM/FC Detector panel

At the end of its path, the secondary ion beam reaches the detectors. Each of the multi-collection detectors can be either an Electron multiplier (EM) or a Faraday Cup (FC). Number of detectors and EM/FC availability may differ depending on instrument model and options. Using either EM or FC depends on:

- the expected count rate (cps) that will hit the detector. Typically, an EM is suitable for a signal up to around one million counts per second, while a FC is used up to mid- E8 or mid-E9 c/s respectively with FC preamplifier of 1 E10 or 1 E11 ohm resistor.
- The acquisition mode: working in single ion pulse counting mode EM allows fast imaging with short dwell-time per pixel (e.g. 50µs) as well as long acquisition times (several seconds for isotopic ratio down to the permil reproducibility), as well as the use of electronic window in depth profiling.
- Due to a large time constant, the FC cannot be used for imaging. It is ONLY used for large dwell-times of several seconds typically. But it will ensure the best precision and reproducibility for very precise isotopic ratios (down to a few tenth of permil reproducibility).

The following panel displays controls for an EM/FC detector (Figure 123). The panel is identical for EM and FC apart from the counting unit option (cps or pA) on top of the panel that is only available for FC. For EMs, the signal is always given in cps (=c/s).

Figure 123: two EM/FC detector control panels

[1] The position of the detector in the chamber is given by the radius value and can be adjusted by either typing a different value (in mm) or using the arrows of the scrollbar. Sometimes it is practical to write down a radius for a given B-field in order to be able to come back to the position after some movements, by retyping it in this field. When changing the radius, the mass (in amu) is refreshed using the current B/R calibration.

[2] "Mass" indicates the mass (in amu) corresponding to the position, depending on the current Magnetic Field and EOW HV (= 1/2 impact energy). When changing the mass (in amu) the radius (in mm) is refreshed using the current B/R calibration.

The second box allows the user to enter the isotope expected to be detected by the detector. It can be typed in or selected from a Mendeleyev table by clicking on "Symbol". It must be typed like:
"28Si2 16O3" (with a space between each atomic species). *It is only an information, with no action on the instrument*.
In italic next to it, the theoretical mass of the isotope is given. When the position and the theoretical mass match, the isotope appear in **black** (see Trolley 2 in Figure 123). However, when the trolley position (thus the mass detected) is too far from the theoretical value, the isotope name appears in **red** (see Trolley 3 in Figure 123). *It is only an information or warning*. The user can adjust the maximum interval of acceptance between the mass detected and the theoretical value by clicking on "dM" (for example, if dM is set at 0.3, every position within ±0.3 of the theoretical mass will be accepted and the isotope name will appear black)

When **in multi-collection** tuning mode, **entering a mass** (in amu) followed by "Enter" **will move the detector** to the corresponding position (except for detector #7 where it will move the B-field).

- ⇨ It makes sense to start by positioning first the highest mass of interest into the highest radius in order to determine and fix the B-field, then move the others trolleys by decreasing radiuses (see chapter 5.2.9 on how to check a trolley configuration).

When in **magnetic peak switching or combined** analysis mode, **entering a mass** will **change the magnetic field** value. The detector will not move. Note that changing the magnetic field value affects all the detectors following R = sqrt(2*m*HV/q) / B.

[3] Because of the hysteresis of the magnet, the calibration between the magnetic field and masses is delicate. For more details on how to finely adjust the calibration for one peak, see the procedure described in 0.

[4] Each detector is preceded by two deflection plates (Pd) before the exit slit. To finely position the peak in the detector, the user can adjust this electrostatic deflection. However each detector has an maximum deflection range. It is recommended to stay within about 100 V of the optimum value, otherwise the

transmission/MRP relation will be degraded. It is then recommended to move the trolley to reduce the need for electrostatic deflection introducing aberration (peak shape degradation).

[5] - A positive or negative voltage can be added (+/- 200V max) to *both* deflection plates (creating a stigmator/lens effect) to optimize the mass resolving power at the detector. For this and the use of the Pd/ESA calibration see expert operation chapter 0

[6] For each detector, 3 exit slit sizes are available. The mass resolution of the mass analyzer is determined mostly by the entrance slit sizes and residual aberrations and noises. The exit slit size will change the shape of the peak. Depending on the analytical need one will work whether with large exit slit to obtain flat top peaks (in the case of inorganic peaks on one (light/left) side of the peak group) or with small exit slit when the peak of interest is in the middle of a range of peaks at one given mass unit.
On the NS50L it is possible to switch slit automatically by clicking on “Change Slit.”

5.2.3 Global control panel

The following panel displays global controls for the tuning (Figure 124).

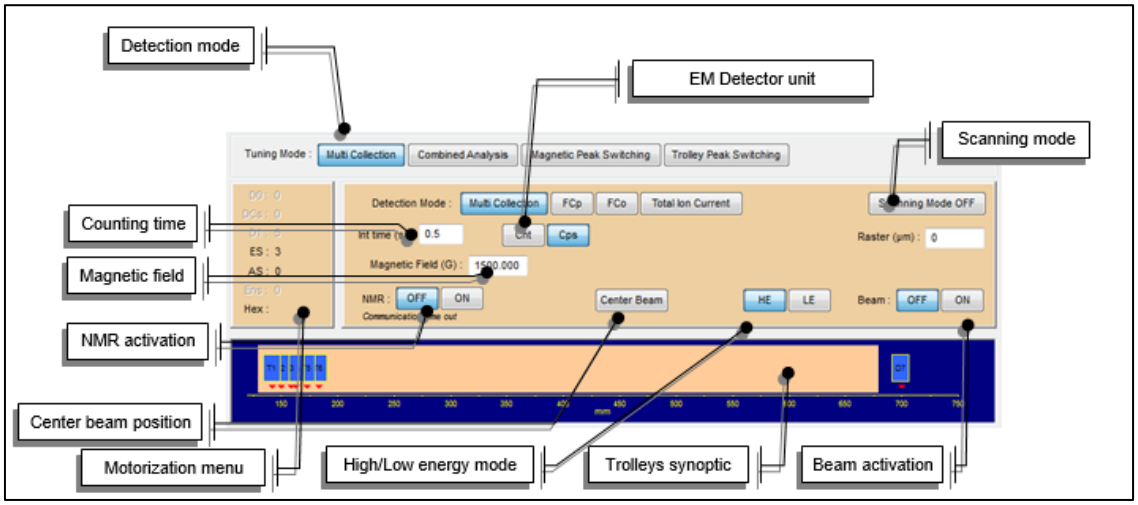


Figure 124: Tuning control panel

- The **Detection Mode** allows the user to send the beam in different detectors along the path of the NanoSIMS (Figure 125):
  - **FCp** is a Faraday Cup on the primary column. It measures the primary beam current after the source (and the first lenses) when the beam is OFF.
  - **FCo** is a Faraday cup located behind the sample (object). When moving the stage to a position letting the beam go through, it allows to measure the primary beam current reaching the sample when the beam is ON.
  - Total Ion Current (**TIC**) is located on the secondary beam section, before the mass spectrometer. Therefore, it receives all the secondary ion signal of one polarity coming from the sputtered sample, without mass filtering.
  - At the end of the course is the **multi-collection**, where the EM/FC detectors are positioned to receive the signal of selected isotopes. In Multi-collection mode, all multicollection detectors (see above) as well as the **secondary electron (SE)** detector are active.

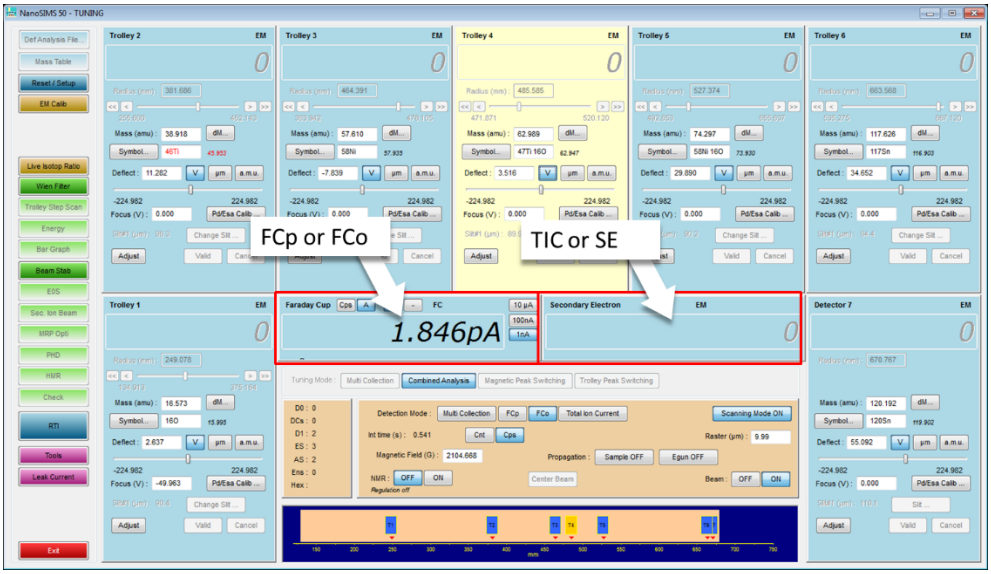


Figure 125: the central part of the TUNING window shows either FCp (in FCp mode), FCo (in FCo mode), the Total Ion Current (in TIC mode) or the Secondary Electron signal (in Multicollection mode)

The user can also:

- Adjust the counting time for all the detectors (only available if the scanning mode is OFF, otherwise the counting time is determined by the scanning mode, see Figure 126)
- Set the Magnetic Field (value given in Gauss)
- Change the EM detector display unit (count per second (cps = c/s) is traditionally used)
- Put the NMR regulation ON or OFF
- Put the beam ON or OFF. Note that beam "OFF" means the primary beam is blanked by sending it sent into FCp. Beam "ON" means the primary beam is sent toward the sample or FCo.
- Set the Raster, and the size of the rastered area.
- Adjust the raster parameters by right-clicking on the "scanning mode ON/OFF" button (Figure 126). In most cases it is recommended to keep the raster simple and fast, defined with 64x64 px, with a counting time of 0.54s per frame. Only for imaging with NEG on difficult heterogeneous insulators might it be useful to tune with the scan conditions of the future image.
    - Max Frame (=working frame) is the maximum area **in pixels** allocated in the memory of the scanning board. The maximum is 1024 x1024 pixels.

    - Scanning frame defines the scanning of the beam**, in pixels**. It is defined by the coordinates of its upper left corner inside the max frame (in pixels: NXLow, NYLow) and its width and height (in Nb of pixels in X and Y).

    - Counting frame: an electronic window (=gate) permits to count the signal only when the primary beam is inside this window. It is used mostly for depth profiling to reject signal coming from the edge of the crater but count the signal only coming from the (flat) center of the crater bottom. It is defined by the coordinates of its upper left corner and nb of pixels in X and Y.

In general, the pixel size is taken between half to a third of the beam size. Over-pixelizing wastes time and do not bring anything more except making the drift correction easier. Under-pixelizing brings loss of information by drilling holes at each pixel center with untouched sample space in between.
e.g. if the spot size is 200nm, the max area will be 1024*0.1 ~ 100µm. With 50nm spot size and 512 pixels, 12.8µm max before becoming under-pixelated (= losing information). With 1µm spot size (depth profiling), 64 x64 pixels allows crater size up to 32x32µm, which is generally sufficient.

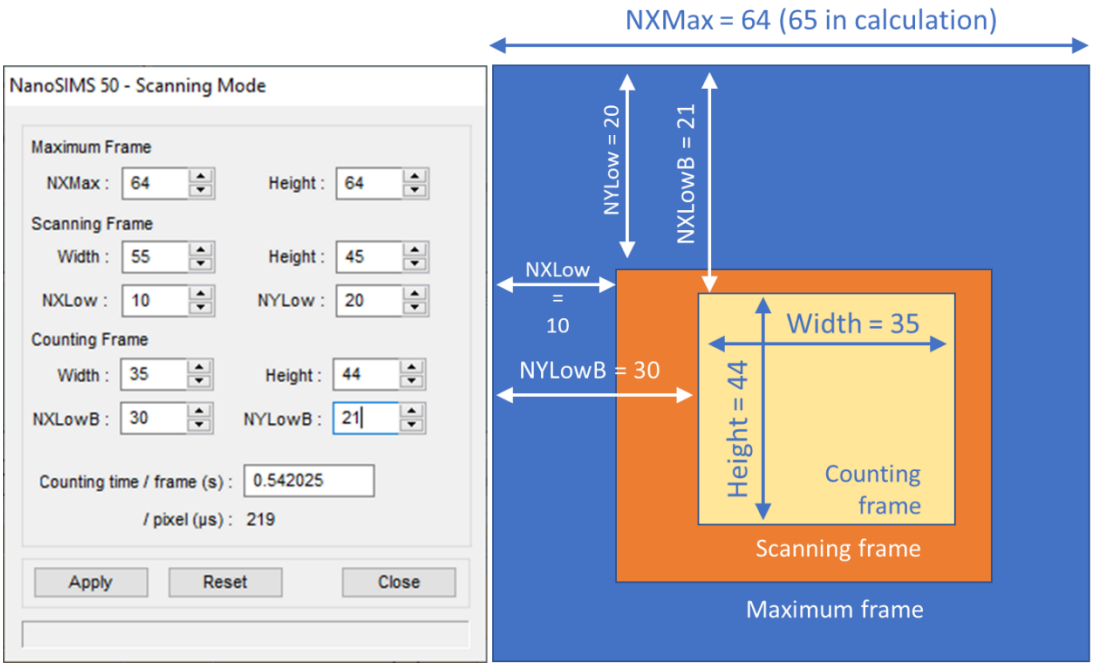


Figure 126: Scanning mode configuration window and schematic showing the definition of the various parameters with numerical example of a counting frame smaller than the scanning frame, which is itself smaller than the maximum frame.

- If the option is available on the instrument, the user can also switch between high and low energy modes (HE/LE), the latter being used for ultra-low energy implantation/deposition without analysis (see 9.2.8 for details)

Note: the “Center Beam” option is not available.

5.2.4 Tuning mode selection

At the top of the global control panel (Figure 124), the Tuning mode allows the user to select the way they want to tune the multi-collection.

- In “**Multi-collection**” mode, when entering a mass (in AMU) in the EM/FC detector panel, the detector mechanically moves in the chamber to place itself in the position to “catch” atoms from said mass, at a fixed magnetic field value.
- In “**Combined Analysis**” mode, all detectors are available, but they do not move. When the user enter a mass (in AMU) in a detector panel, It is the magnetic field that adjusts so that the detector will receive the signal of the desired mass. Be careful that, as the magnetic field is common to all detectors, all detectors will be affected by this magnetic field switch.
- The “**Magnetic Peak Switching**” mode is like Combined Analysis. It is the magnetic field that adjusts, while detectors do not move. Here it is necessary to select each detector one by one.
- The “**Trolley Peak Switching**” mode is similar to the “Multi-collection” mode. The selected trolley mechanically moves to the mass position.

For more details on image acquisition in Multi-collection mode, see advanced operations 9.2.1. For how and why use Combined analysis, Magnetic and Trolley peak switching, see expert operations 9.3.6.

5.2.5 Slit and diaphragm panel

The following panel displays controls to manage motorized slits and diaphragms (*Figure 127*). For each diaphragm or slit, several positions are available (1 to 5 or 6). Each of these positions match a specific size of diaphragm/slit (may vary from one instrument to another – For diaphragm and slit dimensions, refer to the SETUP (chapter 5.9).

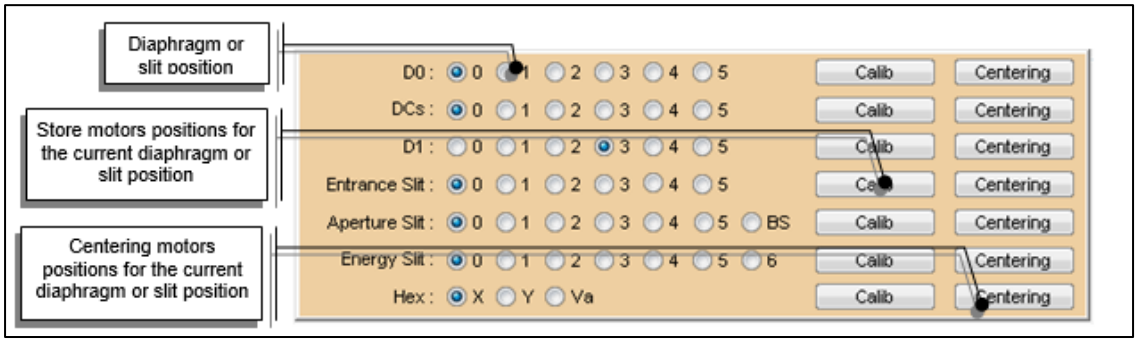


Figure 127: Slit and diaphragm control panel

The Aperture slit also has a "beam stop" (BS) position, which stops the secondary beam. This is useful to protect the TIC or multicollection EMs when implanting the sample at high current.

Those positions are motorized. When clicking on a position, the diaphragm or slit pin will move to the selected position. However, the motors are not perfect and the positions can shift with time. The user can then readjust manually a position via the keyboard (for example, to reposition D1, select D1 on the keyboard and adjust X and Y to optimize the signal). To save the new X and Y positions in the keyboard, click on "calib". Those positions can also be adjusted by doing a scan via the "centering" function associated with each slit and diaphragm (See chapter 9.1.7 in Basic Operations for more details on diaphragm and slit centering).

The energy slit can be used between 0 (wide opened, no energy filtering) up to position 6, depending on the aim (MRP increase, reduction of molecular interference, etc…). Each EnS setting can be adjusted in position and width and stored in the set-up. Refer to advanced operation chapter 0 for more details on adjusting the energy slit.

This slit and diaphragm panel also allow to recall X and Y positions of the hexapole (HEX) (See 9.2.1.1) as well as centering the Hexapole (see alignment procedure 9.3.1.3).

5.2.6 Synoptic panel

The following panel displays the detectors synoptic positions (*Figure 128*) in the multi-collection chamber.
The scale can be displayed in mm or a.m.u. (according to the magnetic field value). Click on the scale to switch between units.
No action is possible from this panel (it is for information only). The trolley currently selected in TUNING appears in yellow.

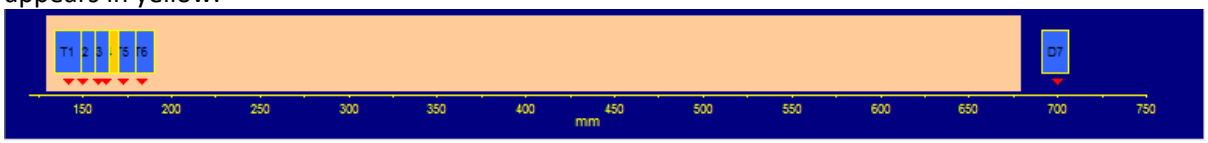


Figure 128: Trolleys position synoptic

5.2.7 TUNING side panel

5.2.7.1 List of TUNING side panel functions

The Tuning side panel shows all the tuning programs and settings directly accessible to tune the instrument. They will be described one by one in the next paragraphs.
Note that depending on the detection mode, certain programs might not be accessible.

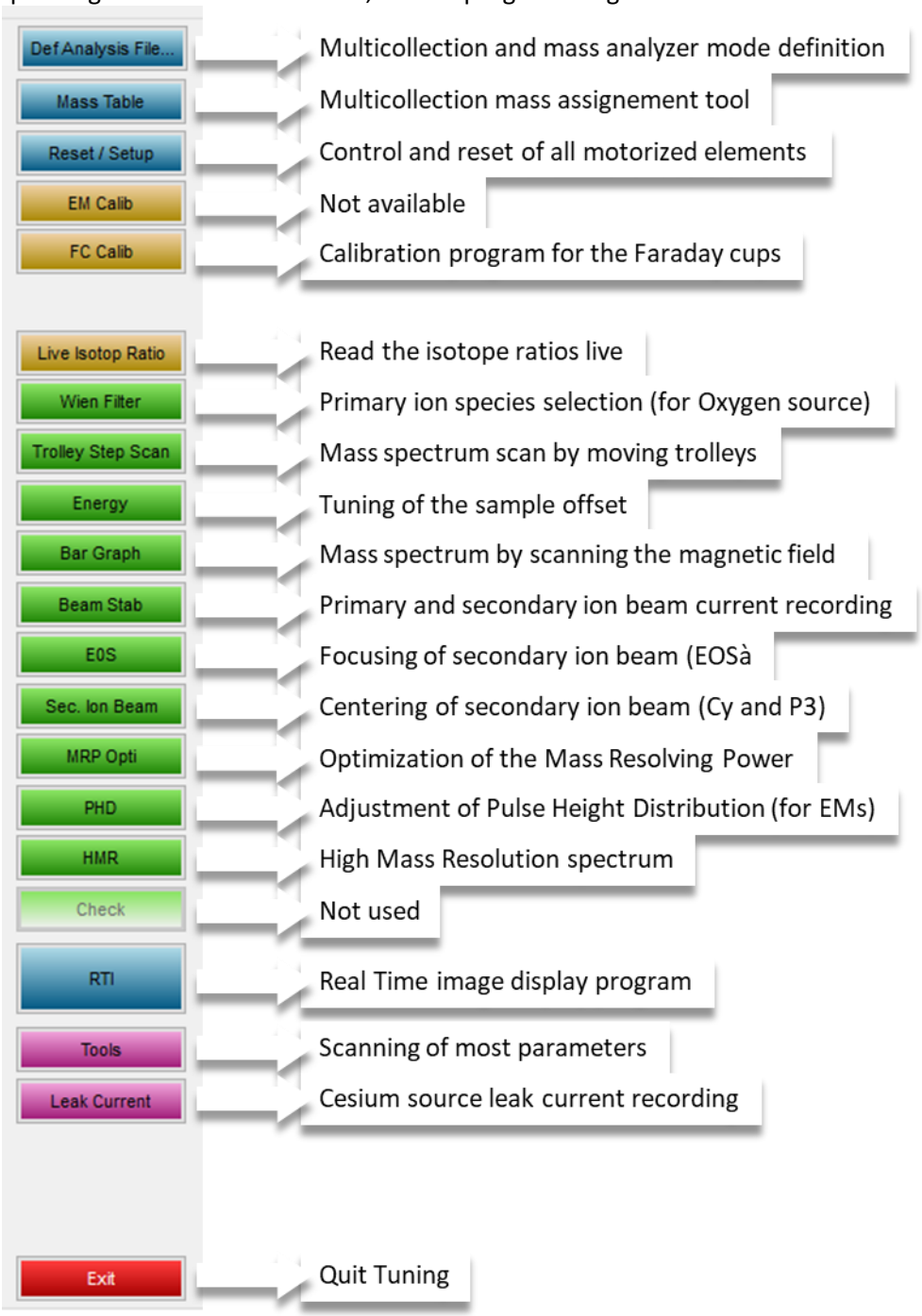


Figure 129: Tuning side panel

5.2.7.2 Common software interface for scanning a parameter

Many of those programs use similar scanning functions. Figure 130 shows the shared features of all those scanning functions.

1- Choose one (or when applicable, several) detector.
2- Set the scan parameters. When unsure of what to set, check the scanned lens value via the keyboard and set the “start voltage” and “voltage step” parameters as to have the current lens value at the center of the scan. When the signal is low, you can accumulate several scans (“number of scans”) to smooth the curve.
3- When parameters are set, launch the scan by clicking on “Start”. You can stop the scan at any time by clicking on “abort” or “stop”.
4- When the scan is done, the program calculates the central line (CL, in yellow on the graph), as well as various widths of the peak. If you are satisfied with the centering of the CL, click on “apply CL”. Otherwise you can readjust it manually by clicking on the graph (green line). In this case, click on “Apply value” to apply the green line value.

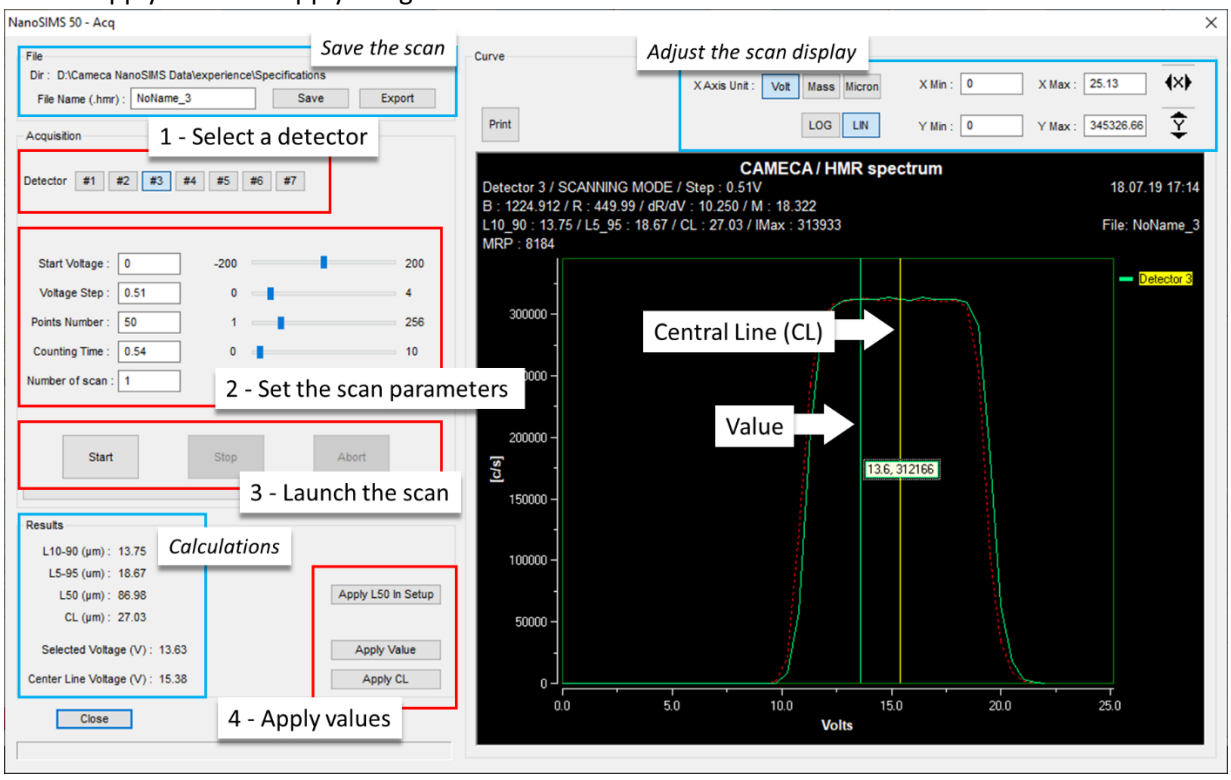


Figure 130: Common organization of all scanning windows of TUNING.

Additional information (in blue):

- You can change the graph display depending on your needs: The X scales can be displayed either in Volt (recommended), Mass units or microns. You can also adjust the Y scales in Log or Lin depending if you’re looking at peak flatness of flank shape. You can also readjust the X and Y coordinates manually. Note however that it will not affect the actual scanning scale defined by the parameters on the left. The X and Y double arrows allow to switch back to the default (auto) X and Y values.
- At the end of the scan, the program calculates the center of the line and shows Results in the bottom left corner of the window. In addition, depending on the program, it calculates peak width (for instance, L50 is the peak width at 50% of the peak) and slope width (L10-90 is the slope width between 10% and 90% of the peak signal). These are used for automatic peak centering (see below)
- You can also save the scan. Click on “save” to save a file that can then be read via WinCurve. Click on “Export” to automatically export data into an Excel file. In both cases the file will be saved in the noted directory, which Is where everything from a session is saved (scans, acquisitions, etc…) This directory can be changed through Def Analysis.

Certain programs also allow an automatic peak centering modes. Those modes are described in Operations (chapter 9).

5.2.8 DEFANALYSIS file display

This program is used to display and save MULTICOLLECTION (B-field and trolley position) configurations. It allows an easy reload of a previous configuration and only retune it for new analytical needs, instead of sending all trolleys one by one and adjusting the B-field. It is slightly different depending on Tuning mode (thus type of analysis) selected.

5.2.8.1 DEFANALYSIS in multi-collection mode

In **Multi collection**, this program can be used as a simple display (Figure 131). It shows the detectors settings (mass, corresponding species, exit slit, radius position and deflection values) as well as the magnetic field, EOW offset, Q, LF4 and hexapole values. When opening the program, the window will display the current configuration. This configuration (comprising magnetic field, trolley configurations, EOW, Q, LF4 and Hex) can be saved in a def analysis file (Save As).
Alternatively, a previously saved configuration can be loaded (Load). When applied (Apply), all saved parameters will be applied (overwriting any previous value sent to those parameters), and the trolleys will move accordingly.

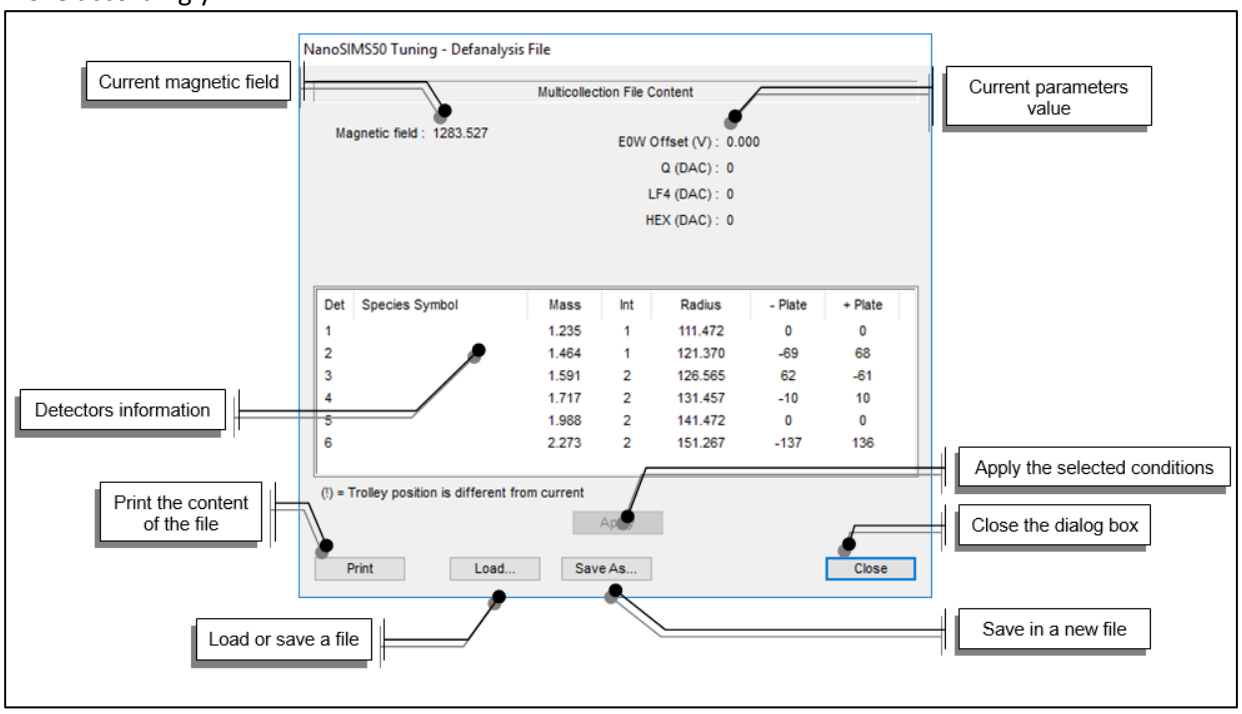


Figure 131: DefAnalysis window in Multi Collection mode

5.2.8.2 DEFANALYSIS in Combined Analysis mode

In **Combined Analysis** mode, the magnetic field cycles between 2 or more values during the analysis. An individual "multi-collection"-like trolley configuration can be associated to each value of the magnetic field. This program is then necessary to set magnetic field settings and their associated trolley parameters before running an analysis. The Def Analysis File window looks as follow (Figure 132):

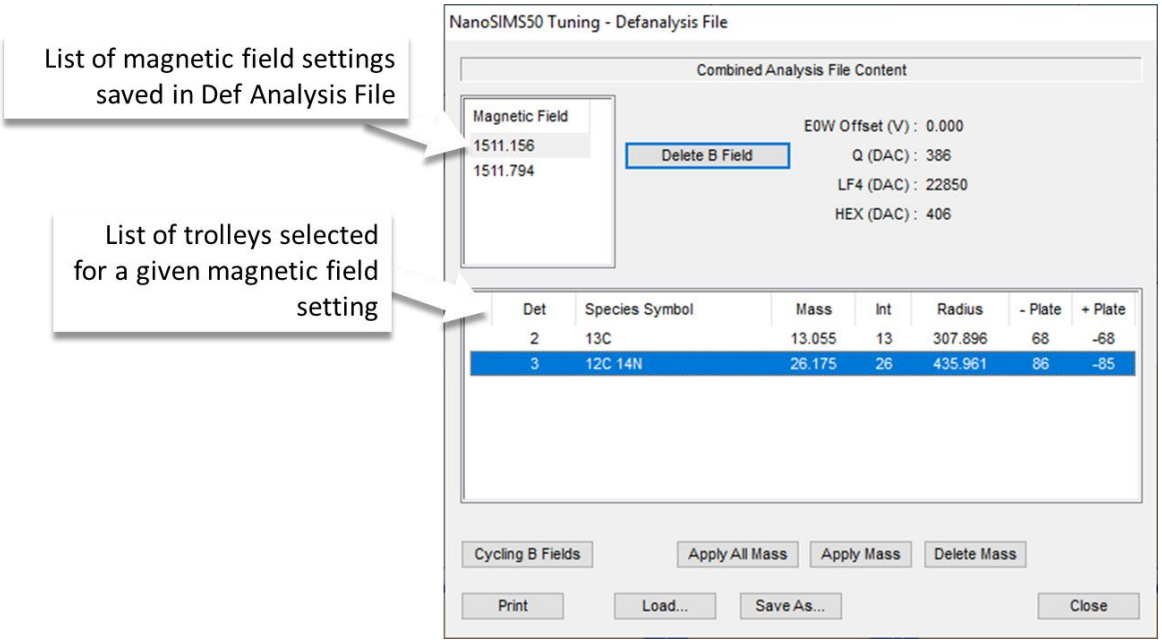


Figure 132: Def Analysis File window in Combined Analysis mode

Edit the content of a Def Analysis File:

- Remove a magnetic field setting, including all its associated trolley parameters: Select a magnetic field value and click on Delete B Field.
- Remove a trolley from a Magnetic Field setting: Select the magnetic field setting, then click on the trolley you wish to remove, and "Delete Mass".
- Add a magnetic field setting: In Tuning, when the magnetic field and trolley positions are set, click on "Save to Def Analysis" for each trolley you will use.

Apply the displayed setting:

- Apply a Magnetic field setting (including all its associated trolley): Select a magnetic field setting and click on "Apply all Mass".
- Apply parameters to a single trolley: Select the magnetic field setting associated with the trolley, then select the trolley you wish to move, and click on "Apply Mass".

Cycling B-Fields: launch a cycling of the magnetic field between the different magnetic field setting values. It is necessary to let the magnetic field cycle for a few minutes before launching a Combined Analysis acquisition, otherwise it will not be reliably reproducible. See chapter 9.3.6 on Combined Analysis for more details.

Similarly to the Multi-collection mode, a given configuration can be saved in a def analysis file for further use (Save As) and later recalled (Load). Note that it is not necessary to save a Def Analysis File to launch an analysis acquisition. The acquisition will use the data configuration as it is displayed in the Def Analysis File window.

Note that a given trolley can be used for several values of magnetic field.

In addition, any change in a trolley configuration, such as adjustment of the deflector values between two cycles (for instance to measure $^{12}C^{15}N$ and $^{13}C^{14}N$ at mass 27 on same detector and same B-field but alternating between two sets of Pd deflector) can be saved in distinct magnetic field settings, even if the magnetic field value is identical.
For more details on how to configure the data displayed in the Def Analysis File and set a Combined Analysis acquisition, see chapter 9.3.6 in Expert Operation.

5.2.8.3 DEFANALYSIS in Magnetic Peak Switching mode

The **Magnetic Peak Switching** mode is a mono-collection mode. Only one detector is used, and it is the magnetic field that cycles between 2 or more values to measure the different elements or isotopes. Here the trolley is fixed (it has been selected in the Tuning window, Figure 133), and only the different magnetic field values need to be set in the Def Analysis File (Figure 134).

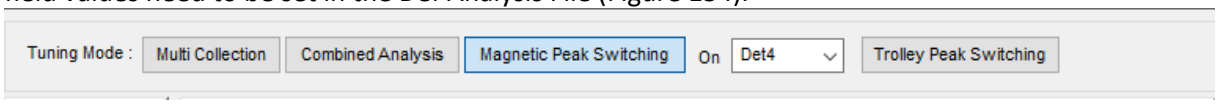


Figure 133: partial view of the Tuning window, showing the selection of detector 4 (Det4) in Magnetic Peak Switching mode)

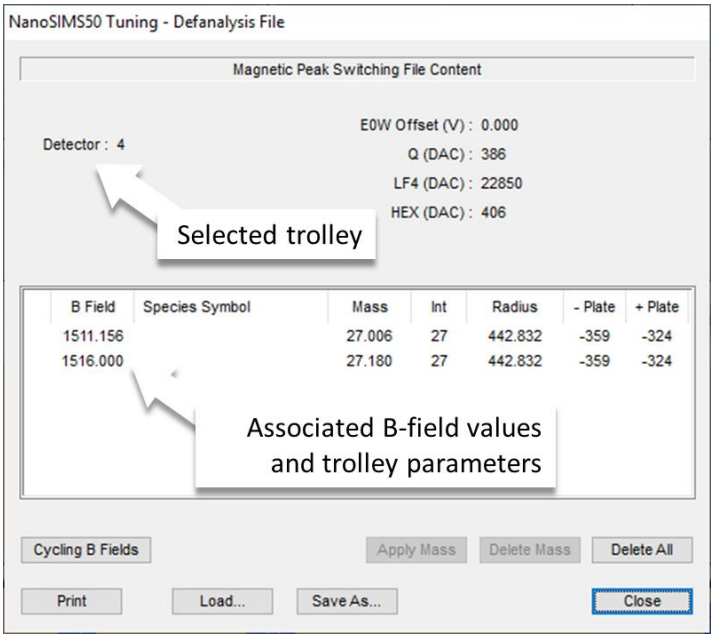


Figure 134: Def Analysis File window in Magnetic Peak Switching mode

Edit the content of a Def Analysis File:

- Remove a magnetic field value: Select the magnetic field value in the list and click on Delete Mass.
- Clear all the list content: Click on “Delete Mass”.
- Add a magnetic field value: In Tuning, set the magnetic field, and click on “Save to Def Analysis” for the used detector.

Apply the displayed setting:

- Apply a Magnetic field value: Select a magnetic field value and click on “Apply Mass”.

Cycling B-Fields: launch a cycling of the magnetic field between the different magnetic field values. It is necessary to let the magnetic field cycle for a few minutes before launching a Magnetic Peak Switching acquisition, otherwise it will not be reliably reproducible. See chapter 9.3.6 for more details.

Again, a given configuration can be saved in a def analysis file for further use (Save As) and later recalled (Load). Note that it is not necessary to save a Def Analysis File to launch an analysis acquisition. The acquisition will use the data configuration as it is displayed in the Def Analysis File window.

5.2.8.4 DEFANALYSIS in Trolley Peak Switching mode

In Trolley Peak Switching mode, the magnetic field is fixed but this time it’s the trolleys that are cycling between positions. The Def Analysis File then looks as follow (Figure 135):

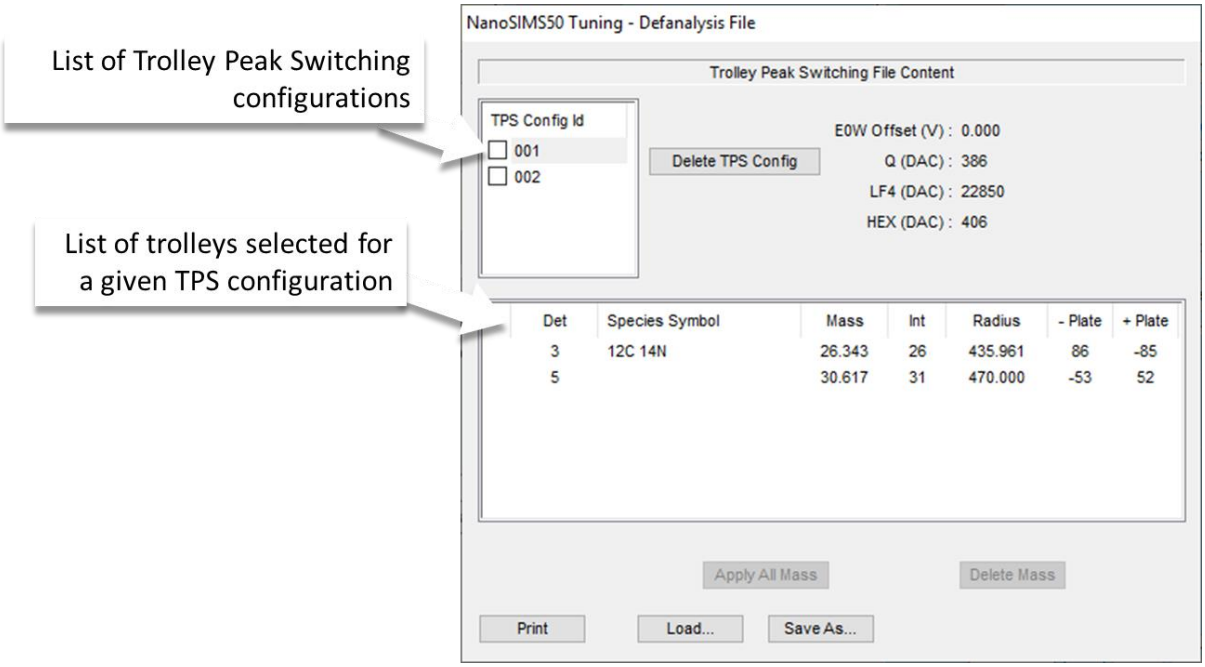


Figure 135: Def Analysis File window in Trolley Peak Switching mode

In this mode, the detector window in TUNING shows two new options/buttons: Save in New or Save in Current (Figure 136). When moving a trolley to a desired position, click on "Save in Current" to add it to the selected Trolley Peak Switching (TPS) configuration in the Def Analysis window. The trolley and its position will be added to the list of trolleys associated to the selected TPS configuration. To add a trolley position to a separate TPS configuration, click on "Save in New". The trolley and its position will then be added to a separate TPS configuration.

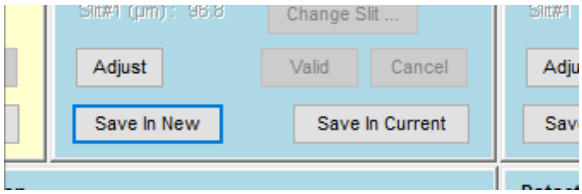


Figure 136: partial view of a detector window in TUNING showing the options specific to the Trolley Peak Switching mode

Edit the content of a Def Analysis File:

- Remove a TPS configuration, including all its associated trolley parameters: Select the TPS Configuration and click on "Delete TPS Config". in the list and click on Delete Mass.
- Clear the trolley list of a given TPS configuration: Select the TPS configuration and click on "Delete All Mass".
- Remove a trolley position from a TPS configuration: Select the TPS configuration, then click on the trolley you wish to remove and click on "Delete Mass"
- Add a TPS configuration: In Tuning, move a trolley into position, and click on "Save in New"
- Add a trolley to an existing list of a TPS configuration: Select the TPS configuration in the Def Analysis File window, then in TUNING, move the trolley into position, and click on "Save in Current".

Apply the displayed setting:

- Apply a TPS configuration value: Select a configuration in the "TPS config" list and click on "Apply All Mass".

Again, a given configuration can be saved in a def analysis file for further use (Save As) and later recalled (Load). Note that it is not necessary to save a Def Analysis File to launch an analysis acquisition. The acquisition will use the data configuration as it is displayed in the Def Analysis File window.

5.2.9 Mass Table Edition

This program (Figure 137) allows the user to edit, build, test, save and apply to the instrument a **multicollection setting**, including B-field value and trolley positions with the names of the ions. Trolleys can be moved to a given position either by entering a mass value, or a radius. This Mass table program makes it very quick to reload a previous configuration and only retune it for new analytical needs, instead of sending all trolleys one by one to their position and adjusting the B-field.

Reminder: The two basic limits of the NS50L multicollection are:

- M_{max}/M_{min} = 22. For example, if M_{min} = 10 amu, M_{max} is 220 amu.
- Neighbor trolley minimum interval dmin = $M_{max}/58$. For example, if M_{max} = 58 amu, dM_{min} = 1 amu. If M_{max} = 70 amu, dM_{min} = 2 amu, if M_{max} = 200 amu, dM_{min} = 4 amu, etc…

This is controlled more precisely by the values stored in the SETUP.

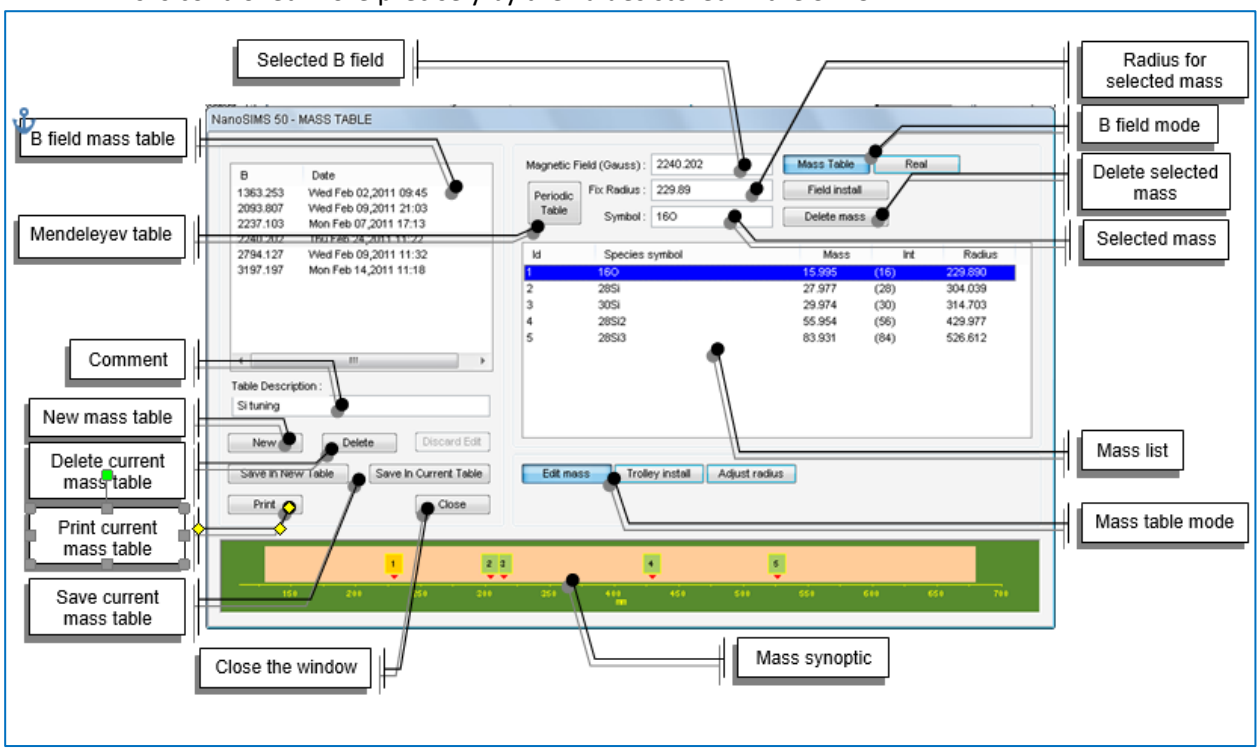


Figure 137: Mass table window

The "**Edit mass**" mode allows the user to **edit** an existing configuration (listed on the left) or **create** a new one ("new" button on the left).
You can **add** an element by typing in its symbol on the "Symbol" field, or **delete** an existing one from the list by selecting it and clicking on "delete mass".
"**Mass Table**" will display the magnetic field for the configuration you are working on, while "**real**" will show the **current** magnetic field value.
"**Field install**" allows you to retrieve the current (= "real") magnetic field value and apply it to the configuration.
You can **edit** the configuration of the mass table by selecting a trolley in the list and changing the associated element or radius.

Once done with the mass table configuration, click on "**trolley install**" or "**adjust radius**" to move the trolleys or adjust the magnetic field radius, respectively.

5.2.10 Motor Reset/Setup

This program (more of a service tool than a tuning function) allows the user to reset the motorized parts of the instrument, such as detectors, slits and diaphragms. This is useful when one of them starts acting up, or when there is an error in communication.
For each of those elements, it is possible to either “RESET” or “initialize” (INIT) it.
RESET means the trolley or slit goes **back to its zero position** (an electrical stop contact mounted on the next trolley or a fixed support) before moving back to a **default, *Reset position*** defined in the SETUP.
With **INIT**, the trolley moves **from one end to the other** of its course (defined by the two electrical stops), before moving **back to Reset position.** When resetting the motorization of the Multicollection trolleys, it is recommended to use the ***Init*** command, while ***Reset*** is enough for all other motors.
In the particular case of the MC trolleys, which are all moving together, the first trolley is stopped by a fixed electrical contact at the end of the rail, while all following trolleys are stopped by an electrical stop on the previous trolley (trolley 1 is stopped by an absolute stop, while trolley 2 will be stopped by the electrical stop mounted on trolley 1, etc…)

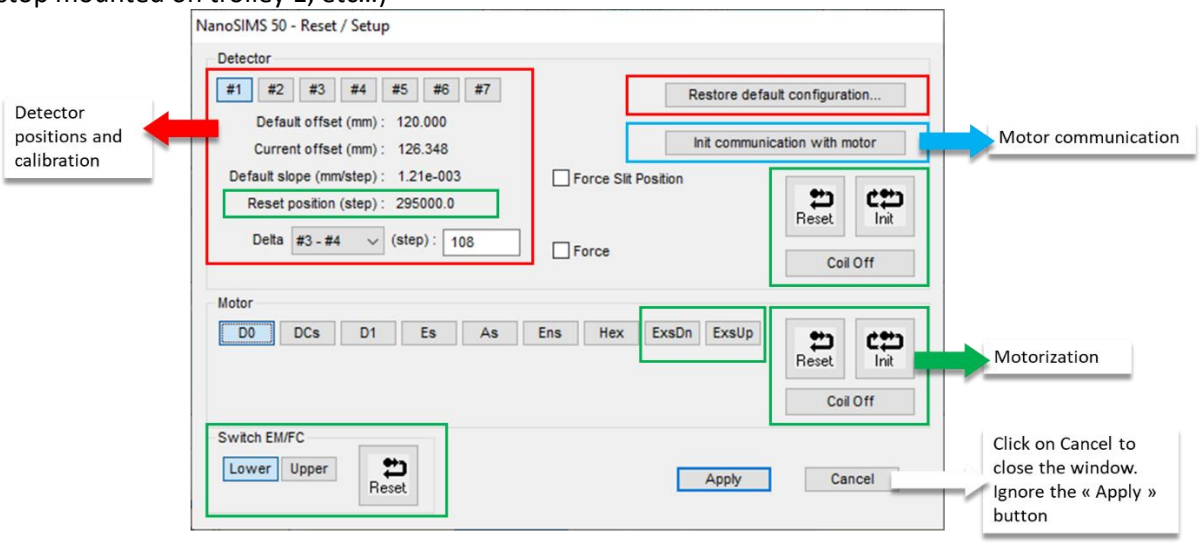


Figure 138: Reset/Setup window

[1] Detector positions and calibration:
- Default offset: original position set in the factory. Offset from the 0 mm radius position defined by the mechanical stop at the inner side of the muticollection chamber.
- Current offset: most recent offset value. It is updated each time a calibration with the magnetic field is done (see advanced operation 0)
- Default slope: size of a step in mm
- Reset position: position the trolley goes back to after “Reset” or “Init”
- Delta: difference (in steps) over the whole course of the multi-collection chamber between 2 consecutive moveable detectors. This difference must be under 400 steps. If the interval is over 400 steps, an “INIT” is necessary to recalibrate the trolley course.
- Restore default configuration: reset the current offset to default offset. This is to be used with caution as it will impact the magnetic field calibration (see advanced operation 0) .

[2] Motor communication:
Every time the motorization electronics has been shut down (Cabinet B), the communication must be re-established. Do so by clicking on “Init communication with motor”.

[3] Motorization:
- **Detectors**: If the trolley position seems inaccurate (for instance, you get a collision warning message that trolleys are touching each other while they, in fact, are not), you can re-calibrate the position reading by doing an **INIT**. **All trolleys** will then move to their Reset position.

- **Motor** (diaphragms, slits, Hexapole): Similarly, if the motors of the diaphragms and slits do not work properly, do a **RESET**.
- **particular case of the EM/FC switch and exit slits**: if a reading error on the EM/FC switch motor or the exit slit motor occurs, then, when trying to move a trolley, you will get an error message and the trolley will not move. You then need to do a **RESET** of either the exit slits or the EM/FC switch.
    - Exit slits (in Motor section of the window): Click on ExsDn (Exit slit Down), then **RESET**. When the motors stop moving, Click on ExsUp (Exit slit Up), then **RESET**.
    - Switch EM/FC (in Switch EM/FC section of the window): Click on Lower, then **RESET**. When the motors stop moving, click on Upper then **RESET**.
- **Coil Off** (both Detectors and Motor sections): shut down the motors. To be used if anything wrong happens when moving a motor (strange noise, unexpected collision, a motor doesn't stop, etc…). Once the problem has been solved, any command to move a detector will automatically switch the coils back on.

When the RESET or INIT process is done (all motors have stopped moving), click on Cancel to close the window.

Note: The *Force* options are for Cameca staff only. Do not use them.

5.2.11 FC Calib : Faraday cup intercalibration

This Faraday Cup calibration acquisition is used to inter-calibrate the Faraday cups, i.e. adjust their responses so that they would all be the same. Waiting time and counting time are independently adjustable by user. The following window (Figure 139) displays settings for FC Calibration acquisition.
The principle is to switch successively a calibrated electronic voltage (0V, 9V, -9V and 0V) on the different preamplifiers, measure the various responses and calibrate them numerically in order to get a uniform gain and offset. See 9.2.13.1 for procedure details.

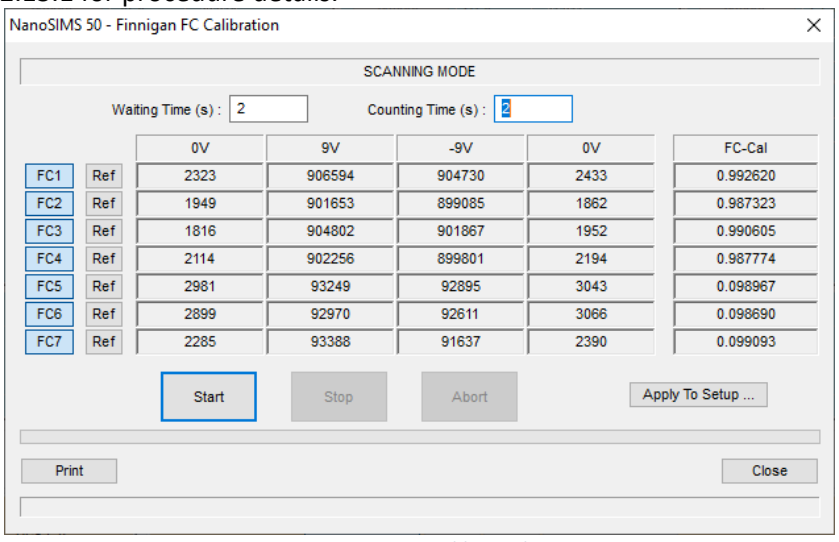


Figure 139: FC calib window

5.2.12 Live Isotopic ratio Monitor

This tool (Figure 140) allows the user to monitor "live" the element or isotope ratio between the signals of 2 different detectors. Its use is limited to the **Multi-collection mode**, with the beam ON.

The "ratio" is the simple ratio between numerator and denominator, while "sigma" translates the difference between the ratio measured here (R) with a known standard ratio (R0) defined by the user*. "Sigma" is expressed as σ = (R/R0 -1) x1000 (‰). You can also smooth the variations by integrating the data

over several measures. The measure is defined by the integration time in TUNING (usually set at 0.54s, see 5.2.3). The displayed ratio is a raw ratio based on detected counts, and does not take into account typical corrections such as instrumental fractionation, dead-time correction, QSA effect, detector efficiencies, etc…

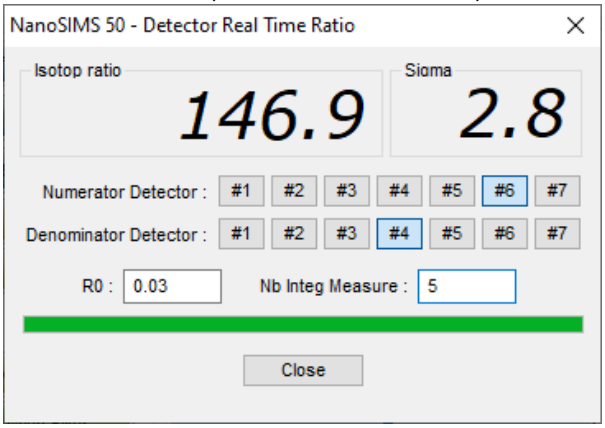


Figure 140: Live isotopic ratio window

5.2.13 Wien Filter mass spectrum recording

The cesium beam is sufficiently pure when reaching the sample in the NanoSIMS and no filtering is necessary. In contrast, the RF-plasma source generates various primary species. The Wien filter is then used with the oxygen primary ion source in order to filter only the desired O ionic species (mostly O_2^+, O^-, O_2^- and O_3^-). O_2^+ gives the highest current but is of limited use on the NanoSIMS because of its opposite polarity design: the negative ions are rarely intense under O_2^+ bombardment. With negative primary ions, one generally uses O^- (giving the highest beam density and current, and smallest spot size) but also O_2^- or O_3^- in some cases (better ionization yield and depth resolution compared to O^- but slower due to the lower generated current). Adjusting RF-plasma parameters (pressure, coil) or internal cesium contamination can optimize slightly one peak vs the others.

To select the desired O species, a mass spectrum is recorded using the Wien Filter function. It works identically whether in negative or positive mode. This Wien filter acquisition program measures the variations of the beam current in FCp as a function of the voltage applied to the Wien filter Coil (CWf). Hence, it records a mass spectrum of the ionic species emitted by the source, allowing the user to select the primary ion of interest.
Start value, step value, number of point and counting time are independently adjustable by user:

- You can choose to do the scan in FCp or FCo (if selecting FCo, remember to go to FCo in the Navigator)
- Set the scanning parameters. Typical values are:
    - Start voltage: -40 V
    - Voltage step: 0.3 V
    - Point numbers: 130
    - Counting time: 1 s
- Launch the scan and apply CL/Value on the desired peak.

Figure 141 displays settings for Wien filter mass spectrum acquisition.

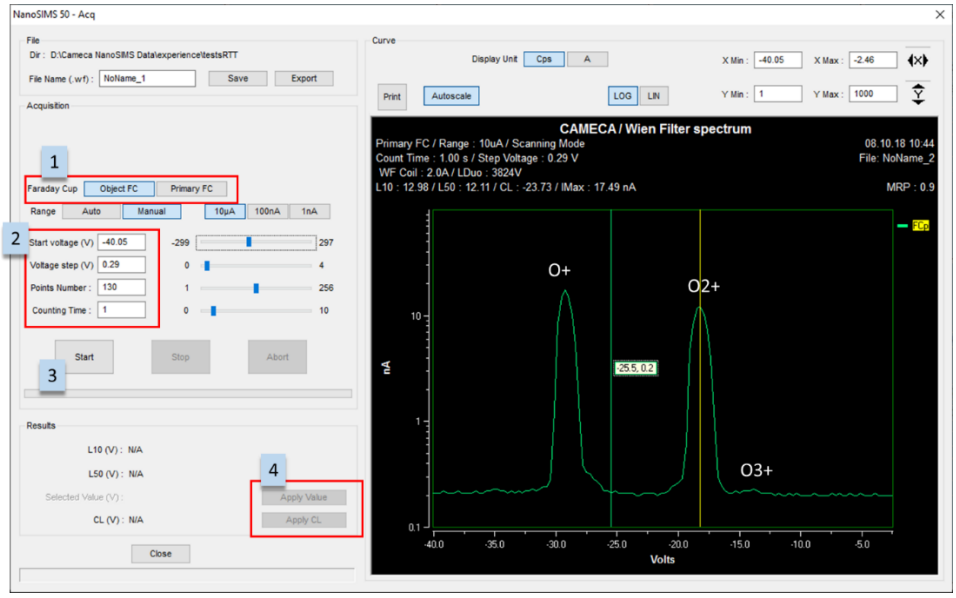


Figure 141: Wien Filter acquisition window

5.2.14 Trolley Step Scan mass spectrum acquisition

This Trolley Step Scan acquisition is used to record a mass spectrum on one trolley by moving the trolley while the magnetic field remains constant. This allows the user to find the position of a given mass and position the detector at the right mass. This can be useful when the user is looking for a mass but doesn't want to change the magnetic field (for example when other trolleys have already been tuned to the right masses).

The trolley Step Scan thus measures the variations of the EM counts as a function of the trolley position. The user can define the range of the scan either in term of start and end position (radius, in mm), or in mass (Figure 142). Trolley step value, number of point and counting time are independently adjustable by user.

Note: For large mass scans, one needs to ensure not to miss any peak by setting too large a detector step. From the basic relation R = sqrt (2mU/q) /B , one gets R_{max}/R_{min} = k * sqrt (M_{max}/M_{min}): the unit mass lines are physically nearer at higher radiuses than at lower radiuses. However, the mechanical steps are constant. Assuming, as an example, a MRP of 6000 at M_{max} = 60amu for R_{max} =400mm. Using dm/m = 2 dR/R, dR = 400/6000 /2 = 33µm. Choosing ~15µm steps will ensure not to miss information.

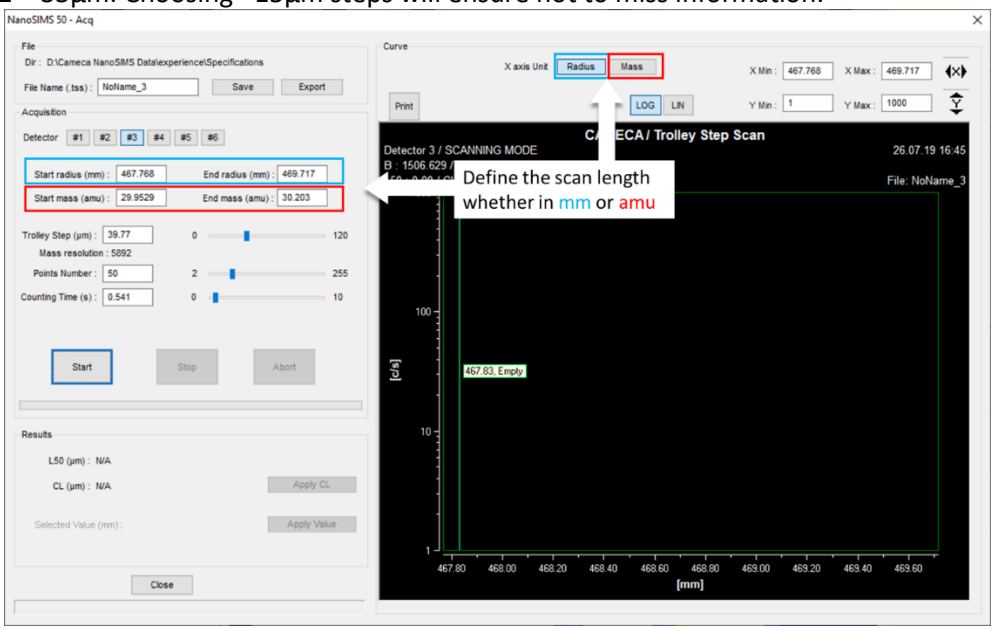


Figure 142: Trolley Step Scan acquisition window

5.2.15 Energy (EOW scanning)

When a sample is slightly charging of a few eV (i.e. the signal is not stable without completely dropping) it can be possible to compensate the charging effect by adjusting the sample HV (E0W). For a few eV the primary focusing with EOP will not be changed. EOS might need fine tuning.
However, for high precision analyses, where the stability of the signal is primordial, it is recommended to use conductive thin coating (C, Pt, etc…) and the e-gun to compensate charges, especially for non-homogeneous samples.
The primary ion source voltage being kept constant, varying EOW (= adding an offset to the sample HV) will vary the primary ion impact energy as well as the secondary ion kinetic energy. The program measures the variations of the secondary ion intensity as a function of the E0W HV offset. The increment of the sample HV offset, the number of point and the counting time per point are independently adjustable. The following window (Figure 143 and Figure 144) displays settings for Energy acquisition.

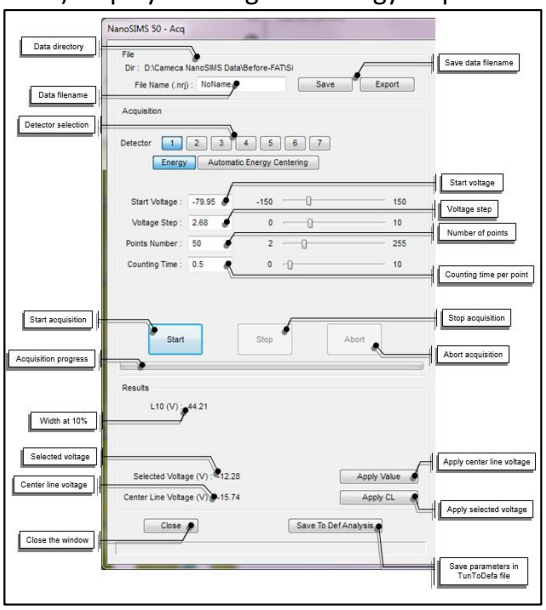


Figure 143: Energy acquisition window

Figure 144 displays the Automatic Energy Centering part of the Energy acquisition window which allows to center automatically the maximum of the Energy curve.

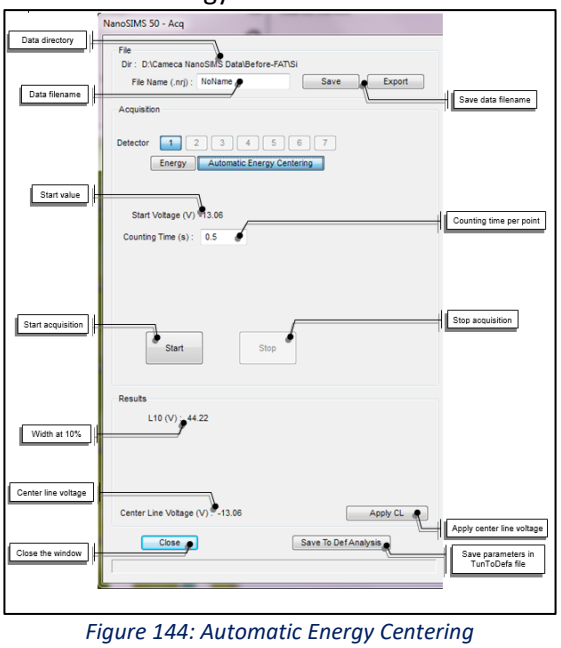


Figure 144: Automatic Energy Centering

5.2.16 BarGraph mass spectrum acquisition

This program is used to record a mass spectrum over a defined mass range by scanning the B-field and measuring the signal variation on one or several multicollection detectors. A *bargraph* analysis is recorded at a constant number of data points per peak width which means *the B-field step varies over the mass range*. This program is very useful to identify the presence of certain species in samples of unknown composition, or the exact "position" of the peaks when the mass table is not perfectly calibrated. In order to properly see all main masses over the mass range, it is recommended to use a BField step (δB) of 0.3 G maximum.

Scanning over a large range of masses thus takes time. If you need to scan over a large mass range (several dozen of masses), it can be useful to use in parallel several trolleys located over the length of the multi-collection chamber. In this case, it is best to set the masse ranges for each detector in such a way that they will partially overlap, in order to identify common peaks and make it easy to reconstruct the whole spectrum.

Note that this is different from the high mass resolution (HMR) program which deflect the secondary beam at the exit slit in order to precisely scan a given mass and identify interferences.

B_i and B_f are defined by the user. Over the B field range [B_i, B_f] and for a given mass resolution, the B field is scanned with an increment δB computed with the relationship following:

$$\delta B = \frac{1}{2} \times \frac{B_f - B_i}{\text{Mass resolution}}$$

Inversely, if δB is set by the user, the mass resolution is computed via the same relationship. When setting the parameters, the user must thus keep in mind that all parameters are interconnected.

How to use this program (Figure 145):

1- select one or several detectors
2- for each detector selected, enter a start and end mass. Beware that because trolleys are fixed and the magnetic field is common to all, changing the range of a scan will affect all of them. Ranges for all detectors will automatically adjust when changing one.
3- adjust the Bfield step (<0.3 G)
4- Start

Once the scan is done, you can set a detector to a desired mass. To do so:

5- If multiple detectors, select the one you want to set, and simply move the cursor to the desired peak
6- "Apply Value" to change the magnetic field so that the mass will be on the selected detector. Keep in mind that this will change the magnetic field value and thus affect all detectors.

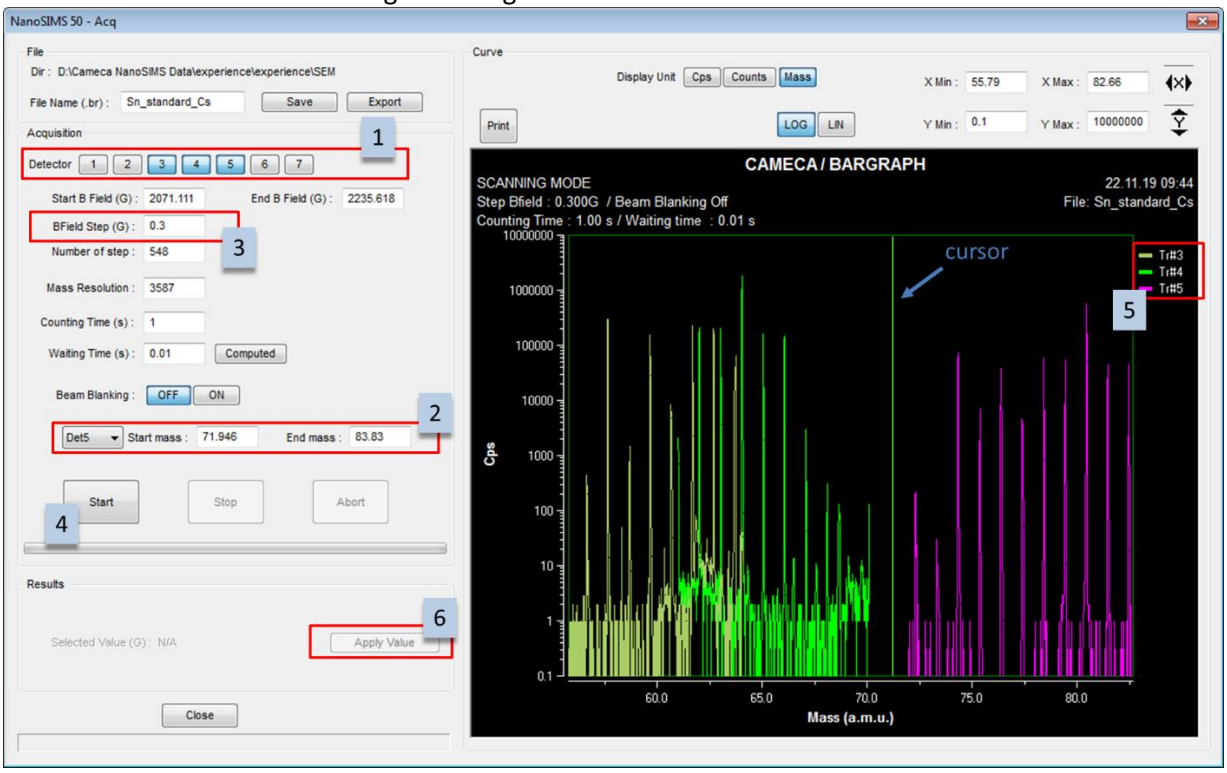


Figure 145: Bar Graph acquisition window

5.2.17 Beam Stability recording

This program is used to check the stability of either the primary or the secondary beams. The primary (or secondary) beam intensity is measured as a function of time.

To start a primary beam stability test:

- Open the "Beam Stability" window (Figure 146).
- If you want to save the file, enter a file name.
- Select the signal to be recorded: primary beam current (FCp, FCo) or secondary ion signal (TIC, multicollection detector)
- Enter the total acquisition time and/or the number of points and/or the counting time per point needed for the beam stability recording.
- Click on Start. The analysis is running and a real-time curve signal/time is displayed (the analysis can be stopped or aborted).

- At the end of analysis, some statistical values are computed and displayed in the result window. Click on “Show Results” to open this result window (Figure 147).

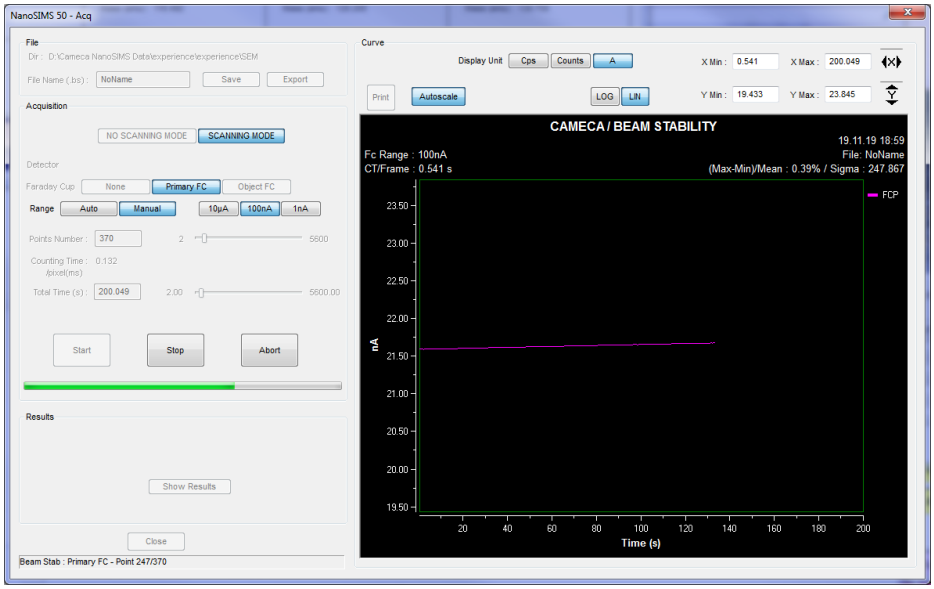


Figure 146: Beam Stability acquisition window

For the stability measurement, parameters are printed in the results window (Figure 147):

- *Maximum*: is the maximum intensity measured.
- *Minimum*: is the minimum intensity measured.
- *Mean*: is the mean intensity measured.
- *Geom Mean*: geometrical mean
- *S.D (%)*: is the (relative) standard deviation computed for all data points.
- *Poisson (%)*: is the Poisson statistic computed for all data points.
- *Sigma*: is the sigma computed for all data points.
- *Radius (mm)*: is the radius of the selected detector.
- *Mass (a.m.u.)*: is the mass of the selected detector.

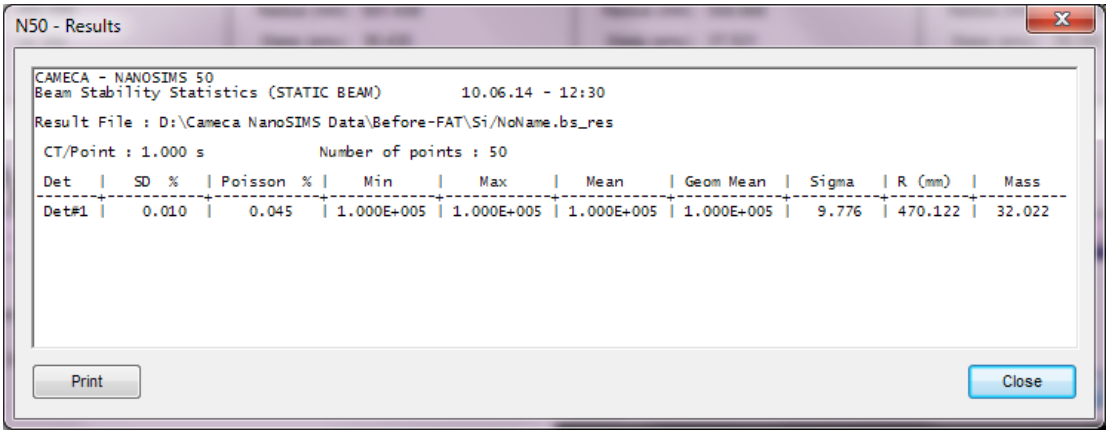


Figure 147: Results window for a beam stability acquisition

5.2.18 E0S Focusing Scan

This E0S scanning routine is used to measure the variations of the signal from a multicollection detector as a function of E0S. This allows the user to focus a secondary ion beam waist at the level of the entrance slit, thus maximizing the mass analyzer transmission. It is generally used in combination with the centering of the beam in the entrance slit using P2-P3 and Cy (see chapter 0).
The final EOS scan must be done with the selected entrance slit in position.
E0S start value, step value, number of point and counting time are independently adjustable by user.
Once the EOS scan curve is recorded, it is possible to apply the optimum EOS voltage to the instrument (Apply CL or, if the program fails to calculate a CL, position manually the cursor on the scan and "apply value").

Typical values are:

- Start Voltage: -7300 V (refer to your installation documentation for reference of the expected CL value)
- Voltage Step: 10
- Points number: 40
- Counting Time: 0.54

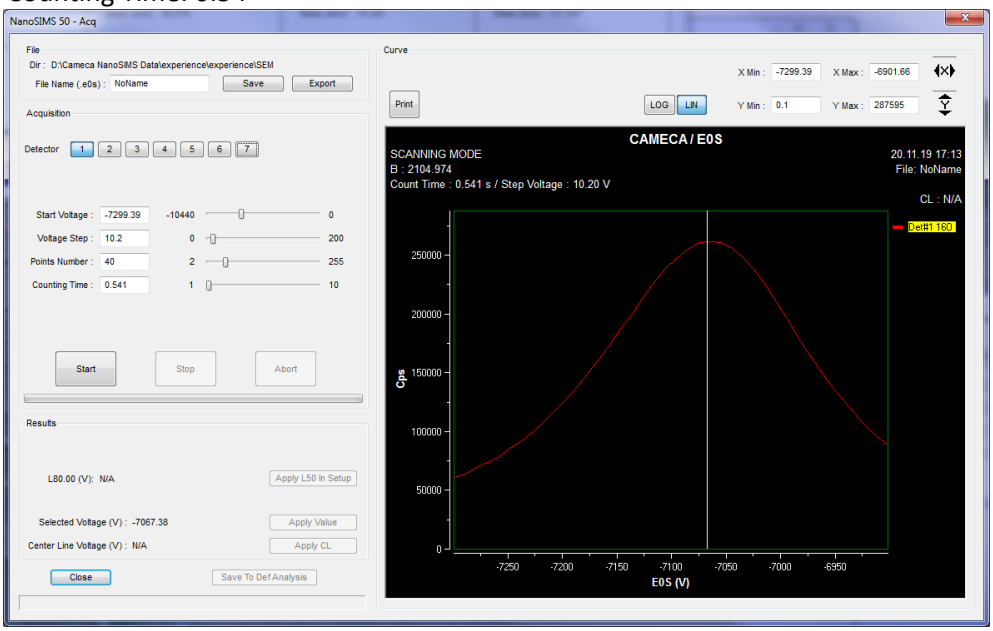


Figure 148: EOS acquisition window

It is also possible to do an *automatic* EOS centering. This routine will determine automatically the value of E0S voltage providing the maximum of secondary beam signal.
Note that the term "centering" does not refer to the centering of the beam waist in the entrance slit (this is performed by P2-P3 and Cy) but the generic centering of the recorded curve to its max or center position.

Please refer to chapter 0 for details on automatic centering routines.

5.2.19 Secondary Ion Beam Centering scan

This Secondary Ion Beam scanning window is used to center the beam in the entrance slit by varying P2 and P3 deflection voltages in synchronism (in the VERTICAL plane) and the Cy deflection voltage (in the HORIZONTAL plane), in order to maximize the transmission (= the percentage of beam going through the slit). It is generally used interlaced with EOS optimization (see previous chapter).
The tool records the variations of counts on a selected multicollection detector by scanning either Cy (HORIZONTAL) or P3 (VERTICAL). Start value, step value, number of point and counting time are independently adjustable by user (Figure 149).

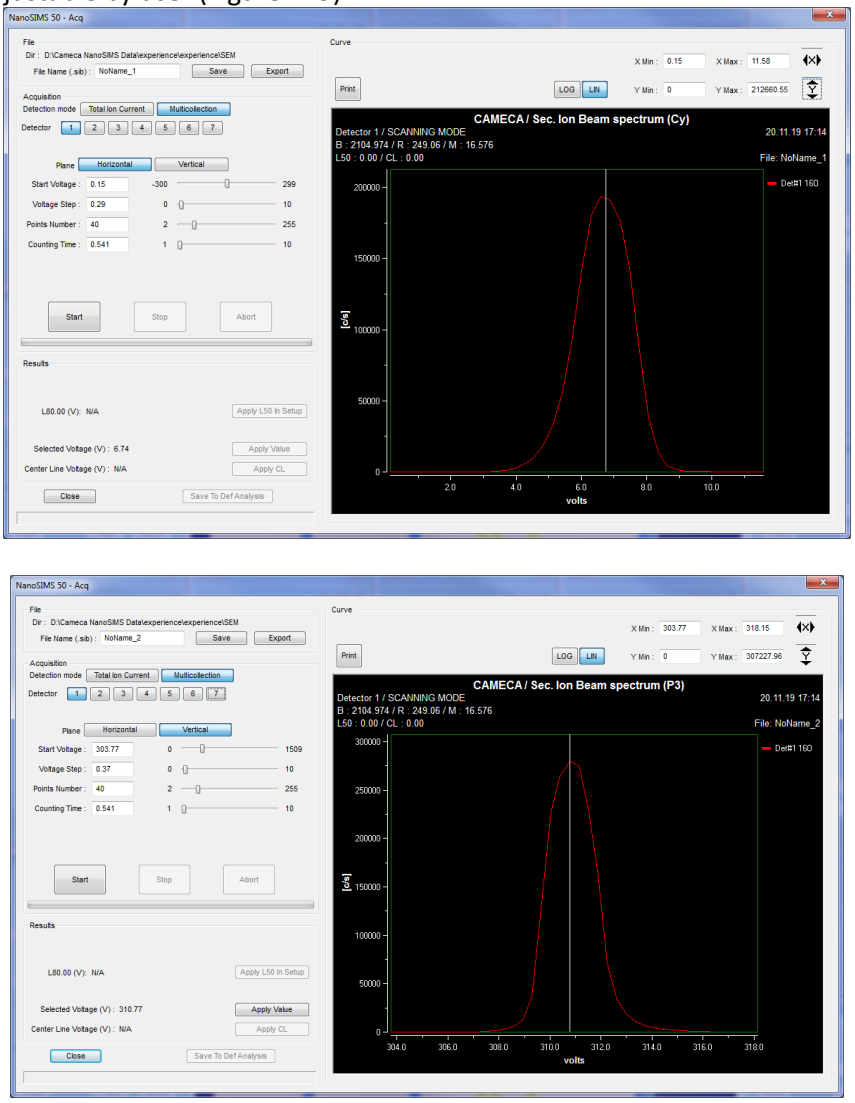


Figure 149: Second Ion Beam centering, horizontal (top) and vertical (bottom)

Similarly to the EOS centering, the Automatic Beam Centering part of the Secondary Ion Beam acquisition window allows to set automatically the maximum of the secondary ion beam signal (Cy and P3 values). Please refer to chapter 9.3.3 for details on automatic centering routines.

Caution: While using the Sec. Ion Beam centering software which determines the optimum values for Cy, P2 and P3, the ratio P2/P3 has to be properly set. This ratio allows to keep the secondary ion beam parallel to the horizontal axis while changing its height in ES by varying P2 and P3 in synchronism. This ratio is very sensitive to the setting of LF2. As LF2 is generally set to 20 000 bits, the ratio P3/P2 must be set near 0.36. This coefficient is introduced in the Setup chapter 5.9.4.6 Secondary Ion Beam. We recommend to NOT modify the value determined by CAMECA production or service engineer. This tuning goes beyond the scope of this users guide.

5.2.20 MRP OPTI

This program is an automated routine for the adjustment of Q and LF4 in order to optimize the mass resolving power (MRP) on a chosen detector. The procedure is all automated. The HMR window parameters are taken from the last HMR acquisition. Simply select the trolley and click on start. The program will first adjust Q, then LF4, and finally check Q again, so as to optimize the MRP. Q will be incremented by +/- 1 between scans, while LF4 steps can be adjusted by the user (steps of 100 bits are recommended). The graph on the right shows progression of each HMR scan, while the small graph shows evolution of the MRP for each value of Q (or LF4). When the program is done, the best Q and LF4 values are automatically applied. The user can check via the keyboard the applied values.

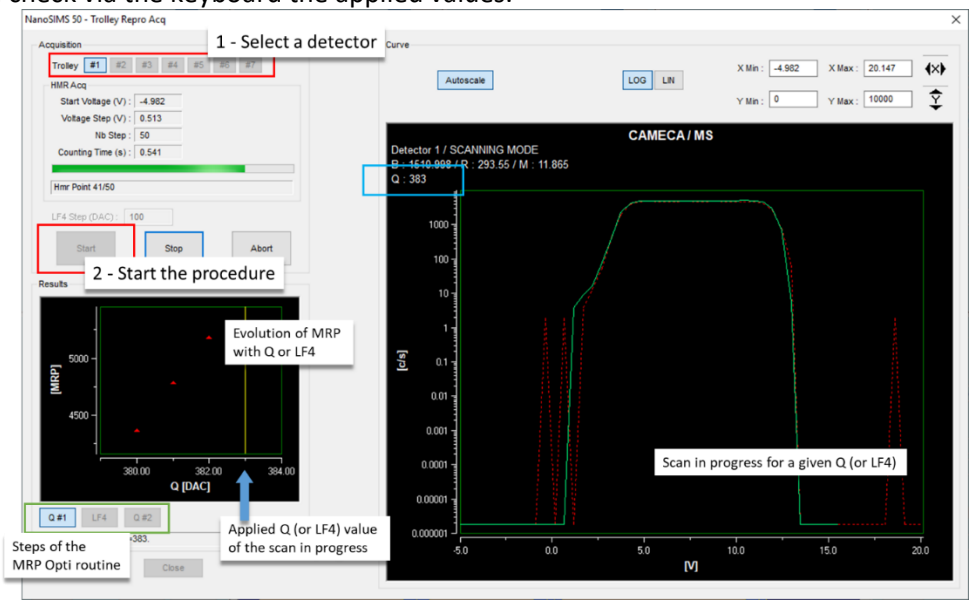


Figure 150: MRP Opti program

Note that the optimum Q might differ from one trolley to another. It is recommended to note the optimum Q for each detector and then find the best compromise (see chapter 9.1.13 for details on the MRP and how to adjust it manually).

5.2.21 PHD acquisition window

Refer to chapter 2.1.4.3 to learn EM and PHD principles and background.
Electron multipliers age as they are used. Therefore, it is necessary to periodically readjust their pulse height distribution by increasing the applied high voltage. The Pulse Height Distribution (PHD= acquisition program records the EM PHD distribution curve, where the EM threshold is the scanned parameter and the EM counts the acquired signal.
EM Threshold start value, step value, number of point and counting time are independently adjustable by user. The following window (Figure 151) displays settings for PHD acquisition.

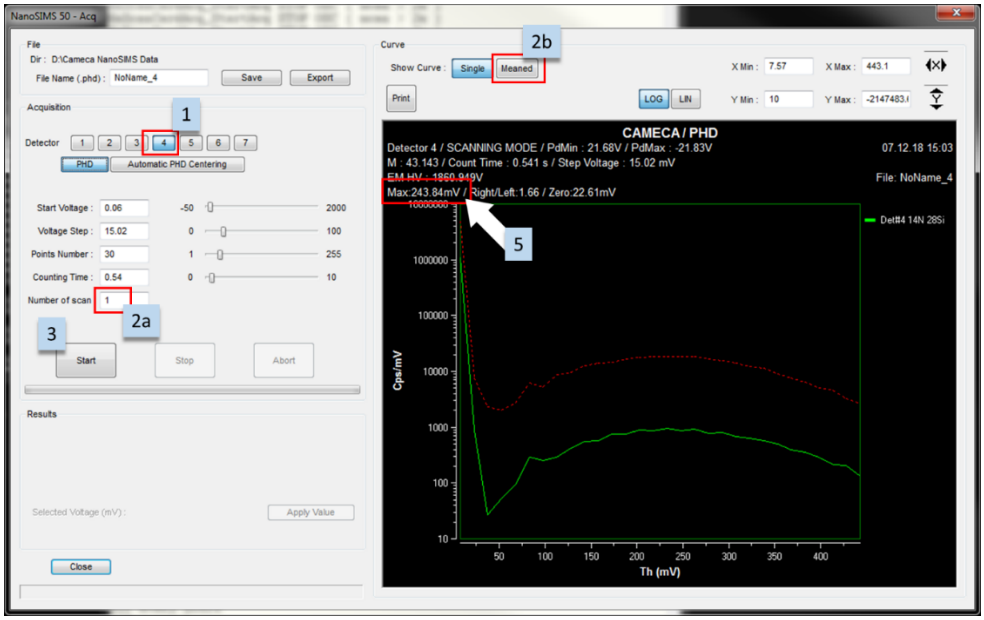


Figure 151: PHD acquisition window

Parameters should usually set as:

- Start voltage: 0
- Voltage step: 15
- Point number: 30
- Counting time: 0.54

For a good accuracy of the measurement, it is recommended to check an EM PHD with the EM set to receive a high count (of the order of several 100 000 cps). If necessary, the user can average several scans to smooth the PHD scan: Set the number of scan so that the addition would reach about a million counts per second, and click on “meaned” in the scan window (2a and 2b steps in Figure 151).

The maximum of the curve (“Max”, see Figure 151) should be around 220 mV, though can be increased to 250 mV for high precision isotope analyses (the higher the PHD_{max} the stronger the aging effect; but at the same time at higher voltage the effect of a shift of PHD has less influence on the count rate; 250mV seems a good compromize).

If PHD_{max} is too low, the user needs to adjust the EM high voltage via the keyboard (see part 2.3.6). The EM HV will have to be increased as the EM ages. For a new EM, the HV is around 1400 Volts.

For detailed procedure on manual adjustment of threshold and PHD, see Basic Operation chapter 9.1.10.
For automatic PHD & HV_{EM} control during high precision isotopic ratio, see Expert Operation chapter 9.3.3.4

5.2.22 HMR Acquisition configuration

This High Mass Resolution (HMR) spectrum acquisition is used in order to visualize the peak shape and flatness, potential mass interferences, and center the signal in the detector. To do so, it measures the variations of the detector intensity as a function of the deflection plates (Pd) before the exit slit.
Pd deflection plate’s start value, step value, number of point and counting time are independently adjustable by the user. Figure 152 displays the HMR acquisition window.

5.2.22.1 Manual peak centering

In the easiest case (Figure 152), there is a single species at the mass scanned by the HMR program. In that case, the HMR scan shows a single peak. The program will calculate the central position of the peak. You can then click on “Apply CL” to adjust the deflection plate voltage to the center of the flat top of the peak. The deflection voltage is sent to the instrument as the current one for this detector.

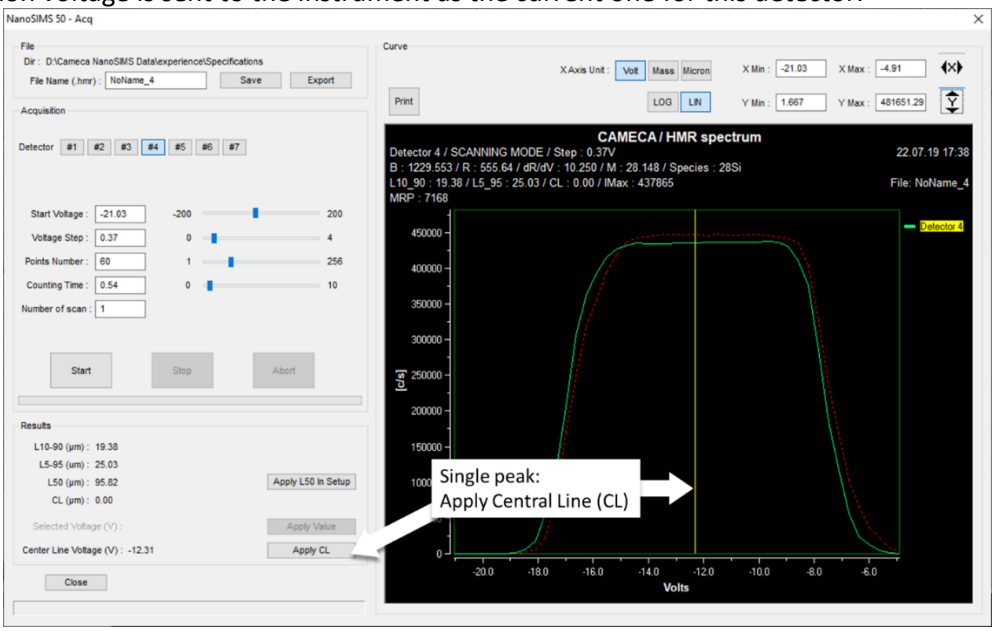


Figure 152: HMR spectrum in the case of a single peak

However, mass interferences often occur. In that case, the HMR spectrum will show a more complex shape (Figure 153). Here, the program will try and calculate a center as usual, placing it at the center of the higher peak. Whether to avoid contribution of interfering species, or because he/she is interested in a lower peak, the user can **manually** select the detection position by clicking with the cursor on the graph.
When using large exit slit for working with flat top peaks, it is important to keep in mind the hidden part of interfering peaks, adding to his neighbor(s). If one peak is much smaller than his intense neighbor the flat top peak of the intense one will not reveal any change. But the interference is nevertheless there ! And will become important if the large peak vanishes (e.g. in a depth profile).
In the example shown in Figure 153, the line is placed on the *left* side of the ^{29}Si peak *(but still on the flat part*), in order to avoid contribution of the (nearly invisible) $^{28}Si^{1}H$ peak tail.

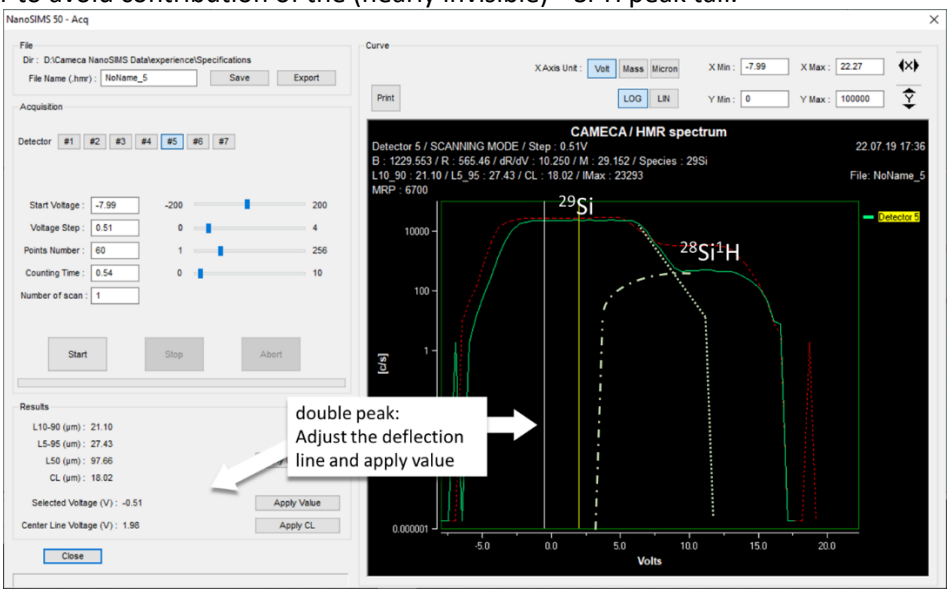


Figure 153: HMR spectrum in the case of a mass interference

5.2.22.2 Automatic Peak Centering (APC) during long acquisitions

For long isotopic analyses, if there is a risk that the magnetic field might shift and thus shift the centering of the beam in the exit slit, it is possible to set an automatic peak centering routine at intervals **during the analysis**. The interval will be defined in DefAnalysis.

To set this routine, an automatic peak centering must be done and saved before the analysis.
Then the "automatic peak centering" option appears. When clicking "start", the program reproduces the peak centering automatically by searching the peak side(s), then the top (red points in Figure 154)
For more details on how to set/use the HMR automatic peak centering routine for isotopic analyses see 9.3.3.2.

Note: the option "Ftp" (flat top peak) is not used.

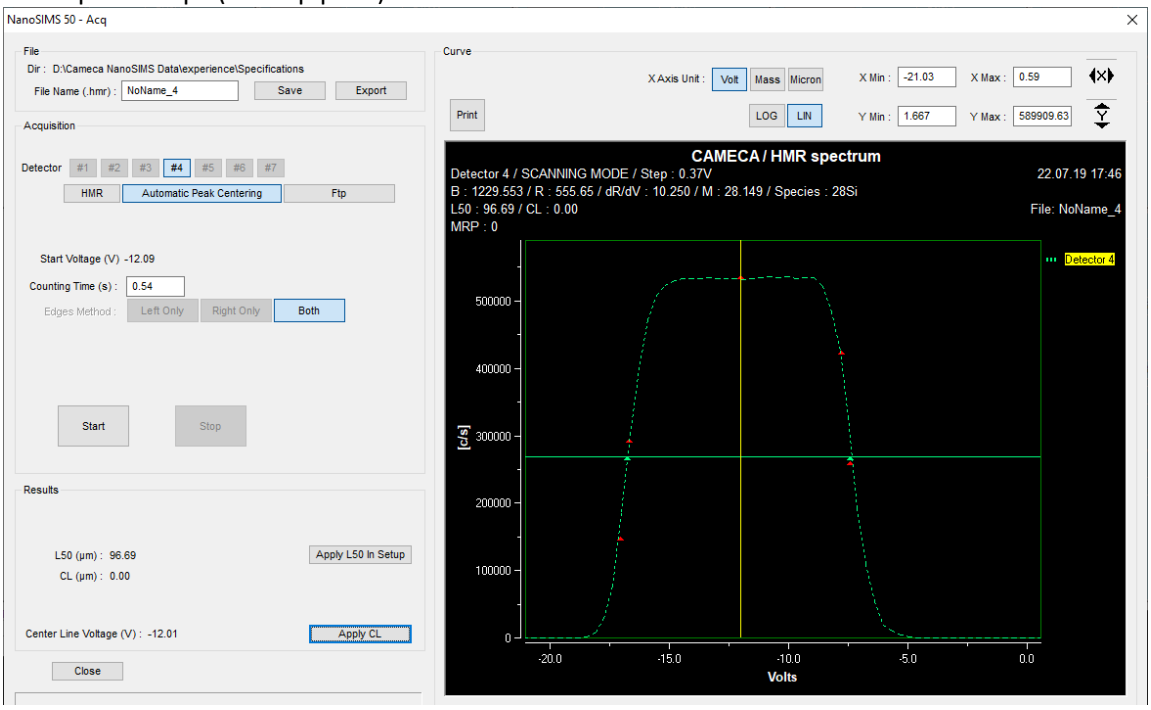


Figure 154: Automatic peak centering window

5.2.23 Real Time Imaging window

The Real Time Imaging (RTI) allows the user to make an ion or electron image of the sample. Note that this is different from a proper analysis acquisition. It is only a "live" display and does not record successive frames.

When opening the RTI program, the following window appears (Figure 155).

- 1) The detection mode (TIC or multicollection) will be automatically selected depending of the detection mode in TUNING. In the case of multicollection, you can choose up to 2 detectors, which will be displayed in the main RTI window, while the others will appear as thumbnails.
- 2) The scanning speed is defined by the counting time/px. A fast scanning (such as 50 µs) is usually preferred for tuning purpose, while a slower scanning (200 µs) gives a better signal/noise ratio, useful for low signal.
- 3) The raster can be adjusted via the this RTI window, or via the Tuning.
- 4) The Working and Scanning frame resolutions are by default set at 256x256 px. The raster defines the size in microns of the working frame (= max scan memory size). The scanning frame is the area actually imaged within this max working frame. It can be reduced by decreasing the number of

pixels in Width and Height. Its position can be adjusted by adding an offset from the top left corner of the working area. This option is rarely used in RTI mode as the RTI imaging is mainly used for tuning purposes.

- 5) Click on Start to launch the imaging.

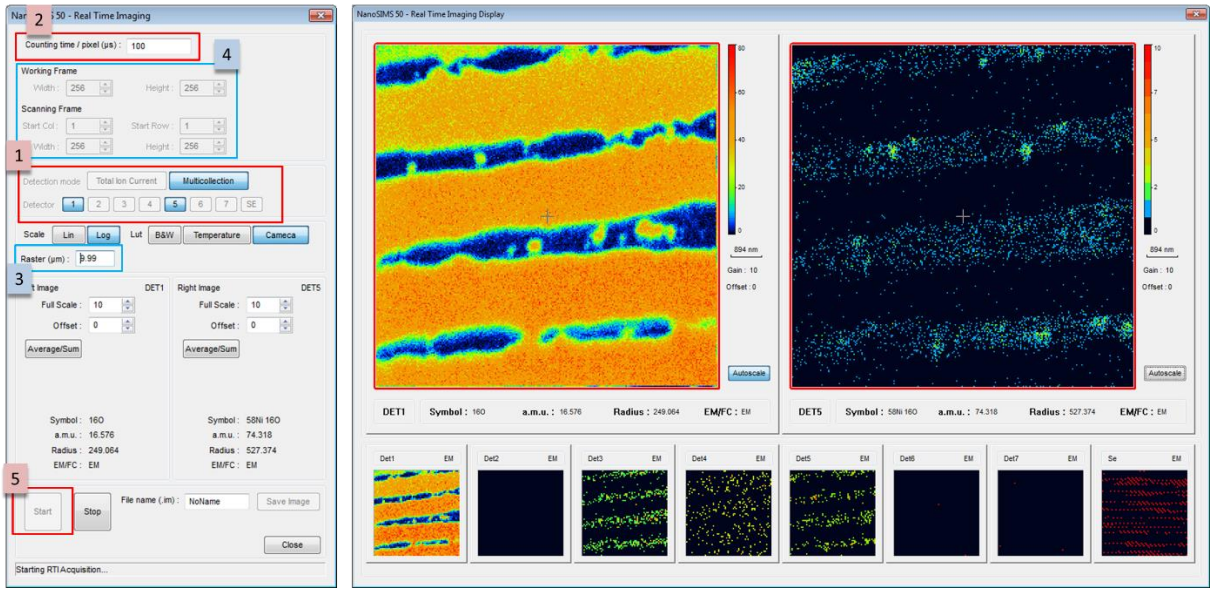


Figure 155: main features of the Real Time Imaging window

- Several parameters allow the user to adjust the two live images from the chosen detectors:
    a. The signal intensity scale can be linear (lin) or logarithmic (log)
    b. The user can choose between three types of color scales: black and white (B&W), “temperature” or “Cameca”.
    c. The maximum (full scale) and minimum (offset) can be adjusted separately for each image
    d. Average/Sum allows to average or add several images. Click once on the button to activate the “average” option, then set the number of planes you wish to average. Click twice on the button to activate the “sum” option, then set the number of planes you wish to add. Click a third time to deactivate this option.
- It is possible to save a “capture” image of the two displayed signals, as a .im file, similar to an image acquisition file. This .im file can then be opened in WinImage (The difference with a proper image acquisition is that here you can only save one plane (frame)).
- When done, click on “stop” to stop the imaging (turn the beam off), or click on “close” to exit the program (it will also turn the beam off)

Real Time imaging can be used to:

- Select the analysis position when moving the stage.
- Follow in real-time the pre-implantation process increasing the useful yield
- Control good charge compensation or good tuning through homogeneous signal or collection
- Check the centering of D1 (A large raster will show the edges of diaphragm D1)
- Adjust the focus of the primary beam with EOP and correct the astigmatism with the octopoles 45 and 90.
- etc…

5.2.24 Tools acquisition window

This Tools acquisition is used to measure the variations of the EM counts on TIC or selected detectors of the multicollection as a function of a selected parameter in order to optimize the tuning. You can select a parameter to scan and the relevant detection mode (FCp, FCo, TIC, multicollection detectors) (Figure 156). All the parameters accessible from this program are: C0X, C0Y, CY, C2X, C2Y, C3X, C3Y, C4X, C4Y, CDuoX, CDuoY, CWF, L0, L1, EOW, EOS, LF2, LF3, SS100 int, SS100 ext, LF4, LF5, Esa int, Esa 1, Esa 2, Esa 3, Esa 4, Esa 5, Esa 6, Esa 7, E-gun Be, C5X, C5Y, C6X, C6Y, LF7, Z axis (refer to the instrument schematics in part 2.1 of this manual for details of all those elements).

As for all scanning tools, start value, step value, number of point and counting time are independently adjustable by the user.

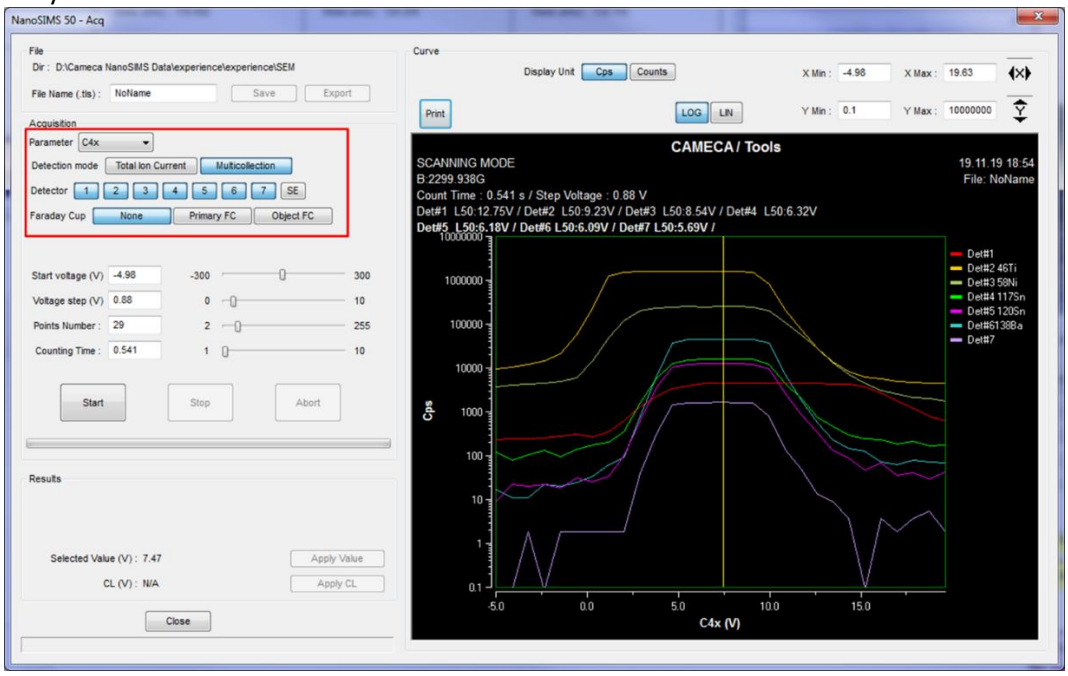


Figure 156: Tools acquisition window, here showing the scanning of parameter C4X on the multicollection Ems

5.2.25 Cesium source leak current recording

This Leak Current acquisition is used to measure the variations of the leak current of the Cs^+ source versus time. Number of point and counting time are independently adjustable by the user. The following window (Figure 157) displays settings for Leak Current acquisition.
The HV source power supply delivers a total current = I ionizer + I reservoir + Ileak. The latter, I leak, comes from plasma possibly created around the polarized source and from secondary electrons re-emitted by the extractor toward the source. If too much cesium has been coating the extractor I leak might increase, show bursts or be unstable. It is often the sign that one should open the source and clean the contaminated extractor electrode.

The leak current of a cesium source in good condition should be stable and stay below 0.2 mA.

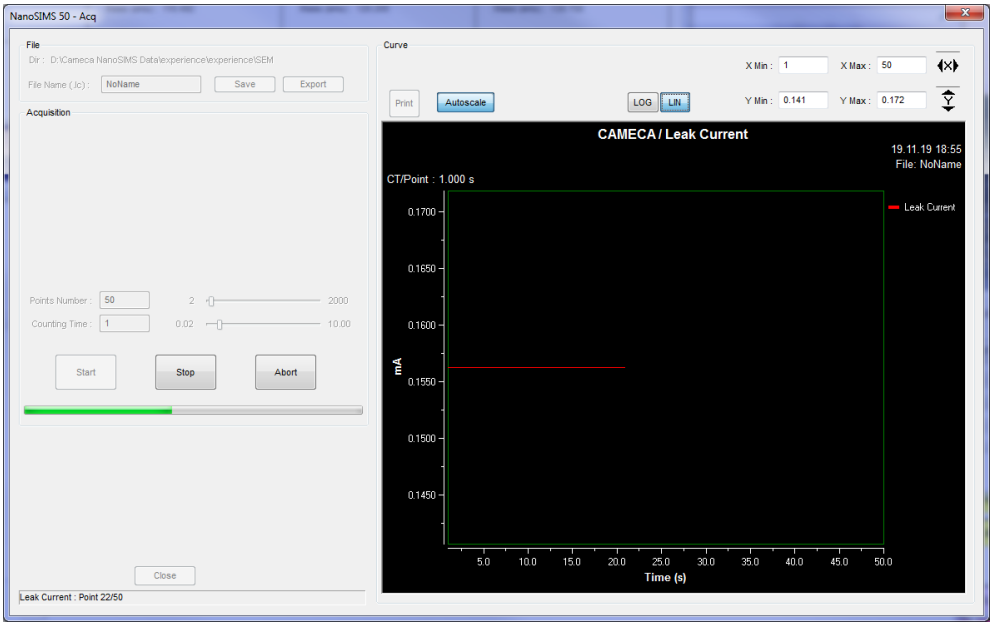


Figure 157: Leak current acquisition window

5.2.26 The sum-up window

This window is a reduced view of the main window and appears during certain acquisitions. This allows to see the signal live during acquisitions.

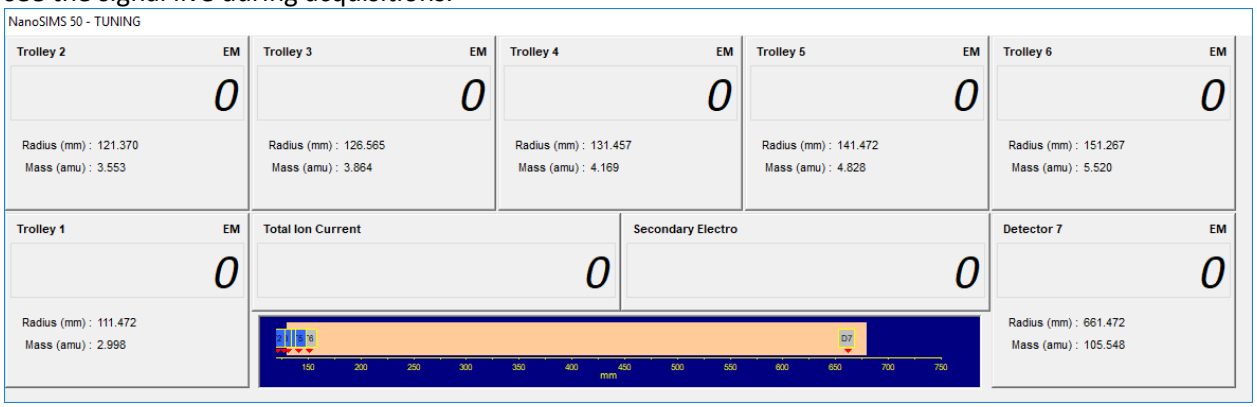


Figure 158: Sum-up tuning window

5.3 Navigator

5.3.1 Main window

5.3.1.1 Terminology

Holder represents the physical sample holder object which is used to carry samples to be analyzed in the analysis chamber. There are different holder types which will be selected according to the sample size, but all of them are designed as *Holder* in this document. These holders are equipped with ***Windows*** (holes) of various sizes in which the samples are loaded.
The *Navigator* program is divided in two main sections: *Navigation* and *Edition*, and in both modes, it uses the concept of *present* holder, *present* window or *present* sample.
Navigation mode: dedicated to the navigation over the holder loaded in the analysis chamber.
Edition mode: dedicated to the edition mode for defining the holder configuration, the sample ID's….
In the *Navigation* mode,

- The present holder is the holder which is loaded in the analysis chamber.
- The present window is the window of the present holder which is in the analysis position (i.e. within the field of view of the secondary ion optics)
- The present sample is the sample loaded in the present window.

In the *Edition* mode,

- The present holder is the holder being under edition.
- The present window is the window being under edition.
- The present sample is the sample being under edition

5.3.1.2 General concepts of the Navigator

The *Navigator* program allows to fully define the sample holder loaded in the analysis chamber by associating three different types of information:

- Sample holder configuration: it defines the size and the position of the windows present in the holder. A file system allows to save different sample holder configurations (e.g. “Biology”, “24 holes”, “WU-MPI”, etc…)
- Sample configuration: it defines the features (name, matrix…) of the sample loaded in every window. A file system allows to save different sample configurations.
- Preset positions: they are positions saved by the operator. A file system allows to save different Preset position configurations.

When a sample holder has been physically loaded in the sample stage and using the *Load* command, the sample holder is displayed with its last sample configuration. The operator can then change the sample configuration and the preset point configuration to be consistent with the targeted analyses.
Note that the software controls the consistency between sample configuration and sample holder configuration (i.e number of windows and number of samples) that the operator is attempting to associate.

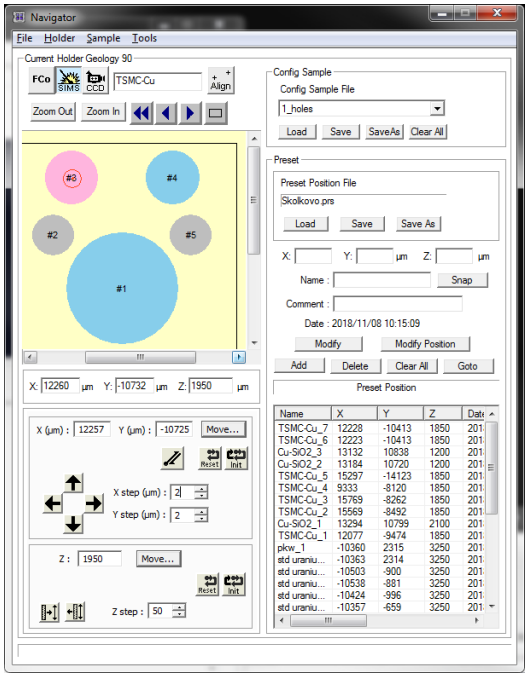


Figure 159: Navigation window

5.3.1.3 Getting started

The *Navigator* program is used to drive the sample stage along the three axes X, Y, Z. Thus, it allows the operator to navigate on the sample holder loaded in the analysis chamber. The *Navigator* program also offers edition functions in order to define the name and the position of the samples mounted on the holder, and preset analysis positions.

The main functions available in this program are visible in the Figure 160 below:

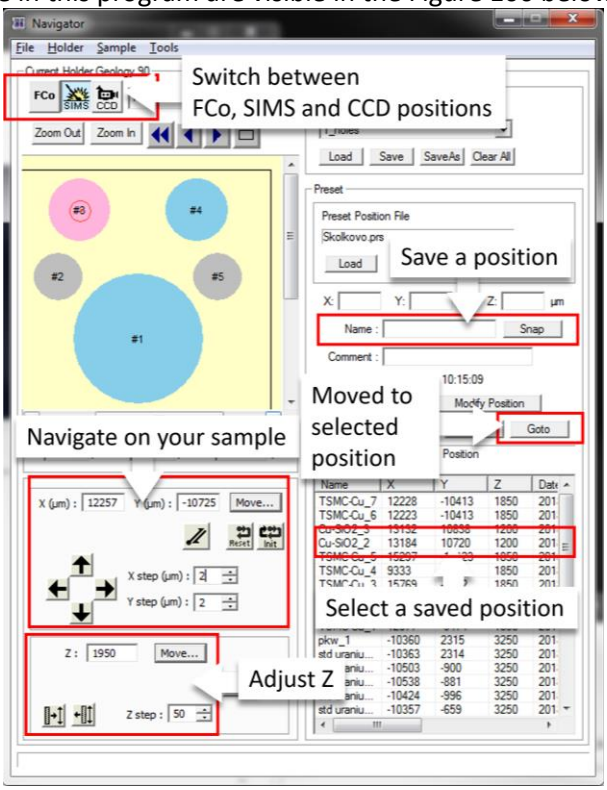


Figure 160: main window of the navigator

Among the main functions are:

- **Switch between FCo, SIMS and CCD mode (**Erreur ! Source du renvoi introuvable.**)**.
  - In SIMS mode, the sample is placed on the path of the primary beam and is thus sputtered away. This is the mode for everything that requires a secondary signal or for primary ion implantation.
  - The FCo mode moves the sample holder away (up) so that the primary beam reaches, through a hole in the stage the fixed Faraday Cup located in front of the beam behind the moveable sample holder, thus giving the primary beam current intensity at the sample.
  - Finally, the **CCD** mode moves up the sample from the current SIMS position to the CCD camera of the microscope, allowing the user to see optically the exact same sample area as in SIMS and navigate in optical mode without sputtering the sample.

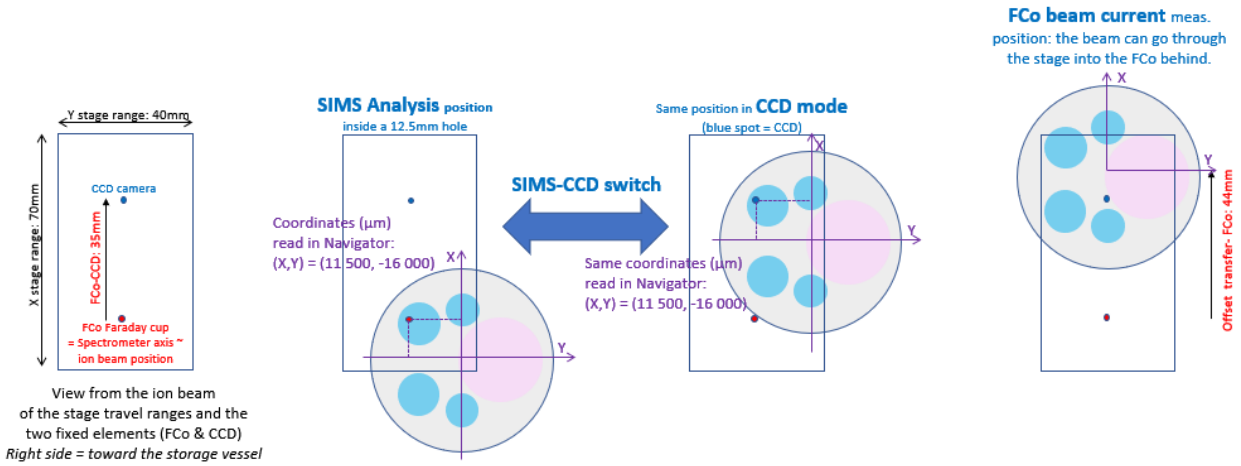


Figure 161: FCo/SIMS/CCD positions and relative X-Y axes, here with a large sample holder

X-Y accessible area:

Due to the two different positions of SIMS analysis and optical camera observation (CCD) the area that can be BOTH accessed in SIMS and CCD is ~39 x 39 mm. It is shown in green in Figure 162: Some sample holders showing in green the area accessible BOTH by SIMS and CCD the schematic below and superimposed on some of the sample holders explaining positions of the holes.

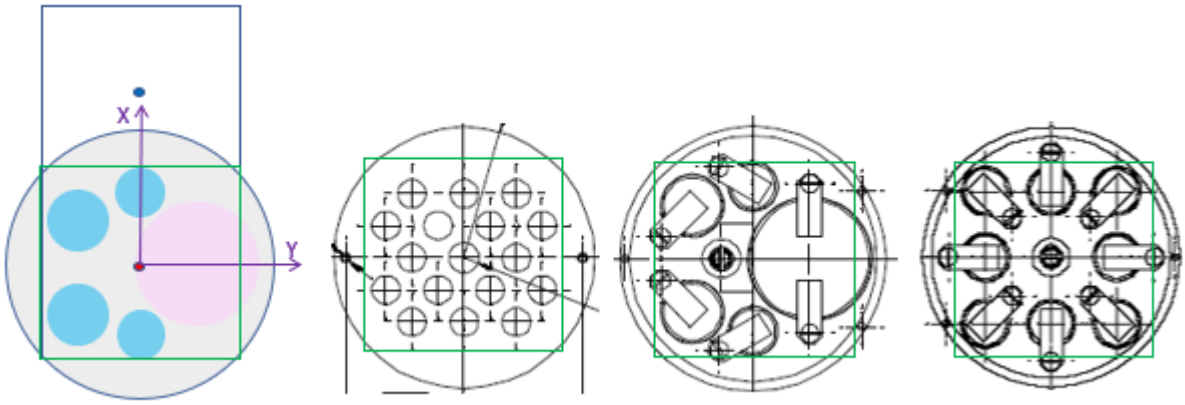


Figure 162: Some sample holders showing in green the area accessible BOTH by SIMS and CCD

Sample stage motion (see chapter 5.3.1.6 for more details):

- The user can first move the stage using the X-Y-Z arrows buttons,
- or directly type X, Y, or Z coordinates and hit “move” to go directly to the position,

- or access a position by clicking on it in the sample holder synoptic.

Note: in addition to these functions available through the Navigator, other options are available to navigate the sample:

- moving the stage with the keyboard thumbwheels (see chapter 3)
- Moving the stage by clicking on the in-situ optical (CCD) microscope image (see chapter 5.1 on the Optical Image functions)
- Clicking on an imported external image after alignment (see the Point logger chapter 9.2.7)

Save/Go to a position. It is possible to save or call back a position. To save a position, move to the position, then enter a name and click on "snap". To recall a saved position, select the desired position in the list and click on "go to". The stage will automatically move to the position.

5.3.1.4 Navigator file types

The *Navigator* program handles different types of files which contain the information defined by the operator. The hereunder table summarizes the different file types and where they are stored. All the files of the *Navigator* program are stored in the *Navigator* directory. They all are text files, except for the image files which are in a TIFF format.

File type	File extension	Directory
Sample holder	.hld	Navigator
Sample configuration	.spl	Navigator/Config_sample
Preset position	.prs	Navigator/Preset_position
Crater	.crt	Navigator/Crater
Sample type	.ptm	Navigator/Sample_type
Image	.tif	Navigator/Image
Logbook	.log	Navigator/Logbook

Refer to their respective section to see how to configure those files:

- *Sample Holder* files: see section 5.3.2
- *Sample configuration* files: see 5.3.2.4
- *Preset position* files: see section 5.3.2.5
- *Crater* files: contains the information relative to analysis craters sputtered in every sample. For every analysis run on a sample, the *Navigator* program stores the raster size and the position of the crater. There is one crater file per sample defined. See section 5.3.3.3.4
- *Image* files: contains the images of the alignment patterns. There is one file per pattern type.
- *Sample type files:* see 5.3.3.3.3
- *Logbook*: contains the information of the *Analysis* logbook (see chapter 5.3.3.4.1)

5.3.1.5 Sample holder synoptic

The holder synoptic is displayed in the light-yellow graphic in both *Edition* and *Navigation* windows (see Figure 159 above). Inside this graphic area, there is a frame (continuous black line) which defines the sample holder area which can be mechanically explored by the sample stage. Therefore, any window defined for a given holder must be defined within this frame.

Window color code (see Figure 163: color code of sample holder synoptic below):
The area covered by **any window** defined for a given sample holder is drawn in light **grey color.**
When a **sample is loaded** in a window (= **sample_ID edited**) light grey turns into **light blue**.
When a window becomes the **present window** (on the mass spectrometer axis) it turns into **light red**.

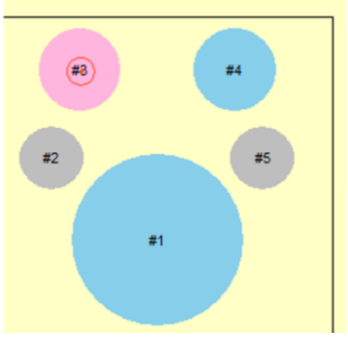


Figure 163: color code of sample holder synoptic

This synoptic can handle rectangular and circular shaped windows. See 5.3.2.

- Zoom Out Zoom In Clicking on one of these 2 buttons allows the operator to change the magnification of the sample holder synoptic.

Options for the information displayed in the synoptic can be selected from the menu: *Tools>Options…*. The corresponding dialog box is shown in Figure 164.

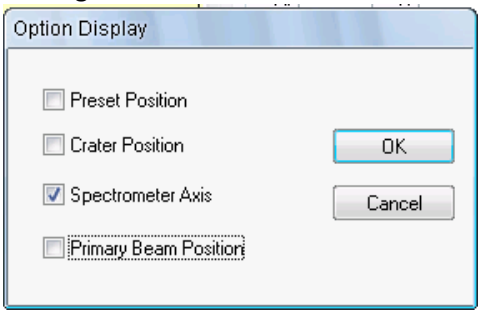


Figure 164: Navigator synoptic display options

- **Preset position**: when checked, the preset positions are represented by green squares on the synoptic
- **Crater position**: when checked, for every analysis performed, there is a brown square marking the analysis position of the corresponding analysis.
- **Spectrometer Axis**: when checked, there is a marker on the synoptic indicating the current position of the mass spectrometer axis (a red circle in SIMS mode, and a green square in CCD). When the sample stage is moved, the marker moves on the synoptic.
- **Primary beam position**: when checked, there is a marker on the synoptic indicating the current position of the primary beam position. When the sample stage is moved, the marker moves on the synoptic.

Note: in most cases, the marker for the spectrometer axis and the marker for primary beam position overlap.

5.3.1.6 Analysis position coordinates – Axis systems

The X,Y position read in the Navigator indicates a position within the sample holder. This internal axis system has been adjusted to match the mass spectrometer axis in SIMS mode: the (0,0) X,Y stage position read in the Navigator is defined by the mass spectrometer axis (= the SIMS position).
This zero position is also adjusted to roughly match the transfer (unload) position.

There are ~ 35 mm between the CCD position and the SIMS position. Hence, in order to switch *accurately* between the CCD and the SIMS positions (keeping the same sample detail at the center of the FOV in both cases), the user must store in the SETUP the *exact* X and Y distances (offsets) between the two positions. (see chapter 9.2.9). Once this is calibrated, **the (X,Y) coordinates of a small detail on the sample read in**

the Navigator appear *THE SAME* in CCD mode and in SIMS mode, despite the fact that in reality the sample holder has been moved by the X-Y offset between the two positions.

The operator can read the **X, Y coordinates of the *analysis* position** in the Navigator window (Figure 165). Figure 166 shows the X-Y axes relative to the stage and sample holder while Figure 167 shows the definition of Z.
Note that opposite to usual conventions X is here the *vertical* axis with positive X coordinates oriented toward the sky and Y is the *horizontal* axis with positive Y coordinates oriented toward the storage vessel chamber. The Z axis is along the ion beam axis, with positive Z coordinates oriented toward the inside of the instrument. The **Z coordinate** read is the external linear Z-movement coordinate (see detail below)

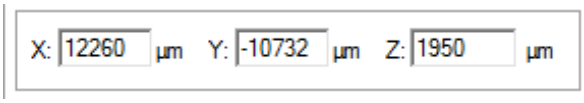


Figure 165: External translation coordinates displayed in the Navigator panel

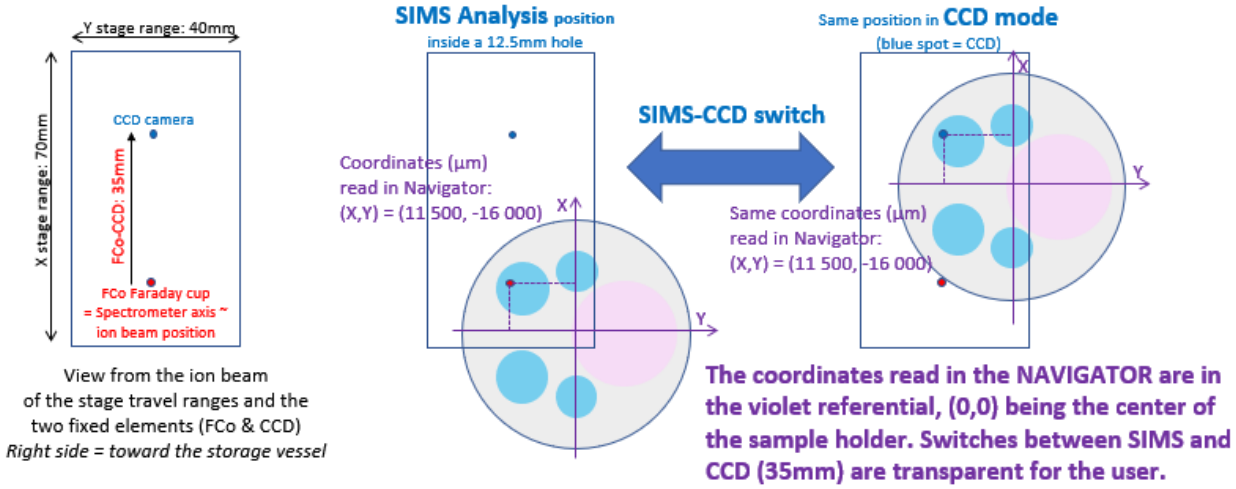


Figure 166: X-Y axes of the positioning system viewed from the ion beam as seen in Navigator

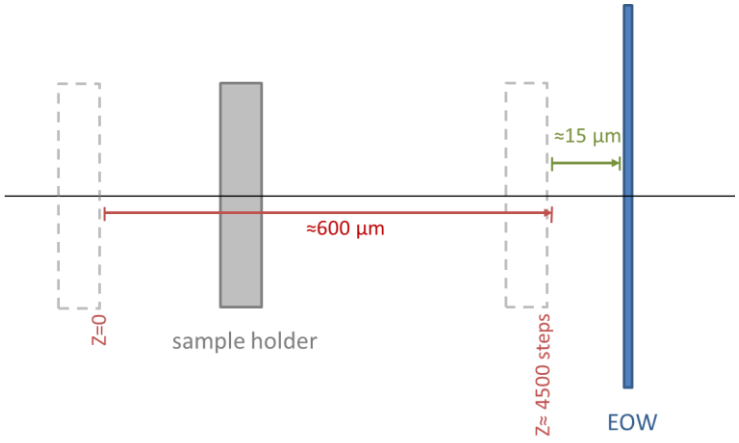


Figure 167: definition of the Z axis.

Note on Z:
All motors are mounted perpendicular to the beam axis. However, the Z movement, adjusting the distance between the sample and the co-axial lens E0W, is in the beam axis. The perpendicular movement (external movement, EM) is then translated inside the analysis chamber into a co-axial movement (internal movement, IM) via a rotor. An external movement of 1 mm translates as an internal movement of 170 µm, which is the actual movement of the stage.

IM = 0.17*EM or EM ~5.9 EI

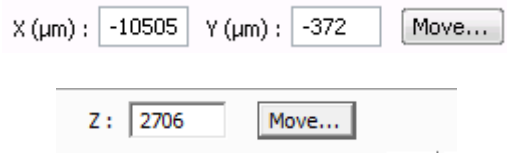
One Z step is equivalent to 1 µm on the external movement, thus a movement of 0.17 µm of the stage.
The exact length of one Z step is given by the slope parameter in the Setup (1 to 1.2 µm/step, see chapter 5.9.2). The total (internal) sample holder Z course is ~ 600 µm corresponding roughly to 4500 motor steps. As shown in Figure 167 , the sample is farther from the E0W lens (600 µm + 10-20 µm) when Z=0, and is the closest (10 to 20 µm) from the E0W lens when Z=4500 (steps). Note that the exact number of steps and the closest distance might vary from one instrument to the other.

The Z-0 (outer) position is defined by an electrical stop (contact). It is adjusted at the **sample transfer position**. The full motor range available is around 4500 steps (corresponding to a travel of 600µm in Z). At the other end, toward the immersion lens, a second (inner) electrical stop limits the travel toward the immersion lens. By precaution, in order to prevent a collision of the sample holder with E0W lens a mechanical stop is adjusted behind the electrical one to always ensure a minimum distance of a couple tens µm between the sample holder and E0W.
The Z course is adjusted by Cameca engineers upon installation.

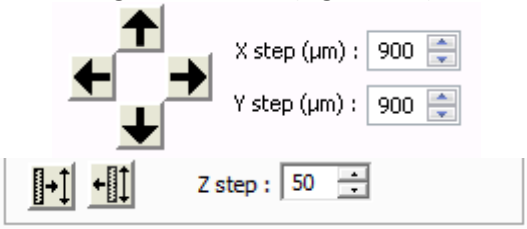
5.3.1.7 Stage motion

From the *Navigator* panel, there are several ways to move the sample stage:

1. Click in the sample holder synoptic on the targeted new analysis point. The stage moves to bring this point of the sample holder onto the mass spectrometer axis.
2. Edit X, Y or Z in the dedicated editing fields and click on *Move…*. The stage moves to this new position.



3. Step by step motion. Enter the step size and click on the arrow. Every click moves the stage along X (up and down), Y (left and right), Z (back and forth).
   Steps of the X-Y stage motions are adjustable in µm.
   Steps of Z are indicated in motor steps (steps of 50 or 100 motor steps are advised). Step length is defined in the SETUP (tab "holder" > Z motor) and should be around 1 µm of the external movement, thus 0.17 µm for the stage movement. (Figure 168).



4. Recall a preset point from the preset point list. (see chapter 5.3.1.10)
5. Click in the CCD image (see chapter 5.1 for details),
6. Click in the Point Logger image (see chapter 6.2 for details)

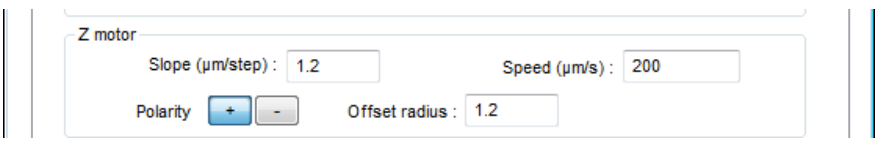


Figure 168: Z motor step slope in SETUP.

There are also “macro” functions to easily move to specific positions or from window to window and within a window:

- 

    - SIMS: default mode for the NanoSIMS. The sample stage is moved to set the analysis area in front of the primary ion beam.
    - CCD: moves the present sample area in SIMS position to the corresponding position in front of the optical microscope (CCD position), ~35 mm above the optical axis. During the moves from/to the CCD position the sample Z is set to the 0 position. The image shown by the CCD camera is displayed in a separate window.
    - FCo: moves the sample holder away (up by ~44 mm from the transfer position) so that the primary ion beam can reach (and refocused into) the fixed Faraday Cup located in the analysis chamber behind the stage, in front of the primary beam and spectrometer axis. The primary ion beam current is displayed in the FC part of the Tuning window.
- 
    - moves the stage from the present window to the next one. (◄: previous, ►: next, ◄◄: first one)
- 
    - moves the stage in order to bring the center of the current window onto the analysis position (i.e the mass spectrometer axis).

Note: in order to avoid any unintentional sputtering of the sample surface, the ion beam is automatically turned off (= blanked into FCp) during stage movements from/to/between SIMS, CCD and FCo positions. It is necessary to re-activate it with Beam ON when necessary.

5.3.1.8 Backlash correction

Function tentatively introduced in earlier version but not available.

5.3.1.9 Motor Initialization and Reset Procedure

The initialization and reset procedure are under the control of the 68030 microprocessors. To initialize or reset the stage motors, click the *Init* or *Reset* icons 

in the *Navigator window.*

The sample stage incorporates on each of its X, Y and Z axes two internal electrical contacts defining ends of travels.

In the Z-axis the rear (or outer) electrical contact is adjusted at the **sample transfer position corresponding to zero Z position**. The full motor range available is around 4500 steps (corresponding to a travel of 600µm in Z). At the other end, toward the immersion lens, in addition to the (inner) electrical stop, a mechanical stop is added behind it by precaution, in order to prevent a collision of the sample holder with E0W lens. This mechanical stop maintains a minimum distance of a couple tens µm between the sample holder and E0W.

For Z axis:
RESET means that the sample stage moves **back to its zero-position** defined by the rear internal electrical stop (rear, exterior, Z=0).

With **INIT** the stage will move up to the (inner) electrical stop closest to the E0W lens (max Z), then back to the electrical stop at the farthest point from the E0W lens (Z=0), while counting the motor steps.

The X axe is the vertical movement of the stage. The full motor range, between the two electrical contacts is of 75000 steps. The Y axe is the horizontal movement of the stage. The full motor range is of 40000 steps.

For **X and Y axes:**
RESET means the motor moves to the internal electrical stop on one side (lowest position for X and left = storage direction for Y), then moves back to a position defined by an hardware offset of X=55000, Y=20000, defined as the X=0, Y=0 coordinates of X,Y axes in the navigator.
With **INIT,** the stage moves to one electrical stop then back to the opposite electrical stop counting the number of motor steps between the two, and finally moves to a position defined by an hardware offset of X=55000, Y=20000, defined as the X=0, Y=0 coordinates of X,Y axes in the navigator.
When resetting or initializing X and Y, both motors move together.

This offset position corresponds roughly to the *unload* position. A software offset is added by Cameca engineers to define the exact *unload* position. This additional offset is visible in the Setup (Setup > Holder > Analysis Sample).

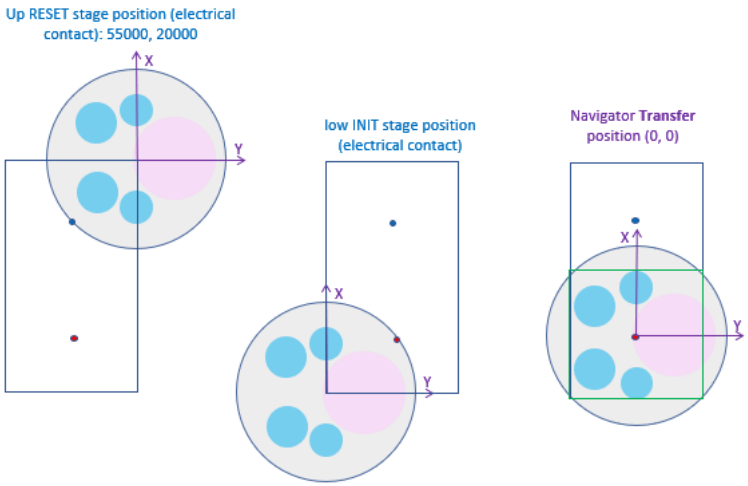


Figure 169: X-Y reset/Init and transfer (=load/unload) positions

5.3.1.10 Preset positions

In the *Navigator* program, the operator can memorize positions of interest, called **Preset points**, in Preset position files that can be saved and recalled later on allowing to revisit the preset points.
A preset point is defined by the 3 coordinates X, Y and Z and a name which is used for its identification.
Figure 170 shows the section of the *Navigator* window dedicated to handling the preset positions.

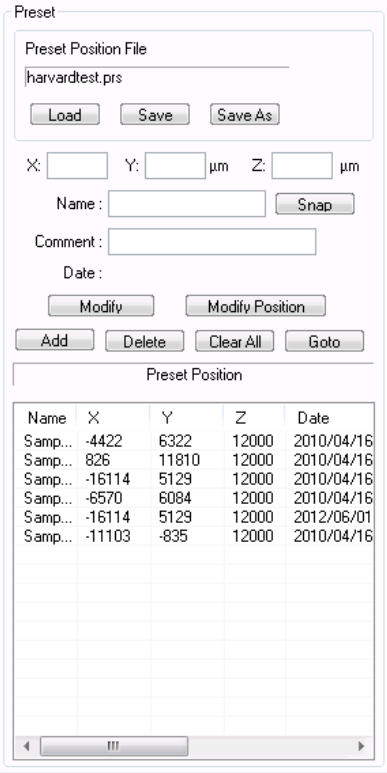


Figure 170: Preset position handling in the Navigator

- *Preset position file* combo box: the first box is used to select the file name of the preset position series to be loaded. The current file name is shown at the top.
- *Load*: click on this button to load the preset file displayed in the combo box
- *Save*: click on this button to save the modifications of the current preset position series in the last loaded file. The previous file will be overwritten *with no confirmation prompt*.
- *Save as*: click on this button to save the current preset position list in a new file. There is a prompt to enter a new file name.
- *X, Y, Z*: editing fields to define coordinates a new preset point (e.g. keeping same name but updating the coordinates).
- *Name*: editing field to enter the ID of the new preset point. Two preset points cannot have the same ID in a given series.
- *Comment*: editing field to enter a comment about the new preset point.
- *Date*: static field concerning the saving date of the preset point.
- *Snap:* click on this button to add a new preset point with the current position X, Y and Z of the sample stage and the *Name* and *Comment* editing fields value.
- *Modify:* click on this button to update the preset information selected in the list (blue line) with the *X*, *Y*, *Z*, *Name* and *Comment* editing fields value.
- *Modify Position:* click on this button to update the preset information selected in the list (blue line) with the current position X, Y and Z of the sample stage (name and comment remain the same).
- *Add*: click on this button to add a preset point with the *X*, *Y*, *Z*, *Name* and *Comment* editing fields value. Note: if there is no value for *X*, *Y*, or *Z*, zero is saved by default. If there is no value for *Name*, "Point" is saved by default.
- *Delete*: click on this button in order to delete the preset position selected in the list (blue line).
- *Clear all*: click on this button in order to delete all the preset positions present in the list.
- *Preset position list*: table displaying the current preset position series.
- *Goto*: click on this button to move the stage in order to bring the selected preset point (blue line) onto the mass spectrometer axis.

❖ Procedure to edit a new preset point
- Edit the *X*, *Y*, *Z*, coordinates fields.

- Edit the *Name* and *Comment* fields.
- click *Add* to add the point to the preset list. Alternatively, click *Snap* to add a new point with the current sample position.

❖ Procedure to Modify a preset point
- Select in the list the preset point to be modified by clicking on the corresponding line
- Modify the *X*, *Y*, *Z*, coordinates fields.
- Modify the *Name* and *Comment* fields.
- Click *Modify* to save all the edited information or *Modify Position* to save only the sample coordinates.

❖ Procedure to delete a preset point
- Select in the list the preset point to be deleted by clicking on the corresponding line and click on *Delete*.

❖ Procedure to move to a preset point
- Select in the list the targeted preset point by clicking on the corresponding line and click on *Goto*

5.3.1.11 Sample configuration

Every sample holder can be associated to a *Sample configuration* file containing the description of all the samples loaded in the *Holder*. This file contains information on: sample name, matrix name, etc…
Figure 171 shows the section of the *Navigator* window dedicated to the handling of the Sample configuration

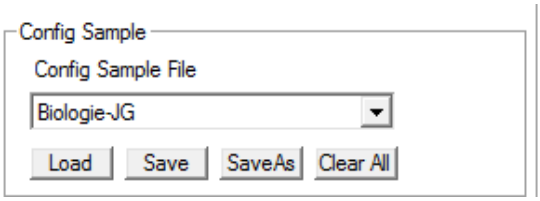


Figure 171: Interface for the Sample configuration handling in the Navigator window

- *Load*: click on this button to load the sample configuration list displayed in the combo box
- *Save*: click on this button to save the modifications of the current sample configuration in the last loaded file. The previous file will be overwritten *with no confirmation prompt.*
- *Save as*: click on this button to save the current sample configuration in a new file. There is a prompt to enter a new file name.
- *Clear all*: click on this button in order to delete all the samples defined in the current sample configuration.

When loading a new holder, the last saved *Sample configuration* matching the Holder configuration will be loaded.

❖ Procedure to Save a new sample configuration file
- Edit the samples loaded in the current Holder.
- Click Save as. A dialog box opens
- Edit the new file name and click OK.
- The new file name is displayed in the combo box

❖ Procedure to Load a sample configuration file
- Select in the combo box the file to be loaded and click on Load

5.3.2 Edit holder window

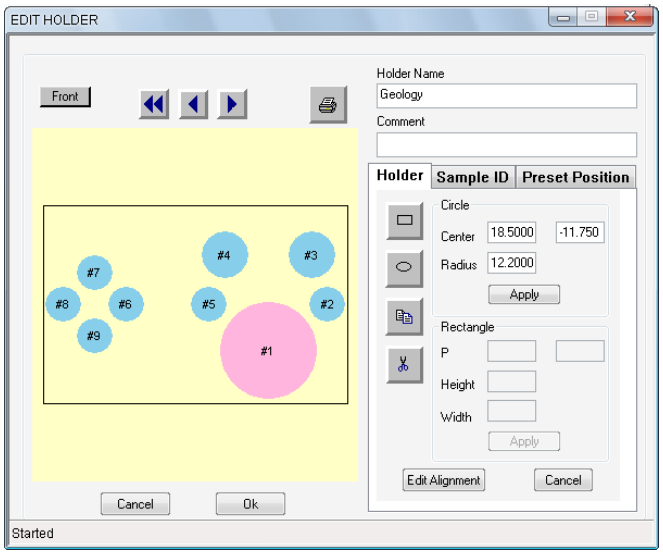


Figure 172: Holder edition window

This mode is used by the operator to define the *Holder* to be used on the instrument (Figure 172). The operator can:

- Edit a new *Holder* configuration, i.e. definition of the position and the size of the windows for the holder.
- Edit the *Sample ID* (definition of the sample ID for every window).
- Edit *Preset positions* (preset positions are stage positions that the operator wants to memorize to be able to come back on it by a single command).
- Save a new *Holder* configuration.
- Duplicate a holder definition.
- Modify an already existing Holder configuration.
- Delete a Holder definition.
- Restore the last deleted Holder configuration.

The holder editor is accessible via the Holder menu.

5.3.2.1 Main commands

The operator can handle the *Holders* by selecting the following commands in the *Holder* menu:

- *New*: to create a new Holder. The *Edit Holder* dialog window automatically opens (see paragraph 5.3.2.2 below).
- *Modify*: to modify an already defined *Holder* configuration (cannot be used for the current holder).
- *Save as*: to save the present Holder with a new name.
- *Duplicate*: to copy a Holder configuration with a new name (cannot be used for the current holder).
- *Delete*: to delete the *Holder* selected in the list (cannot be used for the current holder).
- *Restore*: to restore the last deleted Holder configuration.
- *Sort*: to modify the list order of the *Holder* names.

5.3.2.2 Holder configuration edition

This edition is divided in 3 different steps (Figure 173):

- Holder configuration (window layout in the holder)

- Sample configuration (which sample in which window)
- Preset positions (memorized positions over the useful area of the sample holder)

The operator activates one of these 3 edition modes by clicking on one of the 3 tabs (Holder, Sample ID, Preset position). During the edition, there is a synoptic of the *Holder* presently edited which is updated according to the configuration under definition.
The available commands are:

- *Front / Back*: the label of this button indicates the representation mode of the holder in the synoptic. Switch between *Front* and *Back* representations is made by clicking on this button. The representation change is made by means of a symmetry relative to the horizontal axis of the figure.
-  Click on these buttons to change the present window selection. (◄: previous, ►: next, ◄◄: first one)
- *OK*: close the *Edit Holder* window. All the modifications edited in the different menus are automatically saved in their respective files (*Holder*, *Sample configuration*, *Preset positions*). If new file name(s) has(ve) been edited in the editing fields, OK command is equivalent to the *Save as* command.
- *Cancel*: click *Cancel* to close the Edit Holder mode. None of the modifications edited in the different menus are saved.

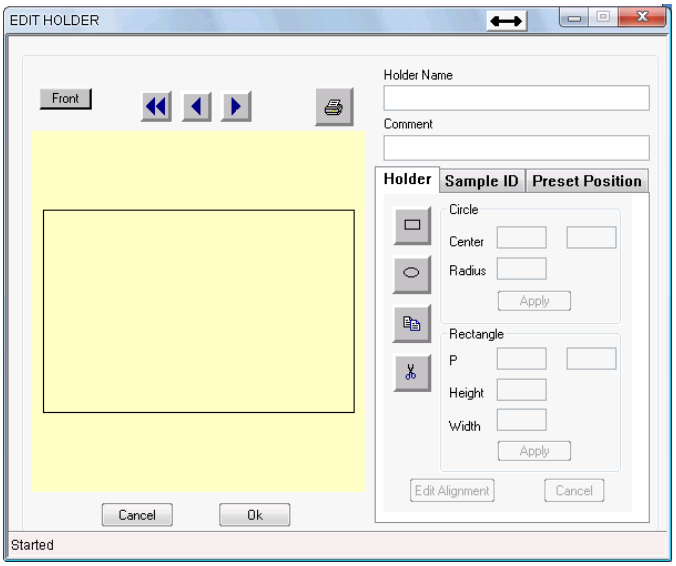


Figure 173: Holder edition, window configuration

5.3.2.3 Holder edition (window configuration)

The *Holder* editor allows the operator to define the window configuration of a given holder. This window configuration is graphically represented in the synoptic during the edition procedure.
The available commands are:

-  : to toggle in the edition mode of rectangular (squared) window.
-  : to toggle in the edition mode of circular window.
-  : to paste a window.
-  : to cut a window.
- *Copy*: to copy a window.
- *Delete*: to delete a window.
- *Center:* editing fields for defining the X and Y coordinates of the center of a circular window. The zero of the XY coordinate system is the center of the holder.
- *Radius:* editing field to define the radius of a circular window.

- *P:* editing fields to define the X and Y coordinates of the lower left corner of a rectangular window. The zero of the XY coordinate system is the center of the holder.
- *Height:* editing field for defining the height of a rectangular window
- *Width*: editing field for defining the height of a rectangular window
- *Edit alignment:* command to open the dialog box used to define the alignment point of the Holder under edition.

❖ Procedure to edit a window
- Select either *Holder > New* (for a new holder definition) or *Holder > modify* (for modifying an already existing holder). In the former case, select in the list the holder to be modified.
- Select the *Holder* tab. Select the type of window to be added (rectangular or circular)
- Edit a *Holder name* to save all the information
- Edit the position and the size of the new window
- Click *Apply*. The new window is displayed in the synoptic. Note that the software does not allow the overlay of two windows. If it happens there is a prompt asking the operator to modify the *Centre* or *P* values.

Note: the windows are numbered according to their edition order.

❖ Procedure to copy a window:
- Select a window type (rectangular or circular) identical to the window to be copied. Editing fields for the window definition are empty.
- Click right on the window to be copied and select Copy
- Click on the Paste icon. The parameters of the copied window are displayed in the parameters of the new window
- Modify P (or Center) parameters in order to avoid window overlay.
- Click Apply. The new window is displayed in the synoptic.

❖ Procedure to delete a window
- Click right on the window to be removed.
- Select Delete. The window is erased in the synoptic.

5.3.2.4 Sample ID edition

The sample edition in the *Edit Holder mode* is the same as in the *Navigator* window.
The editions performed in this menu are saved in a file with the name displayed in the combo *Configuration Sample File*. For a new holder, the default file name is the holder name (Figure 174).

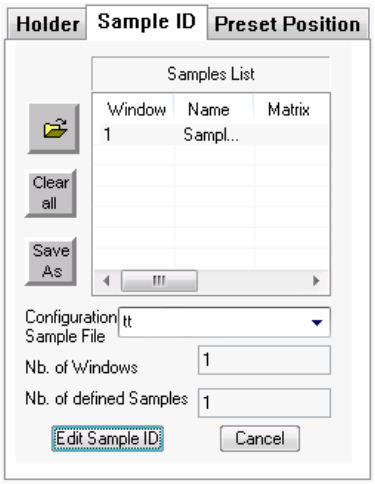


Figure 174: Holder edition, Sample ID

5.3.2.5 Preset Position edition

The sample edition in the *Edit Holder mode* is the same as in the *Navigator* window.
The editions performed in this menu are saved in the preset position file with the name displayed in the combo *Preset position*. For a new holder, the default file name is the holder name (Figure 175).

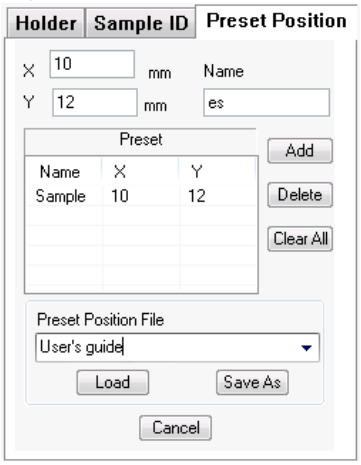


Figure 175: Holder edition, Preset position

5.3.3 Menus

The main bar contains 4 menus giving access to the functions listed hereunder:

- **File**: Print, Remove, Exit
- **Holder**: New, Save as, Duplicate, Delete, Restore, Modify, Sort, Load, Unload
- **Sample**: Edit sample ID, Copy sample, Paste sample, Delete sample, Undelete sample, Edit pattern mapping, Crater.
- **Tools**: Logbook, Options, Holder offset.

5.3.3.1 File menu

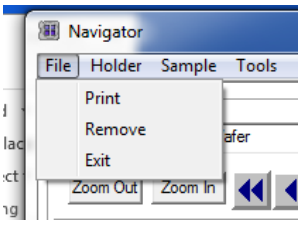


Figure 176: File menu in the main bar of the Navigator panel

- ***Print:*** A dialog box opens for the print option selection. The operator can select to print: the holder synoptic, the sample list, the preset position list.
- ***Remove****:* allows to manage the *Navigator* files (holder, preset positions, etc… see section 5.3.1.4 for details). Files can be deleted, copied, moved. Figure 177 shows the dialog box.
- ***Exit:*** quit the Navigator program. Note that all information not saved will be lost!

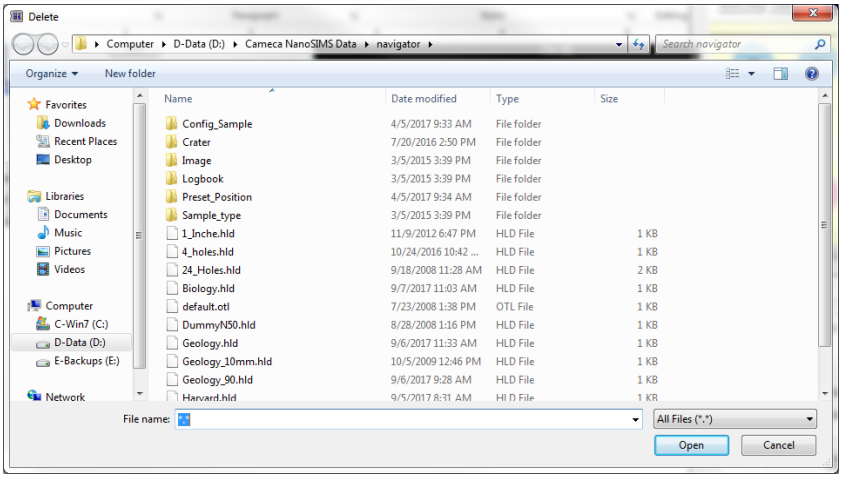


Figure 177: Navigator file manager

5.3.3.2 Holder menu

This menu regroups two main functions:

- The first few commands (from “New” to “Sort” are for *Holder* definition (see chapter 5.3.2 above).
- “Load” allows to load an existing holder configuration, which must be done every time a new sample holder into the analysis chamber. See below for details.
- “Unload” allows to move the sample holder back to the “unload” position so that the user can take it out of the analysis chamber (see below).

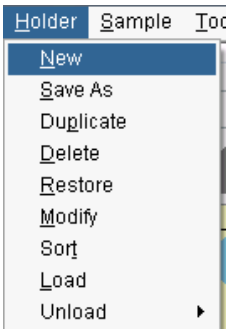


Figure 178: Holder menu from the the main bar of the Navigator panel

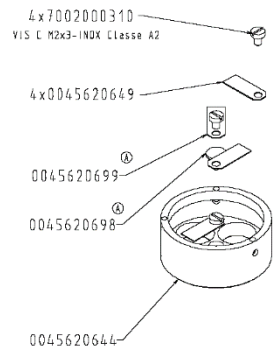
5.3.3.2.1 Load

This function is used to define the current sample holder which is the holder loaded in the analysis chamber. This is a software definition only and the holder will have to be physically loaded by the operator.
Procedure to load a Holder:

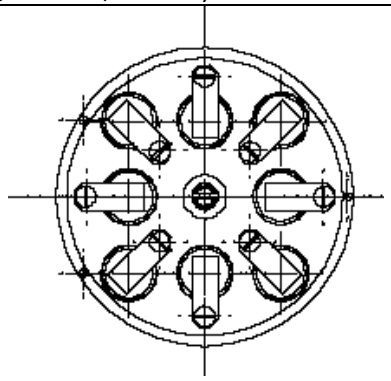
- Select Holder > Unload. The stage moves to the Load/Unload position
- Check with the optical microscope that the stage is well set in the Load/Unload position.
- Physically (unload the holder from the stage and) load the new holder from the storage chamber into the analysis chamber.
- Select Holder>Load. The Holder list dialog opens. Select in the list the holder file name corresponding to the holder loaded in the analysis chamber.
- Click OK, the schematics of the holder now appears in the synoptic window.

This Holder file is loaded in the Navigator software with its last associated Sample configuration and Preset position files. Once loaded, using the Load function for Sample configuration and Preset positions files, a given holder can be associated with different samples and preset position configurations. It is thus practical to save these files to reload them (e.g. standards are often always mounted in same windows).

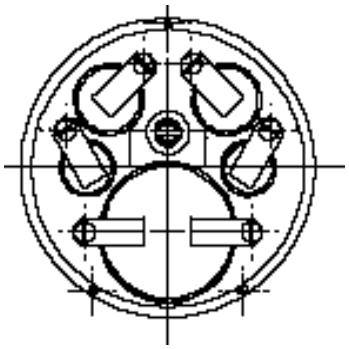
The available holders are shown in the Figure 179: Various sample holders below, with various number of holes and diameters (25.8mm, 10.4mm, 7.2 mm, 5.4mm):



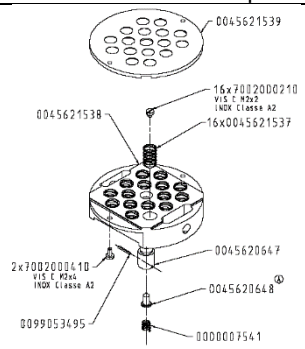
45620641
"1-INCH STANDARD" sample holder



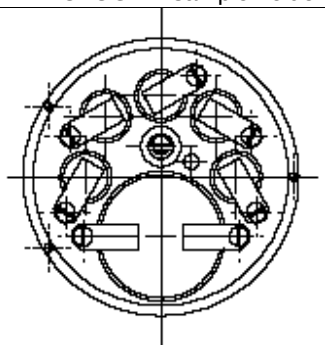
45620642
« BIOLOGY » sample holder



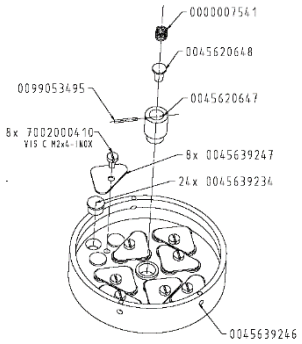
45620643
" WU-MPI » sample holder



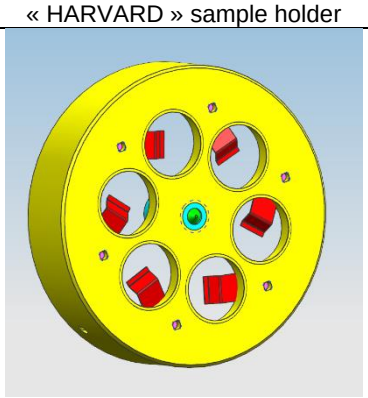
45621567
« HARVARD » sample holder



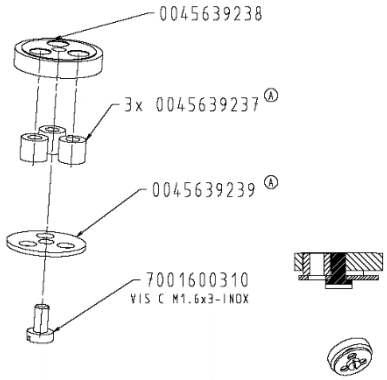
45639165
« GEOLOGY 10mm » sample holder



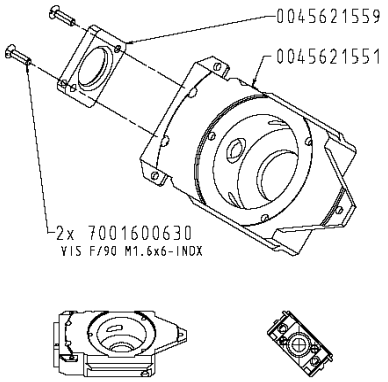
45639248
« 24 holes » sample holder



45641320
"JAMSTEC" sample holder



45639345
« 3 TEM grids » sub sample holder



45631183
« NS50 SHUTTLE »

Figure 179: Various sample holders

5.3.3.2.2 Unload

This function is used to unload the Holder from the stage. The Unload command moves the stage to the Load/Unload position but the holder will have to be physically unloaded by the operator with the transfer road, back to the Storage vessel.

❖ Procedure to unload a Holder

- Select Holder > Unload. The stage moves to the Load/Unload position. The synoptic shows an empty stage.
- Check that the stage is well set in the Load/Unload position.
- Perform the holder load/unload operation using the transfer rod between the analysis chamber and the vessel chamber (see chapter 9.1.1 for details on the procedure).

When the Unload command is applied all the modifications made for the Sample configuration and Preset Positions are saved in their respective current file name.

5.3.3.3 Sample menu

This menu (Figure 180). gives access to the commands used to perform

- *Sample configuration* definition (Edit Sample ID, Copy Sample, Paste Sample, Delete Sample, Undelete Sample)
- Alignment procedures (Edit Align point, Edit Pattern mapping)
- List coordinates for all analyses done on the samples of the loaded holder (crater).

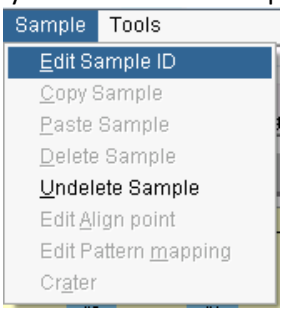


Figure 180: Sample menu from the main bar of the Navigator panel

5.3.3.3.1 Edit Sample ID

For every window of a given holder, the sample ID can be defined from the Navigator window or from the Edit Holder mode. Do not forget to systematically fill at minimum *sample ID* and *Comment* fields that will be saved in the acquisition conditions, to keep track of data in the future (Data Explorer CAMECA search engine software uses this information). For a given holder, the sample ID must be different for every window. For any window the sample ID can be displayed by a right click in the corresponding window (Figure 181Figure 181).

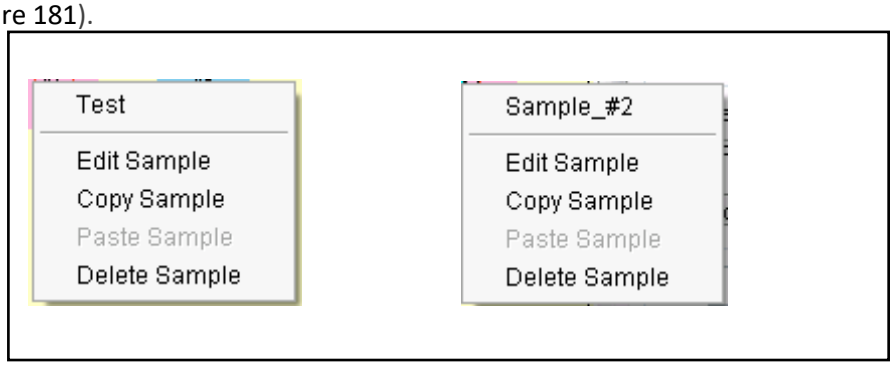


Figure 181: Menu for Sample ID edition

- ***Edit sample*:** This command opens the dialog box used to edit the Sample ID and associated parameters. (Figure 182)
- ***Copy sample*:** This command is used to copy the sample description of the current window in the clipboard.
- ***Paste sample:*** This command is used to paste the sample description from the clipboard to the current window. Note that as two samples cannot have the same sample name in a given holder, the Paste command will update the sample name from *sample_name* to *sample_name_copy.*
- ***Delete sample*:** this command erases the current sample ID (and associated parameters) of the window.
- The ***Undelete sample*** command available in the *Sample* menu allows the operator to restore the parameters of the last deleted sample.

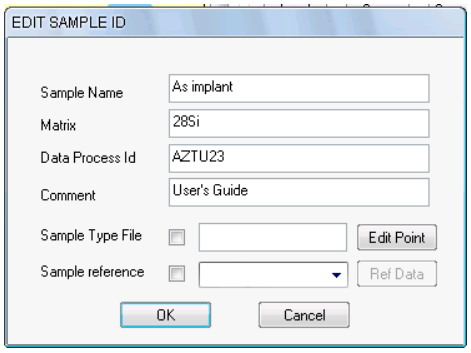


Figure 182: The Edit Sample ID dialog box

- *Sample name:* Editing field for the Sample ID
- *Matrix:* Editing field for the Matrix name. This information will be used in the Curve processing for the RSF data base.
- *Data process ID:* Editing field to enter a label defining a data processing recipe. This label is saved in the Raw data file.
- *Comment:* Editing field to enter a comment. Only the Navigator program can display this comment.
- *Sample type File*: check this editing field and enter a *Sample type* name. The parameters defining the *Sample type* will be saved in a *Sample type* file (the file name will be the *Sample type* name). These parameters are edited in the *Edit Sample Type* dialog box (see 5.3.3.3.3)
- *Edit point:* Click on this button to open the *Edit Sample Type* dialog box.
- *Sample reference:* Check this editing field in order to declare the sample being edited as a *Sample reference*. If the sample being edited is not a reference sample, do not check this editing field and select a *Reference sample* in the list to be associated to the sample being edited. Note that in the first *Navigator* program release, this *Sample reference* option is just used as a comment by the software.

❖ Procedure to edit a Sample ID:
- Right-click on the target window in the synoptic,
- Select Sample ID in the menu displayed,
- Fill the different editing fields (all are optional but *Sample name*)
- Click *OK* to validate the edition (or *Cancel* to abort the edition)

5.3.3.3.2 Edit Align point

This function is meant to help navigation on the sample in other CAMECA SIMS. However it is not suited to NanoSIMS and we recommend the use of Point Logger instead (see chapter 6.2).

5.3.3.3.3 Edit Pattern mapping

This function is generally used in nanotechnology on other SIMS tools, for samples containing repetitive structures with lines and rows. This function permits to use a referential internal to the sample itself, with pattern repetition distances helping for navigation.
These files are used to save the information relative to a type of samples. A type of samples is defined by:
- Alignments points
- The coordinates in the window axis system of the origin of the Sample axis system
- A series of analysis points defined with their coordinates in the Sample axis system.

Therefore, a *Sample Type file* is mainly oriented to the definition of a sample with patterns.
In order to edit these parameters defining the sample type, the operator must open the *Edit Sample Type* dialog box by clicking on *Edit point*. This dialog box is shown in Figure 183.

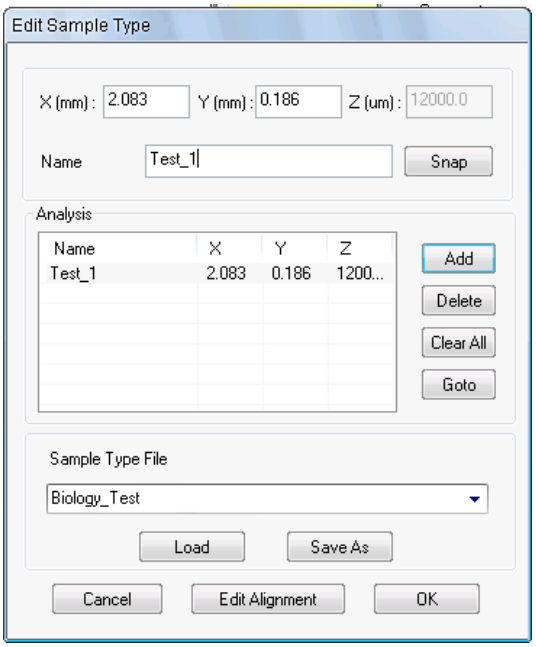


Figure 183: Edit Sample Type dialog box

- *X, Y:* editing field to enter the X&Y coordinate of a new analysis position. These coordinates are given in the sample axis system. The current sample position can be loaded in the editing field with the *Snap* button.
- *Name:* editing field to define the name of the new position analysis position.
- *Add:* add to the list the analysis position being edited. Two analysis positions cannot have the same name. There is a prompt before overwriting a former point with the same name. Note that this function adds the analysis point in the current list but not in the corresponding *Sample Type File*. A Save action is required to update the file.
- *Delete:* Delete the position selected in the list (the blue line).
- *Clear all:* Clear the analysis position list
- *Goto:* Click on this button to move the stage and bring the selected analysis position (blue line) onto the mass spectrometer axis.
- *Sample type file* list: Display the name of the current Sample Type File on the top. Allow to select a new *Sample Type File*.
- *Load:* Click on this button to load the Sample Type File display selected in the list.
- *Save*: click on this button to save the modifications of the current analysis positions series in the last loaded file. The previous file will be overwritten with no confirmation prompt.
- *Save as*: click on this button to save the current analysis positions series in a new file. There is a prompt to enter a new file name.

5.3.3.3.4 Crater

For every analysis launched from the *Analysis* program, the Navigator program stores information in a file which can allow the operator to control the relative crater position for different analyses on a given sample. Note that all craters sputtered outside of the *Analysis* program (e.g. during a tuning) are not considered by this function.

The command *Sample > crater* in the *Sample* menu opens a dialog box which lists this information. This dialog box is shown in Figure 184.

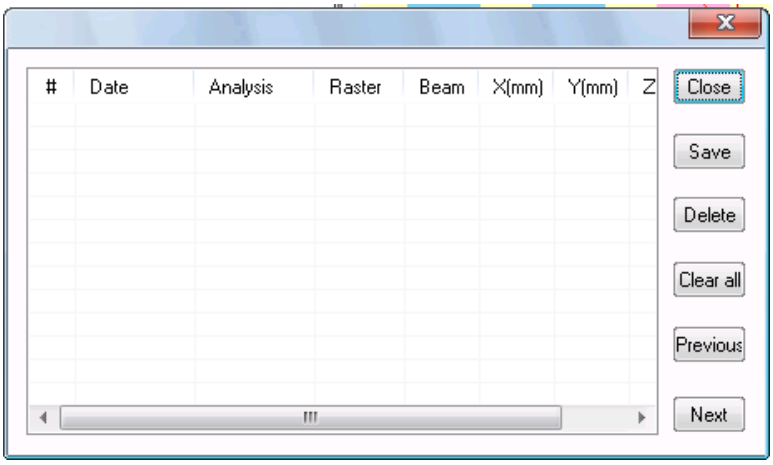


Figure 184: Crater position dialog box

- *#*: Analysis rank. Note that the maximum number of analysis per sample is 50.
- *Date:* date of the analysis.
- *Analysis*: Analysis type (Depth profile, mass spectrum….).
- *Raster:* raster size used for the analysis.
- *Beam:* spot size in the case of the use of a static primary beam (raster = 0µm).
- *X(mm) and Y(mm):* X and Y coordinates of the analysis position in the stage axis system.
- *Close:* to close the dialog box.
- *Save*: to save the crater position list.
- *Delete:* to delete a line in the list.
- *Clear All*: to get an empty crater position list.
- *Next/Previous:* To move back and forth in the list. When a crater (i.e. a line in the list) is selected, the corresponding symbol representing the crater in the synoptic (see 5.3.1.5) turns to a different colour in order to identify it.

5.3.3.4 Tools menu

This menu (5.3.3.4) shows a few more options regarding the Navigator. However, as the Navigator program is common to most CAMECA ion probe products, certain options are not available to the NanoSIMS (e.g. sample rotation). Those options appeared greyed in the menu and cannot be accessed. The other options are described below.

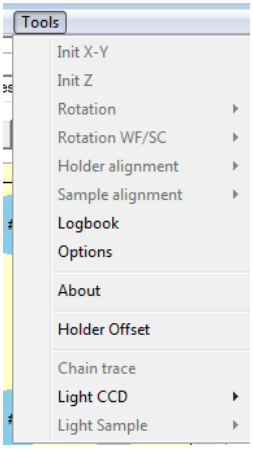


Figure 185: Tools menu from the main bar of the Navigator panel

5.3.3.4.1 Logbook

The *Navigator* program updates the content of a logbook at the end of an analysis. The *Logbook* dialog box (Figure 186) opens with the command Tools > Logbook. In this logbook, the operator can read the following parameters:

- *Date*: completion date of the analysis.
- *Window:* window number where the sample is loaded in the holder.
- *Point*: analysis point number (see 5.3.3.3.4).
- *X(mm), Y(mm), Z(µm)*: analysis position coordinates.
- *Sample:* sample name.
- *Data file:* name of the raw data file saved at the end of the analysis.
- *Holder*: holder name.
- *Preset file*: preset position file name associated to the holder.
- *Pattern file*: Pattern file name associated to the sample.

There are commands to handle the content of the logbook:

- *Sort criteria*: this menu offers the choice between criteria to sort the information listed in the logbook (Date, holder name, Analysis type, Sample name, Data file name).
- *Save:* to save the content of the logbook in a dedicated file.
- *Print:* to print the content of the logbook.
- *Previous/next*: Allows to move back and forth in the logbook list. When an analysis (i.e. a line in the list) is selected, the corresponding symbol representing the crater in the synoptic (see 5.3.1.5) turns to a different color in order to identify it.
- *Close:* to close the *Logbook* dialog box.

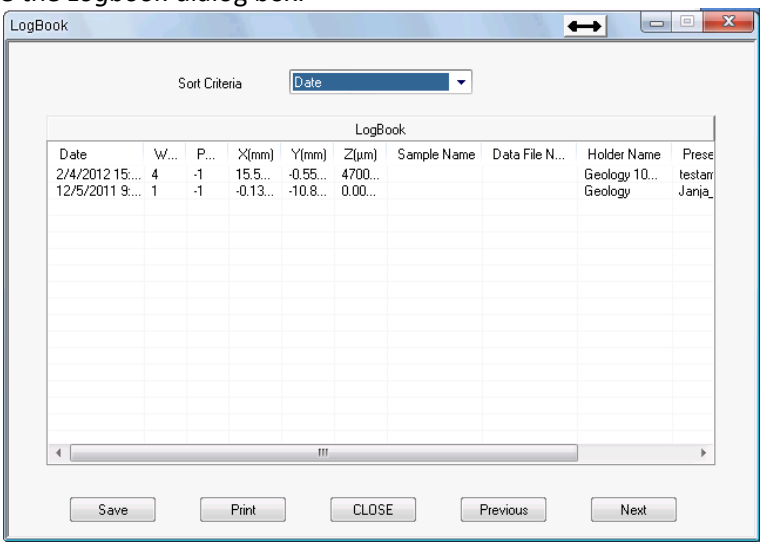


Figure 186: Logbook dialog box

5.3.3.4.2 Options

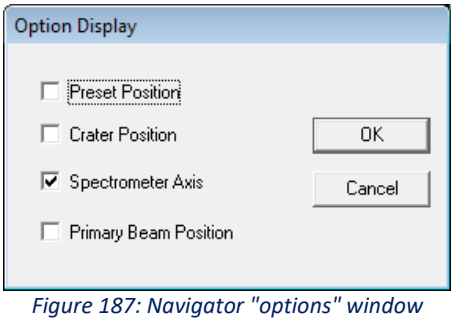


Figure 187: Navigator "options" window

- Preset position : when checked, the preset positions are represented by green squares on the synoptic
- Crater position : when checked, for every analysis performed, there is a brown square marking the analysis position of the corresponding analysis.
- Spectrometer Axis : when checked, there is a marker on the synoptic indicating the current position of the mass spectrometer axis. When the sample stage is moved, the marker moves on the synoptic.
- Primary beam position : when checked, there is a marker on the synoptic indicating the current position of the primary beam position. When the sample stage is moved, the marker moves on the synoptic.

Note : in most of the cases the marker for the spectrometer axis and the marker for primary beam position are overlaid.

5.3.3.4.3 About

"About" indicates the version of the Navigator program

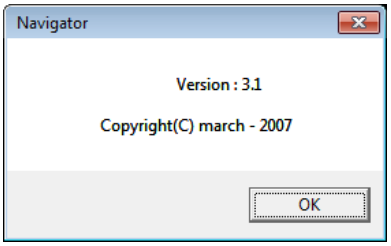


Figure 188: Navigator "about" window

5.3.3.4.4 Holder Offset

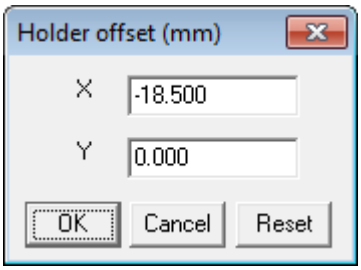


Figure 189: Holder offset window from the Navigator

An X and Y holder offset can be applied to adjust the CCD and SIMS. The reset button set the X and Y offset to 0.

For historical reason this window is now redundant with the CCD/SIMS offset and thus we recommend not to change this Holder Offset here.

5.3.3.4.5 Light CCD

This function allows the control of the light intensity in the analysis chamber. It can be turned off (OFF) and set at a given percentage of the max intensity (25%, 50%, 75%, 100 %). Note that the light is turned on only in CCD mode and turns off when the user switches back to SIMS.

5.3.3.4.1 Light sample

This function allows the control of the light in the **airlock**. It can be turned off (OFF) and set at a given percentage of the max intensity (25%, 50%, 75%, 100 %). It is mainly used to heat the airlock to help degas a new introduced sample. Use it with caution, many samples do not react well to the heat.

5.4 (ion and electron) Sources

5.4.1 The main window

The source window (Figure 190) is used to control the two ion sources and the electron-gun. The Cesium ion source is used in positive polarity, while the Oxygen RF-Plasma source can be used in both negative and positive polarity.
Using positive primary ions (Cs^+, O_2^+) with the sample negatively biased, only negative secondary ions and electrons will leave the surface. Hence the surface of an insulating sample will charge positively. We then can use the normal incidence electron flood gun (NEG) to compensate the charges. Using O-, O_2^- or O_3^- and extracting positive secondary ions will charge the sample negatively and the e-gun is not used in the impact energy range generally used on the NS50L.

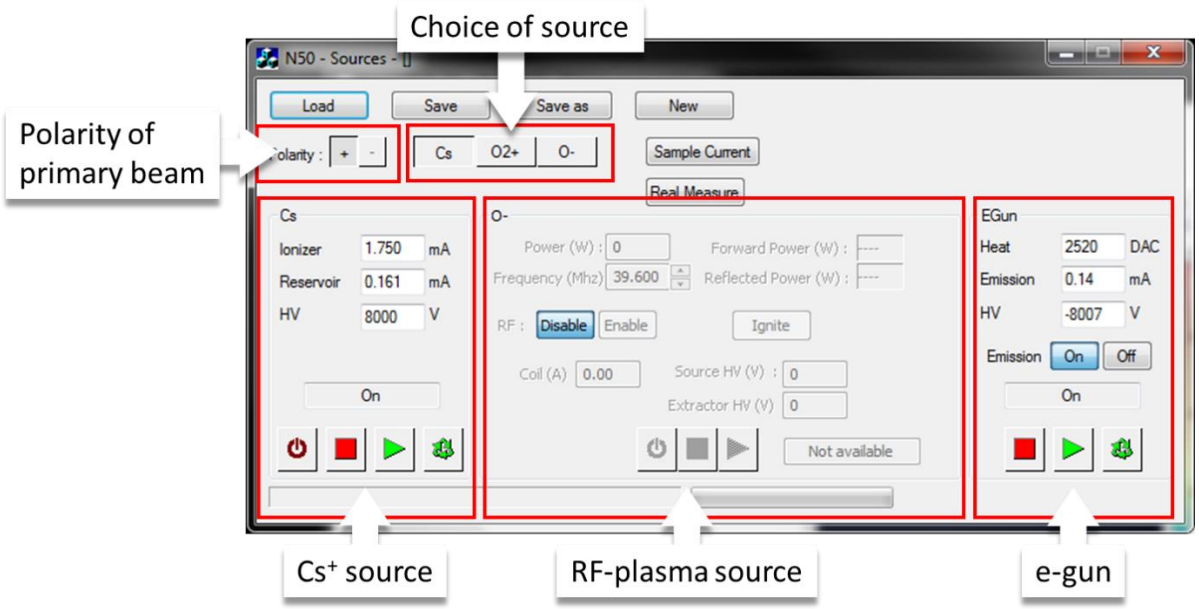


Figure 190: Sources window – Main parts

In this window, only one source can be used at a time: Cs^+ or $O^{-/+}$ (RF-Plasma). The source is linked to the polarity, so when the operator selects the ion source (Cs, O_2^+ or O^-, Figure 190), the polarity is switched automatically. The operator can load, save or create files containing sources parameters information, allowing to apply saved values. By default, the previous session's parameters will be applied. The *Sources* window is separated in three main parts for the Cs^+ source, the RF-plasma source, and the e-gun. The source currently used **is not** darkened.

5.4.2 The Cs^+ control

The Cs^+ source controls (Figure 191) allow to set the source parameters, to stop or start the source and eventually add delays to the start and stop procedures.

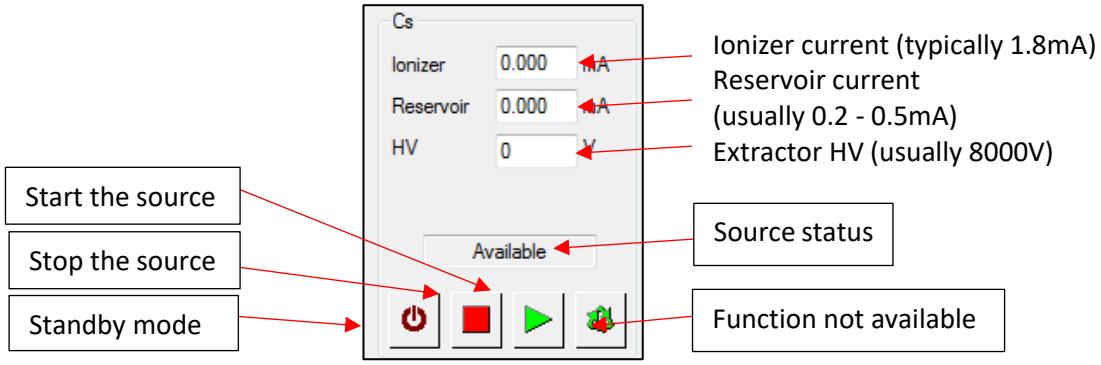


Figure 191: Cs+ controls

The "Start the source" button opens a dialog box (Figure 192) where the operator can enter/modify the source parameters. If a source file has been loaded, then, the values are already filled. After having filled the values, the operator can click on OK to continue or click on Cancel to abort the process.

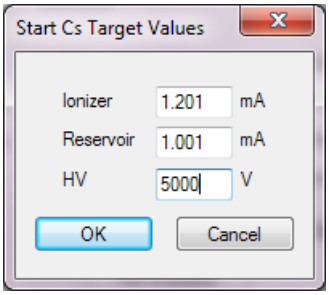


Figure 192: Start Cs+ target values

If the operator chooses OK, a small window opens (Figure 193) where one can add a delay to start the source. The delay unit is hours and minutes: XX:XX (for example, if the operator wants to start the source in 1 hour and 12 min, he has to fill : "01:12"). If no delay is necessary, fill 00:00 and click on OK.

It is also possible to turn off all HVs of the instruments (lenses, detectors…,) except the source HV which is independent) after the source start. by clicking on "stop HV after". So the source is kept stabilized while the rest of the instrument is at rest.

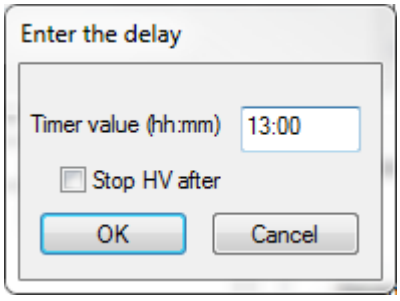


Figure 193: Cs+ start delay

Click on OK. The starting process will automatically start, progressively raising current and voltage until the target current and voltage values are reached (as defined in the Setup, see 5.9.12). Wait for the end of the process. Note that once the starting process is over, it still takes about thirty minutes for the source temperature to stabilize and thus for the primary beam intensity to stabilize.
Note that the Start process cannot be interrupted once launched.

To STOP the Cs+ source, click on the "Stop" button  and wait for the process (as defined in the Setup in 5.9.12) . A new window opens (Figure 194). You can decide to totally stop (all parameters are set to zero)

or put the source in standby. Like for the start, it is possible to add a delay to the stop procedure. This is particularly useful when one wants to program the stop of the source at the end of a long analysis.
Note that the Stop process cannot be interrupted once launched.

If the user wishes to program a restart of the source (for the next morning or next Monday), it is also possible. Check the "restart after cooling" option. New parameters appear. Enter the Ionizer, Reservoir and HV values, and the time delay. Note that the countdown for the restart only starts **after** the stop procedure is complete.

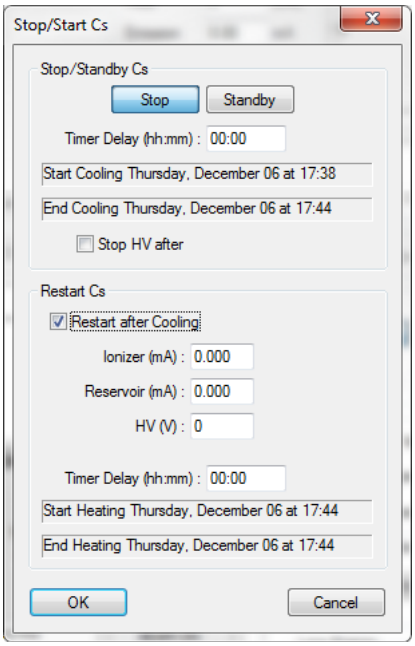


Figure 194: Stop and auto-start window for the Cs source

5.4.2.1 Cs^+ use clock

When hovering the cursor over the source status (Figure 195), the number of hours of use of the Cs^+ source is showed. This clock can be reset by clicking on the reset button.  We recommend resetting the clock every time a new source is installed.

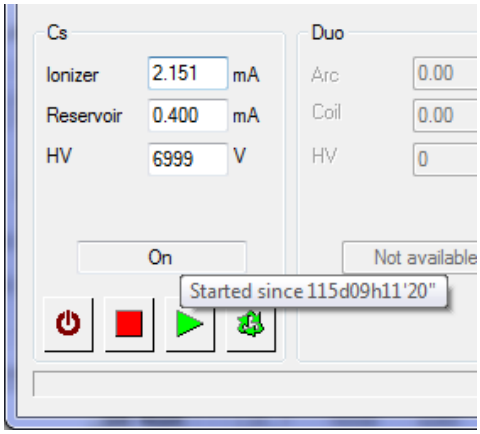


Figure 195: Cs^+ source clock

5.4.3 The RF-Plasma control

The O source control window (Figure 196) allows to start automatically the RF-plasma source. Procedure is the same in both negative and positive mode.

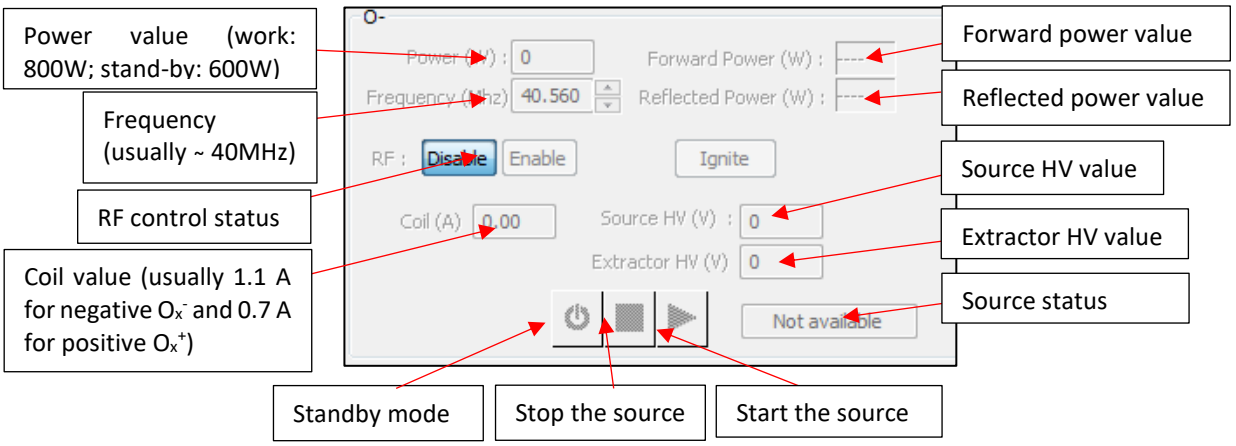


Figure 196: O- source control

All the source values necessary to start the source, such as Power value, Frequency, Coil value, Source and Extractor HVs, are saved in the Setup file and does not need to be changed by the operator (see 5.9). Note that for O_2^+ mode, because the polarity is set as positive, the *slit, diaphragm* and *hexapole* values, which are applied from the *Setup* will be the ones from the Cs mode.

To start the source automatically, select overall the “Enable” button of the RF control status 

Then click on the “Start” button  and follow the steps displayed on the following pop-up window (Figure 197).

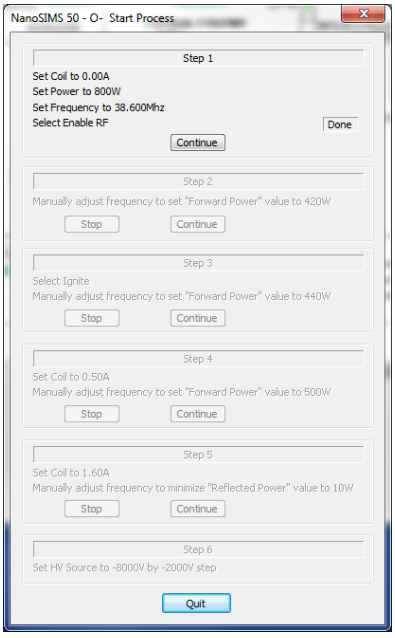


Figure 197: RF-Plasma source start window

To stop the source, click on the “Stop” button  and wait for the end of the process.

If, for stability reasons, the user wishes to leave the RF source on even though no analyses are running for a period of time of several hours (typically, overnight), we recommend to put the source in standby mode. You can do so directly from the front panel of the RF cabinet, or from the source window of the NS50 software.

Standby mode:
1- Lower the power to 600W.
2- Lower the Source HV and Extractor HV (Set to 0).
Note: the order is important. This will ensure a safe mode for the source and maintain the best source stability when needed.

To restore normal operation:
1- Restore the HV (8000 V under normal conditions), restore the Extraction voltage (4000 V under normal conditions),
2- Rise the power to the usual setting (800W Max)
Note: the order is important.

5.4.4 The Electron gun control

The E-Gun control (Figure 198) only needs three parameters:

- The **Heat** which corresponds to a DAC parameter of the heating current applied to the filament.
- The **Emission** value (current delivered by the HV power supply = electron current extracted from the filament by the grounded extraction electrode, plus eventual electrical leaks through incorrect insulation) is controlled by the **Wehnelt voltage** (0-200V) applied to this intermediate electrode focusing the emitted electrons. With HV on and a heated filament, a wehnelt voltage at zero will allow all generated electrons to be extracted, resulting in the highest possible $I_{emission}$. A higher wehnelt voltage value will reduce the emitted current to a point where it will cut all emission (if the filament is correctly mounted).
- The **HV** value which is the voltage applied on the filament heating current power supply in order to extract the electrons from the polarized filament toward the grounded extraction electrode.

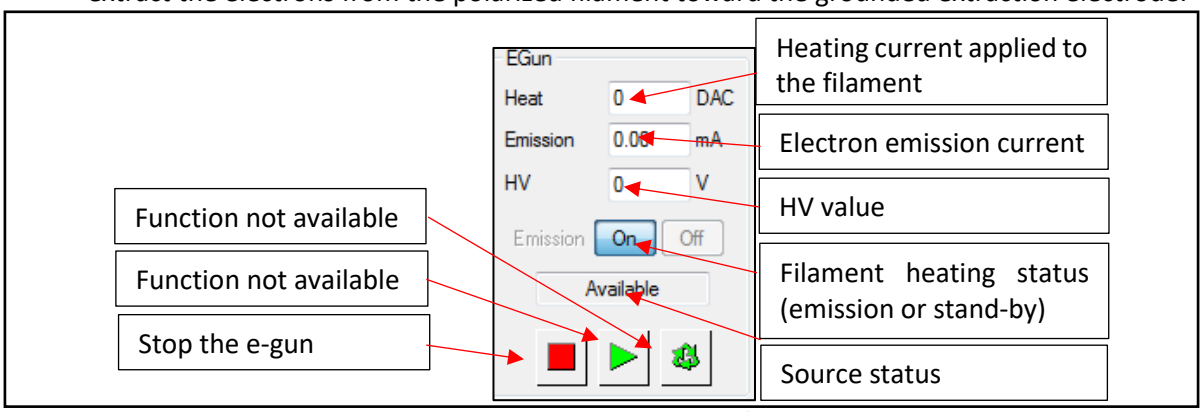
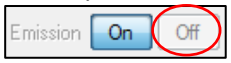


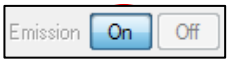
Figure 198: E-Gun control

Starting the E-Gun **is NOT** an automatic process, so the "Start" button  does not work. The start-up is manual. To see the starting procedure, refer to 9.3.2.

To stop the E-Gun, click on  and wait for a few seconds.
When moving the stage (including when switching between FCo/SIMS/CCD modes and when unloading/loading sample), it is recommended to turn off the e-gun emission. Some catastrophic degasing could happen if the electron beam was left with high impact energy on some fragile or uncured resin.

To keep the e-Gun turned on without emission, select the OFF button off the Filament emission status 

. The filament Heat current DAC value is automatically decreased to 1700 to keep it warm while the wehnelt voltage is set to its maximum value, cutting all emission.

To re-start the e-Gun emission, click on the ON 

button .

When "On", the e-gun parameters are usually as follow:

- Heat = 2500 DAC (never exceed 2700 DAC), with emission 0N, 1700 DAC with emission OFF.

CAUTION ! an emission above 3000 DAC will lead to a sublimation of the filament requiring its replacement soon !

- Emission = 0.14 mA (never exceed 2 mA)
- HV = -8000 V (never exceed a voltage of -8050 V)

5.5 Preset (and ISF)

5.5.1 Overview (Setup, ISF and Preset)

It is important to distinguish the "SETUP" , "ISF" and "PRESET" files.
The CAMECA NanoSIMS 50L is entirely computer controlled. All the instrument parameters are contained either in the SET-UP file or in the Instrument Status File (ISF).

The "**SETUP**" is a set of values defining **ALL** the parameters and their values for the **whole instrument** configuration. The "Setup" values, contained in the setup table, are automatically read and applied to the instrument as **"default" values**.
For example, if the operator asks for a CCD mode in the Navigator window, the instrument reads in the "Setup" the stage motorization positions independently of the other values already applied. Similarly, if the customer blanks the beam, the instrument reads the L1 HV and the C1 deflector values from the setup and applies them to direct the primary beam in FCp. Most of the time these values are the same for all analyses and the operator does not modify them from a sample to another.

The **ISF** (instrument status file) is a set of values for the whole instrument, defining a general mode of operation (e.g. Cs+ mode or O- mode,…).

The "**PRESET**" file is a smaller file containing a **reduced number** of specific parameters for **a specific adjustment or tuning** (e.g. lens HV value, deflection value, detector HV values…) and **belonging to a complete "ISF"** (Instrument Status File). The principle is to constitute a reduced set of selections to simplifying the instrument operation.

From one sample to the other, the EOS HV value or the Cy deflection value, for example, can be different. It is possible to *create* a "Preset" file and choose each parameter value to be in this file (see 5.5.5). Consequently, when the operator sends (applies) such preset files to the instrument, only the short list of values defined by the operator will be modified.

5.5.2 The main *Preset* window

Before working with presets, it is necessary to load an ISF files from the *Preset* window (Figure 199).

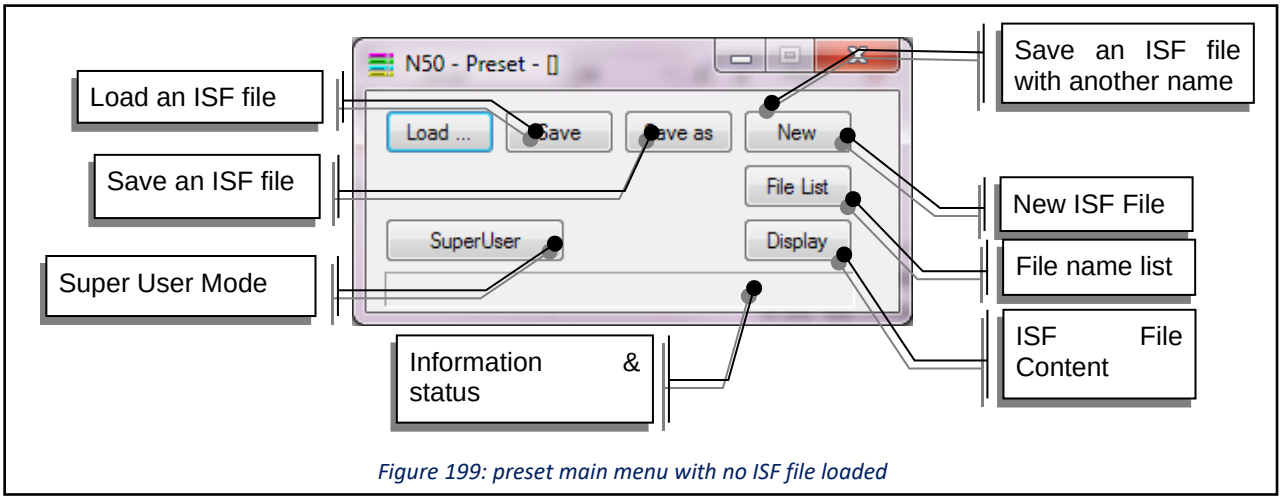


Figure 199: preset main menu with no ISF file loaded

5.5.3 Loading an ISF File

Select the Load ... button to load an ISF file and open the ISF loading dialog box (Figure 200).

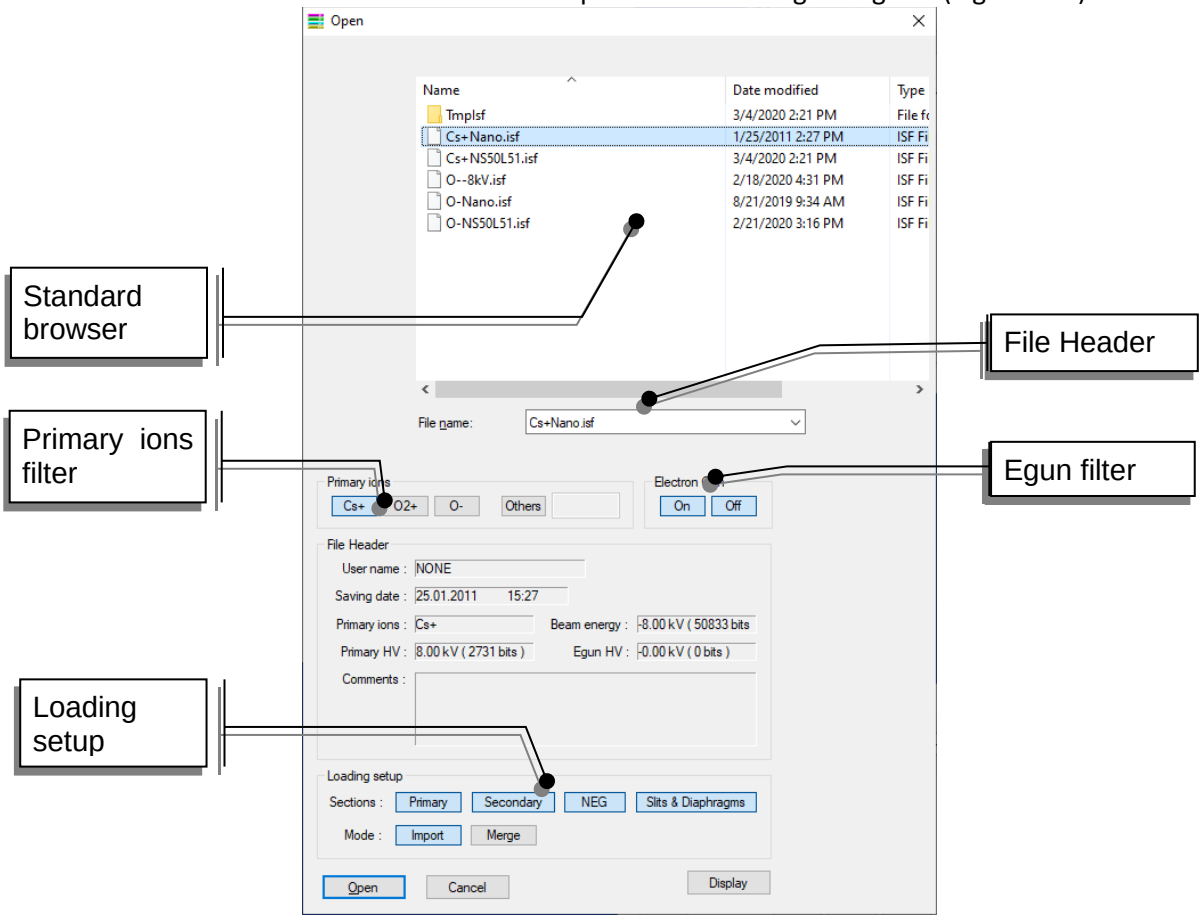


Figure 200: ISF file loading

The standard browser allows to **sort** the ISF files by name, by size or by date. Additionally, the *Primary ions* field allows the user to **filter** the ISF files according to their primary ions (Cs+, O-, O2+) and the *Electron gun*

use. The selection of an ISF file shows the file header content (User name, Saving date, Primary ions, Beam energy, Primary HV, Egun HV and Comments).
The *Loading setup* allows to **select the sections** (*Primary, Secondary, NEG*) to be loaded and **the loading mode**: *Import* or *Merge*.

Note that most of the time, all sections are selected and when you import a new ISF file, all preset sections are updated. However, if for any reason you wished to import only one section into the active file, it is possible. Just select the section(s) you would like to import from a given file.

We recommend to regularly save your presets into a new .ISF file, in order to keep a record of previous conditions. This way, if anything goes wrong, it is always possible to restore a previous configuration and start again from there.
Select the Open button to load the selected file or the Cancel button to return to the main menu bar without loading any file.

5.5.4 Main Preset dialog box

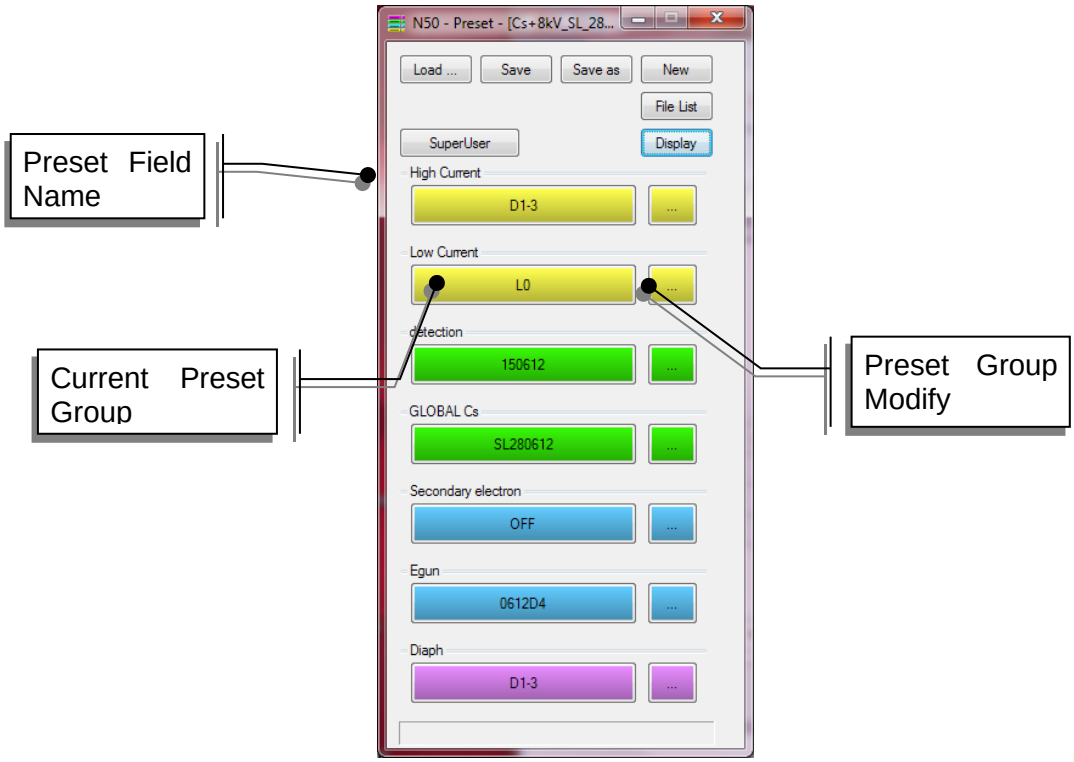


Figure 201: Preset main window with ISF loaded

The “ISF” file contains several presets defining distinct groups of values. Each **preset category** contains 10 possible group of values which can be sent to the instrument. The operator can select one group of values to send to the instrument (Figure 202).

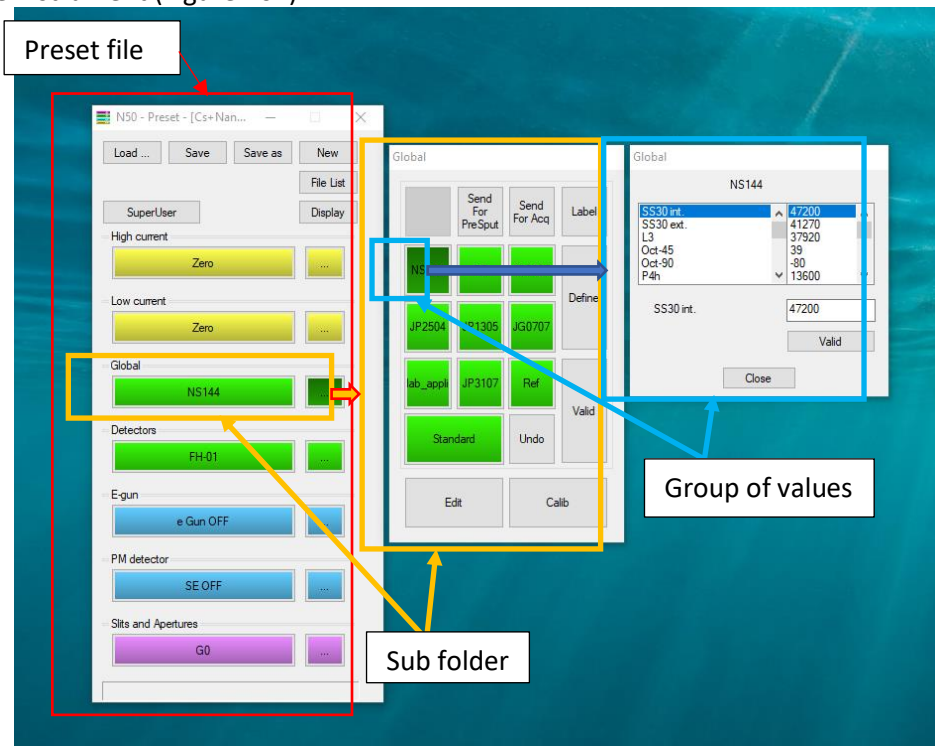


Figure 202: "Preset” files configuration

Each preset category is usually dedicated to one part of the instrument is in a specific color:

- **Primary beam: YELLOW**

Usual values for the primary beam Presets: L0, Lduo, C0x, Coy, Cduox, Cduoy, L1, C1x, C1y, Wf coil, Cwf, LCs
Commonly, the primary beam Presets are “bistable”, meaning that by clicking on the preset main button, the operator switches between two groups of values: the standard value (on the bottom left of the Preset window) and one of the nine selected values (one of the nine other groups of values).
For example, when the operator applies a high current to sputter a sample, he can come back to a normal current just by clicking again on the same button (Figure 203).

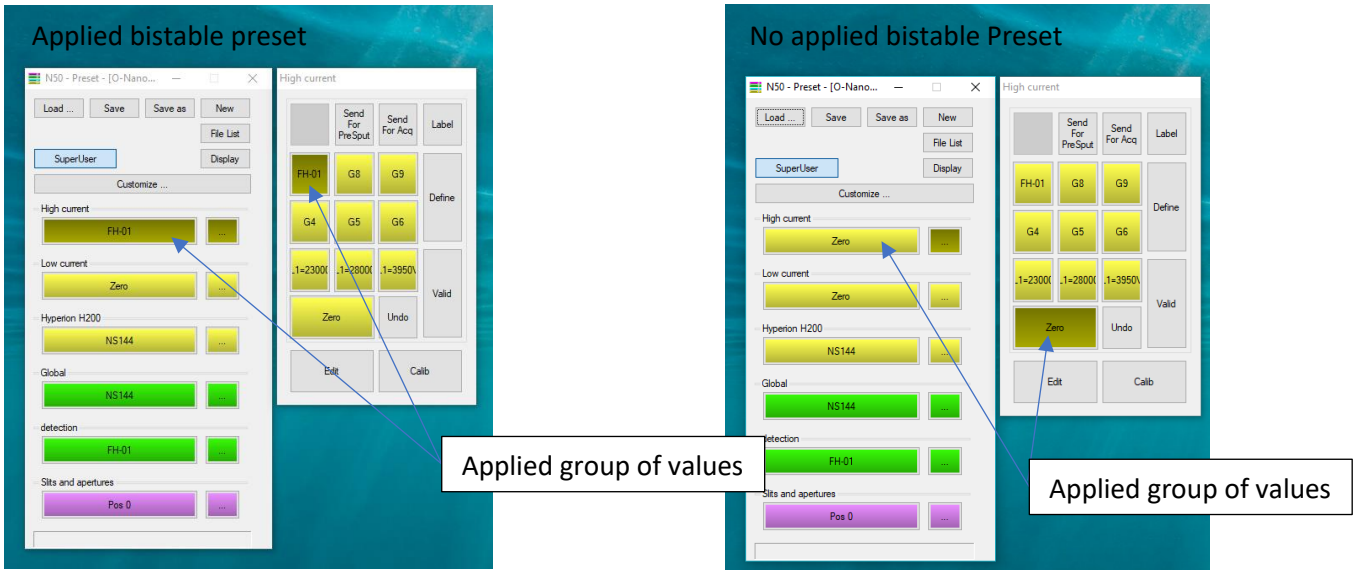


Figure 203: Switch between two group of values in "bistable" mode after having clicked twice on the “high current” Preset main button

- **Secondary beam: GREEN.**

Usual values for the secondary beam Presets: SS30int, SS30ext, L3, Oct-45, Oct-90, Octb-45, Octb-90,P4h, P4b, P1h, P1b, L4, EOS, EOP, EOW, EOW Ref, EOW offset, P2h, P2b Cy, P3h, P3b,LF2, LF3, C2x, C2y, Hex, SS100int, SS100ext, C3x, C3y, LF4, C4x, C4y, LF5, Q.

Frequently, the secondary beam presets are “monostable”, meaning that by clicking on the preset main button, the operator applies all the value with no turning back.

- **Electron gun (Egun) and Secondary electron detector (SED): BLUE** (Cs+ mode only)

Usual values for the Egun: C5x, C5y, LF6, C6x, C6y, LF7, EgunBe.
Usual values for the SED: PM, PM Offset, EgunBe
Commonly, the Egun and SED Presets are “bistable”, meaning, by clicking on the Preset main button, the operator usually switches between an ON state and OFF state. For example, when the operator wants to use the Egun or the SED, he just has to click on the Preset main button and when he does not want to use them, he just has to click again on the button.

- **Slits and Diaphragms: PURPLE.**

Values: D0 Cmd Pos, D1 Cmd Pos, Es Cmd Pos, As Cmd Pos, EnS Cmd Pos

This Preset can be “bistable” or “monostable” depending on the analysis requirements.

5.5.5 Editing a Preset group

Select the EDIT button beside the preset group to edit the groups of values. The following dialog box appears:

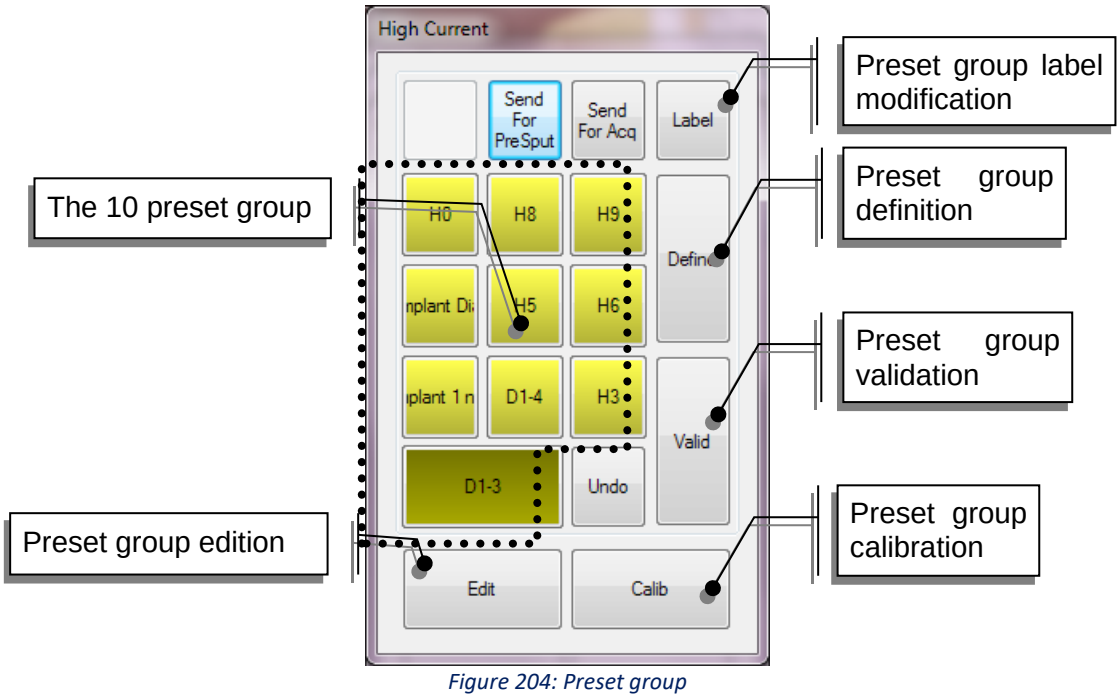


Figure 204: Preset group

hover the mouse cursor over a preset button to display its last calibration date (Figure 205):

Figure 205: display of the last calibration date of a preset (here preset "D1-3")

If a modification is applied to one of a preset group, the owner preset field in the Main Preset Dialog box appears with a '*' after the field name (Figure 206):

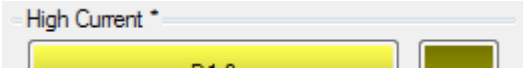


Figure 206: Preset field title displaying a '' after a modification has been applied to one its the group of values*

5.5.5.1 Preset group label modification

To modify a group label, select the button 

and the preset group to be renamed, then change the label in the dialog box (Figure 207):

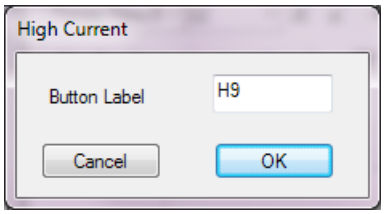
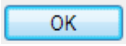
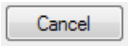


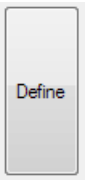
Figure 207: label editing dialog box

Select the 

button to validate the new label or the button 

to restore previous label.

5.5.5.2 Preset group definition

To define the preset group, select the 

button. A new dialog box appears (Figure 208):

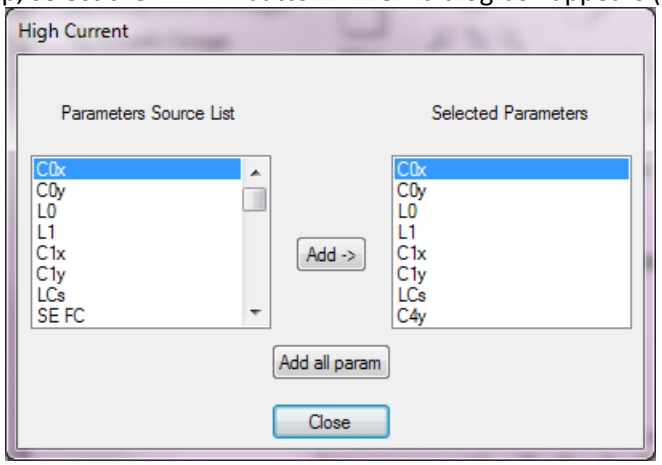
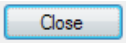


Figure 208: Value selection during a preset construction/modification

Select the 

button to add all the parameters in the preset list.

To add a single parameter, select the parameter in the "Parameter Source" list (left) and select the 

button.
Then the added parameter appears in the "Selected" list (right).
To delete a selected parameter in the "Selected" list, select this parameter and press the "Delete" key.
Select the 

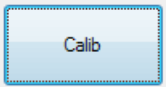
button to close the define dialog box.

5.5.5.3 Valid: Preset group application

To validate *and apply* a preset group, select a preset and press the 

button. The preset parameters values are then **sent to the instrument** and appear simultaneously **on the keyboard** and the dialog box disappears. The validated preset replaces the previous one in the preset main dialog box.

5.5.5.4 Calib: Preset group updating

To update a preset group with the current values (as can be read on the keyboard), select a preset and press the 

button. The following dialog appears (Figure 209):

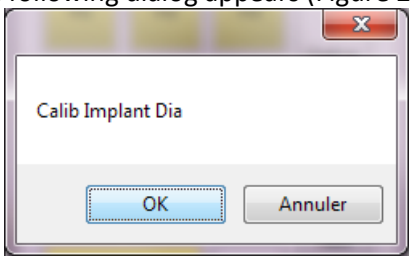
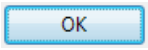
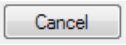


Figure 209: preset calibration confirmation window

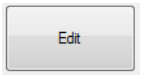
Select the 

button, then the preset parameters values are retrieved from the keyboard.

To abort calibration, select the 

button.

5.5.6 Preset group edition

To edit a preset group and modify each parameter manually, select a preset and press the 

button. The following dialog window appears (Figure 210):

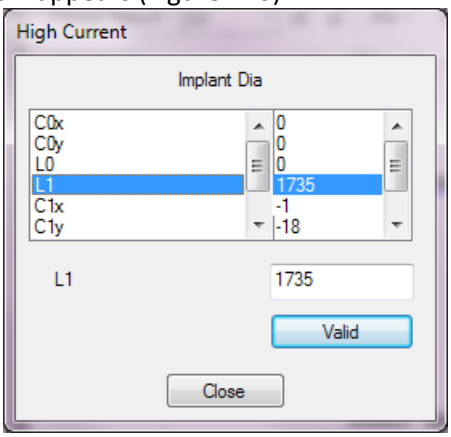
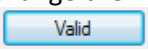
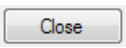


Figure 210: window to edit individually each value in a preset group

Select the parameter to modify in the list on the left side and change the value in the editable text below the list. To store the new value in the parameter list, select the 

button.

Select the 

button to exit the edition mode.

5.5.7 Saving an ISF File in another name

Select the Save as button to save an ISF file under another name. It opens the ISF saving dialog box (Figure 211):

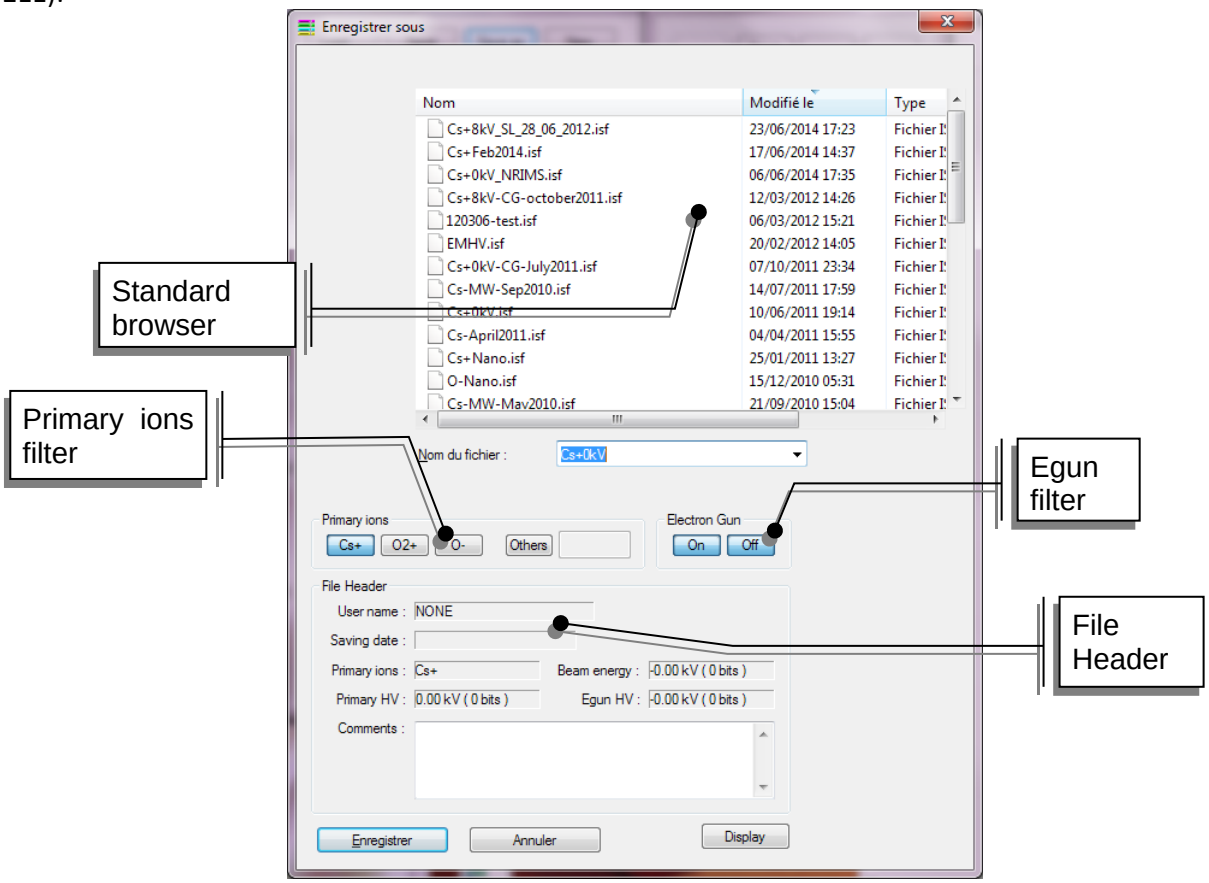


Figure 211: dialog box to save an ISF file under a new name.

The standard browser allows to sort the ISF files by name, by size or by date. Additionally, the *Primary ions* field allows to filter the ISF files according to their primary ions (Cs, O2+, O-, Other ions) and the *Electron gun* field filters according to the Egun status. The file header content (User name, Primary ions, Beam energy, Primary HV, Egun HV and Comments) show the header values to be saved. The comment editable box allow to add a comment in the file.

Select the Enregistrer button to save the selected file or the Annuler button to return to the main menu bar without saving.

5.5.8 Create a new ISF File

Select the New button to create an empty ISF file.

5.5.9 Super user mode

Like the SETUP (see below, chapter 5.9), the PRESET program offers a “Super User” mode. Protected by a password, this mode offers additional options that are meant to be accessible only to experienced users. It is thus recommended to be careful when sharing this password with NanoSIMS users and collaborators. And always proceed with caution when changing parameters protected by this “Super User” mode.

Select the SuperUser button to activate the super mode and enter the password (Figure 212):

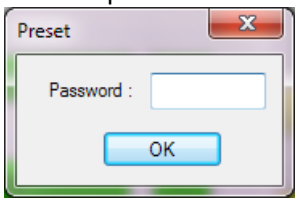


Figure 212: password window protecting the "super-user" mode

The following interface appears (Figure 213), showing a new option “customize”:

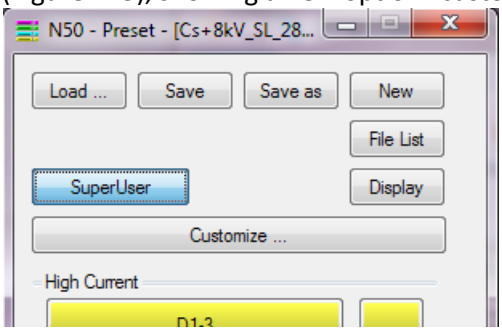


Figure 213: Preset main window in super-user mode

See the following chapter for how to use the customization option.

Do not forget to leave the SuperUser mode once you are done. To do so, simply click again on “SuperUser”.

5.5.10 Preset field customization

The Customize ... button allows the edition (add, delete or modify) of the current Preset fields. See Figure 214: preset field customization window below:

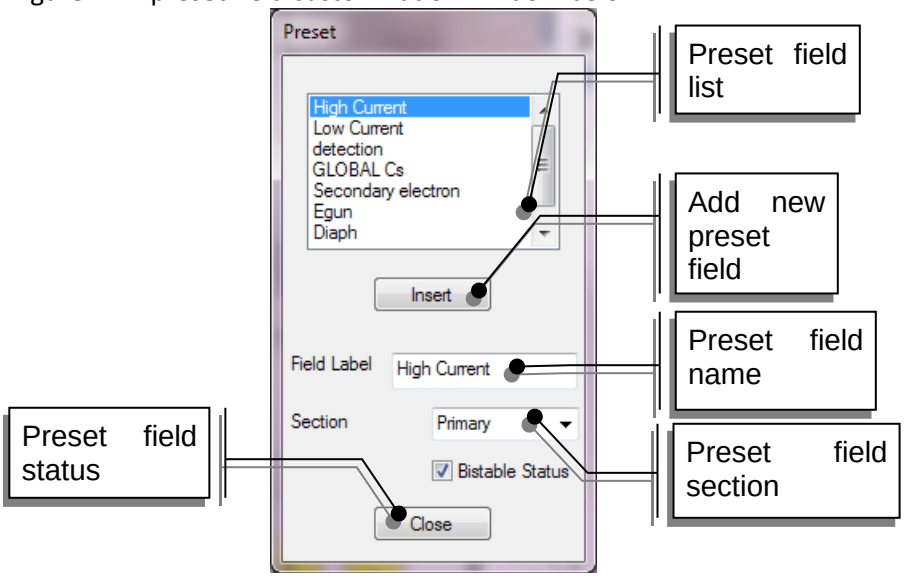


Figure 214: preset field customization window

To delete a preset field, select it in the preset field list and press the "delete" key.
To modify a preset field, select it in the preset field list and:

- Change the name in the *Field Label* edit box,
- Select the Section (Primary, Secondary, Neg),
- Check the status *Bistable* or not (Monostable).

To add a new preset field, select the position in the preset field list and select the Insert button. The new preset field is inserted before the selected position.

Select the Close button to return to the Main Preset dialog (Figure 201).

5.5.11 File list

Select the File List button to see the list of ISF file loaded for each preset section (Figure 215):

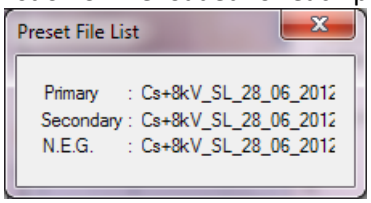


Figure 215: Preset file list

Check chapter 5.5.3 above to see how to load presets from distinct ISF files.

5.5.12 Display

Select the Display button to see the content (**=all the saved presets**) of the current .isf file:

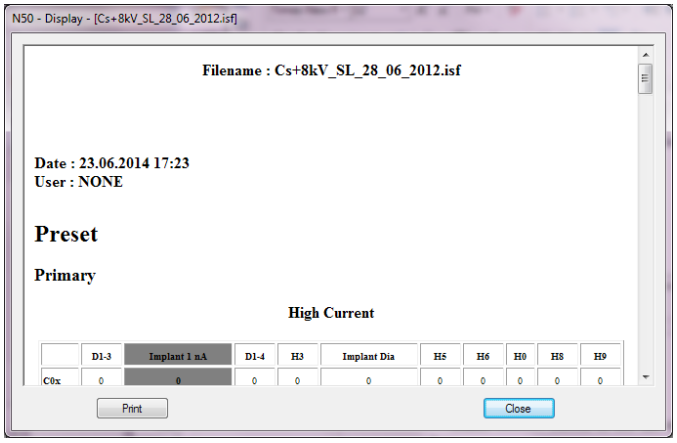


Figure 216: display window showing all saved presets from an isf file

Select the Print button the print the display window content.

Select the Close button to return to the Main Preset dialog box (Figure 201).

5.6 DefAnalysis

5.6.1 Getting Started

The Def Analysis program allows the user to set all the parameters for an analysis (i.e. define the analysis). When opening the Def Analysis program, the following window appears (Figure 217):

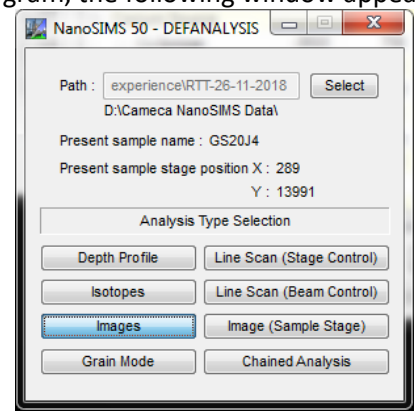


Figure 217: Def Analysis, Analysis selection window

Here the user can select the folder (path) where the analysis files will be saved and the type of analysis. Except for Chained Analysis, all programs show similar settings. When selecting the "Images" mode, for example, the following window opens (Figure 218):

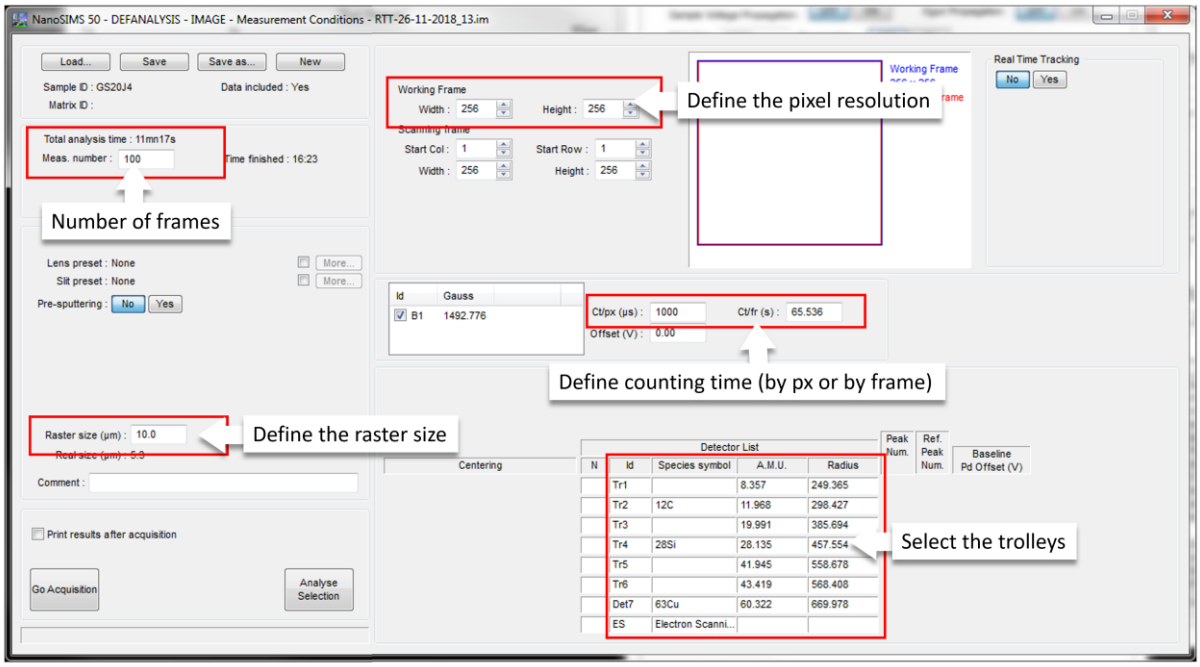


Figure 218: Images Def Analysis window

The main parameters the user needs to define to launch an image acquisition are:

- The working frame resolution (in pixels)
- The number of planes (or frames)
- The raster size (in µm)
- The counting (dwell) time (by px or by frame)
- The desired detectors

Once all those parameters are defined, the user is ready to launch an image acquisition. Click on Go Acquisition to go to the Analysis program (see chapter 5.7).

See the full description below for more details on the various options available in Def Analysis.

5.6.2 Full description of the Def Analysis

5.6.2.1 Different types of analyses

The first window of the Def Analysis file allows the user to select different kind of analysis:

- **Depth profile:** allows to record depth profiles of selected elements or isotopes. For details, see chapter 9.2.6.1.
- **Isotopes:** allows to define and record isotopic and elementary ratios. For details, see chapter 9.2.6.2
- **Images:** allows to record SIMS and SE images of selected elements, possibly over multiple cycles (frames). See chapter 9.1.14.
- **Grain mode:** allows to run isotope analyses over selected grains of an image. For details, see chapter 9.2.6.3.
- **Line Scan (Stage control):** allows to record a lateral isotopic or elemental profile by moving the stage under the beam. See chapter 9.2.6.4.
- **Line Scan (Beam control):** allows to record a lateral isotopic or elemental profile by scanning the beam over a line defined within an image. See chapter 9.2.6.5.
- **Image (Sample Stage):** allows to image or pre-implant a large area by recording a mosaic of images moving the stage between each image. See chapter 9.2.6.6.
- **Chained Analysis:** allows the user to set a series of analyses that will run automatically. See 9.2.6.7.

5.6.2.2 Save a Def Analysis setting

It is possible to create specific settings for an analysis. To create a new one from scratch, click on “new”. To load an existing configuration, click on “load”. To update a configuration, click on “save”. To save the settings as a separate file, click on “save as”.

5.6.2.3 Use of presets

By default, the analyses will run with the lenses and slits as **last tuned** by the user.
It is however possible to call **specific lens and slit presets**, either for the analysis run or for the pre-sputtering. To do so, the user must **first send the desired preset** either to pre-sputtering (**Send for PreSput**) or to acquisition (**Sent for Acq**) in the Preset window (Figure 219).

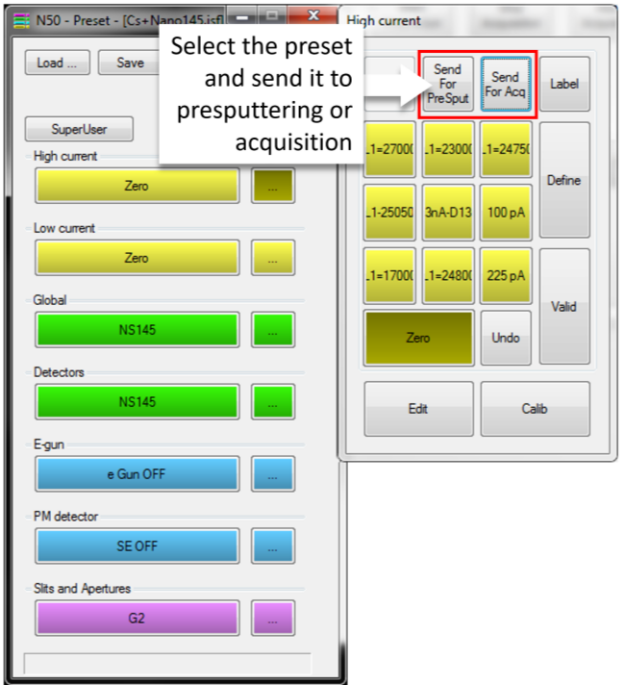


Figure 219: sending a preset to presputtering and acquisition Def Analysis files

Then, in the Def Analysis window, the user can select the Lens and/or Slit presets, for both acquisition and pre-sputtering (Figure 220). Note that only the last preset sent can be selected in Def Analysis.
Clicking on "More" allows to read the list of parameters in the preset.

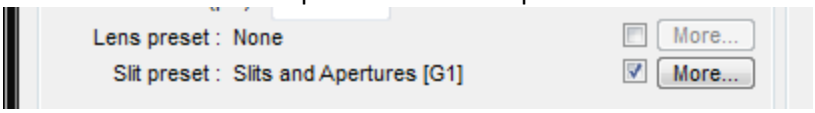


Figure 220: selection of presets in Def Analysis. Here the preset "G1" has been sent to Acq, allowing the user to select it.

5.6.2.4 Pre-sputtering

It is possible to add a pre-sputtering time before an analysis. It is particularly useful when doing a series of analysis where one wants the pre-sputtering conditions to be identical. To add a pre-sputtering before analysis, select "yes" to the Pre-sputtering option. An additional menu then appears (Figure 221). The user can then choose to have the pre-sputtering at high energy or low energy (more a deposition than a sputtering), the pre-sputtering time, the raster of the pre-sputtering, as well as specific Lens (high/low current) and Slit presets (see paragraph on Presets).

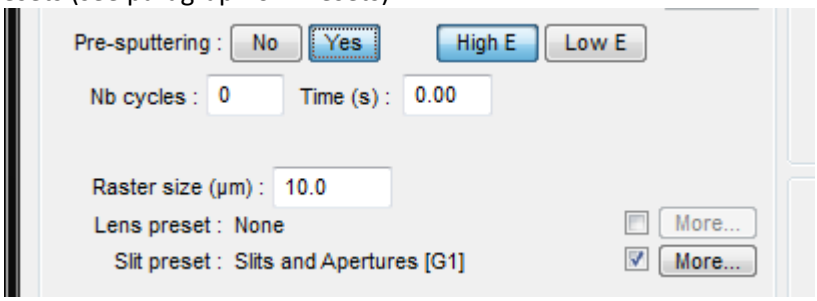


Figure 221: pre-sputtering definition in Def Analysis

5.6.2.5 Different types of frames

The program defines three types of frame (Figure 222) described below.
Units: *frames* are defined in **pixels** while *sizes* are defined in **microns**.

- The **Working Frame**: This is used in certain modes (grain mode or RTT) to acquire a first image of a larger area in which subsequent analyses in smaller areas will be defined. The "**Raster size**" as noted in Tuning, as well as in Def Analysis (Figure 223) is the one of the Working Frame.

- The **Scanning Frame**: This is the area that is actually sputtered by the beam during the analysis. In most case it is as large as the Working Frame. In case it is not, keep in mind that Raster and pixel resolution are defined by the Working Frame. The size (in µm) of the scanning frame is noted sometimes as the "**Real size**".

- The **Blanking Frame** or **Counting Frame** (= electric window or gate): in certain modes, it is possible to add a counting blanking. In that case, a third frame is defined. The whole Scanning Frame is sputtered, but the signal is measured only when the primary beam is inside this Blanking Frame. This can be useful when one wants to reject the signal coming from the edges of the crater during direct depth profiling or precise isotopic ratios. Note that the detector will always receive secondary ions (with risk of saturation !) but the signal will not be counted outside the blanking frame. The "**counting size**" is then the size of the counting frame.

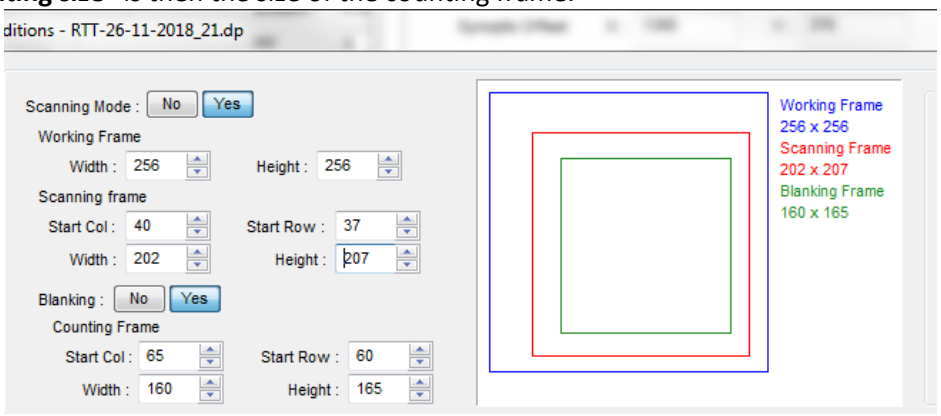


Figure 222: display of the three types of frames in Def Analysis

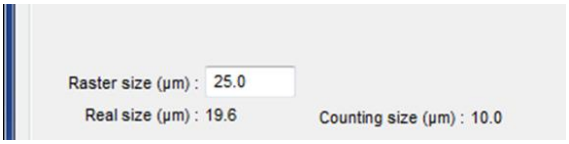


Figure 223: raster size (in µm) for each type of frame.

5.6.2.6 Ratios

In Isotopes, Depth Profile and Grain modes, it is possible to record elementary or isotopic ratios. When in those modes, click on the “Ratios” button. A new window “isotopic ratios” opens (Figure 224). There, enter the desired ratios, using the N reference appearing in the Detector list. Note that a N reference is only given to the selected detector. Therefore, their numbers are distinct from the trolley IDs.

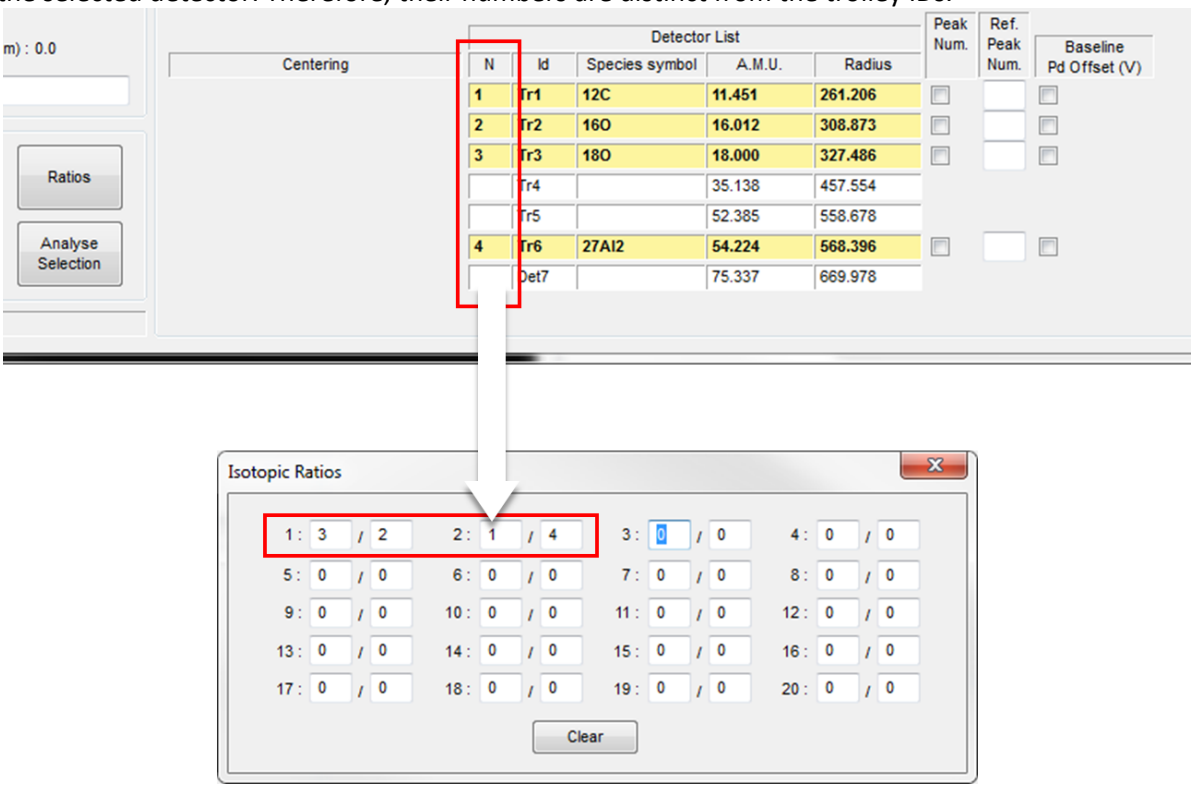


Figure 224: Ratio definition through Def Analysis in Isotopes mode

5.6.2.7 EOS centering

It is possible to add an EOS and P3/Cy centering during the analysis, either every frame (= scanning cycle or scan) or each *n* number of frames. This option requires that an automatic EOS centering has been set earlier.

- Open the EOS program in Tuning and launch an EOS scan. Preferentially select the trolley with the highest signal that will stay high during the whole acquisition. Click on “Apply CL”.
- Select “Automatic EOS Centering” and launch another scan. Click on “Apply to Setup” and “Save to Def Analysis”.
- Open Secondary Ion Beam, select “vertical” and launch a P3 scan. Click on “Apply CL”.
- Select “Automatic Beam Centering” and launch another scan. Click on “Apply to Setup” and “Save to Def Analysis”.
- Select “Horizontal and launch a Cy scan. Click on “Apply CL”.
- Select “Automatic Beam Centering” and launch another scan. Click on “Apply to Setup” and “Save to Def Analysis”.

Now in Def Analysis, under « centering » the options « EOSC » (EOS centering) and « SIBC » (Secondary Ion Beam centering) are now available (Figure 225). Select EOSC, SIBC and EOSC (they appear blue when selected). It is also possible to define the number of frames between each centering.

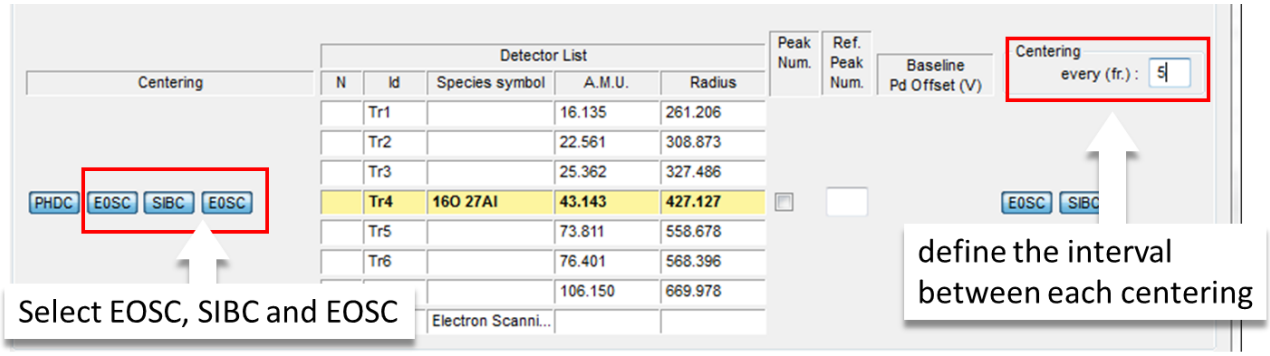


Figure 225: optional centering of EOS (EOSC), P3 and CY (SIBC) in Def Analysis

5.6.2.8 HMR Peak centering

It is possible to check the centering of the HMR peak during the analysis, either every frame or each *n* number of frames. This option requires that an automatic centering has been set earlier. Open the HMR program in Tuning and for each detector used:

- Launch an HMR scan. Click on “Apply CL”.
- Select “Automatic Peak Centering” and launch another scan. Click on “Apply to Setup”.

Then in Def Analysis, check the box next to the detectors you wish to center during the analysis (Figure 226). A number (Peak Num.) is then associated to the detector. It is possible to use a different detector as reference to center a detector. In that case, enter the Peak Num. of the reference detector in “Ref. Peak Num.” This can be useful in case the centering of a given peak might fail (in case of low signal or peak interference, for instance). In this case the shift is calculated proportionally to the measured shift from the peak used as a reference: $shift_{peak\ 2} = (radius_{peak\ 2})/(radius_{peak\ 1})*shift_{peak\ 1}$
It is also possible to specify the interval of frames (= cycles or scans) at which the peak centering is checked.

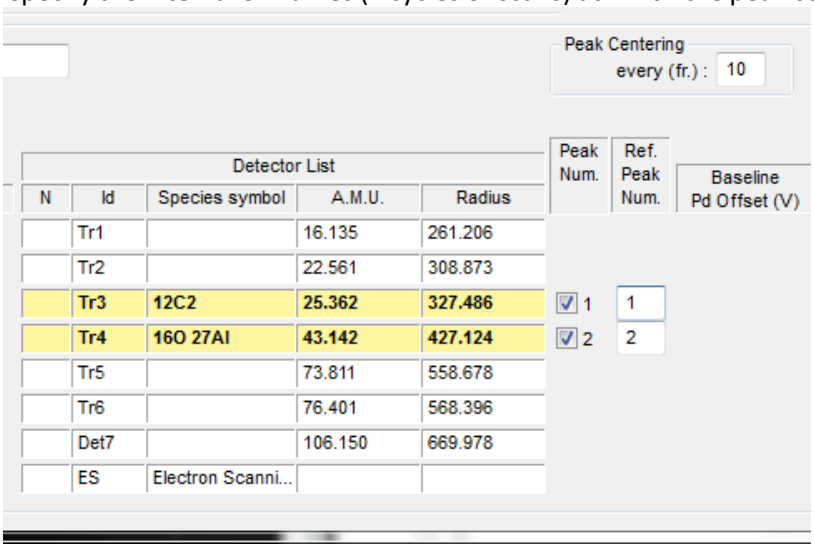


Figure 226: HMR Peak centering option in Def Analysis

5.6.2.9 Automatic PHD adjustment

It is possible to add a PHD centering (re-centering the EM PHD_{max} by re-adjusting HV_{EM}) during the analysis, either every frame or each *n* number of frames. This option requires that an automatic PHD centering has been set earlier. Open the PHD program in Tuning and for each detector used:

- Launch a PHD scan. Click on “Apply CL”.
- Select “Automatic PHD Centering” and launch another scan. Click on “Apply Ref to Setup” and “Save to Def Analysis”.
- Check that in the Setup program > Centering > PHD, the “Automatic correction” is ON.

Now in Def Analysis, under « centering », the option PHDC (PHD centering) is now available (Figure 227). Select this option to apply it to the Def Analysis.

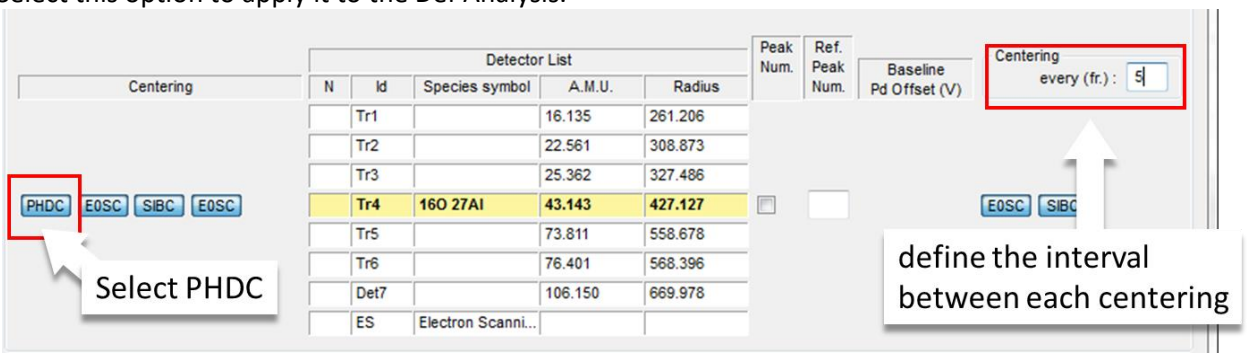


Figure 227: optional adjustment of PHD in Def Analysis

5.6.2.1 Baseline correction for isotope analyses

This option is only available for “isotopes” analyses. It allows a correction of the signal from the “baseline” noise that can occur between masses when the beam is ON. This is distinct from the measurement of the detector & electronics “background”, which is measured when the beam is OFF.
To measure the baseline on a detector, select the “Baseline” option as shown in Figure 228. The Pd plates will then deviate the beam so as to measure the baseline next to the peak. A deviation of about ±50 V is advised. Measurement time (Baseline measure) and interval between each measurement (“every”) can also be adjusted.

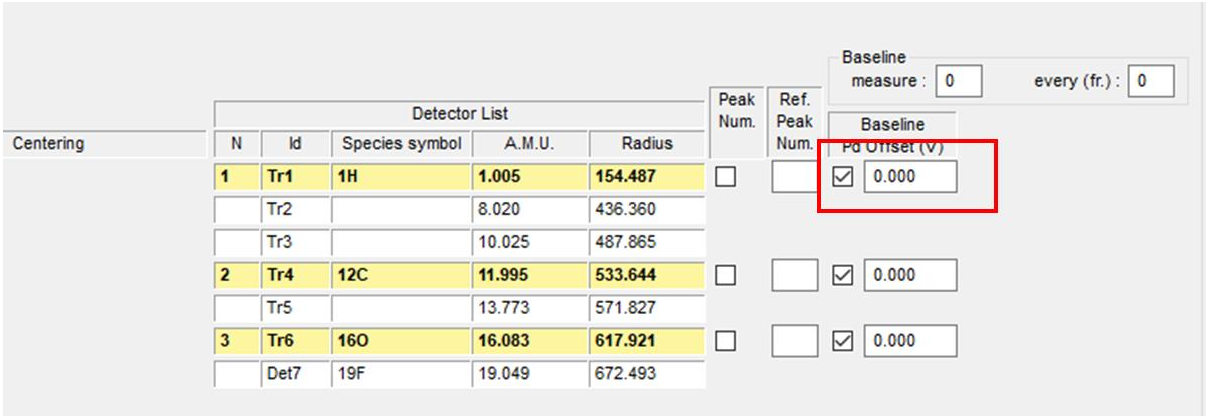


Figure 228: optional measurement of a detector’s baseline.

5.7 Analysis

Once all the parameters are defined in Def Analysis, the user click on “Go Acquisition” in the Def Analysis window. The Analysis window then opens. In image mode it will show the image acquisition (Figure 229), while in depth profile and isotope modes it will show a profile acquisition (Figure 230) as well as ratio data. Here are the basics of the Analysis program, a main NanoSIMS application:

- **Start Acquisition:** Click on Start to launch the acquisition. On the right a live preview of the acquisition is shown.
- **Stop:** To interrupt the acquisition and save what has already been analyzed, click on Stop.
- **Abort:** to interrupt an acquisition without saving the unsaved data, click on Abort.
- **Control:** Shows the tuning parameters of the analysis, such as primary beam intensity, lenses, slits, X, Y and Z positions, etc..
- **Analytical Parameters:** shows all parameters and slit used for the primary and the secondary beam, as well as the presets.
- **Change MC:** After an analysis, click on “change MC” to go back to Def Analysis and modify analysis settings.
- **# of planes (=cycles)**: Shows the number of planes defined in Def Analysis. It is possible to change the number of planes during an acquisition without interrupting it.

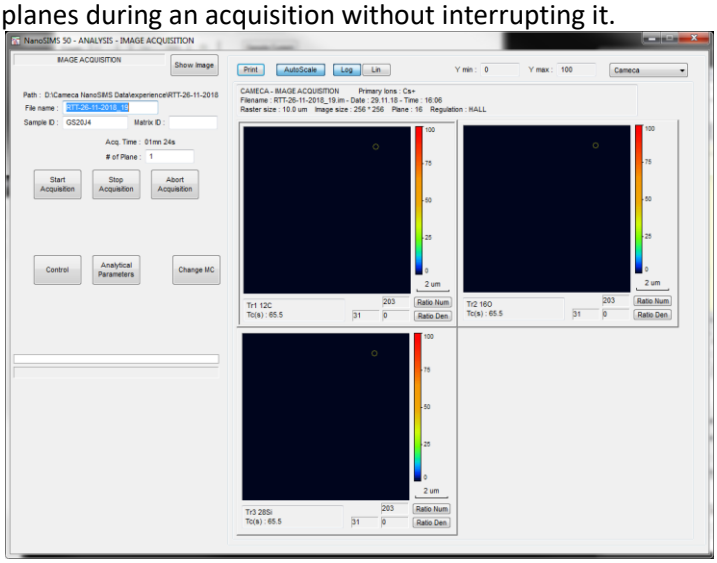


Figure 229: Analysis main window in Image mode

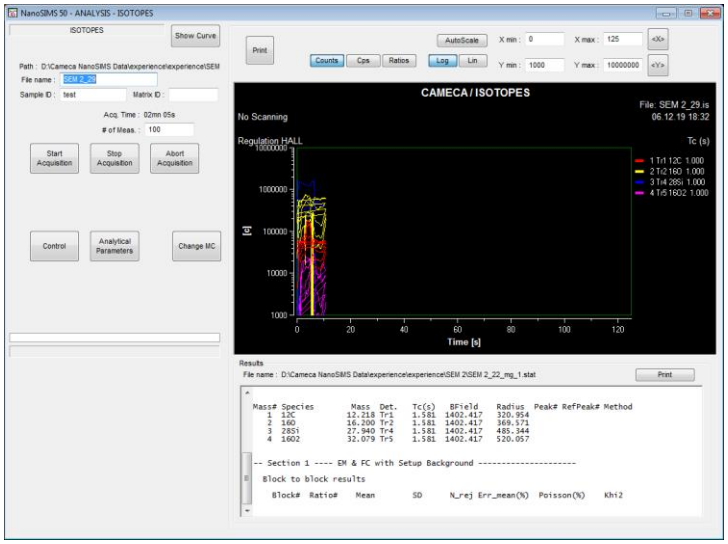


Figure 230: Analysis window in Isotope mode

Some additional options can appear, depending on the analysis mode or the options selected. Specificities of the different analysis modes (direct depth profiles, isotopes, grain, line-scans, stage-scan, etc..) are described in chapter 9.2.6.
Options specific to alternated sample drift correction are described in chapter 9.3.8,
Options linked to hybrid and monocollection modes are described in Expert operations 9.3.6.

5.8 WinImage and WinCurve

These two buttons allow accessing the two CAMECA SIMS data processing software:

- **WinImage II** allows processing and extracting information from SIMS image stacks. In addition, it allows exporting line-scans or depth profiles in WinCurve format for specific processing. Finally, data can be exported to external formats (e.g. Excel, TIFF...)

- **WinCurve** allows processing SIMS depth profiles and line or band-scans.

- A third CAMECA software, **DataExplorer**, is a dedicated search engine allowing to filter, pre-visualize and retrieve very efficiently your (numerous) CAMECA data stored on a hard disk.

- A fourth CAMECA software is **EditSpecies.exe**, allowing you to modify the chemical element in an image file, when it is empty or wrong for some reason.

Refer to the separate users' manuals for these software.

5.9 Setup

5.9.1 Introduction

The *Setup* file contains all the instrument configuration parameters. For each parameter, this file contains an identification number and the parameter value. Note that some parameters are editable only in super-user mode.

Like for the Presets the SETUP program offers a "Super User" mode. Protected by a password, this mode offers additional options that are meant to be accessible only to experienced users or Cameca engineers exclusively. It is thus recommended to be careful when sharing this password with NanoSIMS users and collaborators. And always proceed with caution when changing parameters protected by this "Super User" mode.

⇨ **Be very careful when modifying those parameters.** It is recommended not to, unless you are sure of what you are doing! Contrary to ISF files for presets, there is only ONE setup file and there is no tool to load/save different Setup files. Any modification to the Setup file will overwrite previous data. It can be wise, especially when leaving the instruments in the hands of beginners, to save a back-up of the Setup by making a copy of the Setup folder (D:\Cameca NanoSIMS Data\setup) and change its name as to not disturb the program.

⇨ Note that the Setup is compatible with all NanoSIMS instrument and takes into account options and evolutions that your machine might not have.

5.9.1.1 Setup browser

The setup window allows the user to view and modify all saved parameters easily. They are arranged in categories: Holder, Tuning, Keyboard, Hardware, Slit, Diaphragm, Detector, B Field, Directories, Sources. and Centering.

The common setup user interface looks like Figure 231 below:

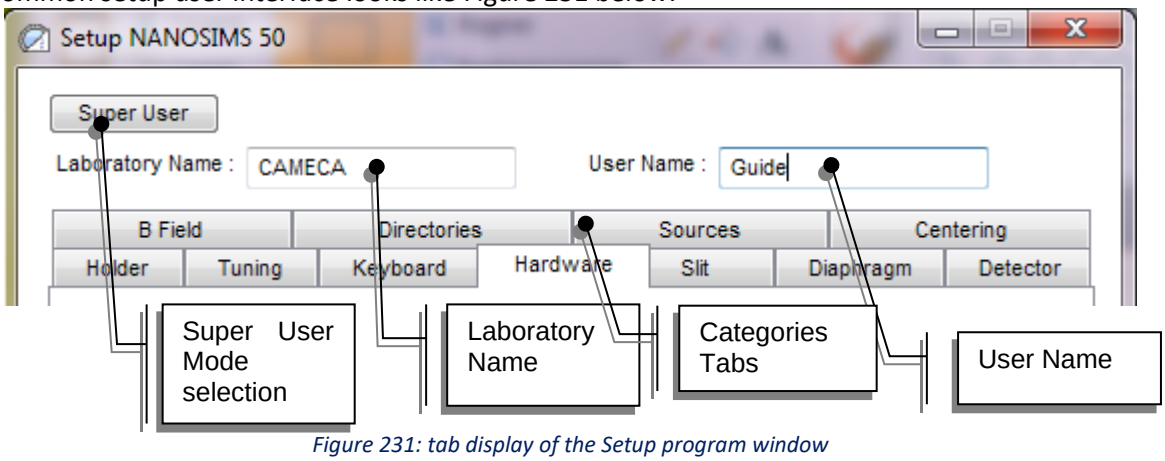


Figure 231: tab display of the Setup program window

Some parameters need super-user mode to be editable. To activate this mode, you have to select the Super User button and enter the password in the dialog box (Figure 232):

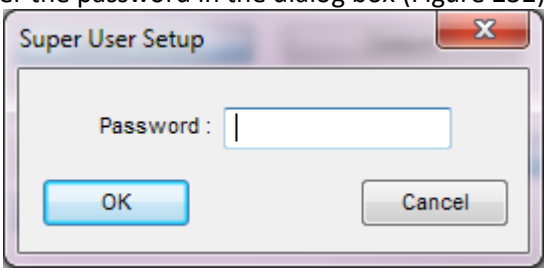


Figure 232: password window for the super-user mode of the Setup program

Ask your lab manager (or Cameca staff) for the password.

The super user setup user interface looks like this (Figure 233):

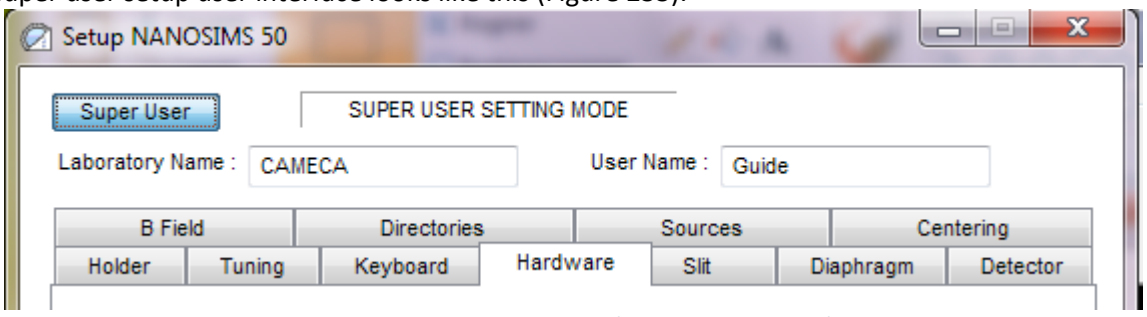


Figure 233: Setup program window in super-user mode

We advice to always switch back to “normal” mode once you are done with the “super user” mode. Click again on “Super User” to quit the “super user” mode.

For all the parameters, you can have information (subject, min and max limits, unit) by hovering the mouse pointer on the graphical item. For example, Figure 234 below shows min and max values for the Standard sample X position.:

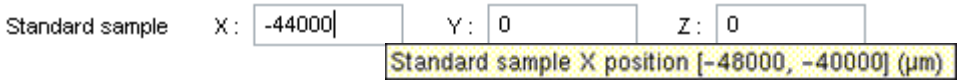


Figure 234: additional information displayed when hovering over a parameter.

To apply your setup parameters, click the Apply button. All the parameters are saved in a private file and sent to all connected software. To restore parameters from private file, click the Restore button. To print parameters, select the Print button (Figure 235).

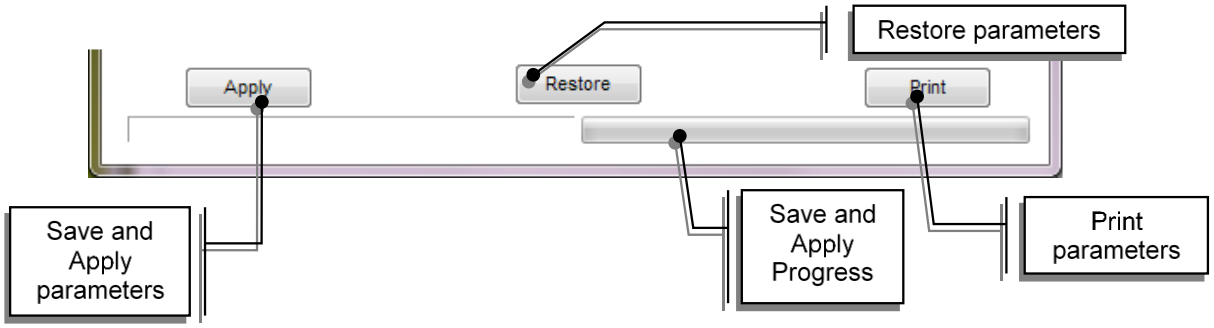
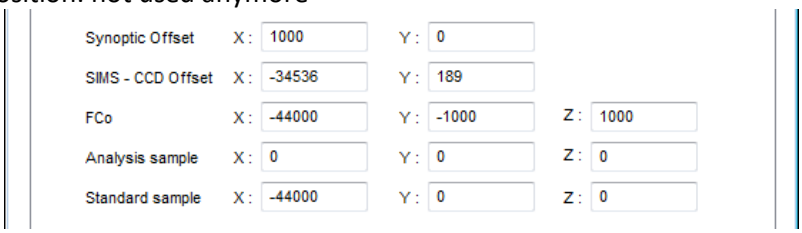


Figure 235: bottom of the Setup window.

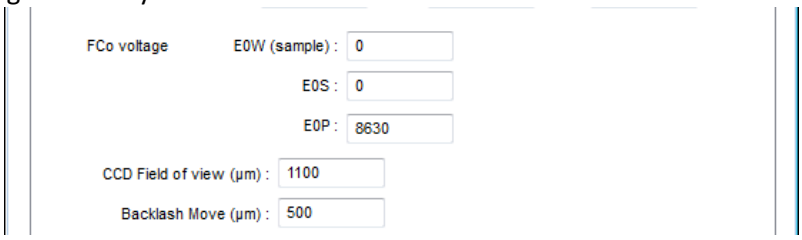
5.9.2 Holder

This tab allows the user to modify the parameters used by the Navigator and Optical Image programs.

- Synoptic Offset (fine adjustment of the current position on the holder graphics) :
- SIMS – CCD Offset: X and Y offset so that the CCD image and the beam position in SIMS mode match. (See chapter 9.2.9)
- FCo: coordinates of the stage to read FCo current. For CAMECA engineers only.
- Analysis sample: X, Y, Z coordinates of the center of the sample holder in SIMS mode (should be all at 0)
- Standard position: not used anymore

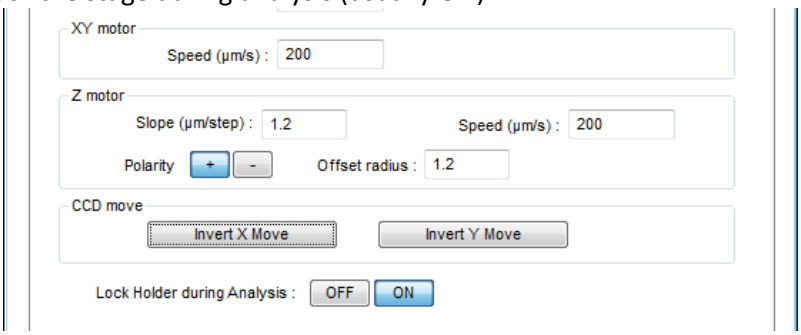


- FCo voltage: coaxial lens voltage values applied in FCo mode. For CAMECA engineers only.
- CCD field of view
- Backlash move: movement length for the backlash correction (see chapter 5.3.1.8 for details). For CAMECA engineers only.



- Stage motor speeds (X, Y and Z). For CAMECA engineers only.
- Z µm/step conversion (it should be around 1-1.2)
- Option to invert the X and Y moves (usually X is inverted, Y is not)

- Option to lock the stage during analysis (usually ON)



5.9.3 Tuning

This page allows the user to modify parameters used by the Tuning software. It contains several groups of parameters:

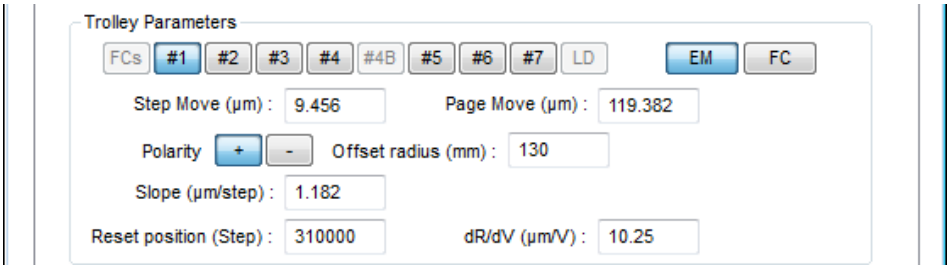
5.9.3.1 Active detectors

This group shows the active detectors for Tuning. The choice of available detectors depends on the NanoSIMS version (50, 50L).

5.9.3.2 Trolleys parameters

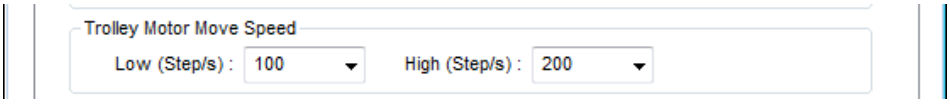
This group is used to define various parameters for each selected trolley:

- Detection mode (EM or FC). The switch is automatic when switching from one mode to the other (see chapter 9.2.11)
- Trolley step (</>) and page (<</>>) moves in Tuning. Those can be adjusted at the user's preference.
- Slope and Offset parameters for step to µm conversion. For CAMECA engineers only.
- Reset position for when a motor reset is necessary (see chapter 5.2.10). For CAMECA engineers only.
- dR/dV conversion coefficient to calibrate the trolleys in mass. For CAMECA engineers only.



5.9.3.3 Trolley Motor Move Speed

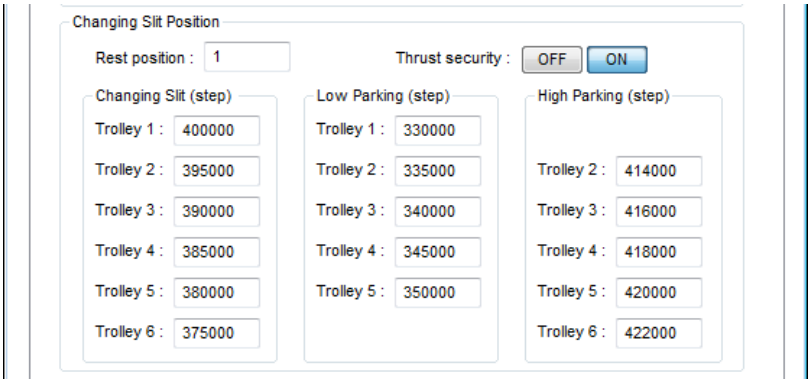
This group handles the motor speeds for the trolley movements. This option is only available with the most recent instruments with the new motorization. For CAMECA engineers only.



5.9.3.4 Changing slit position

This group is used to define the trolley positions for the exit slit change procedure. All those parameters are defined at the Cameca factory and are for CAMECA engineers only:

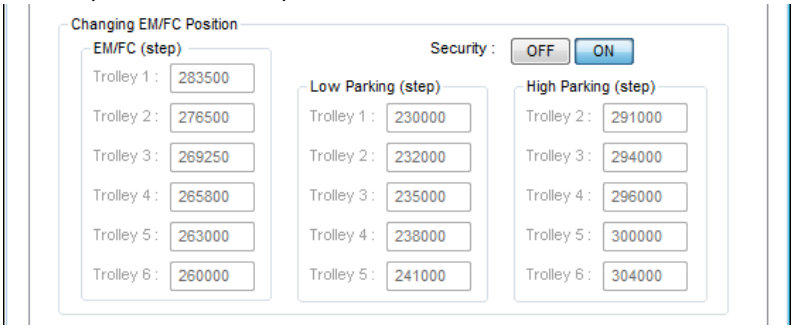
- Rest position
- Position of the trolley for slit change procedure
- Low and high parking positions
- Thrust security activation (it should be ON).



5.9.3.5 Changing EM/FC position

This group is used to define the trolley position for the EM/FC switch procedure. For CAMECA engineers only :

- Trolley position to operate the EM/FC switch
- Low and high parking positions
- Security activation (it should be ON).



Refer to chapter 9.2.11 for details on the FC/EM switch procedure.

5.9.4 Centering

This page allows you to modify parameters used for the various functions in the Tuning program.

Note: for certain centering programs, a percentage for the central line computation is asked. Depending on the parameter, it will be of 10, 50 or 80%. This corresponds to the percentage of the peak height at which

the peak width is measured. In the Tuning programs, those parameters are referred to as L10, L50, L80 and are used for automatic centerings. Figure 236 shows the example of the measurement of L80 for the automatic centering of E0S.

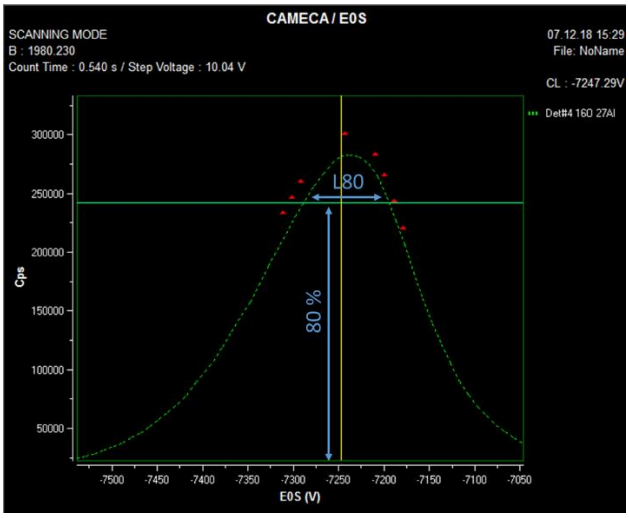


Figure 236: measurement of L80 parameter (=peak width at 80% of the signal max).

5.9.4.1 FC and Background

This group is used to define the waiting time and the counting time for the FCp check (primary ion beam current in the upper column) before and after an analysis. When relevant, the background noise of the multicollection FCs is checked at the same time as well :

- Waiting time (for the detectors) before measurement. Recommended values: 2s in EM, 10 s in FC (in particular for isotope measurements) or when using the e-gun.
- Counting time for measurement. Recommended values: 3s in EM, 10 s in FC (in particular for isotope measurements) or when using the e-gun.

5.9.4.2 Preset Lens

This group is used to define the waiting time (for the detectors) after applying a lens preset. It is recommended to use a waiting time of 1s in EM and 5s in FC or when using the e-gun.

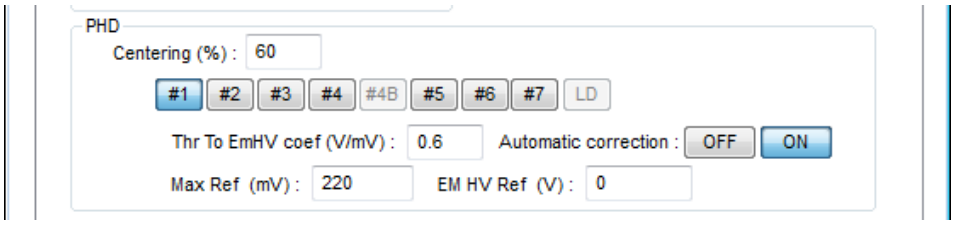
5.9.4.3 All Centering

This group is used to define the waiting time and pre sputtering time before a centering scan. Those parameters are applied to all centering functions. Recommended values 3s in EM (can be extended to 5s if using the e-gun and a high intensity primary beam), 10s in FC.

5.9.4.4 PHD

This group is used to define the PHD centering parameters for the automatic PHD adjustment:

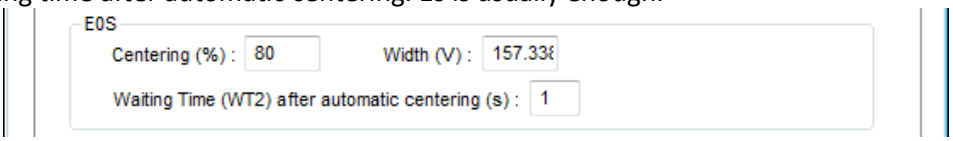
- Percentage for the center line computation (60% is recommended)
- EMHV to Thr conversion coefficient. It should be at 0.6 V/mV
- Automatic conversion flag. It is recommended to leave it ON.
- The Max voltage reference. 220 mV is recommended, though it can be increased to 250 mV for high precision isotopic analyses (see chapter 5.2.21).



5.9.4.5 E0S

This group is used to define the E0S centering parameters necessary to the E0S automatic centering function:

- Percentage for the center line computation (80% is recommended).
- Apparent width of the corresponding graph at x %, which is measured from the E0S scan (see chapter 0 for details on how those parameters are used)
- Waiting time after automatic centering. 1s is usually enough.



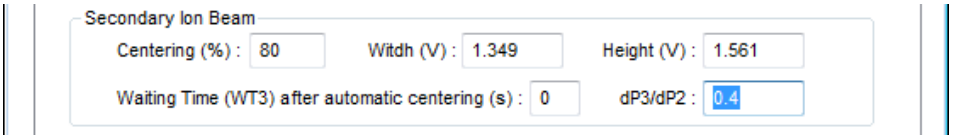
5.9.4.6 Secondary Ion Beam

This group is used to define the secondary ion beam centering parameters necessary to the CY and P2/P3 automatic centering functions:

- Relative percentage for the centerline computation (80% is recommended).
- Apparent width and height of the corresponding graph at x % which is measured from the Cy and P3 scans (see chapter 9.3.3 for details on how those parameters are used).

Dependence coefficient for P3 and P2. (This ratio value that keeps the beam horizontal while varying its height position in ES through P3, is set by CAMECA engineers. It should be in the range of 0.36-0.4. We recommend to NOT modify the value determined by CAMECA production or service engineer. This tuning goes beyond the scope of this users guide.

- Waiting time (for detection) after automatic centering (1s in EM, 5s in FC or when using the e-gun)



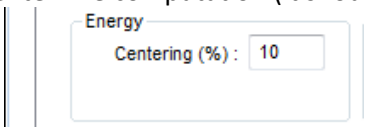
5.9.4.7 E0S vs Z

This function is not available.

5.9.4.8 Energy

This group is used to define the E0W automatic centering functions. In this group, only one parameter is accessible:

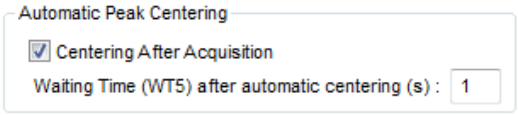
- Relative percentage for the center line computation (it should be at 10% of max signal)



5.9.4.9 Automatic Peak Centering

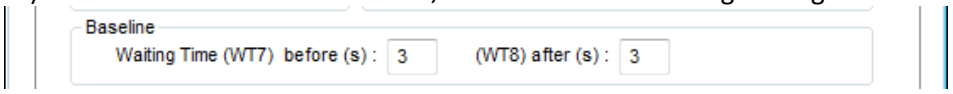
This group is used to define the automatic peak centering parameters:

- Position of the centering in the acquisition (if unchecked, the centering will be before the acquisition)
- Waiting time after automatic centering



5.9.4.10 Baseline

This group is used to define the waiting time before and after measurement of the FC baseline (see chapter 5.6.2 for details). Recommended values: 1s in EM, 3-5s in FC or when using the e-gun:



5.9.4.11 IMF-AS (instrumental mass fractionation at AS)

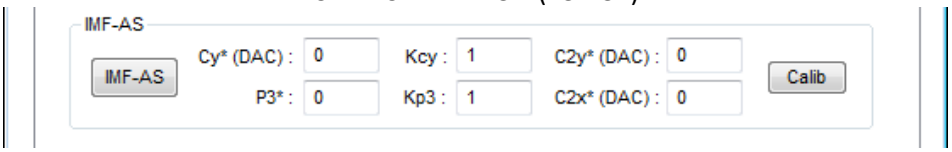
When scanning Cy the secondary ion beam rotates around a center point located between ES and AS. Hence scanning the beam in ES moves it in AS too. It can be a problem for small AS size. It is possible to keep the secondary beam centered in the Aperture Slit and centered also in the entrance slit by adding some compensating voltage to C2y and C2x. (See chapter 9.3.1.3.4)

This group is used to define the IMF-AS mode parameters. When ON (“IMF-AS” button blue), this function links C2x and C2y to Cy and P3. When you first hit “calib”, it will retrieve the current Cy, P3, C2x and C2y from the keyboard (saved as Cy*, P3*, C2y* and C2x*) and calculate the coefficients Kcy and Kp3.

Thus, when later adjusting P3 and Cy, the new C2y and C2x will be calculated as follow:

$$C2y = C2y^{*}+kCy \times (Cy - Cy^{*})$$

$$C2x = C2x^{*} + kP3 \times (P3-P3^{*})$$

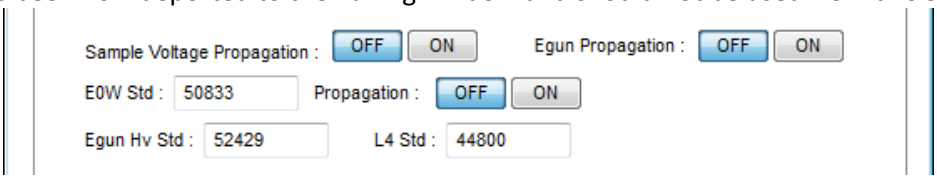


5.9.5 Keyboard

This page allows the modification of the parameters used by the Keyboard.

5.9.5.1 Propagation

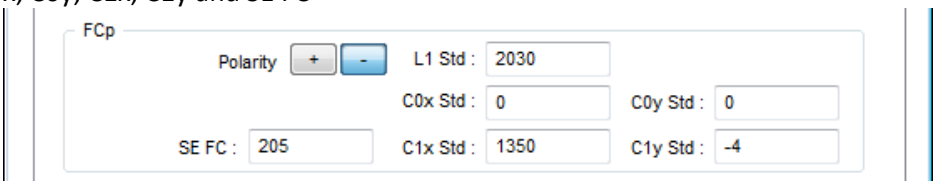
This group has been now deported to the Tuning window and should not be used from this Setup window.



5.9.5.2 Primary Faraday cup

This group is used to define lens and deflector values to deflect the primary beam toward the primary FC and read the primary beam intensity, in positive and negative mode:

- L1, C0x, C0y, C1x, C1y and SE FC



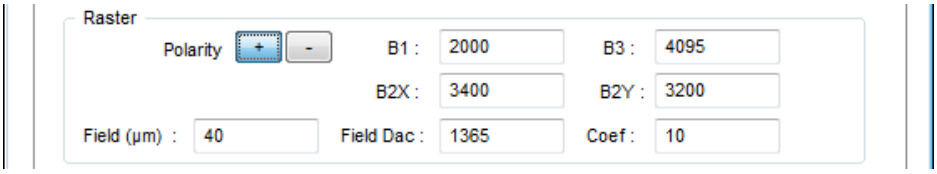
L1 (31 200 bits in most recent instruments) and C1 X (-1350 in positive mode, and 1350 in negative mode) are fixed values, which must not be changes. However, C0 X, C0 Y and C1 Y should be adjusted every time the primary column is tuned.

5.9.5.3 Raster

This group is used to define the Raster element values:

- B1, B2 and B3 relative value of the scanning plates.
- Relation between the field of view in microns and in bits.

All those values are calibrated by Cameca engineers upon installation of the instrument and are for CAMECA engineers only.



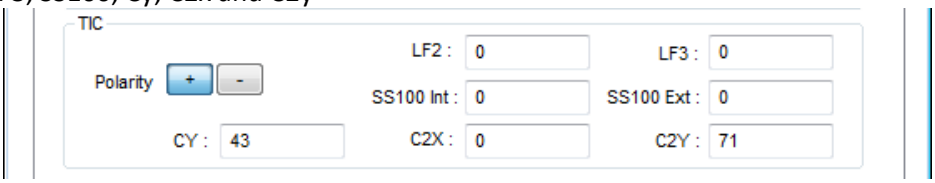
5.9.5.4 LF4 dependency

This function is not available.

5.9.5.5 Total Ion Current

This group is used to define the values of necessary elements to send the secondary beam into the TIC detector:

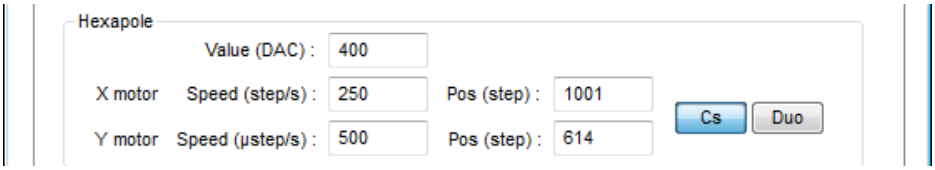
- LF2, LF3, SS100, Cy, C2x and C2y



5.9.5.6 Hexapole

This group is used to define the Hexapole motorization parameters values:

- Hex value
- X and Y motor speed, for each source
- X and Y motor position, for each source



5.9.5.7 E0P Compensation

The E0P compensation is a function that allows to automatically adjust C1 X and Y while adjusting E0P's focus in order to keep the beam at the same position on the sample. This group is used to define E0P compensation relationship parameters:

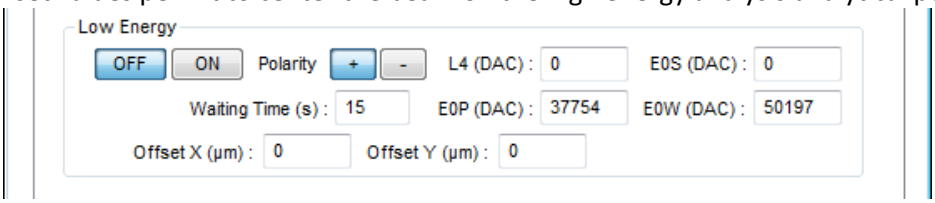
- Dependence coefficient between C1x and E0P
- Dependence coefficient between C1y and E0P

Those coefficients vary depending on the tuning of the primary and secondary beam. It is thus necessary to manually determine those coefficient before using this function.

5.9.5.8 Low Energy

This group is used to define the Low Energy parameters for pre-implantation (or deposition) at ultra-low energy.

- L4, E0S, E0P, E0W values in positive and negative polarity are typically set as follow for a 50eV impact energy: L4=0, E0S= 0, E0P≈37754 to unfocus the beam (for pre-implanting large areas uniformly), E0W=50197, i.e. a +50 V offset from the normal value in the opposite polarity).
- Waiting time (typically 15s)
- The offset values permit to center the beam on the high energy analysis analytical position.



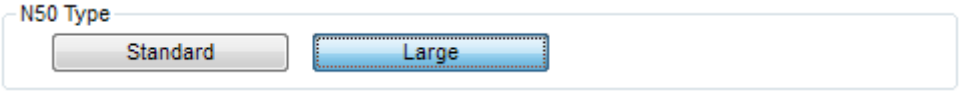
These parameters are defined by Cameca engineers upon installation for 25eV impact energy deposition. See chapter 9.2.8 for details on how to use and adjust the parameters.

5.9.6 Hardware

This page allows the definition of the Hardware functionalities. Those define your instrument's options and should not be altered.

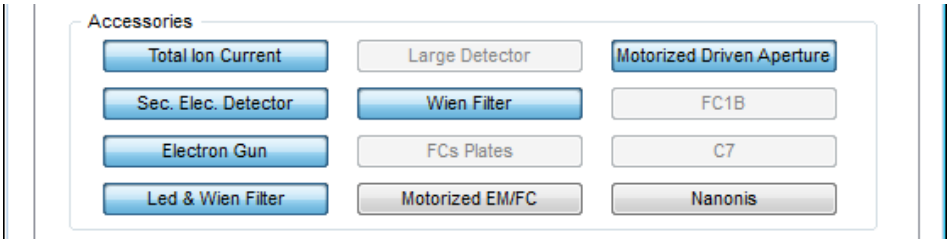
5.9.6.1 N50 type

This group is used to select the NanoSIMS type: standard (NS50) or large (NS50L):



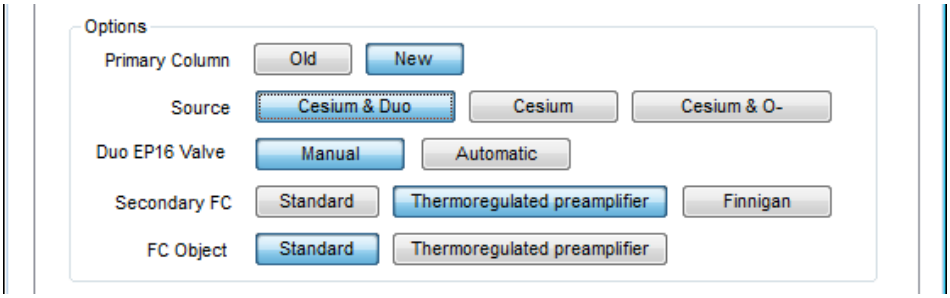
5.9.6.2 Accessories

This group is used to define the available accessories on the instrument. The accessories present on your machine are underlined in blue.



5.9.6.3 Options

This group is used to define the available options on the instrument:



5.9.6.4 Motorizations

This group is used to define the available motorizations on the instrument:

5.9.6.5 Exit Slits

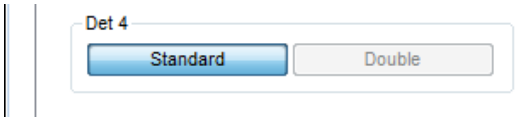
This group is used to define the exit slit set on each detector:

5.9.6.6 Detection

This group is used to define the available detection (EM and/or FC) on each detector, as well as the current pre-amplifier resistor configuration for FCs (10 or 100 GΩ – only for Finnigan FC):

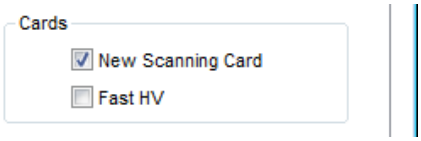
5.9.6.7 Double Det4

This group is used to select the double detector 4, for the instruments which have this option (not available on most recent instruments):



5.9.6.8 New scanning board

This group is used to define the new electronic boards on recent instruments):

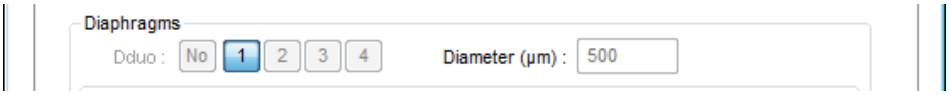


5.9.7 Diaphragms

This page allows the definition of the parameters for the different diaphragms. For each diaphragm set (see below), you can click on the diaphragm position to see its diameters. X and Y motor speeds are also indicated. Those parameters are for CAMECA engineers only.
For each diaphragm position, its X and Y coordinates are also shown. Those parameters are updated each time a position is calibrated ("calib") via the Tuning window (see chapter 5.2.5)

5.9.7.1 Diaphragm Duo

This group was formerly used to define Dduo diameters for each diaphragm position (unavailable on recent instruments)



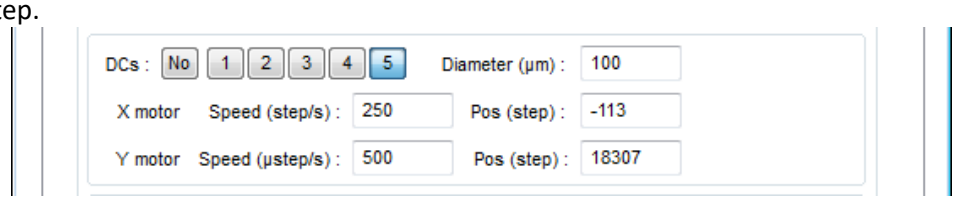
5.9.7.2 Diaphragm D0

This group defines D0 X-Y coordinates and diameter for each diaphragm position (0 to 5), and motor speed.

D0 sizes are as follow: D0-1 = 200µm, D0-2 = 150µm, D0-3 = 100µm, D0-4 = 100µm, D0-5 = 50µm.
Motor speeds are 250 steps/s for X motor and 500 steps/s for Y motor.

5.9.7.3 Diaphragm DCs

This group defines DCs X-Y coordinates and diameter for each diaphragm position (0 to 5), motors speed and motor step.



DCs sizes are: DCs-1 = 200µm, DCs-2 = 200µm, DCs-3 = 150µm, DCs-4 = 100µm, DCs-5 = 100µm.
Motor speeds are 250 steps/s for X motor and 500 steps/s for Y motor.

5.9.7.4 Diaphragm D1

This group defines D1 X-Y coordinates and diameter for each diaphragm position (0 to 5), motors speed and motor step.

D1 sizes are as follow: D1-1 = 750µm, D1-2 = 300µm, D1-3 = 200µm, D1-4 = 150µm, D1-5 = 100µm.
Motor speeds are 250 steps/s for X motor and 500 steps/s for Y motor.

5.9.8 Slits

This page allows the definition of the slit parameters for all slits. For all the slits defined below, slit dimensions (width and height) are fixed and should not be changed, while the positions are updated each time a position is calibrated ("calib") via the Tuning window (see chapter 5.2.5)

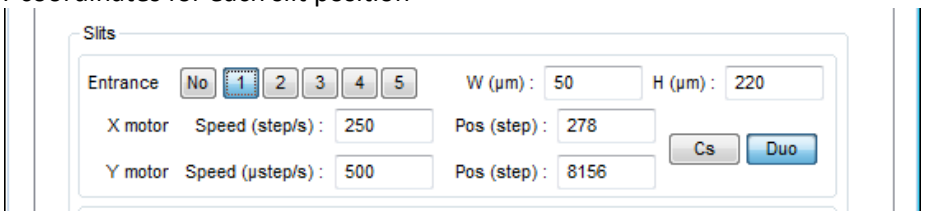
5.9.8.1 Entrance Slit

This group is used to define the parameters values for the Entrance slit:

- width and height for each slit position:

Standard slit sizes are as follow: ES-1: W = 30µm, H = 180µm. ES-2: W = 25µm, H = 160µm, ES-3: W = 20µm, H = 140µm, ES-4: W = 15µm, H = 120µm, ES-5: W = 10µm, H = 100µm

- motors speed (250 steps/s for X motor and 500 steps/s for Y motor).
- X and Y coordinates for each slit position



5.9.8.2 Aperture Slit

This group is used to define the parameters values for the Aperture slit:

- width and height for each slit position:

slit sizes are as follow: AS-1: W = 350µm, H = 250µm. AS-2: W = 200µm, H = 200µm, AS-3: W = 150µm, H = 150µm, AS-4: W = 80µm, H = 80µm, AS-5: W = 40µm, H = 40µm

- motors speed (250 steps/s for X motor and 500 µsteps/s for Y motor).
- X and Y coordinates for each slit position

5.9.8.3 Energy Slit

This group is used to define the parameters for the Energy slit:

- motors speed (250 steps/s for X motor and 500 steps/s for Y motor).
- X and Y coordinates for each slit position

5.9.8.4 Exit Slit

This group is used to define the parameters for the Exit slit for each detector:

- Selection of the slit “slit position”: 1, 2, or 3.
- width and height for each slit position.
- motor speed (400 steps/s) and additional steps (“play”, as calibrated by CAMECA engineers) when the motor moves in an opposite direction.
- It also indicates the type of exit slit set installed for each detector, as defined in the Hardware page of the Setup: Normal, Large or XLarge. This needs to match the setting declared in the Hardware section. By the default, instruments are equipped with the XLarge slit set.

The table below summarizes the width and height of each slit position for each set (Normal, Large, XLarge).

Normal			Large			Xlarge		
position	W	H	position	W	H	position	W	H
1	50	1600	1	80	1800	1	100	2400
2	20	1600	2	50	1800	2	70	2400
3	10	1600	3	25	1800	3	40	2400

5.9.8.4.1 Large Detector (option)

For older models equipped with a large detector, this group defines its slit dimensions

5.9.9 Detectors

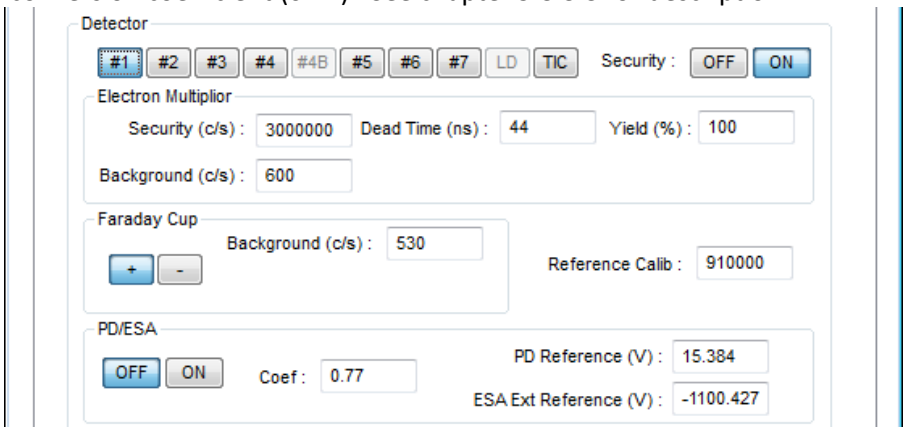
This page is used to define the parameters for the different detectors.

5.9.9.1 Detector

This group is used to define the parameters for the EM and FC:

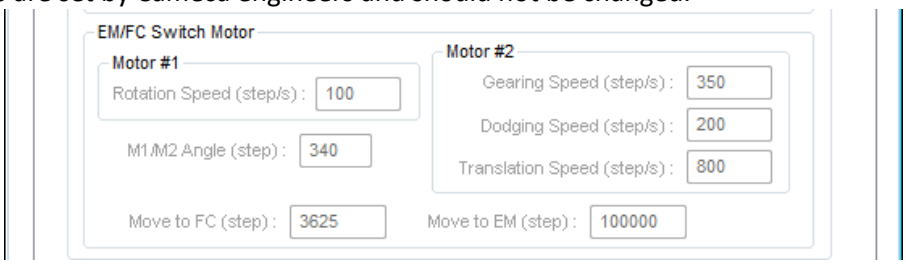
- Security: you can define a maximum signal beyond which the EM shuts down to protect it. It is recommended to set this max value to 2 or 3 M c/s

- dead time (44ns on the NS), yield and background for EM
- Background and calibration for FC (see chapter (see chapter 9.2.13)
- Pd/ESA conversion coefficient (0.77). See chapter 9.3.3.3 for description.



5.9.9.2 EM/FC Switch Motor

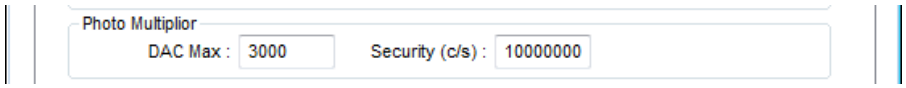
This group is used to define the motor parameters for the EM/FC switch procedure. It defines various movement speed for the two motors involved in the procedure, as well as the step moves for FC and EMs. All those values are set by Cameca engineers and should not be changed.



5.9.9.3 Photo multiplier (of the SE detector)

This group is used to define the parameters for the photo multiplier of the SE detector:

- max photomultiplier HV value (in DAC units) and Max count-rate security (respectively set at 60 000 bits and 10 000 000 c/s). If the count rate exceeds the security count, the HV of the photo-multiplier is turned off.

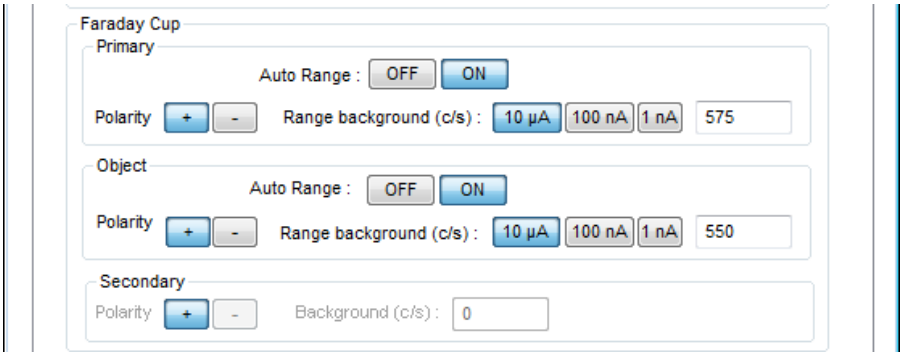


5.9.9.4 Faraday cup

This group is used to define the parameters for FCp and FCo:

- Autorange: when ON, this function allows the FC measurement to automatically select the measurement scale (10 µm, 100 nA, 1 nA) to optimize the reading of the current.
- Range background: for each range of FC preamplifier one can enter a noise threshold, that will be subtracted to the measurement. So 3 values in positive and 3 values in negative. Those values are usually determined upon installation and do not need constant adjustment.

Note that the secondary Faraday Cup (FCs) is no longer available.



5.9.10 B Field

There is only one group on this page dedicated to the parameters of the magnetic field.

5.9.10.1 Waiting time computed

This group is used to define the computed waiting time when a jump in the B-field is asked (either by the operator or the program).
The waiting time (WT) is calculated via the equation:

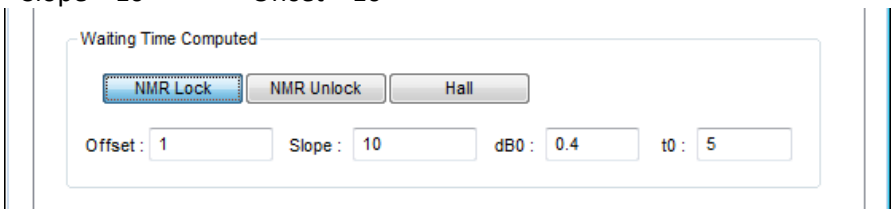
$$WT = 2 * | B2 - B1 | / (B2 + B1) * Slope + Offset$$

With B1 and B2 the two B-field values.
The offset is defined as:

$$Offset = t0 - Slope*dB0$$

Parameters should be defined as follow:

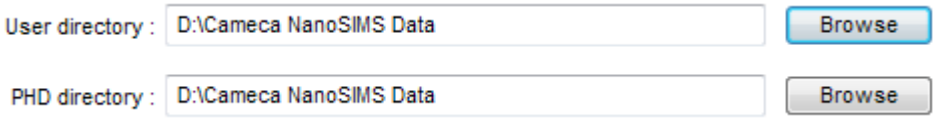
NMR Lock :	Slope = 10	Offset = 1	t0 = 5	dB0 = 0.4
NMR Unlock :	Slope = 10	Offset = 1	t0 = 5	dB0 = 0.4
Hall :	Slope = 10	Offset = 10		



5.9.11 Directories and Misc

5.9.11.1 Directories

This group is used to define the root directories in which all acquired data will be saved. Sub-directories can then be created via Def Analysis (see chapter 5.6).



5.9.11.2 Spy info duration

This group is used to define the number of days information is kept for the SpyEdit software (See chapter 7.5) This is for Cameca personnel only.

5.9.11.3 Numeric format

This group is used to define the numeric format for numeric data saving.

5.9.11.4 Autograin

This group is used to define which software to use for the Autograin function in Grain mode imaging. The standard mode is the CAMECA one. CIW mode links to a customized version requiring an external homemade software.
See 5.7 for description of the Analysis program and 9.2.6.3 for details on how to use the Autograin function in Grain mode imaging.

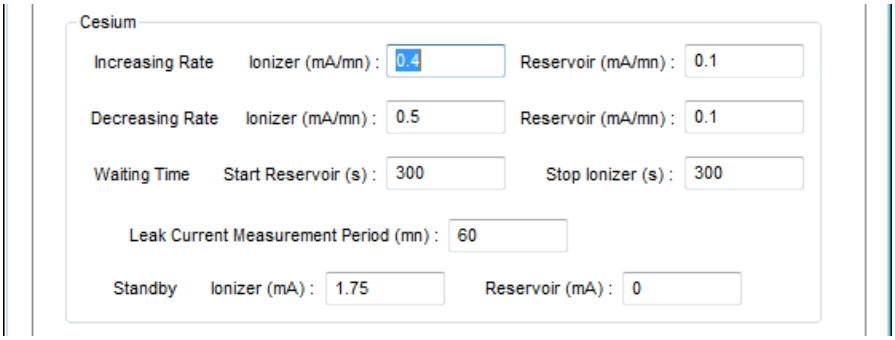
5.9.12 Sources

This page is used to define the parameters settings for the sources.

5.9.12.1 Cesium ion source

This group is used to define the parameters for the Cesium source start and stop procedures:

- Increasing rate for ionizer (0.4 mA/min) and reservoir (0.1 mA/min) for the start procedure.
- Decreasing rate for ionizer (0.5 mA/min) and reservoir (0.1 mA/min) for the stop procedure.
- Waiting time before starting the reservoir during the start procedure (300s) and stopping the ionizer during the stop procedure (300s).
- Leak current measurement period (60 min)
- Standby ionizer and reservoir values (respectively 1.75 mA and 0 mA)

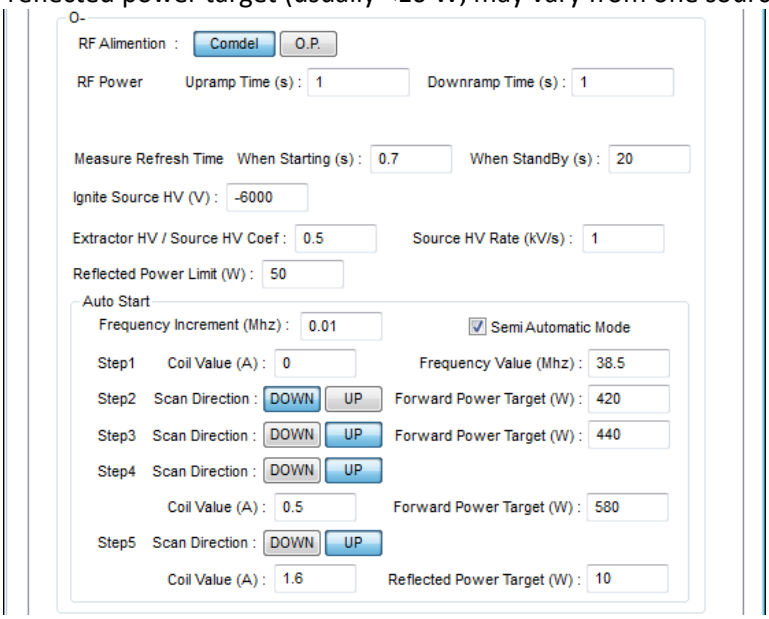


Those are the recommended values for a good use of the Cs source. It is not advised to change them. In particular, speeding the start or stop procedure would only lead to a premature degradation of the source and could lead to an unstable primary beam.

5.9.12.2 RF-plasma ion source

This group is used to define the parameters for the RF-plasma oxygen ion source start and stop:

- RF source power supply manufacturer (initially Comdel, now Oregon Physics)
- Speed of power increments (both up-ramp and down-ramp must be set at 1s)
- Refreshing time when reading the parameters in the source window: when starting and during operations: 0.7s, in standby: 20s.
- The ignition voltage (Ignite source HV = 6000V)
- The ratio between Extractor HV and Source HV (=4000/8000=0.5)
- Source HV rate (1 kV/s) and reflected power limit (100 W)
- Set all the parameters for the starting procedure of the RF source:
    - The semi-automatic mode: when checked, the starting procedure requires user confirmation at each step (see chapter 9.1.2.2 for details on the procedure).
    - Frequency increment: 0.01 MHz and Frequency target at the end of step 1 (38-39 MHz)
    - Frequency scan direction for each step (from 39 to 42 MHz), the program will then look for Frequency resulting in the lowest Reflected power.
    - Coil value applied (0A from step 1 to 3, 0.5A at step 4, then 1.1A at step 5)
    - forward power target at each step (step 2: 460W, step 3: 460W, step 4: 580W)
    - Final reflected power target (usually <10 W, may vary from one source to another)



Those parameters are determined by Cameca engineers upon installation of the instrument (or source upgrade) and should not be changed.

5.9.12.3 Electron gun

This group is used to define one parameter of the Egun: The value set when the e-gun **emission** is turned-off (see Source window description 5.4 and e-gun tuning 9.3.2 for details). This value is usually of 1700 bits.

5.10 Vacuum

The vacuum synoptic program is available on the "Board" by clicking on this button: 

It is the interface that allows the user to communicate with the vacuum automaton. The vacuum automaton surveys the pumping system, controls the pumping/venting sequences, valve opening and HV safeties (refer to chapter 5.10.4). From the vacuum synoptic, the user can monitor the state of the pumps, the valves and read the pressure in the various chambers of the instrument.

5.10.1 Synoptic in "Auto" mode

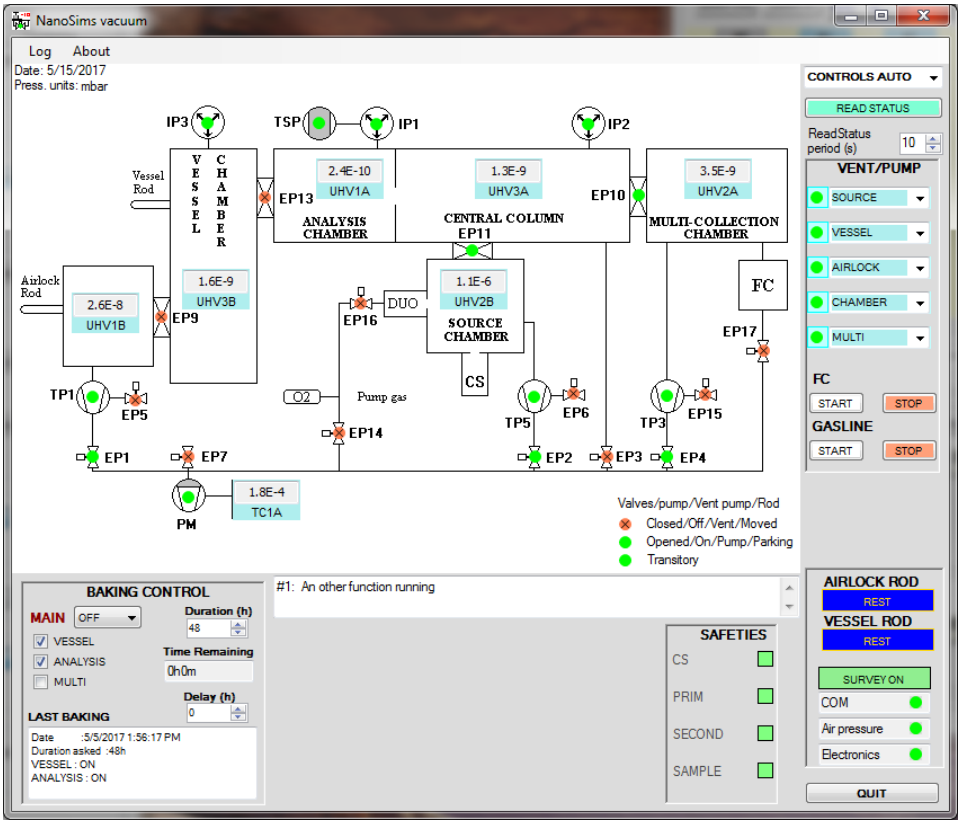
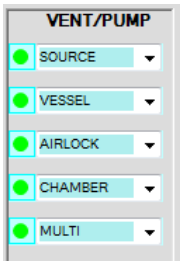
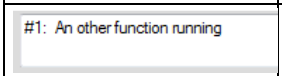
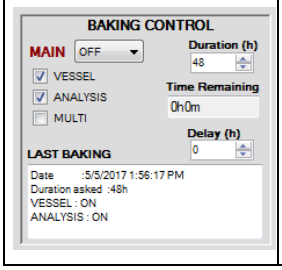
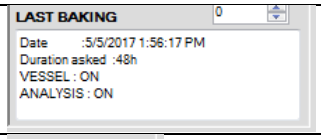
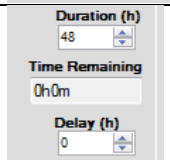
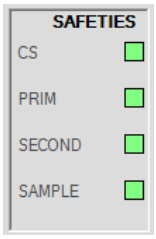
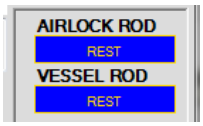
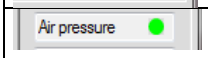
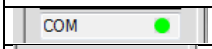
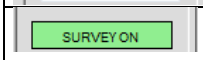
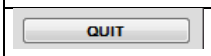


Figure 237: vacuum synoptic in AUTO mode

	Closed or off	
	Open or on	
	Transitory or problem	
READ STATUS ReadStatus period (s) 10	**Turquoise color**: "Read Status" (=between updates) "read status period" (synoptic update interval). Minimum value: 15sec. **Green color**: "reading " The computer reads the NanoSIMS status (vacuum, valves, pumps, HV..)	

CONTROLS AUTO	**3 modes are selectable:** **AUTO Mode** - The automaton survey is ON **MANUAL Mode**

		- Every vacuum component can be switched by clicking with the mouse on the synoptic. - A password will be asked: “ims” **RECORDER Mode** - The vacuum evolution is recorded (see the synoptic “Pressure recorder”)
	VENT/PUMP switch	Clicking on each selector gives access to 3 possibilities - Vent - Pump - Change status (Only in manual mode) **Green Led:** - Pumping is active **Green/orange blinking:** - Repumping or Venting process is running **Orange Led** - Venting is done Change status (only available in manual mode) - Change the pumping status. All vacuum states are recorded in a NVRAM **Note:** For each action, a confirmation message (OK - Cancel) is required. **WARNING: for security reasons, in Auto mode only one process is available at a time e.g. if a load-lock venting is started no other valve can be actuated.**
	START/ STOP	**FC** - If the instrument has a multi-FCs acquisition electronics, this option allows to pump the FC pre-amplifier box (on the top of the multicollection) **Gasline** - Start: primary pumping of the O_2 line between the bottle and the ion source leak valve - Stop: abort the primary pumping sequence **<u>Only 2 states are available</u>** - “Start” green: pumping sequence running - “stop” orange: pumping done
		The last error message is displayed in this panel. This panel is always displayed
	BAKING ON/OFF. Selection of parts to bake.	 **Vessel** (vessel chamber) **Analysis** (analysis chamber) **Multi** (multicollection chamber) All checked chambers will be baked

	Information about the baking	
	Time configuration	 **Duration**: time of baking in hours **Delay**: recorded moments before (in h and mn)
		Status of Cesium, Primary, Secondary and Sample high voltage: **Green**: High voltage is permitted **Red**: High voltage is not permitted (by hardware)
		**Blue** The airlock (or vessel) rod is pulled back in its rest position: its transfer valve can be actuated **Red** The airlock (or vessel) rod is not in its rest position: its transfer valve cannot be actuated
		Electronics ON (green) or OFF (red)
		Green: The compressed air is > 4 bars Red: the compressed air is < 4 bars
		The communication is operational
		The survey (= gauge and valve status reading) is operational
		Quit the synoptic display application. The vacuum automaton keeps running.
		Electrovalves for Nitrogen venting
		Electrovalves to pump or vent specific parts of the NS
		Linear gate valve

5.10.2 Synoptic in “Manual” mode

The manual mode allows the user to stop/start the pumps and gauges, and open/close the valves. This mode overrides the vacuum automaton and **disables all pressure safeties**. It must thus be used with EXTREME CAUTION and only by experimented users. It is therefore primordial to only use the manual mode (accessible via the vacuum synoptic) when absolutely necessary and to switch back and stay in Auto mode the rest of the time.

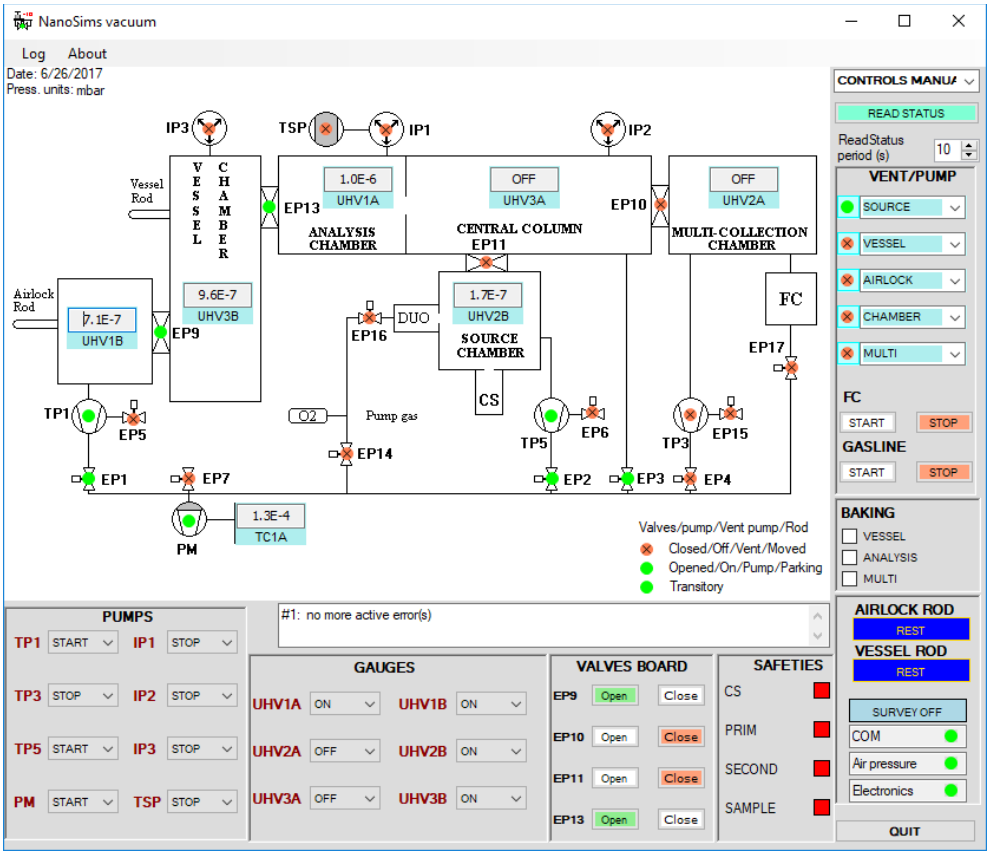


Figure 238: Vacuum synoptic in manual mode

CONTROLS MANUA	Manual control selected
PUMPS TP1 START IP1 STOP TP3 STOP IP2 STOP TP5 START IP3 STOP PM START TSP STOP	**IP ON:** starts the ion pump HV **IP OFF:** Stops the ion pump HV Protection: If the vacuum is too high, the high voltage of the ion pump is stopped automatically
	TP ON: starts the turbomolecular pump **TP OFF:** stops the turbomolecular pump
	Starts or stops the titanium sublimation pump. Note: The intensity, the periodicity and the sublimation time are set directly on the front panel of the Sublimation pump power supply. Refer to the maintenance chapter 10.5
GAUGES UHV1A ON UHV1B ON UHV2A OFF UHV2B ON UHV3A OFF UHV3B ON	**ON:** starts the gauge (will not start until vacuum is below 10^{-4} mbar) **OFF:** stops the gauge

BAKING VESSEL ANALYSIS MULTI	All checked chambers will be baked
VALVES BOARD EP9 Open Close EP10 Open Close EP11 Open Close EP13 Open Close	Close or open the Linear gate valve
SAFETIES CS PRIM SECOND SAMPLE	Vacuum software safeties allowing application of HV on the different parts of the NanoSIMS.

5.10.3 Pressure recorder

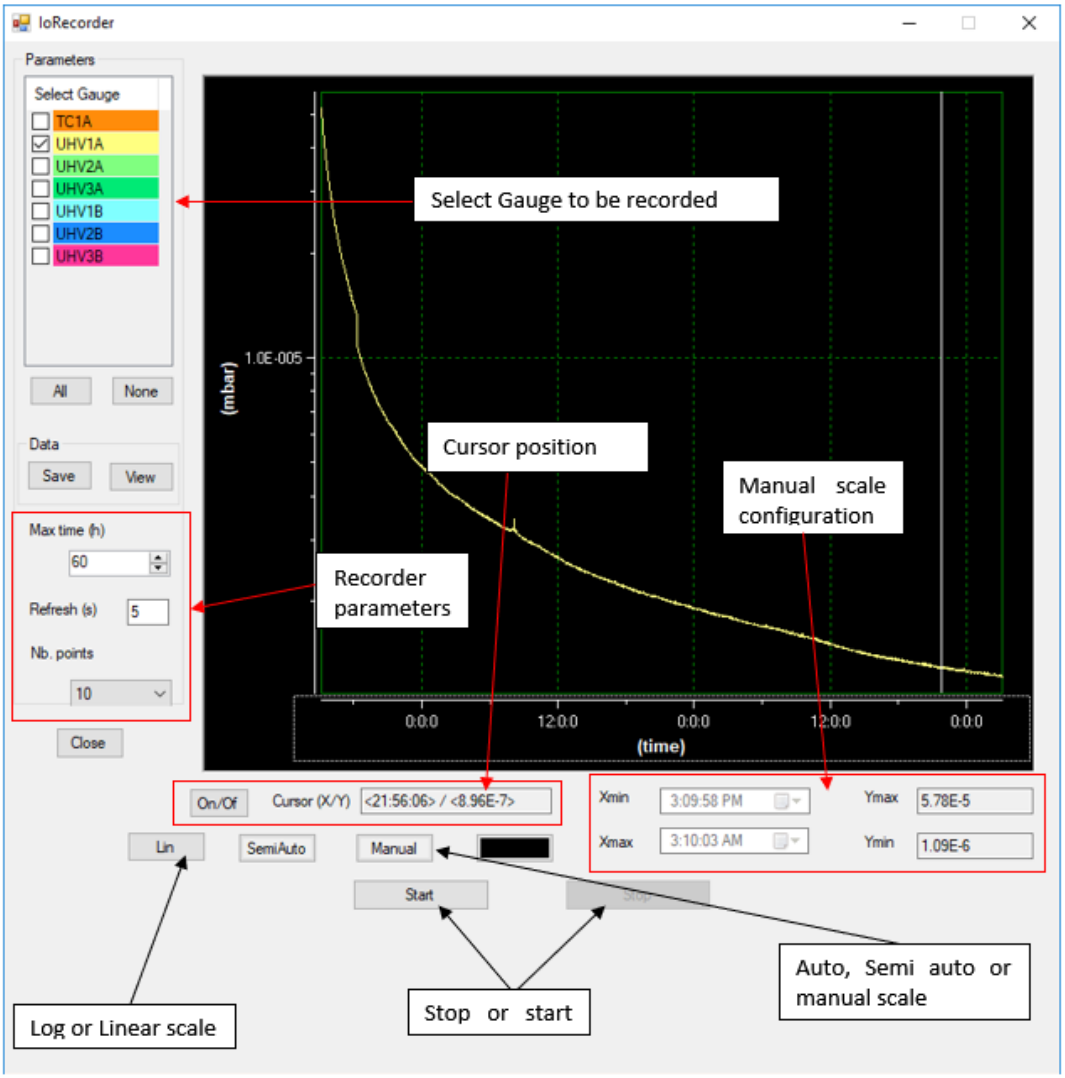


Figure 239: Vacuum pressure recorder

5.10.4 Safeties

Several safeties are in place to insure a good use of the instrument. Most of these safeties are controlled by the Auto mode of the Vacuum Automaton. It is therefore primordial to only use the manual mode (accessible via the vacuum synoptic) when absolutely necessary and to switch back and stay in Auto mode the rest of the time.

5.10.4.1 High voltage safeties

The high voltages in the **primary and secondary columns** (lenses, detectors, e-gun) will apply only if:

- Pressure in the source chamber is < 1.10^{-5} mbar (Cs mode), < 5.10^{-5} mbar (O mode).
- And pressure in the analysis chamber, the central column and the multicollection chamber is < 10^{-6} mbar everywhere.

The **sample** high voltage will apply only if:

- High voltage is applied in the primary and secondary columns.
- And the valve between the analysis chamber and the vessel chamber is closed.

The Cs^+ **source** high voltage will apply only if:

- Pressure in the source chamber is < 1.10^{-5} mbar.
- And the cooling water is running through the red “physics” circuit (a flowmeter wheel is visible on the side of the RF source cabinet).
- The leak current from the source is <0.2 mA

High Voltages can also be manually stopped by clicking on “HV” (light off) on the keyboard. This will stop all HV, except the Cs^+ source, which is controlled by the Source software window, and the RF source, which is controlled independently by the RF source chassis.

The **RF oxygen source** securities are handled independently by the RF source chassis. The RF source will start only if:

- Pressure in the source chamber is < 5.10^{-5} mbar.
- The power supply of the RF source is on and in “enable” position.
- There is no polarity switch in progress.
- There is galden in the reservoir.
- The galden flow is sufficient (measured as 1.1 V on analog flowmeter).
- The galden temperature < 55°C.
- The oxygen pressure in > 0.5 bars.

5.10.4.2 Valves safeties

Two valves are constantly monitored by the vacuum Automaton (in AUTO mode only!):

EP11 (between the source and the central column). It will **open only if:**

- The “source” switch on the airlock valve control box is in “open” position

AND

- Pressure in the source chamber < 1.10-5 mbar (Cs mode), < 5.10-5 mbar (O mode), pressure in the central column is < 10-6 mbar, and the pumping system is on.
- OR if the pumping system is off, and both the source chamber and central column are vented.

EP10 (between central column and multicollection chamber). It will **open only if:**

- The “multi” switch on the airlock valve control box in in “open” position

AND

- Pressure in the central column and in the multicollection is < 10-6 mbar, and the pumping system in on.
- OR if the pumping system is off and both the central column and the multicollection chamber are vented.

If any of these conditions are not met, the valve will **automatically close**, to protect the source, analysis or multicollection.

The vacuum automaton also controls the opening of the valves between the airlock and the vessel chamber, and between the vessel chamber and the analysis chamber. Those valves are actioned via the airlock valve control box:

EP9 (between airlock and vessel chamber). It will **open only if:**

- The airlock-vessel transfer rod is in parking position.

AND

- valve EP13 (between vessel and analysis chamber) is closed.

AND

- Airlock and vessel chamber are both vented and pumping system is off.
- OR pressure in the airlock is < 5.10-6 mbar and pressure in the vessel chamber is < 1.10-6 mbar, with the pumping system on.

EP13 (between vessel chamber and analysis chamber). It will **open only if:**

- The vessel-analysis transfer rod is in parking position.

AND

- valve EP9 (between airlock and vessel chamber) is closed.

AND

- Vessel and analysis chambers are both vented and the pumping system is off.
- OR pressure in the vessel chamber is < 10-7 mbar and pressure in the analysis chamber is < 10-8 mbar.

EP9 and EP13 will close only if the rods are properly in their parking position.

5.10.4.3 Pump safeties

Pumping and venting sequences are handled by the vacuum automaton. However, the pump safeties are defined directly by the pump controllers. In “protect” mode, the ion pumps will stop if the pressure increases above 2.10^{-5} mbar. Turbo pumps do not have pressure safeties.

5.11 NMR

5.11.1 Introduction

Working at high mass resolution with small mass line widths, the stability of the magnetic field of the Mattauch Herzog-like NS50 mass analyzer is crucial. It is first governed by the stability of the current flown in its coils. Both the power supply and the magnet are water cooled. This is a first crucial reason to require a stable water chiller.

The B-field is then regulated by a feedback loop made by a Hall probe inserted inside the gap of the magnet, measuring the B-field and regulating the current power supply (up to 0.35 Tesla). The bandwidth of the Hall probe is large enough to allow magnetic peak jumps between different magnetic B-fields when working in monocollection or hybrid modes. But this flexibility limits the ultimate B-field stability.

Hence, a third feedback can be given by an NMR probe, keeping the magnetic field more stable in the long term than the Hall probe but is much slower and thus does not allow peak jumps. The NMR is used ONLY in multicollection***, on request*** from the operator. Two NMR probes are inserted inside the gap of the magnet connected to the same electronic rack. The two probes cover different ranges and switch automatically.
In option one can purchase a third NMR probe called “NMR H/D” with even better stability for the low magnetic field required for precise hydrogen/deuterium ratios (i.e. when 7th detector mass less than 23Na).

- Standard NMR Bfield range: 0.09 to 0.26 Tesla (probe 2) and 0.17 to 0.52 (probe 3)
- H/D NMR Bfield range: 0.043 to 0.13 Tesla. (probe 1, optional)

The NMR regulation is particularly useful during hour-long analyses where the magnetic field is otherwise likely to drift. The following chapter describes the main windows and capabilities of this software.

5.11.2 Reduced Panel Display.

Figure 240 shows the reduced display of the NMR program window. A click on the “NMR” button opens more controls.

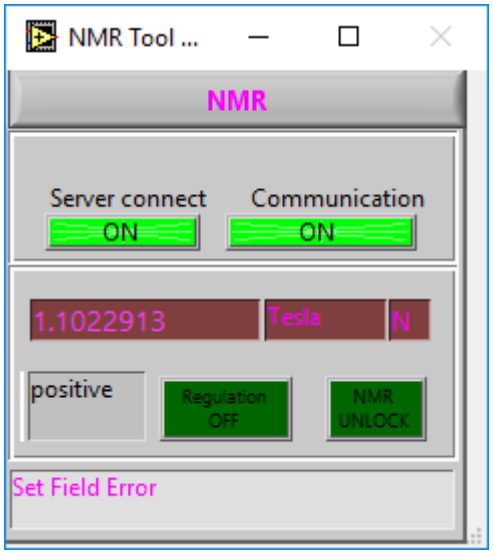


Figure 240: reduced display of the NMR window

Server connect: Status of the interface connection with the server. This status has to be green in order to send field commands or to read the Bfield value.
Communication: Status of the Communication line (RS232) controlling the NMR Teslameter.

1.1022913 Tesla N: NMR Field Reading in Tesla or Gauss

- N: Status of the Teslameter - N= not Locked, S = NMR Signal, W = Wrong reading, L = Locked (Reading OK).

Polarity Positive: Displays the Instrument Polarity

Regulation OFF: Status of the Regulation Process, if Regulation = OFF the field is under the Hall probe control only.

NMR UNLOCK (or LOCK): Status of the Teslameter for a New field value. It is Blinking during the field setup, then turns to LOCK = reading OK ; or UNLOCK = reading not OK. Typically, after a modification of B-field it will take a few second for the NMR to re-LOCK.

"Set Field Error": Message from the NMR Interface.

When the NMR program opens, communication with the NMR electronics will be established. The buttons "Sever connect" and "communication" will then appear "ON" in bright green (Figure 240). If one or both appear dark green (OFF), quit the NMR program, and restart the NMR electronics via the electronics cabinet front panel (Figure 241). Click on the black switch to turn off the NMR, wait a few seconds, then click again to re-start it.

Figure 241: NMR electronics panel

When opening the NMR program, it will also attempt to "lock" the NMR (bottom right corner button). However, it will automatically unlock when the user changes the magnetic field. If it does not relock by itself when the user stops adjusting the magnetic field, reset the NMR.

Start the NMR regulation : If Server connect, Communication and NMR lock are all bright green, then you can launch an NMR regulation to prevent your magnetic field to drift. To launch an NMR regulation, go to the Tuning window and put the NMR regulation ON (Figure 242). It is advised to check the centering of your mass peaks via an HMR scan before launching an analysis as the regulation process might have slightly affected the magnetic field.

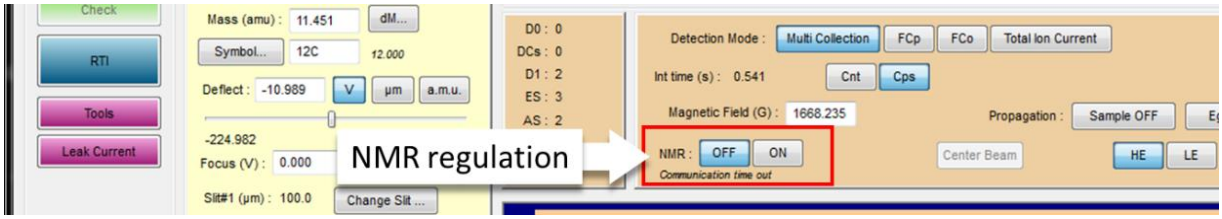
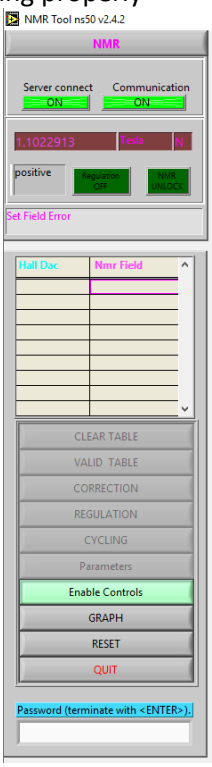


Figure 242: set the NMR regulation via the Tuning window

Note that it is recommended to put the regulation back OFF before changing the magnetic field value. Otherwise, after a modification of B-field it will take typically a few second for the NMR to re-LOCK.

5.11.3 Full Control Panel.

Click on the Pink “NMR” button on top of the window to display the full menu. New options appear, which can be useful for when the NMR is not working properly



All greyed options are for Cameca engineers only and require a password to access them. They are used to calibrate the NMR. Changing the calibration of the NMR is very risky and it is recommended not to use those options without a Cameca engineer.

GRAPH: Opens A Graph that displays Field Measurements and Statistics Computation. (see below)
RESET: Reinitialize the Interface. When communication issues appear with the NMR, a simple reset can sometimes restore the communication and solve the issue.
QUIT: quit the NMR program.

5.11.4 Recording Field values with the Teslameter

It is possible to record the variations of the magnetic field with the Teslameter on (Figure 243). This can be useful to check the magnet natural drift (record a graph with the regulation OFF), or the proper regulation of the NMR (record a graph with the regulation ON).

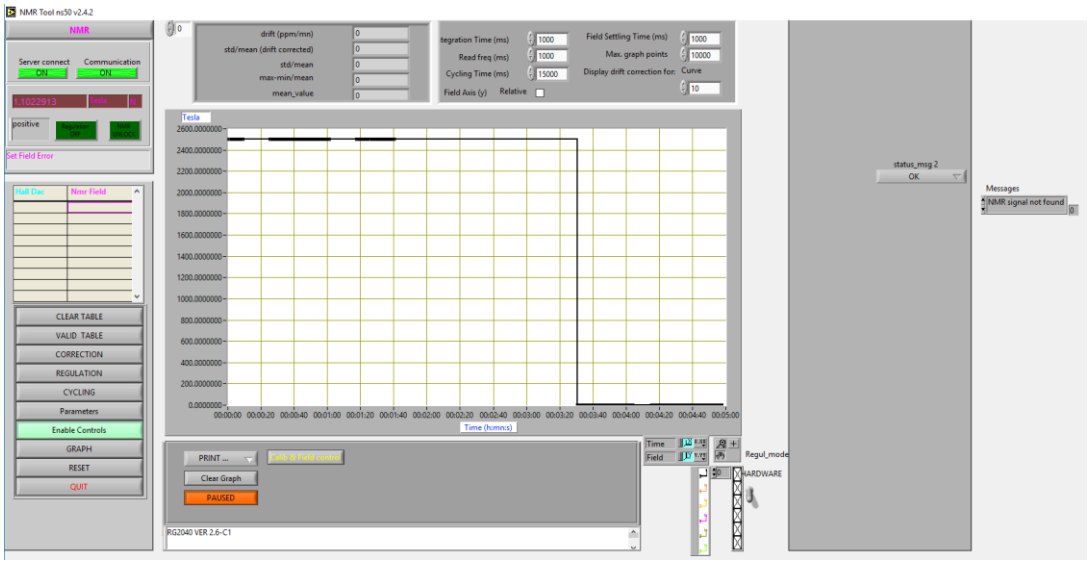


Figure 243: recorder window of the NMR program

Recording Field values with the Teslameter:

- Select: GRAPH, Release the PAUSE button, the field value is now recorded and statistics are computed for all data displayed in the graph.
- Starting Regulating the Field with the NMR:
- Press 'Enable Controls', enter the password (nano50)
- Press 'REGULATION'
- The Regulation process starts by setting the Teslameter in order to take an accurate measurement of the field, then starts the actual regulation process.
- This process will take 30 seconds the first time the regulation starts upon a reset, or Interface open. The next regulation process will take maximum 12seconds.
- If the 'VALID TABLE' button is pressed the Regulation will be performed on the NMR value stored in the 'Hall_Dac/NMR_Field' Table, If no NMR Field value corresponding to the current Hall DAC is stored in the Table, a new reading is taken.

To stop the NMR Regulation: Release the 'REGULATION' Button.

5.11.5 Restoring NMR communication

When the communication fails between the NMR and the software, it is necessary to reset the NMR:

- If the regulation was on, put it off in Tuning.
- Quit the NMR software.
- Restart the NMR electronics via the electronics cabinet front panel (Figure 241) by clicking on the black switch.
- Wait a few seconds, then click again to re-start the NMR electronics.
- Restart the NMR software. Wait for "server connect" and "communication" to turn ON (bright green)
- Put the regulation back ON if you wish to.

6 NS50L Software: The “TOOL” taskbar

6.1 Param

This program is extremely useful for troubleshooting and localizing a problem with an electronic component, a leak or a short circuit between cables, feedthroughs or optics.
For each parameter, the program displays (in the ***Measure*** column, Figure 244) the voltage measured directly at the output of the electronic board sending the voltage to the component (lens, deflector, etc…) and compares them to the programmed voltage (***Applied*** column in Figure 244). A difference (***Diff***) is then calculated:

- A difference of 1% is acceptable, due to the inferior precision of the DAC feedback measurement compared to the command.
- A different > 5% usually indicates a problem in the voltage supply, the inter-connexion, cabling, plugs, or the component’s failure.
- A difference of 200% indicates a reversed polarity, which often happens during a polarity switch. To solve this issue, switch again the polarities.
- Note that the coils are not monitored and the difference calculated is meaningless.

To check all voltages are properly working, open the Param program and click on “Update” to launch a scan of all parameters. Click on “update” again whenever you want to refresh the reading.
If you wish to keep a record, you can save the table in a text format by clicking on “Save”.

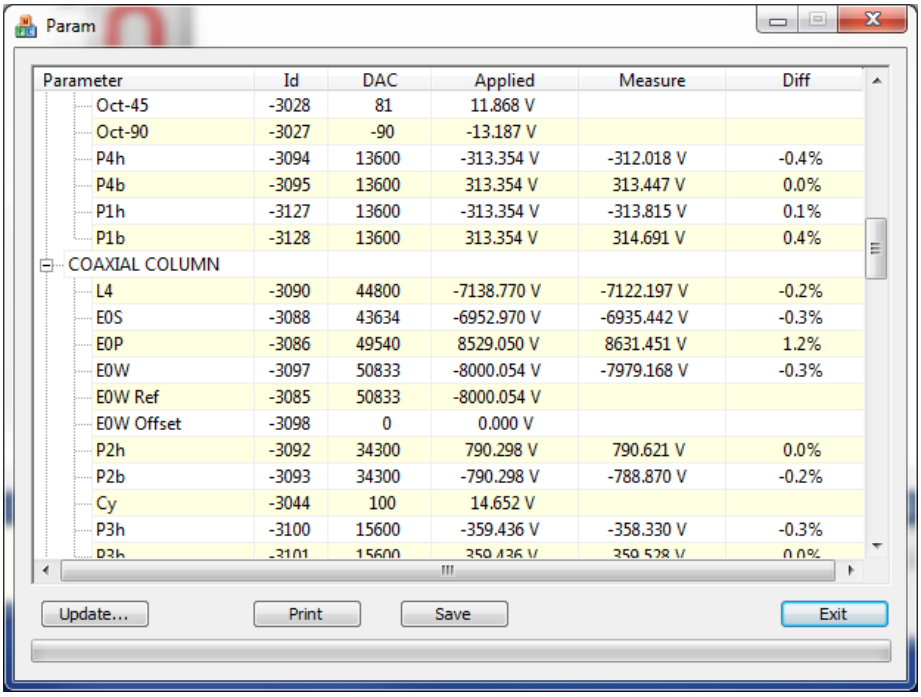


Figure 244: "Param" window

6.2 Point logger

6.2.1 Introduction

Point Logger is a navigation software allowing **navigation using an imported image** instead of the optical microscope. This feature can be extremely useful for example to navigate over large distances inside a holder hole or on a coated sample resulting in low contrast in the optical image. It can facilitate correlative microscopy between different instruments.

The imported point logger image can be a simple scanned image of the full sample holder placed on a photocopier machine or a SE/BSE image from an electron microscope, or a fluorescence or other optical microscopy image.
The Cameca Point logger program accepts external images of JPG format only.

The key point is to use an external image with as low X-Y distortion as possible (a square should be square, lines must be straight, X-Y axis should be orthogonal). And its pixelization should be high as it will contribute also to the ultimate precision of the coordinate transfer.

The principle is:
a) to note two sharp details (natural or *intentionally made*: deposited FIB marks, scratching, writing, etc…) in the imported image, with large X-Y space between them,
b) move the sample in order to bring these 2 points successively in the SIMS analysis (or CCD optical microscope) center position and record these two points in the X-Y stage coordinate reference
c) run a routine of transfer of X-Y referential (alignment procedure).
d) the Navigator program will be then able to use the imported image for navigation: when clicking on a point in the imported image the stage will move to it.
The NS CCD optical microscope image can be used instead of the SIMS image. Just make sure that CCD and SIMS positions are well aligned (see chapter 9.2.9 on CCD/SIMS adjustment).
Algorithms used for the reference system transfer are given below in chapter 6.2.5.

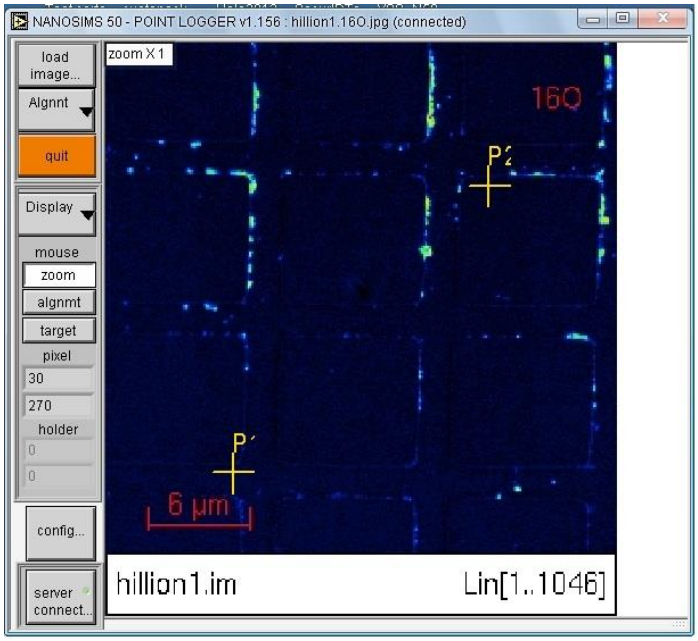
6.2.2 Getting started

6.2.2.1 Automatic connection to NS50 Holder

Click on “pointlogger” button in order to launch the Point Logger program. It opens the last validated alignment (unless the alignment file has been moved or deleted meanwhile; in such case a message indicates that last validated alignment file failed).
An “alignment” means both an imported image and the set of points on which the alignment is based. If there is no previous picture nor alignment point, the window appears blank.

6.2.2.2 Loading an external image

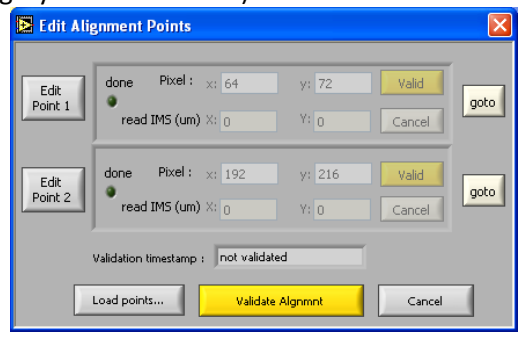
On the main toolbar (inner left side of the NS50 pointlogger window), click on the “load image…” button, then select the external image to load.
The NS50 PointLogger accepts only **jpeg** format (jpg suffix).



Picture loaded

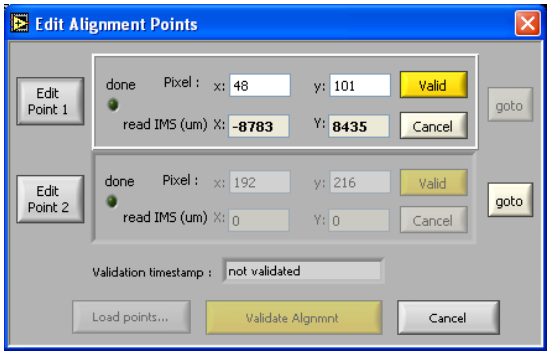
6.2.2.3 Input of two points of alignment

Move the stage to bring the sample to the first point of reference at the center of the SIMS (or optical microscope) image.
On the main toolbar (inner left side of the NS50 pointlogger window), click on the "**Algnnt**" mouse mode button. Then choose the item labeled "Open…". The dialog box as seen below appears (in case no picture is loaded, item "Open…" looks grey and invalidated).



Alignment point set dialog box

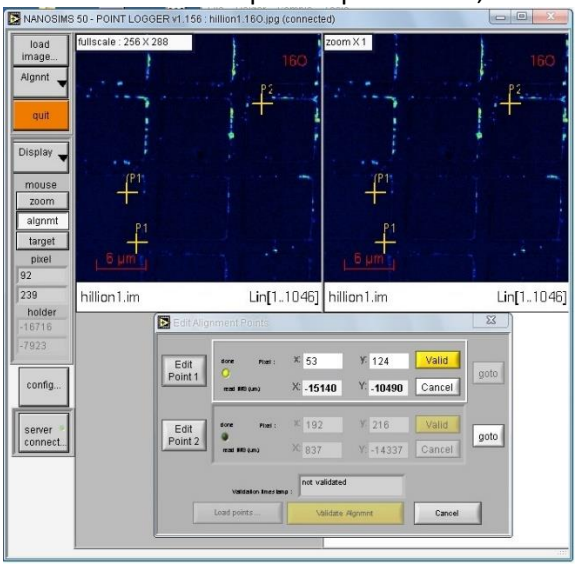
Input of the first point:
Click on "Edit Point 1" Button. The **current** sample stage coordinates are then sampled and displayed in µm in the "read IMS" fields.



Input of the first point

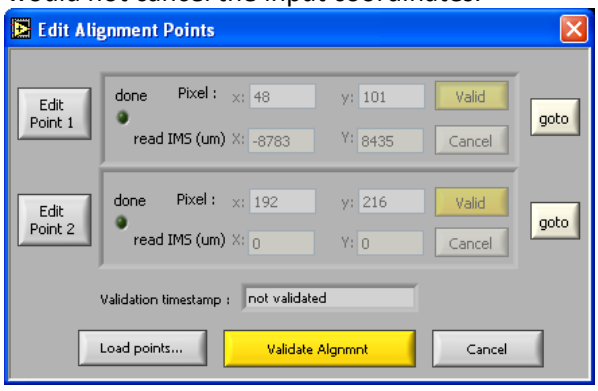
The user must now indicate with the mouse the corresponding point in the picture matching the current stage position
A label "P1" between parenthesis is labeled besides the yellow cross-shaped pointer. The small red diode "done" is now blinking. The user is free to click as many times as necessary before point validation to refine location. Zooming/de-zooming is available (button on left side of the PL window).
Once the user considers the point to be correct vs the stage position, the user valids the point by clicking the button "Valid". The LED "done" switches to GREEN.

Input of the second point:
Move the stage to bring the center of the SIMS (or CCD optical microscope) image to the second point of reference selected on the external image.
The process is identical to those used for the first point input. Use P2, Edit Point 2, Valid.



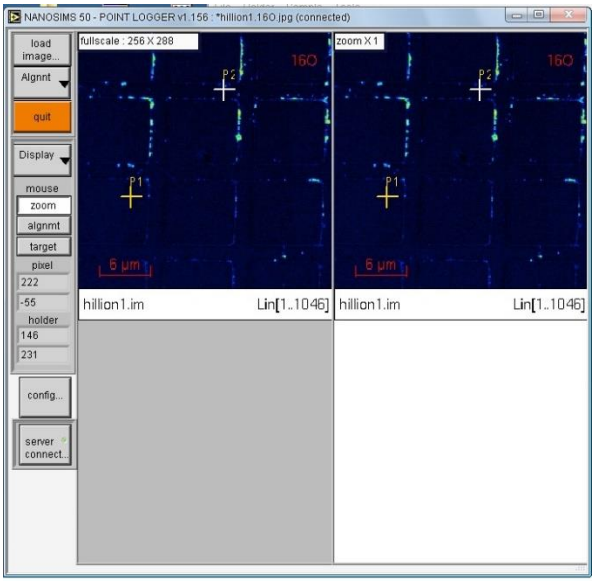
Input of the P1 pixel coordinates on imported image

The user will eventually valid the set of two points with the button "Validate Algnmnt". Closing the Edit Alignment Points dialog box would not cancel the input coordinates.



Alignment front panel before final validation

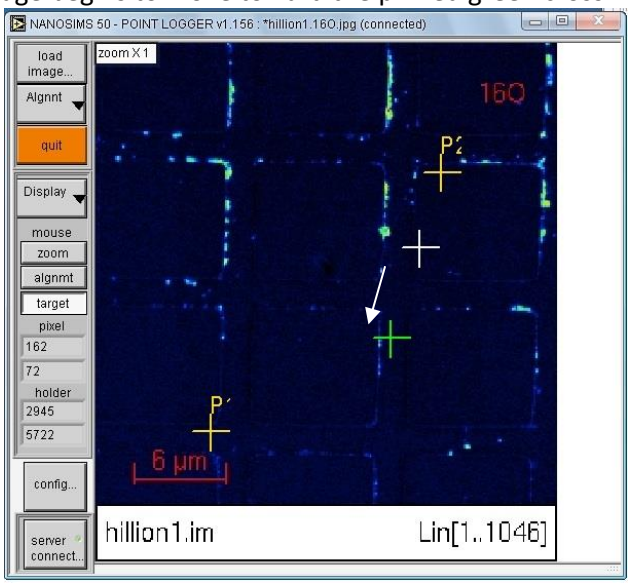
As soon as alignment has been done, the current **STAGE POSITION** is periodically sampled and displayed superimposed over the external image, symbolized by A **WHITE CROSS**-shaped pointer.
The related coordinates are displayed in the fields labeled "holder" on the left side vertical toolbar.



The external image with stage position (white cross) superimposed after alignment processed

6.2.2.4 Driving the stage by clicking in the imported image

From that point on, the user can select the "**TARGET"** mouse mode (left side of PL window) and click in the imported image to drive the stage:
First, select the **TARGET** mouse mode by clicking the button "target". Click on a point of interest of the picture. A **GREEN CROSS**-shaped pointer appears on the point you clicked. That **GREEN CROSS** pins the target. Immediately, the stage begins to move toward the pinned green cross.



Defining a target

6.2.3 Pointlogger in more detail

6.2.3.1 Pointlogger Window

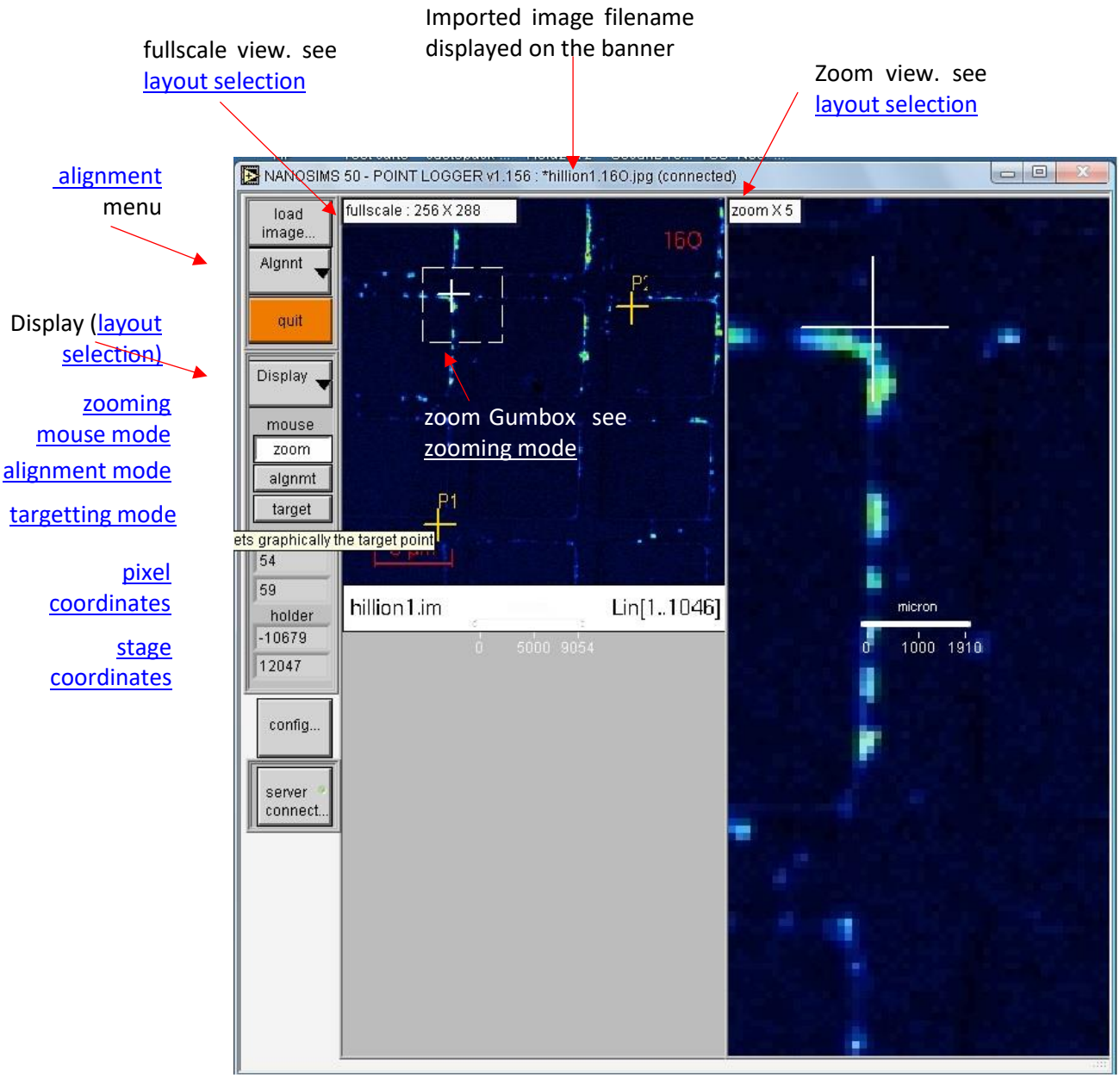


Figure 245: Main point logger window

The pointlogger main window is resizable. Size and position on the screen are automatically saved in setup file and reset at the next launch.

Pixel and stage coordinates:
Current mouse position expressed in pixel and simultaneously in holder coordinates is displayed in frames on lower part of the toolbar.
Before an alignment is performed or with no connection to the holder, the holder coordinates display is inactive (field in grey). After an alignment and the holder being connected, the holder coordinates display are activated.
Pixel coordinate referential: pixel coordinates are given relatively to a referential centered on the **lower left** and whose axes are oriented **rightwards and upwards.** Coordinates are expressed in pixels.

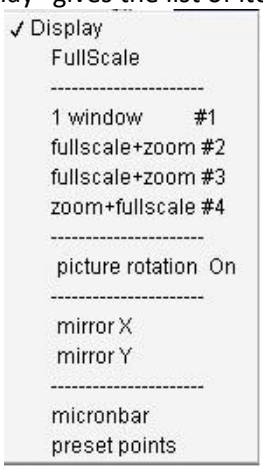
Stage coordinate referential: stage coordinates are given relatively to holder motorized axes coordinates and are expressed in micrometers.

6.2.3.2 Load Image

The button "Load Image…" permits to load an external image that will be used to drive the stage
The default path is retrieved from NS50 setup file.
The sole image format is jpeg.
As the loading might last some time in case of high pixelization, a progress bar indicates the completion of the loading.
The image filename is displayed on the upper banner of the PL window.

6.2.3.3 Display menu

Clicking on popdown button labeled "Display" gives the list of items shown below:



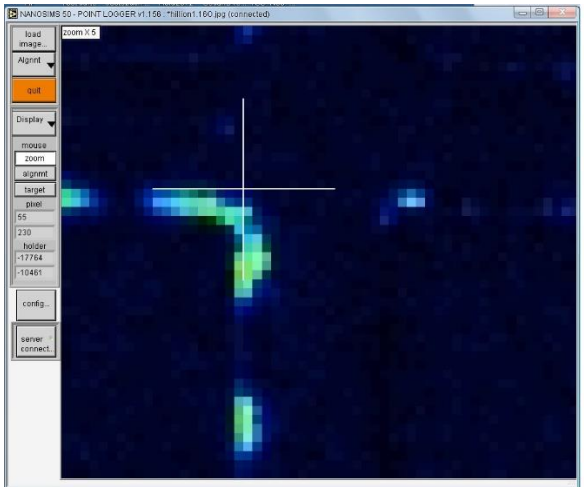
Display popdown button menu

6.2.3.3.1 Four possible window layouts

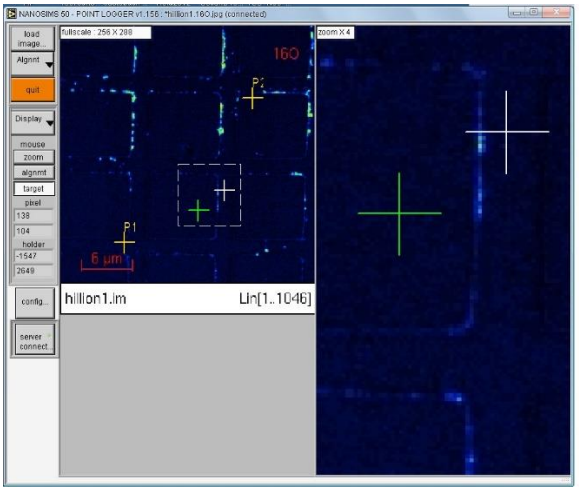
Pointlogger allows the user to display a fullscale picture and a **zoomed view** side by side.

layout #1 : 1 window : full view or zoomed view
layout #2 : 1 fullscale + zoom : 1 fullscale view and 1 zoom view side by side.
layout #3 : 1 fullscale + zoom : 1 large fullscale view and 1 small zoomed view.
layout #4 : 1 zoom + fullscale : 1 small fullscale view and 1 large zoomed view.

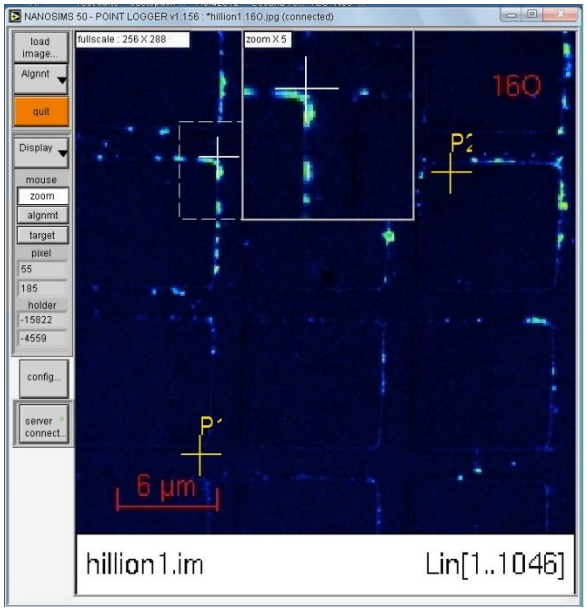
The default layout is loaded at pointlogger initialization. To change the default layout, see PointLogger configuration below.
The picture own scaling is taken unchanged (i.e the picture appearance is not altered due to screen resolution). In another terms, a pixel keeps its squared shape, even at a high magnification.



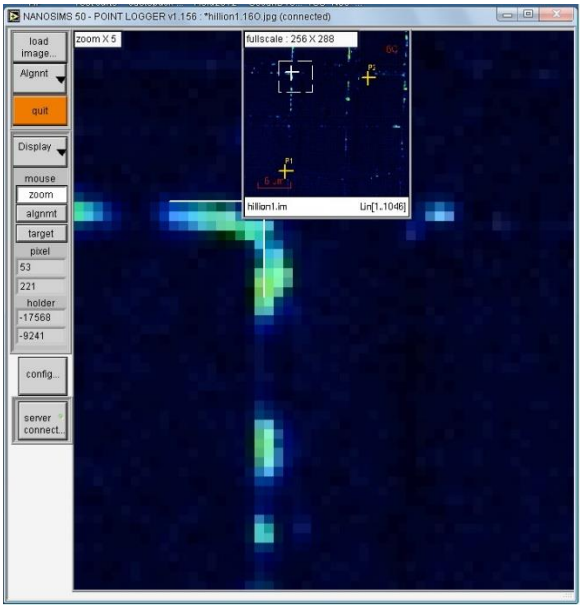
Layout #1 : 1 single view (here : zoomed view)



Layout #2 : 1 fullscale view + 1 zoomed view with the same size



Layout #3 : 1 large fullscale view + 1 small zoom view



Layout #4 : 1 small fullscale view + 1 large zoom view

6.2.3.3.2 Rotation

A rotation may be applied to align the whole picture in respect with the sample holder axis.
The rotation is computed using the algorithms used for conversion (APPENDIX 1 : coordinates conversion formulae). As a consequence, it requires a point alignment to be performed before (APPENDIX 2 : ROTATION formula).

To point out the principle of a rotation, select the alignment points so that the 2 points will have an equal holder value. The rotation rotates the picture so that the 2 points P1 and P2 appear horizontal.

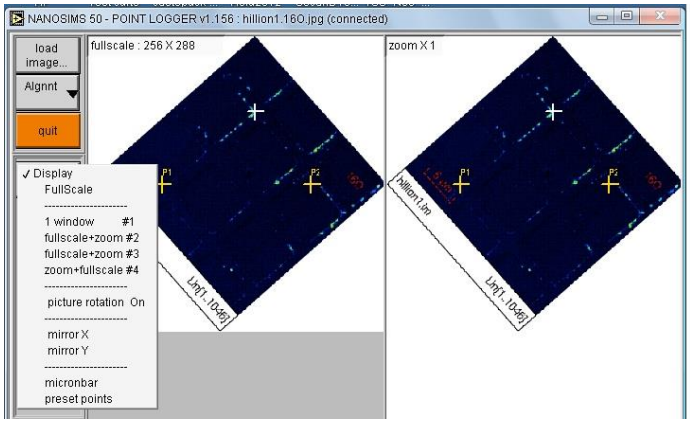


Figure 246 : rotation

Once a rotation is made, the other functions: mirrorX, mirrorY, zoom can be carried out as for non rotated picture.
The rotation can be done, whatever the functions done before are. For instance the zoom area is kept unchanged.

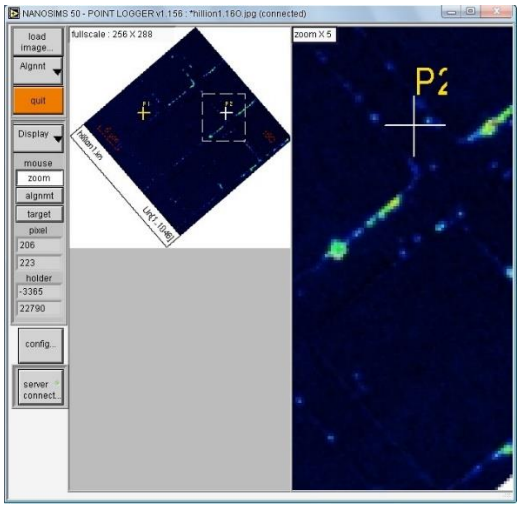


Figure 247 : zoom processed after a rotation

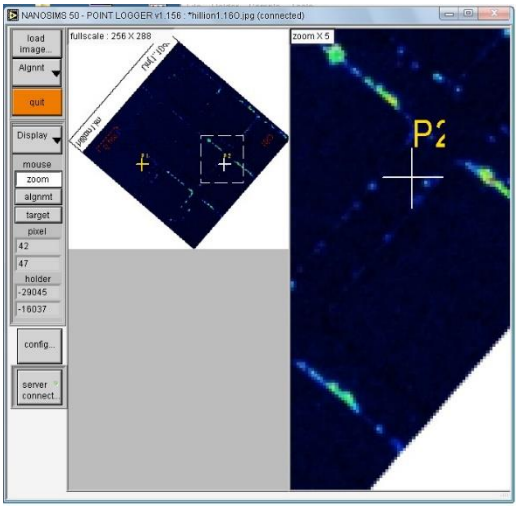


Figure 248 : mirrorX processed after a rotation

6.2.3.3.3 mirrorX

Enables picture to be flipped symmetrically to a horizontal axis.

6.2.3.3.4 mirrorY

Enables picture to be flipped symmetrically to a vertical axis.

6.2.3.3.5 micronBar

This function displays the pixel bar if no alignment is done, or the micron bar otherwise. The micron bar display status is kept in setup to be applied at each program start. The micron bar object can be moved by drag and drop mouse process.

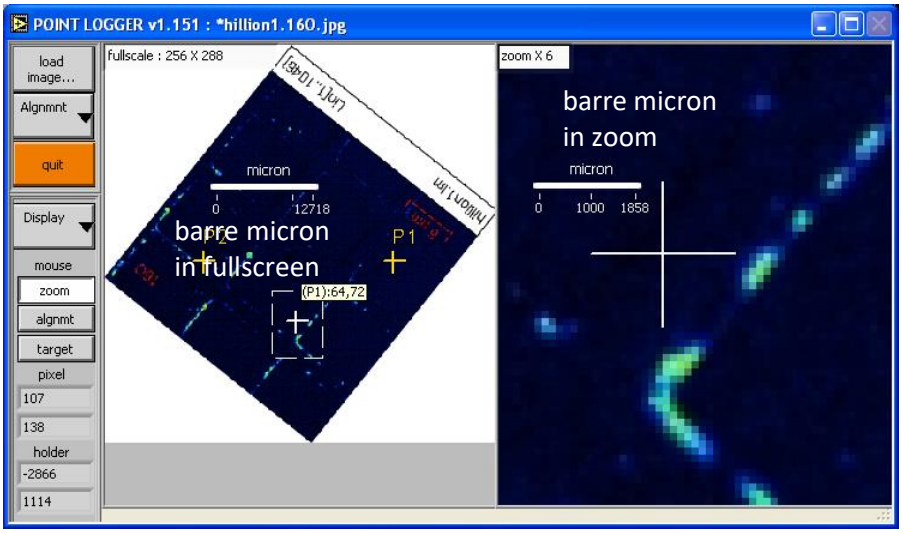
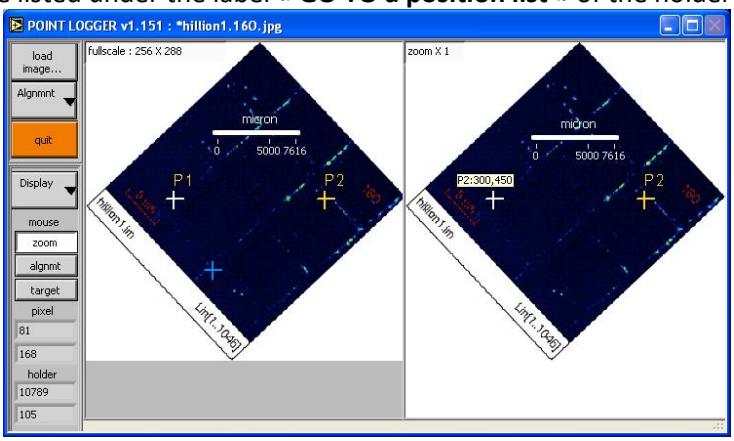


Figure 249 : display of a micronbar

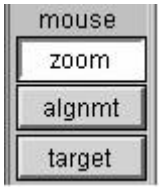
6.2.3.3.6 Preset Points as BLUE crosses

PRESET POINTS are displayed as **BLUE CROSSES**. They are only displayed on full screen view. The displayed preset points are those listed under the label « **GO TO a position list** » of the holder front panel.



display of preset points

6.2.3.4 Mouse mode selection



A set of 3 buttons are dedicated to mouse assignment.

- a button labeled "zoom" assigns the mouse to defining a zoomed view.
- a button labeled "algnmt" assigns the mouse to defining the picture coordinates during an alignment process.
- a button labeled "target" assigns mouse to defining a target for stage moving.

6.2.3.4.1 Mouse zooming mode

This mode allows shaping a gumbox to define a zoom view. Opening a gumbox consists of hitting the left mouse button, dragging while keeping the left button down and releasing it up.
The zoom factor is labeled on the upper left coin of the zoomed view.

Note 1 : except for layout # 1 ("1 single view"), zooming can be made only on "fullscale" view.
Note 2 : zoomed view is cancelled with function "FullScale" in the "Display" menu.

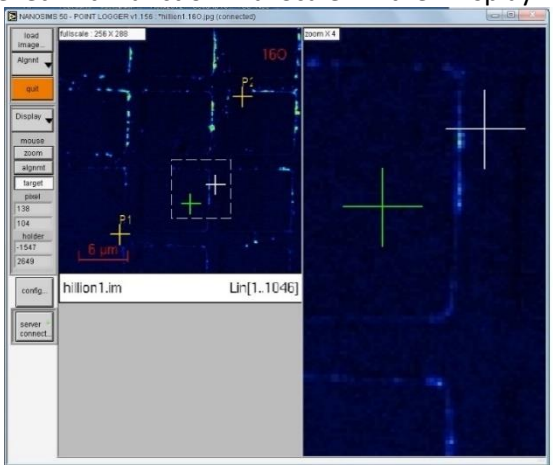


Figure 250 : zoom procedure

The white dotted rectangle isolates the zoomed section. If the user selects a section too small, the zooming procedure is cancelled.

6.2.3.4.2 Mouse Alignment mode

Pointlogger automatically switches to the editing alignment mode whenever Alignment dialog box is shown. The mouse in editing alignment mode is used to sample the 2 alignment points.
Note: During the alignment process, the user is free to switch to zooming mode to change viewing conditions.

6.2.3.4.3 Mouse Target mode

In target mode, any mouse click on picture sets off a stage motion. Target mode gets enabled only if alignment is validated and holder connected. For a detailed description see moving the stage

6.2.3.5 Cross pointers

Overal there are 4 different cross-shaped pointers:

6.2.3.5.1 PRESET Points as BLUE crosses

PRESET POINTS are displayed as **BLUE CROSSES**. They are only displayed on full screen view. The displayed preset points are those listed under the label « **GO TO a position list** » of the holder front panel.

6.2.3.5.2 The 2 alignment point YELLOW cross-pointers

Those **YELLOW** crosses are labeled "P1" and "P2". During an alignment process, the point under editing sees its **label** lead and trailed by **parenthesis,** for instance "P1" to "(P1)"

6.2.3.5.3 The current STAGE position WHITE cross pointer

Displayed in **WHITE**, this cross-shaped pointer is displayed as soon as an alignment is validated. This cross-shaped pointer is not labeled.

6.2.3.5.4 The TARGET GREEN cross pointer

This **GREEN** temporary cross-shaped pointer appears as soon as the user clicks on a picture point in target mode. This green target cross-shaped pointer turns to the **WHITE** current stage position when the stage reaches the target position.

6.2.3.5.5 Cross display accuracy

The cross coordinates are expressed in pixel. The coordinates are related to the crossing point of the 2 thin lines of the cross-shaped pointer. The vertical thin line is always set to the right side of a pixel.

6.2.3.6 Alignment menu

6.2.3.6.1 Alignment file loading

The user can load an alignment file by calling the item "Open…" from menu "Algnmt ". Alignment files have a suffix "ref". In case where the current validated alignment has not been previously saved, the user is prompted to save it before loading.
The image with which the alignment had been processed is loaded as well and the alignment points displayed.
This new alignment will be saved and reloaded at the next pointlogger start.

6.2.3.6.2 Saving current alignment

One can save an alignment file under current name (Save) or under a new name (save as). The path selected at the saving will be used further as the default path.
The saved alignment file is becomes the "last alignment file" : at the next pointlogge restart , PointLogger will load this alignment file.

When an alignment has been done but has not been saved yet, an asterix caracter appears ahead of the filename displayed on the upper window banner.

6.2.3.6.3 Editing the alignment point set

Once an external image is loaded, this command is allowed. The user can update the alignment at any moment. The diode labeled «done» are set on or off depending if the alignment points have been previously validated or not.
The “Validation timestamp date” field is filled with the last validated alignment date.

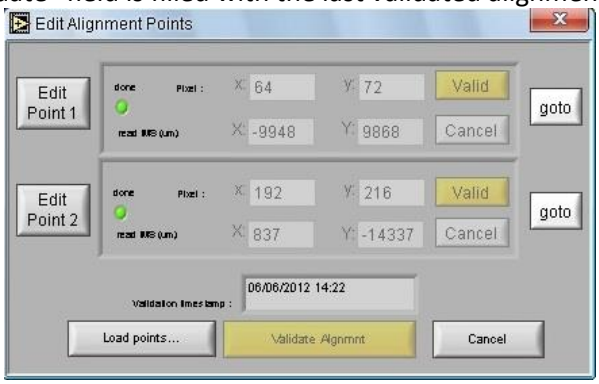


Figure 251: dialog box filled with an already validated alignment

6.2.3.6.4 Loading a set of points from another alignment file

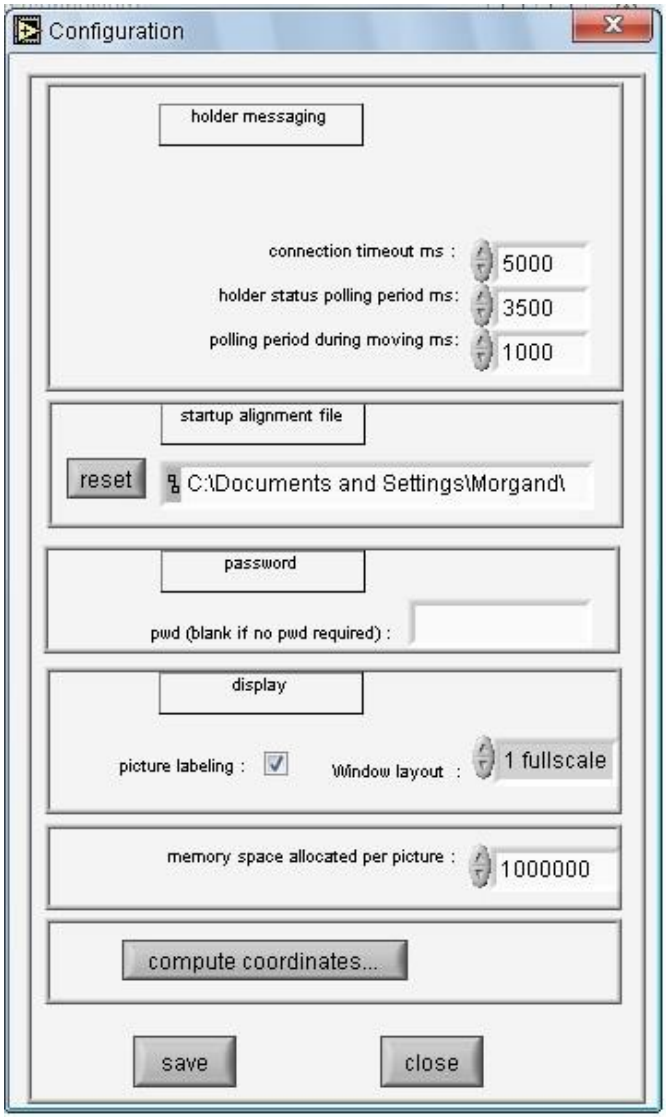
The user can pick alignment points from another alignment file. This can happen when the user has captured a new picture from the stage and is willing to re-use the alignment points input from a former picture. The alignment cross-shaped pointers displayed on the picture are updated with the new values.

6.2.3.6.5 Quitting a point alignment

At any moment, the user can quit the alignment process with button “Close”.

However the alignment points are memorized until the user re-opens the alignment dialog box.

6.2.3.7 Point Logger configuration



Underlaying messaging configuration for connection to holder. Set in factory.
not supposed to be changed

Last validated alignment file.
Reset deletes the reference to the file.

Access to that configuration dialog can be restrained by a password. Blank if no pwd.

Picture labelling controls the display of labeling on the upper side of picture views.

Window layout used the starting of PL.

Tool to simulate conversion from pixel to stage and vice-versa. Used for checking conversion formulae.

Figure 252 : configuration dialog box

6.2.4 Troubleshooting

If any problem occurs at starting, an efficient way to fix that is to remove the pointlogger setup file, called "PointLoggerSetup.xml" and installed in camims/data/holder. Pointlogger will rebuild a default setup file when restarting.

6.2.5 Appendix i: coordinate conversion formula

6.2.5.1 Prerequisites

The camera device main axis is assumed to be orthogonal in consideration with the stage plate.
The referential local to the camera (also named picture referential) should be rotated to be aligned with the stage main axis.

The own picture referential named {r0} is centered on the lower left corner of the picture and the axes are oriented rightwards and upwards.
The optical system applies a magnitude factor which is direction independent.

6.2.5.2 Conversion from external picture ref to sample holder ref

Assuming the constrains listed above, we consider a triangle made up of the 2 alignment points and a free point with known coordinates in the external picture referential.
It is asserted that the angles inside the triangle are kept unchanged during the transformation (rotation + translation).

P1 coordinates are noted (m1x, m1y) in the picture referential and (M1x, M1y) in the holder referential.
P2 coordinates are noted (m2x, m2y) in the picture referential and (M2x, M2y) in theholder referential.
P3 is a point with unknown holder coordinates.

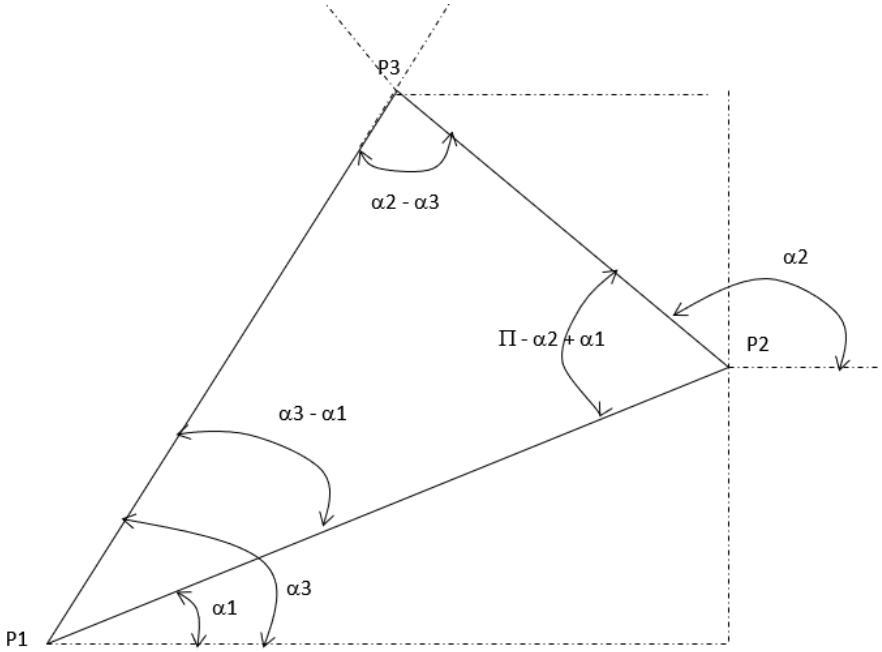


Figure 253 : triangle P1P2P3 and its inner angles

The angles $\alpha 1$, $\alpha 2$, $\alpha 3$ can be expressed (in the picture referential) as :
$\alpha 1$ = ATAN ((m2y - m1y) / (m2x - m1x))
$\alpha 2$ = ATAN ((m3y – m2y) / (m3x – m2x))
$\alpha 3$ = ATAN ((m3y - m1y) / (m3x - m1x))
As asserted above :
$\alpha 3 - \alpha 1$ keeps unchanged during transformation
$\alpha 2 - \alpha 1$ keeps unchanged during transformation
The angles $\alpha 1$, $\alpha 2$, $\alpha 3$ can be expressed (in the holder referential) as :
$\alpha 1$ = ATAN ((M2y - M1y) / (M2x - M1x))
$\alpha 2$ = ATAN ((M3y – M2y) / (M3x – M2x))
$\alpha 3$ = ATAN ((M3y - M1y) / (M3x - M1x))

To simplify, intermediairy $\alpha 1$, named alpha1 in respect with picture referential and ALPHA1 respect with holder referential.
We obtain the following equation system :
ATAN ((m3y – m2y) / (m3x – m2x)) – alpha1 = ATAN ((M3y – M2y) / (M3x – M2x)) – ALPHA1
ATAN ((m3y - m1y) / (m3x - m1x)) – alpha1 = ATAN ((M3y - M1y) / (M3x - M1x)) – ALPHA1

We gather terms M3x and M3y in one side :

TGT (ATAN ((m3y – m2y) / (m3x – m2x)) – alpha1 + ALPHA1) = (M3y – M2y) / (M3x – M2x)
TGT (ATAN ((m3y - m1y) / (m3x – m1x)) – alpha1 + ALPHA1) = (M3y - M1y) / (M3x - M1x)

We assert 2 intermediairy variables a and b expressed like :
a = TGT (ATAN ((m3y – m1y) / (m3x – m1x)) – alpha1 + ALPHA1)
b = TGT (ATAN ((m3y – m2y) / (m3x – m2x)) – alpha1 + ALPHA1)

a = (M3y – M1y) / (M3x – M1x)
b = (M3y – M2y) / (M3x – M2x)

a * (M3x – M1x) = M3y – M1y
b * (M3x – M2x) = M3y – M2y

a * (M3x – M1x) – b * (M3x – M2x) = – M1y + M2y

M3x * (a – b) = – M1y + M2y + a * M1x – b * M2x

M3x = (M1x * a - M1y + M2y - M2x * b) / (a - b)
M3y = M3x * a - M1x * a + M1y

In the end, M3x and M3y are expressed as :
M3x = (M1x * a - M1y + M2y - M2x * b) / (a - b)
M3y = M3x * a - M1x * a + M1y

6.2.5.3 Inverse conversion from holder ref to picture ref

Equations are symmetrical to equations defined above :
m3x = (A * m1x – m1y + m2y – B * m2x) / (A – B)
m3y = A * m3x + m1y – A * m1x

where A and B are expressed as :
A = TGT (ATAN ((M3y – M1y) / (M3x – M1x)) – ALPHA1 + alpha1)
B = TGT (ATAN ((M3y – M2y) / (M3x – M2x)) – ALPHA1 + alpha1)

6.2.6 Appendix II: Rotation formula

For the rotation angle, only rotation noted ($\alpha + \varphi$) is taken,ignoring the following other parameters :

. the homothetical magnitude noted (β)
. the origin move of pixel referential (O -> P1)
. the origin move of holder referential (P1 -> holder origin)

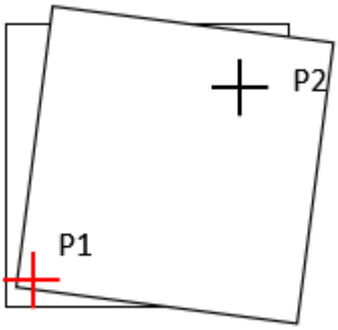


Figure 254 : rotation principle

Note that P1 is invariant in consideration with the rotation.
The α angle is computed as:

α = arctangent ((y2-y1) / (x2-x1))

with : x1,y1 pixel coordinates of P1 and x2,y2 pixel coordinates of P2

φ is computed as:

φ = arctangent ((Y2 – Y1) / (X2 – X1))

with : X1,Y1 holder coordinates of P1 and X2,Y2 holder coordinates of P2

6.3 Editor

The editor allows the user to load the full analytical conditions of a previous analysis (Figure 255). It is then possible to export or print the file.

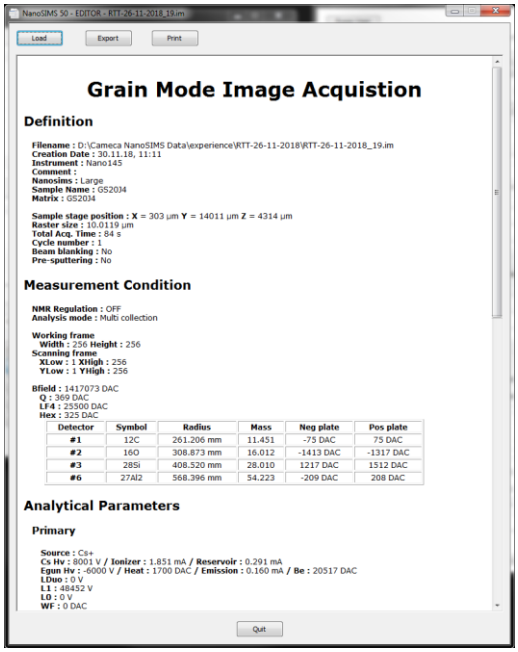


Figure 255: Editor window showing all acquisition parameters

7 NS50L Software: The “OTHER” taskbar

7.1 Periodic table

The Periodic table (Figure 256) provides information about natural element abundances and interferences from a Mendeleïev table.
By clicking **left** on elements, the operator can add elements to the list of “selected elements” or he can delete them by clicking on the button “delete” in the selection editor.
By clicking **right** on the elements of the Mendeleïev table, the operator can see the natural isotope abundance of the element.

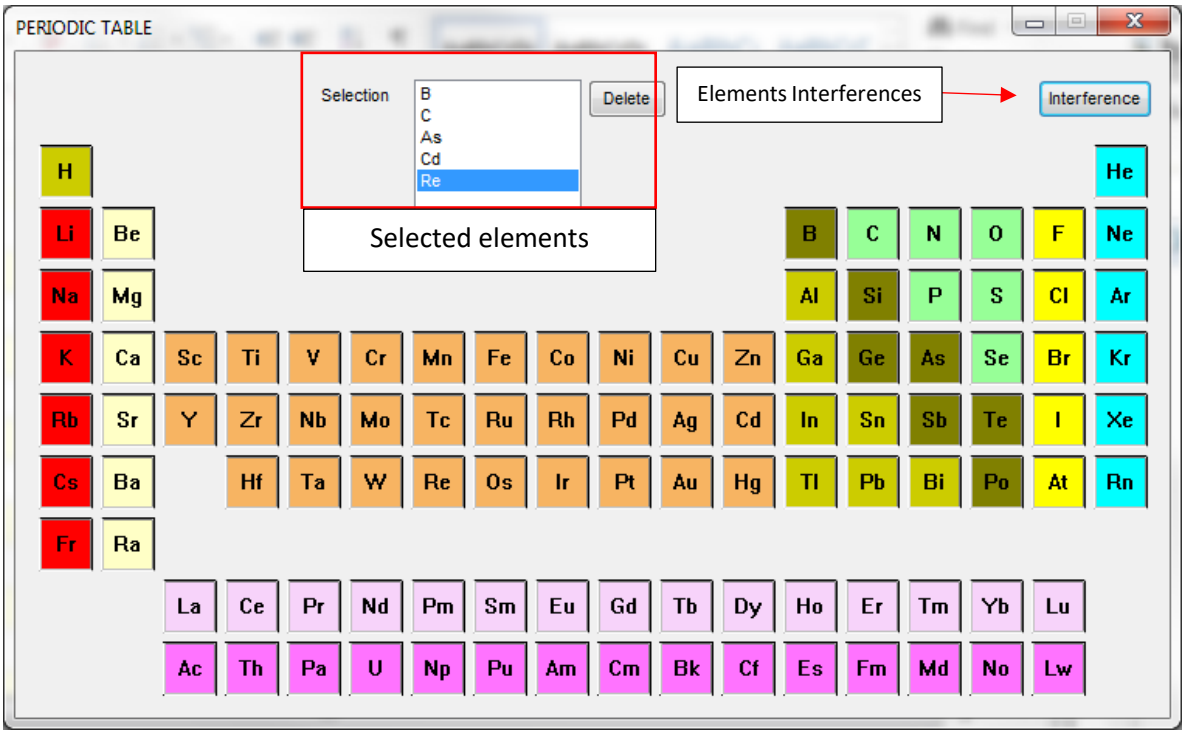


Figure 256: Periodic table window

When clicking on the “interference” button, a window opens, allowing to see all possible mass interference coming from the selected elements on an ion formula to enter (Figure 257).
Enter **the ion chemical formula or a mass number** in the “**nominal masses**” box. The following formats are allowed (Figure 257):

- Chemical formula without specifying isotopes, with no space between letters. In this case, the major isotope will be selected. Ex: CN (→26.0031 amu)
- Chemical formula with specific isotopes. This time, a space between species is necessary. Ex: 28Si2 14N4 (→111.9662 amu)
- Mass number Ex: 26.

When entering a formula in order to filter through all possible interferences, you can also specify a minimum mass resolving power (Min MRP) and a minimum species abundance (Min. Abundance). Species not matching those criteria (e.g. too far in mass or too low in abundance) will not be shown in the results to make it more readable.
You can also choose to see interferences for all combination of the selected masses (“All masses”) or only on the “nominal masses” you have entered.
Click “Start” for the list of interferences to appear.

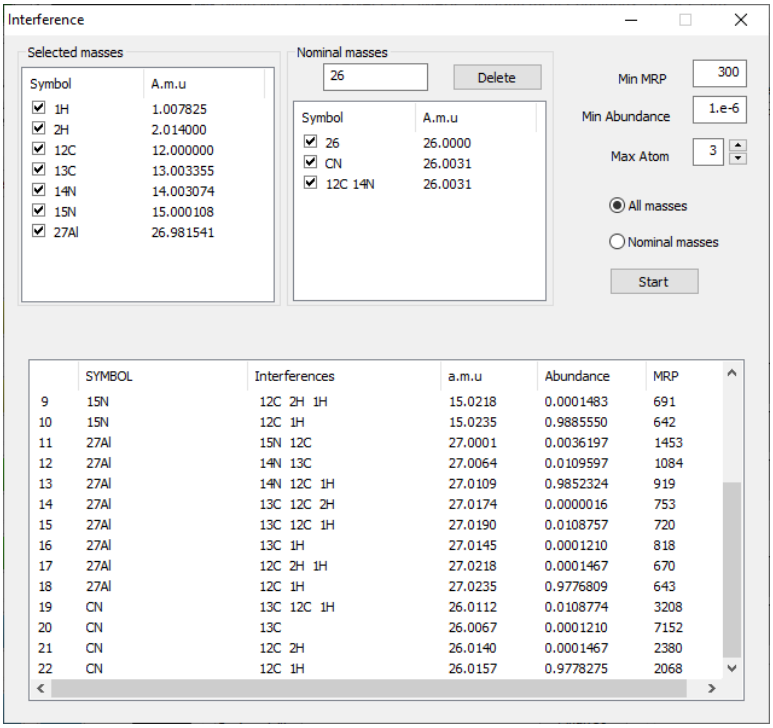


Figure 257: Interferences window

7.2 Ana2Excel export to Excel format

This export program (Figure 259) allows the user to export a data summary from isotopic data acquisition files (.stat) into an Excel spreadsheet after a chained analysis. Each .stat acquisition file shows a list of analysis parameters, as well as the results of the analysis, as the ratios defined before the analysis (see chapter 9.2.6.2 on isotopic analyses).

Below a header giving the acquisition conditions, the results are shown in four sections (Figure 258), corresponding respectively to:

- **Section 1:** results are given with **no correction** applied for EM detectors and with a **simple correction** of the background noise as defined in the Setup for FC detectors.

- **Section 2**: results are given with **yield and dead time corrections** applied for EM detectors and with a **simple correction** of the background as defined in the Setup for FC detectors.

- **Section 3:** results are given with **yield and dead time corrections** applied for EM detectors and with a **correction of the baseline** as defined **in the Setup** for FC detectors.

- **Section 4**: results are given with **yield and dead time corrections** applied for EM detectors and with a **correction of the background** as measured **during the acquisition** (average background noise over 10 seconds before and 10 seconds after the analysis, as set in the SETUP program. See 5.9.4.1).

CAMECA \ GRAIN MODE - ISOTOPES \ Sample : Sample_#1

29.08.19 20:33

```
Stage Position   : x=-12149 um  y=1837   um  z=2200   um
Raw data file    : D:\Cameca NanoSIMS Data\experience\IsoCeline\IsoCeline_3_mg_12.is
Block number     : 10
Meas. per block  : 30
Rejection(sigma) : 2
Slit Preset : None
Lens Preset : None
Pre Sputtering Time (s) : 499.5.
               Raster (um) : 35.0
               Slit Preset : None
               Lens Preset : None
Scanning On : Frame:91,207-115,231 / Blanking:100.0% / Raster:3.4um
Tuning Mode: Multicollection
Regulation Mode: NMR
Check every (fr.): 300
Secondary Ion Beam Centering : ON / during Acq : OFF
        Width Horizontal(V): 1.160 / Vertical(V): 1.034
E0S Centering : ON / during Acq : OFF
        Width(V): 155.092
E0P Offset (V): 0.000

Primary Current before acq :   31514 pA / after acq :   31509 pA


Mass# Species        Mass  Det.   Tc(s)   BField    Radius  Peak# RefPeak# Method
   1  28Si          27.687 Tr4    0.540  1228.747   551.436     1      1     BOTH       SIBC      E0SC
   2  29Si          28.716 Tr5    0.540  1228.747   561.582           1
   3  30Si          29.713 Tr6    0.540  1228.747   571.250           1


-- Section 1 ---- EM & FC with Setup Background ---------------------

  Block to block results

    Block#  Ratio#    Mean        SD      N_rej Err_mean(%)  Poisson(%)    Khi2

       1     2/1   5.082E-002  3.83E-004    2   1.42E-001    1.97E-001   5.2E-001
       1     3/1   3.344E-002  4.91E-004    1   2.73E-001    2.37E-001   1.3E+000

       2     2/1   5.069E-002  4.37E-004    1   1.60E-001    1.93E-001   6.9E-001
       2     3/1   3.331E-002  3.90E-004    0   2.14E-001    2.32E-001   8.5E-001

       3     2/1   5.082E-002  5.79E-004    2   2.15E-001    1.96E-001   1.2E+000
       3     3/1   3.335E-002  4.94E-004    1   2.75E-001    2.36E-001   1.4E+000

       4     2/1   5.068E-002  4.01E-004    1   1.47E-001    1.93E-001   5.8E-001
```

*Figure 258: a *.stat file from an isotope acquisition analysis (partial view).*

To export data into Excel:

- Launch Ana2Excel.
- Click on “File and select the .is analysis file you wish to extract, then choose the section you wish to extract (1, 2, 3 or 4), depending whether you wish to extract data with or without corrections. We recommend section 4.
- Click on “Extract”. This will create a new .csv file in the same directory where your analysis file is stored.
- Launch the Excel software. In the “Data” menu, click “extract from text/CSV” and select the newly created file. In the new window, select “Delimited” and click on “next”. Select “semi-colon” and click on Finish. The resulting excel file should now be correctly formatted.

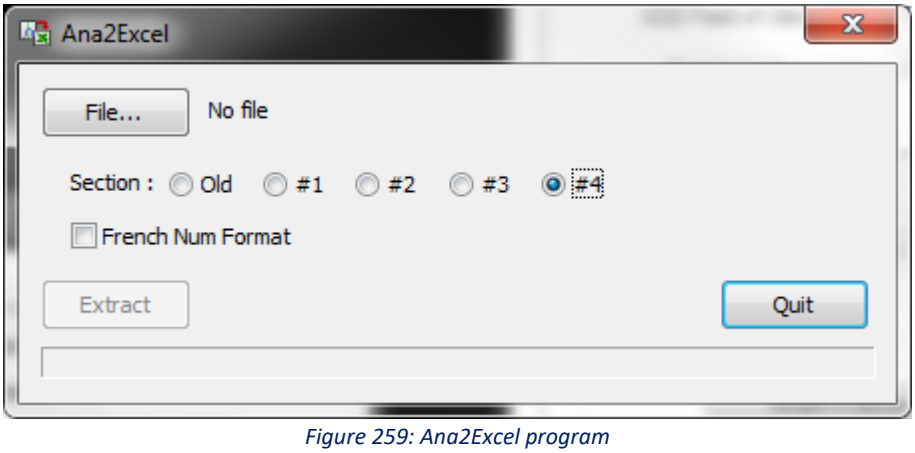


Figure 259: Ana2Excel program

*Other options were from previous export versions and do not work. Always use option 4.

7.3 Virtual Keyboard

This program displays a virtual keyboard (Figure 260), identical to the physical dedicated keyboard and its three thumbwheels. It can be used locally, replacing the dedicated keyboard, and controlling all parameters. But mostly it is used for remote control of the NanoSIMS.

In order to change a parameter, the user can:

- Enter directly the numerical value to be sent (and type enter), or
- Use the **mouse wheel** to modify the value. By moving the mouse wheel, the numerical value will be changed one bit by one bit. Pressing the **CTRL key** while moving the mouse wheel will move **x10 faster** (by ten bit increments). Pressing **Ctrl + Shift buttons** will move **x100 faster** (by 100 bit increments). Finally activating the "**Fast"** button on the virtual keyboard will multiply the speed by another factor **X10**.
- Or use the PC keyboard instead of the mouse wheel as:
  - **Letf/right** arrows are used to move **X** parameter,
  - **Up/down** arrows are used to move **Y** parameter,
  - **m/n** key are used to move **Z** parameter,

  combining with **Ctrl key** for **x10** ; **Ctrl + Shift keys** for **x100** and **Fast** for **x10** speed.

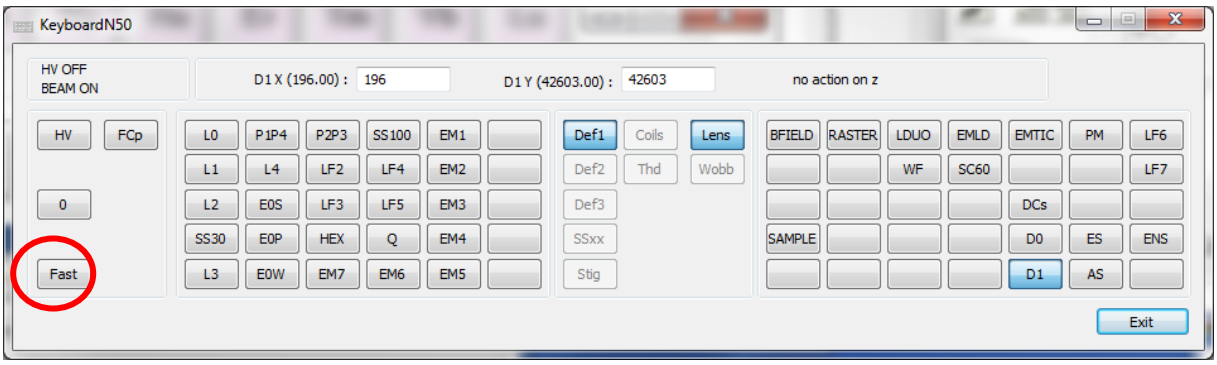


Figure 260: Virtual Keyboard

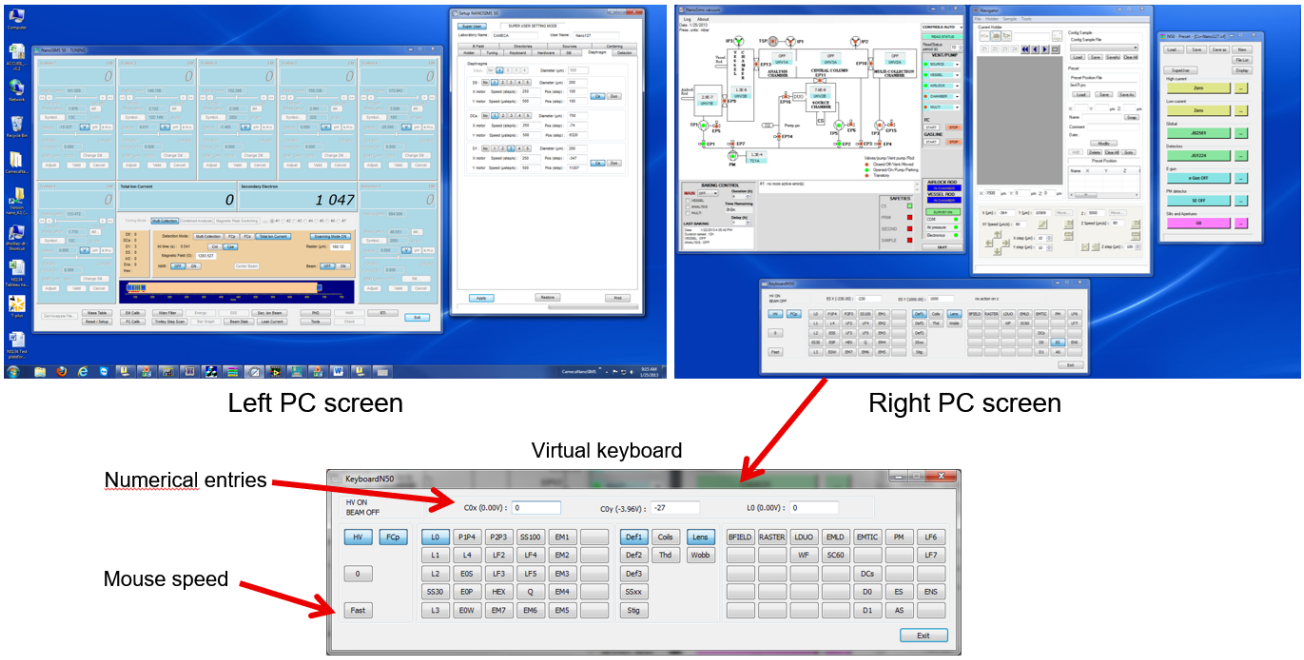


Figure 261 : example of Virtual keyboard used in a two-screen configuration

The Virtual keyboard can be used to control the instrument from a nearby control room, with a duplicated control configuration and two display screens.
The Virtual keyboard is, above all, very useful to control the instrument over the internet in conjunction with Team Viewer software. Service engineers will often use this Virtual keyboard to tune and check an instrument and diagnose remotely the origin of a problem.

7.4 SerialServer

This program allows communication between the software and the Hyperion source. Note that when starting the instrument and programs, the Hyperion electronics must be switched on before starting SerialServer. When clicking on the SerialServer icon, a small window appears (see Figure 262). To make sure the RF source is communicating, click on "Show Console". A second window opens (Figure 263). Make sure that it reads "RFGen Connection ON". If not, click on "Reconnect" to check the connection. If the connection fails, quit the SerialServer program and make sure the RF source electronics is on before restarting SerialServer.

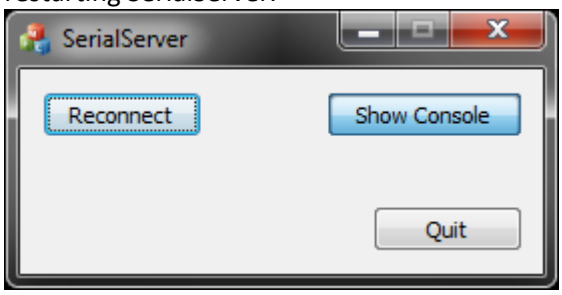


Figure 262: SerialServer window.

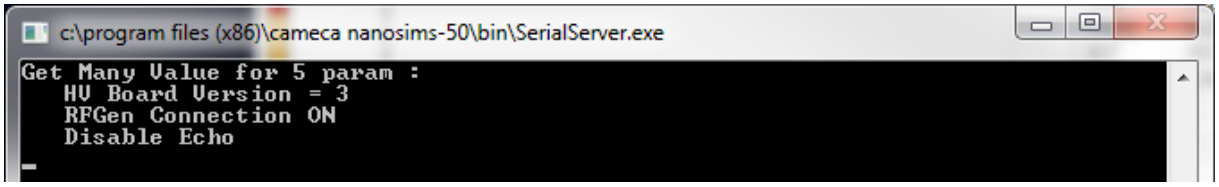


Figure 263: SerialServer console

If everything is working, you can hide the console by clicking again on "show console", but ***do not close or minimize*** **the SerialServer window** (Figure 262). This window must be displayed at all time or the communication with the source electronics will be interrupted.

7.5 SpyEdit

This program records every command entered by the operator and stores the information for a given number of days (as set in the Setup, see chapter 5.9.11.2). It can help solving software issues. It is for Cameca programming purpose only.

8 Communication between computer and instrument

The real-time unit is the link between the computer and the CAMECA NanoSIMS 50L instrument. Furthermore, all the signal acquisition, scanning and keyboard values go through this unit.

The communication between the computer and the CAMECA NanoSIMS 50L can be made thank to four programs:

- The **MachServer** which is the communication server
- The Real-Time terminal called "**Mach.Ter**" which is an interaction window where all Real-Time information are written and can be read
- The **Load68** program which allows to download a program establishing the communication between the computer and the instrument
- The Vacuum terminal called "**Vac.Ter**" which is an interaction window where all Vacuum information are written and can be read

Most of the time, the MachServer (8.1), the Real-Time Window program (8.2), the Load68 program (8.3), the Vacuum Terminal (8.4) **are only used to connect the computer to the instrument** when starting the software. Those programs must remain open, but the operator does not normally interact with them while running analyses.

8.1 MachServer

The MachServer is a program making the communication between the instrument and the PC computer (Figure 264).
This program must be open when the instrument is running. This black window can be read only. The operator cannot type instructions inside.

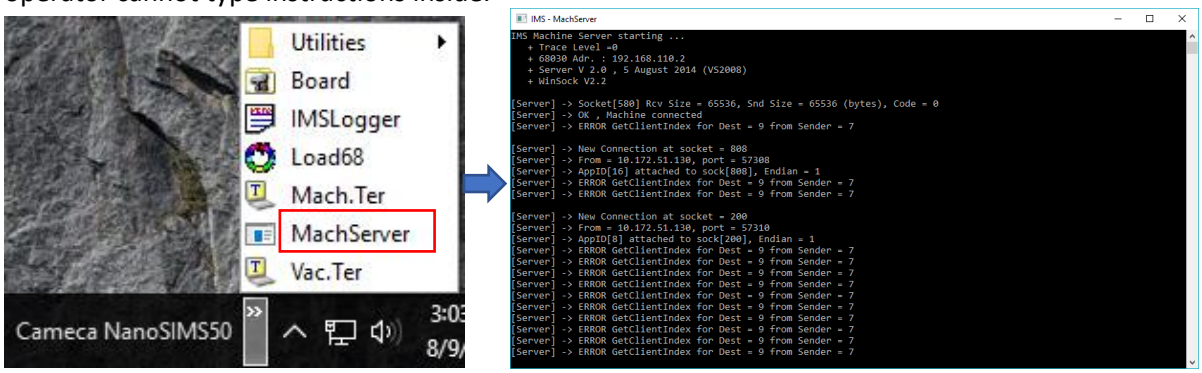


Figure 264: MachServer window

8.2 Mach. Ter (Real Time Terminal)

This program is a terminal window which allows the communication with the Real-Time Unit (RTU) of the instrument (Figure 265). In this window, the operator can **read** Real-Time information or **write** Real-Time instructions from and to the instrument. Moving motors, writing keyboard values or even changing the Bfield polarity can be done in this window. However, **there is no safety when the operator uses this window and some mistake could damage the instrument**. Good knowledges are needed to control the instrument through this program and it is usually not recommended.

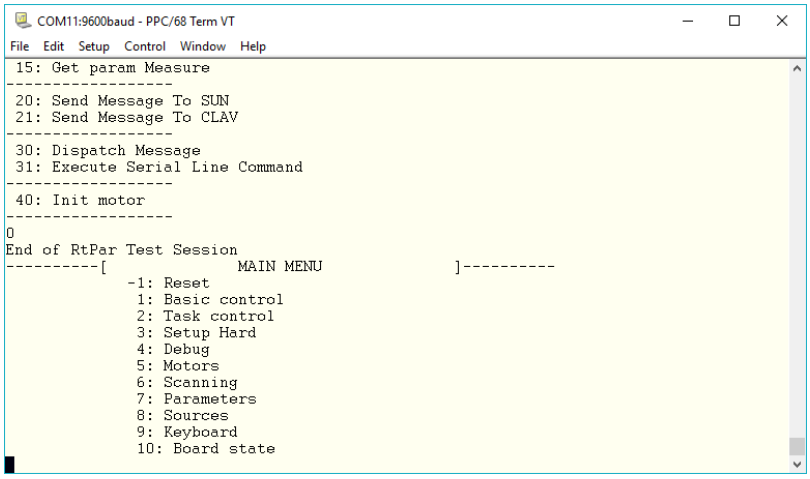


Figure 265: Real-Time Terminal window

8.3 Load68 program

This program (Figure 266) allows sending necessary information to the instrument to start the RTU.
This program is only used to start the instrument when the electronic has been turned off for any reason (see 10.1)

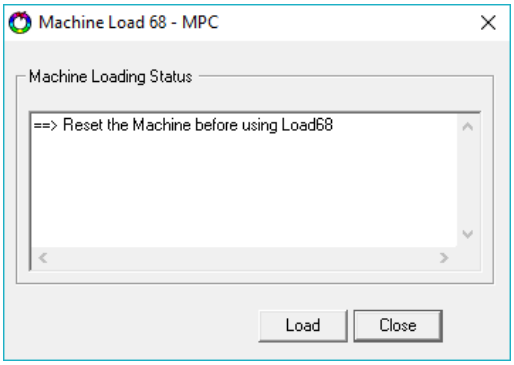


Figure 266: Load68 program

Two buttons are available:

- Load  : When the instrument has been reset, all the information is sent by clicking on this button. The loading is automatically done.
- Close  : Close the program

8.4 Vacuum terminal

8.4.1 Vacuum terminal window

This program is a terminal which allows to communicate with the vacuum automaton. In this window, the operator can **read** vacuum information or **write** vacuum instructions from or to the instrument. Reading gauge values, controlling a pump or even switching off the survey can be done in this window. However, **there is no safety when the operator uses this window and some mistake could damage the instrument. Good knowledge is required to control the vacuum through this program.**

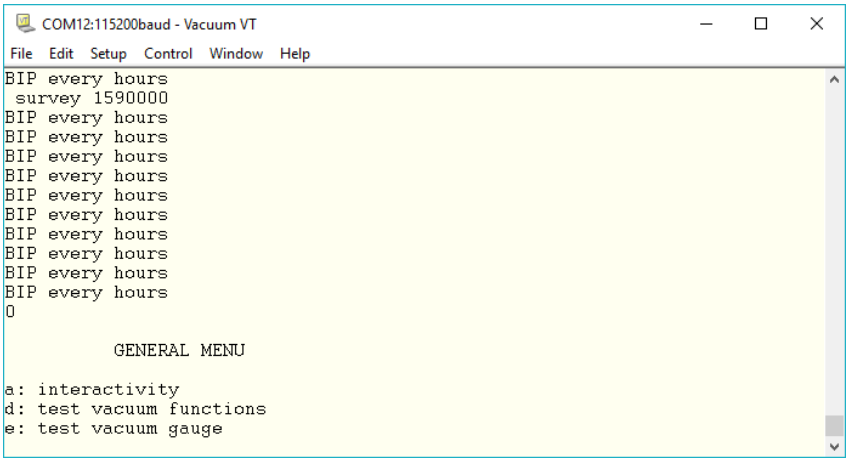


Figure 267: Vacuum terminal window

Several menus allowing a control or a simple display of all the automation functions are available from the RS232 serial link of the microprocessor 68070, through the Vacuum PC window interface or through the "VacTer" Vacuum Terminal.

Terminal VT mode:
Several menus allowing a manual control of all the functions of the automation are available from the RS232 serial link of the microprocessor 68070 (P631) link 45629330 PORT2, through the PC window interface vacuum or through the Vacuum Terminal "Vacter"

Synoptic PC mode:
All the statuses are available in the vacuum synoptic through the RS232 serial link of the microprocessor 68070 (P631) link 45629330 PORT1.
All procedures allowing to set the NanoSIMS under vacuum and to control the various parts (valves…) are available.

8.4.2 Vacuum terminal organisation

8.4.2.1 Vacuum window general menu

The general menu is separated in three parts: interactivity, test vacuum function and test vacuum gauges.

GENERAL MENU	
a = "Interactivity"	: Access to the « Interactivity menu »
d = "Test vacuum functions"	: Access to the « Basic functions »
e = "Test vacuum gauges"	: Access to the « Vacuum measurement »

Type "a" and Return to activate the 'interactivity menu'.
Type "xxxx " and Return to activate the selected function.
Type "0" and Return go back to the 'general menu'.

8.4.2.2 Interactivity menu

INTERACTIVITY MENU	
c: print_on	Displays every dialogue 68070/user
d: print_off	Deactivation of previous displays
e: print status	Pump or Vent status reading
f: chge_stat_source--> PUMP	Status Pump of source

g: chge_stat_source--> VENT	Status Vent of source
h: hours_baking	Baking hours reading
i: chge_stat_vessel--> PUMP	Status Pump of the vessel
j: chge_stat_vessel--> VENT	Status Vent of the vessel
k: chge_stat_ multicollection --> PUMP	Status Pump of the multicollection
l: chge_stat_ multicollection --> VENT	Status Vent of the multicollection
m: print stat selector	keyboard address table
n: chge_stat chamber --> PUMP	Status Pump of the main chamber
o: chge_stat_chamber --> VENT	Status Vent of the main chamber
p: stop_survey	Vacuum survey system desactivation
q: autorisation_survey	Vacuum survey system activation
r: chge_stat_airlock --> PUMP	Status Pump of the airlock
s: chge_stat_ airlock --> VENT	Status Vent of the airlock
v: PROM version	EPROMS Version reading
0: general menu	Return to general menu

8.4.2.3 Basic functions

Function	Closed	Middle position	Open
EP10	= 1	3	= 2
EP13	= 4	12	= 8
EP9	= 64	192	= 128
EP11	= 256	768	= 512

Function	Full speed	Stop
TP1	0	4
TP3	0	16
TP5	0	64
PM	1	3
PM	Accel 2	3

Electronic	0	OFF
Electronic	32768	ON

8.4.2.4 Vacuum measurement

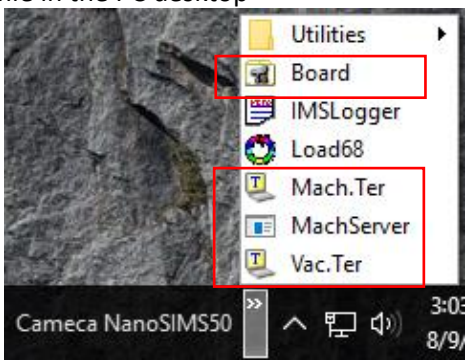
Type “e” and « return » to activate the test ‘vacuum gauges'.
Type “xxxx” and « return » to activate the selected function.
To go back to the 'general menu', type « 222. »

Function	Address	Pressure value (mbar)
TC1A	0	$1.33.10^{-3}$
UHV1A	1	4.10^{-6}
UHV2A	2	$9.36.10^{-7}$
UHV3A	3	4.10^{-7}
UHV1B	4	4.10^{-6}
UHV2B	5	6.10^{-7}
UHV3B	6	9.10^{-7}

8.5 Connecting the instrument and the PC

If the instrument has been turned off, refer to 10.1.
If the instrument is already turned on but the PC is off, follow this procedure to connect the PC to the instrument:

- In the CAMECA NanoSIMS50 file in the PC desktop



- Open the Real-Time (RT) terminal “Mach.Ter”
- Open the Vacuum terminal “Vac.Ter”
- Open the MachServer
- Open the Board program

The instrument should be connected to the computer and the operator car run an analysis.

9 NanoSIMS 50L operation

9.1 Basic operation

9.1.1 Sample introduction

A few recommendations:

- all elements going into the instruments need to be manipulated with clean gloves.
- Samples must be dehydrated, tools (screwdrivers, tweezers, …) must be kept clean (clean with acetone or alcohol).
- Sample mounting materials (resin for embedding, tapes and adhesives) must be UHV compatible.

Remember that the NanoSIMS works in the mid 10^{-10} mbar pressure range , typically 1000 times lower than electron microscopes !!!

- The NanoSIMS is a near-lens microscope with a high electrical field. Any particle, whisker, cell or piece of tissue that is not properly fixed can (will!) jump and stick to the immersion lens, resulting in arcing. This will ultimately require instrument venting, immersion lens cleaning, and baking.

Failure to observe these drastic rules will result in vacuum degradation or internal contamination. Depending on the severity it can require additional pumping time, additional titanium sublimation, infinite pumping time, baking, dismounting for cleaning (alcohol, re-polishing, sanding, …) or part replacement.

Figure 268 and Figure 269 show the introduction system configuration:
Note the three positions A, B, and C for both sample transfer rods.

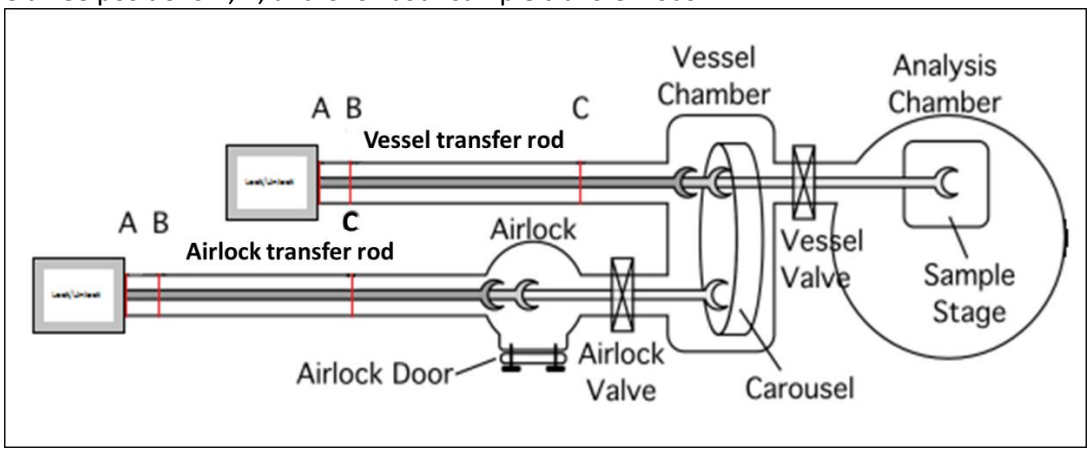


Figure 268: Schematic of the sample Airlock system

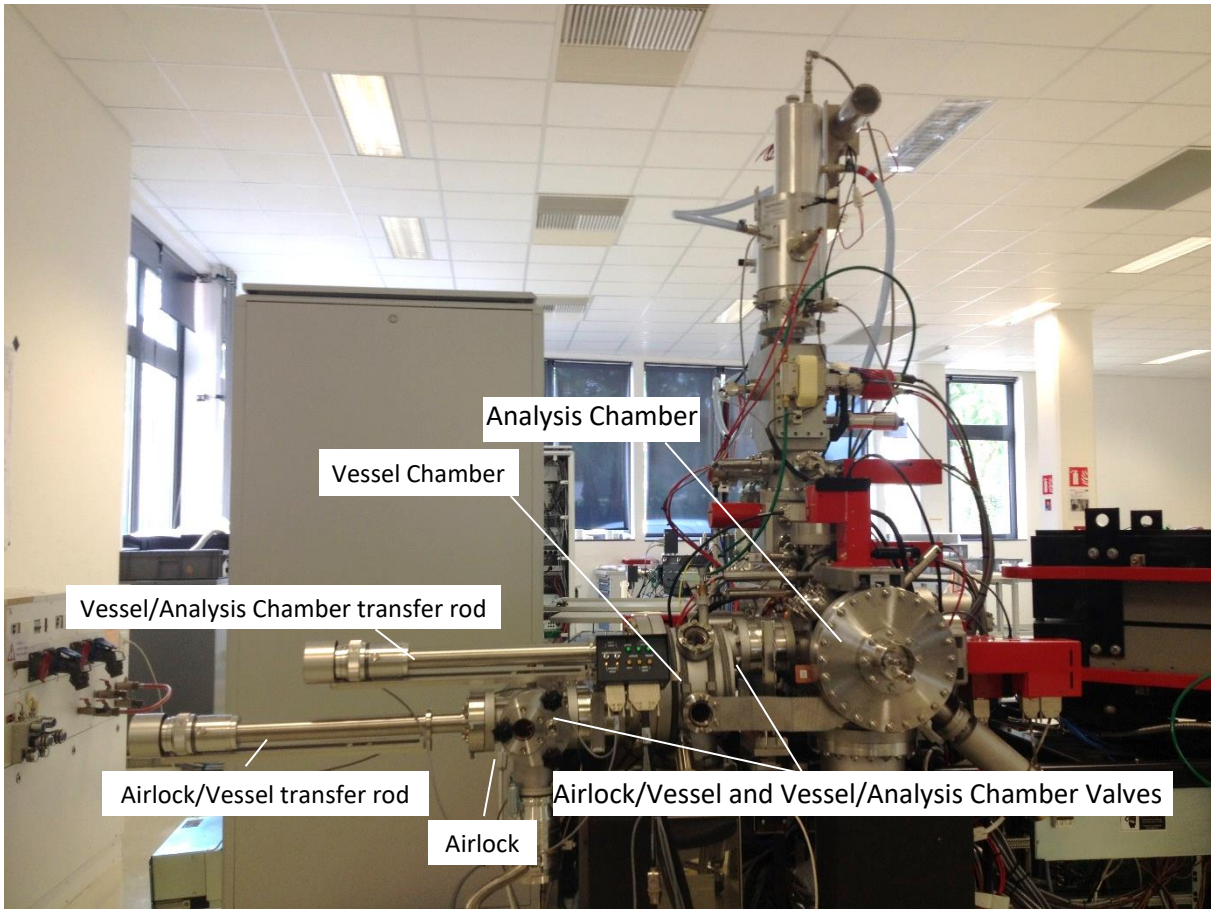


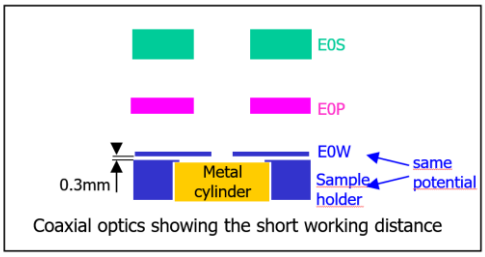
Figure 269: Photo of the sample Airlock system

The process described here assumes that a sample is introduced from the outside into the analysis chamber. However, in many cases samples will remain in the carousel for intermediate storage.

9.1.1.1 Sample mounting

To ensure good analyses, there are a few things to remember regarding the sample:

The overall performance of the NanoSIMS relies on the ***homogeneity of the electrical extraction field.*** The NanoSIMS, with its very short extraction gap, small slit size and high mass resolution, is more sensitive to sample surface height variations than other Cameca magnetic sector SIMS. Hence, the possibility to add automated secondary ion beam alignment & focusing routines to maintain good reproducibility.

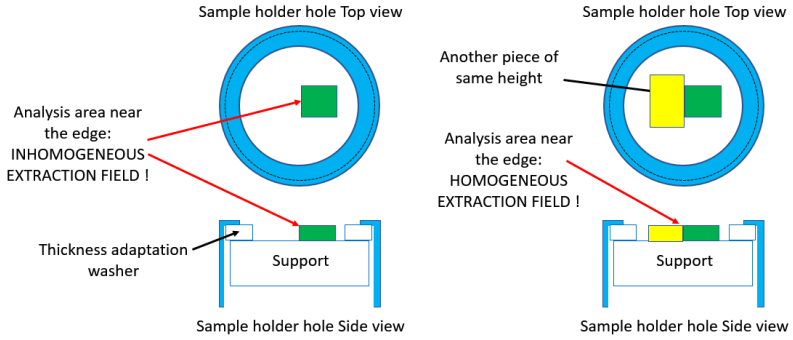


The sample's surface to be analyzed must be flat and parallel to the sample holder surface, itself mechanically adjusted to be parallel to the immersion lens surface. The analyzed area must remain at a constant distance of the E0W immersion lens: 400µm (which translates to ~300µm between the holder and E0W).
Strong topography at the surface will impact the sputtering, collection and transmission homogeneities. When applicable (e.g. in geological samples), it is recommended to polish the sample's surface to insure its flatness. Also, insulating samples should be metal-coated when possible.

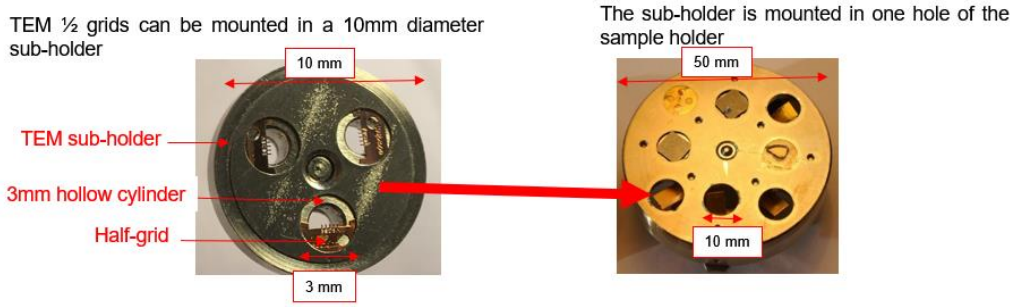
Similarly, for extraction field homogeneity:

- one should avoid analyzing sample areas less than 1mm away from the 100µm-thick sample holder hole lip.

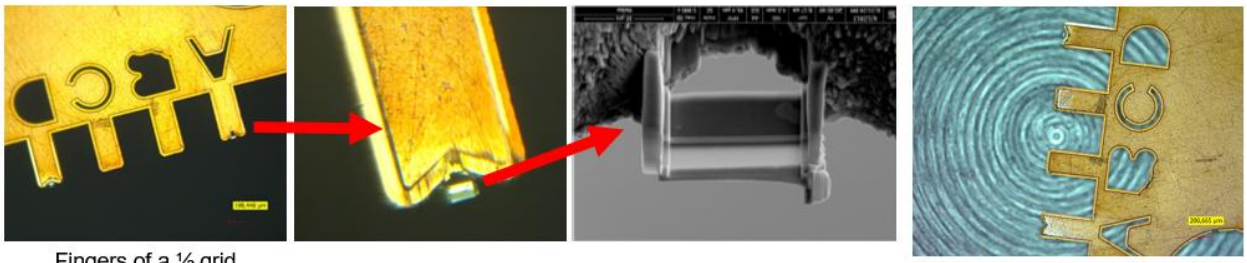
- If the area of interest is near the edge of a “thick” sample (several 100s µm) it is necessary to mount aside and in contact with the edge another piece of material with the same height.



- TEM grid: it can be either pressed flat on a double-sided UHV compatible sticky tape (e.g.: Cu or C) or mounted in the special sub-holder for 3 grids.



- If welded at the extremity of a half-TEM grid finger, with the half grid mounted in a special sub-holder, it must be backed by a flat surface a few hundreds µm behind (e.g. a small 3mm metal cylinder). Lamellas should be welded inside a V-type finger of the half-grid.



Fingers of a ½ grid

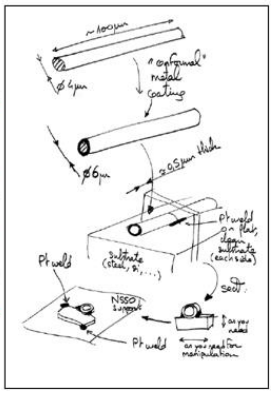
Zoom on a V-type finger with lamella welded on it

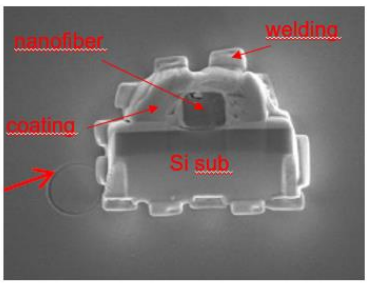
½ grid mounted with metallic cylinder below (in blue)

- FIB section: can be deposited flat on a Si wafer (with welding at the corners to avoid it jumping around). If the border of the lamella is the interest, the sample must be thickly coated (e.g: 1-2 µm thick coating if the section is ~1µm thick) prior to fibbing, in order to avoid field perturbation at the edges.

FIB-SEM lamella from a very insulating organic nanofiber.

1) welding to substrate,
2) Coated with GIS metal for uniform extraction field,
3) Fib section cut and laid flat,
4) Welding to the substrate to avoid jumping under charging





The surface potential must be well defined and as homogeneous as possible. In case of electrically insulating samples, the sample should be metal-coated, typically 10-30 nm of gold, platinum or other conductive species (*additional* charge compensation can be achieved in negative secondary ions by flooding with low energy electrons). For trace element analysis one should be careful about the purity of the coating material to avoid background signal or interference on the peak to detect. In SIMS, re-deposition can happen and in some cases surface migration, especially with cesium.
The **coating must be done *PRIOR* to mounting** the sample in the sample holder. Once the sample mounted, the user must check with an ohmmeter the good electrical contact (< 1 ohm) between the coating and the sample holder hole lip.

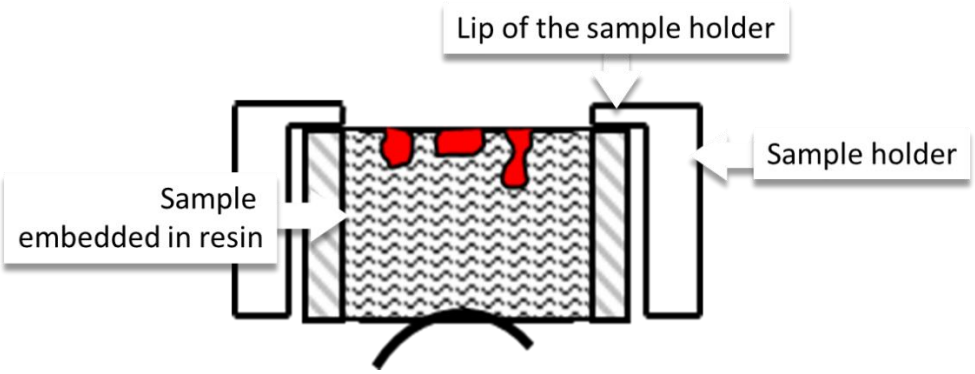


Figure 270: cross-section view of a sample embedded inside a ring, mounted in a sample holder.

The ideal sample is a cylindrical sample of the size of the holes (5 mm, 10 mm, 0.5 in or 1 in) with a flat surface. But in reality, samples come in all sizes and shapes. For this reason, CAMECA offers several sample accessories allowing to fit the sample to the sample holders. Sample holders are 50 mm wide, with hole sizes being 5 mm, 10 mm, 0.5 inch and 1 inch, depending on the type of holder. The thickness of the sample must be of around 5mm maximum.
Geological samples are usually embedded in 1-inch resin disks. The best results are achieved when the standards and the sample are embedded in the same resin block, positioned close to each other and near the center of the same hole. The embedding resin must be compatible with UHV and devoid of bubbles (some must be cured under vacuum). Depending on sample (roughness, shape,..) and subsequent required steps (e.g. polishing) or the required absence of one specific chemical element (e.g. nitrogen) different embedding media will be used. We recommend to read scientific articles based on NanoSIMS analyses. One can download a compilation of such articles from the CAMECA website.
Among the embedding resins commonly used, we can cite without commercial interest: Korapox 439 epoxy, LR White, LR Gold, EpoCure, EpoxiCure, Varian Torr Seal Low Vapor Pressure Resin. Also used: Wood metal (In-Bi alloy melting at 78°C).
Alternatively, particles can be pressed into a gold film pressed on a scratched surface (see Cosmochemistry articles) or pressed on a UHV-compatible sticky tape (e.g. carbon tape or copper tape – such as 3M EMI Copper Foil Shielding Tape).

FIB sections should be deposited flat on a conductive substrate (e.g. on a silicon wafer) but one should not forget to add platinum welding spots at corners to prevent the lamella from jumping during transport/loading/transfer/analysis!

While smaller samples can be accommodated with the addition of a home-made ring (Figure 271) the maximum sample diameter possible in the NanoSIMS is 1 inch. Such ring must be as thin as possible (0.1mm) while still flat and rigid.

Figure 271: examples of rings accommodating unregular shape samples

Below, a sample is first mounted in a sample holder (Figure 272) which is assembled onto a shuttle with three screws (Figure 273)

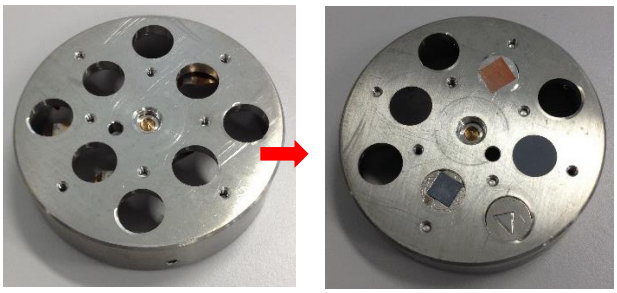


Figure 272: A sample holder before and after having mounted samples

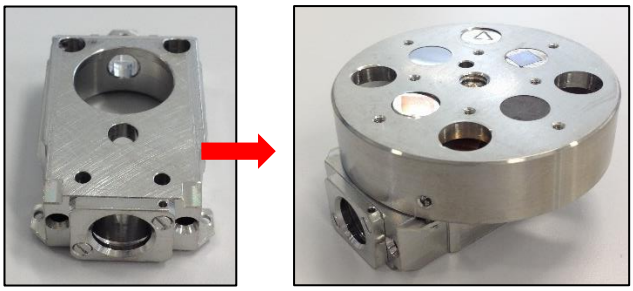


Figure 273: complete assembly of a sample holder and a shuttle ready to be introduced

To mount a sample in the sample holder, follow this procedure:

- Put the sample holder upside down and unscrew the copper spring.

Figure 274: back view of a sample holder with no samples (left) and with four samples (right)

- Place the sample(s) in the appropriate hole(s), so that it comes in contact with the lip (Figure 275). Be cautious not to bend the lip which is very thin (100µm). Potentially, add a 100µm-thick ring if the sample does not fit perfectly to the hole.

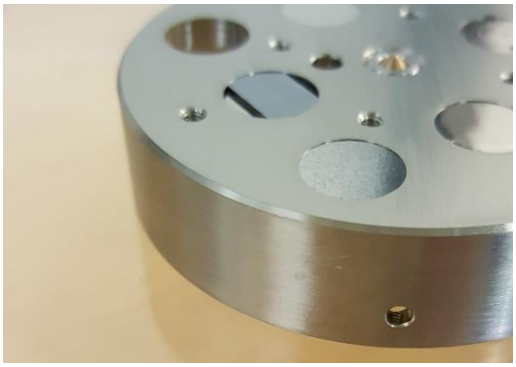


Figure 275: sample perfectly flat and in good contact with the holder's lip

- If the sample is too thin, add an extra height element (Figure 276) to facilitate the spring push.

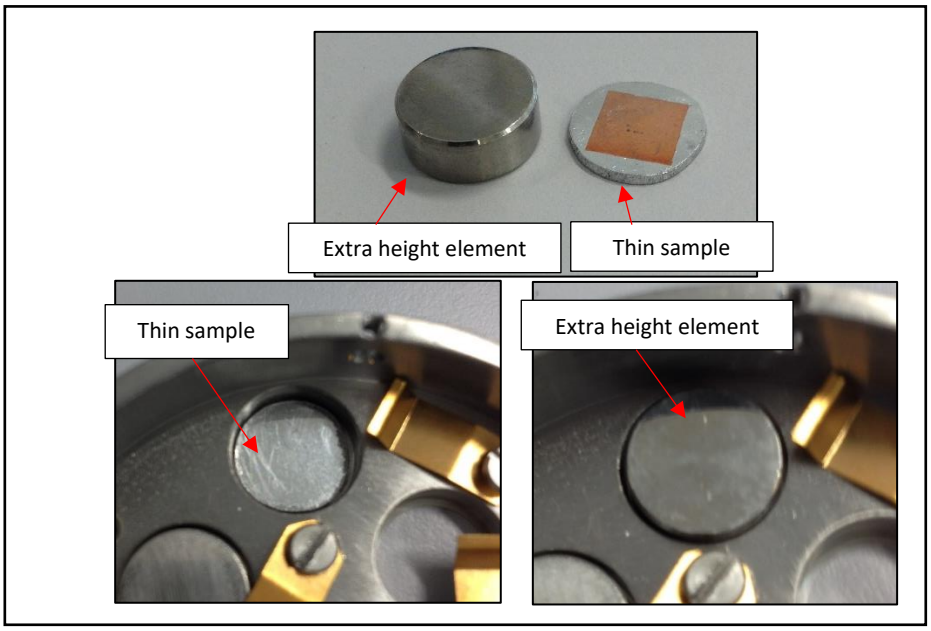


Figure 276: Extra height element for thin samples

- Screw the copper strips in order to block the samples (Figure 277).

Figure 277: all samples are held tight with a copper strip so that they wouldn't move.

Remark: All the strips need to be tightened even if there is no sample in the hole to avoid losing a strip or a screw in the chamber!

- Make sure that the sample is perfectly flat and not tilted once mounted in the holder and that the contact between the sample surface and the holder is secure (no space between the sample surface and the holder lip).
- Using an ohmmeter check the electrical conductivity between the sample surface and the sample holder.
- Dust off with compressed air the surface of the sample to make sure there is no dust polluting the sample surface.
- When the sample holder is ready to be introduced in the Airlock, screw it to the shuttle (Figure 278) and pay attention on the flatness of the assembly.

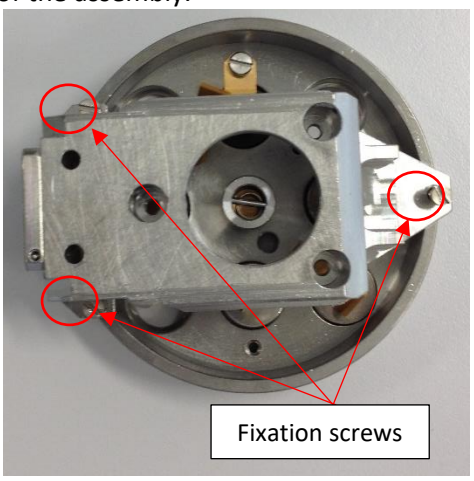


Figure 278: shuttle fixation on the sample holder with three screws

The three holes on the side of the shuttle are where the transfer rod grabs the shuttle (Figure 279).

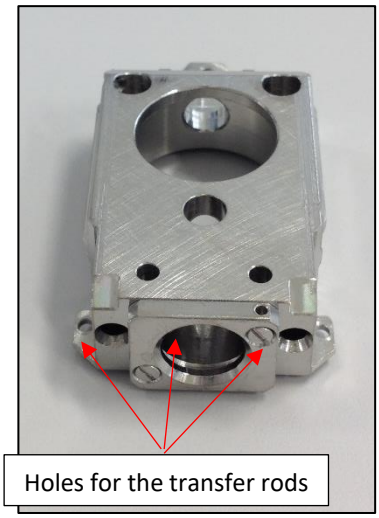


Figure 279: holes made to introduce the transfer rods

9.1.1.2 Sample loading in the Airlock

Make sure the airlock transfer rod is pulled all the way back (position A,

- Figure 268) and the Airlock/storage valve is closed.
- Verify that the inflatable dry nitrogen tank is full. This dry nitrogen (N_2) will be blown in the Airlock. For a good ventilation, it is important to have enough N_2 during all the process. Using such a balloon ensures there will be no overpressure. In case of using a bottle, check that the overpressure is not more than 0.1bar.

Figure 280: The inflatable dry nitrogen tank

- Vent the Airlock by pressing the 'Airlock Vent' button on the Airlock control pad. There is a 30s delay, during which the venting process can be aborted by pressing 'Pump'. Wait until the light stops blinking and the 'Vent' light stays ON (yellow means vented) (see Figure 281 and Figure 282).

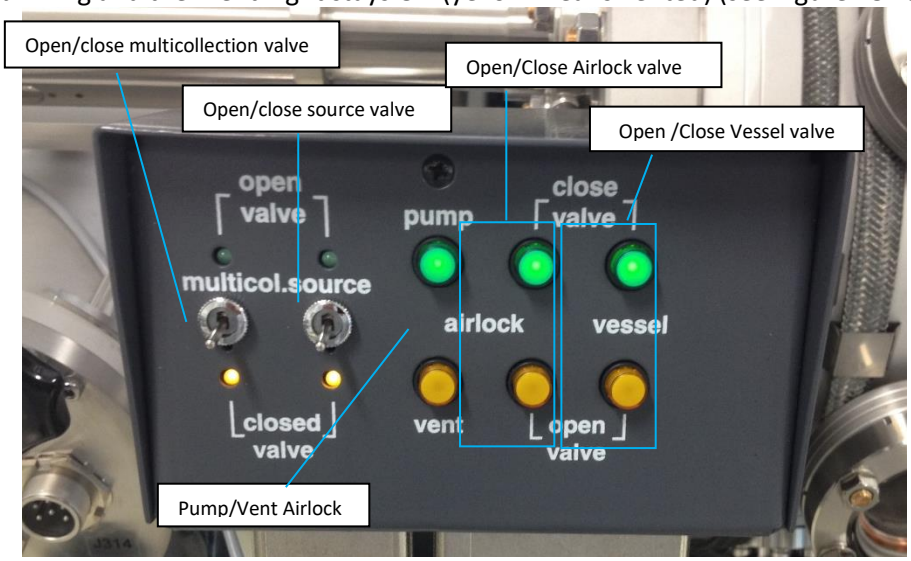


Figure 281: The Airlock control pad

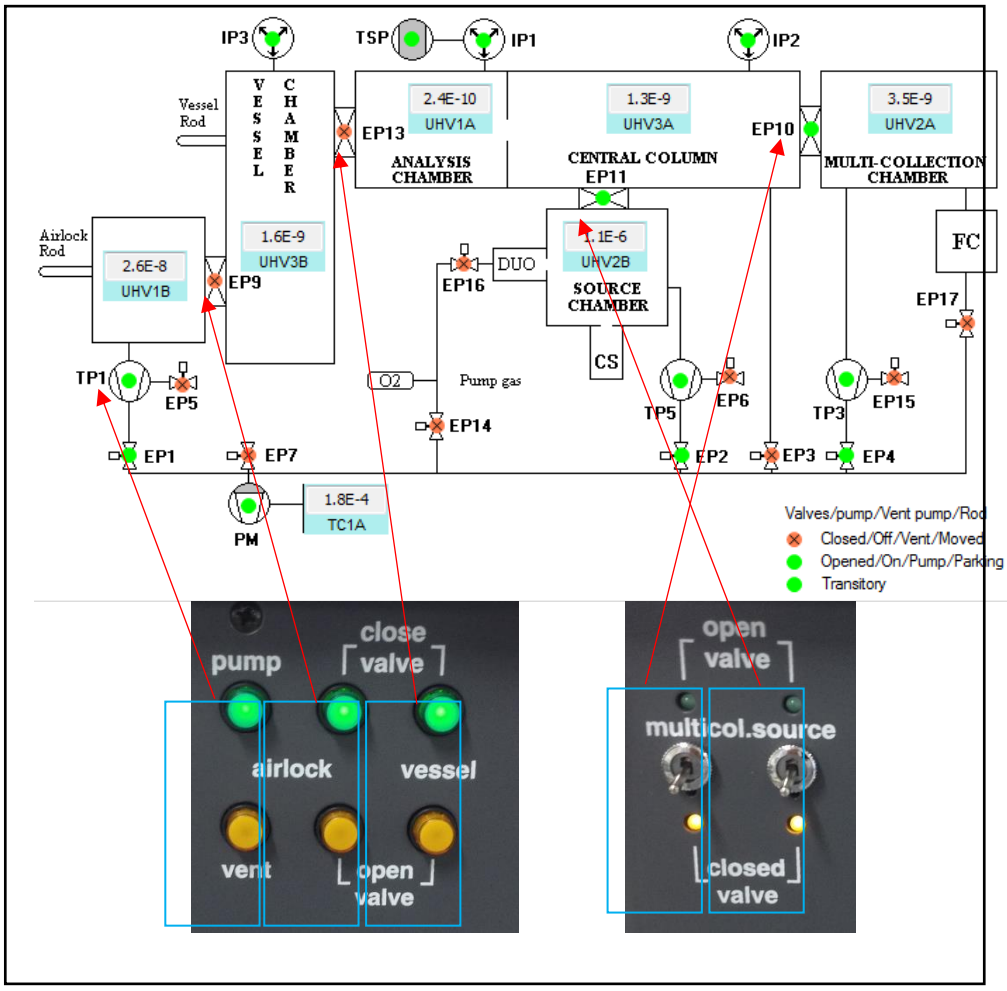


Figure 282: link between the Airlock control pad buttons and the vacuum synoptic

- Open the Airlock door by unscrewing the three black knobs. Pull out the door until the rods are fully extended (Figure 283).

Figure 283: The Airlock door open

- ***Use clean gloves*** to insert the holder/shuttle assembly into the bracket on the inside of the Airlock door. Check that the rear finger (Figure 284) can be easily inserted in the shuttle.
  Remark: when the Airlock is closed and the shuttle locker down (more details below), this rear finger (1 in Figure 284) is pushed down and, pushing the stick (2) into the shuttle. Consequently, when later the rod is pushed by the airlock sample locker to grab the shuttle, the shuttle does not move.

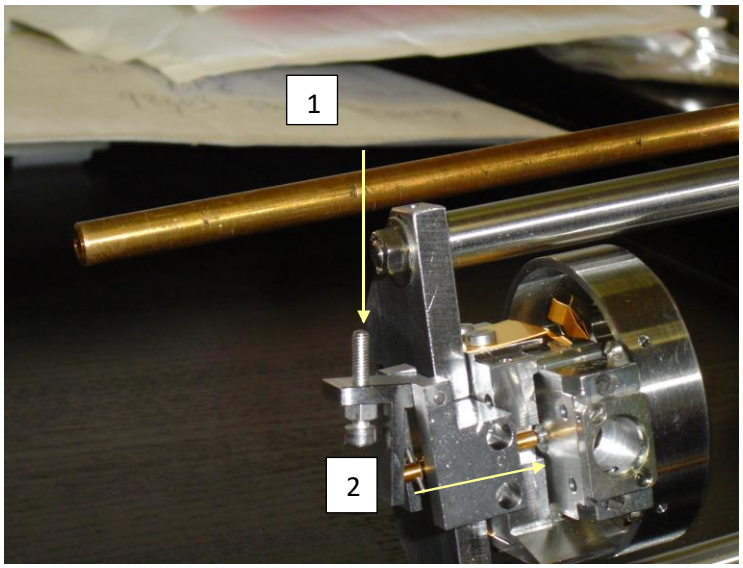


Figure 284: positioning of the shuttle in the Airlock

- After inserting the shuttle into the Airlock bracket, check that the small shaft at the rear can be freely inserted in the shuttle.
- Close the Airlock door and tighten the three knobs (no need to force, the atmospheric pressure will do the job).
- Lock the sample holder/shuttle in place by pulling the outer part of the airlock sample locker up, turning it, and pushing it down. If it does not go down all the way, the sample is not inserted correctly.

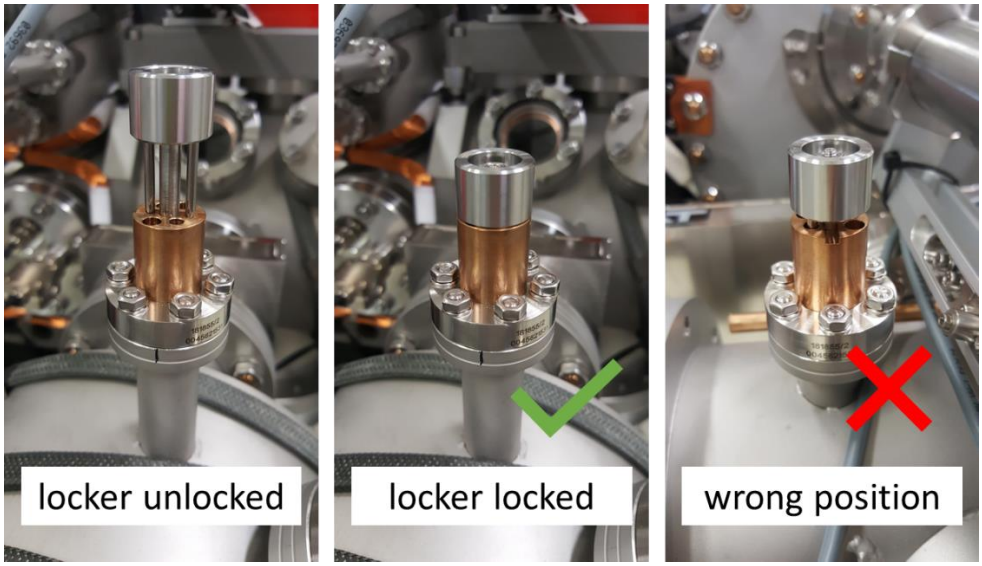


Figure 285: shuttle locker positions.

Locker Unlocked: the sample holder can be moved with the transfer rod.
Locker locked: the sample holder is hold in position by the internal finger: one can connect or disconnect the rod from it safely.
Wrong position: Danger ! the sample holder might well fall in the load-lock chamber when pushing it with the rod !!

- Pump the airlock by pressing the 'Airlock Pump' button on the Airlock control pad. There is a 30 second delay. Wait until the light stops blinking and the 'Pump' green light stays on (*Figure 281*).

9.1.1.3 Sample transfer from the Airlock to the Vessel chamber

- The transfer rod handle has two positions (Figure 286):
    - The "LOCK" position attaches the shuttle on the rod. Rotating the ring by 60° changes the arm to the 'UNLOCK' mode.
    - The "UNLOCK" position release the shuttle. Rotating the ring by -60° changes the arm to the 'LOCK" mode.

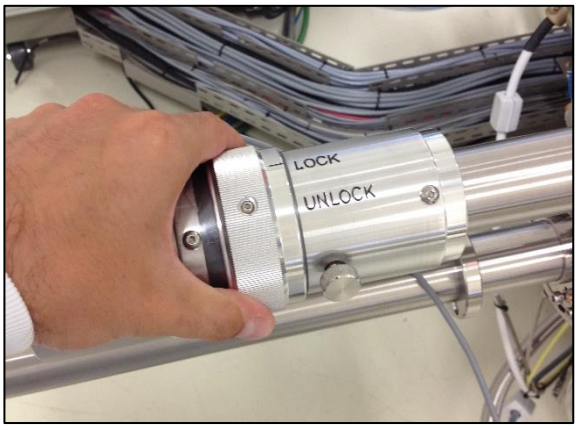


Figure 286: Lock the sample holder/shuttle to the transfer rod

- Check through the storage vessel chamber viewport that there is an empty position on the carousel.
- Check that the pressure in the airlock is below 5 x 10^{-7} mbar, low enough not to contaminate other samples in the storage vessel.
- Move the Airlock/vessel chamber transfer rod to position B (

- Figure 268) and attach the shuttle by moving the rod's ring from "unlock" to "lock" position (Figure 286).
- Open the airlock valve by pressing airlock 'open valve' on the airlock control pad (Figure 281). Wait until the 'open' light stays on (yellow).
- Pull the outer part of the airlock shuttle locker up (Figure 285).
- Make sure the carousel is in a correct position.
- Move the Airlock/Vessel chamber transfer rod to position C (
- Figure 268) and release the shuttle by rotating the rod ring to unlock position.
- Move the Airlock/vessel chamber transfer rod back to position A (
- Figure 268).
- Close the Airlock valve by pressing 'Airlock Close Valve' on the Airlock control pad (Figure 281). Wait until the 'close' light stays on (green).

9.1.1.4 Sample transfer from the Vessel to the Analysis chamber

- Rotate the carousel to move the sample to the position (upper position) where the Vessel/Analysis chamber transfer rod can reach it (Figure 287).

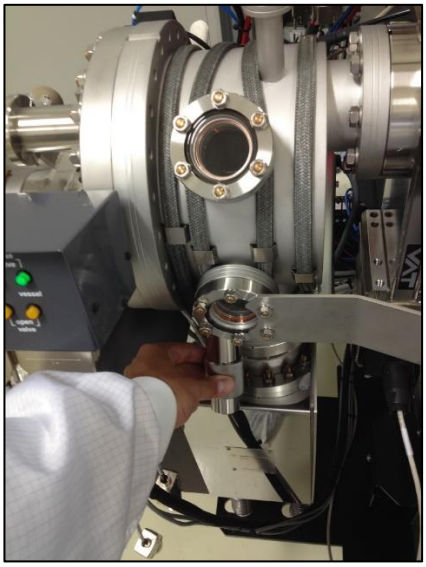


Figure 287: Rotate the carousel by turning the crank

- Lock the shuttle by closing Vessel shuttle locker.
- In order to preserve the analysis chamber vacuum quality, it is better to check that the vessel pressure is no more than x10 time worse than the analysis pressure level. If the pressure in the Vessel is over 10^{-8} mbar, a security will prevent the valve between vessel and analysis chamber from opening.

Note: It is always possible to transfer at a higher Vessel pressure by switching the vacuum program to manual mode (see chapter 0 to force the valves open, but it is **not** recommended. For studies (such as hydrogen isotope studies) where a low pressure is critical, it is best to leave the samples to degas in the storage chamber for at least 12-24 hours, hence, to plan measurement sessions in advance.

- Make sure that there is no sample on the analysis sample stage and that the sample stage is in the loading position: In the *Navigator* window, go to Holder menu: Unload → Analysis (Figure 288).

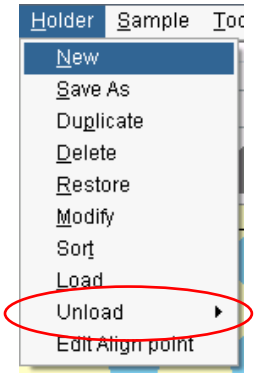


*Figure 288 : Moving the sample to the "unload" position by clicking on "**Unload → Analysis**"*

- Move the Vessel/Analysis chamber transfer rod to position B (
- Figure 268) and attach the shuttle by moving the ring from "unlock" to "lock" position (Figure 286).
- Open the vessel valve by pressing vessel 'open valve' on the Airlock control pad (Figure 281). Wait until the 'open' light stays on (yellow).
- Pull the Vessel sample locker up to release the sample.
- Move the Vessel/Analysis chamber transfer rod to position C (
- Figure 268) and release the shuttle by moving the ring from "lock" to "unlock" position (Figure 286).
- Move Vessel/Analysis chamber transfer rod back to position A (
- Figure 268).
- Close the Vessel valve by pressing the vessel 'close valve' on the airlock control pad (Figure 281). Wait until the 'close' light stays on.
- On the Navigator, click on "Holder" and "Load" (Figure 289).

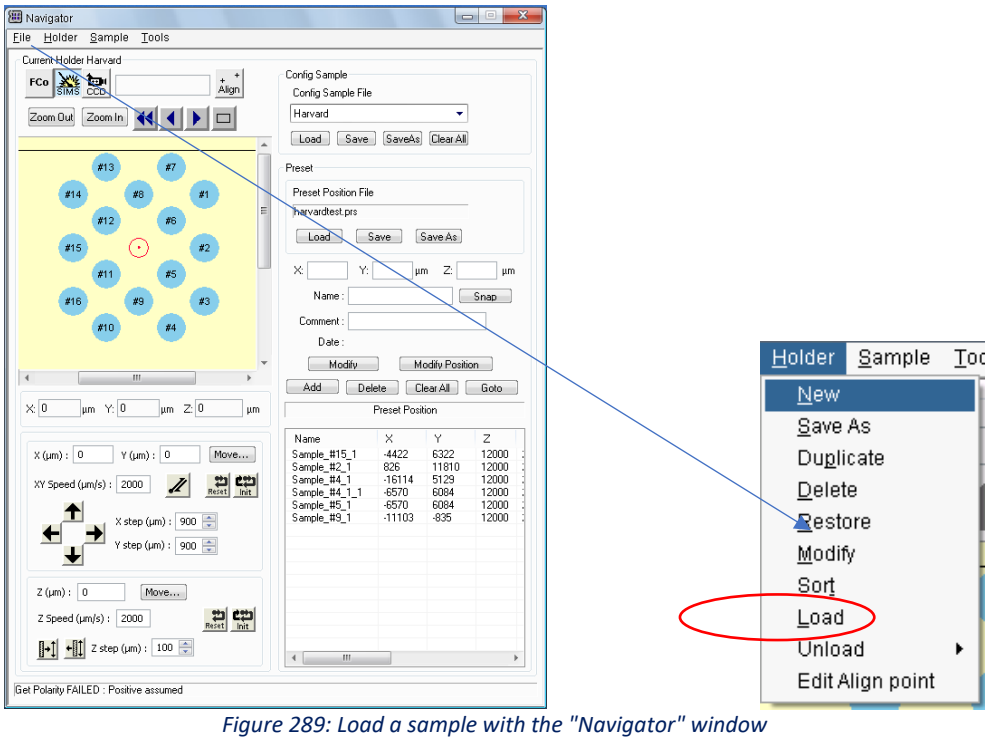


Figure 289: Load a sample with the "Navigator" window

- Select in the Holder→Load window, the type of sample holder which is introduced in the analysis chamber (Figure 290). The synoptic will display a schematic of this holder type.

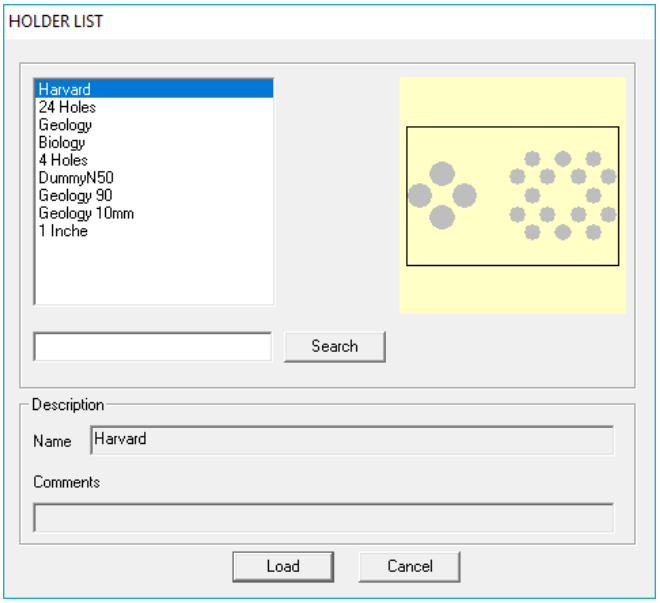


Figure 290: window allowing to choose the sample holder type introduced in the analysis chamber

9.1.1.5 Unloading samples from the Instrument

The procedure for unloading samples is analogous to the loading procedure described above, only backwards.

9.1.1.5.1 Sample transfer: from Analysis chamber to Vessel

- Check the vessel vacuum level. It must be less than 10x times the analysis chamber vacuum.
- In the Navigator, click on Holder > Unload > Analysis to place the sample holder in transfer position.
- Open the Vessel valve by pressing 'open valve' on the airlock control pad. You should hear the valve open, and the 'open' light for the vessel should turn on.
- Move the Vessel/Analysis rod all the way until it reaches the sample (position C). Rotate it to put it in "lock" position.
- Pull back the rod to position B. On the side of the rod, a little knob should rise and fall as the rod reaches position B. (Figure 291)

Figure 291: knob on the side of the rod indicating when the rod reaches position B. When the user pulls back the rod, the knob will rise (left) then fall back into its position (right), indicating the rod is in position B.

- Insert the shuttle locker to prevent the shuttle from moving. If the locker doesn't fall all the way, it means the rod, thus the sample, is not in the right position (Figure 292).

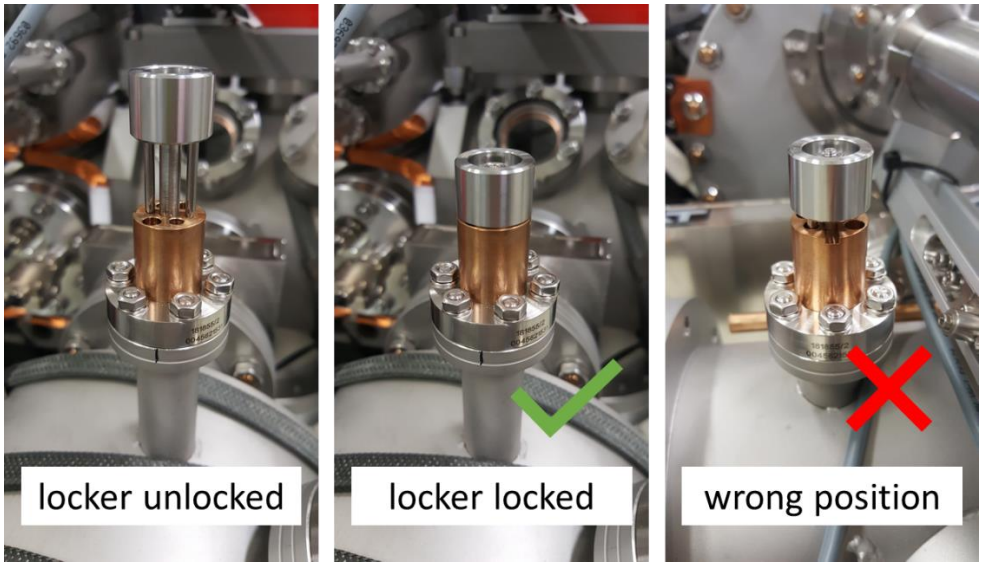


Figure 292: shuttle locker positions.

- When the lock is inserted, rotate the rod into 'unlock' position and pull it back all the way, to position A.
- Close the valve between the analysis chamber and the vessel by clicking on 'close valve' for the vessel.
- If you wish to rotate the carousel inside the vessel, pull up the lock that will release the shuttle.

9.1.1.5.2 Sample transfer: from Vessel to Airlock

- If you want to preserve the storage vessel vacuum quality, check that the airlock vacuum is better than 5 E-7mbar.
- Rotate the carousel so that the sample you want to transfer is in bottom position, facing, vertically the Airlock/Vessel rod.
- Open the valve between the airlock and the vessel by clicking on the airlock 'open valve' button on the airlock control pad.
- Push the Airlock/Vessel rod all the way until it reaches the sample (position C). Rotate the rod to put it in "lock" position.
- Pull back the rod to position B. On the side of the rod, a little knob should rise and fall as the rod reaches position B (Figure 291).
- Insert the airlock shuttle lock to prevent the shuttle from moving and push back the rod all the way to position A.
- Close the valve between the vessel and the airlock by clicking on 'airlock close valve' on the airlock control pad.
- If you wish to retrieve the shuttle, vent the airlock by clicking on 'vent' on the airlock control pad.
- When the airlock is vented, unscrew the door and pull it out gently to retrieve the sample.

9.1.2 Source start-up and shutdown

9.1.2.1 Cs+ primary ion source start

If the instrument is equipped with a Oxygen source:

- Check that the oxygen valve on the RF source is closed or that the Oxygen bottle is closed.
- If Oxygen gas has been introduced in the source during a previous session, pump the gas by clicking on GASLINE > START in the vacuum synoptic window (Figure 293).

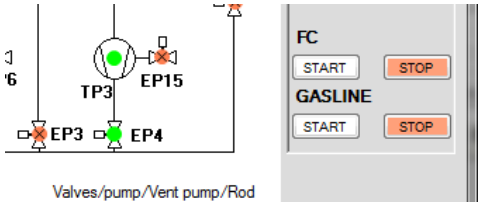


Figure 293: Launch the pumping of the Oxygen line by clicking on GASLINE > START

Procedure to start the Cesium source:

- The cesium source water cooling must be running and the source vacuum must be below the value set in the setup (1.10^{-5} mbar) otherwise the source will not start.
- In the TUNING window (motorization section), choose a diaphragm D1-2 or D1-3 and remove (position 0) all other slits and diaphragms such as this configuration (Figure 294):

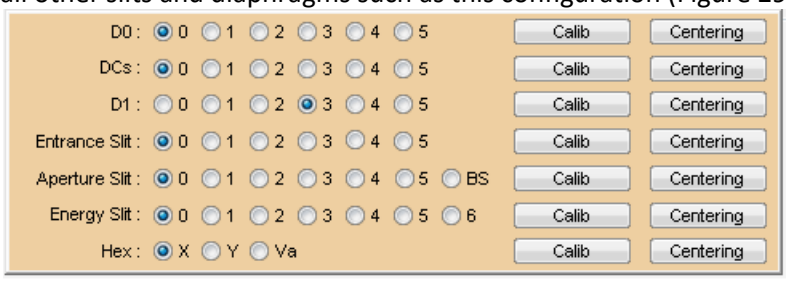
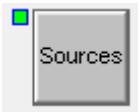


Figure 294: slit and diaphragm configuration when starting the Cs source.

- In the "BOARD" select 'Sources' and wait for the window to open 

.
- In the 'Sources' window, the polarity should be positive and 'Cs' should be selected.
- All three values for Cs (Ionizer, Reservoir, and HV) should be at zero (otherwise the Cs source is already on).
- Click on the Start button (green arrow, Figure 295).

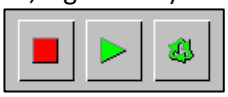


Figure 295: stop and start button for all sources

- A box opens with three values corresponding to the Cs source start-up values: Ionizer, Reservoir and HV (Figure 296). If an 'isf' setup file has been loaded earlier, these values are different from zero. Otherwise, the three values are equal to zero. In any case, it is possible to change the values before starting the source. Usual values are: Ionizer= 1.8mA, Reservoir= 0.2mA and HV=8000V.

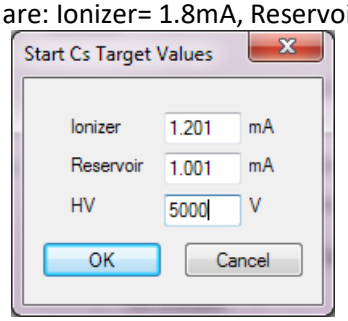


Figure 296: Window to define the target values during the starting process of the Cs source.

- Once the three parameters are entered, start the Cs source by clicking on "OK".
- In the next pop-up window, enter the number of hours until you want the Cs source to start. This is useful if you go through this whole routine the evening before you want to use the Cs source.

You can set a delay, so that everything is up and running by the time you come to the lab in the morning. If you want to start right now, enter nothing or 0:00 and continue.

- The Cs source should now slowly start. The HV will go to the set value in about one minute. Then the ionizer current (i.e. the current of electrons bombarding – thus heating – the tungsten source ionizer) will gradually increase, which will also take one minute. Next comes a waiting time of 10 minutes after which the reservoir current (the current of electrons bombarding – thus heating – the cesium carbonate reservoir) will be turned on gradually (also 1 minute).
- The Ionizer and Reservoir lights on the electronic cabinet should then be ON (green light, Figure 297).

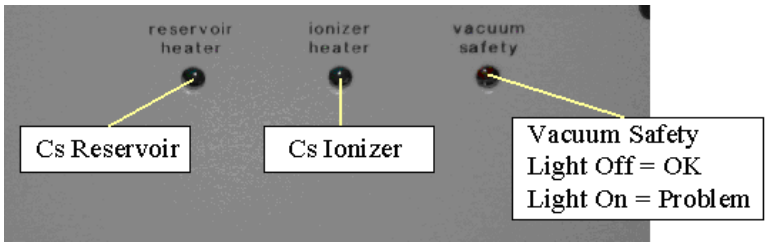


Figure 297: Front face of the Cesium chassis

- In the TUNING window, select 'Detection Mode: FCp'. (the light on the FCp button ("CFp") on the keyboard should be ON 

). You should see the primary beam current slowly increasing.
- A typical value for the Cs+ beam current measured in FCp is between 30 and 100 nA for HV = 8kV. A current below 30 nA is considered too low.
- Monitor the source ramping up and stabilization. Typically, the beam reaches a stability good enough for use after 30 minutes. You can use the "Beam stability" option in the TUNING to monitor the ramping and stabilization.

It is a good practice to check and note from time to time (every week for example) the ratio of beam currents FCo/ FCp, for a given primary column configuration (diaphragms, voltages). It should be stable and any noticeable deviation should be investigated and corrected/compensated: cleaning of the extractor, increasing of reservoir current,… Depending on usage, typical Cs ionizer lifetime is around 3-4 months.

9.1.2.2 RF-Plasma primary ion source

Do not start the RF-Plasma source if the Cs^+ source has been turned off less than 45 minutes before. This could severely damage the Cs^+ source as Cs^+ is very reactive causing oxidation and contamination in the primary column.

In the BOARD, open the SOURCES window, select the O^- source. The source polarity is switched automatically to negative. Starting on software version 4.5, it is also possible to use the instrument in O^+/O_2^+ mode. In that case, you can select "O_2^+". The source polarity will switch automatically to positive.

Note: It is preferable to turn off the high voltage prior to switching polarity. To do so, click on the HV button on the keyboard to turn the green light off.

In order to use the source the RF chassis must be ON and "enable" (Figure 298). The power supply is then controlled by CAMECA software.

Figure 298: RF source chassis

Open the "Serial Server" program from the BOARD and click on "Show Console". A message "RFGen connection OK" must be written. This means the communication is ok between the source and the electronics. If this is not the case, click on "reconnect" to reset the connection.

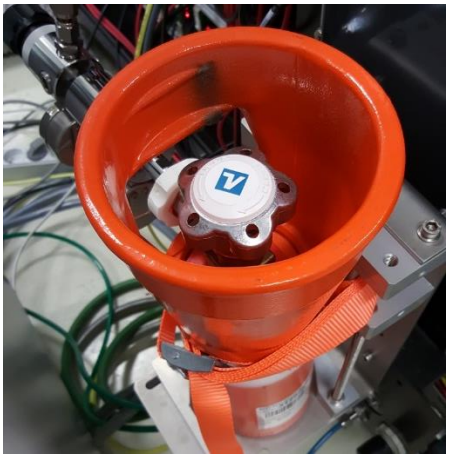


Figure 299: oxygen bottle, located at the rear of the NanoSIMS

Make sure valve EP14 is closed and open the oxygen bottle (Figure 299) to release oxygen in the pipe then close the bottle. If the source hasn't been used for over a month, pump away the residual oxygen by clicking on "start" below "gasline" in the vacuum window (Figure 300). Let the pumping process run.

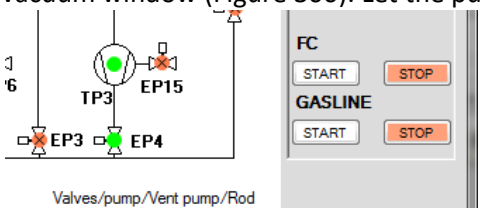


Figure 300: Launch the pumping of the Oxygen line by clicking on GASLINE > START

Once it's done and the valves are closed again, open and close the oxygen bottle again to release a new dose of oxygen. Note that if you intend to use the O source for a while (> 4 days), it is better to leave it open. Or even to let the ion source running permanently.

Gradually open counterclockwise the grey knob (the leak valve EP16) on top of the Hyperion source to reach the vacuum value needed to start the RF source, typically between 2 E-6 mbar and 1 E-5 mbar of oxygen. The vacuum value can be read directly on the gauge display (cabinet B) or on the vacuum synoptic (however, be carefully that the Vacuum synoptic only refresh every 30s).

Regularly check the source vacuum value. If it's too low or too high, the source will not start or will shut off.

Note: some operators always close the leak valve after source usage. They re-adjust completely the leak valve at each new use. Other operators prefer to leave the leak valve open and close the bottle valve instead. Then, they just repump the gas line when not using the O source and fill the gas line again at the next session. But in both cases one will probably have to increase slightly the gas at start, then reduce it

slightly: the source leak valve flow is varying (increasing) as the source (and valve) temperature is increasing, until it stabilizes (within ~half an hour).

Check the HVs are ON on the keyboard. 

From the "BOARD" open the 'PRESET' window 

(Figure 301).
Note: For more details on Presets definition and use, read chapter 5.5.2.

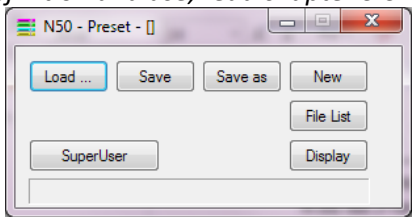


Figure 301: Preset window

If no file has been loaded yet or if you want to use a file different from the one already loaded, click on 'Load'.
Choose the needed '.isf' file within the list (Figure 302):

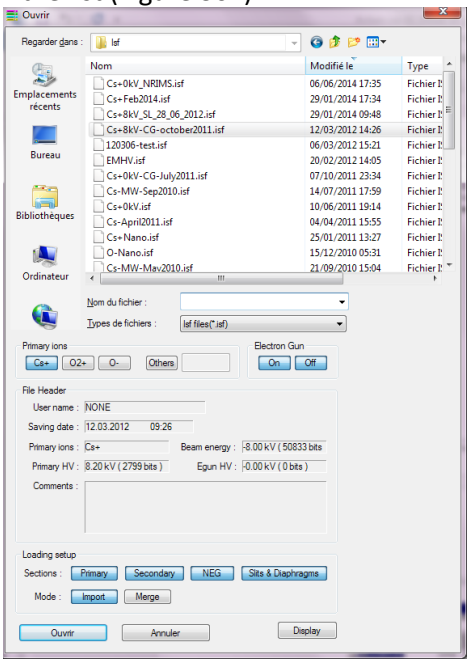
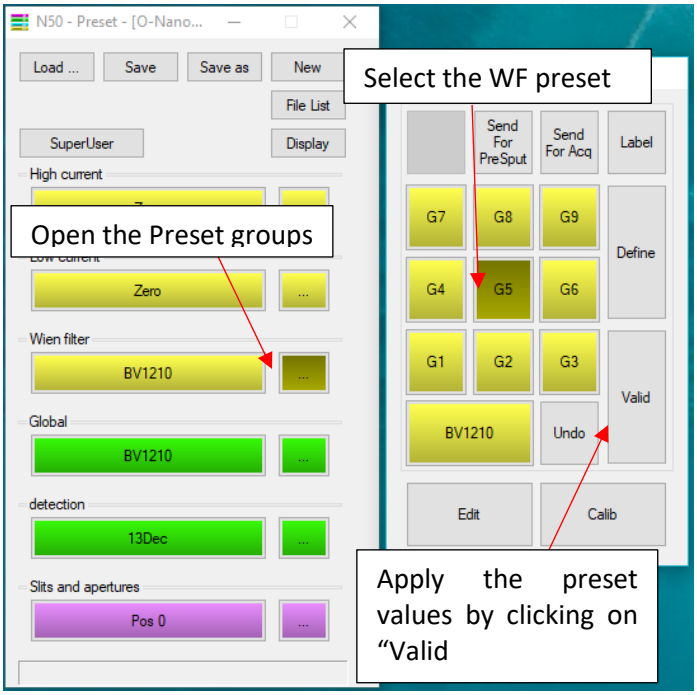


Figure 302: Preset filtered list which can be selected

Click on OK to open the file.
Open the **Wien Filter Preset** groups (click on "…" colored key on the right) and select the Wien Filter Preset. Click on 'Valid' to apply the values. This will send all the Wien Filter settings from this file to the dedicated keyboard of the NanoSIMS (and thus to the instrument itself).



Position the Cs/RF-Plasma goniometer switch in the usual O^- source position (usually around 3.4).
In the TUNING window (motorization section), select D0 diaphragm (often D0-2) and D1 diaphragm (D1-2 or D1-3) and remove (position 0) all other slits and diaphragms (*Figure 303*):

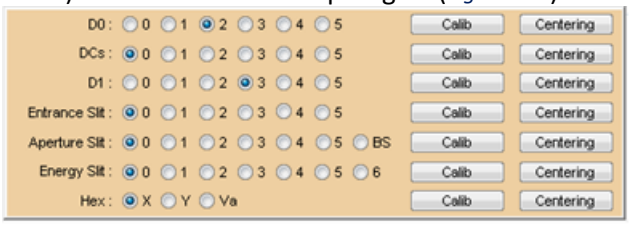


Figure 303: diaphragm and slit configuration when starting the O^- source.

Go to the "SOURCES" window (*Figure 304*):
Make sure the RF source is "enable" and click on the start button (green arrow inside the O- part).

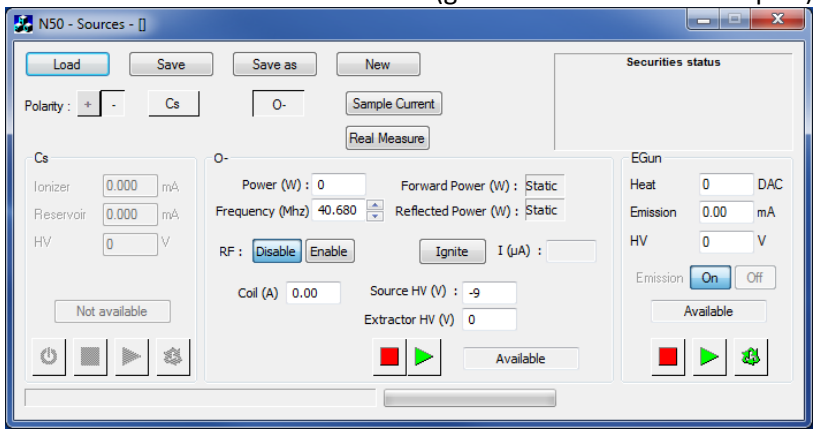


Figure 304: Source window in RF-Plasma source mode

A process window pops up.
The source will progressively be turned on, via six successive steps. Coil, power and frequency values will progressively be applied, as defined in the Setup (chapter 5.9.12.2). Depending on the configuration stored in the setup the starting can be semi-automatic or automatic.
In automatic mode, the process jumps to the next step once the previous one is successful.

In semi-automatic mode, the operator has to click the “continue” buttons for the process to continue to the next step.

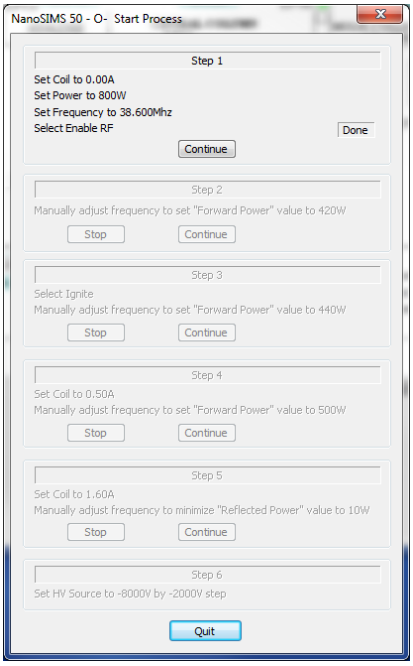


Figure 305: RF source starting window

At the end of the process, the Source window shows the “Power” (target value) at 800 W. The optimum Frequency is usually around 40 MHz, the coil value is set at 1.1A. If the source successfully started, the forward power should be of 800 W, the reflected power should be less than 10 W. You can manually decrease it by adjusting the Frequency, either on the SOURCE window (Figure 304) or directly on the RF source chassis. (Figure 298)

Make sure the beam is in the primary FC 

(the light on button ‘CFp’ on the keyboard should be on).
Typical values of the FCp current with the RF source on are around 200 nA with D0-2
Monitor the source ramping up and stabilize. In only takes a few minutes for the beam current to reach the order of 200 nA, though it might take up to 30 min for it to be properly stable. You can use the “Beam stability” option in the TUNING to monitor the beam increase and stabilization. Adjust the source leak valve (grey knob) in order to keep the pressure in the source chamber to its optimal value. The ideal value is the one giving at the same time a stable beam current on the sample and the highest beam density (beam current in a given spot size) which is approximated by the highest beam current in FCp.
It is a good practice to check and note from time to time (every week for example) the ratio of beam currents FC0/ FCp, for always the same primary column configuration. It should be stable and noticeable deviation should be investigated.
For optimization of the primary beam current, all the parameters might need a slight adjustment depending on the selected ions (O-, O2-, O2+).

9.1.3 Automatic Source Shutdown & Restart

9.1.3.1 Cs+ source Shutdown & Restart

To stop the Cs+ source:
Go to the Sources window and click on the Stop button (red square). A new window opens (*Figure 306*).
Two choices are available. “Stop” or “Standby”. The “Stop” will totally shutdown the source, meaning the three values (Reservoir, Ionizer and HV) will be set to 0 with an internal timed sequence. The “Standby” will

set only the reservoir value to 0 and keep the values for the ionizer and the HV, keeping the source thermalized, degassing but saving the Cs carbonate in the reservoir. It is possible to stop the instrument HVs (lenses, detectors, etc…) by checking the "Stop HV after" box.

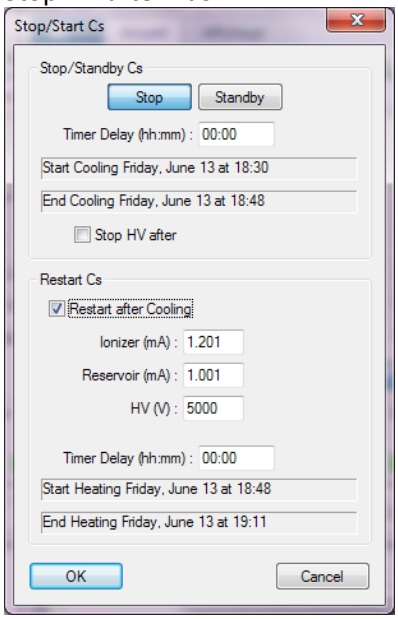


Figure 306: stop and restart the Cs+ source window

Choose a delay time to stop or standby mode (empty = 0.00 = now) and continue. Then check or uncheck the box "Restart after cooling" to decide whether you want to restart the source after it cools down.

a. If checked, then enter the number of hours you want until the source starts up again. *Don't forget that the time needed to cool down the source is not included in the delay.*
b. If unchecked, the source will remain off after the shutdown process.

When all is set, click on "OK". The Cs source should now start the stop procedure: first cooling of reservoir, then cooling of ionizer and finally HV to zero. The overall sequence will take ~half an hour.

For an optimum long term stability when the source is used over several days, it is recommended to let the source run permanently and simply switch it to "STAND-BY" mode with the source HV ON when the source is not in use for intervals of several hours (e.g. nights). In the morning one just needs to raise the reservoir heating current (possibility to set an auto-start). For longer intervals (e.g. week-ends) one can stop completely the source and HV.

Wait ~45min after a Cs+ source "Stop" process before switching the RF-plasma on.

9.1.3.2 RF-Plasma Standby mode

If, for stability reasons, the user wishes to leave the RF source on even though no analyses are running for a period of time of several hours (typically, overnight), we recommend to put the source in standby mode. You can do so directly from the front panel of the RF cabinet, or from the source window of the NS50 software.

Standby mode:

1- Lower the power to 600W.

2- Lower the Source HV and Extractor HV (Set to 0).

Note: the order is important. This will ensure a safe mode for the source and maintain the best source stability when needed.

To restore normal operation:

1- Restore the HV (8000 V under normal conditions), restore the Extraction voltage (4000 V under normal conditions),
2- Rise the power to the usual setting
For longer periods of inactivity (weekends or holidays), we recommend to completely stop the RF source following the procedure below.

9.1.3.3 RF-Plasma source Shutdown, Restart

To stop the RF-Plasma source:
Go to the SOURCES window and click on the Stop button (red square) (Figure 304).
Close the grey leak valve on top of the Hyperion source, and wait for a source vacuum recovery, generally below 5.10^{-7} mbar.
Alternatively, if you do not wish to touch the grey leak valve, close the Oxygen tank and pump the remaining gas by clicking on GASLINE > START in the VACUUM window.

As most users will simply leave the source on for the entirety of a session, there is no special "auto-start" procedure. However, it is possible to start the source with an added delay (see 9.1.2.2 on how to start the source).

9.1.4 Applying Presets

9.1.4.1 Reminder: Setup, ISF, Preset

It is useful to recall the three different notions in the software, to avoid confusion:

The **SETUP** is a file containing the **complete list of** instrument parameters. There is only one Setup file defined for a given instrument. It is thus advised to modify it with particular caution. It is advised not to modify the SETUP unless by expert authorized users and to keep a back-up file with all original parameter values.

The **ISF** (instrument status file) regroups all the presets groups defined for a given **mode of operation** (i.e. Cs^+ mode, O^- mode or O^+ mode). Users can save as many independent ISF files as they want, so as to reload them later.

The "**PRESET**" file is a smaller file containing a **reduced number of specific parameters for a type of adjustment or tuning** (e.g. lens HV value, deflection value, detector HV values…) and **belonging to a complete "ISF"** file. The idea is to reload or save a subset of parameter values with a click, in order to ease the instrument operation.
For example, from one sample to the other the EOS HV value or the Cy deflection value can be different. It is possible to *create* a "Preset" file and choose each parameter value to be in this file (see 5.5.5). Consequently, when the operator sends (applies) such preset files to the instrument, only the short list of values defined by the operator will be modified.

Each "ISF" file contains **several Presets which are groups of a few parameter values**.
Each Preset contains 10 possible groups of values which can be sent to the instrument. The operator can select one group of values to send to the instrument per Preset (Figure 307, Figure 202).

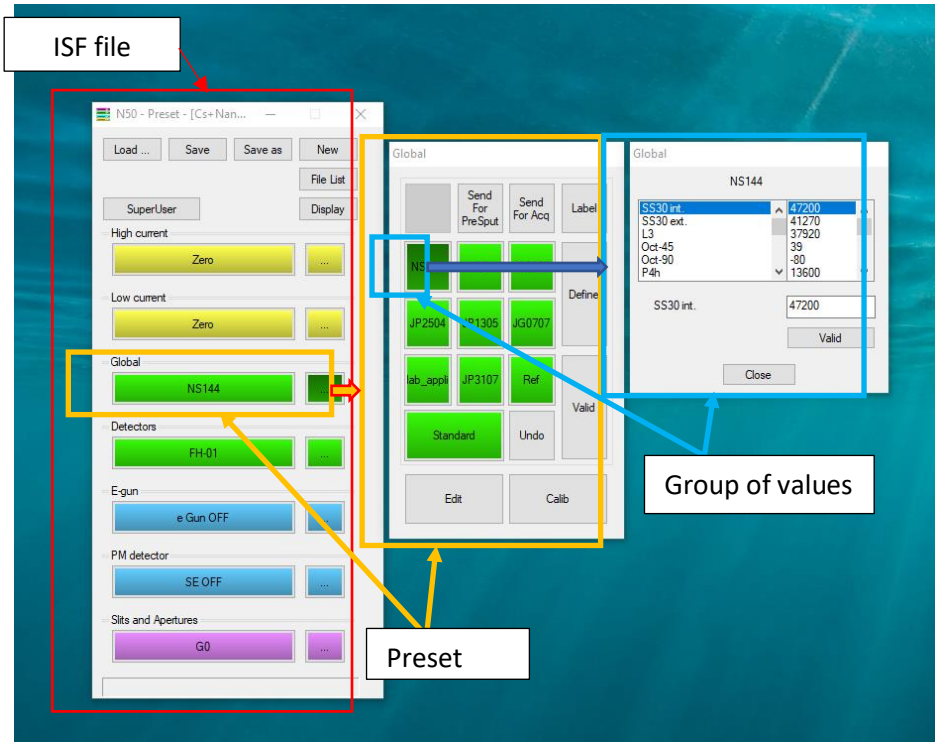


Figure 307: "Preset" files configuration

Each Preset is usually dedicated to a given part of the instrument which is identified by a specific color:

- **Primary beam (yellow)**

Commonly, the primary beam Presets are "**bistable**", meaning by clicking on the long rectangular Preset main button, the operator **applies alternatively one of the** two groups of values: the **standard** value (rectangular button on the bottom left of the Preset window) and the **one** selected among **the nine** other values.

The **ACTIVE** Group of values appears **DARKER within the same color.** For example, when the operator applies a high current to pre-sputter quickly a sample, he can come back to a normal analysis current simply by clicking again on the button (Figure 203).

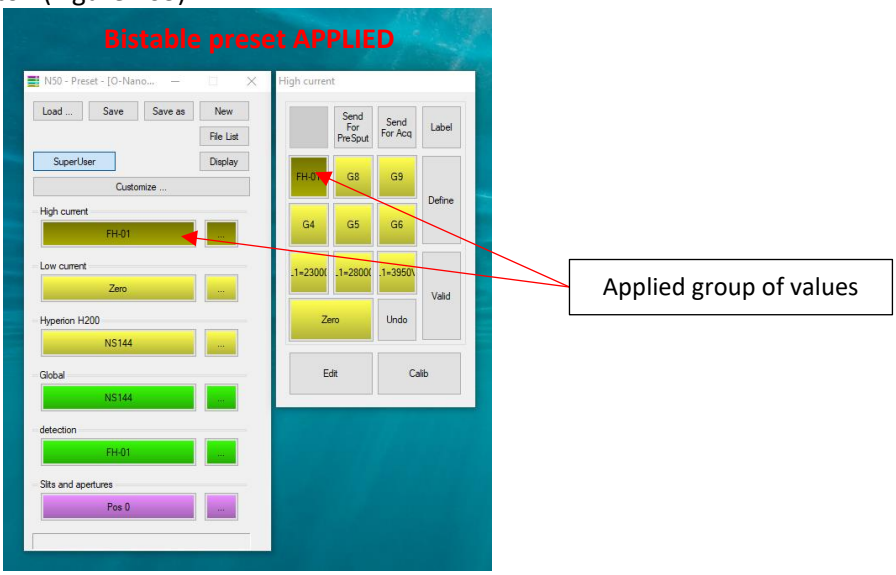


Figure 308: Switch between two groups of values in "bistable" mode

- **Secondary beam (green)**.

Frequently, the secondary beam Presets are "**monostable**", meaning that by clicking on the long rectangular Preset button, the operator **applies** all the value with no turning back.

- **Electron gun (Egun) and Secondary electron detector (SED) (blue**, Cs+ mode only)

Commonly, the Egun and SED Presets are "**bistable**", meaning that by clicking on the long rectangular Preset button, the operator usually **switches** between an **ON** state and **OFF** state. For example, when the operator wants to use the Egun or the SED, they just have to click on the Preset main button and when they don't want to use them, they click again on the button.

- **Slits and Diaphragms (purple).**

This Preset can be "bistable" or "monostable" depending on the analysis requirements.

9.1.4.2 Cs+ mode

For this basic operation description it is considered that the instrument has been tuned before the analysis and the Presets are already stored, ready to be used for the analysis. To send this Presets to the instrument, follow this procedure:

- Check that the HVs are ON on the keyboard 

(green light ON)

- Click the *PRESET* icon in the MAIN taskbar. The icon indicator must now be green 

.
- The following window opens (*Figure 309*):

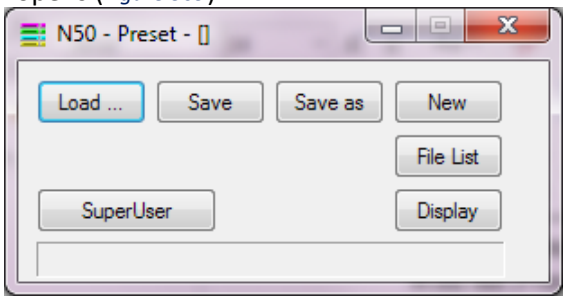
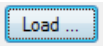


Figure 309: Preset window

- Load an ISF file (see 5.5.1) by selecting the 

button which open the following ISF loading dialog box (Figure 310):

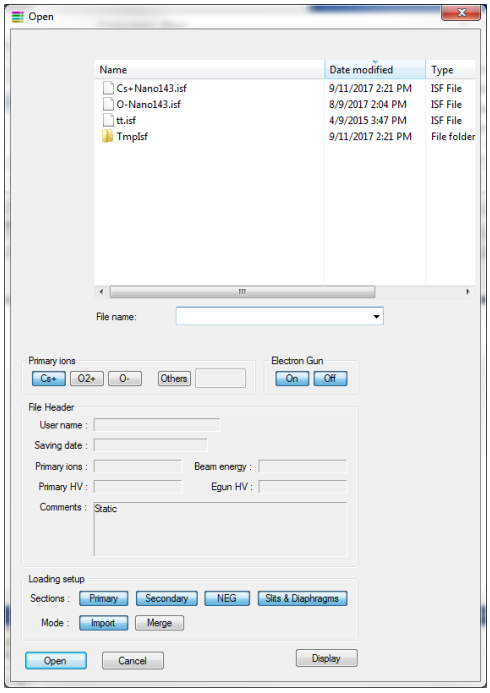


Figure 310: ISF files selection list

- Possibly using the filters available, select the desired file (Cs^+, O^- or O_2^+ depending on your analysis type, so that the correct preset options will be available) and click on *Open*. The following window appears (Figure 311):

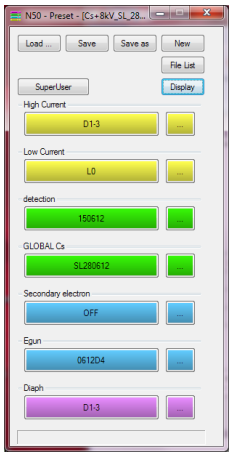


Figure 311: example of a Presets window for Cs^+ analyses with e-gun preset available.

- In the NAVIGATOR window, check that the sample stage is in SIMS mode 

And on the TUNING window, click on the multicollection mode (Figure 312).

Always make sure to be in SIMS and Multi collection modes before applying presets, so that all preset values are properly applied.

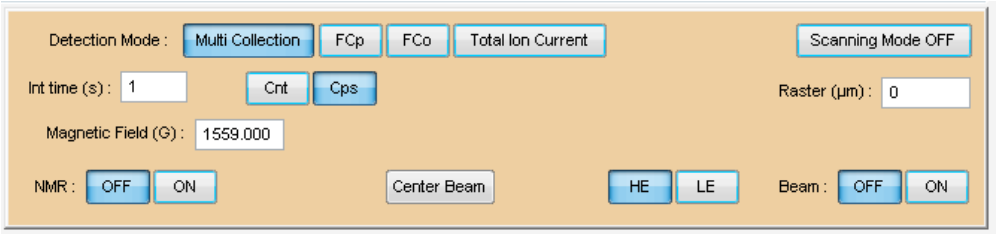


Figure 312: partial view of the tuning window

Come back to the PRESET window and check that all presets you want to apply are ready to be used (Figure 311). If not, click on the right button of each Preset and select the one you want to launch (Figure 313). Select the desired preset and click on "**Valid**". The preset is immediately applied to the instrument and keyboard.

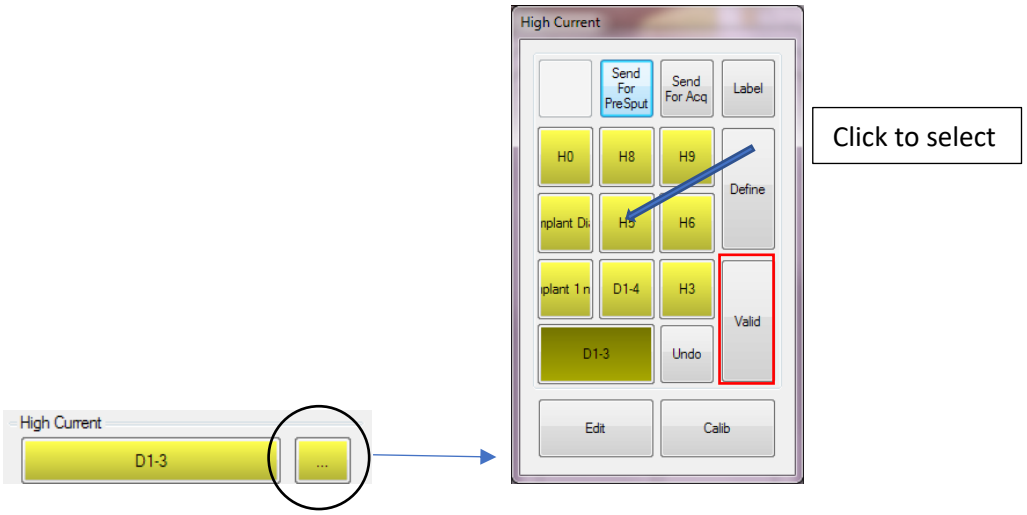


Figure 313: window showing all 10 presets for a given category (here: High current)

Go back to the PRESET window and click at least on the Preset "DETECTION" and "GLOBAL". These files (secondary beam Presets) are "monostable", which means all values are sent with no turning back (Figure 314).

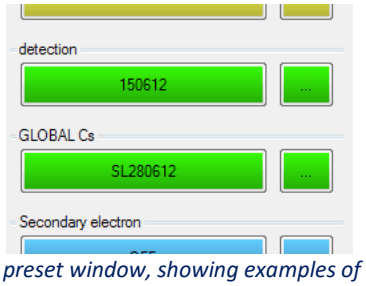
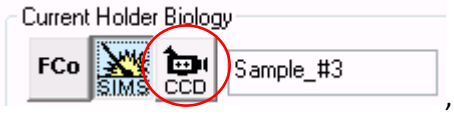


Figure 314: partial view of the preset window, showing examples of Detection and Global presets.

It is possible to check on the keyboard that the parameters have well been applied to the instrument by browsing the buttons (such as EOS, P2P3, Cy, SS100, etc…)

9.1.5 The optical microscope

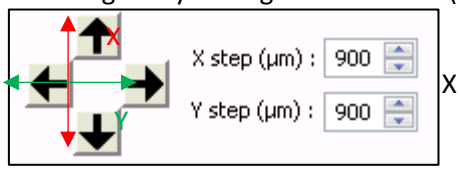
9.1.5.1 Moving the sample under optical microscope (CCD)

After having introduced the sample and selected the type of sample holder in the Navigator window, the sample is already in SIMS position with Z=0. Navigation on the sample holder is usually performed in the optical CCD microscope mode. To position the sample before starting a SIMS analysis:
In the NAVIGATOR window, select the CCD mode. The stage will move automatically to the corresponding CCD position 

, 35mm above the SIMS position in the X (vertical) axis.

- From the "BOARD" launch the "OPTICAL IMAGE" program by clicking the icon 

. A window showing a live optical image of the sample opens. The center of the CCD image, marked by a blue cross, should match the center of the SIMS image (see 9.2.9 on how to set the CD/SIMS offset).
- Adjust the Z position in order to obtain an image well in-focus. External Z steps of 50 to 100 are adequate, equivalent to real sample Z steps of 8.5- 17 µm.
  (N.B: with the mechanical reduction there is a factor 0.17 between external steps and sample steps).
- The NanoSIMS 50L is designed and adjusted to have the same Z (~ 300 µm between the sample holder surface and EOW immersion lens or 400µm between the sample surface and EOW) for a focused image in CCD and SIMS modes. Thus**, if your image is in focus for a Z position in CCD mode, this Z position will be the same for the SIMS mode.** It is the usual method for correct Z-positioning. Note that if you reach the end of the course on Z and haven't reached the focus point, your sample is "too deep". You must then find a better way to mount it, closer to the sample holder's level.
- It is now possible to explore your sample moving the sample holder by clicking on the X and Y arrows using steps that can be adjusted in the editor on the right. (the conversion step/µm, usually 1, can be modified in the Setup>Holder (see chapter 5.9.2)).
- Adjust the X and Y position to center the image on your region of interest (Figure 315).



X and Y position

Z position

Move the sample toward the immersion lens

Move the sample away from the immersion lens

Figure 315: NAVIGATOR window: navigation arrows to move the sample along the X, Y and Z axis.

The sample stage can also be moved in all directions (X, Y and Z) using the thumbwheels of the keyboard by selecting "sample".

Once you have centered the area of interest of your sample in the middle of the optical microscope image (on the white cross), you can go back in the SIMS position with: 

in the NAVIGATOR window

9.1.5.2 Main functions of the *Optical Image* program

Zoom: You can zoom in and out in the optical image pixels by clicking on the magnifying glass icons on the right of the menu bar (Figure 316). The number indicates the zooming factor (1= no zoom, 2= x2, 3= x3). Note that this is a numerical zoom and the more zoomed in you are, the more pixelized your image will be.

Figure 316: zoom in and out by clicking on the + or – magnifying glass icons

Save an image: You can capture an image of the sample via the CCD via the "Camera" menu: File→Save→Image in the "Optical Image" window (Figure 317).

➔ ***For more details about the optical CCD microscope image functions, refer to 5.1***

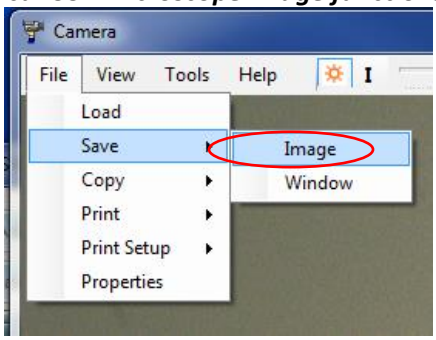


Figure 317: main menu of the CCD window showing how to capture the image displayed by the CCD camera.

There are three distinct crosses to mark positions in the Optical Image program:

- The beam position (blue cross)
- The working position (white cross)
- The user position (green cross)

Display positions: all crosses can be displayed or hidden by selecting (or de-selecting) them in the menu "view".

Edit positions: To edit the position of the blue or the white cross, in the menu Tools, and select "working position adjustment" (for the white cross) or "beam position adjustment" (for the blue cross). To edit the position of the green cross, simply click on the CCD image.

Beam position vs. working position: The use of a "beam position" and a "working position" are inherited from other SIMS, where the "beam position" is the tuning position, used as a reference during session, and from which the current beam position "working position" should not divert too much. This distinction is meaningless with NanoSIMS and the user can choose to use either the beam position or the working position. It is however recommended to keep, for instance, the "beam position" at the center, and use it to do the CCD/SIMS offset calibration (see chapter 9.2.9 for details), and then adjust the "working position" to the exact position of the beam when navigating a sample. Both positions should not drift too far apart. if the working position wanders too far from the center of the image, it is recommended to adjust the SIMS/CCD offset (see chapter 9.2.9 for details).

Labelling a position and saving an image: It is possible to add labels, which can be sometimes useful to mark specific features of an image, or distinguish analysis points, before saving an image (Figure 318).

- In view, make sure "labels" is checked.
- Go to Tools > Label Properties.
- In the *Label* list, Select <new label>.
- In *Text*, type a name.

- In the main camera window, position the green cross (user position) at the proper point.
- Click on “Get User Pos”
- Make sure “visible” is selected.
- You can also choose the label color.
- When all is set, click on “apply”.
- You can then save the image with the labels via File > Save > Window.

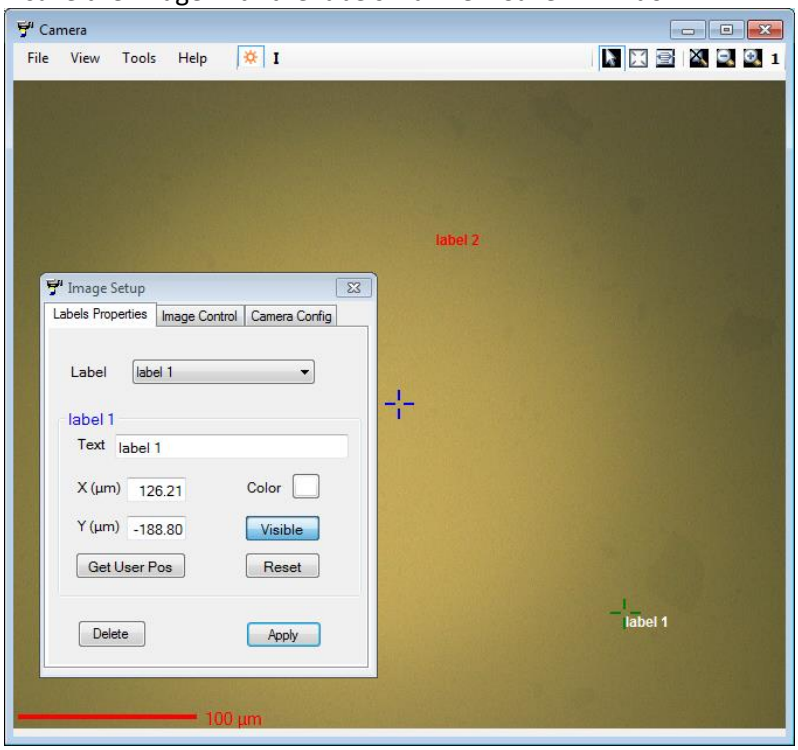
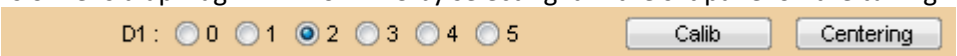


Figure 318: labelling positions in Optical Image

9.1.6 FCp and FCo beam current checking

This step is the same for both Cs^+ and RF-Plasma sources.
Use an immersion lens diaphragm D1-2 or D1-3 by selecting it in the slit panel on the tuning window:



To check if the source and the alignment are ok, it is interesting before starting any analysis to check and write the FCp (Faraday Cup Primary = source current after D0 and DCs) and FCo (Faraday Cup Object = final beam current on the sample). For a given configuration of diaphragms and lenses this ratio should be roughly stable over time. Otherwise there might be an adjustment to perform on the source conditions or alignment.

In scanning mode (Scanning Mode ON in Tuning, Figure 319), with a resolution set to 64x64 px (recommended) the integration time corresponds to the scanning of a complete frame and is fixed to 0.541 s. In fixed mode (Scanning Mode OFF, Figure 320), you can define the integration time for the measurement of FCo (0.5 s or 1 s is recommended).

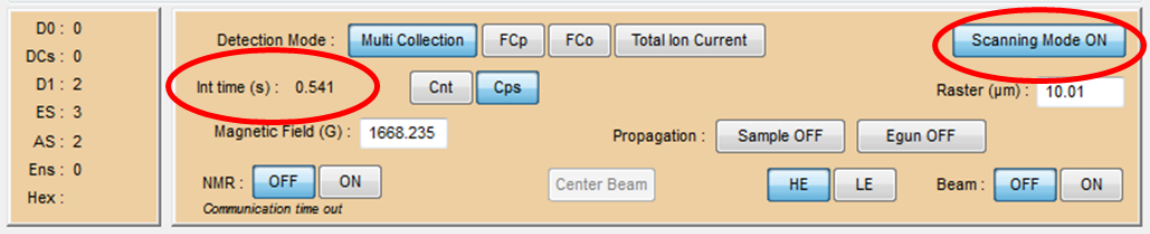


Figure 319: integration time to measure FCp and FCo in Scanning Mode ON

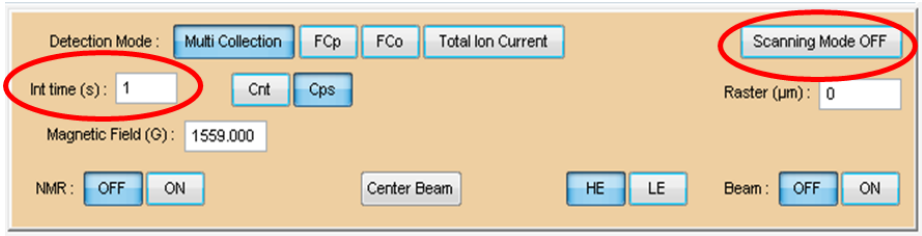
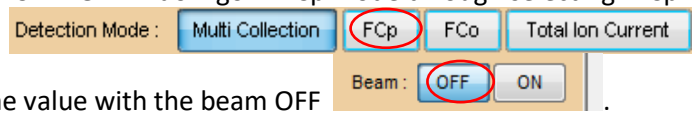


Figure 320: The integration time is adjustable when the Scanning Mode is OFF

For FCp (Figure 319**):**

FCp is a Faraday cup placed in the primary column allowing to measure the primary ion beam current emitted from the source (after D0 and DCs). This value can be read **when the primary ion beam is OFF** (= deflected into FCp = blanked).

- In the "TUNING" window go in FCp mode through selecting "FCp" key:



- Read the value with the beam OFF .

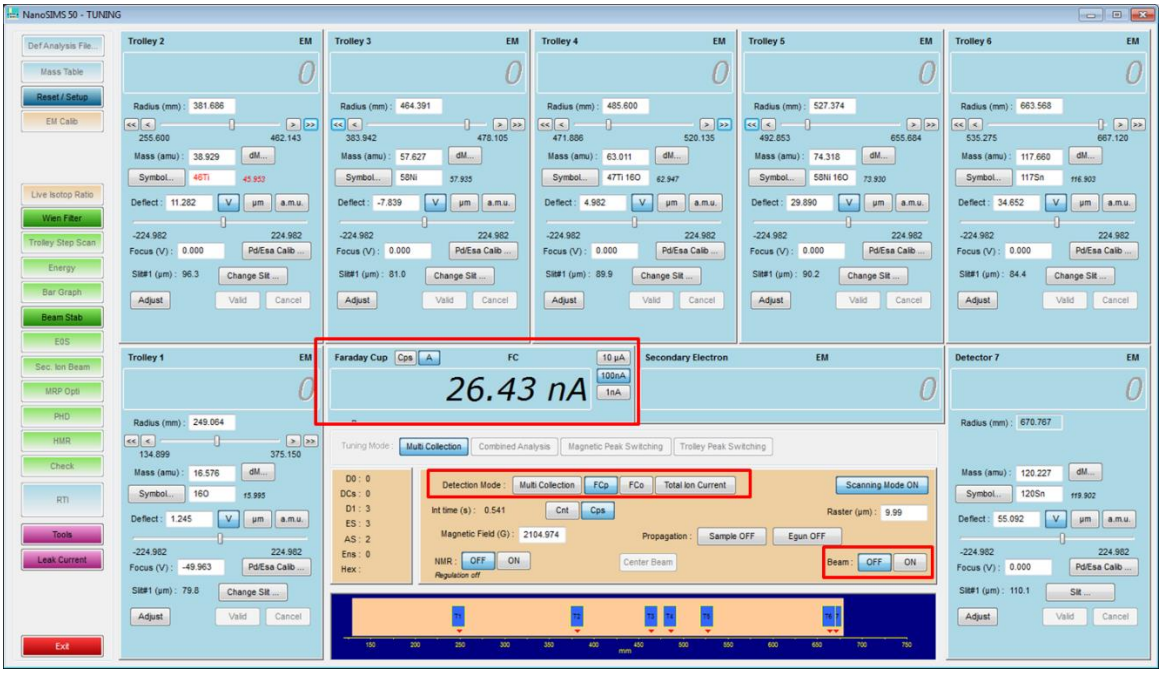
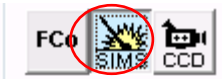


Figure 321: Reading the primary beam current intensity in FCp

For FCo (Figure 322**):**

FCo is a fixed Faraday cup placed just *behind* the sample holder allowing to measure the primary ion beam current arriving on the sample ("object"). It is thus necessary to move the stage to the FCo position and refocus the beam into the further Faraday cup hole, adjusting EOW, EOS and EOP.

- In the NAVIGATOR window send the sample to the FCo position by selecting the corresponding key: 

.
- In the Tuning window, the detection mode has been changed to FCo. If not, click on FCo: 

FCo parameters are applied, as defined in the Setup: EOW and EOS are set to zero and EOP is set to 1400 V (for HV source = 8kV) to focus the beam into FCo.

- **Put the beam ON** 

. You can now read the FCo value on the TUNING window. Note that the Faraday cup has to be put in positive mode (click on + in the FC window in TUNING 

) when using Cs^+ or O_2^+, and in negative mode (click on -) when using O^- or O_2^- primary ions.

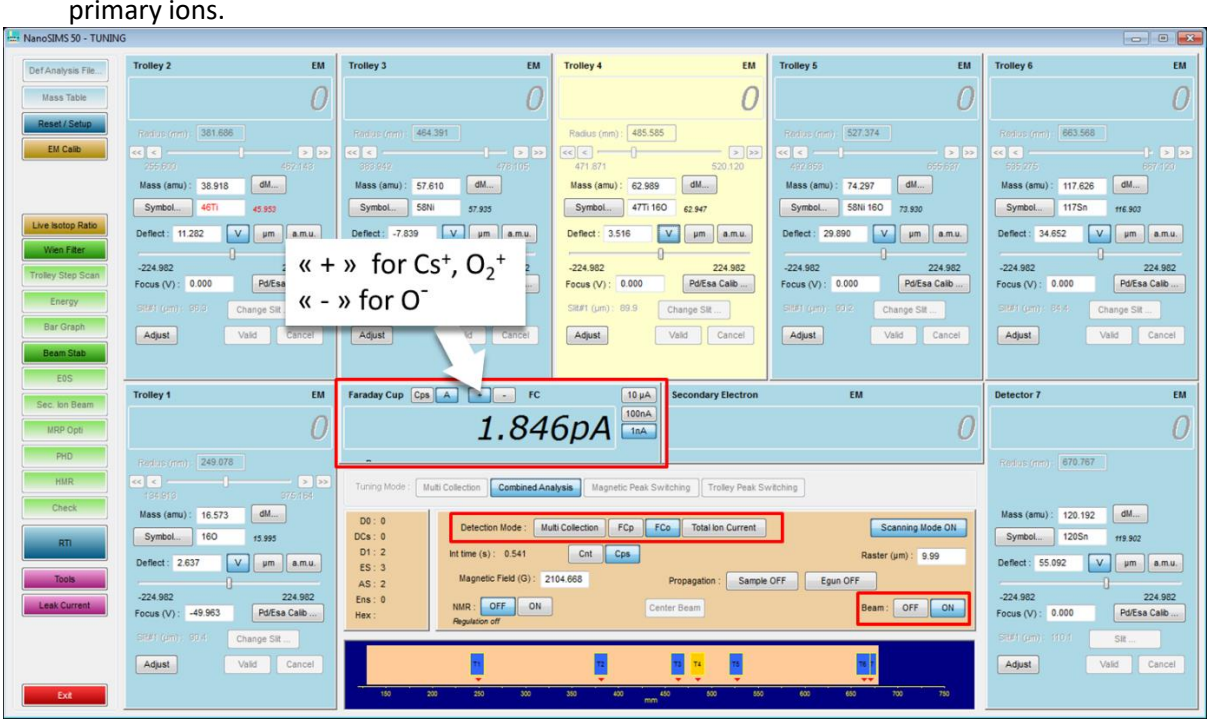


Figure 322: Reading the primary beam current intensity in FCo

*When done, do not forget to put the detection mode back to **Multi-collection in TUNING**, and **SIMS mode in the NAVIGATOR**.*

9.1.7 Diaphragm and slit centering

Because the reproducibility of the motors is not perfect, it is often necessary to check the centering of a diaphragm or a slit when inserting one. This is easily done **manually**, by selecting the diaphragm (D0, D1) or slit (ES, AS, EnS) on the keyboard and **adjusting** its associated X and Y with the thumbwheels in order to obtain the highest possible signal on a detector (which can be, depending on the diaphragm or slit in question, FCp, FCp, the TIC detector or a detector of the multicollection).

It is also possible to use the **automated centering functions**, available via the Tuning window (Figure 323).

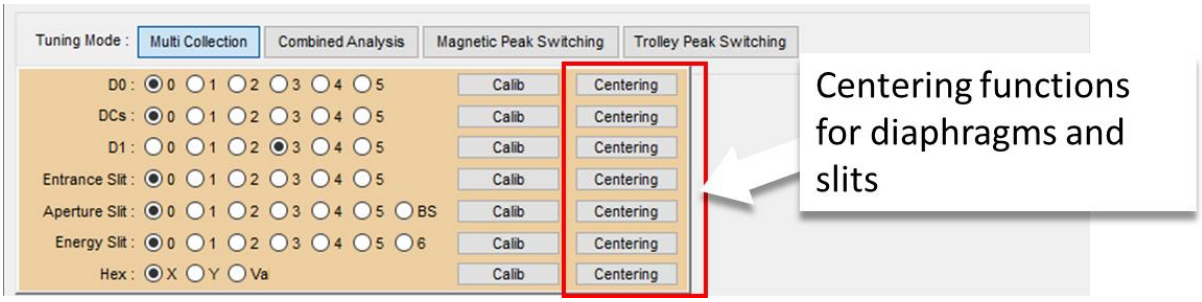


Figure 323: Partial view of the TUNING window, showing the centering functions for diaphragms and slits.

9.1.7.1 Diaphragm centering

The centering window is the same for all diaphragms (Figure 324). For general information on the organization of a scanning window, refer to *Figure 130*.

To center a diaphragm:

1- Select the *horizontal* motor axis.
2- Select the detector in which you wish to read the signal (FCp or FCo if checking the centering of D0, FCo if checking D1).
3- Adjust the scanning parameters. For this, check on the keyboard the current value and set the parameters so that it will be approximatively at the center of the graph. We suggest steps of 500 bits horizontally, and 50 bits vertically.
4- Launch the scan and Apply CL.
5- Repeat the operation with the *vertical* motor axis.

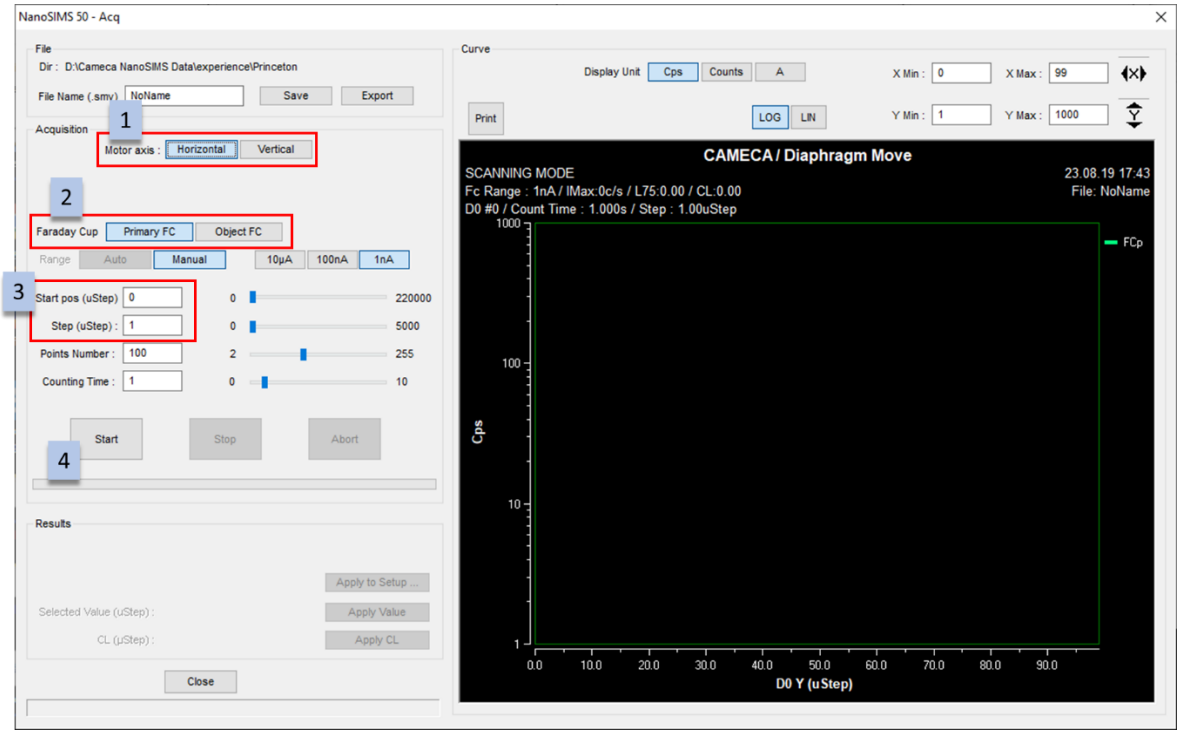


Figure 324: Diaphragm centering window (here D0)

9.1.7.2 Slit centering

The centering window is the same for all slits (Figure 325). For general information on the organization of a scanning window, refer to *Figure 130*.

To center a slit:

1- Select the *horizontal* motor axis.
2- Select the detector in which you wish to read the signal (TIC or Multicollection. If selecting Multicollection, also select a detector).
3- Adjust the scanning parameters. For this, check on the keyboard the current value and set the parameters so that it will be approximatively at the center of the graph. We suggest steps of 500 bits horizontally, and 50 bits vertically.
4- Launch the scan and Apply CL.
5- Repeat the operation with the *vertical* motor axis.

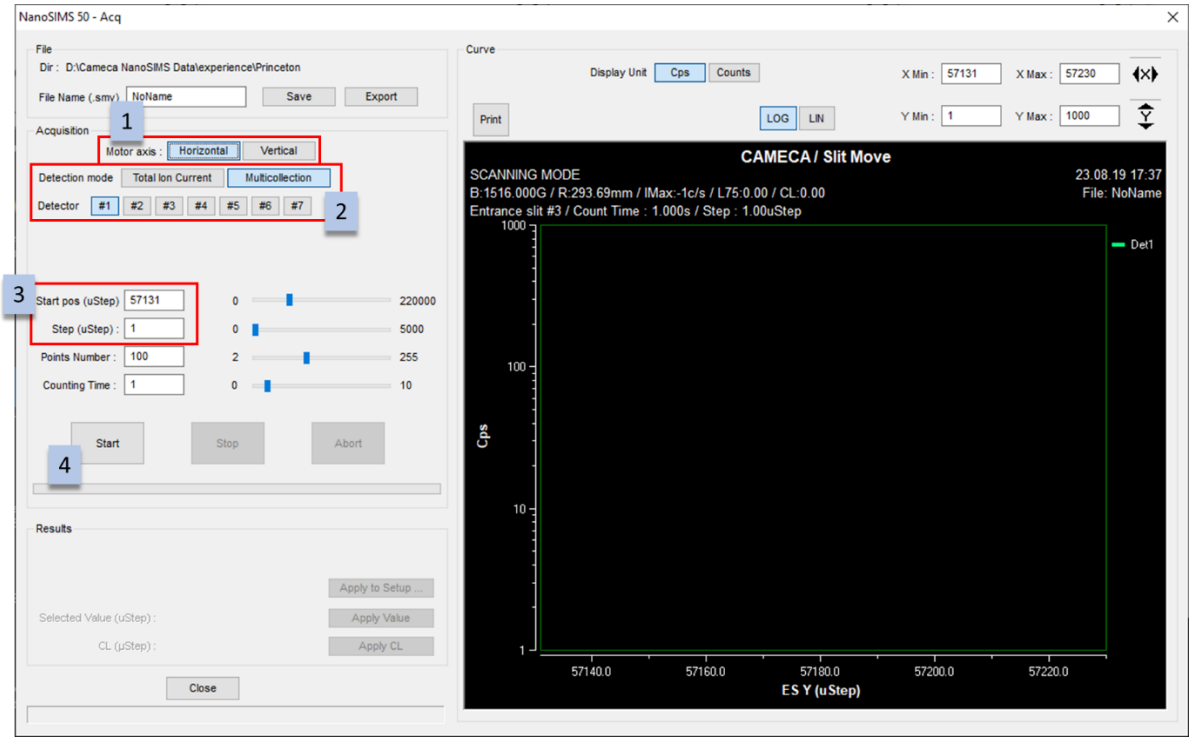


Figure 325: Slit centering window (here ES)

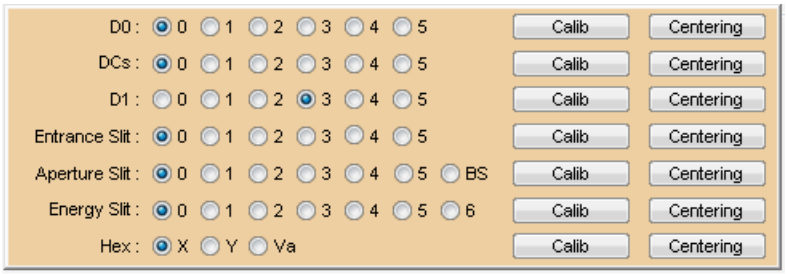
9.1.8 TIC image

The total ion current detector is an electron multiplier (like those of the multicollection) working similarly in pulse counting mode. It detects all, non-mass filtered secondary ions. In order to let the secondary beam reach this detector going through the energy sector, LF2 and LF3 slits are set to zero, ES and AS must be removed and of course SS100 is set to zero.

In the following procedure, the instrument is already aligned and tuned. To make a TIC image, proceed as follow:

- Go to SIMS position: 

(in the NAVIGATOR).
- As an example, choose a diaphragm D1-2 or D1-3 and remove Entrance slit and Aperture slit (select 0 position) like in the configuration below:

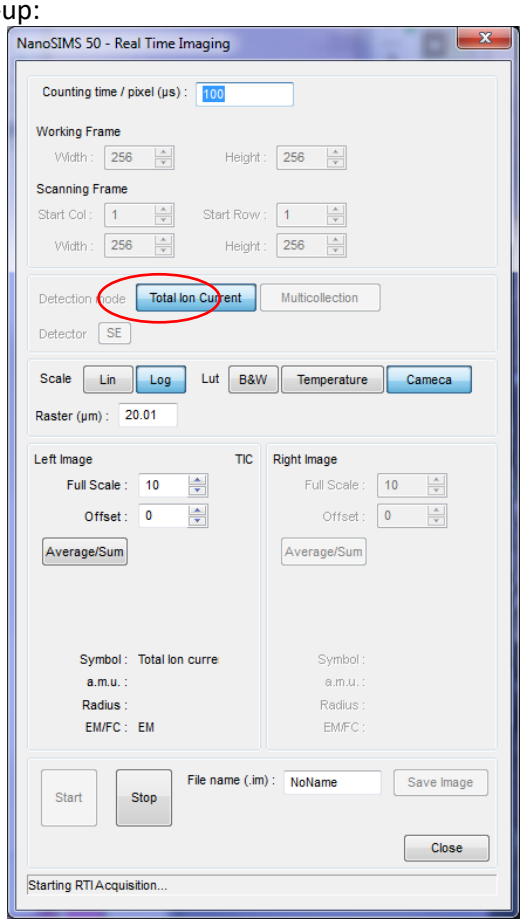


- In the TUNING window
    a. select the “Total Ion Current” (TIC) detection mode:

    b. In TUNING, choose a raster size of 50µm or 60µm.
    c. Click on “Beam ON 

or disactivate the keyboard button CFp (no green light) to turn the beam on.
- You now normally have a signal in the Total Ion Current signal square in the “TUNING” window:
- If the signal is too strong (above 2 000 000 c/s), change the D1 diaphragm to a smaller one (D1-4 or D1-5).
- On the left side of the TUNING window, select the RTI (real time image) button 

- The following window pops-up:



- Choose the counting time per pixel (e.g. 50, 80 or 100 µs/pix)
- Select the Working and Scanning Frames (e.g. 256 x 256 pixels for both)
- Select the “Total Ion Current” detection mode
- Select the desired Y Scale (e.g. linear) and Look Up table (e.g. black and white).
- Select the Raster size (= the field of view), e.g. 50 x 50 µm
- Click on “Start”: a Real-time TIC image of the sample appears in another window (Figure 326).

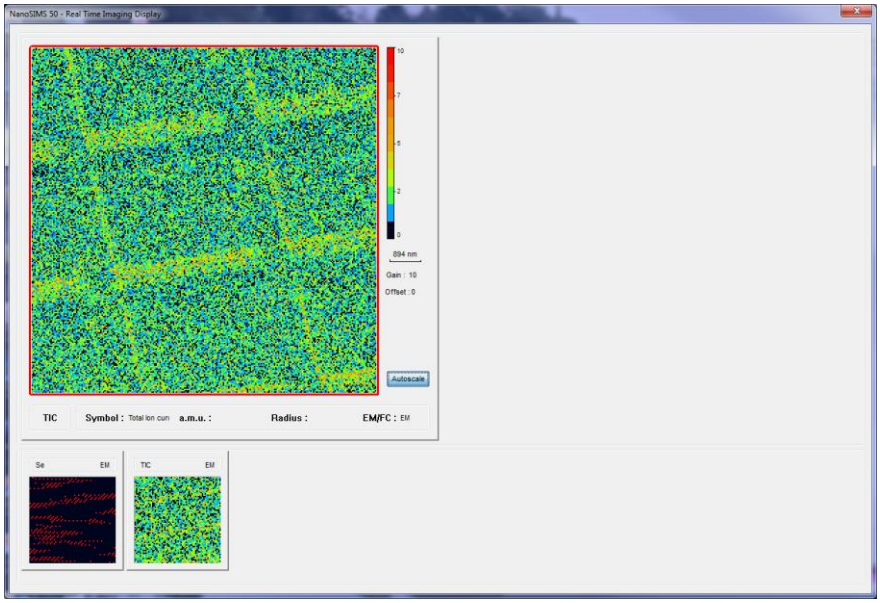


Figure 326: RTI in TIC mode

- The signal intensity will progressively increase with the cesium surface enrichment. You can now adjust the focus with EOP and correct the astigmatism with the octopoles (45 and 90), in order to get a sharper image, using the dedicated keyboard.
- You can now travel over the sample moving the stage to localize an area of interest.

9.1.9 Secondary electron detector (SE)

When using positive primary ions (Cs^+ or O_2^+) ***and without NEG*** e-gun (which is emitting a strong light that would saturate the photomultiplier), it is possible to record images in Secondary Electron (SE) induced by primary ions. Extracted together with the *negative* secondary ions, they are deflected by a magnetic field toward an electron detector, an ensemble made of a scintillator (Sc) and a photomultiplier (PM), located on the side of the central column (Figure 327). The same Be coil used for deflecting primary electrons from the NEG toward the sample is used here to deflect the collected secondary electrons toward the SE detector. The secondary electrons emitted with a few eV initial energy from the sample at E0W potential will hit the scintillator, positioned under vacuum and polarized at ground level with an energy of ~EOW eV, generating light. This light is detected through a viewport and amplified with a photomultiplier positioned at air side made of a photocathode converting light into electrons and subsequent electron dynodes for signal amplification. The signal is detected, like for other electron multipliers, counting the pulses above a discriminator.

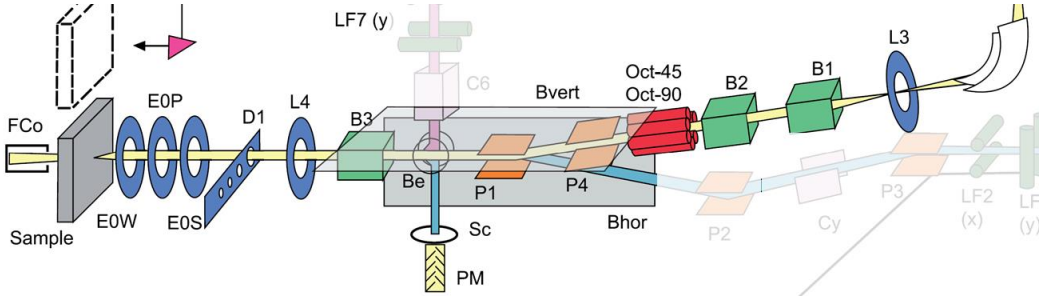


Figure 327: secondary electron detector option

Contrarily to ion images which show composition contrasts, SE images give mostly indications on the sample topography (cracks, grains, edges, cells, holes,…) and secondary electron emission. Topographical contrasts are usually less pronounced in the NS than in a SEM microscope as the collection of secondary electrons is normal to the sample and based on a huge electrical field removing most shadowing.

The SE detector can be used in RTI or in image acquisition. It can thus be a useful complementary tool to localize for example small oxide grains, cracks, etc… or sometime gives nice contrast even from a simple hole, very useful for drift correction.

9.1.9.1 Basic operation

To use the SE detector, apply the SE presets in the PRESET window (Figure 328) by clicking on “SE OFF” to turn it to “SE ON”.

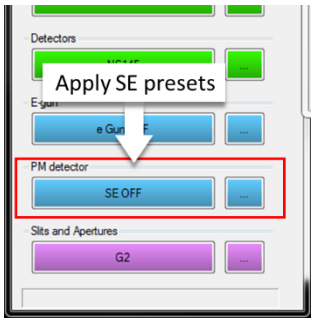


Figure 328: Apply SE presets

RTI imaging: Make sure you are in “multicollection” or “TIC” mode in TUNING. Launch the RTI program and select the “SE” detector. You should now have a secondary electron image.

Image acquisition: it is also possible to acquire an SE image alongside the ion images in an image acquisition. Simply select “SE” in list of detectors in Def Analysis (Figure 329). The SE image will then be acquired in parallel of the ion images during the analysis.

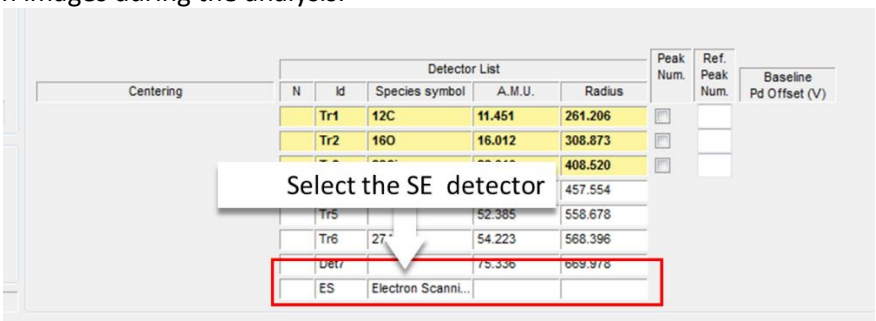


Figure 329: select SE detector in Def Analysis

9.1.9.2 Tuning

The SE image is controlled by three parameters: the e-gun coil current, a photomultiplier (PM) voltage and an offset. Usually, recalling the SE presets (see above) is enough to obtain a good SE image. However, in certain cases, it might be necessary to adjust those parameters. All three parameters are accessible from the keyboard:

- the e-gun Be coil (PM > Coils). Note that this is the coil also used by the e-gun. **The Be coil current value will have a different value for the e-gun and for the SE image**. Adjust the Be coil to maximize the image signal intensity.
- The voltage (PM > PM). The PM voltage controls the image brightness through the gain of the electron multiplier part. You can increase the PM voltage to increase the signal. Never exceed 700 V.

- The offset (PM > Offset). This parameter controls the contrast of the image by added an electronic threshold to the signal. Increasing the offset will decrease the signal. An offset of 0 is usually recommended.

You can save those new settings as a new SE preset in the PM detector group. As mentioned above, the Be coil is common with the e-gun, so make sure to save the SE settings in the SE presets, as to not change the e-gun settings (saved in the e-gun presets).

9.1.10 Pre-implantation

9.1.10.1 Background

It is necessary to enrich the sample surface with reactive species (Cs or O) in order to increase the ionization yield and stabilize the secondary signals to a “steady state” sputtering regime (the **transient regime** primary ion dose will differ from species to species due to their electronic affinity in the case of cesium, or work function in case of oxygen).
Typically, reaching the steady state requires to sputter at least the depth of the implantation range of the primary projectile in the considered matrix. This can be simulated very easily using freeware such as SRIM (J.F Ziegler et al.). The ion range also gives a good feeling of the **depth resolution** that can be achieved, governed in this range by the **atomic mixing** introduced by the ion bombardment. In the Figure 330 below one can see simulations of implantation range of the primary ions in a silicon sample with typical NanoSIMS conditions (normal incidence, Cs+, O-, O_2^{+or-}, Ep 4keV and 16keV)°:

16keV Cs+ (Rp: 16nm) 4keV Cs+ (Rp: 7.6nm) 16keV O^- (Rp: 42nm) 4keV O^- (Rp: 13.4nm).

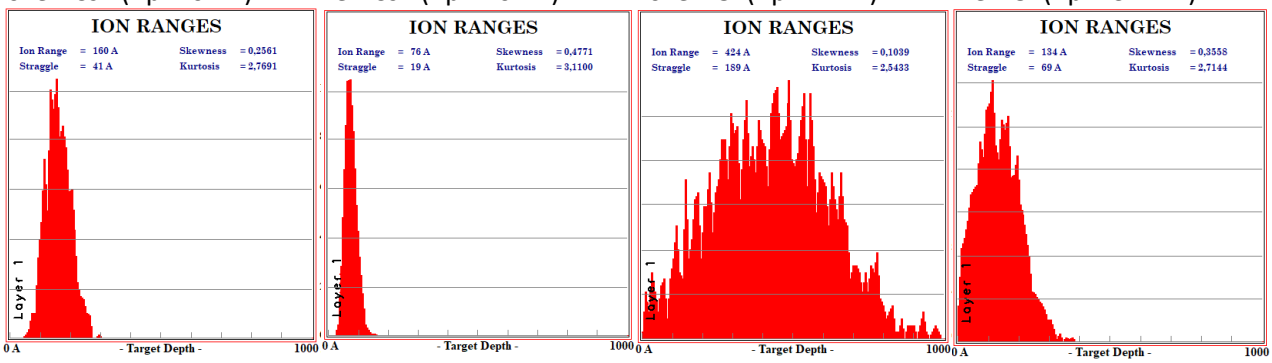


Figure 330: SRIM simulations of implantation depths at various impact energies for Cs^+ and O^- primary species.

Note that 4keV O^- will give same result as 8keV O_2^{+or-}.
This permits the operator to optimize the analytical conditions depending on depth resolution and transient regime depth requirements.

In an embedding resin a typical dose required to reach the steady state is around 5.10^{16} Cs^+ ions/cm^2.
For a field of view (FOV) of 50x50µm, one needs 5 E16 * 50 E-4 * 50 E-4 = 1.25 E12 PI (primary ions). Hence with I (FCp) = 2pA this will require 1.25 E12 * 1.6 E-19 / 2 E-12 = 100 000 seconds !! This is not practical. It is then required to pre-implant with a higher primary beam current: for example, with I (FCp) = 1nA this time will become 100 s, a more acceptable duration in term of throughput. This is described in the two following chapters

Nevertheless, for very small area analysis or depth profiling, or when it is forbidden to sputter around the crater, it is possible just to start the analysis without pre-sputtering: with 2x2µm at 2pA, the approximative time for reaching the steady state becomes 100 000 * (2*2) /(50*50) = 160s. After the acquisition one will take care to avoid the transient regime depth for quantification.

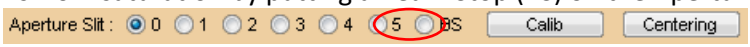
A special case is the need to analyze the very top surface. On the NS50L there are mostly three directions:

- work at a lower impact energy (4 - 16 keV), still compatible with the lateral resolution requirement,
- coat the sample so that the sample top surface will be reached pre-implanted. In such case it is recommended to use a (conductive) coating with similar density as the sample in order to avoid large sputtering yield variations going through the interface,
- *deposit* primary ions at ultra-low energy (ULE: 10-100eV) without (much) sputtering in order to enrich the surface before the analysis and thus reduce the transient depth and optimize sensitivity of the top surface : see chapter 9.2.8 .

9.1.10.2 Simplest pre-implantation using a larger D1 diaphragm

Follow these steps:

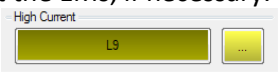
- Scan the beam on the desired Raster size (it is sometime useful to scan slightly larger than the desired subsequent image)
- Protect the EMs from saturation by putting a Beam Stop (BS) on the Aperture Slit (AS):



- Alternatively, you can temporarily increase the value of C4x (200 bits should be enough) to deflect strongly the secondary beam.
- Change the D1 diaphragm to D1-1 (the largest, 750µm diameter) and wait for the desired sputtering. The beam current is proportional to the square of diaphragm diameter. E.g switching from D1-4 (150µm) to D1-2 (300µm) will increase the beam current by roughly a factor X4.
- When the sputtering is finished, go back to the initial values and slits.

9.1.10.3 Pre-implantation with a high current

To implant faster, it is also possible to increase the primary beam current. Two options are available:

- Loading a high current preset:
    a. Put a BS on AS or increase C4x to protect the EMs, if necessary.
    b. Click on the High Current Preset button. 

    c. Change the D1 diaphragm to D1-1.
    d. Start an RTI acquisition in multicollection mode during the sputtering to avoid a pixelized implantation.
    e. When the sputtering is finished, stop the RTI acquisition.
    f. Replace D1-1 with the initial diaphragm.
    g. Click again on the High Current Preset button to stop the high current sputtering.
    h. Remove the AS BS or put C4x back to zero.

Note: During an acquisition, it is possible to load a high current preset automatically just before making an image with the “DefAnalysis” program: see 9.2.3.3

- No preset is ready, and the implantation needs to be done quickly:
    a. In order to protect the EMs put a BS on AS or, if it is not set in your preset, put a strong C4x value.
    b. Change the D1 diaphragm to D1-1.
    c. Increase the value of L1 to increase the primary beam current. (L1 ≈ 10 000 – 15 000 will give an FCo current of a few 10 pA. L1 ≈ 24 000 – 25 000 will give you an FCo current of a few hundreds pA)
    d. Start an RTI acquisition during the sputtering to avoid a pixelized image.
    e. When the sputtering is finished, stop the RTI acquisition.
    f. Replace the D1-1 with the initial diaphragm.

g. Put L1 back to 0.
h. Remove the As or put C4x back to zero.

9.1.11 Multicollection setting

Following the path of this Basic operation chapter you should now have a TIC image and – assuming the alignment of the spectrometer is fine (= has been previously done by an experienced operator) – the next step will be to get a multicollection signal:

- On the Tuning window, select the Multicollection **detection mode**:

- Now, the Tuning window should have this appearance (Figure 331):

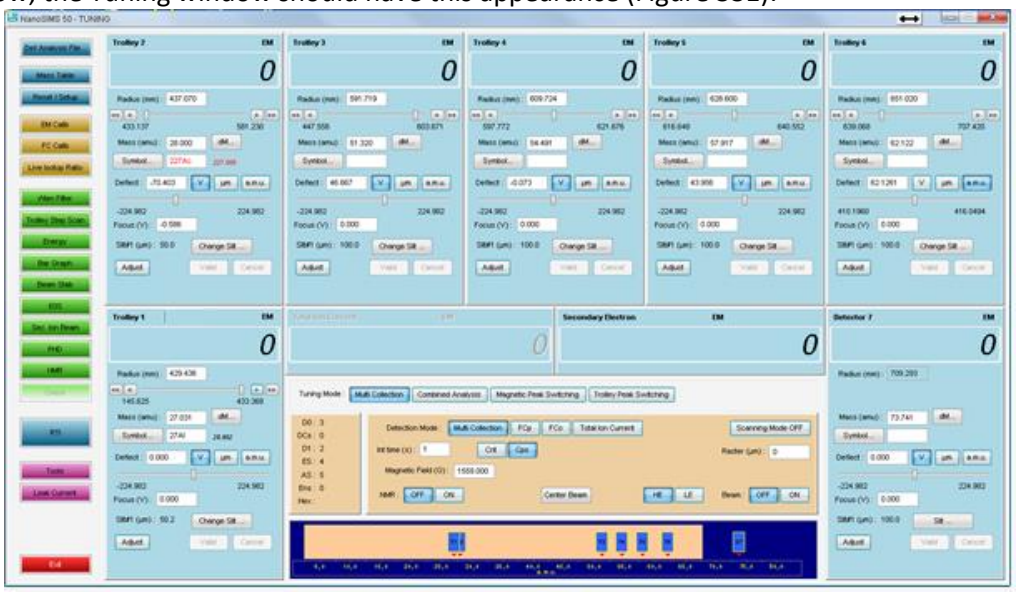
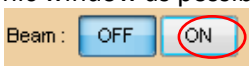


Figure 331: Tuning window in muticollection mode

Note that in order to record fast scanning images, all trolleys to be used must be in EM mode (upper right corner of trolley window). The FC detectors have a too long time constant for fast imaging. It is used for "long" integration dwell-times (seconds, not sub-ms), the signal always coming from the entire scanned area, without ROI or electronic window as possible with EMs.

- Click on "Beam On" 

or deactivate the keyboard button CFp (no green light) to turn the beam on (= deblank it and let it reach the sample).
- Enter the mass (in AMU) of the element to detect on any trolley, e.g 26.982 for 27Al (Figure 332)

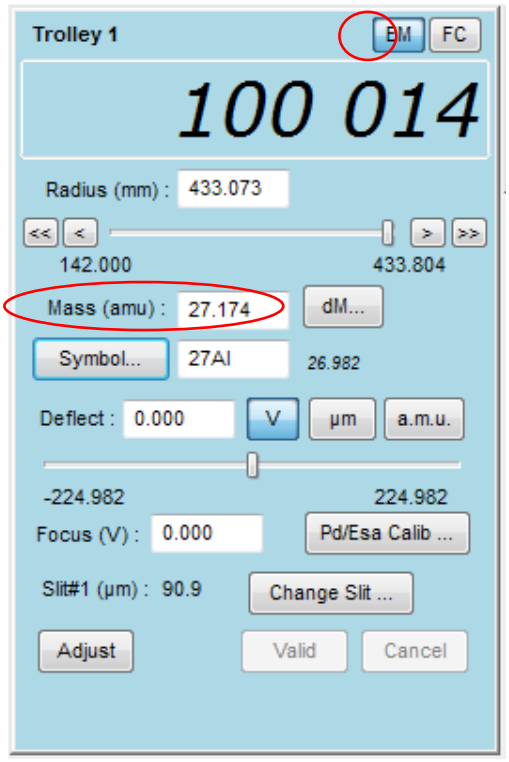
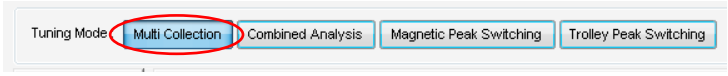


Figure 332: Trolley dialog box here set at the mass 27 to detect ^{27}Al.

- Adjust the B-field with the thumbwheel and the keyboard to have the maximum signal at the top of the trolley window.
- Do not forget to enter the Symbol (e.g. 27Al) of the desired peak; it will appear in the real time display and be stored in the image file.
- Note that the magnetic field/trolley position calibration is not perfect, hence the difference between theorical mass (in the case of 27Al, 26.982 amu) and the actual mass displayed on the trolley (here 27.174 amu) which depends on the calibration.
- If another mass needs to be acquired at the same time, select the **Multicollection Tuning mode** in the tuning window.



Remark: In this Multicollection mode, the trolleys are moving when you select a mass, contrary to the Combined Analysis Tuning Mode where the B-field changes and the trolleys remain at their position.

- On another trolley, enter the mass of the other element to detect (Figure 332). The trolley will move to the position corresponding to the mass asked by the user.
- Move the trolley with the arrows (Figure 333) to finely adjust the trolley position to the maximum of the signal.

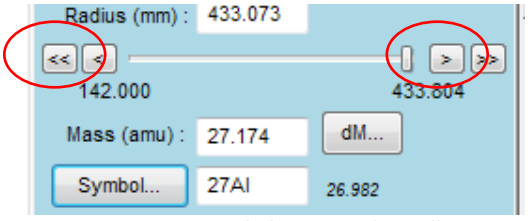


Figure 333: Arrows provided to move the trolley positions

Repeat the maximization for all desired trolleys/masses. Do not forget to enter the Symbol of the desired peak; it will appear in the real time display and be stored in the image file.

9.1.12 Manual EM pulse height distribution (PHD) adjustment

It is advised to previously read the chapter 2.1.4.3.1 explaining the EM and PHD concepts. To check the PHD on a detector, it is recommended to send a strong signal – of the order of several 100 000 c/s and ideally around 10^6 c/s. When checking consecutively several detectors, use a major element (classically $^{28}Si^-$, $^{28}Si_2^-$ in Cs^+ on a Si sample) and change the magnetic field to center the signal on each detector one by one.

In the Tuning window, click on the "PHD" button 

. The following window opens (Figure 334):

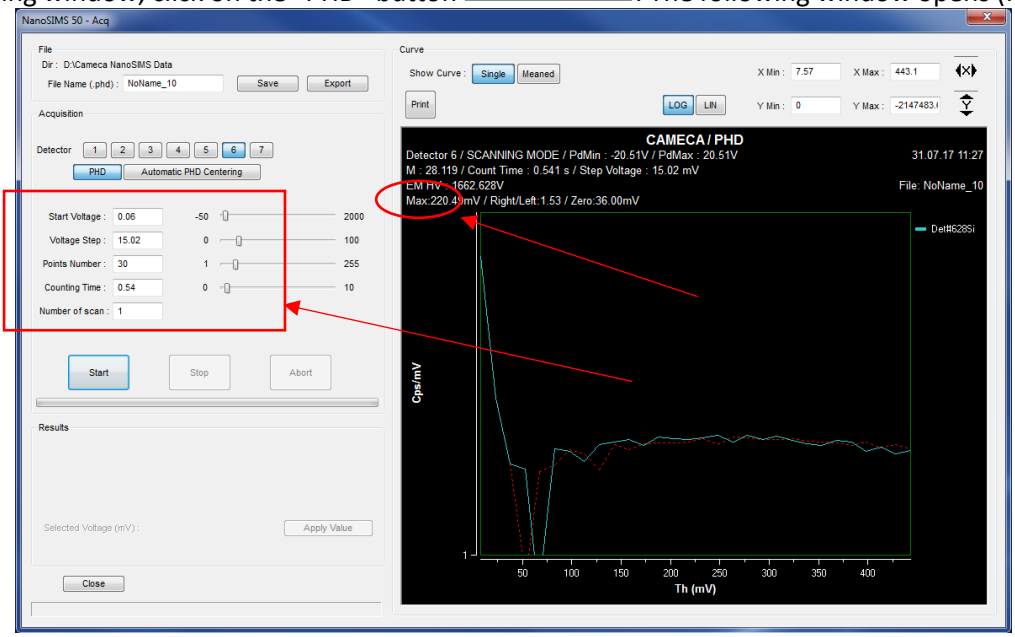


Figure 334: PHD window

9.1.12.1 EM HV adjustment

EM age significantly with use. Therefore, this operation needs to be done frequently. At least at the beginning of each session, and before each analysis in the case of isotope analyses. In must be checked on each detector one by one.

In the "PHD" window, select the detector number and set the following parameters:

a. Start voltage: 0
b. Voltage step: 15
c. Point number: 30
d. Counting time: 0.54
e. Number of scans: 1

Click on start and read the "Max value" on the top left of the curve (Figure 334). If this value is higher than 220 mV, decrease the EM HV with the keyboard, if it is lower, increase the EM HV. Make a new scan for each value of HV until the "Max value" is around 220 ± 10 mV.

Note that if doing high precision isotopic ratio analyses, it can be interesting to adjust the EM HV so that the max value would be around 250 mV. The higher the PHD_{max} the stronger the aging effect; but at the same time at higher voltage the effect of a shift of PHD has less influence on the count rate; 250mV seems a good practical compromise for highest precision measurements (e.g. below permil level reproducibility).

9.1.12.2 Threshold adjustment

EM thresholds do not change much with time. This operation does not need to be checked as regularly as the EM HV. However, if a detector is either too noisy and/or not efficient enough, it can be good to check that the threshold value is correct.

To check the threshold, set the following parameters:

f. Start voltage: 0
g. Voltage step: 5
h. Point number: 30
i. Counting time: 0.54
j. Number of scans: 3
k. Choose “Meaned” on the top of the window

You obtain a curve like the following one (Figure 335):

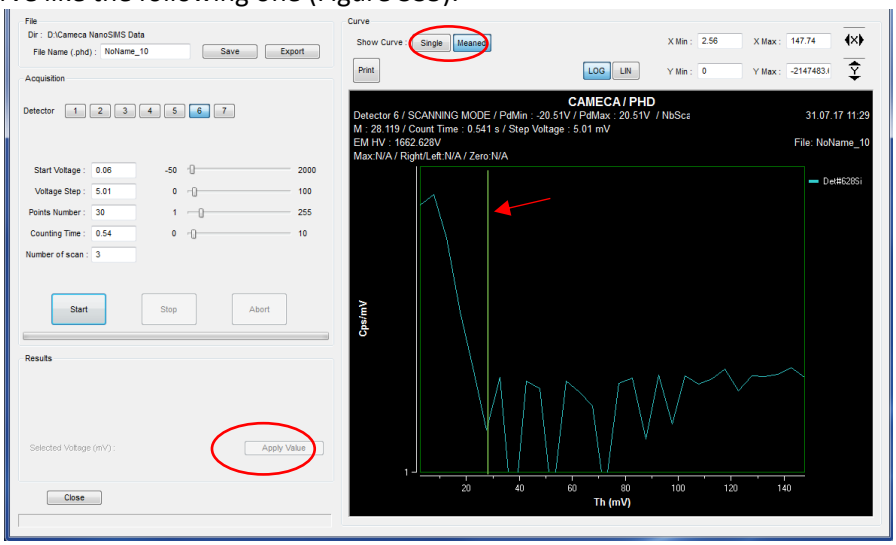


Figure 335: PHD window with a scan set to adjust the threshold

Select manually the threshold by clicking on the curve and valid it by clicking on the “Apply value” button. The threshold is usually around 30-40 mV.

9.1.12.3 Optimizing Mass analyzer transmission

For a tuning that will be valid for all masses, it is of course recommended to work with an intense peak. In order to optimize the mass analyzer transmission at a given mass resolution (mostly defined by the size of the entrance slit), you first need to introduce such an entrance slit:

- Select an entrance slit, e.g. ES-3:

- This new slit ES-3 will decrease the signal of about 50% compared to ES-0 (no slit).
- In order to compensate for imperfect sample parallelism, working distance, surface potential and ES slit position, one needs to center the secondary ion beam in the **entrance** slit (ES) acting on deflectors P2-P3 and Cy, and to focus it in this slit by acting on EOS. You can do so either manually or using the Tuning programs.
  a. Manually: On the keyboard, press the button EOS and adjust with the thumbwheel the Cy, P2/P3 couple and EOS values in order to maximize the detector signal at the top of the detector window.
  b. Via the programs: Scan first EOS followed by Cy then P2/P3 via the programs “EOS” and “Sec. Ion Beam” respectively. It is usually advised to scan Cy (horizontal), *then* P2/P3 (vertical), and finish with EOS *again*. See chapters 0 and 0 for details on EOS and Secondary Ion Beam scans, respectively)

- Repeat the operation (Cy, P2/P3, EOS) until it does not increase the signal anymore.

Note: EOS should be between 43 200 and 44 800 bits. If optimizing the transmission leads to EOS value to far from this range, the sample distance is not optimal. It is then better to readjust Z (via the *Navigator*) to keep EOS within the recommended range.

Caution: While using the Sec. Ion Beam centering software, which determines the optimum values for Cy, P2 and P3, the relative ratio P2/P3 has to be properly set. This ratio allows maintaining the secondary ion beam parallel to the horizontal axis while varying its height in ES changing P2 and P3 in synchronism. This ratio is very sensitive to the setting of LF2 (Figure 336). As LF2 is generally set to 20000 bits, the relative ratio P3/P2 must be set to 0.36.
This coefficient is introduced in the Setup chapter 5.9.4.6 Secondary Ion Beam. We recommend to NOT modify the value determined by CAMECA production or service engineer. This tuning goes beyond the scope of this users guide.

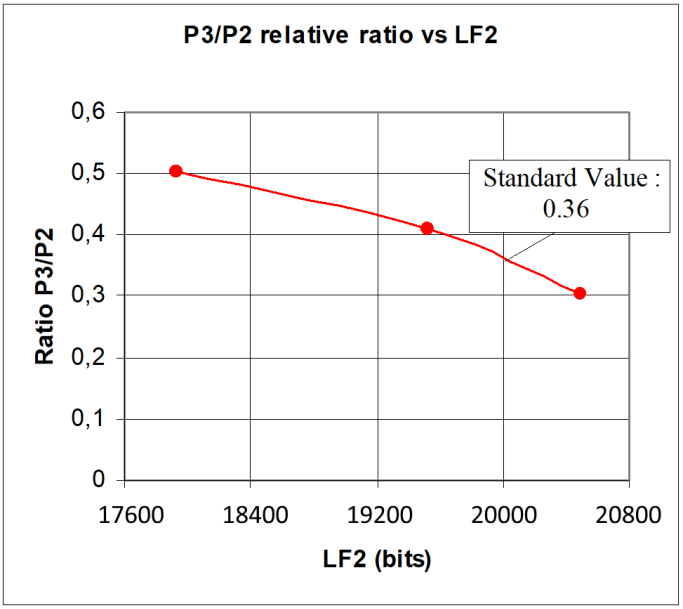


Figure 336: P3/P2 ratio variation with LF2.

9.1.13 Optimizing Mass Resolving Power

To simplify extremely, the MRP is mostly governed by the size of the entrance slit (ES), then by the aperture slit (AS), and then by other secondary factors. It is defined by the sharpness of the sides of the peak, not by the width of the peak. The width of peak is governed mostly by the size of the exit slit. In order to optimize the peak shape, and to reveal the peak tails several decades lower in intensity, one needs a large dynamic range. Thus, one needs to tune on high intensity peaks. For instance, when in Cs^+ mode, it is best to first check peak shape on ^{28}Si on a Si wafer sample and then repeat the operation **for each detector one by one:**

- click on the "**HMR**" button on the left side of the Tuning window 

.In this "HMR" window, the detector trolley deflection plates Pd_x are scanned before the **exit** slit, allowing:
  a) to record a **high mass resolution mass spectrum** (HMR) around the mass of interest,
  b) to position with a cursor the precise peak position in this spectrum, to use in subsequent image, profile, isotope acquisitions.

- Select the detector. The start value, step value, number of point and counting time are independently adjustable by user (Figure 337):

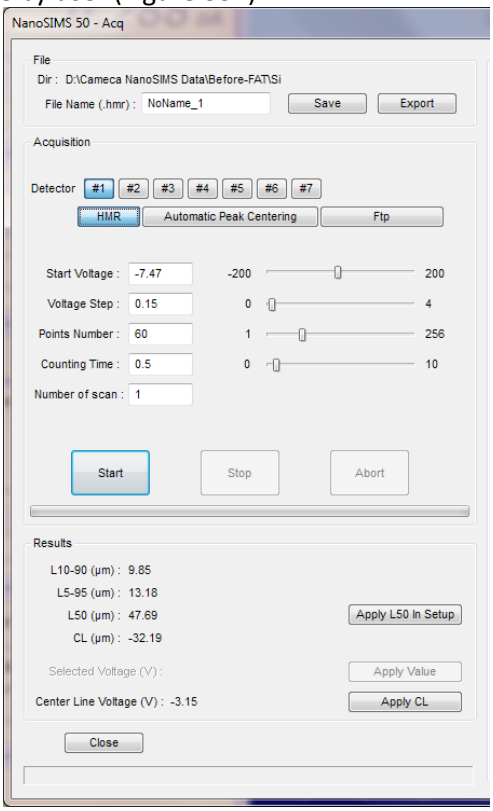


Figure 337: HMR window showing the adjustable parameters for a scan.

- When all the scanning parameters are set, click on *start* to record a High Mass Resolution (HMR) spectrum for the mass measured by the selected detector (Figure 338).

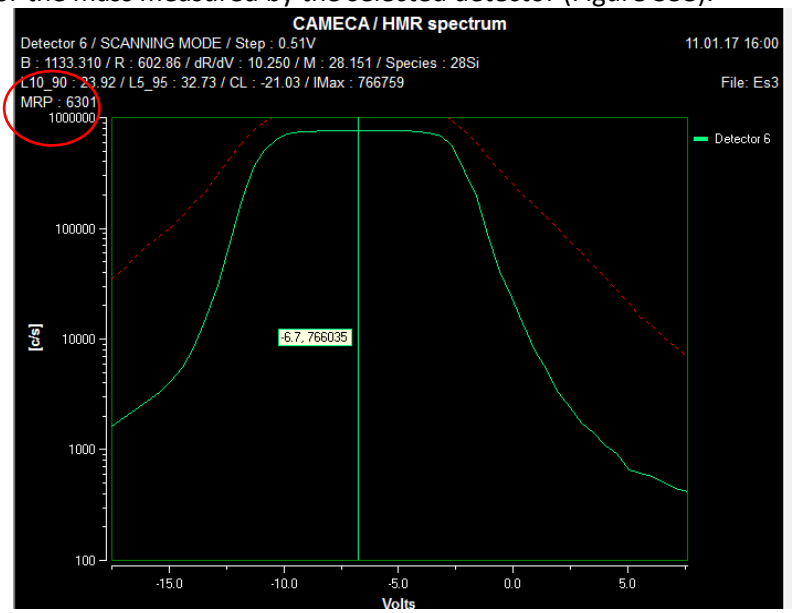


Figure 338: High Mass Resolution spectrum. The green curve is the last one, the red curve is the previous one, for reference.

- The mass resolving power (MRP), automatically calculated, is written on the top left of the HMR curve (Figure 338).
- To finely select the position of the peak (= mass line) used for detection in the subsequent acquisition (isotope, image, profiles…), two options are possible:

a. Automatic centering: The peak center is automatically calculated at the end of the analysis. A vertical green line appears. To apply this deflection plate value click on the center line (CL) button: “Apply CL” 

.

This automatic method is efficient when there is a single peak with no interference because the software is able to calculate the peak center only for single peaks.

b. Manual centering: click in the middle of the peak, a second vertical line (**white**) appears corresponding to the chosen deflection value. To apply this value of deflection plate, click on the button “Apply Value” 

This manual method is used when there are interferences (several unseparated peaks). The software is not able to detect each peak, therefore the operator needs to select manually the peaks.

Whatever the method used to select the peak to measure, it is important to remain on the flat top part of the peak, and not on the edges, as it would result in an unstable signal.

- To improve the MRP, adjust the quadrupole before the magnetic sector by adjusting Q value via the keyboard. For each detector, there is a Q value that will optimize the MRP (Figure 339). Q may vary depending on the detector’s position in the multicollection chambers. If Q varies from one detector to another, find the best compromise: either an average Q that gives similar peak shapes to all detectors, or a Q that would favor the peaks showing interferences, where it is most critical to have a good MRP.

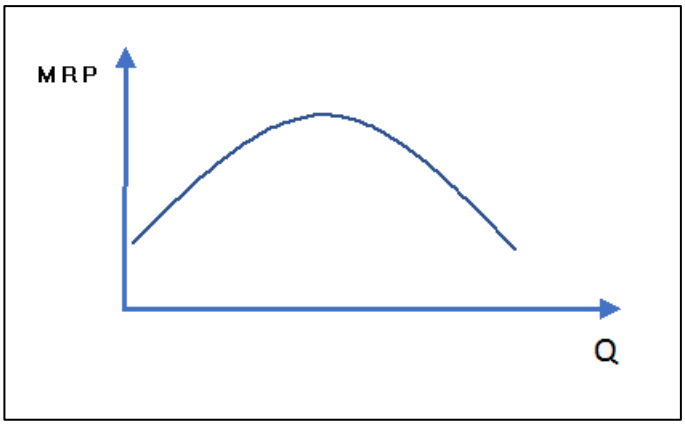


Figure 339: theoretical relation between MRP and Q

- If a higher MRP is needed, put a smaller entrance slit (ES) (which implies a re-adjustment of Cy, P2/P3 and EOS) or add an aperture slit (AS): in the same conditions of analysis, the two of Figure 340 show two HMR peaks, one with ES-3 (MRP=6301) and the other with ES-4 and AS-3 (MRP=9390).

Of course, adding or reducing slits will reduce the transmission for all detectors.

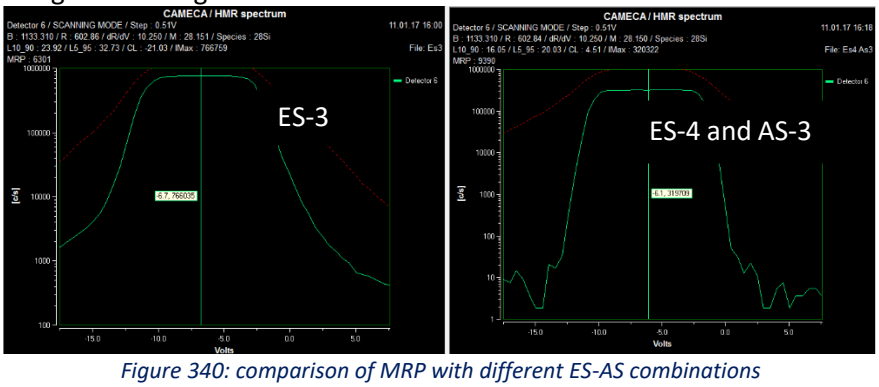


Figure 340: comparison of MRP with different ES-AS combinations

More advanced peak shaping & positioning is available in expert mode chapter 9.3.1.3.

Notes:
On a multicollection system with an adjustable B-field, several moveable detectors and their electrostatic deflectors, it is impossible to keep a perfect mass calibration. One cannot simply move a detector to a theoretical mass and select the good peak to image. It is thus necessary to use standard samples, preferentially mounted on the same holder as the sample to analyze, in order to easily identify peaks and center the detector on it. In case of interferences, to make sure the correct peak has been selected, one can check that the ratios are correct between the different isotopes of a given elements (by varying the B-field manually or recording a bargraph).

At this stage, it is always very useful or necessary to check the good tuning of the spectrometer. On a standard sample (ex: a silicon wafer) and using always the same NS50L conditions measure FCp beam current and check the expected sensitivity (in c/s) on a matrix peak (ex: $^{28}Si^-$ with 16 keV Cs^+) for the given mass resolution setting, and the noise around the peak.
For example, on a Si wafer, with a primary beam of Cs^+ at 16 keV, the $^{28}Si^-$ signal is of about

- 625 000 c/s per pA of Cs+ with no slit.
- 250 000 c/s per pA of Cs+ with ES-3, AS-2 (MRP > 6000)

This test should be performed regularly (e.g. every week) and documented ensuring a follow up of the good condition of the instrument.

9.1.14 Basic image acquisition

When the analysis area is set and the instrument is ready for an image acquisition, follow this procedure:

- On the "Board", open the "**Def Analysis**"  and the "**Analysis**" program 
- In the "Def Analysis" main menu, use "Select" button to select the directory in which you want your analyses to be saved (this directory will also be used for any other file you might save using other functions, CCD screenshot, scan, RTI image, etc…).
- Click on the "images" button (Figure 341)

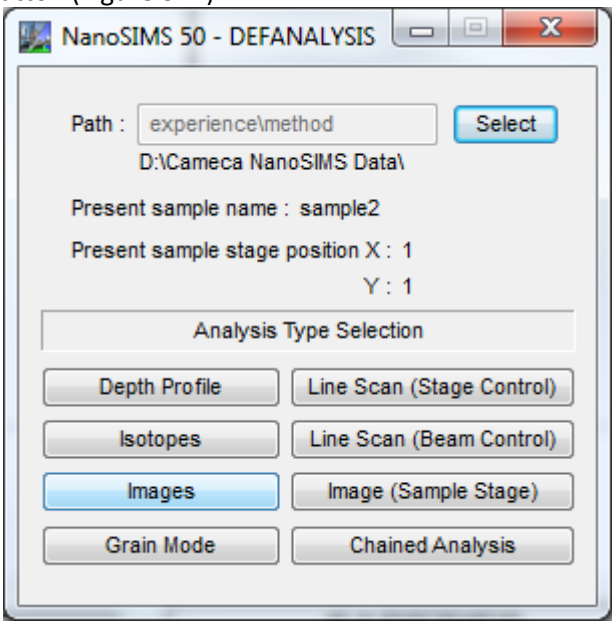


Figure 341: Def Analysis main menu

- The following Image **DefAnalysis** window opens:

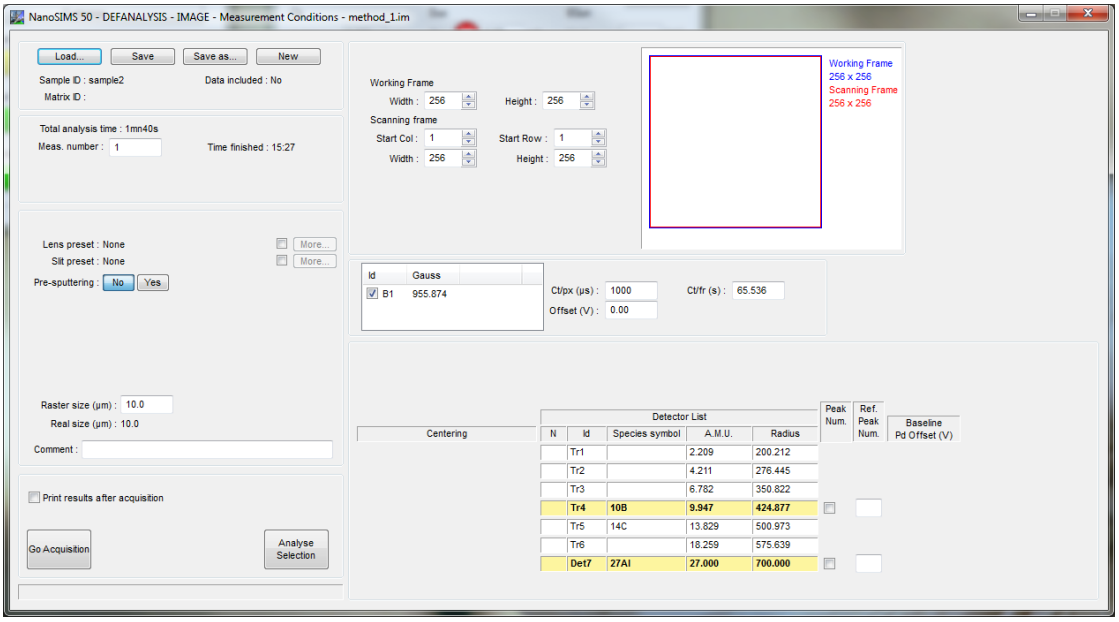
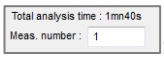
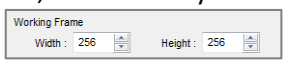
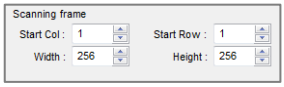
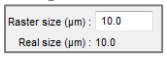


Figure 342: IMAGE "Def Analysis" window

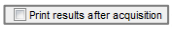
- Fill in the following parameters necessary for the analysis. All these (and many more) will be stored in the resulting *.im file:
    a. Meas. number 

: number of frames (planes) to be acquired (in Images mode, 1 measure = 1 frame, also called cycle in WinImage software)
    b. Working Frame 

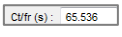
: Maximum accessible frame size (in pixels), defining the memory allocation size.
    c. Scanning Frame 

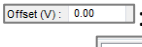
: Size and position of the scanned image, in pixels, within the working frame. In most cases, working frame = scanning frame.
    d. The raster size 

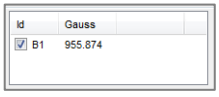
: Field of View (FOV), in µm= the area physically rastered by the beam on the sample.
    e. Comment 

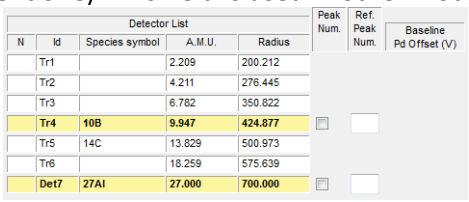
: very useful for documenting a data inside an analytical session, for future data analysis.
    f. Print results after acquisition 

: If checked, the results will be printed after acquisition.
    g. Ct/px 

: the counting time (= dwelltime) per pixel, to be entered in µs
    h. Ct/fr 

: the counting time per frame (=cycle)
        i. Note that Ct/px and Ct/fr are linked. Modifying one will automatically modify the other and the total acquisition time will be adjusted automatically.
    i. Offset 

: Function not used anymore. Should be left at 0.00 V.
    j. B_{field} selection 

: In Multicollection Tuning mode, only one Bfield value is available (the current one). This field is used in other modes.
    k. Detector selection 

: Select the detectors to be used for the imaging by clicking on their respective “id”. The Species field filled automatically

from the EM/FC detector panel. Be sure it is filled properly, otherwise it will be tricky to identify them later on! (see chapter 5.2.2 for details)

l. See advanced and expert operations for additional options such as use of presets (9.2.3.3) and peak centering (0)

m. Once all necessary DefAnalysis parameters are documented (e.g. 256x256 pixels for both, 1ms/pixel (~1min per cycle), Raster 30x30µm, trolleys selected), click on "Go acquisition" button 

to access the image Acquisition window.

n. The Image acquisition "**Analysis**" window opens:

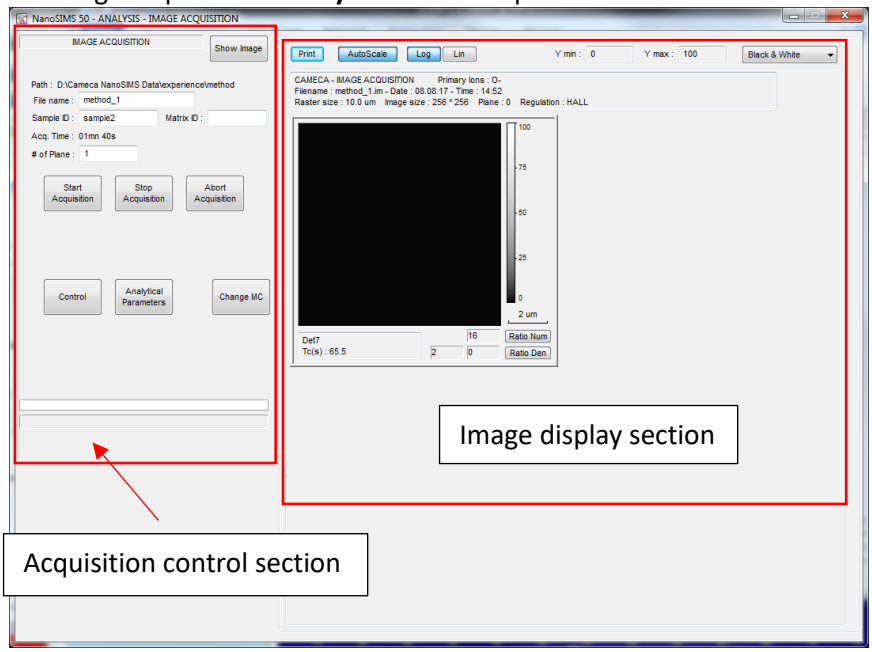
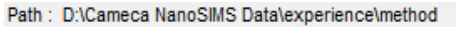
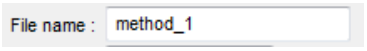
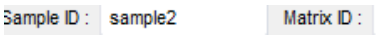
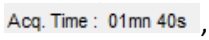


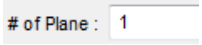
Figure 343: Analysis window

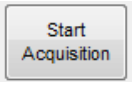
- "Path" shows the disk/directory where the data file will be saved
  

- Enter the file name (*.im) for the image to be saved 

  and press enter. By default, the file name is the name of the directory, with a number increment.
- You can document your image with a sample and a matrix comment (by default, the information displayed are taken from the Navigator. See chapter 5.3.2 :
  

- The acquisition time is indicated 

  , as calculated from the frame acq. time defined in DefAnalysis and the number of planes (= cycles) defined just below.
- The total number of planes is indicated: 

  If need be, this can be changed, even during the acquisition, to shorten or extend the analysis.
- When everything is set, click on "Start Acquisition" 

  .
- At the end of the analysis, the image will be stored under an *.im file. Such files can be opened with the WinImage program.
- Note: opening it in WinImage and just re-saving it as is will create a new *.im_rpc file in which the data are re-organized for smaller file size and much faster future opening.

During the analysis, the operator can:

- Select the temporary image display parameters in this section:

- Stop or abort the acquisition by clicking on the "Stop Acquisition" or "Abort Acquisition": 

"Stop", will stop the acquisition, including the last plane (=cycle) partially acquired. "Abort" will save the acquisition *without* the last partially acquired plane (=cycle). If the acquisition is aborted before the first plane is completed, the analysis will not be saved at all.
- View the analysis parameters by clicking on "Control" 

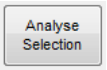
- View the instrument configuration 

Once the acquisition is done, you can:

a. Launch the same acquisition again by clicking on "Start" once again.

b. go back to the Def Analysis window (Figure 342) by clicking on *Change MC* 

if you wish to change any acquisition parameter for the next analysis.

c. Change the type of analysis by clicking on Change MC to go back to Def Analysis, then click on *Analysis Selection* 

to go back to the list of analysis types.

9.1.15 Real time imaging (RTI) in Multicollection

When the peaks are selected and tuned, a live SIMS image (RTI) can be started.
Note 1: the sample field of view (FOV) as viewed by the analyzer is firstly limited by the diameter of diaphragm D1 (near which an image of the sample is formed), following the equation FOV = 0.6 * diameter of D1. A secondary ion image using a larger primary beam raster will show the black edges of the selected D1 on the circumference of the scanning.
Note 2: if an entrance slit ES is used,it is necessary that the dynamic transfer (B2) is well tuned and the secondary beam waist right in the middle of ES and AS. This latter part is obtained with a sample surface at the good Z-position in order to be able to use a fairly constant EOS value.
If the dynamic tranfer is not well tuned and/or EOS far from its normal value, the image might be cut on the two vertical sides for fields of view over ~45µm when using small entrance slit.

To obtain a live RTI image:

- On the left side of the Tuning window, select the RTI button 

- The following window pops-up (Figure 344):

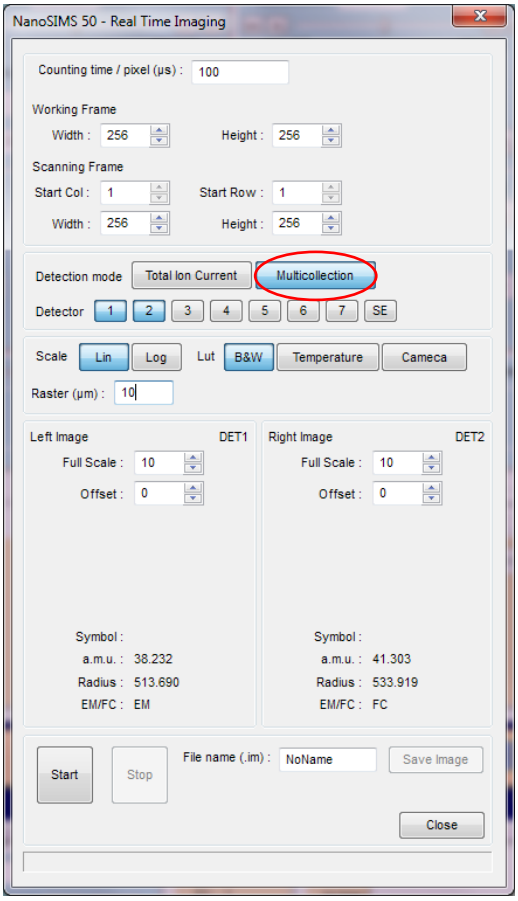


Figure 344: RTI window. This time "multi-collection" detection mode is selected

- Enter the counting time per pixel (e.g. between 50 and 100 µs)
- Select the Working and Scanning Frame pixelization. The working frame is the total number of pixels available. The scanning frame defines the number and position of pixels physically scanned by the beam during this acquisition. For this Basic operation let's select 256x256 for both.
- "Multicollection" detection mode (Figure 344) is automatically selected.
- Select the detectors you want to activate (2 maximum)
- Select the field of view needed (raster size or FOV, in µm).
- Click on "Start", a real-time SIMS image of your sample appears in a second window

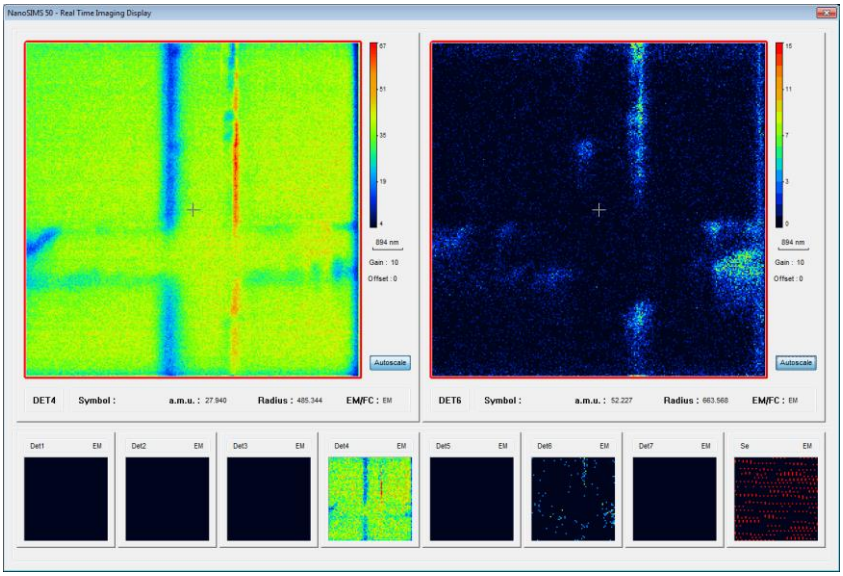


Figure 345: RTI acquisition window in multicollection mode with detectors 4 and 6 selected

You can now:

- Move the stage to adjust the image position.
- Adjust the focus with EOP and correct the astigmatism with the octopoles 45 and 90.

If the sample was not pre-implanted it will take several scans before the ion signal starts to increase and reach a steady state.

- Change the image to display in large, by dragging one of the eight thumbnails at the bottom toward one of the two larger images.
- Use the buttons available to adjust the image display: color scale, Lin-Log scale, Rej and Sat (manual or autoscale).
- Use “Save Image" button to save a graphical copy of the screen in a .im format which can be opened in WinImage.

Important Notes:

Real Time Imaging only offers a **live imaging** (for tuning, area selection, pre-implantation control, …) and while you can save a “screenshot” of the live image, you must go through the **image acquisition programs** (Def. Analysis and Analysis, see next paragraph) to record a proper analysis.

In RTI, when the counting time is set to 50 µs/px, with a Full Scale (scale maximum) set at 100 counts per pixel, the image appearing mainly red means that you are saturating your EM detectors and should reduce the signal to avoid early aging of the EM: 100 counts per 50µs is equivalent to a count rate of 100/ 5 E-5 = 2 E6 c/s.

9.2 Advanced operation

9.2.1 Tuning Check

Assuming the instrument had previously been tuned, you do not need to manually re-tune everything. This paragraph details the procedures to check routinely the good alignment of the instrument at the beginning of a session, assuming no maintenance operation has been performed.

Note that it is always easier to first check the alignment and the proper functioning of the instrument always on the same known standard, such as a Si grid, or any flat, conductive sample that can give strong signal and some sort of image features.

9.2.1.1 Switching Sources

If switching sources (and polarity):

- Shut down the High Voltage (HV) on the keyboard.
- In the source window: Select the primary beam species you intend to use (Cs^+, O_2^+, O^-/O_2^-), and wait for the polarity to switch.
- When switching from O to Cs, make sure D0 is in position 0. Make sure the Oxygen tank is tightly closed and do a gasline purge (In the Vacuum window → Gasline → Start; wait for the end of the procedure).
- Put the HV back on.
- In the Preset window, load an .isf file corresponding to your settings.
- In SIMS mode (Navigator) and Multicollection mode (Tuning), send the presets to the keyboards by selecting the correct presets and click on “valid”. Do this for:
    a. Global
    b. High current

c. Detectors
d. Wien Filter (in Oxygen)

- In the Tuning window:
  a. Click on Hex X and Y as well as D1 to recall the correct positions associated with the new polarity
  b. Press enter in the Magnetic Field box to recall the correct value.
- If in Oxygen, insert a D0 diaphragm (usually D0 -2 or 3)
- Move the source goniometer to the correct position previously noted (Cs or O) and adjust it if necessary to maximize the FCp current.

9.2.1.2 Primary beam alignment

- In CCD mode, adjust Z so that the sample surface appears as focused as possible.
- In SIMS mode on the Navigator and Multicollection mode in Tuning, select a preset in the Preset window and send it to the keyboard by clicking on "Valid".
- When using the RF source: In FCp (beam OFF), adjust slightly (a few bits) the Wien Filter voltage to optimize the signal (on the keyboard: WF → CWF)
- Go to FCo mode in the Navigator and adjust the centering of D1 (via D1 X and D1 Y on the keyboard) to optimize the signal in FCo. Centering can also be done via the "centering" function of D1 (see chapter 5.2.5) though it is just as easy and faster to do it manually.

9.2.1.3 Secondary beam alignment

- If some slits are inserted (ES, AS, EnS) put them all back to position 0.
- Go back to SIMS mode and select TIC mode in the Tuning window. Adjust EOS, Cy and P2/P3 to optimize the TIC signal. Do it several times if necessary.
- Select the Multicollection mode in the Tuning window and adjust the Magnetic field so that a major mass matches with the position of a given detector. For instance, select $^{28}Si^-$ in Cs^+/O_2^+ or $^{27}Al^+$ in O^- .
- Insert an entrance slit (usually ES-3) and adjust ES X and Y to optimize the signal on the detector. If the position has changed, click on "calib" in the Tuning window.
- Re-adjust EOS, Cy, P2/P3
- Insert an aperture slit (usually AS-2) and adjust AS X and Y to optimize the signal on the detector. If the position has changed, click on "calib" in the Tuning window.
- Open the RTI program and select a detector with a good signal. On the scanning image, adjust EOP, Oct-45 and Oct-90 to sharpen the image. Use a fast enough scan so that the image would refresh quickly (typically 50-60 µs/px). However, if the signal is low, find the best compromise between clarity of features and scan speed.

Note: adjustment of EOS, Cy and P2/P3 can be done manually via the keyboard by adjusting those parameters to maximize the signal in the detectors, or via the EOS and Secondary Ion Beam functions in TUNING (see chapters 5.2.18, 5.2.19 and 9.1.12.3)

9.2.1.4 Multi-collection

Move the detectors to their positions. Then, on each detector to be used:

- Check the PHD for all used EMs (see chapters 5.2.21 and 9.1.12 for details).

It is recommended to use a major element, giving a strong signal (0.5 to 1.10^6 c/s). If the signal is too low, you can double or triple the number of scans to obtain a total of 10^6 c/s (ex: if the signal is of 300 000 c/s, do 3 scans) and average them by selecting "meaned" in the PHD window (see chapter 9.1.12).

- Record an HMR spectrum and center the peak of interest (either by adjusting the trolley position or the deflection plate voltage (Pd).
- Adjust Q to maximize the MRP (see chapter 9.1.13 above). If the optimum value for Q is different from one detector to another, optimize the MRP for the detector where it is likely to be the most critical, i.e. where there might be mass interferences.
- If a higher MRP is required, an energy slit can be added. The energy slit can be used between EnS-0 (wide opened, no energy filtering) up to position EnS-6, depending on the aim (MRP increase, reduction of molecular interference, etc…), though most of the time EnS-1 (cutting 10% of the signal) is enough. See chapter 9.2.2 below for details on Energy filtering.
- Do a C4 X scan (Tools → parameters: C4 X) selecting all detectors used to check their centering in height. Find a C4X position that falls on the peak flat top for all detectors.

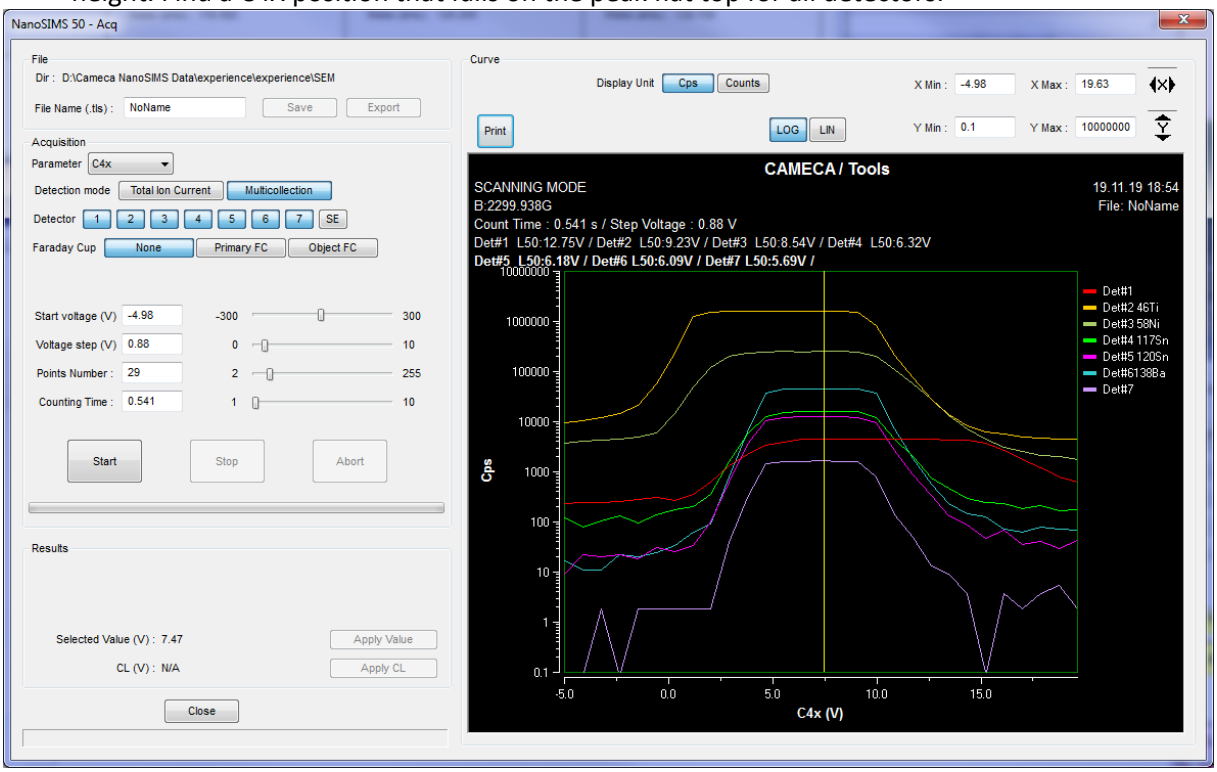


Figure 346: positioning of C4X.

- At this stage, it can be good to check that the transmission of the instrument is good by assessing the sensitivity (in c/s/pA) on a peak of a known standard sample (ex: Si grid) for a given configuration. For example, with 16keV Cs+ in pure Si, with no ES, no AS, the ^{28}Si signal should be around 625 000 c/s/pA. With a MRP > 6000 (ES-3, AS-2), the ^{28}Si signal should be around 250 000 c/s/pA. Whatever the reference used, it is good to check at the beginning of a new session that this reference signal remain reproducible. It is a good idea to always add this standard in the sample holder.

9.2.2 Energy filtering

9.2.2.1 Energy filtering background

Dynamic SIMS is characterized by a variable energy dispersion which depends on secondary ion species. It is recommended to read scientific literature (e.g. Stevie FA, *Secondary Ion Mass Spectrometry – Applications for Depth Profiling and Surface Characterization*, (Momentum Press, LLC, New York, 2016). Generally, molecular ions (e.g. $Si_2O_3^-$) will have a narrow energy distribution of a few eV, starting at zero relative to

the sample HV. In contrast, atomic ions (e.g Si-) have a much larger distribution, reaching several hundreds of eV.

Resolving mass interferences can be done by improving the mass resolving power (MRP), hence, at first level selecting small entrance slits. Although this method is optimized on the NS50L, reducing the entrance slit size will ultimately reduce the mass analyzer transmission.

Another route (which can be complementary to using small slits) is to cut the lower part of the energy bandwidth in order to attenuate the molecular species interfering on atomic species of interest. This is a classical method for rare earth element analysis.
On the opposite cutting the higher part of the energy spectrum will also reduce chromatic aberrations and help improving the MRP while reducing the atomic signal moderately.

Note that if the sample is charging (usually positively), especially if non homogeneously, its surface potential will vary, and so will the energy of the secondary ions. Consequently, using a small energy slit can be problematic with a higher sensitivity to varying charging effects (laterally over the surface or in depth at interfaces). Hence it is recommended on the NS to start analysis of insulators without energy slit.

It is possible to store seven settings for the energy slit (EnS-x) that can be then recalled by a single click in the slit & diaphragm control panel of Tuning. See below in 9.2.2.2 how to memorize the desired settings.

9.2.2.2 Calibrating Energy Slit setup values

The energy slit (EnS) can be used to either increase the mass resolving power (MRP) or reduce the interference of low energy molecular ions on monoatomic ions.

To set up the energy slit and improve the MRP, follow the procedure below:

- With the beam ON on a conductive sample (e.g. doped silicon), mass spectrometer well-tuned, with a good and stable signal on a detector, start with the energy slit wide open: EnS Y > 6 000 and EnS W > 20 000. The zero position for both is on the optical axis.
- Decrease Y value (= move the whole EnS slit (both sides together) toward larger radius or energy; see red arrow on the schematic below) until you reach the point where you start cutting the signal.
- Decrease W value (= close EnS by only moving the exterior side of the slits, see green arrow in the schematic) until the signal has decreased of about 10 %. This will cut the high energy ions that are introducing chromatic aberrations.

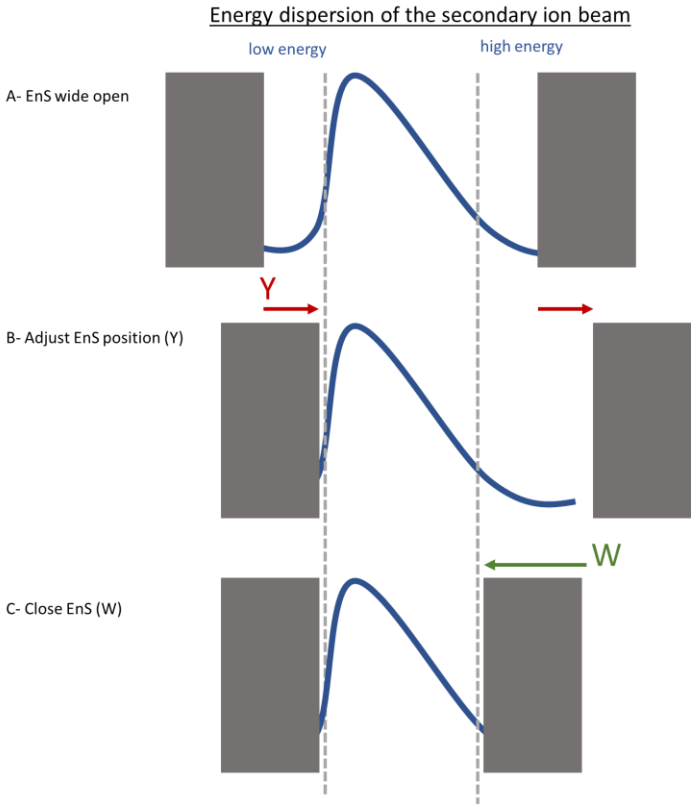


Figure 347: adjustment of the energy slit (EnS)

In Setup → Slit → Energy → [1], enter the Y and W values. Now in Tuning, EnS-1 will correspond to a 10% cut of the signal.

You can repeat steps B and C for EnS-2 by cutting the signal 20% with W. Then, in Setup → Slit → Energy → [2], enter the Y and W values. Now in Tuning, EnS-2 will correspond to a 20% cut of the signal. You can repeat the same procedure for all Energy slit slots, though the signal is rarely cut beyond 20%.

Cutting high energy ions will improve the MRP by reduction chromatic aberrations.

For applications requiring cutting low energy ions (for instance, reducing an interference from organic or molecular ions), simply move Y (step B) a little further to slightly cut the signal on the low energy side of the spectrum.

Be careful that for insulating samples, especially with non-homogeneous conductivity, if the energy slit is too small or not well centered, it might introduce sensitivity variations!

9.2.3 High beam current and high lateral resolution settings

It is possible to increase or decrease the primary beam intensity at the object (read on FCo) independently of the source settings. The primary parameter must always be D1 diaphragm selection.

But in order to optimize the primary beam density and vary the beam current on the sample, L1 can also be used. See chapter 2.1.1.2.3 for detailed explanation.

It is possible to SAVE those settings as “high current” or “low current” presets. This can be useful for tuning purpose, to easily and rapidly switch between different current settings or to define pre-sputtering conditions different from the analysis conditions.

9.2.3.1 Using L1 for High beam current

To increase the current, increase L1. If the primary beam has been tuned properly, you should be able to increase L1 up to 27 000 bits (4300 V) and reach about 3 nA (Figure 348). It will also increase the spot size.

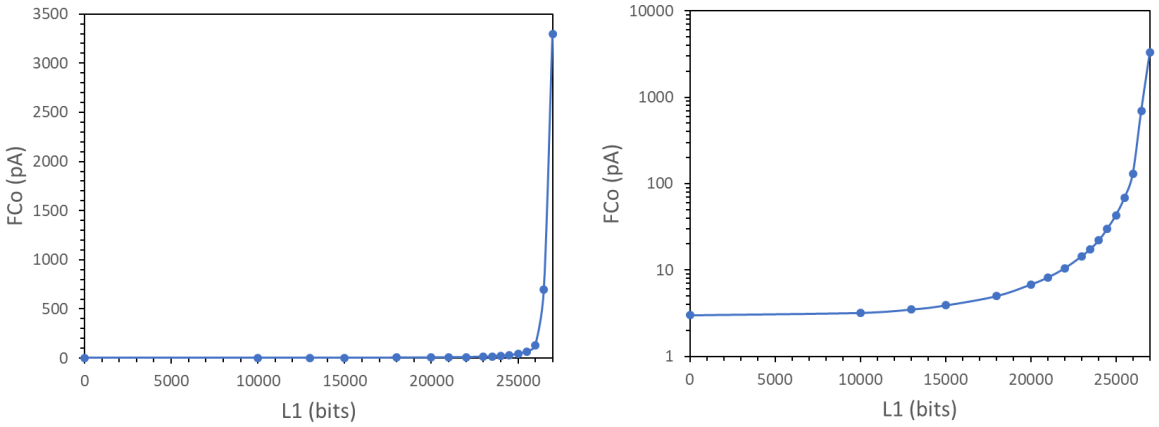


Figure 348: Evolution of FCo with the increase of L1, on a linear scale (left) and LOG scale (right), with D1-3 and FCp = 50 nA.

In RTI mode, adjust C1 X and C1 Y so that the image remains in the same position when L1 = 0 and with a high value of L1.
Note that if the current (in FCo) is 3 times higher with a given L1 than with L1 = 0, the secondary signal should be 3 times higher as well.
Select a new preset slot in “high current” and hit “calib” to SAVE the L1 and C1 values in a new preset.

Note: increasing L1 will induce a deformation of the beam, making images inhomogeneous (especially for images of more than 10 µm raster the crater bottom with start to become not flat for hundreds of pA). When making images at current over 100 pA, it can be better to use a combination of L1 and L0, increasing both moderately. This is to be done with caution by an experimented user. Make sure to check in RTI that the image is homogeneous. Readjust C1 X-Y and C0 X-Y if necessary. For more details on the effects of L1 and L0 on the beam, see chapter 2.1.1.2.3.

9.2.3.2 Using L1 for High lateral resolution

To increase the spatial resolution (=reduce spot size) L1 can be used to demagnify more the source size (at the cost of a lower beam current). To do so, L1 has to be increased until the beam current peaks and starts decreasing again. Typically to reach a resolution around 50 nm, L1 should be around 8500 V, associated with a D1-4 or D1-5 diaphragm to limit aberrations.
Similar to high current settings, adjust C1 X and C1 Y so that the image remains in the same position between different L1 settings (doesn’t move laterally).
Select a new preset slot in “low current” and hit “calib” to SAVE the L1 and C1 values in a new preset.

9.2.3.3 Use of Presets in Analysis Definition

Whether in “high” or “low” current, once a preset is saved (“calib”), it can be sent to the pre-sputtering or analysis condition files. To do so, select the preset, then click either on “Send For PreSput” or “Send For

Acq" (Figure 349). Note that this step is mandatory to make those conditions available for analysis and only one preset can be sent to each category (one preset for pre-sputtering and one preset for the analysis).

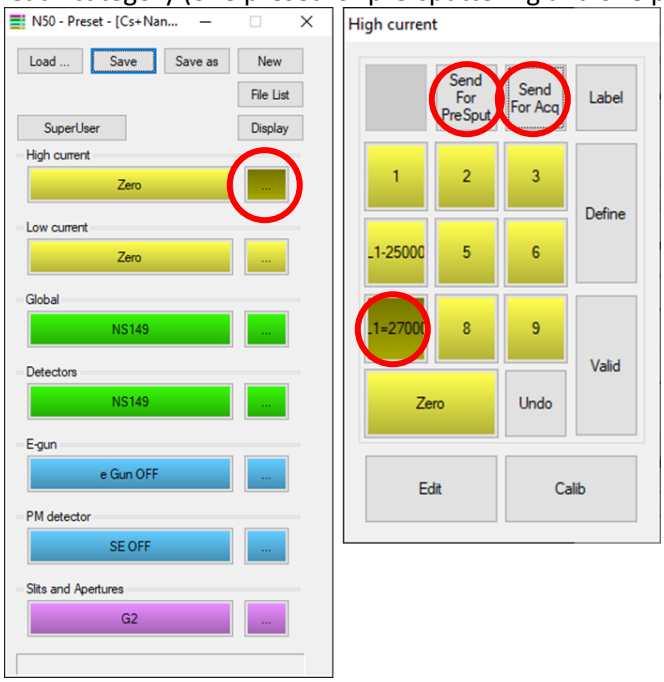


Figure 349: High current presets

If the user then wishes to use those presets during analysis, in the **Def. Analysis** window, check the "lens preset" and/or the "slit preset" boxes. If the user wishes to do a pre-sputtering prior to the acquisition, select "yes". The pre-sputtering menu appears below. Select similarly the lens and slit presets if you wish to use any (Figure 350). Note that if no preset is selected, the analyses will be run under the instrument's current conditions.

The "more" button shows you the list of parameters under the selected preset.

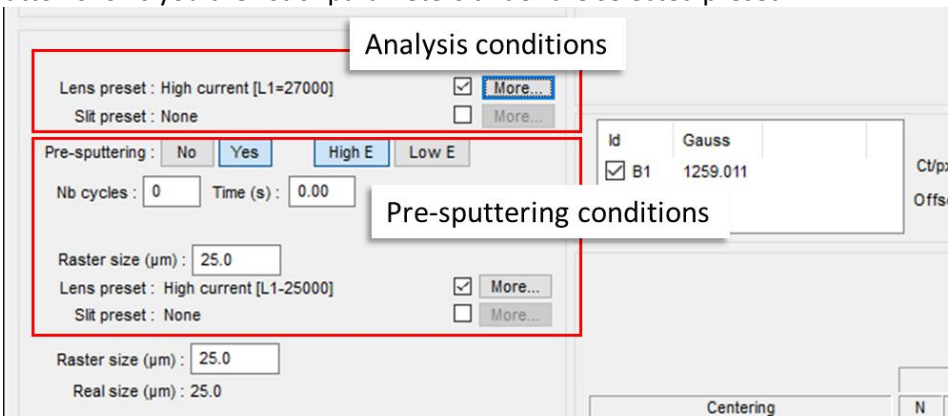


Figure 350: DefAnalysis : select presets for the acquisition and the pre-sputtering

9.2.4 Slit preset

For both Analysis and pre-sputtering conditions, it is also possible to define particular slit and diaphragm presets in case the user wishes to use different diaphragms or slits for pre-sputtering and for the analysis. The setting is the same as for the use of lenses presets: **First in Presets**, send the desired presets to pre-sputtering and to acquisition (Figure 351). **Then in Def Analysis**, send select those presets if you wish to you use it (Figure 352). See chapter 5.6.2.3 for more details on how to send presets to Def Analysis.

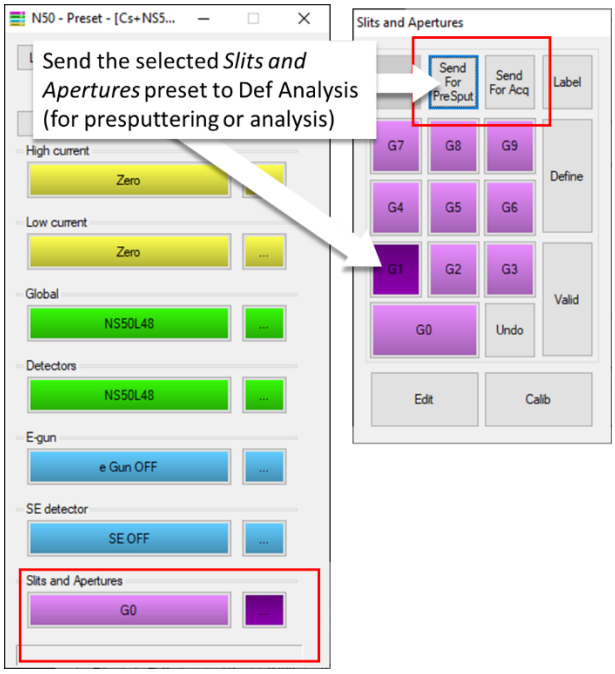


Figure 351: sending "Slits and Apertures" preset to Def Analysis

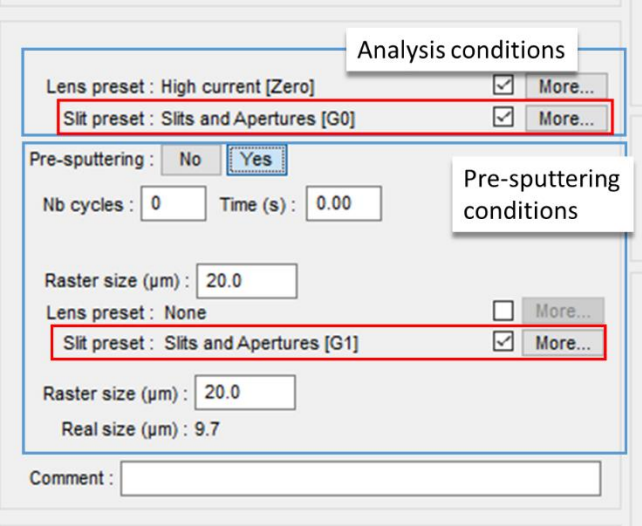


Figure 352: Def Analysis: adding a slit preset to the analysis conditions and to the pre-sputtering conditions.

9.2.5 NMR

9.2.5.1 Use of NMR in static multicollection mode

The magnetic field is regulated with a Hall probe sensing the B-field and sending a feedback signal to the electromagnet coil current power supply. This system also allows magnetic peak jumping and for long analyses at very high mass resolution the stability of the B-field can be not good enough to keep the peaks stable enough. In order to improve the B-field stability, NMR probes are positioned “in” the magnet. Such NMR regulation loop is more stable but has a longer response time (it is good for static multicollection mode but does not allow fast peak jumping). Each NMR probe has a range of B-field over which it can work. For example, a separate NMR probe (named H/D) is proposed as an option to regulate lowest B-field for hydrogen. The switch between NMR probes is done automatically by the NMR cabinet and is transparent for the user.
To activate the NMR probes:

- Open the NMR program by clicking on the button 

on the “Board”
- Wait for the end of the NMR connection and locking process (the “NMR locked” button should turn from dark green to bright green, Figure 353). It can take a minute.

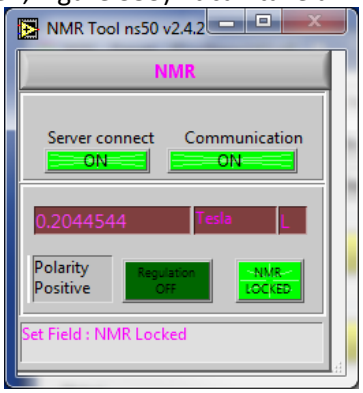


Figure 353: NMR reduced window, showing the NMR locked

- In the Tuning window, click on the NMR regulation ON

- Wait for the regulation process (the Regulation button will turn from dark green to bright green, Figure 354)

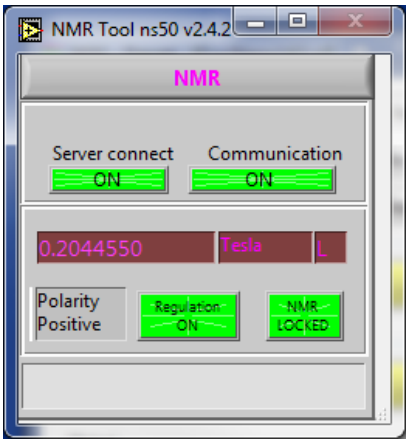


Figure 354: NMR reduced window with the Regulation ON

Note: when the operator wants to change the magnetic field with the keyboard and/or the thumbwheels, it is better to turn off the NMR regulation. Deactivate it temporarily by clicking on NMR Regulation OFF in the Tuning Control panel (Figure 124), and reactivate it when the B-field is set by clicking on NMR regulation ON.

9.2.6 Other multicollection acquisition modes

From the first **DefAnalysis** window, it is possible to select different acquisition modes (Figure 355).

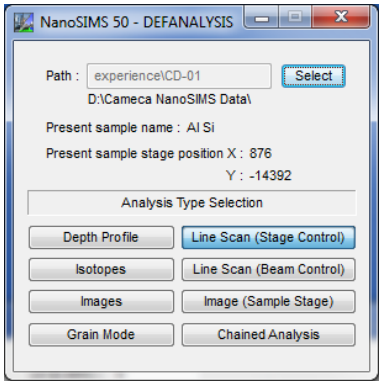


Figure 355: First DefAnalysis window, allowing the user to choose the mode of data acquisition

For all modes (except Chained Analysis) a similar window will open. See Basic Image acquisition 9.1.14 for the basic settings required for all analyses. There are also some optional settings that are common to all those modes:

- For the use of presets see chapters 5.6.2.3, 9.2.3.3 and 9.2.4
- To define pre-sputtering conditions, see chapter 5.6.2.4
- To define the different scanning frames, see chapter 5.6.2.5.
- For Real Time Tracking (live alternated drift correction option) see 9.3.8.

There are also **options in DefAnalysis that are specific to certain modes**. They are described below:

9.2.6.1 (direct) Depth Profile

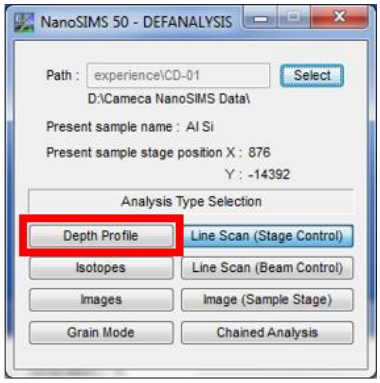


Figure 356: DEFANALYSIS: selection of Depth profile

Depth Profile allows the user to record the signal intensity on selected detector as a function of depth. It is a conventional ion microprobe direct depth profile, without storage of image and post-reconstruction.

The user can define the duration of the analysis and the number of cycles (Figure 357). In this mode, the scanning mode is optional (but generally used to generate a flat crater bottom). If the scanning mode is not selected, the beam will be fixed and the analyzed area will be equal to the size of the beam. If the scanning mode is selected, the scanning frame will govern the physical beam scanning.

The tails of the primary beam leaching the crater edges will limit the dynamic range of a depth profiles. Hence direct depth profiles are always recorded with an electronic gate (called here Blanking) that will avoid crater edge contribution. It will be adjusted before the acquisition for the whole profile. Before setting the profile conditions, leaving the beam erode the sample in fixed position (without scan) on an homogeneous sample for a while, then dezooming and imaging the induced hole will reveal the beam tails (“moustaches”) caused by a wrong adjustment of EOP and stigmator, aperture aberrations with large D1 diaphragm, other aberrations…). Typically, the crater size will be adjusted no less than X10 larger than the observed beam shape. And the electronic gate may be 10% in area or 30% in width.

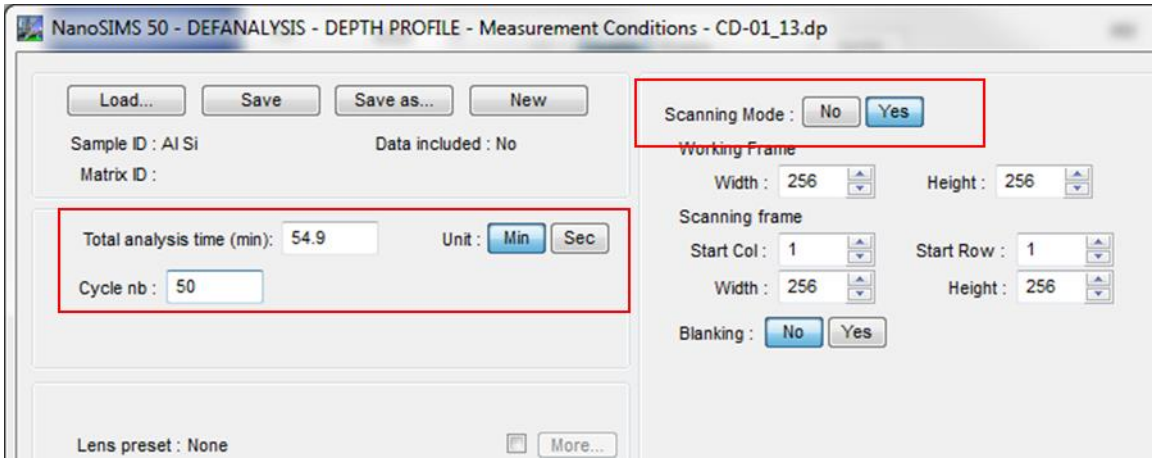


Figure 357: specificity of the Depth Profile acquisition mode

When everything is set, click on “Go Acquisition”. The Analysis window opens (Figure 358). Click on “Start Acquisition”. The Analysis window shows a preview of the data during acquisition.
If necessary, you can modify the number of cycles (# of Meas.) during the acquisition to shorten or extend the analysis.

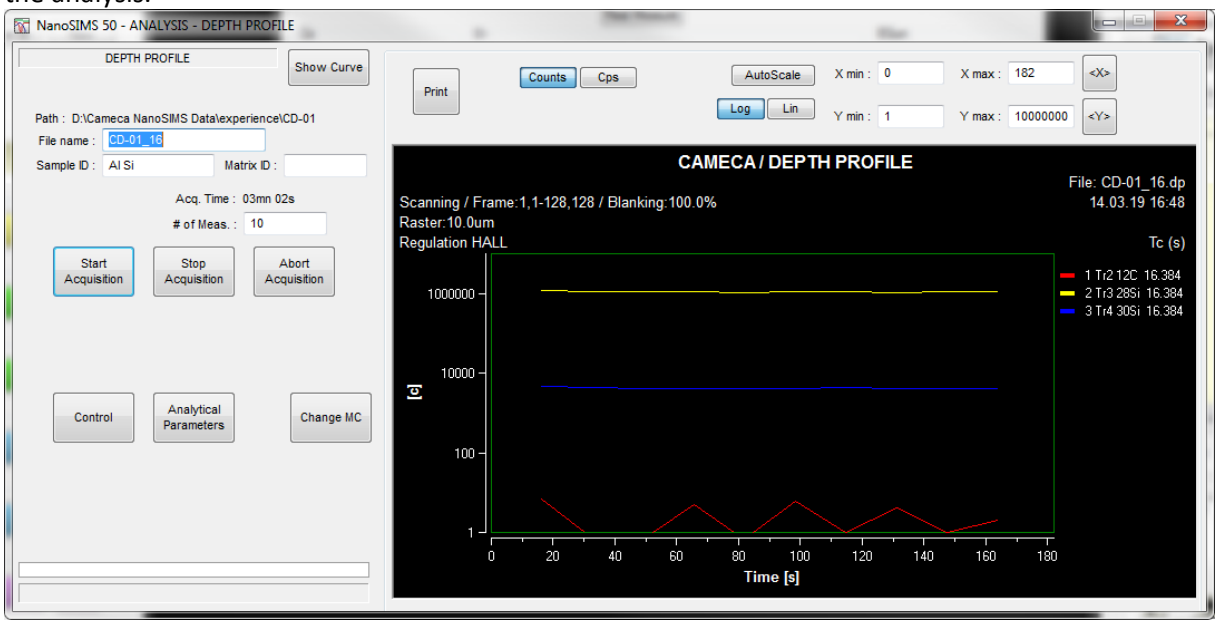


Figure 358: Analysis window in Depth Profile mode

The other Analysis buttons have been already described:

- Control: Shows the tuning parameters of the analysis, such as primary beam intensity, lenses, slits, X, Y and Z positions, etc..
- Analytical Parameters: shows all parameters and slit used for the primary and the secondary beam, as well as the presets.
- Change MC: After an analysis, click on “change MC” to go back to Def Analysis and modify analysis settings.

Once the analysis is done, data can then be saved in a .dp file for data processing with WinCurve Cameca software.

Advantages of direct depth profiling: such depth profile is directly visible during the acquisition without the need of reconstructing it afterward. It will be stored in a very small size file. The conditions are easily reproducible. No time is spent for defining ROIs and reconstruction. When there is no contrast in the FOV,

it is the fastest and most convenient method. For very small FOV e.g. below 5x5µm, alternated real-time drift correction (RTT) can be added when necessary (see chapter 9.3.8).

Disavantages of direct profiling: no replay or repositioning of the electronic gate is possible as opposed to an image stack where one can process the image stack after the acquisition: correct for sample drift (when there is some contrast in the FOV), finely position multiple ROIs of any shape for profile replay, post-acquisition.

9.2.6.2 Isotopes

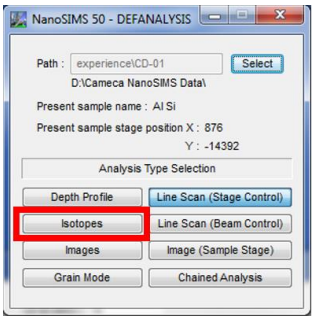


Figure 359: DEFANALYSIS: selection of Isotopes

The Isotope mode allows the user to measure isotopic or elemental ratios on defined areas. In order to evaluate variations and stability within the analysis, the data are grouped in internal blocks of scanning cycles. A final table of results gives the main statistical information among and between the blocks.

Click on the "Ratios" button and define in the "Isotope Ratios" table the ratios by entering detector numbers corresponding to the isotopes or elements available in the Detector List at the bottom right of the window (Figure 360). When done, close the window by click on the red cross.

Note that at this level some optional automatic routines are often added to refine the isotopic analysis conditions (automatic EOS focus and beam centering in the entrance slit; automatic peak centering in the exit slit, , automatic PHD adjust; baseline Pd offset). All these routines must be first tuned then sent to DEFANALYIS before they can be selected here. They are described in the expert operation chapters ***Erreur ! Source du renvoi introuvable.****, 9.3.3).*

Define then the number of blocks and the number of measurements per block, as well as the (optional) rejection condition.

Note on rejection conditions: at the end of the analysis, the mean and the standard deviation (σ) of each ratio is calculated. Setting the rejection conditions, for instance at 2 σ, means that if the value measured at a given cycle deviates from the mean more than 2 σ, it will be rejected and the mean recalculated.

Select the scanning mode (yes/no). If yes, define the usual parameters (Working and scanning frames, dimensions, and if an electronic gate ("blanking") must be used to remove crater edges. The raster sizes in µm of the working, scanning and blanking frames are noted respectively as "raster size", "real size" and "counting size" (Figure 360).

Enter the acquisition time as:

- A count per point (Ct/pt) if no scanning mode
- A count per frame (Ct/fr) or count per pixel (Ct/px) if the scanning mode is on. Only the scanning frame will be taken into account in the calculation of the total analysis time (= time/px * px/ scanning frame * nb of frames).

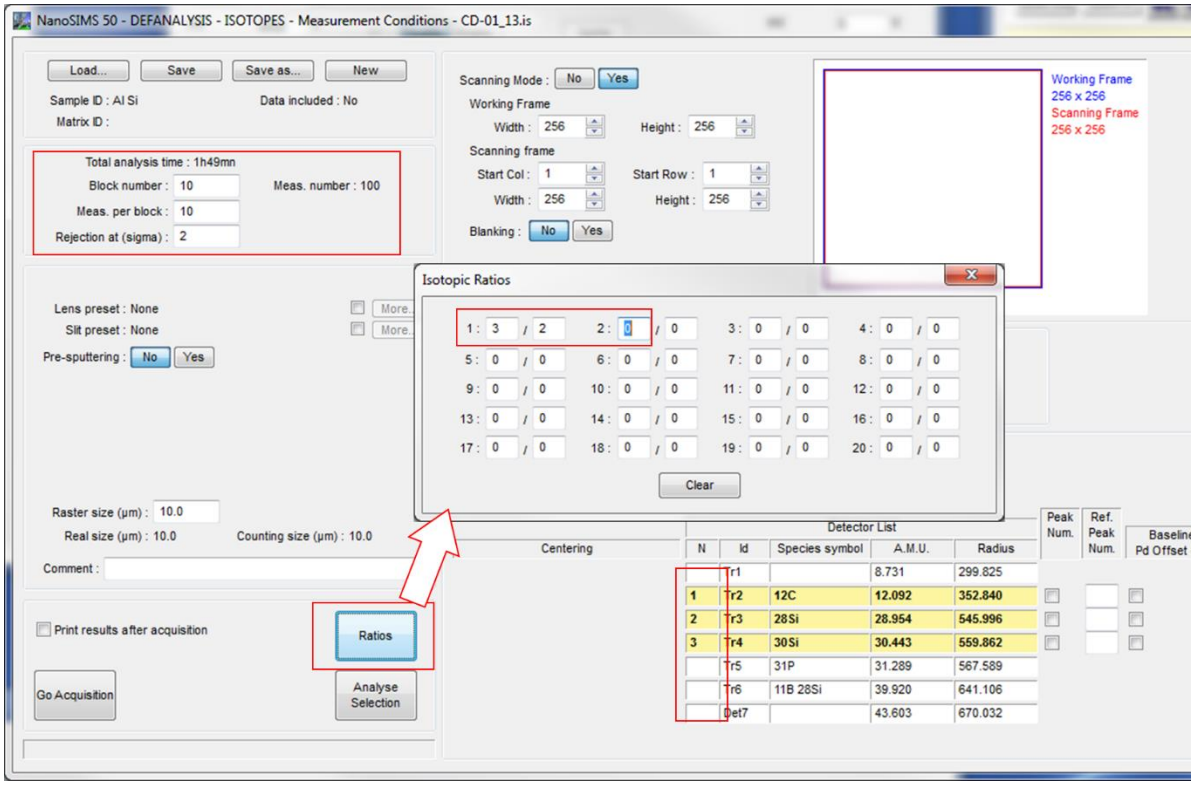


Figure 360: Isotopes acquisition mode

When everything is set, click on “Go Acquisition”. The Analysis window opens (Figure 361).
Click on “Start Acquisition”.
The Analysis window shows a preview of the data during acquisition (table of results and evolution of the detector signal over the measurement number).
Once the analysis is done, the table of results data are stored in a file with *.is_txt extension. The data can be imported and processed through Excel via the “import text/csv” module.

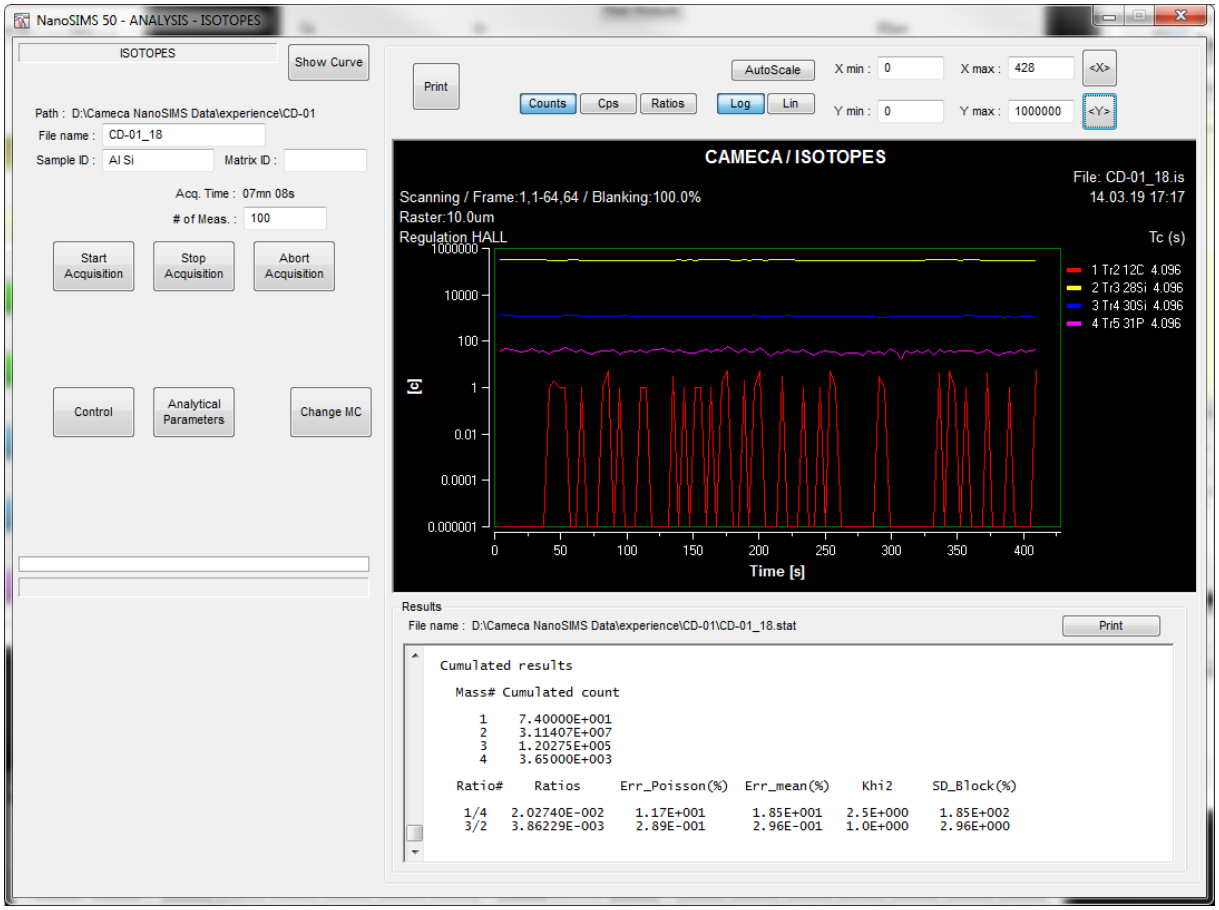


Figure 361: Final Analysis window in Isotopes mode

9.2.6.3 Grain Mode

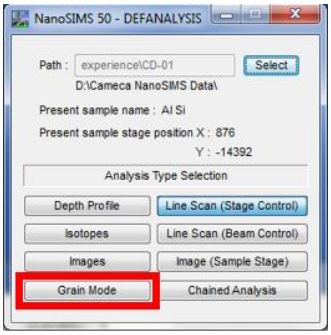


Figure 362: DEFANALYSIS: Grain mode selection

This is the mode often used for cosmochemistry with many tiny grains or particles fixed on the sample surface (e.g. pressed in a gold foil). The principle is to record a Reference image containing the grains then chain the analyses of all selected individual particles or areas in this image.

There are two different types of acquisition in grain mode depending on their output:

- an IMAGE acquisition around each grain. These small image files of *.im extension will be reprocessed with WinImage software, or
- an ISOTOPIC RATIO acquisition ending in a numerical result table file of *.is extension with cycles and blocks. Typically this is exported to Excel or other program for further processing.

To use this mode, proceed as follow:

- Select the Grain mode.
- When selecting this mode from Def Analysis, the Def Analysis window will appear as a usual “Image” mode window (Figure 363), allowing the acquisition of a single frame image acquisition (“Meas. number” is fixed = 1) in order to image the general area where the user will then select the grains. Define all parameters as you would define a regular image acquisition.

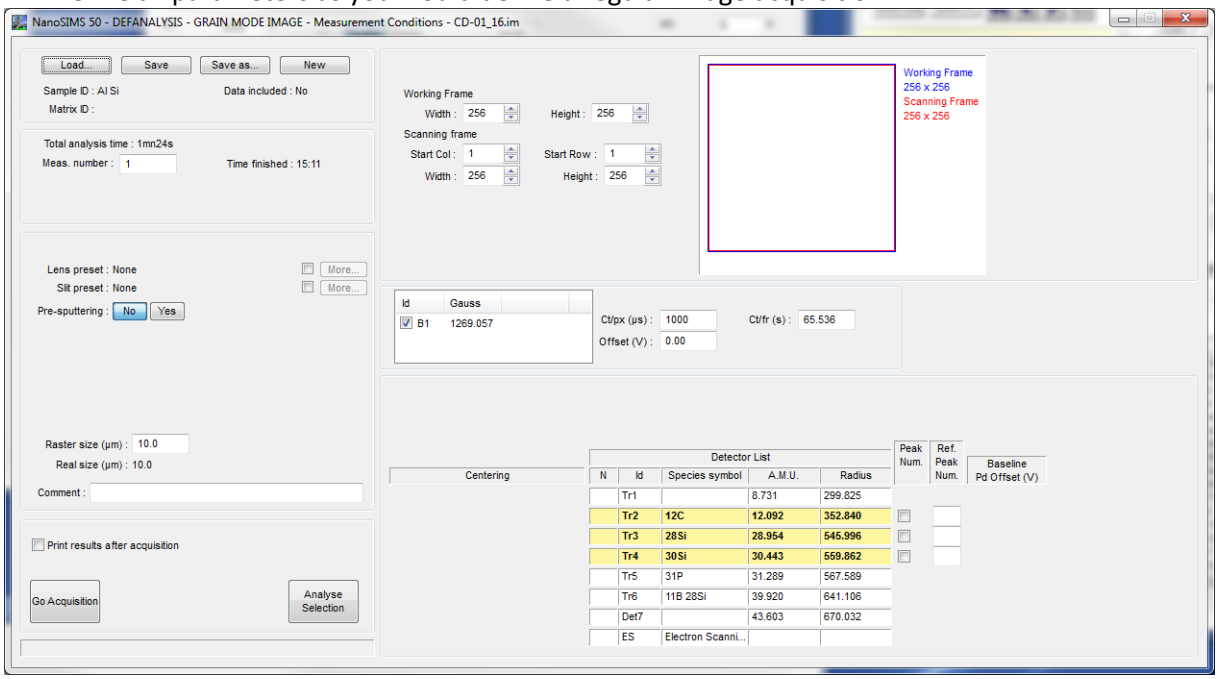


Figure 363: first step of the Grain mode

- Click on “Go Acquisition”. The Analysis window opens.
- Click on “Start Acquisition” and let the program acquire the reference image.

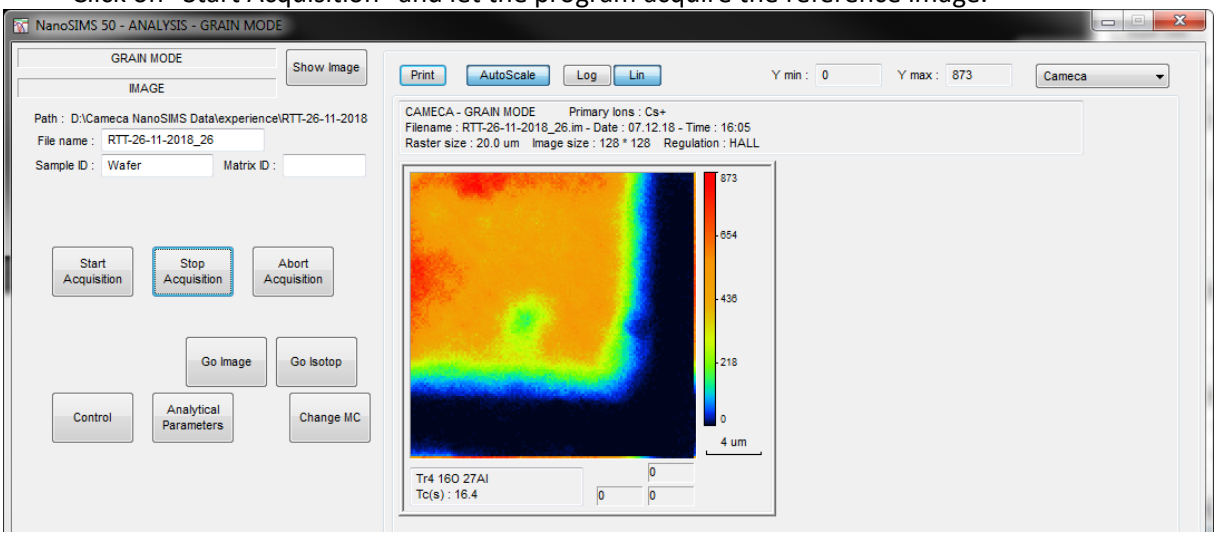


Figure 364: Analysis window in Grain mode.

- Click on “Go Isotopes” or “Go Image” (to select the output file type), depending on the type of analysis you wish to acquire (Figure 364). You are then redirected to a specific Def Analysis window corresponding to your choice (Figure 365). Define analysis parameters like for a regular Isotope or Image analysis. In “isotopes” mode, do not forget to define the isotope ratios.

The specificity of the grain mode is that you have an option to select an area with the scanned image. You have four selection modes available:

- **Graphic**: define a rectangular ROI manually with the mouse within the re image. You can repeat the operation as many times as you want between analyses, using the same rectangle or drawing a new one.
- **Semi Graphic**: Define a rectangle size (dX and DY, in px) following the equation: (2 dX+1)(2 dY+1) in Def Analysis, then position a rectangular ROI with the mouse in Analysis. You can reposition the rectangle as many times as you want between analyses.
- **Auto Grain**: detects grain positions following specified criteria and proposes successive acquisitions on rectangular ROIs of size adjusted to the grains.
- **Spec**: for CAMECA engineers: twelve pre-defined neighbor square ROIs in a square for testing local isotopic reproducibility during acceptance tests

Each mode is described below.

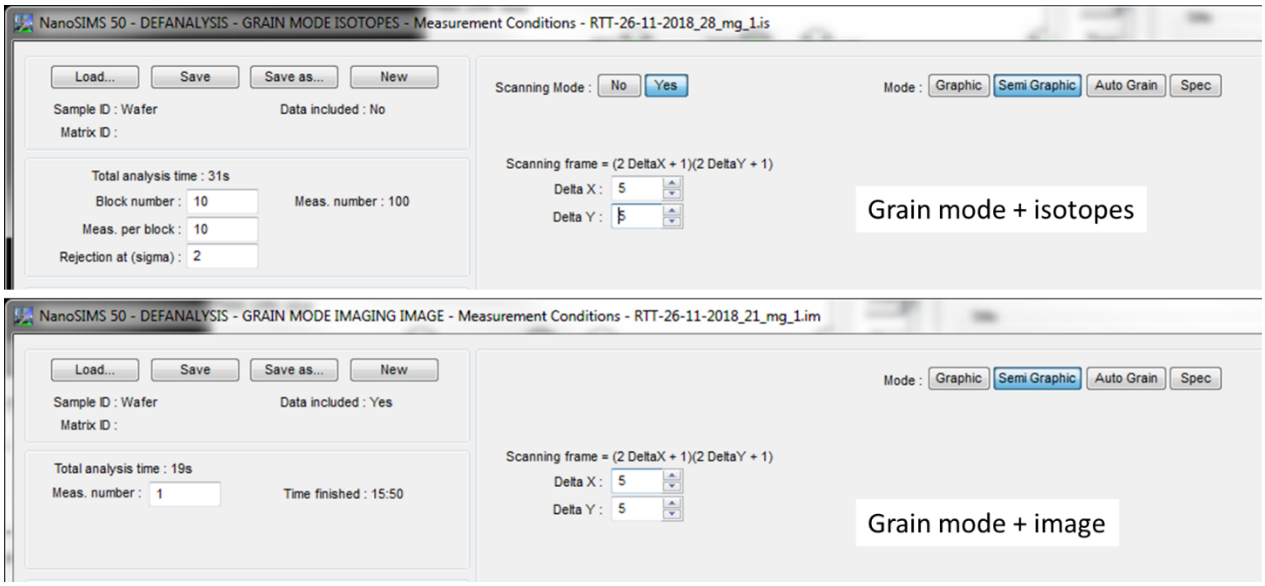


Figure 365: Scanning mode options in the Def Analysis window in Grain mode.

9.2.6.3.1 Graphic

The Graphic mode permits to repeat analyses on *any* rectangular ROIs manually and successively *defined* with the mouse within the ref image.

- Define the image/isotopes analysis conditions as usual then click on “go acquisition”. There is no specific option to the Graphic mode. Go directly to Analysis

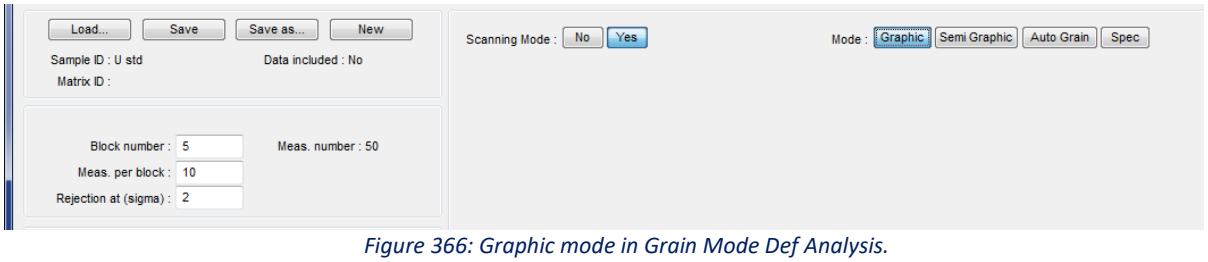


Figure 366: Graphic mode in Grain Mode Def Analysis.

- In Analysis, draw the rectangle of the desired analysis area with the mouse and click “valid” in the small pop-up window (Figure 367) then “start acquisition” in the main window. Depending whether you are in isotope or image mode, you will obtain an isotope or image file.

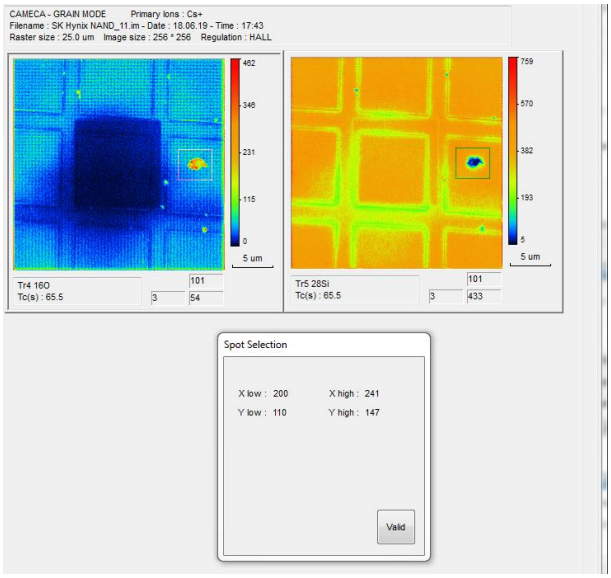


Figure 367: Analysis window in Grain Mode/Graphic. Manually draw the analysis area.

Only one zone can be drawn at a time. To analyze a second grain, after the first acquisition is over, in the Analysis window click on “show spot” to display the reference image showing the already acquired spots (Figure 368). Draw a new zone and click on “Start Acquisition”. Acquisition for this new spot will then start.

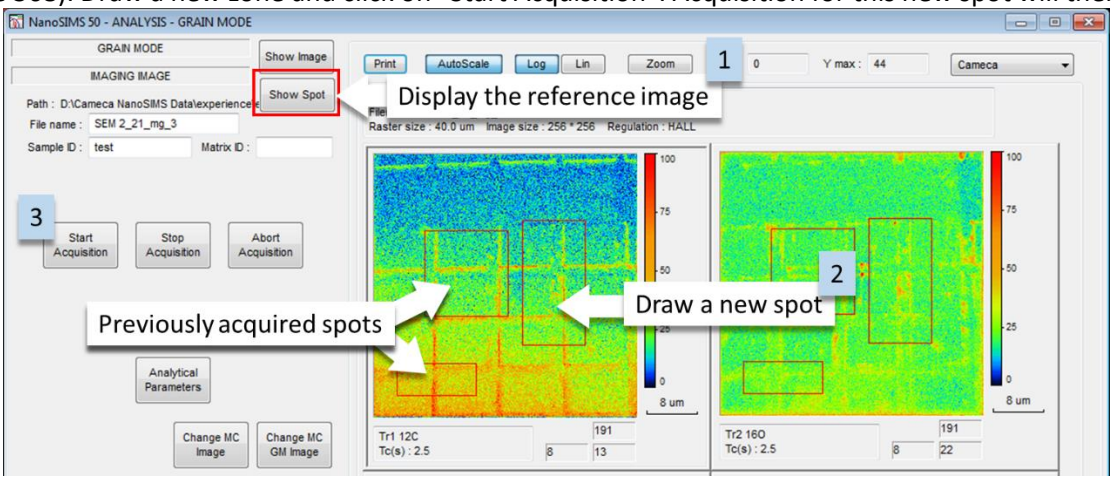


Figure 368: acquire successive spots via the Graphic grain mode

9.2.6.3.2 Semi Graphic

The Semi Graphic permits repeating analyses on the *same* rectangular ROI manually and successively *positioned* with the mouse within the ref image,

- Define the size of the selection area (Figure 369).
  Delta X and Delta Y are the half-length of the rectangle sides in pixels.

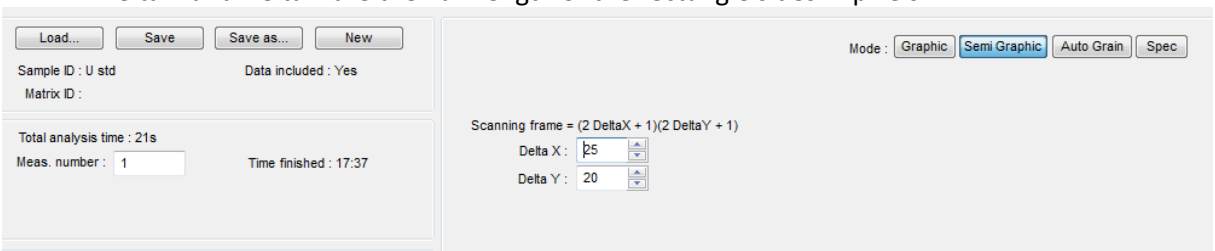


Figure 369: Semi-Graphic option in Grain Mode Def Analysis

- Define the image/isotopes analysis conditions as usual, then click on “go to acquisition”.

- the Analysis window looks as shown in Figure 370. A pop-up window shows the grain coordinates. Click on the image to position the grain then click on “valid”. If you wish to change the selection position, drag it with the mouse, or click on “remove” and select again on the image.
- Once the selection area is ok, click on “valid” in the pop-up window and then “start acquisition” in the main window. Depending whether you are in isotope or image mode, you will obtain an isotope or image file.

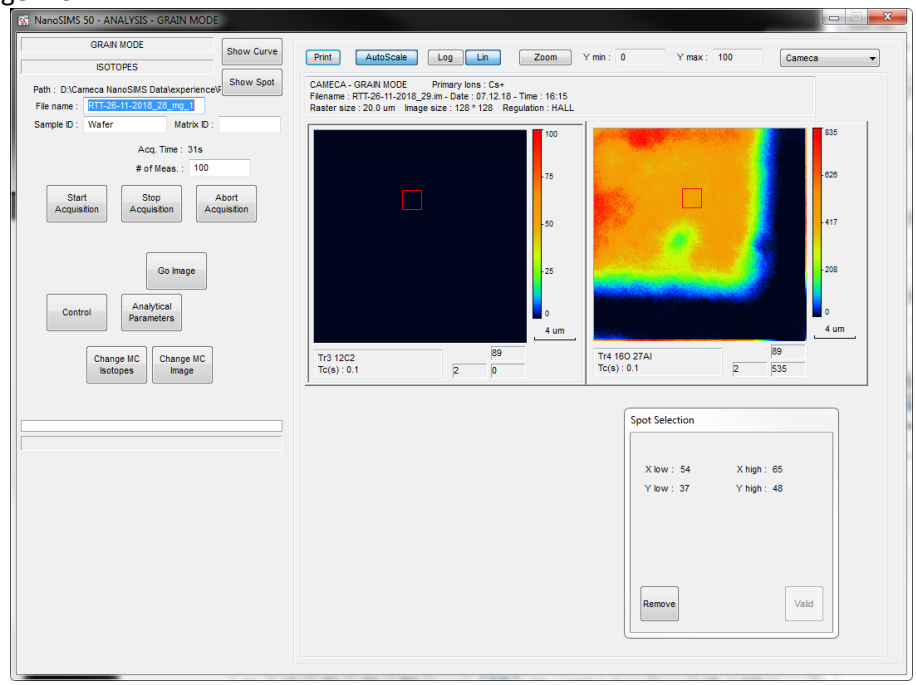


Figure 370: Analysis window after scanning mode “Semi-Graphic” in Grain mode.

Only one zone can be drawn at a time. To analyze a second grain, after the first acquisition is over, in the Analysis window click on “show spot” to display the reference image showing the already acquired spots (Figure 368). Draw a new zone and click on “Start Acquisition”. Acquisition for this new spot will then start.

9.2.6.3.3 Auto-grain

The Auto Grain mode detects grain positions following specified criteria and offers successive acquisitions on rectangular ROIs of rectangular ROIs of size adjusted to the grains.
Two different Autograin search algorithms buttons are available: Cameca and CIW (a customized version requiring additional software not available in the standard NanoSIMS software). The selection is done in the setup menu (See chapter 5.9.11.4). But for all users but two, the Autograin mode to select is the CAMECA one.

- Define the image/isotopes analysis conditions as usual, then click on “go to analysis”
- In the pop-up window, define the conditions of the “auto-grain” selection. Several options are available (Figure 371):
- First select the type of search within the image: by pixels or by Pins
    - Pixel: the search will look at all the pixels individually matching the criteria defined below. The pixels will be grouped into grains of all sizes
    - Pins: in this mode, a fixed grain size (of 16x16px, 25x25px or 32x32 px) is first defined and when an area is matching the criteria, it will apply the grain size to it.
- Second, select the image type used for the search:
    - **Threshold**: Select an ion, and define an intensity range (in cps/px), by entering a low and a high threshold on the pixel intensity.
    - or **Ratio**: define a ratio between two ions (e.g. 18O/16O), then define a lower (>) or upper (<) limit.

- It is possible to define successively different criteria and *combine* them to your search selection (e.g. 16O > 500 counts *and* 28Si > 2000 counts, or 30Si/28Si > 0.2 *and* 30Si > 500 counts). For this, define your criteria then use Add Criteria, Modify Criteria and Delete Criteria.

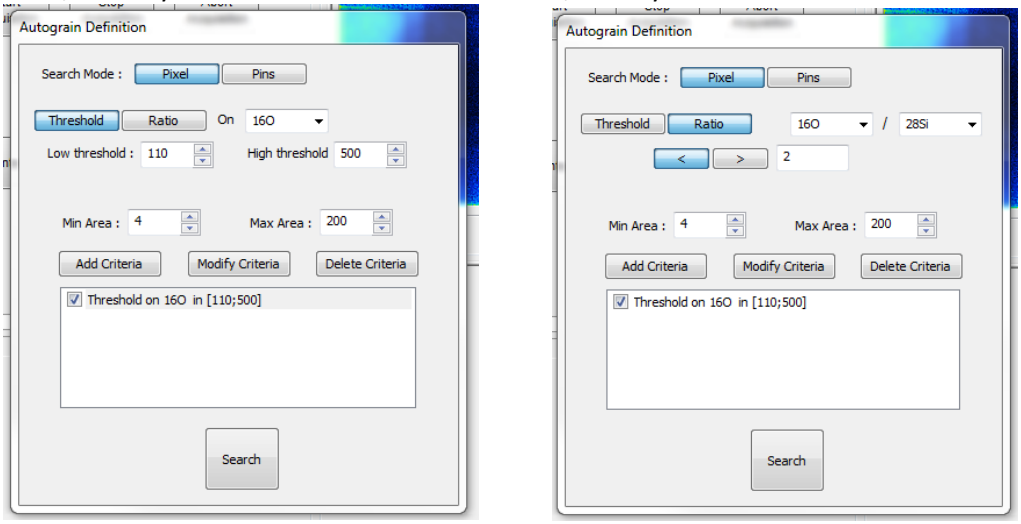


Figure 371: Options of the grain selection in Auto-grain mode. By pixel intensity thresholds (left) or by signal ratios (right)

- You can also limit the grain size range via “min area” and “max area” (in pixels) (Note that the area cannot be smaller than 16 px).

- When all conditions are set, click on “**Search**”. A new pop-up window opens, listing all areas matching your conditions (Figure 372).
- You can review the grains and select/deselect the grains you wish to analyze/skip. When a grain is selected the area appears with a red cross on the image.
- Click on “Start Acquisition” to launch the chained analysis of all selected grains. A separate .is file will be created for each grain.

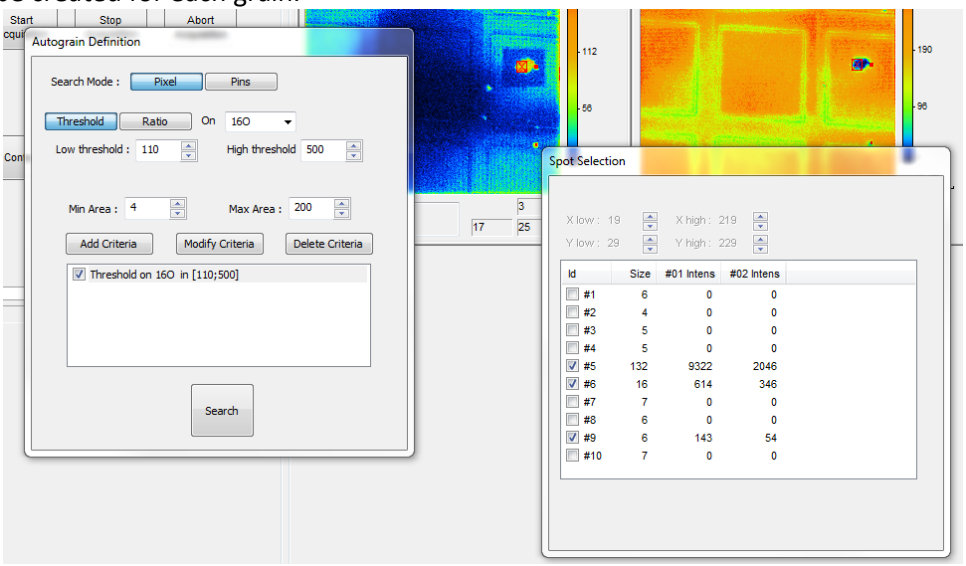


Figure 372: Auto-grain mode: List of grains matching the criteria and selection for analysis.

9.2.6.3.4 Spec

This mode has been designed to run isotope specification tests on the instrument. It automatically selects 12 areas in a cross pattern (Figure 373). The size of the squares will be a tenth of the raster size (i.e. for a raster size of 40 µm, the squares will be of ≈ 4 µm). This can be useful in isotope mode to check the stability and reproducibility of the instrument.

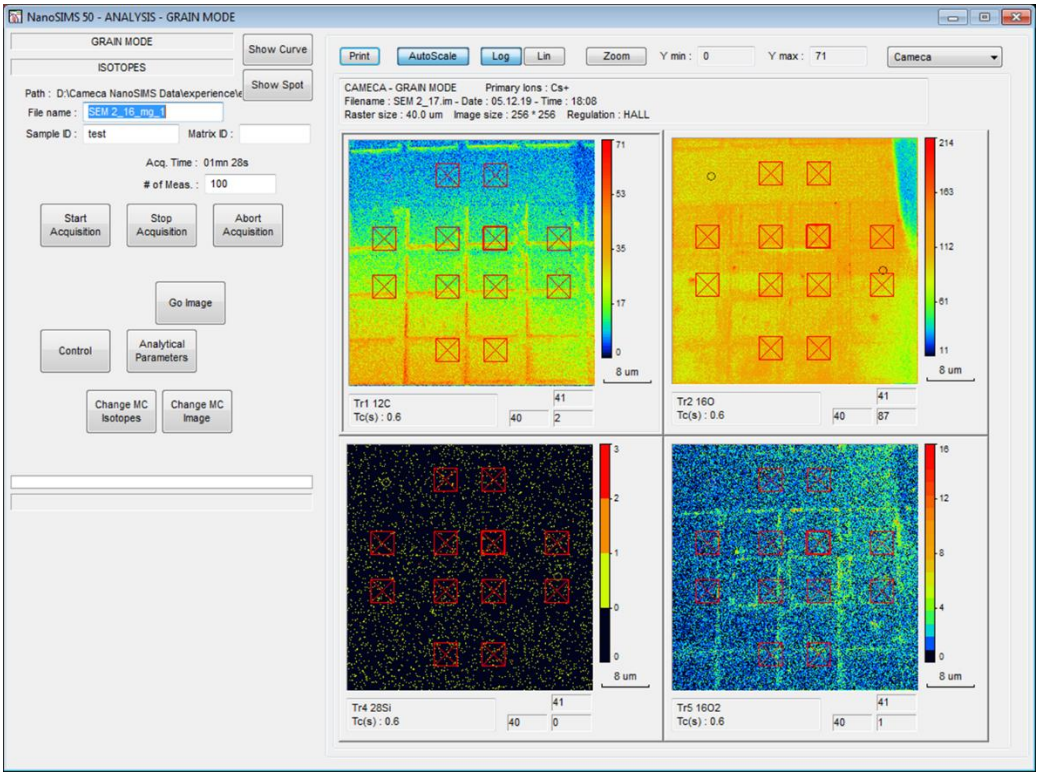


Figure 373: Grain mode: Analysis window in "Isotopes" and "Spec" modes

9.2.6.4 Line Scan (Stage scan)

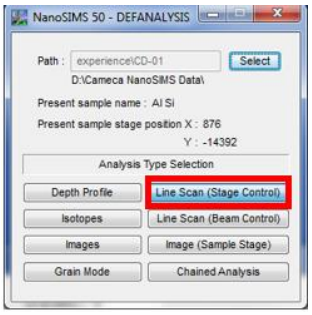


Figure 374: DEFANALYSIS: Line-scan by Stage scan, selection

This mode allows the user to make a lateral isotopic profile moving the stage step by step.
It is usually used for lines exceeding the dimension of images (e.g. > 40-50µm) or/and when long acquisition time/pixel is needed for statistical reasons and where the recording of a full image would take too long.

In this mode, the starting point must be set beforehand in CCD mode. Then in SIMS mode, when opening the “Line Scan (stage Control)” mode, (Figure 375), the user can define the number of steps and the distance in X and Y between steps.

The scanning mode is optional. However, even if the scanning mode is selected, no image will be recorded. Once the analysis is done, the table of results data are stored in a file with *.is_txt extension. The data can be imported and processed through Excel via the “import text/csv” module.

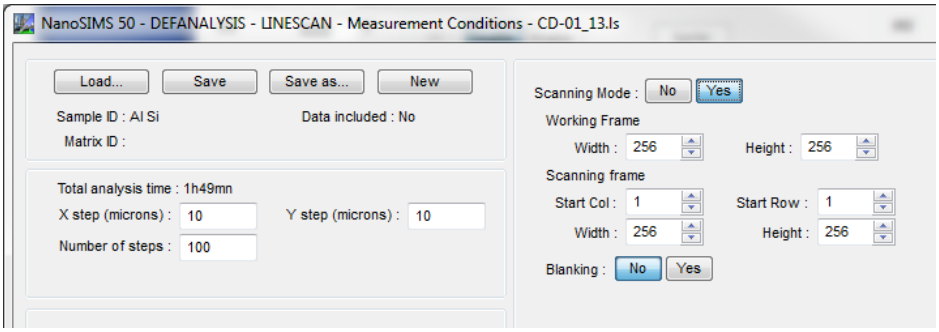
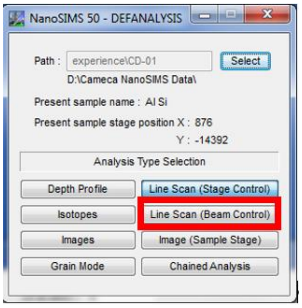


Figure 375: DefAnalysis options in Line Scan (Stage scan) mode

9.2.6.5 Line Scan (Beam scan)



For small line scans, it is also possible to move the beam electrostatically.

- Click on “Line Scan (Beam Control)”. Def Analysis opens an “Image” window, allowing the user to first acquire an image of the area.

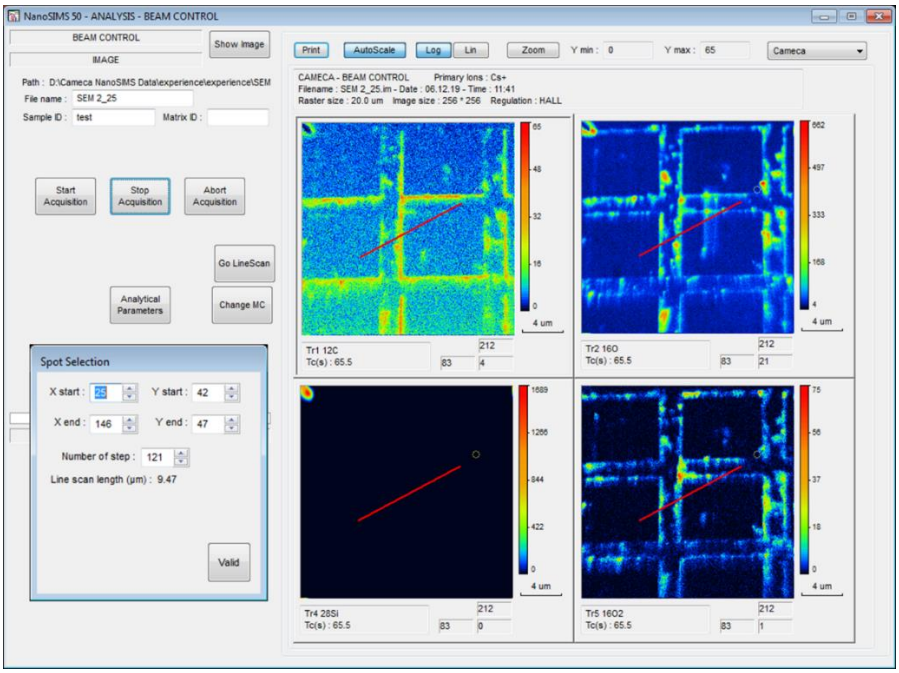


Figure 376: DEFANALYSIS: drawing the line prior a linescan (beam control)

- Once the image is acquired, draw a line by clicking and dragging the cursor over the image (Figure 376). You can adjust the position of the line by modifying its coordinates via the “spot selection” window.
- Enter the number of steps, depending on beam size, (optional) raster size and desired pixelization.
- Then click on “Valid” and “Go LineScan”.

- A new Def Analysis window opens, allowing you to set the parameters for the line scan analysis (Figure 377). You can define the number of cycles over the line scan (cycle number). You can also choose to do the line scan
    - with the scanning mode off.
    - With the scanning mode on. In this case you can define the size of the scanning frame with Delta X and Y following the equation: scanning frame = (2*DeltaX + 1)(2*DeltaY +1)

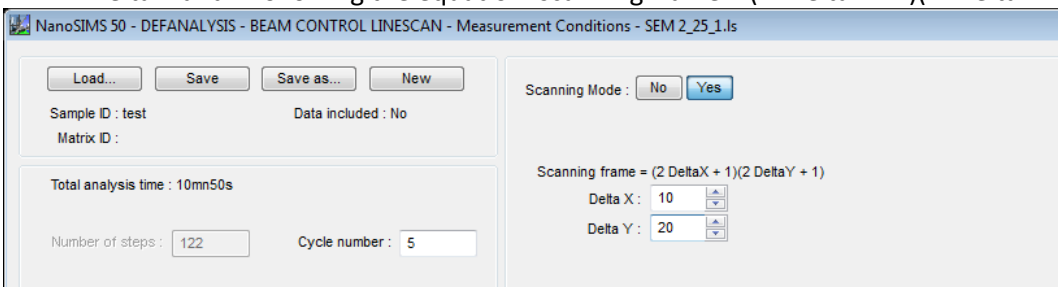


Figure 377: options of the Def Analysis in linescan mode

- When everything is set, “Go Acquisition” and in Analysis “**Start Acquisition**.”

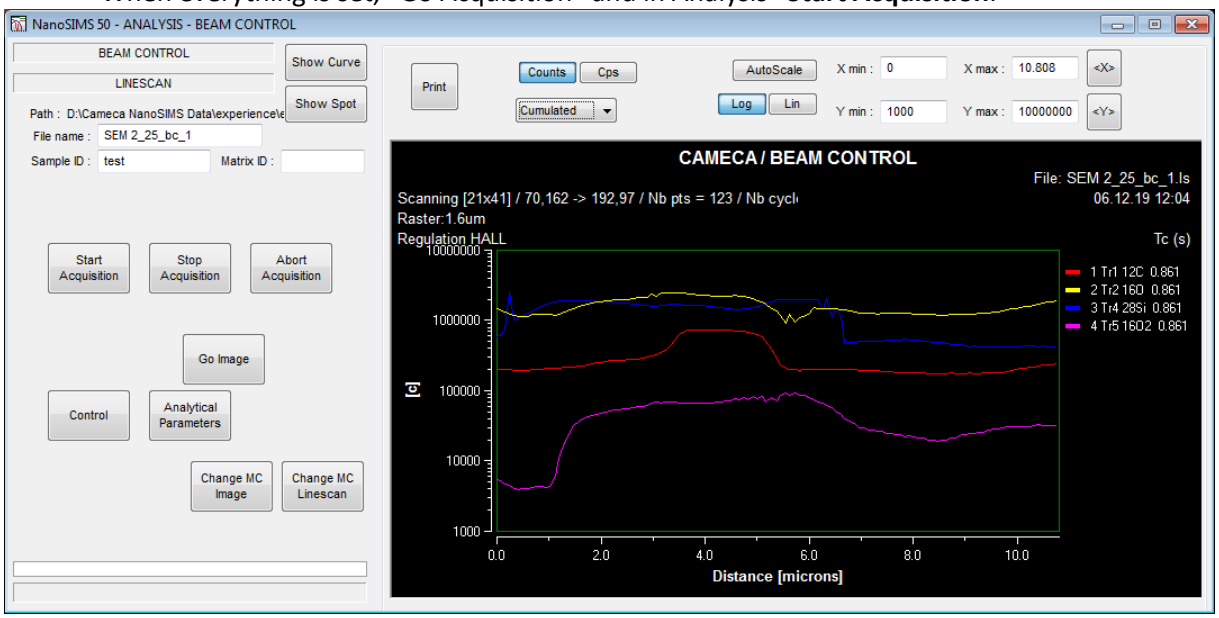


Figure 378: analysis during a linescan acquisition

- The table of results data are stored in a file with *.is_txt extension. The data can be imported and processed through Excel via the “import text/csv” module.

- When the analysis is over, the program shows the option “go image” to acquire a second image of the area in the exact same conditions as the original image. This can be useful to record an image of the scanned area.

9.2.6.6 Images (Stage Raster)

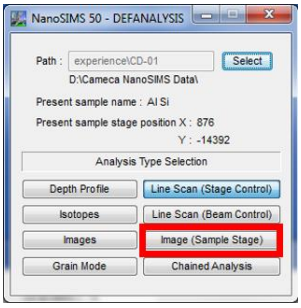


Figure 379: DEFANALYSIS: Stage Raster selection

The NanoSIMS is limited by design to "small" FOVs, i.e. 50x50µm. When the user wishes to map a very large area (e.g. a few hundreds microns width) keeping high spatial resolution, it is possible to record a mosaic of small images using Chained Analysis (see chapter 9.2.6.7) and stitch them together with WinImage software.

Instead this Stage Raster imaging mode is more intended for ***fast screening*** of very large areas (up to several mm) at more modest lateral resolution and high current.
Another typical use can be a ***large area pre-implantation***, before a navigation searching for interesting areas at smaller scale.
Let's take a numerical example:
Cs+/ 100pA / 0.7µm spot. Recording a 128 x 128 (mechanical) pixel image over 500x500µm is possible. But the pixel (=step) size being 500/128 = 3.9µm, and the spot size being 0.7 µm the image will be underpixelated (a series of holes with untouched sample in between). One can then add in such case a (fast) electrostatic scanning of say 8x8µm raster size of 16x16 pixels to be sure not to miss sample area.
See Figure 380 for the definition of parameters:

The user first defines the width and height of the whole image (in microns) and the number of stage steps (mechanical pixels) to cover the whole field.

The (electrostatic) scanning mode is optional (recommended for large areas as explained above).
The Ct/pt (s) parameter to fill next is the requested time spent per mechanical pixel or step. The total acquisition time is calculated depending on the number of pixels. A mechanical stage movement can not be as fast as an electrical beam deflection; hence times are usually in the range of seconds. This leads rapidly to Total analysis time reaching hours.

The dwelltime/pixel of the electrostatic image is automatically adjusted depending on the number of selected pixels of the scanning frame. Only on cycle (= frame) is available.

For each position of the stage, the program will sum all signals from the electrostatic image (if any) to generate one pixel of the stage scan image. The data is finally saved as a regular *.im image file, to be opened in WinImage.

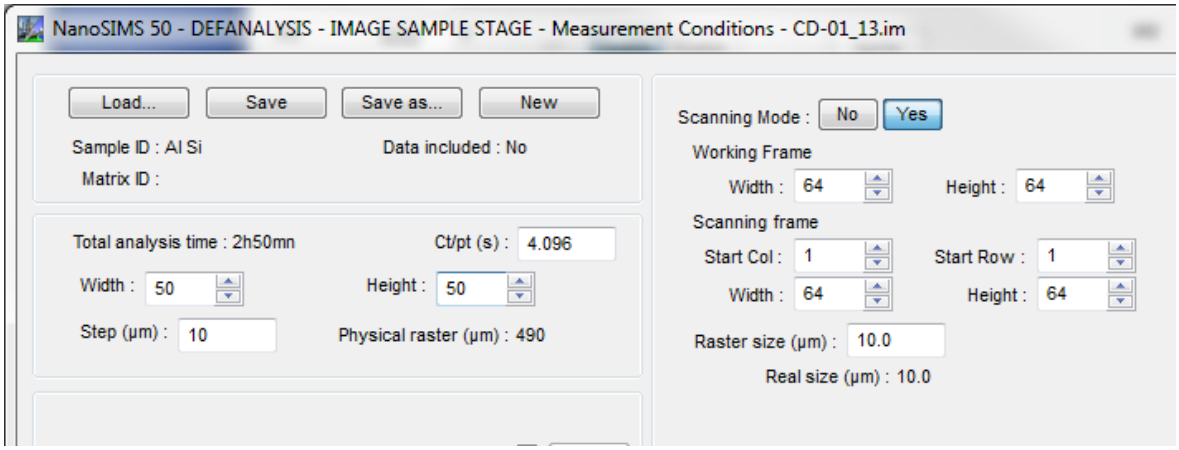


Figure 380: Def Analysis options for the Image with sample raster mode

Note: Once saved the parameters can be quickly recalled and this Stage Raster program can also be used for unattended reactive ion **pre-implantation over large areas** in preparation for images.
If the sample is then maintained under UHV, the subsequent image acquisitions on small selected FOVs inside this large area will require limited or no pre-implantation before acquisition.

9.2.6.7 Chained Analysis

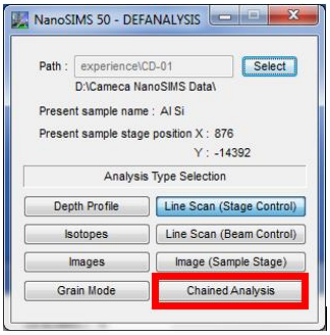


Figure 381: DEFANALYSIS: Chained Analysis selection

The **Chained Analysis** program allows the user to schedule a series of analyses or actions and launch the sequence to run automatically.
The main concept to remember is that it is necessary to run and save first an analysis that will then serve as a "**Reference**" (containing the conditions and eventual presets) for the chained analysis. In the case of a chain that would mix different types of analyses or different settings, it is then necessary to run and save a reference analysis for each. Note that this automation is restricted to:

- same ion source & polarity,
- same ion source voltage (the program is not able to apply different source conditions),
- Using recent Reference files that can be reloaded without conflict (e.g. presets deleted or ion optic alignments retuned after a service).

Once the reference analysis(ses) are obtained, go to "Chained Analysis". The following window opens (Figure 382):

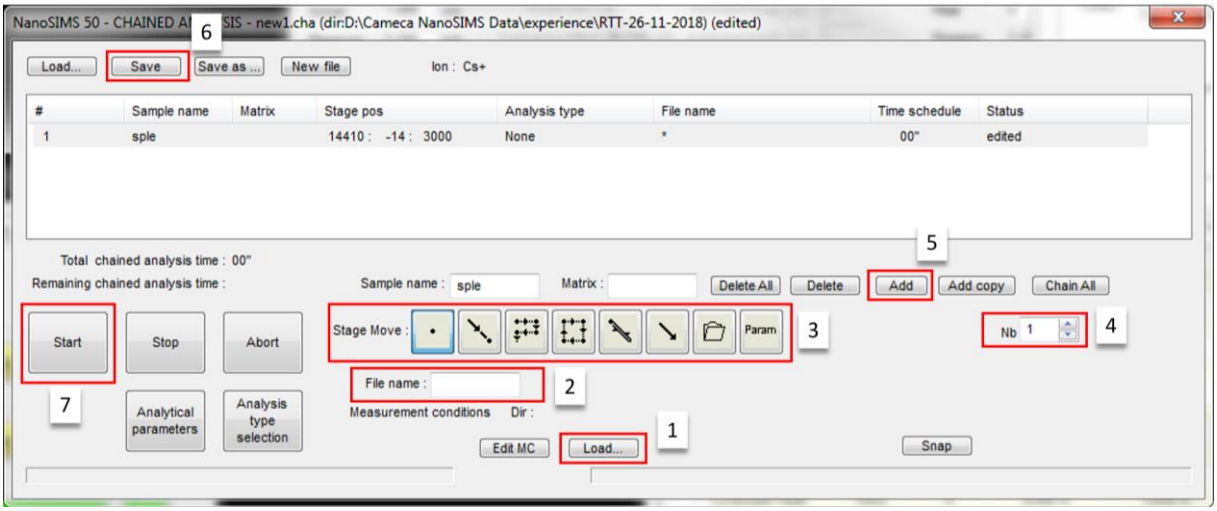


Figure 382: Chained Analysis window

To launch a chained analysis follow the steps below that are indicate on Figure 382:

1- Load the Reference analysis file (.im, .dp or .is)
2- Give a name to your chained sequence. Each analysis will be incremented as *name*_1, *name*_2, etc…
3- Select and document the Stage Move (see below for details on each options)
4- Enter the number of times the analysis/sequence will be repeated.
5- If you want to add a different series (from a different reference file or with a different stage move), select “add” to add a second line to the chain and follow the same procedure as before.
6- Save the chain (a .cha file will be saved in the root directory)
7- Click “Start” to launch the acquisition. The Analysis window will open, and the chain will automatically launch. You can interrupt the chain by clicking on “Stop” or “Abort”.

Stage Move types:

1-  The stage doesn’t move. All analyses will be recorded at the same stage position.
2-  The stage will move in line, by a step (in microns) defined by the user
3-  “snake”. The user defines the number of horizontal steps (Y), the step length (dY, in µm), the number of vertical steps (X) and the step length (dX, in µm). During the sequence, the stage will first move horizontally by the number of Y steps defined, then move vertically by the number of X steps defined, and finally move back horizontally by the number of Y steps. The stage makes a final vertical move (without analysis) as to be in position for the next “snake”.

Practical example 1: a horizontal mosaic of 8 images (4x2) of 50x50µm can be recorded by programing ONE snake: 3 horizontal Y moves of 40µm step (to keep an overlap of 10µm), and one vertical X move of 40µm before coming back.
Practical example 2: a square mosaic of 4x4 images can be recorded by setting the number of snake “Nb” = 2. Each snake being composed of 3 horizontal Y moves and 1 vertical X move.

4-  “square”. The user defines the number of horizontal steps, the step length (dY, in µm), the number of vertical steps (X) and the step length (dX, in µm). During the sequence, the stage moves horizontally then vertically, horizontally in the opposite direction, and then vertically to close the square
5-  The stage will move in line, by a step (in µm) defined by the user, then back in reverse to the point of origin.
6-  The stage will simply move without acquisition (this move can be useful when chaining different motives).

7-  Folder: clicking on this icon will open a new window allowing the user to select positions saved in the Navigator. To add a position, select it and click on “add”. Move the chosen positions up and down to rearrange the order of analysis.

8- 

(*Param* is not used)

Note that in order to save definition time it is possible to loop a chain by clicking on “chain all” and indicating the number of times one wishes to repeat the chain. E.g. for a set of 3 small area depth profiles performed, to be repeated at four locations (= test pads), one can just program two lines (the description of the three acquisitions and the stage movement) and repeat this four times with “Chain all” function.

9.2.7 Point logger navigation (using an imported sample image)

Point Logger is a navigation software allowing **navigation using a large field imported *.jpg format image** (e.g. SEM, optical, fluorescence, photocopier image of the holder,…) instead of the optical microscope or SIMS images. This feature can be extremely useful for example to navigate over *large distances inside a holder* hole or on an image of the sample *before metal coating* resulting in low contrast in the optical image. It can facilitate correlative microscopy between different instruments.

See the software description in chapter 6.2 containing all details on the use of Point Logger.

9.2.8 Ultra-low energy (ULE) pre-implantation/deposition

Note: earlier instruments did not have this capability that requires software and electronics updates.

In SIMS, surface enrichment of reactive species (cesium or oxygen) is necessary prior to the real useful measurement, in order to maximize the secondary ionization yield of elements. Most of the time it is performed with a first phase of **pre-implantation** in one of the following ways:

a) by using the current analysis conditions (it can be too slow, especially for large fields of view);
b) by increasing the beam current through the selection of a larger D1 diaphragm;
c) by combining this with an increased L1 value in order to further increase the beam current.

The possible inconvenience is that this implantation process will in parallel sputter a given volume of sample, which can be a problem for top surface analysis (few nm), very thin/limited samples, fragile or charging samples. This is not always an issue, as in some soft materials surface, atoms can be pushed/mixed to be ejected some nm deeper with better yield. Or when using a coating, the sample surface is reached when it is already mixed/enriched through the coating. In addition, this interface can still be smooth, especially if the coating has similar density as the sample.

But for **uncoated ultra-thin** layers or ultra-small features it is possible to use the alternative of ultra-low impact energy pre-implantation or rather **pre-deposition** mode: by polarizing the sample very close to the source voltage in a quasi-mirror mode and energizing lenses appropriately , Cs^+ or $O^{+/-}$ primary ions will reach the sample with an impact energy equal to the difference between ion source and sample voltages.
Below a threshold energy (50-100eV depending on ions and sample), sputtering stops and there is a deposition of primary species on the surface governed by the local sticking coefficient. However, note that ***this mode does not allow analysis*** during it. It is only aimed at **surface enrichment** before an analysis at higher energy that will benefit of a ***reduced transient regime***.
The primary ion beam size at a few tens eV energy is enlarged to several microns so this pre-implantation/pre-deposition is restrained mostly to “large” craters of a few tens microns.

9.2.8.1 Switching to ULE using the Setup settings

To switch to this ULE pre-implant mode, select the option “LE” (low energy) in the Tuning window and put the beam on. The primary beam impact energy will be reduced from e.g 16 000 eV to e.g.100 eV by switching the polarity of the sample HV (EOW) to +7900 volt (for + 8000v on the source). Other lens potentials will be adjusted as illustrated in Figure 383.

The incoming primary beam ions will be deposited “delicately” on the sample, without sputtering much of the surface. The sputter threshold varies depending on materials but is usually in the range 50-100eV, so the deposition or implantation energy will be adapted depending on the type of samples typically between a few tens eV to a few hundreds eV. The sticking coefficient will also vary from species to species.

Typical pre-implantation time is of 15-20 min at 2 pA for a FOV of 30-40 µm.

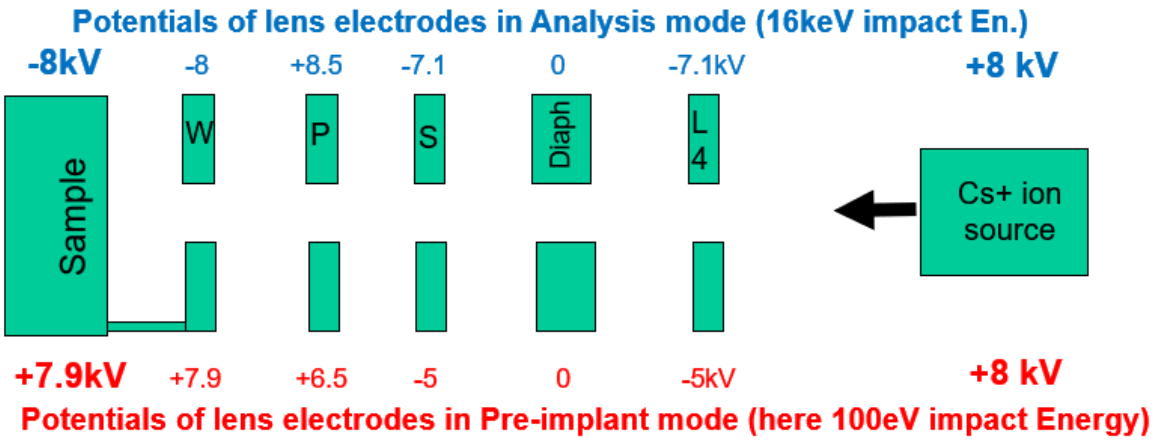


Figure 383 Example of different voltages applied to the optics in 16keV ANALYSIS (=HE) and 100eV (=LE) pre-implant modes.

Once the pre-implantation is done, click on “HE” (high energy) to restore the sample and other lens voltages for analysis mode.

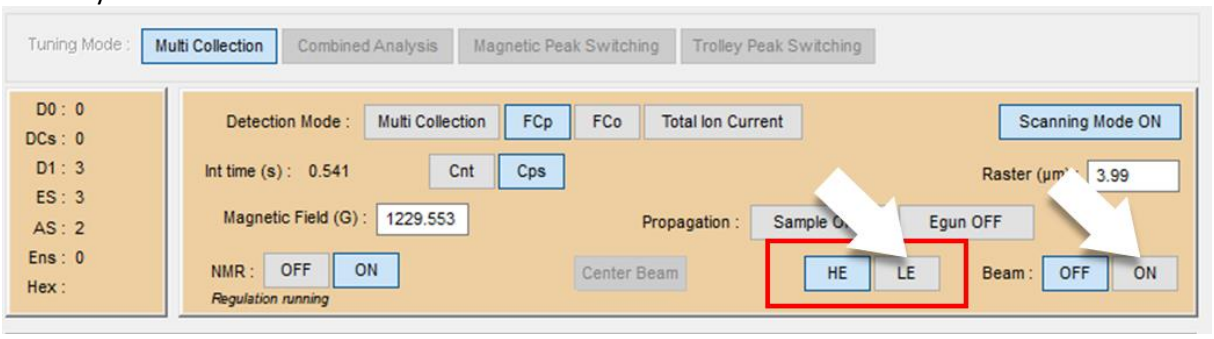


Figure 384: Low Energy option in the Tuning window

It is also possible to program such a ultra-low energy pre-implantation as part of the “pre-sputtering”, before an analysis. To do so, simply select the pre-sputtering option in Def Analysis and within, select “low E” (Figure 385)

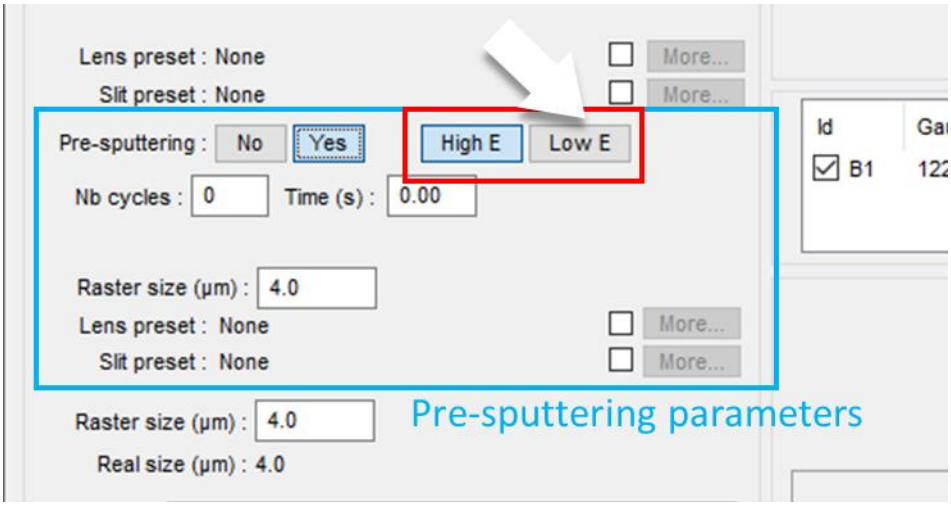


Figure 385: Def Analysis : High Energy or Ultra Low Energy pre-sputtering option

It is important to note that the raster is affected by the lowering of impact energy. When using the LE mode, the actual raster size is no longer matching the raster value entered in TUNING. You must thus enter a raster value larger than the desired raster size following the empirical law:

$$raster_{SIZE} = 2 \times raster_{TUNING} + 2.9$$

Thus, if you want to implant a zone of 15x15 µm, you must enter a raster of 33 µm.

9.2.8.2 Adjusting the ULE parameters

When the option is available on your instrument, Cameca engineers have set a ULE preset (in the SETUP) for a deposition at 25 eV.
If you wish to change the ULE parameters, follow the procedure described below. **NOTE that this requires an habilitation to electricity and must only be performed by users with this electric knowledge.**

To proceed, it is recommended to use a Si wafer for Cs^+ and a Al/Cu sample for O^-. Before starting, the procedure, save two high current presets with D1-3 and high L1, for a current around 200 and 500 pA. They will be used later.

In the SETUP > Keyboard > Low Energy (Figure 386: Low Energy parameters from the SETUP), click on “ON” to activate the option and put L4 and E0S at 36000 bits.

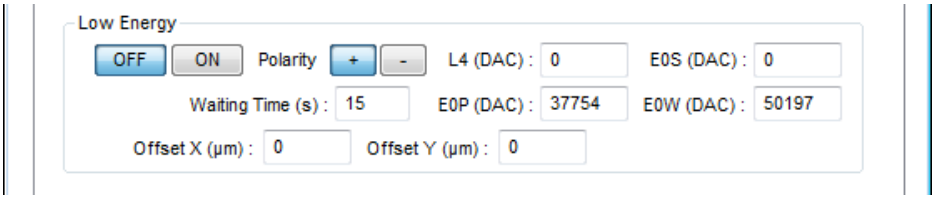


Figure 386: Low Energy parameters from the SETUP

In the Tuning window, click on “LE” to activate the ULE. This will switch the polarity of E0W.

Adjusting E0W:
With the keyboard, adjust E0W to 50833 bits. This is the theoretical value for a E0W at -8000V (with the Cs^+ source at +8000V) or at +8000 V (with the O^- source at -8000V). In those conditions, the source and E0W cancel each other out.

However, the exact voltage may vary by a few volts. You thus need to measure the exact voltage applied on the source and E0W. On the instrument, use a high voltage probe to precisely measure the voltage at the source and on E0W (Figure 387).

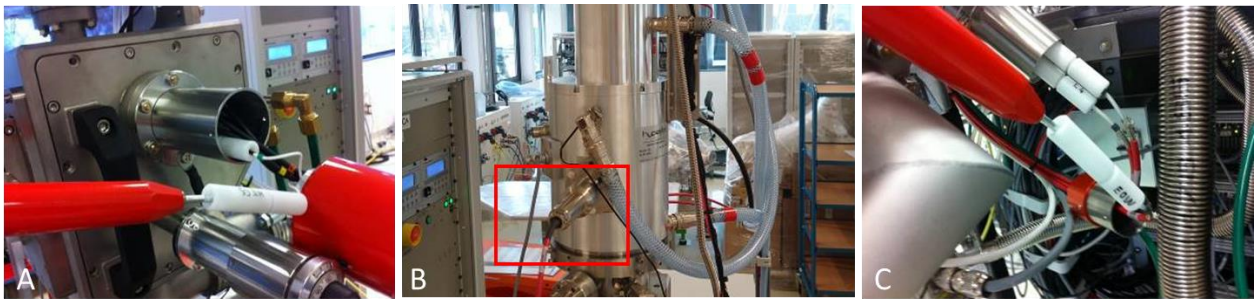


Figure 387: measuring the HV on the Cs source (A), the RF source (B) and E0W lens (C)

To apply a 25 eV acceleration to the deposition, apply the following equation:

$$E0W_{ULE} = 50833 \times (|HV_{measured,\ source}| - 25) / (|HV_{measured,\ E0W}|)$$

This equation can be generalized to any voltage (X):

$$E0W_{ULE} = 50833 \times (|HV_{measured,\ source}| - X) / (|HV_{measured,\ E0W}|)$$

Enter the new $E0W_{ULE}$ value in the *E0W (DAC)* field in the Setup (Keyboard > Low Energy). Make sure you are in the right polarity ('+' if in Cs^+, '–' if in O^-). Click on Apply.

Adjusting E0P:

Here, the goal is to reduce the size of the probe.

- In the SETUP > Keyboard > Low Energy, enter 33 000 in the *E0P (DAC)* field, and click on Apply.
- In TUNING, select "LE" and put the scanning mode OFF.
- Apply the high current preset previously saved.
- On your sample, go to a clean area and put the beam ON. Wait for about 60 seconds in order to obtain a clear mark of the beam on the sample (Note: in Oxygen, it might be necessary to wait longer in order to obtain a clear mark of the beam on the sample).
- Turn the beam OFF and go back to HE (= high energy, normal imaging conditions)
- Switch to a low current preset, put the raster to 120 µm, and the scanning mode ON.
- Open RTI to see the image. A small circle should appear, showing the mark of the beam in LE obtained above.
- Note the approximate size of the LE beam.
- Change E0P (with steps of 1000) in SETUP > Keyboard > Low Energy, Apply.
- Go to a clean zone on your sample and repeat the procedure until you obtain the E0P value that gives the smallest beam (Figure 388).

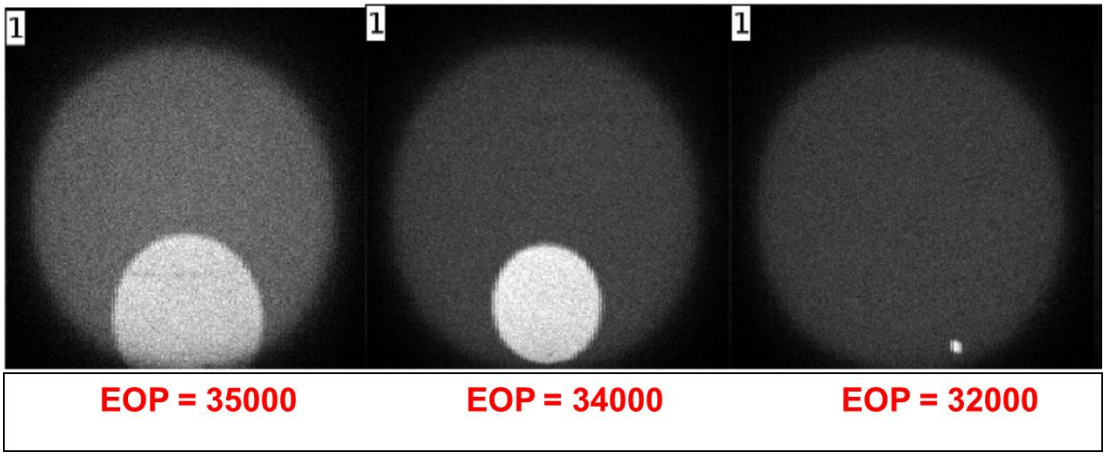


Figure 388: Influence of EOP on the beam size

Adjusting the beam position:

Once all lenses parameters are adjusted and applied in the Setup > Keyboard > Low Energy, you can adjust the X and Y offsets, so that the beam position will remain the same in high energy and low energy:

- In the SETUP > Keyboard > Low Energy, enter offset values (start with a few bits) in the *Offset X* and *Offset Y* fields. Apply
- In TUNING, select "LE" and put the scanning mode OFF.
- Apply the high current preset previously saved.
- On your sample, go to a clean area and put the beam ON. Wait for about 60 seconds in order to obtain a clear mark of the beam on the sample (Note: in Oxygen, it might be necessary to wait longer in order to obtain a clear mark of the beam on the sample).
- Turn the beam OFF and go back to HE (= high energy, normal imaging conditions)
- Switch to a low current preset, put the raster to 120 µm, and the scanning mode ON.
- Open RTI to see the image. A small circle should appear, showing the mark of the beam in LE obtained above.
- Note the position of the LE beam.
- Adjust *Offset X* and *Offset Y* in SETUP > Keyboard > Low Energy, Apply.
- Go to a clean zone on your sample and repeat the procedure until you obtain the beam mark appears at the center of the image.

9.2.9 Adjusting the CCD/SIMS offset

The (0,0) X,Y position of the NanoSIMS stage read in the Navigator is defined by the mass spectrometer axis. There are ~40mm between the CCD position and the SIMS position. In order to switch *accurately* between the CCD and the SIMS positions (keeping the same sample detail at the center of the FOV in both cases), the user must store in the SETUP the *exact* X and Y distances (offsets) between the two positions. Once this is performed, the (X,Y) coordinates of a small detail on the sample read in the Navigator appear the same in CCD mode and in SIMS mode, despite the fact that in reality the stage has been moved by the X-Y offset between the two positions.

To adjust precisely the (X,Y) CCD/SIMS offset value, use the following procedure. This should be re-adjusted at least at each stage dismounting (e.g. immersion lens cleaning):

STEP 0: Before adjusting the offset, make sure the beam position (blue cross) is selected in OPTICAL IMAGE and at the center of the window. If not:

- To show the blue cross: in the menu "view", select "beam position."

- To position the blue cross at the center of the field of view, in the menu “tools”, click on “beam position adjust”, then click at the center of the CCD field of view. This will position the blue cross at the center.

STEP 1: In the NAVIGATOR, select the **SIMS mode**. In TUNING, choose either the TIC or multi-collection mode, and open the RTI. Find a noticeable feature that will also be easy to find in CCD (such as the edge of a grain or sample) and move it with the sample stage to the center of the RTI window (blue cross = beam position). Note the X and Y coordinates as noted in the NAVIGATOR (X_{SIMS}, Y_{SIMS}).

STEP 2: In the NAVIGATOR, select now the **CCD mode** and find your previously selected feature. Move the stage so as to place it in the center of the CCD image (blue cross). Note the X and Y coordinates (X_{CCD}, Y_{CCD}).

STEP 3: In SETUP → Holder, (chapter 5.9.2) note the previously saved X and Y offsets ($X_{old\ offset}$, $Y_{old\ offset}$).
Calculate the new offset values:
$X_{New\ offset} = X_{old\ offset} – (X_{SIMS} - X_{CCD})$
$Y_{New\ offset} = Y_{old\ offset} – (Y_{SIMS} - Y_{CCD})$
Enter those new values in the X and Y offsets in SETUP → Holder and *Apply*.

Switching now between SIMS and optical microscope should be accurate within the mechanical construction specification (for a new stage, within +/- 5µm @ 1 sigma over more than ten movements)

9.2.10 Adjusting the Magnetic Field calibration

Because of the hysteresis of the magnet, the calibration between the magnetic field and masses changes with time and mass jumps. It is independent for each detector and can be readjusted following the procedure below for each detector:

1. Adjust the magnetic field to maximize the signal of a known peak (the middle of the flat top if there is one) on the chosen detector. It is recommended to use a known and easy standard so that there wouldn’t be any ambiguity when identifying the peak. For example, use a Si grid and set your detector on ^{28}Si.
2. In the detector panel (Figure 389), click on **Adjust**.
3. In the “Mass” box of the detector, **type in the theoretical mass value** of the isotope (27.977 for ^{28}Si).
4. Click on **Valid**.

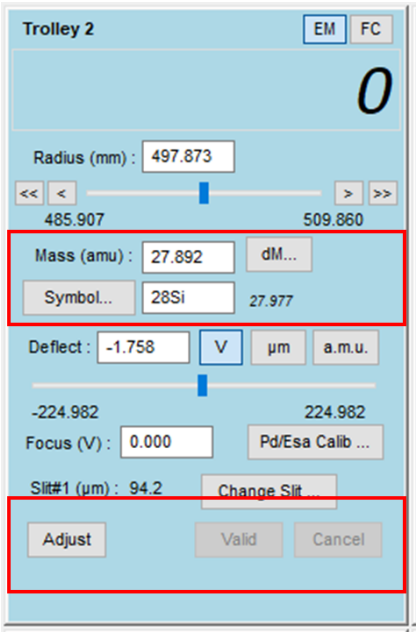


Figure 389: detector panel useful to calibrate the Magnetic field for a given detector.

This feature can be very useful to help identify peaks on an unknown sample (by measuring the mass difference with a known peak for example), however it needs to be used with caution, as a wrong calibration will set the whole table off for this detector, making it even more difficult to identify peaks!

9.2.11 Automated Switch of detector (EM/ FC)

To switch a detector from EM to FC on one of the six mobile trolleys on a NS50L equipped with this automation (and the 7FC option), proceed as follow:

1- In TUNING, on the detector window (Figure 390) click on FC.

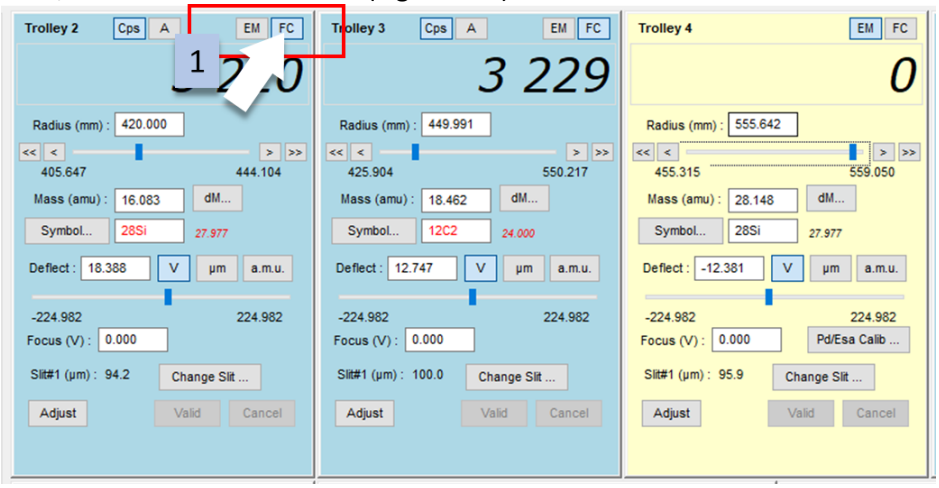


Figure 390: EM/FC button in the detector window of the TUNING

A new window pops-up (Figure 391, left).

2- Click on “Start”. All necessary trolleys will automatically move to a parking position (either high or low, depending on which trolley is being switched) to free the way so that the selected trolley can move to its EM/FC exchange position. Parking and Exchange positions have all been defined in factory and are recorded in the SETUP. They should not be changed.

3- When all trolleys have stopped moving, click on “continue”. The trolley will be switched to FC mode.

4- When all motors have stopped moving, click on “continue”. All trolleys will move back to their initial position.
5- When all trolleys have stopped moving, click on “Quit”. In the SETUP > Tuning, check that the detector has been switched to “FC” mode. You should also now see in TUNING a baseline of noise on the detector (a few thousands cps), even though the beam is off (see chapter 9.2.13 on how to adjust the baseline).
6- You can now repeat the procedure for the next detector you wish to switch.

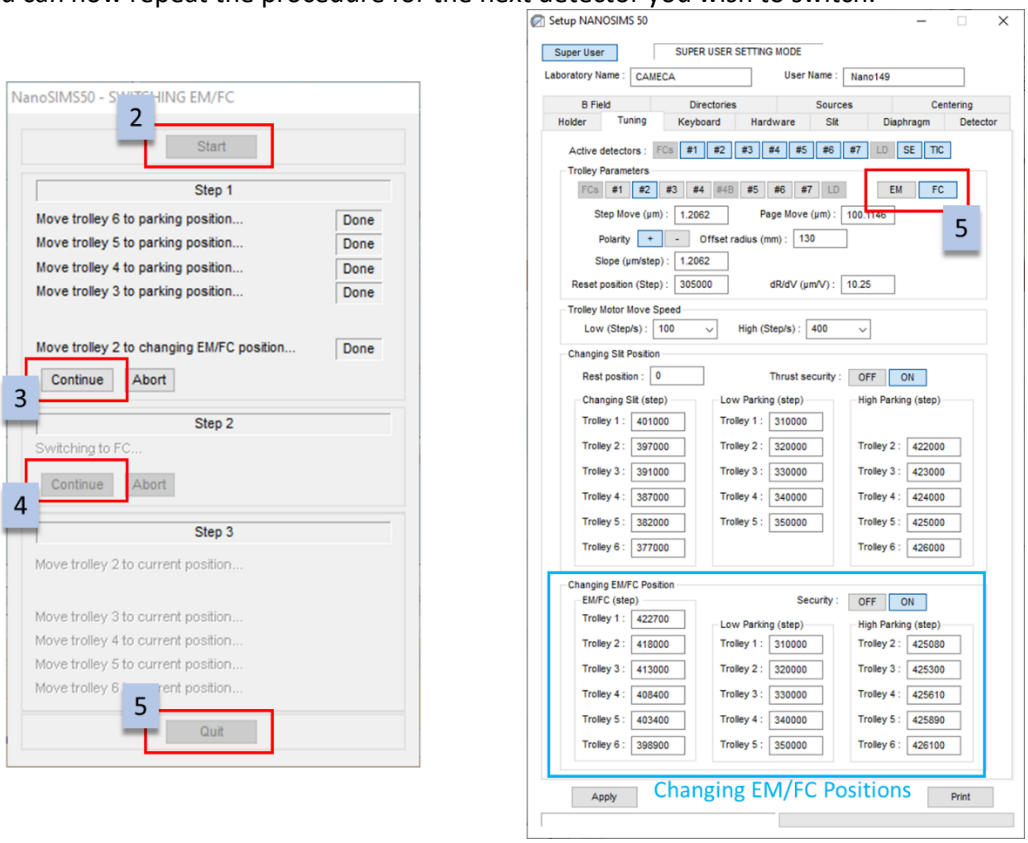


Figure 391: Left: EM-FC switching window. Right: SETUP window (Tuning tab) showing the EM/FC exchange and parking positions and the configuration of the detector (here, Trolley 2 is in FC mode).

To switch back from FC to EM, follow the exact same procedure reversely. Check in the Setup at the end that the detectors are well back in EM mode.

In Setup → Centering, adjust the waiting times (WT)

- For Ems: WT9 = 2s, WT0 = 3s, WT1 = 3s (5s when using the e-gun at high current)
- For FCs: WT9 = 10s, WT0 = 10s, WT1 = 10s

9.2.12 Switching the FC pre-amplifier resistor (1 E10 / 1 E11 ohm)

Faraday Cup pre-amplifier boards are equipped with two resistors of different values:
- A 10^{10} Ω resistor, suited for secondary ion current up to 1000 pA (=1E-9/1.6 E-19 = 6.25 E9 c/s),
- A 10^{11} Ω resistor, suited for secondary ion current up to 100 pA (=1E-10/1.6 E-19 = 6.25 E8 c/s).

Depending on the signal measured, it is thus necessary to use one or the other. The 10^{11} Ω one is better suited for small signals but will saturate at 100 pA. Thus, when measuring a signal above 100 pA, it is necessary to switch to a 10^{10} Ω resistor. While both resistors are present on the board, the switch must be

done manually. The Multicollection FC electronics is located in the thermostated cylindric box above the multicollection chamber (Figure 392).

Figure 392: thermostated chamber holding the FC electronics.

To access the FC pre-amplifier board, remove the chamber cap, the foam and unscrew the metallic plate (Figure 392). Note that while the lower part of the chamber is under vacuum, this upper part is not. Thus, no venting/pumping procedure is needed for this operation.
Figure 393 shows a schematic and a photo of the FC pre-amplifier board. The labels FC1 to FC7 correspond to the FC of detectors 1 to 7. FC8 is not used.

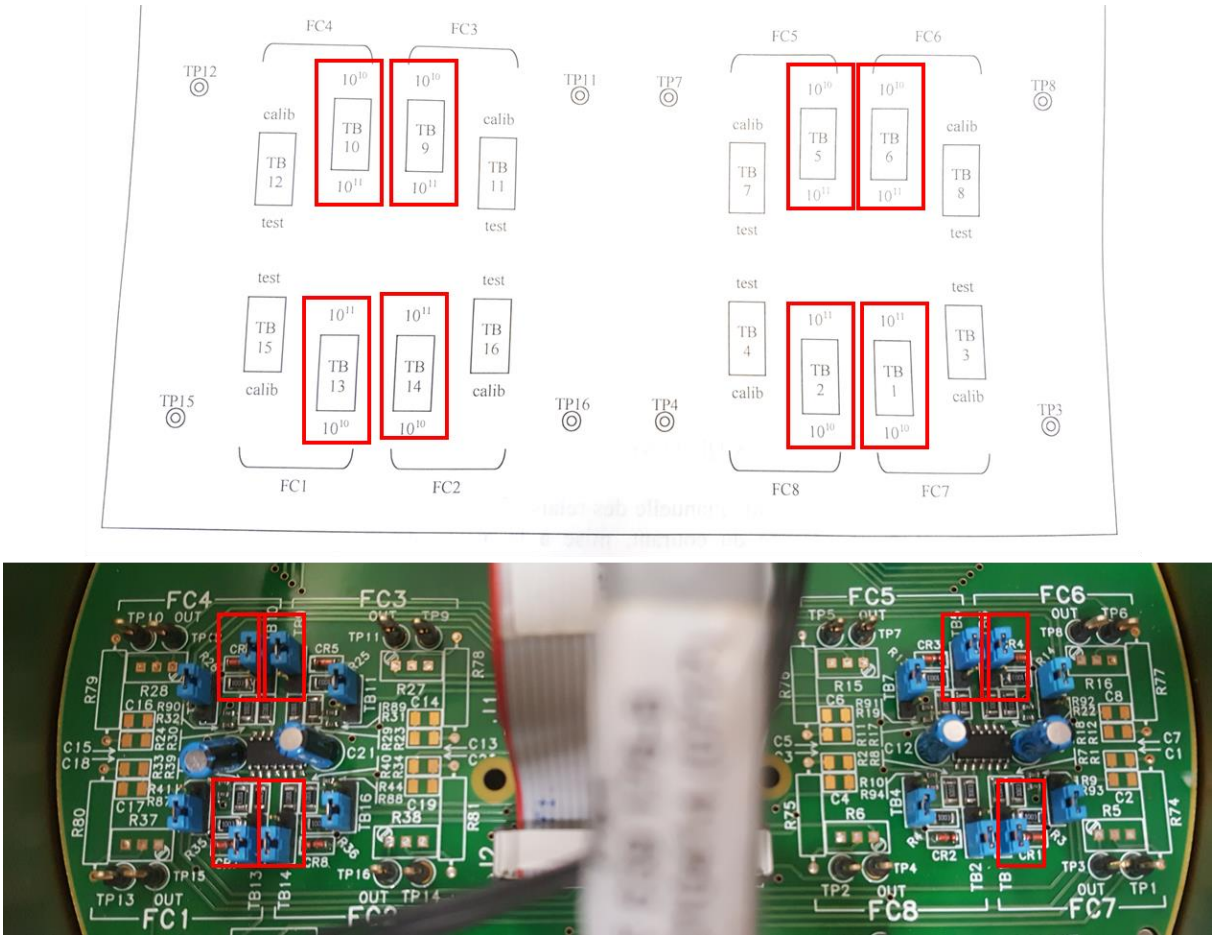


Figure 393: schematic and picture of the FC pre-amplifier board.

Each FC has an independent circuit, where a jumper can be switched between two positions to connect either the $10^{10}\,\Omega$ or the $10^{11}\,\Omega$ resistor. Figure 394 shows the 2 positions of the jumper, which connects two of three pins on the board. For all FC circuits, the positions are as follow:

- To use the $10^{10}\,\Omega$ resistor, put the jumper in outer position (the jumper connects the outer pin with the center pin).
- To use the $10^{11}\,\Omega$ resistor, put the jumper in inner position (the jumper connects the center pin with the inner pin).

Important note: *it is crucial not touching the boards with the hands or dirty gloves/tools, and above all avoid touching the high resistors. Any grease or other contaminant can introduce electrical conductivity change and lead to poor electrometer performance !!!*

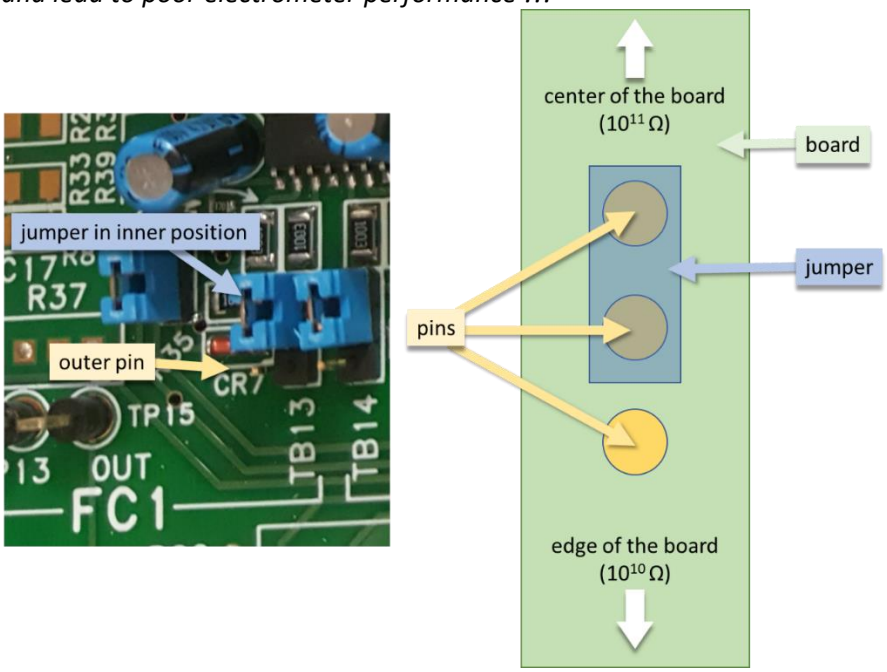


Figure 394: (left) zoom on the jumper of FC1. Here the jumper is in inner position, covering the center and the inner pins. The outer pin is visible. FC1 is thus in $10^{11}\,\Omega$ mode. (right) schematic of the FC1 jumper position.

Each time a switch is done, it is necessary to put it in the SETUP. In the detection section of the Hardware tab (Figure 395), select the FC you have switched and then select the correct resistor values. Do not forget to click on Apply to validate the change.

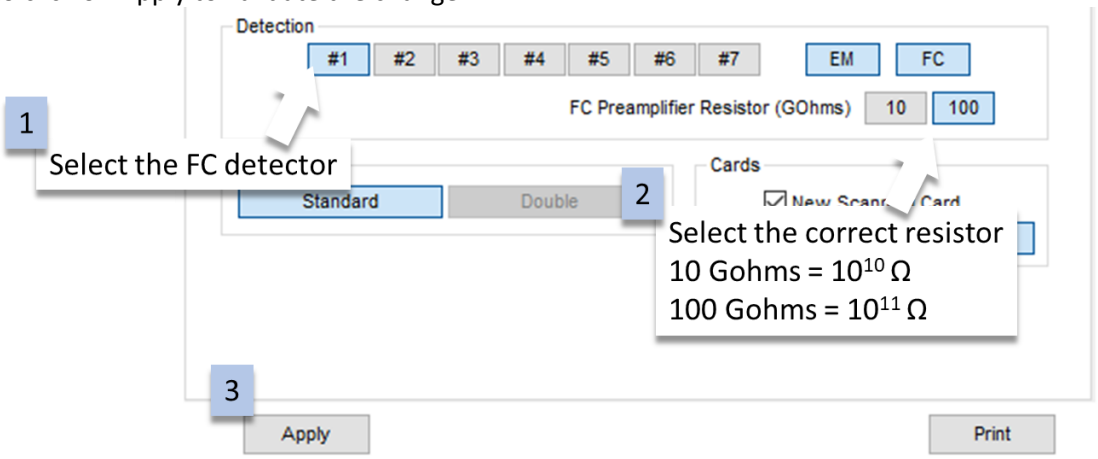


Figure 395: SETUP > hardware > Detection. Select the resistor value of the FC pre-amplifier

9.2.13 Faraday Cup calibration and background adjustment.

9.2.13.1 FC Gain Calibration

When switching from EM to FC or when switching the FC pre-amplifier resistor or typically once a week, it is necessary to do a FC gain calibration to make sure the FC are working properly. The principle is to switch

successively a calibrated electronic voltage source (0V, 9V, -9V and 0V) on the different preamplifiers, measure the various responses and calibrate them numerically in order to get a uniform gain and offset.

The procedure is computerized, via the FC Calib function in TUNING:

1. Open the “FC Calib” program (Figure 396).
2. Select the FC you are using.
3. Set waiting time and counting time both at 10 seconds and click on START.
4. Let the program run. When it’s done, “FC-cal” must be close to 1 (±0.02) for all checked FC.
5. If everything is in order, click on “Send to Setup”.
6. Close the program.

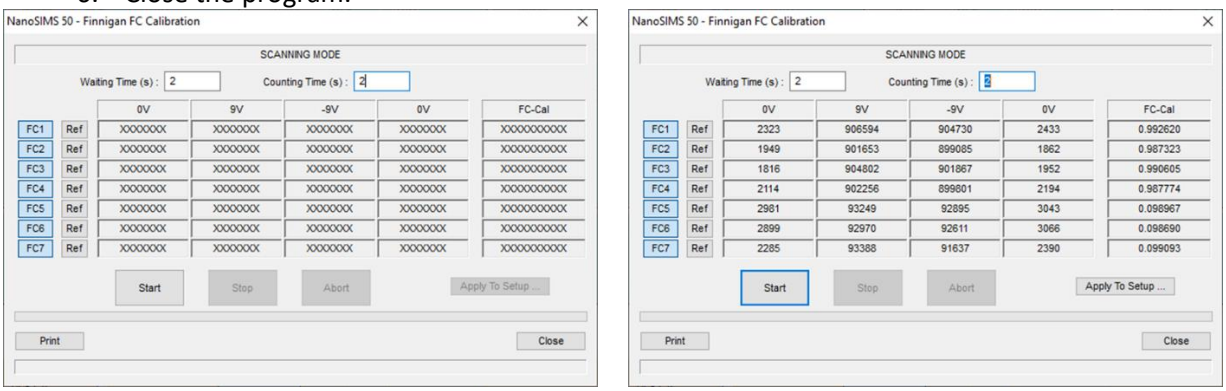


Figure 396: FC calibration program before (left) and after (right) calibration.

9.2.13.2 Background adjustment

A Faraday Cup and its pre-amplifier always show a background noise, usually around 3000 count per seconds on the NS50L. This background current must be measured precisely for each detector set in FC mode, stored in a Set-up file and subtracted to each subsequent secondary ion current measurement. It is critical for maintaining best reproducibility over long term especially for low FC signals. This is checked everyday or for the purists before every series of high precision measurements (e.g. during the pre-sputtering, with the spectrometer blanked).

This should be done in both polarities (positive and negative secondary ions).

To adjust the FC background level,

1. open the “Beam Stab” program
2. select all necessary FC detectors and put the display units in cps
3. with the beam OFF, let it run for about 5 minutes
4. Stop the acquisition and select each detector one by one to read the average signal
5. In the SETUP, enter the background signal in c/s for each FC detector and click on “Apply” (Figure 397).

In Tuning, put the signal reading in A and check that it’s at 0 pA (±0.05).
Typically, when starting a high precision session in FC one should always check the background level to be zero on all used FCs.

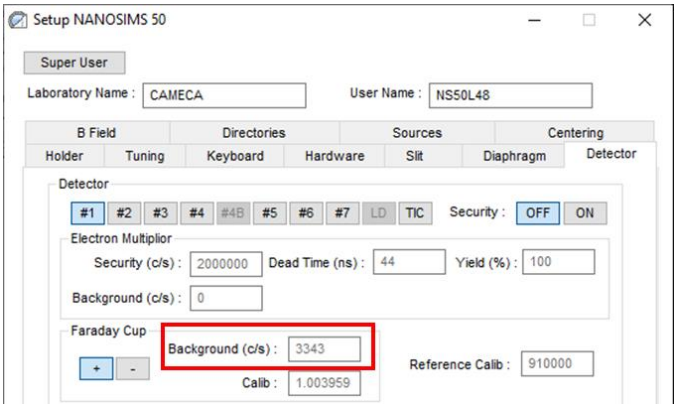


Figure 397: adjust the FC background in the SETUP

9.3 Expert operation

9.3.1 Primary and secondary Beam tuning

It is better to tune the instrument first on a standard sample such as a Si grid (when the primary beam is positive) or Al grid (when the secondary is negative). While it does not matter for the primary beam tuning, it will be useful to tune the secondary and multicollection.

9.3.1.1 Primary beam tuning

9.3.1.1.1 Cs+ beam tuning

1- Start the Cs^+ source with Ionizer and Reservoir values used the last time (HV = 8000 V), and wait 30 to 45 min for it to stabilize. The primary column should be as follow: D0-0, L0 = 0, C0 X = 0, C0 Y = 0, L1 = 0, C1 X = 0, C1 Y = 0, D1 = 1.
2- In FCp mode (beam OFF), adjust the gonio on the source block to optimize the intensity of the primary current in FCp (it should be around 1.1 mm). You should obtain the same intensity as during the previous run (50 nA is recommended).
3- Switch to FCo mode in the *Navigator* and the *Tuning* window. Put the beam ON and a raster of 10 µm. Select diaphragm D1-3. On the keyboard, select L1 and Def 2 to display L1, C1 X and C1 Y. Start with L1 = 0, C1 X = 0 and C1 Y = 0.
4- Increase progressively L1. FCo should increase. When FCo starts decreasing again, adjust C1 X and C1 Y to maximize the current in FCo. Continue to increase L1 and readjusting C1 X-Y until you reach L1 = 27 000. FCo should be around 10 nA.
5- Put C1 X back to 0 and readjust the gonio to maximize the current intensity in FCo.
6- To make sure the primary beam is properly aligned, vary L1 from 0 to 27 000. It should increase continuously.

9.3.1.1.2 RF-Plasma beam tuning

1- In FCp mode, adjust the gonio to maximize the intensity of the primary current in FCp.
2- On the keyboard, select LDuo. It shows LDuo, CDuo X and CDuo Y. Adjust CDuo X and CDuo Y to improve the current. *Do not* touch LDuo.
3- In the Tuning window, insert a D0 diaphragme (D0-1) and via the keyboard (D0) adjust D0 X and D0 Y back and forth until you reach the maximum current. In Tuning, click on “calib” to memorize the position of D0-1 (D0 X and Y will be sent to the *Setup* file)
4- If necessary, you may check the centering of the Wien Filter. Check on the keyboard that the WF Coil is set at 26200 (= 2 amperes). In the tuning window, check that you are in FCp mode. Then open the Wien Filter window. Set the scan as: Start = -40, step = 0.3, number of points = 130. Let the range in “auto”, put the scan in “log” and press START.
5- With a gas bottle of pure oxygen you should see 3 peaks, as shown on Figure 398. From left to right: O, O_2, O_3. Center the cursor on the desired peak – usually O^- and O_2^+ (by default the program will center on the most intense, thus O).
Using pure oxygen, O^- and O^+ will be the most intense. The relative height of O_2 and O_3 varies depending on source (duoplasmatron or RF-plasma), polarity and on cesium contamination.
In most case O^- is used for ultimate resolution due to the higher source brightness but O_2^- and O_3^- can be interesting for reducing charge, improving depth resolution and sputtering yield, and ionization for some elements.

O_2^+ is preferred as positive ion due to a high source brightness, better sputtering and depth resolution compared to O^+, and a higher ionization for some elements.

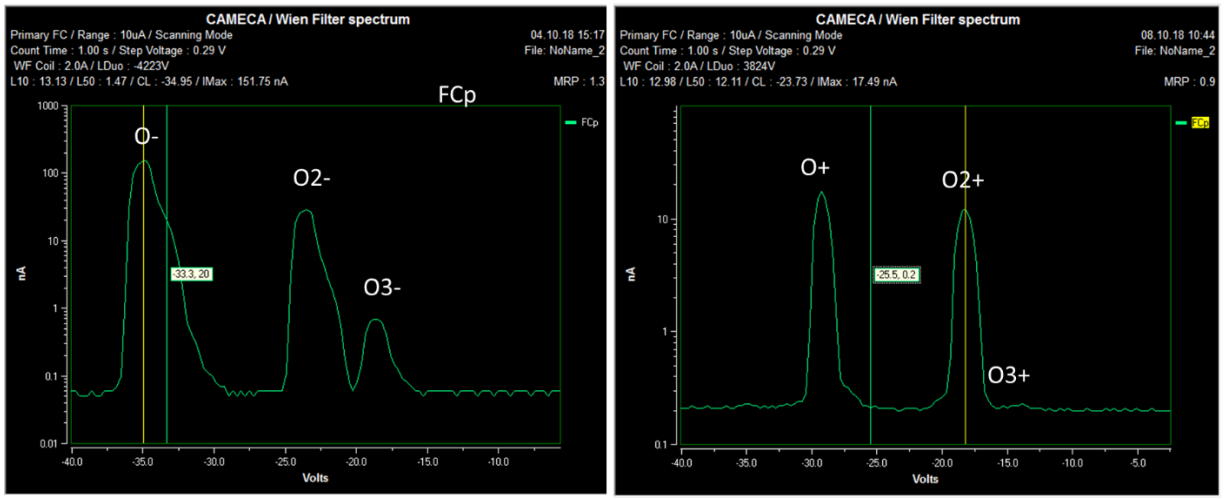


Figure 398: scan of the Wien Filter in both negative (left) and positive (right) modes.

6- Switch to FCo mode in the Navigator and Tuning windows.
Note: In the FC reading in the Tuning window, make sure the selected polarity (+/-) for FCo is the same as the primary beam. Put the beam ON and apply a raster of 10 µm. Insert the diaphragm D1-3.
7- Adjust D0-1 X and Y as well as CDuo X and Y to optimize the intensity of the primary current in FCo. Save the Wien Filter preset.
8- Put the beam OFF and switch to SIMS mode in the Navigator.

9.3.1.2 Co-axial lens tuning

1- Adjust sample Z: Open the "Optical Image" window. In the Navigator window, go to CCD mode and visually adjust Z (steps of 50 or 100) to get the most focused image of the sample on the optical image. When it's done, go back to SIMS mode.
2- In the Tuning window, select the TIC tuning mode. This puts lenses LF2 and LF3, as well as sector SS100 to zero. Make sure ES and AS are all open (= 0). Select diaphragm D1-3, put the scanning mode ON, with a raster of 10 µm. Put the beam ON. On the keyboard, select EOS, and adjust Cy, P2/P3 and EOS (in this order) to maximize the TIC signal. Select LF2 and adjust C2 X and Y.
3- Click on RTI and start a real time image. Choose a large raster so that you can see the edges of the D1 diaphragm: if using D1-1, select a raster of 250 µm. For D1-2, a raster of 200 µm. D1-3, raster 120 µm. D1-4, raster 80 µm. D1-5, raster 60 µm. From the keyboard, adjust D1 X and Y to center D1 in the image. For each diaphragm size, when the image is centered, save the D1 X and Y positions by clicking on "calib" in the tuning window. The X and Y positions associated with each D1 size will be saved in the Setup file.
4- Check the centering of SS30. Select SS30 → SS30 Y and adjust SS30 Y so that in RTI mode, when adjusting Oct-45 (EOP → Oct-45), the image doesn't move vertically. The SS30 value should be around 41500 bits.
5- Readjust EOS to maximize the signal and proceed to adjust EOP. In RTI, choose a raster of 20x20 µm and adjust EOP, oct-90 and oct-45 (EOP → EOP, oct-90, oct-45) to sharpen the image and correct from distortion. Try to keep Oct-90 < ± 200 bits and Oct-45 < ± 150 bits.

9.3.1.3 Secondary beam tuning

9.3.1.3.1 Secondary tuning in TIC mode

1- Put the beam on and implant an area of 20x20 µm. Then put back the raster to 10x10 µm
2- Centering on the horizontal plane (LF3) by adjusting Cy and C2 Y:
   a. Tuning → Tools → Parameters → C2 Y
      i. Starting voltage: -50 V
      ii. Voltage steps: 2.5
      iii. 50 points
   b. Do a series of scans for LF3 = 0 and LF3 = 28 960 varying Cy. Note the ΔC2Y between LF3 = 0 and LF3 = 28960 until you find a Cy value where ΔC2Y < 20 bits. Set C2Y to an intermediary value and save the settings in the Setup file (Setup → keyboard → TIC. Apply) as well as in the Global Preset file (Valid).
3- **Centering on the vertical plane (LF2)** by adjusting P2, P3 and C2 X (Figure 399):
   a. Tuning → Tools → Parameters → C2 X
      i. Starting voltage: -50 V
      ii. Voltage steps: 2.5
      iii. 50 points
   b. Do a series of scans for LF2 = 0 and LF2 = 20 000 varying P2 and P3. Adjust P2 and P3 until you find a configuration where C2 X ≤ ±50 and ΔC2X ≤ ±20. Set C2 X to an intermediary value and save the settings in the Setup file (Setup → keyboard → TIC. Apply) as well as in the Global Preset file (Valid).
4- Switch to Multicollection mode in the Tuning window and adjust Cy, C2 X and C2 Y to the new values. Send those values in the preset (Preset → Global → Calib). Save the .isf Master File.

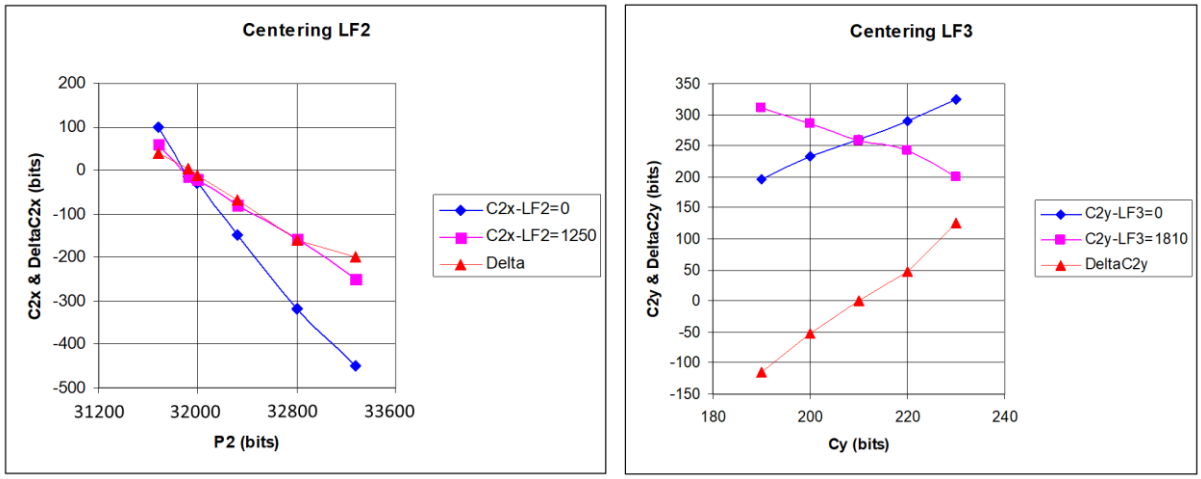


Figure 399: Centering of the beam in LF2 (left) and LF3 (right) in order to minimize aberrations along the secondary ion beam path. Optimum Cy is 210 bits with C2y set at 250 bits.

5- **Centering of the Entrance Slit.** Go back to TIC mode and manually adjust LF2 and LF3 to their multicollection values: LF2 = 20 000 and LF3 = 28960. Insert Entrance slit ES-3 and adjust ES X and ES Y to maximize the TIC signal. Adjust EOS to maximize the TIC signal, and readjust ES Y and ES Y. Calib ES-3.
6- Take out ES-3, and put LF2 and LF3 back to zero. Start RTI and make a TIC image. Adjust EOP, Oct-90 and Oct-45 until the image is perfect.

Here, it can be necessary to check the dynamic transfer tuning, especially if the mass spectrometer has to be used at high mass resolving power and if E0S has been largely modified:

7- **Adjust the dynamic transfer.** Do a 80x80 µm raster in order to pre-implant the sample, then reduce it to 50x50 µm. Insert ES-3. The image must be homogeneous. Adjust B2 X and Y if necessary.

9.3.1.3.2 Secondary tuning in multicollection mode

1- Switch to multicollection mode. With a Si grid in the analysis chamber, put ^{28}Si on a detector. Implant a large area (25x25 µm) then switch to 10x10 µm. Insert D1-3.
2- **Optimizing the transmission**. Insert ES-3 and adjust ES X and ES Y to maximize the signal on the detector. Select Calib ES-3.
3- Do a series of HMR and adjust Q to obtain the highest MRP possible. Adjust the HMR window settings to center the peak.
4- **Adjustment of the Hexapole**. In the Tuning window, go to Hex → centering
   a. Start with *horizontal*.
      i. Starting voltage: -5000 V
      ii. Voltage steps: 500
      iii. 15 points
      ➢ Apply value and Apply Setup.
   b. Start with *horizontal*.
      I. Starting voltage: -5000 V
      II. Voltage steps: 100
      III. 15 points
      ➢ Apply value and Apply Setup.
   c. DAC
      I. Starting voltage: 250
      II. Voltage steps: 25
      III. 10 points
      ➢ Apply value and Apply Setup.
5- Do a new HMR. The MRP should be better.
6- **Adjustment of the Aperture Slit**. Insert ES-3 and AS-2. Adjust AS X and AS Y to maximize the signal on the detector.
7- **Control of the beam height** by scanning C4 X
   a. Tuning → Tools → Parameters → C4 X in LOG scale
      i. Starting voltage: -27
      ii. Voltage steps: 0.3
      iii. 50 points
   b. Adjust C3 X (LF5 → Def 3 → C3 X) as to obtain a peak as symmetrical as possible when scan ing C4 X : **case (b)** in the Figure 400 below.

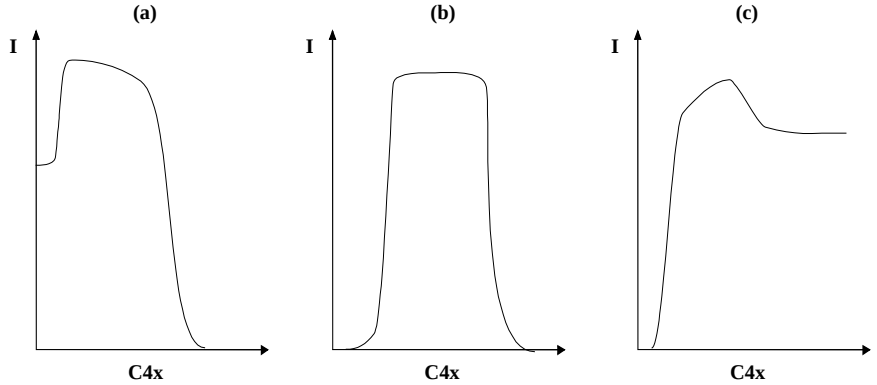


Figure 400: peak shape when tuning C3 X while scanning C4 X.

   c. Tuning → Tools → Parameters → C4 X in LIN
      i. Starting voltage: -27
      ii. Voltage steps: 0.3
      iii. 50 points

d. Adjust LF5 as to obtain peak edges as sharp (vertical) as possible.
e. Re-do a scan in Log to check the symmetry. Re-adjust C3X if necessary. Apply CL for C4 X.

8- Do another HMR scan and re-adjust Q if necessary.

9- **Adjustment of LF4**. Here we are looking for the LF4 value that gives the smallest ΔCL in an HMR scan when adding an offset of ± 5V to EOW. For each value of LF4, do a scan with an EOW offset of -5 V and another scan with an EOW offset of +5 V (EOW → offset = 32 bits = 5V). Adjust LF4 for the smallest ΔCL. When done, do not forget to put the EOW offset back to zero.

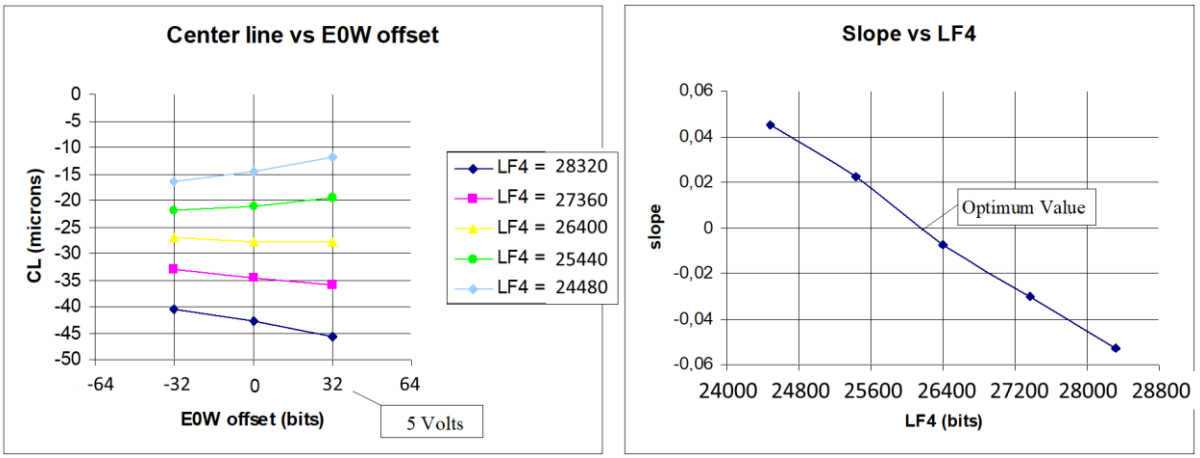


Figure 401: left: variation of peak position (HMR scan central line) with the E0W offset, depending of the value of LF4. Right: slope of E0W variation with LF4 value. The optimum LF4 value is for peak that does not move with E0W (E0W slope = 0).

10- Redo an HMR scan and Apply CL.

11- **Adjustment of SS100**. Here we are centering the beam in LF4 and the quadrupole, Q. We only adjust SS100 ext.

a. First, we look for the SS100 ext value that gives the smallest ΔCL in an HMR scan when adding an offset of ±1000 to LF4. Note that you may need to readjust the magnetic field to keep the signal on LF4. Restore LF4 to its original value.
b. Then we do the same with Q. We look for the SS100 ext values that gives the smallest ΔCL in an HMR scan when adding an offset of ± 5 to Q.
c. Set SS100 to an intermediary value between the best value for LF4 and the best value for Q. Readjust the magnetic field.
d. Readjust Q to optimize the MRP.

9.3.1.3.3 Reduction of mass fractionation at entrance slit ES using B-field coils

To check that the B-field coils are properly set, you must have several masses set on several detectors, with a good signal (> a few hundreds counts) and spread over a large mass range on the multicollection: e.g. 12C, 16O, 28Si, 28Si3, 197Au-, etc... Go to “Secondary Ion Beam” (TUNING > Sec. Ion Beam), select all used detectors and scan Cy (horizontal) and P3 (Vertical) in Log scale (Figure 402). The central line (CL) for all masses must be close (within 0.1 V). If certain masses have a CL significantly different from the others, you must adjust the horizontal and vertical B-field coils (Bf-hor and Bf-vert, respectively).

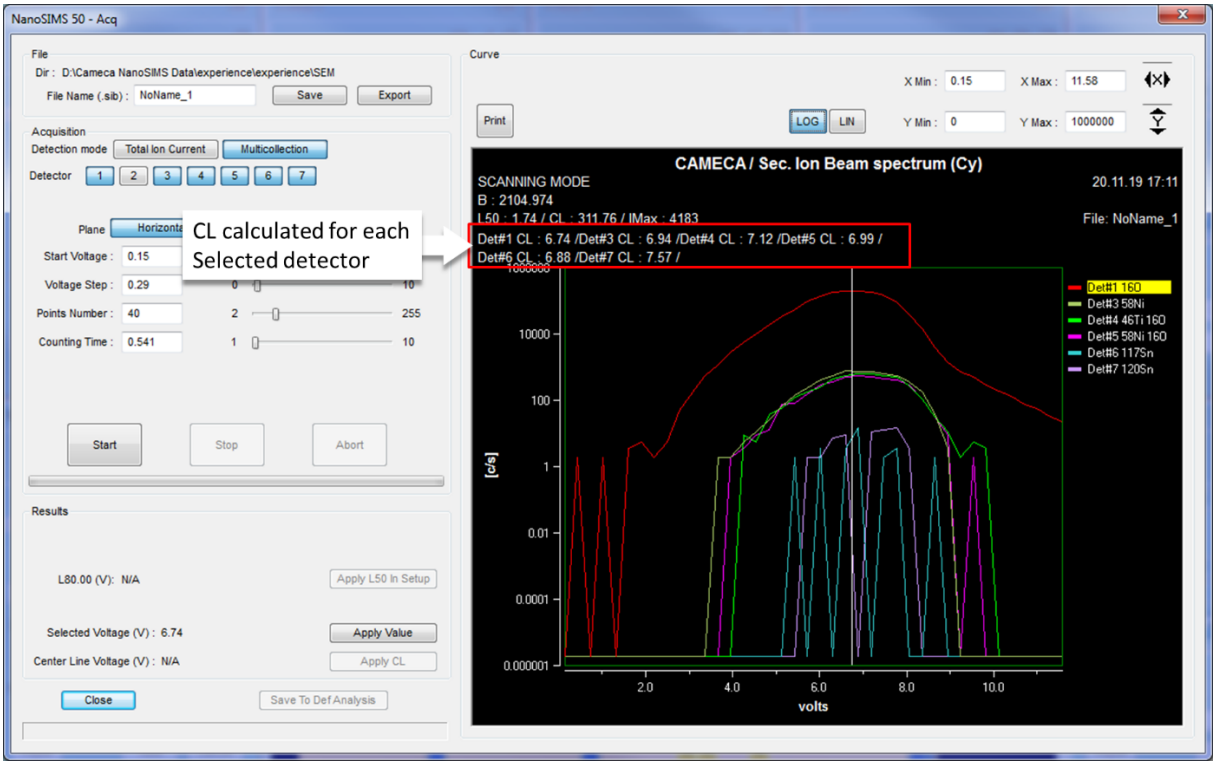


Figure 402: scan of Cy in Log scale and on multiple detectors to check the adjustment of the B-field coils.

On the keyboard: PM > Def-2 > X= Bf-hor, Y = Bf-vert

For each value of Bf-hor and Bf-vert
- do a Cy scan
- note the CL value for each detector selected. Ex: CL(T1, hor), CL(T3, hor), CL(T6, hor)
- calculate the interval ΔCL(hor) between the two most extreme CL(hor) values
- do a P3 scan
- note the CL value for each detector selected. Ex: CL(T1, vert), CL(T3, vert), CL(T6, vert)
- calculate the interval ΔCL(vert) between the two most extreme CL(vert) values
- adjust Bf-hor and Bf-vert so as to reduce ΔCL(hor) and ΔCL(vert). both ΔCL must be < ± 0.1 V

It is advised to check the mass fractionation reduction every time you are setting a new multicollection configuration, in particular is the detectors are widely spread across the multicollection chamber and for hydrogen.

It is also recommended to check the mass fractionation when moving significantly on the sample holder (from one hole to another), as the optimum Bf-vert and Bf-hor might vary slightly.

Note: if the Bf-vert and Bf-hor have been significantly modified, it will probably be necessary to readjust the Be-coil values for both the e-gun (see chapter 9.3.2.2) and the SE detection (see chapter 9.1.9.2)

9.3.1.3.4 IMF-AS: reduction of mass fractionation for smallest aperture slit AS

When scanning Cy, the secondary ion beam rotates around a center point located between ES and AS. Thus, scanning the beam in ES moves it in AS too. This can induce a problem of instrumental mass fractionation with AS of smallest size. It is possible to keep the secondary beam centered in the Aperture Slit and centered also in the entrance slit by adding some compensating voltage to C2y and C2x.

Once you have optimized the secondary beam alignment by following the steps described in chapter 9.3.1.3.1 above, turn on the IMF-AS option via the SETUP (Setup > Detector > IMF-AS, “IMF-AS” must be blue) (Figure 403)

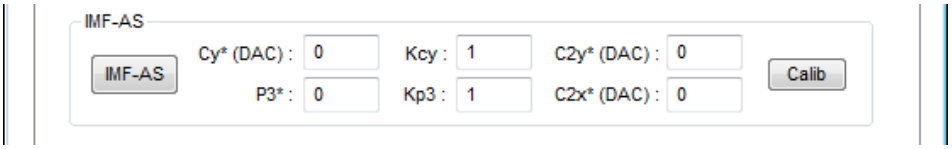


Figure 403: IMF-AS option from the Setup (Detector > IMF-AS)

When ON, this function links C2x and C2y to Cy and P3. When you first hit “calib”, it will retrieve the current Cy, P3, C2x and C2y from the keyboard (saved as Cy*, P3*, C2y* and C2x*) and calculate the coefficients Kcy and Kp3.
Thus, when later adjusting P3 and Cy, the new C2y and C2x will be calculated as follow, so as to keep the beam centered in AS:

$$C2y = C2y^* + kCy \times (Cy - Cy^*)$$
$$C2x = C2x^* + kP3 \times (P3 - P3^*)$$

9.3.2 Normal incidence electron flood gun (NEG) for charge compensation

9.3.2.1 Use of the e-gun

It is not necessary to re-tune the e-gun before every use. Most of the time, the user will only need to start the e-gun and adjust slightly e-gun Heat, HV and coil (eGunBe).
The starting process is manual:

- In the Preset window, click on the E-gun preset to turn it ON.
- In the Navigator window, go to FCo mode.
- In the Tuning window, put the FC reading in negative mode.
- In the Source window (Figure 404), put the emission ON.
- Put Heat = 500 DAC, Emission = 2.00 mA, HV = -1000 V.
- Increase progressively the Heat to 1500, 1800 up to 2000 DAC and the HV from –1000 to –8000 V by increments of 1000 V.
- At that stage, FCo should start reading a current (up to 1 µA with D1-2)
- Decrease the emission to 0.14 mA

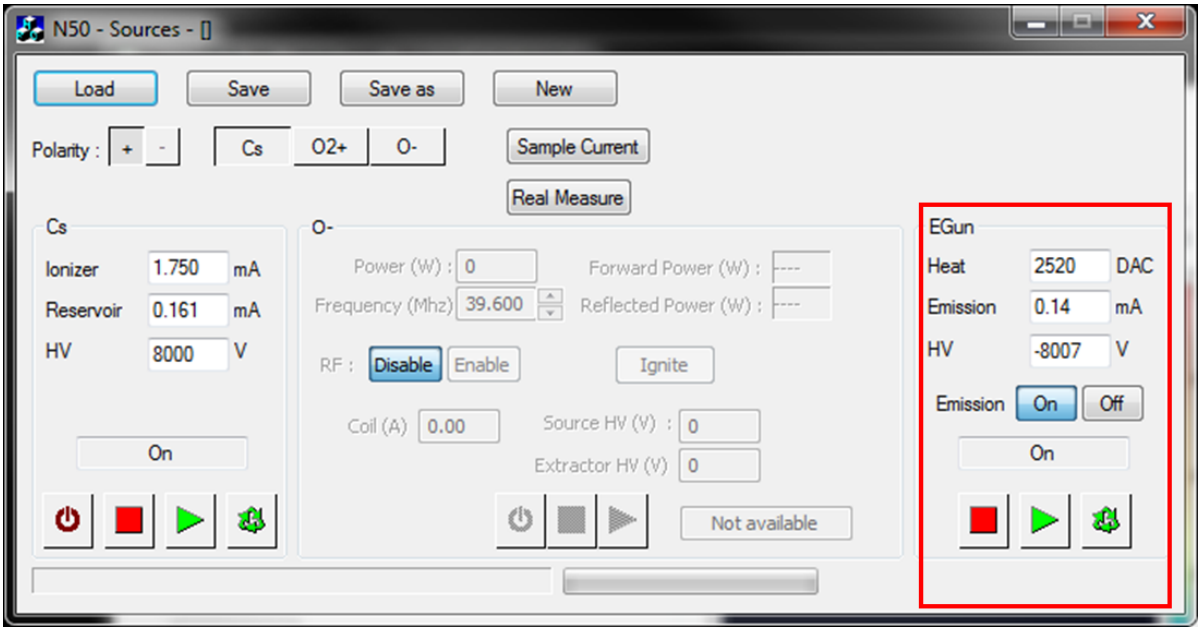


Figure 404: The e-gun controls are located on the Source window

Then you can adjust the e-gun to optimize the charge compensation:

- Turn the emission OFF before switching to SIMS mode in the navigator window, and to multi-collection mode in the Tuning window. If you haven't done so previously, move to your insolated sample. Put a species with a strong signal on one detector (for instance ^{18}O).
- Put the emission back ON, and adjust the HV by steps of ± 10 V so as to optimize the signal on your detector. In necessary, adjust also the Heat of about ± 20
- Finally adjust the e-gun coil (LF6 → Coils → egun Be)

Note that you might need to readjust EOS and C4 X after you've turned on the e-gun.

Important: always turn OFF the emission of the e-gun (in the Source window) when you move your sample or switch between FCo/SIMS/CCD modes.

To achieve ultimate reproducibility (such as low tenth permil with FC) on strong insulator, it is wise to let the e-gun thermally stabilize and degas for several hours. It is thus recommended to start the e-gun half a day prior to analyses or the night before. Start the e-gun following the above procedure, then turn OFF the Emission. Similarly, the e-gun can be kept warm between measurements with the emission set to OFF, it will reduce the filament current to a preset value (1700 DAC).

9.3.2.2 Tuning of the e-gun

It is recommended to tune the e-gun using a quartz sample coated with gold or platinum.
When you need to readjust the e-gun, follow the procedure below:

- Switch to TIC mode (in the Tuning window) and put the beam ON with a raster of 10x10 µm.
- Select D1-2 and make sure it is centered.
- Turn on the e-gun following the procedure above. At the end of the procedure, you should have Heat = 2500, emission = 0.14 mA and HV = -8000 V
- In the Navigator, go to FCo mode and in the Tuning window, select negative polarity to read the electron current.
- In LF6 → Coils → egun Be, adjust the coil (e-gun Be) as to find the maximum current intensity. It should be around 24700 bits.
- Adjust LF6 to maximize the current.
- Adjust C5 X and Y to maximize the current.

- Readjust the coil (e-gun Be), LF6 and C5 XY.
- Select and increase LF7. The current will decrease, then increase again. Find the maximum
- Adjust C6 X and Y and readjust e-gun Be.
- Repeat the procedure: LF6, C5 X-Y, LF6, C5 X-Y, LF7, C6 X-Y, LF7, C6 X-Y. Until FCo reads 10 000 nA with D1-0 (don't forget to put D1-2 back when doing the adjustment). At the end, LF6 should be around 21 300 bits.
- Save the new parameter values into an e-gun preset (PRESET window, see chapter 0), so that you can you can later recall those parameters (note that e-gun Be coil is also used by the SE detector with a different value than for the e-gun, saved in the SE PRESET).
- Back in TIC or multicollection mode and beam ON, sputter away the sample coating on a 80x80 µm raster, then reduce the raster to 50x50 µm. Adjust alternatively the HV and heat of the e-gun to maximize the signal. The signal must be stable.

Important: always turn OFF the emission of the e-gun (in the Source window) when you move your sample or switch between FCo/SIMS/CCD modes.

9.3.2.3 Stopping the e-gun

To stop the e-gun, turn the emission OFF, then click on the red icon to stop the e-gun.

9.3.3 Automatic routines for high precision analyses

For certain high precision/high reproducibility analyses (isotopic ratios, profilometry, near mass interference, etc…), the stability of the secondary beam is primordial. Therefore, it is necessary to include centering checks before launching or/and during the analyses. It can also be necessary to set an automatic PHD adjustment to keep a constant EM response throughout the analysis.
Such software tuning routines can be:

- individually launched (manual),
- saved in the setup (automatic) and added to DEFANALYSIS (see chapter 9.3.3.5 below) to be used automatically in the coming analysis or the many analyses to be launched in a chained analysis.

All the routines described below can be executed either in EM or FC (with the exception of the Pd/ESA centering, which is specific to EM detectors).

9.3.3.1 Automatic centering of the sec. beam in the entrance slit

Prior to defining the analysis, set up the three secondary centering routines on EOS, Cy and P3, and Pd/ESA coupling. All these routines must be set in the same conditions as the analysis (current intensity, ES, AS, etc…) This is necessary because the automatic centering algorithm uses the peak width (parameter L50 or L80) to find its center. So before an analysis using these routines is launched it is necessary to run them once for the routines to get a first reference point.

a. Open the **EOS program** in TUNING to run a first EOS scan to determine proper ranges and parameters:
b. step 1 (in Figure 405): preferentially select the trolley with the highest signal,
   step 2: launch an **EOS scan**
   step 3: click on "Apply L50 to Setup". This sends the current EOS curve width (L80, despite "L50" label on the button…)) to the SETUP.
   step 4: Click on "Apply CL".

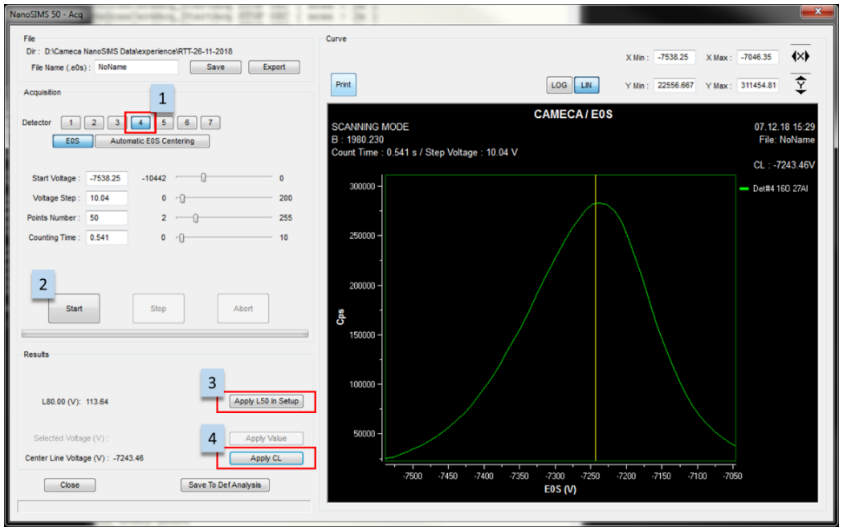


Figure 405: EOS centering

Note: although called" EOS centering" it is not really a centering in the entrance slit but more a focusing of the beam waist at the ES level, thus letting a maximum portion of the beam going through.

c. Activate the automated EOS centering for the upcoming acquisition by selecting "**Automatic EOS Centering**" (step 5 in Figure 406). Now that the program has the reference CL and EOS curve width (L80) it can "reproduce" the centering automatically as follow:
step 6: launch another EOS scan by pressing on Start,
step 7: click on Apply CL
step 8: click on "Save to Def Analysis".

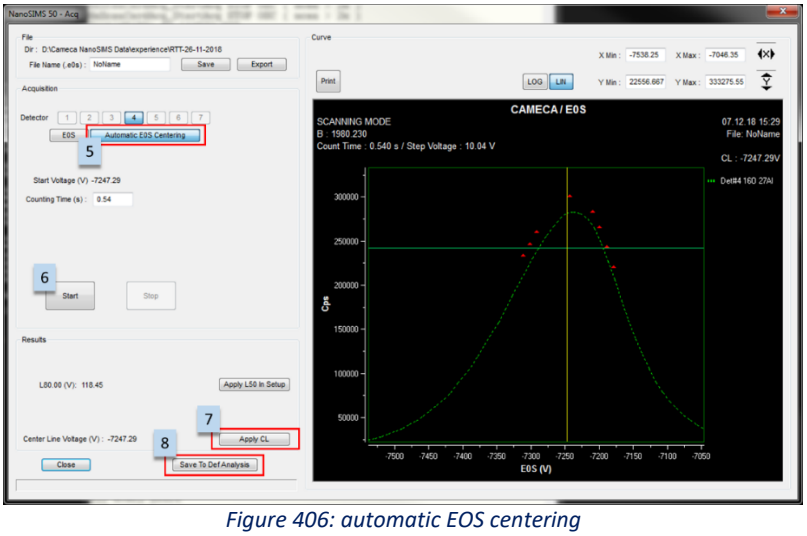


Figure 406: automatic EOS centering

d. Open the **Secondary Ion Beam (SIB) program** from TUNING to run a first Cy scan to determine proper ranges and parameters:
step 1 (in Figure 407): Select "**Horizontal**" and
step 2: launch a **Cy scan**.
step 3: click on "Apply L50 to Setup".
step 4: Click on "Apply CL".

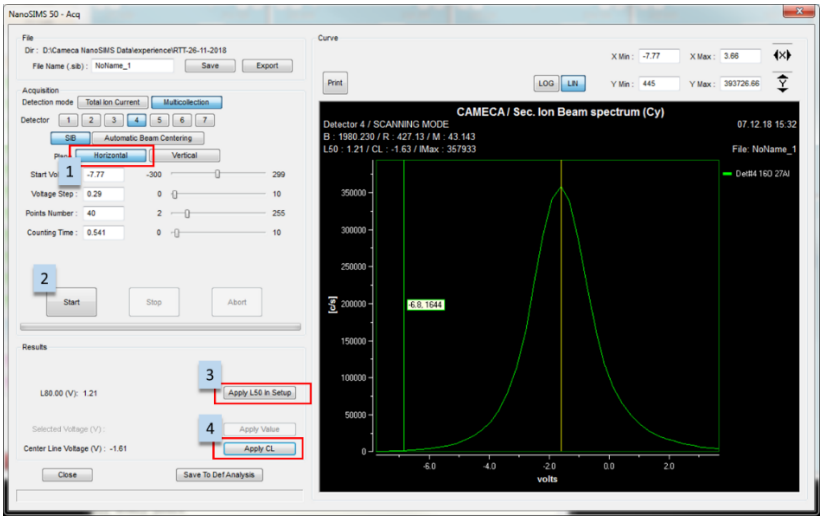


Figure 407: Cy centering (Secondary Ion Beam - horizontal)

e. Step 5 of Figure 408: Activate the automated Auto beam centering with Cy for the upcoming acquisition by selecting "**Automatic Beam Centering**"
step 6: launch another **Cy scan** by clicking on Start.
step 7: Click on "Apply CL",
step 8: "Save to Def Analysis".

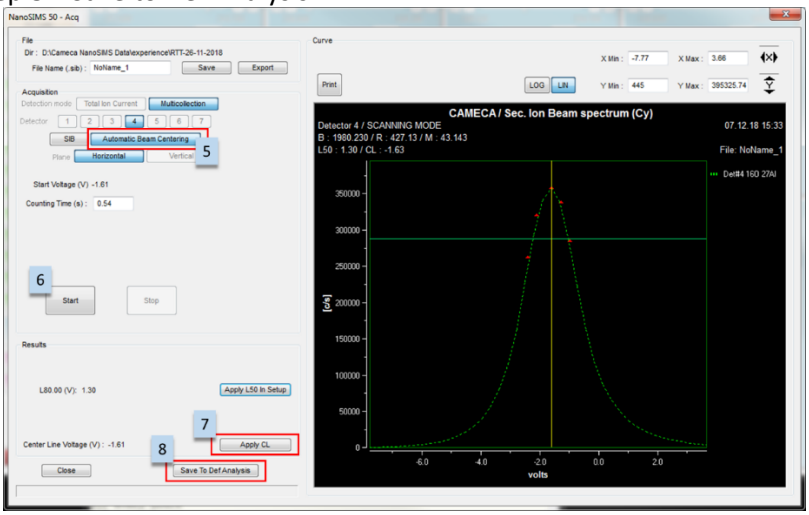


Figure 408: automatic Cy centering

f. Click on **SIB** (Figure 409) to run a first P3 scan to determine proper ranges and parameters: select "**vertical**" and launch a **P3 scan**. Click on "Apply L50 to Setup" and "Apply CL".

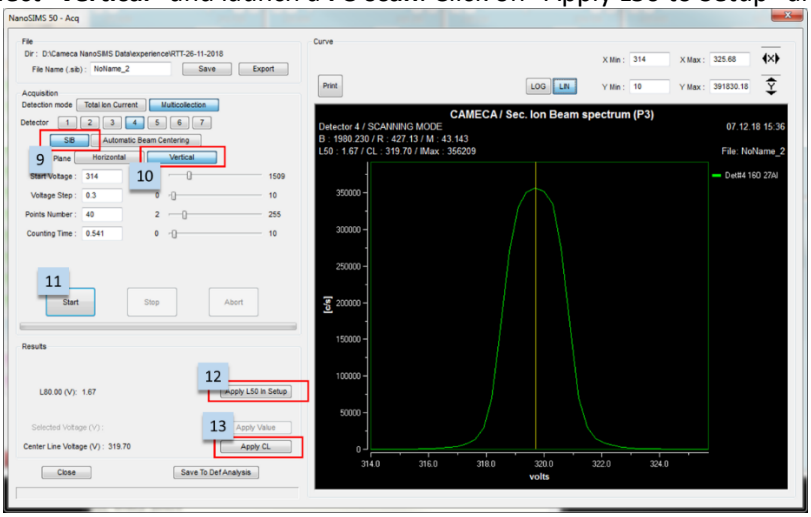


Figure 409: P3 centering (Secondary Ion Beam – vertical)

g. Activate the automated Auto beam centering with P3 for the upcoming acquisition by selecting "**Automatic Beam Centering**" and launch another **P3 scan** (Figure 410). Click on "Apply CL" and "Save to Def Analysis".

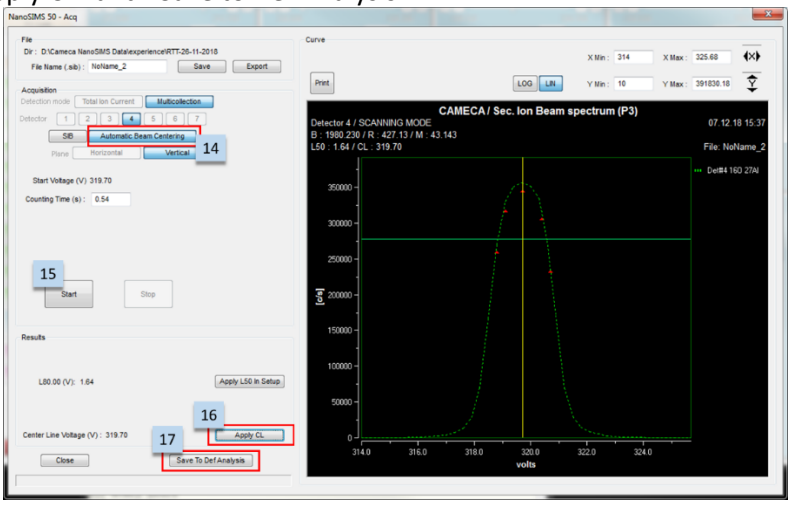


Figure 410: automatic P3 centering

9.3.3.2 Automatic centering of the sec. beam in the exit slit

In case of close mass interferences, very narrow or not flat-top peak (with small exit slit), fluctuating lab temperature or other reasons, the stability/reproducibility of the measurement can be limited due to mass spectrum line instability. It is then crucial to monitor the peak position regularly and adjust it if necessary. This can be performed automatically by this routine.

Note that to make sure that this automatic peak centering works correctly, it is recommended to set it on **a single intense peak (case 1),** showing no interference during the whole measurement (it could change for example during a depth profile). You then can use this "clean" intense peak as a reference to adjust the centering of more difficult peaks (e.g. mass interferences or low signal). Indeed, each detector can be programmed in DEFANALYSIS *without* an automatic peak centering, re-centered *on itself* (in which case, you must use the automatic centering routine below*)*, or re-centered using one reference *peak from another trolley* (then no automatic peak centering is necessary on this peak*)*.

If there are no single peaks, it is also possible to do an automatic peak centering using **one flank of the peak (case 2).**

See chapter 5.6.2.8 in DEFANALYSIS and chapter 9.3.3.5 below for adding a reference peak.

<u>Case 1 (isolated peak):</u>

a. Refer to Figure 411:
   Step 1: Launch an HMR scan,
   Step 2: Click on "Apply L50 to Setup" to send the current peak width to the Setup,
   Step 3: "Apply CL". The line to measure is positioned at the center of the peak.

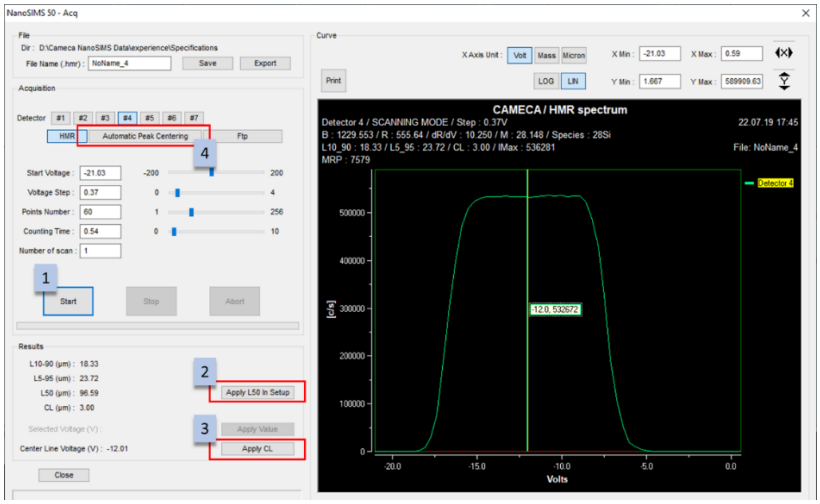


Figure 411: HMR centering with a single peak

b. New options appear (see Figure 412).
Step 4: Select “Automatic Peak Centering”,
Step 5: select “Both” (if the peak is symmetrical without interference or shoulder from near peak that could vary during the acquisitions to come),
step 6: launch another scan.
step 7: Click again on “Apply CL”.

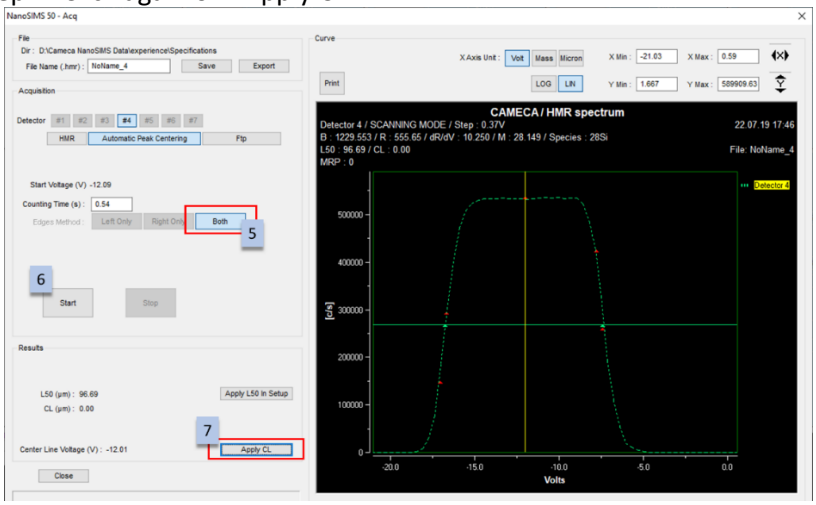


Figure 412: HMR automatic peak centering with a single peak

Once this is programmed, the acquisition will pause regularly, record the intensities of the same 5-points, deduce the new center peak position (in volt), correct accordingly the peak position by re-adjusting Pd voltages and re-start the acquisition. The signal will thus be measured from the same position of the peak even if the peak has drifted for any reason. The information will be stored in the acquisition file.
One can realize why a high intensity peak (= high signal to noise) is required: a low intensity peak with low S/N will create a very poor positioning as in the steep sides a variation in intensity will result in incertitude in X positioning.

Case 2 (one flank of a group of peaks) :

a. Step 1: Launch an **HMR scan** (refer to Figure 413),
Step 2: Click on “Apply L50 to Setup” to send the current peak width to the Setup,
step 3: Position the cursor at the desired peak position and press “Apply Value”.

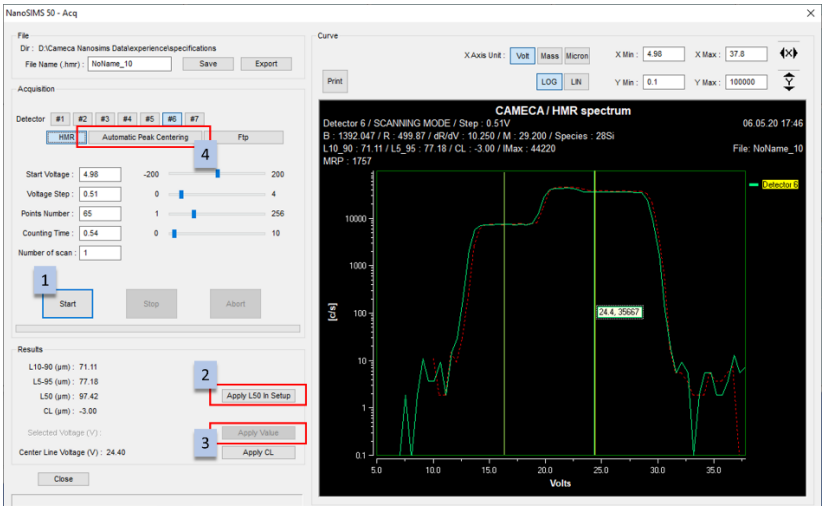


Figure 413: HMR centering with peak interference

b. New options appear (see Figure 414). Select “Automatic Peak Centering”,
Step 5: select “Left only” or “Right only”.
step 6: launch another scan.
step 7: Click again on “Apply CL”.

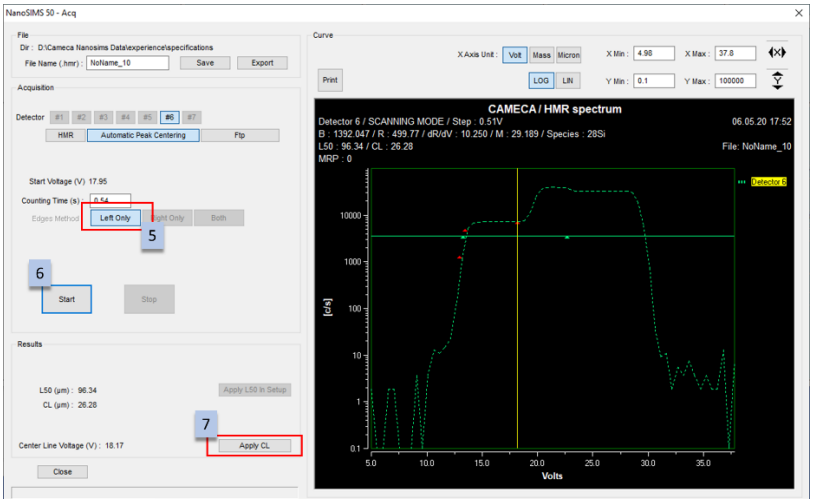


Figure 414: HMR automatic peak centering with peak interference

Once this programmed, the acquisition will pause regularly, record the intensities of the same 3points, deduce the 50% position of the selected peak flank, calculate the drift compared to the original flank, correct accordingly the peak position by re-adjusting Pd voltages and re-start the acquisition: the signal will be measured from the same position of the peak even if the peak has drifted for any reason. The information will be stored in the acquisition file.

9.3.3.3 Pd /ESA automatic coupling for high precision analyses

As the secondary beam hits an EM at a given position of the first electrode, the aging of the EM is not uniform spatially and the most hit point of the EM will age faster, making the response of the EM dependent of the position where the beam hit the first electrode. This can become inconvenient for high precision analyses, as readjusting the deflection plates to keep the peak centered in the exit slit will induce a slight shift of the beam on the dynode. To prevent this, it is possible to compensate this shift via the external ESA plate of the detector. The empiric correlation for the correction is Pd/ESA = 0.77.

- First, adjust the Pd deflector by doing an HMR scan for all used detectors.
- Then, activate this option: turn it ON in the SETUP (Setup > Detector > PD/ESA, Figure 415)

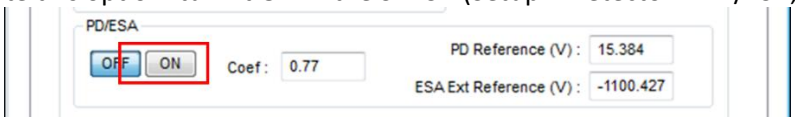


Figure 415: Pd/ESA option in SETUP > Detector

- In the TUNING window, click on “Pd/ESA calib” to save the Pd and ESA values into the SETUP (Figure 416). From now on, Pd and ESA values will compensate so that the beam will always hit the EM at the same position.

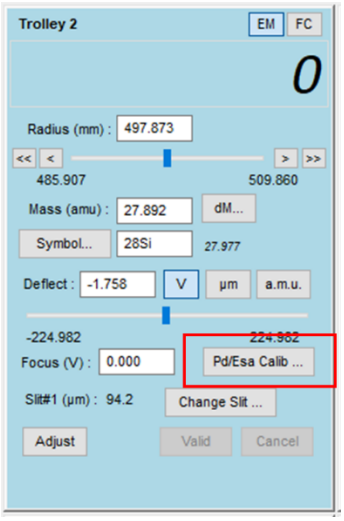


Figure 416: "Pd/ESA Calib" in the Tuning window

- If you wish not to use this option, simply turn it OFF in the SETUP. Pd and ESA will then change independently.

9.3.3.4 PHD automatic adjustment

PHD adjustment is primordial for isotopic analyses (see 2.1.4.3.1 and 9.1.12) When an EM receives a high signal (> 500 000 cps), it can age fast enough during an analysis that it will degrade the stability required for high precision measurement. It can therefore be necessary to set an automatic adjustment of the EM HV to counter the aging of the EM *during the analysis*.
The principle of this routine is to pause the acquisition at regular interval, record an EM pulse height distribution (PHD), extract the PHD_{max} value (in mV), re-adjust the EM HV in order to reset PHD_{max} always to the same value, re-start the acquisition. Set the automatic PHD adjustment:

a- Open the PHD program in TUNING,
Step 1 of Figure 417: choose the detector,
step 2: launch a PHD scan.
Adjust the EM HV if necessary (see 9.1.12 for details)

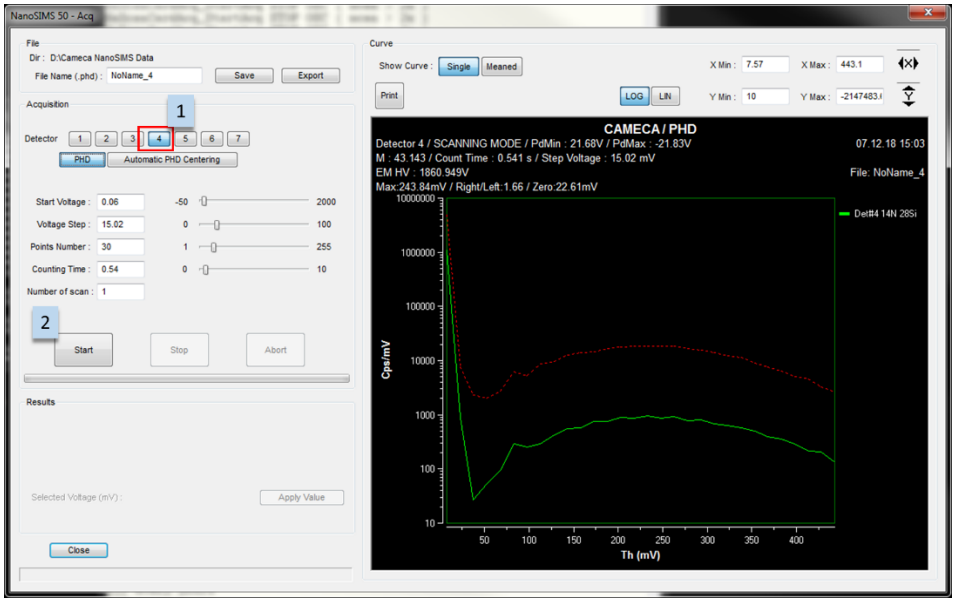


Figure 417: PHD scan

b- When the EM HV is correct,
step 3 of Figure 418: select “automatic PHD centering”,
step 4: launch another scan.
step 5: Click on “Apply Ref to Setup”

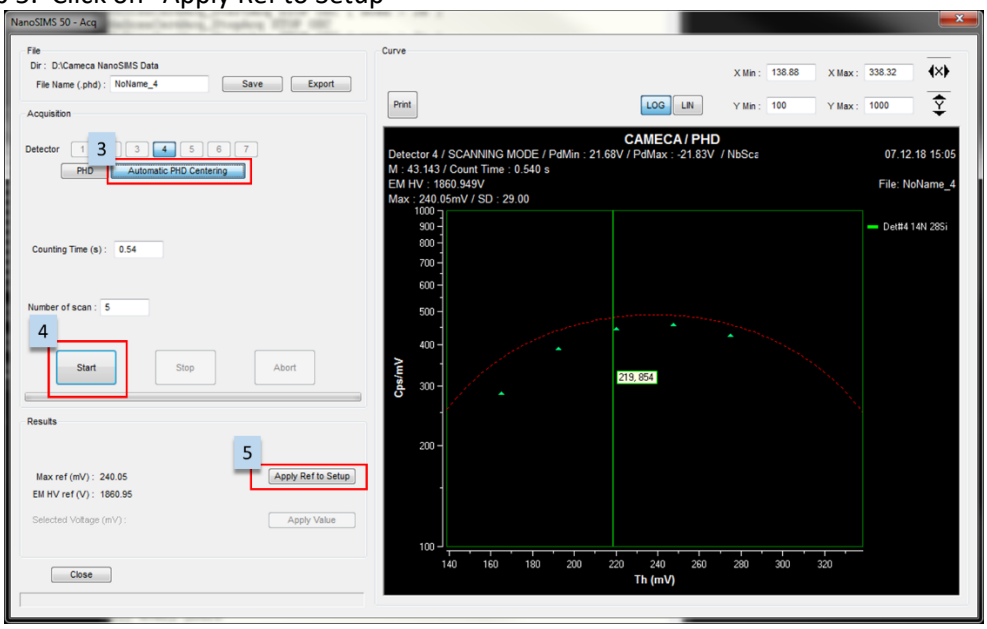


Figure 418: Automatic PHD adjustment

9.3.3.5 Adding the automatic routines to Def Analysis

Once all necessary automatic centering routines have been set, it is now possible to include them to the analysis via Def Analysis.

1- select the acquisition mode.
2- Set all parameters as usual. If in “isotopes” mode, do not forget to define your isotopic ratios as seen in 9.2.6.2
3- Set the automatic routines you wish to include in your analysis:

a. **EOS/Secondary Ion Beam centering**: under "centering", select EOSC, SIBC and EOSC (it is always better to re-do an EOS scan at the end) and define the number of frames between each centering (Figure 419).

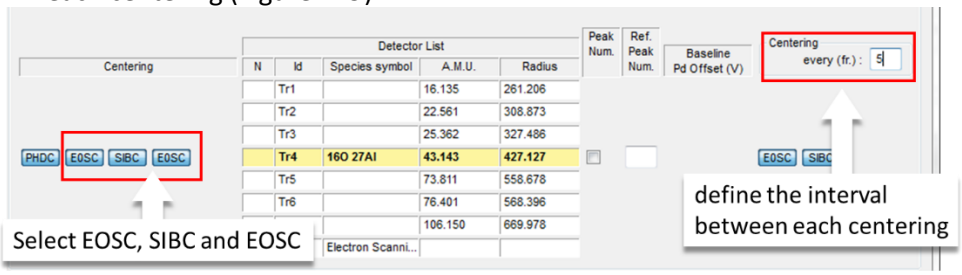


Figure 419: add EOS and SIB centering to an analysis

b. **Peak centering**: Select the masses for which you wish to do a peak centering and their reference peak, if necessary (Figure 419).

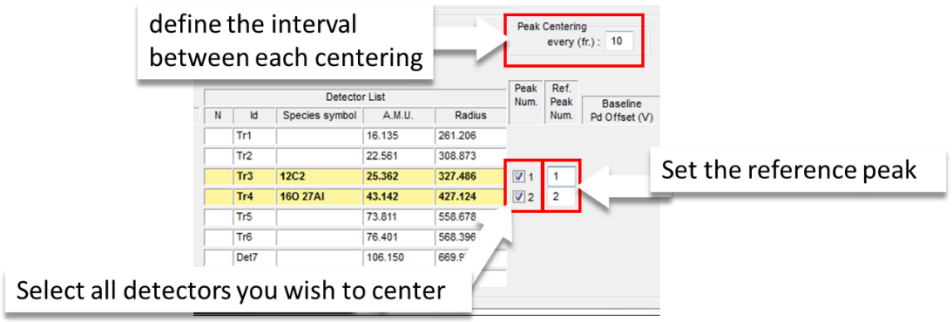


Figure 420: add a peak centering to the analysis

c. **PHD adjustment (EM only)**: under "centering", select "PHDC" and define the number of frames between each centering (Figure 421).

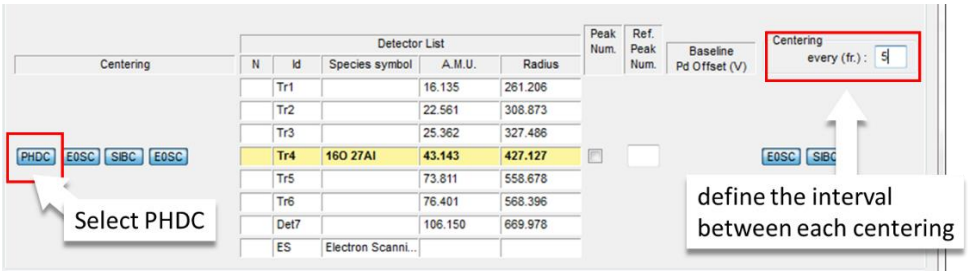


Figure 421: add automatic PHD adjustment to the analysis

4- Go to Analysis and launch the analysis.

9.3.4 High precision isotopic ratios with Faraday Cups

Using electron multipliers and making use of all available routines (automated alignment and focusing, EM aging correction, peak recentering, etc…), it is possible to achieve sub-permil isotopic ratio reproducibility on major elements on the NanoSIMS. Nevertheless some limitations remain (QSA effect, statistics, instabilities…).

It is possible to get isotopic ratio reproducibility in the low tenth permil range by switching to FCs and, if necessary, using higher beam currents (up to nA range). In order to achieve this, many aspects must be controlled:

- The sample must be perfectly flat and well-polished.

- The sample must be mounted parallel to the sample holder surface, and with a good sample-holder contact for a good diffusion of the charges.
- E0S must not vary much between spots.
- sample and standards should be as close to the center of the mount as possible and be coated with the same recent metal-coating.
- Standards must be homogeneous at the NanoSIMS scale! Standards working well with techniques using mm area might not be sufficient for nanoscale analysis.
- Use of charge compensation (e-Gun) for insulated samples.
- Optimizing the vacuum to reduce the contribution of hydrides.
- Optimizing the tuning to obtain sufficient MRP and sufficient flatness of peak top (reducing variation of signal in case of any line shift).
- Use of the automated centering routines to optimize the reproducibility of data over time.
- Use of the NMR to avoid magnet drift over time.
- Keeping a low background noise and adjustment of its correction (for FCs),
- ion source stability,
- sufficient pre-implantation,
- temperatures of the laboratory, and the cooling water should remain as stable as possible to avoid drifts. It is crucial that the average room temperature is stabilized using a mild continuous flow of fresh air and not sudden alternations of freezing air blowing directly on the instrument! Refer to lab requirements. Cooling water temperature variations are killing factors for magnet and source stabilities.

It is recommended to start on easy samples (e.g. pure silicon) and then practice by reproducing the acceptance tests made by Cameca engineers during installation. A detailed procedure is given in chapter 9.3.9.

Reminder on the statistical aspect:
If the signal received by a detector exceeds 10^6 counts per second, it is advised to switch from electron multiplier to Faraday cups. For a high-count rate (several 10^6 cps), FCs offer the advantage of a better reproducibility than EMs. However, this implies certain limitations that the user must keep in mind:

- Faraday cups' response time is slower than EMs. Therefore, it is not possible to make images with FC or set an electronic gate. Only isotopic ratios and depth profiles (without electronic gate) are possible.

- On the NS50L, the background noise of a FC with a 10^{11} ohm resistance is given as 5 $x10^{-16}$ A, measured over 5s. Noise, count rate and integration time will determine the incertitude for short term measurements (long term baseline drift must be controlled also): the basic rules applies: I = n*q / t e.g the equivalent count rate of the FC background noise is: n = 5 $x10^{-16}$ / 1.6 $x10^{-19}$ = 3 100 c/s. This noise will be reduced when integrated over a longer time *t* instead of 5s, following sqrt(*t*/5).

Examples:
- Case 1: with 5 $x10^5$ c/s over 80s, the relative incertitude will be: [3100/ sqrt(80/5)] / (5 $x10^5$) = 1.5 $x10^{-3}$ = 1.5 permil. It is poorer than with EM as above.
- Case 2: with 1 $x10^7$ c/s (not possible with EM) over 80s, the incertitude will be: 0.75 $x10^{-4}$ = 0.07 permil, which is much better than with EM (other factors will limit the overall isotopic reproducibility to a few tenth permil on the NS50L).

9.3.5 Data Acquisition in Magnetic Peak Switching mode

The practical design of the NanoSIMS 50L multicollection implies two main constraints:

1- The ratio between the highest mass (7th detector) and the lowest mass (1rst trolley) **Mmax/Mmin must be under 22.** E.g. from ^{1}H up to 22 AMU or ^{10}B up to 220AMU).

One classical limitation in cell biology can be parallel detection of ^{2}D/^{1}H, ^{13}C/^{12}C and ^{15}N/^{14}N plus ^{31}P and ^{32}S. If Mmax is 32 amu then Mmin is 32/22= 1.45 precluding ^{1}H detection. One solution can be to change the protocol to adapt to the constraint, e.g. measuring instead: ^{12}C, ^{13}C, ^{12}C^{1}H, ^{13}C^{1}H, ^{12}C^{14}N, ^{12}C^{15}N, ^{31}P and ^{32}S if the sample is full of carbon (resin-embedded section).

2- The minimum mass interval between two adjacent trolleys, defined mostly by the physical width of the EMs, is defined by: **$interval_{min}$ (AMU) = Integer (M_{max}/59) +1.**

Hence if for instance Mmax (on 7th detector) is ^{58}Fe, one can position the 5th and 6th detectors on ^{56}Fe and ^{57}Fe (one mass unit interval). However if one wants to measure uranium isotopes (^{234}U, ^{235}U, ^{236}U, ^{238}U, ^{235}U^{16}O, ^{238}U^{16}O, ^{238}U^{16}O$_2$), here $interval_{min}$ = Integer (254/59) +1 = 5 AMU!

In this case, magnetic peak switching can be a solution: **use one single detector** and with the magnet jumping from B-field to B-field as in a monocollection SIMS instrument.

Note 1: in some cases, monocollection with one single EM can be a positive point (no risk of variation of gain between detectors). The information of each element will not come from the exact same volume but this can be acceptable if the analyzed volume is large and homogeneous and you program many cycles interlacing the various element measurements.

Example: zircon dating: one EM, B1:203.5 amu (background), B2: ^{204}Pb, B3: ^{206}Pb, B4: ^{207}Pb.

Note 2: when there are only a few ions to follow, monocollection can be a good option, but when there are a few tens ions it can become too long and a strategy of hybrid collection (**Combined Analysis**) can be more advantageous (see chapter 9.3.6)

To program a Magnetic Peak Switching analysis, follow the steps indicated on Figure 422:

1- Select the mode “Magnetic Peak Switching” in the Tuning window and choose the desired detector.
2- Adjust the magnetic field to center an element’s signal on the selected detector. In HMR, do a manual peak centering then an automatic peak centering. Apply CL.
3- In Tuning, in the detector’s window, click on “Save to Def Analysis” to save the magnetic field value.
4- Repeat 2- and 3- for each mass you wish to add to your analysis. All the magnetic field values are stored in the “Def Analysis File”.

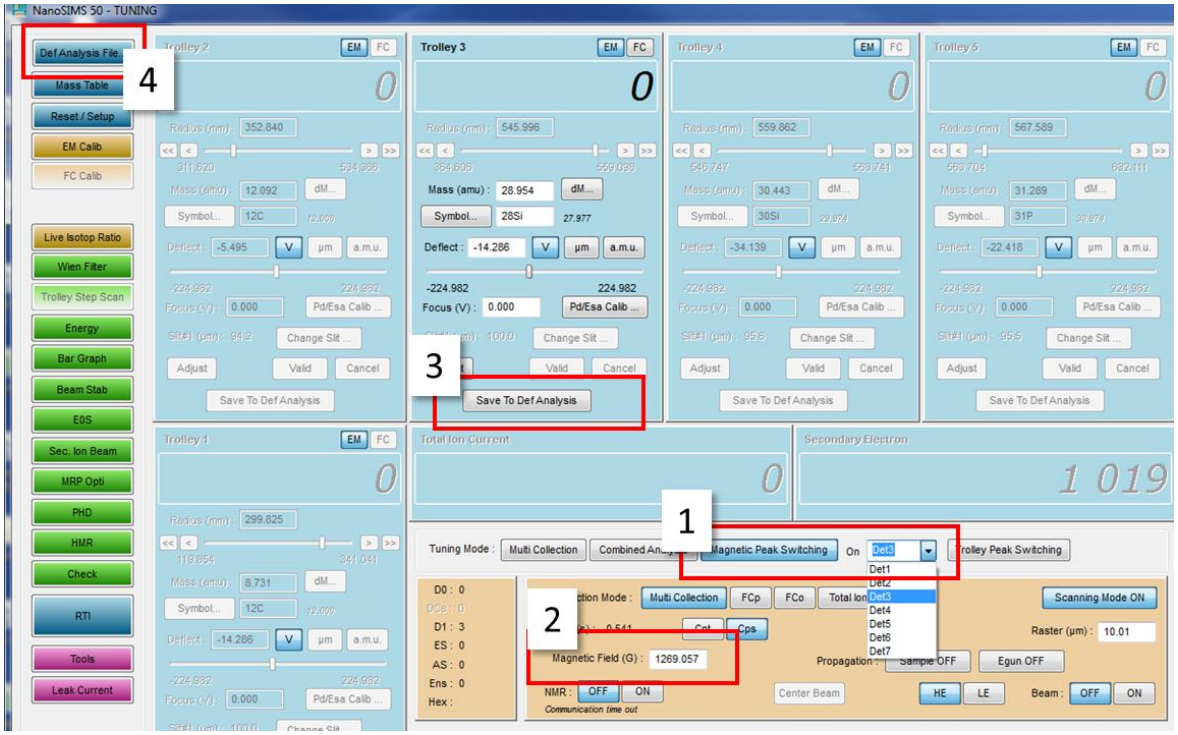


Figure 422: Selection of the "Magnetic Peak Switching" mode in Tuning.

Before launching an analysis, it is necessary to stabilize the magnetic field, by cycling it repeatedly between the saved masses:

1- Open the Def Analysis File and click on "Cycling B Fields" (Figure 423). Let it cycle for say 20 min (this can be optimized case by case depending on the B-field interval to jump and the peak shape/mass resolution)
2- The magnetic field will likely have shifted. Readjust the value of the magnetic field for each mass, re-do a HMR and an automatic peak centering, then save the new values to the Def Analysis File (see above). Erase the previous values by clicking on "delete mass".
3- Do another cycling for around 20 min.
4- Once the field is stable enough, adjust the centering more finely by adjusting the deflection ("Deflect" value in the detector's window in Tuning).
5- Cycle one more time for around 20 minutes
6- If the magnetic field cycles properly between masses, go to Def Analysis.

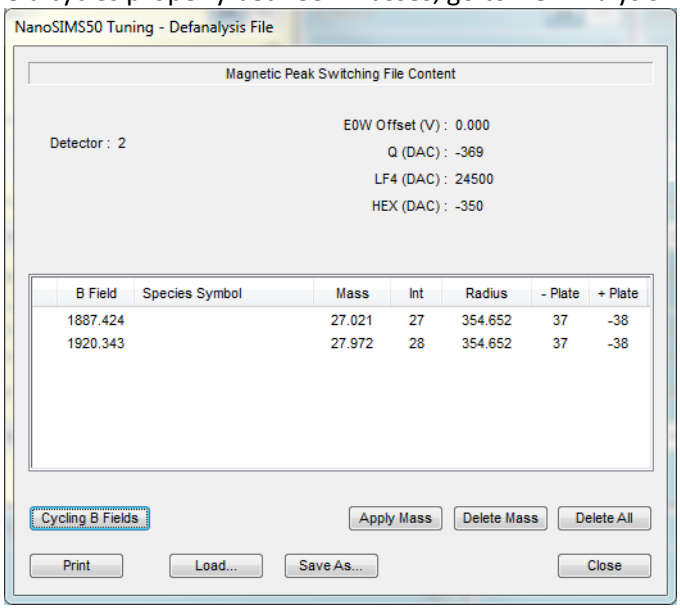


Figure 423: Def Analysis File in "Magnetic Peak Switching" mode

In Magnetic Peak Switching mode, each magnetic field values appears in the Def Analysis window (Figure 424). Select the desired B field values and set all other parameters as usual (see options in “Multi-collection” mode). Then start the acquisition. Note that it is recommended to start the acquisition quickly after the cycling as to not let the magnetic field drift again.

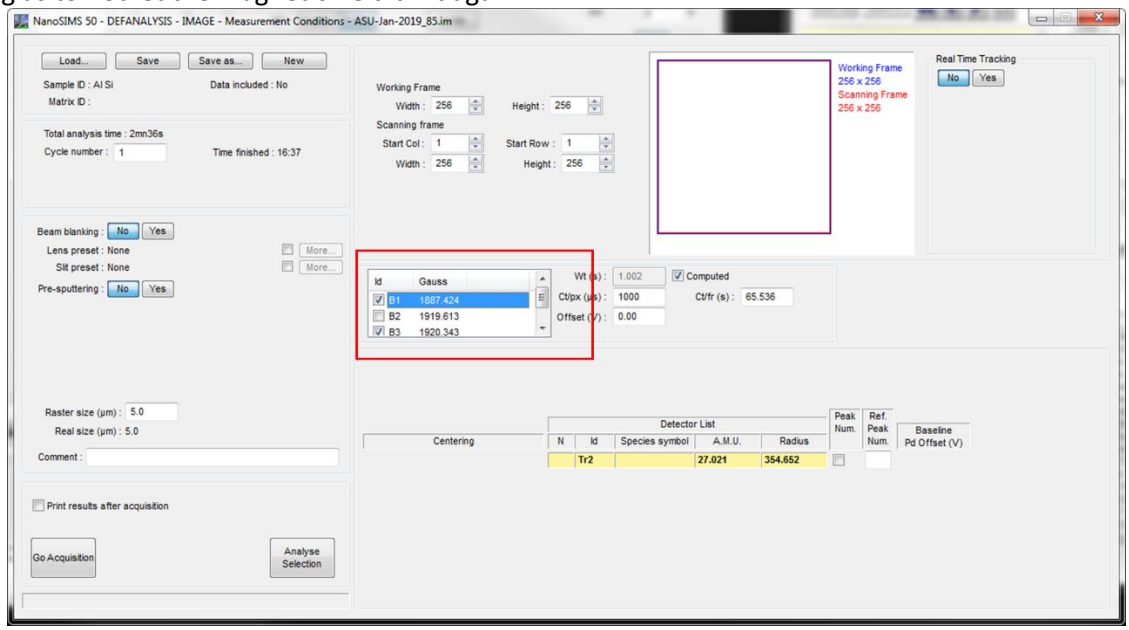


Figure 424: Def Analysis window in Magnetic Peak Switching mode

Note: all automatic options (HVmulti drift corr, EOS auto, Pd/ESA,… others) are available in magnetic peak Switching mode like in the Multicollection mode. They appear in the menu if one launches an automatic centering.

9.3.6 Data acquisition in Combined Analysis modes

9.3.6.1 Combined Analysis 1: multicollection with several B-fields

As the name implies, the “Combined Analysis” mode combines Multi-collection and Magnetic Peak Switching modes.
In this example, the trolleys do not move. Several multicollection settings (each using its **own B-field**) can be defined using some of these fixed detectors (a same detector can be part of several multicollection settings). The acquisition will interlace cycles as: B1 with MC1; B2 with MC2, Bn with MCn, back to B1 with MC1, …etc.

Before starting programing such a combined mode, it is important to test numerically the physical possibilities, given the mass range and mass interval constraints. For this, one can use the tool Mass Table Edition described in chapter 5.2.9.

Example of combined analysis taken from literature for zircon U-Pb dating (from Hu at al., 2016):

Magnetic field	EM2	EM3	EM4	EM5	EM6	EM7
B1			203.5amu			
B2	$^{28}Si_2{}^{16}O_3$		204Pb		$^{90}Zr_2{}^{28}Si_2$	
B3	$^{89}Y^{16}O$		206Pb	238U	$^{238}U^{16}O$	$^{238}U^{16}O_2$
B4		$^{96}Zr^{16}O$	207Pb			

To program a Combined analysis, select the mode “combined analysis” in the Tuning window (Figure 425).

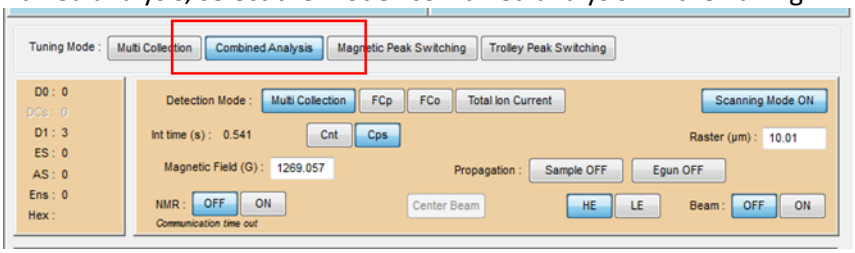


Figure 425: Selection of the tuning mode “combined analysis”

1- Position the trolleys used for the first value of the magnetic field (first multicollection setting).
2- For each trolley used, click on “Save to Def Analysis”.
3- Switch the magnetic field to a different value so that the other masse(s) you wish to analyze can be measured by some (other or same) detectors.
4- For each detector used, click again on “Save to Def Analysis” to save the new magnetic field value for the second configuration.
5- Repeat 3- and 4- for as many magnetic field values as necessary.

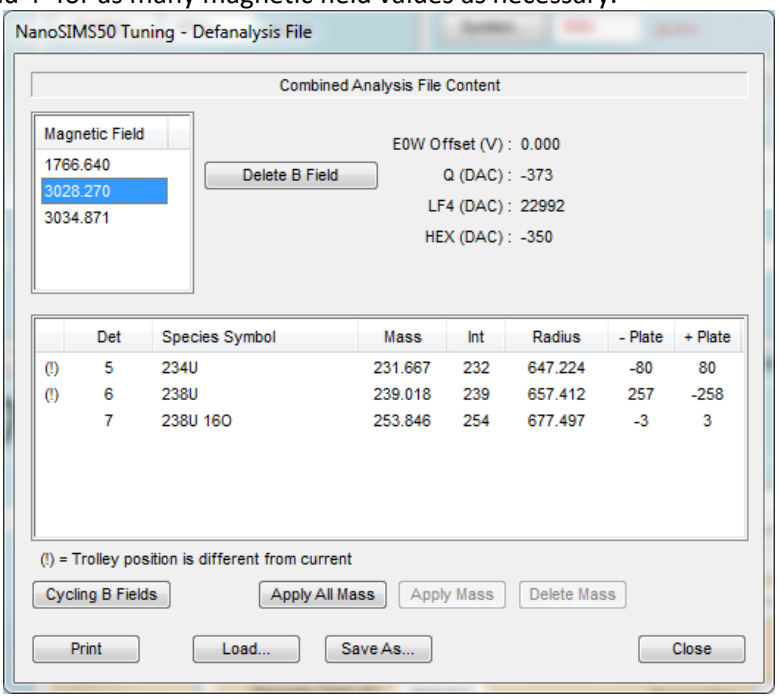


Figure 426: Def Analysis file in Combined Analysis mode.

Now the Def Analysis file contains info on all necessary detectors for each magnetic field value (Figure 426). Once all magnetic field configurations are set, start cycling the magnetic field, similarly as for the Peak Switching mode:

1- Open the Def Analysis File and click on “Cycling B Fields (Figure 426). Let it cycle for say 20 min (depending on B-field jump and peak shape/mass resolution).
2- The magnetic field will likely have shifted. Readjust the value of the magnetic field for each mass and save the new values to the Def Analysis File (see above). Erase the previous values by clicking on “delete mass”.
3- Do another cycling for say 20 min.
4- If the field is stable enough, do not touch it. This time, adjust the centering more finely by adjusting the Pd deflection (“Deflect” value in the detector’s window in Tuning)
5- Cycle one more time for say 20 minutes
6- If the magnetic field cycles properly between masses, go to Def Analysis.

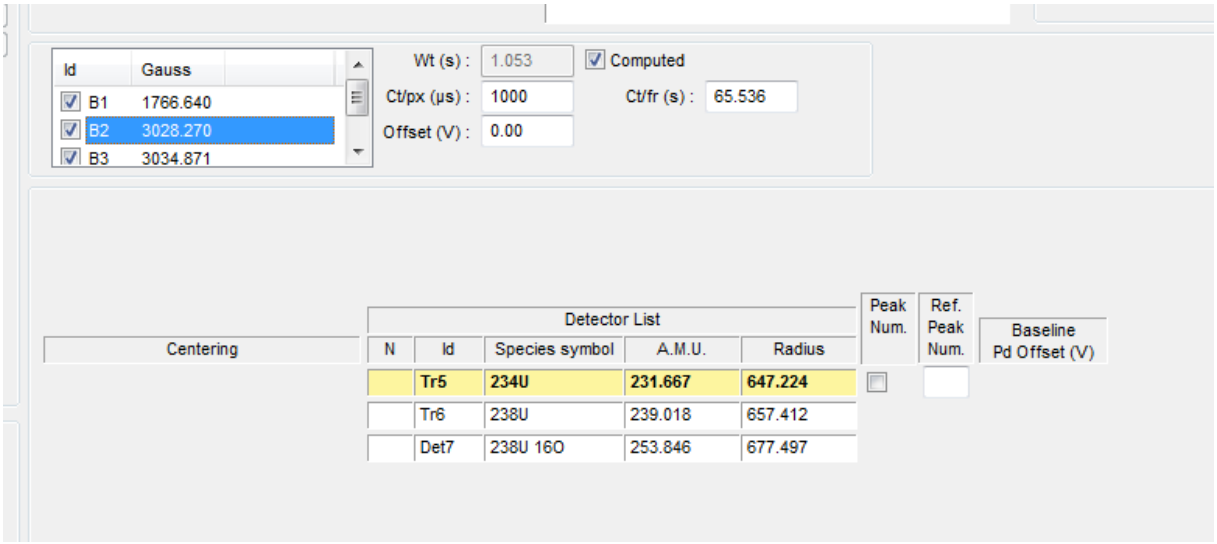


Figure 427: Def Analysis window in Combined Analysis mode

In Def Analysis (Figure 427), select the desired B-field value (B1, B2, etc…) and for each magnetic field value, select the associated detectors by clicking on "id". Then set all other parameters as usual (see options in "Multi-collection" mode). Finally, start the acquisition. Note that it is recommended to start the acquisition quickly after the cycling as to not let the magnetic field drift again.

9.3.6.2 Combined Analysis 2: one B-field, trolleys moving / Trolley peak switching

It is also possible to do a combined analysis with alternating positions of the trolleys. This can also be done via "Trolley peak switching". The setup procedure is pretty similar to the one above.

In Combined Analysis mode:

1- Position the trolleys according to the first set of positions needed.
2- For each trolley used, click on "Save to Def Analysis".
3- Move the necessary trolleys to their next position.
4- For each of these trolleys, click again on "Save to Def Analysis" to save the new positions for the second configuration.
5- Repeat 3- and 4- for as many trolley positions as necessary.

In Def Analysis, select the associated detectors. Then set all other parameters as usual (see options in "Multi-collection" mode) and start the acquisition.

Note: the trolley reproducibility is not sufficient to ensure a perfect peak centering especially at high radius and high MRP. Hence it is mandatory to activate the peak centering option.

9.3.6.3 Combined analysis 3: one B-field, electrostatic peak jump on exit slit

You can also use the combined analysis mode to adjust the Pd deflector value at the exit slit of a trolley. This can be useful when one wishes to record several peaks at a same mass, for instance to clarify a mass

interference. An example of such setting in cell biology is taken from C. Guillermier et al (2014), allowing detection of nine ions with seven detectors:

scan	EM1	EM2	EM3	EM4	EM5	EM6	EM7	Ratio measured
1	16O	18O	12C2H	12C14N	12C15N	31P	32S	15N/14N and 18O/16O
2	16O	18O	12C2H	12C14N	13C14N	31P	32S	13C/12C and 18O/16O
3	16O	18O	12C2H	12C2D	13C14N	31P	32S	D/H and 18O/16O

To set this up, proceed as follow:

In Combined Analysis mode:

1- On all detectors used, do an HMR to adjust the exit slit deflector on the right peak.
2- For each trolley used, click on “Save to Def Analysis”.
3- For the trolleys with multiple peaks, do another HMR to adjust the exit slit deflector on the next peak.
4- For each of these trolleys, click again on “Save to Def Analysis” to save the new deflection value for the second configuration.
5- Repeat 3- and 4- for as many peaks as necessary.

In Def Analysis, select the desired configurations (B1, B2, etc…) and for each, select the associated detectors. Then set all other parameters as usual (see options in “Multi-collection” mode) and start the acquisition.

9.3.7 Analysis at low energy

For the best spatial resolution, the impact energy is usually of 16 keV (8 keV on the primary beam + 8 keV on the secondary beam). For a better depth resolution, it is possible to reduce the impact energy of the primary beam by reducing the source voltage. Due to the geometry of the instrument (opposite polarities), this will also lower the voltage of the secondary beam. On the NanoSIMS 50 & NanoSIMS 50L, starting on software version 4.5, this impact energy can be reduced down to 4 keV (2+2). Reducing the impact energy will improve the depth resolution through a reduction of the atomic mixing depth (roughly in square root law: reducing Ep by 4 will improve mixing depth by 2). However, this will degrade the spatial resolution due to many factors: a lower brightness of the ion source, a larger contribution of chromatic aberrations, degraded transmission and mass resolution, as well as a reduced ion-electron conversion on the first EM dynode. In addition, beams at lower energy (primary ions, secondary ions and electron flood gun) are more sensitive to magnetic spurious and external fields.
This impact energy change on the NanoSIMS requires four steps:
1) modify the source HV and heating conditions,
2) set the PROPAGATION mode ON then modify EOW (=sample) voltage: this will adjust accordingly most HV, voltages and coil currents,
3) set PROPAGATION mode OFF,
4) Update or check the independent parameters not sensitive to the PROPAGATION mode: B-field/mass correspondence, Raster size and EM HV/PHD

9.3.7.1 In Cesium

The Cs source should be on, the instrument tuned.

- Note the values for Ionizer, Reservoir and HV (8000 V). Note FCp as a reference (typically 30 to 50 nA for Ionizer=1.75 mA, Reservoir=0.12 mA). Note also EOW, the B-field on the magnetic sector and the masses on the detectors. Note the raster size. If needed save the current presets

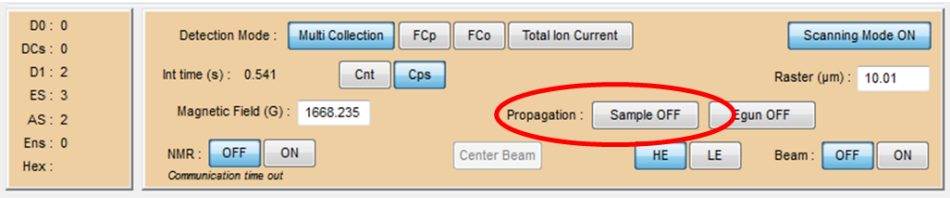
Decreasing the Impact energy:

- Decrease the source HV by steps of 2000 V and at each step, adjust the Ionizer heating current (emitted from the filament and bombarding the ionizer cap) value in order to keep the Cs+ beam current in FCp constant. Wait a few minutes after each increase of the Ionizer to give time to the source to stabilize. The principle is to keep both electron bombardment heating power HV x I constant.
- Lower the source HV down to the desired value (ie. down to 2000 V if you want a total impact energy of 4 keV).

Note 1: down to 4000 V, increasing the ionizer heating current should allow to keep the same primary beam intensity, but it is not as efficient below. For reference, at 2000 V, the Ionizer heating current should be around 6.5 mA.

Note 2: as explained in chapter 2.1.1.1.3 the heating power is more sensitive on the reservoir than on the ionizer, so the optimum reservoir emission current might need re-adjustment around this "theoretical" constant power value. There is a balance to find: a too low reservoir heating will reduce the beam current; a too high reservoir heating will reduce the beam current too but also reduce the source lifetime through increased consumption of cesium atoms.

- In Tuning, press the *Sample* propagation button to turn it from off to **ON**.



- Adjust the value of EOW (= sample voltage = half of impact energy) following a simple proportion law: if you want to decrease the high voltage by a factor of 2 (from 16 to 8 keV), divide EOW by 2. All lens and deflector values in the NanoSIMS primary and secondary optics and spectrometer will automatically adjust by applying the same factor, while coils will adjust following a square root law.
- Put the *Sample* propagation back OFF
- The **magnetic field** of the magnetic sector analyser does not automatically readjust. You must do so manually. To readjust the Magnetic field, select B-field on the keyboard and decrease the B-field value as to display to correct masses on the detectors.
- The **raster** does not adjust automatically either. Do not forget to adjust the raster size in Tuning.

It is then necessary to

- do a PHD scan and adjust (generally increase at lower energy) the HV of all EM used. Check the thresholds (they should not move much)
- Do an HMR scan to check the centering of the beam in the exit slit and adjust the deflection plate voltage, as at lower energy, this parameter is more sensitive.

When everything is set, it is possible to save all the new parameter values in new presets (in Global, Detection, low current, etc…) to be recalled when using low energy.

To increase the impact energy, follow the exact same but reversed procedure:

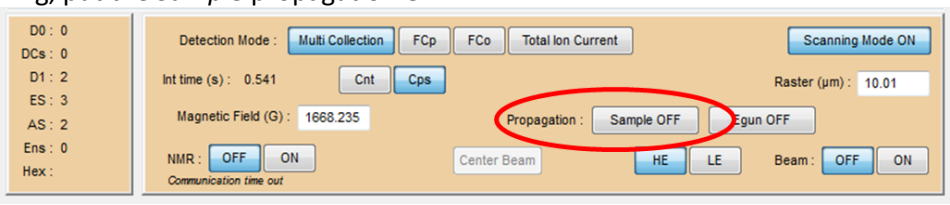
- If needed, save the low energy settings in new Global and Detection presets before going back to high impact energy.
- Put the *Sample* propagation ON.
- Set EOW to the desired value.
- Turn off the *Sample* propagation.
- Re- Adjust the Magnetic field.
- Re-Adjust the Raster.
- Re-adjust the EM HVs.
- Put back the corresponding values for the source: proceed by energy steps waiting a few minutes for stabilization, decreasing first both source heating currents, then increasing the source HV.

9.3.7.2 In Oxygen

Starting point: the instrument must be tuned at high energy with the RF-plasma source ON. Note the source parameter values. Note FCp as a reference. Note EOW, the B-field on the magnetic sector and the masses on the detectors. Note the raster size. If needed save the current presets.

Decreasing the Impact energy:

- In the Source window, decrease the source HV down to the desired value (for instance from 8000 V to 2000 V) and reduce the extractor HV by the same factor (from 4000 V to 1000 V if dividing the HV by a factor of 4). Note: this factor might need fine tuning in order to optimize the O- beam current in FCp.
- In Tuning, put the *Sample* propagation ON.



- Adjust the value of EOW (= sample voltage = half of impact energy) following a simple proportion law: if you want to decrease the high voltage by a factor of 2 (from 16 to 8 keV), divide EOW by 2. All lens and deflector values in the NanoSIMS primary and secondary optics and spectrometer will automatically adjust by applying the same factor, while coils will adjust following a square root law.
- Put the *Sample* propagation back OFF.
- The **magnetic field** of the magnetic sector analyzer does not automatically readjust. You must do so manually. To readjust the Magnetic field, select B-field on the keyboard and decrease the B-field value as to display to correct masses on the detectors.
- The **raster** does not adjust automatically either. Do not forget to adjust the raster size in Tuning.

It is then necessary to

- do a PHD scan and adjust (generally increase when reducing E) the HV of all EM used. Check the thresholds (they should not move much).
- Do an HMR scan to check the centering of the beam in the exit slit and adjust the deflection plate voltage, as at lower energy, this parameter is more sensitive.

When everything is set, it is possible to save all the new parameter values in new presets (in Global, Detection, low current, etc…) to be recalled when using low energy.

To increase the impact energy, follow the exact same but reversed procedure:

- If needed, save the low energy settings in new Global and Detection presets before going back to high impact energy.
- Put the *Sample* propagation ON.

- Set EOW to the desired value.
- Turn off the *Sample* propagation.
- Re- Adjust the Magnetic field.
- Re-Adjust the Raster.
- Re-adjust the EM HVs.
- Put back the normal Source and Extraction HV values on the RF-plasma source. Wait a few minutes for stabilization.

9.3.7.3 Low impact energy with the charge compensation e-gun

Tuning of the e-gun for low energy analyses can be done either before or after the rest of the instrument has been tuned for low energy, though it is easier to do it before.
Starting point: the e-gun must be ON in regular conditions. With diaphragm D1-2, one should read ≈ 1 µA in FCo (negative polarity).

To decrease the e-gun energy:

- In Tuning, put the propagation of the e-gun ON, with the "HV tuning" option unchecked (Figure 428: e-gun HV Tuning option unchecked in TUNING.
- Change step by step the e-gun HV in the Source window (-6000 V, -4000 V, -2000 V).
- At each step, ajust by iterations:
    a. The e-gun Be coil (Keyboard: PM > Coils > e-gun Be)
    b. C5 X-Y (Keyboard: LF6 > C5 X, Y) and C6 X-Y (Keyboard: LF7 > C6 X, Y)

  to keep the electron current in FCo constant.

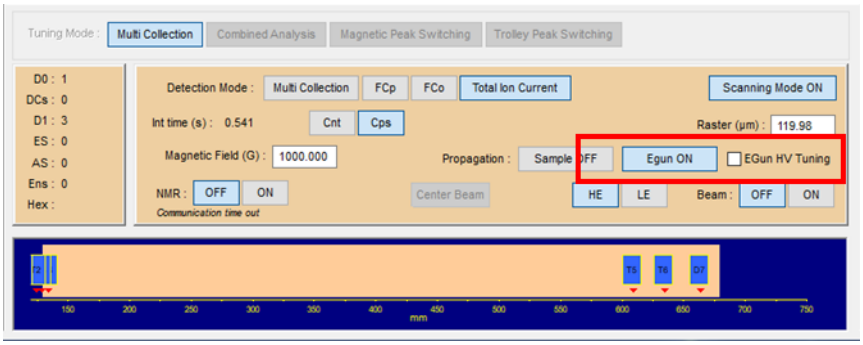


Figure 428: e-gun HV Tuning option unchecked in TUNING

Note: If the instrument is already tuned for low energy analyses, the L4 value will be decreased, and the reading of FCo will not be possible. You must then temporarily readjust L4 proportionally when decreasing the e-gun HV step by step, starting from 44 800 at -8000 V, 33 600 at -6000 V, 22 400 at -4000 V, etc…

Once the e-gun has been tuned to the desired low energy (for instance -2000 V), check "HV tuning" to refine the tuning of the e-gun (see chapter 9.3.2 on e-gun tuning) without affecting the other parameters.

To increase the e-gun energy, follow the exact same but reversed procedure:

- With the e-gun propagation ON in Tuning and HV tuning unchecked,
- Increase the e-gun HV
- Readjust C5 X-Y and C6 X-Y

When done with the e-gun at low energy, put the e-gun propagation OFF.

9.3.8 RTT: alternated real time tracking (sample drift correction)

9.3.8.1 Introduction

Due to several parameters (including room temperature variations inducing dilatation-contraction of the instrument, spurious magnetic or voltage instabilities, or sample charging), the relative position of the ion beam on the sample can vary over time. This is a particular concern for long analyses (several hours) at high magnification (raster < 5 µm). This drift can be a limitation when one wants to merge successive cycles of an image or when one wants to reconstruct a depth profile from a very small ROI.
To counter this issue, two strategies are available:

- *Post-analysis* drift correction:

Using WinImage II software, it is possible to correct this drift, provided there are some sharp structure or contrast in the FOV.

- Real Time Tracking (RTT):

In cases where there is no contrasted detail in the images, or for direct depth profiling (no image recorded) of very small FOV (ex: 2x2µm), it is better to correct the drift in quasi real-time, during the stack acquisition. This is the purpose of this *quasi real-time* drift correction function.

9.3.8.2 Alternated RTT Principle

A sharp feature must be available outside (near) the FOV of the image or profile to record. One first SIMS or SE image of this feature is recorded *before* the start of acquisition. Then at regular time intervals, the software will *pause* the image acquisition, go and re-image this detail, *determine the drift* (number of pixels in X and Y) comparing with original detail image (using the selected algorithm from WinImage II), *shift* inversely the *beam* position by this amount, and *restart* the image acquisition.
If there is no sharp contrasted detail around the FOV of the image, one must *create one* prior the acquisition (ex: a point (hole without scanning) or a small, sharp and deep square (e.g. ≤ 1x1µm) sputtered crater).

9.3.8.3 RTT Implementation

Inside a larger Working Frame, the user must define two areas: the ***Analysis area*** and the ***Reference area***. The reference area will image a characteristic feature on the sample that will allow to monitor the drift during the analysis and apply a correction to the analysis area.

To use the RTT option, select “yes” (top right corner of the **Def Analysis window**). New options appear (Figure 429).

Note that in the case of the RTT, the *Working* Frame and the *Scanning* Frame will be of different sizes. The analyzed area (the image to record) is the Scanning Frame smaller than and inside the Working Frame. The working frame will determine the pixelization of the two sub-images inside. This is important to consider when defining Raster and Pixel size of the Working Frame in order to keep adequate pixel number and size in the two smaller images.

1- With “Image Ref Definition” **not selected** (i.e. grey, see figure Figure 429: RTT in Def Analysis below), set all necessary parameters as usual. In particular, define the raster size and pixel size of the **Working Frame**, as well as the dwell time for the **analysis area** (Scanning Frame). You can also adjust the Scanning Frame size and position within the Working Frame but it will be resizable &

repositionable precisely a few steps later after the acquisition of the working frame image. The analysis scan frame can be rectangular.

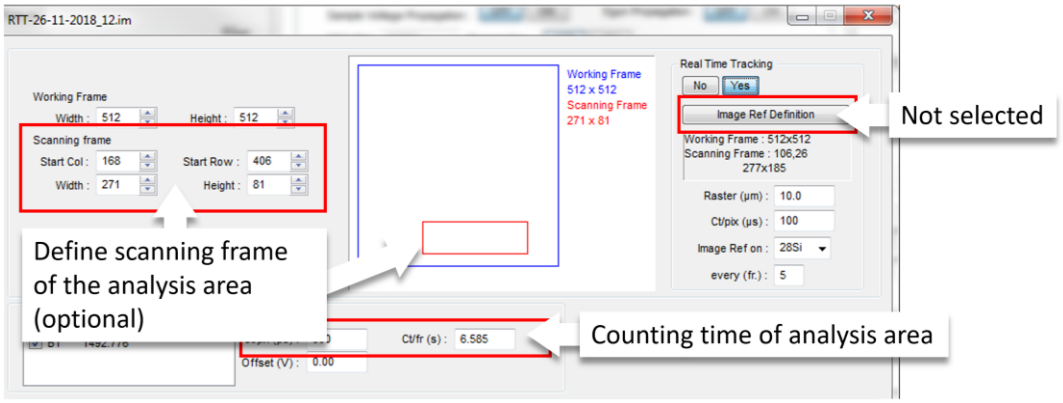


Figure 429: RTT in Def Analysis; Definition of the working and the scanning frames

With “Image Ref Definition” **selected** (i.e. blue, see

Figure 430: RTT in Def Analysis; definition of the reference image

2- below), you can adjust the parameters for the **reference image** (= the drift correction image). Make sure the Working Frame raster and pixel sizes match the ones of the analysis area. Define the dwell time for the reference image, the detector (SIMS or SE) for the drift correction program to use to compare images, and the intervals at which the acquisition pauses to record a reference image (i.e. every 10 analysis images). You can also adjust the Scanning Frame size (this time it is the reference image) but it will be resizable & repositionable precisely a few steps later after the acquisition of the working frame image. The reference scan frame can be rectangular. In the screen copy below it is large; in reality one will try to minimize this ref image size to minimize the time taken by the drift correction process, mostly for minimizing recontamination of the analysis crater by residual gas (e.g. for hydrogen analyses).

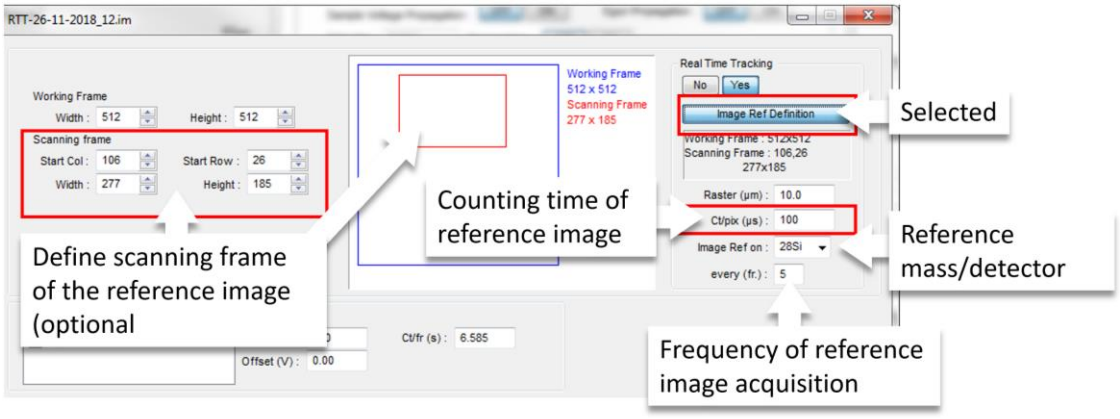


Figure 430: RTT in Def Analysis; definition of the reference image

3- Go to **Analysis**.
First acquire an image of the whole working frame by clicking Start Acq Def Zone.
When the acquisition is done, a new window appears, allowing you to optimize the analysis area as well as the reference image (Figure 431).
It also shows the drift area within the reference image, which is the pixel area used by the program to run the drift correction routine between cycles (Algo 1 Ref1 or Algo 2 Ref N-1, refer to WinImage program).
Each of these 3 areas can be adjusted. To do so, select the area you wish to change, and click on “remove” to erase the existing area. Then, with the mouse, draw the desired area. When satisfied, click on “valid” to save the new area. When all three areas are good, click on “close”.

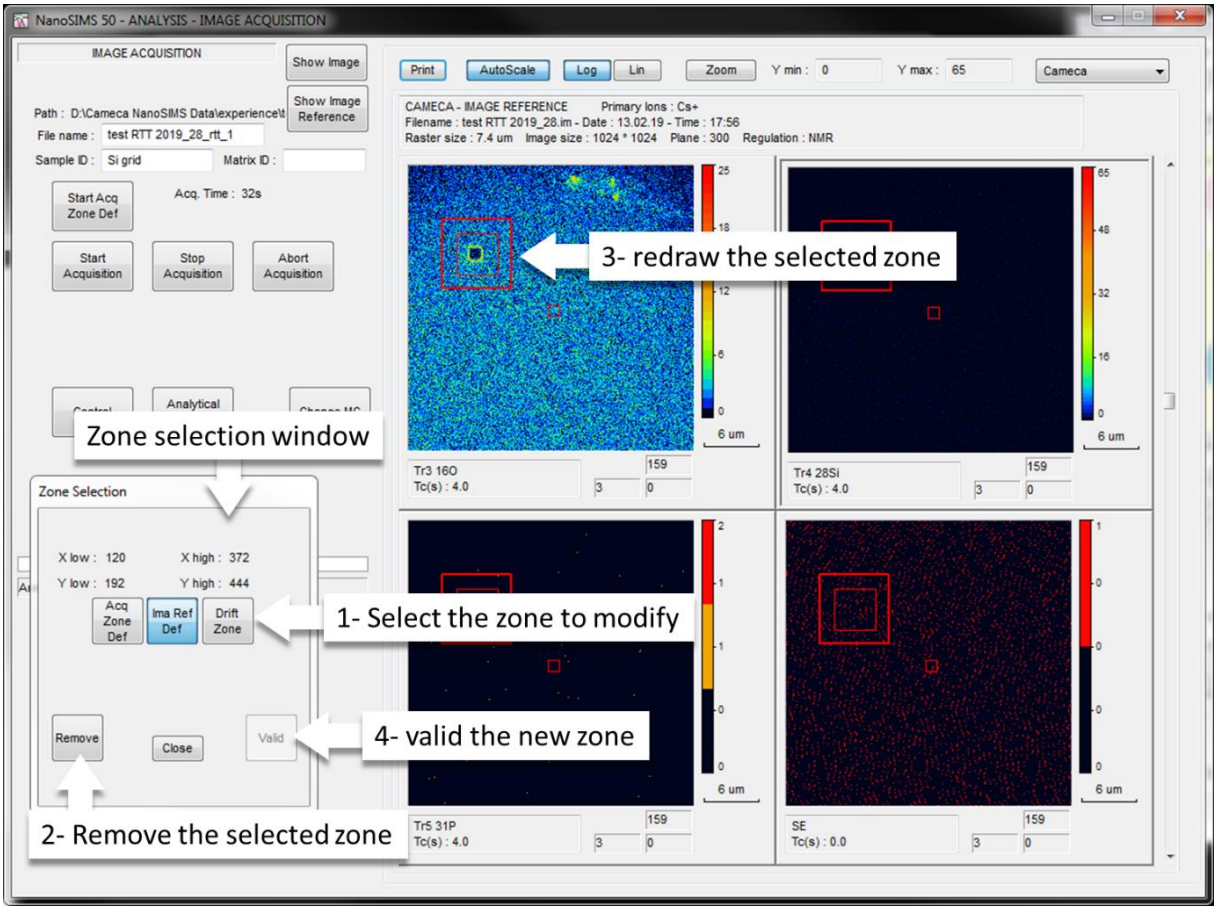


Figure 431: RTT : how to modify the Analysis area, the Ref area and the drift correction area before launching an analysis

4- Launch the analysis by clicking on “Start Acquisition” in the main Analysis window.

At the end of the acquisition there will be three files stored:

- the (multiple)-SI-SE analysis image stack (virus.im) with n cycles,
- the “largest” working frame (virus_rtt_1.im) with always one cycle, and one SI or SE.
- the reference image (virus_rtt_2.im) with less cycles than n, and one SI or SE.

It is sometimes useful to check the ref image to understand a problem of drift correction. Otherwise it can often be discarded as well as the working frame one.

9.3.8.4 Practical use

The Figure 431 displays a typical example requiring the use of a RTT: one wishes to record a long direct depth profile on a crater of only a couple µm in size. And there is quasi no contrast or detail on the surface: it would be impossible to do a drift correction after the acquisition !

The strategy is then here to start by creating a sharp detail by sputtering a small crater deep enough of 1.5X1.5µm with sharp edges and contrast. It is visible as a black square in the 16O image. This will be used as the detail to track during the RTT.
It could also be a simple spot on the sample, leaving the beam without scanning for a while; it would be faster to generate but possibly less effective for drift detection (to be tested).

A red square around the small crater is the reference scanning frame, of several µm width.
The depth profile crater will be positioned elsewhere within the Working frame.

As SIMS is destructive **the most delicate part** is to optimize the size and pixelization of the reference scanning frame around the detail to track:

- The acquisition of the ref scan should be as fast as possible in order to avoid recontamination of the real analysis crater by residual gas during the tracking process,
- the sputtering of this ref image should not be so strong that it destroys or smoothes the detail or changes its contrast (e.g. during a multi-layer depth profile the black square on light background should not turn white over black).
- But not too fast in order to get a good signal to noise required for proper drift detection routine (note that SE image can also be used, which can give good S/N),
- And the pixelization must be high enough to allow for proper drift detection: typically, pixel size = one third of the beam size. And this might limit the maximum size of the working frame. For example (it is not mandatory but to fix ideas) a beam size of 100nm will define a pixel size of 33nm, so with 1000x1000pixel for the working frame will give its max size as 33x33µm.

9.3.9 Annex: internal procedure of isotopic tests at NS50 installation

The present chapter concludes in some way the Expert operation chapter. It is a *condensed, internal* procedure allowing *an "expert" operator* to achieve the advanced isotopic specifications demonstrated at the end of all NS50L installation. Such tests require an instrument perfectly tuned, good samples (Si and quartz) and an understanding of all advanced instrumental and analytical aspects (primary and spectrometer tuning, multicollection, charge compensation, EM and FC detector tuning, statistics, etc…) and software.
It is a good internal test for expert operators wanting to check their mastering of the instrument as well as a test of the instrument shape since the installation.

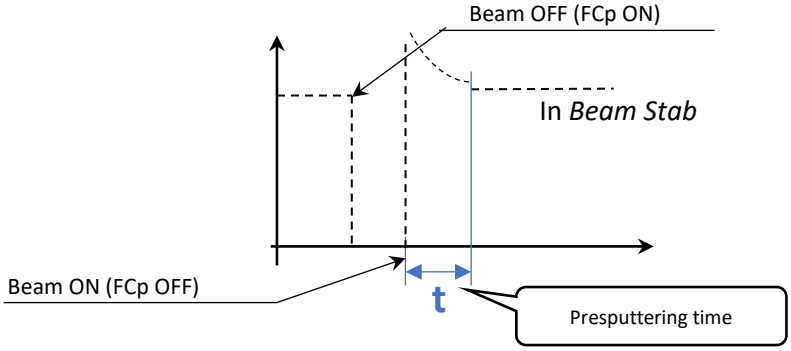
Note : All analyses are made in 64x64 px, 132 µs/frame (or 0,54 s/fr).

9.3.9.1 EM on Si wafer.

Tuning :

- Source Cs^+ ≈ 50 nA. D1-3, ES-3, AS-2. L1= 0.
- Adjust HV of used EMs for PHD max around 260 mV.
- Tune the NanoSIMS to have the 3 Silicon isotopes (^{28}Si, ^{29}Si, ^{30}Si) on 3 detectors and adjust the primary beam intensity to obtain ≈350 000 c/s on ^{28}Si and 12 000 c/s on ^{30}Si.
- Put the NMR regulation ON.
- Check that the mass fractionation is properly corrected by doing Cy and P3 centering with all 3 trolleys selected. CL for the 3 masses should be close. If not, B-field coils must be adjusted.

Determine the pre-sputtering time for a series of analyses in a single crater : Beam Stab → beam ON. Put the beam OFF for 10 seconds and ON again. Note the delay (t) before the signal stabilizes



Determine the length of an analysis (number of cycles necessary): the statistical noise or dispersion must always be neglectable compared to the reproducibility spec to demonstrate !
For sufficient statistics, if we accumulate 2 x10^6 counts on the minor isotope ^{30}Si, this will give a S/N ~(1/sqrt(N) = 7 E-4 = 0.7permil, a statistical fluctuation (just) low enough to demonstrate permil reproducibility.

Example : If we measure 1,5.10^4 c/s on ^{30}Si, to reach 2.10^6 cumulated counts will require 2.10^6/1,5.10^4 = 133 sec. We must them run an analysis of 133 sec. A cycle being set to 0.54 sec., the analysis must be of at least 270 cycles (for example 10 blocks of 27 cycles).

Launch an « isotopes » analysis, of 10x15 cycles (or more, as necessary) with a 4x4 µm raster, and including pre-sputtering as determined above. Do not forget to define the ratios (ratio 1: ^{29}Si/^{28}Si, ratio 2: ^{30}Si/^{28}Si). This analysis will be used a reference for the following *chained analyses* :

A-1 – 10 times the reference analysis in a single crater. Sigma (standard deviation) on ^{30}Si/^{28}Si must be < 1.25 ‰ (0.125 %).

For the next analyses, automatic EOS, SIB and Peak Centering are also necessary:

Centering the beam in the entrance slit

Tuning → EOS
Select the ^{28}Si detector
Start.
Write down L80.00 (= ES width).
Setup → Centering, check EOS *width*. If the new value is significantly different, Apply L50.
Apply CL. Save to Def Analysis.
Select *Automatic EOS Centering*
Start.
Apply CL. Save to Def Analysis.

Centering the beam at spectrometer « entrance ».

Tuning → Sec. Ion Beam

Horizontal (Cy)

Voltage step=0.15 V
Start.
Apply L50 if necessary
Apply CL. Save to Def Analysis.
Select *Automatic Beam Centering*
Start.
Apply CL. Save to Def Analysis.
Go back to *SIB*

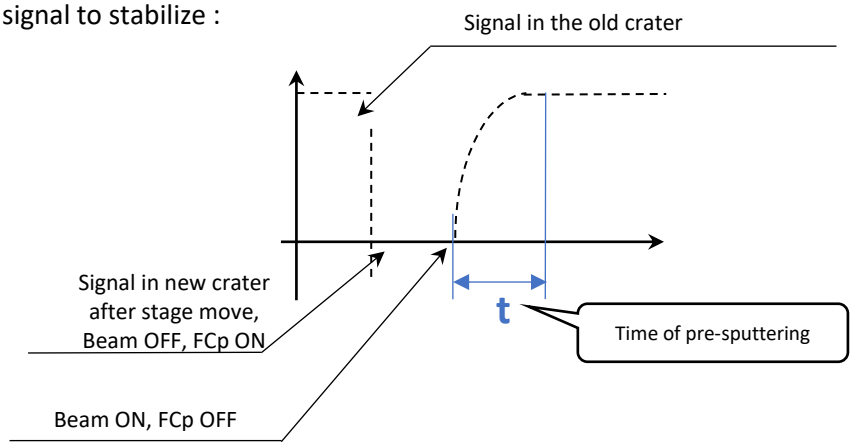
Vertical (P3)

Voltage step=0.3 V
Start.
Apply L50 if necessary
Apply CL. Save to Def Analysis.
Select *Automatic Beam Centering*
Start.
Apply CL. Save to Def Analysis.
If necessary, add a Peak Centering :

Automatic Peak Centering

Tuning→ HMR
Select the ^{28}Si detector. Start – Apply CL
Select *Automatic Peak Centering*
Start.
Apply L50
Apply CL. Save to Def Analysis.

You also need to determine a new pre-sputtering time for crater-to-crater analyses: Beam Stab → beam ON. Then put the beam OFF and from the Navigator, move the stage of about 20 µm. Put the beam back ON and wait for the signal to stabilize :



Do a new reference analysis. This time, in Def Analysis, add EOSC, SIBC, EOSC and select « peak centering » for the used detectors. Use ^{28}Si as the centering reference for the others. Do not forget to add the new pre-sputtering time.

A-2 – This test checks the reproducibility in « beam deflection » mode. To run this test of 12 analyses, use the « Grain Mode » analysis type and « Spec », where the test is pre-defined. Set the analysis as follow: 256x256 px, raster 35 µm, 0,54 ct/fr. Go to Acquisition and launch the analysis. The program will acquire a first (empty) image of 35x35 µm that we do not care about. Then click on « Go to Isotopes ». This is where you define the test parameters : Add the pre-sputtering time, the EOS, SIB, EOS centering, and the peak centering. Here sigma (standard deviation) on ^{30}Si/^{28}Si must be < 1.4 ‰ (0.14 %).

A-3 – This test checks the reproducibility when moving the stage by doing a series of 16 analyses over a 8 mm square. On the 1 inch Si wafer, move the stage from the Navigator so that the starting coordinates are around: X = -8000, Y = -4000.

In chained analysis, use the reference analysis, choose movement option #3 and set the moves as follow:

X= -2670 x1 Nb = 2
Y= 2670 x3

Sigma (standard deviation) on ^{30}Si/^{28}Si must be < 1.4 ‰ (0.14 %).

A-4 – The last test is a series of 12 analyses on 5 different samples on a sample holder of the *Geology* type. First you need to define manually the coordinates as follow : 2 analyses on sample 1, 2 analyses on sample 2, 2 analyses on sample 3, 2 analyses on sample 4, 2 analyses on sample 5 and 2 analyses on sample 1 again. Sigma (standard deviation) on ^{30}Si/^{28}Si must be < 1.65 ‰ (0.165 %).
Here, Z might significantly vary from one sample to another, making the automatic centering on EOS difficult. If necessary, determine the correct Z for each point by doing as follow:

- Pick a reference point (for instance on sample 1), check Z and do a EOS centering. Note the EOS centering CL value.
- Move to the next sample. Keep the same EOS value and adjust Z as to maximize the signal.

- Note Z and and repeat the operation for all the analysis points. For each point, Edit its coordinates with the new Z value.

9.3.9.2 Faraday Cups on Si wafer

Tuning :

- Source Cs^+ ≈ 50 nA. ES-3, AS-2.
- Switch to FC, with 10^{11} Ω pre-amplifiers on the 3 detectors.
- Tune the NanoSIMS to have the 3 Silicon isotopes (^{28}Si, ^{29}Si, ^{30}Si) on 3 detectors and adjust the primary beam intensity to obtain ≈25 pA on ^{28}Si and 0,7 pA on ^{30}Si (Ex : D1-2 with L1 = 25000 or D-3 with L1 = 26600)
- Calibrate the FC background.
- Put the NMR regulation ON.

Set the EOS/SIB automatic centering as well as the Peak centering (see above).
Determine the crater-to-crater pre-sputtering (see above).
Do an « isotopes » analysis of 10x15 cycles, with a raster of 10x10 µm. Add EOS, SIB, EOS centering, Peak Centering and pre-sputtering. This analysis will be used a reference for the following *chained analyses* :

B-1 – 10 times the reference analysis in 10 different craters 20 µm apart. In *chained analyses*, choose move option 2:

dY = 30 µm Nb = 10

Sigma (standard deviation) on $^{30}Si/^{28}Si$ must be < 0.45 ‰ (0.045 %).

B-2 – A series of 16 analyses over a 8 mm square (see test A-3). On the 1 inch Si wafer, move the stage from the Navigator so that the starting coordinates are around: X = -8000, Y = -4000.

In chained analysis, use the reference analysis, choose movement option #3 and set the moves as follow:

X= -2670 x1 Nb = 2
Y= 2670 x3

Sigma (standard deviation) on $^{30}Si/^{28}Si$ must be < 0.6 ‰ (0.06 %).

B-3 – A series of 12 analyses on 5 different samples on a sample holder of the *Geology* type (see test A-4). Sigma (standard deviation) on $^{30}Si/^{28}Si$ must be < 0.08 %.
For each series, make sure the FC background remains stable (small σ on the background signal).

9.3.9.3 Faraday Cups on Quartz

Tuning :

- The e-gun must have been started the day before to degas and stabilize. You will tune it properly the next day.
- Source Cs^+ ≈ 50 nA. ES-3, AS-2.
- 10^{10} Ω pre-amplifier on ^{16}O, and 10^{11} Ω pre-amplifier on ^{18}O (change the ^{16}O pre-amplifier and do not forget to do a *FC Calib*)
- Tune the instrument to have ^{16}O and ^{18}O on 2 FC detectors and adjust the primary beam intensity to obtain ≈250-350 pA on ^{16}O and 0,5-0,7 pA on ^{18}O. Ex : D1-1, L1 = 26800.
- Note : for high currents you might need to re-adjust the secondary beam tuning. Check ES and AS positions, and LF3, LF4, C3X and C4X centering.
- Carefully tune the e-gun as you need a very stable signal at high current.
- Put the NMR regulation ON.

Set the EOS/SIB automatic centering as well as the Peak centering (see above).
Determine the crater-to-crater pre-sputtering (see above).
Do an « isotopes » analysis of 10x15 cycles, with a raster of 10x10 µm. Add EOS, SIB, EOS centering, Peak Centering and pre-sputtering. This analysis will be used a reference for the following *chained analyses* :

C-1 – 10 times the reference analysis in 10 different craters 20 µm apart. In *chained analyses*, choose move option 2:

dY = 30 µm Nb = 10

Sigma (standard deviation) on $^{18}O/^{16}O$ must be < 0.8 ‰ (0.08 %).

C-2 – A series of 16 analyses over a 8 mm square (see test A-3). On the 1 inch Si wafer, move the stage from the Navigator so that the starting coordinates are around: X = -8000, Y = -4000.

In chained analysis, use the reference analysis, choose movement option #3 and set the moves as follow:

X= -2670 x1 Nb = 2
Y= 2670 x3

Sigma (standard deviation) on $^{18}O/^{16}O$ must be < 1 ‰ (0.10 %).

Notes :

Switch from EM to FC (or FC to EM)

1- Click on FC/EM in the Tuning window for all trolleys you want to switch (for instance trolley 1, 2 and 3) and follow the procedure for each of them.
2- In Setup → Tuning, check that the trolleys are now noted as FC.
3- In Setup → Centering, adjust the waiting times (WT)
- For EMs: WT9 = 2s, WT0 = 3s, WT1 = 3s (5s when using the e-gun at high current)
- For FCs: WT9 = 10s, WT0 = 10s, WT1 = 10s
4- Do a *FC Calib* for the 3 detectors.

Change a FC pre-amplifier

1- Open the cylindrical box containing the pre-amplifier boards and manually switch the jumper of the pre-amplifier.
2- Setup → Hardware →Detection
Select the detector whose pre-amplifier you just switched and change the resistor info:
FC pre-amplifier resistor : 10 for 10^{10} Ω (for very high currents)
100 for 10^{11} Ω
3- Tuning →FC Calib
Waiting time : 5s
Counting time : 5s
Select all used detectors
Start

Extract isotope data from a chained analysis

1- Launch Ana2Excel
2- Click on “file” and select a file from the chain you wish to extract data from.
3- Select ‘section’ 4 and click on “extract”. This will create a .csv file containing data from *all* the files from the chain. Sélectionner la ‘section’ 4, et cliquer sur “extract”.
4- In Excel, click on “extract from text/CSV and select the newly created csv file. Select “delimited”, then, depending on your Excel version:
   a. click“semi-colon”, then “finish”.
   b. click on “load”.

The Excel file thus created is a summary file containing data from each analysis of the chain. At the bottom right, average, standard deviation and standard error (in ‰) are calculated.

Notes on pre-sputtering

To gain time, it is possible to do the pre-sputtering by creating a High Current preset file for pre-sputtering :

- Increase L1 and adjust C1 X-Y so that the image doesn't move beam pre-sputtering conditions and analysis conditions.
- Save (Calib) those values in a new *High Current* preset.
- Also save (Calib) a preset with the normal analysis conditions.
- Send both presets to Def Analysis (« send for pre-sputt » and « send for acq » respectively).
- In Def Analysis, select those presets when setting the analysis.

9.4 Remote control through the internet

Remote access is currently possible via Team Viewers software. This allows the user to take control of the two computer screens of the instrument, and thus do everything that is accessible via the instrument's computer.

Of course, the laboratory firewall must be deactivated to grant the access to the instrument's PC from the outside. And real-time tuning will require a good network bandwidth.

This solution allows:

- Tuning
- Running analyses (limited to a single sample holder as exchange is manual)
- Monitoring the state of the instrument (sources, vacuum, etc…)
- Retrieving and processing data

This functionality is daily used by Cameca service team to diagnose, or tune customer's instruments.
It is also very practical to follow or check from time to time long chained acquisitions, from another location or room.
The keyboard located on cabinet A is replaced by a virtual keyboard accessible via the *keyboard* program in the "other" taskbar of the Board (see chapter 7.3).

The local dedicated three-roller pad is replaced by the use of the user's keyboard arrows, keys and mouse. In order to change a parameter, the user can:

- Whether enter directly the numerical value to be sent (and type enter), or

- Use the **mouse wheel** to modify the value. By scrolling the mouse wheel, the numerical value will be changed one bit by one bit.
  If the operator presses the **CTRL key** while scrolling the mouse wheel, the value will be changed x10 faster: ten by ten bits.
  Pressing **Ctrl + Shift keys** will change parameters **x100 faster** (100 by 100 bits).
  The **"Fast" button** is equivalent to the "x10" of the physical keyboard, thus when "on" (blue) while scrolling the mouse wheel, whatever the key combination the speed will be accelerated by x10.

- Or use the PC keyboard instead of the mouse wheel as:
  - **Letf/right** arrows are used to move **X** parameter,
  - **Up/down** arrows are used to move **Y** parameter,

- **m/n** key are used to move **Z** parameter,

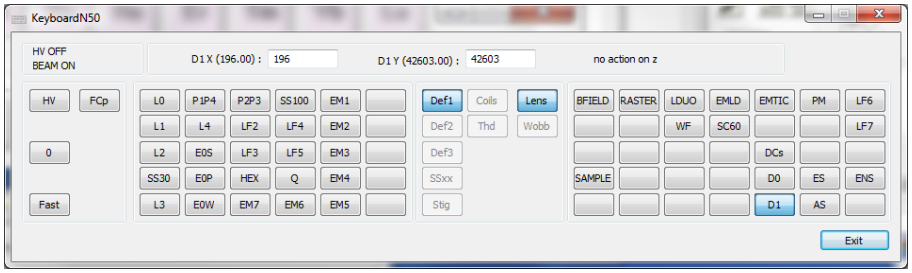
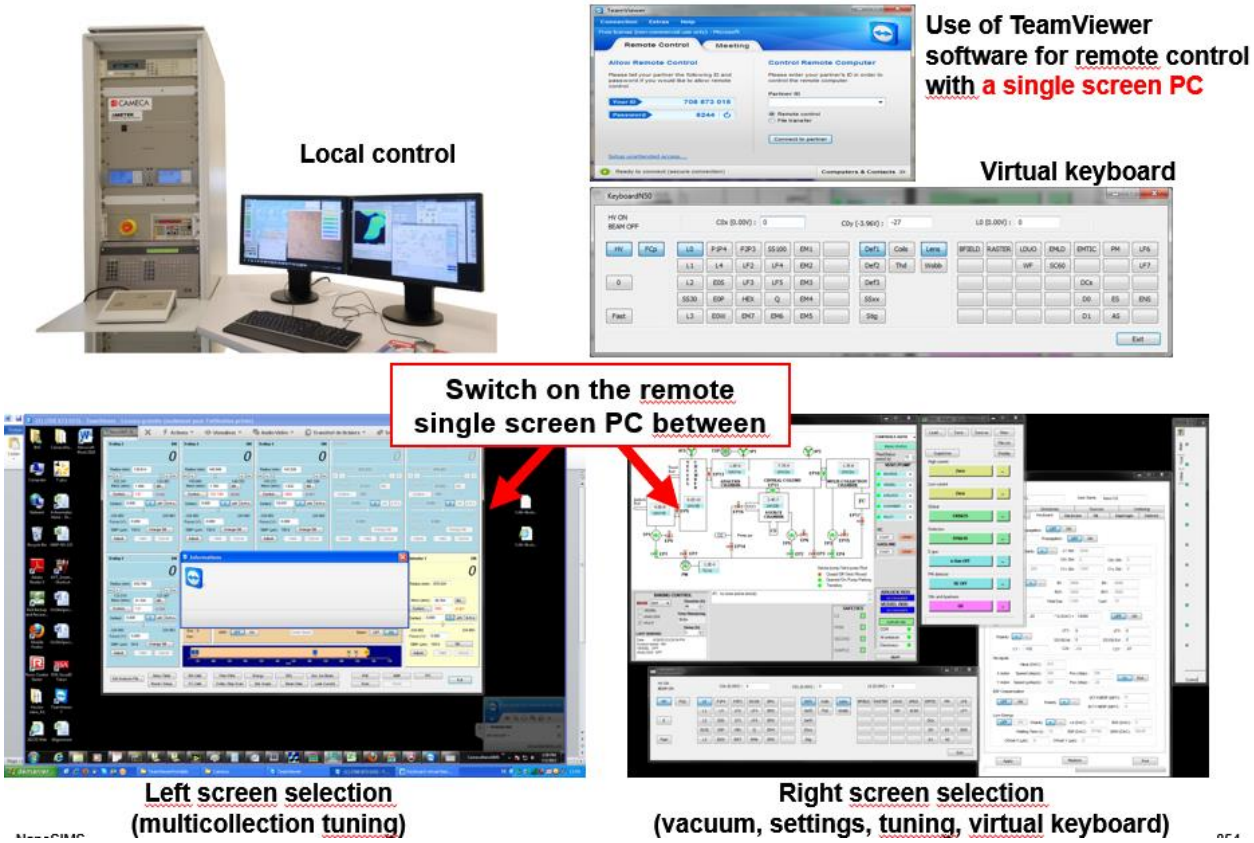


Figure 432 : virtual keyboard



As illustrated above the control of the two instrument screens can be performed on a PC equipped with a **single screen**. A tab in the Team Viewer program is available to toggle between the two screens.

Alternately, the use of a remote PC with **two large screens** is more comfortable. This can be configured in the “View” menu of Teamviewer.

However, one must keep in mind that there are instrumental limitations that prevent certain operation to be done remotely:

- Sample transfer from vessel to analysis chamber is manual. Therefore, it is not possible to change sample remotely.
- It is only possible to restart the NMR on site.
- Most electronics chassis need to be turn on/off on site. The electronics cannot be turned off remotely (for baking of analysis chamber and vessel for instance)

- The Oxygen source cannot be completely operated remotely (Open/Close the Oxygen bottle, adjust the leak valve).
- The goniometer cannot be adjusted remotely, so it is not possible to switch between sources.
- The Ti sublimator controller is only accessible on site.

Hence the presence of a local operator is still required for these operations.

In addition, it is always good to be able to listen to the instrument, to hear motor movements (when it starts, when it stops), or to identify unusual noises like a turbopump whistling, etc. This diagnosis-by-ear is not possible remotely or require a separate communication software aside (e.g. a separate laptop inside the lab running Teams or other).

10 NanoSIMS 50L Maintenance

10.1 Stop and start of the instrument

For many reasons, the operator may have to switch off the instrument completely (for example, laboratory electrical maintenance) or just the real-time electronics (for example, baking).

10.1.1 Partial Stop of the Electronics and Restart

This chapter details the procedure to turn off the electronics. When the electronics is off, only the main functions are left on, such as the vacuum automation, the pumps, etc… while the Real Time Unit, the low and high voltages are turned off.

10.1.1.1 Stop

To stop the CAMECA NanoSIMS 50L electronic, follow this procedure:

- Place your sample in “Unload mode” and, if needed, remove it (see 9.1.1.5)
- Turn off the source (see 9.1.3) Cs+ or RF-Plasma, as well as the e-Gun if it was used (see 9.3.2.3.
- On the keyboard, Turn the HV off (green light off) 

- Put the Magnetic Field to 0 in Tuning, and switch the Bfield chassis and the motorization chassis off via the two switches on the front face of the Cabinet B (Figure 433)

Figure 433: front face of electronics cabinet B

- On the front face of the cabinet A, turn the key to its “OFF” position (Figure 434). All electronics, including the RF source cabinet, will be turned off.

Figure 434: front face of electronics cabinet A

10.1.1.2 Re-start after a partial stop

To restart the CAMECA NanoSIMS 50L electronics, follow this procedure:

- In the Cameca NanoSIMS50 menu on the PC desktop (Figure 435):

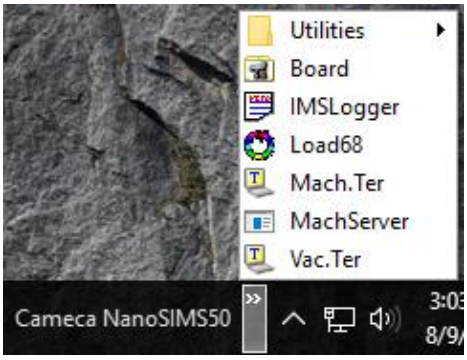
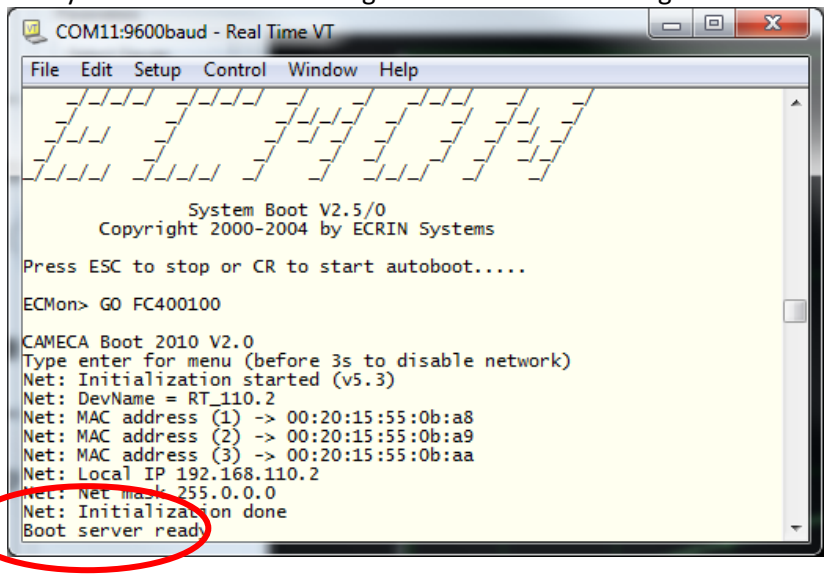


Figure 435: Cameca software are accessible from the Windows taskbar

a. Open the Real-Time terminal **Mach.Ter** (see Figure 265)
b. Open the **Load68** program (see Figure 266)
c. On the front face of the cabinet A, turn **the key in its "ON" position** (Figure 434)

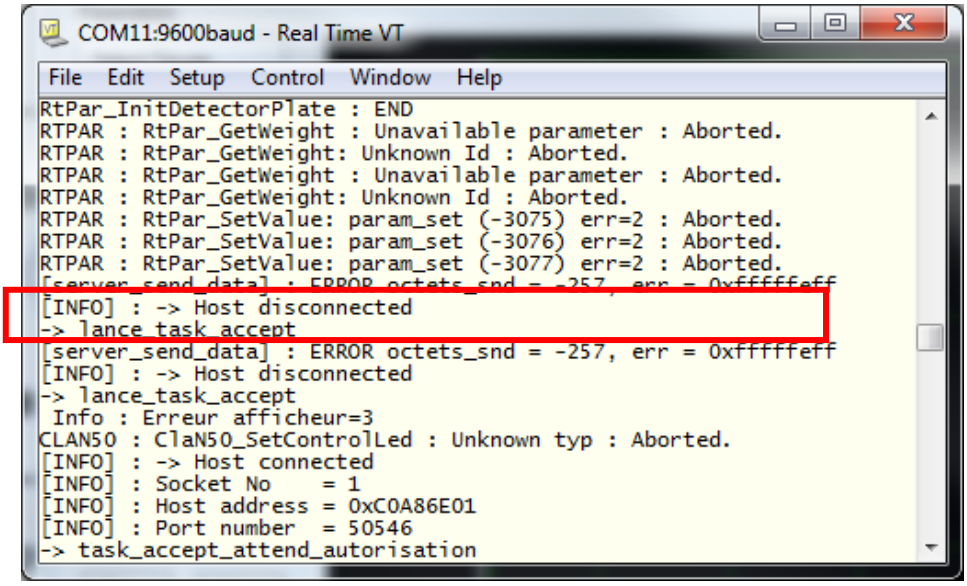
- Switch on the **Bfield chassis** and the **motorization chassis** on the front face of the Cabinet B (Figure 433)

- On the RT-terminal you should have a message similar to the following:

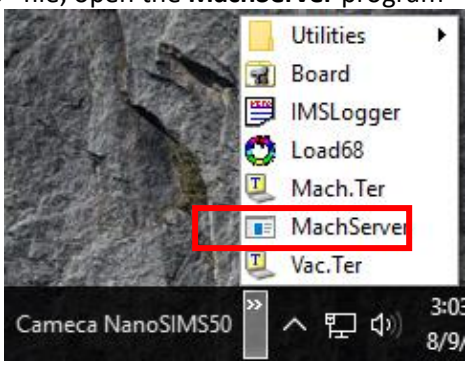


- The last message of the dialog box must be "**Boot server ready**".
- On the Load68 program, click on load 

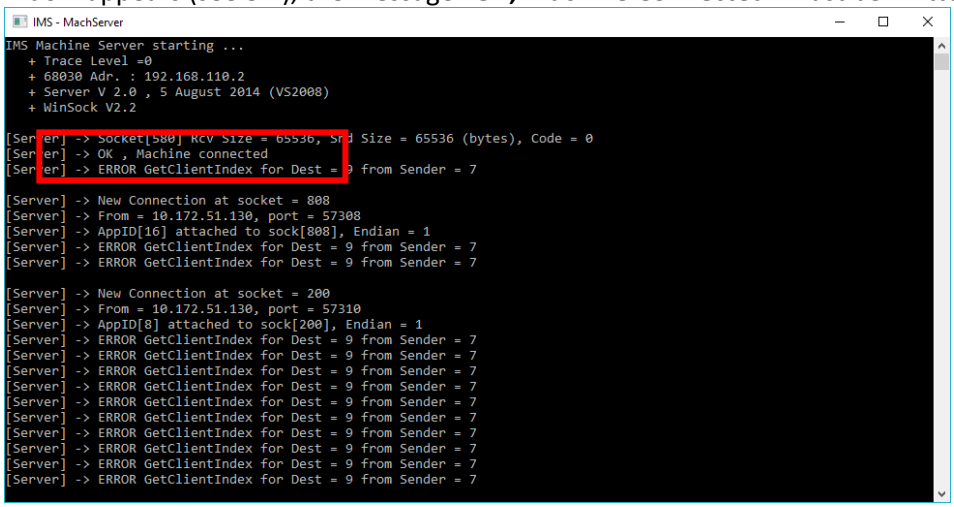
(see 8.3)
- Wait for the download until the message "**Host disconnected**" appears



- On the “Cameca NanoSIMS50” file, open the **MachServer** program

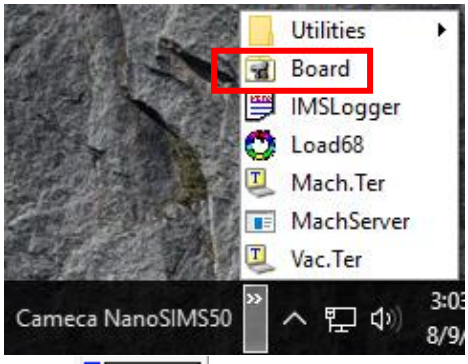


- A black window appears (see 8.1), the message “**OK, Machine Connected**” must be written



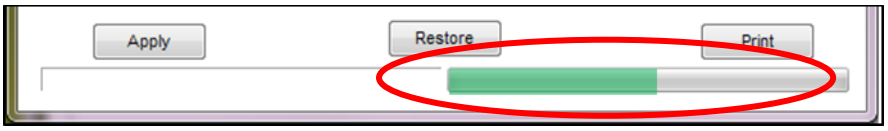
- Leave this window activated. Minimize this window but **do not close it** (otherwise, the communication will be lost)
- **Turn on the HV** on the keyboard 

- In the “Cameca NanoSIMS50” file, open the “**Board**” program



- In the “Board”, open the “**Setup**” 

- On the bottom right of the “Setup” window, wait for a green loading bar



- In TUNING, open Motor Reset/Setup and click on “**Init communication with motor**” to reestablish the communication with the motors.

The instrument is ready to use.

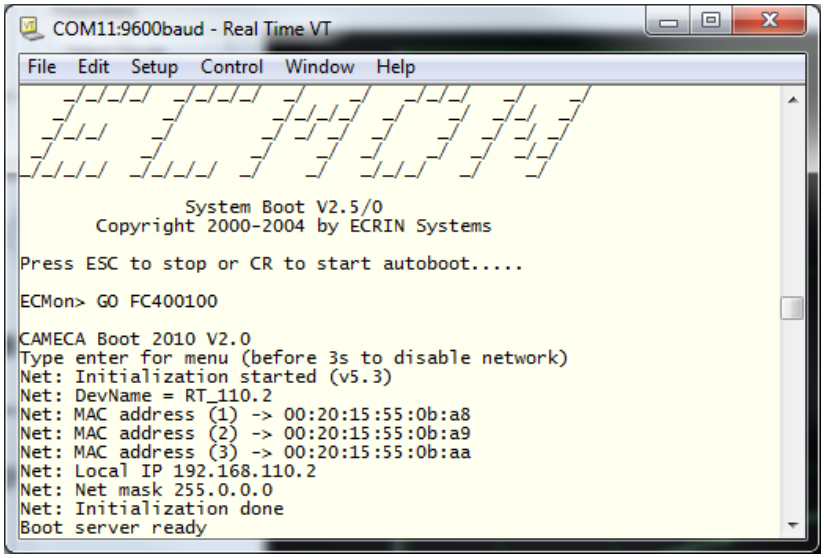
10.1.1.3 Real Time Electronics Reset

In some situations, the operator must restart the RT electronics without all the electronics stopping procedure (see 10.1.1.1). This operation can be useful for instance when the operator wants to restart the RT electronic without turning off the source.
To reset the CAMECA NanoSIMS 50L Real-Time, follow the procedure:

- **Turn OFF the HV** on the keyboard 

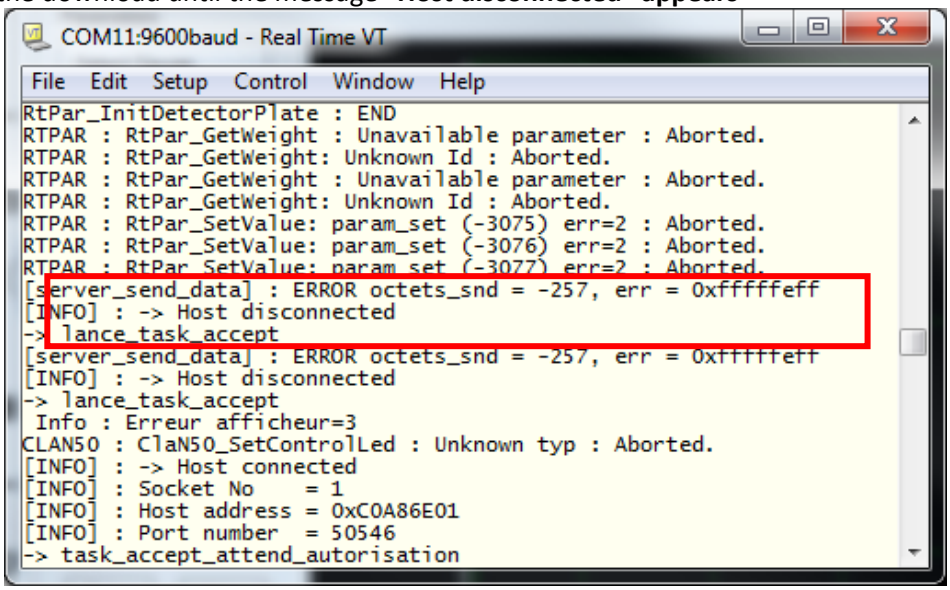
- Press the **reset button on cabinet A** front side once 

- On the RT-terminal you should have a message similar to the following, with the last message of the dialog box being: “**Boot server ready**”.

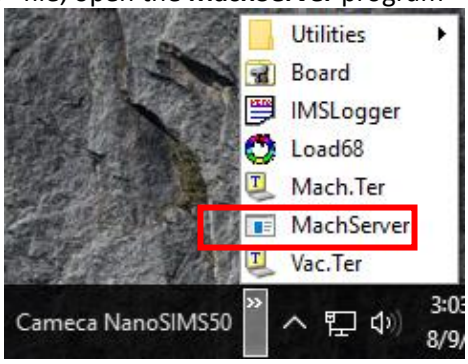


- Close the current MachServer program
- On the **Load68** program, click on load 

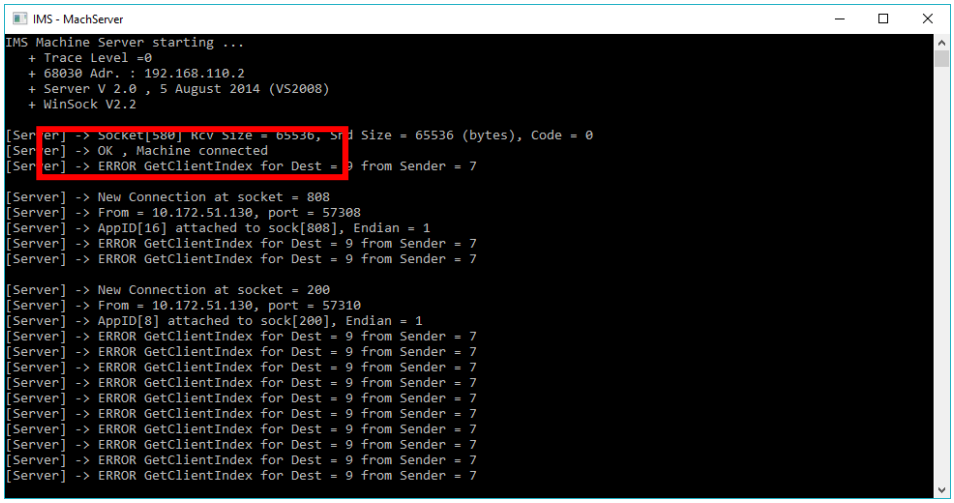
(see 8.3)
- Wait for the download until the message "**Host disconnected" appears**



- On the "Cameca NanoSIMS50" file, open the **MachServer** program



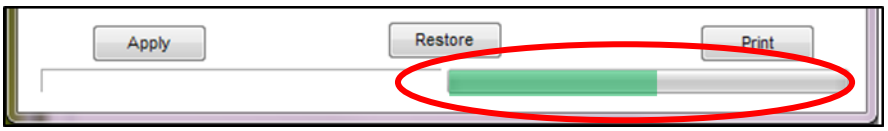
- A black window is open (see 8.1), the message "**OK, Machine Connected**" must be written



- Leave this window activated. Minimize this window but **do not close it** (otherwise, the communication will be lost).
- **Turn on the HV** on the keyboard 

.
- "Board" and "Setup" 

should be open (if not, open them)
- On the bottom right of the "Setup" window, wait for a green loading bar



10.1.2 Complete stop/start of the NS50L

Contrary to the partial stop, where the main functions stay on, during a complete stop everything is stopped (including pumps, automaton, etc…). This procedure is necessary, for instance, when the lab is closed for a long period of time, or if a power cut is scheduled.
Note that these instructions below are to stop the instrument. Some elements, such as the air compressor, the water chiller or the UPS are completely independent from the instrument.

10.1.2.1 Complete stop

For a complete stop of the instrument, follow this procedure:

- Stop the ion source, wait for the end of the process.
- Stop the electron flood gun (NEG).
- If the Oxygen source was in use, make sure the Oxygen bottle is closed, then pump the remaining oxygen gas of the oxygen source by click on "gasline start" in the Vacuum synoptic window.
- Turn off the HV on the keyboard
- Put the magnetic field to zero in the Tuning window and turn off the magnet chassis (Figure 436).

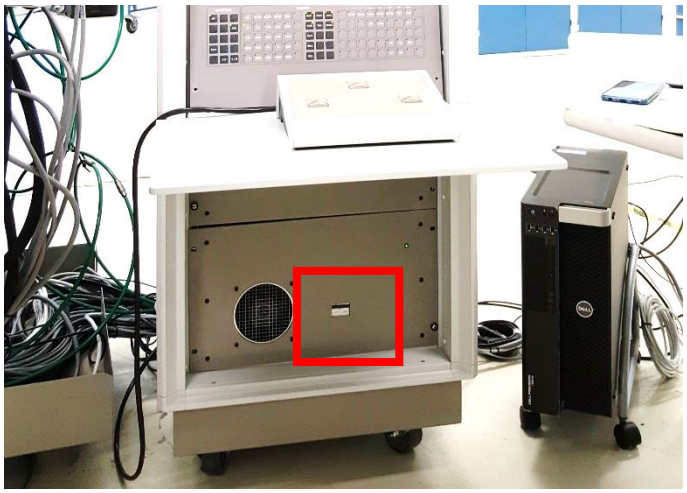


Figure 436: switch of the magnet chassis on cabinet B

If you need to stop the pumping system (for instance, a power cut that will stop the pumping system, or for a long stop), you must vent the part of the instrument pumped by the turbo pumps. This will stop the turbo pumps as well as fill the instrument with nitrogen. This will limit the amount of atmospheric air (and thus moisture) that could leak into the instrument in the absence of pumping. Ion pumps are controlled by the automaton and will be turned on when the equipment is turned off (see below).
To stop the turbo pumps:

- Make sure the dry nitrogen tank or bottle is filled and connected.
- Starting point: the system is in normal operation (all turbos and primary pump are running, all gauges are on). Main connecting valves are closed (EP9, EP10, EP11 and EP13) as well as venting valves (EP3, EP5, EP6, EP7, EP14, EP15, EP17).
- Using Vent/Pump switches on the vacuum synoptic (see chapter 5.10) vent the source, the multicollection and the airlock, to stop the turbo pumps and the primary pump. During the sequence, the vacuum gauges UHV1B, UHV2A, UHV2B will turn off. This may take a few minutes.
- Make sure all presets are saved and close all the programs. Turn the computer OFF.
- On cabinet B, turn OFF the NMR and the motor chassis (Figure 437):

Figure 437: top of cabinet B, with the motor chassis and the NMR

- On cabinet A, turn the **Equipment OFF** by pressing the **red button**. Then turn the **key** to the "OFF" position (Figure 438). Everything should now be shut down (including ion pumps, Ti sublimation and RF source cabinet). You should hear the turbo pumps slowing down and stop. You can now unplug the main supply cable of the instrument.

If you do not need to stop the pumping system (for instance, a temporary lab closure)**:**
On cabinet A, only tun the key to the “OFF” position. All the electronics should now be shut down (including the Ti sublimation and the RF source cabinet) but the “equipment” will remain ON, thus keeping all turbo and ion pumps on.

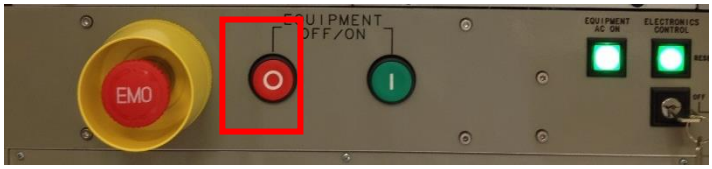


Figure 438: equipment, electronics and main switches on cabinet A.


Figure 439: main supply cable plug

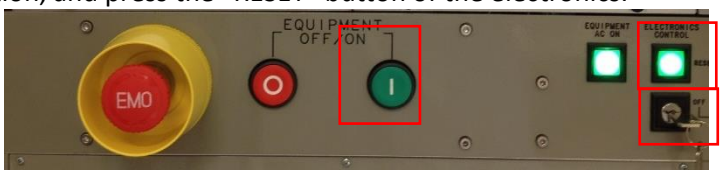
- In case of a global power cut of the lab requiring everything to be shut down, do not forget to stop the air compressor, the water chiller and the UPS as well. In this case, it is better to close all the monovalves.

10.1.2.2 Complete Start

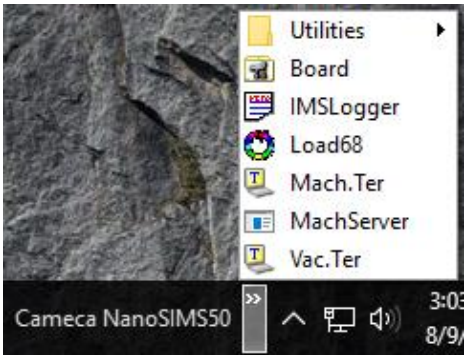
This procedure is applicable whether the stop of the instrument was voluntary following the procedure above, or the result of a power failure.

To completely start the instrument (instrument being totally off, vented and unplugged), follow this procedure:

- Plug the main supply cable of the instrument
- On cabinet A, switch ON the Equipment by pressing the green button “Equipment ON”, turn the key to the ON position, and press the “RESET” button of the electronics.



- Turn the PC computer on and open the programs via the NanoSIMS 50 menu from the Windows task bar:
    a. First open VacTer. Position the cursor in the VacTer window and press Enter.
    b. Open Mach.Ter
    c. Open Load68
    d. Open MachServer



- On cabinet B, turn the motor chassis, the NMR and the magnetic field chassis back ON.
- Turn the compressor ON.
- Launch the “Board” program. Inside it, launch the Vacuum program to get the vacuum automaton to start pumping the instrument. Everything should be automatic. Let the automaton follow its programmed procedure.
- You can now put the HV ON.
- Via Board, open the Serial Server to connect the Oxygen source. Click on “show console” to make sure the connection is ok (if not, click on “reconnect”)
- In TUNING, open Motor Reset/Setup and click on “Init communication with motor” to reestablish the communication with the motors.

- When the vacuum is restored in all the parts of the instrument, you can start the source and start working. However, it will take some time for all elements to stabilize (pumping system, source heat, magnet, etc…).

After a long venting of the instrument, the vacuum will take hours to improve. To restore an optimum vacuum, which is particularly critical for light element analyses, one might need to bake the system, degas the titanium filaments and the BA gauges.

Even after a short period of venting, it is recommended not to start high precision analyses right after a complete stop and start but to wait a few hours. This time however can be used to tune the instrument. In particular one can:

- slowly degas the electron multipliers by raising them up to a few M c/s then recheck all PHDs,
- slowly restart the cesium source and let it degas/pump down before setting it to full power. It could generate contamination on the extractor and lead to a re-opening for cleaning it.
- Let degas the NEG as it warms up.
- Turn up the immersion lens voltage carefully as some debris might fly around during a venting or pumping. Never force on HVs. It might create arcing marks on insulators leading to a re-opening for sanding them. Arcs between electrodes and ground can be detected for example when the pressure rises as HV are raised (without beam on). Raising HVs slowly to burn a dust or reversing the polarity might cure some insulation problems.

- Reset/Init the motors via the Motor Reset/Setup program (see chapter 5.2.10) and the Navigator program (see chapter 5.3.1.9).

10.1.3 Emergency Stop with EMO and restart

The instrument is equipped with an emergency off (EMO) button. Note that if you can safely shutdown the instrument following the procedure above, it is always recommended to do a normal shutdown. However, sometimes a quick stop of all voltage is required, such as fire or a water leak that may cause short circuits.

In case an emergency stop of the instrument is necessary, press the red EMO button. This shuts down all power supplies to the instrument including the RF source cabinet (i.e. a complete shutdown).

After an EMO shutdown, you need to unlock the EMO button to be able to start the instrument. To do so, turn the button clockwise. Then follow the regular starting procedure for a “complete start”, as described in chapter 10.1.2.2.

10.1.4 General lab power failure and restart

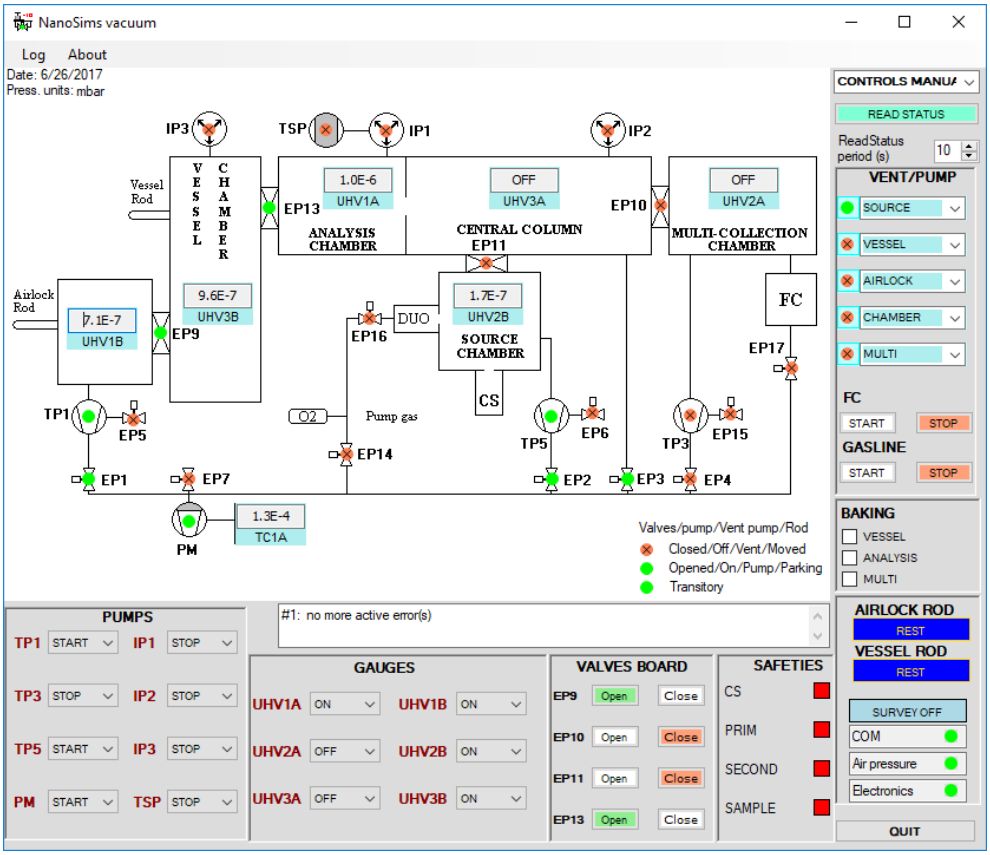
In case of a general power failure at the lab, and in the absence of a UPS, the instrument will shut down. The air compressor will fail as well, causing all the valves to close to preserve vacuum inside the instrument, except for the valves between airlock and the vessel chamber and between the vessel chamber and the analysis chamber (see chapter 2.3.3).

If the lab is equipped with a UPS, it will provide power to the instrument for 45 to 60 min. In case the lab power failure is expected to last, it is recommended to take advantage of this extra time to shut down the instrument properly, following the “complete stop” procedure above.

Whether or not the instrument has been shut down properly or not, once the power is back up, the instrument will not restart by itself. You must follow the “complete start” procedure above to restart the instrument.

10.1.5 Restoring vacuum after a long instrument down time

When the down time of the instrument is relatively short (a few hours to a couple days), the vacuum inside the instrument will be preserved enough to allow the vacuum automaton to restart the pumping system following the automatic procedure (see “complete start” procedure above). However, after a long down time, the vacuum might have degraded too much and when launching the vacuum automaton, the pumps will stop. In this case, it is required to start the pumps manually.



From the vacuum synoptics:

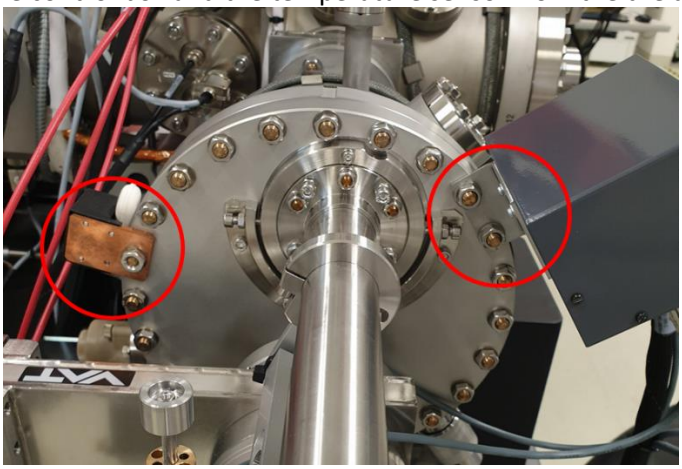
- Open the valves between the different chambers (EP9, EP13, EP11 and EP10).
- Switch the vacuum synoptics controls to manual mode in the vacuum synoptics.
- Start the primary pump (PM) until the vacuum reaches around 1.10^{-4} mbar at the TC1A gauge.
- Then, you can start the turbo pumps (TP1, TP3 and TP5). The pump icons on the synoptic will blink, indicating the pumps are starting.
- Once they stop blinking and stay green, you can try and turn on the UVH gauges. If the vacuum is not good enough, they will turn back off. Keep trying until they are all on.
- Once the vacuum reaches 1.10^{-4} mbar on the UHV gauges, you can turn on the ion pumps.
- On Cabinet A, check the ion pump voltages. As the pumps start, it should increase from 1000V to 7000 V. If it does not, stop the pumps and wait for a better vacuum.
- One the ion pumps reach 6500 V, switch on the “protect” mode in the vacuum synoptics. This will allow for the vacuum automaton to turn off the ion pumps if the vacuum degrades.
- Once the vacuum is restored in the instrument, it is recommended to switch the vacuum synoptics controls back to “auto”.
- In the vacuum synoptic, click on “FC Start” to pump the pre-amplifier chamber above the multi-collection

10.2 Opening the storage chamber

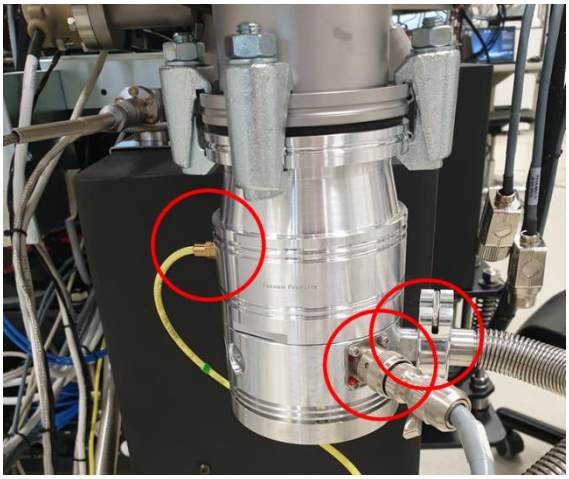
In case of a sample dropped or stuck in the storage chamber, it can be necessary to open it. However, keep in mind that it is a delicate operation and it requires a lifting crane to support the detached airlock+rods part.

- Turn off the HV from the keyboard.
- Vent the “vessel” chamber from the vacuum synoptic.
- Vent the airlock.

- Secure the airlock+rods part with straps attached to the crane. Be careful that the weight should not be supported by the transfer rods, as they are fragile.
- Unscrew the valve control box and the temperature sensor from the the airlock.



- Unplug the pumping tube, the power supply and the nitrogen feed from the airlock pump (but leave the pump in place).

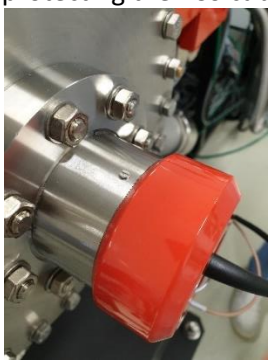


- Unscrew the left side vessel flange supporting the load-lock and the vessel-analysis rod.
- With the help of the crane, slowly move the airlock+turbo+rods part away from the vessel chamber.
- Retrieve your sample. Either put it into place on the carousel or take it out.
- Replace the copper gasket with a new one.
- Carefully reposition the airlock+rods part so that the vessel flange adjusts right against the vessel chamber. Adjust the crane if necessary. It is important for the flange to be at the right height and parallel to the vessel chamber.
- Screw back the flange by tightening them progressively, and alternatively on one side then the other, in order to progressively press the gasket as homogeneously as possible. This will insure the best airtightness possible.
- Re-plug the pumping tube, the power supply and the nitrogen feed to the airlock pump.
- From the vacuum synoptic, launch the pumping procedure (vessel>pump) and wait for the end of the process.
- Re-attach the airlock valve control box and the temperature sensor.
- If necessary, bake the vessel chamber (see chapter 10.6.2.1) to restore an optimum vacuum. Make sure there is no sample left in the vessel before baking.
- Put the HV back on.

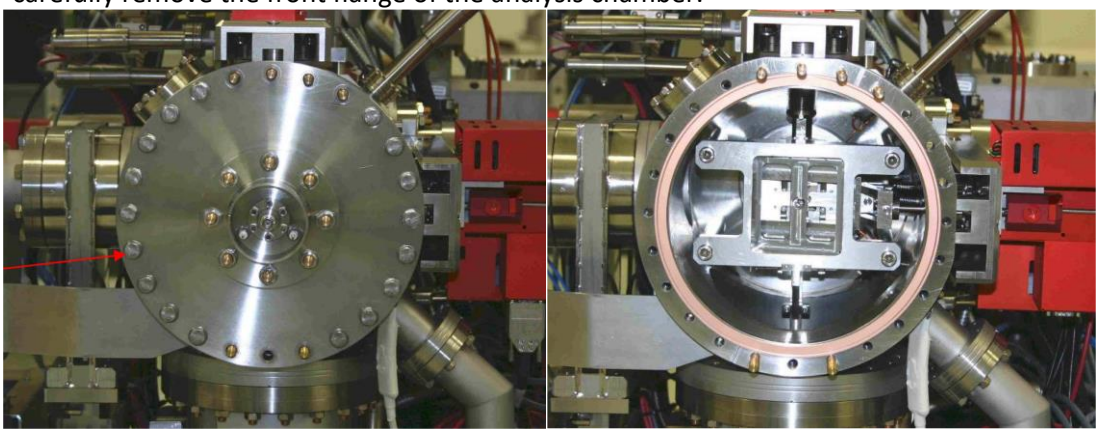
10.3 Opening the analysis chamber

If a sample is stuck or has dropped in the analysis chamber and cannot be removed by the “unload” procedure, it might be necessary to open the analysis chamber. The procedure is described below. Going further and dismounting the stage or the immersion lens are not described as they should preferably be performed by trained service engineers.

- If possible, put the stage in FCo mode (it will move the stage upward, freeing space in the lower part of the chamber)
- Stop the HV on the keyboard.
- Vent the analysis chamber via the vacuum synoptic (referred as “chamber”) and wait for the end of the process.
- Unscrew and remove the red cap protecting the FCo cable



- Disconnect the cables attached to the front of the analysis chamber: P270 (FCo), P485 (FCo repeller).
- Unscrew the front flange. Hold it against the chamber so as to not let it fall. Put clean gloves on and carefully remove the front flange of the analysis chamber.



- With gloves, retrieve your sample. Be careful, as much as possible, not to touch the sample stage and the different wires and springs inside the chamber.
- When done, replace the copper gasket with a new (unused) one and carefully put back the flange into place. Make sure the gasket is properly inserted in its groove.
- Screw all the screws by tightening them progressively, and alternatively on one side then the other, in order to progressively press the gasket as homogeneously as possible. This will insure the best airtightness possible.
- From the vacuum synoptic, launch the pumping procedure (chamber>pump) and wait for the end of the process.
- If necessary, bake the analysis chamber (see chapter 10.6.2.1) to restore an optimum vacuum.
- Put the HV back on.

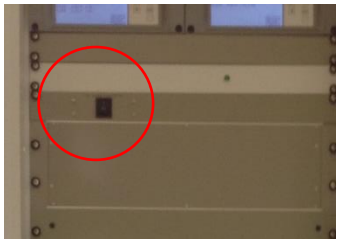
10.4 Opening the multicollection for ExS exchange and EM/FC selection on detector #7

For all detectors, the EM/FC or exit slits changes are automated except for the detector 7 which is fixed. To change the EM/FC or the exit slit of the detector 7, the operator needs to open the multicollection.

10.4.1 Venting the multicollection

- On the keyboard, switch of the HVs 

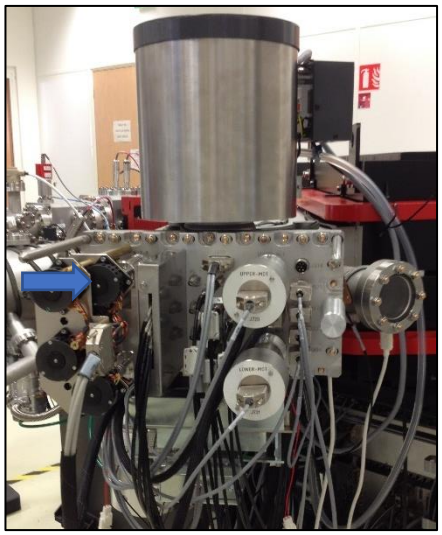
(green light OFF)
- Switch OFF the motorization unit on the front face of the cabinet B



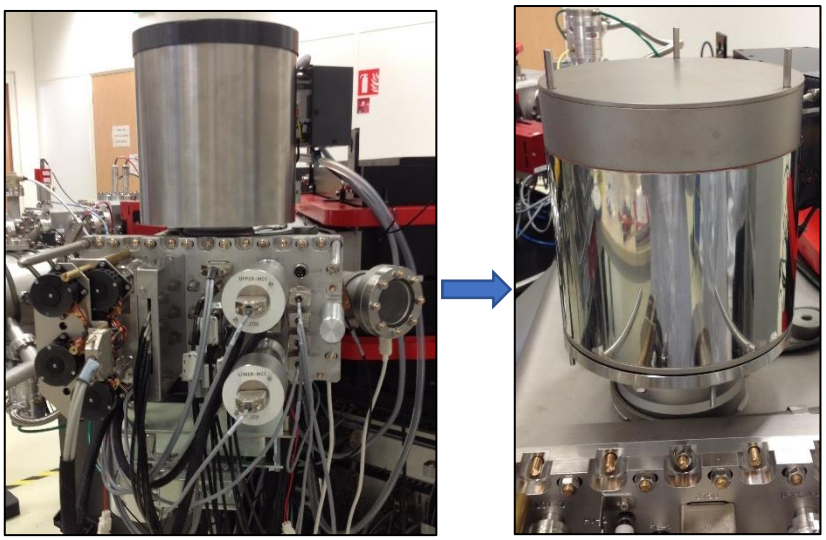
- Fill the dry nitrogen tank or ballon
- On the vacuum synoptic window, vent the multicollection only.
- Check the nitrogen volume in the tank during the ventilation and fill it if necessary

10.4.2 Multicollection opening

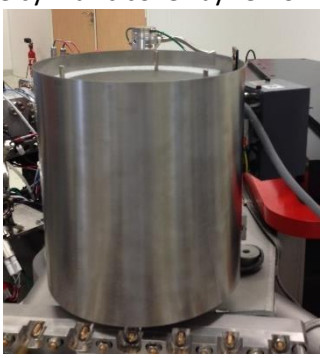
- Remove the motor cover on the front face by unscrewing the three screws on the front face off the multicollection



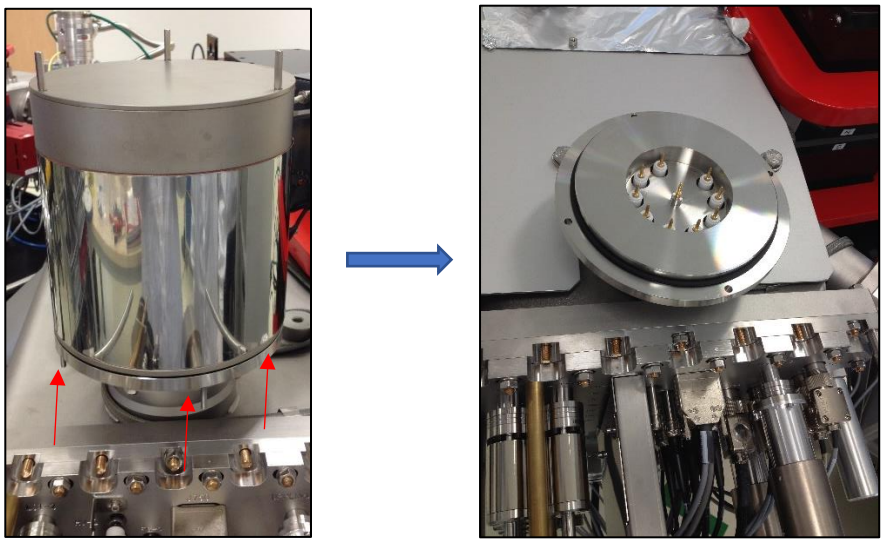
- Remove all the cables on the flange
- Remove the Faraday Cup thermostatic cover



Remark: for this step, first, remove the top of the cover
Secondly, remove the cylindric cover by removing the lateral screws.



- Remove the vacuum tube and the cables
- Unscrew the four small bolts fixing the Faraday cup unit to the multicollection chamber and remove the Faraday unit



- Unscrew the bolts of the Faraday Cup unit flange and remove it slowly
- Inside, unscrew the ceramic from the chamber and release it slowly in the chamber. A screw screwed in the middle of the ceramic is helpful to do this operation.

- Unscrew the multicollection flange (the Figure 441 shows the bolts which needs to be

- unscrewed) from the chamber and leave only one “security” bolt screwed on the top of the flange
- Fix the multicollection table on the chamber by screwing it at the backside of the front face and check the stability (Figure 440)

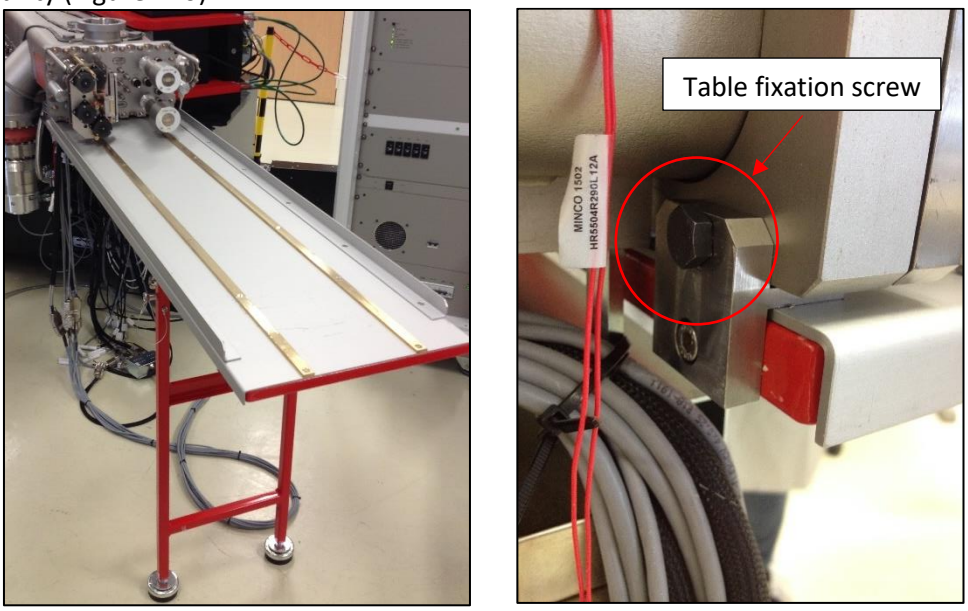


Figure 440: Multicollection extraction table fixation

- Pull the multicollection out by holding the handles to open in (Figure 441).

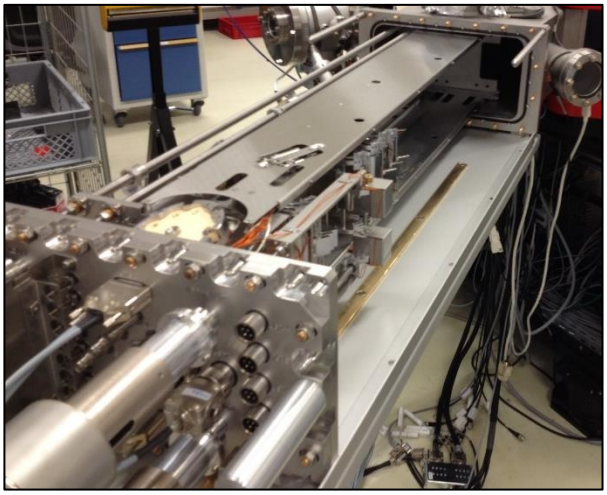


Figure 441: Multicollection out of its chamber

Remark: Pull the chamber cautiously and make sure you do not feel any resistance. There could be an element which has not been unscrewed.

10.4.2.1 EM/FC switch for Detector 7

The detector 7 is held on the trolley which is the closest to the multicollection flange (high radius). To change the EM/FC position, follow this procedure:

- Prepare the dedicated tool:

- Notice the EM and FC position marks on the detector (Figure 442)

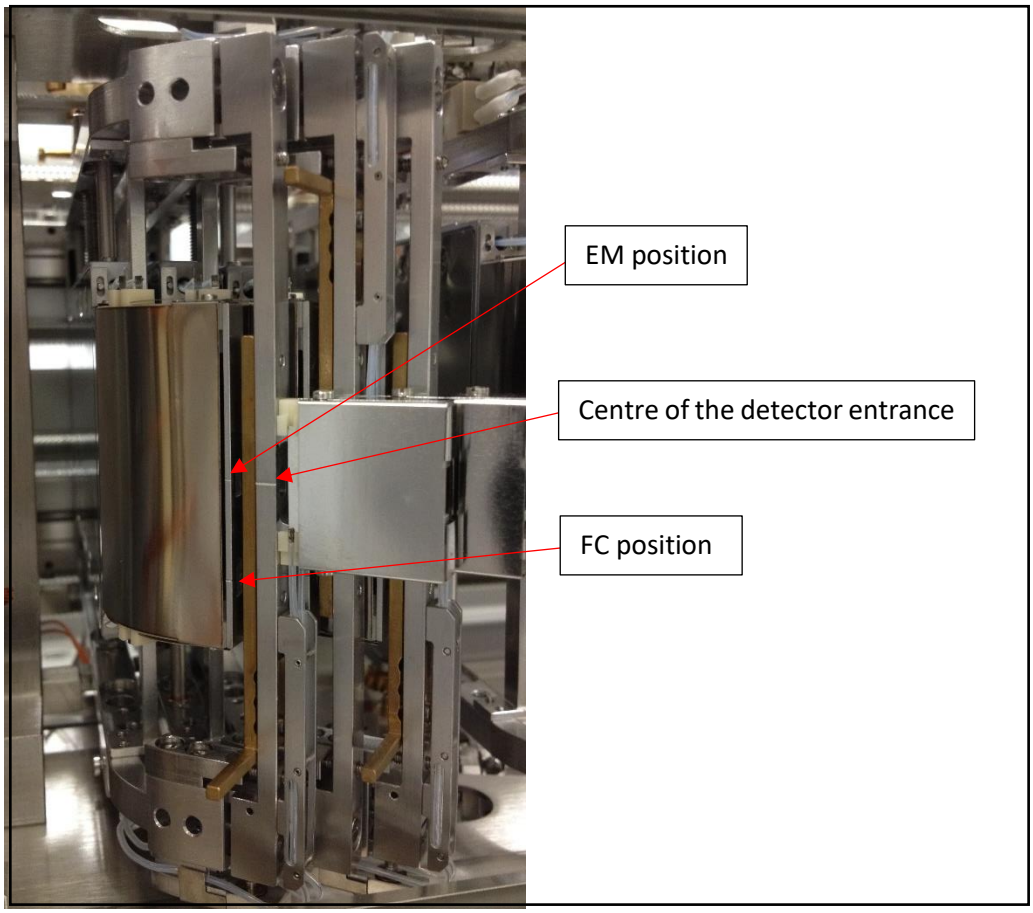


Figure 442: EM/FC Trolley

- On the top of the detector, notice the EM/FC switch screw head (Figure 443)

Figure 443: EM/FC manual switch for the trolley#7

- Move the detector position with the tool by screwing or unscrewing (Figure 444: EM/FC switch tool use for the trolley 7

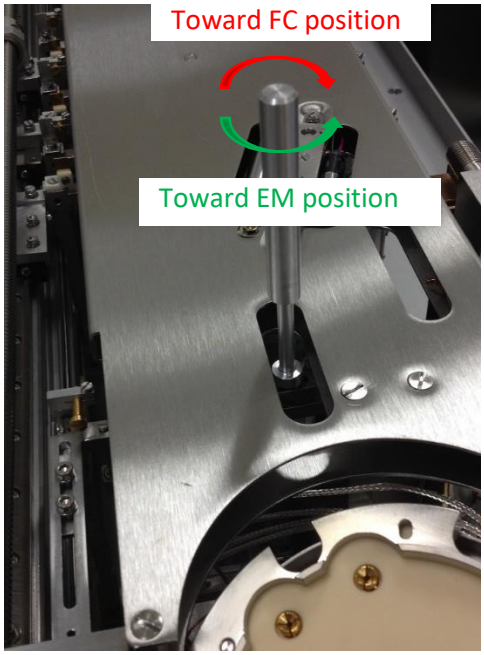


Figure 444: EM/FC switch tool use for the trolley 7

- Align the center of the detector entrance with the EM or FC mark (Figure 442).

10.4.2.2 Exit Slit switch for Detector 7

To change the exit slit at the entrance of the detector, lift the brass stick up or lower it (Figure 445).

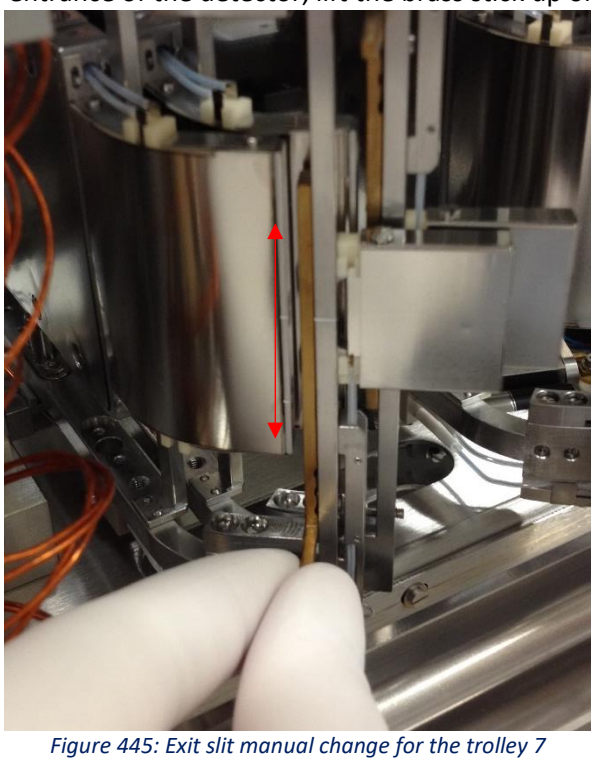


Figure 445: Exit slit manual change for the trolley 7

When moving the brass stick, you can feel notches. Each notch corresponds to an exit slit. Stick in the upper position: the biggest exit slit is used. Slit in the lower position: the smallest exit slit is used.
Reminder of the standard slit sizes on the NS50L:
100 um X 2400 um (lowest position), 70 um X 2400 um and 40 um X 2400 um (upper position).

10.4.3 Re-pumping the multicollection chamber

To restore the vacuum in te multi-collection chamber, on the vacuum synoptic, select “multicollection” and “pump”. Wait for the process to stop.

It is also necessary to repump the pre-amplifier chamber: on the vacuum synoptic, under “FC”, click on “Start” to launch the pumping. Once done, it is recommended to do a “FC Calib” (In Tuning > FC Calib, see chapter 5.2.11 for details)

If the multicollection chamber stayed opened too long, a baking might be necessary to get a vacuum in the 10^{-9} mbar range : refer to chapter 10.6.2.2 for the baking procedure.
Note that it is recommended not to start high precision analyses right away after a multicollection venting, as the instrument usually take a couple hours to stabilize. However, this time can be used to tune the instrument, degas the EMs by raising progressively the count rate and recheck their PHDs, etc...

10.5 Titanium Sublimation

10.5.1 The Titanium Sublimation Pump controller

The Titanium Sublimation Pump (TSP) is operated via the vacuum synoptic and via a controller, located on Cabinet 1 (Figure 447). The Ti sublimator is generally used as a complement to ion pumping for reaching UHV. It is used more frequently at high pressures to help obtaining a good vacuum and is then less frequently used at lower pressures. The sublimation intervals can be defined on the controller.

The TSP is equipped with three Titanium filaments located inside the ion pump below the analysis chamber. A current will periodically go through the selected filament in order to heat it and sublimate Ti onto the walls of the ion pump. Fresh Ti being very reactive, it will form chemical compounds and thus fixate molecules (mostly H_2O, CO, CO_2 and O_2) improving the analysis chamber’s vacuum without rejecting anything outside.
Each filament is consumed progressively until it is time to switch to the next. A degassing procedure is necessary before first use of the filaments (see below.)
A typical sublimation sequence happens as follow:

- a first degassing (increase of pressure),
- a pumping period (decrease of pressure)
- then a re-increase of pressure (degassing all around due to the heat). It is time to stop the sublimation.

This process typically last less than a minute. The Ti layer will now pump until it is fully reacted (this can last several days under a $P < 5.10^{-10}$ mbar, requiring a less frequent sublimation).

From the vacuum synoptic, the user can turn ON or OFF the sublimation routine (Figure 446). Note that this can only be done in Manual mode and must therefore be handled with caution by an experienced user.

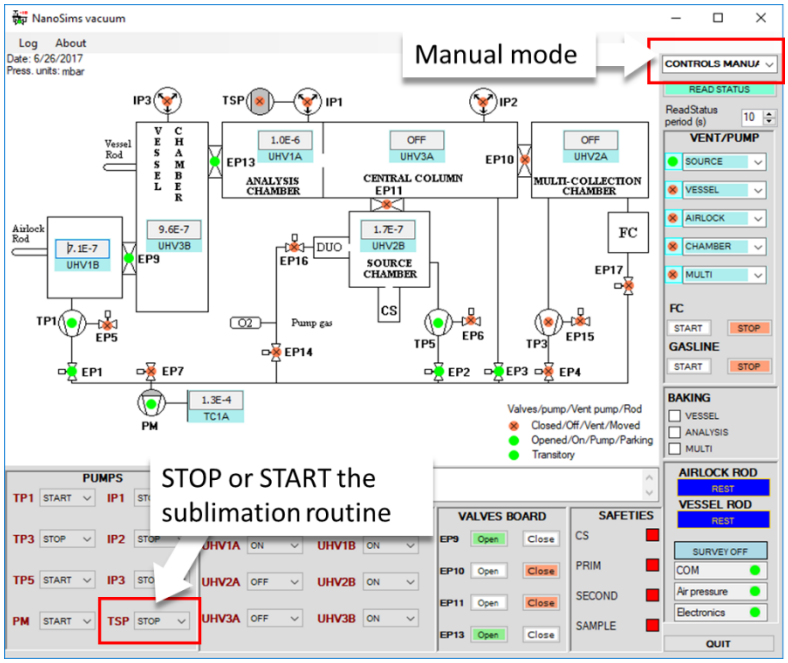


Figure 446: Stop or Start the TSP from the vacuum synopic

The TPS controller allows the user (Figure 447):

- to control the settings of the automatic sublimation routine (periodicity, duration and intensity)
- select one of the three filaments available and display the state of the filaments.
- Launch/stop manually a sublimation.

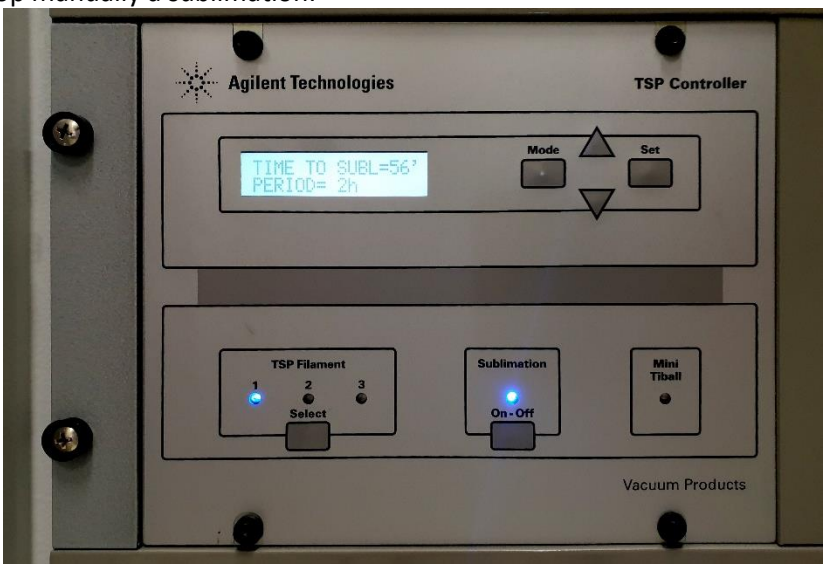


Figure 447: Titanium Sublimation Pump controller

To **adjust the settings of the automatic routine**:

- Click on “Set”. The ID of the filament currently selected will appear on the TSP controller screen

- Click on the up/down arrows to select the parameter to adjust. There are three parameters

a. SUBL. PERIOD: interval between two sublimations. When the high vacuum is established, a period of 32 hours is recommended.
b. SUBL. CURRENT: current intensity during the sublimation. Usually set at 42 A.
c. SUBL. TIME: duration of a sublimation. Set at 1 min.

- To select the parameter to adjust click on “Set”. You will notice that the arrow symbol on the screen moves from the right side of the first line, to the left side of the second line, indicating the value of the selected parameter can now be adjusted.

- Then click on the up/down arrows to adjust the parameter.
- Click on set to validate the change and go back to the upper menu.

Note that the “Mode” should always be set in “Manual”. This Automatic/ Manual mode option of the TSP controller is distinct from what we call automatic routine and manual sublimation. The Manual mode means that the TPS controller is controlled by the Vacuum automaton (in particular, *it will prevent sublimation during analyses*). In Automatic Mode, everything is controlled by the TPS controller and it will not allow the vacuum automaton overrides.

Filaments: The controller also shows the state of the 3 filaments in the TSP:

- LED ON: selected filament.
- LED OFF: filament not selected
- LED blinking: broken filament. Needs replacement.

For a good use of the filaments, it is recommended to regularly switch the selected filament (every month or so), as unused filaments are getting clogged by the other filament’s regular Ti sublimation. Each filament will last several months. Replacement of the filament set (see below) will be necessary every year or so.

10.5.2 Maintaining an optimum vacuum, manual sublimation

To maintain an optimum vacuum in the analysis chamber, it is recommended to use the Titanium Sublimation Pump (TSP). When the vacuum is established (< $.10^{-9}$ mbar in the analysis chamber), the sublimation is set to trigger every 32 hours and lasts one minute. If for any reason, the vacuum in the analysis chamber is not optimum, it is possible to do a manual sublimation. Usually, a single sublimation is enough to improve the vacuum.

This manual sublimation can be useful in particular when introducing a degassing sample in the analysis chamber (sample not perfectly dried, or embedded in not-cured resin) or when an optimum vacuum is required (analysis of atmospheric elements).

To **manually start a sublimation**, go to the TSP controller, located on Cabinet 1, and click on “Sublimation On-Off”. Use the arrows to display “Start ? Yes” on the controller screen and click on “set” again to confirm. You can always click on “Sublimation On-Off” to interrupt the sublimation.

10.5.3 Restoring the vacuum

After a maintenance operation requiring to open the instrument, or a power failure that degraded the vacuum in the instrument, it can be useful to adjust the sublimation program to set a more frequent sublimation. If the pressure in the analysis chamber is between 10^{-6} mbar and 5.10^{-9} mbar, it is recommended to set the sublimation between two-hour and several hours interval, until a good vacuum is restored in the analysis chamber (< $.10^{-9}$ mbar). Then a sublimation every 32 hours should be enough to keep a good vacuum in the chamber

10.5.4 Changing the filaments

Three filaments are attached to the TSP cartridge but only one is used at a time, when one fails the next one starts automatically. When all the filaments are broken, it is necessary to change them.
CAUTION 1: Titanium flakes are flammable and may spontaneously ignite when exposed to air. Do not clean flakes with a vacuum cleaner or leave the flakes in contact with any flammable materials. Flakes should be stored in a metal container until they can be disposed of.
CAUTION 2: Titanium flakes easily fly around during venting or re-pumping. They can relocalize near a HV feedthrough or polarized electrode, inducing electrical arcs. Hence a slow venting with a low nitrogen flow is always recommended.

- Turn off the HV on the keyboard. Vent the Analysis Chamber through the Vacuum Synoptic window.
- From the back of Cabinet A, switch off the TSP controller via the main power button located at the back (Figure 448):

Figure 448: back of the TSP controller

- Remove the NanoSIMS black cover panel (Figure 449, A) in order to see underneath the Analysis chamber ion pump (B) and unplug the TSP cartridge supply cable.

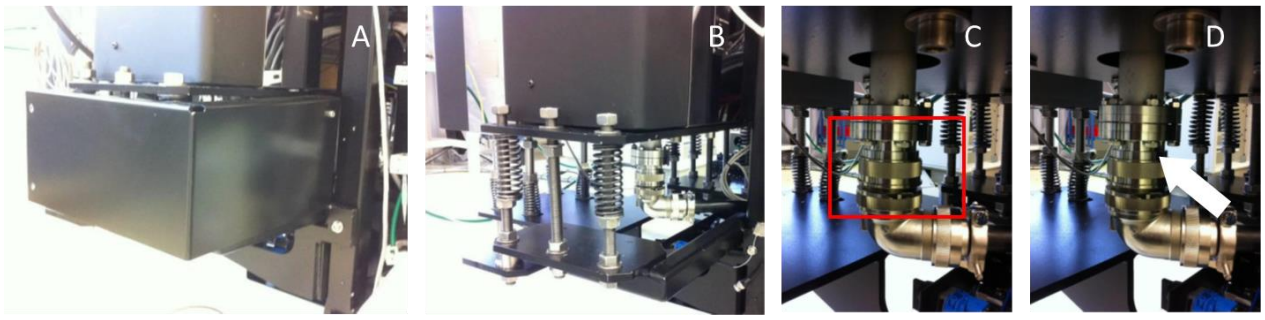


Figure 449: unmounting the TSP cartridge

- Unscrew the cartridge nuts and bolts (Figure 449, C, then D). Hold the cartridge during this process in order to avoid the cartridge flange falling down.
- Lower the cartridge very carefully until it is completely removed (Figure 450).

Figure 450: Ti filament cartridge

- Loosen the golden screws (Figure 451, A) with the 8-32 Allen key provided in the Agilent kit and use the special Spline key and loosen the screws that pinch the filaments (B).

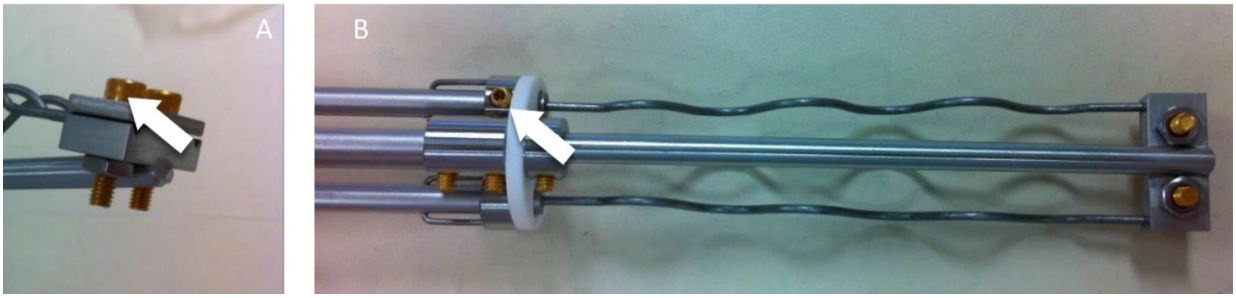


Figure 451: Unscrewing the Ti filaments from the cartridge

- Remove the filaments and replace them by new ones. Re-tightens the golden screws. Be careful that the three filaments are spaced properly and do not touch.
- Use a new copper gasket CF35 and put it on the cartridge feedthrough.
- Carefully insert the cartridge into the ion pump and orientate it in order to get the TSP power cable oriented as you want.
- Tighten the nuts and bolts.
- Pump inside the Analysis chamber
- Plug the TSP power cable back into the connector.

10.5.5 Degassing a new set of filaments

Once the new set of filaments is in place, it is necessary to degas them before their first use. To degas the filaments, follow the procedure below:

- Select filament 1
- Set the sublimation settings as follow:
    a. Sublimation period: 30 min
    b. Sublimation time: 1 min
    c. Sublimation current: 30 A
- Click on Sublimation On-Off and start a sublimation
- Check the pressure in the analysis chamber (gauge UVH1A). When the pressure increases beyond 2.10^{-5} mbar, stop the sublimation (click on sublimation On-Off), and let it decrease.
- When the pressure is below 10^{-7} mbar, start a new sublimation.
- Repeat this process until it is possible to run a full sublimation (1 min) while keeping the pressure below 2.10^{-5}.
- When this is done, repeat the process for filaments 2 and 3.
- Once all 3 filaments have been degassed at 30 A, put the sublimation current at 35 A and repeat the degassing process for all three filaments at 35 A.
- Then repeat the operation for all three filaments at a current of 40 A
- And finally, repeat the operation for all three filaments at a current of 45 A.

When the degassing process has been achieved for the three filaments, put back normal settings (32 hours period, 1 min sublimation time, 42 A)

10.6 Instrument baking

10.6.1 Introduction

The NanoSIMS is an instrument working under high vacuum. The analysis chamber vacuum must be kept as low as possible for obvious sample contamination reasons (especially for H, C, N, O analysis and all hydride, carbide, oxide, nitride peaks creating mass interference and risks of detection limit degradation). After a proper baking allowing the degassing of the vessel and other internal surfaces, the continuous ion pumping and regular titanium sublimation pumping should maintain the vacuum below 5 E-10 mbar level in the analysis chamber.

But vacuum can be degraded easily, e.g. (and not limited to!):

- if the user introduces degassing samples (non-UHV-compatible embedding resin, polymers-lubricant-oils, hydrated samples, porous samples),
- if the user touches the sample holder or samples with skin or dirty gloves/tools,
- if the user does not respect transfer vacuum thresholds,
- if there is a leak or degassing part left inside or a Viton gasket is used,
- After opening the analysis chamber for maintenance (e.g. cleaning of the immersion lens).

In such cases a baking is necessary in order to restore real UHV (after solving the problem if any).
Without sample inside and without ion sources off the NanoSIMS should stay typically under 5 E -10 mbars. Applying baking, degassing and Ti sublimation, and introducing only degassed, clean sample holders from a low vacuum storage chamber it is possible to stay in the low E-10 or enter the E-11 mbar range.

For baking, the CAMECA NanoSIMS 50L is equipped with heater bands and heating resistors which can be independently activated, to bake certain parts of the instrument: (1) the vessel storage chamber, (2) the analysis chamber and the central column, and (3) the multicollection. This operation can improve the vacuum in the instrument and is often necessary when parts of the instruments have been vented.

10.6.2 Preparation

Before starting baking, the electronics must be turned off, unless only the multicollection chamber is being baked.

10.6.2.1 Baking the vessel, analysis and central chambers

Baking of the main parts of the instrument for 12 hours is mandatory after every maintenance that required to open the instrument. It is also recommended to bake the instrument for a few hours if the vacuum does not seem to go down to expected value (around 5.10^{-10} mbar in the analysis chamber when empty). If the instrument has been left opened for over a day, it might be useful to do a 48 hour baking in order to restore optimum vacuum conditions (< 5.10^{-10} mbar). Make sure there is no sample inside the vessel or the analysis chamber during the baking of these parts.
The load-lock is not bakeable.

- Stop the electronic as described in 10.1.1.1

- In the vacuum synoptic window, in the baking options, check ANALYSIS (analysis chamber + central column) and/or VESSEL (vessel chamber) and/or MULTI (multicollection) accordingly (Figure 452):

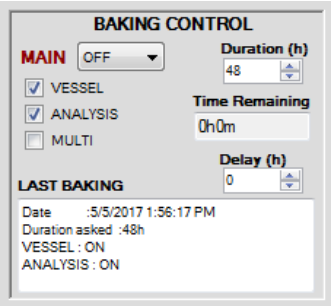


Figure 452: bottom left part of the Vacuum synopic

- Select a baking time in “Duration” and, a start delay if needed in the “Delay”.
- Start the baking by selecting ON in the MAIN list box

10.6.2.2 Baking the multicollection chamber only

Baking of the multicollection chamber for a dozen hours is mandatory after any maintenance that required opening the multicollection chamber.
A good vacuum in the multicollection will reduce ion **scattering** and contribute to a low **background noise**. As seen before carbon contamination on the last dynode of EMs is a degrading factor to pulse counting **stability**. Hence maintaining the multicollection vacuum below 10^{-8} mbar is important.

- Switch off the HVs on the keyboard. 

- In the vacuum synoptic window, in the baking options, check the MULTI box and uncheck the ANALYSIS and VESSEL boxes:

Figure 453: baking control section of the Vacuum synoptic.

- Select a baking time in “Duration” and add a delay if necessary.
- Start the baking by selecting ON in the MAIN list box

Note: for more information on the baking control, refer to 5.10.1

10.6.3 Restarting the instrument after baking

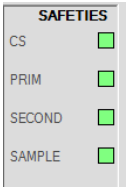
10.6.3.1 After baking the vessel, analysis and central chambers

- Let the instrument cool down approximately 12 hours after the end of the baking.
- Turn ON the electronic – see 10.1.1.2
- Put the HV back on and apply the Presets to activate the HV in the instrument (see 9.1.4)

The instrument is now ready to be used.
Note that it is recommended not to start high precision analyses right away, as the instrument usually take a couple hours to stabilize. However, this time can be used to tune the instrument.

10.6.3.2 After baking the multicollection only

- Let the instrument cool down approximately 12 hours after the end of the baking.
- In the Vacuum window, check that all the safeties are ON (green)



- Switch the HVs ON 

(green light is on) on the keyboard.
- Apply the Presets to send back the HV values to the instrument (see 9.1.4)
- The instrument is now ready to be used.

Note that it is recommended not to start high precision analyses right away, as the instrument usually take a couple hours to stabilize. However, this time can be used to tune the instrument, degas the EMs by raising progressively the count rate and recheck their PHDs, etc...

10.7 Cs+ Source

10.7.1 Source trouble-shooting

Low current: If the current read in FCp is too low but stable, start by gently increase the reservoir by 0.05 mA increments. The current should increase. Wait a few minutes between increments to let the source stabilize. If the reservoir value reaches 0.5 mA and the current is still too low, it is time to replace the Cs carbonate.

Unstable current: If the current is unstable, it is possible that some Cs deposit is obstructing the source.
It is possible to try to clean it without opening the instrument. To do so, put the Cs source in Standby mode (see chapter 5.4.2) overnight, with HV ON. This will put the Reservoir heating to zero, while keeping the Ionizer heated and extracting the Cs+ ions.

High leak current (and unstable current): the extractor has probably been contaminated by too much cesium deposition, due to poor vacuum conditions or a repeated switch between O and Cs sources, causing the formation of some Cs hydroxide or other compounds. When the positive source HV is applied to extract Cs+ ions from the source, it can extract electrons emitted from the Cs-coated extractor edges or particles toward the source which can be seen as a (false) ion emission current. Often it is necessary to open and clean the extractor.
Such problem can also occur when heating the reservoir too much without heating the ionizer enough: cesium is then evaporated but not ionized. Similarly, it can happen also when running the source without polarizing it with HV (= without extracting the Cs ions).
Finally, this can happen when heating too fast a newly-mounted ionizer: a burst of degassing could coat the extractor.

No heating current (reservoir or ionizer): A filament in the source may have broken. Heat or incorrect mounting of a filament may have cause it to deform, causing to touch the source body.
To test this, stop the source. Let it cool. Disconnect cables and test with an ohmmeter:

- the good isolation between each filament pin of the feedthrough and the source body. See Figure 454: source filament feedthrough.: pins 1 and 2 are connected to the ionizer filament. Pins 3 and 4 are connected to the reservoir filament.
It should be several M Ω. If it shows 0 Ω, the filament is in contact with the source body.

- the continuity of each filament. The resistance should be a few Ω. If it is at zero, there is a short-circuit, the filament is touching the source body. If it shows an infinite resistance, the filament is broken.

Note that if the ohmmeter doesn't show anomalies but there is still current in the source, then the problem probably comes from the source chassis (possibly a broken fuse).

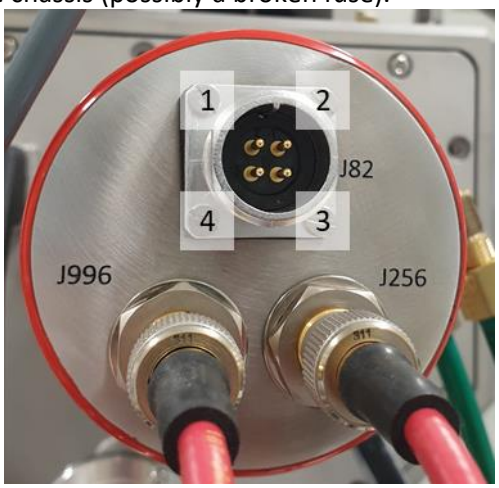


Figure 454: source filament feedthrough.

10.7.2 Source disassembly

10.7.2.1 Removal of Wien Filter and Cs+ source from the chamber

When a maintenance is needed on the Cs+ source, it is important to turn it off and wait for 45 minutes before venting it. Then, follow this procedure:

- Turn off the HV via the keyboard.
- On the vacuum synoptic window, vent the source only, by clicking on "source" in the menu on the right, and select "vent" (Figure 455)

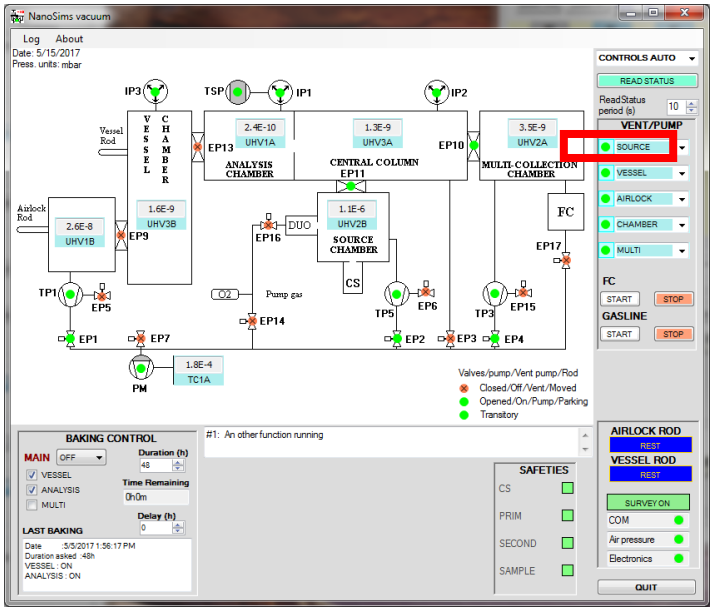


Figure 455: vacuum synoptic window

- Put the magnetic field to a low value (200 gauss or below)
- Close the water circuit for the physical part (Figure 456 and cooling circuit of figure 2.3.6)

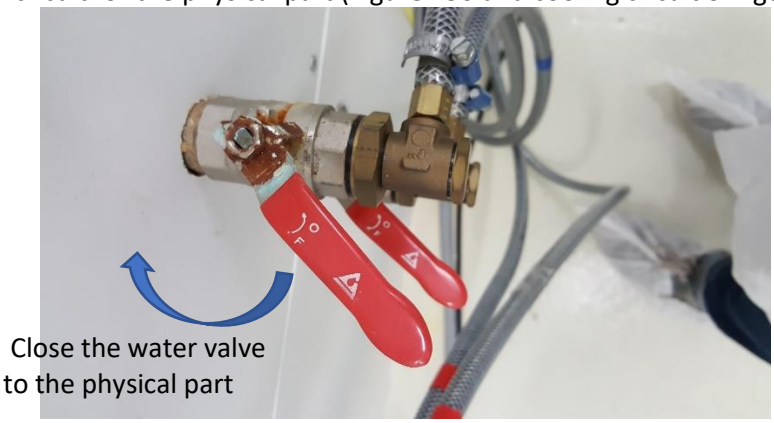


Figure 456: valve for the water circuit.

- Purge the water remaining in the pipes via the purge valve on the fluid panel at the rear end of the instrument (Figure 457).

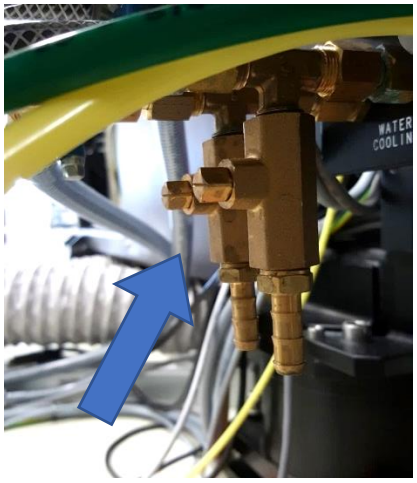
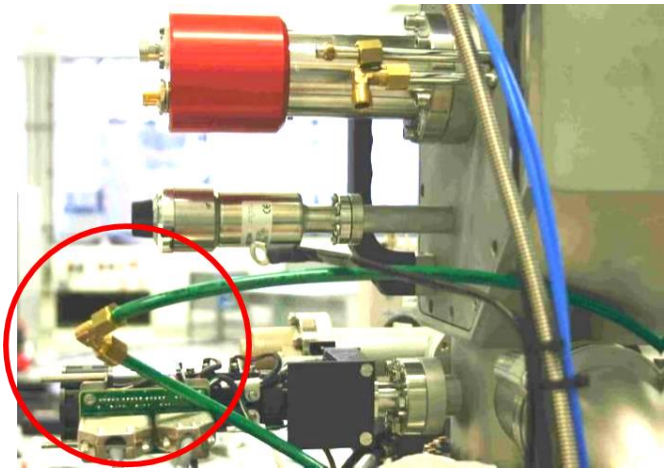


Figure 457: water purge valves

- Disconnect the Cs source water circuit from the rest of the water circuit by connecting the two water plastic tubes from the Cs source together and the two tubes from the rest of the instrument together.



- Unplug all cables attached to the Wien filter and unscrew the Wien filter chiller (Figure 458, a).

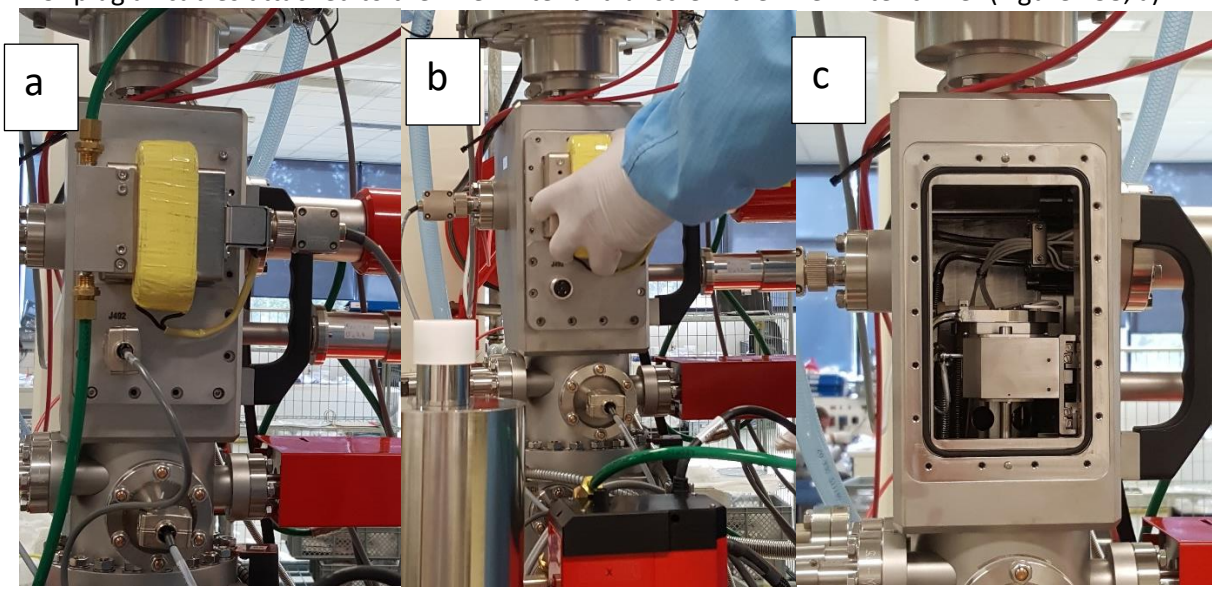


Figure 458: unmounting the Wien filter from the source chamber

- Unplug all cables connected to the removable part of the Cs+ source chamber (Figure 459).

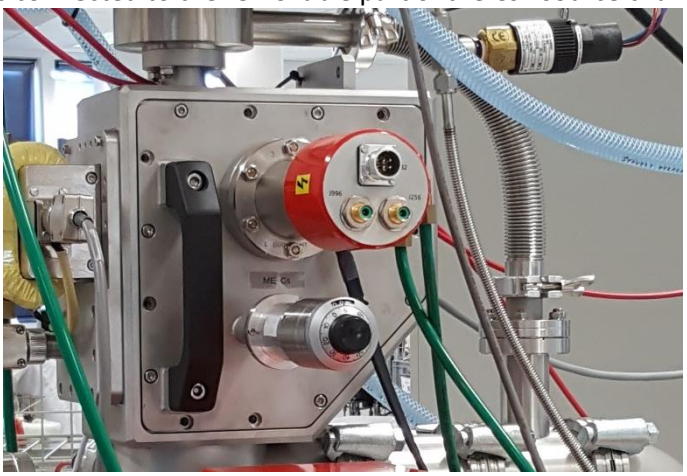


Figure 459: Source chamber side panel. All cables have been disconnected and it can now be unscrewed and removed

- Turn the gonio clockwise till the end (position 1.0)
- Unscrew and remove the Wien filter (Figure 458 (b,c)).
- Move the source to its exit position by turning the gonio anti-clockwise till the 3.0 mark.
- Remove the Cs+ assembly by unscrewing it.

CAUTION: Always use clean gloves/tools to touch inner parts ! Rapidly cover opened flanges with clean aluminum paper to limit entrance of dust, particles or moisture.

10.7.2.2 Cs+ source cage disassembly

To access the extractor, the filaments and the Cs+ source, position the source like on Figure 460:

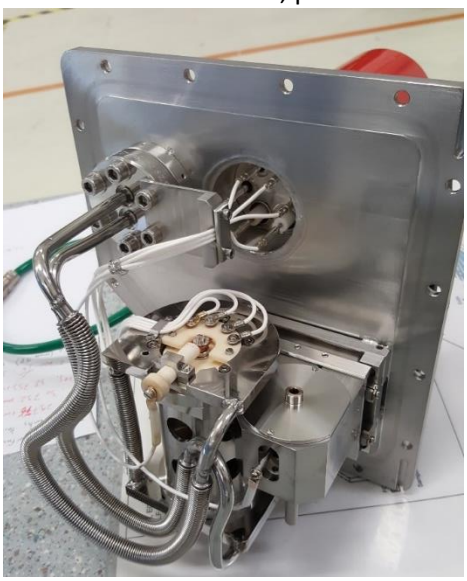


Figure 460: Cesium source block, detached from the primary column

The Cs+ source is included in a cage we need to separate it from the rest of the mechanism:

- Remove the HV ceramic locker by unscrewing the two screws: Figure 461a).
- Unscrew the central HV screw and release the HV ceramic: Figure 461b)

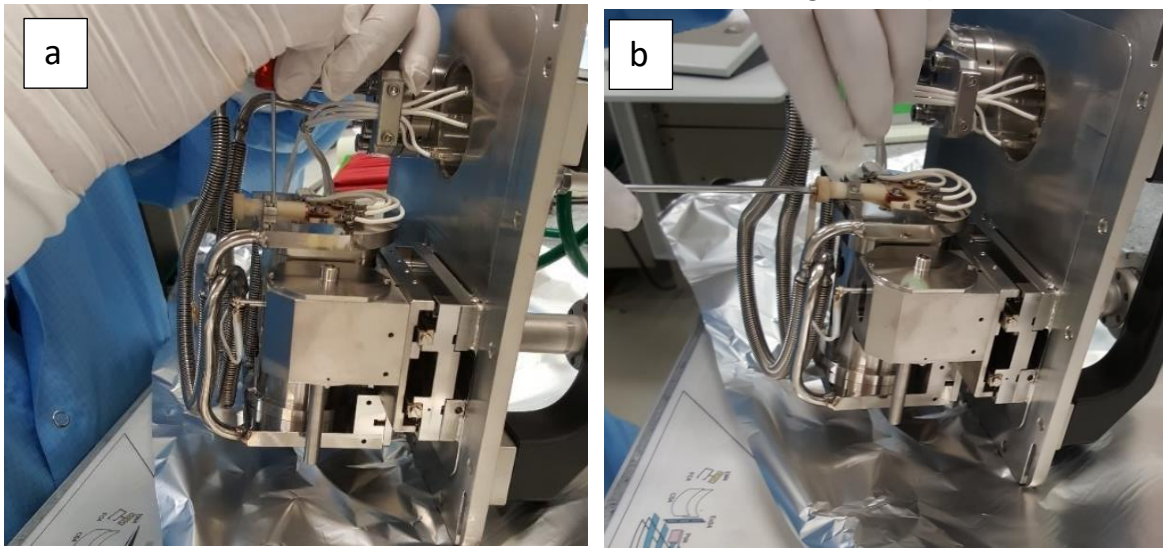


Figure 461: unscrewing the HV the ceramics

- Remove ionizer & reservoir current cable locker by unscrewing the two screw: Figure 462a.
- Remove ionizer & reservoir current ceramic screws, remove **carefully** the ceramic: Figure 462b.

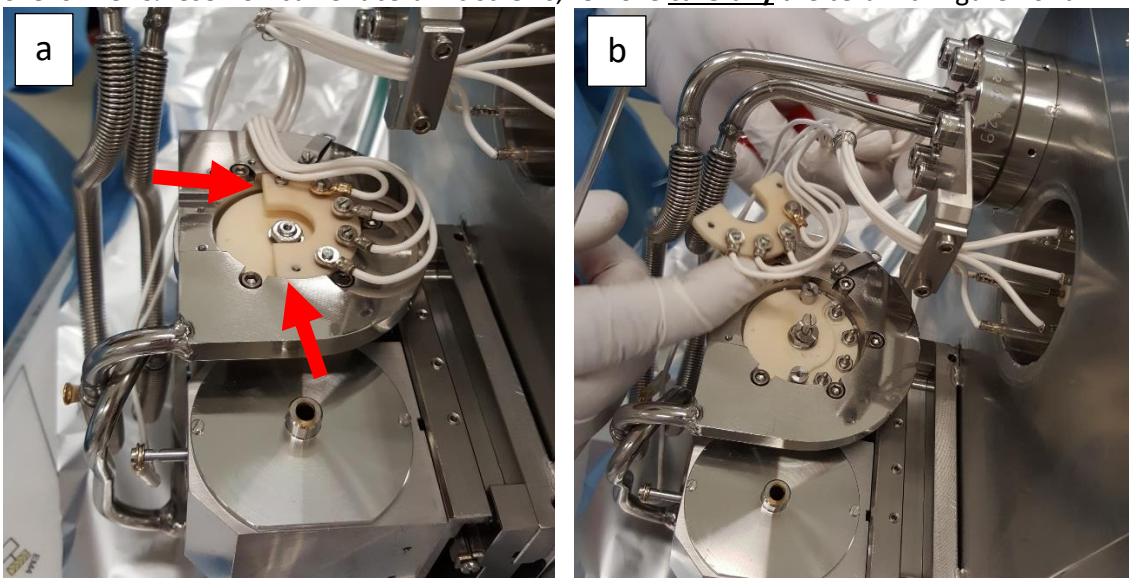


Figure 462: unscrewing the ionizer and reservoir current ceramics.

- Unscrew the five fixation screws on the top of the cage.

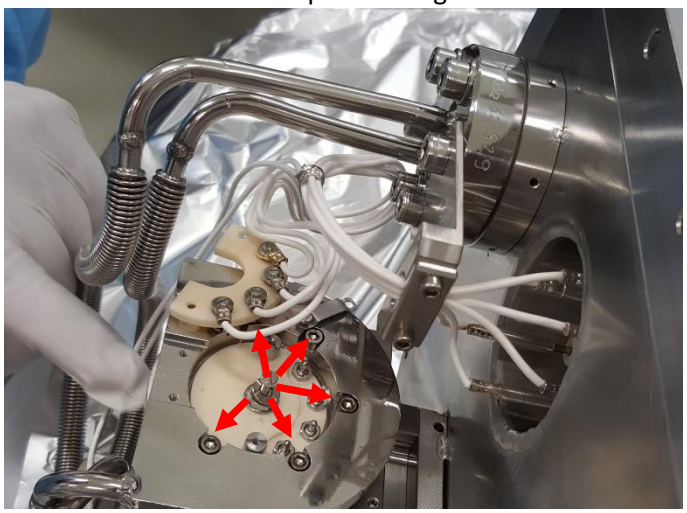


Figure 463: fixation screws on top of the Cs source cage

- Unscrew the four fixation screws under the cage (Figure 464).

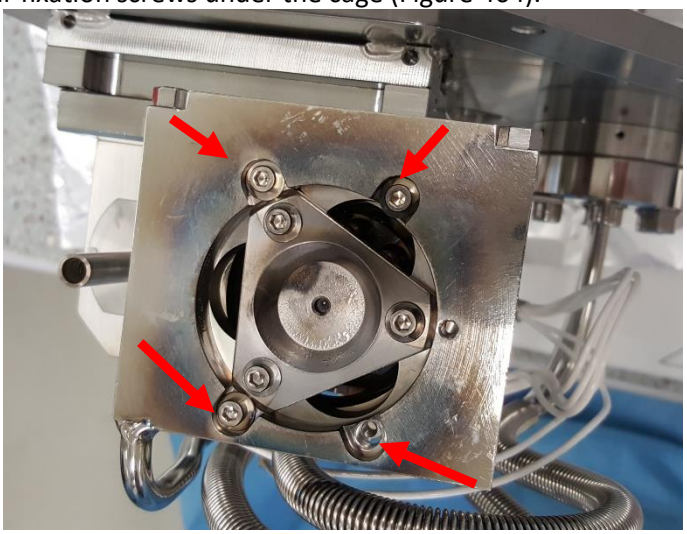


Figure 464: bottom of the fixation cage

- Remove the cage from the rest of the mechanism (Figure 465).

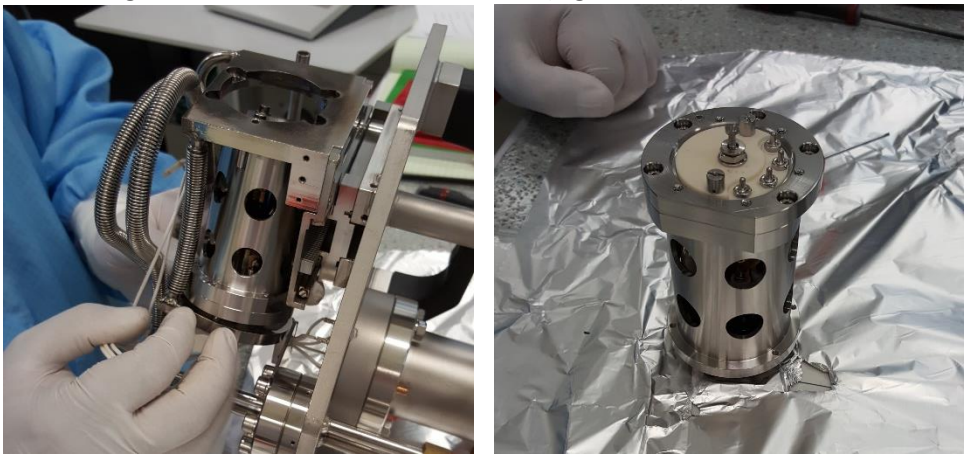


Figure 465: cage separated from the rest of the source mechanism

CAUTION: Always use clean gloves/tools to touch inner parts ! Rapidly cover opened flanges with clean aluminum paper to limit entrance of dust, particles or moisture. Do not touch ceramics with metallic tools with the risk of creating leakage paths. Cleaning ceramics can be done by sanding them, followed by alcool/acetone cleaning and drying.

10.7.3 Extractor cleaning

When the primary Cs+ beam is not stable or the image shows vibration, it is most of time because the extractor is dirty. The operator can clean it easily by following this procedure:

- Unscrew the three fixation screws and remove the extractor.

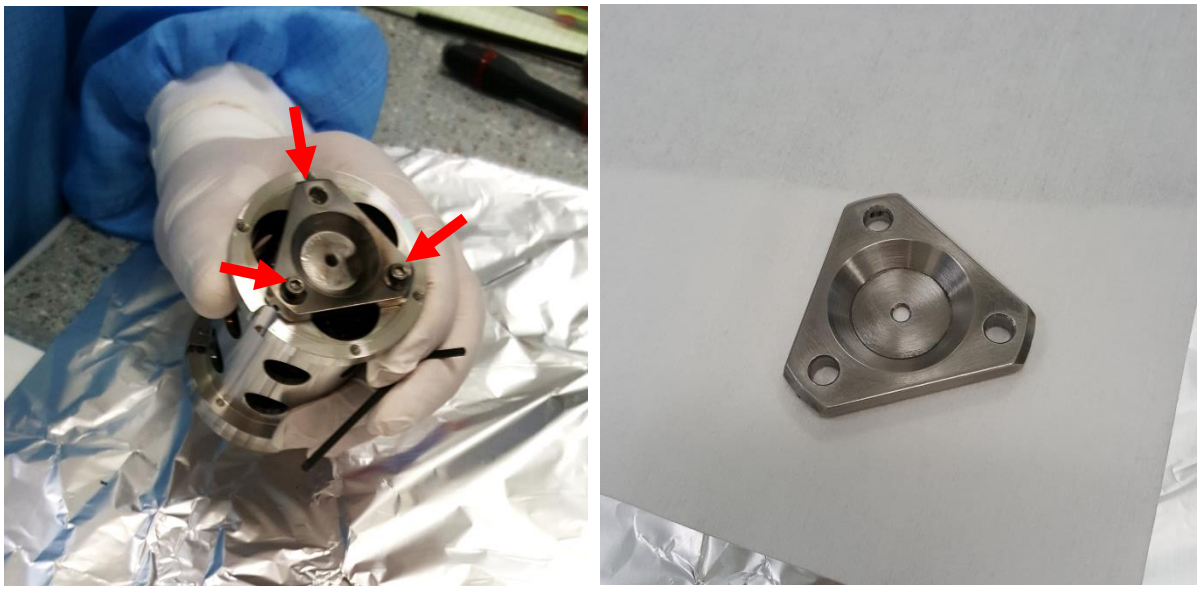


Figure 466: separation of the extractor for cleaning

- Clean the extractor with alcohol and a clean tissue.
- Place the source cage on its support.
- Position the extractor without screwing it (Figure 467 a).

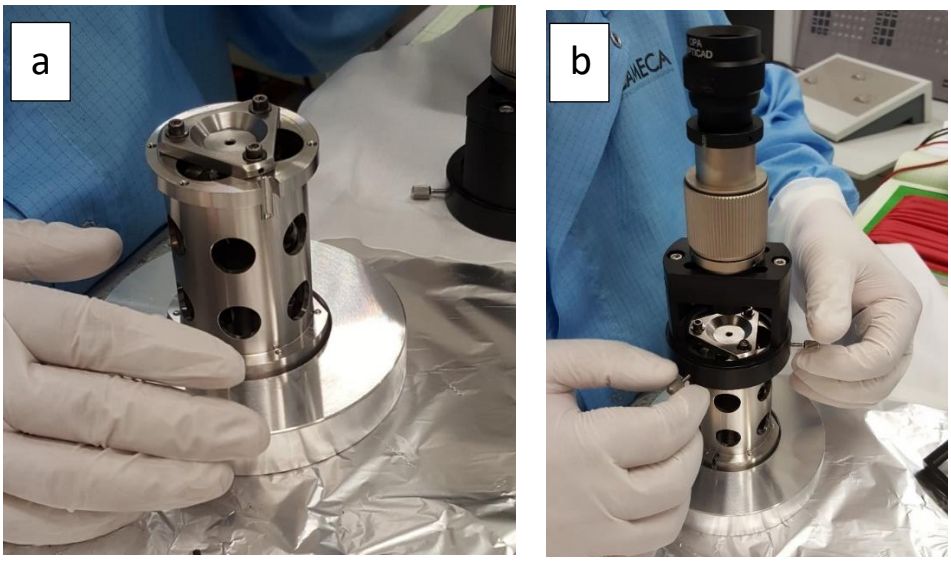


Figure 467: centering of the extractor on the Cs source

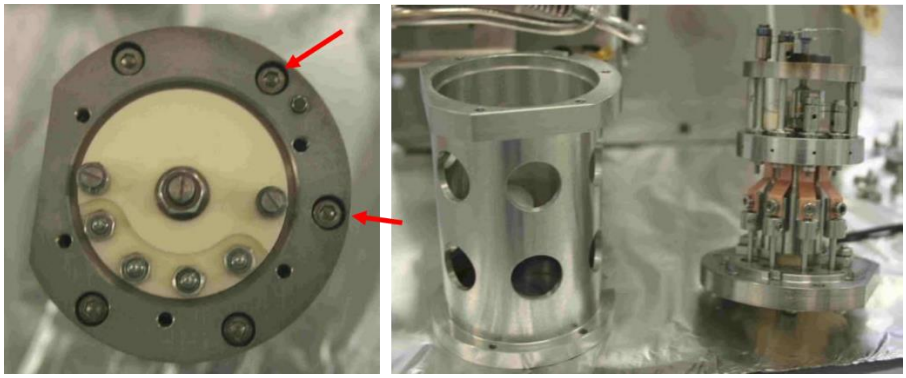
- Place the centering microscope on the top of the rest (Figure 467, b).
- By looking on the lens, focus the hole of the Cs+ ionizer.
- Use the three screws to center the black-cross (visible in the microscope optic) on the hole of the ionizer).
- Screw the three fixation screws strongly.
- The extractor is now centered.

10.7.4 Cs+ Source replacement

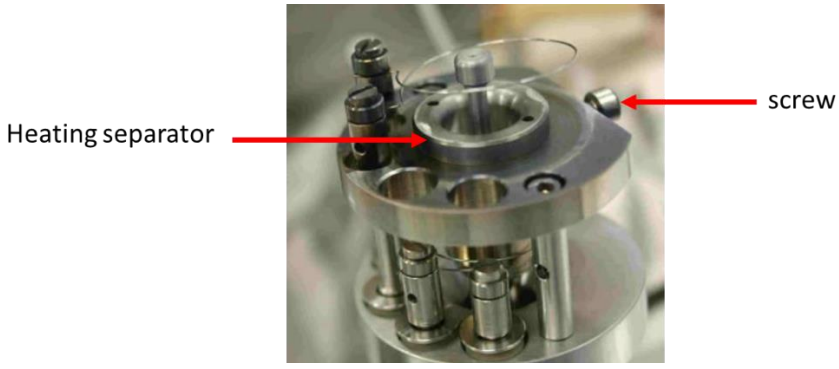
After some time using the Cs^+ source (typically 3-4 months), the primary current becomes too low and cannot be restored by increasing the reservoir current. The source then probably needs to be replaced. Note that it can be useful to place the new source for a night or more in the airlock to help it degas in the vacuum, prior to this operation. This will shorten the degassing procedure later.
To replace the Cs^+ source, follow the procedure below:

- Remove the Cs+ cage (see 10.7.2.2)
- Unscrew and remove the extractor

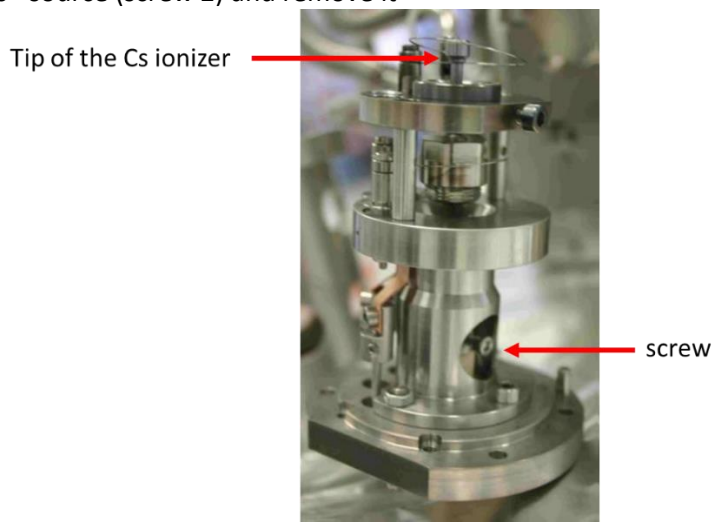
- Turn the source upside-down with caution and unscrew the five fixation-screw on the top of the cage



- Separate the source support and the cylinder of the cage and put the source back to its upward position.
- Release the heating separator ring by unscrewing the screw (screw 1) on the side and pull out the separator



- Unscrew the Cs+ source (screw 2) and remove it



- Put the new Cs+ source in without tightening the screw.
- Place the positioning tool allowing to give the good distance to the source. Carefully turn the source upside-down and ensure the top of the ionizer is touching the top of the gauge tool. Tighten the screw (screw 2) to old the source in place.

- Put the source back in its upward position and remove the gauge tool.
- Put back the heating separator plate and screw it, but not too much.

- If changing the filaments, see chapter 10.7.6. Otherwise, continue below:

- Attach the source's cage to the source's body and place the source cage on its support.
- Position the extractor without screwing it (Figure 467 a).

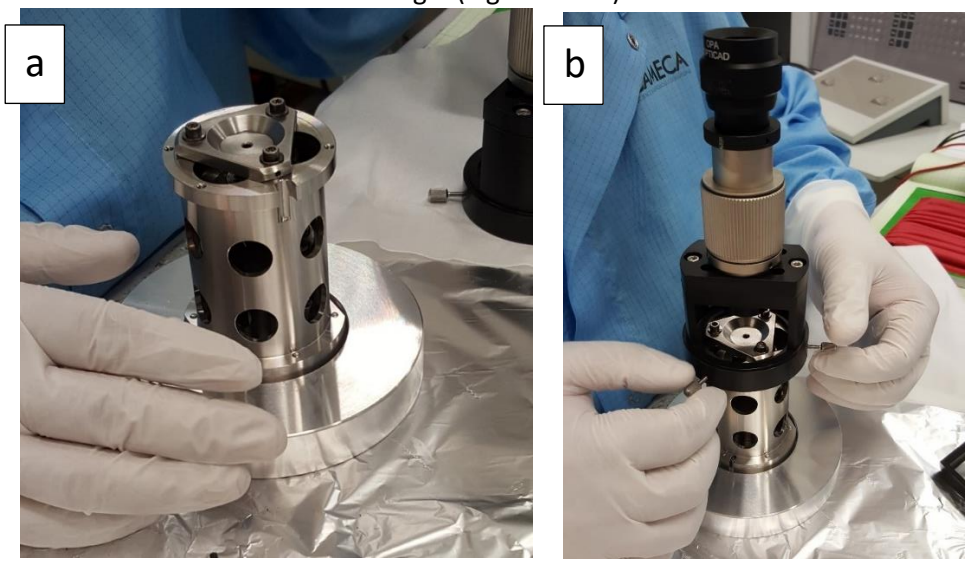


Figure 468: centering of the extractor on the Cs source

- Place the centering microscope on the top of the rest (Figure 467, b).
- By looking through the lens, adjust the focus on the hole of the Cs+ ionizer.

- Use the three screws to center the black-cross (visible in the microscope optic) on the hole of the ionizer).
- Screw the three fixation screws strongly.
- The extractor is now centered.

10.7.5 Cs+ outgassing and restarting

Note that in order to help the outgassing of a new source, it can be useful to put the new source in the airlock for a night, with the airlock light at 75%, before installing it and proceeding to the following sequence.

10.7.5.1 Outgassing of a new source

After remounting the source and once the vacuum has been restored in the Source chamber, it is important to properly outgas the source before use:

- You must be in “Cs” mode in the Source window, and the goniometer must be set in Cs position. Put the HV ON on the keyboard so that you can read FCp.
- In the vacuum synoptic put the control in “manual” mode (this will temporarily disable the security in order not to stop the source during the degassing process).
- Open the vacuum recorder to monitor the vacuum in the source chamber (display UVH2B).
- In the Source window, put the source HV at 500 V.
- Increase the ionizer current up to 0.1 mA. Pressure in the source chamber will increase. Wait for it to stabilize and decrease again.
- Progressively increase the ionizer current by steps of 0.1 mA until you reach 1.6 mA while keeping an eye on the pressure in the source chamber. If the pressure reaches 1.10^{-5} mbar, wait until the pressure decreases again before incrementing the ionizer current.
- Decrease the ionizer current to 1.0 mA and wait for the pressure to go down to 1.10^{-6} mbar.
- Increase the source HV by increments of 1000 V up to 8000 V while keeping an eye on the pressure in the source chamber. If the pressure reaches 1.10^{-5} mbar, wait until the pressure decreases again before incrementing the HV.
- Once at 8000 V, wait for the pressure to decrease to 1.10^{-6} mbar, then increase the ionizer current to 1.7 mA.
- Put the reservoir current at 0.05 mA, and wait for the pressure in the source chamber to stabilize.
- Once the pressure starts going down (and it well below 10^{-5} mbar), **put back the controls in auto mode in the vacuum synoptic** so as to put back all the securities.
- If necessary, increase the reservoir current by steps of 0.05 mA to reach your usual working primary beam current in FCp (usually around 50 nA).

10.7.5.2 Starting the source after a maintenance

After outgassing a new source, or after a source maintenance that did not require a change of the source, it is recommended to start the source manually and gently the first time.
The starting pressure, with the source OFF, should be < 10^{-6} mbar. Always keep an eye on the pressure, it should not exceed 10^{-5} mbar. If the pressure increases too quickly, wait or go back to the previous step.

- In the source window, put the HV at 4000 V.
- Increase the ionizer emission current by increments of 0.2 mA until you reach 1.6 mA.
- Increase the HV to 8000 V.
- Increase the reservoir by increments of 0.01 up to 0.05 mA.

- Check FCp, you should start seeing current. Adjust the gonio to optimize the current in FCp, if necessary.
- Increase the ionizer emission current to 1.7 mA.
- Increase the reservoir step by step until you reach the desired FCp current (usually around 50 nA)

10.7.6 Cs source filaments replacement

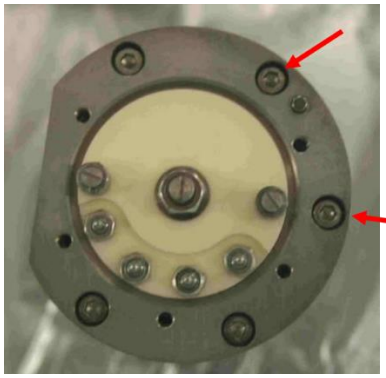
One filament heats the ionizer by electron bombardment and a second one heats the reservoir. Because filaments are heated and cooled often, they can sublimate, recrystallize, break or deform and need to be changed about once or twice a year.

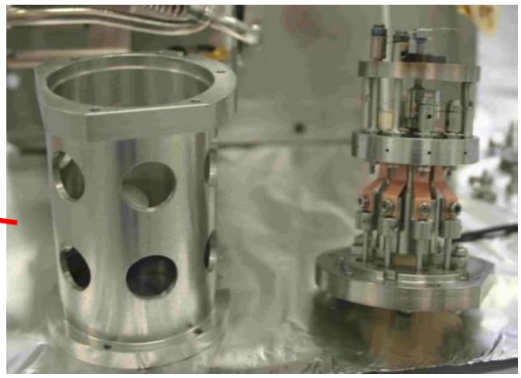
Follow the instructions as described in chapter 10.7.2 . to dismount the source, then proceed as follow:

10.7.6.1 Ionizer filament replacement

- Remove the Cs+ cage (see 10.7.2.2)
- Unscrew and remove the extractor

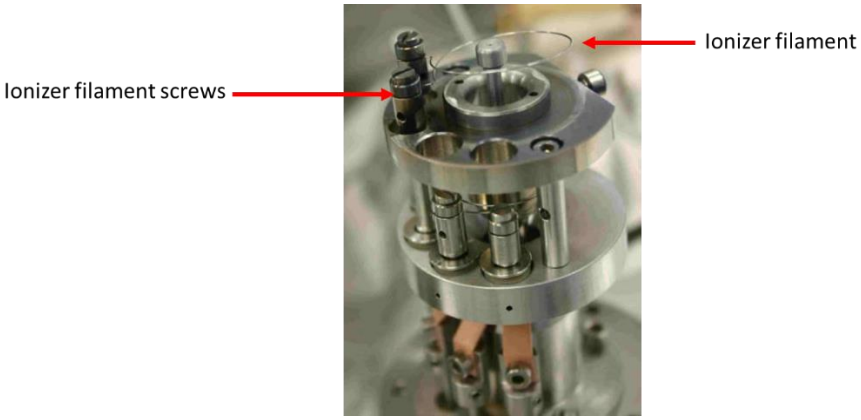
- Turn the source upside-down with caution and unscrew the five fixation-screw on the top of the cage





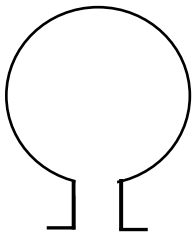
- Separate the source support and the cylinder of the cage and put the source back to its upward position.

- Loosen the screws holding the ionizer filament and cautiously remove the filament. Be careful when handling the filament, as after a long use, filaments become thin and brittle.

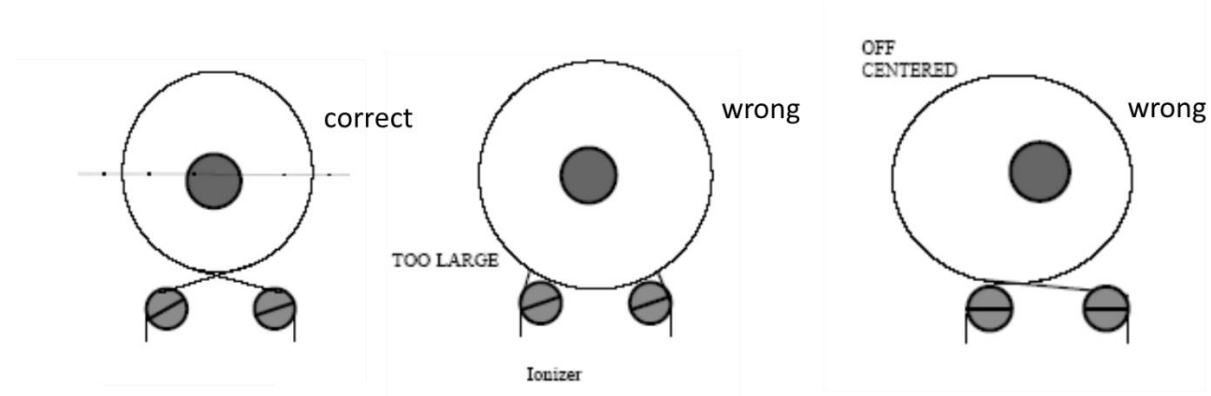


- If you are only replacing the Ionizer filament, follow the instructions below. If you need to replace also/only the reservoir filament, see the next chapter.

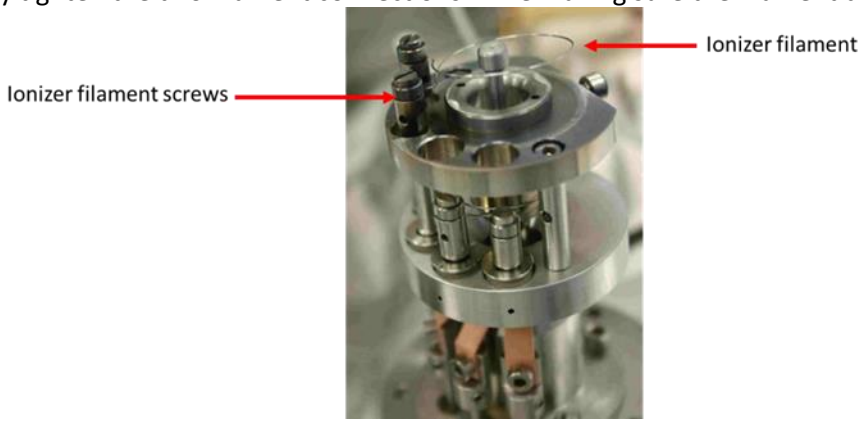
- Prepare the new filament by folding the two tips with this configuration



- Position the filament like showed in the following picture, perfectly centered around the ionizer. Once centered, very slightly push the filament away from the extractor, so that when heated the filament does not come in contact with the extractor. The source being vertical, the filament has a tendency to move down with time.



- Delicately tighten the two filament connections while making sure the filament doesn't move.



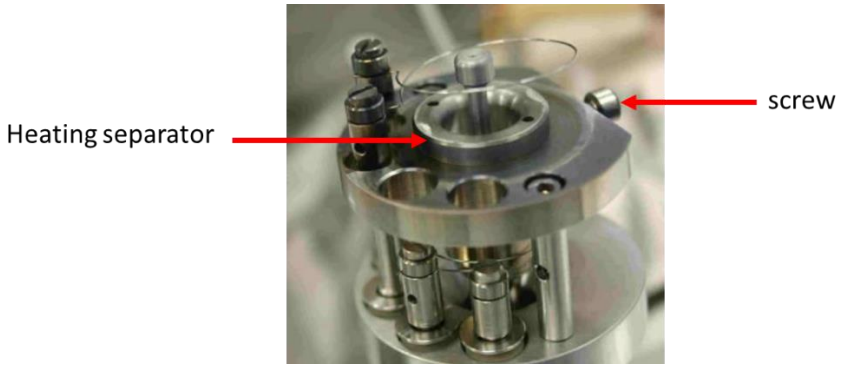
- Assemble the source support and the cylinder of the cage

- Put the extractor back and realign it (see chapter 10.7.3)
- Follow the instructions from chapter 10.7.2 in reverse order to reassemble the source and put it back on the instrument.

10.7.6.2 Reservoir filament replacement

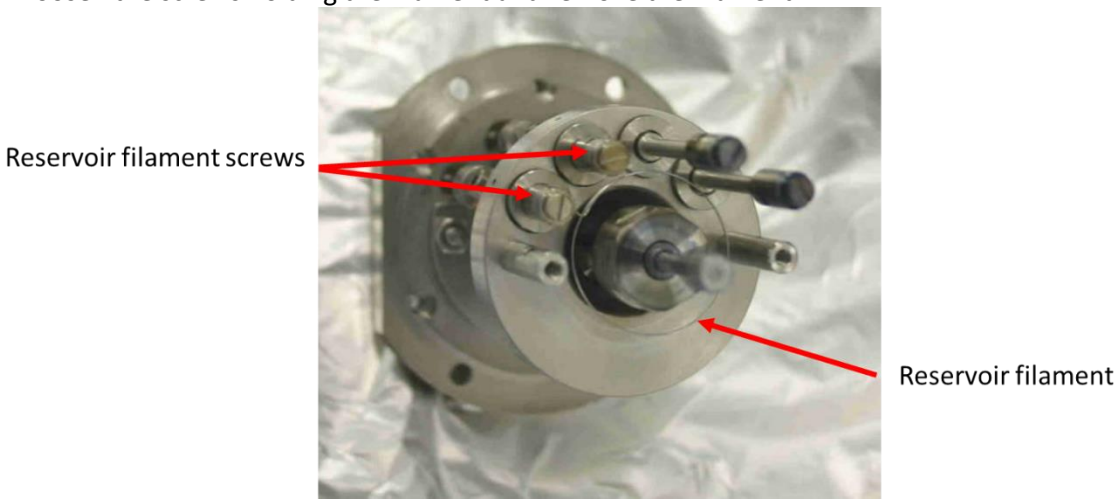
The reservoir filament rarely breaks on its own, this operation is rather infrequent. If the reservoir filament needs to be replaced, disassemble the source up to the point described in the previous chapter.
After you have unmounted the source and removed the ionizer filament (note: a used ionizer filament might break during this step so be sure to have a new one available just in case).

- Release the heating separator ring by unscrewing the screw (screw 1) on the side and pull out the separator

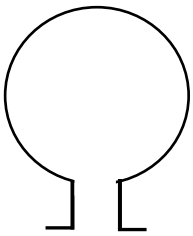


- Then, unscrew and remove the separation plate between the ionizer and the reservoir

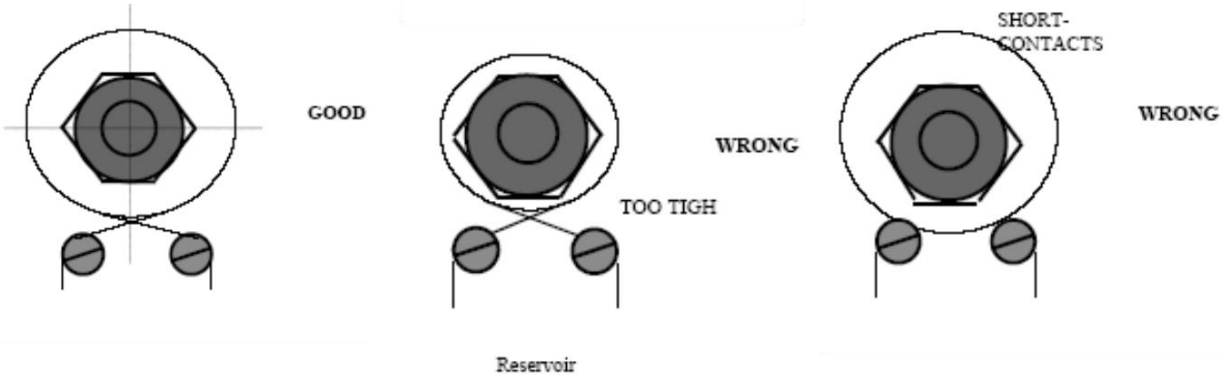
- Loosen the screws holding the filament and remove the filament.



- Prepare the new filament by folding the two tips with this configuration



- Position the filament like showed in the following picture, perfectly centered around the ionizer. Once centered, very slightly push the filament away from the intermediate shield, so that when heated the filament does not come in contact with it. The source being vertical, the filament has a tendency to move down with time.



- Delicately tighten the two filament connections while making sure the filament doesn't move.
- Put back and screw the separation plate, as well as the heating separator ring
- Put back the ionizer filament as detailed in chapter 0
- Assemble the source support and the cylinder of the cage
- Put the extractor back and realign it (see chapter 10.7.3)
- Follow the instructions from chapter 10.7.2 in reverse order to reassemble the source and put it back on the instrument.

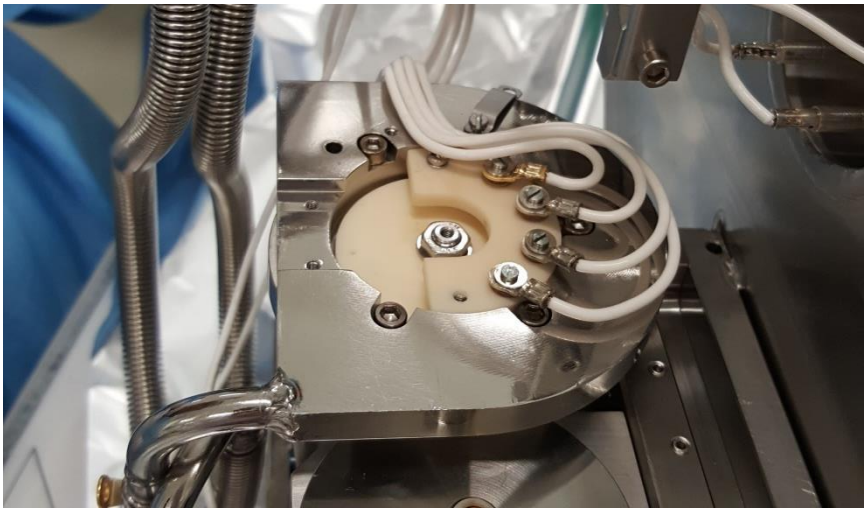
10.7.6.3 Degassing the source after a filament replacement

After remounting the source with a new filament, similarly to when changing source, you must degas it. Follow the procedure described in 10.7.5. If you changed a filament (or both) but did not change the ionizer, you can shorten the advised waiting time, as long as the pressure remains under 10^{-5} mbar.

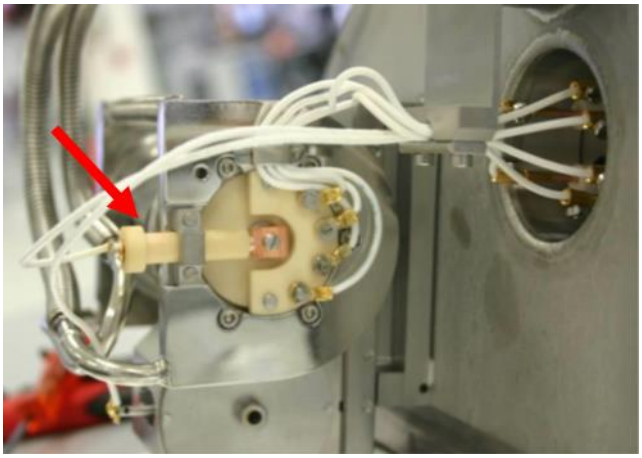
10.7.7 Considerations during re-assembly of the source

When reassembling the source, there are two main points that require caution:

- The half-moon ceramic must be put in place correctly and lugs on the half-moon ceramic must neither touch one another, nor should they touch the inox cooling system surrounding the ceramic.



- When putting back the HV ceramic, you must not push it too far. The picture below shows the right position of the ceramic.



10.8 RF-plasma oxygen ion source

The RF-plasma has replaced the duoplasmatron source due to a higher brightness, better long term stability and minimum maintenance/cleaning.

10.8.1 Coolant and cooling system

The RF source is cooled via a separate cooling circuit filled with galden. The galden circuit is a closed system and is cooled by the water circuit through the RF source chassis (see chapter 2.3.6). Always use the specific galden referred by Cameca and NEVER put water inside the galden circuit. The galden amount in the circuit is constant. It is important to keep the right quantity of galden in the circuit, as galden will expend when heated by the operating source, and then retract when the source cools off after being shut down. Too much galden might thus cause an overflow of galden, while a low level of galden might cause an insufficient cooling of the source, and then damage it.

Regularly check the galden level. When the source is off and cold, the galden level in the source reservoir should be mid-height of the glass (Figure 469).

Figure 469: gladden level in the RF source reservoir, when the source is off and cool.

10.8.2 Source disassembly/cleaning

The user should not, at any time, try to dismantle the RF source. In case of malfunction, it is advised to request an intervention from Cameca engineers. In addition, it is recommended to have the RF source serviced by Oregon Physics after about 2500 hours of use.

10.8.3 Galden circuit proper connection

It is primordial that the galden flows correctly in the circuit connecting the source and the cooling water. Figure 470 shows the proper connection of the galden suppy tubes to the source at one end and to the heat exchange unit at the other end. The cool galden comes out of the cooling system (located in the RF source cabinet) via the “source out” port. The tube, labelled by a blue tape, connects to the RF source body at the bottom. The galden warmed by the source comes out of the source body via the port on the side at the top of the source body. The tube, labelled by a red tape, connects to the heat exchange unit via the “source in” port.

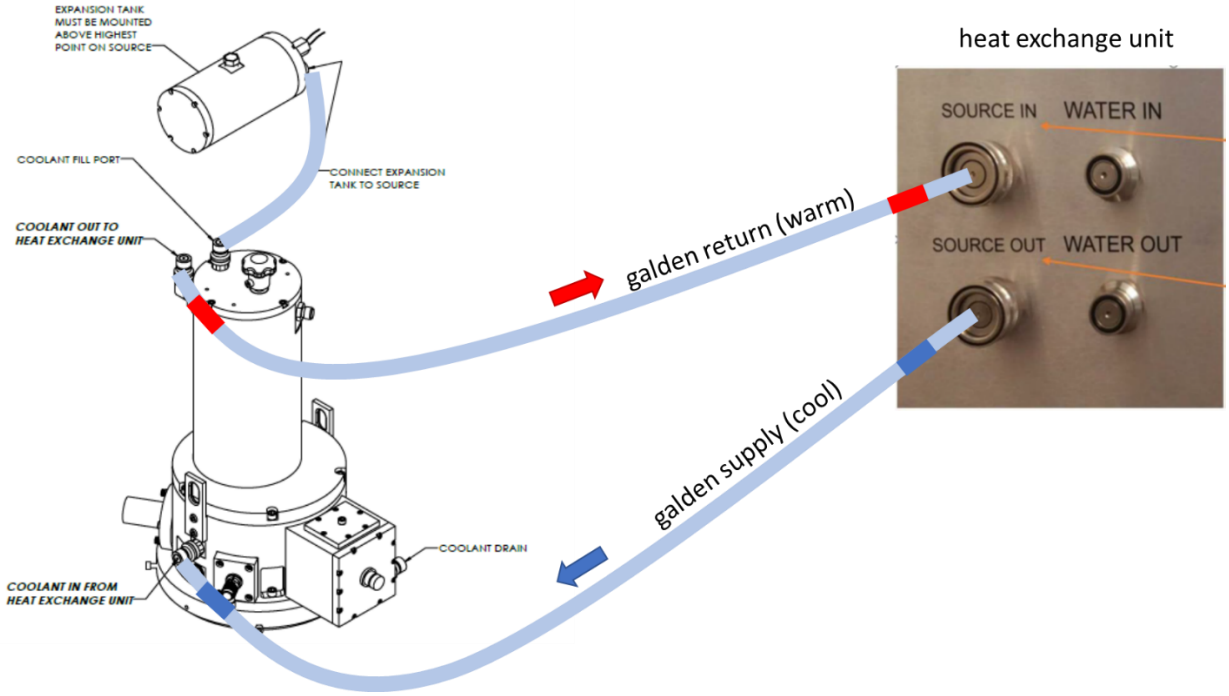
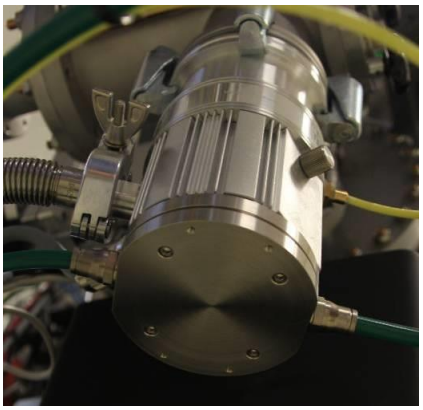


Figure 470: connection of the galden cooling circuit to the RF source body.

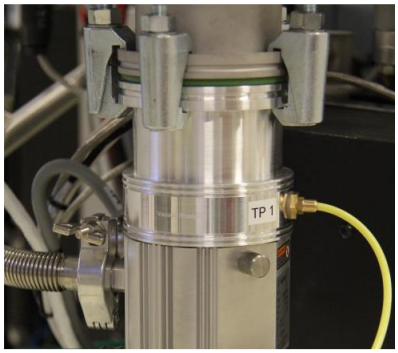
10.9 Replacing a turbo pump

10.9.1 Replacing the airlock Agilent V84 turbo pump (TP1)

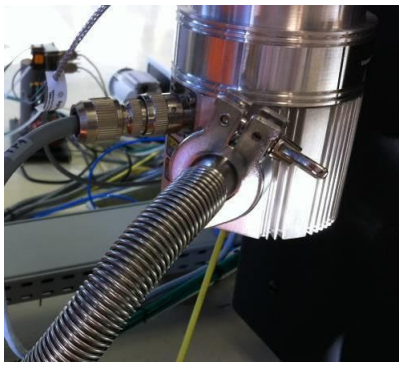
- Vent the airlock and wait for the end of the venting process,
- Unscrew the cooling plate at the bottom of the turbo pump. You do not need to stop the cooling circuit.



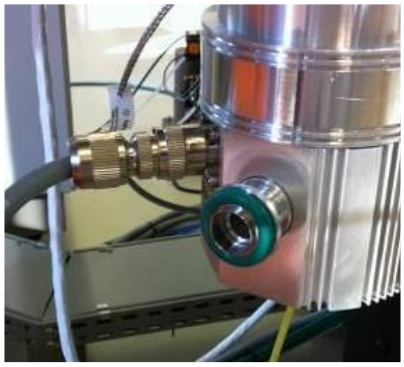
- Disconnect the venting line (yellow tubing)



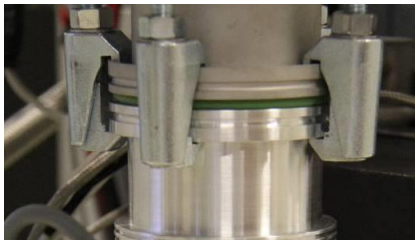
- Disconnect the rough pumping line by unscrewing the gasket clamp. Leave the gasket on the bellow tube and cover it with an aluminum foil



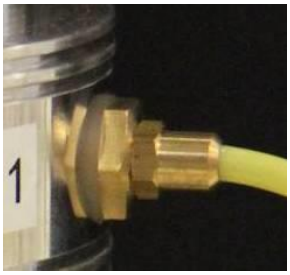
- Disconnect the turbo pump control cable



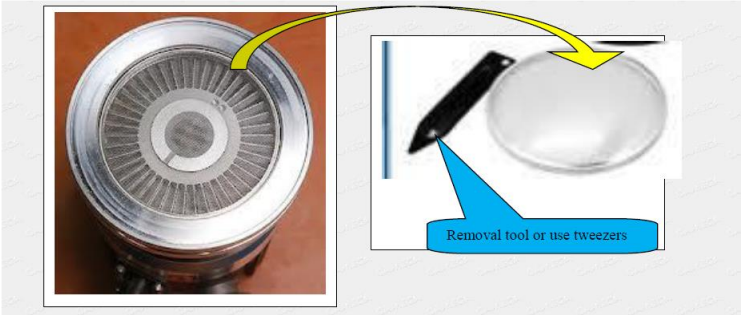
- Separate the pump from the instrument by removing the four clamps holding it. Cover the gasket with aluminum to keep it clean.



- Remove the dry nitrogen venting line inlet connection (nylon gasket) from the old pump and place it on the new pump. If it's damaged, replace it with a new one. Carefully tighten it to avoid leaks.



- Remove the grid from inside the old pump and place it in the new pump. Be carefully that the grid should not touch or rest on the rotor blade.

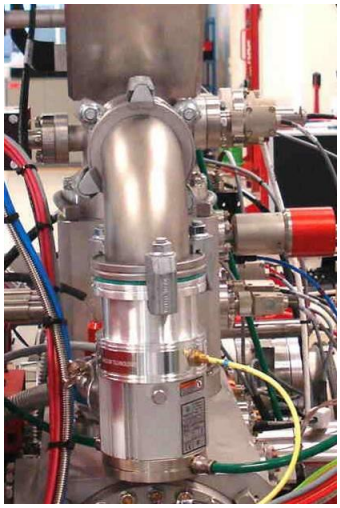


- Place the gasket on top of the new turbo pump. Add a small amount of vacuum grease (Apiezon) on the rubber part of the gasket if it is too dry.
- Install the pump on the instrument and hold it with the clamps. Be aware of the positions of the different inlets and position the pump accordingly. Tighten the clamps.
- Attach the control cable
- Attach the bellow and its gasket clamp to the pump. Add a small amount of vacuum grease (Apiezon) on the rubber part of the gasket if it is too dry.
- Reconnect the dry nitrogen venting line (yellow tubing)
- Screw the cooling plate into place.
- To establish communication with TP1, go to the vacuum synoptic and select the manual control mode. Start TP1. Wait 5 seconds and Stop TP1. Click on “read status” to refresh the window and make sure TP1 appears stopped (orange cross) 
- Put the synoptic back to automatic mode and launch the pumping of the airlock.

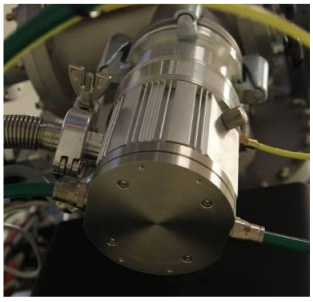
10.9.2 Replacing the Source chamber Agilent V84 turbo pump (TP5)

- Vent the source chamber via the vacuum synoptic and wait for the end of the venting process.

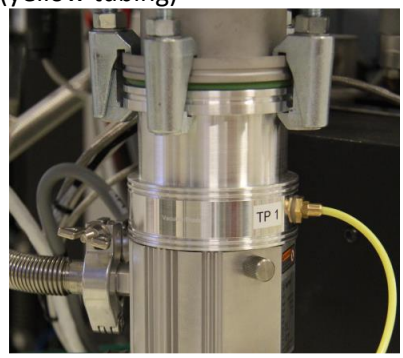
- Dismount the source turbo pump from the elbow by unscrewing the clamps. Cover the gasket and top of the pump with aluminum foil.



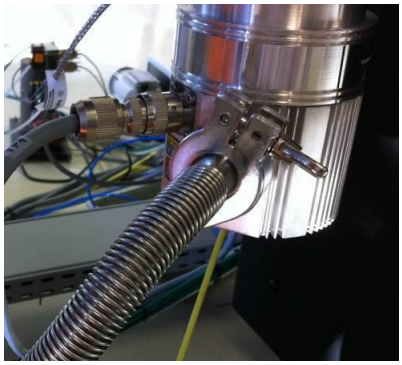
- Unscrew the cooling plate at the bottom of the turbo pump. You do not need to stop the cooling circuit.



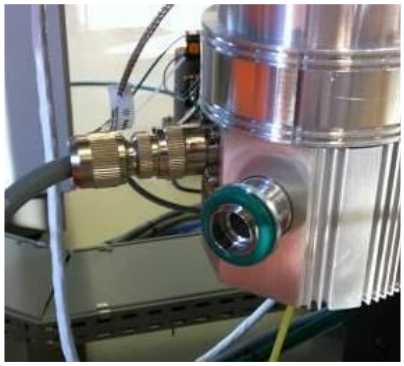
- Disconnect the venting line (yellow tubing)



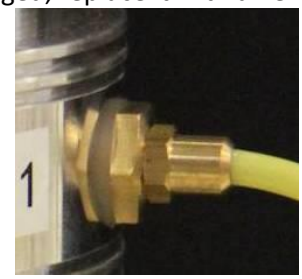
- Disconnect the rough pumping line by unscrewing the gasket clamp. Leave the gasket on the bellow tube and cover it with an aluminum foil



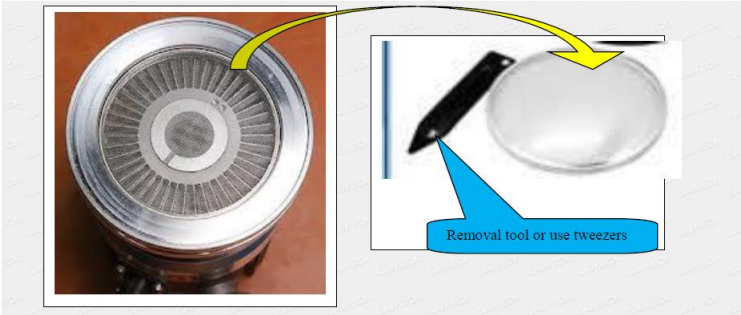
- Disconnect the turbo pump control cable



- Remove the dry nitrogen venting line inlet connection (nylon gasket) from the old pump and place it on the new pump. If it's damaged, replace it with a new one. Carefully tighten it to avoid leaks.



- Remove the grid from inside the old pump and place it in the new pump. Be carefully that the grid should not touch or rest on the rotor blade.



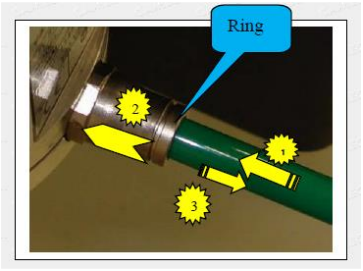
- Place the gasket on top of the new turbo pump. Add a small amount of vacuum grease (Apiezon) on the the rubber part of the gasket if it is too dry.
- Install the the pump on the instrument and hold it with the clamps. Be aware of the positions of the different inlets and position the pump accordingly. Thighten the clamps.
- Attach the control cable
- Attach the bellow and its gasket clamp to the pump. Add a small amount of vacuum grease (Apiezon) on the rubber part of the gasket if it is too dry.
- Connect the dry N2 venting line (yellow tubing)
- Screw the cooling plate into place.
- To establish communication with TP5, go to the vacuum synoptic and select the manual control mode. Start TP5. Wait 5 seconds and Stop TP5. Click on "read status" to refresh the window and make sure TP5 appears stopped (orange cross) 
- Put the synoptic back to automatic mode and launch the pumping of the source.

10.9.3 Replacing the multicollection Agilent TV551 turbo pump

- Vent the source chamber via the vacuum synoptic and wait for the end of the venting process.

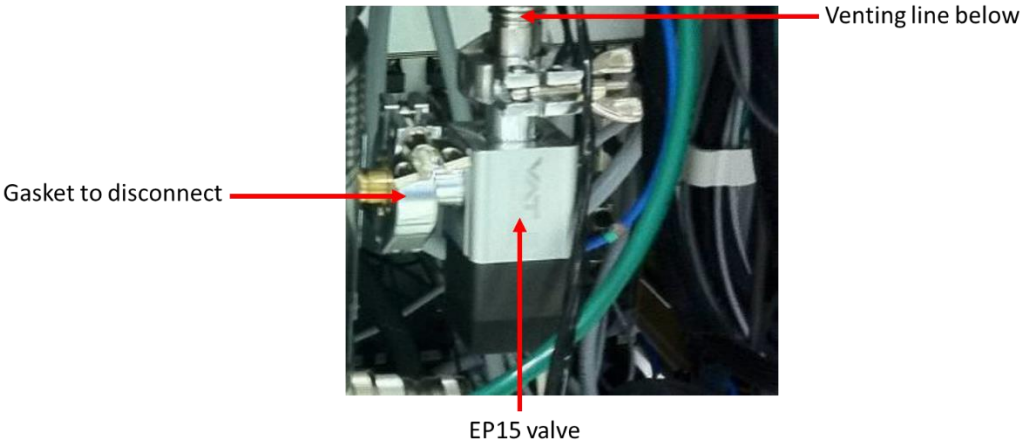
- For this pump, you need to interrupt the water circuit. You must then put the magnetic field value to 0 in the Tuning window and switch off the magnetic field chassis:

- Stop the water circulation by closing the taps.
- Disconnect the water tubes (green) from the turbo pump. To do so: push the tube to disconnect into the connector, hold the ring toward the connector and pull the tube.

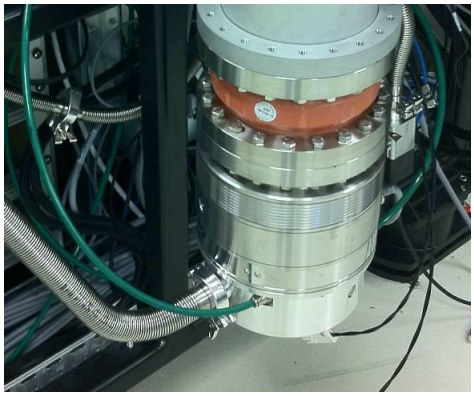


- Temporarily connect the two tubes together with a union connector.

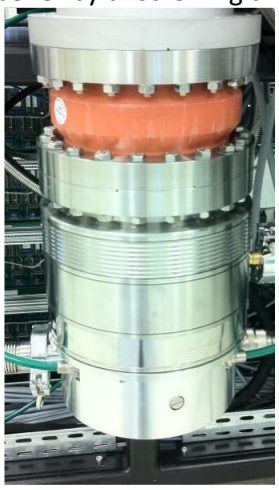
- Remove the venting line (below). Unscrew the clamp and put an aluminum foil on it to protect it from dust.



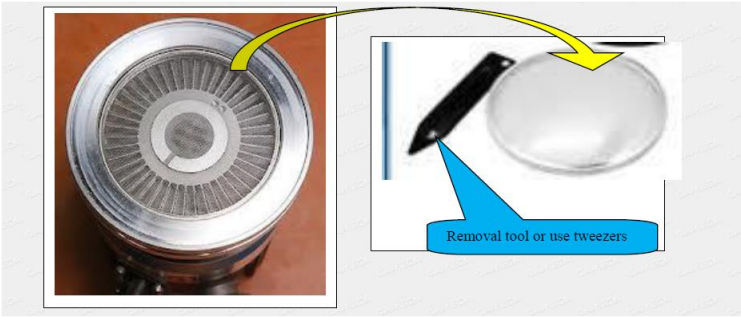
- Disconnect the rough pumping line by unscrewing the gasket clamp. Keep the gasket attached to the below and cover it with aluminum.



- Disconnect the turbo pump controller cable
- Remove the pump from the dampener by unscrewing the bolts and nuts.



- Unscrew the venting inlet and the rubber or nylon gasket and install it on the new pump. Replace the gasket if it appears damaged. Carefully tighten the inlet to avoid leaks.
- Remove the grid from inside the old pump and place it in the new pump. Be carefully that the grid should not touch or rest on the rotor blade.



- Remove the cooling kit from the old pump and place it on the new one. Unscrew the screws and don’t forget to transfer the seal.

- Put a new copper gasket on top of the new turbo pump and install the pump on the damper. Be aware of the positions of the different inlets and position the pump accordingly. Tighten the bolts and nuts.
- Plug in the controller cable.
- Attach the rough pumping below and gasket with the gasket clamp. Add a small amount of vacuum grease (Apiezon) on the rubber part of the gasket if it is too dry.
- Re-connect the dry N2 venting line. Add a small amount of vacuum grease (Apiezon) if the rubber part of the gasket is too dry.
- Reconnect the water tubes and open the taps.
- To establish communication with TP3, go to the vacuum synoptic and select the manual control mode. Start TP3. Wait 5 seconds and Stop TP3. Click on “read status” to refresh the window and make sure TP3 appears stopped (orange cross) 
- Put the synoptic back to automatic mode and launch the pumping of the multicollection.

10.10 Changing a UHV gauge filament

It is necessary to change a UHV gauge when

- its controller displays the following errors:
  - EO4 (short cut on the filament)
  - EO5 (filament cut)
  - EO6 (grid grounded)
- When the pressure reading is wrong and/or unstable (and a leak has been ruled out).

To change a gauge filament, proceed as follow:

- Vent the chamber whose gauge is faulty, and wait for the end of the venting process
- Uncrew the UHV cable connecting the gauge. Be advised that the gauge feedthrough and housing may be hot.

- Unscrew the six nuts on the rim of the gauge feedthrough.

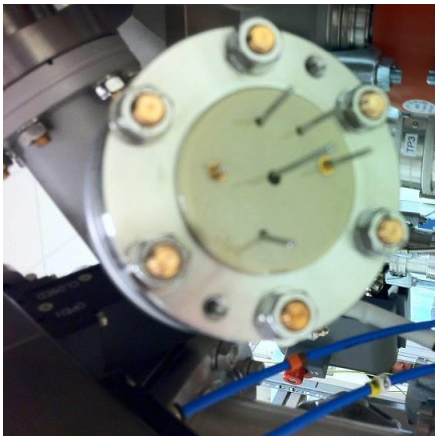
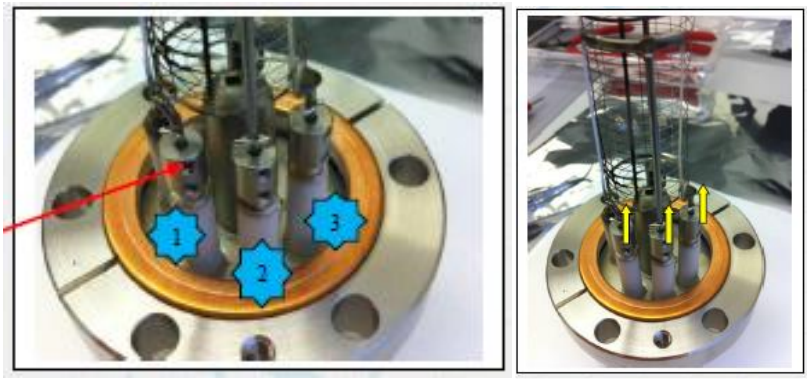


Figure 471: UHV gauge flange external view

There are five feedthroughs:

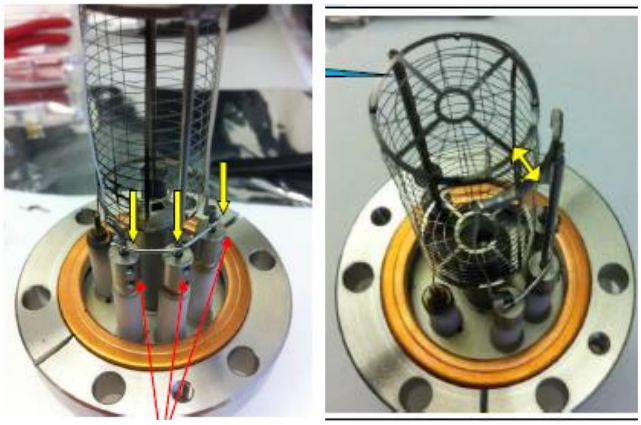
- The central one is connected to the ion collector (attracting all positively ionized molecules within the volume defined by the grid).
- The three close to each others, are connected to the two filaments (the middle pin being common to both). They are generating electrons to ionize the residual gas within the grid space.
- The isolated one on the opposite side is connected to the grid (accelerating the electrons emitted by the filament toward the ionization space defined by the grid).

- Put clean gloves and carefully remove the gauge from the instrument. Place it on the table. Be careful not to break the pins or the gauge, they are fragile.
- Cover the opening of the instrument with an aluminum foil to prevent dust.
- Unscrew the 2 screws on each of the 3 feet of the filament kit and gently pull out the filament kit with the broken filament.

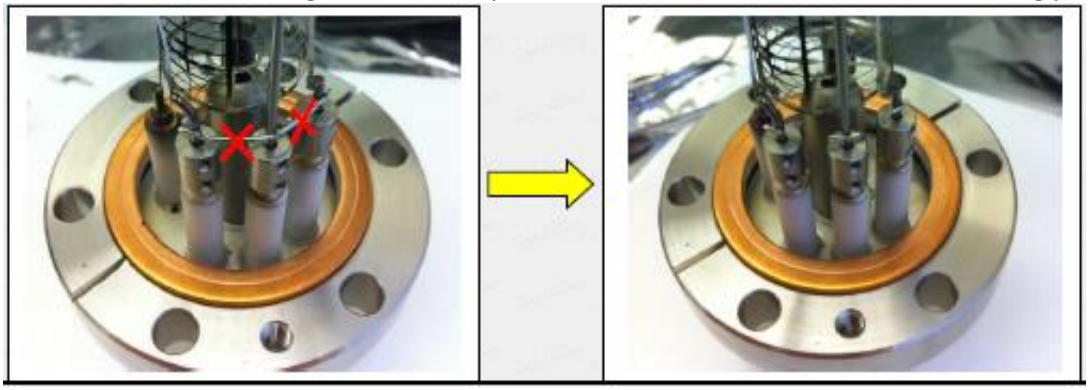


- Prepare the new filament kit and insert it into the gauge.

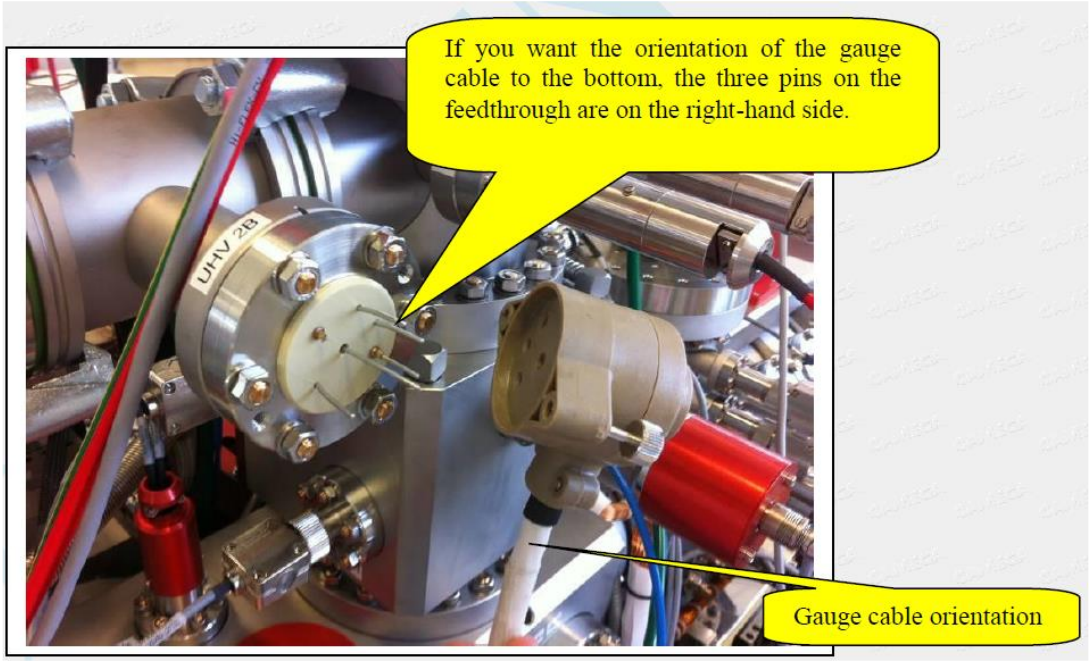
- Tighten the screws and make sure that the filaments do not touch the gate.



- Once the filaments are tightened and in place, cut the bottom metal brace with cutting pliers.



- Replace the copper gasket with a new one and put the gauge back on the instrument.
- Be mindful of the orientation: To have the gauge cable coming from under the gauge, put the three filament pins on the right side, as shown on the picture below.



- Screw the gauge
- Plug the cable back
- Launch the pumping of the chamber via the vacuum synoptic and wait for the end of the process.

10.11 Tuning the pneumatic antivibration system

If you notice vibrations on your images (in RTI mode, for instance), it might be caused by the vibration of the instrument that is not properly suspended.
To check the good suspension of the instrument,

- Try to move it by pushing against the instrument body (on the analysis chamber for instance), with two fingers. You should feel the instrument move (slightly).
- Check the four feet of the instrument. None of them should reach neither the bottom stop not the upper stop. If it has reached the upper stop, decrease the foot's air pressure via the manometer under the instrument. If it has reached the bottom stop, increase the foot's pressure. Every time you adjust the pressure on one foot, check that the other feet are not touching a stop either, as adjusting the pressure on one foot will re-equilibrate the entire instrument.

It is recommended to have the "level" about one centimeter above the bottom stop, each foot pressure independently adjusted with about 5 bars of pressure on most feet and more (~5-6 bars) on the right-side feet, supporting the magnet.