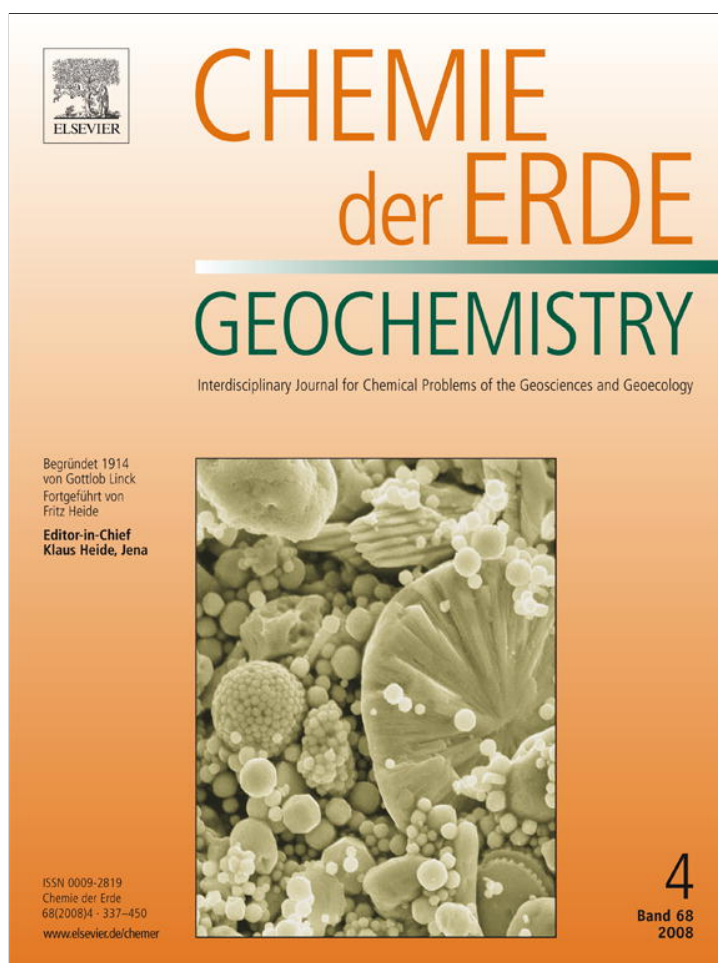


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INVITED REVIEW

I–Xe dating: From adolescence to maturity

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Abstract

The I–Xe chronometer is based upon decay of now-extinct ^{129}I where the ratio of accumulated daughter ^{129}Xe to stable ^{127}I reflects the iodine isotopic ratio at closure of the host mineral. Since none of the parent remains, I–Xe is by nature a relative chronometer but, when referenced by a standard mineral of known age, the I–Xe system becomes an absolute chronometer reflecting true closure times. Most iodine hosts are secondary minerals so the I–Xe system is unique in providing details of post-formational chronometry not readily available with other chronometers. The short half-life of ^{129}I gives it exceptional precision. However, the secondary nature of iodine host minerals, combined with the inherent precision of I–Xe, were responsible for a large database of “whole-rock” I–Xe ages that were not easily interpreted. As this problem evolved historically, doubts were cast upon the viability of the I–Xe system as a chronometer which persisted until it was tested against other chronometers in single-mineral systems. Properly calibrated, absolute I–Xe ages reflect the true closure time of the host minerals, and sequences of closure times in different hosts provide cooling rates for the parent object.

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Keywords: I–Xe chronometer; Meteorite evolution; Cooling rates

1. Introduction

The I–Xe chronometer is now 48 years of age, an age that signals seasoned maturity for most of us. In its growth from adolescence to maturity, the I–Xe chronometer has gone through the typical teenage years of being misunderstood, asserting itself when it should not, and generalizing to the broader application when its talents are more restrictive. In the journey toward maturity, the I–Xe chronometer has demonstrated its

limitations and promoted its unique features, redefining itself in terms of its strengths.

It was first proposed by Brown (1947) that the age of the elements might be determined if the decay product of an extinct radionuclide could be found in meteorites. Excess ^{129}Xe from the decay of ^{129}I (half-life = 17 Ma) was first reported in the Richardton H4 meteorite by Reynolds (1960a). Radiogenic ^{129}Xe in Richardton was confirmed by Signer (1960) and Zähringer and Gentner (1961), and a more extreme excess was discovered by Reynolds (1960b) in the Indarch enstatite chondrite, with a measured $^{129}\text{Xe}/^{132}\text{Xe}$ ratio of 3.4, whereas the normal ratio is 0.98. Proof that this excess came from decay of living ^{129}I was made by the association of this ^{129}Xe excess with iodine-derived ^{128}Xe , produced by neutron irradiation where stable ^{127}I is converted to

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^{128}Xe . The first application of the I–Xe chronometer to estimate the age of the elements was presented by Reynolds (1960c). Fish and Goles (1962) realized that, by clever treatment of data from neutron-irradiated samples, correlations between these two iodine-derived isotopes yield straight lines in 3-isotopes plots, and the slopes of these lines are proportional to the $^{129}\text{I}/^{127}\text{I}$ ratio at closure. Thus, high-precision I–Xe ages can be obtained, with proper monitors, by measurement of Xe isotopic ratios alone, avoiding the complications of isotope dilution techniques.

Following these early demonstrations of the I–Xe chronometer, it was widely applied to a variety of meteorites of different types and different metamorphic grades, with mixed results. It was demonstrated that the I–Xe system is inherently quite precise, resolving “formation” ages of many different meteorites with resolutions often less than a million years. The results, however, did not adequately track the highly developed and largely accepted metamorphic trends, raising the question of the viability of I–Xe as a reliable chronometer (cf. Swindle and Podosek, 1988). Various explanations were given for such failures, ranging from purported isotopic heterogeneities of iodine within the solar nebula (Crabb et al., 1982; Jordan et al., 1980) to interpretations of the observed “isochrons” as simply mixing lines between reservoirs with different isotopic compositions, perhaps with ^{129}Xe as surrogate for “dead” ^{129}I in extrasolar grains (Clayton, 1975; Crabb et al., 1982), thus of no chronological significance. Moreover, some I–Xe ages appeared to be older than the formation ages of calcium–aluminum-rich inclusions (CAIs) which are among the earliest solar system condensates (Amelin et al., 2002). Thus, reported I–Xe ages began to fall on deaf ears and were dismissed, because the high precision with which I–Xe ages could be obtained did not correspond to interpretable chronologies.

2. I–Xe chronometry: addressing historical problems

The discovery of CAIs in meteorites, and the realization that these were early solar nebula condensates, brought to focus one of the early interpretational difficulties of the I–Xe system as a chronometer. Allende CAIs contain large quantities of radiogenic ^{129}Xe , and they are rich in sodalite and nepheline, the host phases for this component. The presence of low-temperature halogen-containing secondary minerals, presumably emplaced by aqueous processes, are clearly not associated with initial condensation, and the presence of excess ^{129}Xe , the daughter product of ^{129}I , suggested that Allende CAIs had gone through significant

secondary alteration shortly after their high temperature formation. The parent–daughter pair indicated that the I–Xe chronometer in these objects reflects closure of these secondary phases, not the formation time of the refractory CAI itself. This clearly points out interpretational difficulties for I–Xe in whole-rock samples, since little is generally known about either the iodine host minerals or the processes which formed them. It is, therefore, no surprise that conventional metamorphic sequences in chondrites, reflected in their numeric petrographic types (e.g., Van Schmus and Wood, 1967), do not always track the measured I–Xe ages, certainly not within the high reported precision. Many iodine host phases are low-temperature minerals while others are not and little is generally known about which phases in whole-rock samples are responsible for the I–Xe isochrons. In fact, the meteorites for which I–Xe ages *do* reflect the petrographic type are the enstatite chondrites where the primary iodine host phase is enstatite itself, the major mineral in these objects (cf. Kennedy et al., 1988). The lesson here is that I–Xe is seldom a “whole-rock” chronometer, but a mineral-specific chronometer capable of obtaining different closure times for different phases within a given meteorite. While this created a problem of interpretation for early I–Xe studies, it is now exploited, providing details for post-formational alteration and thermal history (Pravdivtseva et al., 2005a, b, 2006a, b, 2008). Recent advances in resonance ionization for Xe measurements, such as RELAX developed by the Manchester group, yield greater sensitivity than conventional mass spectrometry and provide unique opportunities for single-mineral studies (Whitby et al., 2000).

There have also been difficulties in measuring the conversion efficiency of ^{127}I to ^{128}Xe which is required to obtain the $^{129}\text{I}/^{127}\text{I}$ ratio present at the time of closure. These difficulties are due to the extreme differences in the iodine concentrations found in meteorites and in typical iodine-bearing compounds used as conversion monitors. This leads to two sources of calibration error. The first arises from self-shielding at the sharp neutron capture resonances, which account for about $\frac{1}{2}$ of neutron captures by ^{127}I . The second arises from the extreme isotopic dilutions required when pure iodine compounds are used as monitors to obtain the initial isotopic ratio. Both of these are addressed in Section 3.

Due to the difficulties in interpretation of “whole rock” I–Xe ages, and the errors introduced by improperly monitoring neutron capture probabilities, damage to the credibility of the I–Xe chronometer had already been done. The scientific community at large became skeptical that the I–Xe system was capable of understandable results. To resurrect the I–Xe chronometer it was necessary to prove that well-defined ratios between parent and daughter could be interpreted as meaningful ages.

3. I–Xe dating: the technique

In I–Xe dating, the ratio of accumulated radiogenic ^{129}Xe (denoted $^{129*}\text{Xe}$) from the decay of 17 Ma ^{129}I (now extinct) to stable ^{127}I would seem to be the quantity of interest. (Various values exist in the literature for the ^{129}I half-life, ranging from 15.6 (Russel, 1957) to 19.7 Ma (Kuhry and Bontems, 1973) with latest recommended value 17 Ma (Holden, 1990).) However, a tracer for ^{127}I is iodine-derived ^{128}Xe (denoted $^{128*}\text{Xe}$), produced by neutron capture on ^{127}I in a reactor, so the $^{129*}\text{Xe}/^{128*}\text{Xe}$ ratio, properly monitored, provides the chronometry. Since both are isotopes of xenon, this ratio can be determined by simply measuring isotopic ratios alone, thus avoiding the problems associated with measurements of absolute quantities of either parent or daughter. Mass spectrometers are most precise for ratio measurements, and noble gases are easy to chemically purify eliminating any effects from isobaric interferences, so the I–Xe technique has inherent advantages over other methods. Each irradiation, of course, has its unique ratio of $^{128*}\text{Xe}$ to ^{127}I , which would be nominally established by careful monitoring of the neutron capture probability in each irradiation, thus yielding the chronometrically significant $^{129}\text{I}/^{127}\text{I}$ ratio at closure of the iodine host mineral. However, direct monitoring of the neutron capture probability is impractical for several reasons. An alternative calibration procedure by means of a reference meteorite standard is the usual method used in I–Xe dating. This method yields the highest precision of both relative (to the irradiation standard) and absolute closure times but it fails to directly provide the value of the $^{129}\text{I}/^{127}\text{I}$ ratio at closure, a quantity of considerable interest.

During neutron irradiation, $^{128*}\text{Xe}$ is produced at iodine sites by neutron capture on stable ^{127}I . These sites will also contain $^{129*}\text{Xe}$ provided ^{129}I was still alive at the closure time of the host mineral. A suite of iodine sites in a common mineral phase, or a collection of phases that closed simultaneously, will have a unique $^{129}\text{I}/^{127}\text{I}$ ratio at the time of closure, assuming that iodine was isotopically homogeneous in the solar nebula. Following the neutron irradiation, this ratio is reflected by a similarly unique ratio of $^{129*}\text{Xe}/^{128*}\text{Xe}$ at these sites if no $^{129*}\text{Xe}$ has been lost. In any given iodine host phase, the total quantity of ^{129}Xe is the sum of $^{129*}\text{Xe}$ from ^{129}I decay, and ^{129}Xe that was previously present in that phase, denoted $^{129}\text{Xe}_t$, for the “trapped” component. Similarly, the total quantity of ^{128}Xe is the sum of $^{128*}\text{Xe}$, from neutron capture on ^{127}I , and ^{128}Xe previously present. Thus, at these isotopes simple two-component mixtures will exist, and it is the presence of I–Xe isochrons, linear mixing lines, between these components that demonstrate the initial assumptions are met. Normalized to either ^{132}Xe , the most abundant

Xe isotope, or ^{130}Xe , an isotope shielded from production by fission, we can easily derive an equation for the I–Xe isochron in terms of isotope ratios:

$$^{129}\text{Xe}/^{132}\text{Xe} = [(^{129}\text{Xe}/^{132}\text{Xe})_t - S(^{128}\text{Xe}/^{132}\text{Xe})_t] + S(^{128}\text{Xe}/^{132}\text{Xe})$$

This will be an equation of a straight line in the form $y = Sx + b$, where y is the measured $^{129}\text{Xe}/^{132}\text{Xe}$ ratio, x is the measured $^{128}\text{Xe}/^{132}\text{Xe}$ ratio and S , the slope, is the ratio of the two iodine-produced isotopes, $^{129*}\text{Xe}/^{128*}\text{Xe}$. Thus, the presence of a straight line indicates simple two-component mixing between “trapped” Xe, the lower left endpoint, and a unique iodine-derived component, at infinity to the upper right of a 3-isotope plot (Fig. 1). The intercept b of the straight line, the term in brackets, need not be known and the “trapped” ratios, denoted by the subscript t , usually correspond to the common Xe composition found in the most Xe-rich meteorites, though it too need not be known for I–Xe dating (only the slope is important). This component, previously referred to as AVCC, for Average Value Carbonaceous Chondrite, “planetary” Xe, or OC–Xe (Signer and Suess, 1963; Pepin and Signer, 1965; Lavielle and Marti, 1992) is now generally known as Q or P1–Xe (Busemann et al., 2000; Ott, 2002), and is present in exceedingly high concentrations in the enigmatic phase- Q (for “Quintessence”) (Lewis et al., 1975). It is fortunate that trapped Xe does not differ much in composition from that in the terrestrial atmosphere, so, even though blank corrections are usually negligible, any corrections applied

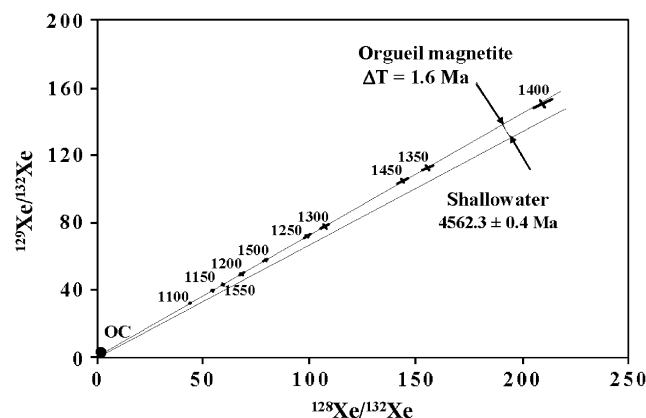


Fig. 1. I–Xe isochrons for magnetite separated from the Orgueil meteorite (cover photo) and the Shallowater meteorite, irradiation standard. The difference in slopes of the two correlation lines corresponds to the difference in closure times of the I–Xe system, and subsequently to the age difference. The numbers represent the extraction temperatures in step-wise heating analysis; OC stands for the Ordinary Chondrite component (Lavielle and Marti, 1992) and the errors shown are one sigma.

neither change the isochron slopes nor affect the I–Xe ages. Since all of the ^{129}I present at closure has decayed to ^{129}Xe , $S = ^{129}\text{Xe}/^{128}\text{Xe} = (^{129}\text{I}/^{127}\text{I})_i / (^{128}\text{Xe}/^{127}\text{I})$. Thus, the isochron slope S is proportional to the initial iodine isotopic composition at closure ($^{129}\text{I}/^{127}\text{I}$)_{*i*} and the denominator $^{128}\text{Xe}/^{127}\text{I}$ is the neutron capture probability for ^{127}I , a constant for that particular irradiation.

Following neutron irradiation, the sample is generally heated to incrementally higher temperatures (step-wise heating) with the isotopic composition of Xe measured in each release. In the lower temperature steps, non-correlated excesses of ^{128}Xe are usually observed, indicating prior losses of ^{129}Xe from the less retentive mineral sites. I–Xe ages are determined when many successive stepped extractions yield straight lines (isochrons), indicating a constant $^{128}\text{Xe}/^{129}\text{Xe}$ ratio, and thus a constant $^{129}\text{I}/^{127}\text{I}$ ratio at closure. In order to determine an age from this isochron, a standard calibration meteorite is included with the irradiation. The ratio of the slopes of the two isochrons is the ratio of the initial iodine at closure, thus providing the age of the sample relative to that of the standard. An example is shown in Fig. 1 where the I–Xe standard is the enstatite achondrite Shallowater (Hohenberg, 1967). The iodine carrier for Shallowater is associated with its primary mineral phase enstatite (Kehm et al., 1994). In Fig. 1, the isochron for Orgueil magnetite is shown along with the isochron for the Shallowater reference (data points are not shown for Shallowater). Since the iodine isotopic compositions at closure ($^{129}\text{I}/^{127}\text{I}$)_{*i*} are proportional to the isochron slopes, the ratio of the two slopes provides the relative I–Xe age since the constant of proportionality, the ^{127}I neutron capture probability, is identical in the same irradiation and cancels out. The difference in closure times ΔT between the two samples is simply given by ΔT (Ma) = 23.6 ln(slope ratio), where 23.6 Ma is the mean life of ^{129}I . The normalization isotope, ^{132}Xe or ^{130}Xe , is chosen to provide the best precision.

Iodine in the sample must have an identical ^{127}I neutron capture probability as the reference standard. Thermal neutrons are generated by water moderation in most reactors, yet the high neutron fluences generally used (typically 2×10^{19} n/cm²) require positions in the pool area relatively close to the core. In such regions there are usually relatively steep radial and vertical neutron flux gradients, effects that must be neutralized for proper normalization. The effect of the radial neutron gradient is minimized by the constant rotation of the capsule about its vertical axis during the irradiation. The effect of a vertical neutron gradient can be minimized by selecting locations at the central point of the vertical flux distribution, where the flux is maximal and the variation with vertical position is minimal, and by arranging the samples in a single plane

around the perimeter of the irradiation capsule at that vertical position. To monitor actual fluence variations among the samples, a set of Co-doped aluminum flux wires are positioned around the perimeter of the capsule. These flux wires are later analyzed with a gamma-ray spectrometer to quantify any fluence differences between samples, typically less than 0.5%, which is good considering that the neutron flux gradient across the one inch diameter of the capsule can be greater than 10%. The samples themselves are loaded into fused quartz vials and sealed under vacuum, and the capsule itself is punctured so that it is water-flooded for proper thermal control.

The most direct way to measure the neutron capture probability is to determine the ^{128}Xe produced from a known quantity of ^{127}I . Iodine salts have been used in the past but this has led to problems. One comes from the fact that meteoritic iodine occurs at ppb levels, a billion times less than those found in such compounds. This extreme concentration makes KI, or similar iodine salts, unusable for an irradiation monitor for two important reasons. The most fundamental problem associated with large iodine concentrations comes from the fact that about half of the ^{127}I neutron captures are due to sharp resonance captures of epithermal neutrons. The two major neutron capture resonances for ^{127}I have cross sections of more than 10^4 barns, with three others resonances between 300 and 1000 barns, collectively accounting for half of the ^{127}I neutron captures given a typical reactor neutron spectrum (Kennedy, 1981; Hohenberg and Kennedy, 1981). These large capture cross sections, which exceed scattering cross sections at these energies, and the high concentration of I in KI (1.4×10^{22} /cm³), yield mean capture lengths for resonance neutrons of less than 40 μm , much smaller than the dimensions of KI, or similar, monitors. Neutrons at these resonance energies will be rapidly removed as captures in the outer layers of these monitors occur, producing gaps in the energy spectra at these resonances. This self-shielding reduces the effective capture cross sections for such iodine salts, making them inappropriate as irradiation monitors. This effect does not occur at the ppb concentration levels in meteorites, making meteoritic standards the best choice for irradiation monitors. A second benefit of meteoritic irradiation monitors is that the differences in isochron slopes between the monitor and the sample provide the relative I–Xe age directly without the need of any other measurements or intricate calculations.

Another problem with high concentration iodine monitors is purely experimental: preparation of accurate iodine standards for isotopic dilution measurements when concentration differences are so extreme and adequate mixing for proper isotope dilution. Typically, atmospheric Xe, with its known isotopic composition and concentration, is used for isotopic dilution. The

amount of $^{128*}\text{Xe}$ produced in the irradiation is then determined by measuring the $^{128}\text{Xe}/^{132}\text{Xe}$ ratio in a mixture containing a known amount of atmospheric Xe. The atomic ratio of $^{128*}\text{Xe}$ and number of iodine atoms irradiated, given by the weight of the iodine salt, provides the neutron capture probably of ^{127}I in that irradiation. For an irradiation of $2 \times 10^{19} \text{ n/cm}^2$ the neutron capture probability is $\sim 10^{-5}$ if a neutron capture cross section for ^{127}I of about half a barn is used (ignoring self-shielding by resonance captures). One milligram of KI (about the smallest practical monitor), irradiated with $2 \times 10^{19} \text{ n/cm}^2$, will produce about 3×10^{13} atoms of $^{128*}\text{Xe}$ (actually twice this much when resonance captures are included). A comparable amount of atmospheric ^{128}Xe for proper isotopic dilution would require an air spike of about 50 cm^3 STP. Even after removal of chemically active species, the gas pressure is high enough to be in the viscous, rather than the molecular flow regime, making complete mixing a serious problem. Although careful mixing by repetitive exchange between two activated charcoal fingers, by sequential application of liquid nitrogen and heating, was used to obtain reproducible values for initial iodine in Bjurböle of $(^{129}\text{I}/^{127}\text{I})_0 = (1.095 \pm 0.029) \times 10^{-4}$ (Hohenberg, 1968; Hohenberg and Kennedy, 1981) that value is clearly too low due to self-shielding in the KI monitor. Although the high pressures involved could have been alleviated, to some extent, by using pure Xe for isotopic dilution, the fact remains that extreme difference in iodine concentration between iodine salts and meteorites makes iodine compounds very poor choices for irradiation monitors in I–Xe dating due to the effects of self-shielding. Errors in the neutron capture probability caused by self-shielding are controlled by the physical size of the KI itself (or other iodine salts) and the neutron energy spectrum. Thus we have no good value for the initial iodine isotopic ratio in Bjurböle, or any other material, but this ratio is not needed for I–Xe dating if a meteorite standard, such as Shallowater enstatite, is used.

An example of the problems is the spectacular result of Lewis and Anders (1975) in which the apparent I–Xe ages of Orgueil and Murchison magnetites required them to form earlier than Allende CAIs, among the first solar system condensates. From this irradiation, the initial $^{129}\text{I}/^{127}\text{I}$ ratios in Orgueil and Murchison magnetites were determined to be 1.451×10^{-4} and 1.465×10^{-4} , respectively, indicating formation some 8.2 Ma before Shallowater enstatite, and 4.3 Ma before condensation of CAIs. To make matters worse Crabb et al. (1982), and others, used the apparent initial iodine $^{129}\text{I}/^{127}\text{I}$ value of 1.465×10^{-4} for Murchison magnetites determined by KI monitor (Lewis and Anders, 1975) as an irradiation monitor for many other samples, creating a large number of published I–Xe ages that were too old

by about 6.2 Ma. This greatly contributed to the mistrust of the I–Xe system since the magnetites are clearly secondary minerals, and other samples calibrated by them, are all unlikely to have formed before the refractory condensates.

Fig. 1 shows a comparison of the I–Xe isochrons from the Shallowater meteorite, used as the irradiation standard, and a magnetite separate from the Orgueil meteorite (Pravdivtseva et al., 2003c). In fact, this magnetite was separated following the exactly same procedure used by Herzog et al. (1973) and Lewis and Anders (1975). In Fig. 1 the relative age, given by the difference in isochron slopes, places Orgueil magnetite 1.8 ± 0.3 Ma older than Shallowater enstatite, thus younger than the CAIs by 2.1 Ma. The Shallowater absolute I–Xe age uncertainty of ± 0.4 Ma is mostly due to uncertainties in its age normalization, not the precision of its isochron.

4. Verification of the I–Xe chronometer

The inherit precision of apparent I–Xe ages is determined by the uncertainties of isochron slopes, and is often less than 100,000 years, so the I–Xe chronometer has the potential of resolving fine details of meteorite evolution. Instead, it has brought frustration because the resulting ages did not generally correspond to the record established by chemical/petrographical studies of meteoritic minerals (Kennedy, 1981). The I–Xe system was consequently viewed by most researchers as unsuitable for chronology, perhaps due to isotopic heterogeneity in the early solar nebula (Jordan et al., 1980; Crabb et al., 1982) or due to “fossil” ^{129}Xe brought into the solar system on pre-solar grains, “stardust” (Clayton, 1975). However, it is hard to imagine how either could have occurred for an element as volatile as iodine. The required heterogeneity is far greater than that found in oxygen, which exists both in refractory and gaseous phases and the $^{129*}\text{Xe}$ is, in fact, derived from the decay of live ^{129}I since it is intimately associated with stable ^{127}I as demonstrated by the linear isochrons. However, with the skepticism already in place, proof of viability and interpretability of I–Xe as a chronometer must be provided.

To effectively test the I–Xe system as a chronometer, comparison with another chronometer is required. One first has to identify a chronometer that is comparable in precision and then find a meteoritic mineral that can be dated by both methods. Even this may be risky, because there is no guarantee that both systems closed at the same time. The Pb–Pb system has been demonstrated as a chronometer with high levels of precision by successfully developing the “onion-skin” model of ordinary chondrites (Göpel et al., 1994),

illuminating small differences in ages among petrographic types. Simultaneous closure of Pb–Pb and I–Xe systems seems assured only if iodine were present as a structural member of the mineral. The logical choice seemed to be chlorapatite, where uranium would follow the calcium, and iodine the chlorine. However, Ca-phosphates occur in two types, apatite, containing the halogens, and merrillite, and these are difficult to separate except visually in a tedious grain-by-grain process.

The Acapulco meteorite contains several percent phosphate and many grains are large enough to be separated by hand. Moreover, Acapulco phosphates have been dated by Pb–Pb, though with larger errors than phosphates from other meteorites (Göpel et al., 1994). Samples of phosphates from Acapulco were separated both magnetically, where merrillite and apatite could not be distinguished, and by hand, where a number of the larger grains were identified as merrillite or apatite (Nichols et al., 1994). The phosphate separate, containing both merrillite and apatite, was dated and each of the grains of merrillite and apatite were individually dated, all obtaining I–Xe ages relative to the Shallowater standard. All of the apatites had identical ages within statistical errors of the I–Xe age of the phosphate separate, and the merrillites contained no ^{129}Xe or ^{128}Xe , indicating they never contained ^{129}I , no indigenous iodine, and only superficial contamination by modern ^{127}I (Nichols et al., 1994). Thus, the presence of merrillite in the magnetic separate served only to dilute the iodine concentration, not change the I–Xe age. Therefore, it is not necessary to separate merrillite from apatite; a simple magnetic separation is all that is required, at least in Acapulco. Apatite from Acapulco formed 8.8 ± 0.2 Ma after the Shallowater enstatite reference mineral (Nichols et al., 1994; Brazzle et al., 1999). This set the stage for a detailed comparison between I–Xe and Pb–Pb ages for phosphates from a large number of meteorites.

Phosphate separates from several ordinary chondrites were irradiated at MURR, along with the Shallowater standard, and results, reported by Brazzle (1997), Brazzle et al. (1997, 1999), and Hohenberg et al. (1998a, b), demonstrate concordancy between the I–Xe and Pb–Pb systems. Moreover, feldspar separates, whose concentration of ^{129}Xe was generally greater than that of the phosphate separates, also formed a convincing correlation with the Pb–Pb ages of phosphates, though the ages were slightly offset, presumably reflecting differences in the closure times of phosphate and feldspar in the same meteorite (Brazzle et al., 1997, 1999). The convincing agreement between ages obtained by Pb–Pb and I–Xe among phosphate and feldspar separates formed the proof that was needed for the I–Xe method as a chronometer.

5. Absolute I–Xe ages

While I–Xe is a relative chronometer, with no further accumulation of ^{129}Xe since the parent nuclide ^{129}I is now extinct, the Pb–Pb system is an absolute chronometer with ages based upon Pb accumulation from ^{235}U and ^{238}U continuing up to the present time. If the Pb–Pb age of the reference standard is known, the absolute I–Xe age of every mineral whose relative I–Xe age has been measured, is also known.

The traditional problem has been that most iodine host phases contain little uranium and vice-versa. This situation changed once the effort to verify I–Xe began and it was found that apatite frequently contains both iodine and uranium. The age of Acapulco phosphate was determined by Pb–Pb and the relative age of individual Acapulco apatites to the Shallowater reference sample was determined by I–Xe dating of individual grains, providing absolute I–Xe ages (Nichols et al., 1994). The absolute I–Xe age of Shallowater enstatite reference was then refined using the added precision of the entire phosphate separate once it was determined that the presence of merrillite did not affect the observed I–Xe ages. The Pb–Pb age of Acapulco phosphate of 4557.0 ± 2.0 Ma (Göpel et al., 1994), and the I–Xe age of the Acapulco phosphate separate relative to Shallowater provided the necessary normalization, yielding an absolute age of the Shallowater enstatite reference of 4566 ± 2.0 Ma. The uncertainty here is almost entirely due to the uncertainty of the Pb–Pb age of Acapulco phosphate.

In addition to pure mineral phases, material that has been rapidly quenched without post-formational alteration also provides samples where I–Xe ages can be readily interpreted. This assumption lead to the earlier I–Xe studies of chondrules from meteorites of low metamorphic grade (Caffee et al., 1982; Swindle et al., 1983, 1991b). Later, a number of individual chondrules were split and dated by both I–Xe and Pb–Pb. In case of Richardton H4 chondrules (Pravdivtseva et al., 2002a, b), both techniques probably dated pyroxenes. However, it was discovered that the absolute I–Xe ages of two Richardton chondrules that were dated by both techniques were a few million years older than the Pb–Pb ages when using the previous Shallowater value (Fig. 2), suggesting that the absolute normalization of the I–Xe chronometer might be slightly in error. A subsequent I–XeI–Xe and Pb–Pb study of Elenovka chondrules strongly supported this observation, suggesting a refined absolute age for Shallowater of about 4563.1 Ma (Pravdivtseva et al., 2004a). Gilmour et al. (2006, 2007) integrated data from a large number of samples and techniques for an even more convincing test of I–Xe. This led to a best-fit normalization for absolute I–Xe ages, now based on a variety of chronometers (Mn–Cr, Al–Mg, Pb–Pb), and an expanded data base,

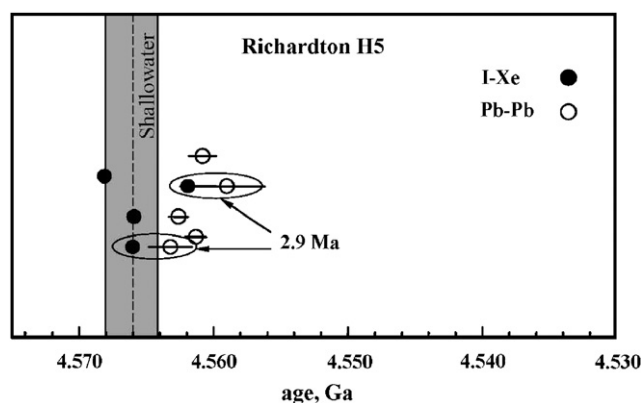


Fig. 2. Absolute I–Xe and Pb–Pb ages of chondrules from the Richardton H5 chondrite. Circled data points represent I–Xe and Pb–Pb ages measured in the same chondrule.

leading to a refined Shallowater enstatite age of 4563.3 ± 0.4 Ma. This current best Shallowater age forms the basis for absolute I–Xe ages whose accuracies can now be computed with confidence from the precision of the relative I–Xe isochrons and relative neutron fluences.

6. Recent results of I–Xe chronometry

As was previously mentioned, the only way for chronometric data to have a meaningful interpretation is to know what mineral phase is being analyzed. This is particularly true for I–Xe, where the host mineral is typically secondary, as was emphasized during discussion of its historical problems. In case of the carbonaceous chondrites Kaba, Bali and Mokoia, the I–Xe chronometer was able to date a range of secondary alteration processes. Samples from these meteorites were crushed and the highly magnetic fraction was separated with a hand magnet. Two nonmagnetic fractions were then hand-picked and tentatively identified by EDS as enstatite and mixtures of plagioclase-rich mesostasis and Al-rich phyllosilicates. Most mineral fractions yielded I–Xe isochrons (shown for Kaba in Fig. 3). Considering the very fine scale of primary and secondary mineral intergrowth in CV chondrites, it is difficult to expect good mineral separation. Nevertheless, I–Xe ages of Kaba and Bali enstatites are the oldest, Kaba at 4565.3 ± 0.8 Ma and Bali at 4565.4 ± 0.7 . I–Xe system in magnetic fractions closed at 4559.1 ± 0.3 Ma in Kaba and 4554.4 ± 0.2 Ma in Bali, and in phyllosilicates even later at 4554.4 ± 0.7 Ma in Kaba and 4554.3 ± 0.9 Ma in Bali (Pravdivtseva et al., 2001). The absolute I–Xe ages for these samples are obtained by adding the Shallowater enstatite age of 4563.3 ± 0.4 Ma to the relative ages. This yields 4565.4 Ma for the oldest, Kaba enstatite, and 4554.3 for the youngest, Bali phyllosilicates, so the

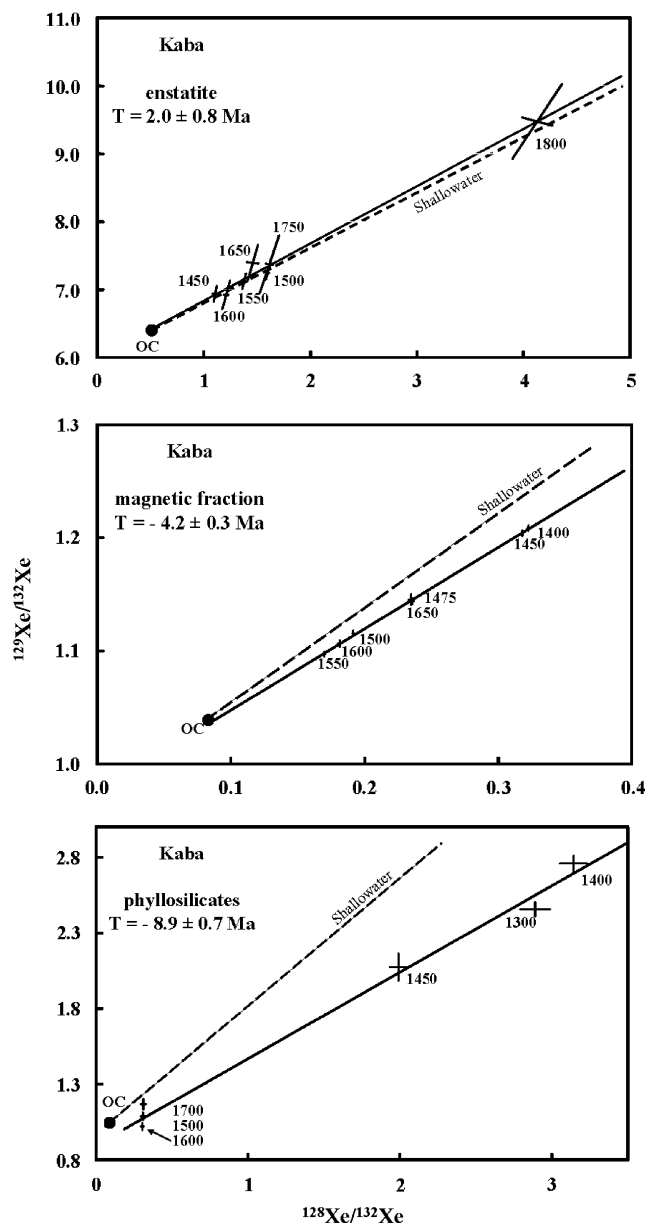


Fig. 3. I–Xe ages of different mineral fractions, separated from the Kaba CV chondrite.

collective I–Xe data from these samples suggest that aqueous alteration on the CV parent body lasted at least 10 Ma.

I–Xe studies of dark inclusion from CV meteorites also lead to similar conclusions. Krot et al. (2001) proposed that rimmed dark inclusions in Allende experienced at least two stages of aqueous alteration. The first stage took place in an asteroidal setting, the second stage *in situ* during the alteration in the Allende host. I–Xe studies of 17 Allende dark inclusions yield well-defined I–Xe isochrons with absolute ages ranging from 4563.8 ± 0.4 to 4566.1 ± 0.4 Ma. These ages are interpreted as first stage aqueous alteration of the dark inclusions, prior to incorporation into the parent body.

The second stage of aqueous alteration most probably is revealed in the I–Xe ages of three Allende CAIs, more precisely, the sodalite contained within them, of 4560.2 ± 0.2 , 4560.3 ± 0.2 and 4559.6 ± 0.2 Ma (Pravdivtseva et al., 2003a). Dark inclusions in the reduced CV chondrites Efremovka, Leoville and Vigarano experienced varying degrees and styles of aqueous alteration (Krot et al., 1999). This is reflected in their I–Xe ages (Swindle et al., 1998; Pravdivtseva et al., 2003b), with Efremovka E53 being 4568.2 ± 1.8 Ma and Vigarano and Leoville much younger: the I–Xe system in Leoville LV2 closed at 4553.8 ± 2.3 Ma. Overall, the I–Xe ages of these phases in CV dark inclusions span ~ 14 Ma, also suggesting a long period of aqueous alteration in the CV parent body (Krot et al., 2006), in agreement with conclusion drawn from the I–Xe studies of mineral phases from Kaba and Bali.

I–Xe studies of chondrules sometimes yield more questions than answers. The iodine host phases are difficult to identify when the chondrules are small and the separation of pure mineral phases is not possible. In these cases the interpretation of I–Xe ages is difficult, since chondrules potentially contain multiple iodine-carrier phases with different closure times. Nevertheless, age information from different mineral components in chondrules or other complex samples can be successfully resolved if, during step-wise heating, radiogenic xenon from different hosts is released at significantly different temperatures, resulting in multiple isochrons. Among the 43 LL chondrules studied to date (Swindle et al., 1991a,b; Gilmour et al., 1995; Pravdivtseva et al., 2004b, 2005a), 10 have yielded two distinct isochrons, with higher temperature releases corresponding to the older apparent I–Xe ages, probably related to chondrule formation in the lower metamorphic grade chondrules. Lower temperature I–Xe ages generally reflect post-formational alteration. Not only do LL chondrules tend to be younger with increasing metamorphic grade, but the spread between high- and low-temperature I–Xe ages increases also, suggesting longer or multiple alteration events for higher metamorphic grade LL meteorites (Fig. 4). Within each type the I–Xe ages of chondrules from the higher metamorphic grade ordinary chondrites tend to be younger, indicating longer post-formational processing or multiple secondary alteration events (Caffee et al., 1982; Pravdivtseva et al., 2002a, b, 2004a, 2005b, 2006a, b).

The absolute I–Xe ages of the oldest chondrules from ordinary chondrites cluster tightly at 4565.5 ± 1.8 and 1.8 ± 1.8 Ma after the formation of CAIs (Pravdivtseva et al., 2006b). Qualitatively similar, though less precise, results were obtained for ordinary chondrites (Gilmour et al., 2000) and for chondrules from enstatite and carbonaceous chondrites (Whitby et al., 2002, 2004). This age interval is consistent with the time of the chondrule forming event derived from Pb–Pb (Amelin

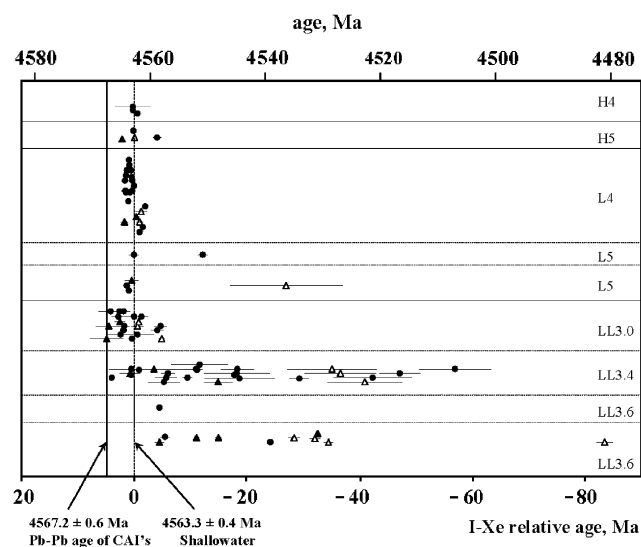


Fig. 4. I–Xe ages of chondrules from ordinary chondrites. Triangular symbols represent I–Xe ages from chondrules with two iodine carriers: solid symbols – high temperature isochron ages, open triangular symbols – low temperature ages: H4 – NWA267 (Pravdivtseva et al., 2005b); H5 – Richardton (Pravdivtseva et al., 2002a, b); L4 – Bjurböle (Caffee et al., 1982; Pravdivtseva et al., 2006b); L5 – Saratov, Elenovka (Pravdivtseva et al., 2004a, 2006a); LL3.0 – Semarkona (Swindle et al., 1991b); LL3.4 – Chainpur (Swindle et al., 1991a; Holland et al., 2005); LL3.6 – Parnallee, Antarctic meteorite (name under consideration) (Gilmour et al., 1995; Pravdivtseva et al., 2004b, 2005a).

et al., 2002, 2004) and Al–Mg chronometry (Hutcheon and Hutchison, 1989; Kita et al., 2000; Srinivasan et al., 1999; Huss et al., 2001; Mostefaoui et al., 2002; Bizzarro et al., 2004; McKeegan and Davis, 2004). Therefore, based on high-precision Pb–Pb ages for CAI's and chondrules from various carbonaceous chondrites, chondrule formation began simultaneously with CAI's, or shortly thereafter, and lasted for at least 4 Ma.

7. Cooling rates

If closure times and closure temperatures of different minerals from a given object can be found, cooling rates are established. While I–Xe and other chronometers can determine closure times, it is much more problematic to know the temperature of closure. However, progress is being made.

The IAB iron meteorites have complicated textures and chemical-mineralogical features that indicate the parent body had a complex history. These meteorites commonly contain abundant silicate inclusions embedded in the metal phase. In an attempt to develop the least complicated formation scenario, Benedix et al. (2000) explored a hypothesis in which the differentiation

process on the parent body is interrupted by a catastrophic impact. The subsequent gravitational re-assembly mixes together lithologies from different parts of the parent body. Although this hypothesis does not completely explain all properties of these meteorites, it does explain the emplacement of solid silicates within molten metal as well as the slow cooling rates (<700 K/Ma) of the reassembled debris.

The ^{129}Xe in chlorapatite, pyroxene, and perryite grains from a graphite-troilite nodule of Toluca is dominated by ^{129}Xe from ^{129}I decay (Pravdivtseva et al., 2008). Pyroxenes in this nodule exhibit a clear correlation between the Mg/(Fe+Mg) ratio and the radiogenic ^{129}Xe concentration, with the highest concentration and the oldest I–Xe ages in the high-Mg grains. The high-Mg grains closed 6.1–10.8 Ma earlier than the low-Mg pyroxenes (Fig. 5). To arrive at closure temperatures, one observation is the temperature in which Xe is released during laboratory extraction, the bulk of which occurs upon melting at 1830 and 1419 K,

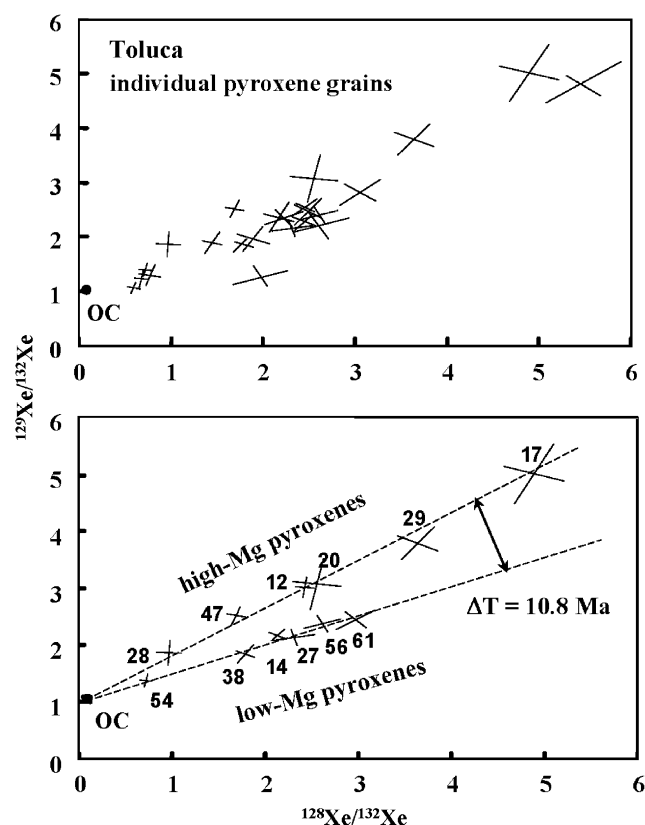


Fig. 5. Three isotope plots for individual pyroxene grains (indicated by number), separated from a Toluca IAB graphite-rich troilite nodule. Because of the presence of fission-produced Xe, the best isotope of normalization would have been ^{130}Xe . However, since these were very small grains, the lower abundance of ^{130}Xe compromises the precision, so we normalize to ^{132}Xe after correction for fission contributions, based upon the observed $^{134}\text{Xe}/^{132}\text{Xe}$ ratio. This introduces correlated errors which show up here as oblique error bars.

respectively, for the high- and low-Mg grains. It is clear that closure, occurring over millions of years, must occur at a much lower temperature than laboratory extraction, so the observed melting temperatures cannot be directly compared with closure temperatures. However, it is the difference in closure temperatures between these two phases that is important, not the values themselves. The difference in melting temperatures for these phases is 411 K, which provides an upper limit for the difference in closure temperatures. This can be compared with the difference in *tammann* temperatures (the temperature at which self-diffusion begins (Tammann, 1926)) for enstatite and ferrosilite of 214 K, which provides a lower limit. With only a factor of 2 between upper and lower limits in closure temperature, a cooling rate for the temperature range of Xe closure in Toluca pyroxenes can thus be calculated to be $\sim 40 \pm 20$ K/Ma. This cooling rate is in a good agreement with the metallographic cooling rate values reported for 7 IAB meteorites (Herpfer et al., 1994), where long extrapolations from laboratory diffusion models are similarly problematic.

The reason that cooling rates can be estimated using laboratory extraction temperatures is that it is the difference in temperatures, not the temperatures themselves that, along with the closure times, that provides the cooling rates. In Fig. 6, we show I–Xe data for Colomera feldspar (Pravdivtseva et al., 2000). Model mixing lines for the 1250–1500 °C extractions systematically increase in slope, hence in model age, approaching the linear isochron defined by the fractions 1550 °C

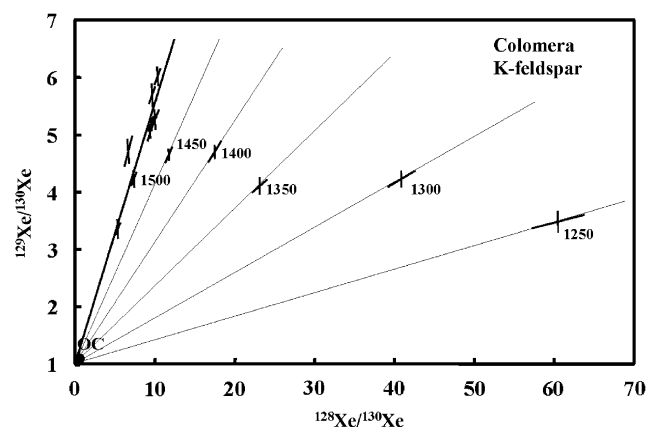


Fig. 6. Three isotope correlation plot for Colomera feldspar along with the Shallowater standard. The higher temperature points (1550–1900 °C) form an I–Xe isochron while the lower temperature points suggest systematic cooling. Xe isotopic ratios were measured with respect to ^{132}Xe . Because of the presence of fission-produced Xe, these ratios are renormalized to ^{130}Xe by multiplying by the $^{132}\text{Xe}/^{130}\text{Xe}$ value, thus a correlated error component is introduced, resulting in the oblique error bars shown. With ^{130}Xe the smallest and least precise isotope, the error bars inherit a larger correlated uncertainty toward the origin.

and above. If one assigns closure time intervals by the difference in slopes of successive lines and approximate closure temperature intervals from the differences in the extractions temperatures, the apparent cooling rates decrease monotonically from 16.8 °C/Ma for the 1500–1550 °C interval to 3.9 °C/Ma for the 1300–1350 °C interval. If grain geometry is limited to simple models, diffusion coefficient D_0/a^2 (the frequency factor normalized to the square of effective diffusion length) and the activation energy can be estimated from the Arrhenius plots (Dodson, 1973). These values are clearly model dependent, and the assumptions on which this diffusion model is based are probably not correct in detail. However, within the context of uniform diffusion models, these parameters allow us to estimate closure temperature intervals for each extraction temperature interval. In both cases, the closure temperature interval is about a factor of two less than the extraction temperature interval. Due to the logarithmic nature of the relationship, these closure temperatures are not very sensitive to either cooling rates, or the chosen geometry of diffusion, giving an 8.4 °C/Ma cooling rate for the higher temperature interval and 2 °C/Ma for the lower temperature interval. Approaching the isochron, the cooling rate of Colomera feldspar is thus estimated to be 12 ± 6 °C/Ma for the temperature range 1500–1550 °C and 3 ± 2 °C/Ma for the 1300–1350 °C interval, the values based on the average between model closure temperatures, which probably is an underestimate of true closure temperatures and laboratory extraction temperatures, which are certainly upper limits.

Data from Allende and Bjurböle chondrules, and some many mineral separates, also display systematic trends in the $^{129}\text{Xe}/^{128}\text{Xe}$ ratios in step-wise heating for successive fractions released just before the data settle onto the well-defined isochron. This too suggests regular cooling. With the observation that the difference in extraction temperatures between successive steps may be within a factor of two of the difference in closure temperatures, a cooling rate of the chondrule or phase can be plausibly estimated.

8. Conclusions

The I–Xe system has proven itself by direct comparison with other chronometers, a confirmation process no other chronometer had to endure. However, in the case of I–Xe, this was prudent here because the early database for whole-rock samples was largely misinterpreted. Moreover, these thorough intercomparisons led to a precise absolute age for Shallowater enstatite, the most widely used irradiation monitor, and provided a sound basis for absolute I–Xe ages which could be directly compared with chronologies derived from other

systems. The source of early interpretational difficulties, the secondary nature of many iodine hosts, has turned out to be one of the great assets of I–Xe dating, providing a detailed history of post-formational evolution. Studies of minerals with different closure times can establish cooling rates or, if non-thermal, the rates for relaxation of post-formational metamorphic processing. Only in extreme cases do all fractions released on step-wise heating fall on the I–Xe isochron. Typically early fractions, those released from the less retentive sites, have suffered diffusive losses. When applied to single-mineral systems this means that, while the isochron itself provides I–Xe ages, defined by closure of iodine-bearing phases, the approach to the isochron during step-wise heating involves mineral sites where diffusive losses have occurred. To the extent that those losses can be approximated by diffusion theory, cooling rates may be estimated from model ages and approximate closure temperatures derived from diffusion models. It is clear that while exploitation of the I–Xe system in the study of early solar system evolution is still in its infancy, we now have a new tool with which to study an old problem.

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