Meteoritics & Planetary Science 41, Nr 1, 19–31 (2006)
Abstract available online at http://meteoritics.org

The I-Xe chronometer and the early solar system

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(Received 31 January 2005; revision accepted 05 August 2005)

Abstract–We review the development of the I-Xe technique and how its data are interpreted, and specify the best current practices. Individual mineral phases or components can yield interpretable trends in initial $^{129}I/^{127}I$ ratio, whereas whole-rock I-Xe ages are often hard to interpret because of the diversity of host phases, many of which are secondary. Varying standardizations in early work require caution; only samples calibrated against Shallowater enstatite or Bjurböle can contribute reliably to the emerging I-Xe chronology of the early solar system.

Although sparse, data for which I-Xe and Mn-Cr can be compared suggest that the two systems are concordant among ordinary chondrite samples. We derive a new age for the closure of the Shallowater enstatite standard of 4563.3 ± 0.4 Myr from the relationship between the I-Xe and Pb-Pb systems. This yields absolute I-Xe ages and allows data from this and other systems to be tested by attempting to construct a common chronology of events in the early solar system.

Absolute I-Xe dates for aqueous and igneous processes are consistent with other systems. Consideration of the I-Xe host phases in CAIs and dark inclusions demonstrates that here the chronometer records aqueous alteration of pre-existing material. The ranges of chondrule ages deduced from the Al-Mg and I-Xe systems in Semarkona (LL3.0) and Chainpur (LL3.4) are consistent. Chainpur I-Xe data exhibit a greater range of ages than Semarkona, possibly reflecting a greater degree of parent body processing. However individual chondrules show little or no evidence of such processing. Determining the host phase(s) responsible for high temperature correlations may resolve the issue.

INTRODUCTION

The underlying principle of I-Xe dating is as follows. ^{129}Xe produced from decay of now extinct ^{129}I is quantitatively retained at iodine sites in a host mineral after isotopic closure. The present-day $^{129*}Xe/I$ ratio represents the $^{129}I/^{127}I$ ratio upon closure of the host mineral to xenon loss. The uniformity of this initial iodine ratio, and its application as a chronometer, is confirmed by means of linear isochrons similar to those provided by other radioisotope dating systems.

In this paper we review developments in the technique since the discovery that ^{129}I was present in the early solar system and outline the state-of-the-art of I-Xe analysis, pointing out the most reliable techniques in practice today. We also compare I-Xe ages with ages derived from other chronometers, such as Pb-Pb and those based upon other extinct radionuclides, and derive a new absolute age for the Shallowater standard.

Excess ^{129}Xe ($^{129*}Xe$ hereafter) in primitive meteorites was first identified by Reynolds (1960a, 1960b). By using neutron irradiation (which converts stable ^{127}I to ^{128}Xe) Jeffery and Reynolds (1961) were able to demonstrate that these excesses correlated with iodine in stepped heating experiments, proving that the $^{129*}Xe$ was produced by the in situ decay of ^{129}I incorporated in iodine-bearing minerals of the host meteorite. This was the first evidence for the presence of an extinct radionuclide in the early solar system (Reynolds 1963). It also provided the first potential chronometer with resolution high enough to study the sequence of events that occurred during the formation of our solar system and the subsequent reprocessing of solar system material that occurred shortly thereafter.

TECHNICAL ISSUES OF I-Xe DATING

I-Xe dating requires determination of the $^{129}I/^{127}I$ ratio that was present in an iodine host mineral on closure to xenon loss. The technique used here is similar to the methodology of

Ar-Ar dating (which was, in fact, inspired by the I-Xe technique) in that neutron irradiation is used to convert a proportion of stable ^{127}I to ^{128}Xe by neutron capture: ^{127}I (nγ, β) ^{128}Xe. Refined by Fish and Goles (1962), this method requires only measurement of the isotopic ratios of a single element, which can be done with great precision. It shares this feature with Ar-Ar dating. ^{129*}Xe is produced by ^{129}I decay in the early solar system, while ^{128*}Xe (denoting iodine-derived) is produced by neutron irradiation of the sample in a reactor. The ^{129*}Xe/^{128*}Xe ratio is thus proportional to the ^{129}I/^{127}I ratio present in the iodine host mineral at the time of isotopic closure. To obtain a quantitative I-Xe age two critical requirements must be met. The relationship between ^{127}I and neutron-produced ^{128*}Xe must be quantified. And the value of the ^{129*}Xe/^{128*}Xe ratio and its uniformity among iodine-bearing sites must be determined.

In practice, not all of the iodine present in a sample exhibits the same ^{129*}Xe/^{128*}Xe ratio. Step-wise heating (often referred to as step pyrolysis) is used to order iodine-bearing sites by means of their thermal properties. The irradiated sample is heated through a sequence of increasing temperature steps, with the xenon evolved at each step analyzed separately. Usually, low-temperature extractions exhibit lower ^{129*}Xe/^{128*}Xe ratios than higher temperature extractions. This reflects either loss of ^{129*}Xe from the less retentive iodine-bearing sites, or contributions from superficial contamination of these sites with modern terrestrial iodine. In many samples a constant ^{129*}Xe/^{128*}Xe ratio is obtained in the higher temperature extractions, reflecting a constant ^{129}I/^{127}I ratio at isotopic closure in these more retentive sites. In disturbed samples, a constant ^{129*}Xe/^{128*}Xe ratio may not be observed and precise chronological information cannot be obtained. Some samples show progressively increasing ^{129}I/^{127}I ratios with increasing extraction temperatures before a constant (isochron) value is observed, with possible implications for slow cooling. We now discuss the detailed theoretical and practical aspects of I-Xe dating as currently practiced.

Irradiation

Typical neutron irradiations using fluxes of 7×10^{13} n cm^{-2} s^{-1} and integrated fluences of a few $\times 10^{19}$ n cm^{-2} lead to ^{127}I conversion efficiencies of the order of 10^{-4}. This is chosen to be a good match to the initial solar system ^{129}I/^{127}I ratio of ~10^{-4}, ensuring similar ^{129*}Xe and ^{128*}Xe ion signals during analysis. Both thermal and epithermal neutrons contribute to the production of ^{128*}Xe during the irradiation since neutron capture resonances are prominent for iodine in the energy ranges found in typical reactor neutron spectra. Contributions from these resonance captures are estimated to be about 50% of the total production (Hudson 1981; Kennedy 1981), so direct calculation of the conversion efficiency is impractical. Conversion of ^{127}I to ^{128}Xe can be directly determined using irradiation monitors of iodine-bearing compounds from which the resulting ^{128*}Xe is measured, but this leads to the significant problems detailed below.

To avoid these problems, iodine host minerals from a "standard" meteorite are irradiated along with the samples. This sample is used to directly determine the relative I-Xe ages and, once calibrated, becomes a standard from which to compute the absolute I-Xe ages and the initial ^{129}I/^{127}I ratios. Minor variations in conversion efficiency due to fluence differences across the sample container can be minimized by placement of the samples in a fixed vertical plane and rotation of the container during the irradiation. Moreover, any variations that may persist can be detected by cobalt-doped aluminum flux wires included all around the perimeter of the sample plane. Samples from separate irradiations can be compared because each irradiation is calibrated by the standard meteorite so that relative I-Xe ages, absolute I-Xe ages, and the inferred initial iodine are independent of the specific neutron capture efficiencies.

A variety of meteorite standards have been used in the past including Bjurböle whole rock and Shallowater enstatite. The use of pure compounds, such as KI, and CI magnetite calibrated with KI monitors (Lewis and Anders 1975) introduce additional complications that appear to have led to misleading results when compared with I-Xe ages based upon the more direct meteoritic standards (Hohenberg et al. 2000).

Use of a meteorite sample as an irradiation monitor has the distinct advantage that the differences in closure times can be determined directly from the measured differences in the ^{129*}Xe/^{128*}Xe ratios alone, independent of the ^{127}I capture probability. When the same meteorite standard is used in separate irradiations, the principle can be generalized in a straightforward manner so that the relative closure times of all samples can be determined independent of the specific irradiation or the specific capture probability. In contrast, if an iodine compound such as KI is used as a monitor, the absolute capture probability must be determined. This depends, in turn, on knowledge of the number of moles of parent iodine present and precise measurement of the ^{128*}Xe daughter. However, the ^{128*}Xe produced from macroscopic quantities of KI vastly exceeds the dynamic range of noble gas mass spectrometers. A 1 mg sample of KI produces ~10^{-5} cm^3 STP ^{128*}Xe in a typical irradiation, whereas a Xe sample of 10^{-13} cm^3 STP is typical of many meteoritic measurements. Reducing the released ^{128*}Xe to the range where it can be determined with precision in a conventional noble gas mass spectrometer requires extensive dilution, introducing mixing problems and new sources of systematic error. The production of ~50% of the ^{128*}Xe from ^{127}I by resonance captures of epithermal neutrons causes an additional problem. Resonances are typically narrow in energy, which raises the possibility of self-shielding for samples so rich in iodine that neutrons have little chance of energy transfer by scattering between potential captures. Typical meteorite samples are at most a few parts

per million iodine and thus do not suffer from self-shielding effects. Therefore, a monitor such as KI can introduce serious systematic offsets leading to underestimation of the production of ^{128*}Xe and hence to anomalously early apparent ages (Hohenberg et al. 2000). For these reasons, iodine compounds are generally not suitable for use as monitors, especially for direct comparison of I-Xe ages. In Figs. 1 and 2 we illustrate the reproducibility and the consistent high quality isochron that have led to the adoption of Shallowater enstatite as the preferred irradiation monitor for I-Xe experiments. Variations in neutron fluence can also be assessed by using multiple aliquots of the standard in each irradiated package.

One irradiation of Bjurböle included KI monitors. After a set of careful dilutions this yielded an apparent ^{129}I/^{127}I ratio of 1.125×10^{-4} in Bjurböle (Hohenberg and Kennedy 1981). Subsequent experiments in which Bjurböle and Shallowater were irradiated together led to the adoption of ^{129}I/^{127}I = 1.072×10^{-4} for Shallowater enstatite (Nichols et al. 1994). These nominal values are subject to all the caveats associated with KI outlined above, but this is of no consequence as long as they are used exclusively and consistently to derive nominal ^{128*}Xe/I ratios for I-Xe analyses or nominal values of the initial ^{129}I/^{127}I ratios that will be compared only to other values similarly calibrated. In effect, all data referenced to Shallowater or Bjurböle may be subject to the same systematic offset from their actual ^{129}I/^{127}I ratios but this is irrelevant to I-Xe chronology as long as all samples have been normalized to one of these standards. Data referenced to separate determinations from KI are subject to systematic offsets from data referenced to Shallowater/Bjurböle and so cannot be integrated into the same chronology.

Relative I-Xe ages, and absolute I-Xe ages calibrated by Pb-Pb or other absolute chronometers, are not subject to any of the uncertainties associated with the elusive determination of the actual ^{128*}Xe/I ratios. Until initial iodine in Shallowater or Bjurböle is measured independently, initial iodine isotopic ratios inferred in I-Xe dating should only be used as relative values.

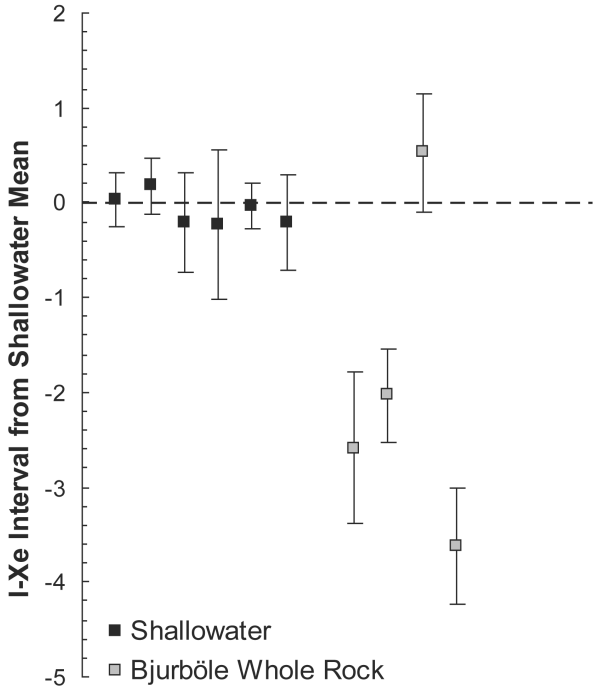

Fig. 1. Comparison of derived I-Xe intervals from analyses of Shallowater enstatite and Bjurböle whole rock (i.e., samples not dominated by one chondrule) from the same irradiation. Typical sample sizes were 0.5–1 mg. The excellent reproducibility of the Shallowater sample contrasts with more variation among the Bjurböle samples. For this reason, Shallowater enstatite is now the preferred standard for calibration of the I-Xe technique.

Recoil

The Ar-Ar technique, which also employs a neutron irradiation to allow determination of the parent element in geological samples, is known to exhibit problems with recoil, especially when fine-grained materials are analyzed. ^{39}Ar is produced by neutron capture from ^{39}K, as is ^{128}Xe from ^{127}I. Although the two processes might seem analogous in that both involve neutron capture, there are major differences that prevent recoil effects from being a problem with I-Xe technique.

Recoil occurs in the K-Ar technique because conversion of ^{39}K to ^{39}Ar is an (n, p) process. This requires capture of a fast neutron and emission of a proton with a minimum energy established by the Coulomb barrier of the nucleus. Momentum conservation results in a relatively high kinetic energy for the recoiling nucleus (tens of keV), sufficient for it to move many lattice spacings. In contrast, none of the processes involved in conversion of ^{127}I to ^{128}Xe (thermal or epithermal neutron capture, gamma, beta, and UV emission) lead to significant momentum in an emitted particle. The recoiling ^{128}Xe nucleus, nearly three-fold more massive than ^{39}Ar, is no more than 3 eV kinetic energy, whereas the activation energy for radiogenic ^{129}Xe in meteorite samples is >3.6 eV (Burkland et al. 1995). Recoil effects will not be an issue with the I-Xe technique and no such effects have ever been observed.

I-Xe Data Reduction

Step-wise heating orders the released Xe in terms of the thermal properties of the host sites. The less retentive Xe sites are sampled in the lower temperature fractions from which losses of radiogenic ^{129*}Xe over the 4.6-billion-year storage time may be significant, or superficial iodine contamination has occurred. This leads to lower measured ^{129*}Xe/^{128*}Xe ratios. Higher temperature fractions represent the more retentive sites. A series of sequentially increasing temperature

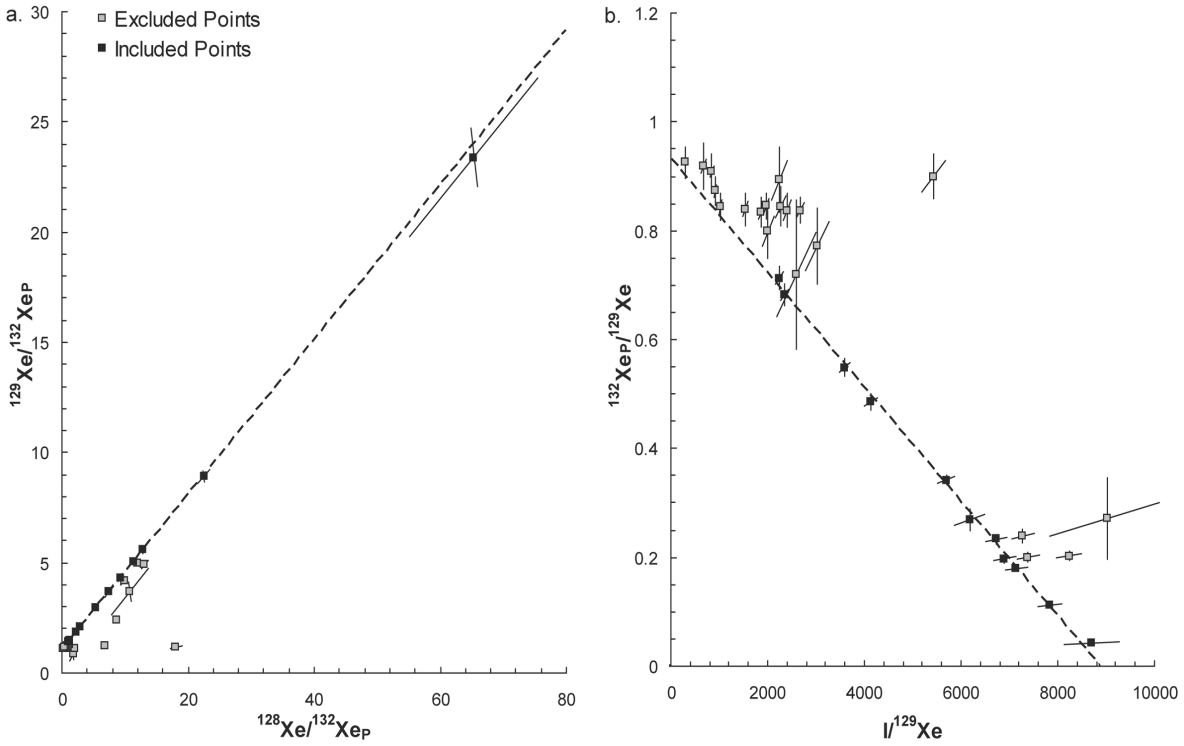

Fig. 2. Data from laser stepped heating of a 1 mg sample of Shallowater enstatite illustrating the high-quality isochron typical of this standard. Errors correspond to the axes of ellipses encompassing the data with 68% confidence. In (a) choice of scale prevents many excluded points from being plotted. Two formally equivalent ways of presenting the data are contrasted. In both cases, isochrons (shown with broken lines) have been fitted to the high temperature releases using the method of York (1969). In (a) the gradient of the isochron (broken line) defines the $^{129*}Xe/^{128*}Xe$ ratio of the high temperature releases from the sample. This can be converted to $^{129*}Xe/^{127}I$ (=$^{129}I/^{127}I$ on closure) using a $^{128}Xe/^{127}I$ conversion factor, though only a ratio of gradients is required to establish an interval. The $^{129}Xe/^{132}Xe$ ratio associated with a trapped (iodine-free) component is defined at the point on the isochron corresponding to the $^{128}Xe/^{132}Xe$ ratio of planetary xenon. In (b), excess ^{128}Xe has been calculated over the planetary $^{128}Xe/^{132}Xe$ ratio and converted to iodine using the nominal $^{128}Xe^*/I$ ratio, for the irradiation of which this sample formed a part (calculated with reference to an irradiated standard). The intercept of the isochron with the horizontal axis is $I/^{129}Xe$ (with no contribution from planetary xenon), while the intercept of the isochron with the vertical axis is $^{132}Xe/^{129}Xe$ (with no contribution from iodine). These are the reciprocals of the quantities determined in (a) (see also Table 1).

steps with constant $^{129*}Xe/^{128*}Xe$ indicate a constant initial iodine ratio at closure of the associated iodine sites. In a given neutron irradiation, iodine-bearing sites in different minerals and different meteorites will have different $^{129*}Xe/^{128*}Xe$ ratios in these stable sites, corresponding to different relative I-Xe ages. Comparisons among different irradiations are made through the common standard meteoritic mineral, such as Shallowater enstatite.

In the past, the different groups contributing to this paper have adopted superficially different means of reducing and presenting I-Xe data. We now demonstrate that these are equivalent. The differences amount to a different sequence of the calculations but yield identical results. In each case, the primary aim is to determine the iodine-xenon age relative to the included irradiation monitor. Sometimes the isotopic composition of the accompanying trapped xenon is also derived. This can be an important indicator of shock which may have disturbed the I-Xe system (Gilmour et al. 2001; Caffee et al. 1982) or effects associated with the trapping process itself (Hohenberg et al. 2004).

In the "St. Louis" approach (Fig. 2a), the ratios of ^{128}Xe, ^{129}Xe to a normalization isotope are displayed as a 3-isotope correlation plot. The measured ^{128}Xe and ^{129}Xe contain mixtures of iodine-derived and normal Xe. Normal Xe, often called "trapped" Xe, "planetary" Xe, "Q-Xe," "OC-Xe" (for ordinary chondrite Xe), or "AVCC" Xe (for average value carbonaceous chondrite) is the ubiquitous and uniform component that dominates the solar system. The choice of ^{132}Xe or ^{130}Xe as the normalization isotope is made to optimize the precision. Although both isotopes are generally dominated by normal Xe, small corrections are sometimes necessary for fission-produced ^{132}Xe or spallation-produced ^{130}Xe, so that the best choice for normalization is dictated by the precision after these corrections are made. Very often the relative contributions from spallation and fission are so small that no corrections are necessary and the measured ratios can be plotted directly.

In such three-isotope correlation plots, after progressing beyond the low-temperature fractions, constant $^{129*}Xe/^{128*}Xe$ ratios are observed. A series of sequential temperature steps

from sites that contained uniform initial iodine, now preserved as a unique $^{129*}Xe/^{128*}Xe$ ratio, form a linear mixing line between trapped Xe and a single iodine-derived Xe component with its distinct $^{129*}Xe/^{128*}Xe$ ratio. The value of this $^{129*}Xe/^{128*}Xe$ ratio is simply the slope of this mixing line, the I-Xe isochron. Knowledge of this ratio in a sample and the meteorite standard is sufficient to obtain the closure interval between the sample and the standard, and the only uncertainty of that age is provided by uncertainties of the slopes of these two isochrons. Any variation of the neutron fluence between the sample and standard is monitored by the Co-Al flux wires and seldom exceeds 1%, corresponding to an uncertainty of about 200,000 yr.

The "Manchester" approach has been to convert excess ^{128}Xe over the nominal planetary composition into an equivalent amount of iodine using the observed $^{129*}Xe/^{128*}Xe$ of the standard and its nominal $^{129}I/^{127}I$ ratio. Ratios among ^{129}Xe, I and the isotope representing "normal" xenon corrected for any spallation of fission contribution are then used to determine the desired quantities. In Fig. 2b, the dependence of $^{132}Xe_P/^{129}Xe$ on $I/^{129}Xe$ is examined ($^{132}Xe_P$ denotes ^{132}Xe corrected for fission). The intercept of the isochron with the ordinate yields the reciprocal of the $^{129}Xe/^{132}Xe$ ratio of trapped xenon, while the intercept with the abscissa yields the reciprocal of the $^{129}Xe/I$ ratio ($^{129}I/^{127}I$ ratio on closure).

The two approaches are equivalent, provided the isochrons are determined with fits that take into account the correlated errors of all operations including correlations in the uncertainties of the ratios plotted. In Table 1 we demonstrate this by comparing the values of the derived quantities for the data of Figs. 2a and 2b.

Measured $^{129*}Xe/^{128*}Xe$ ratios, in principle, provide the $^{129}I/^{127}I$ ratios on closure in the sample if the nominal $^{129}I/^{127}I$ ratio of the standard is adopted. It should be stressed that the initial iodine ratio itself is of no importance for the relative I-Xe ages, or the absolute I-Xe ages found by normalization. It does, however, become important when inventories of short-lived radionuclides in the solar system are compared.

We now consider the chronology the I-Xe system provides for the early solar system and compare it to those based on other radionuclides.

Table 1. Comparisons of derived quantities from the data set of Fig. 2 using the "St. Louis" and "Manchester" methods described in the text.

	$^{129}I/^{127}I$ ($/10^{-4}$)[a]	$^{129}Xe/^{132}Xe$[b]
St. Louis method[c]	1.0735 ± 0.0138	1.073 ± 0.020
Manchester method[c]	1.0737 ± 0.0143	1.074 ± 0.018

[a]Initial iodine ratios calculated relative to the mean of 6 Shallowater monitors included in this irradiation assuming $^{129}I/^{127}I$ for Shallowater = 1.072×10^{-4}.
[b]Derived $^{129}Xe/^{132}Xe$ ratio of iodine-free trapped xenon component.
[c]All errors are 1σ.

I-Xe AND THE CHRONOLOGY OF THE EARLY SOLAR SYSTEM

Early Whole-Rock Studies

While a large number of meteorites had been dated over the years by the I-Xe method, until recently there was no general agreement that I-Xe ages were meaningful; whether the I-Xe system worked as chronometer was even considered doubtful (Jordan et al. 1980; Swindle and Podosek 1988). This was not because the I-Xe ages were discordant with other chronometers, because few, if any, could reliably measure the age intervals of a million years or less determined by the I-Xe method. The problem was rather that, "whole-rock" I-Xe ages seldom seemed to correlate with observable petrographic properties, the enstatite chondrites being an exception (Kennedy et al. 1988). Many reasons were proposed for the lack of such correlations, but the most prevalent concluded that iodine may not have been isotopically homogeneous in the early solar system (Swindle and Podosek 1988; Jordan et al. 1980; Crabb et al. 1982).

On the other hand, inhomogeneities in $^{129}I/^{127}I$ are not expected on theoretical grounds. As a volatile element, iodine should be dominated by the gaseous component of precursor material. Furthermore, in contrast to the more short-lived species (such as ^{26}Al), the observed initial $^{129}I/^{127}I$ ratio in the early solar system of $\sim 10^{-4}$ does not require an admixture of recently synthesized material (Reynolds 1960b; Wasserburg et al. 1960). ^{129}I itself is an r-process-only nucleus and its daughter, ^{129}Xe, has also chiefly been produced via the r-process (i.e., by ^{129}I decay). While the stable isotope of iodine, ^{127}I, is on the s-process track, its proximity to the peak of r-process production means that it too is mostly from an r-process source. Thus, when the solar system formed, the galactic budget of ^{129}Xe, $^{129}Xe_0$, had already been produced from ^{129}I over the lifetime of the galaxy leading to

$$\frac{^{129}Xe_0}{\tau_{Galaxy}} \approx \frac{^{129}I_t}{\tau_{129}} \quad (1)$$

for the average ^{129}I budget of the galaxy, $^{129}I_t$. So $^{129}I/^{129}Xe$, which approximates the $^{129}I/^{127}I$ since $^{127}I/^{129}Xe \sim 1$ (Anders and Grevesse 1989) was $\sim 10^{-3}$, the approximate ratio of the half-life of ^{129}I (15.7 Ma) to the lifetime of the galaxy before the formation of the solar system (~10 Ga). Thus, the observed initial ratio is less than that expected for average galactic material when the solar system formed, an expectation confirmed by more detailed modeling (Meyer and Clayton 2000). It follows that there is little reason to doubt the isotopic homogeneity of iodine in the early solar system. There is even less cause to believe that a uniform value for the initial $^{129}I/^{127}I$ ratio at iodine-bearing sites in minerals should be established by something other than simultaneous closure, and that its variation among different samples caused by anything other than the passage of time. It appears simply implausible for the I-Xe system not to be a chronometer.

The solution to this conflict rested both in improved technology that allowed more detailed investigation of the I-Xe system and in the knowledge of iodine host phases. We now know that many important iodine host phases are post-formational, therefore reflecting secondary processes in the early solar system (Brazzle et al. 1999). It is not clear exactly what closure event(s) were dated by the high resolution, high temperature isochrons observed in many whole rock samples. Consider the "near simultaneous" closure of iodine sites in the 5 chondrites measured by Hohenberg and Reynolds (1969). While the result stands, the differences in closure times of a million years or so between the individual chondrite whole-rock samples cannot be interpreted simply because the phase being dated, and hence the event leading to closure of the I-Xe system, is unknown. The enstatite chondrites are an exception to this uncertainty (Kennedy et al. 1988) because the major iodine carrier is enstatite. Since enstatite dominates the petrology of these meteorites, the correlation observed between the measured whole rock I-Xe ages and their petrographic properties is expected.

Recently, Gilmour (2000) revisited the early whole-rock data and showed that model ages (total ^{129*}Xe/total I) for equilibrated meteorites do tend to correlate with petrologic type within individual groups. In contrast to high temperature isochrons, which may reflect closure of specific iodine host phases, within any meteorite group this ratio could be a rough property of the bulk rock. Whatever the details of the phases responsible for high temperature isochrons, meteorites of higher metamorphic grade were more intensively (or longer) processed, so tend to have retained less ^{129*}Xe.

Final verification of the I-Xe system as a precise and reliable chronometer arrived with direct comparisons between measured I-Xe age intervals and those derived from absolute Pb-Pb ages. Such comparisons are best attempted on single-mineral systems where isotopic closures, the events being dated, are more likely to be related. The view that the I-Xe system provides good chronological information was vindicated when Brazzle et al. (1999) found excellent agreement between the Pb-Pb ages of phosphates, and the I-Xe ages of the phosphates and feldspars separated from 12 different meteorites. In addition, Hohenberg et al. (2000) revisited the anomalously old apparent I-Xe ages obtained when iodine salts were used as neutron monitors. The apparent I-Xe ages of Murchison and Murray magnetites (and other meteorite samples referenced directly or indirectly, to the same monitors) in these earlier studies (Lewis and Anders 1975; Swindle and Podosek 1988), were shown to be too old when compared with data obtained using the Bjurböle/Shallowater standardization. This was due to a systematic offset between I-Xe ages derived using the neutron capture probability obtained from analyses of KI and I-Xe ages obtained from the Bjurböle/Shallowater standard where no capture probabilities are needed. Only data successfully normalized to Shallowater or Bjurböle can be incorporated into the emerging I-Xe chronology.

Four decades after the discovery that ^{129}I was alive in the early solar system, it is now apparent that I-Xe ages, properly measured and standardized, and properly interpreted with regard to the likely iodine host phases, can be successfully applied to the study of meteorite evolution in the early solar system.

Integrating I-Xe into the Wider Picture

We now seek to compare the I-Xe system with those based on other short-lived radioisotopes and on the decay of uranium to lead isotopes. In doing this, we rely almost entirely on the somewhat limited number of meteoritic samples or components in the literature on which determinations of closure time of more than one isotopic system are available. Previously unreported I-Xe ages from two Richardton chondrules are also employed. Of course it is not necessarily the case that two systems determined in the same meteorite, or even in the same mineral within it, have been set or reset by the same event; this must be considered for each candidate data point contributing to the comparison.

Our first objective is to further test the hypothesis that variations of the initial ^{129}I/^{127}I ratios across a range of sample types and across the region of the solar system sampled by meteorites do indeed serve as a chronometer. This will be the case if ^{129}I was homogeneously mixed across this region. We also wish to establish the I-Xe system as an absolute chronometer by calibrating the I-Xe Shallowater standard, to which relative I-Xe ages naturally emerge, against the Pb-Pb time scale.

Absolute calibration of the I-Xe time scale was first attempted by Nichols et al. (1994), who compared the I-Xe age of individual Acapulco phosphate grains with the Pb-Pb age of Acapulco phosphate (4.557 ± 0.002 Ga) (Göpel et al. 1994). Their work established that in Acapulco the Cl-bearing phosphates (apatites) contained all of the iodine and radiogenic ^{129*}Xe, and that the merrillites, which are Cl-free, contained neither. Based on this observation, Brazzle et al. (1999) were able to use bulk phosphate separates (not separated into apatite and merrillite) from Acapulco for comparison of the I-Xe and Pb-Pb systems. Acapulco phosphate was found to be 8.8 ± 0.2 Ma younger than Shallowater enstatite, which led to an absolute age of Shallowater enstatite of 4.566 ± 0.002 Ga, the error arising chiefly from the uncertainty in the Pb-Pb age of Acapulco phosphate. Absolute I-Xe ages for all other samples calibrated against Shallowater could then be obtained and integrated into a time scale of solar system evolution. However, the normalization to obtain absolute I-Xe ages from the measured relative ages is based upon one measurement and is subject to further refinement.

Previous authors (e.g. Gilmour 2000; Gilmour and Saxton 2001; Pravdivtseva et al. 2002; Pravdivtseva et al. 2004) have examined the consistency of the chronometers based upon a few short-lived radioisotopes by accepting

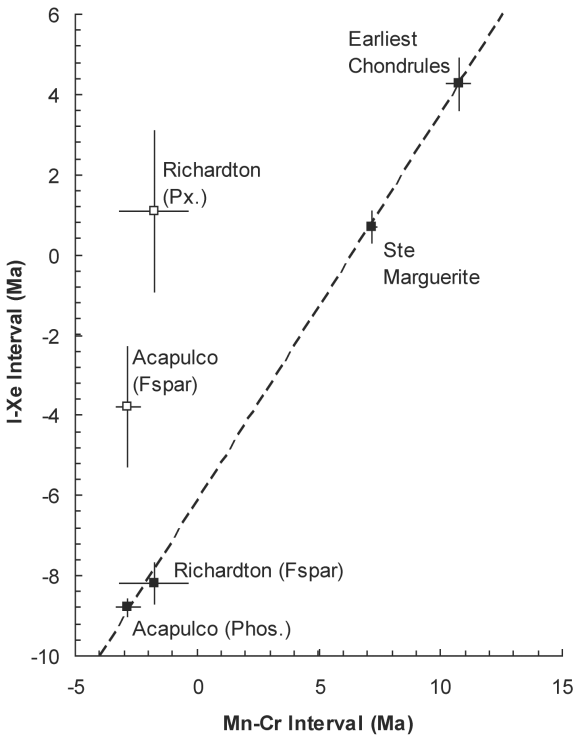

Fig. 3. Comparison of intervals in the I-Xe and Mn-Cr systems. Whole-rock Mn-Cr ages for Ste. Marguerite, Richardton, and Acapulco are compared with I-Xe data from mineral separates (Px-pyroxene; Fspar-feldspar; Phos.-phosphate). Mn-Cr model ages from Semarkona chondrules are also compared with the earliest I-Xe chondrule ages from the same meteorite. The two chronometers appear concordant if Mn-Cr ages approximate the ages of the later I-Xe ages derived from minerals with lower closure temperatures. Error bars are 1σ and data are collated with references in Table 2.

proposed calibrations at face value and constructing proposed chronologies. These chronologies have then been examined and adjustments or alternative calibrations proposed to make interpretations more robust. This led to a proposal that the I-Xe to Pb-Pb calibration based on Acapulco phosphate resulted in slightly (1–3 Ma) early ages. Here we adopt a different procedure. We use relative calibrations for each of the chronometers and plot one chronometer against another and against the Pb-Pb chronometer. This procedure, first applied by Busfield (2004) and Gilmour et al. (2004), is equivalent to plotting logarithms of ratios with the further advantage that, because the natural logarithm of isotope ratios are multiplied by half-lives in the calculation of age intervals, gradient one correlation lines are expected for concordant chronometers. Linear arrays with gradients different from unity would suggest errors in the accepted half lives of the one of the radionuclides, or some other failure in the underlying assumptions. From fits to these linear arrays we obtain conversion algorithms between the chronometers being examined.

We first examine the relationship between the I-Xe chronometer and that based on decay of ^{53}Mn to ^{53}Cr with a half-life of 3.7 Ma. Intervals in the I-Xe system are reported relative to closure of Shallowater enstatite (Hohenberg 1967; Brazzle et al. 1999), while intervals in the Mn-Cr system are reported relative to isotopic closure in the angrite Lewis Cliff (LEW) 86010 (Lugmair and Shukolyukov 1998). Although a large number of samples have been dated in each system, there are few samples that are dated in both systems and can contribute to an examination of their consistency. Variation in initial ^{53}Cr/^{52}Cr ratios have been interpreted as evidence of inhomogeneity in ^{53}Mn/^{55}Mn (Lugmair and Shukolyukov 1998). While this interpretation is disputed it is unclear how to integrate data from enstatite chondrites into our comparison. In contrast, there is no evidence of inhomogeneity in ^{53}Mn abundance among ordinary chondrites (Lugmair and Shukolyukov 2001), so we restrict our comparison to data from these meteorites.

Identical Mn-Cr model ages of chondrules from Semarkona and Chainpur were interpreted as dating isolation of chondrule precursors from a parent reservoir, providing an early limit for the date of subsequent chondrule formation (Nyquist et al. 2001). We thus compare the earliest chondrules ages in the I-Xe system, which are reported from analyses of Semarkona and Chainpur (Swindle et al. 1991a, 1991b), with the Mn-Cr data. Whole rock Mn-Cr data from the ordinary chondrites Ste. Marguerite (H4) and Richardton (H5) (Polnau and Lugmair 2001), and from Acapulco (Zipfel et al. 1996) are also available, and should record the last equilibration of chromium isotopes as these meteorites cooled. I-Xe data from various mineral separates of these meteorites are also available (Brazzle et al. 1999). The Richardton data fall into two groups, minerals with early I-Xe ages and minerals with late I-Xe ages (Pravdivtseva and Hohenberg 1999). We adopt pyroxene as representative of the former and feldspar as representative of the latter. Ste. Marguerite feldspar yields a precise I-Xe isochron, while phosphate has a consistent age within larger errors (Brazzle et al. 1999). Both feldspar and phosphate data are available for Acapulco (Brazzle et al. 1999). In this meteorite, a significant difference in age between phosphate and feldspar exists, whereas in Ste. Marguerite the equivalent mineral separates exhibit identical I-Xe ages within error. This can be understood in terms of the factor of 10 lower cooling rate of Acapulco compared to that inferred for the ordinary chondrite parent body (Bennett and McSween 1996; Pellas et al. 1997).

In Fig. 3 we present these data from the Mn-Cr and I-Xe systems. The two systems appear to correlate provided that the I-Xe age of the minerals recording the latest age is adopted in each case. Thus I-Xe data from Acapulco phosphate and feldspar from Richardton and Ste. Marguerite appear to define a line of concordance with the Mn-Cr system in whole- rock samples. Zipfel et al. (1996) noted that their Mn-Cr age for Acapulco was close to that measured for phosphates in the Pb-Pb system, while Shukolyukov et al. (1994) showed that

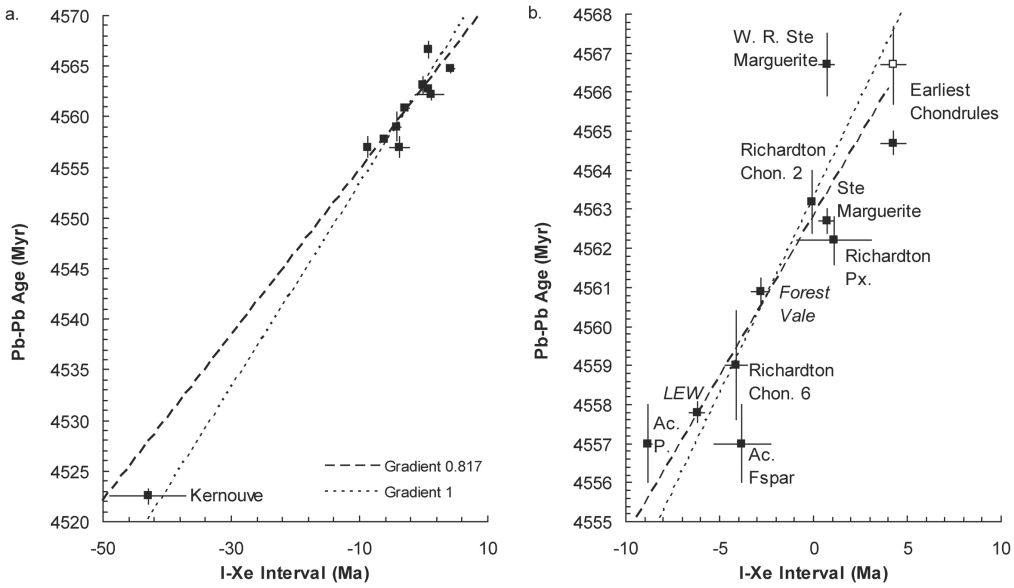

Fig. 4. A comparison of I-Xe intervals with ages recorded by the Pb-Pb chronometer; the region with most details in (a) is shown in expanded scale in (b). The lines drawn correspond to the average intercepts (Shallowater ages) and gradients of the free fit and fit with gradient forced to equal 1 from Table 3. The data points corresponding to "earliest chondrules" relate to the Pb-Pb data from Acfer 059 (Amelin et al. 2002) and Allende (Amelin et al. 2004); see text for discussion. Error bars are 1σ and data are collated with references in Table 2.

xenon retention ages and U-Pb ages are comparable in terrestrial samples. Thus, it is at least consistent that whole-rock Mn-Cr and phosphate I-Xe ages should agree. In addition, the faster cooling of the ordinary chondrites and the Ste. Marguerite data suggest that feldspar I-Xe ages for the ordinary chondrites are reasonable approximations to phosphate I-Xe ages, as found by Brazzle et al. (1999). While acknowledging that data are sparse, our tentative conclusion is that the I-Xe and Mn-Cr data can be unified and considered together as a single time scale, though the specifics are subject to some modification as more data becomes available for the comparison.

We now consider integration of this unified time scale with that based on the decay of uranium isotopes to lead isotopes. In contrast to those chronometers based on extinct radionuclides, the Pb-Pb system is based on decay of isotopes that are still present in measurable quantities, and so allows absolute ages to be determined. However, it should be noted that ages derived from Pb-Pb data are not based on isochrons. They are model ages derived on the assumption of single-stage evolution, an assumption that is not necessarily valid for any sample. Furthermore, Wasserburg et al. (1996) have suggested that relative abundances of actinides and ^{129}I require distinct supernova sources and that contributions of actinides ceased as recently as ~10^7 years before the formation of the solar system. It is conceivable that this would have led to incomplete mixing of the parent isotopes and a detectable discordance between the I-Xe and U-Pb chronometers. On the other hand. successful calibration between the Pb-Pb and I-Xe time scales would lend support to the chronological interpretation of both systems since only if both are evolving consistently with time would agreement be expected. It would also allow conversion of formation intervals determined using chronometers based on short-lived radionuclides into equivalent absolute Pb-Pb ages. We now examine the extent to which the I-Xe and Pb-Pb systems agree.

Several samples allow comparison of Pb-Pb and I-Xe data. Göpel et al. (1994) published a systematic study of Pb-Pb ages from phosphate separates of ordinary chondrites and Acapulco. Among these, Acapulco, Ste. Marguerite and Kernouve phosphates yielded I-Xe isochrons with sufficient precision to allow direct comparison. Acapulco feldspar also yielded an accurate I-Xe isochron (Brazzle et al. 1999). In addition, Ste. Marguerite feldspar yielded a precise I-Xe isochron that may be compared with the Pb-Pb age of the phosphate separate. Some insight into the effects of comparing the chronometers in different minerals may be gained from the existence of a concordant whole-rock age for this meteorite that predates the phosphate age by some 4 Ma. At the time of writing, the earliest chondrules measured in the Pb-Pb system are those from the CR chondrite Acfer 059 measured by Amelin et al. (2002), which might thus be considered to be comparable to the earliest I-Xe ages measured in Semarkona chondrules (Swindle et al. 1991a, 1991b). I-Xe and Pb-Pb data (courtesy of Y. Amelin) for two Richardton chondrules also dated in the Pb-Pb system are presented here for the first time, while both I-Xe (Pravdivtseva et al. 1998) and Pb-Pb (Amelin 2001) data are available for Richardton pyroxene. Finally, we employ the

correlation between the Mn-Cr and I-Xe time scales derived above to convert the Mn-Cr intervals measured for LEW 86010 (Lugmair and Shukolyukov 1998) and Forest Vale (Polnau and Lugmair 2000) into equivalent I-Xe formation intervals; Pb-Pb data are available for both of these meteorites (Lugmair and Galer 1992; Göpel et al. 1994) .

In Fig. 4, we plot Pb-Pb ages and I-Xe intervals for the above samples (the data are collated with references in Table 2). There is a general trend suggesting that chronological information is recorded by both systems. A line fitted to the data yields a gradient significantly different from 1. This might be seen as evidence of some error in one or more of the accepted half lives. However, the apparent concordance of I-Xe and Mn-Cr data suggests both these half lives would need to be modified to similar extents. We consider it more likely that it indicates that one or more of the candidate data points should not be included. Indeed, this is inevitable since both Acapulco phosphate and feldspar I-Xe intervals are considered as potentially corresponding to the phosphate Pb-Pb age, and both phosphate and whole rock Ste. Marguerite Pb-Pb ages are considered potentially corresponding to the feldspar I-Xe age. It might be seen as encouraging that the alternate Acapulco I-Xe ages and Ste. Marguerite Pb-Pb ages bracket the correlation lines.

To proceed further, we investigated the effect of excluding various combinations of points (Table 3) that seemed particularly likely to skew the results, those derived via the Mn-Cr calibration, those depending on Ste. Marguerite and those depending on identifying Pb-Pb chondrule ages from Acfer 059 with Semarkona chondrule I-Xe intervals. In addition, we investigated which points yielded the greatest improvement in χ^2 of the fit forced to have gradient one when they were excluded. These turned out to be the earliest chondrules and the data derived from Ste. Marguerite. Exclusion of these points brought the gradient to within 2σ of 1, as did exclusion of the earliest chondrules datum alone. During the review stage of this paper our attention was drawn to ongoing work on Allende chondrules reported by Amelin et al. (2004), who report an age of 4566.7 ± 1.0 Ga. If verified, this would make the earliest chondrule datum more consistent with the trend defined by the other samples. Incorporating this number into our total fit yields a gradient of 0.87 ± 0.06 and an intercept of 4563.1 ± 0.03 when all data are included.

Clearly the variation in the absolute age derived for Shallowater under the various selections of points in Fig. 4 and Table 3 is greater than the error associated with the Shallowater value determined for any one selection of points. Accordingly, as a cautious estimate, we adopt the average value and standard deviation across the group as the best absolute age for Shallowater and associated 1σ error of 4563.3 ± 0.4 Gyr. Shallowater ages derived from varying gradients and gradients set to 1 are consistent within error (Table 3). The trend observed in Fig. 4 suggests that both I-Xe and Pb-Pb are recording chronological information, though the scatter around the trend indicated in the χ^2 values of Table 3 suggests that the dates recorded by the systems diverge from true consistency by greater than the reported margins of error. This is not unexpected given the probable differences in closure conditions of the two chronometers.

The correlation of Fig. 4 is strongly suggestive. However, it involves only a small fraction of the data from proposed chronometers available in the literature. The remaining data are for events dated in only one system, so no direct comparison is possible. Nonetheless, examining how these events would relate to one another should these calibrations be adopted can act as a test of the proposed calibrations

Table 2. Compilation of data used to examine the correlations among the Mn-Cr, I-Xe, and Pb-Pb chronometers. All errors are 1σ.

Sample	Interval	Source
Mn-Cr intervals		
Chondrule precursors	10.7 ± 0.5	Nyquist et al. (2001)
Richardton whole rock	−1.8 ± 1.4	Polnau and Lugmair (2001)
Ste. Marguerite whole rock	7.2 ± 0.2	Polnau and Lugmair (2001)
Forest Vale whole rock	3.5 ± 0.3	Polnau and Lugmair (2000)
Acapulco whole rock	−2.8 ± 0.5	Zipfel et al. (1996)
LEW 86010	0.0 ± 0.1	Lugmair and Shukolyukov (1998)
I-Xe intervals		
Earliest chondrules	4.3 ± 0.6	Swindle et al. (1991a, 1991b)
Richardton pyroxene	1.1 ± 2.0	Pravdivtseva et al. (1998)
Richardton feldspar	−8.2 ± 0.5	Brazzle et al. (1999)
Ste. Marguerite feldspar	0.7 ± 0.4	Brazzle et al. (1999)
Acapulco phosphate	−8.8 ± 0.2	Brazzle et al. (1999)
Acapulco feldspar	−3.8 ± 1.5	Brazzle et al. (1999)
Kernouve phosphate	−43.0 ± 6.0	Brazzle et al. (1999)
Richardton chondrule 2	−4.1 ± 0.6	Pravdivtseva (unpublished data)
Richardton chondrule 6	−0.1 ± 0.1	Pravdivtseva (unpublished data)
Pb-Pb ages		
Richardton pyroxene	4562.2 ± 1.2	Amelin (2001)
Richardton chondrule 2	4559.0 ± 1.4	Amelin (unpublished data)
Richardton chondrule 6	4563.2 ± 0.8	Amelin (unpublished data)
Ste. Marguerite whole rock	4566.7 ± 1.6	Göpel et al. (1994)
Ste. Marguerite phosphate	4562.7 ± 0.3	Göpel et al. (1994)
Forest Vale phosphate	4560.9 ± 0.4	Göpel et al. (1994)
Acapulco phosphate	4557.0 ± 1.0	Göpel et al. (1994)
Kernouve phosphate	4522.5 ± 0.7	Göpel et al. (1994)
LEW 86010	4557.8 ± 0.3	Lugmair and Galer (1992)
Chondrules	4564.7 ± 0.3	Amelin et al. (2002)

Table 3. Comparison of the results of Williamson fits to the data set of Fig. 4 and subsets thereof as defined in column 1. Errors are 1σ throughout.

	Free fit[a]			Fixed gradient[b]	
Data set	Gradient	Shallowater[c]	χ^2	Shallowater[c]	χ^2
All 11 candidate points	0.79 ± 0.05	4562.7 ± 0.2	31	4563.2 ± 0.2	43
All but those derived via Mn-Cr (9 pts)	0.81 ± 0.06	4562.5 ± 0.3	30	4562.7 ± 0.3	37
Al but those from Ste. Marguerite (9 pts)	0.76 ± 0.05	4562.4 ± 0.3	13	4563.3 ± 0.3	28
All but earliest chondrules (10 pts)	0.87 ± 0.08	4563.1 ± 0.3	24	4563.5 ± 0.2	27
All but earliest chondrules and Ste. M. data (8 pts)[d]	0.86 ± 0.07	4563.1 ± 0.4	7	4563.8 ± 0.3	9
Average[e]	0.82 ± 0.05	4562.8 ± 0.3		4563.3 ± 0.4[f]	

[a]Gradient and error allowed to vary.
[b]Gradient fixed equal to 1, forcing accepted values of half-lives.
[c]The intercept of the fitted line determines the Pb-Pb age equivalent to closure of the I-Xe system in Shallowater enstatite.
[d]These three points have the largest contributions to χ^2.
[e]Simple average of the 5 candidate fits and standard deviation.
[f]Preferred absolute age and error for the Shallowater standard.

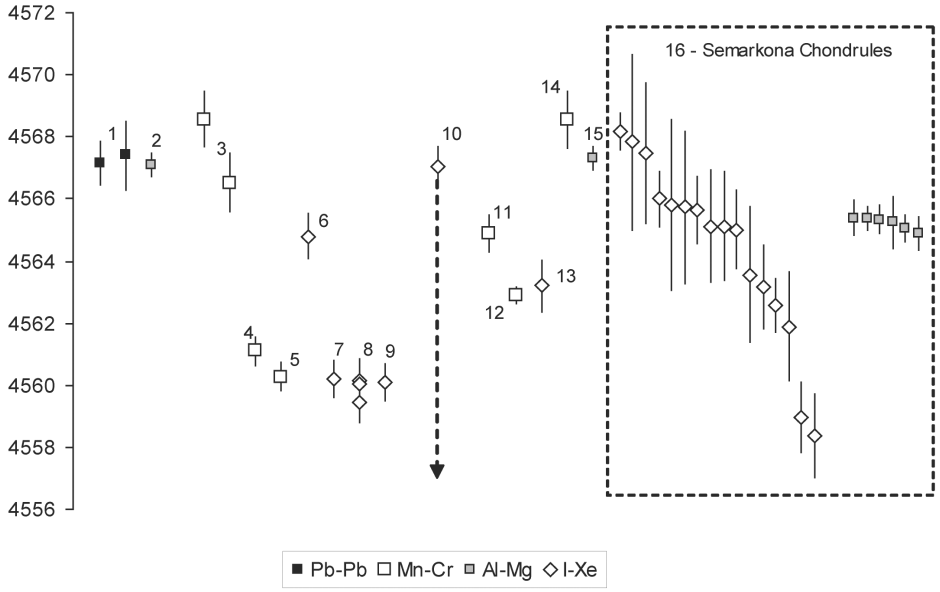

Fig. 5. The time scale of the early solar system that would result from adopting our preferred absolute age for the Shallowater standard of 4563.3 ± 0.4. Data from the Mn-Cr and Al-Mg systems have been incorporated based on the accepted age of LEW 86010 (Lugmair and Shukolyukov 1998) and assuming the CAI Pb-Pb age of Amelin et al. (2002) corresponds to $^{26}Al/^{27}Al = 5 \times 10^{-5}$. Error bars are 1σ and include errors arising from the conversion between chronometers. Data have been chosen to represent major processes as follows: 1–2: CAIs; 3–9: aqueous alteration; 10: earliest igneous process, range extends to younger ages than that displayed; 11–13: earliest achondrites; 14+: chondrules. Data: 1, 2: CAIs-Amelin et al. (2002); 3: CM, CR carbonates-Hutcheon et al. (1999); 4: Mokoia fayalite-Hutcheon et al. (1998); 5: CI carbonate-Endress et al. (1996); 6: Allende dark inclusions (average excluding one outlier)-Pradivtseva et al. (2003); Hohenberg et al. (2004); 7: CI magnetite-Hohenberg et al. (2000); 8: CAI alteration (sodalite)-Swindle et al. (1988); Pradivtseva et al. (2003); 9: OC halide-Busfield et al. (2004); 10: Barwell igneous clast-Gilmour et al. (2000); 11, 12: HED parent body and LEW 86010-Lugmair and Shukolyukov (1998); 13: Shallowater pyroxene-Brazzle et al. (1999); 14: Chondrule precursors-Nyquist et al. (2001); 15: Allende chondrules-Bizarro et al. (2004); 16: Semarkona chondrules-Swindle et al. (1991b), Kita et al. (2000), Huss et al. (2001).

(Fig. 5). Although there may be a hint that the earliest dates of aqueous alteration could predate the Pb-Pb age of CAIs, the errors clearly overlap. Igneous processes also appear to have commenced early in solar system history. That aside, there is little that is controversial in the relationships among these ages. While it would be premature to conclude that we have a definitive and unproblematic chronology of the early solar system, data that are available to date are encouraging.

We note that the model ages of chondrules in the Mn-Cr system and the earliest Al-Mg data are comparable. The range of I-Xe ages for Semarkona chondrules might be interpreted as extending from this formation time for several million years, perhaps reflecting alteration. Uncertainties in these ages, which are dominated by the individual measurements rather than the calibration via Shallowater and are thus independent, suggest that the I-Xe data are also comparable

with the Al-Mg data of Kita et al. (2000). The only other meteorite in which (separate) suites of chondrules have been measured in both systems is Chainpur. Huss et al. (2001) reported Al-Mg measurements on 7 chondrules, 4 of which yielded ages corresponding to 4562.5–4565.5 Gyr in the scheme adopted here, the remainder having no detectable ^{26}Mg excesses. I-Xe data have a somewhat similar distribution: Swindle et al. (1991a) reported 12 chondrules of which 3–4 had I-Xe ages consistent with those datable in the Al-Mg system, while Holland et al. (2005) reported 5 I-Xe ages from a suite of 10 chondrules, of which 2 were within the Al-Mg age range. There is thus some evidence that Al-Mg and I-Xe tell a consistent story in the analyses of chondrules from these meteorites.

What process the I-Xe chronometer dates in chondrules is more than somewhat controversial. Two factors are clearly relevant: the process(es) that led to the incorporation of iodine into a chondrule (primary formation or secondary alteration), and the process that led to Xe closure (primary mineralization or secondary alteration). Iodine is a volatile element that would be severely depleted during chondrule formation. This has been used to argue that I-Xe ages can be completely discounted as ages of primary formation. However, typical iodine concentrations in Chainpur chondrules are ~10 ppb (Holland et al. 2005) versus 430 ppb in CI chondrites (Anders and Grevesse 1989), and the correlated sites responsible for the I-Xe isochrons are a fraction of this. So it is not inconceivable that correlated iodine in some chondrules is primary.

Knowledge of the origin of the iodine carrier phase in chondrules is clearly crucial for the proper interpretation of I-Xe ages in chondrules. Kennedy et al. (1988) and Whitby et al. (2002) concluded that enstatite was the carrier of correlated ^{129*}Xe in EH3 chondrites, and Kehm et al. (1994) determined it was contained within micro-inclusions in the enstatite, suggesting that, in these samples, it is primary mineralization that is being dated. Secondary processes may be responsible for the I-Xe ages of many chondrules, as indeed they are thought to be responsible for resetting the Al-Mg system in chondrules from most meteorites. The I-Xe system in Allende CAIs clearly dates secondary processes since the carrier is sodalite, a product of aqueous alteration (Swindle et al. 1988; Kirschbaum 1988; Swindle 1998; Pravdivtseva et al. 2003). The spread of I-Xe ages in Chainpur chondrules is suggestive of secondary processing (Swindle et al. 1991a), but Holland et al. (2005) sought separate evidence of such processes in the Chainpur chondrules they dated without success. Pravdivtseva et al. (2005) argued that the spread of I-Xe ages among individual chondrules from LL chondrites increases with metamorphic grade, with chondrules from the low metamorphic grades clustering tightly shortly after CAI formation, and inferred that formation and alteration play complimentary roles in the I-Xe ages of chondrules. Nonetheless, in the absence of independent evidence of secondary processes in the chondrules dated and of the association of iodine with altered phases, it would be premature to conclude that secondary processes are responsible for the I-Xe age of any sample, chondrules included. Final resolution of this issue must await identification and characterization of the host phases of correlated iodine in chondrules and their associated ages.

Acknowledgments–This work was supported by the Particle Physics and Astronomy Research Council and by NASA grant NAG5-12776. We thank J. A. Whitby and D. A. Papanasstassiou for useful reviews.

Editorial Handling—Dr. Timothy Swindle

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