4 Noble Gases in the Moon and Meteorites: Radiogenic Components and Early Volatile Chronologies

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INTRODUCTION

One of the reasons noble gases make such good tracers of processes occurring in rocks is their scarcity. Thus, a process that converts a small fraction of a relatively rare element into a noble gas can have a large effect on the noble gas. Radioactive decay can be such a process. In a meteorite which has formed very early in the solar system's history and been little altered since, the effect can be even more dramatic. Furthermore, since radioactive decay proceeds at a known rate, radiogenic noble gases, those produced by decay, have been crucial in deciphering the chronology of the solar system.

Several "radionuclides," radioactive isotopes that decay to noble gases, are listed in Table 1. In a radioactive decay, the radioactive isotope is referred to as the "parent" isotope, while the decay product, the noble gas isotope, is referred to as the "daughter" isotope. In some cases listed, there is more than one mode of decay possible. In other cases, a single decay starts a chain that will ultimately produce several noble gas atoms. To take both into account, the yield (the number of noble gas atoms produced for each parent atom) is also given. Finally, radionuclides that fission may produce any of the several different isotopes of Xe in a characteristic spectrum (Table 2). All of the systems listed, with the exception of the decay of U and Th to Xe, have been exploited in extraterrestrial samples at one time or another.

Table 1: Radiogenic noble gas isotopes and their parents

Parent isotope	*Half-life (years)*	*Daughter isotope(s)*	*Yield*[c] *(atom/atom)*	*Proxy*[a]	*Abundance*[b]
^{22}Na	2.602	^{22}Ne	1		
^{36}Cl	3.01×0^{5}	^{36}Ar	1	^{37}Cl→^{38}Ar	^{36}Cl/^{35}Cl ~ 10^{-6}
^{40}K	1.277×10^{9}	^{40}Ar	0.1048	^{39}K→^{39}Ar	0.0117
^{129}I	1.57×10^{7}	^{129}Xe	1	^{127}I→^{128}Xe	^{129}I/^{127}I ~ 10^{-4}
^{232}Th	1.405×10^{10}	^{4}He	6		100
^{235}U	7.038×10^{8}	^{4}He	7		0.72
^{238}U	4.468×10^{9}	^{4}He	8		99.28
^{238}U	4.468×10^{9}	$^{131, 132, 134,1\ 36}$Xe	3.50×10^{-8}	^{235}U→$^{131-136}$Xe	99.28
^{244}Pu	8.08×10^{7}	$^{131, 132, 134,1\ 36}$Xe	7.00×10^{-5}	^{235}U→$^{131-136}$Xe	^{244}Pu/^{238}U~5×10^{-3}

[a]Nuclear reaction(s) producing noble gas isotope(s) from stable or long-lived isotope during an irradiation in a nuclear reactor. [b]For parent isotopes with half-lives less than 10^{8} years, typical or suspected abundance at the time of formation of the solar system. For longer-lived parent isotopes, current abundance (%) of element. [c]For actinides, ^{4}He yields are the number of atoms produced per decay chain, fission Xe yields are branching ratio for ^{136}Xe. Yields for other isotopes are given in Table 2.

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Table 2: Xenon fission spectra of actinides

Actinide	^{131}Xe	^{132}Xe	^{134}Xe	^{136}Xe	*Reference*
^{238}U	0.078	0.595	0.832	1.0	(Wetherill1953)
^{235}U^{a}	0.669	1.0	1.841	1.475-2.5	(Hohenberg & Kennedy1981; Hyde1971)
^{244}Pu	0.246	0.885	0.939	1.0	(Lewis1975)

[a]The lower number for the abundance of ^{136}Xe is for low-flux environments. Typical irradiation experiments give values more like 2.5, because the half-life of ^{135}Xe is long enough (9.14 hours), and the neutron-capture cross-section high enough to produce a significant amount of ^{136}Xe (Hohenberg and Kennedy1981).

A typical analysis of radiogenic noble gases involves trying to find the ratio of the daughter isotope to some other, stable, isotope of the parent element, for example, the ratio ^{129}Xe*/^{127}I or ^{40}Ar*/^{40}K, where the asterisk refers to radiogenic gas. For a long-lived radionuclide like ^{40}K, the result is usually quoted as an age, the length of time it would have taken for that much of the radiogenic gas to build up. For a short-lived radionuclide, where all the radionuclide has long since decayed, the result is often converted into the relative abundance of the parent isotope at the time of isotopic closure, such as ^{129}I/^{127}I. There is also chronological information in this ratio, although there are other complications, such as the possibility of isotopic inhomogeneity, the question of when and how the radionuclide was created, and the calibration of what the ratio was at some known time, details that will be discussed later in this chapter for several radionuclides.

Isotopic closure will not necessarily be the same thing as formation of the host rock. Since noble gases diffuse more readily than other radiogenic isotopes, they are uniquely suited to studies of thermal processes. However, one of the things that has become increasingly apparent is that it is crucial to determine what sample is required to date a particular process (or, to determine what process a particular sample is dating).

In this chapter, we will first consider the long-lived nuclides, such as ^{40}K and the actinides U and Th, and then the short-lived "extinct" radionuclides.

LONG-LIVED NUCLIDES: CHRONOLOGY OF SOLAR SYSTEM EVOLUTION

Of the parent isotopes listed in Table 1, ^{40}K is by far the most abundant. Only 0.017% of the potassium in a modern rock is ^{40}K, but potassium is at least a minor element in many rocks, and the half-life of ^{40}K (1280 Ma) is relatively long. Because ^{40}K is so abundant, it is not surprising that the ^{40}K-^{40}Ar system is the most widely used in meteorites as well as terrestrial samples. Although the ^{40}K-^{40}Ar system can provide some useful information about crystallization ages, in most cases in meteorites and lunar samples it is most useful for piecing together the thermal history, since it is more sensitive to low-temperature heating events than the Rb-Sr, Sm-Nd or even U,Th-Pb systems. For example, metamorphic heating of chondrites or heating by the deposition of warm ejecta by an impact event can frequently be dated using the K-Ar system.

The actinides, Th and U, also decay by α decay en route to the stable Pb isotopes. For each actinide nucleus, several α particles (^{4}He nuclei) are produced. However, the same problems that make it difficult to use U,Th-He dating in terrestrial studies (Farley 2002, this volume), the recoil upon creation and the extremely low temperature at which it is lost, make it difficult to apply to meteorites as well. So the K-Ar system, particularly the ^{40}Ar-^{39}Ar version, is used far more often than is the U-Th-He system, and hence will be mentioned far more often in applications.

Another chapter in this book (Kelley 2002) contains details about the history of the technique and pertinent technical details about its application. These will not be repeated here, except to note that the most common application of the technique for meteorite and lunar studies is step-heating ^{40}Ar-^{39}Ar dating. In this application, a sample is irradiated with neutrons, converting some ^{39}K into ^{39}Ar through neutron capture. The sample is then heated to progressively higher temperatures, and the gas released at each temperature is analyzed. This yields an "age spectrum" of apparent age vs. temperature (Kelley 2002).

Solar system impact history

The dominant process occurring on most solid surfaces in the solar system for the last 4.5 Ga has been impact cratering. When Galileo turned his new telescope to the Moon in 1609, what he noted most was the myriad of near-perfect circles. Spacecraft views of asteroids, planetary moons, and even planets have looked much the same, except for cases like Venus or Jupiter's moons Io and Europa, where there has been enough internal heating to cause very recent resurfacing. Our understanding of how the cratering process works has grown in recent decades, spurred in part by the observations of other solar system bodies and by a recognition that craters might be important to Earth's history, and aided by nuclear weapons tests (which provide the closest analog to an impact) and the development of sophisticated hydrocodes (Melosh 1989). Recognition of the inevitability of the cratering process has led to the use of relative crater densities to determine the relative chronology of surfaces on the Moon, Mars or any other heavily cratered body. However, crater densities determine relative, not absolute, chronology. Crater densities can be turned into absolute chronology only if the cratering rate is known, a difficult proposition at best (Strom et al. 1992).

Although an impact-cratering event carries a huge amount of energy and can disrupt a vast area of a planet's surface, it will not cause isotopic resetting of every rock that it moves. In fact, a shock event will not even cause isotopic resetting of every rock in which it leaves mineralogical evidence. The shock wave caused by the impact can move through fast enough that it may disrupt mineral grains without giving radiogenic isotopes an opportunity to move and equilibrate. Even if radiogenic isotopes are mobilized, it may simply be enough to disturb a system without resetting it. Hence, finding the age of a crater is often difficult. In a few cases on the Moon, there are craters young enough (<100 Ma) that their ages can be determined by the clustering of cosmic ray exposure ages of rocks they have uncovered (Arvidson et al. 1975). In most cases, an impact event has to be dated through the collateral heating that it produces. Since the ^{40}Ar-^{39}Ar system is the most sensitive to heating of the major chronology systems, it will be reset in many more rocks than systems such as Rb-Sr or Sm-Nd. Bogard has written two good reviews of this application of ^{40}Ar-^{39}Ar studies, a decade and a half apart (Bogard 1979, 1995).

For an impact event to be able to reset the ^{40}Ar-^{39}Ar system, the rock has to be at a high enough temperature for a long enough time. There are many cases in meteorite and lunar samples, as for terrestrial samples (Kelley 2002, this volume), where samples have apparently not reached high enough temperatures to completely degas—mineral sites that degas at low-temperatures yield lower apparent ages, and the age spectrum can be interpreted in terms of diffusive loss. On the other hand, even if a sample is melted, the ^{40}Ar-^{39}Ar system will not be reset if it cools quickly. This has been shown dramatically in the cases of the meteorites Peace River (McConville et al. 1988) and Chico (Bogard et al. 1995), both of which have glassy areas that were melted in a shock event, but then quickly cooled. In each case, detailed ^{40}Ar-^{39}Ar studies, comparing multiple samples, have shown that some samples have retained ("inherited") a fraction of the ^{40}Ar that they contained before the impact, and yield age spectra that would be very difficult to interpret without multiple samples. Hence, in trying to find the age of a crater, one would like to find an impact melt that cooled slowly enough to fully degas (for example, one that cooled slowly enough to recrystallize). Such slow cooling requires being near the center of a fairly large (perhaps 100 km in diameter) crater. In many cases, we will be limited to imperfect samples, either because we are trying to date a smaller crater or because of the fundamental limitation of the available samples, so it becomes necessary to interpret a complicated age spectrum (Kelley 2002).

Lunar impact history: a cataclysm? Given its proximity to the Earth, the Moon's impact history presumably is indicative of the impact history of the Earth as well. The majority of published ^{40}Ar-^{39}Ar ages of lunar rocks come from the 1970s, most of them published in the annual *Proceedings of the Lunar Science Conference*. However, these early studies raised questions that are still being sorted out by detailed studies with more advanced technology.

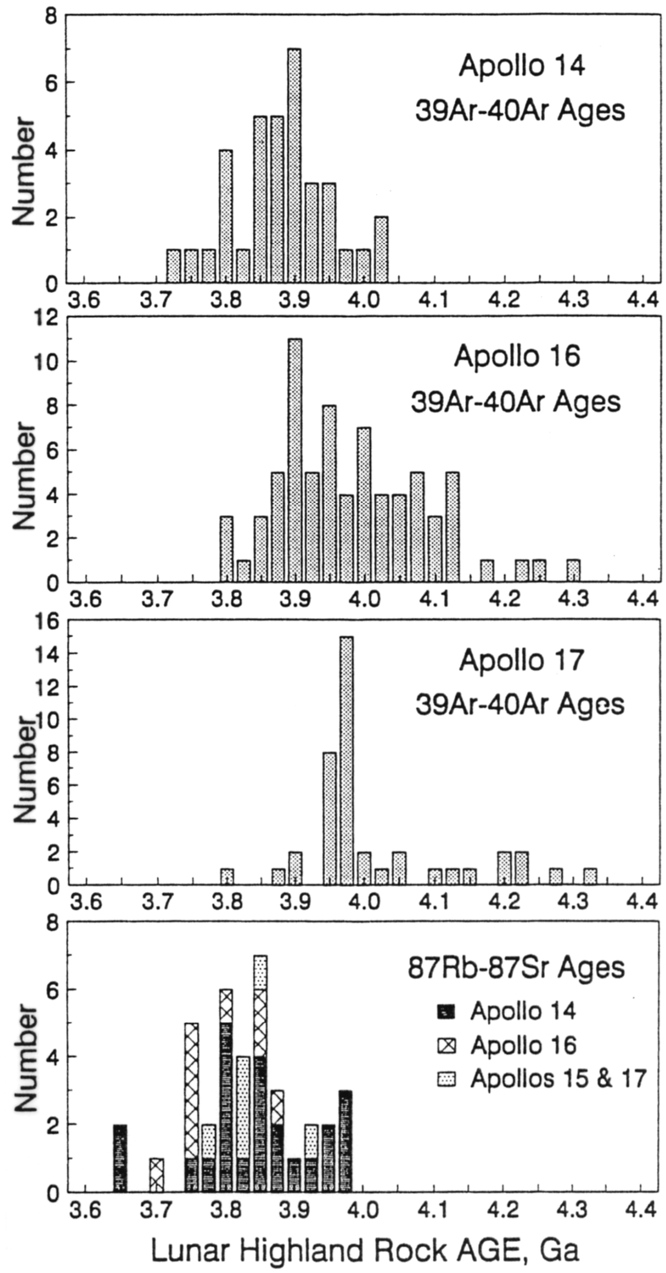

Figure 1. Histogram of apparent ^{40}Ar-^{39}Ar ages of lunar samples from three Apollo landing sites and Rb-Sr ages from four sites. Note the prominence of ages between 3.8 and 4.0 Ga. [Used by permission of the editor of *Meteoritics*, from Bogard (1995), Fig. 6, p. 256].

The most notable aspect of ^{40}Ar-^{39}Ar ages of lunar impact melts is that the majority of them cluster at an age just younger than 4.0 Ga (Fig. 1), first noted by Turner et al. (1973). A similar effect is seen in the U-Th-Pb- and Rb-Sr-systems (Fig. 1), which led Tera et al. (1974) to suggest that a "lunar cataclysm" occurred 3.8 to 4.0 Ga ago. No one suggested a good reason why such a cataclysm should have occurred, and many lunar geologists quickly argued that instead of a "cataclysm," the isotopic systems might be recording the end of an epoch of "heavy bombardment," (Bald-win 1974; Hartmann 1975). It has also been suggested that the apparent "cataclysm" might be reflecting a single event, the impact that formed Imbrium. Imbrium is one of the most recent of the basins, craters several hundred kilometers in diameter that dominate lunar geology. The returned lunar samples all come from a relative small area on the Near Side of the Moon, and that region can be all be seen to have suffered some effects of the Imbrium impact, so there simply might not be any material surviving with a record of previous impacts. On the other hand, attempts to date other basins based on impact melts that could have come from them still suggested that the basins had all formed in a relatively short period of time (see Dalrymple and Ryder (1996) and Stöffler and Ryder (2001) for discussion of many isotopic attempts to find the ages of basins).

Two studies that appeared less than a year apart led to detailed experimental studies that have lent strong support to the idea of a cataclysm. Although the idea still cannot be considered verified, some of the earlier questions have been answered, and means have been found to address a new set of questions.

First, G. Ryder revived the cataclysm hypothesis (Ryder 1990). He looked at the petrology of samples that had been dated, and argued that none of the ages older than 4.0 Ga were actually dating impacts. Instead, he suggested that those ages all came from samples that were either igneous rocks, or rocks that had been at most partially reset by impacts. This led to a detailed study by Ryder and Dalrymple of clasts that definitely were impact melts. With the advances in techniques that had occurred, they were able to date much smaller samples than had been possible in the 1970s. In a series of studies (Dalrymple and Ryder 1993; Dalrymple and Ryder 1996) they found many impact melts 3.8 to 4.0 Ga old. They suggested that most of the visible basins may have formed in as little as 55 to 60 Ma, implying a very high flux of very large objects for a very short period of time. They found several other samples that were older than 4.0 Ga, but none of the old samples were impact melts. Since some older samples were observed, they argued that the lack of old impact melts was not a result of all old rocks having been reset, but rather the result of a cataclysm.

Another insight came from G.J. Taylor (1991), who suggested a way to test the influence of Imbrium. In a study of a lunar meteorite, MacAlpine Hills 88105, he pointed out that the meteorite's bulk chemistry was inconsistent with an origin in the part of the Near Side from which samples had been returned, and that it had impact melt clasts with compositions different from anything expected from impacts on the Near Side. Hence, he suggested that if those clasts could be dated, the ages would assuredly not be reflecting Imbrium. Cohen et al. (2000) determined ages from 31 impact-melt clasts in MacAlpine Hills 88105 and three other lunar meteorites, and still failed to see any ages older than 4.0 Ga. The source of the cataclysm is still unexplained, but its reality now seems more likely.

In reviewing impact ages of extraterrestrial bodies, Bogard (1995) pointed out that there may also be evidence for a cataclysm in ^{40}Ar-^{39}Ar ages of other samples from the inner solar system. He noted that several of the eucrites and diogenites, differentiated meteorites that apparently come from the outer portion of the Main Belt asteroid Vesta (Drake 2001), show impact ages of 3.5 to 4.0 Ga. The same is true of many of the mesosiderites and IIE iron meteorites, which are both types of metal-rich meteorites that presumably come from near the cores of differentiated asteroids. Furthermore, the only Martian meteorite old enough to have been in existence during the cataclysm, Allan Hills 84001, has also apparently been reset by impact about 3.9 to 4.0 Ga ago. Although there is not a strong signal of a 3.9 to 4.0 Ga impact among the most common meteorites, the chondrites, K-Ar ages have been determined for far more chondrites, and many of these do appear to have lost Ar within the last 4 Ga (Heymann 1967).

Lunar samples have also been used in an attempt to determine the frequency of smaller cratering events on the Moon and, by extension, the Earth. Culler et al. (2000)

analyzed many samples from an Apollo 14 "soil" (lunar regolith) sample. In contrast to Dalrymple and Ryder (1993, 1996) and Cohen et al. (2000), who restricted themselves to impact melts that had cooled slowly enough to recrystallize, Culler et al. (2000) chose to analyze glass spherules, which must have cooled quickly, reasoning that this would increase their chances of sampling smaller craters, which in turn would increase their chances of sampling many different craters. Many of their samples had ages less than 1 Ga (some, though gave ages approaching 4.0 Ga), and while there are certainly some ages that appear more often than others, the meaning of the age spectrum, (e.g., whether there is a periodicity), remains to be seen (Culler et al. 2000). In addition, the use of glass spherules also makes such a study more vulnerable to samples that have not been completely degassed in the impact event, as shown by the Peace River data (McConville et al. 1988) discussed above.

Many of the advances in the last decade of the 20th Century were possible because of advances in techniques. Dalrymple and Ryder (1993, 1996), Cohen et al. (2000), and Culler et al. (2000) all used laser extraction techniques that allowed them to use smaller samples (typically no more than a few hundred micrograms, sometimes as small as 1 μg). This in turn, enabled them to determine ages of samples that would have been completely undatable in the 1970s. Lasers were used for ^{40}Ar-^{39}Ar experiments as early as 1973 (Megrue 1973). However, the early experiments used the lasers as microprobes, basically finding the K-Ar ages of small regions within a sample. More recent experiments have used lasers in step-heating experiments as a heating device that generates extremely little background interference. The latter approach has proven to be more widely applicable.

Ordinary chondrites. The ordinary chondrites, the most common meteorites, do not show a strong signature of a cataclysm at 3.9 Ga. Bogard (1995), in pointing this out, suggested that it was a selection effect, that chondritic asteroids that suffered large impacts in the cataclysm were simply destroyed, so we preferentially sample those that were not affected. There are, however, more recent impact events that are clearly recorded in ordinary chondrites.

The most prominent signature is of one or more events that affected, perhaps destroyed, the L (low-iron) chondrite parent body less than 1 Ga ago (Fig. 2). This can be seen even in the simplest of noble gas radiometric ages, K-Ar and U-Th-He ages. Because L chondrites all have roughly the same K and actinide contents, the distribution of ^{40}Ar and ^{4}He, respectively, are equivalent to age distributions. Several authors (Heymann 1967; Wasson and Wang 1991; Zähringer 1968) noted a preponderance of ages of roughly 350-500 Ma, although it is impossible to be more precise on this basis.

More detailed ^{40}Ar-^{39}Ar ages have only slightly clarified the situation. Bogard and Hirsch (1980) analyzed seven severely shocked chondrites and found most showed either well-defined plateaus at 500-600 Ma or age minima at 700-1000 Ma. In a more detailed study of a single sample, with multiple smaller samples from various locations within a shock-melted L chondrite, Chico, Bogard et al. (1995) documented the prevalence of partial resetting in this event—the lowest ^{40}Ar-^{39}Ar ages clustered around 550 Ma, but even that is higher than the Rb-Sr age of about 470 Ma, so they suggested that at least 1-2% of the radiogenic ^{40}Ar was retained in all of the samples. In a similar study of another shock-melted L chondrite, Cat Mountain, Kring et al. (1996) argued that Cat Mountain suffered a shock event 800-900 Ma ago, an age also shown by a few other L chondrites. Whether this truly represents a second shock event, or simply reflects partial resetting 400-500 Ma ago, will require future studies to resolve.

Metamorphic heating of chondritic meteorites. In terrestrial studies, one of the most common uses of the K-Ar system is to date metamorphic events (Kelley 2002; McDougall and Harrison 1999). In meteorites, the most prominent metamorphic situation is the heating of ordinary chondrites. Terrestrial studies have demonstrated that the "age" of a metamorphosed sample depends on the closure temperature, which in turn depends on both the time-temperature history of the sample and the specific mineral under consideration. In addition, whether a particular question can be answered depends on the precision of the system being considered. The story of ordinary chondrite metamorphism provides examples of all of these facets.

Ordinary chondrites are divided into chemical classes, based primarily on their iron abundance (H – high; L – low; LL – very low). Each chemical class is further divided into metamorphic classes or petrographic grades (3 to 6 from least to most metamorphosed), based on, among other things, the degree of equilibration of mafic silicates and the degree of recrystallization of textural components (Dodd 1981; Wasson 1985).

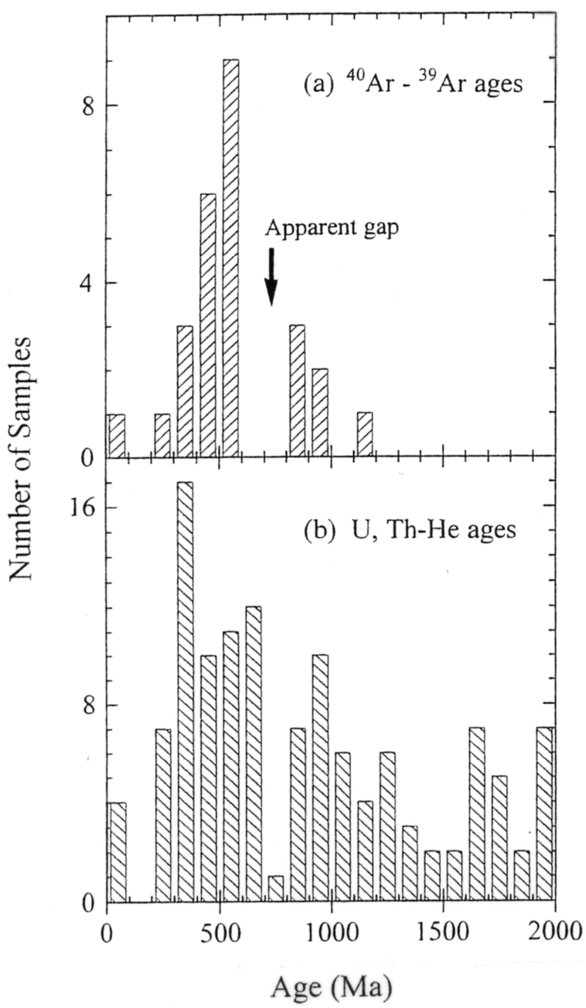

Figure 2. Histogram of reported recent shock-reset ages of L chondrite meteorites. Note the apparent presence of one peak at about 500 Ma and the possibility of another near 900 or 1000 Ma. [Used by permission of the editor of the *Journal of Geophysical Research*, from Kring et al. (1996), Fig. 12, p. 29,367].

The question is how and where that metamorphism occurred. Is it prograde or retrograde (i.e., is it reflecting a reheating, or does it merely reflect slow cooling from some very high initial temperature)? Did it occur within an internally heated, stratified parent body, or within pieces of a rubble pile that were heated, broken apart and reassembled? Within a stratified parent body, the expectation would be that the more equilibrated (higher petrographic grade) meteorites would have come from closer to the center of the body, so they would have stayed hot longer (younger ages) and would have cooled more slowly.

For many years, the chronological and cooling rate data did not give a coherent picture. Cooling rates based on the size and composition of metal grains typically showed no correlation with petrographic grade (Taylor et al. 1987), nor did I-Xe ages ((Jordan et al. 1980; Swindle and Podosek 1988); note that these experiments were on whole-rock samples, rather than the mineral separates that have proven more amenable to interpretation, as discussed below). On the other hand, cooling rates based on damage tracks from fissioning ^{244}Pu, really cooling ages based on that extinct radionuclide, did seem to show the expected correlation (Pellas and Störzer 1981). A ^{40}Ar-^{39}Ar study of

13 ordinary chondrites, meanwhile, concluded that the ages were nearly indistinguishable at about 4.5 Ga old (Turner et al. 1978). However, these typically had uncertainties of 30 Ma or more, comparable to the expected variations, and 10 of 13 were of a single one of the three chemical classes (H). When all available analyses were considered, a correlation was suggested, particularly for the LL chemical class (Lipschutz et al. 1989).

Later radiometric studies finally began to show a correlation. The most telling evidence came from Göpel et al. (1994), who found a correlation in Pb-Pb ages from apatites. Later, Brazzle et al. (1999) found that the I-Xe ages of apatites (in some cases, aliquots of the samples analyzed by Göpel) and feldspars also showed a correlation. The I-Xe work will be discussed more in the next section.

In this case, the crucial data appears to require higher precision than the ^{40}Ar-^{39}Ar technique was capable of when the problem was first attacked. Using mineral separates, instead of whole rock samples (Pb-Pb and I-Xe), was the other key step. Although ^{40}Ar-^{39}Ar experiments are capable of the required precision, whether any minerals within the meteorites will have remained closed systems for the entire 4.5 Ga remains to be seen. If so, the logical next step is to use mineral separates so that the extensive terrestrially-derived database on diffusion properties can be used to define closure temperatures (McDougall and Harrison 1999).

Other applications of the K-Ar system to meteorites. The K-Ar and/or ^{40}Ar-^{39}Ar techniques have been applied to virtually every type of meteorite. However, while precise ages can be obtained, in many cases it is not clear exactly what those ages are dating. While many of them show evidence of impact events 4 Ga ago or less (Bogard 1995), others give ages of nearly 4.5 Ga, including some eucrites and most of the IAB iron meteorites (Niemeyer 1980). A detailed summary will not be attempted here, but the individual studies are most commonly found in *Meteoritics and Planetary Science*, *Geochimica et Cosmochimica Acta*, and the *Journal of Geophysical Research*.

^{40}Ar-^{39}Ar dating has been performed on most Martian meteorites. Depending on the meteorite, and on what results have been obtained from other radiometric chronometers, the results have been interpreted in different ways. Results are discussed in the chapter on Martian samples (Swindle 2002).

Extinct radionuclides: Chronology of solar system formation

There are several cases in Table 1 (those with half-lives less than 10^8 years) where the half-life of the parent isotope is so short that any that was incorporated into a rock at the time of formation of the solar system 4.5 Ga ago will have long since decayed away. However, the noble gas daughter isotopes may still be present, and can be used to deduce the very early history of the solar system. Freshly fallen meteorites do contain some of these "short-lived radioactivities," as a result of bombardment by cosmic rays (Wieler 2002, this volume). But ratios like ^{36}Cl/Cl and ^{129}I/I resulting from cosmic ray bombardment are typically 10^{-10} to 10^{-15}, compared to 10^{-4} to 10^{-6} at the start of the solar system, so they are measured by accelerator mass spectrometry or direct counting, rather than by measuring noble gases. Noble gas measurements are only useful for determining the abundances of short-lived radionuclides within about 10 half-lives of their synthesis.

Extinct radionuclides have a huge advantage in precision over long-lived radionuclides. For example, a change in a factor of two in the abundance of ^{129}I requires only one half-life, 15.7 Ma, while the same length of time will cause a change of less than 1% in the abundance of ^{40}K, since it is only about 1% of a half-life. On the other hand, since the extinct radionuclide is, by definition, completely decayed away, it is necessary to determine its abundance at some particular time to determine ages, a problem for all extinct radionuclides.

The relative amount of attention given to extinct radionuclides compared to the ^{40}Ar-^{39}Ar system in this chapter does not reflect the relative number of studies. However, the greater applicability of the ^{40}Ar-^{39}Ar system means that there are more reviews available for that technique (Bogard 1979, 1995; Kelley 2002; McDougall and Harrison 1999).

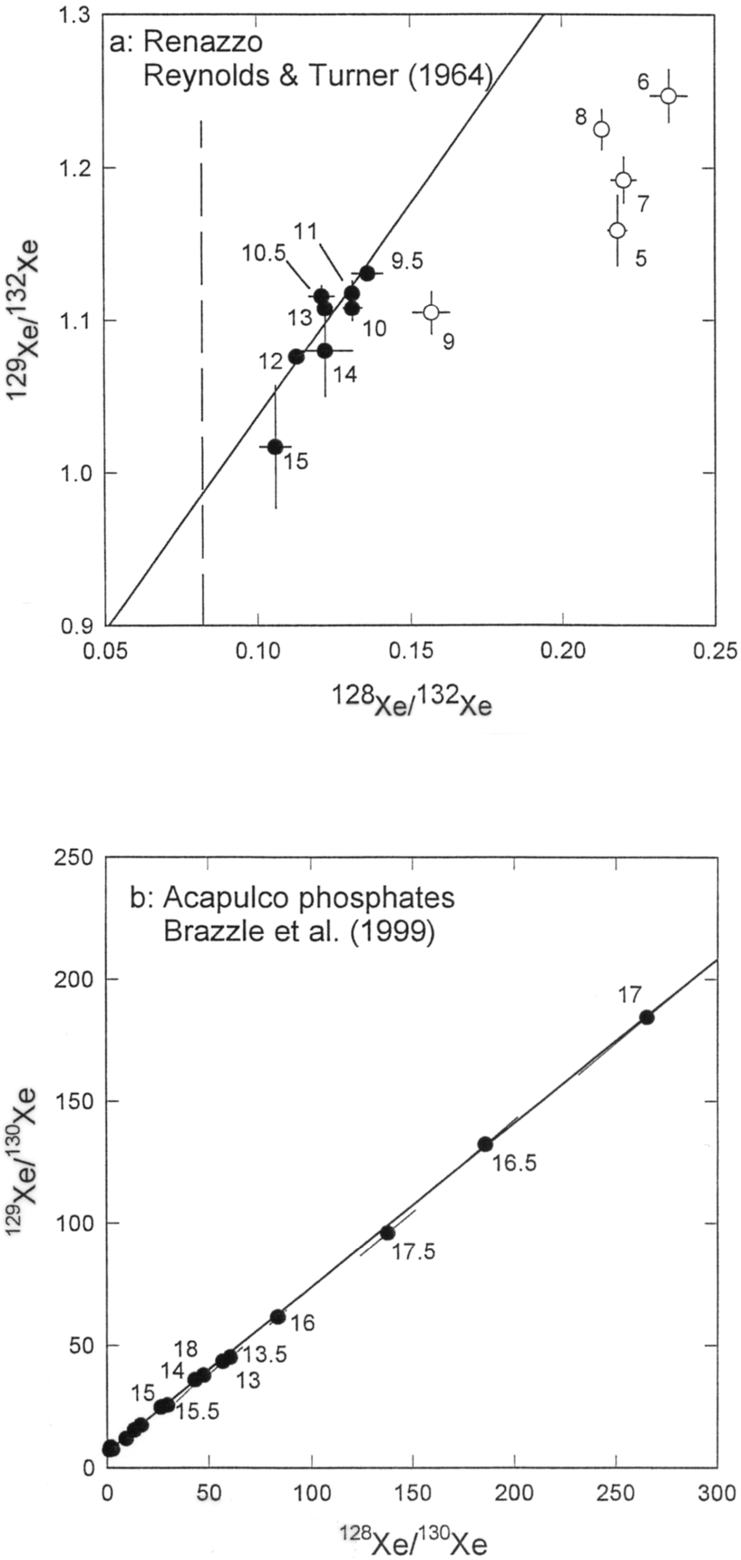

Figure 3. Three-isotope plots for I-Xe experiments. Data in (a) are from Reynolds and Turner (1964). Labels next to points are extraction temperatures (in hundreds of degrees Celsius). Solid points are those defining the correlation line. Dashed line is at a $^{128}Xe/^{132}Xe$ ratio typical of chondritic meteorites. Data in (b) are from Brazzle et al. (1999). The extraction temperatures in (b) are actually the temperature of a heating coil containing the sample, rather than the sample itself. In this case, even the low temperature extractions fall on the correlation line. A different denom-inator is used because the ^{132}Xe in the Acapulco phosphates is dominated by fission-produced gas. For the sake of conversion, the trapped $^{130}Xe/^{132}Xe$ ratio is typically about 0.16 (Ott 2002, this volume). The equivalent dashed line would be indistin-guishable from the ordinate, because the I/Xe ratios are so high.

Iodine-xenon

The first extinct radio-nuclide studied, and by far the most commonly studied of the extinct radionuclides, is ^{129}I. Most chondrite meteorites contain ^{129}Xe that appears to be a result of the decay of ^{129}I (as do the atmospheres of Earth and Mars). We can illustrate the basic technique commonly used in studies of many of the short-lived radiogenic noble gases in meteorites with two examples (Fig. 3). Figure 3a is data from one of the first I-Xe analyses, a study of the carbonaceous chondrite Renazzo. Figure 3b is data from an important recent analysis, of a phos-phate mineral separate from the meteorite Acapulco. The data from Renazzo are less precise, and more difficult to interpret, than more recent data, and the data from Acapulco are among the most precise and simplest to interpret. Between them, they illustrate several of the basic principles that will be discussed. We will then compare that to a typical ^{40}Ar-^{39}Ar analysis, which has been described elsewhere in this volume (Kelley 2002), and then briefly consider the complications involved in analyzing other systems.

Technical details. First, assume that the ratio ^{129}I/^{127}I is the same in all mineral sites, no matter how much iodine they contain. The validity of that assumption has been the subject of many discussions of the I-Xe system, but for the moment, we will make it. Then, since all of the ^{129}I has decayed to ^{129}Xe*,

$$^{129}\mathrm{Xe}^*/^{127}\mathrm{I} = (^{129}\mathrm{I}/^{127}\mathrm{I})_I = R \tag{1}$$

where the subscript $_I$ refers to the "initial" ratio, the ratio at the time of formation. Next, the sample is irradiated with neutrons. As a result of neutron capture, some of the denominator isotope is converted to noble gas, through a reaction like

$$^{127}\mathrm{I}(\mathrm{n},\beta^-)^{128}\mathrm{Xe}$$

The noble gas isotope then becomes a proxy for the parent. The isotopes used this way are listed in Table 1. The rest of the analysis is quite similar to a standard three-isotope plot for geochronology systems such as Sm-Nd or Rb-Sr. The major difference is that instead of using an isotope of the parent element, noble gas-based techniques use only isotopes of the daughter element, the noble gas. Because mass spectrometers are generally better at measuring relative isotopic abundances than at measuring absolute amounts, this is a decisive advantage.

After this neutron irradiation, the sample is then heated to progressively higher temperatures (stepwise heating), and the gas released at each temperature is analyzed. The original idea was to do the equivalent of a mineral separation, assuming that different minerals would degas at different temperatures. While that is true to some extent, stepwise heating is now often performed on mineral separates with the understanding that information on the diffusion properties of the mineral is contained in the degassing results (Bogard and Hirsch 1980; Burkland et al. 1995; McDougall and Harrison 1999). In the case of Renazzo, the temperature steps were 100°C each, in the Acapulco phosphates, the steps varied. Ratios, rather than absolute amounts, are usually reported, in part because the measurements of ratios are usually much more precise than the measurements of absolute amounts, and in part because it is the ratios that will be important in the next step, which is to plot the data (Fig. 3).

In an idealized case the gas now comes from three sources: (1) the background (or "trapped") noble gas that was present in the rock at the start; (2) the radiogenic gas that was produced by radioactive decay within the rock; and (3) the gas produced as a parent proxy by the nuclear irradiation. If we let the subscript "T" refer to an isotopic ratio or the abundance of a particular isotope in the total sample, "t" refer to the trapped noble gas, and "n" refer to the gas produced by the nuclear irradiation, then for ^{129}Xe and ^{128}Xe we have

$$^{129}\mathrm{Xe}_\mathrm{T} = {}^{129}\mathrm{Xe}^* + {}^{129}\mathrm{Xe}_\mathrm{t} \quad \text{and} \tag{2a}$$

$$^{128}\mathrm{Xe}_\mathrm{T} = {}^{128}\mathrm{Xe}_\mathrm{n} + {}^{128}\mathrm{Xe}_\mathrm{t}\,. \tag{2b}$$

Also, $^{128}\mathrm{Xe}_\mathrm{n} = C\ ^{127}\mathrm{I}$, where C is the conversion factor, which is determined by the neutron capture cross-section and the neutron flux. Then we can combine Equations (1) and (2) and write

$$^{129}\mathrm{Xe}^* = R\ ^{127}\mathrm{I} = CR\ ^{128}\mathrm{Xe}_n\ . \tag{3}$$

The presence of the trapped Xe means that the total ^{129}Xe/^{128}Xe ratio will differ from one mineral to another, because the ratio of I to Xe in the minerals will differ.

Some other Xe isotopes, notably ^{130}Xe and ^{132}Xe, may be entirely "trapped" Xe (in reality, a fraction of these may have been produced by spallation, in the former case, or fission, in the latter case, but those are often second-order complications). If we measure, for example, ^{129}Xe/^{130}Xe and ^{128}Xe/^{130}Xe, then we can use Equations (2a) and (2b) to write

$$(^{129}\mathrm{Xe}/^{130}\mathrm{Xe})_T = {}^{129}\mathrm{Xe}^*/^{130}\mathrm{Xe} + {}^{129}\mathrm{Xe}_t/^{130}\mathrm{Xe} \quad \text{and} \tag{4a}$$

$$(^{128}\mathrm{Xe}/^{130}\mathrm{Xe})_T = {}^{128}\mathrm{Xe}_n/^{130}\mathrm{Xe} + {}^{128}\mathrm{Xe}_t/^{130}\mathrm{Xe}. \tag{4b}$$

But since $^{129}\mathrm{Xe}^* = CR\ ^{128}\mathrm{Xe}_n$, we can substitute in Equation (4a), and write

$$(^{129}\mathrm{Xe}/^{130}\mathrm{Xe})_T = CR\ ^{128}\mathrm{Xe}_n/^{130}\mathrm{Xe} + {}^{129}\mathrm{Xe}_t/^{130}\mathrm{Xe}\ , \tag{5a}$$

we can also rewrite Equation (4b) as

$$^{128}\mathrm{Xe}_n/^{130}\mathrm{Xe} = (^{128}\mathrm{Xe}/^{130}\mathrm{Xe})_T - {}^{128}\mathrm{Xe}_t/^{130}\mathrm{Xe}\ . \tag{5b}$$

Making one last substitution of Equation (5b) into Equation (5a), we have

$$(^{129}\mathrm{Xe}/^{130}\mathrm{Xe})_T = CR\ [(^{128}\mathrm{Xe}/^{130}\mathrm{Xe})_T - {}^{128}\mathrm{Xe}_t/^{130}\mathrm{Xe}] + {}^{129}\mathrm{Xe}_t/^{130}\mathrm{Xe}\ . \tag{6}$$

This is the equation of a line involving the two measured ratios. Much of the data in Figure 3, where the two ratios are plotted, clearly do fall along a line, which is referred to as an "isochron", since all the points falling along the line have the same ("iso") initial ratio or age ("chron"). We will return later to the significance of the Renazzo points that deviate from the isochron. Note that if the I/Xe ratio is higher, as is the case for most of the data from the Acapulco phosphates, the trapped Xe is less significant. However, there are also cases where the isochron is defined by fewer points than the Renazzo plot in Figure 3.

The slope is *CR*, the product of the initial iodine isotope ratio and the conversion factor. The real point of the experiment is to determine *R*, the initial iodine isotopic composition. To do so, however, it is necessary to know *C*, the efficiency with which ^{127}I is converted to ^{128}Xe. This is normally done by including an "irradiation monitor" in the nuclear irradiation, a sample with a known $(^{129}\mathrm{I}/^{127}\mathrm{I})_0$ ratio. The data from the monitor are then plotted on the same type of isochron plot, but for the monitor, the unknown is *C*, the conversion factor for that particular experiment. Then, knowing the conversion factor, *R* can be determined for the other samples irradiated at the same time.

It might seem that the best monitors of the conversion factors would be relatively pure samples, such as potassium iodide (KI) for either K-Ar or I-Xe experiments. In fact, such samples are difficult to analyze, because they contain so much more neutron-derived gas than the unknown samples, and have so much iodine that they may produce self-shielding effects in the reactor (Hohenberg et al. 2000). Instead, the monitors are usually samples with known ratios similar to those expected in the experiment. For meteorites, the most commonly used monitors in recent years have been the meteorites Bjurbole and Shallowater (Table 3). Other meteorites have been used as monitors, as well, but only for these two have enough samples been analyzed to demonstrate that the $(^{129}\mathrm{I}/^{127}\mathrm{I})_0$ ratio is uniform. In experiments where other monitors have been used, the data should be approached with caution (Hohenberg et al. 1981, 2000; Swindle and Podosek 1988).

Table 3: Iodine-xenon monitors and calibrations.

Sample	$^{129}I/^{127}I$	*Relative age (Ma)*	*Absolute age (Ma) relative to*	
			Acapulco	*Ste. Marguerite*
Shallowater	≡1.072	≡0	4566	4564
Bjurbole	1.095±29	-0.46±0.15	4566	4564
Acapulco phosphates	0.731	+8.8±0.2	≡4557	4555
Ste. Marguerite feldspars	1.105	-0.7±0.4	4567	≡4565

From Brazzle et al. (1999), Gilmour and Saxton (2001).
Relative ages are positive for later ages.

Another technique that might seem sufficient for calculating the conversion factors would be to simply calculate the conversion efficiency of ^{127}I to ^{128}Xe from the neutron flux and the cross-section of the reaction. However, just as in the case of ^{39}Ar production, much of the production comes not from the thermal neutrons but from resonances at higher energies, and the details of the energy spectrum can be a function of the geometry of the irradiation package, as well as self-shielding.

The other piece of information available in a plot such as Figure 3 is the initial isotopic ratio of the daughter element, in this case $(^{129}\text{Xe}/^{130}\text{Xe})_t$, the ^{129}Xe/^{130}Xe ratio before iodine decay began. The initial ^{129}Xe/^{130}Xe ratio, however, is not the intercept of the isochron with the y-axis, as it would be for Rb-Sr or Sm-Nd. Instead, Equation (6) shows that the ordinate-intercept is $(^{129}\text{Xe}/^{130}\text{Xe})_t$ - $CR(^{128}\text{Xe}/^{130}\text{Xe})_t$. A better way of thinking of this is that if the I/Xe ratio is zero, $(^{128}\text{Xe}/^{130}\text{Xe}) = (^{128}\text{Xe}/^{130}\text{Xe})_t$, which is usually well known (Ott 2002, this volume). Thus, $(^{129}\text{Xe}/^{130}\text{Xe})_t$ is the intercept of the isochron with a vertical line at $(^{128}\text{Xe}/^{130}\text{Xe})_t$, as given by the dotted line in Figure 3. If the I/Xe ratio is high, as for the Acapulco phosphates, $(^{129}\text{Xe}/^{130}\text{Xe})_t$ is poorly defined.

All the data will be colinear in Figure 3 only if all the mineral sites that are degassed started out with the same ^{129}I/^{127}I ratio and the same ^{129}Xe/^{130}Xe ratio, and there were no subsequent events that affected different sites differently. The latter assumption is the one mostly likely to be invalid. In the common case of a later event that leads to partial resetting, some of the points will fall off the line. For example, in Figure 3a, it is the data points from the higher temperature steps that define the line. Gas released at lower temperatures comes, by definition, from sites that are easier to degas, so it is quite likely that the Renazzo meteorite has been partially degassed. In fact, those are also the sites that would be easier to contaminate with terrestrial iodine, which would produce the same effect, so it is often impossible to tell which has happened. In some cases, though, the pattern of apparent initial iodine isotopic ratios is consistent enough with the pattern expected from diffusive loss that it is possible to determine apparent thermal histories.

The initial ^{129}Xe/^{130}Xe or ^{129}Xe/^{132}Xe ratio, as well, potentially contains information about the evolution of the system before decay began (Swindle 1998; Swindle et al. 1991b), just as the initial isotopic compositions of Sr or Nd do. However, it is not commonly used, and Gilmour et al. (2001) suggest that it should not be used, in part because some of the initial Xe isotopic ratios derived (such as the one for Renazzo in Fig. 3a) are less than 1, less than any suspected trapped Xe component (Ott 2002). They argue that plots such as Figure 3 are more complicated than described above, and that the trapped Xe must be accompanied by some trapped iodine as well.

Comparison with ^{40}Ar-^{39}Ar. There are several differences between I-Xe dating and ^{40}Ar-^{39}Ar dating. The most fundamental difference is that a ^{40}Ar-^{39}Ar age is intrinsically an absolute age, while I-Xe ages are not. Traditionally, I-Xe ages have been reported as either initial $^{129}I/^{127}I$ ratios or as ages relative to other I-Xe ages. Since it is often not obvious what event last reset the xenon isotopic composition, and most absolute age systems do not have the resolution that I-Xe does, calibration is difficult. However, the Pb-Pb system can now produce 1-2 Ma scale precision, and has been applied to phosphates in meteorites that also have enough iodine to produce precise I-Xe ages. The most common intercalibration currently in use (Table 3, below) involves the assumption that the I-Xe and Pb-Pb ages of Acapulco phosphates date the same event, and hence they represent the same age (Brazzle et al. 1999; Nichols et al. 1994). To try to better calibrate I-Xe with chronometry based on the extinct radionuclide ^{53}Mn, Gilmour and Saxton (2001) have suggested an alternate calibration, assuming that the Pb-Pb and Mn-Cr ages given by the meteorite LEW86010 represent the same event and that the Mn-Cr and I-Xe ages of feldspars from the ordinary chondrite Ste. Marguerite give the same age. The difference in absolute I-Xe ages between the two calibration schemes is less than the uncertainty in the Pb-Pb age of Acapulco phosphates, so they are not really in conflict with one another.

To determine an age from an initial iodine isotopic composition, we can rewrite the equation of radioactive decay as

$$(^{129}I/^{127}I)\ (t_2) = (^{129}I/^{127}I)\ (t_1) \times \exp[-\lambda\ (t_2 - t_1)]\ ,$$

where t_1 and t_2 are two different times, and λ is the decay constant for ^{129}I. From this, it is possible to solve for $(t_2 - t_1)$, the time difference. Without the calibration to give the absolute age of one of those times, it is only possible to give relative ages. Incidentally, when the first I-Xe experiments were performed, the best determination of the half-life of ^{129}I was 17.2±0.9 Ma (Katcoff et al. 1951), which was used in early I-Xe experiments, and then was often used for consistency even after a more precise determination of 15.7±0.4 Ma became available (Emery et al. 1972). At present 15.7 Ma is most commonly used (Table 1), even though 17 Ma may actually be more accurate (Holden 1990). Relative ages are proportional to the half-life, so conversion from one to the other simply requires multiplying by the ratio of the adopted half-lives.

Another fundamental difference is that while potassium is a minor element, and there are minerals that can be identified that contain relatively large amounts of potassium (e.g., orthoclase or biotite), iodine is a trace element, and is seldom a major part of a mineral. Hence it is usually not known what mineral contains the bulk of the iodine in an extraterrestrial sample. A notable exception is the mineral sodalite in the Allende meteorite, which contains the bulk of the iodine in that meteorite (Kirschbaum 1988). Although the siting of the iodine has been identified in a few other cases, discussed below, it may be that most of the iodine is along grain boundaries and in interstitial sites.

Although the early ^{40}Ar-^{39}Ar and I-Xe experiments were one and the same (consisting of xenon and argon analyses of the same irradiated samples), they are optimized in slightly different conditions. For ^{127}I, there is a large capture cross section from thermal neutrons, while the highest neutron-capture cross section of ^{39}K is at higher energies, so different reactors are preferred for the two types of experiments (see McDougall and Harrison (1999) for a list of reactors commonly used for ^{40}Ar-^{39}Ar dating). In addition, since Ar diffuses more readily than Xe, the crucial temperature steps in a ^{40}Ar-^{39}Ar stepwise heating experiment are often at lower temperatures than the crucial steps in an I-Xe experiment.

There are also several differences between the ways I-Xe and ^{40}Ar-^{39}Ar data are usually presented. I-Xe data are usually presented in isochron form, ^{40}Ar-^{39}Ar data usually are not. Instead, an initial argon isotopic ratio (in this case $^{40}Ar/^{36}Ar$) is assumed, and an apparent age (really, an apparent $^{40}Ar^*/K$ ratio) calculated. Next, the data are presented as an age spectrum plot (Kelley 2002). In this representation, if all the mineral sites formed at the same time with the same $^{40}Ar/^{36}Ar$ ratio, they will all have the same apparent age, and the result will be a flat series of points across the plot. Or, if some low temperature sites have been degassed more recently, the result will be a series of steps up to what appears to be a plateau, in which case the value of the plateau is usually interpreted to be the age of resetting, and the lower temperature steps may be used to define the thermal history. In some cases, including the studies of the shock-melted meteorites discussed above and studies of the shergottite Martian meteorites that will be discussed in the chapter on Martian noble gases (Swindle 2002), there can be a combination of partial degassing and even injection of gas from the atmosphere or surrounding rock that makes the plateau plot difficult or impossible to interpret. Similar problems can occur with the interpretation of the I-Xe system, particularly of shocked meteorites (Hohenberg et al. 2000). It should be mentioned that in cases where the I/Xe ratio is high, so the assumed xenon isotopic composition has little affect on the apparent age, I-Xe experiments are also often presented as age spectrum plots.

Finally, it should be noted that just as a typical ^{40}Ar-^{39}Ar irradiation also produces argon from calcium and chlorine, a typical I-Xe irradiation also produces xenon from tellurium, barium and uranium (Turner 1965) and krypton from bromine. Conversion factors for many or all of these reactions have been measured or calculated by several authors, most recently by Irwin and co-workers for use in studies of fluid inclusions (Irwin and Reynolds 1995; Irwin and Roedder 1995). Many of these elements are not routinely determined by other techniques, so direct comparisons with other techniques are rare (but see (Garrison et al. 2000; Swindle et al. 1991b)).

Applications. The iodine-xenon system has been reviewed by Swindle and Podosek (1988), and, more recently, by Gilmour (2000). In this chapter, we will highlight some of the areas in which I-Xe has been, or is likely to be, applied the most.

The first I-Xe experiments, like the Renazzo experiment described above, were on whole-rock samples, and served primarily to roughly establish the initial $^{129}I/^{127}I$ ratios typical of the early solar system. This, combined with nucleosynthetic calculations, made it possible to make estimates of the time between the synthesis of ^{129}I and the formation of solids in the solar system (e.g., Hohenberg et al. 1967). Initial results suggested a difference of approximately 100 Ma, and provided the first experimental constraint of this important parameter. More recently, other extinct radionuclides with even shorter half-lives have been documented within meteorites. They do not necessarily refer to the same nucleosynthetic event as ^{129}I, so the ~100 Ma timescale for ^{129}I still appears to be correct (Cameron 1993).

Later, as more I-Xe experiments were performed, they were used in an attempt to develop higher precision chronology within the early solar nebula. For example, the I-Xe system was applied to ordinary chondrites of different classes in an attempt to determine the details of chondrite metamorphism, as discussed above. A lack of correlation with petrographic grade was taken as an indication as either that metamorphism occurred in rubble piles, so cooling rates and times did not correlate with petrographic grade (Taylor et al. 1987) or that ^{129}I was distributed inhomogeneously in the early solar nebula (Clayton 1980; Clayton et al. 1977), so that the I-Xe system could not be used as a chronometer. In fact, the biggest problem was probably the fact that whole-rock samples were being analyzed, and different rocks had different amounts of various iodine carriers that required different time-temperature histories to reset. Burkland et al. (1995) showed that a single chondrite, Bjurbole, contained at least three different iodine carriers, some of which would not have been reset by the metamorphism that chondrite experienced. When samples of feldspar and the phosphate apatite, both secondary minerals, were analyzed (Brazzle et al. 1999), petrographic grade did correlate with apparent age (Fig. 4). Feldspar and apatite are presumably not the only minerals that contains enough iodine use for I-Xe, and that could be reset or created at temperatures low enough to be dating some aspect of chondrite metamorphism, but they are the only ones so far applied.

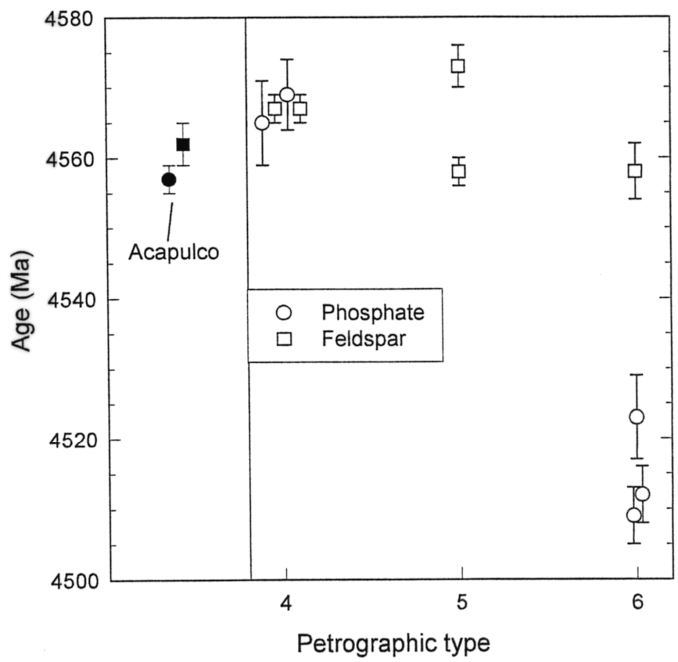

Figure 4. Correlation of I-Xe ages with petrographic type for phosphates and feldspars from ordinary chondrites. Data are from Brazzle et al. (1999). Note that type 6 (more equil-ibrated) chondrites have later ages than type 4 chondrites. The very old age for one of the type 5 chondrites may only be apparent, an artifact of a shock event (Hohenberg et al. 2000). The absolute ages are calibrated by assuming the I-Xe and U-Th-Pb systems for Acapulco phosphates reflect the same time.

Another process that the I-Xe system should be effective at dating is aqueous alteration, since iodine is extremely mobile in water. Several I-Xe experiments on objects that have been affected by aqueous alteration have been interpreted in this fashion (Krot et al. 1999; Swindle et al. 1991a; Whitby et al. 2000). The biggest problem is separating out the details of the complicated histories that these objects have experienced.

Iodine-xenon experiments have also been applied, but only with minimal success, to dating the formation of chondrules and calcium-aluminum-rich inclusions (Fig. 5). These two types of objects make up large portions of many meteorites, and are believed to be among the earliest solids formed in the solar nebula.

The formation of chondrules, the spherical objects that make up the bulk of many of the most common, "chondritic," meteorites, is particularly contentious. Two conferences more than a decade apart (Hewins et al. 1996; King 1983) each failed to reach consensus on the mechanism of the origin of chondrules, although formation from molten droplets of rock is a feature of most of models of their origin. Clearly, a chronology of chondrule origin would be desirable. I-Xe experiments have been performed on nearly 100 individual chondrules (see Swindle et al. 1996; Whitby et al. 2002). A wide variety of ages has been observed, of which the oldest must represent a minimum time of the formation of the first chondrules. Much of the variation probably represents secondary processing, rather than differences in formation ages (Swindle et al. 1991a,b). But even when meteorites with very simple histories are considered, there is still a range in ages. For example, a 2.6 Ma range in I-Xe ages in enstatite chondrites has been interpreted as representing the minimum duration of chondrule formation (Whitby et al. 2002).

Calcium-aluminum-rich inclusions (CAIs) are among the most refractory objects within meteorites (MacPherson et al. 1988), and hence are often thought to have been among the first objects formed in a solar system that was cooling from high temperatures. They are primarily found in meteorites that have not been heavily metamorphosed. I-Xe experiments have been performed on more than a dozen individual CAIs (Swindle and Podosek 1988), again giving a range in ages. However, there is a problem with this. Most of the analyses have come from CAIs within a single meteorite, the carbonaceous chondrite Allende. Within Allende, most of the iodine is contained within the mineral sodalite (Kirschbaum 1988), which is known to be a secondary phase. Hence, the I-Xe ages of Allende CAIs presumably reflect alteration, rather than formation. Swindle et al. (1996) argued that the oldest whole-rock I-Xe ages of meteorites containing CAIs are older than the oldest I-Xe ages from chondrules by several Ma, suggesting that CAIs formed that much earlier. Further, they also pointed out that it had been argued that CAIs have higher abundances than chondrules of two other extinct radionuclides, ^{53}Mn (3.7 Ma halflife) and ^{26}Al (0.7 Ma half-life). However, when Gilmour and Saxton (2001) tried to intercalibrate all of the chronometers and put absolute ages on the various samples (Fig. 5), they concluded that all of the old CAI ages are problematical. In fact, they suggest that the isotopic systems in the CAIs, particularly Al-Mg, may have been disturbed by isotopic heterogeneity in the early solar system. In addition, they suggested that the fact that they found I-Xe ages older than 4570 Ma for some other samples, even though the best Pb-Pb age of a CAI is 4566 Ma (Göpel et al. 1991) might mean that the I-Xe system is less easily reset than the Pb-Pb system (Gilmour et al. 2000).

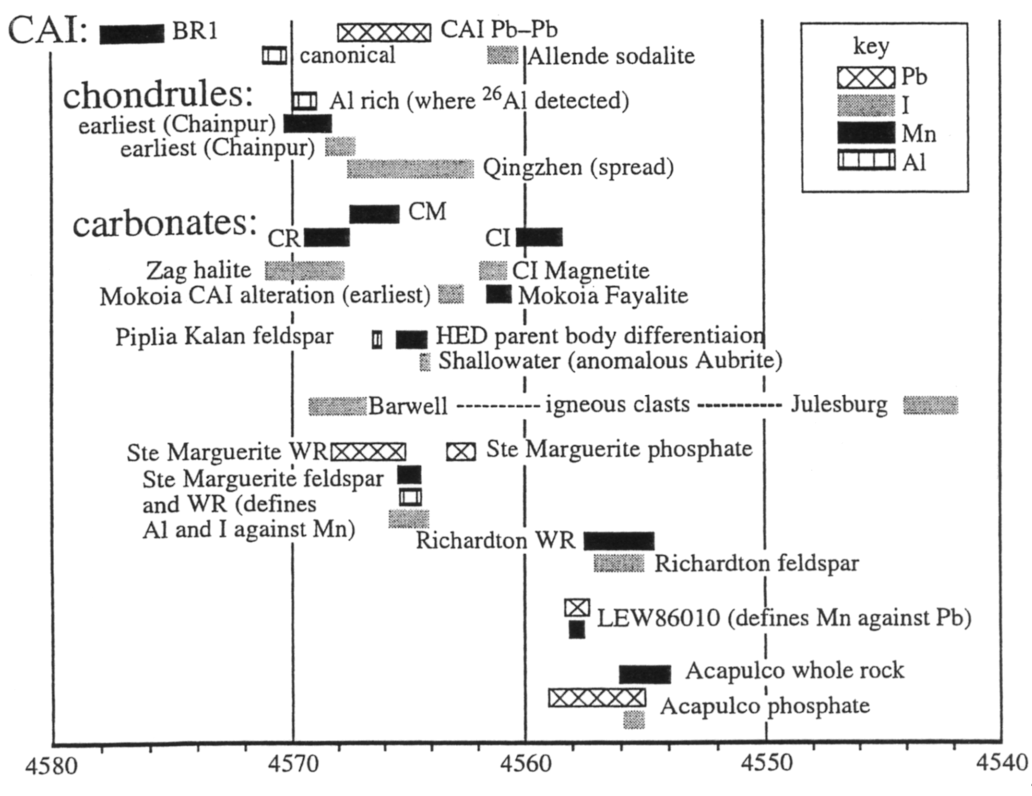

Figure 5. Summary of a variety of ages in the early solar system, as determined by I-Xe, Al-Mg, Mn-Cr and Pb-Pb. Absolute I-Xe are generated by assuming Acapulco phosphate (near bottom of figure) became closed to the I-Xe and U-Th-Pb systems at the same time, although an alternative calibration point is to assume that Ste. Marguerite phosphates are the same age in both the I-Xe and

Pb-Pb systems. [Used by permission of the editor of *Philosophical Transactions: Mathematical, Physical and Engineering Sciences*, from Gilmour and Saxton (2001), Fig. 2, p. 2044.]

This ties into another unsolved problem: what is the oldest age in the solar system? In the I-Xe system, this translates into the highest $^{129}I/^{127}I$ ratio. Several different authors (Gilmour 2000; Gilmour and Saxton 2001; Swindle and Podosek 1988; Swindle et al. 1996) have all discussed this question and come to different conclusions, so it is probably safest to say that the answer is not yet known, although current arguments range between 1.3×10^{-4} and 1.45×10^{-4} for the $^{129}I/^{127}I$ ratio, corresponding to absolute ages between 4570 and 4580 Ma. There are several factors that can lead to a $^{129}I/^{127}I$ ratio that appears higher than it is, including an improperly calibrated monitor (Hohenberg et al. 1981, 2000) or a complicated history involving shock (Caffee et al. 1982) or aqueous alteration (Turner et al. 2000). Finally, of course, the intercalibration between I-Xe and Pb-Pb is not yet certain. Since the I-Xe system is, by nature, not an absolute chronometer, absolute I-Xe ages are derived by normalization of the two systems. Acapulco phosphate has a Pb-Pb age with about 2 Ma uncertainty, introducing this uncertainty into all absolute I-Xe ages calibrated in this fashion (Brazzle et al. 1999), while I-Xe ages calibrated via Mn-Cr (Gilmour and Saxton 2001) have a smaller statistical uncertainty, but the calibration involves an extra step. However, with increasing analyses of mineral separates for I-Xe making it both easier to intercalibrate systems and to understand the processes being dated, this problem is becoming more tractable.

^{244}Plutonium

With its relatively long 82 Ma half-life, it is not surprising that ^{244}Pu was present in the early solar system. In fact, the presence of ^{244}Pu in the early solar system was postulated at nearly the same time ^{129}I was discovered (Kuroda 1960), although it took another decade for its existence to be definitively proven.

One difficulty is that there is no stable isotope of plutonium with which to compare its abundance. To really quantify its abundance, it is necessary to consider the amount of ^{244}Pu relative to an isotope of a similar element. The definition of "similar" depends on the problem to be addressed. In studies of nucleosynthesis, the "similar" element used is usually uranium, another actinide, which is produced in the same stellar environments. In studies of the history of specific meteorite parent bodies, the "similar" element is more commonly a light rare earth element (LREE) like neodymium, since the geochemical behavior of plutonium is apparently most similar to that of the LREE. We will discuss the details of the experimental technique of each approach below.

The other difficulty is that unlike ^{129}I or ^{40}Ar, each of which decays to a single specific noble gas isotope, the fission of ^{244}Pu can produce any of several Xe isotopes. However, the spectrum of the Xe isotopes produced makes it possible to distinguish the actinides (Table 2). In fact, the original suggestion of ^{244}Pu was made on the basis of its fission spectrum inferred from the composition of the terrestrial atmosphere (Kuroda 1960). Subsequently, the predicted spectrum was indeed measured on artificial ^{244}Pu (Alexander et al. 1971). Only the heaviest xenon isotopes are produced in fission. Since stable nuclei become progressively more neutron-rich at higher atomic number, fission produces two neutron-rich fragments, each of which then undergoes a series of β^- decays until it reaches stability. Hence fission will ultimately produce only those isotopes which are not shielded by stable isotopes to their lower right in the chart of the nuclides. In the case of xenon, the unshielded isotopes are at masses 129, 131, 132, 134 and 136. Fission typically produces unequal sized fragments, and ^{136}Xe is near one of the peaks of production for actinide fission (^{129}Xe is far enough from the peak that there is usually too little production to be usefully measured). The other peak is near krypton, but only ^{86}Kr

is both close to the production peak and unshielded, so there is no krypton isotopic spectrum that can be used to diagnose actinide fission.

Nucleosynthesis studies. For nucleosynthesis studies, a neutron irradiation, much the same as the ones for ^{40}Ar-^{39}Ar or I-Xe, is done. But the reaction of interest is neutron-introduced fission of ^{235}U. As noted in Table 2, this produces a different isotopic spectrum than ^{244}Pu decay. Since there are four isotopes affected, and only two spectra, it is usually possible to determine the ratio of ^{244}Pu fission to ^{235}U fission and hence, with the help of a monitor, the ^{244}Pu/^{238}U ratio (Hudson et al. 1989). The more common uranium isotope, ^{238}U, has a far lower cross-section for neutron-induced fission, but can be used as a reference isotope because ^{235}U/^{238}U is constant in all samples measured so far. Natural samples do contain some Xe from spontaneous fission of ^{238}U, but in most meteorites, the amount is negligible compared to the amount of ^{244}Pu fission Xe, since the fission branching ratio of ^{244}Pu is so much higher (Table 1).

For example, Figure 6 shows a three-isotope plot involving ^{130}Xe, ^{134}Xe, and ^{136}Xe. Points are colinear, suggesting a two-component mixture. In this case, the two likely components are trapped Xe (to the upper right) and a fission component (in the lower left). Note that the fission component is neither ^{244}Pu fission nor neutron-induced fission of ^{235}U, but rather a mixture of the two. Knowing the compositions of the two possible fission components, it is possible to determine how much of each isotope is a result of each actinide. If, for example, we determine the relative amount of ^{136}Xe from each, then

$$[^{136}\text{Xe}]_{244} = B_{244} \times y_{244,136} \times [^{244}\text{Pu}] \tag{7a}$$

$$[^{136}\text{Xe}]_{235} = [\text{n}] \times B_{235,\text{n}} \times y_{235,136} \times (^{235}\text{U}/^{238}\text{U}) \times [^{238}\text{U}] \tag{7b}$$

where the B's represent branching ratios (for fission, instead of alpha decay), the y's

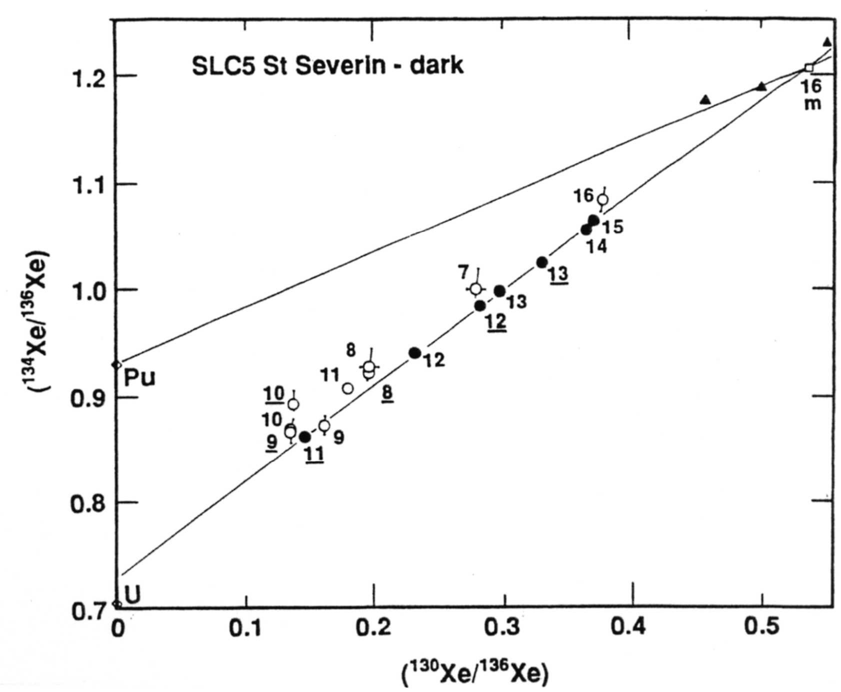

Figure 6. Three-isotope plot for an irradiated sample of the St. Severin meteorite, demonstrating the presence of ^{244}Pu-derived fission Xe. Triangles near the upper right-hand corner represent plausible trapped (fission-free) compositions. "Pu" and "U" represent the fission composition of Xe derived from ^{244}Pu and ^{235}U (for this particular experiment). Labels next to points are temperatures in

hundreds of degrees Celsius. Underlined numbers are 50°C higher (e.g., 13 represents 1350°C). [Adapted by permission of the editor of the *Proceedings of the 19th Lunar and Planetary Science Conference*, from Hudson et al. (1989), Fig. 4, p. 551.]

represent the yield of ^{136}Xe in each of the fission processes, [n] is the total neutron fluence, the isotopic values in square brackets represent the absolute amounts of those isotopes, and the U ratio is the measured ratio. Simply taking a ratio of Equation (7a) to (7b), it is clear that the ratio of ^{244}Pu/^{238}U is proportional to the ratio that can be measured.

The initial ^{244}Pu/^{238}U ratio of the solar system is valuable for studies of the history of the material that became a part of the solar system. The most massive stable nucleus is ^{209}Bi, which contains far less nucleons than ^{244}Pu, ^{238}U or ^{235}U. Hence, these actinides can only be synthesized by rapid neutron capture ("r-process"). Their relative rates of production can be predicted from theoretical considerations, so their abundances at the beginning of the solar system can be used to infer the time between cessation of r-process activity and the formation of the solar system (Hudson et al. 1989; Jones 1982). As mentioned above, it is particularly useful when combined with data on ^{129}I, another r-process nuclide (Cameron 1993), and confirms the ~100 Ma timescale suggested by ^{129}I.

Pu-Xe dating. Decay of ^{244}Pu can be used as a chronometer of the first 100 Ma for some specific meteorite parent bodies. Both Pu and the LREE tend to be concentrated in refractory minerals like phosphates. Which LREE is the best proxy for Pu? Various authors have suggested Nd (Lugmair and Marti 1977), Sm (Jones and Burnett 1987), or Pr or Ce (Boynton 1978). There are no neutron-induced reactions that produce a rare gas from any of the LREE, but all of these, particularly Nd, do produce the light xenon isotopes like ^{124}Xe and ^{126}Xe through cosmic-ray-induced spallation reactions (Wieler 2002, this volume). In fact, in many cases, the LREE (and presumably Pu), are probably not fractionated much from each other. Hence, if the cosmic ray dose (i.e., the cosmic ray exposure age) is known, and the production rate of isotopes like ^{124}Xe and ^{126}Xe is also known, then the abundance of the LREE can be calculated. Then the ratio of $^{136}Xe_{244}$ (Pu-derived fission ^{136}Xe) to $[^{126}Xe]_{spall.}$ (LREE-derived spallation ^{126}Xe) is proportional to the ratio of ^{244}Pu to LREE, since

$$[^{126}\mathrm{Xe}]_{spall.} = P_{\mathrm{LREE},126} \times [\mathrm{T}_{exposure}] \times [\mathrm{LREE}] \qquad (8)$$

where the P is the production rate of ^{126}Xe from LREE and $T_{exposure}$ is the time of exposure to cosmic rays. Once again, $^{136}Xe_{244}$ is as given in Equation (7a).

Varieties of this technique, Pu-Xe dating, have been applied to igneous meteorites like the eucrites (Marti et al. 1977, 1979; Miura et al. 1998; Shukolyukov and Begemann 1996a), which have enough actinides and LREE to make the measurement possible, and have histories which extend over several hundred Ma, so that the measurement is useful. One of the most interesting results to come from this is the fact that in some cases, the Pu-Xe ages are older than Pb-Pb ages on the same meteorite, suggesting that the Pu-Xe system is more resistant to resetting than the Pb-Pb system (Shukolyukov and Begemann 1996a), consistent with the suggestion that the I-Xe system might be more retentive than Pb-Pb (Gilmour et al. 2000).

The Pu-Xe technique does have its drawbacks. The determination of LREE abundances is dependent on the determination of the cosmic-ray exposure age, although by analyzing Kr at the same time, it is possible to determine precise ^{81}Kr-Kr exposure ages (Miura et al. 1998; Shukolyukov and Begemann 1996b). Another potential problem with the spallation-produced Xe is that Ba also produces Xe isotopes through spallation. Two approaches to dealing with Ba have been tried. Marti et al. (1979) used the isotopic composition of the spallation Xe to determine the Ba/LREE ratio. Other authors (Miura et al. 1998; Shukolyukov and Begemann 1996a) have used measurements of Ba and LREE determined by other techniques on other samples of the same meteorite. More problematical, the correlation of Pu with Nd (or any other LREE) is unlikely to be perfect (Crozaz et al. 1989). As a result of this, plus the inherent uncertainties resulting from the determination of the correlation, the best uncertainties achieved are roughly 20 Ma.

Other fissioning nuclides?

Only a few years after the presence of ^{244}Pu had been suggested, and well before it had been demonstrated definitively, a different fission-like Xe spectrum had been identified in Renazzo (Reynolds and Turner 1964) and other carbonaceous chondrite meteorites. The isotopic spectrum did not match any known fissioning nuclide, and there were other properties that were also inconsistent with actinide fission. Most prominently, the light Xe isotopes such as ^{124}Xe and ^{126}Xe, which could not be produced by fission, were also enriched. The component was dubbed "carbonaceous chondrite fission" (or "CCF") Xe, even though there were questions from the start about whether it was truly a fission component. There were other actinides that were potential progenitors, such as ^{247}Cm, with a half-life of 15.6 Ma, but these were ruled out on various grounds. If it was a fission component, the most likely progenitor seemed to be a "superheavy" element, with a mass number of roughly 115, lying in a predicted island of stability far beyond the elements which either occur in nature or had been synthesized (Anders et al. 1975). In fact, "CCF" Xe was not a fission component at all, but rather a nucleosynthetic effect, now known as "H"-Xe, contained in pre-solar grains (Ott 2002, this volume). In a classic example of dogged pursuit of a scientific problem, the same laboratory that put together the most convincing story for a superheavy progenitor was also the one that laid that theory to rest after a decade of progressively more detailed studies (Lewis et al. 1987).

Other radiogenic noble gases

Two other short-lived radionuclides may have produced measurable amounts of radiogenic noble gases in meteorites. ^{22}Sodium has a half-life of only 2.6 years (Table 1). Remarkably, there are meteorites that contain evidence of ^{22}Na decay. However, ^{22}Na is a special case—the samples in which evidence of its decay is found are presolar grains, and the decay produces the component known as Ne-E(L) or Ne-R (Ott 2002). So while the ^{22}Na was incorporated into a solid within a few half-lives of synthesis, its existence puts constraints on the formation of grains around supernovae rather than on the timing of any process within the solar system.

Another radionuclide, ^{36}Cl, has a half-life (300 ka) that is not as short as that of ^{22}Na, but is still shorter than ^{129}I or ^{244}Pu. Hence, it would be expected to have been less abundant in the early solar system. Furthermore, it is more difficult to determine its abundance relative to other Cl isotopes. The problem is that while there is a stable chlorine isotope which will produce a noble gas through a neutron capture (^{37}Cl producing ^{38}Ar), the only Ar isotope left that could be used as the denominator in a plot such as Figure 3 is ^{40}Ar, and the change in the ^{36}Ar/^{40}Ar ratio is most likely dominated by K decay rather than Cl decay. Clayton (1977) suggested a way to get around that problem by using only the ^{36}Ar/^{38}Ar ratio and comparing two identical samples, one irradiated and one unirradiated, but this has not been successfully applied.

The only meteorite in which evidence of live ^{36}Cl has been found is Efremovka, a carbonaceous chondrite that has suffered less processing than most meteorites. Murty et al. (1997) have reported the presence of excess ^{36}Ar, presumably from the decay of ^{36}Cl, in a sample of matrix from Efremovka, but no one has successfully measured the ^{36}Cl/^{35}Cl ratio. Murty et al. suggested a ^{36}Cl/^{35}Cl ratio of a little more than 10^{-6}, based on the excess ^{36}Ar and the Cl abundance. Since the half-life of ^{36}Cl is so short, this would be one of the shortest-lived radionuclides yet identified in the solar system. Since evidence of a radionuclide generally disappears within a few half-lives, the very presence of ^{36}Cl would require it to have been synthesized no more than 1 to 2 million years before incorporation into solar system solids. However, another study (Swindle et al. 2001) failed to find any evidence for ^{36}Cl in a similar Efremovka matrix sample, so its existence remains only a tantalizing possibility at this point.

With ^{36}Cl, the roster of potential noble-gas-producing radionuclides is probably exhausted. However, with the exception of ^{36}Cl, studies of radiogenic noble gases have moved from the age of discovery and confirmation to the age of detailed investigation of what they have to say about solar system history.

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