Reviews in Mineralogy & Geochemistry
Vol. 55, pp. 25-64, 2004

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An Overview of Isotopic Anomalies in Extraterrestrial Materials and Their Nucleosynthetic Heritage

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INTRODUCTION

Isotopic anomalies are expected in primitive meteorites since astronomical observation and astrophysical modeling of stars predict a great variety of stellar processes. Protostellar clouds should partially preserve the memory of this diversity in solid grains. Since 1970, high precision mass spectrometry and high resolution ion probes have led to the discovery of numerous isotopic anomalies, which were rapidly associated with nucleosynthetic processes. A general rule is that small isotopic effects (parts in 10^3–10^4) are observed in centimeter size samples, whereas order of magnitude variations are observed at the micron scale in circumstellar grains.

Refractory materials in primitive meteorites were investigated first as they have the best chance of escaping homogenization in the early solar system. Inclusions in C3 carbonaceous chondrites exhibit widespread anomalies for oxygen and the iron group elements. Only a few members, dubbed "FUN" (for "Fractionated and Unknown Nuclear" effects), also display anomalous compositions for the heavy elements. Anomalies in inclusions have generally been connected with explosive or supernova nucleosynthesis.

Several types of presolar circumstellar grains have been separated from the matrix of chondrites: diamonds, silicon carbide, graphite, oxides. The isotopic ratios of the light elements (C-N-O) vary over several orders of magnitude in these grains. Only a few measurement have been performed for heavier elements with generally s-process signatures. AGB stars at different stages of their evolution are thought to be the sources of most circumstellar grains. Nevertheless grains with supernova signatures have also been found. For Cr and Mo in bulk primitive carbonaceous chondrites (C1, C2), large isotopic differences exist between the different major mineral phases of the bulk rock.

A number of now extinct radioactive isotopes have existed in the early solar system. This is shown by the variations that they induce in the abundances in their daughter nuclides. Their main use is in establishing a chronology between their parental presolar stellar sources, and the formation of the solar system and the planets. An active debate is presently going on whether some of these short-lived nuclides could have been made within the early solar system by an intense flux of energetic protons from the young sun.

This chapter presents an updated report of the data with necessarily some limitations, but I will try to keep the most striking features and highlight the clearest relation with nucleosynthesis in stars.

1529-6466/04/0055-0002$05.00

Historical background

In 1970 only three kinds of isotopic variations were known from measurements on solar system samples. Isotopic ratios involving the decay product of long-lived radioactive nuclei (^{40}K-^{87}Rb-^{147}Sm-^{176}Lu-^{187}Re-U-Th) constitute the first family. The second relates to physico-chemical fractionation of the light elements (H-C-N-O) which is mass dependent (hereafter referred to as "linear effects"). The third comprises the products of cosmic-ray spallation which produce very minor amounts of some nuclei (see Reedy et al. (1983) and Honda (1988) for an overview). A number of investigations for extinct radioactivities and isotopic heterogeneities had been carried out earlier but failed to demonstrate resolved isotopic variations (Wasserburg and Hayden 1955; Anders and Stevens 1960; Murthy and Sandoval 1965; Eugster et al. 1969; Schramm et al. 1970; Huey and Kohman 1972). The searches for isotopic variations due to irradiation by an early active Sun failed as well (Fowler et al. 1962; Burnett et al. 1966). Hence until the early 1970s, it was generally accepted that the solar system had undergone a high temperature stage volatilizing all, or almost all solids, and homogenizing at least the inner solar system where the samples were coming from (Cameron 1969). Nucleosynthetic theory in the same time was directed toward reproducing the solar system abundances (Burbidge et al. 1957).

One striking exception was the very early discovery of ^{129}I decay to ^{129}Xe (Jeffery and Reynolds 1961). This discovery reflects the particular properties of rare gases which are nearly absent in telluric planetary bodies. Because they are not diluted by high abundances of isotopically 'normal' noble gases, anomalies in rare noble gas components were the first to be detected. This is also the reason for the Xe record of the fission of ^{244}Pu (Rowe and Kuroda 1965). From the available data on short-lived nuclides at that time, it was concluded that the last nucleosynthetic input into the protosolar cloud predated the formation of the planets by 100-200 Ma.

The first evidence for non-mass dependent isotopic variations came from oxygen in the refractory inclusions of the carbonaceous chondrite Allende (Clayton et al. 1973). The large effects found for oxygen, a major constituent of rocks, acted as a trigger for numerous high precision isotopic studies for as many elements as were possible to measure, and creating a new field in planetology. The outcome of this profusion of successful studies has been summarized in several past reviews, with special emphasis on groups of elements or objects (Clayton et al. 1988; Lee 1988; Anders and Zinner 1993; Zinner 1997; Goswami and Vanhala 2000). The major advances in the field roughly correspond to two waves of new data: the high precision isotopic measurement in the cm size inclusions of the C3 chondrites and the discovery of presolar grains which thereafter were investigated with microprobe techniques. This is not fortuitous and connects to two significant instrumental evolutions. The lunar exploration program led to the development of a new generation of mass spectrometers, able to resolve 10^{-4} (100 ppm) relative isotopic differences (Papanastassiou and Wasserburg 1969; Wasserburg et al. 1969). The sample sizes of the inclusions is the same order of magnitude as some precious lunar samples. The development of high resolution SIMS/ion probe (Lepareur 1980) was also under way during the 1970s and was shown to be suitable for natural micro sample isotopic analysis (Hutcheon 1982).

The definition of isotopic anomalies

In a strict sense "isotopic anomalies" are defined here as isotopic variations that are not understood to have been generated from a once uniform reservoir by processes acting within the solar system. They may result from incomplete homogenization of isotopically very diverse nucleosynthetic components. They potentially possess two types of information: the nature of the nucleosynthetic sources of the material, and the processes acting during their transfer from within stars to their preservation in meteorites. The definition of the "anomalous" composition reflect the status of knowledge around 1970 and by extension has been extended to all variations which were not identified in 1970. Effects related to extinct radioactivities, are also usually referred to as "Anomalies" as well as non-linear physico-chemical effects (Thiemens 1999; Clayton 2002) which will be briefly discussed at the end of this review. Extinct radioactivities can induce isotopic variations within the solar system starting with an isotopically homogeneous reservoir. They then do not strictly correspond to the first definition. Nevertheless they enter into the extended definition and are obviously related to nucleosynthesis occurring shortly before the formation of our planetary system; it is therefore useful to include them in this chapter.

THE MEASUREMENT OF ISOTOPIC ANOMALIES

Many of the isotopic anomalies would not be accessible without the ability to measure isotopic ratios with precisions down to 20-100 ppm. This is well below the level of instrumental fractionation for traditional thermal ionization mass spectrometers. From the experimental point of view, the data are often not interpretable in a straightforward manner and an isolated set of data may lead to ambiguities. These ambiguities will be greatly reduced or eliminated if more than one isotope ratio for the element under study can be measured, an appropriate normalization scheme is used to correct for instrumental fractionation, the presence of anomalous isotopic patterns in neighboring elements, and other astrophysical considerations are also taken into account. In addition, natural linear (or Rayleigh) mass fractionation is often present in primitive meteoritic samples as a result of partial evaporation or condensation. Nevertheless, the main observations, which will be presented in the following review, can be divided in four categories.

Large isotopic variations

Isotopic variations are large enough to exceed terrestrial variations by more than about an order of magnitude. As an example, this is the case for ion probe work on the CNO isotopes of circumstellar grains (see Section "Presolar stardust grains..."). These effects can be attributed unambiguously to nucleosynthetic reactions.

Two isotope elements and small effects

For some elements the variations are commensurate with, or smaller than the instrumental reproducibility or terrestrial variations, and it is difficult to distinguish between linear fractionation due to physico-chemical effects and nuclear effects. All the interpretation depends on the connections between the variation in the different elements and the phase they are in. This is also relevant to a number of radiochronometric systems in which the decay product belongs to an element having only two isotopes (e.g. ^{107}Pd-^{107}Ag, ^{205}Pb-^{205}Tl). Modern ICPMS instruments are able to resolve differences much smaller than the possible natural mass dependent effects (e.g., Carlson and Hauri 2001a, b). A correlation with the parent-daughter elemental ratio usually indicates that radioactive decay has occurred. Nevertheless, this interpretation must be used with caution when the overall isotopic ratio variations in the daughter element are small. It may be possible that in some cases processes such as volatilization, producing the spread in the parent-daughter ratio, could also produce mass dependent fractionation in the daughter element, resulting in a pseudo-isochron. Cross-checking with similar mineralogical situations without radioactive decay from the parent, alleviates this problem.

Three isotope elements and small effects

Non linear variations can be smaller (a few ε units) than instrumental fractionation in TIMS instruments (a few ‰ per mass unit). It is then the choice of the isotopic pair used for normalization which determines that the third isotope displays non-linear effects. ICPMS is able to measure natural fractionation with a precision close to that obtained for non-linear effects (Carlson and Hauri 2001a; Kehm et al. 2003) but, if we take the instrumental fractionation observed in TIMS instruments as the possible extent of fractionation in natural processes, ICPMS does not lift this particular ambiguity. The choice of the normalizing pair is not unambiguous and has to be sustained by interelement comparison and theoretical considerations based on nucleosynthetic models. As an example when an extinct radioactivity is examined, the variations are expected on the daughter isotope in correlation with the elemental parent daughter ratio.

Elements having 4 and more isotopes

They are used as above, but in general they lead to more straightforward interpretations. The correction for instrumental fractionation involves an isotope pair for which the measurements are in agreement with the terrestrial ratio, whether this choice results from the measurement itself or from model considerations. In cases where all the ratios are different from the terrestrial ratios, model considerations are used to interpret the data. In the most common cases, one isotope displays wider variations than the others and constitutes a guideline for modeling the origin of the anomalies.

Double spike techniques have been used occasionally to quantify the fractionation produced in natural processes, but this does not change the magnitude of non-linear effects as the natural fractionation follows closely the same laws as the instrumental fractionation. For the following, the instrumental fractionation correction is not discussed as this is described in other chapters of this volume. I suggest that the reader consult the original papers reporting the data for the details. In general the exponential law is used (Russell et al. 1978). In its outcome, it is very close to the Rayleigh law which has a better grounding in the physics of evaporation, but is simpler to use in on line ratio calculations.

Units

Units which are used in isotopic work depend on the precision of the measurements. Generally δ units are used for stable isotopes and correspond to permil relative deviation. It is used occasionally also for non linear effects and then they are permil (‰) deviations without reference to mass differences between the isotopes. Since the beginning of the 70s (e.g., Papanastassiou and Wasserburg 1969) thermal ionization data are often given in ε units which are fractional deviation from the normal in 0.01%. With the new generation of more precise instruments, results are sometimes given in ppm (parts per million) relative to a terrestrial standard sample.

For the following text, isotopic "anomalies" always stands for non-linear or non-mass dependent variations; "linear" or "mass dependent" have the same meaning although mass dependent fractionation may not be strictly linear (Rayleigh). Usually, in the first approach the difference is not essential for description

STELLAR EVOLUTION AND NUCLEOSYNTHESIS

Almost all of the elements heavier than He are synthesized in the interiors of stars. The work of Burbidge et al. (1957) gives the theoretical framework for the synthesis of the elements. The experimental evidence of active nucleosynthesis came from the discovery of the unstable nuclei of technetium in the spectra of red giants (Merrill 1952). The solar elemental and isotopic abundances which are taken from the primitive carbonaceous chondrites constitute the guidelines for testing such models (Anders and Grevesse 1989). A minimum of eight basic processes are required to reproduce the observed compositions. Nucleosynthetic theory has expanded since the initial work, but this frame is essentially still valid today. It was also realized that materials expelled into the interstellar medium by stellar winds or explosions contribute to the chemical evolution of the galaxy (Timmes et al. 1995) and become part of the raw material for the next generations of stars. As a result the materials constituting the solar system are a mixture of materials from a vast number of stellar sources (see Clayton 1988) for the primary nucleosynthetic sources of the elements up to Ni). The understanding of isotopic anomalies requires some basic concepts of stellar evolution and nucleosynthesis. The purpose here is only to give a broad outline. For further details, many textbooks (Clayton 1983; Rolfs and Rodney 1988; Bernatowicz and Zinner 1997) and review articles are available if the reader is interested in more details (e.g., Woosley 1986; Woosley and Weaver 1995; Arlandini et al. 1999; Busso et al. 1999; Rauscher et al. 2002 and references therein).

The energy powering stars results from nuclear fusion reactions transforming light nuclei into heavier ones. During their different evolutionary stages, a number of different nucleosynthetic process occur in different types of stars. Stars spend most of their lives in the core H-burning phase producing helium. When H is exhausted the star evolves into a red giant where part of the core material mixes convectively with the envelope and changes the surface composition. The next step is the onset of He combustion into ^{12}C, some of which gets converted into ^{16}O. When He is exhausted the future of the star depends on its mass. In the lighter stars (M < 8 Mo, where Mo = 1 solar mass), the core pressure and temperature are too low to proceed with further fusion reactions and becomes inert. The outer layers expand into an Asymptotic Giant Branch star (AGB) while H and He fusion continues just outside the core in thin shells producing large amounts of ^{12}C and heavy elements by slow neutron capture (s-process). Periodic convection episodes bring freshly synthesized materials to the surface. Large stellar winds expel the outer layers of the stars into a planetary nebula leaving a white dwarf star.

Massive stars

Cores of stars heavier than about 8 Mo continue to produce heavier nuclei within their cores, first the silicon mass region and ultimately the iron group nuclei. The resulting structure is an onion shell structure in which each layer has experienced more extensive fusion history than the next overlaying layer. When the core is composed mostly of iron, nuclear fusion can produce no more energy, and the structure collapses and rebounds on the core in an energetic event called supernova. The shock wave expels most of the mass of the star e.g. more than 90% for a 25 Mo star in a type II supernova (Woosley and Weaver 1995). This shock wave also heats the material in the different layers that it crosses and causes extensive nuclear reactions known as explosive nucleosynthesis. Taking together the products of hydrostatic fusion combustion and explosive nucleosynthesis, type II Supernovae are considered to be major producers of elements heavier than H in the galaxy. They are also thought to be the major site of the r- and p- nucleosynthetic processes. In the last stages of the evolution of massive stars, the core reaches very high temperature, above about 4×10^9 K, at which point the nuclear reaction network attains a thermal equilibrium called “nuclear statistical equilibrium” (NSE) or e-process. Variants having high neutron densities of this process have also been described to account for the production of neutron-rich isotopes (Hainebach et al. 1974; Cameron 1979; Hartmann et al. 1985).

Massive stars from 25 to 100 Mo already lose a substantial fraction of their mass in strong stellar winds ranging from 2×10^{-6} Mo/y during the H-Burning phase up to 5×10^{-5} Mo/y in the He-burning phase also known as Wolf-Rayet phase. As a large convective core develops in these stars, fresh nucleosynthetic product are soon exposed on the surface and ejected with the stellar winds (Prantzos et al. 1986). Wolf-Rayet stars may have been the principal source of ^{26}Al in meteorites (Arnould et al. 2000).

The fate of stars also depends on the presence of a companion. Other violent episodes known as Novae or type Ia supernovae result from the accretion of materials from a partner star. Supernovae of type I are thought to be responsible for most of the production of the neutron-rich isotopes of the iron group (Woosley et al. 1995; Höflich et al. 1998). One of the key parameters in stellar evolution, and consequently the nucleosynthetic outcome, is the "metallicity," defined as the proportion of elements heavier than He.

p-r-s-processes

Elements heavier than iron are not produced by the stellar fusion reactions. They are endothermic products and, to reproduce observed isotopic abundances, three production mechanisms are required (Burbidge et al. 1957). Two of them are neutron addition reactions starting with nuclei from the iron group. They are divided according to their slow (s-process) or rapid (r-process) time scale, as compared with the ß decay from unstable nuclei encountered along the neutron capture path. In the s-process, the orders of magnitude of the exposure to neutrons is: neutron densities of 3×10^8 n/cm^3 at temperatures close to 3×10^8 K for pulses of a few years occurring at intervals in the scale of 10^4 y. The r-process is at the origin of the elements beyond bismuth, and especially U-Th. r-process nuclei are produced in a high temperature ($>10^9$ K) stellar environment having neutron densities in the range 10^{20}-10^{25} n/cm^{-3} as highly unstable precursors which then return to the stability valley by ß-decay (Matthews and Cowan 1990; Meyer 1994). The neutron-rich isotopes of the elements from Zn to U result at least partially, if not totally, from this process. The neutron-deficient nuclei also called the p-nuclei cannot be produced by the two former processes. They represent no more than 1% of the bulk elemental abundances. It is generally considered that radiative proton capture and (γ, n) photodisintegration on preexisting neutron-rich nuclei are playing a key role in the p-process (Rayet et al. 1990; Lambert 1992; Meyer 1994).

In considering the mass balance of the solar system, the main production of s-process nuclei is attributed to the AGB phase. For the r- and p-process the relation with a particular astrophysical site is less straightforward but most models referred in the above reviews relate them to supernova events (Matthews and Cowan 1990; Rayet 1995).

As a concluding remark of this section, the theoretical models of nucleosynthesis within stars show that the isotopic compositions of the elements are highly variable depending on star size, metallicity, companion's presence. From the isotopic data obtained in diverse solar system materials it turns out that most of this material was highly homogenized in the interstellar medium or by the formation of the solar system. The presence of isotopic anomalies preserved in some primitive materials are the last witnesses of the initial diversity of the materials constituting our planetary system.

THE ISOTOPIC HETEROGENEITY IN THE REFRACTORY COMPONENTS OF THE EARLY SOLAR-SYSTEM

The contraction of the interstellar cloud at the origin of the solar system was a strongly exothermic process and the inner solar system went through a temperature peak which caused the loss of a number of volatile elements. This episode also promoted the isotopic homogenization of the elements. The completeness of the later process depends strongly on physical factors, such as the temperature and duration, but also on the mobility of the individual elements. In addition, parent body thermal metamorphism tends to erase isotopic heterogeneities and to reset radioactive chronometers (Göpel et al. 1994). Refractory mineral phases or phase assemblages are expected to better preserve any initial isotopic heterogeneity, and have therefore been the prime focus of isotopic measurements.

Carriers of anomalies were first investigated in carbonaceous chondrites, which are the most primitive meteorites available. For reasons that are not well understood, there is considerable variation in the size, morphology and mineralogy of refractory components between primitive meteorite families. Millimeter to several cm sized inclusions are found in the CV3 carbonaceous chondrites (Christophe 1968). Their compositions are close to those predicted for early condensates in a high temperature gaseous nebula with solar composition (Grossman 1972; Grossman and Larimer 1974). They were first to be investigated for practical reasons, such as, ease of separation.

Other meteorite classes like C2, CO and ordinary chondrites contain much smaller inclusions less than 1mm (MacPherson et al. 1988) and require ion microprobe techniques to evaluate the isotopic compositions. On the least metamorphosed side, C1 have very few inclusions or oxide grains, but are the carrier of the greatest amounts of stellar nanodiamond and other carbides (Anders and Zinner 1993). As will be shown for Cr anomalies in carbonaceous chondrites, the survival of the mineral carriers of the anomalies also depends on the metamorphic grade of the meteorites. Nevertheless, isotopic anomalies have also been found in higher metamorphic grade from other classes, especially in the reduced enstatite chondrites.

Refractory inclusions of the CV3 carbonaceous chondrites (CAIs)

The initial data on isotopic anomalies resulted from the abundant Allende meteorite, of which about 3 tons were recovered in 1969. Inclusions often found in CV3 meteorites like Allende, are straightforward to separate from the surrounding matrix with traditional mineral separations. Their sizes were within the same order of magnitude as the small lunar samples, for which a large experimental development effort had just been made (Gast et al. 1970; Tera et al. 1970). Refractory inclusions are often easily identified in hand-specimen as whitish objects in the dark matrix. They make up about 5% of the bulk mass of the meteorite. Other CV3s contain similar inclusions with some petrographic differences, but their isotopic data fit within the range of observations of Allende inclusions and will not be discussed here. The isotopic compositions for inclusions clearly divide them into two groups.

The first group called "common" or "normal" inclusions, represents the overwhelming majority of the samples. These display isotopic anomalies up to 4% in O and in the iron group elements. For the other elements investigated to date, they have normal isotopic compositions within instrumental resolution. The second group is composed of a very few inclusions showing mass fractionated light elements, much larger and variable effects in the iron group elements, and anomalies in all other elements investigated so far, especially the heavy elements Sr, Ba, Nd, Sm. They have been dubbed "FUN" inclusions for "Fractionated and Unknown Nuclear" effects. "FUN" signatures are indistinguishable from the main group on petrographic or mineralogical grounds, except for an enhanced occurrence in purple spinel-rich inclusions (Papanastassiou and Brigham 1989). They clearly show that in some cases, specific nucleosynthetic products were able to escape complete rehomogenization between their ejection from stars and their incorporation into meteorites.

Common inclusions.

Oxygen. The first isotopic measurements were for oxygen (Clayton et al. 1973). In the 3 isotope diagram (Fig. 1) all terrestrial samples plot on a mass dependent fractionation line of slope 0.52. Refractory inclusions and their minerals plot on a slope 1 line, which deviates by up to 2% from the terrestrial line for the most anomalous samples. This indicates a mixing line between an ^{16}O enriched reservoir that is distinct from the Earth-Moon with a composition of $\delta^{17}O = -42‰$ and $\delta^{18}O = -40‰$, and a terrestrial-like reservoir (Young and Russell 1998). Detailed analysis of mineral separates from a number of inclusions indicate that the most refractory minerals, like spinel and pyroxene, possess the highest ^{16}O enrichments, whereas the smallest enrichments are found in melilite and feldspathoids. This suggests that the carrier of the anomaly is a solid that underwent exchange with a nebular gas close to terrestrial

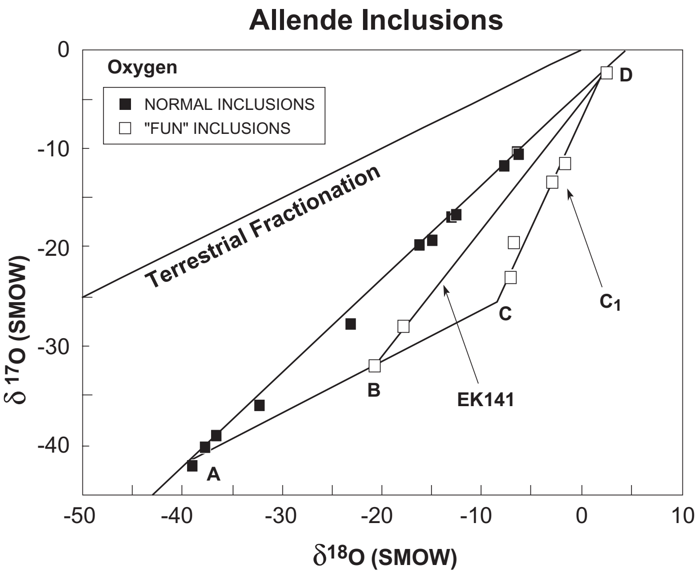

Figure 1. Three isotope plot of O isotopes in Allende inclusions. Deviations are plotted in δ units which are ‰ deviations relative to the terrestrial SMOW standard. In a two stage model, "normal" inclusions had initially a composition close to point A and exchanged with a reservoir poorer in ^{16}O in the region of point D (Clayton et al. 1973). "FUN" inclusions underwent an intermediate step along a fractionation line between point A and point C. Then each inclusion exchanged with the same ^{16}O poor reservoir D (Clayton and Mayeda 1977).

composition. In Figure 1, the most resistant mineral phases are assumed to retain their original isotopic composition. That the refractory inclusions have undergone secondary exchanges is also suggested by the observation of mineralogical alteration by a volatile rich and oxidizing gas (Wark 1986).

The ^{16}O excess was first suggested to have a nuclear origin in stars. Almost pure ^{16}O is produced in He-burning shells in massive stars, and in supernovae. On the other hand it has been shown that non-mass dependent fractionation can be produced in the laboratory by non-nuclear processes (Thiemens and Heidenreich 1983; Thiemens 1988). Similar non-linear effects have been found for O isotopes in atmospheric gases (Schueler et al. 1990; Thiemens et al. 1995). Although stellar nucleosynthesis is indeed at the origin of the O observed in the universe, the link between O isotopic anomalies in inclusions and nucleosynthesis is still under debate (Thiemens 1999; Clayton 2002).

Magnesium. The next element investigated was Mg as it is close to oxygen in several aspects: it is a major element, and has a relatively low atomic mass number. It possesses only three isotopes, and ^{26}Mg variations could possibly be induced by radioactive decay of now extinct ^{26}Al, so other non linear effects cannot be resolved unambiguously. If ^{24}Mg/^{25}Mg is assumed to be un-altered by nucleosynthetic inputs, then variations of a few ‰/amu around the average terrestrial value may reflect condensation-evaporation processes in the solar nebula (Wasserburg et al. 1977) and the alteration process already mentioned for oxygen (Young et al. 2002).

The iron group elements (from calcium to zinc). Some typical results on this group of elements are displayed in Figure 2. This group of elements have the most stable nuclei of the

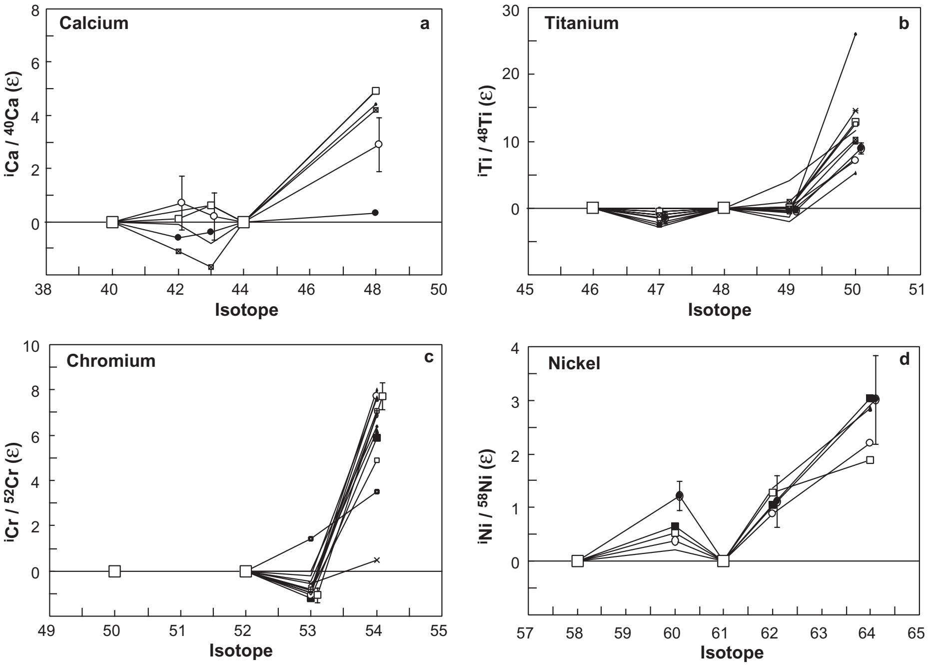

Figure 2. Typical isotopic compositions of the iron group elements in "normal" Allende inclusions (Niederer et al. 1981; Niemeyer and Lugmair 1981; Birck and Allegre 1984; Jungck et al. 1984; Birck and Allegre 1988; Birck and Lugmair 1988; Bogdanovski et al. 2002). The data are plotted as deviations in ε units (fractional deviation in 0.01%) from the terrestrial composition after normalization relative to the two isotopes represented with large open squares. The errors on the individual data points are not represented to not confuse the Figure. Typical experimental errors are represented only on one point in each sub-figure. Variations are clearly resolved for the most neutron-rich isotopes of this group of elements. Inclusions have an excess of a component produced in a neutron-rich nuclear statistical equilibrium.

periodic table. They may constitute the ultimate ashes of quiescent nuclear fusion in massive stars (see Section "Massive stars"). Ca and Ti belong to the refractory group of elements which appear in the first condensates above 1400K, whereas Zn is volatile (Larimer 1967). Cr and Ni are not considered to be refractory elements in the condensation sequence, and are close to the common major elements: Fe, Si, Mg. They are expected to condense with the bulk mass of a nebula of solar composition.

- Calcium. Ca is abundant in refractory inclusions. It has seven isotopes that span a 20% relative mass range. The nucleosynthetic pathway leading to the formation of Ca is expected to be varied because of its location between the silicon group and the iron group (Niederer and Papanastassiou 1984; Clayton 1988). ^{40}Ca which constitutes 96% of the total Ca, is also one of the decay products from ^{40}K , and can vary within the solar system with time, depending on the K/Ca ratio. Fortunately K is a volatile element and is strongly depleted in inclusions relative to Ca. Thus radioactive ingrowth of ^{40}Ca from ^{40}K over the lifetime of the solar system can be neglected in inclusions. This allows the use of the ^{40}Ca/^{44}Ca ratio to precisely determine the instrumental mass fractionation. In "Normal" or "Common" inclusions only the most neutron-rich isotope ^{48}Ca shows clearly resolved (more than 5σ) excesses (Fig. 2a). These excesses relative to average solar system composition are small, up to 6 ε with an average of about 3 ε (Lee et al. 1978; Jungck et al. 1984; Niederer and Papanastassiou 1984). No real correlation exists between O and Ca besides the presence of isotopic effects in the same objects.

^{48}Ca unlike the other isotopes of Ca, can be produced in significant amounts only in high neutron density regions of stars. Several processes can produce ^{48}Ca enrichments: neutron-rich Si burning (Cameron 1979), nβ−process (Sandler et al. 1982), s-process (Peters et al. 1972) and neutron-rich statistical equilibrium (Hainebach et al. 1974; Cameron 1979; Hartmann et al. 1985). All these processes take place in massive stars (>8 M_0). Addition of material from a heterogeneous supernova remnant to the solar system could produce regions with ^{48}Ca excess. In fact, Ca isotopic measurements eliminate the r-process, because it would overproduce ^{46}Ca well beyond the detection level despite its small abundance. No such excess is observed. The Ca isotopes alone do not permit one to distinguish between the other possible processes, and the other iron group elements are needed.

Unlike O, mass dependent fractionation is widespread for Ca in inclusions; it ranges from −3.8 to 6.7 ‰ (Niederer and Papanastassiou 1984) which is about four times the terrestrial range (Schmitt et al. 2003). However, 80% of samples fall within an interval of 2‰. The mass fractionation is the result of complex sequences of condensation and vaporization. The connection to Mg isotopic fractionation is not obvious for these samples as the resolution of Mg measurements is much smaller.

- Titanium. Ti has 5 contiguous isotopes from mass 46 to 50. As with Ca, Ti shows clearly resolved effects only on the most neutron-rich isotope: ^{50}Ti (Heydegger et al. 1979; Niederer et al. 1981; Niemeyer and Lugmair 1981). As for ^{48}Ca, a narrow range of excesses is found but the magnitude is about twice that of ^{48}Ca from 7 to 25 ε with an average of about 8 ε (Fig. 2b). The choice of the pair of isotopes to correct for instrumental *fractionation* is not of much importance. The high precision of the measurements gives hints of variations in other isotopes like ^{47}Ti or ^{49}Ti, but their size is an order of magnitude less than for ^{50}Ti. At least 3 different nucleosynthetic components are suspected, but this is better demonstrated in FUN inclusions to be discussed later. The main feature in "common" inclusions is the clear excess in ^{50}Ti.

  The *nucleosynthetic* sources for Ti isotopes are very similar to those of the isotopes of Ca, and ^{50}Ti requires a neutron-rich zone to be produced in significant amounts. In addition to the nonlinear effects, absolute isotopic compositions have been measured in a number of samples using double spike techniques (Niederer et al. 1985). Mass dependent fractionation effects are rarely resolved and are small, below 1 ‰/amu except in one sample, where it reaches 1.3 ‰/amu. In general the fractionation is in favor of the heavy isotopes; partial condensation or evaporation may explain of this observation.

- Chromium. Cr has four isotopes from mass 50 to 54. The choice of the normalizing isotope pair can be more ambiguous for this element (Birck and Allegre 1984; Papanastassiou 1986). ^{50}Cr and ^{52}Cr were used for this purpose because: 1) their nucleosynthetic properties are close to the non-anomalous isotopes of Ca-Ti, 2) excesses are expected on the neutron-rich isotope ^{54}Cr and 3) ^{53}Cr may be subject to variations related to the possible extinct radioactivity of ^{53}Mn. Indeed systematic ^{54}Cr excesses from 3 ε to 8 ε are found (Fig. 2c; Birck and Allegre 1984; Papanastassiou 1986; Birck and Allegre 1988; Bogdanovski et al. 2002). Variations of ^{53}Cr between −1.5 ε to +1 ε are related to ^{53}Mn decay and are discussed in section "53-Manganese –53-Chromium." When comparing with ^{48}Ca and ^{50}Ti excesses, the ^{54}Cr variations are of similar magnitude and suggest a nucleosynthetic component resulting from neutron-rich statistical equilibrium in a presupernova massive star (5 to 20 solar masses), near the cut-off between the ejected layers and the neutron star (Hainebach et al. 1974; Hartmann et al. 1985).

- Iron. Fe has 4 isotopes of which the heaviest ^{58}Fe has a very small abundance of about 0.3%. The precision of thermal ionization mass spectrometers is around 10 ε on this isotope and there is only a hint in some "normal inclusions" for an excess in ^{58}Fe (Völkening and Papanastassiou 1989). Recent ICPMS measurements at the 2 ε precision level display normal isotopic compositions for Fe in planetary materials but no Allende inclusion was reported in this study (Kehm et al. 2003). If excesses of similar magnitude to ^{48}Ca, ^{50}Ti, ^{54}Cr were present they would not be clearly resolved in agreement with the observations. When ^{54}Fe and ^{56}Fe are used to correct for instrumental *mass* fractionation, ^{57}Fe exhibits normal abundances, suggesting all three isotopes are present in solar relative abundances.
- Nickel. Ni has 5 isotopes, of which ^{60}Ni can be the product of the extinct radioactivity of ^{60}Fe. When considering the possible nucleosynthetic processes at the origin of Ca,Ti,Cr anomalies, several heavy isotopes of Ni may be affected by anomalies leaving only ^{58}Ni the major isotope and ^{61}Ni a minor isotope to correct for instrumental fractionation (Birck and Lugmair 1988). There are only a few data available indicating a systematic excess of ^{62}Ni and ^{64}Ni averaging at about 1ε and 3ε respectively (Fig. 2d). Again considering the other iron group elements, the anomalies suggest a particular nucleosynthetic source: neutron-rich nuclear statistical equilibrium with multiple zone mixing (Hartmann et al. 1985).
- Zinc. Zn is the upper end of the iron group. Although a strong hint exists for excesses of about 0.6 ε in ^{66}Zn in some samples, the results are not as clear as for the other elements of the group (Loss and Lugmair 1990; Völkening and Papanastassiou 1990). The *measurements* show that the effects are about a factor ten smaller than predicted by the models of nucleosynthesis. This does not constitute a problem for the model, but is most probably due to the properties of Zn. Zn is among the volatile elements and has been shown to be orders of magnitude more mobile than the other members of the iron group in circumstellar envelopes (Van Winckel et al. 1992). The longer residence time in the gas phase probably results in a more thorough homogenization of Zn isotopes between the various reservoirs constituting the solar system.

*Elements beyond zinc***.** Despite extensive investigations carried out in parallel with the "FUN" inclusions, clear-cut mass-independent isotopic heterogeneities (e.g., more than 5 σ of a single measurement) have not been identified yet in "normal" inclusions for the rest of the elements besides extinct radioactivities. Nevertheless, there are consistent hints for excesses of about 2 ε in ^{96}Zr (Harper et al. 1991; Schönbächler et al. 2003) which is an r-process isotope for which some coupling may exist with neutron-rich statistical equilibrium (Meyer 1994).

As a partial summary, in "normal" C3V inclusions material from the neutron-rich nuclear statistical equilibrium nucleosynthetic process is in excess relative to the average solar system composition, as well as an ^{16}O-rich component. Nevertheless, the exact composition of this material is somewhat blurred by secondary processes (nebular or interstellar) as the observations show no strict interelement correlation (Jungck et al. 1984; Birck and Lugmair 1988).

"FUN" inclusions.

This group is made up of a very few individual inclusions found among Allende studies. In fact it can be reduced to the two most striking samples: C1 and EK141. EK141 is unique in its isotopic properties, whereas a number of inclusions are closely related to C1 in their isotopic compositions (Papanastassiou and Brigham 1989; Loss et al. 1994). The discussion will therefore be limited to these two particular objects. C1 and EK141 have very similar isotopic patterns for O and Mg but differ radically in the other isotopes (Clayton and Mayeda 1977; Wasserburg et al. 1977).

Oxygen and magnesium. FUN inclusion samples do not plot on the O mixing-line between a solar nebular component and the exotic ^{16}O rich component (Fig. 1). They plot on the ^{18}O-rich side of this line and are thought to have been produced in several successive steps (Clayton and Mayeda 1977). They started as normal inclusions with a 4% ^{16}O enrichment. Then there is an intermediate stage where O mass fractionation occurs along a slope 0.52. The last stage is the same as for normal inclusions: isotopic exchange with a reservoir having a more nearly normal composition, which reduces the anomalies in the most sensitive minerals like melilite, and leaves spinel almost unaffected.

Magnesium is mass fractionated by 2 to 3 %/amu in favor of the heavy isotopes (Wasserburg et al. 1977). This is about 2 times the fractionation of O. When canceling out this mass fractionation by normalizing to the terrestrial $^{25}Mg/^{24}Mg$ ratio, a deficit of ^{26}Mg of about 3 ‰ is found in both inclusions. This is generally not believed to be a deficit of radiogenic ^{26}Mg (from ^{26}Al) but to some unidentified nucleosynthetic effect on one of the 2 other isotopes of Mg: ^{24}Mg, ^{25}Mg.

Silicon. Normal inclusions are spread on a mass dependent fractionation line over a few ‰/amu around the solar system average. FUN inclusions display a heavy isotope enrichment from 5 to 15 ‰/amu in a similar way to Mg. Non-linear effects are small and indicate an excess of ^{29}Si smaller than 0.5 ‰ (Clayton et al. 1984).

The iron group elements. Figure 3 displays the isotope ratios for this group. As a general result, the most neutron-rich isotope nuclei of this group display the largest variations relative to the other isotopes. Deficits are seen for C1, and excesses for EK141.

- <u>Calcium.</u> ^{48}Ca has a 20 ε deficit in C1 and a 141 ε excess in EK141. Clear differences from terrestrial compositions are also apparent for all other isotopes, with magnitudes ranging from 5 to 20 ε. Whatever the choice is for the pair of normalizing isotopes (^{46}Ca is not taken because of its very small abundance), all the others are non-solar. Also, a large mass dependent fractionation of similar amplitude to the non-linear features is apparent between the two samples (Fig. 3a; Lee et al. 1978; Niederer and Papanastassiou 1984) . All this together does not allow one to determine which isotope pair should be used for the normalization. Nevertheless, besides the excess in EK141 and the deficit in C1 of the component originating in neutron-rich statistical equilibrium, there is at least one other nucleosynthetic component present in variable amounts. This component may originate in explosive He or O burning shells, which are the main producers of the lighter Ca isotopes (Clayton 1988).
- <u>Titanium.</u> The isotopic effects are very similar to those of Ca, with the largest effects on the most neutron-rich isotope (Fig. 3b; Niederer et al. 1981). A deficit and an excess on ^{50}Ti for C1 (−51 ε) and EK141 (+37 ε) are observed respectively and agree with Ca data, suggesting a mixing model with a component produced in a neutron-rich nuclear statistical equilibrium. As for Ca the presence of isotopic anomalies up to 15 ε for the other isotopes of Ti and of mass dependent fractionation of up to 0.8 ‰/amu leaves an ambiguity in attributing the anomalies to a component that was produced by a specific nucleosynthetic process (Niederer et al. 1985). Nucleosynthetic processes which can contribute to the production of Ti isotopes are numerous and have been discussed extensively in conjunction with the Ca isotopic data (Niederer et al. 1981; Clayton 1988). Nevertheless in C1, the pattern of Ti isotopes may indicate a deficit of ^{46}Ti, the second most anomalous isotope after ^{50}Ti. In that case one can speculate that the signature of explosive O burning in a supernova shell (Woosley et al. 1973) is indicated.

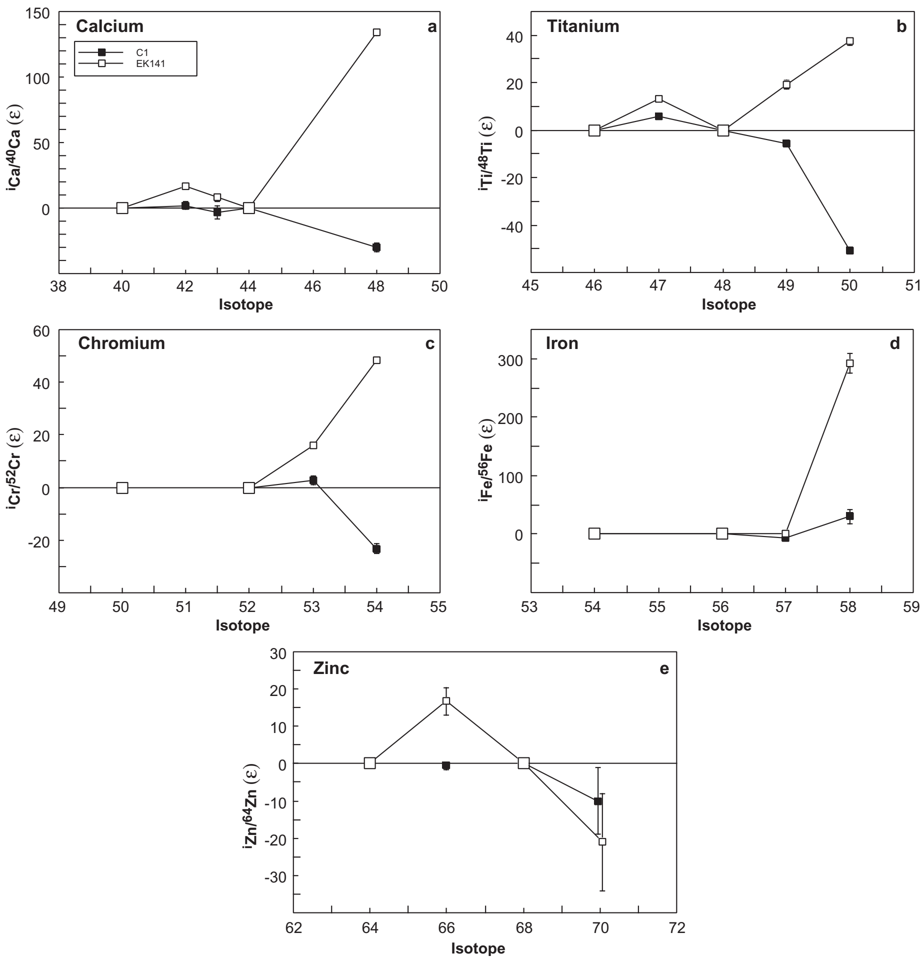

Figure 3. Isotopic compositions of the iron group elements in the "FUN" inclusions: C1 and EK141 (Lee et al. 1978; Niederer et al. 1985; Papanastassiou 1986; Völkening and Papanastassiou 1989; Völkening and Papanastassiou 1990). Scales are as in Figure 2. ■ and □ are used for C1 and EK141 respectively. The individual errors are represented but are often smaller than the dots representing the data. Isotopic variations in "FUN" inclusions are more than an order of magnitude larger than in "normal" inclusions. Both excesses and deficits in the neutron-rich statistical equilibrium produced component are present. EK141 is unique among inclusions, in showing large isotopic variation of similar amplitude for several isotopes in most elements.

- Chromium. The pattern is again very similar to Ti and Ca, with large excesses of the most neutron-rich isotope ^{54}Cr (48 ε) in EK141 and a large deficit in C1 (−23 ε) (Fig. 3c; Papanastassiou 1986). Unlike normal inclusions, ^{53}Cr exhibits a large excess in both inclusions. In nucleosynthetic models, this isotope is mostly produced as ^{53}Mn which decays to ^{53}Cr (Hainebach et al. 1974; Hartmann et al. 1985). Whether this happened in the source of EK141 or that some unidentified process produced ^{53}Cr directly, is not settled yet. The presence of mass dependent fractionation has not been investigated in details for Cr. This is also the case for the 3 remaining iron group elements: Fe, Ni and Zn.

- Iron, zinc (Fig. 3d,e). Nickel has not been investigated in FUN inclusions. For Fe (Völkening and Papanastassiou 1989) and Zn (Völkening and Papanastassiou 1990) only EK141 displays clear anomalies, with excesses in ^{58}Fe (292 ε) and in ^{66}Zn (15.5 ε). C1 has only a hint of an excess in ^{58}Fe (30 ± 15 ε). In an inclusion similar to C1, but extracted from the Vigarano meteorite, a deficit in ^{66}Zn has been evidenced by Loss et al. (1994). This is in agreement with the deficit in neutron-rich isotopes of the other iron-group elements in the same inclusion. The data of EK141 are in agreement with the model deduced from the other iron group elements

Heavy elements (Fig. 4). This is the domain where the FUN inclusions differ the most from normal inclusions. With anomalies in the range of a few ε to a few ‰, and also possible mass dependent fractionation, there can be an ambiguity in using a given isotopic pair to normalize for instrumental fractionation. The corrections may result in attribution of some isotopic anomalies which are actually normal. This can be avoided with a minimum number of assumptions. The heavy elements beyond Zn in the solar system are produced through 3 nucleosynthetic processes: p, r, and s (Burbidge et al. 1957). Heavy elements often have between 5 and 10 isotopes, and the three processes are required to produce all the various isotopes of a single element. To interpret the observed anomalies, the normalization of the measured isotopic ratios can be tried in using a pair of isotopes produced in only one of these processes. This is not always possible and inter-element comparison is also required (Lugmair et al. 1978; McCulloch and Wasserburg 1978a; Lee 1988).

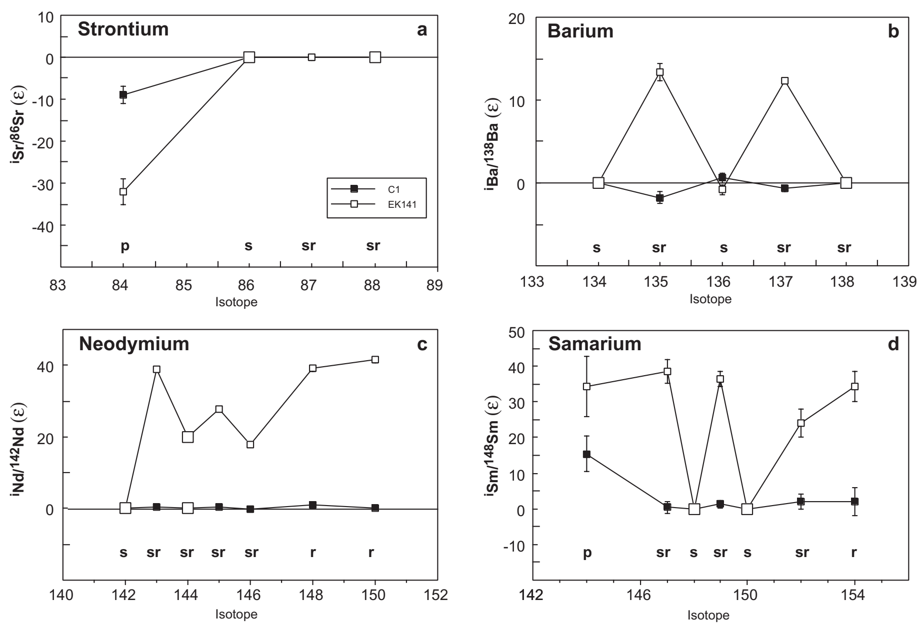

Figure 4. Non-linear effects for Sr, Ba, Nd and Sm in the "FUN" inclusions: C1 and EK141 (McCulloch and Wasserburg 1978a,b; Papanastassiou and Wasserburg 1978). Relative deviations from terrestrial standard ratios are plotted after normalization with the isotope pair represented with large open squares. Each isotope is labeled with its primary nucleosynthetic source. In using s-process isotopes for normalization, clear excesses in r-process nuclei are seen for Ba and Sm in EK141. Sr is normal in both inclusions except for a deficit in the p-process ^{84}Sr. As Nd has only one pure s-process isotope at mass 142, the data in EK141 have been further corrected to yield an excess in ^{150}Nd identical to that of ^{154}Sm as these two isotope are pure r-process nuclei expected to be produced in comparable abundances.

- Strontium. C1 and EK141 display deficits of 8 ε and 32 ε in the least abundant isotope ^{84}Sr which is a p-process nucleus (Fig. 4a; Papanastassiou and Wasserburg 1978). The other three isotopes ^{86}Sr-^{87}Sr-^{88}Sr, which result mainly from s-process nucleosynthesis, are in normal proportions. The isotopic pattern could be interpreted as an extra addition to average matter of a s-process enriched component. This seems implausible because this s-process component would have to have precisely the solar composition for the 3 heavy Sr isotopes, and it is expected that the many stars which contributed to the solar system mix did not produce all 3 isotopes in exactly the same pattern. In C1 and EK141, the contrasting p deficits in the Sr mass region with p-r excesses in the Ba-REE region require that the nucleosynthesis in these two mass regions occurred in distinct nucleosynthetic sites. Two components can be sought: one with the p deficit contributes to the mass region A <90 and the second to the mass region >130.
- Barium, neodymium, samarium (Fig. 4b,c,d). For these 3 elements, C1 displays normal isotopic compositions, except for an excess of the p-produced isotope ^{144}Sm. In EK141, and using s-only isotopes for normalization, clearly resolved excesses of 10 to 40 ε are found for r-produced isotopes of Ba, Nd and Sm (McCulloch and Wasserburg 1978a; McCulloch and Wasserburg 1978b). The remarkable feature is that the particular r-process component found in the FUN inclusion EK141 is like that of the average pattern of r-process isotopes in the solar system.

Inclusions of the CV3 led to the search for isotopic signatures of individual nucleosynthetic processes, or at least for components closer to the original signature than average solar compositions. They have also begun to demonstrate the isotopic variability of matter emerging from these processes in agreement with astrophysical and astronomical expectations. The principal features of inclusions are: an up to 4% ^{16}O enriched reservoir in the early solar system, variations in a component produced in a nuclear neutron-rich statistical equilibrium, and variations in the contribution of p- and r-process products to the heavy elements.

Hibonite rich inclusions of the CM2 and oxide grains in carbonaceous chondrites

Hibonites $CaAl(Ti,Mg)_{12}O_{19}$ is one of the most refractory minerals found in primitive meteorites (Grossman 1972; Lattimer et al. 1978) and is expected to condense at high temperatures just after corundum from a cooling gas of solar composition. Hibonite-bearing inclusions and spherules are common in CM2 meteorites, of which Murchison is the most studied (MacPherson et al. 1983). These inclusions are similar to Allende inclusions in several aspects: they carry isotopic anomalies and they have undergone secondary alteration on their exterior. Hibonite inclusions are too small to be extracted by traditional means (Ireland et al. 1985; Hinton et al. 1987). In general, data are reported on single grains of 10-100 μm in size which have been classified according to their morphology, which correlates reasonably well with the isotopic characteristics (Ireland 1990). Almost all isotopic data available are ion probe measurements, and are often accompanied by measurements of a number of trace element abundances, especially REEs. REEs are refractory elements, and have the signature of earlier volatilization-condensation processes. One group of hibonite grains has a condensation pattern in which the most refractory REEs are depleted (Ireland 1990). Ion probe measurements have a reduced precision by about an order of magnitude (‰ level) relative to thermal ionization measurements. This is due to the small sample size and sometimes to the necessity to correct for isobaric interferences (e.g., ^{48}Ca-^{48}Ti). For minor or trace elements, the precision is even lower and the data base is not very large. However, large isotopic effects are present in these grains.

Magnesium. Corundum-hibonite associations are what could be the first condensates from a solar composition gas. Mg is not a refractory element and is strongly depleted in these inclusions. For an object with such a primitive composition, one would also expect the presence of ^{26}Al in significant amounts. ^{26}Mg effects related to ^{26}Al are rare, but correlated excess is present in others (Fahey et al. 1987; Ireland 1988). The uniform ^{26}Mg composition must be characteristic of the reservoir from which the corundum-hibonite formed. Formation of these primitive objects after ^{26}Al decay is difficult to reconcile with solar system formation models. ^{26}Mg heterogeneity in presolar reservoirs is preferred by some of the authors. Fahey et al. (1987) argue for heterogeneous distribution of ^{26}Al in the solar nebula. Mass-dependent fractionation favoring the heavy isotopes is present in some samples up to 1.8 %/amu resulting from distillation processes (Ireland 1988; Ireland 1990). Effects resulting from ^{26}Al decay (see below) range from 0 to the canonical value found in Allende Inclusions ($^{26}Al/^{27}Al = 5.1\times10^{-5}$ in Fig. 9b) and are mostly restricted to certain morphological types of hibonites (Ireland 1988); others classes show little or no evidence for past ^{26}Al effects.

Titanium-calcium. The first evidence for isotopic anomalies in the iron-group was found in Ti showing up to 10% excesses of ^{50}Ti in hibonites from the Murray CM2 meteorite (Hutcheon et al. 1983; Fahey et al. 1985; Ireland et al. 1985; Hinton et al. 1987). Further studies in Murchison showed that ^{50}Ti extended from −7% to +27% associated with ^{48}Ca variation from −6% to +10% (Ireland 1988; Ireland 1990). Except for the magnitude of the variations, this is similar to the results from Allende inclusions. Only a few samples display mass-dependent fractionation for which it ranges up to 1.3 %/amu. In the majority of the samples, it is absent or very low (less than 1 ‰/amu) for Ca-Ti. There is no correlation between the presence of linear fractionation and the magnitude of ^{50}Ti effects. ^{49}Ti variations are also present, but about an order of magnitude smaller than ^{50}Ti. Variations affecting these two isotopes are related but not strictly correlated (Ireland 1988).

As for Allende's inclusions, variable contributions of a component produced in neutron-rich nuclear statistical equilibrium best explains the ^{50}Ti-^{48}Ca data. Some parts of the solar nebula were depleted in these isotopes as deficits are also seen. There are several possibilities for explaining the variations in ^{49}Ti. 1) The neutron-rich component itself may be heterogeneous and incorporate locally less neutron-rich statistical equilibrium products (Hartmann et al. 1985). 2) ^{49}Ti may result from another process like explosive Si or He burning (Clayton 1988). This component would be associated with the neutron-rich component but not completely homogenized. In all cases, carriers are solid grains which may have behaved differently than the gaseous nebula during the formation of the solar system. A minimum number of components may be calculated to account for the Ca and Ti isotopic data, which number up to 3–4 (Ireland 1990) but to be conservative at the 5σ level, clearly resolved effects are present only on 3 isotopes (^{48}Ca,^{49}Ti, ^{50}Ti).

Despite anomalies up to 27%, titanium is still 98.4% of average solar isotopic composition which is a strong argument in favor of a solar system origin for the hibonite grains or aggregates. Little information is available on the isotopic ratios from the other elements. Oxygen isotopic compositions are within the range of Allende's inclusions based on the few data available (Hashimoto et al. 1986; Olsen et al. 1988).

"Presolar" stardust grains: diamond, graphite, carbides and oxides

The subject of this section is "acid-resistant" phases obtained from the matrix by dissolving away the major matrix minerals. They are understood as refractory phases, which is true in general but not strictly equivalent. Diamonds represent at most a few hundred ppm of the total mass of the meteorite, and silicon carbide a few ppm only, but nevertheless, thousands of grains have been analyzed and the presolar origin of the grains is indicated by enormous isotopic ratio differences relative to solar system average. Isotopes of the CNO elements are normally not part of this volume, but in these grains variations are so large that there is no doubt of their nucleosynthetic origin. The word "stardust" is a generic term for the grains.

The first indications of carriers of fresh nucleosynthetic products came from the work on the isotopes of Ne which indicated a component enriched in ^{22}Ne (Black and Pepin 1969; Black 1972). Further work eventually led to the isolation of a carrier of more than 99% pure in ^{22}Ne (Jungck and Eberhardt 1979; Eberhardt et al. 1981). In the mean time, Xe also revealed several components located primarily in trace phases. It turned out that Xe-HL showing the most striking isotopic differences from the planetary average noble gas patterns resides in acid-resistant phases (Lewis et al. 1975). The preparation procedures for isolating presolar grains have been improved over time and involve dissolution of the matrix of the rock and further physico-chemical separations by grain size and density (Tang et al. 1988; Amari et al. 1994). There is now a data base of isotopic analyses on thousands of individuals grains and this chapter can only be a rapid glimpse to the isotopic variety in these objects. For more details see Bernatowicz and Zinner (1997).

Nanodiamonds. Nanodiamonds were first isolated by Lewis et al. (1987). They are very small, with an average size of 2nm and are a stable phase relative to graphite in H-rich environments (Nuth 1987; Badziag et al. 1990). Their small size precludes the analysis of single grains, and isotopic data are averages of many grains. C isotope ratios are close to the average solar value and N which constitutes up to 0.9% of the diamond, is depleted by as much as 35% in ^{15}N (Virag et al. 1989; Russell et al. 1991). Three distinct noble gas components are present in diamonds: two with close to normal isotopic compositions and one with anomalous isotopic compositions, in particular Xe-HL and Kr-H (Fig. 5; Huss and Lewis 1994). The favored explanation of the Xe-HL pattern is that it was produced in supernovae by a combination of p-and r-process (Clayton 1989). According to the small size of the diamonds, only 1 grain in 10^6 has a single Xe atom. Thus it is possible that most diamonds come from other sources (Alexander 1997) and that most of them could in fact be produced in the solar system (Dai et al. 2002). Thermal processing before accretion is also indicated by the data (Huss et al. 2003). Very few other isotopic data exist on these objects. A study on Sr and Ba isotopes showed only marginal effects occurring at the r-process isotopes, which is in agreement with a supernova origin (Lewis et al. 1983, 1991). Te isotopic variations point to a component with virtually no light isotopes and requires a separation of the r-process isotopes of Te-Xe from their precursors within a few hours (Ott 1996; Richter et al. 1998). The supernova component found in nanodiamond has to go through series of complex processes to account for the observation.

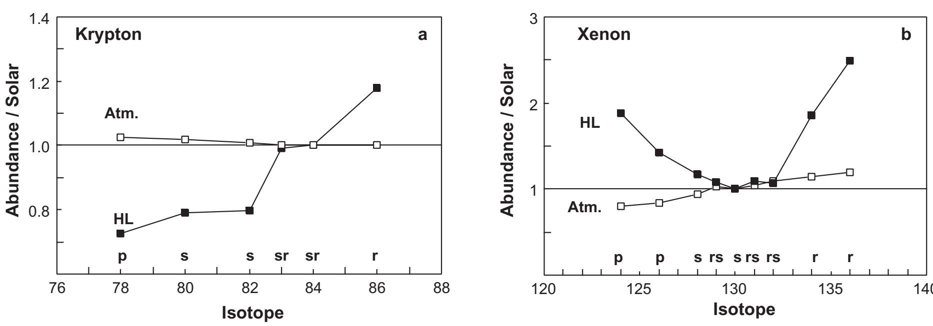

Figure 5. Relative abundances of the Kr, Xe isotopes (Huss and Lewis 1994) in presolar diamonds have been measured in bulk samples (= many grains) and are plotted relative to solar wind abundances. The terrestrial atmosphere is shown for comparison and displays a pattern close to mass dependent fractionation relative to the solar wind. The primary nucleosynthetic processes at the origin of the different nuclei are also listed. Both Kr and Xe are elevated in the r-process isotopes, whereas only Xe is also enriched in the p-isotopes. These patterns are a strong argument in favor of a supernova origin for the diamonds. Ne isotopes in presolar diamond is within the field of bulk meteorite data.

Silicon carbide and graphite grains. An overview of the thousands of data available is displayed in Figures 6 and 7. This fraction resulting from the separation procedure has been shown to be the carrier of s-process Xe (Xe-s). Graphitic carbon and silicon carbide have been shown to be the carrier of Ne E (Fig. 7a). The nucleosynthetic source of Neon E (pure ^{22}Ne) has long been thought to be ^{22}Na ($T_{1/2}$ = 2.6 yr) (Black 1972; Brown and Clayton 1992), but can also be produced in He-burning shells of AGB stars, like the CNO anomalies (Ott 1993). This question is not settled yet.

Grains larger than 0.5 μm are common and can be individually analyzed. Tremendous variations, over 4 orders of magnitude, are observed in the isotopic ratios of CNO (Fig. 6) and over 1.5 order of magnitude for Si. If the isotope ratios reflect the nucleosynthetic processes without too much intercomponent mixing and fractionation, then the variations in most grains reflect various degrees of He shell and H shell burning, and mixing in AGB stars. The data base is now numerous and reviews exist in which references can be found (Anders and Zinner 1993; Busso et al. 1999; Nittler 2003).

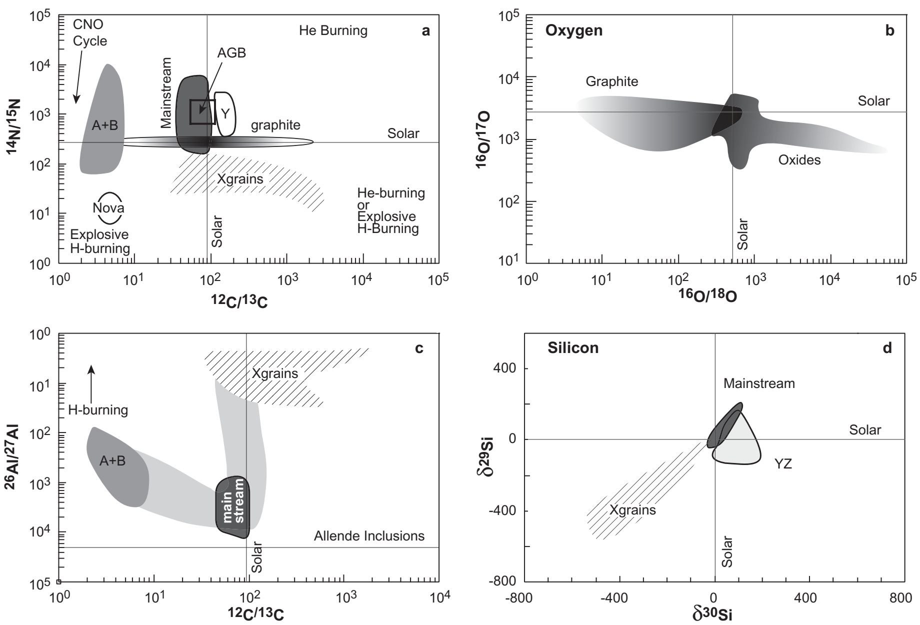

Figure 6. Single presolar silicon carbide grains exhibit a tremendous isotopic variety and are separated in several populations on the basis of grain size and C-N-O-Si isotopic properties. Graphite grains are also represented in (a) for comparison. Figure after Hoppe and Ott (1997), Zinner (1997), Amari et al. (2001) and Nittler (2003). The data are plotted as absolute values in (a), (b), (c), and the solar average value is also shown. Si is plotted as ‰ deviations relative to the terrestrial values in (d). ^{26}Al/^{27}Al is calculated assuming that all excess ^{26}Mg found in the samples results from ^{26}Al decay. All data displayed have been obtained on individual grains ranging from 0.3 to 5 μm in size by ion probe measurements (Amari et al. 1994, 2001). Model predictions in AGB stars are also shown. X grains are thought to be produced in supernovae (Nittler et al. 1996; Besmehn and Hoppe 2003). The data represented here represent thousands of individual grains. The main stream of grains (more than 90% by number) are produced at different stages of evolution of AGB stars. Oxygen isotopic data are only available in graphite and oxide grains. In (a) and (b) the gradation of the shading reflects roughly the data density of population in the given areas of the diagram. There are a few grains located in the space between the defined populations; they are represented by shaded areas. There are also a very few grains with extraordinary isotopic signatures located outside the fields displayed here and which are not represented here for clarity.

Large excesses in ^{26}Mg have been interpreted as resulting from the decay of ^{26}Al (Fig. 6c; Hoppe et al. 1994; Huss et al. 1997). Initial values as high as 0.5 have been found in ^{26}Al/^{27}Al. High ^{26}Al is produced in the H-burning shell, but, if it can match such high ratios in agreement with other elements, is not known yet. Calcium and titanium display excesses in ^{42}Ca, ^{43}Ca, ^{44}Ca and a V shaped pattern for Ti (Fig. 7b; Amari et al. 2001). The results are qualitatively consistent with a s-process during He burning.

The heavy elements carry clear excesses in a s-process component as can be seen from Figure 7. This has been demonstrated for Kr, Sr, Xe, Ba, Nd, Sm (Ott and Begemann 1990; Prombo et al. 1993; Lewis et al. 1994; Richter 1995; Hoppe and Ott 1997). The models can be made to fit very precisely the measured data (Lattanzio and Boothroyd 1997; Busso et al. 1999). Mo and Zr can occur as microcrystals of Mo-Zr-C within graphite grains. Typical s-process patterns are observed, with isotopic variations of about a factor of more than 5 (Nicolussi et al. 1997; Nicolussi et al. 1998a).

Some X-SiC grains possess r-process excesses (see Nittler, 2003, and references therein) as well as a fraction of the graphite grains (Nicolussi et al. 1998c). The variability in the grains is such that subgroups (the X SiC, low density graphite and Si_3N_4 grains) carry the signature of supernova nucleosynthesis in Si (Amari et al. 1999), as well as for excess ^{44}Ca interpreted as decay product from ^{44}Ti (Nittler et al. 1996; Amari and Zinner 1997; Besmehn and Hoppe 2003).

Oxide grains. Corundum, hibonite and spinel represent a very minor proportion of the acid resistant residue (Nittler et al. 1994; Zinner et al. 2003) in the range of 0.05 ppm of the bulk meteorite. This may also be due to their having a smaller size distribution or the grains are more susceptible to destruction in the meteorite and in sample preparation. Only a small number of such grains has been analyzed and among these less than 10 have proven to possess very exotic isotopic signatures (Huss et al. 1994). A few other grains with similar signatures have been found in Murchison and Bishunpur which is a low metamorphic grade ordinary chondrite of type LL3 (Choi et al. 1998).

In these investigations, due to practical limitations, measurements have been limited to grains larger than about 1 μm in size, but progress in the instrumentation will lead to more subtle phases and intra-grain analyses. Most probably new effects will be seen, and will increase the observed variability in isotopic components. The modeling of nucleosynthesis and circumstellar processes is also at stake. The puzzle at the moment is quite complicated as different nucleosynthetic sources (AGB stars, supernovae) are represented in the major families of grains, except for the first one containing the supernova diamonds. It turns out that some stellar zone are very specific isotopic "spike" factories, which thereafter mix with other components depending on stellar dynamics.

Solar system processes are also among the targets, because of the disappearance of this heterogeneity with the metamorphic grade of the meteorites. Studies of temperature and duration of metamorphism are still in their early stages (El Goresy et al. 1995; Huss 1997)

THE ISOTOPIC HETEROGENEITY IN NON-REFRACTORY EARLY SOLAR SYSTEM MATERIALS

Presolar stardust discovered to date in meteorites constitute no more than 0.5‰ of the total mass of the samples, and one common chemical property is that they are acid resistant. Isotopic heterogeneity could also be present in less refractory phases like silicates, provided parent body metamorphism did not erase the differences. Noble gases are not discussed here because they are depleted by many orders of magnitude relative to the Sun and can be dominated by trace exotic minerals. Nitrogen is not discussed for the same reason. The

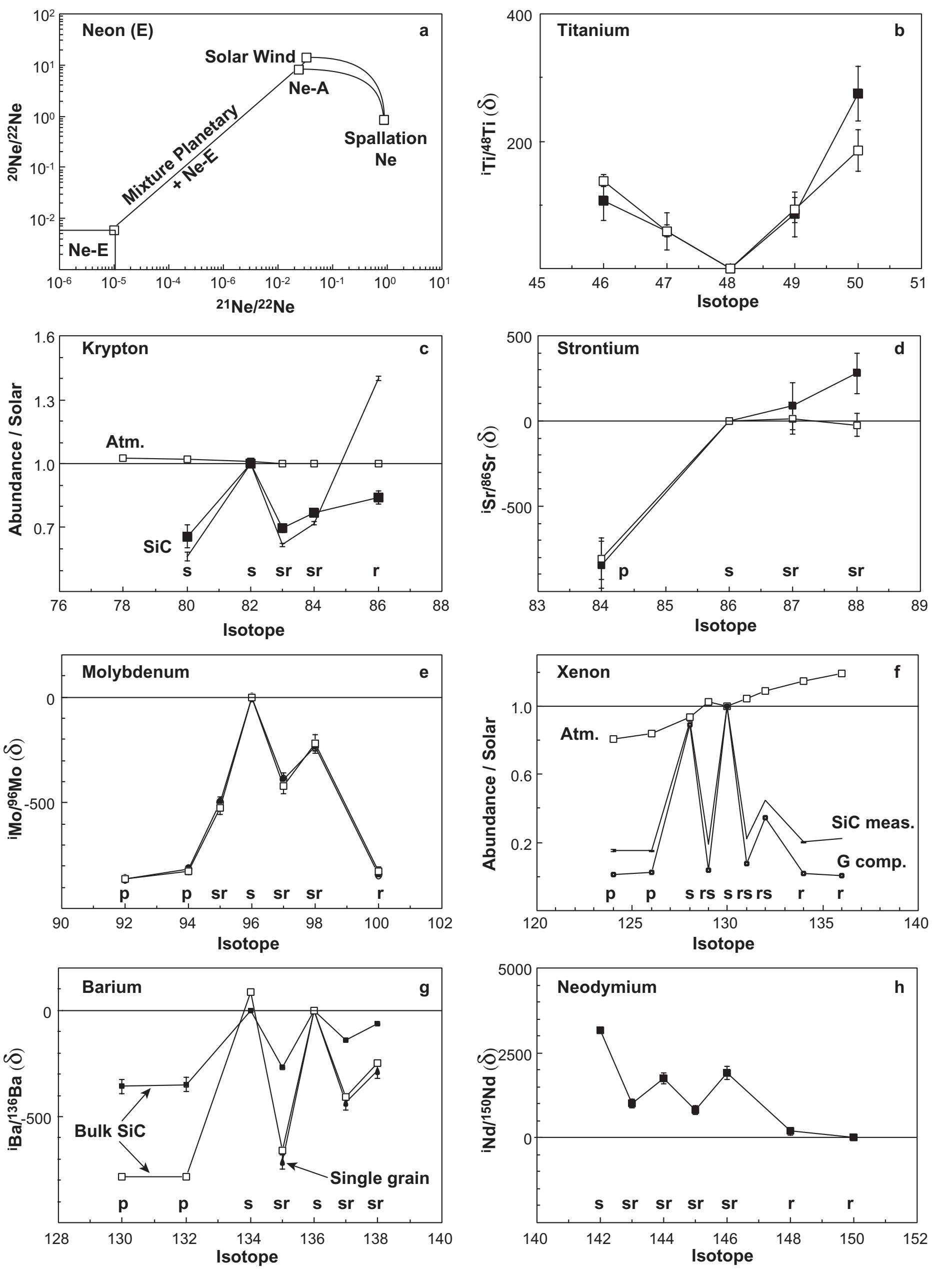

Figure 7. A few examples of isotopic patterns of Ne, Ti and heavy elements in SiC and graphite grains are displayed. Absolute ratios are plotted for Ne (a) whereas abundance ratios relative to solar wind composition are plotted for Kr (c) and Xe (f). The remaining elements are plotted as ‰ deviations from laboratory standards. The data have been obtained on bulk SiC separates by traditional mass spectrometry for Ne (Jungck and Eberhardt 1979), Kr (Ott et al. 1988; Lewis et al. 1994), Sr, Ba (Ott and Begemann 1990; Prombo et al. 1993) and Nd (Richter 1995). SIMS techniques *(caption continued on facing page)*

purpose here is to look to elements which are close to bulk solar proportion when compared to Si as a reference. This field has not received as much attention as the refractory grains but a few striking examples of isotopic diversity exist in separates from bulk samples.

Oxygen

The O isotopes show significant heterogeneity between the different meteorite classes (Fig. 8a; Clayton et al. 1976, 1977). Differences are small, but, each chondrite group has a distinct bulk O isotopic composition. O isotopes also indicate the close ties between the Earth and the Moon. O therefore can be used to identify members of a family that formed from a common reservoir, which is the definition of a tracer. Such differences are also found between chondrules within the same meteorites related to their size (Gooding et al. 1983). This is a survival of the initial isotopic heterogeneity in already high temperature processed materials like chondrules.

Chromium

C1 chondrites have very fine-grained predominantly phyllosilicate mineralogy. Mineral separates of phyllosilicates are not practically achievable. In using increasing strength acids, the stepwise dissolution/leaching procedure exhibits large isotopic heterogeneity for Cr isotopes (Fig. 8b) which represents about 0.3% of the mass. The size of the non-linear variations ranges up to 2.2% for the most neutron-rich isotope ^{54}Cr (Rotaru et al. 1992; Podosek et al. 1997).Variation on ^{53}Cr are smaller and have been interpreted as resulting from ^{53}Mn decay.

54-Chromium. The isotopic heterogeneity is limited to this isotope which can be compared with the "normal" refractory inclusions of Allende. Both ^{54}Cr deficits and excesses are found ranging from −7.6 ε to +210 ε (Fig. 8b). The fractions showing the highest enrichment in ^{54}Cr with no correlated effects in ^{50}Cr, ^{52}Cr, ^{53}Cr points towards a nucleosynthetic component, which is 99% pure in ^{54}Cr. This component is probably the same as the component found in the CV3 inclusions, and which is produced in a neutron-rich nuclear statistical equilibrium in presupernova massive stars.

None of the dissolution fractions has normal isotopic compositions. This implies that the solar system results from mixing of major Cr-bearing components, none having the solar (terrestrial) isotopic compositions. Data obtained from higher metamorphic grade carbonaceous chondrites show that the isotopic heterogeneity is erased by parent body metamorphism with an amplitude decreasing from 220 ε in the C1 chondrites, through 40 ε in

Figure 7 (*caption continued from facing page*).

have been used for single grain analysis of Ti (Amari et al. 2001) and by laser-ablation-RIMS techniques for Sr (Nicolussi et al. 1998b), Mo (Nicolussi et al. 1998a) and Ba (Savina et al. 2003a). Bulk analyses mostly reflect the isotopic ratios of the main stream population. The magnitude of the effects does not require one to correct for mass fractionation. As displayed on one sample for Ba, individual grains are often close to the bulk samples. With the exception of Nd for which a r-process isotope is taken for reference, abundant s-process isotopes are usually taken as reference to compare the compositions. In the resulting patterns, the isotopes produced by p-, r-process are highly depleted or absent within the resolution of the experiments. These results point to strongly s-process dominated components originating in AGB stars. The spectrum of Kr is somewhat more complex with, in some sets of data, an over-abundance of r-process ^{86}Kr. Adjustments in the neutron exposure within the star can resolve this apparent discrepancy. It is also noticeable that ^{86}Sr, ^{87}Sr, ^{88}Sr are produced in proportion close to the average solar system composition despite the variability in the neighboring elements. In general for an element, numerous individual grain data are available and the measured composition can be interpreted as a mixture of a component close to normal solar system composition and a component representing the signature of the nucleosynthetic process (G-component). This G-component is represented for Xe. It can be seen that with carefully prepared samples the G-component is dominant and precisely defined. For the other elements, grains very close to the original nucleosynthetic isotopic signature are also found.

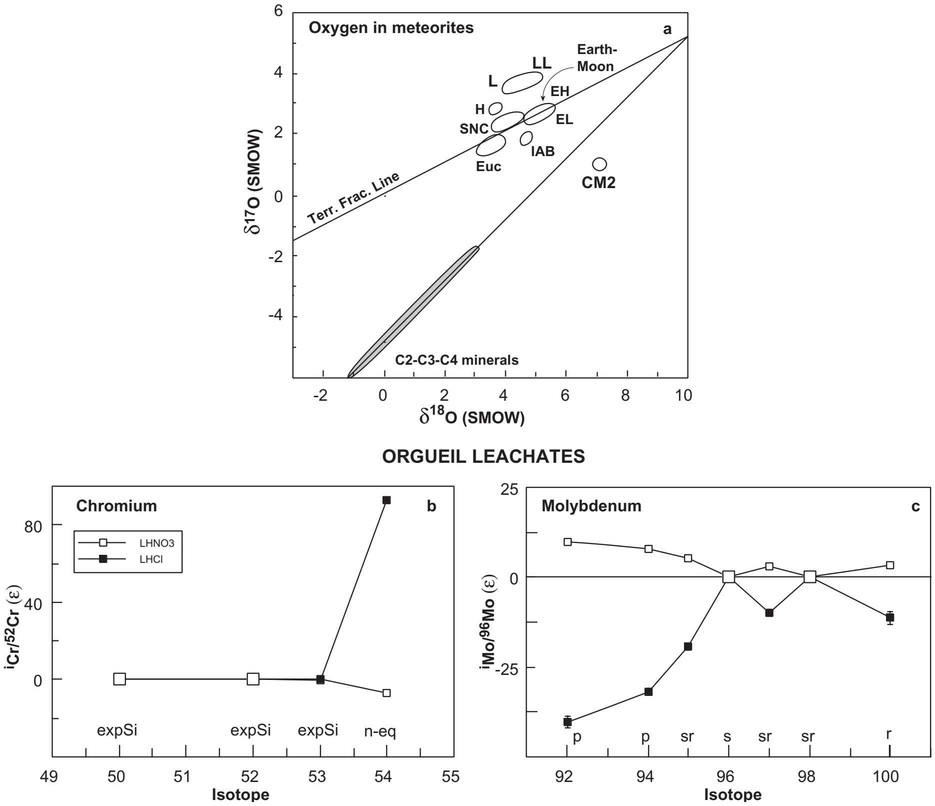

Figure 8. Figure (a) after Clayton et al. (1976, 1977). The scales are as in Figure 1. The O isotopic compositions of the different meteorite classes are represented: ordinary chondrites (H, L, LL), enstatite chondrites (EH, EL), differentiated meteorites (eucrites, IAB irons, SNCs) and some components of the carbonaceous chondrites. As the different areas do not overlap, a classification of the meteorites can be drawn based on O isotopes. Cr (b) and Mo (c) isotope compositions obtained by stepwise dissolution of the C1 carbonaceous chondrite Orgueil (Rotaru et al. 1992; Dauphas et al. 2002), are plotted as deviations relative to the terrestrial composition in ε units. Isotopes are labeled according to their primary nucleosynthetic sources. "ExpSi" is for explosive Si burning and "n-eq" is for neutron-rich nuclear statistical equilibrium. The open squares represent a HNO_3 4 N leachate at room temperature. The filled square correspond to the dissolution of the main silicate phase in a HCl-HF mix. The M pattern for Mo in the silicates is similar to the s-process component found in micron-size SiC presolar grains as shown in Figure 7.

the C2 chondrites, and to below 5 ε in the C3 chondrites. Ti and Fe isotopic analysis at the 2 ε precision level have shown no evidence for isotopic anomalies in the same fractions of the C1 Orgueil. From the CAI observations of related effects between ^{54}Cr, ^{48}Ca, ^{50}Ti, ^{58}Fe effects of a few tens of ε are expected for Ti and Fe. This is not observed and is most probably due to the mineralogy of the matrix (Rotaru et al. 1992). Cr is present in at least 2 different carriers behaving differently in the dissolution procedure: one with depleted ^{54}Cr and one with the neutron-rich component carrying pure ^{54}Cr. Ti and Fe are obviously not in the same carriers. The two nucleosynthetic components of Ti and Fe are probably also present in the samples but they are located in similar mineralogical sites which so far could not be resolved.

Bulk rock analyses of carbonaceous chondrites exhibit a ^{54}Cr excess from 1 to 2 ε (Rotaru et al. 1992; Shukolyukov et al. 2003). The carbonaceous chondrites are not exactly solar in their Cr isotopic bulk composition, but taking into account that the components are more than 220 ε apart, the match is very close and the idea that C chondrites are a fair representation of the solar system average is still reasonable.

53-Chromium. Much smaller variations are evident in this isotope: usually a few ε. They are not correlated with ^{54}Cr variations but with the Mn/Cr elemental ratios. They are also present in the higher metamorphic grades with even larger spreads. ^{53}Cr is not produced directly in stars but through ^{53}Mn which then decays to ^{53}Cr. These arguments favor the interpretation of ^{53}Cr as solely due to in situ ^{53}Mn decay (Birck et al. 1999).

Molybdenum

The same stepwise dissolution procedure revealed Mo isotopic variations of up to 50 ε (Dauphas et al. 2002). Mo belongs to the heavy elements beyond the iron peak. It has r-, s-process isotopes but differs from most other elements in having two abundant p-isotopes ^{92}Mo (14.8%) and ^{94}Mo (9.2%). ^{94}Mo may also be s-produced. The largest differences from the terrestrial or average solar composition appear in the same steps as the most striking ^{54}Cr isotopic effects (Fig. 8c). As for Cr, very little Mo has average solar system composition. The largest variation corresponds to an excess of s-process Mo as was observed in SiC presolar grains (Nicolussi et al. 1998a) and the host phase of this particular component is thought to be SiC (Dauphas et al. 2002).The complementary pattern is not expected to be in a specific host phase because r and s components have no reason to be coupled. Variable Mo isotopic compositions have also been found in iron meteorite with differences between classes of up to 4 ε on ^{92}Mo but with no differences within a given class . The Cr and Mo data demonstrate that the signature of nucleosynthesis is also present in major mineral phases of primitive meteorites and parent body metamorphism is an efficient homogenization process (Rotaru et al. 1992).

There are also indications that Ru displays systematic deficits in ^{100}Ru in iron meteorites and in Allende relative to terrestrial composition (Chen et al. 2003). A deficit in s-process isotopes of Ru is the favored interpretation.

EXTINCT RADIOACTIVITIES

Isotopic variations can be generated from a once uniform reservoir by decay of radioactive nuclides. Extinct radioactivities are the radioactive nuclides which were present in the early system, but have now completely decayed. Table 1 shows the radioactive nuclei that have been positively or tentatively identified. The separation from long period chronometers is between ^{40}K ($T_{1/2}$ = 1.2 b.y.), which is still present, and ^{146}Sm ($T_{1/2}$ = 104 m.y.) of which a few atoms could theoretically still be detected, but not with standard instrumentation. Half-lives from a few m.y. to 100 m.y. cover the time scale which is expected for solar system and planet formation. Occasionally nuclides of a few years half-life have been sought to explain isotopic variations as for NeE in presolar silicon carbide. A number of reviews have been published on extinct radioactivities and their possible nucleosynthetic sources (Wasserburg and Papanastassiou 1982; Arnould et al. 2000; Goswami and Vanhala 2000; Russell et al. 2001). I present here only the broad outlines of this field. Extinct radioactivities are demonstrated by the existence, in coexisting phases, of a positive correlation between the elemental parent-daughter ratio and isotopic variation involving the daughter isotope. Due to their short half lives, the nuclides have small abundances compared to stable isotopes accumulated during 10 b.y. of galactic evolution. Moreover the protostellar cloud could have stayed closed to nucleosynthetic inputs before contraction into the solar system (Wasserburg and Papanastassiou 1982). This would further reduce the abundances of the short lived

Table 1. Radionuclides of half-life from 0.01 m.y. to 150 m.y.

Parent	*Daughter*	*Half-Life (m.y.)*	*Detected (§)*	*Initial solar system Abundance*	*Reference*
^{10}Be	^{10}B	1.5	+	$^{10}Be/^{9}Be = 9\times10^{-4}$	McKeegan et al. 2000
^{26}Al	^{26}Mg	0.73	+	$^{26}Al/^{27}Al = 5.1\times10^{-5}$	Lee et al. 1977
^{36}Cl	^{36}Ar	0.28	−		
^{41}Ca	^{41}K	0.1	+	$^{41}Ca/^{40}Ca = 1.4\times10^{-8}$	Sahijpal et al. 1998
^{53}Mn	^{53}Cr	3.7	+	$^{53}Mn/^{55}Mn = 4.4\times10^{-5}$	Birck and Allegre 1985
^{60}Fe	^{60}Ni	1.5	+	$^{60}Fe/^{56}Fe \approx 1.5\times10^{-6}$	Birck and Lugmair 1988
^{79}Se	^{79}Br	0.06	−		
^{81}Kr	^{81}Br	0.21	−		
^{92}Nb	^{92}Zr	36	+	$^{92}Nb/^{93}Nb \approx 1\times10^{-5}$	Schönbächler et al. 2002
^{97}Tc	^{97}Mo	2.7	?		
^{98}Tc	^{98}Ru	4.3	?		
^{99}Tc	^{99}Ru	0.21	?		
^{107}Pd	^{107}Ag	6.5	+	$^{107}Pd/^{108}Pd = 2.4\times10^{-5}$	Chen and Wasserburg 1990
^{129}I	^{129}Xe	16	+	$^{129}I/^{127}I \approx 1\times10^{-4}$	Reynolds 1963
^{135}Cs	^{135}Ba	2.3	?		
^{137}La	^{137}Ba	0.06	−		
^{146}Sm	^{142}Nd	103	+	$^{146}Sm/^{144}Sm \approx 0.005–0.015$	Lugmair et al. 1983; Prinzhofer et al. 1989
^{150}Gd	^{146}Sm	1.8	−		
^{182}Hf	^{182}W	9.4	+	$^{182}Hf/^{180}Hf \approx 1\times10^{-4}$	Yin et al. 2002
^{202}Pb	^{202}Hg	0.6	−		
^{205}Pb	^{205}Tl	14.1	?		
^{239}Pu	α,SF	0.02	−		
^{242}Pu	α,SF	0.35	−		
^{244}Pu	α,SF	81	+	$^{244}Pu/^{238}U \approx 0.004–0.007$	Rowe and Kuroda 1965
^{247}Cm	^{235}U	16	?		
^{248}Cm	^{244}Pu	0.35	−		

§ (+) positive detection (?) hint (−) undetected or uninvestigated nuclides. The detectability of their decay products is always a difficult task, requiring state of the art precision and sensitivity in mass spectrometry. Due to their rapid decay, they have a potential of being high resolution chronometers, but this assumes that these nuclides were homogeneously distributed in the early solar system. Since they are now extinct, a reference object with a well known age determined with a long-lived nuclide (U-Pb generally), is used to convert relative ages determined with short-lived radionuclides to absolute ages. The ideal reference object would be a common well-equilibrated meteorite which was not disturbed thereafter. Half-lives and early solar system contents of the short-lived nuclides are given in Table 1. Examples of the correlations resulting from a number of extinct radioactivities are given in Figure 9.

10-Berylium - 10-Boron ($T_{1/2}$ = 1.5 m.y.)

B has only 2 isotopes and natural fractionation may be present as observed on the Earth. Only highly radiogenic phases avoid this ambiguity. B is a mobile and volatile element and alteration by a gas phase has partially re-equilibrated oxygen in inclusions. Nevertheless, the presence of live ^{10}Be has been established by ion probe work (Fig. 9a; McKeegan et al. 2000). The interest of this extinct radioactivity is that ^{10}Be is not produced significantly in stellar nucleosynthesis and an irradiation process is required; the question being if it was by the early sun or somewhere else close to a presolar star. As B isotopes are measured by ion-probe, the Al-Mg system can be readily investigated on the same analytical spots. There is a good connection between the two chronometers ^{10}Be-^{10}B and ^{26}Al-^{26}Mg (MacPherson et al. 2003) but differential behavior relative to temperature and to mineralogy also occurs (Sugiura et al. 2001) and contributes to establishment of the thermal history of the inclusions.

26-Aluminum - 26-Magnesium ($T_{1/2}$ = 0.7 m.y.)

This nuclide is of particular interest for two reasons: its very short half-life and its potential as a heat source for early planetary objects (Urey 1955; Lee et al. 1977). Detailed reviews with emphasis on this nuclide are available (Wasserburg and Papanastassiou 1982; MacPherson et al. 1995). An early study in feldspars which have high Al/Mg ratios, extracted from a variety of chondrites and eucrites did not detect evidence for past ^{26}Al (Schramm et al. 1970). The first positive evidence came from the refractory inclusions of Allende with ^{26}Mg excesses correlated with Al/Mg (Gray and Compston 1974; Lee and Papanastassiou 1974) but the clear evidence that this was not a fossil correlation, was demonstrated by an isochron from inclusions which were initially homogenized by melting (Lee et al. 1976; Clayton 1986) (Fig. 9b). Numerous results were also thereafter obtained with ion probe in situ measurements. A number of well-defined isochrons from refractory inclusions give the so-called "canonical" value of $^{26}Al/^{27}Al = 5.1\times10^{-5}$ (see MacPherson et al. (1995) for a review) Many inclusions display lower values which can be interpreted in 3 different ways: 1) ^{26}Al was heterogeneously distributed in the early solar system, 2) secondary processes have partially re-equilibrated the samples with normal Mg, 3) delayed formation after partial decay of ^{26}Al. An illustration of protracted formation is given by the detailed work of Hsu et al. (2000) on different locations within a single CAI. Several stages of melt addition within a few 10^5 years are suggested.

In ordinary chondrites, Ca-Al rich inclusions are also present and give the canonical value (Russell et al. 1996), but other object like chondrules or mineral grains give reduced values by a factor of 5 to 100 (Hinton and Bischoff 1984; Hutcheon and Hutchison 1989). Delayed formation relative to CAIs is a probable cause. High precision ICPMS measurements of Mg have been used to address the timing of chondrule formation and show the importance of gas during the formation process (Galy et al. 2000).

Parent body metamorphism also resets the ^{26}Al-^{26}Mg chronometer as it does for the traditional long-period chronometers. Plagioclases separated from a few of the oldest chondrites display a good agreement between ^{26}Al-^{26}Mg and U-Pb chronometers (Göpel et al.

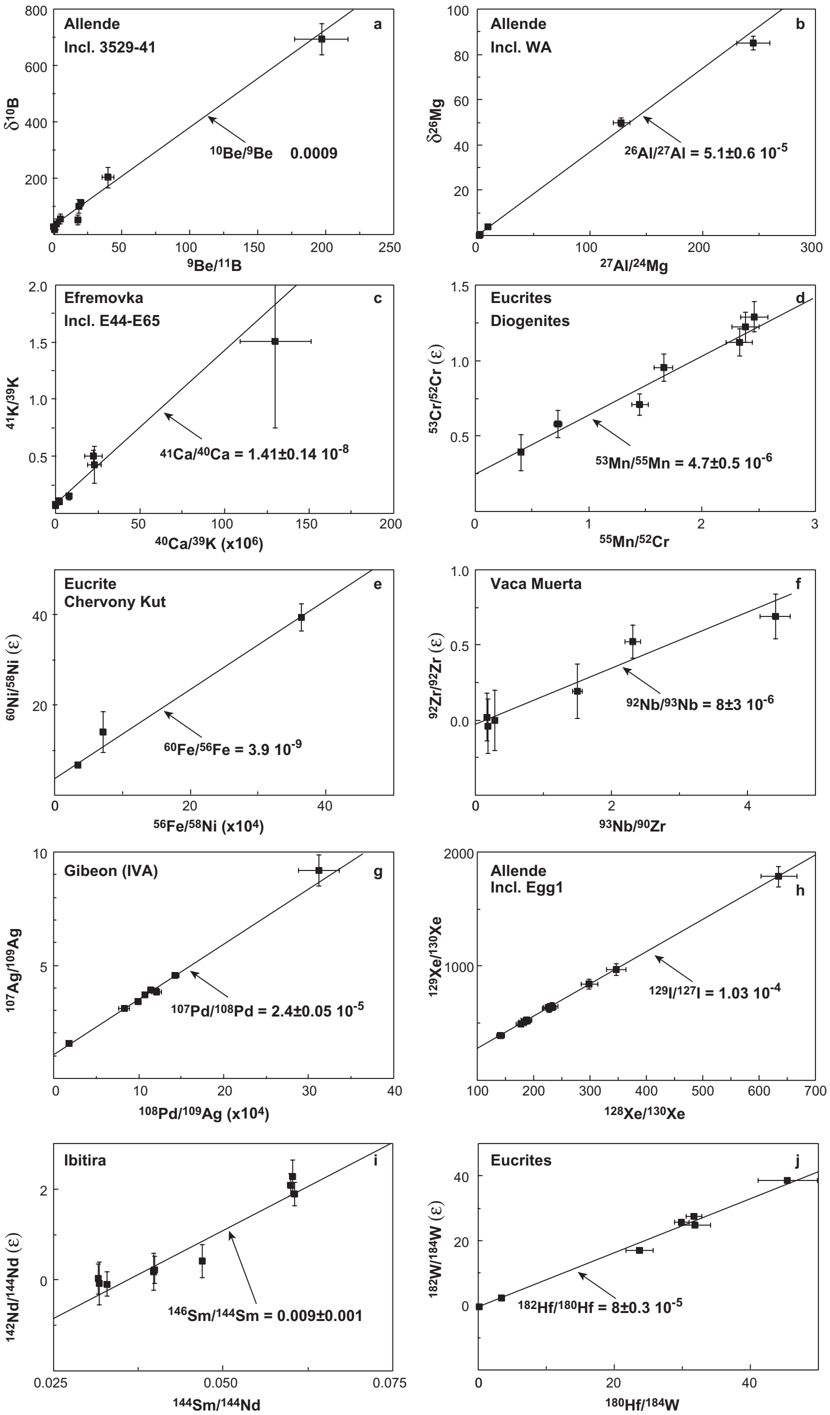

Figure 9. *(caption on facing page)*

1994; Zinner and Göpel 2002). This work is an argument in favor of ^{26}Al being homogeneously distributed in the solar system at least in chondrites (Huss et al. 2001). ^{26}Al has also been found at very low levels in basaltic achondrites (Srinivasan et al. 1999; Wadhwa et al. 2003) and the data show that secondary effects (such as shock?) have often affected the Al-Mg system. It is not known yet if the chronometry stemming from ^{26}Al in eucrites is in agreement with other chronometers or not. The connection so far looks complex. If ^{26}Al was homogeneously distributed in the early solar system, which is not proven yet, it might have melted the interior of bodies of a few km radius and may constitute an efficient trigger for early magmatism.

^{26}Al is produced efficiently in a number of nucleosynthetic processes like hydrostatic burning or explosive H burning in novae (Woosley 1986) or Wolf-Rayet stars (Arnould et al. 2000) or in AGB stars (Wasserburg et al. 1994). Its short half-life requires that this has happened no more than 3 Ma before meteorite formation (Wasserburg and Papanastassiou 1982). It has been argued that a late addition of about 10^{-3} to 10^{-4} solar masses of freshly nucleosynthesized material has been added to the solar protostellar cloud just before the formation of the sun (Birck and Allegre 1988), but other hypotheses have not been completely eliminated, such as local irradiation by energetic protons from the young sun (Heymann and Dziczkaniec 1976; Lee 1978; Gounelle et al. 2001). However irradiation should also produce effects on other elements (Clayton et al. 1977a) which have not been observed yet.

41-Calcium - 41-Potassium ($T_{1/2}$ = 0.1 m.y.)

^{41}Ca is very important because its very short half life provide a strong time constraint between a supernova event and the formation of the solar system. It was identified in very low K grains in C2 and C3 chondrites with an average ^{41}Ca/^{40}Ca value of 1.4×10^{-8} (Fig. 9c; Srinivasan et al. 1996). The presence of live ^{41}Ca in the early solar system constrains the time scale for the collapse of the protosolar cloud to be less than a million years (Cameron 1995; Wasserburg et al. 1996). As for ^{10}Be there is a good correlation of the ^{41}Ca-^{41}Ca system with the ^{26}Al-^{26}Mg data in the primitive samples (Sahijpal et al. 1998). The close connection of ^{41}Ca with ^{26}Al and ^{10}Be has been taken as an argument in favor of a production of these isotopes within the solar system by proton irradiation in an active X-wind phase of the young sun (Shu et al. 2000; Gounelle et al. 2001). This would release the time constraint between presolar stellar nucleosynthesis and planet formation, but would complicate the use of short-lived nuclide as chronometers.

53-Manganese - 53-Chromium ($T_{1/2}$ = 3.7 m.y.)

Most of the ^{53}Cr found in the solar system is produced in presupernova neutron-poor nuclear statistical equilibrium as ^{53}Mn which then decays to ^{53}Cr (Hainebach et al. 1974; Hartmann et al. 1985). The first evidence for the presence of ^{53}Mn in the early solar system

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Figure 9 ***(on facing page).*** Examples of isotopic variations resulting from extinct radioactivities. Isotopic ratios are plotted as absolute values for K (Srinivasan et al. 1996), Ag (Chen and Wasserburg 1990) and Xe (Swindle et al. 1988). They are plotted as deviation from a terrestrial standard in δ units (‰ fractional deviations) for B (McKeegan et al. 2000) and Mg (Lee et al. 1977), and in ε units (0.01% fractional deviations) for Cr (Lugmair and Shukolyukov 1998), Ni (Shukolyukov and Lugmair 1993), Zr (Schönbächler et al. 2002), Nd (Prinzhofer et al. 1989) and Hf (Quitté et al. 2000). Some systems do not exhibit sufficient isotopic dispersion in Allende inclusions to define a clear isochron. The examples displayed here have been chosen for the clarity of correlation in the isochron diagram as more favorable parent daughter ratios are often found in differentiated meteorites: irons for ^{107}Pd-^{107}Ag, eucrites for ^{182}Hf-^{182}W. Solar system initial isotopic ratios of the parent element can be found in Table 1. The meteorites in which the measurements have been carried out, are given in each sub-figure. For I-Xe the parent daughter ratio is not measured as such. The sample is irradiated with neutrons which convert ^{127}I into ^{128}Xe as ^{128}I decays to ^{128}Xe within a few hours (Swindle et al. 1988). The pattern shown here represent the stepwise degassing from 1400–1950°C, starting from upper right of the diagram. The ^{129}I/^{127}I ages are usually given as relative ages to the L4 chondrite Bjurböle (^{129}I/^{127}I = 1.095 10^{-4}).

was found in Allende CAIs with a $^{53}Mn/^{55}Mn$ ratio of 4.4×10^{-5} (Birck and Allegre 1985). Such a value allows this potential chronometer to be of some practical use over about 30 m.y. after CAI formation. Inclusion data show that about 0.02% of present normal ^{53}Cr was ^{53}Mn at the time of inclusion formation.

^{53}Mn was found in chondrites, pallasites and eucrites (Birck and Allegre 1988; Lugmair and Shukolyukov 1998). Its presence in basaltic eucrites unambiguously established the in-situ nature of ^{53}Mn decay (Fig. 9d). In general, these measurements require very high precision mass spectrometry, to 20 ppm precision and better. Ion probe measurements on very high Mn/Cr phases reveal very radiogenic ratios: sulfides in enstatite chondrites (Wadhwa et al. 1997), phosphates of iron meteorites (Davis and Olsen 1990), olivine in chondrites (Hutcheon et al. 1998). They exhibit $^{53}Mn/^{55}Mn$ ratios ranging from 10^{-8} to 10^{-5} which represent a delayed formation by more than 7 m.y. relative to inclusion formation or re-equilibration in secondary processes. Eucrites together with pallasites and diogenites (the HED family) plot along a well defined bulk rock isochron, but individual samples do not display a coherent picture of internal mineral isochrons which are distributed from the $^{53}Mn/^{55}Mn$ value of 4.7×10^{-6} down to zero slope (Lugmair and Shukolyukov 1998).

As for other extinct radioactivities, to provide absolute ages, the $^{53}Mn/^{53}Cr$ chronometer has to be anchored to a sample for which the absolute age is known from a traditional chronometer, usually U-Pb because of its high precision. There is debate going on how to do this with ^{53}Mn and how to compare the ^{53}Mn-^{53}Cr data to other short-lived and long-lived radiometric systems (Lugmair and Shukolyukov 1998; Birck et al. 1999; Quitté 2001). Further high precision work is still required to settle the debate.

60-Iron - 60-Nickel ($T_{1/2}$ = 1.5 m.y.)

^{60}Fe is a neutron-rich nucleus and is expected to be produced in similar processes to those producing the excess of neutron-rich nuclei of the iron group elements in Allende's CAIs. The first hint for the presence of ^{60}Fe was found in the Ni isotopic compositions in CAIs (Birck and Lugmair 1988). The presence of ^{60}Fe has been clearly identified in eucrites which have high Fe/Ni ratios (Fig. 9e; Shukolyukov and Lugmair 1993). In using ion probes to spot phases with high Fe/Ni ratios, the presence of ^{60}Fe was also demonstrated in chondrites (Mostefaoui et al. 2003). The initial solar system value for $^{60}Fe/^{56}Fe$ is estimated around 1.5×10^{-6} with some assumptions. If homogeneously distributed, ^{60}Fe can constitute a rapid heat source to melt small planetary bodies in addition to the possible presence of ^{26}Al.

92-Niobium - 92-Zirconium ($T_{1/2}$ = 36 m.y.)

As ^{146}Sm, ^{92}Nb can be used to place constraints on the site of p-process nucleosynthesis. After a first hint (Harper 1996; Sanloup et al. 2000), its presence was established in an ordinary chondrite and a mesosiderite (Fig. 9f; Schönbächler et al. 2002). The solar system initial $^{92}Nb/^{93}Nb$ ratio was between 10^{-5} and 3×10^{-5} a value in the same order of magnitude as most of the other extinct radioactivities.

107-Palladium - 107-Silver ($T_{1/2}$ = 6.5 m.y.)

Pd is a siderophile element and is concentrated in iron meteorites. Ag also has some siderophilic character, but is predominantly chalcophile. It also belongs to the volatile elements which are depleted in a number of iron meteorite classes. The chemical properties of this system result in high Pd/Ag ratios in the metal phase of iron meteorites and low ratios in the associated sulfide nodules, if they are present. The existence of ^{107}Ag excess correlated with the Pd/Ag ratio in coexisting phases and between bulk meteorites show that ^{107}Pd was widespread in the early solar system (Fig. 9g; Kelly and Wasserburg 1978; Chen and Wasserburg 1983; Chen and Wasserburg 1990). The narrow range around 2.1×10^{-5} for the $^{107}Pd/^{108}Pd$ ratio in all meteorites not disturbed by secondary processes shows that all classes of iron meteorites formed in a narrow time interval of less than 10 m.y. This is in agreement with W isotopic data, although the constraint is not very strong. Silver has only two isotopes and can only be measured down to a precision of c.a. 1‰ in TIMS instruments. This system could only be investigated in radiogenic samples. The introduction of ICPMS allows an order of magnitude improvement in precision and hence the investigation of silicate meteoritic materials (Carlson and Hauri 2001a). The results are in agreement with former Pd-Ag data and other chronometric systems.

^{107}Pd results from the same type of r-process as ^{129}I, but it is not well characterized (Wasserburg and Papanastassiou 1982) and can result from a late addition of a component incorporating a number of other short-lived nuclide as well.

129-Iodine - 129-Xenon ($T_{1/2}$ = 16 m.y.)

This was the first extinct radioactivity detected (Jeffrey and Reynolds 1961) and was made possible by the early high sensitivity of rare gas measurements and the low abundance of Xe in rocks. I has only one stable isotope at mass 127. Its abundance is measured as ^{128}Xe after exposing a sample to an adequate neutron flux. The correlation between ^{128}Xe and ^{129}Xe observed in a stepwise degassing of a sample demonstrates that the excess ^{129}Xe results from ^{129}I decay (Fig. 9h). Results in primitive meteorites and inclusions show that ^{129}I/^{127}I is close to 10^{-4}. Chronometry with ^{129}I-^{129}Xe has been widely used in meteorite work (Reynolds 1963; Hohenberg 1967) but occasionally has some difficulties to agree with the other chronometers due to the sensitivity of I to secondary processes and water alteration (Pravdivtseva et al. 2003; Busfield et al. 2004; see also Swindle and Podosek (1988) for an extensive review) .

146-Samarium - 142-Neodymium ($T_{1/2}$ = 103 m.y.)

^{146}Sm has the longest half life for the extinct radioactivities. Theoretically there should be a few atoms left in present day samples, although their detectability is not obvious and could be confused in meteorites with spallation-produced ^{146}Sm. ^{146}Sm is a p-process nuclide. It was detected first in a magmatic meteorite: Angra Dos Reis but at the limit of resolution of the method (Lugmair and Marti 1977; Jacobsen and Wasserburg 1980). Effects were well resolved in Allende acid-resistant residues (Lugmair et al. 1983) giving an initial ^{146}Sm/^{144}Sm ratio of 0.005. Further studies in eucrites showed that it was widespread (Fig. 9i; Prinzhofer et al. 1989) and give a higher initial ^{146}Sm/^{144}Sm ratio of 0.015, 4.56 Gy ago. Several nucleosynthetic models have been proposed to produce ^{146}Sm, some of which are compatible with a constant production in the galaxy without the need for a late spike to introduce the extinct radionuclide in the protosolar cloud (Prinzhofer et al. 1992). The long half life of ^{146}Sm allows for an investigation of crustal fractionation processes in the early earth, but this requires even higher precision mass spectrometry (Sharma et al. 1996) down to 4-5 ppm to resolve differences (Caro et al. 2003)

182-Hafnium - 182-Tungsten ($T_{1/2}$ = 9.4 m.y.)

^{182}Hf is an r-process nuclide similar to ^{107}Pd, ^{129}I and ^{244}Pu. The main interest of this system resides in the chemical properties of the parent-daughter elements. Hf is highly lithophile and partitions into planetary crusts whereas W is strongly siderophile and concentrates into planetary cores. When metal in equilibrium with silicates separates from these silicates, it traps W without Hf and then freezes the isotopic composition of W at the moment of the separation. On the other hand, the silicate part (which still contains very little W) gets very radiogenic and has a rapidly increasing ^{182}W/^{184}W ratio with time, which yields a high resolution chronometer. A deficit in ^{182}W has been detected in iron meteorites by Harper and Jacobsen (1994) and then by Lee and Halliday (1995, 1996). These data along with ^{107}Pd, show that the segregation of iron meteorites was an early and short episode. The last authors applied these measurements to a broad range of samples to study planetary core formation as well as the crystallization of the Moon's magma ocean (Lee et al. 1997). A large data base exists now for this system for iron meteorites as well as for chondrites and achondrites (Fig. 9i; Quitté et al. 2000). Cosmogenic W may constitute a problem for planetary surface samples having large galactic-cosmic-ray exposure ages like the lunar rocks (Leya et al. 2000). W isotopic data have shown that the silicate part of the Earth is more radiogenic by 2 ε than the bulk solar system (Kleine et al. 2002; Yin et al. 2002). It follows that the Earth's core formed rapidly, within 30 m.y. This system is presently under intense investigation because its half-life gives access to the processes forming the larger planets, like the Earth, the Moon and Mars.

244-Plutonium spontaneous fission ($T_{1/2}$ = 81 m.y.)

Excess fission Xe due to the decay of ^{244}Pu, was found in U-Th rich samples (Rowe and Kuroda 1965; Lewis 1975). Its abundance relative to U is about 7×10^{-3}. This extinct radionuclide is a pure r-process product and its abundance can be used to estimate the timing of the r-process contribution to the presolar interstellar cloud (Wasserburg et al. 1996)

Other nuclides

Other nuclides have also been sought for, like ^{135}Cs (Shen et al. 1994; Hidaka et al. 2001), ^{205}Pb (Chen and Wasserburg 1987; Rehkämper and Halliday 1999), ^{247}Cm (Chen and Wasserburg 1981) but so far have not been detected.

In summary, the extinct radioactivities which have a limited time of existence in the solar system, constrain the time interval between the late stages of stellar nucleosynthesis and the formation of the solar system. Some production may also occur within the solar system during active periods of the young Sun. There have been numerous studies about how this matter was added into the solar system as a late spike of about 10^{-3} solar masses of freshly stellar processed material or from constant production in the galaxy (Wasserburg et al. 1996; Goswami and Vanhala 2000; Russell et al. 2001). These models are refined constantly with the input of new data and will probably continue to evolve in the future.

OPEN QUESTIONS AND FUTURE DIRECTIONS

The measurements of isotopic anomalies in meteorites has contributed greatly to the understanding of mixing processes and time scales in the formation of the solar system as well as strong constraints on presolar stellar evolution but it also left unanswered questions and revealed new complexities which are discussed here.

Mass independent fractionation in physico chemical-processes

The case of oxygen is puzzling and still unsolved. When excess ^{16}O was discovered in the inclusions of Allende (Clayton et al. 1973), the most straightforward interpretation was an addition of supernova oxygen in these particular objects, but this is still debated today whereas many elements are unambiguously thought to display anomalies with stellar nucleosynthetic origins. Laboratory experiments showed that it was possible to create non-mass-dependent fractionating O by ultraviolet light photolysis, RF discharge or electrical discharge (see Thiemens (1988) for a review). Effects mimicking a ^{16}O addition up to 70‰ have been observed (Thiemens and Heidenreich 1983), and the implication of such effects in the solar system formation has been discussed and included in the interpretation of meteoritic data (Thiemens 1999; Clayton 2002). A number of questions include: how many such reservoirs can be created and to what extent are other elements involved?

Early proton irradiation within the solar system

The presence of short-lived nuclides like ^{41}Ca and ^{26}Al imply a very short time scale between stellar nucleosynthesis and the formation of planetary bodies. To accommodate this time constraint, it is tempting to try to include some of the short-lived nuclide production within the early solar system (X-wind model of Shu et al. (2000)). Such models have been developed earlier (Fowler et al. 1962; Heymann and Dziczkaniec 1976; Lee 1978) but their past action failed to be proven unambiguously (Burnett et al. 1966). On one hand, stellar nucleosynthesis satisfactorily explains the observed anomalies as discussed above. On the other hand, an irradiation process is required to produce ^{10}Be. This alternative is presently under debate. The model of Shu et al. (2000) is the latest evolution of such irradiation scenarios based on stellar evolution models. Relevant questions are: irradiation should also produce isotopic heterogeneity for some sensitive stable isotopes, and how homogeneously are the materials exposed to protons ? On the other side, can there be some unsuspected stellar site able to produce sufficient amounts of ^{10}Be ?

Presolar silicates

The huge isotopic variations in presolar grains have so far been found in diamonds, carbides and oxides. This is due to the separation procedures using HF to remove the major minerals from the chondrites. It is expected that presolar silicate mineral grains also existed and future work directed toward their separation is anticipated. What nucleosynthetic signatures they could carry is also an open question. The extent of these large isotopic variations within the bulk of the presolar material is also unknown. Are all the grains constituting the protosolar cloud so isotopically dispersed as the presolar grains found to date or is it some peppering process in which matter synthesized in the last few hundred m.y., before solar birth, is injected in a roughly homogeneous medium. The cosmic chemical memory (Clayton 1982; Jones et al. 1997) is still somewhat model dependent on this aspect.

Instrumentation

The instrumentation is constantly progressing in two directions: increased spatial resolution with the evolution of ion probes (Zinner et al. 2003), and increased precision with the evolution of multicollector TIMS and ICPMS instruments. There is no doubt that the possibility of precisely addressing the mass-dependent fractionation effects, new visions of early solar system history will emerge. Smaller presolar grains can now be investigated and a strong push exists to develop specific instruments to analyze as many elements as possible within a single sub-micron grain, and automated isotopic analysis (Savina et al. 2003b; Nittler and Alexander 2003).

There are many other questions underlying the measurement of isotopic anomalies and future planetary explorations will bring new stones to the construction.

CONCLUSION

Despite the numerous and sometimes huge isotopic anomalies found during the past 30 years, the solar system, at a first glance at the meter scale and at the percent level of precision, still looks remarkably homogeneous. The formation of the sun and the planets was a very efficient homogenizing process. The discovery of the isotopic anomalies in the 1970s has opened a fascinating door to the presolar history of the solar system materials. They allowed characterization of a number of nucleosynthetic processes expected to be active in the galaxy and which are required to produce the isotope ratios of elements found in the solar system. A number of processes have been clearly identified and connected to astronomical observations and astrophysical models. By using individual grains ejected by stars, a somewhat unexpected large diversity of effects has been discovered even within the same process. The interpretation is complex and lies between two extremes. One possibility is that measured ratios are straight production ratios within stars and the very large isotopic variability reflects the variability of stars, and mixing processes within stars, having contributed to the solar system. On the other hand, there can be a limited number of stellar processes producing extreme isotopic compositions, and the observations may reflect various mixing ratios between these relatively few components during the long evolution between stellar interiors and planetary bodies in the solar system. Such mixtures are necessary to explain some observations, such as Xe-HL as one example. That secondary processes, like gas phase alteration operated during the evolution of the solar nebula is demonstrated in Allende inclusions, and parent body metamorphism in chondrites. The complexity of the image that we now have from the forming solar system results from the variety of the isotopic measurements. Nevertheless the accumulation of new data and the improvements in the models of planetary formation, will help constrain more strictly the unknowns.

ACKNOWLEDGMENTS

I wish to thank the anonymous reviewer, M. Kurz and R.N. Clayton for the comments and the improvements of the text of this overview. Many thanks to J. Dyon for his help for the Figures. I am also grateful to C. Göpel, G. Manhès, A. Trinquier, M. Moreira and C.J. Allègre for the numerous discussions about radioactive isotopes and isotopic heterogeneity. This is IPGP contribution no 1961.

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