RADIOMETRIC DATING AND TRACING

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Summary

Advanced search for analytical improvements has produced a substantial variety of precise isotope chronometers presently applied to Earth Science problems. The methods or techniques are based on different radioactive decay processes, different ways of measuring isotopes or different evaluation procedures and have their own applications. The preferred technique (or isotope/nuclide system) depends on the half-life of the parent nuclide and the predicted age of the target of interest. This article reviews radiometric dating and tracing techniques currently employed in geological and archaeological sciences and introduces technological developments and strategies that allow to “read” geological time or to quantify the duration of geological processes. A major focus is given to the application of isotope dating and tracing. Our main motivation here is to discuss some exciting new discoveries that provide insight into processes ranging from the earliest phases of terrestrial evolution to the cradle of humanity.

1. Introduction

Small amounts of natural radioactive nuclides (both long-lived and short-lived) and their decay products occur in nearly all ordinary matter. The long-lived unstable nuclides (^{40}K, ^{87}Rb, ^{147}Sm, ^{176}Lu, ^{187}Re, $^{235/238}U$, ^{232}Th) have not entirely decayed to extinction since the formation of the solar system. The short-lived ones have either become completely extinct by now (e.g., ^{182}Hf, ^{146}Sm), or continuously generated in the decay chain of uranium and thorium (e.g., ^{234}U, ^{230}Th, ^{231}Pa, ^{226}Rn), or produced by cosmic ray bombardment of stable nuclides (e.g., ^{10}Be, ^{14}C, ^{26}Al, ^{36}Cl). Radioactive decay causes the isotopic composition of certain elements to change with time, and materials of different age or different origin contain specific records of isotope variations. Quantitative determination of isotopes and isotope ratios from unstable nuclides or their decay products have been established as a powerful tool for understanding fundamental processes in Earth sciences. One important application which employs radiogenic isotope systems is in the field of radiometric dating. Radiometric dating (or geochronology) provides the key to understanding the evolution of the Earth and other planetary bodies over time. Together, the various dating methods can measure the time elapsed since a molten rock solidified or cooled below a certain temperature within the crust, an ore deposit was generated, or when organic material was once alive. Radiometric dating is the only means that can quantitatively measure geologic time and provides numerical age constraints for the evolution of the solar system and the Earth. Age correlations over more than 90% of Earth history are possible with this technique, much more than with fossil record that covers less than 15%. Relative timescales from fossil records or geomagnetic reversals are calibrated to numerical time by radioactive decay. Since different radioactive nuclides (parent atoms) decay into their radioactive or non-radioactive daughters at different rates, the age of a sample can be verified by using several dating methods. The numerous analytical results and the agreement between independent chronometric dating methods allow scientists to present a coherent picture for the Earth formation and its evolution.

Based on observation in the field, a geologist can generally determine relative geological ages from rock sequences consisting of sedimentary, metamorphic, or igneous rocks. A relative age specifies whether a rock sequence is older or younger than another one. Isotopic dating techniques determine how long an object has been in existence. Whereas relative ages can be deduced from visual observations on a local or regional scale, radiometric dating relies on isotope ratios of high precision and accuracy. It requires a great deal of laboratory study and equipment and needs the specialized experience of isotope geochemists. Interpreting the results in a correct way is rarely straightforward and requires a combination of geological skill and common sense. A large number of isotope systems are currently in use for age determination. A concise overview of available geological dating methods is presented in Igor Villa's article on Geochronology in the EOLSS encyclopedias. The present chapter places emphasis on analytical techniques and applications and includes examples from the field of archaeology.

2. Major Concerns

In many instances Earth scientists are interested in the time when certain geological material was initially formed. Fundamental questions concern the age of the Earth, its reorganization into core, mantle and crust and the formation and growth of continents through time. But also other questions like the appearance of life, the rates of formation and destruction of mountain belts, or the growth history of crystals in a magma chamber can be addressed. Radiogenic isotopes can not only serve as dating tools but also as useful tracers. This is possible because radioactive decay creates variation in isotopic composition in different natural systems or reservoirs. Isotopic tracing techniques utilize the variable radiogenic growth of the daughter isotope, which depends on the age and the ratio of the parent to the daughter atoms (i.e., parent/daughter ratio) of the sample. Isotopic tracers are used to reveal the origin of geological and archaeological material. Important geological applications of isotopic tracing include the study of the Earth's mantle and crust. Apart from the core, mantle and crust are the two major shells (or layers) of the Earth. Mantle and crustal rocks have developed different radiogenic isotope signatures through time, and thus rocks, in isotopic form, provide information about the reservoir in which they were formed. In other words, it is possible to judge whether a particular magmatic rock was derived from melting mantle or crustal material. Radiogenic isotopes can also be used for oceanographic studies to trace the isotopic evolution and the circulation pattern of seawater. In archaeological sciences, the major target of interest is the provenance of artifacts. Anthropological and biological material, such as hard tissue (bone or teeth), is used increasingly for making reconstructions of migration of hominid and historic human populations.

3. Limitations

Naturally occurring material contains small amounts of long-lived radioactive nuclides, so nature provides several dating tools and scientists have learned to read and to interpret these clocks. Each dating method is based on certain assumptions: the sample had to be a closed system, i.e., no loss or gain of parent and daughter isotopes could have taken place (other than that caused by the decay itself) because this would have affected the increase of the radiogenic isotope ratios. Although present-day analytical technologies enable precise measurements of isotope compositions, the ages obtained are sometimes difficult to interpret. The age date only reflects the true age of the mineral if the rock has not been subsequently altered. Such alteration could have occurred if the rock was in contact with a fluid phase deep within the Earth or exposed to the surface during a weathering cycle. In the simplest (and most idealistic) case when geological or biological materials, like rocks or bones were formed, they contained only parent atoms and no daughter atoms. If daughter atoms were present during the formation of the material their abundance needs to be known. The amount of initially present daughter atoms cannot simply be measured since it has changed through the growth of radiogenic daughter material. So, sometimes assumptions have to be made. The technique of isochron dating that is used in this case does not require knowledge of the initial proportions of daughter nuclides. In this technique the age is derived from several cogenetic samples, i.e., samples that formed at the same time from a material in which the relevant elements and isotopes were distributed homogeneously. The isochron technique is reviewed in Igor Villa's article on Geochronology.

4. Radioactive Decay

Radiometric dating and tracing is based on the decay of naturally occurring radioactive nuclides that were incorporated into the material of interest when it was formed. Radioactive decay of a specific nuclide into another nuclide occurs at a constant rate. Decay rates of radioactive nuclides depend on nuclear properties and are independent of changing external conditions like temperature, pressure and chemical environment. Small half-life variations (<1%) have only been reported for some electron capture processes in nuclides like $^{7}\mathrm{Be}$ which are, however, not used for radiometric dating. The rate of radioactive decay is either expressed in terms of the decay constant or the half-life. The half-life, often denoted by the symbol $T_{1/2}$, of the system under consideration is the time by which one half of the initially present atoms of the substance in question is decayed. Half-lives of naturally occurring nuclides used for radiometric dating vary from years (e.g. tritium - $^{3}\mathrm{H}$) to more than a hundred billion years (e.g. $^{147}\mathrm{Sm}$). The decay constant, λ, which represents the probability per unit time for one atom to decay, is related to the half-life, $T_{1/2}$ by:

$$T_{1/2} = \frac{\ln 2}{\lambda} \qquad (1)$$

The rate at which radioactive atoms, N, of a given parent nuclide decay is given by:

$$\frac{\mathrm{d}N}{\mathrm{d}t} = -\lambda N \qquad (2)$$

whereby $\mathrm{d}N/\mathrm{d}t$ is the number of disintegrations per unit of time. Integrating this equation leads to the exponential law of radioactive decay:

$$N = N_0\,\mathrm{e}^{-\lambda t} \qquad (3)$$

where N_0 refers to the number of originally present parent atoms.

The number of radiogenic daughter atoms ($D*$) produced during radioactive decay equals the number of parent atoms consumed. Assuming that the sample of interest had no atoms of this daughter at time $t = 0$, then the total number of daughter atoms $D*$ will be given by:

$$D* = N_0 - N \qquad (4)$$

Substituting for N_0 in the above equation yields:

$$D* = N(\mathrm{e}^{\lambda t} - 1) \qquad (5)$$

In other words, the age of a sample (t) can be determined if the concentration of parent and daughter atoms in the sample is determined, and the decay constant of the radioactive nuclide is known.

When the material was formed there might have been some daughter atoms present (D_0), thus total D after time t is given by:

$$D = D_0 + N(\mathrm{e}^{\lambda t} - 1) \qquad (6)$$

This necessitates knowledge of D_0 in order to calculate the age correctly or a reasonable estimate of the amount of initially present daughter atoms must be made.

In a parent-daughter decay system over time the number of daughter (radiogenic) atoms increases and the amount of the parent (radioactive) atoms decreases as they decay away. The ratio of the parent atoms to the daughter atoms is a direct function of time and, if the half-life of the system is sufficiently well known, by measuring this ratio, the age of the material of interest can be calculated.

5. Chemical Separation Techniques

Modern analytical techniques in radiometric dating and isotope geochemistry offer the ability to analyze elements at sub-microgram levels. Unextracted elements from solid matrix or mineral grains can be investigated using laser ablation techniques (see below). During laser ablation, isobaric interferences, caused by isotopes of different elements present in the sample, can be very troublesome and are sometimes not easily corrected. In order to obtain high-precision isotopic ratios from very small quantities of geological materials the element of interest has to be chemically separated and purified from the matrix prior to analyses. Sample handling, reagent preparation and chemical separation and purification have to be performed in an ultra-clean or contaminant-free non-metal environment (e.g. laminar flow benches, located in a clean room) in order to avoid sample contamination and to achieve ultra-low blank chemistry (background signal). Chemical separation of the elements is usually accomplished by cation or anion-exchange chromatographic techniques. Ion exchange chromatography is suitable to concentrate and purify the elements of interest. In case when the quantity of an element or element ratio needs to be known, as for high-precision geochronology, isotope dilution (ID) analysis is performed. In this procedure, an isotopically enriched spike solution of known isotopic concentration and isotope composition is added to the sample. The spike is normally enriched in a low natural abundance isotope. By measuring the isotopic composition of the mixture (i.e., sample and spike), the absolute concentration of the sample can be calculated. Standard laboratory equipment for high-precision geochronology and isotope geochemistry include laboratory balances, binocular microscopes, high-pressure digestion vessels, centrifuges, pipettes, resins for ion-exchange chromatography, ultra-clean water systems, sub-boiling distillation units for preparation of ultra-clean acids (nitric acid, hydrochloric acid, hydrobromic acid), hot plates for sample digestion and evaporation, and a variety of sample equipment fabricated from Teflon.

6. Mass Spectrometry

The isotopic composition of elements is determined by mass spectrometry techniques. Mass spectrometers, the “work horses” in isotope geochemistry and geochronology, are precision analytical instruments used to detect and quantify very small variations on isotopic compositions (Figure 1). Most dating methods described in Section 7 rely on mass spectrometric measurements. The wealth of information provided by mass spectrometric analyses on the evolution of the Earth is tremendous and this technique has revolutionized Earth Sciences. In general, mass spectrometers consist of an ion source, an ion transfer mechanism, a mass selective analyzer that produces a mass spectrum suitable for isotope ratio measurements, and a detection system. Mass spectrometers are equipped with electronic devices and a high-vacuum system (generally 10^{-7} to 10^{-9} mbar). High-vacuum conditions are achieved by various types of vacuum pumps which remove the air from the beamline so the beam particles have a free path through the mass spectrometer. Most types of mass-spectrometers use either Faraday cups or ion counters to measure the ion beam intensities. Over the past decade, a number of instrumental improvements and refinements have been made. Modern high-precision mass spectrometers are equipped with a moveable array of several Faraday detectors and one or several co-located ion counters, facilitating the simultaneous measurement of different isotopes of a given element over a wide dynamic range. The following discussion focuses on the various types of mass spectrometers which are currently in use for radiometric age determination.

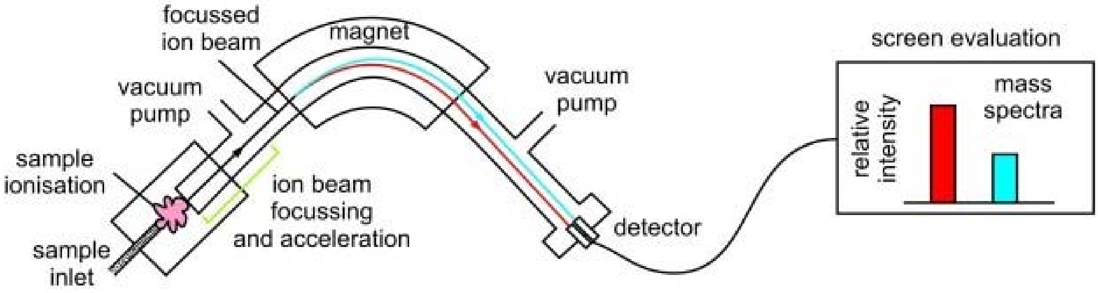

Figure 1. Schematic illustration of the basic features of a mass spectrometer. Red and blue colors represent the light and heavy isotopes of an element and their relative mass intensities (right).

6.1. Thermal Ionization Mass Spectrometry (TIMS)

The TIMS technology provides highly precise measurements of isotope ratios with internal (within-run) precision of 0.001%. For TIMS analyses, a sample is loaded on a metal filament, typically as a chloride or nitrate salt, and transferred in the ion-source of the mass-spectrometer. The sample filament consists of a non-reactive metal such as platinum, rhenium or tungsten. The filament is heated to high temperatures (between 900°-1800°C, depending on the element to be measured) by an electric current until ions are emitted from the sample. The ionization efficiency which is defined as the number of atoms ionized relative to the total number of atoms loaded onto the filament depends on a number of factors including the ionization potential of the element, filament work function and temperature but also on chemical, physical and geometric effects. For TIMS, ionization efficiency is very low and special loading techniques, e.g., the addition of ionization enhancers, or special filament configurations were developed to optimize ionization. Heating of a second filament, opposite to the sample, at an elevated temperature can result in improvement of ionization efficiency. After ionization, the ion beam is focused by a series of electrostatic lenses and is accelerated towards a magnetic mass analyzer. After the ion beam has passed the analyzer, the different mass ion currents (10^{-16} to 10^{-10} A) can be detected with Faraday cups (beams greater than 10^5 ions/s) that measure the voltage drop through large ohmic resistors (10^9 to 10^{11} Ω) or (for beams less than 10^6 ions/s) with ion counting detectors. If only a single-collector is used, ion beam intensities are measured sequentially in "peak jumping" mode by varying the magnetic field so that different ion beams can be cycled through the detector. Multi-collector TIMS instruments with several Faraday cups permit simultaneous detection of different ion beams. This allows the measurement of isotope ratios instead of ion beam intensities. Beam instabilities are thus cancelled out and the precision is highly increased. Standard TIMS does not utilize energy filters because thermal ionization produces an ion beam with a very small kinetic energy spread and doubly ionized particles are almost absent. TIMS instruments used for disequilibrium dating applications are equipped with energy filters, designed to improve the abundance sensitivity which is defined as the tailing contribution from one peak onto an adjacent peak. Such filters are important if extremely large isotope ratios of uranium ($^{238}\mathrm{U}/^{234}\mathrm{U}$) and thorium ($^{232}\mathrm{Th}/^{230}\mathrm{Th}$) have to be measured, to ensure that small ion beams of low abundance isotopes (e.g., $^{234}\mathrm{U}$, $^{230}\mathrm{Th}$) can be measured relative to large peaks (e.g., $^{232}\mathrm{Th}$ or $^{238}\mathrm{U}$) of adjacent masses. The newest generation of TIMS instruments is equipped with multiple ion counters for the simultaneous registration of multiple low-level ion beams. Future generation TIMS instruments might see further improvements in the ionization source (cavity sources or ion guns) which will further enhance ionization efficiency.

Progressive evaporation of the ionized material causes an increasing depletion of the lighter isotopes over the heavier isotopes from the filament surface. This time-dependent instrumental mass fractionation (or discrimination) which is in the order of ~0.1% per a.m.u. in the heavier mass range can be corrected by internal normalization of the measured ratio to an invariant ratio of the same element. For elements with no invariant isotope ratio (e.g., Pb) correction for mass-fractionation is accomplished using external standards. Isotopes of different elements can have almost exactly the same mass (isobars). For example, during strontium isotope measurement isobaric interference can be caused by ^{87}Rb onto ^{87}Sr. Isobaric mass interferences can be minimized by ensuring a clean chemical separation of the sample. If an interfering isotope is present its contribution can be eliminated by pre-heating the sample if it burns off before the sample element (e.g., rubidium during strontium measurements or cerium during neodymium measurement). There are also possibilities to monitor mass interference by measuring another isotope of the interfering element which is not present in the element to be analyzed.

6.2. Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

ICP-MS uses high-temperature plasma (~8000 °C) as ion source causing very efficient production of monatomic ions of most elements. The dissolved sample (untreated solutions or chemically separated analytes) is introduced into the plasma source as an aerosol using a nebulizer. The aspirated sample is introduced into the plasma torch by an argon or helium carrier gas where it is ionized. Although ICP-MS techniques enable the measurement of a variety of elements in solution, efficient separation chemistry is often required in order to reduce matrix effects. This is because every element is ionizes at virtually constant proportions. Matrix effects include elemental isobaric interferences, molecular interferences, double charge interferences, and other non-spectral effects. Instrumental mass discrimination is large (~0.5-1% per a.m.u. for heavy isotopes) but essentially constant over time in contrast to TIMS and can be reliably corrected. Conventional ICP-MS are equipped with a plasma source, an ion separation facility (quadrupole, magnetic sector field or time of flight) and a single detector (usually an ion multiplier). For those instruments precision is limited to 0.1% due to instabilities of the ionization conditions in the plasma source. To overcome these limitations, the new generation of ICP-MS instruments is equipped with a multi-collector system and a magnetic sector field. Although there is still much potential for further development these instruments have already achieved the precision of TIMS, with precision between 0.01 and 0.001% for element concentrations in the 100-10 ng level. One of the current weaknesses of MC-ICP-MS is the low transmission efficiency (typically <1%). Transmission efficiency is defined as the efficiency of transmission of ionized material from the plasma source through the mass spectrometer. Another problem is that the ICP plasma source generates ions with a relatively large energy spread. To achieve a smaller energy range, the newest incarnation of MC-ICP-MS instruments utilize double-focusing (energy and mass) systems or hexapole collision cell energy filters. In these instruments, an ion-energy filter is added between the ion source and the sector magnet. This configuration provides better peak shapes, i.e. it reduces the peak tails in the mass spectrum, thus increasing the abundance sensitivity. ICP-MS (both single and multi-collector instruments) can be combined with in-situ analyses by adding a laser ablation system. The laser ablation technique (LA-ICP-MS) uses highly focused laser light pulses to ablate material from a solid sample. The spatial resolution of a few micrometers of this technique enables surface or depth age profiling. The aerosol produced during the laser ablation process is transferred by a continuous stream of gas into the ICP-MS. These fast growing analytical technologies have, for instance, improved the ability to precisely measuring in-situ isotope ratios of hafnium in mineral targets such as zircons, and the data provide new insight into the chemical differentiation of the Earth. Over the past years MC-ICP-MS has become a widely accepted mass-spectrometric method for obtaining high precision isotope measurements and bears unprecedented, not yet fully explored potential for geochemists. Analytical improvements will be an ongoing future challenge in LA-MC-ICP-MS analyses since it still requires demanding correction methods for matrix effects or laser induced mass fractionation due to the large temperature gradient which occurs in the sample during ablation. To overcome such limitations some teams have already developed a laser system producing infrared light pulses having a pulse width of femtoseconds. It has to be demonstrated if this novel ablation design provides significant advantages like matrix independent and fractionation-free analyses.

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Biographical Sketches

Wolfgang Siebel is currently associate professor at Tübingen University. He received a PhD degree in Geology from Heidelberg University. His general research interests are focused on radiogenic isotopes and the evolution of the continental crust. He was involved in a wide range of geoscience projects at the Federal Institute of Geosciences, Hannover and the GeoForschungsZentrum, Potsdam. These studies have resulted in publications in journals including *Chemical Geology*, *Contributions to Mineralogy and Petrology*, *International Journal of Earth Sciences and Journal of Petrology*. He is a member of the German Mineralogical Society and the Geologische Vereinigung.

Peter Van den haute is a professor at the Ghent University (Belgium). He received his PhD degree in 1983 with a study of the etching characteristics of fission tracks in glass and a Fission Track dating work on apatites from the Precambrian basement of Central Africa. His further research mainly focuses on the calibration of the FT dating method and its application to crystalline basement rocks from Europe, Africa and Asia. He co-authored the first handbook (with G.A. Wagner) on the fission-track dating method. In 1993 he started a luminescence laboratory at his university that at present focuses on the age determination of young Quaternary aeolian sediments. His publications can be found in journals such as *Chemical Geology, Journal of Quaternary Science, Nuclear Tracks, Radiation Measurements and Tectonophysics.* He is a member of the National Geological Committee of the Belgian Academy of Science and of the Mineralogical Society of Great-Britain.