

Revolution and Evolution: 100 Years of U–Pb Geochronology

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U–Pb geochronology has its roots in a spectacular decade of discovery. Within about ten years of the discovery of radioactivity in 1896, old ideas about the nature of matter were overthrown and the seeds of geochronology were planted. After giants of physics like Ernest Rutherford moved on to other research, Arthur Holmes nurtured the new field of geochronology, combining physics, chemistry, and geology to produce the earliest quantitative geologic timescale. Over the following decades, geochronology experienced a series of revolutionary and evolutionary advances, and became a vital part of almost all fields of geology.

KEYWORDS: geochronology, history, radioactivity, Curie, Rutherford, Holmes

INTRODUCTION

Humans have long attempted to calculate the age of the Earth or of specific geologic events by studying either the natural world or religious texts. With the discovery of radioactivity, a new and powerful approach became available. However, it would take decades to fully exploit the new field of “geochronology,” with better instruments and a greater understanding of the nature of matter and the types of geologic materials best suited for dating various kinds of geologic events. This brief review gives a broad overview of the revolution that created geochronology and of some of the key evolutionary developments since then. Caveats: (1) Since almost all of the early history of geochronology is based on the decay of uranium and since many of the advances in geochronology, such as mass spectrometry and isotope dilution, were first applied to the uranium–thorium–lead (U–Th–Pb) system and then used to exploit other decay systems, this review concentrates on the U–Pb system. Other articles in this issue discuss several other methods. (2) Because of length constraints, the bibliography contains only a few specific references and a few useful, broader historical sources.

A MIRACULOUS DECADE

In late 1895, Wilhelm Roentgen discovered “X-rays,” creating a great sensation with the public and in the scientific community. French physicist Henri Becquerel was one of those inspired by Roentgen’s work and, in 1896, he found that uranium compounds emitted penetrating rays similar to Roentgen’s X-rays. He had discovered radioactivity (a term actually coined later by the Curies). Becquerel’s discovery triggered a spectacular decade of research that laid the foundations of modern physics and geochronology,

but not immediately. At first, Roentgen’s X-rays greatly overshadowed Becquerel’s “uranium rays.” Ironically, one of the few scientists who promptly followed up on Becquerel’s work was William Thomson (Lord Kelvin), then 73 years old. Kelvin, whose estimates of the age of the Earth based on simple cooling models had been a thorn in the side of geology for decades, confirmed Becquerel’s observations.

Kelvin’s experiments, his friendship with Pierre Curie, and the surprising lack of interest in

uranium rays evidently encouraged Marie Curie to study radioactivity for her PhD. In 1898, Marie Curie discovered that pitchblende (uranium oxide) was several times more radioactive than pure uranium metal, and she identified two new elements, also radioactive: polonium and radium. Curie laboriously isolated radium for detailed study from large amounts of uranium ore. In 1903, Pierre Curie and Albert Laborde reported that the radioactive decay of radium produced large amounts of heat. This stunning result invalidated a crucial assumption in Lord Kelvin’s calculations of the age of the Earth: that the Earth had no internal source of heat. Meanwhile, foreshadowing a darker side of the early research on radioactive materials, Becquerel and Pierre Curie published a paper on burns resulting from exposure to highly radioactive samples. Ultimately, many of the early workers in the field would pay a high price for their dedication.

By this time, the new field had expanded considerably (FIG. 1). In a series of brilliant papers in 1902, Ernest Rutherford and Frederick Soddy established that radioactivity resulted from the transformation of unstable elements into new forms. A competing theory, that radioactive elements were somehow absorbing then releasing energy from outside sources, soon died out. Rutherford and Soddy derived the mathematical principles of radioactive decay and suggested that helium (He) was a stable daughter product of U decay. Rutherford realized that the ratio He/U could be used to measure ages of geologic materials and presented the first dates based on radioactive decay in a 1904 lecture.

In a 1906 paper, Rutherford also suggested the possibility of U–Pb dates using the Pb/U ratio, based on a suggestion by Bertrand Boltwood in 1905 that Pb was also a stable daughter product of U decay. Rutherford believed that U–Pb dates would be more reliable than He–U dates, because the He/U ratios were commonly too low owing to the escape of helium by diffusion. Encouraged by Rutherford, Boltwood

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FIGURE 1 A few of the giants of the early years. From top left: Henri Becquerel (AIP EMILIO SEGRE VISUAL ARCHIVES, WILLIAM G. MYERS COLLECTION), Marie and Pierre Curie (AIP EMILIO SEGRE VISUAL ARCHIVES), Ernest Rutherford on the New Zealand 100 dollar bill (MATTINSON PHOTO), Frederick Soddy (COURTESY OF UNIVERSITY OF FRANKFURT PHYSICS-RELATED STAMPS), and Arthur Holmes ca. 1910 and ca. 1960 (COURTESY OF LONDON GEODATUM CENTRE, UNIVERSITY COLLEGE LONDON)

in 1907 calculated dates for 43 uranium ore samples based on their Pb/U ratios. These first published U–Pb dates, based mostly on published classical wet chemical analyses predating the discovery of radioactivity, ranged from 410 Ma to 2200 Ma. At this time, the rate of decay of U was poorly known. Nevertheless, the results confirmed the promise of the U–Pb system. Unfortunately the dates lacked specific geologic context, as Boltwood’s primary interest was in confirming that Pb was a final stable product of the decay of U.

ARTHUR HOLMES

Arthur Holmes was a small boy when radioactivity was discovered and a teenager when the first U–He and U–Pb dates were published. He grew up with the intense excitement of the dawning of a new scientific era. Holmes attended a “higher grade school” with a strong science orientation and an exceptional physics teacher who used geologic examples in many of his lectures. In 1907 Holmes began studying physics at the Royal College of Science, London. In his second year, Holmes renewed his interest in geology thanks to a dynamic geology professor, William Watts, and switched his emphasis to geology. Another professor at the Royal College of Science was Robert Strutt, who had published a series of careful studies on helium in a wide variety of minerals, including zircon (the first zircon date) and sphene (titanite), narrowly losing the race to publish the first U–He ages. Holmes seized the opportunity to combine his interests in geology and radioactivity, and began developing improved U–Pb dating techniques under the direction of Strutt.

Holmes’s timing was particularly fortuitous. Boltwood had returned to his primary research interest in the chemistry of the uranium and thorium decay series, leaving the newly born field of geochronology wide open. Almost immediately Holmes made important contributions by developing more sensitive chemical and radiochemical techniques for the measurement of U and Pb. Whereas Boltwood had been limited to U-rich ore minerals, Holmes could analyze minerals much poorer in U and Pb, such as zircon and even feldspar. In 1911, Holmes published a series of new U–Pb analyses and dates and also recalculated Boltwood’s earlier U–Pb dates. Holmes, the physicist, might have

stopped there, but Holmes, the geologist, investigated the geologic settings of all the dated samples and produced the beginnings of a geologic timescale. Building on this success, Holmes (1913) published a 196-page monograph boldly titled *The Age of the Earth*. The book summarizes early efforts to determine the age of the Earth, presents an expanded version of Holmes’s geologic timescale, from Pleistocene to Precambrian, and gives a flavor of the rather bitter controversies that surrounded geochronology at that time. Ironically, many of the same geologists who considered Lord Kelvin’s estimates of the age of the Earth to be much too young now objected that the minimum ages of the Earth based on radioactive decay systems were much too old!

EARLY ISOTOPIC RESEARCH

The shock waves from the discoveries of X-rays, radioactivity, and the radioactive transmutation of atoms into different forms inspired a great outburst of research on the nature of matter. For example, in 1911 Rutherford discovered the atomic nucleus by bombarding thin metal foils with collimated beams of alpha particles. In 1913, Joseph Thomson (Rutherford’s professor) built a “positive ray” apparatus, a simple antecedent of today’s mass spectrometers, and discovered that neon had two different forms, one with a mass of about 20 and another with a mass of about 22. Soddy, based on a suggestion from a friend at a dinner party, coined the term *isotopes* for atoms of the same element but with different atomic masses. The existence of the “neutron” was suggested by Rutherford in 1920 and later confirmed experimentally by James Chadwick in 1932. Thus, the basic picture of the atomic nucleus had been fleshed out.

In 1919, Francis Aston built an improved version of Thomson’s “positive ray” instrument. Aston’s “mass spectrograph” (FIG. 2A) used photographic plates to record the mass spectra of elements and allowed semiquantitative measurements of isotopic ratios. After confirming Thomson’s work on neon, Aston went on to investigate the isotopic composition of many elements over his career, including “ordinary” lead from samples of lead ore in 1927 and “radiogenic” lead from uranium ore in 1929. Lead



FIGURE 2 (A) Aston’s third mass spectrograph, used for Pb isotope analysis (COURTESY OF CAMBRIDGE PHYSICS OUTREACH); (B) Alfred Nier with the flight tube of his 180° mass spectrometer, used to measure the isotopic composition of uranium (UNIVERSITY OF MINNESOTA, COURTESY OF AIP EMILIO SEGRE VISUAL ARCHIVES); (C) for comparison, a modern SHRIMP IIe SIMS instrument (SOURCE: GEOSCIENCE AUSTRALIA)

from the uranium ore unexpectedly contained ^{207}Pb . This discovery led to the conclusion that U consisted of not just ^{238}U but also ^{235}U , a minor, previously unknown isotope that decayed to ^{207}Pb . With the knowledge that two different isotopes of uranium decay at different rates to two different isotopes of lead, the basis for modern U–Pb isotope geology and geochronology was in place.

NIER AND MASS SPECTROMETRY

Over the next several years, improvements in instrument design resulted in the “mass spectrometer,” in which photographic plates were replaced by collection and electronic amplification of the small electrical currents produced by the beams of positively charged ions of each isotope. The brilliant Alfred Nier not only made major contributions to mass spectrometer design (FIG. 2B) but also made several discoveries that were critical to advances in geochronology. In 1938, Nier found that the isotopic composition of “common lead”—for example, lead from lead ores—varied significantly. Earlier measurements of the mean atomic mass of lead from a variety of ore samples gave essentially identical results, leading to the assumption that all common leads had approximately identical isotopic compositions. Nier showed that ^{206}Pb and ^{208}Pb varied in a highly correlated way that fortuitously resulted in little or no variation in mean atomic mass. Nier proposed that common lead was a mixture of “primeval lead” of fixed isotopic composition, dating to the time of the Earth’s formation, and radiogenic lead generated by the decay of uranium and thorium after the Earth’s formation. This stunning conceptual leap was crucial to all subsequent estimations of the age of the Earth.

In 1939 Nier turned his attention to uranium and lead in uranium ores, making accurate measurements of the $^{238}\text{U}/^{235}\text{U}$ and $^{234}\text{U}/^{238}\text{U}$ ratios of uranium and of the decay constant for ^{235}U . Nier and his colleagues then reported lead isotope measurements for still more lead ores. These data attracted the attention of E. K. Gerling, Arthur Holmes, and Fritz Houtermans, who used them to estimate the age of the Earth. Gerling, Holmes, and Houtermans calculated ages for the Earth ranging from ca. 3.0 to 3.9 Ga based on the assumption that the ore with the lowest $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{207}\text{Pb}/^{204}\text{Pb}$ ratios approximated the isotopic composition of primeval lead. This assumption was incorrect, and the calculated ages were too low. The age of the Earth would have to wait just a little longer!

NUCLEAR FISSION AND ISOTOPE SEPARATION

Meanwhile, other momentous discoveries in nuclear physics were underway, with profound implications for geochronology, both directly and indirectly. In 1933, only months before Marie Curie’s death, her daughter and son-in-law, Irène and Frédéric Joliot-Curie, bombarded aluminum with alpha rays and, for the first time, artificially transformed a stable element into a different, radioactive element. Many labs began bombarding a wide range of elements with various particles. Irène Joliot-Curie and Pavel Savitch bombarded uranium with neutrons and produced a radioactive product that appeared to have the properties of lanthanum, ca. 100 atomic mass units (amu) lighter than uranium. As all other experiments to date had yielded irradiation products within a few atomic mass units of the target element, the Joliot-Curie–Savitch result seemed highly unlikely. Joliot-Curie and Savitch reported their results but refrained from making any bold interpretations. The Germans Otto Hahn and Fritz Strassmann replicated the Joliot-Curie–Savitch experiments, positively identified barium (also ca. 100 amu lighter than uranium)

as a product of the neutron irradiation, and concluded that the atom had been split. Lise Meitner, a colleague of Hahn’s who had been forced to flee Germany as a result of Nazi persecution, and her nephew, Otto Frisch, then provided a theoretical explanation for the new phenomenon, which they called “fission.”

The discovery of fission triggered another period of intense research and, with the onset of World War II, the “Manhattan Project” in the USA. An essential part of the Manhattan Project was the development of isotope-separation techniques to concentrate ^{235}U , the isotope of uranium susceptible to “induced fission” by neutron irradiation. The ability to separate isotopes would open the door to a major breakthrough in geochronology, the “isotope dilution method” for precise and accurate measurements of very small amounts of a large number of elements.

TILTON, PATTERSON, AND ISOTOPE DILUTION

After World War II many scientists recognized that the discoveries from military research could be applied to a wide range of research problems in physics, chemistry, and geology. Harrison Brown at the University of Chicago led a group studying trace elements in meteorites using improved mass spectrometry techniques, neutron activation, and radiation counting. The meteorite data would, it was anticipated, better define elemental abundances in the Solar System. Brown also hypothesized that iron meteorites would contain lead, but very little uranium or thorium, preserving the isotopic composition of Pb from the time the Solar System formed and enabling a significant advance in estimating the age of the Earth.

Key members of Brown’s group included Mark Inghram, a physicist and mass spectrometer expert, and graduate students Clair “Pat” Patterson and George Tilton (FIG. 3A, 3B). Patterson’s role was to develop new mass spectrometric techniques for measuring the isotopic compositions and concentrations of the small amounts of lead in meteorites. Tilton was to do the same for uranium and thorium. The meteorite Pb work was slow and very demanding, owing to the extremely low concentrations of Pb in most meteorite samples and the prevalence of lead contamination in the environment. Meanwhile, the Chicago group recognized that the methods they were developing for meteorites would be ideal for analyzing minerals from terrestrial crustal rocks, so Tilton and Patterson honed their techniques by analyzing virtually every mineral in a ca. 1.0 Ga granite sample, including zircon (for which they reported the first zircon U–Th–Pb isotopic ages), sphene (titanite), and apatite.

A major key to Tilton’s and Patterson’s work was the isotope dilution method—the addition of a known amount of a highly purified isotope of the element of interest to a sample. For example, they used a known amount of highly purified ^{235}U as a “tracer” or “spike” to measure natural uranium, which is over 99 percent ^{238}U . They measured the ratio of $^{235}\text{U}/^{238}\text{U}$ of the tracer plus sample by mass spectrometry, allowing accurate calculation of the amount of ^{238}U in the sample. The same approach was used for lead, using a tracer of a highly purified isotope of lead. The analysis of thorium was more complex. There is only one long-lived isotope of thorium, ^{232}Th , so Tilton followed in Marie Curie’s footsteps (albeit on a smaller scale), extracting ^{230}Th from uranium ore. ^{230}Th , like ^{226}Ra , is a radioactive “intermediate daughter” isotope from the decay of ^{238}U . From ca. 1 kg of very pure uranium ore, Tilton laboriously extracted ca. 1 mg of ^{230}Th for use as a tracer.

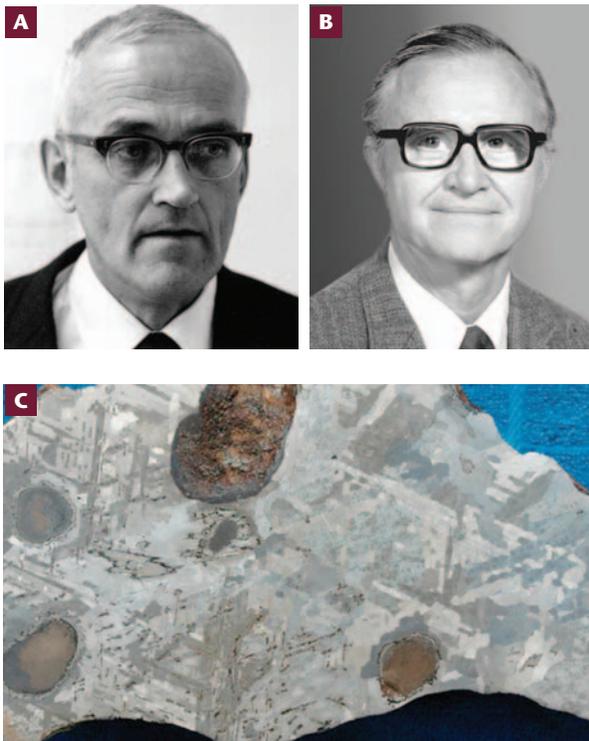


FIGURE 3 (A) Clair "Pat" Patterson (COURTESY OF THE ARCHIVES, CALIFORNIA INSTITUTE OF TECHNOLOGY); (B) George Tilton (COURTESY OF ELIZABETH TILTON); (C) Canyon Diablo Fe–Ni meteorite with nodules of troilite (COURTESY OF RANDY PARRISH); (D) synthesis of ^{205}Pb , ca. 1986; from left: Tom Krogh, Randy Parrish, and Atomic Energy of Canada scientist Steve Oelsher (COURTESY OF RANDY PARRISH)

Another key to success was the development of more sensitive mass spectrometric instrumentation and methods. The mass spectrograph of Aston required ca. 10 grams of Pb for a single analysis. The mass spectrometer of Nier required ca. 10 milligrams of Pb. Tilton and Patterson's improved methods plus Mark Inghram's more modern mass spectrometer, equipped with an electron multiplier developed a few years earlier by James Allen, allowed them to analyze ca. 10 micrograms of Pb—a thousandth of the amount needed by Nier and a millionth of the amount needed by Aston. Today, these methods are commonly referred to as "ID-TIMS" (isotope dilution thermal ionization mass spectrometry). With precise and accurate measurements of Pb and U in minor accessory minerals in common crustal rocks now in reach, a new era of geochronology began. At about the same time as the Tilton and Patterson work with U, Th, and Pb, isotope dilution and improved mass spectrometers were exploited to develop the Rb–Sr and K–Ar dating methods. For many years thereafter, the basic U–Pb, Rb–Sr, and K–Ar methods dominated geochronology.

Meanwhile, Patterson solved the difficult issues of the lead work on meteorites. He analyzed Pb from troilite, an iron sulfide mineral from the Canyon Diablo (FIG. 3C) and Henbury iron meteorites. The troilite contained abundant Pb but almost no U or Th, and thus preserved an archive of primordial Pb from the early Solar System, as hypothesized earlier by Brown. The troilite data, plus Pb data from stone meteorites and young terrestrial samples, the latter presumably a reasonable proxy for the average modern Pb isotope composition of the Earth, gave an age for meteorites and the Earth of ca. 4.5 Ga. Arthur Holmes warmly congratulated Patterson and his colleagues for their great achievement. The long quest had finally succeeded. A final hurdle was cleared when the "Top Secret" classification of ^{235}U from the Manhattan Project was lifted, and Tilton, Patterson, and their colleagues were able to publish their results for the granite and the age of the Earth in the open literature (e.g. Tilton et al. 1955 and Patterson et al. 1955, respectively).

KROGH AND HYDROTHERMAL DISSOLUTION OF ZIRCONS

The mid-1960s to early 1970s brought another series of major advances in U–Pb geochronology. In the field of mass spectrometry, digital data acquisition replaced less accurate manual reading of recorder charts. The development of a silica gel–phosphoric acid "emitter" for mass spectrometry of Pb and UO_2 by Angus Cameron and colleagues allowed a decrease in Pb sample size into the nanogram range. Arthur Jaffey and colleagues used alpha radiation counting on exceptionally pure isotopes of ^{235}U and ^{238}U to improve the precision and accuracy of the U half-lives by an order of magnitude. These advances set the stage for a seminal development by Thomas Krogh (1973). The classic Tilton et al. (1955) analyses used the materials and techniques available at that time for the digestion of zircon, a highly refractory mineral, i.e. decomposition in a borax-based flux at high temperature in a platinum crucible. Large volumes of reagents were required for the subsequent separation of Pb and U from the flux. The Pb "blank" (the amount of Pb introduced during processing of the sample) was large, and severely limited precision and accuracy in general and dating of small and/or young zircon samples in particular. Krogh took advantage of the availability of teflon™ (discovered by Roy Plunkett at Dupont) to develop a hydrothermal-dissolution technique for zircon. In the Krogh technique, zircon, along with highly purified hydrofluoric acid, is placed in a teflon vial. The vial is enclosed inside a stainless steel pressure vessel and heated in an oven at slightly below the failure point of the teflon. Typical zircon samples could be completely digested in several days, with a very low Pb blank. Lead blanks were further reduced by purification of acids in an all-teflon "2-bottle still" system designed by James Mattinson.

The Krogh technique, along with the highly efficient silica-gel emitter and new decay constants, was a generational breakthrough. With ongoing improvements, blanks and sample sizes have been further reduced into the picogram range, and precise, accurate U–Pb dating of single zircon crystals is possible at those labs willing to do the painstaking

ingly careful work required (for example, see Schmitz and Kuiper 2013 this issue). Krogh made many more significant contributions, including the production of ^{205}Pb for isotope dilution (Fig. 3D) and the development of an “air abrasion” technique (Krogh 1982) that could greatly reduce, and in some cases apparently completely eliminate, discordance due to Pb loss from zircon. Air abrasion was so effective that it was supplanted only recently by “chemical abrasion” (Mattinson 2005).

During the decade following Krogh’s development of air abrasion, remarkable new technologies entered the field of U–Pb zircon geochronology. In the early 1980s, microbeam analysis by secondary ion mass spectrometry (SIMS) burst into U–Pb geochronology (Fig. 2C), thanks largely to seminal contributions by William Compston and James Allen. About a decade later, U–Pb dating by the laser ablation inductively coupled plasma mass spectrometer (LA–ICP–MS) method arrived. Both are discussed in more detail by Nemchin et al. (2013 this issue), so are touched on here only briefly. Both techniques provide the spatial resolution to determine U–Pb dates on tiny spots within individual zircon grains, revealing and resolving the complex nature of many zircon populations (several excellent articles that discuss relevant aspects of zircon geochemistry and geochronology are in the February 2007 “Zircon” issue of *Elements*). The history of an individual zircon grain might include inheritance of older zircon from a magmatic source region, a period of new magmatic growth, plus one or more periods of later metamorphic replacement or overgrowth. With techniques that date entire single grains or populations of grains, such complexities can be missed. The price of such high spatial resolution is that precision and accuracy are about an order of magnitude lower than for ID-TIMS. As a result, microbeam techniques alone are less useful for research that requires the highest levels of precision and accuracy, such as high-resolution timescale work. Many labs now use the microbeam and ID-TIMS techniques to complement each other, taking advantage of “the best of both worlds.”

At about the same time as Krogh’s 1982 publication, a new generation of TIMS instruments became commercially available. These instruments can measure several isotopes simultaneously using multiple collectors, advanced

solid-state electronics, advanced vacuum pumps, and sophisticated computer control of data acquisition. The result has been improved precision and accuracy on still smaller samples, plus improved throughput. As analytical capabilities improved, it became more and more important that rigorous, statistical error analysis replace the vague estimates of earlier generations. No one has contributed more to this effort over the years than Ken Ludwig, whose Isoplot program is used around the world. Also vital to continued improvements is the EARTHTIME geochronology community initiative, started about a decade ago by Sam Bowring, Randy Parrish, and Paul Renne. EARTHTIME has promoted, among other things, cooperation and intercalibration of international U–Pb and Ar–Ar labs, driving significant improvements in all participating laboratories and making great progress on Arthur Holmes’s lifelong goal of a highly accurate geologic timescale.

SUMMARY

U–Pb geochronology (1) was conceived during one of the greatest scientific revolutions in history; (2) was born by merging physics, chemistry, and geology; (3) had a sometimes troubled childhood and adolescence; (4) as a young adult, matured significantly thanks to military experience and technological advances; (5) is now widely respected and appreciated, as are numerous younger siblings; and (6) has almost certainly an even brighter future.

ACKNOWLEDGMENTS

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