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# DISCOVERER AND METHODOLOGIST: ALFRED O. C. NIER AND THE MID-20<sup>TH</sup> CENTURY INSTRUMENTAL REVOLUTION IN GEOCHEMISTRY

# ABSTRACT

During the mid-20<sup>th</sup> century, geochemistry—one of the core Earth sciences—underwent a spectacular transformation as a result of the introduction of electronic instruments based on physical principles. In this process, mass spectrometry became the workhorse analytical technique in isotope geochemistry. This essay concerns the dynamic relationship between discoveries of isotope systems and the variations in their relative abundances, on the one hand—discoveries that became the foundation of isotope geology—and the development of mass spectrometry, on the other. This relationship is illustrated by the career of physicist and instrument-builder Alfred O. C. Nier, who was based at the University of Minnesota in Minneapolis. Nier's 60°-sector mass spectrometer design of 1940 endowed the instrument with powerful new capabilities, as well as facilitated its adoption outside the nuclear physics community. In the course of developing and applying the instrument, Nier also made important discoveries about the relative abundances of isotopes that paved the way for geochemical research on the deep past. My thesis is that Nier's early career, spanning the 1930s and '40s, illustrates a dynamic relationship in which science and technology co-evolved synergistically. This pattern of research spread beyond Nier—who largely moved on from this research after the 1950s—to develop into a research tradition, initially based at the University of Chicago's Institute for Nuclear Studies and then spreading to other institutions, notably Caltech, the Carnegie Institution of Washington and the University of California at Berkeley and San Diego.

This tradition made crucial contributions to historical geology, including paleoclimate, solar system history, and the tectonics revolution.

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The following abbreviations are used: NP, Alfred O. C. Nier Papers, University of Minnesota Archives; NGK, interview by Michael A. Grayson and Thomas Krick at the University of Minnesota, Minneapolis, Minnesota, 7-10 April 1989 (Philadelphia: Chemical Heritage Foundation, Oral History Transcript # 0112); PR, *Physical Review*; RSI, *The Review of Scientific Instruments* 

**Key words:** Geochemistry, geochronology, geology, Earth sciences, mass spectrometry, technology.

Abbreviated title: Nier: Discoverer and Methodologist

[FIRST LEVEL HEADING]

# **1** INTRODUCTION

Geochemistry spectacularly transformed scientists' ideas about the Earth and its history during the latter decades of the 20<sup>th</sup> century. Instrumental methods in geochemistry helped 20<sup>th</sup>-century scientists better understand the planetary movement of carbon, global lead pollution, plate tectonics, processes of mineral formation relevant for prospecting and mining, the history of the solar system, and the global hydrological cycle, on geological timescales. All of this has become central to our understanding of the Earth, and how humans can live upon it productively and sustainably.<sup>1,2</sup>

Evidence that supported many of these new ideas came from geochronology, the branch of geochemistry that exploits the constancy of the rate of radioactive decay to measure time.<sup>3</sup> Building upon models of radioactive decay developed in nuclear physics, geochronologists in the middle decades of the 20<sup>th</sup> century learned to determine the absolute age of various materials by interpreting isotopic ratios. By dating samples of earthly minerals, meteorites, and even materials that had formerly been alive—like wood, bone, or coal scientists could better reconstruct the history of these samples.

<sup>1</sup> (a) Peter J. Bowler, *The Earth Encompassed: A History of the Environmental Sciences* (London: W. W. Norton & Co., 2000). (b) Naomi Oreskes, *The Rejection of Continental Drift* (Oxford: Oxford University Press, 1999). (c)
 Spencer R. Weart, *The Discovery of Global Warming* (Cambridge, MA: Harvard University Press, 2008). (d) Sarah Dry, *Waters of the World* (Chicago: University of Chicago Press, 2019). (e) Matthew Shindell, *The Life and Science of Harold C. Urey* (Chicago: University of Chicago Press, 2019). (f) Ronald E. Doel, "Constituting the Postwar Earth Sciences: The Military's Influence on the Environmental Sciences in the USA after 1945," *Social Studies of Science* 33, no. 5 (2003): 635-666. (g) Naomi Oreskes and Ronald E. Doel, "The Physics and Chemistry of the Earth" in *The Modern Physical and Mathematical Sciences*, ed. Mary Jo Nye (Cambridge, UK: Cambridge University Press, 2002).
 <sup>2</sup> (a) William M. White, "History of Geochemistry," in *Encyclopedia of Geochemistry*, ed. William M. White (Cham: Springer, 2018), 670-683. (b) Rhodes W. Fairbridge, "History of Geochemistry," in *Geochemistry. Encyclopedia of Earth Science*, eds. Clare P. Marshall and Rhodes W. Fairbridge (Dordrecht: Springer, 1999). (c) Eville Gorham, "Biogeochemistry: Its Origins and Development," *Biogeochemistry* 13, (1991): 199-239.

<sup>3</sup> William M. White, *Isotope Geochemistry* (Oxford: John Wiley & Sons: 2015).

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Understanding how these significant intellectual changes occurred requires understanding the history of the scientific instruments used to make these measurements. Mass spectrometry was the workhorse analytical technique in isotope geochemistry. One of the most important figures in the development of mass spectrometry as a tool for geochronology was University of Minnesota physicist and instrument-builder Alfred O. C. Nier. Between 1935 and the 1970s, Nier developed a series of influential mass spectrometer designs optimized for measuring the isotopic ratios of various elements. When Nier began his career, mass spectroscopy was the province of physicists, without geological expertise and sustained professional interest in geological questions. During the late 1930s, Nier pioneered the application of mass spectrometry to geochronology, showing how the instrument could be used to measure lead isotope ratios connected to the uranium- and thorium-lead decay chains. Nier's 60°-sector mass spectrometer design of 1940 endowed the instrument with powerful new capabilities, as well as facilitated its adoption outside the nuclear physics community. During World War 2, Nier built prototype mass spectrometers for the Manhattan Project that were produced in volume by General Electric, thus exposing the instrument's capabilities to a greater number of scientists and engineers, including Nier's supervisor, Harold Urey. Nier's early career shows how scientists have worked as instrument-makers to create tools tailored to their research questions, the answers to which have then informed the next generation of tools—an example of the synergistic evolution of instrumentation and discovery. This pattern spread beyond Nier—who largely moved on from geochronology research after the 1950s—to develop into a research tradition, whose early epicenter was the University of Chicago's Institute for Nuclear Studies, and which then spread to other institutions, notably Caltech and

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the University of California campuses at Berkeley and San Diego. This tradition made crucial contributions to historical geology, including studies of paleoclimate, solar system history, and the tectonics revolution.

This paper aims to make three historiographical interventions. First, it aims to elevate Nier's profile as a significant figure in the history of science. When historians of science have paid attention to isotope geochemistry, they have generally focused on post-war developments interpreted through Cold War lenses. These studies briefly mention Nier's role as an instrument inventor, while focusing on the work of people like Harrison Brown, Claire Patterson and Harold Urey.<sup>4</sup> This paper shows the considerable debt these later and more celebrated geochemists owed to Nier—not just for his instrument designs, but also for showing how expertise in mass spectroscopy could be fruitfully applied to geological questions. While Nier did not have the drive (or perhaps hubris) to pursue the use of mass spectrometry as far as it could be taken in geochemistry, his intellectual lineage did.

<sup>&</sup>lt;sup>4</sup> (a) Ana Caneiro and Marianne Klemun, eds., "Seeing and measuring, constructing and judging: instruments in the history of the Earth sciences. Special issue." *Centaurus*, 53 (2011). (b) Matthew Shindell, "From the End of the World to the Age of the Earth: the Cold War development of Isotope Geochemistry at the University of Chicago and Caltech." In *Science and Technology in the Global Cold War*, ed. Naomi Oreskes and John Krige (Cambridge, MA: MIT Press, 2014). (c) Ronald A. Doel, "The Earth Sciences and Geophysics" in *Companion to Science in the Twentieth Century*, ed. John Krige and Dominique Pestre (New York: Routledge, 1997), 391-416.

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Nier's career has received more attention from mass spectroscopists and geochemists.<sup>5</sup> These histories focus on Nier's contributions to the development of mass spectrometry and geochronology. I will highlight how those contributions emerged in the context of two on-going lines of research: mass spectrometric measurement and geologic age determination. These were distinct fields in the 1930s when Nier began his career. I will argue that beyond particular inventions and discoveries, one of Nier's main contributions was to show how the two activities, mass spectroscopic measurement and geologic age determination, could be carried out fruitfully by the same scientist.

Historian and philosopher of science Hasok Chang's notion of an 'epistemic activity' will be helpful for understanding Nier's contribution. Chang defines an epistemic activity as 'a coherent set of mental or physical actions (or operations) that are intended to contribute to the production or improvement of knowledge in a particular way, in accordance with some discernible rules." Epistemic activities are generally practiced in relation to others, forming what Chang calls a 'system of practice.' A set of epistemic activities forms such a system when

<sup>&</sup>lt;sup>5</sup> For a very rich oral history, see NGK, highlights of which are reported in (a) Michael A. Grayson, "Professor Al Nier and His Influence on Mass Spectrometry," *Journal of the American Society of Mass Spectrometry*, 3 (1992): 685-694 (b) John H. Reynolds, *Alfred Otto Carl Nier*, *1911-1994* (Washington, D.C.: National Academies Press, 1998) (c) Konrad Mauersberger, "Alfred O. C. Nier (28 May 1911 – 16 May 1994)," *Proceedings of the American Philosophical Society*, 143, no. 4 (1999): 686-691. (d) Michael A. Grayson, ed., *Measuring Mass*, (Santa FE: American Society for Mass Spectrometry, 2002). (e) John De Laeter and Mark D. Kurz, "Alfred Nier and the Sector Field Mass Spectrometer," *Journal of Mass Spectrometry*, 41 (2006): 847-854. (f) Keith A. Nier, "Alfred Otto Carl Nier," in *The Encyclopedia of Mass Spectrometry, Volume 9: Historical Perspectives, Part B: Notable People in Mass Spectrometry*, ed. Michael L. Gross and Richard M. Caprioli (Amsterdam: Elsevier, 2016), 167-169.

they are performed with a view to achieving certain aims. The system is coherent when the constituent activities combine effectively to achieve the aims of the system.<sup>6</sup> Applying these notions to Nier's career, I argue that he showed how mass spectroscopy and geochronology could be combined to effectively achieve the aim of age determination. In so doing, and to the extent his approach was adopted, geochronology was transformed into a technologically-driven endeavor. It is often noted that science and technology have co-evolved synergistically.<sup>7</sup> I would emphasize, however, that that dynamic is not given, but is an *achievement* that can be more or less successful. Nier helped to establish an extremely successful system of practice in isotope geochemistry in which instrument development and scientific discovery could co-evolve synergistically.

Second, this paper aims to make connections with the literature on the Instrumental Revolution in chemistry. Alfred Nier and his intellectual descendants were part of a bigger change sweeping chemistry during the middle decades of the 20<sup>th</sup> century. Historians of chemistry have identified an "instrumental revolution" that began in the 1930s, picked up steam in the 1940s and had stabilized by the late 1960s.<sup>8</sup> In this period, electronic instruments,

<sup>&</sup>lt;sup>6</sup> Hasok Chang, "Compositionism as a Dominant Way of Knowing in Modern Chemistry," *History of Science* 49 (2011): 247-268, on 250-251.

<sup>&</sup>lt;sup>7</sup> In the case at hand, by John R. de Laeter, "Mass Spectrometry and Geochronology," *Mass Spectrometry Reviews*, 17, (1998), 97-125.

<sup>&</sup>lt;sup>8</sup> (a) George Borg, "On 'the Application of Science to Science Itself:' Chemistry Instruments and the Scientific Labor Process," *Studies in History and Philosophy of Science*, 79 (2020): 41-56; (b) José A. Chamizo, "About Continuity and Rupture in the History of Chemistry: the Fourth Chemical Revolution (1945–1966)," *Foundations of Chemistry*, 21,

many of them based on physical principles, transformed the work practices of analytical chemistry. Like Nier's mass spectrometers, these instruments enabled new ways of analyzing materials that largely replaced the practices of wet chemistry, gravimetric and volumetric analysis, and chemical separation. The instrumental revolution in *geochemistry* multiplied the ways of distinguishing minerals. In addition to chemical and structural properties, scientists could now analyze a mineral's atomic (e.g., absorption and emission spectra) and nuclear characteristics.<sup>9,10</sup>

In this context, Nier's career resembles those of the scientists described in Carsten Reinhardt's seminal book on the Instrumental Revolution in chemistry, *Shifting and* 

<sup>9</sup> For an indication of how chemistry-intensive geochronology was before Nier, see the first attempt, by Arthur Holmes, to quantify the geological timescale using radiometric dates: (a) "The Association of Lead with Uranium in Rock-Minerals, and its Application to the Measurement of Geological Time," *Proceedings of the Royal Society (London) Series A*, 85 (1911): 248-256. For geochemistry more generally see, for example, (b) Henry S. Washington, *The Chemical Analysis of Rocks*, (New York: John Wiley & Sons, 1930), and (c) W. F. Hillebrand, "The Analysis of Silicate and Carbonate Rocks," *United States Geological Survey Bulletin*, 700 (1919): 1-285

<sup>10</sup> For overviews of these developments, see (a) Philip J. Potts et al., "Advances in Analytical Technology and its Influence on the Development of Modern Inorganic Geochemistry: a Historical Perspective," in *Magmatic Processes and Plate Tectonics*, eds. H. M. Prichard et al. (London : Geological Society, 1993). (b) James M. Mattinson, "The Geochronological Revolution," in *The Web of Geological Sciences: Advances, Impacts, and Interactions,* ed. Marion E. Bickford (Boulder: The Geological Society of America, 2013). (c) Clark M. Johnson et al., "Smaller, Better, More: Five Decades of Advances in Geochemistry," in Bickford (ref. 13b).

no. 1, (2019): 11-29; (c) Peter J. T. Morris, ed., *From Classical to Modern Chemistry: The Instrumental Revolution,* (London: Science Museum, 2002); and references therein.

*Rearranging.*<sup>11</sup> Reinhardt focuses on the role of "method makers" in the transfer of physical theory and instrumentation to chemistry. He shows that this process involved the adaptation of the theory and instrumentation to the needs, knowledge and practices of the importing field, and so was more than a case of "applied physics." For this reason, he and others have also emphasized the role of industry in mediating the transfer. As will become apparent, Nier is a good candidate for a "method maker." For the most part, however, my focus will be on the epistemic pre-requisites for a system of practice to come together rather than on the problem of knowledge transfer. Partly for this reason, I will focus on academic developments, though Nier and the scientists he influenced were clearly indebted to industry for certain technical advances. Nevertheless, throughout this paper I will highlight ways in which Nier's work superseded chemical techniques in geochronology.

Third, and most broadly, focusing on Nier's early career also balances out studies that focus on the impact of WWII and the Cold War on the development of geochemistry. Excellent scholarship has amply documented the tremendous transformations in the Earth sciences during and after the world war. The perceived needs of militaries and national security policy makers during WWII, and then the Cold War, led to a vast increase in funding, increased access to distant and extreme environments, the creation of new institutions, and a new availability of expensive new tools and instruments. Scientists put these resources to work in fields ranging from geophysics and space science to oceanography, meteorology, and seismology. Scientists investigated new parts of the Earth, including the deep seas, upper atmosphere, and terrains

<sup>&</sup>lt;sup>11</sup> Carsten Reinhardt, *Shifting and Rearranging*, (Sagamore Beach: Science History Publications, 2006).

that could now be bored and cored.<sup>12</sup> Scientists certainly asked new questions and reoriented what they considered worth studying, sometimes all but abandoning previous questions that had been central to their disciplines.<sup>13</sup> Nier's early career, however, suggests that there were important continuities and precursors to the post-war flourishing of isotope ratio mass spectrometry. In this respect, his career supports Mary Jo Nye's thesis of partial continuity between pre- and post-war chemistry and physics.<sup>14</sup> Before direct US involvement in the war, Nier extended a theoretical framework that had been developed in the early decades of the century to develop instrumentation, methodology and empirical knowledge that would become fundamental to isotope geochronology after the war. Moreover, the post-war black-boxing of the technology required integrating these different kinds of knowledge—theoretical, instrumental, methodological, and empirical—so that non-experts could apply them. Nier's prewar spectrometer designs helped accomplish this integration.

In short, I will argue that Nier should be credited with the following accomplishments:

- Helping to transfer mass spectrometry from physics to geochronology and geochemistry.
- Founding a system of practice based on uniting mass spectrometry and age determination.

<sup>&</sup>lt;sup>12</sup> John Cloud, ed. *Social Studies of Science* 33, no. 5 (2003). Special issue on Earth sciences in the Cold War.

<sup>&</sup>lt;sup>13</sup> E. g., Naomi Oreskes, *Science on a Mission*, (Chicago: University of Chicago Press, 2021).

<sup>&</sup>lt;sup>14</sup> Mary Jo Nye, *Before Big Science*, (Cambridge, MA: Harvard University Press, 1996).

 Demonstrating the exceptional complementarity between the instrument and object of measurement, via the tailoring of the instrument to its object and the making of exceptionally challenging measurements.

I begin by tracing Nier's career. Nier's education in electrical engineering prepared him to improve on existing mass spectroscopy apparatus, and as a graduate student he produced a novel kind of mass spectrometer that could measure low-abundance isotopes. As a post-doc at Harvard, Nier shifted his focus to geochronology, becoming a significant contributor to research in that field. During World War 2, Nier's innovative mass spectrometers developed for the Manhattan Project were built in significant numbers, contributing to the "black boxing" of mass spectrometry and the instruments' becoming accessible to a wider range of users. After the war, Nier disseminated mass spectrometry across the sciences, working in geochemistry, biochemistry, and space exploration. I then explore how mass spectrometers were used in post-war geochemistry. Nier's methodological descendants made key discoveries, propagating a Nierian tradition of research. I conclude by reflecting on the conditions for a synergistic coevolution of science and technology to take off, and Nier's role in bringing about take-off in geochronology.

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# 2 EARLY ACADEMIC CAREER

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# 2.1 From Engineering to Physics at Minnesota, 1927-1936

Alfred Nier was born in 1911 in St. Paul, Minnesota to a family of German immigrants. As a youth, he exhibited a strong interest in the new technology of radio.<sup>15</sup> After graduating high school in 1927, he earned both a bachelor's (1931) and master's (1933) degree in electrical engineering at the University of Minnesota. Nier later recalled that he mainly took physics courses for the master's degree due to the very slim graduate offerings in electrical engineering at the time.<sup>16</sup> His master's thesis was devoted to an analysis of how the magnetic flux changes in an iron bar when the latter is placed inside a variable magnetic field.<sup>17</sup> Nier showed how equations previously used to model current in an electrical transmission line could be applied, by analogy, to the magnetic flux changes. He also criticized previous investigators who "evidently did not realize all of these facts [pertaining to the application of the equations to flux] and consequently applied equations blindly."<sup>18</sup> This knowledge of the interplay between theory and apparatus would serve Nier well when he switched disciplines, but stayed on campus, to pursue a Ph.D. in physics at the University of Minnesota.

<sup>&</sup>lt;sup>15</sup> NGK, 5.

<sup>&</sup>lt;sup>16</sup> NGK, 7.

<sup>&</sup>lt;sup>17</sup> This phenomenon is related to the Barkhausen effect, the noise produced in wires of certain materials when they are placed inside a varying magnetic field. The noise arises from discontinuous changes in the magnetic flux as the field is varied. This effect can be exploited to assess the quality of materials. See K. J. Sixtus and L. Tonks, "Propagation of Large Barkhausen Discontinuities", *PR*, 37 (1931): 930-959, cited in Alfred O. C. Nier, "The Magnetic Transmission Line," Master's Thesis (Minneapolis: University of Minnesota, 1933), NP, Box 10, Folder 21. <sup>18</sup> Nier, ibid., 24.

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Nier joined the lab of physics professor John T. Tate. Tate had become interested in mass spectroscopy as a means for studying electron impact phenomena like the ionization potentials of atoms and molecules. The Tate lab had developed expertise in the construction of apparatus, as well as in high vacuum techniques, thus distinguishing it from other academic labs because, as Nier recalled later, at the time "very few people in the academia [*sic*] had good vacuums."<sup>19</sup> As part of its broad concern with instrumentation, the lab had made a few key improvements in the mass spectrometer that would be key to the eventual success of Nier's own instruments.

For our purposes, the basic design principles of a mass spectrometer may be characterized as follows. Functionally, a mass spectrometer is any instrument "that can sort ions from a sample under investigation by their mass-to-charge ratios and that … could be used to produce a mass spectrum," the record of the different masses in the ionized sample.<sup>20</sup> Structurally, all mass spectrometers consist of five parts: sample inlet, ion source, mass analyzer, detector, and recorder. The core of the instrument, the mass analyzer, separates the ions by electric or magnetic fields, or both. Though many kinds of analyzers have been invented since then, the dominant design at the time of Nier's early career was deflection by a magnetic

<sup>&</sup>lt;sup>19</sup> NGK, 15, 34, 56. According to Nier, these techniques were more widespread in industry at the time. Brian Cathcart's history of the discovery of nuclear fission, *The Fly in the Cathedral* (London: Viking, 2004), 109-111, suggests that top-of-the-line vacuum systems were something of a luxury in academic physics labs at this time.
<sup>20</sup> Keith A. Nier, Alfred L. Yergey, and P. Jane Gale, "Overview," in *The Encyclopedia of Mass Spectrometry, Volume 9: Historical Perspectives, Part A: The Development of Mass Spectrometry,* ed. Michael L. Gross and Richard M. Caprioli (Amsterdam: Elsevier, 2016), 1-3.

field. The first instrument featuring this design had been reported by Arthur Dempster in 1918.<sup>21</sup> Dempster also made electrical detection, via measurement of currents generated by the ions at the detector, his method of choice, as opposed to detection by photographic plate favored by his rival, Francis Aston. Nier situated his own instruments in the Dempsterian tradition and argued that electrical detection was superior for measuring relative abundances.<sup>22</sup>

Nier's first physics paper was, in fact, a piece of applied electrical engineering. He reported the invention of a device to compensate for the fluctuations of the magnetic field of a mass spectrograph. Nier recalled that

[block quote] it was at this point that my engineering background and connections came in...It occurred to me that, since the mass that you collect is proportional to the square of the magnetic field divided by the accelerating voltage, why don't you fool the instrument by monitoring the magnetic field, and changing the ion accelerating voltage so that you stay on the peak ... The result was, the ions didn't know the difference, so the trajectory was stable. I could work with heavy ions where you needed high resolution. [block quote]

Here Nier was describing an instance of feedback control, an electrical engineering concept. The instability was due to a motor generator that was used to produce the large amount of power, 5 kW, required to run the solenoid. Key to the compensator was the use of a

<sup>&</sup>lt;sup>21</sup> Keith A. Nier, "Dempster's Descendants—The Core of the Development of Mass Spectrometry," *Journal of Mass Spectrometry*, 55 (2020): 1-7.

 <sup>&</sup>lt;sup>22</sup>(a) "Determination of Isotopic Masses and Abundances by Mass Spectrometry," *Science*, 121 no. 3152 (19550, 737-744.

magnetron, a kind of vacuum tube whose current can be controlled by an external magnetic field, in this case the spectrograph's. Nier credits the magnetron idea to his master's advisor, H. E. Hartig.<sup>23</sup> Nier did not see a sharp distinction between science and engineering, commenting that "it's so interesting how these things [engineering and experiment] overlap. It isn't quite clear which is which, you see."<sup>24</sup>

From the beginning of his physics career, Nier's approach consisted of pushing the instrumentation to its limits in order to detect extremely low abundance isotopes or abundance variations. Nier's dissertation research involved building an improved mass spectrometer and using it to study low abundance isotopes. He sought to make possible "accurate quantitative measurements on the relative abundances of isotopes of a given element."<sup>25</sup> Nier felt that previous mass spectroscopic apparatus was not sensitive enough to detect weak isotopes and was prone to contamination from water and hydrocarbons present in wax and grease in the joints and stopcocks of the apparatus. To address this problem, Nier first built a new mass spectrometer and published its design in 1935.

The 1935 machine combined Nier's feedback control mechanism to stabilize the magnetic field, with five improvements that had previously been developed by members of the Tate lab. First, graduate student Walker Bleakney had figured out a method for creating ions by

<sup>&</sup>lt;sup>23</sup> See Alfred O. Nier, "Device to Compensate for Magnetic Field Fluctuations in a Mass Spectrograph," *RSI*, 6 (1935): 254-255.

<sup>&</sup>lt;sup>24</sup> NGK, 17-18.

<sup>&</sup>lt;sup>25</sup> Alfred O. Nier, "A Mass Spectrographic Study of the Isotopes of Argon, Potassium, Rubidium, Zinc, and Cadmium," Doctoral Thesis (Minneapolis, University of Minnesota: 1936), p. 2.

bombarding the sample with electrons of controlled energy, known as the "electron impact" method. Descendants of this method are still widely used today. Second, Tate and graduate student P. T. Smith had come up with a novel design that isolated the ionization process from the mass analyzer section of the instrument. This design made it possible to construct instruments with larger magnetic analyzers, which made higher mass resolution possible. Third, the use of larger analyzers required the use of larger magnets, and it so happened that Nier was able to take advantage of a larger solenoid magnet, which had been constructed for unrelated cloud chamber studies. The larger magnet also contributed to enhanced resolving power. Fifth, graduate student Merrill Distad and post-doc John Williams had designed and constructed a novel detector, consisting of an electrometer vacuum tube amplifier of extremely high sensitivity. Nier's 1935 machine used a modified 180° magnetic sector instrument design previously used to study ionization potentials ('sector' here refers to the sector of the circle described by the ion flight path; see Fig. 2). The result was an instrument with exceptionally high stability, resolution and sensitivity. It enabled Nier to make an important discovery that top mass spectroscopists of his day had been unable to make.<sup>26</sup>

Though Nier initially applied the instrument to the study of organic compounds, he quickly moved to that of isotope abundances, which were of growing interest in nuclear physics, itself an increasingly popular field. Tate, as editor of *Physical Review*, was keenly aware of this trend and interested in getting into it himself. So Nier decided to turn his attention to

<sup>&</sup>lt;sup>26</sup> Alfred O. Nier, "Some Reflections on the Early Days of Mass Spectrometry at the University of Minnesota," International Journal of Mass Spectrometry and Ion Processes, 100 (1990): 1-13.

the study of argon and potassium isotopes. Potassium was of particular interest because of its observed, but unexplained, radioactivity. (It's the reason bananas make a Geiger counter click.) Nier later recalled thinking "[w]ell, this is just made to order, because if I'm going to get attention around here, I need to be in nuclear physics, and here it fit in."<sup>27</sup>

Nier was able to detect the extremely low abundance <sup>40</sup>K isotope that was suspected to be the source of the radioactivity.<sup>28</sup> Earlier analyses of the potassium isotopes by the pioneering mass spectroscopists Arthur Dempster, Aston and Kenneth Bainbridge had not detected <sup>40</sup>K. This isotope would become the basis for one of the pillars of geochronology, the K-Ar dating method (section 4).

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## 2.2 Geochronology at Harvard, 1936-1938

While a graduate student, however, Nier had not yet become interested in geochronology. This came during his subsequent post-doc in the Harvard laboratory of Kenneth Bainbridge from 1936 to 1938. On graduating in 1936, Nier won a coveted National Research Council Fellowship in physics, one of two awarded that year. Nier's graduate work had attracted considerable attention due to the power of his technique. Bainbridge thought Nier's

<sup>&</sup>lt;sup>27</sup> NGK, 14, 20-25.

<sup>&</sup>lt;sup>28</sup> Alfred O. Nier, "Evidence for the Existence of an Isotope of Potassium of Mass 40," PR, 48 (1935): 283-284.

precision measurement of isotope abundances could complement his on-going research, and offered Nier \$5000 (1936 dollars) in lab funds, free access to what Nier describes as excellent facilities and shop-work, and freedom of research. Nier accepted.<sup>29</sup>

Bainbridge was a respected physicist who, according to Nier, "was in mass spectrography in the business of measuring precise masses" and "had built this beautiful double focusing machine."<sup>30</sup> The latter would provide crucial inspiration for Nier's 60° sectorfield design of 1940 (*vide infra*). At the time, Harvard was the world leader in precision atomic weight measurements by chemical analysis. The Chemistry Department was home to Gregory P. Baxter, who carried on the "Harvard method" of making such measurements developed by his predecessor T. W. Richards. <sup>31</sup> The atomic weights measured by Richards, Baxter and others had revealed a difference between the atomic weights of what was called "common" or "ordinary" lead, on the one hand, and radiogenic lead from uranium and thorium ores, on the other. Common lead is lead found in non-uranium or -thorium ores, and is so-called because it is found in more common ores like galena. Nevertheless, uranium and thorium ores were often contaminated with this lead. It was thus recognized that the atomic weight difference could be used for dating purposes, namely by correcting the age of a sample for contamination by

<sup>&</sup>lt;sup>29</sup> NGK, 39-40, 53.

<sup>&</sup>lt;sup>30</sup> NGK, 39.

<sup>&</sup>lt;sup>31</sup> For accounts of this method, see Keith Nier, "The Transformation of Atomic Weight: New Instrumentation and Objective Progress," in 5<sup>th</sup> International Conference on the History of Chemistry — "Chemistry, Technology and Society" — Proceedings, ed. Isabel Malaquias, Ernst Homburg, M. Elvira Callapez (Aveiro : Sociedade Portuguesa de Quimica, 2005), 231-240, and references therein.

common lead. As will be explained in the next two sections, however, such correction would only work if one knew the isotopic composition of common lead, and until 1938 it was assumed that the composition was constant. Nier would show this assumption to be false.

[Third Level Heading]

# 2.2.1 State of isotope geochronology in the mid 1930s

When Nier went to Harvard, mass spectrometry was only just starting to be applied to geological questions. The discovery of isotopes in the early 20<sup>th</sup> century opened the way for the development of isotope geochronology, or the dating of processes and events by measuring relative isotope abundances. Ernest Rutherford (1906) was the first to propose that the proportion of radioactive atoms that disintegrate in a given time interval is an unvarying constant and therefore a potential clock. A particularly important kind of radioactive decay, in the early 20<sup>th</sup> century, was that of uranium into lead. Several scientists tried to exploit this process to develop an absolute geological timescale and establish estimates of the age of the Earth.<sup>32</sup> In order to measure the age of a sample, the quantities of both the residual 'parent' atom (e.g. uranium) and the decay product or 'daughter' atom (e.g. lead) had to be measured. Until the 1930s, this was done solely by bulk chemical techniques, involving chemical analysis of the sample followed by gravimetric or volumetric measurements.

The character of the task of radioactive dating was radically transformed by the recognition of the range and complexity of isotopic differences among the elements. In 1919,

<sup>&</sup>lt;sup>32</sup> For accounts of these efforts, see: (a) C. L. E. Lewis and S. J. Knell, eds. *The Age of the Earth: from 4004 BC to AD* 2002 (London: The Geological Society, 2001); (b) Patrick Wyse Jackson, *The Chronologers' Quest* (Cambridge, UK: Cambridge University Press, 2006).

Aston, at the University of Cambridge, constructed the first mass spectrograph to show conclusively that ordinary neon, a nonradioactive element, is composed of two isotopes of different abundances with masses 20 and 22 respectively, resulting in the chemical atomic weight of 20.2. During the following two decades, Aston, Dempster and other scientists continued to apply mass spectroscopy to identify the isotopic composition of elements across most of the periodic table. In 1929, Aston had used a mass spectrograph to detect isotopes of lead in radioactive bröggerite.<sup>33</sup> In articles from that same year, Rutherford, using Aston's data, estimated the age of the Earth while the geophysicists Fenner and Piggot postulated the first isotopic mineral ages.<sup>34</sup> It became clear that one could no longer hope to simply measure the uranium and lead contents of a sample in order to get an accurate age. Age measurements based on radioactivity would require the measurement of isotopes, not just bulk chemical analysis of the radioactive parent element and its daughter, and this would require mass spectroscopy. In particular, since both parents and daughters might consist of isotopes, each with its own decay properties, it was necessary to be able to accurately measure their relative abundances. In the uranium-lead case, for example, it was necessary to distinguish between two different processes, the decay of <sup>238</sup>U and that of <sup>235</sup>U. Moreover, uranium ore samples

<sup>&</sup>lt;sup>33</sup> F. W. Aston, "The Mass-Spectrum of Uranium Lead and the Atomic Weight of Protactinium," *Nature*, 123, no.
3096 (1929): 313.

<sup>&</sup>lt;sup>34</sup> (a) E. Rutherford, "Origin of Actinium and Age of the Earth," *Nature*, 123, no. 3096 (1929): 313-314. Nier
summarized Rutherford's article on an undated sheet (NP, Box 3, folder "Age of earth; computations, 1937, 1941,
1948, & n.d.". (b) C. N. Fenner and C. S. Piggot, "The Mass-Spectrum of Lead from Bröggerite," *Nature*, 123, no.
3108 (1929): 793-794.

were contaminated with common lead, which consisted of both radiogenic and non-radiogenic isotopes. Though traditional gravimetric methods were initially used to sort some of these isotopic complications out, their inherent sensitivity limits restricted the accuracy of this approach.

[Second Level Heading]

2.3 Ordinary Lead: Not So Ordinary

Cambridge, MA was also home to Alfred Lane, a professor emeritus at Tufts. Lane was chairman of the National Research Council Committee on the Measurement of Geological Age, and was excited at the possibility, raised by Nier's graduate work, of accurate isotope measurements of lead samples. Nier considered Lane "one of the very few persons in this country interested in quantitative geological age measurements" at the time, as opposed to qualitative methods based on sequences of rock strata and fossils.<sup>35</sup> In a letter from 1976, Nier recalled that Lane was "intensely interested in the geological age question" and credited him with providing moral support during the post-doc.<sup>36</sup> The extent of Lane's influence is also suggested by Nier's recollection that the former "was very interested in my work, and tried to promote it, getting samples and things for me to work on. So I spent most of my time, or a very large part of it, working on things related to geochronology."<sup>37</sup> Nier's papers contain an agenda for a 1937 meeting of Lane's committee in Boston in which various radioactive decay dating

<sup>35</sup> NGK, 54.

<sup>&</sup>lt;sup>36</sup> Alfred O. C. Nier to Henry Faul, 3<sup>rd</sup> November 1976, NP, Box 3, Folder 21.

<sup>&</sup>lt;sup>37</sup> NGK, 54.

methods were to be discussed alongside the "[i]mportance of work of A.O. Nier."<sup>38</sup> Lane's letters to Nier also exhibit enthusiasm about his results.<sup>39</sup> These sources suggest that Lane's enthusiasm and expertise had a major impact in spurring on Nier's work.

It was in this context that, with Bainbridge's assistance, Nier designed an improved mass spectrometer with higher resolution, thus enabling accurate abundance measurements on lead, uranium, and other heavy elements (resolution is mass-dependent). To improve the resolution relative to the 1935 instrument, Nier decided to double the radius of the mass analyzer and increase the strength of the magnetic field. This modification required a much larger magnet. According to Nier, the use of a larger solenoid would have been impractical, and so he and Bainbridge decided to replace it with a two-ton electromagnet, with the analyzer now sandwiched between the poles.<sup>40</sup> This modification produced a field approximately four to five times stronger than the solenoid without increasing the power requirement, though the latter (5 kW) remained high. This field was "very high for that time," which "pushed the whole thing [spectrometer] up to something that was different." The magnet was still unstable due to the power needed and the stabilizing device Nier had invented in Minnesota would not work with the new design. So Nier had to invent a new compensating device that stabilized the power supply to the magnet rather than the field itself, an approach that was actually facilitated by

<sup>&</sup>lt;sup>38</sup> NP, Box 11, Folder 5.

<sup>&</sup>lt;sup>39</sup> Alfred Lane to Alfred O. C. Nier, 7<sup>th</sup> January and 10<sup>th</sup> March 1939, and 13<sup>th</sup> May 1940, Box 3, Folder 30.

<sup>&</sup>lt;sup>40</sup> Nier, "Some Reflections," (ref. 25), 9.

the switch to an electromagnet.<sup>41</sup> (I mention these details because they are important for understanding the design of the later, seminal 60° sector instrument).

Initially, Nier focused on measuring the isotope abundances of several elements, justifying his research by appeal to the growth of nuclear physics and the power of his instrument for discovering rare isotopes and measuring relative abundances.<sup>42</sup> Under the influence of Baxter and Lane in particular, and with encouragement from Bainbridge, Nier shifted towards applying the new spectrometer to geochronology.<sup>43</sup> As a result of the work of Richards, Baxter and colleagues, the Harvard chemistry department had accumulated a large number of lead samples of high purity, which Baxter made available to Nier. Baxter's willingness to help Nier can be understood by considering the state of radioactive dating at the time. The latter was dependent on chemical atomic weight determinations to estimate isotopic composition, which limited the accuracy of the technique. For example, it was assumed at the

<sup>&</sup>lt;sup>41</sup> NGK, 50-51. In 1935, Nier had not deemed direct stabilization of the power supply feasible.

<sup>&</sup>lt;sup>42</sup> (a) Alfred O. Nier, "The Isotopic Constitution of Osmium," 52 (1937): 885; (b) Alfred O. Nier, "A Mass-Spectrographic Study of the Isotopes of Hg, Xe, Kr, Be, I, As, and Cs," *PR*, 52 (1937): 933-937; (c) Alfred O. Nier, "The Isotopic Constitution of Calcium, Titanium, Sulphur and Argon," *PR*, 53 (1938): 282-286; Alfred O. Nier, "The Isotopic Constitution of Strontium, Barium, Bismuth, Thallium and Mercury," *PR*, 53 (1938): 275-278.
<sup>43</sup> NGK, 53ff. In the latter, Nier states that Bainbridge introduced him to Baxter. He sometimes makes it sound as if Bainbridge might have suggested geochronology as a potential application. See (a) Alfred O. Nier, "Some Reminiscences of Mass Spectrometry and the Manhattan Project," *Journal of Chemical Education*, 66, no. 5 (1989): 385-388, on 385; and (b) Nier, "Some Reflections," (ref. 25), 9. In his letter of November 3, 1976 to the geologist Henry Faul, Nier "suspected" it had been Bainbridge who had informed him of the possibility of working on lead at Harvard: Nier to Faul, 3<sup>rd</sup> November 1976, NP, (ref. 31).

time that common lead is of constant isotopic composition, because its atomic weight was always found to have a value very close to 207.21 and because the U or Th content in its ores was negligible. In order to determine the age of a sample of a uranium ore, the atomic weight of the lead in it was measured, and the amount of common lead contamination estimated in light of the result. For instance, the closer the weight approached 206, the greater the purity of the sample in <sup>206</sup>Pb. This method was less than optimal, however, both because it is an indirect measure of isotopic purity, and also because the chemical methods employed were timeconsuming. In contrast, the mass spectrometric approach potentially offered a direct measure, as well as faster through-put. According to Nier, "I could do in an hour for which [*sic*] the chemists, in making atomic weight measurements, needed weeks." Baxter appears to have been especially excited by the speed of the mass-spectrometric analysis.<sup>44</sup>

For historical studies in isotope geology, three key results came out of Nier's research with this new and improved mass spectrometer. In July 1938, Nier disclosed that common lead consists of a mixture of isotopes the relative abundance of which varies by sample source. This was a great surprise, given all the previous atomic weight determinations, and given that that constancy is not to be expected if the relative abundances vary. These variations are caused by the addition of radiogenic lead, from uranium and thorium that had decayed long ago and was no longer present in the ore, to a "primordial" or "primeval" lead, of fixed isotopic composition, present at the time of formation of the Earth. Nier's discovery had three important consequences for geochronology. First, it showed that U-Pb dating would have to be corrected

<sup>&</sup>lt;sup>44</sup> NGK, 55-56.

for the isotopic composition of the specific common lead that is co-present in a given sample of uranium ore lead. Second, the variations gave insight into the history of the different lead ores. Most importantly, they permitted an accurate calculation of the age of the Earth, provided that one could find a sample of primordial lead (*vide infra*). Thirdly, it showed that massspectrometric measurements were superior to chemical methods for detecting small variations in isotope abundances.<sup>45</sup>

In January 1939, Nier followed up the common lead work with a pair of seminal publications on "The Isotopic Constitution of Radiogenic Leads and the Measurement of Geological Time, II," perhaps his most important foray into geochronology. He made the most precise measurements of the decay constants of <sup>235</sup>U and <sup>238</sup>U to date. He also measured the abundance ratio of the two isotopes. This latter quantity allowed him to develop an "internal" method for dating radiogenic lead samples that dispensed with the need to measure the absolute quantities of uranium and lead (*vide supra*). Instead, the age of a sample could be inferred from the ratio of <sup>206</sup>Pb to <sup>207</sup>Pb.<sup>46</sup> This method obviated the need for chemical analysis and was less susceptible to mineral alterations over time than the U/Pb or Th/Pb methods. In addition, Nier showed that mass spectrometric data could be used to compute ages from the decay of <sup>238</sup>U to <sup>206</sup>Pb and from that of thorium to <sup>208</sup>Pb, thus yielding three independent

 <sup>&</sup>lt;sup>45</sup> Alfred O. Nier, "Variations in the Relative Abundances of the Isotopes of Common Lead from Various Sources,"
 *PR*, 60 (1938): 1571-1576.

<sup>&</sup>lt;sup>46</sup> J. L. Rose and R. K. Stranathan ("Geologic Time and Isotopic Constitution of Radiogenic Lead," *PR*, 50 (1936): 792-796) were the first to suggest this method, but without a mass spectrometer had to determine the abundance ratio indirectly, leading to less accurate results.

methods for determining the age of a sample. He calculated that certain minerals were over two billion years old, which challenged various prior estimates of the age of the Precambrian, the Earth's time of formation and even the beginnings of the universe itself.<sup>47</sup> In 1959, his intellectual descendants George Tilton (see below) and G. R. Davis would credit this paper, and its 1941 follow-up, with reporting "[t]he first complete isotopic age determinations based on mass-spectrometric analysis of lead" and inaugurating "a more precise era in geochronology."<sup>48</sup>

Nier, however, decided not to pursue geochronology as far as his new instrument might take him. Discovering variations in common lead "opened up a whole new area, and I was aware of it at the time, but unfortunately, I didn't exploit it," Nier lamented in 1989. [block quote] If I had followed up on that I'd have been generations ahead of other people, because nobody else had instruments as good as I did for many years. I could have done much more, and could have come up with the age of the solar system...But...I

<sup>&</sup>lt;sup>47</sup> (a) Alfred O. Nier, "The Isotopic Constitution of Uranium and the Half-Lives of the Uranium Isotopes. I," *PR*, 55, (1939): 150-153; (b) Alfred O. Nier, "The Isotopic Constitution of Radiogenic Leads and the Measurement of Geological Time. II," *PR*, 55, (1939): 153-163. The results in (b) were amplified a few years later in (c) Alfred O. Nier, Robert W. Thompson and Byron F. Murphey, "The Isotopic Constitution of Lead and the Measurement of Geological Time. III," *PR*, 60 (1941): 112-116. In (b), Nier cites (on 163) Arthur Holmes' *The Age of the Earth* (London: Thomas Nelson & Sons Ltd, 1937), according to which the universe was thought to be no more than 2 billion years old at the time (on vi).

 <sup>&</sup>lt;sup>48</sup> G. R. Tilton and G. L. Davis, "Geochronology" in *Researches in Geochemistry*, ed. Philip H. Abelson (New York, John Wiley & Sons, Inc.), 190-216 (on 192).

#### NIER: DISCOVERER AND METHODOLOGIST

didn't know enough about geology, and was kind of advised by some people that I shouldn't stick my neck out on things that I didn't know much about. [block quote] <sup>49</sup>

Whoever those people were, Nier chose not to be explicit to historians. Thus, though Nier saw the potential of his discoveries for geochronology, he largely left it to others to connect the ages of individual minerals to the larger context, e.g. the ages of planets and the solar system.

Instead of pursuing radiogenic isotope geochemistry, in 1939 Nier made an important contribution to *stable* isotope geochemistry, of fundamental importance for the study of climate history. Together with Earl Gulbransen, an instructor at Tufts University, Nier recognized that the ratios of the two stable isotopes of carbon, <sup>13</sup>C and <sup>12</sup>C, vary by up to 5 percent depending on the sample source, with limestone-derived samples having the largest amount of <sup>13</sup>C and plant-derived the least. They suggested that the variation may be related to the sample's process of formation.<sup>50</sup> Though Nier and Gulbransen were not the first to observe stable isotope variations—variations in the oxygen isotopes had been discovered by bulk density measurements a few years before<sup>51</sup>—this work demonstrated the discriminating power of mass spectrometry for measurement. Indeed, in addition to theoretical anticipations of carbon variations, testing the power of the technique seems to have been part of the

<sup>&</sup>lt;sup>49</sup> NGK, 259-260 (see also 60-61). Earlier, Nier made a similar remark in a letter to Henry Faul (ref. 35).

<sup>&</sup>lt;sup>50</sup> Alfred O. Nier and Earl A. Gulbransen, "Variations in the Relative Abundance of the Carbon Isotopes," *Journal of the American Chemical Society*, 61 (1939): 697-698.

<sup>&</sup>lt;sup>51</sup> Malcolm Dole, "The Relative Atomic Weight of Oxygen in Water and in Air," *Journal of Chemical Physics*, 4 (1936): 268-275.

motivation for the study.<sup>52</sup> This potential was later recognized by chemist and Nobel laureate Harold Urey. In his 1947 Liversidge Lecture, "The Thermodynamic Properties of Isotopic Substances", Urey developed a statistical mechanical theory explaining why isotope fractionations (the term for changes in isotope ratios arising from some chemical or physical process) could be expected. Urey also suggested that these fractionations could provide useful geologic information. Nier's carbon studies provided some of the initial empirical evidence for Urey's theory. <sup>53</sup> Moreover, on the basis of these results, Urey suggested that Nier's 60° sector spectrometer could be used to measure oxygen isotope ratios, and that the latter would allow the temperature at which the samples were formed to be inferred.<sup>54</sup>

At the end of his post-doc, Nier took up an assistant professorship back at the University of Minnesota. Though the discoveries Nier made in graduate school and his post-doc were certainly important empirical contributions to isotope geology, it was upon his return to Minnesota (1938) that he made his most important *instrumental* contribution to the field. [Second Level Heading]

<sup>&</sup>lt;sup>52</sup> Nier, "Some Reminiscences of Isotopes," (ref. 43), 7-8.

<sup>&</sup>lt;sup>53</sup> Besides the work with Gulbranson, in 1941 Nier published a paper, co-authored with graduate student Byron Murphey, confirming and extending the 1939 results with the newly invented 60° sector instrument. See Byron F. Murphey and Alfred O. Nier, "Variations in the Relative Abundance of the Carbon Isotopes," *PR*, 59 (1941): 771-772.

 <sup>&</sup>lt;sup>54</sup> Harold C. Urey, "The Thermodynamic Properties of Isotopic Substances," *Journal of the Chemical Society* (1947):
 562-581, especially 576-578.

# 2.4 Mass Spectrometry for the Masses, 1938-1940

On his return to Minnesota, Nier worked on the preparative separation of <sup>13</sup>C due to the growing interest in enriched <sup>13</sup>C for use in nuclear experiments and as a tracer in biological and chemical studies. This interest increased the demand for precise isotopic analyses and hence for the construction of more spectrometers.<sup>55</sup> Nier's Harvard instrument had the requisite precision, but its magnet design was impractical for the capacity needed, both due to its heavy weight and its exceptional power requirements, and so Nier set out to develop a more economical system. In 1936, Bainbridge had published the use of a 60° sector magnet as a component of a mass spectrograph built for the precision determination of atomic masses.<sup>56</sup> Bainbridge's design yielded high resolution, which was especially important for the measurement of atomic masses, but it did not work well with electrical detection, which was an important feature for the accurate measurement of isotope ratios (*vide supra*). Nier recounted having the following insight, which also illustrates the constraints imposed on designers by the available technology:<sup>57</sup>

[block quote] Theoretically, any angle would do. If it wasn't for the fringing fields, you could have a 1-degree sector. It seems like the question is if you're going to do this, what angle should you choose? And I thought about this a little bit, and choose 60 degrees...because the 60-degree deflection worked so well in the Harvard machine of

<sup>&</sup>lt;sup>55</sup> Nier, "Some Reflections," (ref. 25), 11-12.

<sup>&</sup>lt;sup>56</sup> Kenneth T. Bainbridge and Edward B. Jordan, "Mass Spectrum Analysis. 1. The Mass Spectrograph. 2. The Existence of Isobars of Adjacent Elements," *PR*, 50 (1936): 282-296.

<sup>&</sup>lt;sup>57</sup> Peter Galison, *Image and Logic*, (Chicago: The University of Chicago Press, 1997).

Bainbridge's...When I thought about other angles, it turns out that it would have taken quite a bit more magnet for 90 degrees. If you stop to think of it, going from 60 to 90 is quite a bit more. Also, if you were going to have a flattened [metal flight] tube that you fit between the poles, it was a lot easier to bend something through 60 degrees, rather than 90. Flattening tubes isn't the easiest thing in the world...you see, if you want to use an electromagnet, you had to have a narrow air gap to exploit it. So, there wasn't room for a glass tube. <sup>58</sup> [block quote]

Such a change in angle would greatly reduce magnet size, since the latter is partially determined by the length of the ion flight path through the field. It would also reduce the magnet's power consumption. So, he combined the 60° sector feature of the Bainbridge design with the electrometer tube amplifier feature of Nier's Harvard instrument. The first prototype was completed in 1939.<sup>59</sup> The new instrument featured greatly reduced magnet and energy source size, power requirements, and cost, and of simpler design, but comparable resolution

<sup>58</sup> NGK, 84-85. See also Nier, "Some Reflections," (ref. 25), 11-12. Here it is appropriate to acknowledge the "hidden labor" that made Nier's career possible. In his recollections, Nier repeatedly acknowledges his debts to the technicians and facilities at Minnesota and Harvard (e.g., NGK, 45, 49, 88-90). For example, the precision machine work for the 60° prototype was done by a machinist, R. B. Thorness, who Nier described as "an utter genius." They would even co-author several papers in the '50s and '60s. Space does not permit going further into the social relations of the laboratories in which Nier worked.

<sup>&</sup>lt;sup>59</sup> Nier, "Manhattan Project," (ref. 42a), p. 386.

and sensitivity (Fig. 2).<sup>60</sup> For comparison, whereas the earlier magnet weighed 4000 pounds and required a large 5 kW generator, the new instrument's magnet weighed a few hundred pounds and could run on 25 W from "a couple of automobile storage batteries."<sup>61</sup> The low power requirement further dispensed with the need for a special stabilizing device.

The title of Nier's report on the instrument was "A Mass Spectrometer for Routine Isotope Abundance Measurements". Nier's inclusion of the adjective "routine" truly reveals the significance of the design because it allowed the mass spectrometer to become more accessible to non-specialists. Reflecting back on this aspect of the instrument design, Nier later commented that "if you could make these tools available to other people, who didn't necessarily have to know how a mass spectrometer worked, but could use it, they could apply it to problems in their fields. I think that this is one of the very important developments of our time."<sup>62</sup> This pay-off was crucial for the widespread adoption of mass spectrometry in geology. Indeed, the development of isotope geology as a discipline independent of physics, i.e. one that was pursued by specialists rather than physicists with an interest in geology, depended on having instruments that were simple and reliable enough to be black-boxed for use by nonspecialists. Moreover, the compact magnet made it more feasible to increase magnet size in future studies, thereby facilitating progress in resolution. At the same time, it also made miniaturization more feasible, a feature Nier would exploit in his later work on space exploration.

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 <sup>&</sup>lt;sup>60</sup> Alfred O. Nier, "A Mass Spectrometer for Routine Isotope Abundance Measurements," *RSI*, 11 (1940): 212-216.
 <sup>61</sup> NGK, 85.

<sup>&</sup>lt;sup>62</sup> NGK, 267.

#### NIER: DISCOVERER AND METHODOLOGIST

The coming of World War 2 focused Nier's research on uranium, and transformed Nier's mass spectrometers from bespoke research instruments into standardized tools used at industrial scale.

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[FIRST LEVEL HEADING]

# 3 NIER AND THE MANHATTAN PROJECT

Nier's expertise and instruments contributed to the development of the atomic bomb. Like many American physicists, Nier's defense related work began even before the United States formally entered the war.<sup>63</sup> In February 1940, Nier used a 180° analyzer instrument, similar to the one he used at Harvard, to separate <sup>235</sup>U from <sup>238</sup>U, permitting the identification of <sup>235</sup>U as the source of fission. <sup>64</sup> As part of the Manhattan Project, Nier reported to Harold Urey as part of the wartime effort to produce enriched uranium. In 1941, the Office of Scientific Research and Development commissioned Urey's uranium program to construct mass spectrometers for uranium analysis. In the following year, Nier's group built seven new 60° instruments specifically for the uranium effort. At the same time, several methods for the preparative separation of <sup>235</sup>U were being considered, and Nier's group established the

<sup>&</sup>lt;sup>63</sup> Daniel J. Kevles, *The Physicists: The History of Scientific Community in America* (Cambridge, MA: Harvard University Press, 1995), chs. 8 and 9.

<sup>&</sup>lt;sup>64</sup> NGK, 73-82.

effectiveness of the gaseous diffusion method. The famous K-25 separation plant in Oak Ridge, Tennessee was constructed to put the method into effect.

Nier built two prototype mass spectrometers for the Oak Ridge plant. One was a leak detector, a portable and extremely sensitive mass spectrometer used to ensure air was not leaking into the uranium separation process. General Electric made many hundreds based on Nier's prototype. Engineers carried them about to check the integrity of the plant's miles of pumps and diffusion barriers. The other prototype was a recording mass spectrometer that could monitor the progress of isotope separation throughout the process stream. General Electric built over 100 of these instruments to create a system for monitoring the separation process in over 50 locations throughout the enormous plant. The recording output of each instrument was transmitted to a central control room, where it could be monitored by a single person (Nier mentions engineers).<sup>65</sup>

Nier's mass spectrometers were among many scientific instruments whose significance was transformed by standardization and substantial manufacturing during World War 2. In some cases, as Yakov Rabkin has described for infrared spectrophotometers, during the same period in chemistry, an existing technique was given a standardized instrumental form and put into commercial production by government order.<sup>66</sup> In other cases, like the Pauling oxygen meter, expected military needs led to a focused effort to invent and manufacture a new class of instrument. Nier's mass spectrometers were black-boxed, mass-produced and, in the case of

<sup>&</sup>lt;sup>65</sup> NGK, 107-124; Grayson, *Measuring Mass* (ref. 5d), 16-17.

<sup>&</sup>lt;sup>66</sup> Yakov Rabkin, "Technological Innovation in Science: The Adoption of Infrared Spectrometry by Chemists" in Morris, *From Classical to Modern* (ref. 8c), 3-28.

the recording spectrometer, partially automated for the war effort. The war exposed more scientists and engineers to mass spectroscopy. As Matthew Shindell observes, mass spectrometers

[block quote] had existed before the [Second World War], but only a few laboratories had had sufficient expertise to build and maintain them. Wartime development of mass spectrometers by US industries under military contract and postwar tinkering by interested engineers, scientists, and oil companies effectively black-boxed the technology (although a trained technician was still required to operate it), increased its precision dramatically, and put it within reach of any university department willing to pay for the still relatively expensive instrument.<sup>67</sup> [block quote]

Scientists like Harold Urey witnessed the flexibility and capability of mass spectrometry, and would put that capability to use in path-breaking research after the war.

Nier's work sheds light on the question of continuity and change between pre- and post-World War II science mentioned in the introduction. Although the Instrumentation revolution in chemistry had begun before the war, the usage and availability of instruments was greatly advanced by standardization, bulk production, and the US government's role in promoting their use by different research groups. Both the leak detector and the recording spectrometer were novel instruments. Nevertheless, all the instruments built by Nier for the war were based on the 1940 sector design which was the fruit of Nier's research in the 1930s. For Nier, as an

<sup>&</sup>lt;sup>67</sup> Shindell, "From the End of the World" (ref. 4b), 109. See also Doel, "Constituting the Postwar," (ref. 1f) and "The Earth Sciences," (ref. 4c), 412, Oreskes and Doel, "The Physics and Chemistry," (ref. 1g), on 539, 552-557.

instrument inventor and research scientist, rather than a manufacturer, it was the pre-war work that truly made mass spectroscopy an accessible tool. "It was not until the late 1930's, when high vacuum and electronic techniques became an essential part of physics laboratory work, that mass spectroscopy became a tool which could serve scientists in a variety of disciplines," he wrote in 1966.<sup>68</sup>

[FIRST LEVEL HEADING]

# 4 NIER'S POSTWAR CAREER

After the war, Nier initially returned to geochronology. In 1948, Nier and his graduate student L. T. Aldrich showed that <sup>40</sup>K decays to <sup>40</sup>Ar, and suggested that this phenomenon "might become extremely useful in the measurement of geological time."<sup>69</sup> This demonstration was based on Nier's earlier discovery of <sup>40</sup>K while in graduate school (section 3.1). The K-Ar method would be developed by others and become one of the pillars of geochronology. Besides continuing to work on the K-Ar dating system, Nier's post-war work in this field largely consisted of collaborations with geologists, like the eminent geologist and pioneer of geochronology Arthur Holmes at the University of Edinburgh or Samuel Goldich at Minnesota, in which Nier's role was to build instrumentation and conduct isotopic analyses. A 1946 letter from Holmes gives a sense of the importance of Nier's work:

<sup>&</sup>lt;sup>68</sup> Alfred O. Nier, "Mass Spectroscopy—An Old Field in a New World," *American Scientist*, 54, no. 4 (1966): 359-384.
For the early use of the technique in the petroleum and chemical industries, see Carsten Reinhardt, "The
Chemistry of An Instrument: Mass Spectrometry and Structural Organic Chemistry," in Morris, *From Classical to Modern Chemistry* (ref. 8c), 229-247.

<sup>&</sup>lt;sup>69</sup> L.T. Aldrich and Alfred O. Nier, "Argon 40 in Potassium Minerals," PR, 47, no. 8 (1948): 876-877.

[block quote] Ever since your isotopic analyses of ore-leads was published I have hoped that it would be possible to calculate from the results the time that has elapsed since the Earth's primaeval lead began to be contaminated by radiogenic lead. The acquisition of a calculating machine a few months ago has now made possible the somewhat formidable calculations and I have just completed the work. The age works out at about 3000 million years by various sets of solutions, the range being about 2750 to 3150 and the average of the best set of solutions ... being 3015. We can, however, afford to neglect the odd 15.! [*sic*] This looks like being the first really reliable estimate of the age of the earth and I should like to salute your work as the means of making it possible.<sup>70</sup> [block quote]

Holmes had been working on the problem of the Earth's age since the beginning of the century. Though Holmes' 1946 estimate was supplanted a decade later by the currently accepted age of 4.56 billion years, provided by Claire Patterson and colleagues, this subsequent revision remained dependent on the techniques and data that Nier pioneered and to which Holmes gives so much credit. Moreover, Holmes is considered the father of the modern geological time scale, which assigns absolute dates to geologic time periods. His early versions of the scale relied on Nier's data.

Over time, Nier shifted to disseminating mass spectrometry across the sciences, making further design improvements, and applying the technique to atmospheric and space

<sup>&</sup>lt;sup>70</sup> Arthur Holmes to Alfred O. C. Nier, 16<sup>th</sup> February 1946, NP, Box 3, Folder 50.

exploration, as well as atomic weight determination.<sup>71</sup> For example, he returned to tracer work and published an improved design of his 1940 spectrometer that was subsequently manufactured by the Consolidated Electrodynamics Company for biochemical research. Whereas the 1940 design was aimed at light elements (C, N, O), the 1947 version could be modified to study masses up to 400.72 Importantly, this design permitted the simultaneous collection of two ion beams, which eliminated errors due to fluctuations in beam intensity. The resulting boost in precision allowed the measurement of tiny variations in isotope composition. This capability made the instrument the cornerstone of stable isotope geochemistry (5.1). The miniaturization of the spectrometer was an important contribution from this period, for it was essential for sending spectrometers into flight.<sup>73</sup> Overall, his research approach consisted in leveraging and enhancing his ability to measure isotope variations in order to study the composition of various objects, such as meteorites or the atmospheres of the Earth, Mars and Venus. Similar in this respect to Nier's early study of carbon isotope variations, these investigations continued to be of historical relevance because they provided information on the origin and mode of formation of their objects, as indicated by a report he co-authored titled the

<sup>&</sup>lt;sup>71</sup> J. R. De Laeter, "Al Nier's contribution to atomic weights," *International Journal of Mass Spectrometry*, 178 (1998): 1-7.

<sup>&</sup>lt;sup>72</sup> Alfred O. Nier, "A Mass Spectrometer for Isotope and Gas Analysis," *RSI*, 18, no. 6 (1947): 398-411. See also NGK,
86-87.

<sup>&</sup>lt;sup>73</sup> NGK, 177-215.

"Isotopic Composition of Nitrogen: Implications for the Past History of Mars' Atmosphere," which discussed results from the Viking mission.<sup>74</sup>

It is worth noting that Nier's topical shift was accompanied by a shift in the sources of funding. As determined from his publications, before the war, Nier was funded by the University of Minnesota, the Milton fund (a Harvard bequest fund), the Rockefeller Foundation and the National Research Council (NRC). After the war, his papers acknowledge the Office of Naval Research (ONR), the Atomic Energy Commission (AEC), and the Committee on Growth of the NRC, which was a funding arm of the American Cancer Society (ACS). For obvious reasons, the ONR was interested in projects relating to nuclear physics, and Nier's work on spectrometers for precision measurements of atomic masses was relevant.<sup>75</sup> The ACS funding was motivated by the use of isotopes as tracers for biological studies.<sup>76</sup> Nier's work on atmospheric and space exploration would receive funding from NASA and the NSF.<sup>77</sup>

The shift in funding may help to explain the topical shift. Nier himself attributed the shift to the intervention of the war and his involvement in the Manhattan Project, which followed on the alleged advice, mentioned above, against venturing further into geology. He also described himself as having a limited attention span. But he also seems to have been motivated by a desire to leverage his instruments, by applying them in "areas where there's interesting things to do." And he did so in a way that made him more than a mere dabbler: As his interviewer

<sup>&</sup>lt;sup>74</sup> Co-authored with Michael B. McElroy and Yuk Ling Yung. *Science*, 194 (1976): 70-72.

<sup>&</sup>lt;sup>75</sup> NGK, 142, 230.

<sup>&</sup>lt;sup>76</sup> Ibid., 156.

<sup>&</sup>lt;sup>77</sup> Ibid., 233.

Michael Grayson has observed, "[a]fter doing seminal work Nier would move on to another area."<sup>78</sup>

[FIRST LEVEL HEADING]

# 5 THE NIERIAN TRADITION IN ISOTOPE GEOLOGY

Even though Nier's own research shifted away from geochemistry, his instrument design, as well as his method of coupling a tailored mass spectrometer to measuring particular isotopes or isotope pairs, produced compelling discoveries in geochemistry during the post-war decades. When mass spectrometrist John Reynolds eulogized Nier for the National Academy of Sciences in 1998, he wrote: "one can categorize a large part of the worldwide geochronological effort as a pyramid of workers with Al at its apex. The Nier pyramid would be all encompassing with respect to geochronologists, if using magnetic sector instruments were the defining category, which is much the case." According to Reynolds, Nier's scientific descendants "frequently acknowledged their debt to the facilities Nier invented and the example he set in their use." <sup>79</sup>

<sup>&</sup>lt;sup>78</sup> NGK, 60-61, 134, 259-260, 275-276; Grayson, "Professor Al Nier," (ref. 5a), 693.

<sup>&</sup>lt;sup>79</sup> Reynolds, *Nier* (ref. 5b), p. 10. The geochemist Harmon Craig made a similar point with regard to Nier's 1947 spectrometer design, on the occasion of presenting Nier with the Geochemical Society's V. M. Goldschmidt Award in 1984: "...the 1947 R.S.I. paper alone would justify the award we are presenting to Professor Nier today ... It would be very nice to have a cladistics diagram of the line of descent from the Nier Machine: we would see the "Reynolds Machine" and the "Clarke Machine", both worthy and important additions to the genus, followed by any number of commercial brands." *Geochimica et Cosmochimica Acta*, 49 no. 7, (1984): 1661-1665.

They also learned how to take the ancient Earth's temperature, gave the planet a new birthdate, found a traumatic period in the Moon's past, and provided key evidence for a mechanism that could cause continental drift, among other applications.

This process was mediated by connections Nier had made through the Manhattan Project. There, Nier came to know one Mark Inghram, a graduate student at the University of Chicago who had studied under Arthur Dempster. Inghram was sent to Minnesota to learn as much as possible from Nier about the instrument and the methods of analysis. Inghram was then sent on to Columbia with two of Nier's instruments to work on uranium enrichment.<sup>80</sup> After the war, Inghram became a physics instructor at the University of Chicago. There Inghram was able to work with some of Nier's 60° sector instruments, which were managed by the Argonne National Laboratory under the auspices of the university.<sup>81</sup> After the war, the university had founded the Institute for Nuclear Studies (INS), which was led by Manhattan Project alumni Harold Urey, Harrison Brown, Arthur Compton and Willard Libby, and attracted a number of students who would become prominent figures in isotope geochemistry. During the late 1940s and throughout the 1950s, Inghram supervised or worked with a number of these students, including Clair Patterson, Gerald Wasserburg, John Reynolds, George Wetherill, George Tilton, and others. According to Reynolds, "Ingram's [sic] skills in having good mass spectrometers were a real important—and perhaps unique—aspect for Chicago's being ... a

<sup>&</sup>lt;sup>80</sup> On these moves of Inghram and the instruments, see NGK, 98 and Reynolds, *Nier*, (ref. 5b), 8 and 10.

<sup>&</sup>lt;sup>81</sup> NGK, 126 and Albert Parr and Roger Stockbauer, "Mark G. Inghram," in *Encylopedia Part B* (ref. 7i), 124-125.

good platform [for geochronology]."<sup>82</sup> These students would go on to set up their own labs and train further generations of students. Their contributions were made possible by a deep knowledge of mass spectrometry, combined with a strong interest in applying it to geological problems. In the next four subsections, I will briefly describe some of the contributions of the INS scientists, focusing on the personal and scientific connections to Nier and his work that made those contributions possible.

## [Second Level Heading]

# 5.1 Harold Urey invents a clam thermometer

Urey's pioneering work in paleoclimatology was indebted to Nier.<sup>83</sup> As previously noted, Urey claimed that past temperatures could be inferred from oxygen isotope ratios in 1947. He also suggested that a Nier-type spectrometer could provide the requisite data. In the late 1940s, however, the commercial production of a Nier-type spectrometer was still in development, so Urey decided to build his own. To this end, Urey recruited two young scientists with experience in mass spectroscopy. One was an electrical engineer, Charles R. McKinney, who had maintained Nier's spectrometers at Oak Ridge during the war. Another was Samuel Epstein, a Canadian post-doctoral fellow who had worked as a graduate student with the Canadian mass spectroscopist Harry Thode. By February 1949, Urey's group had constructed two mass spectrometers based on Nier's 1947 design. Urey, who had supervised Nier on the

<sup>&</sup>lt;sup>82</sup> John H. Reynolds, interview by William Glen. Berkeley, California. January 30, 1978. Bancroft Library, University of California, Berkeley, on 13.

<sup>&</sup>lt;sup>83</sup> For a comprehensive study of Urey's career, see Shindell, Harold C. Urey, (ref. 1e).

Manhattan Project, was in communication with him during this time.<sup>84</sup> Their work with these instruments resulted in a series of foundational papers in paleoclimatology. Paleontologists, marine biologists and curators sent them shells from animals that grew in isotopically stable marine environments around the world. Running these calcium carbonate samples through the mass spectrometers, they matched isotopic ratios to known water temperatures. Then they analyzed the fossilized shells of belemnites, similar to present-day squids, that lived in the Jurassic about 150 million years ago. Concentric layers in the shells revealed regularly alternating temperatures, showing that there were summers and winters at the time. Most remarkably, they showed that one particular specimen lived for about four years, through a succession of summers and winters, experienced a gradual decline in the ambient temperature, and died in the spring. These results showed that given the right sources, mass spectrometry and isotopes could answer extremely fine-grained questions about the deep past, besides age.<sup>85</sup> This work required further improving the stability and sensitivity of Nier's doublecollection spectrometer for the measurement of the very small isotope effects involved in fractionation: "the greatest mass spectroscopist in the world [Nier] had just built a new

<sup>&</sup>lt;sup>84</sup> Ibid., 131-136.

<sup>&</sup>lt;sup>85</sup> H. C. Urey et al., "Measurement of Paleotemperatures and Temperatures

of the Upper Cretaceous of England, Denmark, and the Southeastern United States," *Bulletin of the Geological Society of America*, 62 (1951): 399-416.

spectrometer and it was not good enough," Epstein later recalled .<sup>86</sup> One of the first papers in the series was devoted to describing these improvements.<sup>87</sup>

[Second Level Heading]

## 5.2 Clair Patterson and Harrison Brown Age the Earth

Clair Patterson's first experience with mass spectrometry was on the Manhattan Project, working on uranium separation at Oak Ridge, though in a different plant than Nier. After the war, Patterson undertook a Ph.D. at the University of Chicago, finishing his degree in 1951. His advisor there was the nuclear chemist Harrison Brown, who had published a design for a mass spectrometer in 1941 that cited Nier's 1940 design.<sup>88</sup> Brown had also worked at Oak Ridge, on the separation of plutonium in the same plant as Nier.<sup>89</sup> Patterson's Ph.D. work focused on determining the lead isotopic composition of zircons, a kind of crystal, found in granite, that is free of non-radiogenic lead. He collaborated with fellow Brown graduate student George Tilton, who was charged with determining the uranium contents of zircons. According to an interview Patterson gave in 1995, the amounts of lead in zircons were 1,000 times smaller

<sup>87</sup> C. R. McKinney et al., "Improvements in Mass Spectrometers for the Measurement of Small Differences

<sup>&</sup>lt;sup>86</sup> Samuel Epstein, interview by Carol Bugé. Pasadena, California, December 19 and 26, 1985, and January 10, 1986.Oral History Project, California Institute of Technology Archives, on 22-23.

in Isotope Abundance Ratios," RSI, 21, no. 8 (1950): 724-730.

<sup>&</sup>lt;sup>88</sup> It is worth noting that Nier's designs and geochronological results, discussed in sections 2-4, are routinely cited in the work discussed in section 5.

<sup>&</sup>lt;sup>89</sup> I am not aware of any contact between them there.

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than anything that had been measured before.<sup>90</sup> The attempt to meet this sensitivity requirement revealed to Patterson that his samples were being contaminated by exogenous lead, a discovery that forced him to develop new methods for avoiding such contamination, notably the use of a rigorously decontaminated "clean lab," which was a novelty at the time.<sup>91</sup>

He put these techniques to good use in his post-doc, also with Brown, when he determined the age of the Earth based on the comparison of lead isotope ratios in meteorites and terrestrial samples. In the 1940s, Arthur Holmes, Erich Gerling of the Radium Institute of the Academy of Sciences of the USSR, and F. G. Houtermans at the University of Göttingen had independently estimated the age of the Earth based on data Nier had published in his pre-war research.<sup>92</sup> All three arrived at an age of about 3 billion years. For his own work, Patterson depended on a mass spectrometer developed by Inghram and colleagues at Argonne National Laboratory, a modified version of Nier's 60° sector design.<sup>93</sup> These techniques together were essential for allowing Patterson to settle the question of the Earth's age. Inghram was co-

<sup>&</sup>lt;sup>90</sup> Clair C. Patterson, interview by Shirley K. Cohen. Pasadena, California, March 5, 6 and 9, 1995. Oral History Project, California Institute of Technology Archives, on 14 and 17.

<sup>&</sup>lt;sup>91</sup> Ibid., 19. George R. Tilton, Claire Patterson, Harrison Brown, Mark Inghram, et al., "Isotopic Composition and Distribution of Lead, Uranium, and Thorium in a Precambrian Granite," *Bulletin of the Geological Society of America*, 66 (1955): 1131-1148 provide a detailed description of the procedures used.

<sup>&</sup>lt;sup>92</sup> G. Brent Dalrymple, "The Age of the Earth in the Twentieth Century: A Problem (Mostly) Solved," in Lewis and Knell (ref. 31a), 205-222, on 211-212.

<sup>&</sup>lt;sup>93</sup> Patterson, Cohen interview (ref. 89), 21; Tilton et al., "Isotopic Composition," (ref. 90), 1137-1138.

author on the initial communication of the calculation.<sup>94</sup> Brown and Patterson also planned their approach to calculating the age of the Earth around Nier's discovery that common lead is a mixture of primordial and radiogenic lead, recalled Patterson in 1995: "Now, there's a bunch of equations that these atomic physicists—Al [Alfred] Nier, for example—calculated. It's so marvelous how they worked all this stuff out. And if we only knew what the isotopic composition of primordial lead was in the earth at the time it was formed, we could take that number and stick it into this marvelous equation we had. And you could turn the crank and, *blip*, out would come the age of the earth."<sup>95</sup>

[Second Level Heading]

# 5.3 John Reynolds and Plate Tectonics

The Nier tradition also made an important, albeit indirect, contribution to the plate tectonics revolution. John Reynolds was a student of Inghram's who accepted an assistant professorship in the physics department at UC Berkeley in 1950. He began his career there by improving the technique of potassium-argon dating. Reynolds built an all-glass, 60°-sector spectrometer that could be iteratively baked at high temperature in an oven to release contaminants, and then pumped down. Previous designs did not permit placement in an oven, thus precluding comprehensive baking. This procedure allowed extremely minute amounts of radiogenic argon to be measured very accurately, an essential requirement for dating rocks younger than 10 million years. The Reynolds spectrometer was then used by Garniss Curtis and

<sup>&</sup>lt;sup>94</sup> (a) C. Patterson, G. Tilton and M. Inghram, "Age of the Earth," *Science*, 121, no. 3134 (1955): 69-75. (b) Claire Patterson, "Age of Meteorites and the Earth," *Geochimica et Cosmochimica Acta*, 10, (1956), 230-237.
<sup>95</sup> Patterson, Cohen interview (ref. 89), 16.

Jack Evernden, of the Berkeley geology department, in collaboration with Reynolds' graduate student Joseph Lipson, for the dating of young volcanic rocks. Their data, together with those of other researchers, would allow changes in the polarities of volcanic rocks to be dated. The reversals of polarity would provide key evidence for the reality of sea-floor spreading. Isotope measurements using the Reynolds spectrometer were crucial for establishing the chronology of the sequence of reversals. By 1966, this chronology allowed for the precise identification and correlation of the patterns of remnant magnetism in the ocean floors, allowing for the documentation of the movements of oceanic crust outward from mid-oceanic rifts and ridges. Starting in 1970, the dating of samples, independently of their magnetism, from deep cores showed that oceanic crust is older the farther it is from the mid-ocean rift and ridge yet none of it dates from before the Mesozoic era.<sup>96</sup>

[Second Level Heading]

## 5.4 Gerald Wasserburg and the Lunatic

Gerald Wasserburg may serve as a final example of the kind of research pattern Nier pioneered. Wasserburg earned a PhD in geology in 1954 at Chicago under the joint supervision of Inghram and Urey.<sup>97</sup> He then went on to champion geochronology at Caltech. In the late 1960s, he and his colleagues invented a new 60° sector mass spectrometer, the "Lunatic I", partially in anticipation of the analysis of lunar samples. This instrument was fully digital, which

<sup>&</sup>lt;sup>96</sup> The role of Reynolds and his spectrometer in confirming the theory of plate tectonics is described in detail in William Glen, *The Road to Jamarillo*, (Stanford: Stanford University Press, 1982).

<sup>&</sup>lt;sup>97</sup> G. J. Wasserburg, "Isotopic Adventures—Geological, Planetological, and Cosmic," *Annual Review of Earth and Planetary Science*, 31 (2003): 1-74.

allowed the magnetic field to be changed rapidly via computer programming. Doing so greatly reduced the error induced by ion beam instability. In his 1986 award speech for the Crafoord Prize, a Nobel-equivalent awarded by the Royal Swedish Academy in areas left out of the standard Nobel categories, Wasserburg claimed that "[m]y own ability to carry forward the new instrumental design and development are a direct consequence of the training I received in the laboratory of M. G. Inghram III."98 This technique then permitted measurement of extremely small isotopic differences on very small samples of many elements. Wasserburg applied this capability inter alia to the study of solar system history. For example, the Lunatic I was used to determine the ages of lunar rocks from the Apollo missions and the Soviet Luna 16 and 20 missions. These studies established the time scales for lunar evolution. One remarkable discovery, inferred from a combination of U/Pb and Rb/Sr measurements, was of evidence for a so-called "terminal lunar cataclysm," a large-scale bombardment of the Earth-Moon system by small planetary bodies that might have occurred 500 million years after the formation of that system due to disturbances in the asteroidal belt.<sup>99</sup> In his 2003 memoir, Wasserburg recalled that, when he was looking for an assistant professorship in the mid-1950s, Inghram connected him with Nier, who then championed his candidacy at Minnesota.<sup>100</sup> His first paper, on <sup>40</sup>K/<sup>40</sup>Ar

<sup>&</sup>lt;sup>98</sup> G. J. Wasserburg, "Isotopic Abundances: Inferences on Solar System and Planetary Evolution," *Earth and Planetary Science Letters*, 86, (1987): 129-173, on 136.

<sup>&</sup>lt;sup>99</sup> Fouad Tera, D. A. Papanastassiou, and G. J. Wasserburg, "Isotopic Evidence for a Terminal Lunar Cataclysm," *Earth and Planetary Science Letters* 22 (1974), 1-21.

<sup>&</sup>lt;sup>100</sup> Wasserburg, "Isotopic Adventures," (ref. 96), on 10. The candidacy was ultimately unsuccessful.

dating , was based on dates by Nier.<sup>101</sup> In a personal communication to me, R. Lawrence Edwards, a student of Wasserburg's in the 1980s and himself an expert on the application of Lunatic I-type systems to climate history, corroborated Reynolds' pyramid metaphor and emphasized Inghram's role in disseminating knowledge of how to use Nier-type spectrometers among the geochronologists at Chicago.

Though I have focused on Nier's influence via scientists based at the University of Chicago, other threads could be pursued, such as the disseminating role of L. T. Aldrich, George Tilton and George Wetherill at the Carnegie Institution, mentioned by John Reynolds.<sup>102</sup> That said, enough has been said to show that the post-war period was a time of exciting advances in geochronology and geochemistry, and that Nier's descendants played a major role in making them. Their research displays a common pattern, the adaptation and extension of the capabilities of mass spectrometry to make new kinds of isotopic measurements in order to answer questions of geological structure and history. Beyond the common approach, moreover, there was a good deal of personal and instrumental continuity, involving the transmission of instrumental expertise via individuals like Inghram or the Manhattan Project experience, and of instrumental design in the form of the 60° sector template. The testimony I have cited from Reynolds, Epstein, Patterson, Wasserburg and Edwards, though admittedly retrospective, suggests that the Nier "pyramid" was the result of conscious admiration and emulation. For

<sup>&</sup>lt;sup>101</sup> G. J. Wasserburg, R. J. Hayden, "The Branching Ratio of K<sup>40</sup>," *PR*, (1954): 645.

<sup>&</sup>lt;sup>102</sup> Reynolds, *Nier*, (ref. 5b), 10.

these reasons, I think it is reasonable to speak of a Nierian "tradition," as opposed to weaker forms of dependence, like mere use of his data or instrument designs.

[FIRST LEVEL HEADING]

### 6 CONCLUSION: A CO-EVOLUTION OF SCIENCE AND TECHNOLOGY INSTRUMENTATION

In the decades since Nier's pioneering work, geochemists have developed numerous specialized forms of mass spectrometry tailored to particular isotopic pairs. This led to a corresponding expansion of the questions that could be answered empirically, of which those concerning early solar system history, climate change or plate tectonics are but a few particularly spectacular examples.

While of course Nier himself only initiated these developments, his work established the ways in which those two rich streams, mass spectrometry and isotope science, could be brought together and co-developed to both pose and answer geochemical questions. He was able to engage in this interplay because he was uniquely positioned to combine multiple lines of research and kinds of knowledge: atomic and nuclear physics; radiometric dating; chemical analysis (though he did not perform the analyses himself); mass spectroscopy, and spectrometry in particular; electrical engineering; and vacuum technology. In this sense he illustrates a noted pattern of scientific innovation, creatively integrating ideas and practices from multiple fields.<sup>103</sup> But this integration presupposed that particular lines of research and kinds of knowledge were developed sufficiently so that someone at "the right place at the right

<sup>&</sup>lt;sup>103</sup> Oren Harman and Michael R. Dietrich, *Dreamers, Visionaries, and Revolutionaries*,

<sup>(</sup>Chicago: The University of Chicago Press, 2018), 9-10.

time" could combine them effectively, something Nier keenly recognized about his work.<sup>104</sup> Indeed, instrument development tends to be holistic: instruments embody knowledge of different kinds—such as theoretical or practical knowledge (e.g., glass-blowing skills) produced by different practices and fields, like theoretical physics and electrical engineering. <sup>105</sup> It is rare for one person to master all the different kinds of knowledge required to construct a particular instrument. Nier's unusual skill set corresponded well to the holistic requirements of instrument development. Moreover, he grasped the geochronological problem sufficiently well to use the skills to make fundamental discoveries in that field. Finally, the basic design principles of mass spectroscopy (2.1), which Nier inherited, proved versatile enough to allow an extended series of variants to be derived from it, a crucial feature for meeting geochemical needs.<sup>106</sup>

In order to better characterize Nier as a figure in the history of science, it may be helpful to contrast it with a few others in the literature. Nier was not just a gifted experimentalist, like the particle physicist Patrick Blackett of the same period, for he specialized in instrument design.<sup>107</sup> But nor was he merely a gifted instrumentalist who leveraged his instruments to make discoveries, like Francis Aston or Arthur Dempster, for he was also interested in transferring knowledge from physics to other fields, like the method-makers Carsten Reinhardt has

<sup>&</sup>lt;sup>104</sup> NGK, 18.

<sup>&</sup>lt;sup>105</sup> George Borg, "Discovery and instrumentation: how surplus knowledge contributes to progress in Science," *Perspectives on Science*, 27 no. 6, (2019): 861-890.

<sup>&</sup>lt;sup>106</sup> In this sense, it provided a particularly fruitful *scaffold*, a concept I discuss in the works cited in footnote 109.

<sup>&</sup>lt;sup>107</sup> Mary Jo Nye, *Blackett* (Cambridge, MA: Harvard University Press, 2004).

described.<sup>108</sup> To some extent, his career trajectory resembles that of contemporaries like the geophysicist Victor Vacquier, the inventor of the fluxgate magnetometer, or physicist Arthur Hardy, inventor of the first automated spectrophotomer, who both exploited the flourishing of electronics in the early 20<sup>th</sup> century to develop scientific instruments.<sup>109</sup> In keeping with one of Hasok Chang's key examples of a system of practice, Nier had, in a much smaller way, an effect akin to Lavoisier, insofar as the latter showed how a kind of instrument, the balance, could be optimized to carry out a kind of challenging measurement, of the mass balance in chemical reactions, that could provide the empirical basis for a research program.<sup>110</sup> Similarly, isotope ratios provided "the almost vacant ecological niche waiting for the right machine to come along and fill it."<sup>111</sup> The comparison is all the more apt insofar as Nier's methods helped displace gravimetric, wet chemical ones from their central place in the geochemist's toolkit.

Nier's early career also indicates, by their absence, what factors are needed for the synergistic coevolution of science and technology to proceed on a social scale, namely the institutional, technical and financial conditions for the widespread use of high-tech scientific

<sup>&</sup>lt;sup>108</sup> Ref. 11.

<sup>&</sup>lt;sup>109</sup> (a) Elizabeth N. Shor, "Victor Vacquier (1907-2009)," *Eos*, 91 no. 30 (2010), 264. (b) Jon Eklund and Peter Morris, "Spectrophotometer," in *Instruments of Science*, ed. Robert Bud and Deborah Jean Warner (London: The Science Museum), 558-561.

 <sup>&</sup>lt;sup>110</sup> (a) Chang, (ref. 6). See also (b) Trevor H. Levere, "Lavoisier: Language, Instruments, and the Chemical Revolution," in *Nature, Experiment, and the Sciences,* ed. Trevor H. Levere and William R. Shea (Dordrecht: Kluwer Academic Publishers, 1990), 207-223.

<sup>&</sup>lt;sup>111</sup> Craig, *Introduction* (ref. 80).

instruments by non-specialists. Certainly, these missing factors would emerge during and after the war. That said, his designs, results, and the dissemination of his know-how via students and colleagues, furnished important elements for progress on that scale.

[FIRST LEVEL HEADING]

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