# **Professor Al Nier and His Influence** on Mass Spectrometry

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Professor Alfred Otto Carl Nier has had a singular influence on the art and science of mass spectrometry over the last 55 years. I had the pleasure of conducting an oral history interview of Dr. Nier in the spring of 1989 at his office and laboratory at the University of Minnesota. I was assisted in this endeavor by Thomas Krick of the Biochemistry Department staff. The interview was underwritten by the American Society for Mass Spectrometry. The following highlights are excerpted from 10  $\frac{1}{2}$  hours of material recorded during the interview. The complete set of tapes, a full transcript, pictures, and other material collected during the interview are archived with the Beckman Center for the History of Chemistry in Philadelphia. (*J Am Soc Mass Spectrom* 1992, 3, 685–694)

## From Wireless to Mass Spectrometry

A s a high-school student, Nier pursued a college preparatory curriculum. His immigrant parents directed him along these lines, and his natural aptitude for mathematics and science, as well as a hobbyist's interest in the newly emerging technology of radio, whetted his appetite for the electrical engineering (EE) curriculum at the University of Minnesota.

I was always steered in that direction (by my parents). In high school, that was the time when radio was a coming thing. Keep in mind, this was the middle 1920s and people built their own radio sets, and I had friends who did this sort of thing. You could buy parts and tubes and crystals...so I got into that.... By the time I graduated from high school in 1927, it was kind of understood that I would go into electrical engineering, which I did, and I came here to the University.

As an undergraduate in the electrical engineering department, Nier took a number of courses in the physics department. His outstanding performance rapidly brought him to the attention of one of his undergraduate professors, Dr. Henry Erikson, who taught the beginning physics course. I was in a class of probably 200 students or so. For these big classes we had numbered seats in a big auditorium, and they had somebody going around five minutes after the class started, seeing which seats were vacant. In those days attendance records were kept so the instructor knew where everybody sat. Well, after the third quiz, just before the class, this very dignified professor came up the middle of the room—I sat a few seats from the aisle—and said he'd like to see me. He didn't ask what my name was or anything else, he just came and said he'd like to see me. He asked me at my convenience to come to his office. I didn't know what this was all about, and the people around me began to buzz. They wondered if I'd been caught cheating or something like that. But, anyhow, I went to his office, and made sure he was talking to the right person. What had happened was I had gotten 100's on the first three tests in the physics course, so he raised the question, Had I considered becoming a physicist? I said, Well, no, I really hadn't. But he said I really ought to consider it, and what's more, he gave me a job, working as an assistant, doing some experiments. So this was my real introduction to physics as such.

Despite the interest of his physics professors and laboratory work with Professor Erikson and later with Professor Valasek, Nier completed the undergraduate EE curriculum and graduated with a bachelor's degree in EE. Professor Erikson offered him a teaching assistantship, which Nier turned down as he thought he would prefer to work for a while before beginning graduate studies. At the time, 1931, jobs were hard to find. Nier estimates that only 5 of the 80 graduates

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were offered jobs, and Nier was not among them. He was left without a job or support. Professor Henry Hartig of the electrical engineering department learned of his plight, and Nier took a teaching assistantship in EE while working on a Master's degree. Of the graduate EE curriculum, Nier says:

They had so few (graduate) courses available in engineering departments in those days that in electrical engineering, one took courses in physics and math. So, except for having taken one course in electrical engineering in circuit theory or something like that, I took standard physics courses. It was just as if I'd stayed in physics as far as my course work was concerned and that's mainly what you did the first two years anyhow. But I did...gain valuable experience in instrumentation.

Upon graduation with his master's degree, Erikson again offered Nier a teaching assistantship in the physics department, which he accepted, and he continued his graduate education in physics. John Tate was the advisor of most of the experimental graduate students at the time, but because of the heavy demands on his time as a leader in the physics community he spent little time directing their work. Nier recalls:

In particular, he didn't spend much time with you unless you were getting results. It was really a rugged existence for some people. I was batting around here, trying to work on something related to electrical discharges in gases that he wasn't particularly interested in, and he would come and see me once in a while; he'd look at the glow discharge and say "So, what're you gonna do with that?" I wasn't quite sure what I was going to do with it; then finally, one time he came down, and said "Gee, it sounds a little like you're working on something that General Electric did years ago and never bothered to publish."

Eventually Tate offered Nier some assistance by suggesting that he work for a newly hired postdoctoral appointee, John Williams, who was supposed to continue the study of electron impact phenomena that Tate had an interest in and the department had developed a reputation for. Tate and Williams were collaborating on the construction of a mass spectrometer for the work at a time when rapid advances in nuclear physics began to occur.

Well, Williams barely got here and nuclear physics began to break forth, and people were able to do nuclear disintegrations with relatively low voltages, a few hundred kilovolts. You could perform reactions on the light elements like lithium and boron. We had a 300,000 V x-ray set here in the department, so Williams was encouraged to go into nuclear physics and, of course, he was anxious to do that anyhow. He dropped out of mass spectrometry entirely, and I was left alone—all on my own. Well, in the meantime, I'd built an instrument taking advantage of all of the work of my predecessors.

At the time, Nier probably had the most advanced instrument in the world. He had Walker Bleakney's improved ion source, the granddaddy of the modern electron impact ion source; P. T. Smith's compact improved analyzer layout, an outstanding improvement over those of the earlier instruments; and his own thorough knowledge of circuit design and current awareness of the latest developments in electronics. Add to that a strict tradition of adhering to the highest standards of vacuum technology, and it is unlikely that there were any instruments its equal.

I had a larger magnetic field and a larger radius than had been used before. A 5-kW generator was used to power the solenoid magnet. I could get resolution up to 100 or so. I worked with the cadmium isotopes for instance, and so, to work at mass 78 (benzene) wasn't all that hard. At that moment, I had the highest resolution mass spectrometer in existence. There was nothing going on in Europe along this line, and only a little work was being done in America. Princeton and Chicago were doing some work. But nobody had as good an instrument as I had with the solenoid that I'd inherited when Williams moved into nuclear physics. It was a real break.

Nier's first publication [1] dealt with stabilizing the trajectories of the ions in the mass spectrometer by sensing fluctuations in the magnetic field caused by voltage output variations of the generator powering the magnet. The fluctuations were used to change the ion accelerating voltages in such a way that ion trajectories were not affected. As a result, high-quality spectra were obtained. Initially, the amplifier for the mass spectrometer was a quadrant electrometer, a device that made the acquisition of a mass spectrum a very tedious, prolonged exercise. As soon as electrometer tubes were available, Nier was among the first to put the new technology to work amplifying the miniscule signals from the Faraday cup. Still, the recording of the spectrum was a laborious undertaking as the operator had plenty to do (Figure 1). After selecting the magnetic field strength and getting it stabilized, the operator used a "put-and-take" box of resistors to vary the accelerating voltage, thus bringing different ions to register at the detector. After a coarse scan of the whole spectrum, Nier would return to the region where there were intense signals and very finely step through

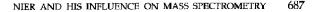




Figure 1. A. O. Nier at control panel of Minnesota  $180^{\circ}$  and  $60^{\circ}$  mass spectrometers (ca. 1940).

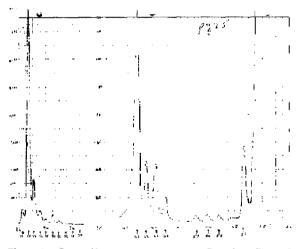
these regions with the put-and-take box. The electrometer signal was fed to a light beam galvanometer and the deflections, along with any attenuating factors, were manually recorded (Figure 2). Subsequent processing of these data resulted in numeric values of ion current, which were plotted by hand (Figure 3).

Nier did some work recording the spectrum of benzene but put aside the study of organic compounds for the same pragmatic reasons that dictate the work of graduate students today.

I thought: "But look, my advisor isn't interested in this (benzene) anymore, so I better get into something to do with nuclear physics. If I can do benzene, I could do such things as argon." <sup>38</sup>Ar had just been discovered spectroscopically by some people in Holland, if I remember correctly, but the exact amount of <sup>38</sup>Ar wasn't known. The 36 isotope had been known before that. Aston may have even seen the 36 along with 40, the most abundant isotope. Sure enough, when we introduced argon, there were three isotopes, without any question. So I made the first measurements on <sup>38</sup>Ar that showed the five-to-one ratio from 36 to 38; I did that in a half an hour one afternoon.

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**Figure 2.** Copy of portion of original data recorded in 1935 of a mass spectrum of benzene. The last column is the intensity after correction for galvanometer attenuation factors.



**Figure 3.** Copy of benzene mass spectrum. Data from Figure 2 is shown as a group of rightmost peaks in the spectrum.

Shortly after this work Nier discovered the potassium-40 isotope and rapidly moved from the bottom to the top of Tate's list. This jump, however, did not occur without some tense moments. Tate was the editor of *The Physical Review* and as such was in the process of publishing the potassium work that Nier had submitted as a letter to the editor [2]. Just as the work went to press, Tate received a manuscript for publication from Keith Brewer, then at the Department of Agriculture. Brewer was a pretty good physicist and he was working on isotope abundances in the alkali metals using thermal ionization. By heating the salt of the element you were interested in, you got ions out. Brewer also was working on potassium, which I didn't know at the time. He had a nice instrument, and with thermal ionization you didn't have the impurity problems—at least of the kind we might have had. While my letter to the editor was in press, Brewer sent in a manuscript to *The Physical Review* in which he stated that potassium 40 could not exist with an abundance even a tenth as much as I said was there!

This put Tate in a devil of a fix. In intense correspondence (Tate was on the East Coast at the time), he guizzed me. Did I do this? Did I do that? Am I sure on that? I finally convinced Tate that what I had done was all right. So he wrote back to Brewer and told him of my work and that this was being published and wouldn't Brewer like to do his work over again to check. Whereupon Brewer did it over again, found the isotope, and measured an abundance of 1 in 8,300 for the <sup>40</sup>K isotope. My abundance figure was 1 in 8,600. Well, I only claimed an accuracy of 10%, so we agreed. What had Brewer done wrong? He had nice flat-topped peaks. If you're looking for a little bitty peak in a valley between two big peaks, between 39 and 41, nice flattopped peaks raise the level in the valley; and he didn't see as sharp a bump in there as I saw.

#### Harvard

Nier's reputation was established, and with Tate's connections in the physics establishment, it was not surprising that, upon graduation, Nier was awarded a National Research Council Fellowhip in physics; one of two awarded that year. Bleakney, a former graduate of Minnesota, encouraged Nier to join him at Princeton for his postdoctoral studies. However, Bainbridge was doing precise mass determination of isotopes at Harvard and had recently enticed Jordan to join him there. Bainbridge thought that Nier's precise isotopic abundance determinations would nicely complement Harvard's work. When Bainbridge sweetened the offer with the promise of \$5000 in laboratory funds and shop support, as well as assurance he could work on anything he wished, Nier couldn't resist.

In the summer before starting his Harvard work, Nier worked at General Electric's laboratory in Schenectady, where he met Dushman and Langmuir. He also was invited up to Bainbridge's summer home in New Hampshire to go over the preliminary plans for the instrument Nier would build that fall and to order long lead-time items. The instrument was to surpass the one that Nier had built at Minnesota in mass range and resolution. In addition, it would have the flight tube in an air gap between pole faces, rather than immersed in a solenoid. At Harvard, Nier found a "first class operation"; all the way from the machine shop to the specification of only the finest materials for the instrument. Square copper tubing was ordered for the cooling coils, and liquid nitrogen, a luxury back in Minnesota, was used instead of dry ice for the vacuum cold traps.

By December 1936, Nier had the instrument running and had recorded the spectrum of mercury everpresent in mercury diffusion pumped vacuum systems. Although the isotopes of mercury were known, the instrument had the mass range and resolution to obtain accurate isotopic abundance information for the first time [3]. He went on to study the abundance of rare gas isotopes, but found his most interesting work related to problems in geochronology, an area of intense study at Harvard. This area of research grew out of the knowledge that radioactive decay of uranium and thorium ended with lead [4]. Wet chemical methods were being used to measure the atomic weight of lead from different sources to determine the common lead content relative to the radiogenic lead content. Clearly when word got around Harvard that an instrument existed that could measure the isotopic abundance of these elements to a high degree of accuracy in a fraction of the time that it took to measure the atomic weight of a sample by wet chemical methods, geochronologists took note. As Nier wryly notes, "Suddenly I had a lot of new friends!"

There were, however, some skeptics among those friends; particularly when Nier's results flew in the face of data derived from atomic weight determinations by wet chemical methods. For a while, Nier was performing analyses of lead samples "as if I were a freshman in analytical chemistry" for Professor Baxter. Eventually, Nier's data won out, and Baxter commented that he was "glad he was nearing retirement." Baxter, an analytical chemist of eminence, became fascinated in the mass spectrometric work and prepared many samples for Nier to help him in his work. As Nier remarked years later, "As a postdoc, I acquired a full professor at Harvard as a lab assistant!" It was unbelievably good luck.

As acceptance of this new technology as a geological dating tool grew, it became apparent that a measurement of the isotopes of uranium would be necessary. Nier went back to Aston's work and found that he had worked with uranium hexafluoride to get the metal into a volatile form. Even in the late 1930s, this was not an everyday common chemical, so Professor Lane, a retired geologist from Tufts College, arranged for a \$500 grant from the Geological Society of America to be awarded to whomever would synthesize a small amount. No one came forward to accept the challenge, and out of frustration, Nier went to the chemical literature in hope of attempting the synthesis of possible substitutes himself. Baxter found out about this and decided to step in. One morning, Nier found that Baxter had left several quartz tubes of uranium tetrabromide and uranium tetrafluoride on his desk. These compounds were volatile at the oven temperatures that Nier used to introduce compounds and fortunately gave rise to a strong set of peaks at the mass of the metal ion, thereby making the isotopic abundance determinations very easy. Fundamental data regarding the isotopes and their abundances were obtained from this work [5, 6]. Nier's results were within 1% of modern values; he admits "the uranium measurements were pretty ragged, but I took a lot of readings and averaged them."

Although this effort comprised the bulk of his work while at Harvard, Nier also got involved in the study of carbon isotopes in collaboration with Earl Gulbranson, then at Tufts University. He was interested in studying the variations in the carbon isotopes from a variety of natural and biological sources: limestone, shell, bone, etc. Nier recalls collecting  $CO_2$  samples from the air "driving with Gulbranson down a side road outside of Cambridge one dark night with a funnel sticking out [of the car] to bubble the air through a solution of calcium hydroxide." The work resulted in a publication [7], but owing to the press of other research, Nier wasn't able to "follow up on the work as I should have."

At the end of his postdoctoral appointment, Nier had several choices: stay on at Harvard with hope for tenure, return to Minnesota, or go to work for Condon, director for research at Westinghouse. Many factors entered into this choice, but the deciding one related to family. Both Nier and his wife were sole surviving children, and both of their parents were advanced in age. The obligation to be close to parents brought Nier back to the University of Minnesota in 1938, although with events looming on the international horizon it wouldn't be for long.

#### Back to Minnesota

Bainbridge was supportive of Nier's decision and offered to let him take his spectrometer tubes back with him to Minnesota. Tate had become dean of the College of Liberal Arts at Minnesota and was able to provide Nier with funding for a new mass spectrometer. So once again, Nier started out building a new instrument. One of the drawbacks at Minnesota, however, was the teaching load. He had two large sections of physics courses along with quizzes and grading. Another drawback was that much of the dirty work of building an instrument, which was done for Nier at Harvard, had to be done by Nier at Minnesota. Thus, the coils for the magnet had to be wound by hand and coated with a tarlike material to conduct heat to the now round water cooling tubes. Despite these problems and the need to redesign and build many of the electronic circuits for the instrument, Nier was able to

get a spectrum from the new machine shortly after Christmas, just in time to get an abstract dealing with the isotopes of iron and nickel [8] in for the American Physical Society meeting in the spring of 1939. It is somewhat comforting to know that even a scientist of Nier's stature was subjected to the nightmare of being placed as the last paper on the last day of the meeting. Beside being the last paper, it was placed in an inappropriate session. "So when my paper was to be presented, everybody walked out except for the session chairman, my wife, and one other individual while I talked about the isotopes of iron and nickel."

Nevertheless, it was at this meeting, through John Dunning at Columbia, that Nier met Fermi. As noted earlier, nuclear physics was progressing rapidly, and a great deal of interest was building to determine the fissionable isotope of uranium.

Dunning had figured out that if I just souped up the spectrometer a little bit, I could collect enough separated isotopes of uranium to make possible a determination of the fissionable isotope. It had been predicted by Bohr and Wheeler that <sup>235</sup>U ought to be the one responsible for slow neutron fission, but it had never been demonstrated experimentally.

At the time of our interview, Nier downplayed the importance of that determination. As with so many items of burning interest, once the determination was made and the values were in the handbook, the intense urgency of obtaining an answer to so fundamental a question was forgotten. A recent publication on Dunning's contribution to nuclear physics [9] includes correspondence that Dunning had with Nier in April of 1939. The rather uncertain state of affairs with regard to which of the uranium isotopes was fissionable is clearly summarized by Dunning in the paragraph from his letter to Nier quoted below:

There is one line of attack that deserves strong effort, and that is where we need your cooperation. The important question is: which uranium isotope is really responsible for slow neutron fission? It is a matter of opinion largely, and some theoretical physicists think one way, some the other. Bohr thinks 235, but Fermi is neutral or inclined toward 238. Bethe and Placzek are on opposite sides of the fence, in fact there is a bet on. It is of the utmost importance to get some uranium isotopes separated in enough quantities for a real test of this whole question.

Fermi encouraged this experiment at his meeting with Nier, and following the meeting expected to see some sort of result. When Nier returned to Minnesota, however, he had his hands full with work in progress, teaching, and the development of thermal diffusion columns for isotope separation. And as at Harvard, word of his analytical prowess had gotten around the campus, so he had plenty of other materials to analyze for his colleagues there. It was during this period that Fermi wrote Nier a brief letter that in so many words said "Get to work on the uranium separation!"

Fermi was insensitive to the fact that solving the problem required both modifying the detector portion of the instrument to collect the separated isotopes and beefing up the ion source to produce fairly high ion currents. Besides, the initial attempt using uranium hexafluoride was a complete failure. The highly volatile salt had enough vapor pressure that a reasonable amount of material reached the collector without becoming ionized and separated. It essentially plated out the interior walls of the vacuum system. At this juncture, the decision was made to use the uranium tetrachloride and tetrabromide salts instead.

Even then, the actual separation was time consuming and difficult. The ion source beam current was running at two to three orders of magnitude higher than normal, and the sample pressure was so high that a discharge would start. The instrument could not be left unattended for long periods of time because the electronics did not have the long-term stability of present-day equipment. Despite these many obstacles, Nier was able to perform the separation, collecting miniscule amounts of each isotope on metal strips placed at the collector of the instrument. These were then sent by special delivery directly to Dunning at Columbia on March 1, 1940, approximately one year after the experiment was suggested. Dunning had the equipment to determine experimentally that <sup>235</sup>U was the radioactive isotope [10, 11]. It is interesting to reflect on the fact that the process Nier used to produce these isotopes represented the state of the art at the time. In the five years immediately following, the technology of separating uranium isotopes would make dramatic strides.

Shortly after returning to Minnesota, Nier became interested in thermal diffusion, a technique for fractionating gases with a small difference in molecular weight. His intention was to use this technique to produce enriched <sup>13</sup>C, charging the diffusion column with methane. The gas enriched with the heavier isotope could be used as a feedstock to produce a variety of compounds for chemical or biological tracer experiments. This work resulted in a series of publications in 1941 [12–18] describing the use of heavy carbon in this manner. That same year Nier also published a paper on the use of thermal diffusion for carbon isotope enrichment [19]. It was coauthored with another physicist who had joined the staff with Nier, an individual by the name of John Bardeen. Nier joked that of the two people to join the physics faculty that year, he would have to be considered the failure, because the other person-Bardeen-later shared in two Nobel prizes. Nier recalls that Bardeen "was really an order of magnitude better than the rest of us. He had a tremendous insight into physical phenomena."

The amount of work demanded that more instruments be built. There was just too much for the one, high-performance instrument. This meant that several smaller instruments with smaller mass ranges were designed and built to handle analyses that did not require the mass range and resolution of the larger instrument. Besides, the original instrument was being used more and more to analyze samples of enriched uranium from several different enrichment efforts around the country. Nier had the only instrument in the world capable of rapidly assaying the relative abundance of these heavy isotopes, thereby determining the degree of success of the enrichment effort. He was also busy building machines for installation at other laboratories to perform the enrichment assay. In late 1941 he went to Berkeley to help E. O. Lawrence in the design of a 180° instrument for the separation of the isotopes by using the cyclotron magnet.

They immediately began to get currents that were 100 to 1000 times bigger than we had ever been able to get.

This instrument was in essence the prototype of the calutron. Nier returned from Berkeley on December 4, 1941.

## The Manhattan Project

Nier was invited along with dozens of other scientists to Washington shortly after the attack on Pearl Harbor. After this meeting, he essentially worked for Harold Urey who was in charge of the various efforts at attempting to separate uranium isotopes. Urey realized the importance of having the analytical capability of the mass spectrometer in several places around the country. Upon returning to Minnesota, Nier immediately set about designing and building four new instruments. Through Arthur Compton, then at the University of Chicago, Mark Inghram, a student of Arthur Dempster, was sent to Minnesota to learn as much as possible about the instrument and the analysis. Later, Inghram would go to Columbia and E. P. Ney, a Minnesota student, to Virginia, each with two instruments to monitor the progress of the diffusion and centrifugation enrichment schemes being pursued at the respective schools.

Nier was relieved of his teaching responsibilities and his laboratories were now totally devoted to the Manhattan Project. All of the locks were changed on his laboratory and office doors, and tight control was exercised over who could gain access. With Inghram and Ney handling all of the analytical work load, Nier was free to develop and build instruments. An additional three uranium analysis machines were made along with ten smaller instruments for performing HD analysis (Figure 4). The impetus for these latter instruments arose in anticipation of the use of heavy water in the bomb production process—a use that never materialized. Although Nier could have had his choice of teams to work with on the Manhattan Project, he chose to go to Kellex, the contractor responsible for building the diffusion plant at Oak Ridge. This selection meant that he would be much more involved in the engineering and production side of the problem, even though his talents were sought by Oppenheimer in Los Alamos, Compton at Chicago, and Lawrence at Berkeley. His decision was driven partly by the fact that he would be working with some acquaintances from his Harvard days, Bob Jacobs and Manson Benedict, but mainly by his recognition of the tremendous analytical problems that would have to be dealt with to get the diffusion plant operational.

Shortly after arriving in New York, Nier and his colleagues at Kellex were searching for ways to make the large diffusion system at Oak Ridge leak tight. By now, gaseous diffusion had proven capable of separating the uranium isotopes using  $UF_{6r}$  a gas. One of the most technically bothersome problems with this technique was that the process vessel had to be completely leak free. Almost as important as keeping the  $UF_6$  from leaking into the environment was the problem of keeping moisture out of the process stream. If  $UF_6$  came in contact with moisture, it would react and plug the pores of the diffusion barriers, thus rendering the plant useless.

It was at this point that the idea arose of using a mass spectrometer as a helium leak detector [20] to

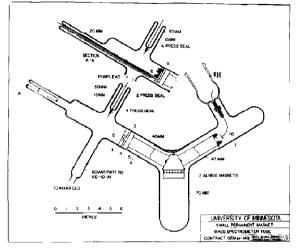


Figure 5. Schematic drawing of prototype helium leak detector mass spectrometer tube (see Figure 6).

check welds and seals for leaks. One of the deuterium analysis instruments was pressed into service to demonstrate the feasibility of the concept (Figures 5 and 6). Plans for the leak detector, as well as a prototype model, were turned over to General Electric, which produced hundreds of the instruments to monitor the multitude of welds and seals as the gaseous diffusion plant was constructed. The technique provided a means of detecting much smaller leaks than

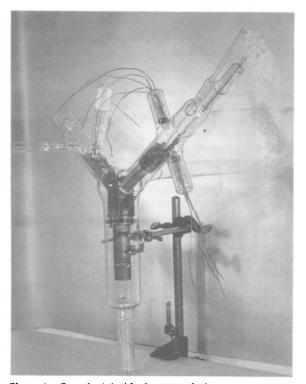


Figure 4. One of original hydrogen analysis mass spectrometer tubes (ca. 1942).

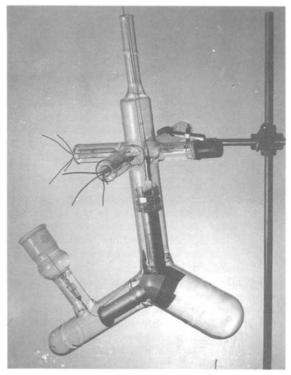


Figure 6. Original helium leak detector mass spectrometer tube.

any other existing method. This single technological development in itself had a major impact in bringing the diffusion plant on line in a relatively short period of time.

Because the development of the helium leak detector was associated with the Manhattan Project, it was kept secret as long as the war continued. Shortly after the end of hostilities, both General Electric and Consolidated Engineering Corporation produced and sold commercial versions of the device. However, two gentlemen who had been involved with the device at Kellex, Al Nerken and Frank Raible, started a small company by the name of Veeco to produce the helium leak detector. In the United States, Veeco was quite successful with the instrument and for many years dominated the American market.

Beside the problem of devising a means of ensuring that the plant did not have any leaks, there existed the fundamental analytical problem of determining the degree of contamination of the process stream. The stream was cooled with numerous refridgerating units, each of which could develop leaks. Hence the process stream could contain a multitude of contaminants, which were monitored with mass spectrometers, 100 of them! In this early application the uranium isotope enrichment was still determined by removing samples and determining the isotopic composition in laboratory measurements.

The plant was divided into some 50 buildings and two mass spectrometers were set up to monitor the process stream in each building [21]. One was used as a backup in case the on-line instrument developed a problem. In this fashion, there was a continuous flow of process data coming from every step in the enrichment. Beside being displayed at the mass spectrometer console of each stage, the information was routed to a central control room where the status of the process could be monitored by one individual. This was a completely new application of mass spectrometer technology. It worked well and provided the chief chemical engineer in charge of plant operation with the necessary information not only to control the process but also to shut down the plant in the event that some process variable got out of range.

Such a case did occur shortly after the plant was put in service. The monitoring instrumentation indicated that an air leak had developed in one of the stages. Unfortunately, the chief chemical engineer was as young as the technology, and his decision to halt the process stream because of the leak was overridden by older hands who "knew how to run things." The system filled with air but fortunately no serious consequences resulted. After the incident was reviewed and the plant put back into operation, the chief chemical engineer had considerably more authority in the control and operation of the plant.

With the end of the war and the analytical problems associated with the diffusion process taken care of, Nier's services were no longer needed. He, along with a great many scientists, went back to pick up their careers where they had left off. This meant a return to Minnesota.

### From Minnesota to Mars

Upon returning to the University in the fall of 1945, Nier found that he was at a distinct disadvantage. All of the instruments that he had built were made under contract to the Atomic Energy Commission. These machines belonged to the Commission and many of them had been placed in the laboratories of other AEC contractors, but Minnesota was not one of them. As a result, Nier—the driving force behind designing and creating dozens of mass spectrometers—had none whereas his colleagues around the country had Nier's! Needless to say, this situation did not last long, but once again, Nier had to build an instrument before he could get back to science.

A variety of interesting problems were pursued at this time, but the problem of accurate determination of the isotopic masses of the elements had a particular impact on mass spectrometry. Originally, Nier had used his instruments and electrical detection to determine precise quantitative information about the relative abundance of the isotopes of the various elements. This was an area in which he continued to do work. essentially leaving the task of precisely determining the masses of the isotopes to others: Bainbridge and his colleagues at Harvard and Mattauch and his collaborators in Germany. Both of these laboratories were using mass spectrographs with photoplate detectors. During Nier's work on isotopic abundance, it became clear that with an instrument of improved resolution, the electrical detection method, which worked so well for abundance determinations, could also be used for precise mass measurement as well.

At the time, a graduate student by the name of Edgar Johnson asked Nier if he had any topics for a master's thesis. Nier gave him the problem of designing the ion optics for a double-focusing instrument with sufficient resolution to permit precise mass determination of the elements [22]. Nier recently wrote a reminiscence about this collaboration to which the interested reader is referred [23]. It is worth noting here that Johnson completed his master's degree work but left Minnesota and had little to do with mass spectrometry afterwards, despite the fact that the high-resolution electrical detection instrument that he helped design had a tremendous impact on the subsequent development of analytical mass spectrometry.

As usual, Nier was always on the lookout for new and exciting areas of application for mass spectrometry. As he notes,

... the problem of studying the composition of the upper atmosphere was an interesting challenge. With all of the experience we have, we ought to build mass spectrometers to do this. This area of work required smaller instruments than had previously been designed. Furthermore, they had to be lighter and more rugged to withstand the forces of satellite launch.

An interesting turnabout occurred in this phase of instrument development. Nier had never used photoplate detection for any mass spectrometer design. His prowess with the Johnson–Nier geometry and electrical detection had given him an edge over his competitors in the precise mass measurement area. Even though he was good friends with Mattauch, Nier was able to produce results much more rapidly once his double-focusing instrument was put in operation. In a sense, he beat out Mattauch and his co-workers in the business of precise mass determinations.

As would be expected, when building new mass spectrometers for the upper-atmosphere studies Nier turned to the Johnson-Nier geometry. He quickly discovered, however, that it had some definite drawbacks, not the least of which was the need to scan the instrument over a mass range and thus sacrifice sensitivity. In addition, the basic design was not as compact and mechanically robust as that of Mattauch's geometry. For upper atmosphere and extraterrestrial studies, Nier quickly abandoned his own geometry and adopted that of his colleague and competitor in the business of precise mass determination. Of course there was a twist; Nier's version of the Mattauch-Herzog geometry used electrical detection. The instrument was designed to focus the ions of low-abundance compounds at two or more electron multipliers located on the focal plane of the instrument. Ions from more abundant species were measured by conventional scanning techniques. By the time Nier adopted this approach to the upper atmosphere and space studies, Mattauch had died. Thus, Mattauch was never able to appreciate the irony that the instrument geometry he had developed for precise mass determination ended up being used successfully by his friend and competitor for a completely different problem.

All of Nier's experience in mass spectrometer design in general and compact instruments in particular came to good use in the early 1970s when NASA solicited scientific teams for Project Viking, a Mars lander. Nier won the position of team leader for the atmospheric analysis on entry and was appointed a team member for the lander experiment, designed to search for evidence of life on Mars. This latter experiment got a lot more attention than Nier's atmospheric analysis on entry in the public press; but the scientists on the Viking team were much more interested in the outcome of Nier's experiment. Some years earlier, a crippled Russian experiment had sent back data that were interpreted to indicate that there was a large amount of argon in the Martian atmosphere. The lander instrument was pumped with a getter pump; thus, if the Martian atmosphere contained as much argon as the Russians surmised, the exobiology experiment would be severely limited because noble gases are not efficiently pumped by getter pumps. As soon as planetary entry began, the scientific team was huddled over the preliminary results from Nier's atmospheric analysis instrument. They discovered that the level of noble gases in the atmosphere was actually quite low and would pose no limitation for the exobiology team's mass spectrometer. Nier recalls this as the shining moment for the atmospheric analysis instrument. Immediately, all attention then focused on the lander instrument's measurements.

#### Conclusion

Clearly, Al Nier's career spans one of the most technologically exciting periods of history: from crystal radio receivers to mass spectrometry on Mars. Probably the single most descriptive characteristic of Nier's career is what he characterizes as his "short attention span." The single constant throughout is mass spectrometry, but with different applications. Typically, an area of study held his interest for five or so years; then another interesting and exciting application came along. After doing seminal work Nier would move on to another area. Thus, his work is referenced in the chart of the nuclides, in radiogenic dating studies, in the use of heavy carbon as a tracer in biological studies, in the design of high-resolution mass spectrometers, in the application of mass spectrometry to the monitoring of patient respiratory gases during operations, in the study of the composition of the upper atmosphere, in the study of the composition of the Martian atmosphere, in the use of mass spectrometry in leak detection.... Truly, the impact of this one man's work on this field of study is immeasurable.

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