Rediscovery of the Elements

Rhenium and Technetium

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Sc (21)	**Ti (22)**	**V (23)**	**Cr (24)**	**Mn (25)**	**Fe (26)**	**Co (27)**	**Ni (28)**	**Cu (29)**	**Zn (30)**
Y (39)	**Zr (40)**	**Nb (41)**	**Mo (42)**	**eka-Mn (43)**	**Ru (44)**	**Rh (45)**	**Pd (46)**	**Ag (47)**	**Cd (48)**
La (57)	**Hf (72)**	**Ta (73)**	**W (74)**	**dvi-Mn (75)**	**Os (76)**	**Ir (77)**	**Pt (78)**	**Au (79)**	**Hg (80)**

Figure 1. The transition metals of the Periodic Table had all been discovered by 1923 except for the eka-manganeses (also known as "eka-" and "dvi-" Mn, "1" and "2" in Sanskrit). Noddack and Tacke reasoned that these two elements might be found in platinum ores (where the shaded blue elements may be found as impurities) and/or columbite (Fe,Mn)(Nb,Ta)$_2$O$_6$ (red shaded elements).[6] Their research showed that element 75 (rhenium) is actually most commonly associated with molybdenum in nature (see double headed arrow), corresponding to the "diagonal behavior"[27,28] occasionally observed in the Periodic Table.

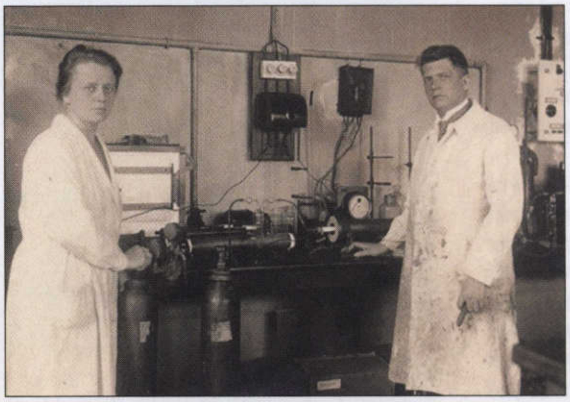


Figure 2. Ida Tacke and Walter Noddack in their laboratory at the Physikalisch-Technische Reichsanstalt (PTR) in Berlin-Charlottenburg, established in 1887, where rhenium was separated and characterized. Noddack is posing with his ever-present cigar and is wearing his stained laboratory coat proving that indeed he was a dedicated bench-chemist. Photo, courtesy, Wesel Archives, Germany.

After the 1923 discovery of hafnium,[1f] only two transition group elements were yet to be discovered. These predicted elements (43 and 75) were homologues of manganese, the predicted "eka-manganeses" of Mendeleev[1c] (Figure 1). However, all attempts to find them in crude manganese preparations ended in failure.[2] The problem of the missing elements was addressed in a comprehensive research project by the husband-wife team of Walter Karl Friedrich Noddack (1893–1960) and Ida Eva Noddack-Tacke (1896–1978) (Figure 2).

The "work-unit" of Noddack and Tacke.[3,4] Walter Noddack was born in Berlin and educated at that city's Friedrichs-Werdersche Oberrealschule. This was the same prestigious institution where Friedrich Wöhler in 1828 disproved the theory of "vital force" in organic compounds by isomerizing ammonium isocyanate into urea.[1a] Noddack then matriculated at the University of Berlin and obtained his doctorate in 1920. His advisor was Walther Nernst (1864–1941; Nobel laureate in chemistry, 1920), who formulated the third law of thermodynamics. When Nernst left to accept the presidency of the Physikalisch-Technische Reichsanstalt (Figure 3) in 1920, Noddack moved with him. Three years later, Noddack became the new head of the Anstalt. It was here that he met Ida Tacke.

Ida Tacke was born in Lackhausen (a suburb of Wesel), a town on the Rhine River 85 kilometers north of Köln (Cologne). She was the daughter of Adelberg Tacke, the owner of a varnish factory. Ida was educated at the Berlin Hochschule, earning her doctorate in engineering (Doktor-ingenieur) in 1921.[3] She specialized in organic chemistry, thinking that she could improve the curing behavior of linseed oil in her father's business. After her graduation, however, she met Walter and was smitten by his quest for new elements. She joined the Physikalisch-Technische Reichsanstalt in 1922 and they married four years later. The two researchers formed, as she called it, the *Arbeitsgemeinschaft* (work-unit)[3] that held them together through mutual research and love in a manner reminiscent of Pierre and Marie Curie — some German journalists have called her "die deutsche Marie Curie."[5] The story is told that when Walter passed away in 1960, it was because of an emotional heartbreak when he heard (incorrectly) that his wife, unreachable by telephone, had died during an illness in Hamburg.[3]

The search for the missing elements. Noddack and Tacke observed that odd-atomic number elements were less abundant than even-atomic number elements.[6] Thus they realized that the eka-manganeses might be particularly rare, and that a very meticulous and methodical search might be necessary to discover them. The team reasoned that since the eka-manganeses had not been found in nature with the parent element 25, they should search

ABOVE: Figure 3. The chemistry building (Chemie Gebäude) of the PTR is now used for administration purposes. The name of the PTR was changed to the Physikalisch-Technische Bundesanstalt (Physics-Technical Federal Institute) in 1945. Presently, among other responsibilities, the Bundesanstalt is accountable for atomic clock standards in Germany.
Inset: *The Waffen (icon) as it appears at the entrance of the Anstalt, Abbestraße (2-12, N52° 30.98 E13° 19.26), Charlottenburg, West Berlin.*

Figure 4. One of the more prolific sources of rhenium-bearing molybdenite is Knaben, Vest-Agder, Norway, where a historic mine is located (N58° 39.55 E07° 04.41). This mine provided strategic molybdenum used in steel armament for the WWII Nazi effort and was bombed twice by the allies in 1943. Note the snow in the foreground despite the summer date (June 30).

RIGHT: Figure 5. The talus scattered about at Knaben, Norway—900-million-year-old Precambrian granitic gneiss— is profuse with molybdenite that looks like splashes of solder. The molybdenite is rich in rhenium, with concentrations as high as 7.5 ppm.[7] Historically, molybdenite, native lead, and graphite were all confused with one another; Scheele distinguished the three in 1779.[1b] Molybdenum is named after the Greek word for lead (molybdos).[1b]

in platinum and columbite ores, in which many low-concentration elements had already been observed (Figure 1). Ida took a full year's sabbatical to conduct a thorough literature search to develop a complete repertoire of chemical separation procedures to help them in their quest.

Working with a variety of platinum and columbite samples, in 1925 they obtained a white sublimate (Re_2O_7). Collaborating with Otto Berg (1873–1939) of Siemens & Halske[1h]— whose laboratories were conveniently located only 400 meters north — Tacke recorded the X-ray spectra of their preparations. Based on the work of Moseley[1e] which predicted the X-ray frequencies based on atomic number, they identified lines from elements 43 and 75. They claimed the discovery of two new elements,[6] which they named *masurium* (Ma) and *rhenium* (Re), respectively (after regions in Prussia and the Rhineland). So far they could not obtain either element in weighable quantities.

After their marriage, Noddack and Tacke traveled to Scandinavia to find richer sources of rhenium; they procured ore samples from 124 mines in Sweden and Norway.[7] An analysis of these indicated relatively large amounts of rhenium in molybdenite (MoS_2) in several of the Norwegian sites[7] (Figures 4, 5). In two years they were able to isolate 1 gram of rhenium from 660 kg of molybdenite, whose X-ray lines confirmed element 75 (rhenium).[8] Rhenium was the last naturally-occurring element to be discovered (Note 1).

Unfortunately, the Noddacks could not reproduce the results for element 43 (masurium). Unknown to them, element 43 has no stable isotopes, and its discovery had to await nuclear synthetic procedures (*vide infra*).

In 1935, the Noddacks moved from Berlin to Freiburg, then in 1941 to Strassburg after Germany annexed the region (Strassbourg in French), and finally the University of Bamberg in 1946, where he founded the Staatliches Forschungsinstitut für Geochemie (State Research Institute for Geochemistry) (Note 2). As an outgrowth of their search for the eka-

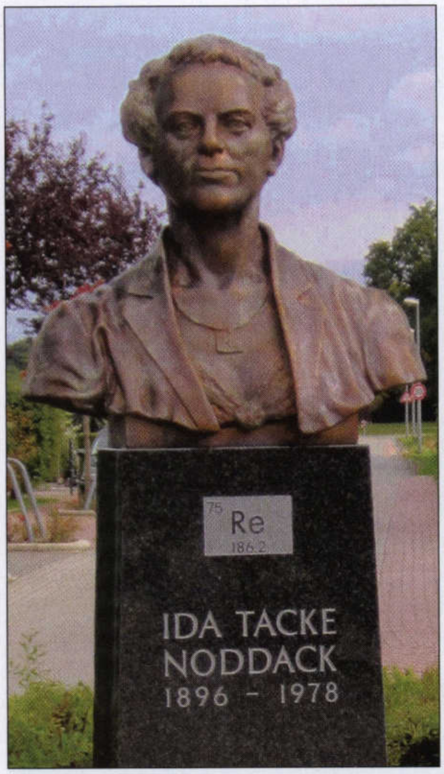


Figure 6. This bronze bust of Ida Noddack-Tacke stands in her hometown of Wesel on Ida-Noddack-Straße (N51° 40.70 E06° 37.96). This two-meter monument, erected in 2006, was constructed by Andreas Krämmer. (For other plaques, see Notes 2 and 3).

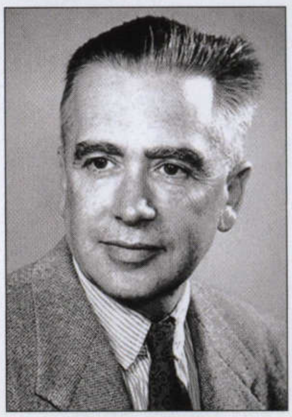


Figure 8. The "Old Radiation Laboratory" at the University of California-Berkeley housed the 27-inch cyclotron invented by Lawrence (later modified to a 37-inch instrument). This laboratory was put into operation in 1931, refurbished from the Civil Engineering Testing Laboratory, built in 1885 (N37° 52.40 W122° 15.37). The building was demolished in 1959 and replaced by Latimer Hall.

Figure 7. Emilio Segrè was co-discoverer of technetium (1937) and later at Berkeley was co-discoverer of the antiproton in 1955 with Owen Chamberlain (1920–2006), with whom he shared the Nobel Prize in Physics in 1958. Segrè moved permanently to the United States in 1938, followed the next year by his wife Elfriede née Spiro. Segrè played a critical role in the Manhattan project, solving problems involving plutonium in atomic bombs.

manganeses, Ida and Walter Noddack originated the concept of "elemental abundances" in the earth's crust ("Die Häufigkeit der chemischen Elementen")[9] and produced estimates for the occurrence of all known elements. Walter retired in 1956.

The Noddacks were nominated several times for the Nobel Prize, but unlike the Curies were not successful. It is suggested that the inability to verify masurium contributed to their failure to win this prize.[3,10] It is ironic that Enrico Fermi (1901–1954) was awarded the 1938 Nobel Prize in Physics for "new radioactive elements"[11] as Ida Noddack-Tacke (Figure 6) was correctly challenging the Italian scientist's conclusions. Refusing to accept the general consensus that neutron bombardment of uranium was creating transuranium elements in Fermi's laboratory, she suggested smaller atomic fragments would be created and one should *first* check the product mixture for all *known* elements.[12] In her article "Über das Element 93," she actually proposed the concept of nuclear fission[12a] five years before the dramatic announcement of Hahn, Strassman, Meitner, and Fritsch.[13] During the period 1935–1938, whenever Walter would remind Otto Hahn (1879–1968) of Ida's suggestions regarding nuclear fission, Hahn would abruptly dismiss the idea with "Ein Fehler reicht" ("One mistake is enough"!),[14a] referring to the Noddacks' masurium research. It is sobering to reflect on the possible outcome of Germany's WWII campaign if Hahn, winner of the 1944 Nobel Prize in Chemistry for "his discovery of the fission of heavy nuclei"[15] at the Kaiser Wilhelm Institut, had not ignored Tacke's early suggestions. It was 30 years later, during a radio broadcast in 1966, Hahn finally admitted, ". . . und die Ida hatte doch Recht." (". . . and Ida was right after all"!)[4a]

The discovery of element 43 — technetium.[16] Many historic claims for "the element between molybdenum and ruthenium" have been proposed, including "polinium," "ilmenium," "pelopium," "davyum," "lucium," "nipponium," "neomolybdenum," "moselium," and "masurium."[16] Unbeknownst to chemical explorers, this element did not exist in the earth's crust in appreciable quantities. The most stable isotope of element 43 is $_{43}Tc^{98}$ with a half-life of 4.2 x 10^6 years — and any primordial element 43 should be long gone since the creation of the earth 4.6 x 10^9 years ago. Hence, the discovery of technetium was not possible until the advent of the artificially produced elements.

While the Noddacks were isolating their first gram of rhenium, Emilio Gino Segrè (1905–1989) was beginning his studies with Fermi at the University of Rome. Segrè (Figure 7) was born in Tivoli, 26 km east of Rome. After a short stint in the military, Segrè became professor of physics at the University of Rome (1932), where he joined the "Via Panisperna boys," a group of scientists under the guidance of Fermi who were pioneering the field of nuclear reactions involving slow neutrons. In

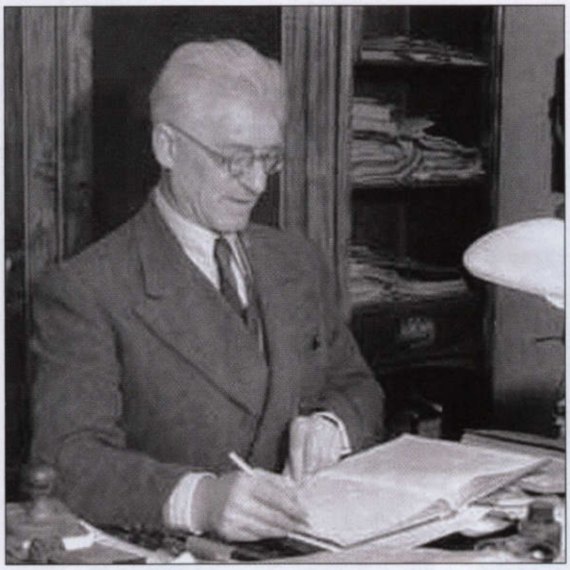
Figure 9. Ernest Orlando Lawrence (1901–1958; Nobel Prize in Physics in 1939) invented the cyclotron in 1931, and improved its design and size through the coming years. A museum devoted to him—the Lawrence Hall of Science—is the best place to be introduced to the extraordinary history of nuclear chemistry at Berkeley. This museum is on Centennial Drive in the Berkeley Hills (N37° 52.77 W122° 14.80) overlooking the University of California at Berkeley. The museum was established in 1982 by Glenn Theodore Seaborg (1912–1999; Nobel Prize in Chemistry 1951; ΑΧΣ - Beta Gamma '35, *John R. Kuebler Award, 1978).*

ABOVE: Figure 10. The magnet used for the 27-inch cyclotron (operational 1932), then modified to the 37-inch cyclotron (operational five years later), is now standing outside the Lawrence Hall of Science. The magnet, originally built for power communication between the U.S. and Europe in World War I, was acquired by Lawrence as war surplus.

BELOW: Figure 11. On Via Archirafi 36, Palermo, Sicily, Italy, is the old Experimental Physics Building (N38° 06.61 E13° 22.39), where technetium was discovered. The first two floors housed physics, and the top floor housed mineralogy. The building is now used for library resources, storage, and the Mineralogy Department. (The modern science buildings are at the new campus on Viale Delle Scienze, 2.2 km to the west.)

1936, Segrè became professor and director of the Department of Physics at the University of Palermo. During a visit to Berkeley in 1936, he became curious about the possibilities of transmutations occurring in the cyclotron (Figure 8) invented by E. O. Lawrence (Figure 9). He returned to Palermo with scrap metal pieces which had been radiated from which he isolated several radioactive elements, including sulfur-32. In February 1937 in Palermo, he received from Berkeley a radioactive strip of molybdenum foil used as a deflector plate in the 37-inch cyclotron (Figure 10). Since the molybdenum had been bombarded with deuterons (${}_1H^2$), Segrè "suspected at once that it might contain element 43,"[17] next to molybdenum in the Periodic Table.

Segrè needed chemical expertise, and he was fortunate that his university building (Figure 11) housed the institutes of both physics and mineralogy. The head of mineralogy was Carlo Perrier (1886–1948) (Figure 12), an Italian mineralogist well versed in the classical analytical procedures in chemistry. In their separation procedures, Segrè and Perrier followed the radioactive material in their separations using the manner pioneered by the Curies when they first separated polonium and radium.[1d] First, they reacted the Mo foil with boiling ammonia to remove the surface impurities, where the nuclear reactions would have occurred. Because of the anticipated small amounts of product (less than 10^{-10} gram,[18]) to the extracted solution, they added various carriers — zirconium (at. no. 40), niobium (41), molybdenum (42), and manganese (25). These were individually isolated, but none showed radioactivity. To the remaining radioactive solution they added a rhenium (75) carrier which was precipitated by H_2S. This radioactive rhenium (sulfide) carrier was converted to the oxide with hydrogen peroxide and distilled to remove the volatile rhenium oxide, which



LEFT: *Figure 12. Carlo Perrier, head of the Mineralogy Institute, worked out the chemistry of technetium[19,29] which allowed its isolation. Despite the importance of his work which allowed the discovery of the first artificially produced element, Perrier never received the Nobel Prize.*

RIGHT: *Figure 13. Prof. Arturo Russo, of the Dipartimento di Fisica e Tecnologie Relative, Università di Palermo, was the host of the authors during their visit to Palermo. Dr. Russo is interested in the science history of his university. From forgotten storage items he has found the old equipment used by Emilio Segrè. Russo is holding the ionization chamber constructed by Segrè.*

exhibited no radioactivity. The remaining radioactive solution was concluded to contain element 43 —but without weighable amounts isolated and without an X-ray spectrum[19] (Figure 13).

Segrè promptly visited the Noddacks, now in Freiburg (September 1937). Segrè, who spoke excellent German, wished to give credit where it was due. He wanted to establish unequivocally whether he had simply confirmed the existence of "masurium," or whether he was actually the first to observe element 43.[17] According to Segrè's account[17] Walter was noncommittal and could not produce the X-ray plates of masurium, which he said "had accidentally been broken." Obtaining no definitive information, Segrè concluded that at the very least, the Noddacks had "no clear-cut results" and that if their claims were indeed lacking, they would probably "fall of their own weight."[17] Two weeks later, the Noddacks and colleagues, outfitted in uniforms with swastikas,[14a] appeared at Segrè's laboratory in Palermo; Segrè showed them his results and they left without comment. No more personal contact occurred between Segrè and the Noddacks.

Subsequent work with the cyclotron at Berkeley allowed material to be produced whose X-ray spectrum confirmed element 43. By the early 1940s, it was generally agreed that element 43 is "missing from our earth"[20a] (technetium *has* since been detected in the spectra of stars[21]) and in five years a protocol was suggested for the naming of artificial elements.[20b] In 1947, a publication appeared in *Nature* dubbing the first artificial element, atomic number 43, as *technetium*.[22]

Could the Noddacks have observed technetium? Ever since Perrier and Segrè's announcement in 1947, there has been a dispute as to whether or not the Noddacks ever could have detected element 43 in their preparations. The short half-life of technetium precludes its presence on earth except in radioactive ores where secondary processes have occurred since the creation of the earth.[23] A subsequent careful analysis has shown that the low concentration of uranium in the Noddacks' material would not have produced detectable amounts of technetium by their X-ray analysis.[24]

The struggle for priority of the discovery of the elements has often been contentious. In a 1947 editorial by Friedrich Adolph Paneth (1887–1958) in *Nature*, this "speaker of authority"[1a] wryly noted, "... the slowness of chemists in taking the obvious action of abandoning suggested names is due to the failure of the claimants to withdraw their statements, although during years of intensive effort they had been unable to substantiate them. In the case of masurium, W. Noddack even went so far ... as to complain to the convener of a chemical meeting in Königsberg for not having invited him to speak on this element, as ... he would have been in a position to disclose the whole chemistry of masurium ... but no communication has ever appeared on this work."[20b]

Despite the lack of further proof of masurium, the most "up-to-date" Periodic Table in the late 1920s and early 1930s was the Antropoff Periodic Table (highlighted in the previous issue of *The HEXAGON*[1i]) which proudly proclaimed "Ma" as element 43. The "largest Periodic Table in the world," constructed in 1935 on the wall of the Metrology Institute in St. Petersburg,[1c] included not only masurium (43) but also illinium (61), both spurious elements which at that time were erroneously "firmly rooted in textbooks and tables."[20b] It is interesting to note that the original tiled "Ma" has been scraped off the Russian muraled Periodic Table, while "Il" remains. One might suspect political reasons are responsible for this removal of "Ma," since the Battle of the Masurian Lakes in World War I was a victory for Germany over Russia. Some believed the Noddacks chose the name for nationalistic reasons, despite the Noddacks' insistence it was done in recognition of his father's homeland (East Poland, then Prussia).[14a]

In defiance of the general rejection of masurium by the scientific community, a plaque on Ida's old homestead in Wesel (Note 3) boldly proclaims [translated] ". . . This worldwide famous woman chemist discovered and described in 1925, together with her husband, rhenium and masurium (today known as technetium . . .)."

Acknowledgements.

The authors are indebted to Dr. Hans Georg Tilgner of Wesel, Germany, local expert on the Noddacks and author[4] of *Forschen, Suche und Sucht [Research, Quest, and Obsession]*, for his generous help of information and photographs regarding the discovery of rhenium, particularly that dealing with the hometown heroine Ida Tacke.

Notes.

NOTE 1. A claim was made in 1908 for a new element *nipponium*[25] found in thorianite (ThO_2) by a Japanese chemist, Masataka Ogawa (1865–1930), in the research group of Sir

William Ramsay (1852–1916, Nobel laureate in Chemistry, discoverer of the noble gases.[1g]) Ogawa placed the element between molybdenum and ruthenium in the Periodic Table, i.e., element 43, or present-day technetium. Upon returning to Japan, Ogawa isolated what he thought to be the same element from Japanese molybdenite. H. Kenji Yoshihara (1929–)[25] long after Noddacks' work, suggested that Ogawa in fact had rhenium,[26, 27] which probably was true based on an analysis of the Noddacks' world-wide analyses[7] which showed Japanese molybdenite to be the richest they ever studied (9.8 ppm Re).

NOTE 2. A plaque devoted to Walter Noddack resides at Künstlerhaus, Villa Concordia, Concordiastraße 28, Bamberg (N49° 53.28 E10° 53.31), in honor of his accomplishments in geochemistry.

NOTE 3. The old Tacke home in Wesel ("Haus Wohlgemuth") is located at Brüner Landstraße 301 (N51° 41.04 E06° 39.12), beside the Brüner Lackhauser Farben (paint store), on the site of the original varnish factory established in 1867. A plaque was erected in 2006, which describes her major accomplishments, notably the discovery of rhenium and masurium, and her 1934 idea of the splitting of the uranium atom by neutron bombardment.

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