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Concepts and Techniques in the Discovery of the Actinide Elements*

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Abstract

The role of new concepts and techniques in the discovery of the actinide elements is interesting. Two of these elements date from long ago, around the time the concept of an element became accepted. Two more date from the classical period of radioactivity. The remaining eleven were identified in the rather short period of 20 years. Both new ideas and experimental techniques were important in this rapid development. As so often is the case with discoveries in science, some rather convoluted stories are found.

Introduction

The history of science is not for those who prefer the logical, neat and tidy; an orderly progression to an objective is seldom found. The course of discovery is commonly tortuous and follows several paths. The dramatis personae are often numerous; each making his own contribution to consolidating and extending that vast body, the corpus of science.

Perhaps this is because at a given point in time the climate of ideas in a subject will lead to several people thinking along similar lines, performing similar experiments and reaching similar conclusions, often quite independently. Science benefits because the sum of the results of such studies is closer to the truth than any individual contributions.

The actinide elements now have a history extending over a little more than 200 years. It is interesting to examine some of the factors influencing their discovery.

Uranium and Thorium

Two of these elements, uranium and thorium have been known for a very long time. When modern chemistry was just beginning there was a great interest in determining the composition of minerals and establishing a classification.

Those elements with coloured compounds and possessing more than one oxidation state responded more readily to the techniques of investigation then available. In 1786 Klaproth examined a sample of pitchblende from Saxony [1], then thought to be a zinc or, perhaps, iron ore, and by 1787 he could claim the discovery of the new element uranium [2] in a paper which gives an easily recognizable account of its simpler chemistry.

The white earths were less tractable to study and identification of new elements in these materials was less certain. Thus in 1815 Berzelius [3] thought he had identified a new element in a Scandinavian mineral and he called the oxide 'thorina'. Some years later, however, he came to the conclusion he had been examining a basic phosphate of yttrium [4]. Later still, examining a mineral now called 'thorite', from Lövön in Norway, Berzelius identified the element we know as thorium [5]. The difficulties of rigorous identification of such elements in the first half of the 19th century led to a number of subsequent supposed discoveries of the same element [6]. For a substantial part of the century the element was known as 'thorinum' [7].

Actinium and Protactinium

The next two actinide elements are associated with the beginning and almost the end of the classical period of the study of radioactivity. The discovery of radioactivity stimulated intense interest in the identification of species showing this remarkable property. It is strange that the third of the new radioactive elements to be identified was the 'rayless' species actinium (^{227}Ac). Not surprisingly, the history of its discovery is fairly complex. In the year following the Curies' discovery of polonium and radium, Debierne found a new radioactive species in the hydroxide insoluble fraction obtained after the separation of U, Ra and Po from a uranium mineral. He likened his product to titanium [8].

About this time the mysterious power of strongly radioactive sources transferring radioactive properties to the surrounding materials was attracting a lot of

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attention and Debierne became much involved in these studies; in fact he noted that actinium preparations also showed this property [9]. He did not return to actinium for two or three years.

Meanwhile, Giesel had been examining, independently, the radioactive species in a rare earth-like fraction obtained from uranium minerals [10]. In 1903 both investigators explored the emanations produced by their products [11a, b]. In the following year a direct comparison of their products showed them to be the same [12].

Giesel had called his new element [11b] 'emanium' and Debierne his 'actinium' [9]. It was agreed that the latter name should be adopted. Debierne found that his product was very like a rare earth and, indeed, could only be separated from lanthanum by fractional crystallization, when it accompanied Nd and Sm [13].

It is interesting to note that during all these studies it was not realised that the radiations detected were not emitted by the actinium, which was later described as rayless [14]; pure actinium compounds were not obtained until many years later using synthetic ^{227}Ac, made from radium [15].

The radioactive displacement laws derived by Russell, Soddy and Fajans indicated that in the transformation of U(I) (^{238}U) to U(II) (^{234}U) two beta-decay steps must be involved and an isotope of the then missing element 91 must be formed. It was already known that UX, the daughter of UI, appeared to have a very soft as well as a hard beta emission, and in 1913 Fajans and Gohring [16] showed that UX comprised UX_1 (^{234}Th) and UX_2 (^{234}Pa). They characterized both the nuclear and chemical properties of the ^{234}Pa and called the new element 'brevium', because of its short half-life. There was much speculation at this time concerning the origin of actinium, then known to be a relatively short-lived species.

Progress was then interrupted by the First World War, but immediately after, both Soddy and Cranston [17] and Hahn and Meitner [18] independently separated and characterized a long-lived isotope of this element, ^{231}Pa. This was indeed the parent of actinium and they called it 'protactinium'. This name has, perhaps improperly, been adopted; it subsequently acquired and then lost a second 'o'.

These developments are most conveniently followed in Soddy's reports on radioactivity in the Annual Reports of the Chemical Society 1904–1920.

An Actinide Group

Although nearly twenty years were to elapse before another actinide element was reported, a very important development took place during this interval.

The concept of the Rutherford–Bohr atom and the application of the embryonic quantum theory to atomic structure led to an understanding of the electronic structure of atoms. The reasons for the existence of the rare earth group became apparent and already in 1913 Rydberg suggested that another such group of elements might exist [19]. A more detailed account of such a group was given by Bohr [20]. For the next fifteen years discussions and calculations focussed on the atomic number of the element at which the 5f orbitals would first be used. There was general agreement that it would be somewhere between $Z = 90$ and 95 [21]. Goldschmidt, who favoured a start at element 93, suggested the new group be known as the 'neptunium group' [22a, b, c]. The idea of an actinide series was established, but it could hardly have been said to have been common knowledge among chemists.

Neptunium

The discovery by Fermi and his collaborators that neutron irradiation of uranium gave rise to beta-active products [23] heralded great activity in the study of these supposed transuranic elements. Bohr had already made prescient suggestions about the difference in the chemistry of a 5f compared to a 4f group of elements [20]. Attention was drawn by von Grosse to the dependence of the chemical properties to be expected for these elements on the point at which use of the 5f orbitals began [24]. Four years of misunderstanding followed until, in January 1939, Hahn and Strassmann came to the conclusion that a 3.5 h activity, already known to have rare earth-like properties [25], could not indeed be separated from lanthanum and must in fact be an isotope of that element: a nuclear reaction of fission had taken place [26].

It was quickly recognized that the products of this reaction had enormous kinetic energy and therefore recoiled vigorously from the target material, distinguishing them from the weakly recoiling products from radiative thermal neutron capture. In February of the same year, McMillan showed that two of the activities, formed by the neutron irradiation of uranium with half-lives of 23 min and 2.3 days did not apparently suffer a strong recoil [27].

The first of these activities had been identified as ^{239}U by Meitner, Hahn and Strassmann two years previously [28]. Later in 1939, Segre showed that the 2.3 day body had rare earth-like properties and could easily be separated from the uranium isotope; he did not recognize any genetic relationship between the two activities. He concluded that the daughter of the 23 min body must be very long-lived and that no direct evidence for transuranium elements existed [29].

This continued to be true for nearly a year. But in May 1940, McMillan and Abelson showed that the 2.3 day beta-active species was the daughter of the 23 min ^{239}U and that the new element had rather similar properties to its neighbour uranium [30].

Plutonium, Americium and Curium

The Second World War was now accelerating and the story of the actinides went undercover for nearly five years. However, what happened during this period is well documented. The experiments on the 2.3 day ^{239}Np certainly indicated that its daughter, an isotope of element 94, must be rather long-lived. Many nuclear physicists were convinced that the daughter species would be a highly fissile nucleus and there was great interest in obtaining enough of it for nuclear measurements. The then available neutron sources (natural sources and particle accelerators) barely produced enough neutrons for this purpose, remembering that the chemistry of element 94 was unknown and that the half-life of this isotope could be very long indeed.

At this point, the University of California at Berkeley gained a unique advantage, the newly constructed 60" cyclotron began working, producing sufficiently energetic particles to achieve nuclear reactions with targets of high Z. The discovery of most of the remaining actinide elements is very much the story of the achievements of Seaborg and his collaborators in Berkeley and, later, in the various laboratories run by the American wartime Manhattan Project.

Thus, at the end of 1940, deuteron bombardment of uranium was found by Seaborg, McMillan, Kennedy and Wahl to produce, in addition to ^{239}Np, a 2.0 day half-life beta-active isotope of the same element. Considerations of yields as a function of the energy of the bombarding deuterons suggested the reaction was ^{238}U(d, 2n)^{238}Np [31].

This isotope of neptunium decayed to yield a moderately long-lived ($t_{1/2} \sim 40$ years) alpha-active isotope of element 94 [31]. In another month, an outline of the chemistry of the new element had been obtained and it was found to resemble its neighbour, neptunium, and uranium [32].

The Manhattan project was now expanding rapidly and a large part of its effort was devoted to developing means of separating ^{239}94 from uranium and the fission products. This was largely achieved using the isotope ^{238}94. In March 1941, it was possible to confirm that ^{239}94 was indeed a highly fissile species, decaying by alpha emission with a half-life of about 24 000 years. By August 1942, a visible (some γ) amount of the isotope had been isolated.

In the spring of 1942 the names neptunium and plutonium were chosen for the two transuranium elements.

For three years all available effort was devoted to the problems associated with the large-scale separation of plutonium. But by 1944 it became possible to return to the problem of the existence of elements of still higher Z values. By now reactors producing enormously greater neutron fluxes were available, but it was cyclotron bombardment that yielded the first results. Initially, progress was slow because it was supposed that elements 95 and 96 would prove very similar in chemical properties to U, Np and Pu. However, a reconsideration of earlier ideas on the actinide series suggested that, perhaps, it would be difficult or even impossible to oxidize these elements to the hexavalent state, indeed, perhaps, the trivalent state would be the common one in aqueous systems. Late in the summer of 1944 Ghiorso, James, Morgan and Seaborg obtained an alpha-active isotope of element 96 by the reaction ^{239}Pu(α, n)^{242}96. It had a half-life of 162.5 days. The irradiations were carried out on the Berkeley 60" cyclotron but the targets were processed at the metallurgical laboratory at the University of Chicago. It was soon apparent that in aqueous solutions the element only displayed one valence and resembled the rare earth elements.

Towards the end of the year, an isotope of element 95 was isolated by the same group but using a preparation by repeated neutron capture, a technique that was to lead to further actinide elements:

$$^{239}\mathrm{Pu}(n, \gamma)^{240}\mathrm{Pu}(n, \gamma)^{241}\mathrm{Pu} \xrightarrow[\text{14 years}]{\beta} {}^{241}95$$

The product was an alpha-active species with a half-life of 433 years [33]. These two new actinide elements were named 'americium' and 'curium', with $Z = 95$ and 96, respectively.

The two elements have the special distinction of having been first revealed to the public on a radio quiz show for children on November 11, 1945 [34]. Their existence had just been declassified for an American Chemical Society meeting taking place at North Western University, Chicago the following week [35].

At first it proved extremely difficult to separate mixtures of the two new elements by chemical means. But in overcoming this difficulty a foundation was laid for chemical characterization of the rest of the actinide elements. It was found that a satisfactory separation could be achieved using ion exchange columns in a similar way to that which had been found convenient for the separation of the rare earth fission products. This led to a careful comparison of the ion exchange characteristics of the trivalent heavier actinide elements with the analogous rare earth elements, supposing the origin of the former group lay at actinium. A one-to-one correspondence between the two sets of data was found, so that the ion exchange characteristics of a new actinide element could be predicted with considerable confidence. Further ion exchange separations could be conducted rather quickly with trace amounts of an element, so that this chemical characterization was possible even with short-lived species.

Parallel progress was made in the prediction of the nuclear properties. Examination of the growing body of data on decay energies and precise relative nuclear masses showed that for similar kinds of nuclei the mass data lay on fairly smooth surfaces so that values for unknown species could be interpolated, or even extrapolated, with some confidence. Thus it proved possible to predict with some accuracy both some chemical and nuclear properties of the isotopes of a previously unknown element. But for these two guides the discovery of the remaining actinides would undoubtedly have taken much longer.

Berkelium and Californium

The next two actinides to be discovered were produced by cyclotron bombardments, using targets with the highest atomic and mass numbers conveniently available. Success rewarded these efforts about five years after the discovery of americium and curium.

Thompson, Ghiorso and Seaborg obtained $^{243}97$ by the nuclear reaction $^{241}\mathrm{Am}(\alpha, 2n)^{243}97$, a species of half-life of 4.5 h, decaying mainly by orbital electron capture [36]. Soon after, Thompson, Street, Ghiorso and Seaborg obtained an isotope of element 98 by the reaction $^{242}\mathrm{Cm}(\alpha, n)^{245}98$. The product was at first assigned the mass number 244 [37]. It had a half-life of 44 min and decayed both by alpha emission and orbital electron capture.

By now the quantities of materials in these experiments had decreased spectacularly. The irradiation involved a target containing only a few micrograms of $^{242}\mathrm{Cm}$ and $^{245}98$ was characterized using only some thousands of atoms.

Einsteinium and Fermium

With one exception, the discoveries of the rest of the actinide elements were much more complex. Elements 99 and 100, called einsteinium and fermium were discovered independently by two groups of scientists working in the United States. Both made use of repeated neutron capture reactions but in rather different ways.

The first group investigated the debris from the first thermonuclear weapon explosion in November, 1952. In this event uranium was exposed to an enormous neutron flux for a very short time. Repeated neutron capture events took place in the $^{238}\mathrm{U}$, producing highly beta-unstable species, such as $^{253}\mathrm{U}$. These decayed and amongst the products was found $^{253}\mathrm{Es}$, a largely alpha-active species of half-life 20 days. The same debris yielded $^{255}\mathrm{Fm}$, an alpha-active species of half-life 20.5 h [38]. They were characterized by ion exchange methods and by their decay products.

These investigations were carried out in three laboratories, the Argonne National Laboratory, the Los Alamos Laboratory and the Radiation Laboratory at the University of California. The einsteinium isotope was identified at the end of 1952 and fermium in the spring of 1953. However, the information was not declassified for publication until 1955.

By this time, workers in Idaho, using the high flux Materials Testing Reactor, had made isotopes of the same elements by an essentially similar, but slower, method, the prolonged neutron irradiation of $^{239}\mathrm{Pu}$ [39]. Atterling, Forsling, Holm, Melander and Astrom had reported the preparation $^{250}\mathrm{Fm}$ by the heavy ion reaction [40] $^{238}\mathrm{U}(^{18}\mathrm{O}, 4n)^{250}\mathrm{Fm}$. The latter experiment was one of the first successful applications of heavy ion bombardment in this area.

Subsequently, macroscopic amounts of all these elements up to einsteinium have been made.

Mendelevium

The thermonuclear weapon data indicated that fermium probably represented the practical limit to the use of repeated neutron capture to obtain actinide elements. In addition it was to be expected that elements with $Z > 100$ would, in general, show still shorter half-lives.

Experiments were then designed in which the recoil associated with the nuclear reaction generating the actinide would eject it from the target foil on to a catcher foil, thus simplifying the isolation of the product. An $^{253}\mathrm{Es}$ target containing only about 10^9 atoms of the actinide element was then bombarded with 40 MeV helium ions in the Berkeley 60" cyclotron. The recoiling product was caught on a gold foil, dissolved and subjected to an ion exchange separation. The fractions of the eluant that could be expected to contain the different actinide elements, including the unknown element 101, could be predicted. No alpha activity was found in the expected 101 fraction, but spontaneous fissions were observed. Eventually Ghiorso, Harvey, Choppin and Seaborg were able to show that the target reaction was $^{253}\mathrm{Es}(\alpha, n)^{256}101$.

The product decayed by orbital electron capture with a half-life of about 1.5 h, giving $^{256}\mathrm{Fm}$ which decays by spontaneous fission with a half-life of 160 min. They called the element mendelevium.

After the elegant and unimpeachable discovery of mendelevium, the discoveries of the two remaining actinides have rather confused histories. The lack of target materials of sufficiently high Z and A values in the required amounts meant that recourse had to be made to heavy ion bombardments. Recoil separations continued to play an important part. Only a few atoms of the new elements were necessary for identification.

Nobelium and Lawrencium

The first report of an isotope of element 102, a species emitting 8.5 MeV alpha particles with a half-life of about 10 min [41, 42], has been discredited by subsequent work. However, in 1958 Ghiorso, Sikkeland, Walton and Seaborg reported [43] the formation of ^{254}102 by the reaction ^{246}Cm(^{12}C, 4n)^{254}102. Their target material in fact contained 95% ^{244}Cm. The ^{254}102 recoiling from the target foil was carried by helium gas to a moving belt. The product from a second recoil, due to alpha decay of material on the belt, was collected on a negatively charged foil and shown to be the known 30 min ^{250}Fm.

From the distribution of the recoils and the speed of the belt, the ^{254}102 was deduced to have a half-life of ~3 s. Later it was shown to emit 8.3 MeV alpha particles. The ^{250}Fm formed by decay was also characterized by ion exchange [44]. Spontaneous fission was also observed.

At the second 'Atoms for Peace' conference at Geneva (1958) Flerov reported some preliminary results on an isotope of 102 and a more detailed account of this work followed in 1960 [45]. An isotope was produced by the reaction ^{241}Pu(^{16}O, 3 or 4n)$^{253/4}$102. The product emitted 8.8 MeV alpha particles with a half-life of 2–40 s. It is now very probable that this product was heavily contaminated with ^{211m}Po which emits 8.87 MeV alphas and might be formed from lead impurity in the target material [^{208}Pb(^{16}O, 3n)^{211m}Po] [46].

In 1961 Ghiorso, Sikkeland, Larsh and Latimer reported an isotope of 102 obtained during their search for element 103. It was obtained by the reaction $^{250-252}$Cf(^{11}B, pxn)$^{255?}$102. The product had a half-life of 15 s and emitted 8.2 MeV alphas [47].

Donets, Shchegolev and Ermakov, working at Dubna using the 300 cm cyclotron, reported in 1964 the production of ^{256}102 formed by ^{238}U(^{22}Ne, 4n) [46] and using a double recoil technique separated and identified the 25 h ^{252}Fm formed by alpha decay of the ^{256}102. A half-life of 8 s was deduced for the ^{256}102 from these experiments. The same species was made by ^{242}Pu(^{18}O, 4n) [48].

In 1965/66 Donets and collaborators obtained ^{254}102 by the reactions ^{238}U(^{22}Ne, 6n)^{254}102 and ^{243}Am(^{15}N, 4n) and showed that its half-life was 50 ± 10 s [49]. In another group [50] the same isotope was made and separated by recoil into a gas jet. The alpha energy was shown to be 8.10 = 0.05 MeV and the half-life 30–40 s.

Subsequent work at Berkeley and Dubna has identified and characterized the isotopes of mass numbers from 251 to 257; they are all pure alpha emitters.

A =	251	252	253	254	255	256	257
$t_{1/2}$	0.8	2.3	102	55	186	3.1	26 (s)
E_α	8.60	8.42	8.01	8.10	8.12	8.44	8.22 (MeV)

It would appear that the early Berkeley experiments produced isotopes of mass numbers 254 and 252, but the half-life obtained for the former was quite wrong. The ^{252}102 probably arose from the ^{244}Cm in the target material.

A well documented account of the discovery of this element has been given by Ghiorso and Sikkeland [51]. It has been decided to keep the name nobelium, given by the earlier unsuccessful investigators [41, 42], for this element.

The discovery of element 103, lawrencium, is also a rather complex story. In 1961 Ghiorso, Sikkeland, Larsh and Latimer [47] bombarded a californium target with boron ions and reported the formation of an isotope of 103 with a half-life 8 ± 2 s emitting alpha particles of 8.6 MeV. The attribution of a mass number was complicated by the target containing $^{249-251}$Cf as well as ^{252}Cf. Consideration of yields with ^{10}B and ^{11}B bombardments suggested ^{257}103. The authors proposed the name lawrencium. Sometime later it was suggested that ^{258}103 was a more likely assignment [52].

Meanwhile in 1965, Donets, Shchegolev and Ermakov [53] reported the production of ^{256}103 formed by the reaction ^{243}Am(^{18}O, 5n). It was reported to have a half-life of about 45 s. Both *alpha* emission and orbital electron capture decay were observed. This claim was well substantiated by the detection of the 25 h ^{252}Fm emitting 7.04 MeV alpha particles, formed by the decay chain

$$^{256}103 \xrightarrow{\alpha} {}^{252}\mathrm{Md} \xrightarrow{\text{o.e.c.}} {}^{252}\mathrm{Fm}$$

o.e.c. = orbital electron capture

In a later paper Flerov *et al.* [54] reported that this ^{256}103 had a complex alpha spectrum with the strongest line at 8.42 MeV. The half-life was corrected to 35 s. In the same paper they reported a similar half-life for ^{257}103 with 8.5–8.6 MeV alphas. Their ^{257}103 was made by the reaction ^{243}Am(^{18}O, 4n)^{257}103. Three years later ^{255}103 was reported by Druin [55]. It had a half-life of 20 s and an alpha energy of 8.38 MeV.

Later work at Berkeley clarified this situation [56]. ^{257}103 was shown to have a short half-life (0.65 s) but ^{258}103 has a half-life of 4.2 s and emits 8.6 MeV alphas. The current data are:

$A =$	255	256	257	258
$t_{1/2}$	21.5	25.9	0.65	4.35 (s)
E_α	8.37	8.43	8.86	8.59 (MeV)

It would appear that the Ghiorso group obtained $^{258}103$ in 1961 but that their half-life was erroneous. The Donets group obtained $^{256}103$ in 1965. The alpha energy reported by Flerov *et al.* is substantially that currently accepted but the half-life was overestimated.

The actinide group is now complete.

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