



## Review of the Accomplishments and Promise of U.S. Transplutonium Research, 1940-1981 (1982)

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**A Review of the Accomplishments and Promise  
of U.S. Transplutonium Research — 1940-1981**

**Prepared under the Auspices of the  
Subcommittee on Nuclear and Radiochemistry  
Committee on Chemical Sciences  
Assembly of Mathematical and Physical Sciences  
National Research Council**

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**NOTICE:** The project that is the subject of this report was approved by the Governing Board of the National Research Council, whose members are drawn from the Councils of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine. The members of the Committee responsible for the report were chosen for their special competences and with regard for appropriate balance.

This report has been reviewed by a group other than the authors according to procedures approved by a Report Review Committee consisting of members of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine.

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## PREFACE

The Subcommittee on Nuclear and Radiochemistry is organized under the Committee on Chemical Sciences to maintain awareness of the chemical aspects of nuclear phenomena, to stimulate scientific and technical progress and to identify opportunities for research. The remarkable progress in the study and utilization of the actinide elements during the past 40 years has been reflected in several monographs and meetings sponsored by the SNR. This seemed an appropriate time to prepare a brief review of the many achievements as well as of the promise of further progress in this vital field of nuclear and radiochemical research.

On behalf of the SNR, Dr. R. G. Wymer and Dr. O. L. Keller contacted a number of scientists from DOE and university laboratories to provide brief summaries which were then collated and edited for the final report. The SNR is grateful to all of these scientists for their role in documenting this record of significant accomplishments. Without the support of the AEC, ERDA and DOE for actinide research, these accomplishments could not be recorded as most would not have occurred.

G. R. Choppin, *Chairman*

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## I

### INTRODUCTION

It has been just over 40 years since the identification of plutonium by Seaborg, McMillan, Kennedy, and Wahl at the University of California, Berkeley. This discovery, and the isolation of plutonium-239 a year later by Kennedy, Seaborg, Segré, and Wahl, laid the cornerstone of one of the most exciting and productive eras of research in American history — research on the transplutonium elements. This research was to span all fields of natural science, and indeed was to have many implications and applications in the biological sciences. In this brief document, we have attempted to touch on only some of the many achievements of the U.S. heavy element program. Many of those whose work has been at the heart of this research have contributed to this report. They have summarized some of what was scientifically important and exciting and they have suggested some of the topics of important research remaining to be done.

Out of this research has come new insight into the basic structure and stability of the nucleus itself. New understanding of the organization of elements in the periodic table — the single most important conceptual tool in all of chemistry — has been gained with the discovery of 15 new elements and the production of weighable amounts of them up through einsteinium. Long-lived isotopes such as  $^{248}\text{Cm}$  and  $^{249}\text{Cf}$  made available through the High Flux Isotopes Reactor (HFIR) and Transuranium Processing Plant (TRU) production program serve as targets in heavy ion accelerators to produce still heavier elements up through atomic number 107, as well as for studies on themselves. Chemical studies have been possible on elements up to the 104-105 region. These elements give us a last chance to understand the symmetry of the periodic table in this region of filling of 5f and 6d electronic orbitals in which significant electronic relativistic effects can occur. Indeed, the studies in the transplutonium region of chemical properties, spectroscopic and magnetic properties, superconductivity, new synthetic routes to metals, alloys, and compounds, thermodynamic and structural studies at extremes of temperatures and pressures, aqueous and non-aqueous chemistry — all have enriched the field of inorganic and physical chemistry in an unprecedented way.

Yet there are many exciting frontiers for today and tomorrow. For example, production of such isotopes as  $^{246}\text{Cm}$  and  $^{250}\text{Cf}$  will provide important possibilities in new nuclear structure and reaction studies.

The needed research to map the structure of the periodic table in the critical region where the actinide series ends (element 103) and the new 6d transition series begins (element 104) has already been mentioned. The discovery of superconductivity in americium suggests new frontiers for exploring important phenomena, including the effects of electronic density of states at the Fermi surface varied by high pressures. No less important is the opportunity to advance our understanding of fission, in which great strides have been made in the last five or six years, showing that it is extremely important to study fission properties of the heavier isotopes of fermium (element 100) and mendelevium (element 101) and, when possible in the future, neutron-rich isotopes of still higher Z in order to understand nuclear fission and the limits of nuclear stability.

Practical uses such as medical applications, radiography, oil well logging, and reactor start-up have been developed for  $^{252}\text{Cf}$ . It is to be expected that additional uses will be found for this isotope, as well as others.

Selected references have been included by some authors of sections of this document; others have included bibliographies. These by no means exhaust the extensive literature in the field of heavy element research, but rather are indicative of the significance of the research that has made the United States the world leader.

Because of the preeminence that U.S. scientists have achieved in fields of research utilizing the isotopes made available to them through the U.S. Transplutonium Production Program, their active collaboration has been sought by premier research groups in nuclear and actinide chemistry and nuclear physics, such as those at Mainz, GSI, Karlsruhe, Heidelberg, Munich, Liege, Orsay, Fontenay-aux-Roses, Grenoble, and Dubna. This worldwide reputation has been achieved and maintained by U.S. scientists primarily because they have had access to these unique materials from the HFIR/TRU and because the scientists in the field have exhibited skill, ingenuity, and imagination in using these isotopes to explore the nuclear and chemical properties of the heaviest elements. This is not a resource that can be stockpiled for the future since many of the isotopes are relatively short-lived. It is consequently of greatest importance to continue producing this resource that has proved so fruitful in the past and that holds so much promise for future contributions.

## II

### HISTORICAL ACCOUNT

We have just passed the fortieth anniversary of the synthesis and identification — i.e., the "discovery" — of the first transuranium elements, neptunium and plutonium. The intervening years have seen the addition of a dozen transplutonium elements whose nuclear synthesis (with two exceptions) has been based on the use of source isotopes produced by neutron irradiation in nuclear reactors. (The thirteenth and last of the known transplutonium elements, atomic number 107, was synthesized by another method.)

Thus, the addition of the transuranium elements to mankind's natural heritage of elements has led to an expansion by about 15% in the fundamental building blocks of nature. Investigation of these man-made elements has led to a tremendous expansion of our knowledge of atomic and nuclear structure. Of particular interest are the unusual chemistry of these elements, their impact on the organization of the periodic table and their sometimes unique nuclear properties.

A key to the leadership of the United States in the transplutonium field has been the widespread availability of the long-lived isotopes in macroscopic quantities as a result of their production by neutron irradiation in nuclear reactors.

Availability of transplutonium isotopes to a limited group of investigators began as early as near the end of World War II, when the nuclear reactors at Clinton Laboratories (Oak Ridge) and Hanford were used. Use of these reactors for this purpose continued for a few years after the war. In the late 1940's the high flux NRX reactor at the Chalk River Laboratory in Canada was made available to U.S. investigators to make possible substantially higher yields of heavier isotopes from multiple neutron capture reactions.

When the newly built Materials Testing Reactor (MTR) at ARCO, Idaho, was made available in 1952, an interlaboratory program for neutron irradiation of plutonium on a large scale led to a substantially augmented supply from this source for the next decade.

The U.S. Transplutonium Production Program got under way in 1959 with neutron irradiation of plutonium in Savannah River reactors. This led to a coordinated program involving chemical extraction facilities at Savannah River and Oak Ridge and the Oak Ridge Research Reactor (ORR) in order to maximize the output of a number of products

of special interest because of their practical importance. This program expanded as the possibilities for very useful practical applications of such isotopes as  $^{244}\text{Cm}$  and  $^{252}\text{Cf}$  came to be more widely recognized. Possibly of greater importance was the growing understanding of the scientific importance of having an adequate supply of isotopes to make it possible for U.S. scientists to study the nuclear properties and reactions, chemical properties, optical properties, and solid state properties of this interesting group of transition elements.

Recognition of the potential importance of this field of research led to the building, at Oak Ridge National Laboratory, of the High Flux Isotopes Reactor (HFIR) and its sister facility, the Transuranium Processing Plant (TRU). These started to operate in 1966, using as input the products from the Savannah River transplutonium programs, and have continued to operate ever since as the mainstay of the U.S. Transplutonium Production Program. An important part of the program has been the operation of Electromagnetic Separators to separate individual isotopes from elemental mixtures of isotopes. The program has been characterized from the beginning by a high degree of interlaboratory cooperation, and this was formalized in 1964 by the creation of a Transplutonium Program Committee consisting of representatives from the Argonne National Laboratory, Oak Ridge National Laboratory, Lawrence Radiation Laboratory, Livermore Laboratory, Los Alamos Scientific Laboratory, Savannah River, and Brookhaven National Laboratory. This committee, which has played an important advisory role in directing the program, is still active. The transplutonium isotopes have been made available to laboratories since 1961.

### III

#### PLUTONIUM

This report is about the elements beyond plutonium – the transplutonium elements. Yet plutonium is where our story begins. All the elements of higher atomic number yet to be discovered were to be synthesized and investigated in ways that were foreshadowed by the approaches developed in the early days at Berkeley when plutonium itself was discovered and in those intense days at the beginning of the Manhattan Project when the nuclear and chemical properties of plutonium first began to unfold.

The very sociology of the transplutonium research community was established in those early days of plutonium research. For example, of its very nature, plutonium discovery, production, and research demanded the complementary talents and cooperation of teams of physicists and chemists. The invention at Berkeley by E. O. Lawrence of the cyclotron brought together an inspired group of physicists and chemists devoted to studying properties of nuclei and to discovering new elements and isotopes. These associations led to the discovery of plutonium by Seaborg, McMillan, Kennedy, and Wahl late in 1940 and early in 1941. The 60-in. cyclotron at Berkeley furnished a 16-MeV beam of deuterons for bombarding a  $U_3O_8$  target to produce  $^{238}Np$ , which decayed to  $^{238}Pu$ . The vanishingly small amount of plutonium produced had to be chemically separated in order to investigate its nuclear characteristics and identify it as a distinctly new element. Since the chemical properties of plutonium were unknown at the time and its supposed placement in the periodic table was actually erroneous, considerable chemical ingenuity was needed to develop these separation procedures.

As a result of tracer level experiments on cyclotron-produced plutonium, it was soon shown that plutonium had at least two oxidation states. The speculation arose that the higher oxidation state might be like U(VI) and the lower like U(IV) and Th(IV). These were the earliest signs pointing towards Seaborg's later pivotal development of the actinide concept, which reshaped the periodic table.

The critical measurement of the fission cross section of the newly discovered  $^{239}Pu$  was made at Berkeley in 1941 by Kennedy, Seaborg, Segré, and Wahl. These measurements established  $^{239}Pu$  as a potential source of nuclear energy like  $^{235}U$ . Therefore, a wartime Plutonium Project was set up to produce and separate plutonium on a large scale for weapons. The large-scale production of  $^{239}Pu$  would have to be from

the neutron irradiation of  $^{238}\text{U}$  in a reactor. The development of the technology of the chain reaction producing excess neutrons from the fission of  $^{235}\text{U}$  to allow such production was largely the domain of the physicists. The separation of pure  $^{239}\text{Pu}$  found at the level of 250 parts per million or less in the discharged irradiated uranium plus fission products was the domain of the chemists.

In 1942, working with cyclotron-produced plutonium, Stan Thompson of Seaborg's group made the unexpected discovery that Pu(IV) is carried quantitatively by bismuth phosphate in acid solution. In a very short time, the "Bismuth Phosphate Process" for separating plutonium was developed to the pilot plant stage using reactor-produced plutonium at Oak Ridge and then scaled up at Hanford to industrial scale.

The development of ultramicrochemical methods for investigations of plutonium was a distinguishing characteristic to be carried forward to the transplutonium elements as they were discovered. The methods developed by Burris B. Cunningham and co-workers in the early 1940's led to the preparation of the first pure compound of plutonium and to the elucidation of many of the new element's chemical properties.

The scientific literature on plutonium is vast, and no attempt has been made in this document to summarize the highlights of plutonium research. However, it has been from the beginning, and continues to be, a key element in the heavy element story.

#### IV

#### SYNTHESIS OF TRANSPLUTONIUM ISOTOPES

The nuclear synthesis of nearly all of the isotopes of the elements beyond plutonium has depended on the availability of target nuclei produced by the neutron irradiation of precursor isotopes. The neutron irradiations to produce such nuclei were performed in the Oak Ridge (Clinton) and Hanford reactors in the 1940's, in the NRX reactor at the Chalk River Laboratory in Canada in the late 1940's, and in the Materials Testing Reactor (MTR) at Arco, Idaho, in the 1950's. This led to the production and identification of about 50 transplutonium nuclear species covering the atomic number range through 103 and mass number range through 257, including those produced through neutron irradiation (the heaviest has the mass number 257) and those produced through bombardment of the neutron-produced nuclei by charged particles. (A number of these were identified for the first time as a result of their production in the "Mike" thermonuclear device test staged in November of 1952, but their production in larger amounts followed through the use of the MTR.)

Neutron irradiations in the Savannah River reactors and the High Flux Isotopes Reactor (HFIR) at the Oak Ridge National Laboratory, followed by chemical separations in the attendant extraction facilities, led to the large-scale production of transplutonium isotopes in the 1960's and 1970's. The use of such isotopes as target materials for bombardments with charged particles, especially heavy ions, allowed the synthesis and identification of three more elements and an additional 50 transplutonium isotopes (the vast majority of which were first identified in the United States as a direct result of the U.S. Transplutonium Production Program).

Since it is not feasible to describe the discovery of 100 nuclear species, this account will be confined to very brief descriptions of the discovery of the first isotope of each transplutonium element.<sup>2</sup>

The first transplutonium elements, americium (No. 95), and curium (No. 96), were discovered<sup>14</sup> in 1944-1945 at the wartime Metallurgical Laboratory of the University of Chicago. Curium was synthesized first as the result of the bombardment of newly available  $^{239}\text{Pu}$  with helium ions in the Berkeley 60-in. cyclotron, and americium was synthesized as the result of neutron irradiation of  $^{239}\text{Pu}$  in the reactors at Clinton Laboratories (Tennessee) and Hanford (Washington). The key to their chemical identification was the recognition<sup>15</sup> that the transplutonium

elements were part of an actinide transition series (second rare earth series), which was also the key to the chemical identification of the following elements.

Berkelium (No. 97) was discovered<sup>13</sup> as the result of the helium ion bombardment of  $^{241}\text{Am}$ , produced through the irradiation of plutonium in Hanford reactors, and californium (No. 98) was discovered<sup>12</sup> following the helium ion bombardment of curium, produced by the irradiation of americium with neutrons in the NRX reactor. The bombardments (60-in. cyclotron) and chemical identification took place at Berkeley in late 1949 and early 1950.

Einsteinium (No. 99) and fermium (No. 100) were discovered<sup>10</sup> in the debris from the "Mike" test in late 1952 and thus did not involve the use of targets produced by neutron irradiation.

Mendelevium (No. 101) was first produced<sup>11</sup> as the result of the helium ion bombardment (in the Berkeley 60-in. cyclotron) of a minute amount (approximately  $10^{-6}$  microgram) of  $^{253}\text{Es}$ , which was produced as the result of neutron irradiation in the MTR. This was the first instance of an identification (chemical in this case) of a new element on the "one-atom-at-a-time" basis.

Nobelium (No. 102) was the first transplutonium element to be discovered<sup>9</sup> through the use of heavy ions. Curium, produced by neutron irradiation in the MTR, was bombarded with  $^{12}\text{C}$  ions in the newly constructed Heavy Ion Linear Accelerator (HILAC) at Berkeley. The actinide transition series was completed with the synthesis and identification<sup>8</sup> of lawrencium (No. 103) by the bombardment of californium, produced by neutron irradiation in the MTR, with boron ions in the HILAC.

Although there are competing claims for the discovery of the next three transplutonium (which are transactinide) elements, criteria for the discovery of elements defined by an international group of experts<sup>4</sup> indicate that the discoveries should be credited to the U.S. teams.\* On this basis, element 104 was first identified<sup>7</sup> as the result of carbon ion bombardment in the Berkeley HILAC of monoisotopic  $^{249}\text{Cf}$  furnished by the Oak Ridge National Laboratory. Similarly, element 105 was identified<sup>6</sup> through the nitrogen ion bombardment of the same  $^{249}\text{Cf}$  target, and element 106 (unnamed as yet) was identified<sup>5</sup> by the oxygen ion bombardment of additional  $^{249}\text{Cf}$  furnished by the Oak Ridge National Laboratory.

On the basis of the knowledge we have gained about the yields and mechanisms of heavy ion reactions for the production of actinide and transactinide isotopes by the bombardment of the available supply of transplutonium target nuclei, we can be assured that a continued and expanded supply will lead to the discovery of many new isotopes and quite possibly to new elements. The possible approaches are rich and varied. One method, which is applicable with the more widely available lighter heavy ions, is that of transfer reactions; these lead to a wide variety of product nuclei extending in mass numbers over the entire

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\*At present, International Union of Pure and Applied Chemistry (IUPAC) has not officially accepted any discovery claim for the elements, and no official names have been designated.

region from that of the target nucleus to that of the compound nucleus corresponding to the sum of the mass numbers of the target nucleus and the heavy ion projectile.<sup>1</sup> Another reaction mechanism is that of deep inelastic scattering in which the heaviest projectiles (uranium ions are a good example) transfer large numbers of neutrons and protons to the target nucleus.<sup>3</sup> Perhaps the compound nucleus mechanism, in which the heavy ion projectile amalgamates with the target nucleus resulting in a potentially interesting product after the loss of excitation energy by particle evaporation, holds the most promise. Each of these mechanisms can only lead to observable actinide or transactinide product nuclei if a sufficient fraction of them survives loss by the prevalent competitive fission reaction. An especially intriguing goal is the production and identification of "superheavy elements" (composed of nuclei stabilized by closed shells of 114 protons and/or 184 neutrons); the compound nucleus and the inelastic transfer mechanisms offer hope for reaching this goal.

A particularly hopeful approach for reaching both superheavy and new isotopes in the actinide and near transactinide region is through the use of the heaviest potentially available isotope,  $^{254}\text{Es}$ . Use of this as a target nucleus might lead to the production and identification of new isotopes by any of the three heavy ion reaction mechanisms.

Also of special value as target nuclei for this purpose are the isotopes of californium and curium. Particularly useful would be enriched  $^{251}\text{Cf}$  (i.e., separated as much as possible from the difficult-to-handle spontaneously fissioning  $^{252}\text{Cf}$ ) and the ultrarare, long-lived  $^{250}\text{Cm}$ .



## V

### NUCLEAR FISSION AND STABILITY, HEAVY ION REACTIONS, AND NUCLEAR STRUCTURE

#### A. NUCLEAR FISSION AND STABILITY

As described earlier, isotopes of the transplutonium elements were produced and made available to researchers through the U.S. Transplutonium Production Program (TPP) beginning in 1959. As a result it became possible to study the spontaneous fission (SF) of the isotopes of elements beyond plutonium, including relatively short-lived rare isotopes such as  $^{255}\text{Es}$  (40 days) and  $^{257}\text{Fm}$  (100 days). Because of the unique availability of such isotopes in this country, American researchers soon took the lead in the measurement of spontaneous fission properties<sup>47,50</sup> even though spontaneous fission had first been discovered by the Russian scientists Petrzhak and Flerov<sup>51</sup> in 1940. Early studies of spontaneous fission were hampered by the low specific activity of the available nuclides, e.g.,  $^{238}\text{U}$  (SF half-life =  $10^{16}$  years),  $^{240}\text{Pu}$  (SF half-life =  $10^{11}$  years), but with the increasing availability of high specific activity sources of the transplutonium isotopes and the development<sup>50</sup> of solid-state detector techniques for measuring fission fragment kinetic energies, information concerning details of mass, charge, and kinetic energy distributions and neutron and photon emission was obtained. These results illustrate one answer to the question: What can one study experimentally in the transplutonium region of nuclei with stable deformation that cannot be studied, or is very difficult to study, in more accessible regions of the chart of the nuclides?

Until about 1970, it was believed that all low-energy fission resulted in very asymmetric mass division and that the SF properties of heavier nuclides could readily be extrapolated from the known systematics of lighter nuclides. However, with the report<sup>47</sup> in 1971 of much enhanced yields of symmetric mass division accompanied by unusually high total fragment kinetic energies in the SF of  $^{257}\text{Fm}$ , a "renaissance" of interest in SF and low-energy fission occurred. (A review of post-fission phenomena studied by the end of 1973 is given in reference 45. Of the 227 references cited, nearly half were dependent on isotopes furnished through the U.S. Transplutonium Production Program.)

The wide availability of high specific activity sources of  $^{252}\text{Cf}$  to researchers outside as well as inside the United States has also been an important factor in promoting detailed studies and an understanding of the fission process using sophisticated instrumental and radiochemical techniques. With the increasing availability of sources of other transplutonium isotopes, studies of fission yields from SF and thermal neutron-induced fission of additional isotopes have been made.<sup>3,5,31,45</sup>

Since 1973, information concerning the SF of the short-lived isotopes  $^{256}\text{Cf}$ ,  $^{258}\text{Fm}$ ,  $^{259}\text{Fm}$ ,  $^{259}\text{Md}$ ,  $^{252}\text{No}$ ,  $^{259}\text{104}$ , and  $^{262}\text{105}$  has been obtained. All of these studies were directly dependent on isotopes furnished by the Transplutonium Production Program. These new data permitted a comparison by Hoffman<sup>28</sup> of the data for the fermium isotopes with both heavier and lighter nuclides. This 1979 review lists some 54 references, and the productivity of the field is shown by the fact that 40 of these were published after 1973. Because of these studies, a reassessment of the theory of low-energy fission has been necessary, and new attempts have been made to develop a comprehensive, dynamic theory of fission that can explain the descent from saddle to scission point, nuclear viscosity, and the time scales involved. It appears that the properties of the heavy fermium isotopes are unique and can qualitatively be explained on the basis of fragment shell effects due to the products of symmetric fission, which are near to the doubly magic  $^{132}\text{Sn}$  configuration. No longer can it be said that all low-energy fission results in highly asymmetric mass division while high-energy fission results in symmetric mass division. Nor can the properties of heavier nuclides be smoothly extrapolated from those of the lighter isotopes. These results have also had a strong effect on our ideas concerning nuclear stability and the properties of a possible island of superheavy elements around atomic numbers 109 to 114. It should be noted that elements through 107 have now been produced, so that region does not seem so far away as it once did.

In addition to the properties of the fission fragments, the systematics of the stability of the heaviest isotopes toward SF have also been of interest.<sup>10</sup> The availability of  $10^9$  atoms or so of  $^{257}\text{Fm}$  made it possible to produce 0.38-ms  $^{258}\text{Fm}$ , which signaled rapid decrease of SF half-lives for the heavier fermium isotopes. It also made it possible to produce<sup>43b</sup> 1.5-s  $^{259}\text{Fm}$ , the most neutron-rich isotope so far known. Use of the exceedingly rare target, 60-d  $^{254}\text{Cf}$ , produced under the TPP program, resulted in the first preparation<sup>20</sup> of 12.3-min  $^{256}\text{Cf}$  (containing the same number of neutrons as  $^{258}\text{Fm}$ ) for study of its SF properties. Its half-life showed that the SF "disaster" did not set in until  $Z = 100$  and its fission properties were similar to those of lighter isotopes. The only other  $N = 158$  nuclide known is 95-min  $^{259}\text{Md}$ , produced by bombardment of  $^{248}\text{Cm}$  with  $^{18}\text{O}$ . Its half-life and properties suggest changes in stability and properties relative to fermium, perhaps due to the effect of the odd proton in Md. The SF properties of  $^{259}\text{Md}$  are quite unusual inasmuch as it fissions symmetrically but with a total kinetic energy that

is not anomalously high as in the case of  $^{258}\text{Fm}$  and  $^{259}\text{Fm}$ . The width of the total kinetic energy distribution is unusually large, perhaps indicating a range of fragment shapes at scission from spherical to highly distorted. It was suggested that the relatively low total kinetic energy might be due to the emission of a  $Z = 1$  particle at scission, but Hulet et al.<sup>25</sup> showed that this was not the case. Another possibility is that because of the odd proton, one of the fragments is highly distorted and the number of prompt neutrons emitted is much larger than for  $^{259}\text{Fm}$ , thus accounting for the additional energy. Neutron emission in the SF of Cf and Fm isotopes has been investigated, and it was found<sup>23</sup> that for the highest total kinetic energy events the average prompt neutron emission was less than 1. It is extremely important to measure the fission properties of still heavier Md isotopes and to measure prompt neutron emission for the SF of both the heaviest Fm and Md isotopes. Again, these studies are vital to our understanding of nuclear fission and the limits of nuclear stability. It will be imperative in the future to examine the properties of the neutron-rich isotopes of other elements with  $Z$  greater than 100 if we are to understand these phenomena.

Fission barrier parameters for a host of heavy element isotopes have been deduced from experiments utilizing heavy element targets and such charged particle fission reactions<sup>22</sup> as  $^{254}\text{Es}(t, \text{pf}; d, \text{pf}; ^3\text{He}, \text{df})$ . Britt<sup>27</sup> reviewed the status of such measurements at the 4th International Conference on the Physics and Chemistry of Fission in Jülich in 1979. Extension of such studies is dependent on target availability. Transplutonium targets have also made it possible to extend the investigation of fission isomers into the transplutonium region.<sup>42,46</sup>

Coulomb fission studies have been carried out by Backe et al.<sup>13</sup> using  $^{184}\text{W}$  and  $^{238}\text{U}$  beams on heavy targets including  $^{248}\text{Cm}$ . This method can be a valuable probe of the fission mechanism because a purely electrostatic interaction can be selected. The current results are not in complete agreement with theory, and further studies are needed.

## B. HEAVY ION REACTIONS

In the past, the major impetus for the study of nuclear reactions using the transplutonium isotopes has been the desire to produce, identify, and study the properties of new heavy isotopes and/or new elements. Recently, however, considerable interest in studying the various interaction mechanisms themselves has been developing, and many theoretical as well as experimental studies are emerging that link observations in this region to those for lighter elements. In addition, it now appears that heavy-ion transfer reactions (Lee et al., ref. 1, section on Synthesis/Transplutonium Isotopes), as well as deeply inelastic and compound nucleus reactions, offer hope for producing new heavy elements and isotopes with relatively low excitation energies so as to avoid complete loss by prompt fission. This field appears particularly fruitful for future exploitation in making new isotopes for systematic studies of their properties and the limits of stability.

In the area of production of heavy element isotopes, one of the classic early articles is that of Sikkeland et al.,<sup>49</sup> which gives excitation functions for the synthesis of nobelium isotopes in bombardment of  $^{244,246,248}\text{Cm}$  targets with  $^{12}\text{C}$  and  $^{13}\text{C}$ . They pointed out that the cross sections depend critically on the value of the ratio of the partial widths for neutron emission and fission, and they gave values of these ratios as a function of  $Z$  and  $A$  for even trans-berkelium nuclides, that are still in use. In 1974, Hahn et al.<sup>44</sup> presented experimental data concerning transfer and compound-nucleus reactions leading to  $^{245}\text{Cf}$  and  $^{244}\text{Cf}$  in bombardments of  $^{239}\text{Pu}$  and  $^{238}\text{U}$  with  $^{12}\text{C}$  ions. They pointed out that noncompound processes (such as transfer of an aggregate from projectile to target followed by neutron evaporation) must be important. The cross sections for the  $(\text{C},\alpha 2\text{n})$  and  $(\text{C},\alpha 3\text{n})$  reactions were too high to be accounted for simply by the evaporation of charged particles, which is expected to be negligible in such heavy nuclei. They measured the recoil ranges and angular distributions as well as the cross sections and excitation functions of the products. There have been essentially no published reports of similarly thorough studies utilizing transplutonium targets, although a study of actinide yields from the reaction of  $^{248}\text{Cm}$  with  $^{16,18}\text{O}$  and  $^{20,22}\text{Ne}$  projectiles has recently been reported by Lee et al.<sup>19</sup> Some unpublished data on recoil ranges<sup>18</sup> and energies are available, and studies of excitation functions for heavy ions on  $^{248}\text{Cm}$  and  $^{249}\text{Cf}$  targets are in progress.<sup>19</sup>

### C. NUCLEAR STRUCTURE

Nuclear structure theories, whether fully self-consistent or based on Strutinsky's macroscopic-microscopic method, contain parameters whose values are adjusted to reproduce properties of known nuclei. By confronting predictions of such theories with experimental data on new nuclei, we are able to both test the underlying theories themselves and also better determine the values of the input parameters. The region of high- $Z$  short-lived isotopes is very critical in this respect because of the smooth variations of the liquid drop model associated with increasing charge and the rapidly changing single-particle effects. For example, small changes in the number of protons or neutrons can lead to large changes in spontaneous-fission and alpha-decay half-lives, a transition from asymmetric to symmetric fission-fragment mass distributions, and anomalous behavior in the average fission-fragment kinetic energy.

Some experimental probes are quite selective as to which nuclear states they populate, whereas others are not very selective but offer assurance of completeness, i.e., that all nuclear states of a certain range of angular momentum and parity values are being populated. In testing nuclear models, this question of completeness can be a very important feature.<sup>24</sup> One example of a nuclear probe that offers completeness is the resonance-averaged neutron capture gamma-ray spectroscopy, which can be performed with filtered neutron beams of 2 and 24 keV at Brookhaven National Laboratory.<sup>6d,26</sup> Another probe that is

24 keV at Brookhaven National Laboratory.<sup>6d,26</sup> Another probe that is reasonably complete is thermal neutron capture gamma-ray and conversion-electron spectroscopy. A very important feature of this kind of spectroscopy is that one obtains an extensive list of secondary gamma transitions along with multipolarities for a significant fraction of the total number of transitions.<sup>6b</sup> Just three nuclides,  $^{244}\text{Am}$ ,  $^{249}\text{Cm}$ , and  $^{250}\text{Bk}$ , have been investigated so far by use of a technique offering completeness, namely, neutron-capture measurements with the high-resolution spectrometers at the Institut Laue-Langevin in Grenoble, France.

One of the limitations in applying various experimental techniques is the question of target size. Measurements of transfer reactions, Coulomb excitation, and inelastic scattering require only micrograms of targets. Hence, these techniques are applicable to the widest range of transplutonic species. In the transplutonic elements, the only isotopes available in multi-gram quantities and in moderately good isotopic purity are  $^{241}\text{Am}$ ,  $^{243}\text{Am}$ , and  $^{244}\text{Cm}$ . In addition, gram amounts of multi-isotopic Cm mixtures (predominantly mass 244, although they may also contain significant amounts of mass 246) also can be produced. In the milligram range, the three isotopes  $^{248}\text{Cm}$ ,  $^{249}\text{Bk}$ , and  $^{249}\text{Cf}$  are extremely important products of the HFIR-TRU complex. Part of their importance is due to the fact they are produced in isotopically pure form by means of chemical manipulations, all of which can be done with very good efficiencies; isotopic purity does not have to be obtained via electromagnetic isotope separation with its attendant low efficiencies and high losses of material. It would be very desirable to have available supplies of isotopically pure isotopes such as  $^{245}\text{Cm}$ ,  $^{246}\text{Cm}$ ,  $^{247}\text{Cm}$ ,  $^{250}\text{Cf}$ , and  $^{251}\text{Cf}$  in quantities up to a few milligrams to permit the extension of certain nuclear structure studies. Of these, perhaps it is most important to produce as much as possible, up to several milligrams, of the two even-even species  $^{246}\text{Cm}$  and  $^{250}\text{Cf}$ . For production of  $^{246}\text{Cm}$ , some means of isotopic enrichment must be employed. The odd-mass members of this set are particularly difficult to produce in quantity due to their fissionability with thermal neutrons. Filtered neutron spectra are of some utility in trying to increase the effective production-to-destruction ratios of these odd-mass nuclei. Again, however, isotopic enrichment will be required.

Although essentially no nuclear structure studies have been done in the actinide region using in-beam gamma-ray spectroscopy at an accelerator, presumably because of severe interference from fission product gamma rays, it appears the  $(t, xn)$  reaction may prove useful. Maier et al.<sup>7</sup> in studying this question conclude that since neutron-rich nuclei have lower fissionability parameters, fission competition may not be too severe for in-beam gamma studies. Their statistical model calculations show that fission is a smaller fraction of the cross section of the  $(t, xn)$  reaction as compared with the  $(\alpha, xn)$  reaction targets of  $^{238}\text{U}$  and  $^{232}\text{Th}$ .

Experimental nuclear structure studies in the actinide region have been an important feature of recent conferences. For example, the 4th International Transplutonium Element Symposium<sup>43</sup> held at

Baden-Baden in September 1975 featured a session on the nuclear stability and synthesis of transactinide elements. E. K. Hulet<sup>43a</sup> reported on the discovery of element 106 and on the identification of  $^{259}\text{Fm}$ . R. W. Hoff<sup>43c</sup> reported on the level structure of  $^{249}\text{Bk}$  from  $^{253}\text{Es}$  alpha decay. Two symposia featuring the nuclear properties of the transplutonium elements took place in 1977 and 1980 at national meetings of the American Chemical Society. At the most recent meeting, the August 1980 meeting in Las Vegas, there were a number of papers presented on the production of heavy actinides and search for superheavy elements in heavy-ion reactions, including the experiments of Hulet and co-workers at GSI, Darmstadt, West Germany, using the near-ultimate combination of  $^{238}\text{U}$  projectiles and a  $^{248}\text{Cm}$  target.<sup>8</sup> Another paper in this symposium by Bemis et al.<sup>32</sup> reported the use of laser-induced nuclear polarization in a study of the  $^{240\text{m}}\text{Am}$  spontaneous fission isomer produced with the ORNL isochronous cyclotron.

At the two most recent international symposia on neutron capture gamma-ray spectroscopy, the latest held in September 1981 in Grenoble<sup>6</sup> and the other in September 1978 at Brookhaven,<sup>39</sup> several nuclear structure studies of actinide nuclei were reported. Among these papers were reports on the level structure of  $^{249}\text{Cm}$ ,  $^{250}\text{Bk}$ , and an interesting series of very precise measurements of actinide K x-rays.

The attached bibliography is not intended to be comprehensive, but rather to give examples of new work published since the last comprehensive review on a given topic. A computer search that selected only Nuclear Properties of the Transplutonium Isotopes yielded 472 references for the years 1975-1981 alone.

## VI

### CHEMISTRY OF THE TRANSPLUTONIUM ELEMENTS

The impact of transplutonium element studies on the chemical community is perhaps most clearly seen through how such studies have changed our understanding of the periodic system of the elements. The formulation of the periodic system of the elements not only gave chemistry an incomparably useful systematics but pointed the way for other fields to develop in analogous ways. [A recent example is "the eightfold way" patterns of elementary particles that are explained by the quark model.] The significance of the periodic system to chemical sciences in particular, and to human thought in general, warrants our understanding it thoroughly. A major contribution of transplutonium element research over the last 35 years has been the substantiation of Seaborg's actinide hypothesis, which reformulated the symmetry of the periodic system and brought out new truths about the electronic systems of the elements that cause periodicity in their properties. In 1944, Seaborg boldly moved thorium, protactinium, and uranium from their long accustomed places in the periodic table to an "actinide" series of heavy, mostly undiscovered elements that would be analogous to the well-known "lanthanide" series. The properties of this new series would be dictated by the filling of a 5f shell of electrons so that the elements would resemble the 4f lanthanides. This was a controversial proposal for many years since Th, Pa, and U were generally considered to be the first three members of a 6d series rather than a 5f. The confusion occurs because the 5f and 6d electrons in the early actinide series have similar energies and the chemical properties of these elements are not governed by the electronic configurations in a simple way as in the 4f lanthanide series. By elements 95 and 96, however, the rare-earth-like character of the actinides becomes prominent. Using his actinide hypothesis, Seaborg correctly predicted the chemical properties of elements 95 and 96, which was the key to their discovery in 1944. Similarly, Seaborg predicted pertinent chemical properties needed for the discovery of the rest of the actinide series through element 103.

A consequence of the actinide hypothesis is that the 6d series of transition elements should start at element 104. Experimental evidence gathered so far supports this conclusion. Further research into the chemical properties of elements 104, 105, and 106 is important in defining whether the remarkable symmetry of the periodic table will persist into this highly relativistic region.

In keeping with Seaborg's reformulation of the periodic table, the transplutonium elements (with varying numbers of 5f electrons), in general, are found to be closely related in chemical behavior to the lanthanide elements (with varying numbers of 4f electrons). The differences in the chemistry of the two groups of elements arise mainly from less effective shielding by outer electrons and lower binding energies of the 5f electrons of the actinides relative to the 4f electrons of the lanthanides. As a result, the chemistry of the transplutonium elements is more diverse than that of the analogous lanthanides.

One example of this greater diversity is the large number of oxidation states in which the actinides can be found. Oxidation states of II through VII are known in aqueous solution for different transplutonium elements. Americium(VI), for example, in phosphotungstate solution, reduces so slowly that Am(VI), Am(V), Am(IV), and Am(III) can all be present for over 20 hours. As with the lanthanides, the III oxidation state is the most common for the elements Am through Lw (except No). However, for Am through Cf, the IV state is stable enough to be used in separations. At the high Z end, No(II) and Md(II) must be studied by fast, radiochemical techniques since the nuclear half-lives are very short. It should be noted that No is the only element in the lanthanide and actinide series that is most stable in the divalent state.

Although there is evidence of some covalent interaction in the bonding of transplutonium elements, their bonding is strongly electrostatic. As a result, these elements are useful in testing models of ionic interaction. Also, due to the ionic nature of the bonding, the number and geometry of the bonding groups around the transplutonium metal cation are not determined by the necessity for orbital overlap. Coordination numbers from 4 to 12 are known with a corresponding diversity of symmetries. Obviously, such systems can provide us with a wealth of information on electrostatic interactions in a wide variety of conditions. The transplutonium cations of oxidation state (III) have been studied with a variety of inorganic (e.g.,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ ) and organic (e.g., alkyl and aryl carboxylates, EDTA, beta-diketones) anions and the data interpreted successfully by a model of ionic interaction. Much more needs to be done with the other oxidation states. Also, despite the importance of separations involving liquid-liquid extractions, transplutonium chemistry in non-aqueous solvents is a relatively unexplored field with much promise as it provides information unperturbed by the strong hydration effects in aqueous systems.

Many of the chemical studies of the transplutonium elements historically have been related to pragmatic problems of separation and purification. However, these elements are unique probes for a variety of chemical phenomena that can be investigated in some cases by radiotracer and in others by macroscopic techniques. In the radiotracer studies, no elaborate safety or instrumental requirements are involved, and the use of these elements to advance our knowledge of chemistry should continue to expand.

From the early days of the Manhattan Project, the properties of transuranium metals have been a source of curiosity because of their

unusual crystallographic and electronic behavior. The preparation of high-purity transplutonium metals is more difficult than making many of the compounds of these elements, partly because the metals themselves are so reactive, and partly because of the limited quantities of the isotopes and the unusual requirements of the metal syntheses. Recently, it has been possible to prepare multimilligram (5-10 mg) amounts of  $^{248}\text{Cm}$ ,  $^{249}\text{Bk}$ , and  $^{249}\text{Cf}$  metal; larger amounts of  $^{241}\text{Am}$  or  $^{243}\text{Am}$  metal can be prepared, but pure einsteinium metal free of a supporting substrate has not been produced to date, even at the microgram level. As a result of these limitations, especially those of the effects of impurities in small samples, the preparation of  $^{248}\text{Cm}$  and the transcurium metals often becomes the most critical phase of experiments, with either the major portion or the entire preparation being needed or consumed (i.e., dissolution of the metal in acid for heat of solution measurements) in the study.

In principle, the transplutonium metals could be prepared by several different synthetic methods, but the reactions outlined in equations (1) and (2) have been the most successful.



The first reaction consists of volatilizing and subsequently condensing the actinide metal, whereas in the second method the excess reductant and lithium fluoride are volatilized. Each method has advantages and disadvantages, and the method of choice depends on several factors, the most important of which is the volatility of the transplutonium metal. Normally,  $^{243}\text{Am}$ ,  $^{249}\text{Cf}$ , and  $^{253}\text{Es}$  metals are prepared by reaction (1), while  $^{248}\text{Cm}$  and  $^{249}\text{Bk}$  metals are prepared by reaction (2). Such contaminants as hydrogen, oxygen, and nitrogen are difficult or even impossible to determine in the milligram amounts of metal, and special procedures are required to avoid their presence in the products.

A great deal of information has been acquired on the transplutonium metals in recent years, but much more data are needed to fully understand their chemical/physical nature. Significant differences are found between the first five ( $Z = 90-94$ ) and the next five (transplutonium) actinide metals. To explain these differences, and to extrapolate the data to even higher members of the series, for which experiments may never be feasible, requires a complete understanding of the available actinide elements.

A good deal of information has been obtained on the vapor pressure and heats of vaporization of the first four transplutonium metals, but only qualitative information is available for einsteinium metal. Heat of solution data for americium, curium, and berkelium metals are established, and preliminary data are available for californium metal. Magnetic measurements have been made, and additional magnetic work will be done on these metals, to help understand their metallic bonding and energy levels. The observance of superconductivity in americium has contributed to understanding actinide metallic bonding.

The metals are used for preparing many compounds such as chalcogenides, pnictides, hydrides, etc. of these elements. Recently, high-pressure x-ray diffraction studies have revealed that interesting changes in metallic bonding occur in americium metal as a function of pressure, although such changes had not been observed for californium metal. Extension of this work to curium and berkelium is in progress.

A variety of experiments is planned, such as preparation of transplutonium monoxides by reacting the metals with oxides under high pressure and temperature. These studies on metals are a rather limited selection illustrative of many needed on the transplutonium metals to fully understand these elements and the actinide series to which they belong.

## VII

### OPTICAL AND MAGNETIC PROPERTIES

The optical spectra of the actinides are like fingerprints. They uniquely identify not only the element, but also its state of ionization. Spectroscopic measurements consequently play an important analytical role in the whole of actinide research. Also, the finer details of such spectra reveal a great wealth of information, as shown below.

Exciting developments in atomic theory are making possible significant advances in our ability to interpret actinide spectra. The result of this interaction between theory and experiment is a growing understanding of the electronic structure in both the neutral and ionized species of these elements, and new insight into the nature of the bonding in actinide molecular species. It is on the basis of the interpretation of spectroscopic structure that the only estimates of thermodynamic quantities (redox potentials, ionization potentials, enthalpies of sublimation, cohesive energies, etc.) essential to the prediction of chemical behavior have been made for some of the actinide elements. The electronic transitions responsible for the importance of the lanthanides in phosphors and in laser technology are also characteristic of the actinides; such transitions are found in these two series of elements but nowhere in the periodic table. Optical spectroscopic studies are thus providing a unique key to the understanding and use of the actinides in science and in technology.

The recent experimental measurement of the atomic spectrum of einsteinium represented in a sense the culmination of many years of effort as well as the beginning of an exciting new era. Einsteinium is the heaviest element that will be amenable to the high-resolution techniques that were used to obtain benchmark spectra of all of the lighter actinide elements. This undertaking was a tour de force in the field of atomic spectroscopy because it put the level of documentation of the einsteinium spectrum (using the highly radioactive isotope  $^{253}\text{Es}$ , with a 20-day half-life) on a par with that of many lighter, inactive, and more readily available elements. It would not have been possible without the collaborative interaction of scientists from many national laboratories (ANL, LBL, LLL, LASL, ORNL) and universities, who progressively refined the measurement and handling techniques and cooperated in the actual experiment.

With this type of high-resolution spectra as a strong underpinning, the path is now open to challenging new and more complex studies using modern electronic and laser technology. Intensity measurements of complex spectra are now being made for the first time; there is a new emphasis on spectra of higher ionization states of interest in plasma research. These new directions also require the continued development of the theoretical basis for interpretation of the spectroscopic results, a separate but interactive effort with its own unique challenges. On the practical side, the theoretical and experimental aspects of actinide spectroscopy meet in attempts to develop new viable techniques for isotope separation.

The study of actinide ion spectra in condensed phases offers unique challenges for interpretation since valence states ranging from 2 to 7 are known. Opportunities to discover particular transplutonium ions stabilized in higher (or lower) oxidation states for the first time still exist. The bonding relationships and types found in some of these compounds are not observed in any other part of the periodic table. While characteristic spectra have been measured, theoretical models of the electronic structure are generally either in an early stage of development or nonexistent. In addition to optical spectroscopy, laser Raman spectroscopy is being exploited as a particularly valuable means for studying bonding and complexation in solution, such as that revealed in the cation-cation complexes of Am(V) and U(VI).

New areas of photophysics are being opened up in recent investigations designed to explore both the storage of optical energy in crystals and liquids containing actinide species, and the dynamics of energy transfer. Ultrasensitive methods of detection that reveal not only the identity of the element but its valence state in solution are emerging from this work. Important applications are found in waste management and environmentally related investigations.

In a series of innovative new experiments, changes in the crystallographic structure and oxidation state of heavy actinide compounds ( $\text{EsCl}_3$ ,  $\text{BkCl}_3$ ) induced by radioactive decay are being sensitively monitored by spectroscopic techniques. This work shows that while the absence of long-range order in a crystalline environment may preclude obtaining structural information from x-ray patterns, the short-range order determined by the arrangement of nearest neighbor ions about a central actinide ion can be characterized spectroscopically.

A milestone in actinide solution chemistry was recently achieved when the first emission spectra and fluorescence emission lifetimes for a transplutonium ion,  $\text{Cm}^{3+}$ , in aqueous solution were recorded using selective laser excitation and digital signal averaging. Closely following this initial success, similar results were obtained with  $\text{Bk}^{3+}$  and  $\text{Es}^{3+}$ . It was concluded that the fluorescence lifetimes of aquo actinide ions obey a simple exponential energy gap law. Changes in the coordination sphere were found to be sensitively related to changes in fluorescence lifetimes. For example, in strong carbonate solution, the fluorescence quantum yield of  $\text{Cm}^{3+}$  was improved by a factor of 4 over that in dilute aqueous solution. Such results are relevant to the extremely sensitive detection of actinides

in waste process streams, or in ground waters, as well as to the modeling of complexation and energy transfer in solution. These experiments are dependent on the availability of macroamounts of  $^{248}\text{Cm}$ .

The monitoring of both the energies and lifetimes of the fluorescing states of actinide ions in solids has provided the basis for uniquely ascribing fluorescences to a particular ion in a radioactive decay chain where interference arises from the daughter or granddaughter of a short-lived parent. New laser-based methods have also enabled detailed investigations of the electronic structure of highly radioactive elements at low temperatures, even though the crystals under study rapidly become opaque to transmitted light.

Crystal field calculations were recently carried out for the first time with lighter trivalent actinide elements in a well-characterized crystal lattice ( $\text{LaCl}_3$ ). This significant breakthrough is now providing the basis for developing a similar detailed analysis of the crystal-field in the spectra of the heavier actinides, such as  $\text{Es}^{3+}$ .

Elegant microchemical techniques have been highly successful in making possible the measurement of the spectra of divalent actinide compounds such as  $\text{CfCl}_2$  and  $\text{EsCl}_2$ . This work is extremely important because it provides the first evidence for the magnitude of the crystal-field interaction in the  $f^N$  configuration for divalent actinides (and lanthanides), and thus the first experimental basis for estimating a complete set of interaction parameters in this valence state. Similar transitions in the lanthanide series and in most lighter actinides are completely masked by intense absorption bands that arise from other types of electronic transitions.

Spectroscopic characterization of electronic structure and the influence of bonding in higher-valent actinide compounds is poorly understood and represents an important new frontier. Not only does it offer the potential for increasing our understanding of the relationship of energy level structure and dynamical processes in compounds that are unique in the periodic table, but there is reason to believe that there are many more classes of compounds containing heavier actinide ions in unusual valence states that remain to be characterized — an equal challenge to the synthetic inorganic chemist.

The use of laser sources with high resolution to explore the photochemical consequences of selective optical excitation will be an important area of future studies, particularly in volatile higher-valent actinide compounds. The potential for isotope separation associated with the coupling of vibronic structure to electronic transitions in visible, near-infrared spectral range has already been demonstrated with lighter actinides.

Magnetic studies provide information about actinide ions and materials complementary to that obtained by optical spectroscopy. Electron paramagnetic resonance experiments also provide a fingerprint of the actinide ions and allow identification of oxidation states and localized site symmetries of the ions. In addition, nuclear information can be obtained from hyperfine structure. These types of studies provided the first microscopic clues for the identification

of the divalent state in the 5f series. Earlier work has been constrained only to the ground state and perhaps one or two close-lying levels. Combining optical laser methods with magnetic techniques will allow the detailed information to be obtained for higher-lying excited states. Questions about bonding in the actinide series and relativistic effects for heavy Z ions should be answered by the analysis of this type of data. These studies are completely dependent on a continuing supply of transplutonium isotopes in macrolevel quantities. Most would be performed more safely and with greater likelihood of success with such isotopes as  $^{248}\text{Cm}$  and  $^{249}\text{Cf}$  from the HFIR/TRU program.

## VIII

### ELECTRONIC STRUCTURE AND SOLID STATE PROPERTIES

The last 10 years have witnessed some remarkable advances in our understanding of the electronic structure and properties of the actinides. The availability of experimental quantities of protactinium, americium, curium, berkelium, and californium, together with the application of many experimental techniques to probe the 5f electron behavior in the more well known elements in the series (thorium, uranium, neptunium, and plutonium), has been instrumental in providing the basis for the development of theoretical understanding of the electronic structure of the actinide series. It also becomes possible to relate the actinide series to the other transition series of the periodic table and to obtain a unifying picture of the relationships among the electronic structures of the actinides, rare earths, and transition metals.

Theoretical energy band structure calculations<sup>1,2</sup> suggested that the actinide metals can be separated into two groups — the "light" (Th to Pu) and the "heavy" actinides (Am and beyond). The 5f electron orbitals in the light actinides must be considered from the itinerant or band point of view. By contrast, the 5f electrons in the heavy actinides form very narrow bands that do not hybridize greatly with the 6d and 7s bands. Further, since the coulomb correlation,  $U$  (the energy required for a 5f electron to hop from one atomic site to another), is larger relative to the effective bandwidth,  $W$ , the localized description of the ionic 5f electrons appears more appropriate. Thus the 5f electrons in the light actinide metals are similar to the itinerant 3d electrons of the transition metals, while at Am, the local character of the rare-earth 4f states is almost attained. However, the degree of localization of the 5f electron (as indicated by the ratio of  $U/W$ ) even in the heavy actinide Bk ( $U/W \sim 7$ ) is much less than that of the 4f electrons in the rare earths ( $U/W$  increases from roughly 20 to about 100 across the series).

The 5f electron behavior in the light actinide metals has been investigated by many experimental techniques: magnetization, electrical transport properties, Mössbauer effect, de Haas-van Alphen effect, x-ray and neutron scattering, heat capacity, and photoelectron spectroscopy measurements (comprehensively compiled in Ref. 3). The results indicated that the filling of the 5f electron shell starts in protactinium; the 5f electron in the light actinide metals (Th to Pu) can best be described in the band picture, and the transition from itinerant to localized 5f electron behavior occurs in americium

metal.<sup>4</sup> The 5f electron behavior in heavy actinide metals, Cm, Bk, and Cf, has been studied by means of magnetization measurements.<sup>5-8</sup> The results, although preliminary, confirmed the localized 5f electron model. The ease of delocalizing the 5f electron in heavy actinide metal (as suggested by the small U/W ratio), has been nicely demonstrated by the structural phase transformation of Am metal from the rare-earth-like double-hexagonal close-packed structure to the orthorhombic U-type under pressure.<sup>9-11</sup> The possibility of phase transition in Cm metal under pressure was studied,<sup>12</sup> but the experimental result was not conclusive.

In the formation of intermetallic compounds by light actinide elements, the actinide-actinide (An-An) spacing can be expanded to decrease the overlap of neighboring 5f orbitals. When the overlap of neighboring 5f orbitals is relatively small, so that the effective bandwidth is smaller than the coulomb-correlation energy, the 5f electrons are, essentially, localized. The magnetic property of binary intermetallic compounds of the light actinides (U, Np, and Pu) can indeed be divided into two groups that are separated by a "critical" range (~3.5 Å) of An-An distances. Compounds with An-An separation less than the critical values are nonmagnetic, except for a small group of compounds with magnetic 3d elements as partners. Compounds with An-An spacing larger than the critical values exhibit a variety of magnetic properties and can be roughly subdivided into two groups. One subgroup has An-An separation close to the critical value. These compounds exhibit localized spin-fluctuation behavior and itinerant electron magnetism. The other subgroup has An-An separation much larger than the critical value, and the magnetic properties of these compounds exhibit localized 5f electron magnetism, except for compounds with Cu<sub>3</sub>Au-type structure. Application of pressure to these compounds can drastically alter the magnetic properties through the change of An-An separation. Since the 5f electrons are more localized in the heavy actinides, the rare-earth-like magnetic properties are expected in their intermetallic compounds. This expectation seems to be confirmed by preliminary magnetization results obtained on NaCl-type compounds.<sup>5,6,13</sup>

The research in the solid state properties of heavy actinides is in its infancy. More systematic studies are required to discern the role of 5f electrons in controlling the physical and chemical properties of their alloys and compounds. Application of other experimental techniques in addition to magnetization measurements to probe the various aspects of the 5f electron interactions in the solid is becoming feasible with the increasing availability of the heavy element. The prospect is exciting and challenging since our understanding of electron-electron and electron-lattice interactions in solid cannot be completed without experimental inputs from the actinide series in the periodic table.

## IX

### ENVIRONMENTAL AND BIOLOGICAL BEHAVIOR OF TRANSPLUTONIUM ELEMENTS

Almost all of the environmental investigations with transplutonium elements have been done with americium and curium. The transplutonium element isotopes used in the various investigations were  $^{241}\text{Am}$ ,  $^{243,244}\text{Cm}$ ,  $^{252}\text{Cf}$ , and  $^{253}\text{Es}$ . P. W. Durbin, in Handbuch der Experimentellen Pharmakologie (1973), has summarized pre-1973 data for the metabolism and biological effects of transplutonium elements. Plutonium research per se is not addressed here. However, because of the large existing body of knowledge on plutonium, comparisons between the transplutonium elements and plutonium will be made where appropriate to establish a continuity of understanding.

Because of the possible health hazard to man through ingestion of transplutonium nuclides, numerous plant studies have been done to determine the ratio of nuclide activity in plants to nuclide activity in soils. The range of concentration ratios is  $1 \times 10^{-2}$  to  $1 \times 10^{-5}$ . The availability of these nuclides to plants decreases in the order: americium > curium > plutonium. Chelation of the nuclides with DTPA or EDTA generally increases the availability and uptake of these nuclides, while addition of organic matter or lime generally decreases the uptake. Different classes of plants take up different amounts of the nuclides (for example, legumes take up ten times more nuclide than grasses). The activity in plant parts generally follows the order: roots > leaves > stems > fruits > seeds for greenhouse experiments. Field studies and deposition studies have shown equal or greater amounts of activity deposited on leaf surfaces than that taken up through the roots. Submicron-size particles containing the nuclide enhance aerial transport but also are not easily dislodged from leaf surfaces, bringing about an incorporation of the nuclide into the plant with translocation occurring to other plant parts. The significance of this difference in uptake, whether through roots from the soil or through leaves from deposition, lies in whether or not a difference is found in the gastrointestinal absorbability of the nuclides causing a change in the dose to man. Ingestion by animals of plant tissues containing the incorporated nuclides may result in greater transfer across the gut than that obtained experimentally by forcing inorganic nuclide solutions by tube into the gut area. Experiments with animals fed plants containing nonchelated nuclides

have shown an increase in nuclide activity in the body, while other experiments, using plants grown on soils to which the DTPA chelator has been added, did not, even though the nuclide content of the plants was increased with chelation.

The chemistry of transplutonium elements in aquatic environments is complex in relation both to element selectivity and to stability of associations formed. In a river system, less than 50% of nuclide activity was associated with particles  $>1 \mu\text{m}$ . In tracing the Mediterranean water movement,  $^{241}\text{Am}$  was found to behave more as a particle-associated nuclide than as a soluble nuclide, and was rapidly deposited in the sediments. An increase by a factor of 3 of americium over plutonium was found in particulates in surface waters of the Mediterranean. The vertical transport of americium is also significantly greater than that of plutonium. The aggregation of particles appears to be an important mechanism in accelerating the removal of surface particulates. An examination of  $^{241}\text{Am}/^{239,240}\text{Pu}$  ratios in deep ocean water and sediments showed an increase in the ratio, suggesting that americium sinks more quickly than plutonium.

Americium has been found to have an especially high affinity for organic detritus in aquatic systems. Separation of the detritus into acid-soluble and acid-insoluble fractions showed ten times more nuclide activity was associated with the acid-insoluble fraction.

Remobilization processes for sediments in marine systems indicate an increased availability and accumulation of these nuclides in marine invertebrates and fish. In one study of the concentrations of americium and plutonium in fish, the concentration of americium was greater than that of plutonium for all organs analyzed.

Very little information exists for curium in natural aquatic environments, making this a worthwhile area for research.

The common (+3) valence state of the transplutonium elements and decreasing ionic radii as the atomic number increases confer several general properties to this group, i.e., significant hydrolysis of aqueous ions, low solubility of the compounds, and stability of complex ions. This is demonstrated by the increased ability of americium to form stable complexes with particles and detrital material in aqueous systems as compared to plutonium. In soil tests, anionic americium complexation, especially with hydroxyl and carbonate anions and/or chemisorption, was determined to be playing a more significant role than either precipitation or ion exchange reactions. When organic substrates were added to well water containing americium, the sorption of americium decreased. Soluble anions of americium were found in a soil test, and these forms were not retained on the soil column. Thus the significance of particle sizes is again shown to be important. Particles  $<1 \mu\text{m}$  are suggested as being responsible for the distribution patterns found between aqueous and soil phases.

The biological impact of the transplutonium elements has been given more attention than the environmental impact, but the primary emphasis has been placed on americium, curium, and einsteinium, with less emphasis on californium. A wide range of responses has been observed in the various animals used for experimental purposes. In general, the transplutonium elements are absorbed in lesser amounts

from the gastrointestinal tract and are removed faster from the lungs than corresponding doses of plutonium. The age of animals used in the experiments causes differences in the amounts of nuclides taken up, and in their deposition sites. In general, the skeleton and the liver are the two primary sites of deposition. The americium concentration in the skeletons of cows originally introduced into a contaminated area showed little increase with increasing time of exposure, while the skeletons of those animals born in the study area showed a continued upward trend with time. The distribution of americium within dog skeletons is dependent on the state of development of the individual bones. The difference in amount of americium deposited in neonatal skeletons was three times greater than for young adult dogs (18 months). With increasing age of animals, curium absorption decreases. Americium and curium are less embryocidal than plutonium; the suggested reason is that there are lower concentrations of these nuclides in the placenta and fetal membranes. Using comparable doses of californium and americium, more californium than americium is found in the bones of rats and hamsters. In comparisons of inhaled aerosols of einsteinium and americium, two times as much americium is excreted as einsteinium. Inhalation of californium aerosols results in a nonuniform distribution pattern in lung tissues leading to pneumosclerosis and lung tumors. The rapid movement of curium from the lungs following an inhalation exposure is thought to be associated with the formation and transport of hydroxyl particles about  $0.001 \mu\text{m}$  diameter. These small negatively charged particles are assumed to diffuse passively through the alveolar walls into the blood with elimination in the urine. Like curium, the metabolic behavior of the oxide of americium more closely resembles a soluble rather than insoluble compound. Injected americium is more uniformly distributed on bone surfaces than plutonium. Curium and americium are less efficient in inducing bone tumors in rats than plutonium.

Inhaled doses of americium, curium, and einsteinium show a propensity for induction of tumors in the lung tissue, followed by bone tissues. The degree of homogeneity of deposition has been suggested as making a difference in tumor formation.

Absorption of organically bound nuclides is twice that of inorganic forms. In vitro studies with americium oxide and serum produces an increase in solubilization of americium. Macrophages produce an increase in the solubilization of americium oxide particles ( $<0.1 \mu\text{m}$  diameter) through cell-mediated phagocytosis.

Information on the impact of curium in the environment is lacking. Particle size and particle formation have appeared in all aspects of this review, yet very little is known about formation, chemical characteristics, effects on the environment and biological systems, and effects of the particles in these systems. In light of the possible influence of particle size on distribution coefficients in soil systems, a reevaluation of the standard  $K_d$ 's seems warranted.

In the area of biological studies, information is lacking on multi-generation effects of chronically exposed animals. Differences

have been noted for uptake, deposition sites, and rates of removal between young and old animals.

Since significant amounts of americium and, possibly, some curium will be present in processed reactor wastes, greatly expanded knowledge of the environmental and biological behavior of the transplutonium elements is a necessary component of a nuclear energy program.

X

HEAVY ELEMENT PRODUCTION

In the earliest days of heavy element research, each experimenter had to prepare the nuclide or nuclides being studied, often in conjunction with the actual measurements. By the 1950's, several radiochemical groups at the major national laboratories had developed joint programs in which batches of material would be prepared and then distributed to several laboratories where complementary investigations would be carried out. This more efficient procedure evolved into the present U.S. Transplutonium Production Program.

The main objective of this program is to supply transplutonium elements for use in research throughout the United States. This currently involves joint operation at ORNL of the Transuranium Processing Plant (TRU), and the High Flux Isotope Reactor (HFIR). Target rods irradiated in the HFIR are processed at TRU for separation, recovery, and purification of the heavy actinides up through  $^{257}\text{Fm}$ . Radioactive target materials are also prepared at TRU and fabricated into suitable form for irradiation in the HFIR. Such materials include recycled americium and curium fabricated into HFIR targets, and heavy isotopes in special forms, including "rabbits" for short-term irradiation. In addition to the normal production functions at TRU, which is the production, storage, and distribution center for the research program, cooperative programs are carried out with research laboratories (both at ORNL and elsewhere) to assist them in their work. When feasible, special isotopes, services, and product forms are supplied at their request. These arrangements have allowed even broader participation in heavy element research by laboratories unable to operate their own production facilities. There remains a major restriction, however, in that all these materials are highly toxic and can be safely handled only by qualified workers in properly designed facilities.

Other facets of the program at TRU include development of chemical separations processes and development of methods for remotely fabricating and inspecting HFIR targets.

Since operations in the ORNL facilities were begun in 1966, production of the transplutonium elements has grown steadily, and by the end of 1981, a total of 600 mg of  $^{249}\text{Bk}$ , 5.4 g of  $^{252}\text{Cf}$ , 24 mg of  $^{253}\text{Es}$ , and 13 pg of  $^{257}\text{Fm}$  had been produced. More than 1100 shipments of these and other isotopes such as  $^{248}\text{Cm}$  or  $^{250}\text{Cf}$ , which are produced in special projects, have been made to about 30 laboratories in the

United States and several foreign countries. Currently about 500 mg of  $^{252}\text{Cf}$  and related amounts of the other materials are produced annually.

In addition to the research-oriented production program described above, there have been a number of other programs in the United States to produce large quantities of transplutonium elements for various purposes. Chief among these were the production of 3 kg of  $^{244}\text{Cm}$  for the Space Nuclear Systems program; the irradiation of multikilograms of  $^{242}\text{Pu}$ ,  $^{243}\text{Am}$ , and  $^{244}\text{Cm}$  at Savannah River to produce over 2 g of  $^{252}\text{Cf}$  for the Market Evaluation, Demonstration Center, and University Loan Programs; development and demonstration of the recovery of  $^{241-243}\text{Am}$  and  $^{244}\text{Cm}$  as by-products of nuclear power fuel processing; and the recovery of commercially valuable  $^{241}\text{Am}$  from decayed  $^{241}\text{Pu}$  in the weapons stockpile. There has been technical collaboration among these programs from which the research program has benefitted greatly, along with the direct benefit of obtaining a number of valuable by-products from the Savannah River californium programs. At the present time, the research program is returning the favor and supplying 50-100 mg/year of "by-product"  $^{252}\text{Cf}$  to Savannah River for distribution worldwide into the industrial, educational, and medical communities.

Future efforts of the heavy element production program will be to continue furnishing needed amounts of transplutonium elements to qualified researchers. A special effort will be made to develop techniques of irradiation and/or processing to produce an order-of-magnitude increase in the quantity of  $^{254}\text{Es}$  and  $^{257}\text{Fm}$  available. Such samples would open the door to new experiments in the formation of superheavy elements and in studying the details of the fission process.

## XI

### APPLICATIONS OF CALIFORNIUM-252

When  $^{252}\text{Cf}$  was discovered, in 1953, its high rate of spontaneous fission immediately suggested the possibility of developing miniature neutron sources of high intensity that might have a multitude of applications in research, in industry, or in education. When the promise of large-scale production of  $^{252}\text{Cf}$  began to be realized in the mid-1960's, development of some of these uses began. By the time of the first  $^{252}\text{Cf}$  Symposium, in October 1968, the AEC had in place a formal industry-government cooperative program for development of commercial uses. Similar programs were set in place for development of medical applications and for use of returned sources by educational institutions.

One of the most exciting fields of development was in the health sciences. Various arrangements of small sources were tested to determine the most effective procedure for radiotherapy of cancer with the least exposure to the therapist. Several hospitals were licensed to use these techniques but interest has decreased in favor of other treatment modalities. Prototypes were also tested to demonstrate in vivo activation analysis for calcium in bone and also the preparation of small quantities of short-lived radioisotopes at the hospital site by activation with californium neutrons. A total of 5.5 mg of  $^{252}\text{Cf}$  has been loaned to the medical community for development and application of these techniques.

Applications in the industrial sector can be grouped into four general areas: natural resources exploration, process control, quality control, and safeguards. Devices that have been developed make use of several techniques, but mainly neutron activation analysis, neutron radiography, and neutron gaging (of either transmitted or backscattered neutrons). The following specific devices have been developed at least to the prototype stage, and many are now in routine use: uranium-borehole analyzers, copper and nickel ore analyzers, oil-well loggers, sulfur meters for coal or oil, analyzers for vanadium in crude oil, moisture monitors, cement analyzers, fuel rod scanners, fissile waste monitors, and instruments to measure the neutron absorber content of control rods and the hafnium concentration in zirconium.

Neutron radiography has been applied to detecting the explosive content of detonators, bonding in aluminum aircraft structures, and evidence of corrosion on operating aircraft. Other applications

within the nuclear industry include calibration of neutron-detecting instrumentation and serving as reactor start-up sources. Altogether 1,535 mg of  $^{252}\text{Cf}$  has been sold or loaned for these applications.

The final category considered is education and research. This includes basic research on biological effects of neutron radiation, on the  $^{252}\text{Cf}$  neutron spectrum, on the fission process itself, and on the identity and energy spectrum of the fission products. Also, sources have been loaned to educational institutions for teaching students to do neutron flux monitoring and neutron activation analyses, and also to produce short-lived isotopes to be used in teaching techniques of radiochemistry and radio-tracing. Through 1981, 134 mg of  $^{252}\text{Cf}$  has been loaned to educational and research institutions for these purposes.

Annual distributions of  $^{252}\text{Cf}$  are approaching the level of 100 mg. The Department of Energy as well as its predecessor agencies has repeatedly assured its clients that  $^{252}\text{Cf}$  would continue to be available at a reasonable price. Clearly, the future extent of application will depend on that price, but it will also depend on many institutional factors, such as problems of licensing, the availability of qualified personnel, and the degree to which management is willing to innovate.

## XII

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