Thermodynamic Studies on Heavy Elements and
Studies in Nuclear Chemistry

Annual Report

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for the

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I. Introduction

This report summarizes research activities carried out under contract AT(11-1)-1716 from January 1, 1971 to December 31, 1971. Work has continued on various aspects of ternary fission, and two papers on this subject were published during the year. Studies on ternary fission induced by heavy ions have been greatly expanded in anticipation of similar future heavy ion research in the super-heavy element region.

Research on the chemically bound neutron under this contract is being phased out and in the future will be supported by the Chemistry Department.

A number of thermodynamic properties of uranyl compounds have been determined and summarized in this report.

The thermodynamic functions for $\text{Eu}^{+2}(\text{aq})$ have been redetermined.

II. Research Activities

A. Nuclear Chemistry Research

1. The Fission of Pu^{239} Induced by Intermediate Energy Helium Ions (McDaniel)

Previously in this laboratory ternary fission research has been confined to elements with Z below neptunium. This has been the case for several reasons; the main ones being that these elements were readily available in reasonable quantities with reasonable purities. Further, metallic foils could be produced which were reasonably inert with respect to air oxidation (for example, uranium oxidizes only slightly to form a protective oxide film which prevents further oxidation).

The results of this research indicated clearly that the study of heavier compound nuclei should lead to further elucidation of the systematics of nuclear fission.¹ To produce heavier compound nuclei one can employ heavier projectile and/or heavier target nuclei. With heavier projectiles angular momentum effects become important (It is known that high angular momentum states have large level widths for fission and small widths for particle evaporation). It is also necessary to consider the much increased Coulomb barrier when using heavy ions as projectiles. This fact requires the increase in bombarding energy and a higher resultant excitation energy of the compound nucleus. In this situation there may be increasing competition from knock-on or direct interaction reactions.

With these facts in mind, it was decided in this project to use a heavier target and alpha particles as projectiles. The data from such a

1. D. L. Uhl, T. L. McDaniel, J. W. Cobble, Phys. Rev. C, 4, 1357 (1971)

project would be easily integrated with the previous work in this laboratory and should add significant data to the present body of knowledge concerning ternary fission.

The only heavy material available for use as a target at a reasonable price was Pu^{239} . As a metal Pu^{239} is extremely unstable with respect to air oxidation to the oxide. This oxidation process is rather rapid and complete. The design problem was to produce a safe, reusable, lasting target that was protected from atmospheric oxidation and stable at the elevated temperatures of cyclotron runs, but to still allow collection of a reasonable fraction of the fission products.

The foil assembly in Figure 1 was decided upon and has proven successful, so far, in the early stages of the project. These foils were fabricated by Union Carbide Corp., Nuclear Division, Oak Ridge, Tenn. One of the foils has been exposed for 14 hours and a total of 42 microampere hours of 44.5 MeV alphas on the Argonne National Laboratory 60" cyclotron with the only noticeable deterioration being a slight discoloration of the silver. While not in use the foils are stored under an argon atmosphere.

The target arrangement for the cyclotron bombardments is shown in Figure 2. This target assembly allows for fission products recoiling in the backward direction (beam side) to escape from the target foil package and be collected in a cylindrical catcher foil along the inside wall of the front portion of the assembly. Subsequent carrier chemistry carried out on this catcher foil and counting gives cross section data for those nuclides which are amenable to radiochemical study. The method used here for collection of fission fragments is superior to the use of stacked foils because here the catcher foil is not activated by the beam. This

Figure 1. Construction of Pu target foil

A: 99.9999% silver cover foil (2.5 cm x 2.5 cm x $2^{\text{mg}}/\text{cm}^2$)

B: 99.11% Pu²³⁹ foil (2.1 cm x 2.1 cm x $10^{\text{mg}}/\text{cm}^2$)

C: Silver disc (4.3 cm diameter x 1 mm)

Figure 2. Target Assembly

- ▨ Ta Collimator
- ▧ Ag-Pu²³⁹ Target
- ▩ Viton "O" Ring
- ▤ Aluminum Target Assembly
- ▦ Ag Catcher Foil

means that the total gross unwanted activity is small and that there are no blank corrections necessary for alpha induced reactions in the catcher material.

The major objective of these experiments is to determine, as completely as possible, the mass yield curve for the reaction $\text{Pu}^{239}(\alpha, f)$ before the foils are destroyed by cyclotron irradiations. Consequently, it is necessary to maximize the number of nuclides determined in each bombardment. Toward this end a separation scheme is being devised which is based primarily / on selective elution or gradient elution from ion exchange columns.

Very briefly the sequence of separations is as follows:

1. Dowex 1 x 8 column: Selective elution with various normalities of HCl (High normality $\rightarrow$ Low normality);
2. Dowex 1 x 8 column: Selective elution with various normalities of HCl (Low normality $\rightarrow$ High normality);
3. Dowex 50 x 4 column: α -hydroxyisobutyric acid gradient elution of rare earth fraction;
4. Dowex 50 x 8 column: formic acid, ammonium formate selective elution of specific alkali metals and alkaline earth elements.

Several other ion exchange columns, solvent extraction procedures, and precipitation steps are used in the general separation scheme. Additionally a separate purification procedure is used for each specific element to insure radiochemical purity. Decontamination of $>1 \text{ part}/10^6$ for several low yield products is obtained. The chemically separated elements are then, after purification, β -counted in standardized low-background counters which have been previously discussed.¹

1. H. Andre, Ph.D. thesis, Purdue University, June 1964.

Work is continuing on a method for identifying and determining cross sections of fission products by examining gamma-ray spectra of the products during various stages of purification. It may be possible with this technique to simultaneously determine several nuclides without rigorous purification and separation of each. The computer programs are essentially complete for this type of analysis and hopefully future runs will soon yield usable data.

The preliminary results in the important region of low Z are shown in Figure 3. The solid line corresponds to the data of Macmurdo *et al.*¹ for the reaction $U^{238}(He^3, f)$. The Ca^{47} upper limit for the Pu^{239} data is from the work of Uhl *et al.*² If possible, later in this project, an extended bombardment may allow us to determine the Ca^{47} point more accurately.

It is expected that the Na^{24} point for the Pu^{239} data will be lowered considerably due to the blank correction which is necessary because of the reaction $Na^{23}(n, \gamma)Na^{24}$ which is induced by the rather high neutron flux near the cyclotron beam. The Na impurities in the silver catcher foil are 2 ppmw. It is hoped that the Na^{24} and Mg^{28} points will be precise enough to determine if the ternary curve has an inflection point or maximum in this region.

2. Ternary Fission of U^{238} Induced by C^{12} Heavy Ions (Otto)

The fission mass yield distribution for C^{12} on U^{238} in the region $20 \leq A \leq 100$ is being determined. Natural uranium foils were bombarded with carbon ions $C^{12}(+5)$ at the Yale Heavy Ion Accelerator.

1. K. W. MacMurdo and J. W. Cobble, Phys. Rev., 182, 1303 (1969).

2. D. L. Uhl, T. L. McDaniel and J. W. Cobble, Phys. Rev. C, 4, 1357 (1971).

Figure 3. Fission cross sections in the mass region $A = 20 - 80$

Open symbols refer to the Pu^{241*} compound nucleus; solid symbols to Cm^{243*} .

Mg^{28} , Ca^{47} , Ni^{66} and Sr^{89} activities were chemically separated (from silver recoil foils and the uranium target foil) using Mg, Ca, Ni and Sr carriers respectively. The beta decay of the separated activities was followed for 3 half lives or longer, using low background gas flow geiger counters. Cross sections for Mg^{28} , Ca^{47} , Ni^{66} , and Sr^{89} were calculated and in the case of Mg^{28} corrected for spallation in the silver recoil foils from a blank run. In addition the percent recoil in the forward and backward Ag catcher foils was determined. The theoretical percent recoil in the backward catcher for fission was plotted against the range in uranium of the fission fragment. The range of the fission products were then extrapolated from previous range studies in this laboratory. The ratio of the theoretical percent recoil to the experimentally determined percent recoil in the backward catcher was calculated. This factor was used as an estimate of the fraction of the measured cross section due to fission. The measured cross sections for Mg^{28} and Sr^{89} were corrected for spallation impurities by this method. These corrected cross sections were then compared with previous fission yields for the reactions $He^4 + U^{238}$ and $He^3 + U^{238}$.

Natural uranium foils (99.27% U^{238}) were placed between two 99.9999% pure 10 mg/cm^2 silver foils as shown in Figure 4. The uranium foils which ranged from 39 to 10 mg/cm^2 were bombarded with $C^{12}(+5)$ ions for a 24 hour period at a flux of 10^{11} particles/sec and an energy of 108 MeV at the front face of the target foil. The fissioning compound nucleus, $^{98}Cr^{250}$ was estimated to have an excitation energy of 79 MeV. The blank cross section for production of Mg^{28} in the silver recoil foils was $1.06 \pm .20$ microbarns. Recoil studies from previous work in this labora-

Figure 4. Diagram of heavy ion target arrangement

tory indicate the range of Mg^{28} and Sr^{89} to be 12 mg/cm^2 in uranium, for the fission of the compound nucleus Pu^{242} at an excitation energy of 35 MeV. The correction factor to the kinetic energy of the assumed recoiling fragment for the Cf^{250} compound nucleus is 2% or less and unimportant. The range of Mg^{28} and Sr^{89} was taken to be 12 mg/cm^2 in uranium for the present system. Figure 5 is a comparison of the corrected fission cross sections for the $\text{C}^{12} + \text{U}^{238}$ reaction with the same cross sections for the $\text{He}^4 + \text{U}^{238}$ and $\text{He}^3 + \text{U}^{238}$ reactions. The $\sigma_{\text{Mg}^{28}}/\sigma_{\text{Sr}^{89}}$ ratio for $\text{C}^{12} + \text{U}^{238}$ fission was found to be 7.33×10^{-3} and 8.00×10^{-4} for $\text{He}^4 + \text{U}^{238}$ fission. This may reflect a substantial increase in the ternary to binary fission ratio.

3. Heavy Ion Induced Ternary Fission in Gold (Kasner)

Earlier studies of ternary fission gave indications that it responds to changes in angular momentum in a manner similar to binary fission. Compound nuclei of high angular momentum can be produced by bombardment with heavy ions. Because the binary fission of the system $\text{Au}^{197} + \text{C}^{12}$ has been studied, it was felt that the ternary fission should also be investigated and the affects of angular momentum examined.

The Yale University Heavy Ion Accelerator is being used as the source of C^{12} . Carbon ions, in a +5 charge state, are accelerated to 10.4 MeV/nucleon (124.8 MeV) and currents up to 25 μ -amperes. The Au target foils are sandwiched between Ag catcher foils. When lower energies are required, Ag foils are used as degraders.

The foils, both catchers and target, are chemically analyzed for Mg and Sr. Mg^{28} activity is used as a ternary fission monitor and Sr^{89} is used to monitor binary fission. An excitation function for the production of Mg^{28} is being determined from the Coulombic barrier (~67 MeV) to ~115 MeV.

Figure 5. Fission mass distribution

Because the C^{12} beam passes through the entire foil stack, there are certain spallation reactions, other than ternary fission, which also have Mg^{28} as a reaction product. Therefore an excitation function for these spallation reactions in Ag must also be determined.

The "blank" excitation function has been determined from -60 MeV to -115 MeV and shows an increasing cross section from $\sim 0.04 \mu b$ to $\sim 0.5 \mu b$. The excitation function for the production of Mg^{28} has been partially determined and varies from $\sim 0.1 \mu b$ at -85 MeV to $\sim 1.1 \mu b$ at -115 MeV. (Fig. 6)

The work on the excitation function is expected to be completed shortly after the first of the year and a partial mass yield curve will then be determined to confirm that Mg^{28} is indeed a ternary fission product.

4. Fission Cross Sections From Gamma Ray Spectra (Hagenauer & McDaniel)

Attempts have been made to measure fission yields by measuring the gamma rays emitted from fission products with a high resolution 30cc Ge(Li) detector. Since most of the short lived fission products emit gamma rays we hope to determine the fission yield curve for the high yield products. The spectra have been collected with a 4096 multichannel analyzer and then computer analyzed for area, energy and half-life.

The computer program that we have developed to handle the analysis we call GASP (for GAMMA-ray SP ectroscopy). GASP is written in Fortran IV for the Purdue University Control Data Corp. 6500 computing system. Due to the unusually large core requirements of the basic program it has been subdivided into overlays where one portion of the program replaces an earlier portion to conserve space.

The first section of the program is basically a minor modification

Figure 6 (Upper) Excitation function for the total fission cross section determined by Gordon et al.¹

(Lower) Preliminary values for the excitation function of Mg^{28} , a ternary fission product.

1. G. E. Gordon, A. E. Larsh, T. Sikkeland and G. T. Seaborg, Phys. Rev., 120, 1341 (1960).

of BRUTAL written originally by R. Gunnink¹ at the Livermore branch of the Lawrence Radiation Laboratory. It's simplified operation is as follows:

1. Read Input parameters (energy calibration information, bombardment time, detector efficiency information etc.) Read spectrum 1.
2. Find peaks in spectrum 1.
3. Examine peaks for multiplet structure.
4. Determine areas for each peak.
5. Find exact energy for each peak.
6. Find counts per minute for each peak.
7. Begin an array containing the following for each γ -ray.
 - A. Spectrum start time.
 - B. Photopeak energy.
 - C. Counts per minute.
8. Read each subsequent spectrum.
9. Repeat steps 2 through 6 for each, in turn.
10. Search the array for a gamma ray with the same energy and either
 - A. Store, start time, counts/minute along with previous gamma-rays of same energy.
 - B. Add a new element to the array as in 7 above.

Once this portion of the analysis is complete the second part of the program is loaded and executed. This section is a modification of FRANTIC written originally by P. C. Rogers² at Massachusetts Institute of Technology.

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1. R. Gunnink, H. B. Levy, J. B. Miday, Lawrence Radiation Laboratory Report - 15140 (1967).
 2. P. C. Rogers, Massachusetts Institute of Technology Laboratory for Nuclear Science, Technical Report No. 76 (1962).

This part of the program does the following:

1. Pulls the stored data out of the array, column by column.
2. Analyzes each gamma-ray that occurs in more than one spectrum for half-life.
3. If the least squares fit for one component is poor, and if certain other criteria are met, analyzes for a good least-squares two component exponential decay.

The final tabulation of the results contains a listing of all gamma rays found in the following categories:

1. Decaying peaks.

lists half-life, uncertainty in half-life, A_0 , channel location in the 1st spectrum, and energy.

2. Growing peaks.

lists some information as 1.

3. Peaks that occurred in only one spectrum.

lists channel number and energy of single peaks (this is helpful in checking energy calibration of the spectra).

4. Peaks with matrix inversion problems.

lists the peaks that the Frantic section could not fit to either a one or two component curve. (These problems are usually due to unresolved multiplets in one or more, but not all, of the spectra.)

This computer software is now nearing final form and in test runs seems to work satisfactorily with spectra containing peaks with a low percentage of doublets.

Experimentally a known aliquot of the solution produced by dissolving the catcher foil was taken (for gamma analysis) while continuing separation

chemistry and β counting of products from the remainder. It appears that this method may give a limited amount of data due to the extremely high number of different gamma rays and the subsequent large Compton background in each spectrum. These effects tend to overwhelm the true photopeaks of elements with lower fission yields. Encouraging preliminary results have been obtained on rare earth samples that have undergone limited lanthanide chemistry. It is hoped that perhaps, in the near future, it will be possible to pass the original solution with carriers through an ion exchange column (e.g. Dowex 1 x 8, See Plutonium Fission Section of this publication) and separate groups of elements; determine chemical yields by atomic absorption or emission, and determine fission yields by gamma-ray spectroscopy of an aliquot of the solution.

At the present time identification of several nuclides has been made and we are in the process of fine tuning the nuclear instrumentations computer software and chemistry so that accurate, reproducible quantitative cross sections will result.

5. Studies on Chemically Bound Neutrons (Hagenauer)

A large number of neutron bombardments were made on γ -irradiated LiF crystals in liquid helium, investigating their ability to bind neutrons at low temperatures (4°K) and release them at room temperature.

Neutron bombardments were made at the Purdue reactor facility. The power was adjusted so that the slow neutron flux was the same as that obtained from the neutron generator where positive results were earlier observed. One-hundred seventy-seven bombardments were made using nine different LiF crystals. Bombardments were made at liquid helium temperatures and room temperatures and their spectra compared. The spectra from most

crystals showed no difference. Two crystals gave positive results after reirradiation in a ^{60}Co source, but after repeated bombardments, the "neutron capture" effect disappeared.

To further investigate this observation, fourteen crystals with different γ -irradiation histories were neutron bombarded with the neutron generator. The crystals were reirradiated with the ^{60}Co gamma source after every 6th neutron bombardment. Seven-hundred forty-four bombardments were made on the targets at various fluxes. Bound neutrons were observed, but no conclusive correlation could be established between the "neutron capture" effect and neutron flux or neutron dose. It was confirmed that spectra from two detectors increased the peak above background a factor of two.

Additional bombardments were then carried out using new detectors whose efficiencies were six times greater than the detectors used in the earlier experiments. Larger targets were made so that the crystals surface was larger than the detector. Four-hundred sixty-four neutron bombardments were made on twelve different crystals. However, no conclusive peak was observed on any crystal.

B. Thermodynamic Studies on Heavy Elements and Related Species

1. Thermodynamic Properties of Uranyl Species (Morss)

This research has been completed and written up for publication. The publication abstract follows:

"A Dewar calorimeter was used to measure the heats of solution of $\text{UO}_2(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ in dilute perchloric acid at six temperatures from 6-95°C. These observations were extrapolated to infinite dilution and the standard partial molal heat capacities of $\text{UO}_2(\text{NO}_3)_2\text{aq}$ were calculated by the

integral heat method. Using data from the literature and the measured heat capacity of crystalline $\text{UO}_2(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, the partial molal heat capacity and entropy of $\text{UO}_2^{+2}(\text{aq})$ and the thermodynamic properties of crystalline hydrates of uranyl nitrate have been calculated. The thermodynamic properties of $\text{UO}_2(\text{NO}_3)_2(\text{aq})$ are discussed in terms of high temperature solution correspondence theory and linear free energy theory."

Table I.

Summary of Thermodynamic Properties of Some Uranyl Species at 25°

	ΔH_f°	S°	C_p°
$\text{UO}_2^{+2}(\text{aq})$	-244.3 kcal	-23.2	25.9
$\text{UO}_2(\text{NO}_3)_2(\text{aq})$	-343.5	46.8	-15.5
$\text{UO}_2(\text{NO}_3)_2(\text{c})$	-324.1		
$\text{UO}_2(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}(\text{c})$	-473.7		66.5
$\text{UO}_2(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}(\text{c})$	-545.7		
$\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}(\text{c})$	-757.9	120.9	112.0
$\gamma\text{-UO}_3(\text{c})$	-293.0		

2. Thermochemistry of Aqueous Europium Ions (Morss)¹

Because of inconsistencies in the various thermochemical measurements among europium species, and because Eu^{+2} is one of the few representative actinide-lanthanide divalent ions which can be measured directly, systematic measurements were undertaken to redetermine the thermodynamic

1. This research was carried out with partial support by the National Science Foundation (in the form of a postdoctoral fellowship to Lester Morss).

properties, particularly the entropy. Earlier investigations had shown that dissolved oxygen was not a suitable oxidant for $\text{Eu}^{2+}(\text{aq})$, since hydrogen peroxide might be an oxidation product. It was decided to attempt to use bromine to effect the reaction

since the reaction is unambiguous and since the thermodynamic properties of $\text{Br}_2(\text{aq})$ are well known.

This reaction was carried out in some dozen calorimetric experiments, using bromine water as the limiting reagent in sealed glass ampoules. The heat of the above reaction was determined as -47.0 ± 0.5 kcal/mol. The limiting uncertainty in these experiments appeared to be establishing the amount of bromine present in each ampoule. The method found to give best results (with an error of about 0.5% at best) was careful and rapid transfer of bromine water with a syringe, weighing the amount of bromine water transferred, and then sealing the ampoules by fusing the glass neck with the bromine water under vacuum at liquid nitrogen temperature.

Alternative oxidation techniques were investigated to decrease this uncertainty in stoichiometry. The use of Eu^{2+} as limiting reagent was ruled out because $\text{Eu}^{2+}(\text{aq})$ is oxidized fairly rapidly -- about 10% per hour -- even in oxygen-free 0.001 M acid. Other oxidants (Br_3^{-} water, Br_2 in CCl_4 , pure Br_2 , and I_3^{-} water) were found to be even less suitable than Br_2 water for a variety of reasons.

An attempt was made to measure the oxidation potential of the $\text{Eu}^{2+} - \text{Eu}^{3+}$ couple by equilibrating a solution of europous and europic chloride or perchlorate with a saturated cadmium amalgam and CdCl_2 or CdClO_4 solution. Redox analyses of the cell solutions, even after careful purging

of air and long equilibration times, showed that the europium ions did not reach equilibrium and that far too much Eu^{3+} was present. A conventional electrochemical cell

was therefore utilized, although there is polarographic evidence that europium ions do not form a reversible couple at a Pt electrode. Experiments indicated that the above cell does reach stable and reproducible (to ± 0.001 volt) potentials. The tentative result is

which is in significant disagreement with McCoy's (J. Amer. Chem. Soc., 58, 1577) 0.43 volt. McCoy's measurements were carried out in concentrated formate solution, which selectively complexes Eu^{3+} .

The above measurements may be combined with other consistent thermochemical data for europium to yield the following standard-state functions:

	$\Delta H^\circ_f(\text{kcal/mol})$	$S^\circ(\text{gbs/mol})$
$(\text{Eu}^{2+}, \text{aq})$	-121.7	-2
$(\text{Eu}^{3+}, \text{aq})$	-140.3	-52.7

3. The Oxidation-Reduction Potentials of the Actinide Elements (Cobble)

The thermodynamic data for the actinide element ions have been brought up-to-date and recalculated for the third edition of "Oxidation-Reduction Potentials". The following potential diagrams are the summaries which will appear in this reference.

Basic Solution

III. Personnel

Dr. James W. Cobble, Project Director

Dr. Richard Hagenauer, Research Associate

Dr. Lester Morss, Research Associate* (Resigned, 8-31-71)

Graduate Research Assistants

T. J. Grant (Resigned 2-10-71)

M. L. Kasner

T. L. McDaniel

R. J. Otto

Ray Weller, Electronics Technician**

Howard J. Gobstein (Undergraduate research technician)**

Mrs. Dorothy Pershing, Secretary**

* N. S. F. Postdoctoral Fellow

** Part-time

IV. Publications and Talks

Publications

1. "Fission of Th^{232} Induced by Intermediate-Energy He^4 Ions," Phys. Rev. C, 4, 1361 (1971). T. C. Roginski, M. E. Davis and J. W. Cobble.
2. "Search for Evidence of Ternary Fission Induced in Au^{197} , Bi^{209} and Pu^{239} by Intermediate-Energy Helium Ions," Phys. Rev. C, 4, 1357 (1971). D. L. Uhl, T. L. McDaniel and J. W. Cobble.
3. "The Partial Molal Heat Capacity and Entropy of Uranyl Nitrate Solutions from 0-100°C and the Thermodynamic Properties of Uranyl Compounds," Inorg. Chem. L. R. Morss and J. W. Cobble, (to be published).

Talks by the Staff

1. "Studies on Chemically Bound Neutrons", J. W. Cobble, Welch Foundation Lecture, Texas A&M University, February 15, 1971.
2. "Studies on Chemically Bound Neutrons", J. W. Cobble, Seminar at University of Florida, April 8, 1971.
3. "Thermodynamics of the Actinide and Lanthanide Elements", J. W. Cobble, Seminar, Long Beach State College, October 13, 1971.
4. "New Techniques in Actinide Thermodynamics", L. R. Morss, Inorganic Seminar, Purdue University, February 25, 1971.
5. "Bromine as an Oxidant in the Thermochemistry of Europium", L. R. Morss, 26th Calorimetry Conference, Orono, Maine, July 12, 1971.