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# RECENT RESEARCH ON TRANSPLUTONIUM ELEMENTS

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associated with each fission. One microgram of $Cf^{252}$ emits about $2 \times 10^8$ neutrons per minute. Exposure at close quarters to the neutrons from microgram amounts of $Cf^{252}$ for prolonged periods of time is a dangerous health hazard since the neutrons have a most probable energy of about 1.5 Mev. A large amount of neutron shielding may be required to reduce the neutron intensity to a safe level. With larger amounts of californium the amount of shielding becomes so large that the use of tongs for carrying out remote operations becomes impractical. Therefore it seems that new types of equipment and new techniques for remote operations must be developed especially for the preparation of targets, sources, and for microchemical investigations.

For investigations of the macro chemistry of berkelium and californium it has now become most convenient to use the isotopes $Bk^{249}$ and its daughter $Cf^{249}$. The former is a beta emitter with a half life of about 280 days; the $Cf^{249}$ has an alpha half life of about 500 years and its spontaneous fission half life is about $10^9$ years. Therefore no unusual hazards are encountered in working with these isotopes. The importance of $Cf^{252}$ in other research, however, requires that remote handling methods be developed. There are obviously important researches on fission and new elements which can only be done with $Cf^{252}$.

In our laboratory the problems of remote operations and shielding are now being studied. Although it may not be necessary to watch all operations visually it may be helpful or even necessary at times to substitute tri-dimensional television as a medium to replace the direct observations. In this case manipulation may be controlled at large distances from the source with power transmission through hydraulic lines or electric motors.

It may be convenient to replace the familiar ion exchange methods for separation and purification of the actinides with solvent extraction methods which are more adaptable to some types of remote control. Peppard and his co-workers at the Argonne National Laboratories have developed some useful solvent extraction methods which will soon be published. Although all of the details of these methods are not available to us at this time the basic steps are as follows: The starting point is a mixture of transplutonium actinide elements and the usual fission products in concentrated hydrochloric acid solution (berkelium is removed first by oxidation to the +4 state with

bromate and extracted with a solvent). Californium and some heavy rare earths are extracted into tributyl phosphate. Curium and some light rare earths remain in the hydrochloric acid solution. Several stages are required to obtain a complete separation. Tributyl phosphate is distilled from the fraction containing californium and heavier rare earths. Then the californium and heavier actinides are separated from heavy rare earths by extraction into a phase composed of 25% di-ectyl-phosphate in kerosene. The aqueous phase in this case is dilute nitric acid.

B. Nuclear Studies

Since the subject of nuclear spectroscopy is excluded from this paper and the research on new isotopes is covered by Ghiorso, the nuclear work to be mentioned here will be largely confined to fission research. The availability of $Cf^{252}$ and the familiarity of many of us in working with this isotope has given rise to a natural interest in the possibilities for research on spontaneous fission. This interest is shared by chemists and physicists alike. In $Cf^{252}$ one has the possibility of preparing very intense fission sources which are at the same time essentially weightless. Deposition in a very small area can be accomplished on foils of thickness less than 100 micrograms per square centimeter. In preparing these sources, in addition to the conventional methods of vaporization and electrostatic spraying, one may obtain sources by a self-transfer method. Californium-252 has the unique property when freshly electroplated, of transferring itself to another foil. In this case the transfer occurs in an evacuated space when the electroplated deposit has not been heated. When the above $Cf^{252}$ deposits are heated to several hundred degrees centigrade the transfer does not occur. Sources prepared in this way are observed to be extremely thin and uniform. The mechanism involved in the transfer has not been studied. Some of the research on fission now in progress is summarized below.

1. Experimental Investigations

(a) Prompt Neutron Angular Distribution and Energy Spectrum from the Spontaneous Fission of $Cf^{252}$.

(H. B. Bowman, W. J. Swiatecki, and S. G. Thompson.)

(a) The neutron velocities and fission-fragment velocities were measured in coincidence by use of a cylindrical drum 1 meter in diameter. The

drum was evacuated to a pressure of $10^{-6}$ mm of Hg. A plastic scintillator 2 by 2 inches attached to a 14-stage photomultiplier tube was located in the center of the drum. A $Cf^{252}$ sample $2 \times 10^6$ spontaneous fissions per minute was placed on a flat portion of the plastic scintillator. The detection of the prompt gamma rays in this crystal indicated the occurrence of fission events. A 0.002-inch thick plastic scintillator was mounted on the face of a 5-inch-diameter photomultiplier located on the curved surface of the drum to detect the arrival of the fission fragments. The arrival of neutrons was detected by a stilbene crystal attached to a 14-stage photomultiplier tube, also located on the curved surface of the drum. The detector outputs were connected to the time-to-pulse-height converters. The pulse heights were in turn converted to digits and punched into IBM cards.

Each fission fragment was characterized as "light" or "heavy" according to its time-of-flight between the source and the fission detector. The time-of-flight of neutrons in coincidence with the fragments was measured at angles $12^\circ$, $26^\circ$, $40^\circ$, and $90^\circ$ relative to the direction of detected fragments. The measured neutron velocity spectra were converted to absolute numbers of neutrons per fission per unit volume of velocity space $\rho^{(\nu,\theta)*}$ by applying the corrections due to the counting efficiency of the stilbene

*The term "per unit volume of velocity space" means that the neutrons have velocity components $\nu_x$, $\nu_y$, $\nu_z$ in a unit cell $\Delta \nu_x$, $\Delta \nu_y$, and $\Delta \nu_z$.

crystal (without arbitrary normalization of any kind). Integration over all angles and intervals of velocity space (above a value corresponding to 1.18 Mev energy) gives the absolute value 2.7 as number of neutrons emitted per fission for the energy range considered. This value is close to the number expected for this energy interval on the basis of previous existing measurements.

The final results of the experiments are shown in Fig. 1 plotted in polar coordinates. The distance from the origin corresponds to the neutron velocity and the angle gives the direction of the neutron with respect to the direction of the light fragment. The contour lines connect points of constant

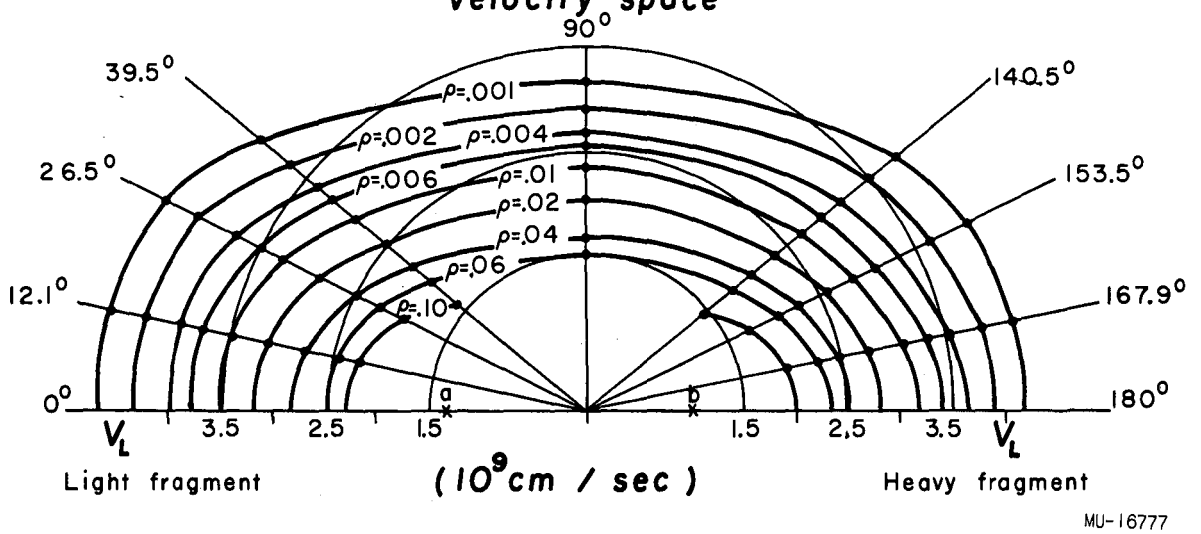


Fig. 1. Angular distribution of neutrons per fission unit volume of velocity space. a-Average velocity of light fragment (1.37 x $10^9$ cm/sec) b-Average velocity of heavy fragment (1.04 x $10^9$ cm/sec) $V_L$ = velocity of neutrons.

$\rho$ at the various measured angles and velocities. The neutrons from the light fragments are shown in the left quadrant. The right quadrant gives the results for neutrons from heavy fragments. The average velocity of light fragments is $1.37 \times 10^9$ cm per sec; the average velocity for heavy fragments is $1.04 \times 10^9$ cm/sec. Points representing the average fragment velocities are shown on the horizontal axis to the left (light) and right of the origin (heavy). It can be seen that the neutron energy and angular distribution in the laboratory system is dominated by the velocity contribution of the fragments.** It can also be seen that the probability of the emission of

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**It may be helpful to realize that the distribution represented in one plane in Fig. 1 is better visualized in three dimensions as that of a prolate spheroid. Thus, the deviation from a spherical shape is a consequence of the velocity component contributed by the fragments to the neutron velocities.

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neutrons decreases as the velocity increases. Analysis of the results also shows that the gross angular and velocity distribution of the neutrons can be accounted for by isotropic evaporation from the fragments but there is a slight indication of a deviation from this in low energy neutrons at $90^\circ$.

The data were replotted making changes in the variables such that a conventional single temperature evaporation spectrum would appear as a straight line on logarithmic paper. The resulting points which clearly deviated from a straight line were resolved in a series of straight lines (using the standard procedure of resolving complex decay spectra into a series of half lives). Each line corresponds to an evaporation at a constant temperature. Three such straight lines were found adequate to represent the data. The results give 43% of component with a temperature of 1.2 Mev, 42% of a component with a temperature of 0.4 Mev, and 15% of a component with a temperature of 0.17 Mev. The lowest temperature component should not be taken seriously because of inherent experimental difficulties.

The results show that the method is capable of giving a complete description as regards the angular and velocity distributions of the absolute number of neutrons emitted in fission process. This experiment is regarded

as preliminary and preparations are being made to begin a more refined series of measurements which have the following advantages over the previous work:

(1) A stronger fission source.

(2) Measurement of the velocities of both fragments over a longer flight path.

(3) Measurements of neutron velocities at more angles.

(4) Measurements of lower energy neutrons.

(b) Fission Studies of $Cf^{252}$ using Photographic Emulsions. By using emulsions which have been impregnated with $Cf^{252}$ it has been possible to study the events in which high energy alpha particles are emitted in the fission process. We have observed a large number of events of this type. The process seems to occur in about the same abundance as it does in $U^{235}$ thermal neutron fission. The alpha particles also seem to have about the same energy distribution. In addition to the long range alpha particles we have also observed a surprisingly large number of triple events in which all fragments have masses much larger than alpha particles. A careful analysis of these events is in progress. Some of them can be accounted for by scattering of binary fission fragments with bromine and silver in the emulsion. However, the possibility remains that some of the events were actually due to fission into three heavy fragments.

## 2. Theoretical Investigations of Fission

(a) The liquid drop model (W. J. Swiatecki). Further study of the potential energy of deformation of a uniformly charged drop has in the main confirmed and extended the considerations of UCRL-3991 (1958 Geneva paper). There is further evidence that in the discussion of the fission of such a drop it is necessary to consider several distinct families of equilibrium shapes. The properties of these families, some of them very unexpected and far reaching in their consequences, are being elucidated by semi-quantitative as well as quantitative electronic machine calculations.

The situation as it appears at the present time suggests that in addition to the several discrete families of equilibrium shapes which tend to strings of spherical fragments in contact for $x = 1$ and to rippled cylinders

for large x, there appears a pair of new equilibrium shapes at a 'point of bifurcation' around x = 0.74. With increasing x one member of the pair, which at first is in the form of a cylinder with rounded ends, tends through prolate shapes for x < 1 through the sphere at x = 1 to oblate shapes for x > 1. This constitutes the conventional family of saddle point shapes. The other member of the pair, also at first in the form of a cylinder with rounded ends, increases in elongation with increasing x. This branch of equilibrium shapes may be considered as a continuation of the conventional family after that family has regained stability against elongation (which it had lost at x = 1 in the form of instability against an $\alpha_2 P_2(\cos\theta)$ type of distortion). Depending on whether other types of instability have set in in the meantime, the new branch of equilibrium shapes might represent stable non-spherical configurations of a charged drop.

The disappearance of both the above branches for x less than about 0.74 calls for a revision of the conventional discussion of fission thresholds.

(b) Influence of angular momentum on fission threshold (John Hiskes). In the formation of compound systems produced by bombarding heavy nuclei with heavy ions, one anticipates the formation of compound systems of very high angular moments. If one assumes that the equilibrium configurations of such a rotating system are determined by a charged drop subject to a surface tension and rotating as a rigid body a relation between the rotational, coulomb, and surface energies can be devised which gives the maximum rotational energy such a drop can support and yet remain stable against fission. If 1/y is the ratio of rotational to surface energy, and x the usual fissionability parameter (ratio of coulomb energy to twice the surface energy), the critical value of rotational energy is given by

$$y_{cr} = 7/5 \ (1 - x)^2$$

in agreement with the formula given by Pik-Pichok.

A calculation of the fission barrier as a function of rotation (the energy difference between the lowest stable equilibrium configuration and the lowest saddle point configuration) leads to a formula for the fission barrier which differs somewhat from that given by Pik-Pichak. We find, (Z = 1-x)

$$\frac{E_B}{4x\,R^2_0} = \frac{49}{135} Z^3 - \frac{7}{3} Zy + \left(\frac{2}{3} y + \frac{14}{45} Z^2\right) \left\{\frac{49}{36} Z^2 + \frac{35}{12} y\right\}^{1/2} .$$

The general conclusion drawn from this work is that the introduction of a certain amount of rotational energy appears to be as significant in terms of lowering the fission barrier as the introduction of an equal amount of coulomb energy.

## C. Investigations of the Chemistry of the Transplutonium Elements

### 1. Americium and Curium Metal Products

(B. B. Cunningham and J. C. Wallmann.)

A considerable effort is being devoted to determining the crystal structures of americium and curium metals. The problem is being approached in two ways: (1) metal is condensed from the vapor phase as a thin film on a tantalum wire, and (2) a bulk phase of solid metal is produced.

The first technique consists of heating La metal with Am oxide in a tantalum crucible in vacuum. One millimeter above the crucible orifice is a Ta wire which may be rotated. The Am metal produced is vaporized from the crucible (crucible temp = 1200°C) and condenses on the Ta wire. The wire can be dropped into an x-ray capillary and sealed off without breaking the vacuum. This procedure has been tried so far only for rare earth metals but will be extended to Am.

The main effort has been devoted to developing the technique for producing bulk solid phases of the actinide metals. The principal refinement has been to remove all refractory oxides from the system and use an all tantalum assembly. (Fig. 2) Lithium or barium metal reductants are used and the scale of production has been varied from a few micrograms, for Cm, to 25 milligrams, for Np, Pu, or Am. The temperature of reduction appears to be critical for at too high a temperature the metal wets the Ta crucible walls and on the submilligram scale is difficult to separate as pure metal. At too low a reduction temperature the metal is poorly agglomerated or even incompletely reduced. The choice of reductant metal is dictated by the vapor pressure of the product metal. If the reductant and product metal vapor pressures are too close to each other then there is frequently difficulty in vaporizing all the excess reductant metal without losing much of

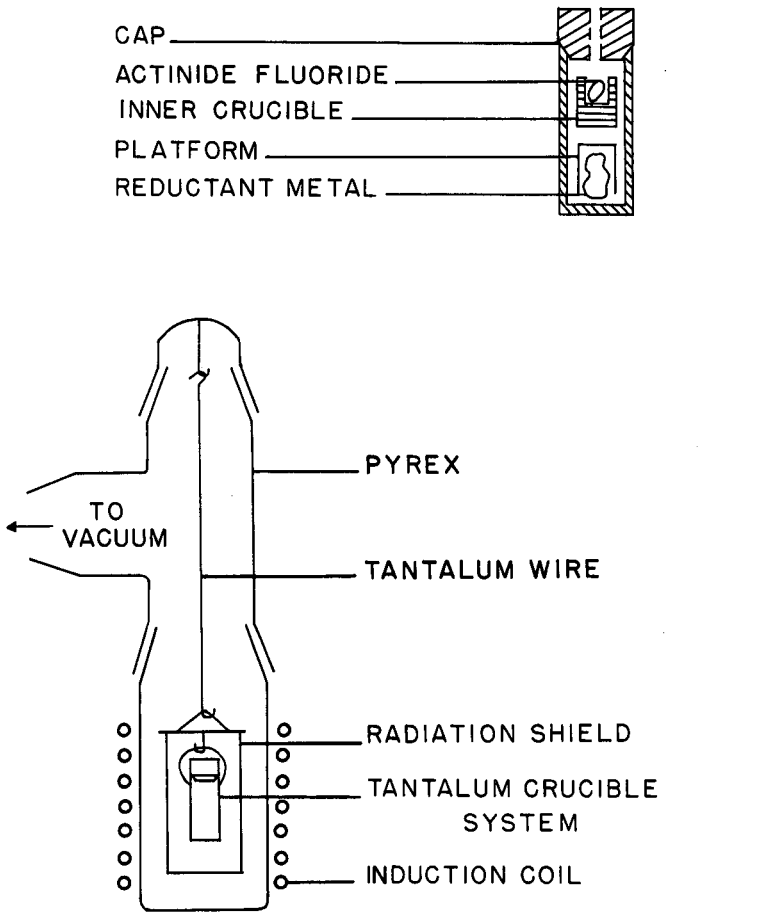


Fig. 2. Tantalum microscale reduction furnace.

the product as well. An additional consideration is the behavior of the LiF or $BaF_2$ produced: The former has a high enough vapor pressure so that it volatilizes completely and does not serve to prevent the product metal from wetting the Ta crucible wall as does $BaF_2$.

The objective in making pure bulk phases of Am and Cm is to determine the crystal structure, both room and high temperature, and prepare metal of known crystal structure for calorimetric studies. The x-ray work is currently being carried on. Curium metal has been produced, but, like Am, appears to crystallize poorly so no structures have as yet been identified. The planned measurement of the heat of solution of Cm metal should be completed by midyear. Relative vapor pressure measurements on Am and Cm may also be undertaken.

A complete report of this work will be presented at the September Munich meeting of the International Union of Pure and Applied Chemistry.

In association with the metal production work some effort is being devoted to producing and characterizing as bulk crystalline phases the compounds AmO and AmN. Both compounds have the fcc-NaCl type structure and we consider it worthwhile to know these lattice constants accurately in order to avoid confusion with fcc Am or Cm metal phases.

## 2. High Pressure Work with Actinide Metals

(D. B. McWahn, J. C. Wallmann, B. B. Cunningham, George Jura, and J. R. Vaisnys.)

We are doing some work on the high pressure transformations in various actinide metals. Our method consists of measuring the resistance as a function of pressure from 30,000 atmospheres up to about 150,000 atmospheres. We are interested in studying americium as the atomic spectra show that the 5f and 6d levels are quite close in energy and an electronic transition similar to that seen in cerium may take place under pressure.

Our sample consists of a piece of metal which fills a 13.5 mil diameter hole in a disk of silver chloride which is 8 mils thick. Around the disk of AgCl is placed a pipestone ring which is very strong with respect to radial pressure. This assembly is loaded between a set of anvils.

We encountered an interesting chemical problem in loading the samples: when a piece of reactive metal, such as one of the lanthanides or actinides, was pushed into the hole in the AgCl disk a reaction started to take place as noted by bubbling around the edges of the hole. This reaction could be stopped by blowing nitrogen on the ring while loading it. The surprising thing was the speed with which the reaction would start again if the stream of nitrogen was taken away (approximately 3-4 seconds). It is believed that the reaction is the dissolution of the metal by water, slightly acid, which is present on the AgCl surface; perhaps it is condensed by capillary action at the interface between the metal and the AgCl.

At present we have run neptunium, plutonium, and americium metal and preliminary results show a transition only in neptunium (at about 95,000 atm.). The possibility exists that some transformations occur below the 30,000 atmospheres pressure at which our studies began. An apparatus to study phase transformations in this low pressure region is being constructed.

### 3. Optical Spectroscopy of Rare Earth and Actinide +3 Ions

(John B. Gruber.)

Investigations are being undertaken to identify the electronic ground states and the low-lying excited states of the rare earth ions $Eu^{+3}$, $Gd^{+3}$, and $Tm^{+3}$ and the actinides $Am^{+3}$, $Cm^{+3}$, $Bk^{+3}$, and $Cf^{+3}$ in $LaCl_3$ as a matrix. Single crystals of the +3 ions in concentrations of 3 to 10% in $LaCl_3$ were grown by passing the sublimed material, sealed in quartz tubes, through a temperature controlled tube furnace at 900° C. The single crystals thus grown contain the +3 ions in $C_{3h}$ point symmetry at the ion sites in the lattice. $LaCl_3$ is isomorphous with the +3 ions investigated. The optical spectra observed for each one of the rare earth ions showed no impurity lines indicating that the impurity in the rare earths used was less than 0.1%.

The spectra of $Gd^{+3}$ (ground state: $^8S_{7/2}$) was studied in the u.v. Our results agree with those reported in the literature. There was observed a remarkable similarity between the $Gd^{+3}$ and the $Cm^{+3}$ spectra. The spectra of $GdCl_3 \cdot 6\ H_2O$ was also studied.

The optical spectra of $Eu^{+3}$ in the range from 2000 to 8000°A (ground state: $^7F_o$) was next considered. Single crystals of $EuCl_3 \cdot 6\ H_2O$ were grown

from solutions of 6 M HCl in a vacuum desiccator. There is some question as to the crystal structure of the hydrate $EuCl_3 \cdot 6\ H_2O$. Hellwege and Kahle[1] claim it is monoclinic, but their claims are based on unpublished data and from an analysis of their spectral data. Low lying states $^5D_0$, $^5D_1$, $^5D_2$ were observed in the hydrate spectra. These were presumably transitions from the $^7F_0$ ground state.

The optical spectra of the anhydrous $EuCl_3$ was also observed and the spectral lines measured. Single crystals containing 3% $EuCl_3$ in isomorphous $LaCl_3$ were grown from the melt. A half atmosphere of $Cl_2$ was placed over the material at all times so that no large amounts of $Eu^{+2}$ would be formed as a result of thermal decomposition of $Eu^{+3}$. The observed spectral lines agreed with those of Sayre and Freed and other workers.[2] One sees no lines in the yellow region of the spectrum (5800° A), two lines in the green (5200°A) and one line in the blue (4650°A). The spectra taken below 4000°A showed a continuum setting is due to the strong f to d electron transition from the very small amount of $Eu^{+2}$ present. We intend to compare the $Eu^{+3}$ spectra with that of $Am^{+3}$ in the near future.

The optical spectra of $Tm^{+3}$ (ground state: $^3H_6$) is being studied in order to find lines which have not yet been reported in the lierature, but which according to theory ought to be present. The excited $^3P_{0,1,2}$ states are expected by theory to be observed in the region from 2500°A to 3000°A. Difficulty in observing any transitions in this region are due to the strongly allowed electronic transition from 4f to 5d shells which result in a broad continuous band.

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1. Hellwege and Kahle, Zs. Physik 129, 62 (1951).
2. Sayre and Freed, BNL-2414

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## 4. Thermodynamics: Americium

(H. Conley.)

Thermodynamics data are essential to understanding the chemistry of any element. In the field of transuranic elements thermodynamic data are somewhat sparse and relatively inaccurate. To help remedy this situation it

seemed necessary to obtain an accurate measurement of the entropy change associated with the following reaction

$$H^+ + AmO_2^+ \longrightarrow AmO_2^{++} + 1/2\ H_2$$

With this entropy change the entropy of $AmO_2^+$ can be determined if the entropy of $AmO_2^{++}$ is taken to be equal to the entropy of $PuO_2^{++}$, namely -18 e.u. Gunn and Cunningham gave $\Delta H = +36.9 \pm 1.0$ kcal for

$$AmO_2^+ (aq) + H^+ (aq) = AmO_2^{++} (aq) + 1/2\ H_2 (g),$$

and using Penneman and Asprey's value of $E_f = -1.60$ v for this couple they calculate $S_{AmO_2^{++}} - S_{AmO_2^+} = -16 \pm 4$ e.u. To afford a check on these data the E and $\Delta H$ for the reaction given above will be measured. The $\Delta H$ will be measured calorimetrically and the E will be measured in the manner to be described below.

A Leeds and Northrup type K-2 potentiometer will be used for the potential measurement. The potentiometer can be read to 0.00002 v. A high sensitivity Leeds and Northrup galvanometer with the following properties will be used,

Sensitivity: .00036 $\mu$ amp/mm,
Critical External Damping Resistance: 10,000 ohms,
Period: 6.8 sec,
Resistance: 553 ohms.

A lamp and scale reading device will be used in conjunction with the galvanometer. An Eppley standard cell will be used in calibrating the potentiometer. The cell may be represented as follows:

$$Pt\ AmO_2^+;\ AmO_2^{++},\ 1\ M\ HCl\ O_4 \parallel 1\ M\ HCl\ O_4,\ H^+;\ H_2,\ Pt$$

An $AmO_2^{++}$ solution will be prepared electrolytically and this solution will be allowed to undergo autoreduction to the $AmO_2^+$ ion as the potential is measured. Total americium concentration will be measured and $AmO_2^{++}$ concentration measured spectrophotometrically thus the ratio $(AmO_2^{++})/(AmO_2^+)$ will be known. The junction between the two half cells will be made by means of an ungreased ground-glass joint so the resistance of the cell will be high and will have to be compensated for if best galvanometer operation is to be had.

The potential of the $Ce^{+3}$ - $Ce^{+4}$ couple has a value of -1.70 in 1.00 N HCl $0_4$ as reported by Smith and Getz. A measurement of this couple will be made to check the experimental procedure and equipment. In the preliminary experiments, the potential of the following cell was measured,

$$Pt,\ Ce^{+3};\ Ce^{+4}\ (\sim.1N),\ 1\ M\ H_2SO_4\ SAT\ KCl,\ Hg_2Cl_2;\ Hg$$

the cell reaction being

$$Ce^{+4} + Hg + Cl^- = 1/2\ Hg_2\ Cl_2 + Ce^{+3}.$$

A small U-shaped glass tube filled with AGAR Saturated with $KNO_3$ was the salt bridge. The ratio $(Ce^{++})/(Ce^{+3})$ was changed by adding $FeSO_4$ $(NH_4)_2SO_4$ -6 $H_2O$. Neglecting any junction potential and using concentrations instead of activities the potential of the cell could be calculated by:

$$E_{calc.} = E^{o}_{Ce^{+3}\ -\ Ce^{+4}} - 0.0591 \log \frac{(Ce^{+4})}{(Ce^{+3})} + E_{KCl\ (SAT);\ Hg_2Cl_2,\ Hg}$$

$E^{o}_{Ce^{+3}\ -\ Ce^{+4}}$ = -1.44 v as reported by Kunz.

$E_{KCl\ (SAT);\ Hg_2Cl_2,\ Hg}$ = 0.2415 v.

$(Ce^{+4})/(Ce^{+3})$ was determined by starting with a known $Ce^{+4}$ solution and adding weighed amounts of $FeSO_4$ $(NH_4)_2SO_4 \cdot 6H_2O$.

| E exp. | E calc. | $(Ce^{+4})/(Ce^{+3})$ |
|---|---|---|
| -1.47 | -1.49 | 8.827 |
| -1.45 | -1.47 | 4.009 |
| -1.43 | -1.46 | 2.323 |
| -1.41* | -1.44 | 1.000 |

*This value is extrapolated from a plot of E exp vs log $(Ce^{+4})/(Ce^{+3})$.

## 5. Chemistry of Berkelium and Californium

Preparations are being made to study the x-ray diffraction patterns of the oxides of berkelium and californium - in this case sub microgram amounts of the elements are available as $Bk^{249}$ and $Cf^{249}$. It is of course necessary to obtain the oxides in a high state of purity therefore considerable work must be done to obtain all of the reagents at ion exchange resins in a very pure form.

Some consideration has been given to the precipitation compounds such as the fluorides and the hydroxides in order to determine the appearances of the precipitates and attempt to estimate solubilities of the compounds.

A program has been undertaken to obtain more precise measurements of the magnetic susceptibilities of Bk and Cf then those reported at the last Geneva Conference. In the work being undertaken an attempt will be made to extend the measurements to liquid hydrogen temperatures since the lowest temperature reached in previous work was $77^{\circ}$ Kelvin. The sensitivity of the apparatus and the precision of measurements especially at very low temperatures was such that it seems feasible to attempt a measurement of the magnetic susceptibilities of einsteinium. Former measurements on californium were done with only 50 millimicrograms. Within this year, it is to be expected that about 10 millimicrograms of einsteinium should be obtained in which case the measurements will probably be attempted.