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HEATS OF FORMATION OF $\text{Pu}^{+3}(\text{aq})$, $\text{PuCl}_3(\text{c})$, $\text{PuOCl}(\text{c})$, $\text{Am}^{+3}(\text{aq})$, $\text{AmCl}_3(\text{c})$ and $\text{AmOCl}(\text{c})$

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Berkeley, California

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HEATS OF FORMATION OF Pu^{+3} (aq), PuCl_3 (c),
 PuOCl (c), Am^{+3} (aq), AmCl_3 (c) and AmOCl (c).

J. Fuger and B. B. Cunningham

March, 1963

HEATS OF FORMATION OF $\text{Pu}^{+3}(\text{aq})$, $\text{PuCl}_3(\text{c})$,
 $\text{PuOCl}(\text{c})$, $\text{Am}^{+3}(\text{aq})$, $\text{AmCl}_3(\text{c})$ and $\text{AmOCl}(\text{c})$.

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March, 1963

ABSTRACT

The following heats of solution have been measured: alpha plutonium in 6M HCl, -138.90 ± 0.9 ; alpha plutonium in 1.5M HCl, -138.65 ± 0.7 ; $\text{PuCl}_3(\text{c})$ in 0.001M HClO_4 -0.099M NH_4ClO_4 , -31.60 ± 0.35 ; $\text{AmCl}_3(\text{c})$ in 1.5M HCl, -30.60 ± 0.2 ; $\text{AmCl}(\text{c})$ in 0.001M HCl, -33.36 ± 0.25 kcal mol⁻¹, respectively.

On combination with other thermodynamic data these results yield the following standard heats of formation at 298°K: $\text{Pu}^{+3}(\text{aq})$, -138.6 ± 0.7 ; $\text{PuCl}_3(\text{c})$, -226.8 ± 0.7 ; $\text{PuOCl}(\text{c})$, -219.7 ± 1 ; $\text{Am}^{+3}(\text{aq})$, -162.4 ± 3 ; $\text{AmCl}_3(\text{c})$, -249.2 ± 3 ; $\text{AmOCl}(\text{c})$, -227.6 ± 3 , all in kcal per mole.

HEATS OF FORMATION OF $\text{Pu}^{+3}(\text{aq})$, $\text{PuCl}_3(\text{c})$,
 $\text{PuOCl}(\text{c})$, $\text{Am}^{+3}(\text{aq})$, $\text{AmCl}_3(\text{c})$ and $\text{AmOCl}(\text{c})$.*

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March, 1963

INTRODUCTION

Much of the available thermochemical data on the transuranium elements is based on measurements carried out a number of years ago when the samples of these materials were very small and often inadequately characterized as to crystallographic homogeneity or chemical purity. Because of the now increased availability of some of these elements in highly purified form a remeasurement of values seems desirable. For example, the only published value for the heat of solution of plutonium metal is that of Westrum and Robinson⁽¹⁾ who reported $-141.64 \pm 0.2 \text{ kcal mol}^{-1}$ in 6M HCl. However, owing to the early date at which these measurements were carried out no statement could be made concerning the allotropic form of the metal used. Since plutonium metal is now known to exist in six distinct crystallographic forms, some of which may be stabilized by relatively minor amounts of impurities and since the heats of transition from the alpha to epsilon phases sum to $1.7 \text{ kcal mol}^{-1}$ the previously reported value could be in error by nearly two kilocalories.

Again, the present values for the heats of formation of $\text{Am}^{+3}(\text{aq})$, $\text{AmCl}_3(\text{c})$ and $\text{AmOCl}(\text{c})$ assume the heat of solution of $\text{AmCl}_3(\text{c})$ to be the same as that of $\text{PuCl}_3(\text{c})$, without experimental verification.

The present paper reports a measurement of the heat of solution of highly purified alpha plutonium (kindly given to us by the Los Alamos Scientific Laboratory) in both 6M and 1.5M HCl, as well as the heats of solution of

$\text{PuCl}_3^{(c)}$ in 0.099M NH_4ClO_4 —0.001M HClO_4 , and of $\text{AmCl}_3^{(c)}$ in 1.5M and 0.001M HCl .

EXPERIMENTAL

Materials.

A. Plutonium Metal.

About two grams of plutonium were furnished by Los Alamos with the identifying designation "A. E. C. 437-SAN-5000-11, HOO 572124-PMR-22". The sample was in the form of a single piece of metal and was labeled as the alpha (monoclinic) form. The X-ray diffraction pattern obtained by us from a small piece of the sample was consistent with the data⁽²⁾ reported for the structure of alpha plutonium. As a further check on the structure a density determination was carried out on the main piece. The density was found to be $19.8 \pm 0.2 \text{ gms cm}^{-3}$ in good agreement with the bulk density value of $19.63 \text{ gms cm}^{-3}$, reported⁽³⁾ for the alpha form.

The chemical purity of the metal, as determined at Los Alamos, is indicated by the data listed in Table I.

Because of the extreme hardness of alpha plutonium we found it necessary to fracture, rather than cut the large piece to obtain samples for calorimeter runs. This was done by wrapping the piece in tungsten foil and applying pressure. The smaller pieces were inspected microscopically for cleanliness before weighing on a quartz fiber torsion balance (Rodder Model E Microtech Services Company, Berkeley, California) sensitive to $\sim 0.05 \mu\text{gm}$.

The samples were weighed in air and were then sealed into pyrex calorimeter bulbs in a dry argon atmosphere, by heating in the neck of the bulb a pyrex bead coated with Apiezon W wax.

B. Plutonium and Americium Trichlorides.

The starting materials were HCl solutions of the chlorides purified by ion exchange. These solutions were evaporated to dryness in a stream of $\text{HCl}_{(g)}$, sublimed in vacuum and finally grown in the form of a single crystal by the technique of gradient solidification from the melt, as described by Gruen, Conway, McLaughlin and Cunningham, ⁽⁴⁾ for lanthanum trichloride. Under the conditions described the hexagonal trichlorides yield a single oriented crystal, the c axis being parallel to the temperature gradient of the furnace. About 100 microgram pieces of these crystals were analyzed by copper spark spectrographic emission analysis. No impurities were detected ⁽⁵⁾ in these samples.

The plutonium trichloride used for the calorimeter measurements was taken from a single crystal, grown as described and weighing about 2.5 grams. The AmCl_3 single crystal weighed about 0.7 gm.

Due to the marked hygroscopicity of the trichlorides, special care was taken to handle them in a suitably dry nitrogen atmosphere, produced by boiling the liquid and subsequently passing the gas through a trap at near liquid nitrogen temperature. The dry nitrogen was introduced at a high flow rate into a metallic gloved box in which the samples were prepared. Items were introduced by means of a vacuum lock. The samples (one or two pieces) were sealed into pyrex calorimeter bulbs of known volume using a pyrex bead coated with Apiezon W and a hot wire.

The weighings were made to a few micrograms accuracy on an Ainsworth Type V. M. Assay Balance. Each gross and tare weighing was repeated at least four times. Corrections were made for differences in density between air and nitrogen and the weight finally reduced to weight in vacuum.

C. Solutions

Solutions of HCl, HClO₄ and HClO₄-NH₄ClO₄ were prepared from analytical grade reagents. The acids were standardized volumetrically to within $\pm 0.2\%$ against constant boiling sulfuric acid. Solvent acids for PuCl₃(c) and AmCl₃(c) were previously de-aerated with argon. Plutonium metal was dissolved in air-free acid saturated with hydrogen. The solutions were loaded and the calorimeter assembled in a gloved box which had been flushed with argon for several hours. Mass analysis of the atmosphere of the box at time of loading indicated that the oxygen content was less than 0.02 volume percent. Although no oxidation to Pu⁺⁴ was to be expected under the conditions of our experiments the solutions were checked spectrophotometrically, with a Cary Model 14 Recording Spectrophotometer. Only trivalent plutonium was found in the calorimeter solutions (Limit of detection of Pu⁺⁴ approximately 1%). We also checked the completeness of dissolution of plutonium metal in 1.5M HCl by centrifuging the calorimeter solution and checking for alpha activity in the microscopic amount of washed solid residue. No plutonium-containing solid was detected.

Calorimeter.

Some features of the microcalorimeter used for our measurements have been described previously^(6,7) but the instrument has since been modified to increase its stability and to convert it to a recording instrument. These modifications will be described in detail elsewhere.⁽⁸⁾ It will suffice here to state that it has a heat capacity of approximately 9 calories, a temperature sensitivity of approximately $1 \times 10^{-5} \text{ }^\circ\text{C}$ and a thermal leakage modulus of about $5 \times 10^{-3} \text{ min}^{-1}$. It was thermostated at 25.03°C , but the steady state temperature was from 0.1 to 0.3° higher

due to radioactive heating, power input to bridge, etc. In some of our measurements the heat of breakage of the calorimeter bulbs was an important limitation on the accuracy of the measurements. Typically, this heat effect, determined by breaking several bulbs containing a small amount of water, was found to amount to $(6 \pm 3) \times 10^{-4}$ calories bulb⁻¹. This correction was applied to all measurements. In addition, the results were corrected, where necessary, for the heat of saturation by H₂O(g) and HCl(g) of the dry gas enclosed initially in the calorimeter bulbs, and for the saturation of the hydrogen evolved from the metal. In all measurements the heat evolution was complete within 3 minutes.

A potential divider in the calorimeter circuit allowed a continuous recording of the calorimeter chamber temperature—hence the computation of accurate corrections for thermal leakage. Heat inputs of the order of 0.2 calories were reproducible to $\pm 0.1\%$. Usually, three or more heat inputs preceded a chemical reaction and three followed it.

As a final check on the performance of the calorimeter a number of runs were made on the heat of solution of pure Mg metal in 1M HCl. The results are given in Table II. The average of these values is in reasonable agreement with the figure of -111.322 ± 0.041 Kcal determined on a much bigger scale by Shomate and Huffman. (9)

Units, Auxiliary Thermodynamic Data, and Limits of Error.

In our thermochemical calculations we have taken the calorie to be equal to 4.1840 absolute joules.

The limits of error quoted for our experimental data are for the 95% confidence interval, but we have not attempted to retain this limit when combining our data with that from other sources, for which we take the error to be that stated by the author.

The heat measurements reported here were made at temperatures a few tenths of a degree above 298.16°K , but within their limits of error they are not distinguishable from 298.16°K values. Hence, we have treated them as such.

On the basis of mass analysis data we calculate 239.11 and 241.07, respectively, for the atomic weights of the plutonium and americium stocks used.

All auxiliary thermodynamic data, for which no reference is given, have been taken from "Selected Values of Chemical Thermodynamic Properties", U. S. Bureau of Standards Circular 500 (1952).

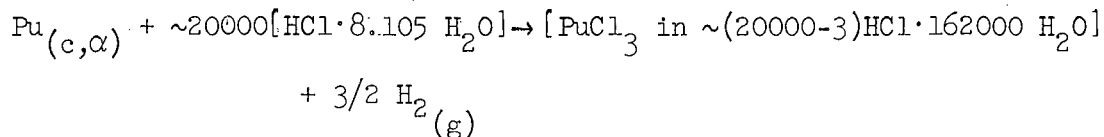
RESULTS AND DISCUSSION

The experimentally observed heats of solution of alpha plutonium are tabulated in Table III.

Our value of $-138.90 \pm 0.9 \text{ Kcal mol}^{-1}$ for the heat of solution of alpha plutonium metal in 6M HCl may be compared directly with that of -141.64 ± 0.2 reported by Westrum and Robinson.⁽¹⁾ A part of the discrepancy of $2.74 \text{ Kcal mol}^{-1}$ may be explained by the fact that early microscale preparations of plutonium yielded soft malleable metal of density ca 16, which corresponds to one of the phases δ , δ' or ϵ —all of which have a substantially higher heat content than that of the alpha phase.

If we assume Westrum and Robinson's metal to have been pure epsilon, the heats of transformation reported by Kay⁽¹⁰⁾ give a calculated heat content of $1.74 \text{ Kcal mol}^{-1}$ above that of the alpha phase. We are unable to account for the remaining one kilocalorie difference. It is of course remarkable that Westrum and Robinson were able to obtain such reliable results at a time when almost nothing was known about the properties of plutonium metal.

Our experimental heat in 6M HCl corresponds to the reaction:



Neglecting the slight change in composition of the 6M HCl due to consumption of HCl in the dissolution of the metal, and taking the relative heat content of HCl in 6M HCl ($\text{HCl} \cdot 8.105 \text{H}_2\text{O}$) to be $-36.81 \text{Kcal mol}^{-1}$, we calculate the heat of formation of PuCl_3 in $\sim 20000[\text{HCl} \cdot 8.105 \text{H}_2\text{O}]$ to be $-249.33 \pm 0.9 \text{Kcal mol}^{-1}$.

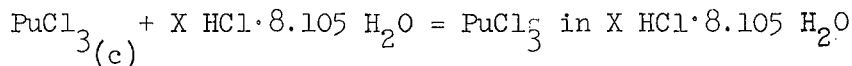
The calculation also ignores the influence of very low concentrations of PuCl_3 on the HCl heat content.

Westrum and Robinson⁽¹⁰⁾ measured the heat of solution of $\text{PuCl}_{3(c)}$ in various concentrations of HCl. They report values of $-22.15 \pm 0.1 \text{Kcal mol}^{-1}$ in 6M HCl, -29.5 ± 0.1 in 1.5M HCl and -31.76 ± 0.1 in 0.1M HCl.

We have measured the heat of solution of $\text{PuCl}_{3(c)}$ in 0.001M HClO_4 - 0.099M NH_4ClO_4 with the results shown in Table IV.

Our value for the heat of solution of $\text{PuCl}_{3(c)}$ in very dilute acid agrees with that of Westrum and Robinson in 0.1M HCl well within the accuracy of the measurements. We therefore believe their results at other HCl concentrations to be accurate.

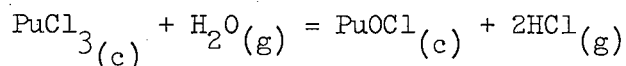
For the process:



Westrum and Robinson report $-22.15 \pm 0.1 \text{Kcal mol}^{-1}$. From this and our metal data in 6M HCl we calculate the heat of formation of $\text{PuCl}_{3(c)}$ to be $-227.2 \pm 0.9 \text{Kcal mol}^{-1}$.

From our measurements on the heat of solution of the metal in 1.5M HCl and Westrum and Robinson's value for the heat of solution of $\text{PuCl}_{3(c)}$ in 1.5M HCl, we calculate $\Delta H_f^\circ \text{PuCl}_{3(c)} = -226.6 \pm 0.7 \text{Kcal mol}^{-1}$. Weighting each

of the two values in proportion to the numbers of observations on which it is based gives -226.8 ± 0.7 kcal mol⁻¹ as our best value for the heat of formation of PuCl₃(c). This value may be combined with the value of 20.8 ± 0.7 kcal for the heat of the reaction



reported by Sheft and Davidson⁽¹²⁾ to give a corrected value, -219.7 ± 1 kcal mol⁻¹, for the heat of formation of PuOCl(c).

Extrapolation of the heat of solution data for PuCl₃(c) obtained by Westrum and Robinson gives for the heat of solution of PuCl₃(c) at infinite dilution -31.9 ± 0.2 kcal mol⁻¹. Combining our value for the heat of formation of PuCl₃(c) with this datum, gives for Pu⁺³(aq), $\Delta H_{\text{Pu}^{+3}}^{\circ}(\text{aq}) = -138.6 \pm 0.7$ kcal mol⁻¹.

Lohr and Cunningham⁽¹³⁾ measured the heat of solution of americium metal, of unspecified crystal structure, in 1.5M HCl and reported -162.3 ± 2.7 kcal mol⁻¹ for the solution reaction.

It is now known⁽¹⁴⁾ that the room temperature stable modification of americium is a double hexagonal close packed phase and that this is normally obtained in small scale reductions. It is likely that this was the form of americium metal used in the heat-of-solution measurements. It is our intention to repeat the measurements using americium metal of known crystallographic form, but for the present we wish only to report our values for the heat of solution of AmCl₃(c) in HCl solutions. As mentioned previously, the samples used for the heat measurements were chipped from a single 0.7 gram crystal of AmCl₃ having the hexagonal LaCl₃ type of structure.

The thermochemical data are summarized in Table V. In calculating their values for the heats of formation of AmCl₃(c) and Am⁺³(aq), Lohr and Cunningham took the heats-of-solution of AmCl₃(c) in HCl solutions of various

concentrations to be the same as those for plutonium trichloride. As is evident from Table V they are actually about 1.1 Kcal/mole more negative. From a compilation of data assembled by Spedding and Flynn⁽¹⁵⁾, the heats of solution of the light rare earth trichlorides (which are isostructural with PuCl_3 and AmCl_3) are shown to become increasingly negative by about 1.3 Kcal/mole per unit increase in atomic number.

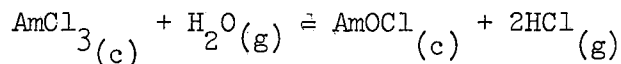
Using our value for the heat of solution of $\text{AmCl}_3(c)$ in 1.5M HCl and the value of -162.3 for the heat of solution of americium metal in 1.5M HCl, we calculate:

$$\Delta H_f^\circ \text{AmCl}_3(c) = -249.2 \pm 3 \text{ Kcal mol}^{-1}.$$

Taking -33.36 ± 0.25 for the heat of solution of $\text{AmCl}_3(c)$ in 10^{-3}M HCl to be equivalent to that at infinite dilution, we find:

$$\Delta H_f^\circ \text{Am}^{+3}(aq) = -162.4 \pm 3 \text{ Kcal mol}^{-1}.$$

Finally, using the data reported by Koch and Cunningham⁽¹⁶⁾ on the temperature dependence of the equilibrium constant for the reaction,



we obtain:

$$\Delta H_f^\circ \text{AmOCl}(c) = -227.6 \pm 3 \text{ Kcal mol}^{-1}.$$

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FOOTNOTES AND REFERENCES

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(b) F. Anselin, Ibid., Chapter XI.
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4. D. M. Gruen, J. G. Conway, R. D. McLaughlin and B. B. Cunningham, J. Chem. Phys. 24, 1115 (1956)
5. Limits of detection (in μgms) by this method are as follows:
0.01—Ag, Al, Ba, Be, Ca, Cr, Eu, Fe, Ga, La, Mg, Mn, Mo, Nb, Sc, Si, Sr, Ti, Yb, Y, Zr;
0.05—Am, Bi, Co, Dy, Er, Gd, Ge, Hf, Ho, In, Lu, Ni, Sm, Tm;
0.1—Ce, Nd, Np, Os, Pb, Pd, Pr, Pt, Re, Rh, Ru, Sn, Ta, Tl, W, Zn;
0.5—As, Ir, Pu, Sb, Tb, Th, U;
1—Au, Cm, K, Na.

Barium was determined specially in its most sensitive wavelength region. Hg, Li, P, Te, Co, Rb were not analyzed for, nor was copper, obviously.

Pulse analysis showed the samples to be free of significant amounts of extraneous alpha emitters.

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TABLE I. Analysis of High Purity Alpha Plutonium

(results expressed in ppm)				
Li < 0.2	Si < 10	Bi < 1	B < 0.5	W = 40 ± 1
Be < 0.1	Pb < 2	Co < 10	F < 2	Zr < 5
Na < 10	Cu < 2	Zn < 10	Ni < 20	Th < 40
Ca < 10	Cr < 10	C = 40	U < 25	Mg < 5
Al = 25	Mn < 1	O = 90	Am = 0.044	Pu = 99.99%
La < 10	Sn < 1	Fe = 45	Ta < 35	

Note: The numbers following "<" are limits of detection.

TABLE II. Heat of Solution of Magnesium Metal in 1M HCl

Weight of Sample (Micrograms)	Heat Evolved (Calories)	$\Delta H_{298.3}$ (Kcal mol ⁻¹)
1. 51.97	0.2877	-111.20
2. 65.85	0.3023	-111.61
3. 84.17	0.3846	<u>-111.10</u>
	Average:	-111.30 ± 0.56

TABLE III. Heat of Solution of Alpha Plutonium Metal in HCl Solutions

Medium	Weight of Sample (Micrograms)	Pu ⁺³ Molarity	Heat Evolved (Calories)	$\Delta H_{298.3}$ (Kcal mol ⁻¹)
HCl 1.500M	494.7	2.587×10^{-4}	0.2877	-139.0
"	597.5	3.124×10^{-4}	0.3454	-138.2
"	590.3	3.087×10^{-4}	0.3413	-138.2
"	901.2	4.712×10^{-4}	0.5247	-139.2
			Ave:	-138.65 ± 0.7
HCl 6.00M	696.8	3.643×10^{-4}	0.4042	-138.7
"	586.6	3.067×10^{-4}	0.3413	-139.1
			Ave:	-138.90 ± 0.9

TABLE IV. Heat of Solution of PuCl_3 in 0.001M HClO_4 - $0.099\text{M NH}_4\text{ClO}_4$.
 3(c)

Weight of Sample (Milligrams)	Pu^{+3} Molarity	Heat Evolved (Calories)	$\Delta H_{298.3}$ (Kcal mol ⁻¹)
3.976 ± 0.005	$1.439 \times 10^{-3}\text{M}$	0.3637	-31.61
4.733 ± 0.005	$1.713 \times 10^{-3}\text{M}$	0.4310	-31.46
5.727 ± 0.005	$2.072 \times 10^{-3}\text{M}$	0.5271	<u>-31.80</u>
Weighted average:			-31.60 ± 0.35

TABLE V. Heat of Solution of $\text{AmCl}_3^{(c)}$ in HCl Solutions

Medium	Weight of Sample (Milligrams)	Am^{+3} Molarity	Heat Evolved (Calories)	$\Delta H_{298.4}$ (Kcal mol ⁻¹)
HCl 10 ⁻³ M	6.138 ± 0.004	2.207 x 10 ⁻³	0.5900	-33.40
"	6.677 ± 0.005	2.402 x 10 ⁻³	0.6402	<u>-33.31</u>
			Ave:	-33.36 ± 0.25
HCl 1.500M	7.193 ± 0.006	2.588 x 10 ⁻³	0.6317	-30.51
"	7.336 ± 0.007	2.639 x 10 ⁻³	0.6478	-30.68
"	5.683 ± 0.004	2.045 x 10 ⁻³	0.5007	<u>-30.61</u>
			Ave:	-30.60 ± 0.20

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