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**The Enthalpies of Solution of
Selected Elements in
Several Liquid Metal Solutions**

by

David F. Bowersox

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ABSTRACT

The calculation of partial molar enthalpies and entropies of elements that form simple eutectic-type binary systems with plutonium are reviewed. The partial molar enthalpies of solution of several elements that form compounds or solid solutions with plutonium have been calculated by regular solution theory from solubility data. The calculated enthalpies are (in kcal/mole): carbon, 10.5; thulium, 9.2; manganese, 4.8; rhenium, 10.5; zirconium, 4.2; titanium, 4.1 from 700 to 750°C, and 5.3 from 800 to 1000°C.

The regular solution theory has also been applied to other systems to calculate enthalpies from published solubility data. These results are compared with an empirical approach.

PART I

THE ENTHALPIES OF SOLUTION OF CARBON, THULIUM, TITANIUM, MANGANESE, ZIRCONIUM, AND RHENIUM IN LIQUID PLUTONIUM

A. INTRODUCTION

The solubilities of tungsten, tantalum, niobium, vanadium, molybdenum, chromium, carbon, thulium, manganese, rhenium, zirconium, and titanium have been measured in liquid plutonium over the temperature range 700 to 1000°C.^{1,2} These studies initially established a series of reliable measurements of the solubilities of sparingly soluble elements in liquid plutonium. The solute elements and the plutonium were of high purity, and the sampling techniques and experimental procedures gave reproducible data that demonstrated solution equilibrium in the ranges of solute concentrations that were examined. The solubility data are of special significance because of possible applications in the liquid-metal reactor field and in studies of corrosion technol-

ogy, heat transfer, and metal processing.

As more data have become available, the measurements have been used for theoretical treatment of the thermodynamic properties of dilute liquid-plutonium solutions. The partial molar enthalpies and entropies of solution for tungsten, tantalum, niobium, vanadium, molybdenum, and chromium in liquid plutonium have been calculated by treatment of the solubility data by least-squares and regular solution theory.³ These binary systems exhibit simple eutectic-type behavior, with the eutectic point occurring very close to pure plutonium.⁴ No solubility in solid plutonium has been reported for these elements.

The binary systems of plutonium with the remaining elements are more complex.^{4,5} Carbon, thulium,

manganese, and rhenium all react to form compounds, and both titanium and zirconium form solid solutions with plutonium in the temperature range of interest.

In this section, the studies of the simple eutectic-type systems will be reviewed and calculations of the partial molar enthalpies and entropies of the more complex systems will be considered in detail.

B. DISCUSSION

The general equation

expresses the equilibrium reaction between a solid component A and a liquid solvent B. In the solubility studies, all measurements were made under conditions such that an excess of the solid component was present. The solubilities of the solute at 850°C and the form of the solid component in equilibrium with the solution are listed in Table I. These components may be classified as pure element (simple eutectic-type binary), compound, and solid solution. The titanium-plutonium system is quite complex. Below 770°C, solid solution β -Ti is found in ϵ -Pu; a peritectic point occurs at 770°C, and above 770°C, Pu is soluble in β -Ti.

The equilibrium expression for all these systems

should be

$$R \ln N_1 = \Delta \bar{S}_1 - \Delta \bar{H}_1^*/T, \quad (2)$$

where R is the gas constant, N_1 is the mole fraction of solute in a saturated solution at absolute temperature T, $\Delta \bar{S}_1$ is the partial molar entropy of solution, and $\Delta \bar{H}_1^*$ is the partial molar enthalpy of solution of solute 1. For Eq. (2) to be valid, it will be assumed that the heat capacities of supercooled liquid solute and solid solute at a given temperature are equivalent. The reference state for the solute in each case will be taken as pure, supercooled liquid solute at the temperature of interest.

1. Empirical Approach

The enthalpies and entropies of solution of the solutes for the simple eutectic-type binaries were calculated from the solubility data by least-squares fitting to Eq. (2).^{1,2} The results are listed in Table II. For these elements, the enthalpy of solution varies from 9.4 to 21.2 kcal/mole, and the entropy from 1.28 to 6.04 e.u./mole. Such large variations in similar solutions would be rather surprising. The entropy of solution of similar solutes in the same solvent would appear, at least empirically, to be rather constant. However, the least-squares treatment is applicable and the resulting

TABLE I
SOLUBILITIES AND SOLID PHASES OF SELECTED
ELEMENTS IN LIQUID PLUTONIUM AT 850°C

Element	Solubility at 850°C $N \times 10^3$ ^a	Solid Phase
C	26.7	PuC_{1-x}
Ti ^b	--	β -Ti in ϵ -Pu
Ti ^c	261	Pu in β -Ti
V	22.9	V
Cr	43.5	Cr
Mn	308	PuMn_2
Zr	80.3	Zr in Pu
Nb	20.5	Nb
Mo	30.8	Mo
Tm	50.4	PuTm_2
Ta	1.90	Ta
W	0.167	W
Re	27.5	PuRe_2

^a Refs. 1 and 2.

^b $t < 770^\circ\text{C}$.

^c $t > 770^\circ\text{C}$.

TABLE II
ENTROPIES AND ENTHALPIES OF SOLUTION
CALCULATED BY LEAST-SQUARES FITTING FOR
SELECTED ELEMENTS IN LIQUID PLUTONIUM

Element	Calculated Constants	
	Entropy (e. u.)	Enthalpy (kcal/mole)
W	1.60	21.2
Ta	3.52	18.0
Nb	1.28	10.0
V	3.66	12.5
Mo	1.51	9.42
Cr	6.04	13.9
Mn	6.90	10.4
C	2.39	10.5
Tm	3.37	10.4
Re	0.89	9.0
Zr	13.1	20.2
Ti ^a	8.69	23.0
Ti ^b	3.38	6.8

^a In temperature range 700 to 750°C.

^b In temperature range 800 to 1000°C

TABLE III

THE PARTIAL MOLAR ENTHALPIES OF SOLUTION
AND δ_i VALUES OF SELECTED ELEMENTS IN
LIQUID PLUTONIUM CALCULATED BY REGULAR
SOLUTION THEORY
(ΔS taken as 2.3 e. u.)

Element	ΔH_i^* , kcal/mole	δ_i^a
W	22.0 ± 0.1	119
Ta	16.6 ± 0.1	110
Nb	11.1 ± 0.1	102
V	11.0 ± 0.1	107
Mo	10.3 ± 0.1	101
Cr	9.5 ± 0.1	106

^aCalculated by Eq. (4).

equations represent an accurate expression of the solubility.

2. Regular Solution Approach

The second approach employed is regular solution theory. In this approach, the excess entropy of solution is assumed to be negligible. For elements that form simple eutectic-type binaries, and for which the entropy of fusion is estimated to be 2.3 e. u.,⁶ Eq. (2) becomes

$$R \ln N_i = 2.3 - \Delta H_i^*/T. \quad (3)$$

This equation was used with the solubility data to calculate enthalpies of solution. Results for tungsten, tantalum, niobium, vanadium, molybdenum, and chromium are given in Table III. The correction factors that are due to increasing concentration of solute with temperature are minor and will be omitted. These enthalpies are internally consistent throughout the 700 to 1000°C temperature range. Standard deviations in the calculated values averaged less than 0.1 kcal/mole.

Hildebrand and Scott⁷ proposed solubility parameters (δ) for binary systems. These constants, which are based upon the values of cohesive densities of the elements, are characteristic of each element and can be applied to calculate a theoretical solubility value. Conversely, an experimental parameter can be calculated if the solubility is known. For these simple eutectic-type systems, experimental solubility parameters were determined by calculating the enthalpies, assuming that δ_{Pu} is 81,⁸ and applying the equation

$$\delta_i = \delta_{Pu} + [(\Delta H_i^* - \Delta H_f) / \bar{V}_i]^{1/2}, \quad (4)$$

where $\bar{V}_i$ is the molar volume of solute i . Results are given in Table III. These parameters averaged 0.84 ± 0.04 of the theoretical values.⁸

3. Enthalpies of Elements Which Form Compounds with Plutonium

Carbon, thulium, manganese, and rhenium form compounds with plutonium.^{4,5} The reference state for these systems will still be the pure, supercooled liquid element at the temperature of interest. The series of reactions at a given temperature may then be expressed in steps as

and

These steps indicate that the overall reaction is analogous to that of the simple eutectic-type binary system if essentially no interatomic bonding remains in the solution. The assumption will be made that the solute, at least in dilute solution, is randomly distributed.

Plots of the logarithm of the solubility (in atom percent solute) against the reciprocal of the absolute temperature are presented for these elements in Figs. 1 through 4. These plots are linear, which indicates that equations having the form of Eqs. (2) and (3) are valid. The equations from the least-squares treatment of the solubility data were used to draw the lines in these figures. Data for the manganese system were limited to three temperatures: 850, 950, and 1000°C. The concentration of manganese is high in these solutions, and correction factors would be large. Extrapolations beyond this temperature range may not be valid. The enthalpies and entropies calculated by the least-squares method are given in Table II. Entropies range from 0.89 to 6.90 e. u. Enthalpies are from 9.0 to 10.5 kcal/mole.

Equation (3) was applied to the solubility data to test the regular solution theory. Results are shown as a function of temperature in Table IV. The correction factor due to the increasing solute concentrations, except for manganese, averages about 5% and was not applied. There is no evidence of temperature dependence

TABLE IV

THE PARTIAL MOLAR ENTHALPIES OF SOLUTION
FOR C, Tm, Re, AND Mn IN LIQUID Pu CALCULATED
BY REGULAR SOLUTION THEORY

Temp, °C	Enthalpy of Solution, ^a kcal/mole			
	C	Tm	Re	Mn
700	10.5	9.5	10.5	---
750	10.7	9.4	10.4	---
800	10.5	9.2	10.5	---
850	10.6	9.2	10.5	5.2
900	10.5	9.0	10.6	---
950	10.4	9.2	10.6	4.7
1000	---	9.2	---	4.5
Av	10.5 ± 0.1	9.2 ± 0.1	10.5 ± 0.1	4.8 ± 0.3

^a Calculated by the equation

$$\Delta \bar{H} = [\Delta S_f / 4.575 - \log N_i] 4.575 T.$$

in the calculated enthalpies. The enthalpies calculated by regular solution theory are consistent, with standard deviations averaging 0.1 kcal/mole.

The solubility parameters would have little, if any, meaning in these solutions. In fact, the thulium-plutonium binary was initially grouped with the simple eutectic-type binaries. However, the solubility parameter calculated for thulium from the data by Eq. (4) was nearly 1.2 times the theoretical value. Ellinger et al.⁹

Fig. 1. Solubility of carbon vs reciprocal temperature.

Fig. 2. Solubility of thulium vs reciprocal temperature.

report an intermediate phase, stable only at high temperatures, for both plutonium-terbium and plutonium-dysprosium at approximately 30 at. % plutonium. Such intermediate phases may be present in other heavy rare-earth binaries with plutonium. In this case, the analogous compound would be PuTm₂.

4. Enthalpies of Solution of Elements Which Form Solid Solutions with Plutonium

Zirconium and titanium form solid solutions with plutonium. Although the solid phase transitions in the plutonium-zirconium system are complex below 640°C, the liquidus-solidus above 640°C is that of simple solid solution.⁴ The reaction may be written as

Below 770°C, the equilibrium reaction in the plutonium-titanium system is⁵

At 770°C, a peritectic isotherm occurs, and above this temperature the reaction is

Equation (2) should remain valid for these solutions, and

Fig. 3. Solubility of rhenium vs reciprocal temperature.

regular solution theory should be applicable if a correction term for the entropy change from pure, supercooled solute to the solid solution state is applied.

The correction factor for changes in activity coefficients with concentration should be approximately equal for these solutions because solubilities are approximately the same in both solid and liquid. The plots of the logarithm of the solubility in liquid plutonium against the reciprocal temperature are shown in Figs. 5 and 6.

Fig. 4. Solubility of manganese vs reciprocal temperature.

The plots are linear, and the lines represent the least-squares, best-fit equation computed with the data. The change in slope for titanium at 770°C is represented by the broken vertical line in Fig. 6. The constants obtained for Eq. (2) by least-squares treatment of data are given in Table II. The entropy terms for zirconium and for titanium below the peritectic are large. There is a definite change in entropy and enthalpy values at the peritectic isotherm.

Fig. 5. Solubility of zirconium vs reciprocal temperature.

Fig. 6. Solubility of titanium vs reciprocal temperature.

If the additional entropy of solution in solid solution is configurational, the total entropy in the system would be the sum of the entropy of fusion, the entropy of configuration (that is, $R \ln N_i^S$ is the mole fraction of solute i in a saturated solid solution at a given temperature), and the excess entropy. In a regular solution, the last term is negligible, and for these systems, Eq. (2) may be written as

$$\Delta \bar{H}^* = T [2.3 - R(\ln N_i^S - \ln N_i)] \quad (11)$$

Values of N_i^S for zirconium and titanium were estimated from the phase diagrams.^{4,5} Partial molar enthalpies were then calculated by Eq. (11) from the measured solubilities in liquid plutonium.^{1,2} Results are given in Table V. These results appear to be temperature-independent. The enthalpy of solution of titanium appears to shift between 750 and 800°C from 4.1 to 5.3 kcal/mole. These enthalpy values are limited by the

assumptions made in deriving Eq. (11) and the accuracy of the estimates of the solid solubilities.

TABLE V
THE PARTIAL MOLAR ENTHALPIES OF SOLUTION
FOR Zr AND Ti IN LIQUID Pu CALCULATED
BY REGULAR SOLUTION THEORY

Temp. °C	Enthalpy of Solution, kcal/mole ^a	
	Zr	Ti
700	4.1	4.2
750	4.3	4.0
800	4.3	5.3
850	4.2	5.3
900	4.2	5.3
950	4.1	5.3
1000	---	5.4
Av	4.2 ± 0.1	---

^aCalculated by the equation

$$\Delta \bar{H}^* = [2.3 - R(\ln N_i^S - \ln N_i)] T.$$

PART II CALCULATED ENTHALPIES IN SEVERAL DILUTE SOLUTIONS

A. INTRODUCTION

The regular solution model approach to liquid-plutonium systems, as discussed in Part I, appears to be successful. It should be applicable to other binary pairs if the underlying assumptions are valid in the system to be considered.

Several such systems will be discussed in this section. In each case, the solute is sparingly soluble, the solubility data appear internally consistent, and the experimental techniques appear to be excellent. The studies, which are all recent, include the solubilities of selected elements in liquid tin,¹⁰ of tantalum and tungsten in liquid rare-earth metals,^{11,12} and of 12 elements in liquid sodium.¹³ The enthalpy of solution will be calculated by the regular solution approach for each solute from the solubility data published in these studies.

1. Selected Elements in Liquid Tin

Darby and Jugle¹⁰ recently measured the solubilities of titanium, vanadium, chromium, iron, and cobalt in liquid tin from 500 to 950°C. Four of these elements form stable intermediate intermetallic com-

pounds with tin; the fifth, chromium, forms a simple eutectic-type binary system with tin.¹⁴ Therefore, the approaches applied to plutonium systems should be valid. Calculations based upon this data are summarized in Table VI. In the empirical approach, once again, large differences occur in calculated entropies for similar solutes in a given solvent. The entropy of solution in the regular solution approach, however, is defined as constant. Variations of the calculated enthalpies by this second approach are less than 4% in each case.

TABLE VI
ENTROPIES AND ENTHALPIES OF SOLUTION
IN LIQUID TIN

Element	Empirical		Regular $\Delta \bar{H}^*$
	$\Delta \bar{S}$	$\Delta \bar{H}$	
V	0.45	17.2	15.0 ± 0.4
Ti	5.7	15.9	12.3 ± 0.4
Cr	7.3	16.2	10.5 ± 0.4
Fe	1.1	8.1	9.4 ± 0.2
Co	1.6	7.3	7.9 ± 0.2

TABLE VII

THE ENTHALPIES OF SOLUTION OF TUNGSTEN
AND TANTALUM IN LIQUID RARE-EARTH METALS

Solvent Element	$\Delta \bar{H}^*$, kcal/mole	
	W	Ta
Yb	51	38
Eu	47	37
La	42	36
Sm	38	35
Pr	37	34
Nd	36	34
Ce	35	32
Gd	33	28
Y	31	25
Tb	30	26
Dy	29	25
Er	28	22
Ho	25	23
Tm	25	22
Lu	24	21
Sc	18	17

TABLE VIII

THE CALCULATED SOLUBILITY PARAMETERS
OF THE LIQUID RARE-EARTH METALS

Solvent Element	Calculated Solubility Parameter, δ_g		
	With W	With Ta	Average
Yb	79	83	81
Eu	82	84	83
La	86	85	85
Sm	89	86	87
Pr	90	87	88
Nd	91	87	89
Ce	92	88	90
Gd	94	92	93
Y	96	96	96
Tb	97	95	96
Dy	99	96	97
Er	100	99	99
Ho	103	98	100
Tm	103	99	101
Lu	105	101	103
Sc	113	106	109

2. Tantalum and Tungsten in Liquid Rare-Earth Metals

The partial molar enthalpies of solution of tantalum and tungsten in liquid rare-earth metals were calculated from the solubility data.^{11, 12, 15} The regular solution theory approach was employed, and it was assumed that these dilute solutions formed simple eutectic-type binaries in the temperature range of interest, 1325 to 2150°C. The entropy of fusion was assumed to be 2.3 e. u. An error of 1.0 e. u. would be equivalent to a change of 2.0 kcal/mole in the enthalpy term at 2000°C and approximately 1.5 kcal/mole at 1500°C. This represents a change of 0.25 kcal/mole over this temperature range, and such a change would not be detected in this approach. Therefore, the first approximation of the entropy term appears justified. The average calculated enthalpies are listed in Table VII in decreasing order. The solubility order, which increases from ytterbium to scandium, is very similar for the two solutes. In each solute pair, moreover, tungsten is less soluble than tantalum. Values of the solubility parameters of these solutes were calculated from the individual enthalpies of solution. These parameters are given in Table VIII. The agreement is within a few percent, which is probably as accurate as can be expected for this type of treatment.

3. Selected Elements in Liquid Sodium

Claar¹³ has reviewed the available solubility data for a large number of elements in liquid sodium and has evaluated the measurements on the basis of experimental technique and internal consistency. On this basis, 12 elements were chosen that appeared to fit a solubility-temperature relation of the form

$$\log N_i = A - B/T, \quad (12)$$

where A and B are constants. These equations were constructed by least-squares analyses of the data.

Ten of these 12 elements form simple eutectic-type binaries with liquid sodium. The other two, bismuth and tin, both form compounds of the type Na₃A.^{16, 17} A break in the solubility against the (1/T) curve occurs in the bismuth-sodium system at 290°C. As in the titanium-plutonium system, there is a definite change in the entropy and enthalpy values at this point. No such break occurs in the temperature range reported for the tin-sodium system, 120 to 300°C. According to the phase diagram,¹⁴ the equilibrium in this temperature range is similar to that for plutonium-carbon and other compound systems.

Equation (12) was used to calculate empirical values for the enthalpies and entropies of solution. In addition, the solubilities were calculated from the equation at the lowest, midpoint, and maximum temperatures

listed for the solubility study. Values for the enthalpy of solution by the regular solution model were then calculated by Eq. (3), assuming that the entropies of fusion are 2.3 e. u. The resulting average values are given in Table IX. There is, once again, a very large variation in the entropy values by the least-squares approach. In the regular solution approach, the solubility increases with decreasing enthalpy of solution in the order given in the table. This order, with the exceptions of bismuth, tin, and silver, reflects the melting point order of these elements, that is, the element with the highest melting point is the least soluble in sodium.

Solubility parameters were calculated from the enthalpies derived by the regular solution approach. These are given and compared with the theoretical values in Table X. The average ratio of calculated to theoretical, 0.84, is the same as for elements in liquid plutonium; however, variations are large. The ratio changes may be due to volume changes or other effects not considered in simple parameter approximations.

III. CONCLUSIONS

The regular solution model appears to fit a variety of dilute systems. These include not only the rather simple eutectic-type binaries, but more complex sys-

Element	δ_i		k, ($\frac{\text{Calc}}{\text{Theory}}$)
	Calc	Theory	
Mo	91	129	0.71
Ni	93	125	0.74
Fe	90	119	0.76
Cu	75	107	0.70
Sb	56	59	0.95
Zn	63	58	1.09
Sn	50	63	0.79
Ag	52	82	0.63
Pb	50	51	0.98
Cd	48	45	1.07
In	48	61	0.79
Av			0.84 $\pm$ 0.15

tems that contain compounds and solid solutions.

However, enthalpy and entropy values calculated from solubility data are strictly applicable only within the temperature and concentration range in which data are available. Changes can occur with changes in phase or activity. Although the approximate enthalpies can be used for qualitative estimates of solubility, it still remains necessary to measure solubilities to obtain quantitative data.

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TABLE IX
ENTROPIES AND ENTHALPIES OF SOLUTION
IN LIQUID SODIUM

Element	Empirical		Regular $\Delta \bar{H}^*$
	$\Delta \bar{S}$	$\Delta \bar{H}$	
Mo	-15.3	18.1	38.8 $\pm$ 1.8
Ni	-19.8	7.2	27.6 $\pm$ 4.1
Fe	- 5.6	19.7	26.8 $\pm$ 1.0
Bi ^a	-22.0	9.7	21.2 $\pm$ 1.5
Bi ^b	- 5.9	18.4	24.7 $\pm$ 1.1
Cu	- 0.04	14.0	15.8 $\pm$ 0.4
Sb	- 6.6	8.4	14.2 $\pm$ 1.3
Zn	4.5	11.7	9.9 $\pm$ 0.3
Sn	12.4	10.6	6.7 $\pm$ 0.8
Ag	2.5	6.8	6.6 $\pm$ 0.1
Pb	14.2	11.3	6.2 $\pm$ 0.6
Cd	4.5	5.5	4.5 $\pm$ 0.2
In	8.2	7.1	4.4 $\pm$ 0.2

^a 125 to 290°C.

^b 290 to 650°C.

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