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Thermodynamic systematics of the formation of liquid alloys of f-elements with bismuth

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Abstract

In order to strengthen the comprehensive understanding of the mechanism of extraction of f-elements in a pyrometallurgical liquid–liquid extraction system, excess thermodynamic quantities of lanthanide and actinide in liquid bismuth are discussed. Excess enthalpy changes of some lanthanides and actinides in liquid bismuth were experimentally determined or estimated, and their systematic variation along the lanthanide and actinide series is examined by using a semi-empirical calculation method. Characteristic features of the thermodynamic stabilities of lanthanides and actinides in liquid bismuth are discussed.

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1. Introduction

The reductive liquid–liquid extraction of f-elements between molten chloride and liquid bismuth deserves an examination as a possible technique for the group separation of actinides and lanthanides [1,2]. In a pyrometallurgical liquid–liquid extraction system, the separation performance of elements mainly depends on the standard Gibbs energy of formation of their chlorides. However, the activity coefficients of the elements in the metallic and molten salt phases also influence the separation efficiency to a great extent. Therefore, in order to evaluate the effectiveness of the separation of lanthanides and actinides by this technique, their thermodynamic activities in the two phases are of great technological interest. From a chemical viewpoint, the thermodynamic properties of the metallic states of f-elements in a liquid metal phase are quite an interesting subject because we can expect an appearance of the characteristic behavior of f-orbitals in their intermetallic bonding with the solvent metals.

In this context, this paper discusses the systematic variation of the thermodynamic stabilities of lanthanide and actinide metals in liquid bismuth. In order to systematize their trend along the 4f and 5f-series, our experimental results about the thermodynamic activity of lanthanides and actinides in liquid bismuth [3] are discussed in conjunction with the reported data [4–8].

2. Experimental

Two different experiments, liquid–liquid extraction and electromotive force measurement were performed. For both experiments, 99.9% pure eutectic mixtures of LiCl and KCl, mole ratio of lithium to potassium = 51/49 purchased from Anderson Physics Laboratory Engineered Materials Inc. was used as molten salt phase. Hereafter the notation LiCl–KCl will be used to represent this eutectic mixture. All the extraction experiments were performed in a vacuum-tight glove box filled with purified Ar of which the humidity and oxygen levels were continuously kept below 1 ppm. In this paper, M represents trivalent actinides or lanthanides. Salts and metal phases are denoted by S and B, respectively, but they actually mean LiCl–KCl eutectic mixture and liquid bismuth, respectively. Bracket (in S or B) attached to M

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or chlorides (MCl_3 or LiCl) specifies that it is a dissolved species in the phases.

2.1. Electromotive force determination of lanthanides

To obtain the excess Gibbs energy of lanthanides in liquid bismuth that is not available in the literature, the electromotive force (EMF) measurement method was applied to the galvanic cell below. La and Gd were tested to compare with the literature values, and Tb and Dy were investigated as the first measurements.

M (solid) | MCl_3 in LiCl-KCl | M (in B)

M : La, Gd, Tb and Dy B : Bi.

About 36 g of LiCl–KCl and 133 g of pure Bi were put in a crucible with about 1 g of one of the lanthanide metals; then the temperature was raised to selected values in an electric furnace. After the desired temperature was achieved, the bottom tip of the pure lanthanide electrode was immersed into the molten salt phase, and then the difference of the electric potentials between the pure lanthanide electrode and liquid alloy electrode was measured by an electrometer. Measurements were performed in a range from 720 to 1100 K. At each temperature, the concentration of lanthanide in Bi phase was changed several times, and the lanthanide mole fraction in bismuth was analyzed by ICP-AES (Shimazu ICPS-1000III). 99.9% pure metallic rods of La, Gd, Tb and Dy were purchased from Rare Metallic, and were used after polishing the surface. 99.999% pure metallic Bi and all other reagents of analytical grade used for his study were purchased from Wako Pure Chemicals Industries.

2.2. Liquid–liquid extraction experiments of actinides

The experimental procedures to determine the distribution ratios of actinides in a reductive liquid–liquid extraction system have been reported elsewhere [1,2]. Distribution ratios of actinides (^{239}Np, ^{241}Am, and ^{242}Cm) were radiochemically determined at 873–1073 K in a two-phase system of LiCl–KCl and liquid Bi. By changing the concentration of metallic Li as reductant, the distribution ratios of actinides (denoted D_M hereafter) were determined as functions of those of Li (denoted D_{Li} hereafter).

3. Results and discussion

3.1. Excess thermodynamic quantities of lanthanides in liquid Bi determined by the electromotive force measurements

The electromotive force between M(solid) and M(in B) can be given by Eq. (1), where $\mu^{ex}_{M(in\ B)}$ is the excess chemical potential of M in B and $\Delta\mu^{fusion}_M$ is the change of chemical potential over the fusion of M which equals the standard Gibbs energy change of fusion of M (ΔG^{fusion}_M).

$$\Delta E = \frac{1}{nF}\Delta\mu^{fusion}_M - \frac{RT}{nF}\ln x_{M\ (in\ B)} - \frac{1}{nF}\mu^{ex}_{M\ (in\ B)}. \quad (1)$$

$\mu^{ex}_{M\ (in\ B)}$ actually equals the excess Gibbs energy change of M in B (ΔG^{ex}[M in B]). Eq. (1) yields Eqs. (2) and (3) where $f_{M\ (in\ B)}$ is the activity coefficient of M in B. ΔE was measured in the range of $x_{M\ (in\ B)}$ from 10^{-5} to 10^{-2}. For every case of measurement, observed ΔE approximately showed a linear dependence on $\ln x_{M\ (in\ B)}$ with slopes which were not exactly equal to the theoretical slopes of Eq. (1) corresponding to $n = 3$ but were close to it. This indicates that the solutes are in trivalent states and that the last term of Eq. (1) may slightly vary with the concentration. By applying the observed ΔE and $\ln x_{M\ (in\ B)}$ to Eq. (2) with $n = 3$, $\log f_{M\ (in\ B)}$ and ΔG^{ex}[M in B] for La, Gd, Tb and Dy were calculated for each temperature. Since a slight concentration dependence was observed, those at $x_{M\ (in\ B)} = 5 \times 10^{-3}$ were selected as representative values. They are listed in Table 1 together with those calculated from the reported activity coefficient data [4].

$$\Delta G^{ex}[\text{M in B}] = -nF\Delta E - \Delta G^{fusion}_M - RT\ln x_{M\ (in\ B)}, \quad (2)$$

$$\Delta G^{ex}[\text{M in B}] = RT\ln f_{M\ (in\ B)}. \quad (3)$$

From the observed temperature dependence of log $f_{M\ (in\ B)}$, the corresponding excess enthalpy change ΔH^{ex}[M in B] was determined and is summarized in Table 2. In Table 2, the values calculated from the temperature dependence reported by Lebedev [4] are listed for comparison. The ΔH^{ex}[M in B] data summarized in the literature [4] were determined by the same method as this study [4–8].

3.2. Excess thermodynamic quantities of actinides estimated from the distribution results of liquid–liquid extraction

The excess thermodynamic quantities of Np, Am, and Cm in liquid Bi have not been precisely reported in the literature, which is considered to be due to experimental difficulties. Thus, in this paper, the excess thermodynamic quantities of Np, Am, and Cm were estimated from the distribution results of the liquid–liquid extraction experiments. The reductive liquid–liquid extraction of trivalent actinide is given by

$$MCl_3(\text{in S}) + 3Li(\text{in B}) \leftrightarrow M(\text{in B}) + 3LiCl(\text{in S}). \quad (4)$$

The standard Gibbs energy changes of formation of four species of reaction (4) are denoted by $\Delta G^{\circ}_f[MCl_3$ in S],

Table 1
Activity coefficients and excess Gibbs energy of lanthanides in liquid Bi

	Temperature (K)	$\log f_{(M\ in\ B)}$		ΔG_{ex}[M in B]	
		Lebedev et al.	This study at $x_{M\ (in\ B)} = 5 \times 10^{-3}$	Lebedev et al.	This study at $x_{M\ (in\ B)} = 5 \times 10^{-3}$
La	766.5	−13.51	−13.63 ± 0.11	−198.3	−199.9 ± 1.7
	865.7	−11.88	−11.92 ± 0.04	−196.8	−197.5 ± 0.6
	963.4	−10.59	−10.53 ± 0.05	−195.4	−194.3 ± 1.0
Gd	778.2	−11.43	−11.48 ± 0.05	−170.4	−171.0 ± 0.7
	873.1	−10.06	−10.17 ± 0.01	−168.1	−170.0 ± 0.2
	963.9	−9.00	−9.04 ± 0.03	−166.0	−166.8 ± 0.5
	1053.3	−8.13	−8.10 ± 0.05	−163.9	−163.3 ± 1.0
Tb	801.3	–	−10.70 ± 0.21	–	−164.2 ± 3.2
	899.9	–	−9.59 ± 0.16	–	−165.1 ± 2.8
	998.5	–	−8.45 ± 0.25	–	−161.6 ± 4.8
	1095.0	–	−7.52 ± 0.10	–	−157.6 ± 2.2
Dy	813.8	–	−10.66 ± 0.04	–	−166.1 ± 0.6
	915.3	–	−9.62 ± 0.08	–	−168.5 ± 1.4
	993.8	–	−8.75 ± 0.06	–	−166.4 ± 1.2
	1088.8	–	−7.85 ± 0.04	–	−163.7 ± 0.8

Table 2
ΔH^{ex}[M in B] determined in this study and reported in the literature

	$V^{2/3}$ ($cm^2/mol^{2/3}$)	ΔH^{ex}[M in B] (kJ/mol)	
		Lebedev et al.[a]	This study
La	7.98	−219.15	−221.54 ± 2.31
Ce	7.76	−225.77	
Pr	7.56	−237.45	
Nd	7.51	−220.07	
Pm	7.43		
Sm	7.37		
Eu	7.36		
Gd	7.34	−200.89	−202.25 ± 1.80
Tb	7.20		−199.83 ± 0.55
Dy	7.12		−193.80 ± 0.99
Ho	7.06		
Er	6.98	−182.46	
Tm	6.90		
Yb	6.86		
Lu	6.81		
U	5.36	−90.40	
Np	5.57		−101.75 ± 25.00
Pu	5.94	−167.24	
Am	6.83		−196.36 ± 25.00
Cm	6.94		−199.85 ± 25.00

[a] Derived from the temperature dependence data of literature [4].

ΔG_f°[Li in B], ΔG_f°[M in B], and ΔG_f°[LiCl in S], which are given by the sum of the standard Gibbs energy changes of formation of their pure liquids ($\Delta G_f^\circ[X, liq]$, and their excess Gibbs energy changes (ΔG^{ex}[M in B], or ($\Delta G^{ex}[MCl_x$ in S] at various concentrations. From the relation between the equilibrium constant K and the Gibbs energy change of reaction (4), Eq. (5) is obtained as a function of the distribution ratios D_M and D_{Li} which are defined by $x_{M\ (in\ B)}/x_{MCl_3(in\ S)}$ and $x_{LiCl(in\ B)}$, respectively.

Table 3
Thermodynamic quantities of actinides adapted

		$\log(D_M/D_{Li}^3)$	$\Delta G^{ex}[MCl_3$ in S] (kJ/mol)	$\Delta G_f^\circ[MCl_3, liq]$ (kJ/mol)
873 K	Np	8.282 ± 0.200	−37.9	−697.4
	Am	9.485 ± 0.202	−46.8	−777.4
	Cm	9.113 ± 0.150	−50.9	−777.4
1073 K	Np	6.468 ± 0.200	−31.9	−664.8
	Am	8.056 ± 0.139	−40.9	−735.6
	Cm	7.690 ± 0.109	−44.9	−735.6

$$\begin{aligned}\log(D_M/D_{Li}^3) = &-\frac{1}{2.3RT}\left\{\Delta G_M^{fusion} + \Delta G^{ex}[M \text{ in } B]\right\} \\ &+\frac{1}{2.3RT}\left\{\Delta G_f^\circ[MCl_3, liq] \right. \\ &\left. + \Delta G^{ex}[MCl_3 \text{ in } S]\right\} \\ &+\frac{3}{2.3RT}\left\{\Delta G_f^\circ[Li, liq] + \Delta G^{ex}[Li \text{ in } B]\right\} \\ &-\frac{3}{2.3RT}\left\{\Delta G_f^\circ[LiCl, liq] + \Delta G^{ex}[LiCl \text{ in } S]\right\}. \end{aligned} \quad (5)$$

About 10–20 pairs of $\log D_M$ and $\log D_{Li}$ for Np, Am, and Cm were measured at 873 and 1073 K, and representative $(\log D_M/D_{Li}^3)$ values were obtained as the intercept of the third-powered linear dependence of $\log D_M$ on $\log D_{Li}$ [1,2]. Because terms other than $\Delta G^{ex}[MCl_3$ in S] and $\Delta G^{ex}[M$ in B] are available from the literature, by knowing the value of $\Delta G^{ex}[MCl_3$ in S], $\Delta G^{ex}[M$ in B] can be calculated by using experimentally obtained $\log(D_M/D_{Li}^3)$. Self-consistent values of $\Delta G^{ex}[MCl_3$ in S] of Np^{3+}, Am^{3+}, and Cm^{3+} for different temperatures are not available, and thus in this paper, estimated values of $\Delta G^{ex}[MCl_3$ in S] were used. Since $\Delta G^{ex}[MCl_3$ in S] is the result of an electrostatic interaction with the component ions of the salt, it can be assumed that $\Delta G^{ex}[MCl_3$ in S] has a linear dependence on the reciprocal of the ionic radius of M^{3+}, and this has been observed for lanthanides [3] in our previous study. For the trivalent actinides, this linear relation obtained for trivalent lanthanide was applied by standardizing it to the point of $\Delta G^{ex}[UCl_3$ in S] which was obtained from the reported formal potentials of U(0)–U(III) in eutectic mixture of LiCl and KCl. For this purpose, temperature-dependent formal potential data of U(0)–U(III) reported in the literature [9] was used since ±1.5% of this reported value covers the scattered data by the other reports. Consequently, the following equations were derived for acinides:

$$\Delta G^{ex}[MCl_3 \text{ in } S]_{873\ K} = -251.3 \cdot 1/r + 210.9 \text{ kJ/mol}, \quad (6)$$

$$\Delta G^{ex}[MCl_3 \text{ in } S]_{1073\ K} = -251.3 \cdot 1/r + 216.8 \text{ kJ/mol}, \quad (7)$$

where r is the ionic radius of the ions for coordination number 6 [10]. By these procedures, $\Delta G^{ex}[M$ in B] was estimated. Some major thermodynamic quantities used are listed in Table 3. From the difference of $\Delta G^{ex}[M$ in B] at two different temperatures, $\Delta H^{ex}[M$ in B] values were estimated and listed in Table 2. Due to

Table 4
Parameters for Miedema's semi-empirical model[a,b]

	$V^{2/3}$ ($cm^2/mol^{2/3}$)	Φ (V)	$n_b^{1/3}$ ($du^{1/3}$)[c]	R/P[d]
La	7.98	3.17	1.18	0.99
Ce	7.76	3.18	1.19	0.99
Pr	7.56	3.19	1.20	0.99
Nd	7.51	3.19	1.20	0.99
Pm	7.43	3.19	1.21	0.99
Sm	7.37	3.20	1.21	0.99
Eu	7.36	3.20	1.21	0.99
Gd	7.34	3.20	1.21	0.99
Tb	7.20	3.21	1.22	0.99
Dy	7.12	3.21	1.22	0.99
Ho	7.06	3.22	1.22	0.99
Er	6.98	3.22	1.23	0.99
Tm	6.90	3.22	1.23	0.99
Yb	6.86	3.22	1.23	0.99
Lu	6.81	3.22	1.24	0.99
U	5.36[e]	3.90	1.44[f]	1.90
Np	5.57[g]	3.80[h]	1.33[f]	1.90
Pu	5.94[i]	3.80	1.27[f]	1.90
Am	6.83[e]	3.80[h]	1.23[f]	1.90
Cm	6.94[e]	3.80[h]	1.23[f]	1.90
Bi	7.20	4.15	1.16	

[a] Number without notes: from References [11,12].
[b] $P = 12.3$ $Q/P = 0.944$ were commonly used.
[c] $du = 6 \times 10^{22}$ electrons/cm^3.
[d] Given by this study.
[e] α-phase.
[f] Given by this study.
[g] γ-phase.
[h] Assumed to be equal to Pu.
[i] ε-phase.

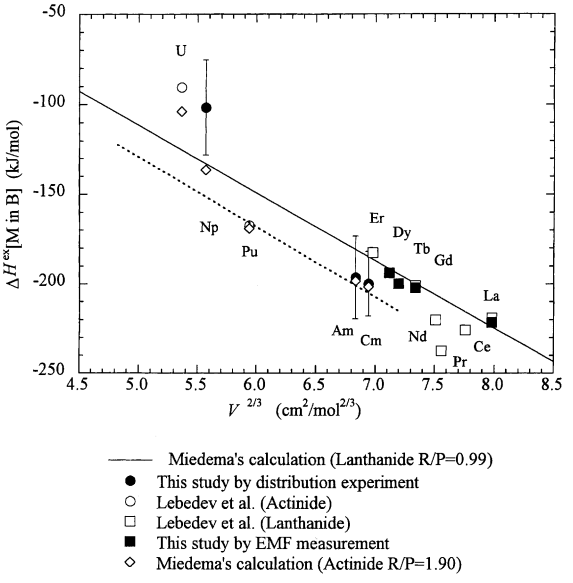

Fig. 1. Dependence of the excess enthalpy change of lanthanides and actinides in liquid bismuth on the 2/3 power of molar volume.

the uncertainty of Eqs. (6) and (7) and other experimental errors of distribution experiment, the values of the derived ΔH^{ex}[M in B] have large errors.

3.3. Systematic analysis of ΔH^{ex} [M in Bi] of lanthanides and actinides

In the liquid alloy, it is considered that the solute metal forms a chemical complex with the solvent metals, i.e. a cluster, and this is considered to be responsible for the thermodynamic stabilization of the solute metals in the solvent [11]. From Miedema's semi-empirical model [12,13], the enthalpy change of solution of metal A in metal B which is denoted as ΔH_{sol}[A in B] is given by

$$\Delta H_{sol}[\mathrm{A\ in\ B}] = V^{2/3}\frac{2P}{n_b(\mathrm{A})^{-1/3}+n_b(\mathrm{B})^{-1/3}} \times\left[-(\Delta\Phi)^2+\frac{Q}{P}\left(\Delta n_b^{1/3}\right)^2-\frac{R}{P}\right], \quad (8)$$

where V is the molar volume of metal A, n_b(A) and n_b(B) are the electron densities at the boundary of Wigner–Seitz cell, $\Delta\Phi$ is the difference of electronegativity between A and B, and P, Q, R are specific constants. R is an additional negative term necessary to account for the experimental results of transition-metals and polyvalent non-transition metals. All the parameters applied are listed in Table 4.

The values of ΔH^{ex}[M in B] obtained by the methods described above and reported values in the literature are plotted in Fig. 1 vs. $V^{2/3}$. The solid line drawn in Fig. 1 represents those of lanthanides calculated by Eq. (8) with $R/P = 0.99$. La, Gd, Tb, Dy, and Er, obviously fit the line, and thus $R/P = 0.99$ correctly represents these lanthanides. On the other hand, those of Ce, Pr, and Nd show more negative values than this line, and this indicates that these elements may be more stable than the group of La, Gd, Tb, Dy, and Er. In Fig. 1, values of ΔH^{ex}[M in B] for the actinides show different systematic features than those for the lanthanides.

In order to evaluate the characteristic features of actinides, Miedema's calculation was performed for U, Np, Pu, Am, and Cm with the parameters listed in Table 4. The electronegativity Φ of Np, Am, and Cm was assumed to be equal to that for Pu which is given in the literature [12]. The electron densities at the boundary of Wigner–Seits cell for actinides, $n_b^{1/3}$, were calculated by Eq. (9) by using their molar volumes V and bulk moduli B [14–18],

$$n_b^2 = C\cdot B/V, \quad (9)$$

where C is a minor correction factor which was determined from the values of lanthanides [13]. For the molar volumes of actinides, their values for metallic phases in the temperature range 600–800 K were adapted [18]. Note that ε-phase for Pu and γ-phase for Np were applied, and this is different from Miedema's treatment which may have assumed α-phase for Pu. Table 5 summarizes the parameters used for estimating $n_b^{1/3}$. For Pu, Am, and Cm, it was found that calculations with

Table 5
Related parameters for $n_b^{1/3}$ of actinides

Element	Phase	Molar volume (V) (cm^3/mol)	$V^{2/3}$ (cm^2/mol$^{2/3}$)	Bulk modulus (Gpa)	References	$n_b^{1/3}$ ($du^{1/3}$)
U	α	12.4	5.36	113	[13,14]	1.44
Np	γ	13.1	5.57	73.5	[13,15]	1.33
Pu	ε	14.5	5.94	47	[13,16]	1.27
Am	α	17.9	6.83	36.1	[17][a]	1.23
Cm	α	18.3	6.94	36.1	[b]	1.23

[a] Derived from compressibility data.
[b] Assumed to be equal to Am.

$R/P = 1.90$, with the use of newly given V and $n_b^{1/3}$, agreed well with the experimentally estimated data which are shown as open diamonds and dotted line in Fig. 1. This is similar to the case of lanthanides which showed linearity for common R/P. The presence of a common R/P for a group of lanthanides or actinides indicates that the constituents of the group show less individuality. The larger R/P (1.90) of the actinide group (Pu, Am, and Cm) than the lanthanide group (0.99), which corresponds to a deeper stabilization of the actinides by 20 kJ/mol, suggests that the actinides metals form more stable bonding with bismuth than the lanthanides.

In contrast, there are elements which show different systematics from the above elements. In the case of lanthanides, Ce, Pr, and Nd showed uniquely deeper stabilization than the others, which suggests that these elements form clusters with different features. Note that these three elements have intermetallic compounds of MBi_2 type while the other elements only have MBi type. It is reasonable to consider that this structural uniqueness of the intermetallic compounds is reflected in the liquid phase. The uniqueness of U and Np suggest totally different characteristics. U and Np which are dense metals are understood to have more de-localized 5f-electrons [13] and behave like tetravalent metals rather than trivalent metals. This could explain the different systematic features of U and Np compared to Pu, Am, and Cm.

4. Conclusions

Excess enthalpy changes of lanthanide and actinide metals in liquid bismuth were determined and estimated from the results of electromotive force measurements and liquid–liquid extraction experiments. Systematic trends for the 4f- and 5f-series were discussed. It is concluded that lanthanides other than Ce, Pr, and Nd behave as a group which is characterized by a constant value of R/P for Miedema's semi-empirical calculation. Pu, Am, and Cm also have a group feature that is characterized by a larger R/P than that for the lanthanides. The uniqueness of U and Np in the systematics of the formation of liquid alloy with bismuth was pointed out.

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