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Estimation of the Activity of Lead in the Binary Pb–Sb and Pb–Bi Systems

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Abstract—The activity of lead in its alloys with bismuth and antimony is estimated by the least squares method and interval analysis. The interval method of processing the experimental results is shown to calculate the reliable ranges of the estimated parameters in the dependences of thermodynamic functions and the actual level of the total measurement errors.

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INTRODUCTION

Based on the thermodynamic data on the binary Pb–Sb and Pb–Bi systems with various antimony or bismuth contents, we estimate the activity of lead in these systems. The estimation is performed in the presence of errors in measuring the emf with an electrolytic cell and measuring the melt temperature. Small chaotic measurement errors can exist because of experimental errors. The samples of measurement results have a rather small volume, the measurement errors are limited in absolute value, and their probabilistic characteristics are known inaccurately. To obtain the most complete information on the temperature dependence of the activity of lead at various second-component contents, the estimation is performed by the standard least squares method (LSM) and interval analysis.

EXPERIMENTAL CONDITIONS AND FORMULATION OF THE PROBLEM

To perform electrorefining of lead–antimony and lead–bismuth alloys in ionic media, we need the knowledge of the electrochemical behavior of lead in these systems under electrorefining conditions. The Pb–Sb phase diagram has one eutectic (T_m = 247°C, 13 wt % Sb) and no chemical compounds. The Pb–Bi phase diagram has one chemical compound, which forms at 184°C and decomposes at –46°C. The eutectic in this diagram solidifies at a temperature of 125°C and a concentration of 56 mol % Bi between this compound and Bi [1].

In this work, we determine the activity of lead in Pb–Bi alloys with 20–80 mol % Pb by measuring the emf of a galvanic cell in the temperature range 720–860 K,

$$(-)(\mathrm{GC})\mathrm{Pb}|\mathrm{KCl{-}PbCl_2}|\mathrm{KCl{-}PbCl_2}|\mathrm{Pb{-}Bi(GC)}(+), \quad (1)$$

where GC is glass carbon.

For comparison, we estimated the activity of lead in Pb–Sb alloys from the experimental data obtained in [2] for the galvanic cell

$$(-)(\mathrm{GC})\mathrm{Pb}|\mathrm{KCl{-}PbCl_2}|\mathrm{KCl{-}PbCl_2}|\mathrm{Pb{-}Sb(GC)}(+). \quad (2)$$

The electrolytic cell consisted of a hermetically sealed quartz glass with an alundum crucible in which molten lead and a KCl–$\mathrm{PbCl_2}$ (50 : 50 mol %) electrolyte (i.e., negative half-cell) were placed. The positive half-cell consisted of a KCl–$\mathrm{PbCl_2}$ (50 : 50 mol %) electrolyte and a Vi0 bismuth–S1 lead alloy. The half-cell electrolytes were separated by a porous diaphragm. The current leads to the metallic electrodes were made of glass carbon rods shielded by alundum straws from the melt.

To prepare electrolytes, we used analytical grade reagents, which were additionally cleaned and dehydrated. Argon supplied to the electrolytic cell was preliminarily dehydrated when passed through concentrated sulfuric acid and calcium chloride.

The temperature was measured using a calibrated chromel–alumel thermocouple. The maximum (in absolute value) error in measuring temperature was ±3 K. The emf was measured with a multimeter with an input impedance of about 10 M Ω. The reliable values of equilibrium potential were taken to be the values that were constant within 60 min. The nominal (in absolute value) error of measuring emf was 0.0001 V.

A small chaotic component of the measurement error was detected in experiments. Therefore, when processing each sample of measurements (for each fixed bismuth content), we additionally estimated modulus $e_{\max}$ of the total error using the special-purpose algorithms from [5, 6] and then used it for an interval estimation of the activity of lead in this sample.

The alloy composition was controlled by atomic absorption spectroscopy before and after an experiment, and no changes in the alloy composition was detected within the limits of experimental error.

The emf polytherms of concentration chains have a linear shape, and the structure of emf measurement is generally described as

$$E = A + (R/Fz)\ln(a)T + e, \{T_n, E_n, n = 1, N\}, \quad (3)$$

$$|e| \le e_{\max}, |t| \le t_{\max}, z = 2,$$

where E is the cell emf (V) measured at a total error $|e| \le e_{\max}$, $R = 8.314$ is the gas constant (J/(mol K)), $F = 96495$ is a constant (C/mol), z is the fixed lead valence in the melt, T is the melt temperature known at an error $|t| \le t_{\max}$, a is the activity of lead in the melt to be estimated, A is a constant component, $\{T_n, E_n, n = 1, N\}$ is the sample of noisy emf measurements, n is the measurement number, and N is the sample volume.

To process such experimental data, researchers usually apply standard statistical methods that are similar to [3, 4] and based on the laws of measurement error distribution. In our case, this information is known inaccurately, the measurement results undergo possible small chaotic perturbations due to the experimental errors, and measurement samples are short because of their complexity. Therefore, it is reasonable to use interval analysis methods along with standard LSM in order to estimate the activity of lead and the possible activity range [5, 6]. To this end, we developed a technique that modifies the interval algorithms of information processing to solve practical estimation problems [7].

Formulation of the Problem

Under uncertainty of the measurement error characteristics, we have to estimate the range of allowable activities of lead in a binary system using standard and interval (for comparison) approaches.

INTERVAL ESTIMATION PROCEDURES

The uncertainty of emf measurement is formalized using the emf measurement uncertainty range (MUR)

$$[E]_n = [{}_{-}E_n, {}^{+}E_n] : {}_{-}E_n = E_n - e_{\max}, {}^{+}E_n = E_n + e_{\max}, \quad (4)$$

where ${}_{-}E_n$ and ${}^{+}E_n$ are the lower and upper MUR boundaries of emf measurement, respectively, and $e_{\max}$ is the limitation imposed on the maximum total measurement error.

Similarly, the uncertainty of temperature measurement is formalized with MUR of temperature

$$[T]_n = [{}_{-}T_n, {}^{+}T_n] : {}_{-}T_n = T_n - t_{\max}, {}^{+}T = T_n + t_{\max}, \quad (5)$$

where ${}_{-}T_n$ and ${}^{+}T_n$ are the lower and upper MUR boundaries of temperature measurement, respectively, and $t_{\max}$ is the limitation imposed on the maximum temperature measurement error.

In the dependence plane, uncertainties (4) and (5) form a number of two-dimensional sets (uncertainties)

$$[E]_n \times [T]_n, n = 1, N. \quad (6)$$

Due to measurement uncertainties (4) and (5), Eq. (3) in an interval form can be rewritten as

$$[E]_n = [A] + [B][T]_n, \{n = 1, N\}, \quad (7)$$

$$[B] = (R/Fz)[\ln(a)], \quad (8)$$

and Eqs. (6) give a system of interval equations in unknown coefficients A and B.

A pair of coefficients (A, B) is allowable if corresponding straight line (3) passes through two-dimensional sets of uncertainties $[E]_n \times [T]_n$ for all $n = 1, N$. Set $I(A, B)$ of all allowable pairs of coefficients A and B is called information set [5–7].

For the case of linear Eqs. (3) and (7), the information set is constructed exactly. The process of its construction is as follows [5, 6]. For each pair $[E]_i \times [T]_i$ and $[E]_j \times [T]_j$ of sets of uncertainties with numbers $i = 1, N - 1$ and $j = i + 1, N$, we construct partial information set $G_{ij}(A, B)$ of coefficients A and B allowable (in the given sense) for this pair of the sets of uncertainties,

$$\begin{aligned} G_{ij}(A, B) = \{A, B : E_i = A + BT_i \in [E_i] \times [T_i] \\ \text{and } E_j = A + BT_j \in [E_j] \times [T_j]\}. \end{aligned} \quad (9)$$

Partial information set (9) has the shape of a convex polygon in the plane of coefficients A and B.

Table 1. Estimated actual level of the total error in measuring emf $e^*_{\max}$ (mV) for the equilibrium potentials of liquid Pb–Sb alloys in the KCl–$PbCl_2$ melt

N_{Pb}, mol %	95	90	70	47	40	25
$e_{\max}$, mV	0.18	0.25	0.40	0.83	0.42	0.93

The desired information set $I(A, B)$ is found as the intersection of all partial information sets of a given measurement sample

$$I(A, B) = \bigcap_{ij} G_{ij}(A, B) \text{ at } i = 1, N-1, j = I+1, N. \quad (10)$$

Information set (10) has the shape of a convex polygon with linear boundaries in the plane of estimated coefficients A and B.

We developed algorithms to provide the stability and convergence of these procedures of calculating information sets $I(A, B)$ and activity ranges. For example, if intersection (10) is empty, this means the incompatibility of sets of uncertainties (6), i.e., the presence of distorted measurements (spikes). In this case, we used the special-purpose algorithms from [5, 6] to analyze an incompatible sample and to reveal and remove distorted measurements.

RESULTS OF PROCESSING THE EXPERIMENTAL DATA

We processed a series of samples of noisy experimental data obtained at various temperatures and second-component contents in the binary Pb–Sb and Pb–Bi systems.

At the first step of processing a measurement sample (at a fixed lead content), the modulus of the total emf measurement error $e_{\max}$ was estimated using the special-purpose interval algorithms from [5, 6]. Recall that this total error is caused by both the measurement error and small chaotic perturbations during an experiment.

Table 1 gives estimated total errors $e^*_{\max}$ of emf measurements for the equilibrium potentials of liquid Pb–Sb alloys in the KCl—$PbCl_2$ melt. Similar estimated total errors $e^*_{\max}$ of emf measurements were found for the equilibrium potentials of liquid Pb–Bi alloys in the KCl–$PbCl_2$ melt (Table 2). An analysis of the data in Tables 1 and 2 demonstrates the presence of very small chaotic perturbations in the measurements; that is, the experiments were carefully performed. These estimates taken with a safety margin were then used to execute interval processing of the experimental data: for example, we used $e_{\max} = 2$ mV to calculate information sets (10), and the maximum error in measuring temperature was taken to be $t_{\max} = 3°C$.

Algorithms and the corresponding programs were tested on a large volume of experimental data. Figures 1 and 2 show the temperature dependences of emf at various lead contents in the binary Pb–Sb and Pb–Bi systems, respectively. To illustrate the high quality of the experimental data, the uncertainty intervals (heavy vertical bars) of emf measurements for each sample correspond to the estimated total error $e^*_{\max}$ from Tables 1 and 2.

It is seen from Figs. 1 and 2 that the polytherms are well described by straight lines, dependence $E(T) = f(T)$ can be considered as linear in advance (dotted lines), and the measurement uncertainty intervals are well arranged with respect to the straight lines.

Set $I(A, B)$ of the allowable coefficients was estimated for describing function (3) and interval equation (7). Note that the average dependences (dotted lines) calculated by the interval estimation and LSM are close to each other due to the high quality of the experiments (Table 3).

Tables 3 and 4 give emf equations for various lead contents in the Pb–Sb and Pb–Bi systems in the KCl—$PbCl_2$ melt and equations $E(T) = A^* + B^*T$ for the average values of coefficients A^* and B^*, which are the middle points of the corresponding $I(A, B)$ information sets. These tables also present the unconditional intervals (external estimation of an information set) of coefficients $[A]$ and $[B]$ and half-width ΔA^* of the section of an information set at coefficient B^*.

For comparison, we present the equations $E(T) = A^{LSM} + B^{LSM}T$ of the straight lines whose coefficients A^{LSM} and B^{LSM} were found by LSM using describing function (3). We also give the standard $3\sigma^{LSM}$ estimate (by LSM) of the scatter of emf measurements E_n with respect to approximating line $E(T) = A^{LSM} + B^{LSM}T$.

The approach based on interval estimation gives more complete information on the actual values of the process parameters and operates successfully at a small experimental data sample in the case of both the Pb–Sb (Table 3) and Pb–Bi (Table 4) systems. Moreover, in contrast to standard LSM, it can also be used to take into account the actual level of the total measurement errors and information on their values.

Table 2. Estimated actual level of the total error in measuring emf $e^*_{\max}$ (mV) for the equilibrium potentials of liquid Pb–Bi alloys in the KCl–$PbCl_2$ melt

N_{Pb}, mol %	74	56	35	18
$e_{\max}$, mV	0.35	0.28	0.64	0.85

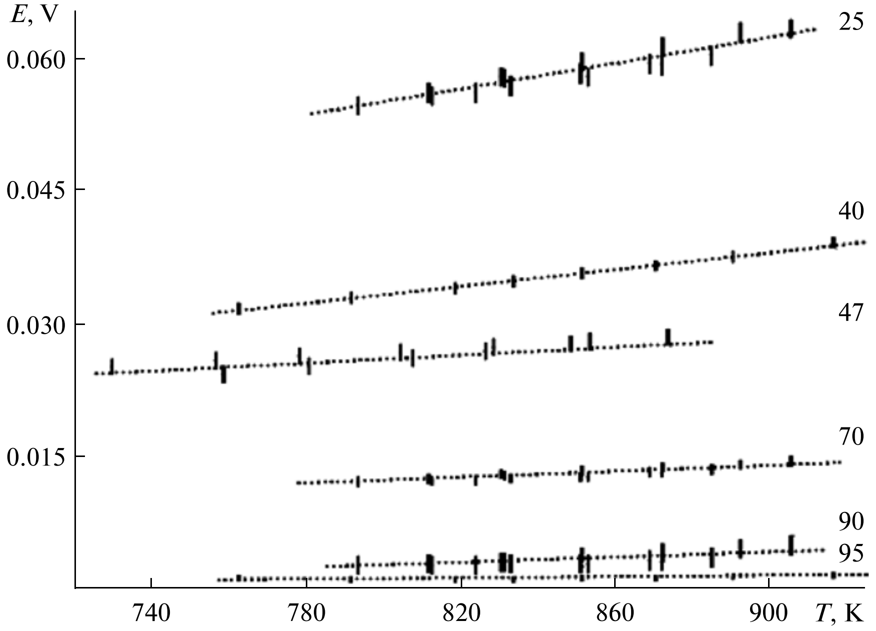

Fig. 1. Temperature dependence of the cell emf at 25, 40, 43, 70, 90, and 95 mol % Pb in the Pb–Sb system.

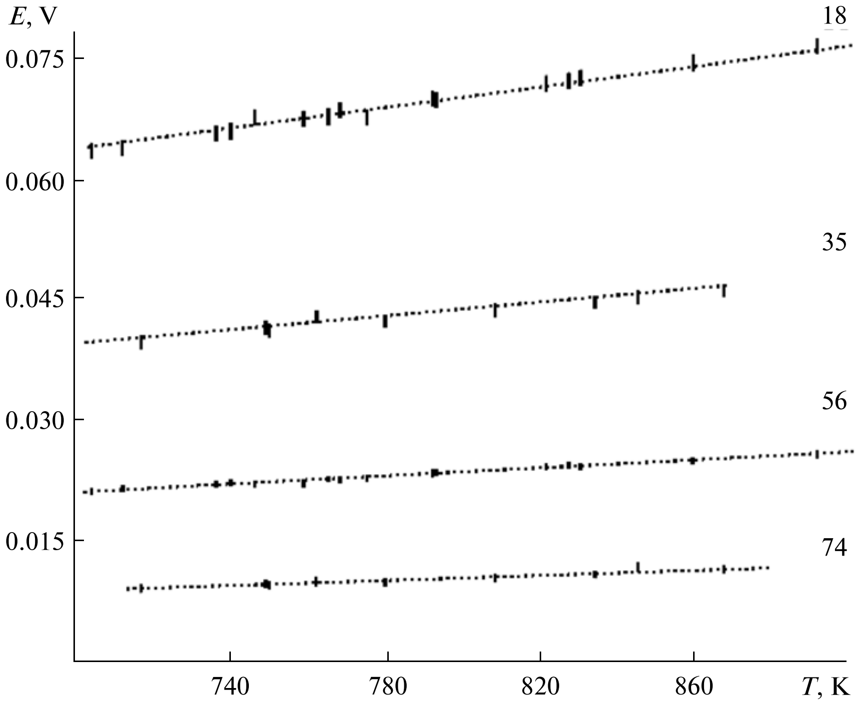

Fig. 2. Temperature dependence of the cell emf at 18, 35, 56, and 74 mol % Pb in the Pb–Bi system.

Figure 3 shows an example of calculating information coefficient set $I(A, B)$ for 18 mol % Pb in the Pb–Bi system. The legend is as follows: information set is gray, the set corners are indicated by solid circles, $[A]$ and $[B]$ are unconditional intervals of the coefficients (dashed lines), A^* and B^* are the midpoint of the information set (open circle), ΔA^* is the half-width of the section of the set at coefficient B^* (thin vertical bar with serifs), A^{LSM} and B^{LSM} represent the point LSM estimation of the coefficients (cross), and

Table 3. Equations for the temperature dependences of emf E (mV) according to interval estimation and LSM for the equilibrium potentials of liquid Pb–Sb alloys in the $KCl–PbCl_2$ melt

N_{Pb}, mol %	Interval estimation $E(T) = A^* + B^*T$, mV	LSM $E(T) = A^{LSM} + B^{LSM}T$, mV
95	$-0.4 + 6.75 \times 10^{-3}\,T$ $[A] = [-21.3; 20.5]$ $[B] = [-2.23 \times 10^{-2}; 2.27 \times 10^{-2}]$ $\Delta A^* = 1.61$	$-0.3 + 2.41 \times 10^{-3}\,T$ $3\sigma^{LSM} = 0.34$
90	$-2.1 + 6.68 \times 10^{-3}\,T$ $[A] = [-23.1; 18.7]$ $[B] = [-1.83 \times 10^{-2}; 3.16 \times 10^{-2}]$ $\Delta A^* = 1.6$	$-2.7 + 7.26 \times 10^{-3}\,T$ $3\sigma^{LSM} = 0.41$
70	$-2.6 + 1.91 \times 10^{-2}\,T$ $[A] = [-31.4; 26,1]$ $[B] = [-1.47 \times 10^{-2}; 5.31 \times 10^{-2}]$ $\Delta A^* = 1.60$	$-2.6 + 1.89 \times 10^{-2}\,T$ $3\sigma^{LSM} = 0.80$
47	$6.4 + 2.56 \times 10^{-2}\,T$ $[A] = [-12.4; 25.2]$ $[B] = [1.83 \times 10^{-3}; 4.93 \times 10^{-2}]$ $\Delta A^* = 1.15$	$5.7 + 2.66 \times 10^{-2}\,T$ $3\sigma^{LSM} = 1.47$
40	$-4.5 + 4.78 \times 10^{-2}\,T$ $[A] = [-12.3; 25.2]$ $[B] = [2.38 \times 10^{-2}; 7.21 \times 10^{-2}]$ $\Delta A^* = 1.49$	$-4.9 + 4.81 \times 10^{-2}\,T$ $3\sigma^{LSM} = 0.54$
25	$-2.8 + 7.26 \times 10^{-2}\,T$ $[A] = [-26.0; 20.3]$ $[B] = [4.56 \times 10^{-2}; 9.96 \times 10^{-2}]$ $\Delta A^* = 0.71$	$-4.7 + 7.47 \times 10^{-2}\,T$ $3\sigma^{LSM} = 2.16$

3σ is the semirange (heavy vertical bar with serifs) of the confidence interval (according to LSM) of the scattering of the measurement results.

A comparison of the $\pm 3\sigma$ interval with coefficient A interval $[A]$ and a comparison of the point estimate of A^{LSM} with coefficient B interval $[B]$ show that the standard LSM method has a lower accuracy (Fig. 3).

Moreover, a strong correlation between coefficients A and B is seen (Fig. 3). This finding demonstrates an obvious advantage of using a "narrow" information set $I(A, B)$ as compared to the traditional box estimation based on rough unconditional intervals $[A]$ and $[B]$.

The activity of lead was calculated from emf measured at several temperatures using the ideal dependence

$$a_{Pb\,n} = \exp(-2FE_n/(RT_n)). \qquad (11)$$

With allowance for estimates (11), we calculated the activity coefficient

$$f_{Pb\,n} = a_{Pb\,n}/N_{Pb}, \qquad (12)$$

where N_{Pb} is the relative mole fraction of lead, at the same temperatures. The estimates performed by Eq. (11) and the activity coefficients calculated by Eq. (12) are given in Table 5 for three (minimum, medium, maximum) temperatures in each experiment.

Over the entire lead content range in Pb–Bi alloys, the deviations from Raoult's law are negative and the activity coefficient of lead decreases.

The partial thermodynamic functions of the characteristics of lead in Pb–Bi alloys were calculated using the average values of coefficients A^* and B^* from the corresponding $I(A, B)$ information sets.

The partial Gibbs energy of lead $\Delta\bar{G}_{Pb}(T)$ (kJ/mol) is

$$\Delta\bar{G}_{Pb}(T) = -2FE(T, A^*, B^*). \qquad (13)$$

The excess energy is calculated by the equation

$$\Delta\bar{G}^{exc}_{Pb}(T) = \Delta\bar{G}_{Pb}(T) - RT\ln(N_{Pb}). \qquad (14)$$

Table 4. Equations for the temperature dependences of emf E (mV) according to interval estimation and LSM for the equilibrium potentials of liquid Pb–Bi alloys in the KCl–$PbCl_2$ melt

N_{Pb}, mol %	Interval estimation $E(T) = A^* + B^*T$, mV	LSM $E(T) = A^{LSM} + B^{LSM}T$, mV
74	$-1.80 + 1.56 \times 10^{-2}\ T$ $[A] = [-22.1; 18.4]$ $[B] = [-1.00 \times 10^{-2}; 4.12 \times 10^{-2}]$ $\Delta A^* = 1.44$	$-3.2 + 1.74 \times 10^{-2}\ T$ $3\sigma^{LSM} = 0.75$
56	$4.9 + 2.34 \times 10^{-2}\ T$ $[A] = [-11.4; 21.3]$ $[B] = [2.84 \times 10^{-3}; 4.41 \times 10^{-2}]$ $\Delta A^* = 1.48$	$3.83 + 2.48 \times 10^{-3}\ T$ $3\sigma^{LSM} = 0.44$
35	$10.8 + 4.07 \times 10^{-2}\ T$ $[A] = [-8.8; 30.3]$ $[B] = [1.59 \times 10^{-2}; 6.55 \times 10^{-2}]$ $\Delta A^* = 1.29$	$11.53 + 3.99 \times 10^{-2}\ T$ $3\sigma^{LSM} = 1.14$
18	$16.7 + 6.93 \times 10^{-2}\ T$ $[A] = [2.9; 30.4]$ $[B] = [4.95 \times 10^{-3}; 8.45 \times 10^{-2}]$ $\Delta A^* = 0.80$	$14.83 + 6.93 \times 10^{-2}\ T$ $3\sigma^{LSM} = 1.16$

To calculate other partial thermodynamic functions, we have to estimate the average value of derivative $(\partial E/\partial T)_p^*$ for the corresponding information set at bismuth concentration p. As noted above (see Figs. 1, 2 and Tables 3, 4), the temperature dependence of emf is rather accurately approximated by a linear equation. Note that information set $I(A, B)$ is found and the average value of coefficient B^* is also

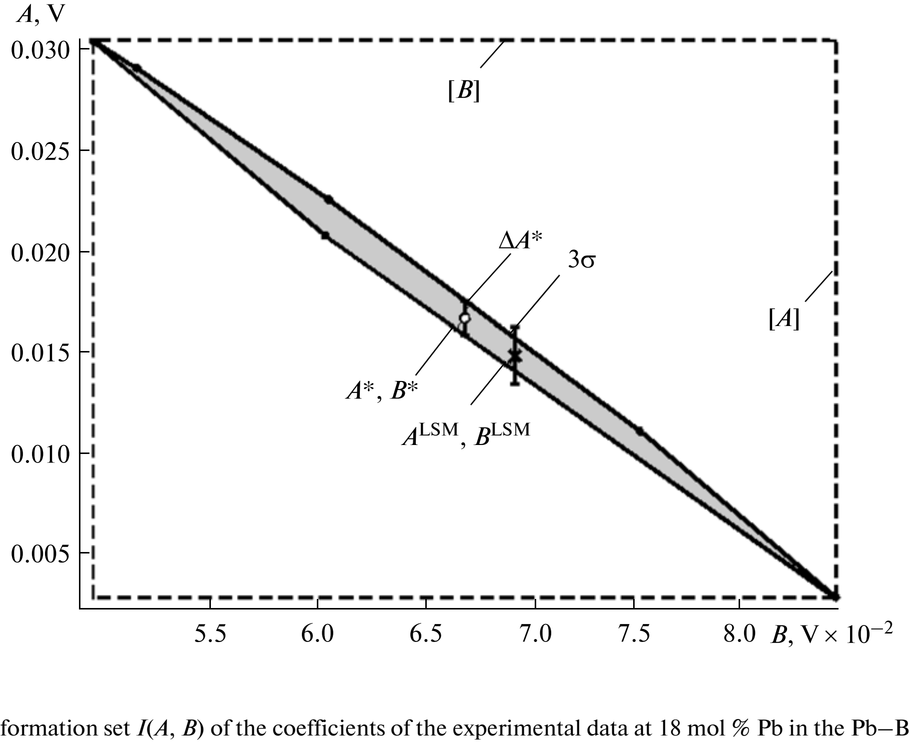

Fig. 3. Information set $I(A, B)$ of the coefficients of the experimental data at 18 mol % Pb in the Pb–Bi system.

Table 5. Estimation of the activity and the activity coefficient of lead in the Pb–Bi system at three temperatures

N_{Pb}, mol %	a_{Pb}	f_{Pb}	a_{Pb}	f_{Pb}	a_{Pb}	f_{Pb}
74	716 K		779 K		866 K	
	0.738385	0.923731	0.742316	0.965763	0.744645	0.930806
56	703 K		792 K		890 K	
	0.493474	0.822456	0.500729	0.834548	0.508891	0.848152
35	716 K		779 K		866 K	
	0.274917	0.687293	0.283271	0.708179	0.290479	0.290479
18	703 K		792 K		890 K	
	0.128128	0.640642	0.129585	0.647926	0.136300	0.681500

Table 6. Interval estimation of the partial functions of lead on liquid Pb–Bi alloys at $T = 866$ K

N_{Pb}, mol %	$\Delta\bar{G}_{Pb}$	$\Delta\bar{G}^{exc}_{Pb}$	$\Delta\bar{H}_{Pb}$	$\Delta\bar{S}_{Pb}$	$\Delta\bar{S}^{exc}_{Pb}$
	kJ/mol			kJ/(mol K)	
74	−2263.2	−656.6	308.8	3.01	1.16
56	−4878.1	−1200.3	−730.5	4.79	5.43×10^{-1}
35	−8885.3	−2289.1	−2082.4	7.85	2.38×10^{-1}
18	−14815.7	−3230.8	−3222.7	13.38	6.00×10^{-3}

found. Therefore, we can determine the derivative irrespective of temperature for each fixed bismuth concentration p according to Eqs. (7) and (8) using the relation

$$(\partial E/\partial T)^*_p = B^*_p. \qquad (15)$$

Then, from E^*_{np} estimated for given temperature T_n and estimated derivative $(\partial E/\partial T)^*_p$, we calculate the following functions:

partial enthalpy of lead $\Delta\bar{H}_{Pb}(T)$,

$$\Delta\bar{H}_{Pb}(T)_n = 2F\{T_n(\partial E/\partial T)^*_p - E^*_{np}\}, \qquad (16)$$

partial entropy of lead $\Delta\bar{S}_{Pb}(T)$,

$$\Delta\bar{S}_{Pb}(T) = 2F(\partial E/\partial T)^*_p, \qquad (17)$$

excess entropy of lead $\Delta\bar{S}^{exc}_{Pb}(T)$,

$$\Delta\bar{S}^{exc}_{Pb}(T)_n = \Delta\bar{S}_{Pb}(T) + R\ln(N_{Pb}). \qquad (18)$$

The calculated values of thermodynamic functions (13)–(18) are given in Table 6 for a temperature of 866 K.

CONCLUSIONS

(1) Using emf measurements, we studied the thermodynamic properties of Pb–Sb and Pb–Bi alloys in the temperature range 720–890 K over a wide composition range.

(2) Small negative deviations from the law of ideal mixtures were detected in the concentration range 18–74 mol % Pb in the systems under study.

(3) With an interval method of processing the experimental results, we were able to calculate the reliable ranges of the estimated parameters in the dependences of thermodynamic functions and the actual level of the total measurement errors. This method can be recommended to estimate the reliability of experimental data at a small number of measurements, e.g., in polarization investigations performed in aggressive media.

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