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Thermodynamic assessment of $EuBr_2$ unary and LiBr–$EuBr_2$ and NaBr–$EuBr_2$ binary systems

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ABSTRACT

As a basis for the design and development of molten salt mixtures, thermodynamic calculations of the phase diagrams and thermodynamic properties were carried out on the $EuBr_2$ unary and LiBr–$EuBr_2$ and NaBr–$EuBr_2$ binary systems over a wide temperature and composition range, respectively. The Gibbs energy of $EuBr_2$ was evaluated using an independent polynomial to fit the experimental heat capacity, the thermodynamic parameters for each phase in the LiBr–$EuBr_2$ and NaBr–$EuBr_2$ systems were optimized by using available experimental information on phase diagrams. A regular substitutional solution model for the liquid phase and Neumann–Kopp rule for the stoichiometric compound $LiEu_2Br_5$ were adopted to reproduce the experimental data with reasonable excess Gibbs energy. Comparisons between the calculated phase diagrams and thermodynamic quantities show that all reliable experimental information is satisfactorily accounted for by the present thermodynamic description. Some thermodynamic properties were predicted to check the suitability of the present calculation.

1. Introduction

Knowledge of the basic physicochemical properties of both pure lanthanide halides and their mixtures with alkali halides can provide useful information for the molten salt mixtures design, application and molten salt technology. This paper deals with the pure $EuBr_2$ and its mixture with light alkali metal halides (LiBr and NaBr) by using the CALPHAD method, aims to critically evaluate the experimental data with the optimized thermodynamic properties for both pure $EuBr_2$ and its mixtures with LiBr and NaBr.

By opposition to the most lanthanide compounds which correspond to the trivalence state (III), europium is one of the few rare earth metals that form stable compounds in the divalent state (II). Experimental investigations [1–4] on pure $EuBr_2$ and its mixture with alkali metal halides were conducted very recently and they are used in the present thermodynamic optimization.

2. Evaluation of experimental data from the literature

2.1. Phase diagram data

By differential scanning calorimeter (DSC) combined with electrical conductivity and spectroscopic measurement, phase diagrams of the LiBr–$EuBr_2$ and NaBr–$EuBr_2$ were, respectively, established by Gadzuric et al. [1] and Ingier-Stocka et al. [2] for the first time. The LiBr–$EuBr_2$ phase diagram [1] was found to be peritectic type with an intermediate compound $LiEu_2Br_5$, which peritectically decomposed at 781 ± 3 K. A eutectic point at 723 ± 3 K and about 31 mol.% $EuBr_2$ was also determined. The phase diagram of NaBr–$EuBr_2$ [2] was found to be of a simple eutectic type without any intermediate compound. The mutual solubility between the $EuBr_2$ and the alkali metal halides LiBr and NaBr was found very limited on the basis of DSC measurements combined with the so-called Tamman construction.

2.2. Thermodynamic data

Before the experimental measurements by Rycerz et al. [3,4], thermodynamic data on pure $EuBr_2$, *i.e.* temperature and enthalpy of fusion [5–7], entropy at 298 K and enthalpy of formation of solid $EuBr_2$ at 298 K [8,9] as well as the heat capacity of solid and liquid $EuBr_2$ [9] were only estimated values based on approximations of thermodynamic information. For this reason, Mucklejohn et al. [10] evaluated the thermodynamic parameters of $EuBr_2$ from the estimated vibrational frequencies. The same authors also concluded that there was a need for reliable experimental information. The estimated enthalpy of formation (−720,000 J/mol), standard entropy (159.75 J/mol K) at 298.15 K and heat capacities of both solid and liquid $EuBr_2$ (108.78 J/mol K) are not used in the present evaluation but only reported for comparison. For the first time, Rycerz et al. systematically

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determined the thermodynamic values of pure $EuBr_2$ [3] and the MBr–$EuBr_2$ binary mixture [4] by DSC and high-temperature Calvet microcalorimetry. Since the experimental procedure was well established and all the manipulations with $EuBr_2$ were performed in an argon glove box with a measured volume fraction of water of about 2×10^{-6} and continuously purified by forced recirculatiuon through external molecular sieves, the measured heat capacities of $EuBr_2$ in the temperature range from 300 to 1100 K [3] are used in the present evaluation for the Gibbs functions of $EuBr_2$. The enthalpy of mixing ($\Delta_{mix}H_m$), in liquid MBr–$EuBr_2$ (M = Li, Na) [4] was found to be so small that it is impossible to measured accurately, therefore, it is not used in this work.

3. Thermodynamic models

The Gibbs energy function ${}^{o}G_i^{\Phi}(T) = G_i^{\Phi}(T) - H_i^{SER}$ for the unary compound i (i = LiBr, NaBr, $EuBr_2$) in the phase Φ is expressed by an equation of the form:

$$ {}^{o}G_i^{\Phi}(T) = a + bT + cT\ln T + dT^2 + eT^{-1} + fT^3 + gT^7 + hT^{-9} \quad (1) $$

where H_i^{SER} is the molar enthalpy of formation of the compound i at 298.15 K and 1 bar in its standard element reference (SER) state, and T is the absolute temperature. The last two terms in Eq. (1) are used only outside the ranges of stability [11], the term gT^7 for a liquid below the melting point and hT^{-9} for solid phases above the melting point.

In the present work, the Gibbs energy functions for pure LiBr and NaBr, *i.e.* ${}^{o}G_{LiBr}^{L}$, ${}^{o}G_{LiBr}^{S}$, ${}^{o}G_{NaBr}^{L}$ and ${}^{o}G_{NaBr}^{S}$, are from the SGTE Molten Salts Database 1996 [12]. As explained in the previous section, the Gibbs energy functions for $EuBr_2$, ${}^{o}G_{EuBr_2}^{L}$ and ${}^{o}G_{EuBr_2}^{S}$, are evaluated in this work, based on the selected experimental thermodynamic data with special attention paid to the recently measured heat capacity [3].

Due to the very limited mutual solubility between alkali metal halides (LiBr and NaBr) and $EuBr_2$, the end-member-component-based solid solutions are treated as pure compounds in this work.

Because the electrical conductivity and the enthalpy of mixing in the liquid can provide powerful structure indication, thus are invaluable in detecting good excess Gibbs energy function to describe the liquid phase satisfactorily and reasonably. In the case of both LiBr–$EuBr_2$ and NaBr–$EuBr_2$ liquid mixtures, the very small negative enthalpies of mixing [4] suggest the low probability of complex formation. And more, the smooth variation of the electrical conductivity with the composition and the simple phase diagram [2] also exclude the formation of any complex species in the NaBr–$EuBr_2$ melts. The electrical conductivity of the LiBr–$EuBr_2$ liquid mixtures shows positive and negative deviations from the Kuroda equation [13], which maybe arise from the specific features of the small Li^+ cation in terms of polarisability [14]. But the structural investigations, by Raman and EXAFS technologies, are considerably desirable to reveal the anticipated existence of associates in the melts. At the present moment, the substitutional solution model, (M^+, Eu^{2+}):(Br^-), is adopted in the present work, and the Gibbs energy of the liquid is described by the Redlich–Kister polynomial [15]:

$$ \begin{aligned} G_m^L - H^{SER} &= x_{M^+}\,{}^{o}G_{MBr}^{L} + x_{Eu^{2+}}\,{}^{o}G_{EuBr_2}^{L} \\ &\quad + RT(x_{M^+}\ln x_{M^+} + x_{Eu^{2+}}\ln x_{Eu^{2+}}) \\ &\quad + x_{M^+}x_{Eu^{2+}}[a_0 + b_0T + (x_{M^+} - x_{Eu^{2+}})(a_1 + b_1T) \\ &\quad + (x_{M^+} - x_{Eu^{2+}})^2(a_2 + b_2T) + \cdots] \quad M = Li, Na \qquad (2) \end{aligned} $$

in which H^{SER} is the abbreviation of $x_{MBr}H_{MBr}^{SER} + x_{EuBr_2}H_{EuBr_2}^{SER}$ and x_{MBr} and x_{EuBr_2} are the mole fractions of MBr and $EuBr_2$, respectively. The coefficients a_j and b_j (j = 0, 1, 2) are the parameters to be optimized.

For the intermediate compound $LiEu_2Br_5$, there was not any experimental thermodynamic data. In this case, the Neumann–Kopp rule is employed:

$$ {}^{o}G_{LiEu_2Br_5}^{S} = {}^{o}G_{LiBr}^{S} + 2\,{}^{o}G_{EuBr_2}^{S} + A_1 + B_1T \quad (3) $$

A_1 and B_1 are the coefficients to be optimized, which are related to the enthalpy and entropy of formation of the compound from the pure bromides.

4. Optimization procedure

The optimization is conducted using the Thermo-calc software package [16]. The critically selected experimental data are processed with a certain weight factor, which should reflect the experimental uncertainty. The step-by-step optimization procedure used in this work is detailed in the following.

The Gibbs energies of solid and liquid $EuBr_2$, in the temperature range where they are stable, are first evaluated from the data measured by Rycerz et al. [3]. Based on the measured data, Rycerz et al. [3] derived C_P expression ($C_P^S = 73.9 + 0.00831T$, $C_P^L = 105.39$) for the solid and liquid $EuBr_2$. In order to check all the original experimental data and to make the experimental data reproduced much better, the C_P expression of $EuBr_2$ is re-evaluated and thus the parameters c, d and e in Eq. (1) for both liquid and solid $EuBr_2$ can be obtained. The melting properties (T, $\Delta_{fus}H_m$ and $\Delta_{fus}S_m$) as well as the thermodynamic properties at 298 K ($\Delta_f H_{298}$ and S_{298}) [3,5–10] are used to evaluated the coefficients a and b in Eq. (1) for both solid and liquid $EuBr_2$. In addition, the Gibbs rule, *i.e.* solid and liquid $EuBr_2$ have the same Gibbs energy at the melting temperature, is taken into account to give the evaluated parameters physical meaning. The obtained thermodynamic parameters for solid $EuBr_2$ below the melting temperature and those for liquid $EuBr_2$ above the melting temperature are subjected to the further extrapolation across the melting temperature, which is necessary for avoiding any risk that a solid phase becomes stable at high temperature, or a liquid phase becomes stable at low temperature. In this way, the method suggested by SGTE [11] is adopted. By introducing T^{-9} term in Eq. (1) for solid $EuBr_2$ above the melting temperature, T^7 term for liquid $EuBr_2$ below the melting temperature, respectively, it forces C_P of the extrapolated phase to approach C_P of the stable phase gradually and produces a kink in C_P at the melting temperature but not a discontinuity, which is more appropriate especially when higher order systems are considered. The finally obtained thermodynamic functions for unary $EuBr_2$ in the temperature where they are thermodynamic stable or metastable are employed in the optimization of LiBr–$EuBr_2$ and NaBr–$EuBr_2$ binary systems.

Since the liquidus lines of LiBr–$EuBr_2$ and NaBr–$EuBr_2$ binary systems have been measured over a wide composition and temperature region, the thermodynamic parameters for the liquid phase are key to the successful description of the considered binary systems. All experimental phase diagram data [1,2] can be satisfactorily reproduced by only introducing a_0 and b_0 two coefficients in Eq. (2). It was impossible to optimize the A_1 and B_1 coefficients in Eq. (3) simultaneously because the experimental data about the intermediate compound $LiEu_2Br_5$ were in a very limited temperature range [1], therefore, a relationship (A_1/B_1 = −3400 K) between the partial enthalpy and excess entropy of the solute suggested by Kubaschewski [17] is applied to reduce the description of the phase to only one independent coefficient.

All the thermodynamic parameters are finally evaluated together and the slight adjustments are made to give the best description of the system. The thermodynamic parameters, finally obtained in this work, are listed in Table 1.

Table 1
Summary of the thermodynamic parameters of the considered systems.

Gibbs energy of pure $EuBr_2$:		
$^{o}G^{L}_{EuBr_2}$	$-753{,}215.9628 + 290.27185T - 66.481301T\ln T - .0082390616T^2 - 280{,}329.34T^{-1} - 7.8121 \times 10^{-19}T^7$	$(298 < T < 941)$
	$-780{,}071.23 + 576.67273T - 105.4T\ln T$	$(941 < T < 6000)$
$^{o}G^{S}_{EuBr_2}$	$-772{,}361.68 + 310.07525T - 66.481301T\ln T - .0082390616T^2 - 280{,}329.34T^{-1}$	$(298 < T < 941)$
	$-804{,}661.0054 + 602.55073T - 105.4T\ln T + 1.3779 \times 10^{29}T^{-9}$	$(941 < T < 6000)$
Gibbs energy of pure LiBr:		
$^{o}G^{L}_{LiBr}$	$-338{,}937.724 - 28.427061T - 4.5652097T\ln T - .070673789T^2 + 1.3999646 \times 10^{-5}T^3 - 430{,}216T^{-1}$	$(298 < T < 823)$
	$-357{,}682.146 + 352.54262T - 65.2704T\ln T$	$(823 < T < 2500)$
$^{o}G^{S}_{LiBr}$	$-368{,}041.904 + 296.462586T - 55.7585T\ln T + .0111371T^2 - 6.7749 \times 10^{-6}T^3 + 169{,}410T^{-1}$	$(298 < T < 600)$
	$-368{,}620.99 + 317.00322T - 59.3618T\ln T + .01832205T^2 - 9.09825 \times 10^{-6}T^3 + 169{,}410T^{-1}$	$(600 < T < 823)$
	$-375{,}338.22 + 373.99593T - 65.2704T\ln T$	$(823 < T < 2500)$
Gibbs energy of pure NaBr:		
$^{o}G^{L}_{NaBr}$	$-353{,}401.598 + 244.433{,}812T - 52.5339T\ln T - .00240438T^2 - 6.31233 \times 10^{-7}T^3 + 129{,}069T^{-1}$	$(298 < T < 300)$
	$-349{,}735.291 + 161.211982T - 38.7385566T\ln T - .022980272T^2 + 3.741877 \times 10^{-6}T^3 - 42{,}439T^{-1}$	$(300 < T < 1020)$
	$-357{,}926.755 + 313.166659T - 62.3416T\ln T$	$(1020 < T < 2500)$
$^{o}G^{S}_{NaBr}$	$-378{,}189.971 + 268.086892T - 52.5339T\ln T - .00240438T^2 - 6.31233 \times 10^{-7}T^3 + 129{,}069T^{-1}$	$(298 < T < 600)$
	$-376{,}481.169 + 242.108585T - 48.5344T\ln T - .006276T^2 + 7 \times 10^{-12}T^3 + 12T^{-1}$	$(600 < T < 1020)$
	$+2{,}125{,}028.83 - 24{,}710.5374T + 3540.45707T\ln T - 2.2933948T^2 + .000273099461T^3 - 3.26719477 \times 10^{8}T^{-1}$	$(1020 < T < 1100)$
	$-384{,}068.177 + 338.794816T - 62.3416T\ln T$	$(1100 < T < 2500)$
The interaction parameters of liquid phase in $LiBr–EuBr_2$ system		
$a_0 = -2630.76, \quad b_0 = 4.64$		
The interaction parameters of liquid phase in $NaBr–EuBr_2$ system		
$a_0 = -2935.6$		
Gibbs energy of intermediate compound $LiEu_2Br_5$:		
$^{o}G^{S}_{LiEu_2Br_5} = {}^{o}G^{S}_{LiBr} + 2{}^{o}G^{S}_{EuBr_2} - 5395.8 + 1.587T$		

In J/mol. Temperature (T) in K.

5. Results and discussion

The present calculated thermodynamic properties of $EuBr_2$, such as $\Delta_f H_{298}$, S_{298}, $\Delta_{fus}H_m$, $\Delta_{fus}S_m$ as well as the melting temperature (T_m) are listed in Table 2 together with the data from the literature [3,5–10]. Fig. 1 shows a comparison of the present calculated heat capacity values of $EuBr_2$ with the measured [3] and the previous estimated data from the literature [9,10]. It is found that the present calculation can reproduce the measured heat capacity of solid $EuBr_2$, temperature and enthalpy of fusion [3,7] and previous evaluated heat capacity of liquid $EuBr_2$ [3] quite well. The previous estimations [9,10] were based on the approximations of the thermodynamic information and were found to be higher by about 8% in the whole temperature range.

The calculated phase diagrams of the $LiBr–EuBr_2$ and $NaBr–EuBr_2$ binary systems following the assessment are shown in Figs. 2 and 3, respectively, with corresponding experimental data marked [1,2]. Table 3 shows the measured invariant reaction points [1,2] and the assessed by the present authors. It can be seen that the present calculation can account for most experimental phase diagrams and thermodynamic data within the experimental errors.

The model-predicted enthalpies of liquid $LiBr–EuBr_2$ and $NaBr–EuBr_2$ mixtures at 1055 K are displayed in Fig. 4. The present calculated enthalpies of liquid $LiBr–EuBr_2$ and $NaBr–EuBr_2$ mixtures at 1055 K show the very small negative with the minimum values at about 50 mol.% $EuBr_2$, which excludes any complex formation in the considered systems and is in consistence with the

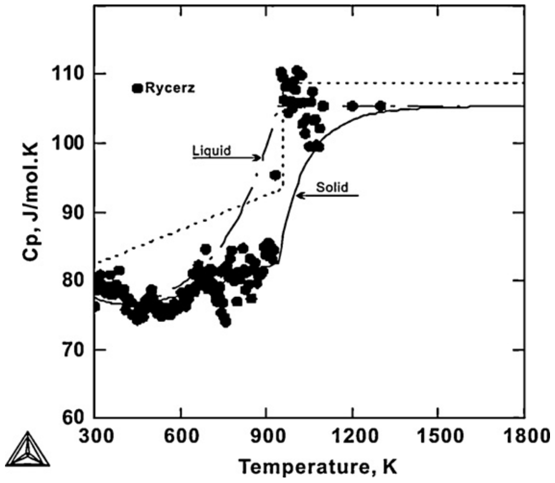

Fig. 1. Calculated heat capacity of $EuBr_2$ compared with the data from the literature [3]. The solid line is for solid $EuBr_2$. The dashed line is for liquid $EuBr_2$. The dotted line is the previous estimations by Mucklejohn et al. [10].

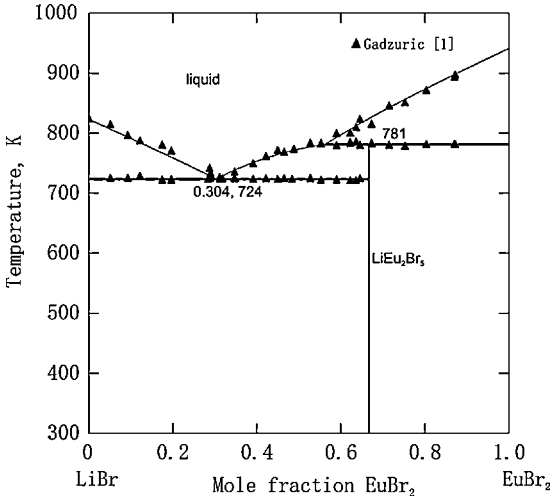

Fig. 2. Calculated $LiBr–EuBr_2$ phase diagram compared with experimental data from the literature [1].

Table 2
Assessed thermodynamic properties of $EuBr_2$ compared with the corresponding data in the literature.

$\Delta_f H_{298}$ (J/mol)	S_{298} (J/mol K)	$\Delta_{fus}H_m$ (J/mol)	$\Delta_{fus}S_m$ (J/mol K)	T_m (K)	Reference
		22,200	23.59	941	[3]
				950	[5]
				975	[6]
		25,100	26.7	940	[7]
−753,700	136.92				[8,9]
−720,000	159.75	25,104	26.26	956	[10]
−753,700	136.91	22,208	23.6	941	This work
Expressions of C_P (J/mol K)					
Solid $EuBr_2$:	$C_P = 73.79 + .00831T$			$(298 < T < 941)$	[3]
	$C_P = 77.41 + .01672T + 9.96 \times 10^{-9}T^2 - 140T^{-2}$			$(298 < T < 956)$	[10]
	$C_P = 66.48 + .016578T + 560{,}658.68T^2$			$(298 < T < 941)$	
	$C_P = 105.4 - 1.2401 \times 10^{31}T^{-10}$			$(941 < T < 6000)$	This work
Liquid $EuBr_2$:	$C_P = 105.39$			$(941 < T < 1300)$	[3]
	$C_P = 108.78$			$(956 < T < 1800)$	[10]
	$C_P = 66.48 + .016578T + 560{,}658.68T^2 + 3.2812 \times 10^{-17}T^6$			$(298 < T < 941)$	
	$C_P = 105.4$			$(941 < T < 6000)$	This work

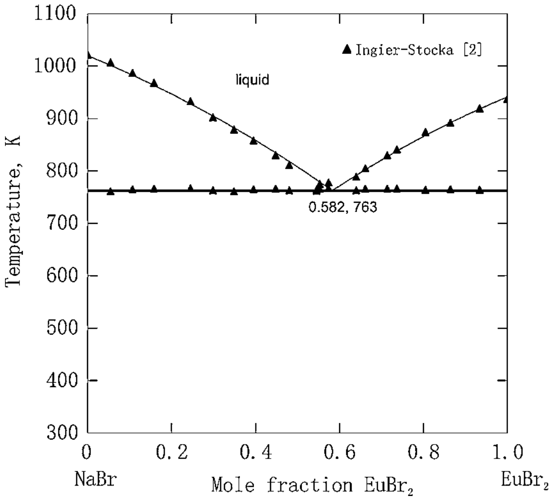

Fig. 3. Calculated NaBr–$EuBr_2$ phase diagram compared with experimental data from the literature [2].

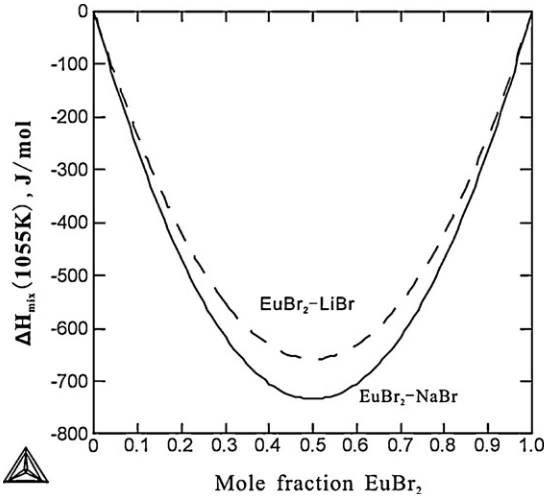

Fig. 4. Model-predicted enthalpy of mixing for the $MBr–EuBr_2$ (M = Li, Na) liquid at 1055 K. The reference states are liquid LiBr, NaBr and $EuBr_2$.

experimental results [4]. A further check on the final optimization is provided by Figs. 5 and 6, where the calculated enthalpies of formation at 298 and 724 K for the LiBr–$EuBr_2$ system, at 763 K for the NaBr–$EuBr_2$ system are shown, respectively. The present calculated enthalpies of formation at both 298.15 and 724 K for the LiBr–$EuBr_2$ system show the most negative value at 66.7 mol.% $EuBr_2$ composition, which corresponds to the compound $LiEu_2Br_5$. The maximum value at 724 K and 30.4% mol. $EuBr_2$ is connected to the eutectic point in the LiBr–$EuBr_2$ system. For the NaBr–$EuBr_2$ system, the calculated enthalpy of formation at 763 K is found

Table 3
Comparison between the calculated and measured invariant reaction points in the LiBr–$EuBr_2$ and NaBr–$EuBr_2$ systems.

Equilibrium composition (mol.% $EuBr_2$), T (K)		Reference
Liquid ↔ LiBr + $LiEu_2Br_5$		
31.9 ± 1	723 ± 3	[1]
30.4	724	This work
Liquid + $EuBr_2$ ↔ $LiEu_2Br_5$		
	781 ± 3	[1]
56.3	781	This work
Liquid ↔ NaBr + $EuBr_2$		
54.6 ± 1	762 ± 3	[2]
58.2	762	This work

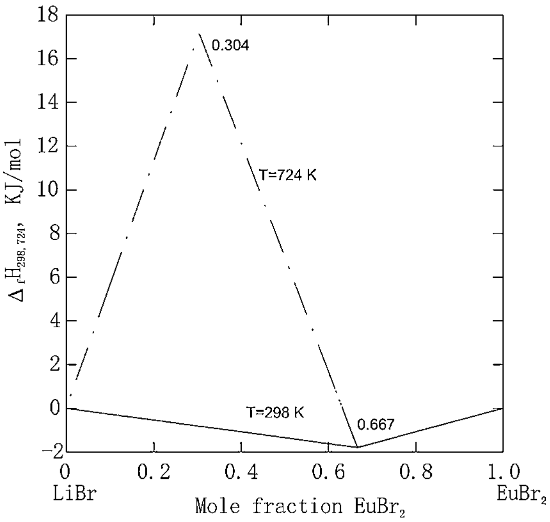

Fig. 5. Calculated enthalpies of formation for the LiBr–$EuBr_2$ system at 298 and 724 K. The solid line is for 298 K, the dashed line is for 724 K. The reference states are stable solid LiBr and $EuBr_2$.

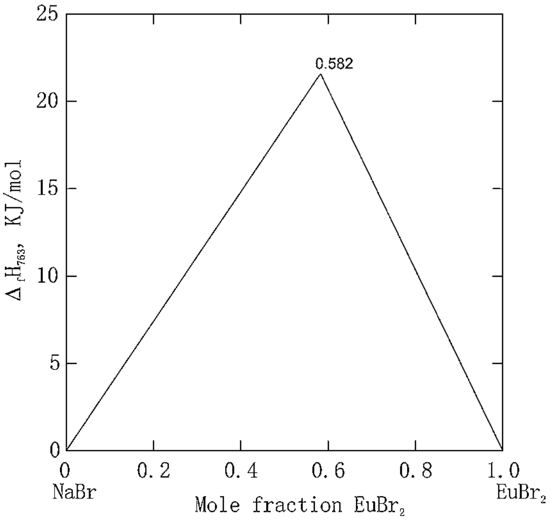

Fig. 6. Calculated enthalpy of formation for the NaBr–$EuBr_2$ system at 763 K. The reference states are stable solid NaBr and $EuBr_2$.

to have the most positive values at 58.26 mol.% $EuBr_2$, which is expected in view of the lowest melting temperatures of the eutectic product.

6. Conclusions

1. The Gibbs energies of unary $EuBr_2$ in the temperature where the phases are thermodynamically stable or metastable have been evaluated. The thermodynamic functions can satisfactorily explain all reliable data from the literature and be extrapolated across the melting point without a jump in C_P. The present obtained thermodynamic parameters have been used to calculate the phase relationships in the $EuBr_2$-included binary systems.
2. A regular substitutional solution model, resembling the structure of liquid phase together with Neumann–Kopp rule for the stoichiometric compound $LiEu_2Br_5$ is capable of giving a quantitative and consistent description of the phase diagrams and thermodynamic data in the LiBr–$EuBr_2$ and NaBr–$EuBr_2$ systems.

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