

Article

Transport Properties of EuCl_2 and Phase Equilibria in LiCl-EuCl_2 System

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Abstract

The ionic conductivity of EuCl_2 was investigated at high temperatures, including the melting point. Measurements were performed on polycrystalline specimens obtained by the solidification of the corresponding melt. The behavior of the conductivity and the apparent Arrhenius activation energy was characterized. It is suggested that the conductivity behavior results from anion sublattice disordering within the crystals up to the melting point. The variation in transport properties correlates well with the crystal structure of EuCl_2 . The phase diagram of the LiCl-EuCl_2 binary system was constructed based on DSC measurements. This system was found to be a simple eutectic type featuring the incongruently melting compound LiEu_3Cl_7 . The CALPHAD method was employed for thermodynamic modeling, incorporating both experimental and literature data for the LiCl-EuCl_2 system.

Keywords: europium dichloride; lithium chloride; phase diagram; phase transition; electrical conductivity

1. Introduction

Although lanthanide elements are usually trivalent, stable divalent compounds exist with Sm, Eu, and Yb. Europium(II) chloride (EuCl_2) belongs to these compounds. It finds applications in various fields, including phosphors, lasers, and optical materials. EuCl_2 is a crucial component in the synthesis of phosphors—materials that emit light when stimulated. These materials are vital for fluorescent lighting, cathode ray tubes, and medical imaging [1]. EuCl_2 is also used to produce various optical devices, including optical fibers, amplifiers, and switches [2], and has been investigated for its potential use in humidity sensors [3], as well as in cryogenic refrigeration due to its significant magnetocaloric effect [4]. Therefore, knowledge of its physicochemical properties is crucial from both a scientific and technological point of view.

According to the literature [5,6], EuCl_2 is orthorhombic at room temperature and undergoes a phase transition to a cubic structure at higher temperatures. However, older literature data regarding the transformation and melting temperatures were contradictory. According to Fink and Seifert [6], EuCl_2 undergoes a phase transformation at 1015 K and melts at 1126 K. Later literature data [7] reported that fusion occurs at 1017 K, and although an effect was detected at 1124 K, it was supposedly attributed to a transition in the liquid phase. This assumption was supported by the authors' interpretation of Raman spectra. In 2001, Da Silva et al. [8] performed DSC studies that confirmed the results of Fink and Seifert regarding the phase transition and fusion temperatures (1014 K



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and 1125 K, respectively). At the same time, their molar heat measurements indicated the possibility of disordering in the high-temperature modification. The experimental method most commonly used to investigate such disorder is electrical conductivity, as disordering should influence the transport properties of EuCl_2 . Thus, we have carried out electrical conductivity measurements in both the solid and liquid phases of this compound. Additionally, we have determined the EuCl_2 – LiCl phase diagram, which is currently missing from the literature. Knowledge of this phase diagram provides thermodynamic and physical guidance essential for pyroprocessing spent nuclear fuel, developing advanced molten salt reactors (MSRs), and lanthanide separations. The studied LiCl – EuCl_2 system is of practical importance due to the fact that LiCl molten salt is used as a reduction medium in electrochemical reduction, which is a key process in pyroprocessing technology for the treatment of spent nuclear fuels. The $\text{EuCl}_3/\text{EuCl}_2$ redox couple serves as a fundamental indicator of the salt's redox potential. Understanding the phase equilibria determines the operating temperatures required to keep these species in a molten, non-precipitating state, allowing for the selective recovery of uranium and the control of corrosion in electrorefining cells [9].

2. Materials and Methods

2.1. Chemicals

Europium dichloride was obtained by the chlorination of europium oxide (Eu_2O_3 , Johnson Matthey, 99.9%, Gliwice, Poland) with thionyl chloride (Johnson Matthey, 99%) according to the method described in detail previously [8].

Lithium chloride was purchased from Merck (min. 99.9%, Poznan, Poland). It was dehydrated by continuous and progressive heating to just above its melting point under an HCl flow in quartz ampoules. Excess HCl was subsequently removed by bubbling argon through the melt.

Since the investigated salts are highly moisture-sensitive, they were handled in a controlled argon-atmosphere glove box (water content < 1 ppm) and stored in sealed glass ampoules.

2.2. Measurements

Electrical conductivity measurements were carried out in a capillary quartz cell with platinum electrodes (described in detail in [10]). It consists of two quartz tubes connected by a capillary with an inner diameter of approximately 1 mm. The tubes were hermetically closed with rubber plugs. A special sluice was welded to the cell for sample loading. The cylindrical electrodes were made from platinum foil ($\sim 4 \text{ cm}^2$) welded to platinum wires with a diameter of 1 mm. The cell was placed into a furnace containing a stainless steel block in order to achieve a uniform temperature distribution. The furnace temperature was controlled by a EUROTHERM regulator (Eurotherm Limited (Watlow), Durrington, UK) with a stability of better than 1 K and measured by means of a Pt-Rh10%Pt thermocouple with an accuracy of $\pm 1 \text{ K}$. The hot junction of the thermocouple was positioned tightly against the cell capillary. A CDM230 conductivity meter (Radiometer Analytical SAS, Villeurbanne, France) was used for the conductivity measurements. The temperature and conductivity values were simultaneously recorded on a computer every 10 s. Sample loading into the cell was performed only through the sluice to prevent any changes in the electrode positions. Measurements were performed in a static atmosphere of pure argon (water content less than 2 ppm). They were started from liquid phase. Argon bubbles adsorbed on the electrode surface can lower the measurement results. Therefore, before starting the measurements, they were removed by repeated pulsed argon flows. This procedure was repeated until a stable measurement result was obtained. The working

frequency of the conductivity meter was automatically selected as 23.4 kHz, i.e., far beyond the frequency range where the resistance is frequency-dependent. The experimental cell was calibrated using a pure NaCl melt [11]. Three independent series of conductivity measurements (heating, cooling) of liquid KCl were carried out in the temperature range of 1050–1150 K. The obtained results allowed us to determine the cell constant as $2509 \pm 2.1 \text{ m}^{-1}$. The temperature was automatically increased and decreased at a rate of $1 \text{ K} \cdot \text{min}^{-1}$. Two series (heating, cooling) of EuCl_2 conductivity measurements were performed. Although the accuracy of the CDM230 conductivity meter is 0.2%, the accuracy of the specific conductivity data is estimated to be $\pm 2\%$ when taking into account all other possible errors.

The temperature and enthalpy values of the phase transitions of EuCl_2 –LiCl binary mixtures were measured with a Setaram DSC 121 differential scanning calorimeter (Setaram, Caluire-et-Cuire, France) equipped with a unique 3D DSC sensor, which totally surrounds the sample and reference crucibles. The sensor used can measure up to 94% of all heat exchanged with the sample/reference crucibles, as compared to the 20% typically achieved (50% absolute maximum) with 2D sensors. The apparatus was calibrated via the “Joule effect” (determination of enthalpy correction coefficients) and by measuring standard substances (high-purity (99.999%) In, Sn, Zn, Sb, and Ag metals) conducted at heating rates of 1, 5, and $10 \text{ K} \cdot \text{min}^{-1}$ (determination temperature correction coefficients). The enthalpy and temperature correction coefficients were calculated using the Setsoft software version 1.54. Prior to the measurements, the apparatus was tested with high-purity metals, and the obtained results showed that the uncertainty in the measurement of enthalpy and temperature across the whole temperature range was 1% and 1 K, respectively. Samples of 300–500 mg were placed in quartz ampoules (approximately 6 mm in diameter and 15 mm in length) sealed under reduced argon pressure. The side walls of the ampoules were designed to achieve a tight fit into the heat flow detector. The experiments were conducted at a heating and cooling rate of $5 \text{ K} \cdot \text{min}^{-1}$. Data acquisition and processing were computer-operated using Setsoft Thermal Analysis Software.

The phase diagram of the LiCl– EuCl_2 pseudo-binary system was evaluated using the CALPHAD method and PANDAT EDUCATION 2022 software.

3. Results and Discussion

3.1. Electrical Conductivity of EuCl_2

The results of the electrical conductivity measurements of europium(II) chloride are presented in Figure 1, together with literature data on the phase transition, melting temperatures, and the molar heat capacity of this compound [8]. Our findings confirmed the correctness of the phase transition and melting temperatures determined by Da Silva et al. [8] (1014 K and 1125 K, respectively) and Fink and Seifert [6] (1015 K and 1126 K, respectively).

Electrical conductivity measurements performed during heating and cooling cycles revealed the existence of a conductivity hysteresis loop. This loop is related to the supercooling effect characteristic of lanthanide halides [12]. The width of this hysteresis is 20–25 K.

The conductivity of the low-temperature modification of EuCl_2 (which has a co-tunnite structure and crystallizes in the orthorhombic Pnma space group [13]) increases monotonically with increasing temperature, from approximately $0.0059 \text{ S} \cdot \text{m}^{-1}$ at 850 K to $0.0547 \text{ S} \cdot \text{m}^{-1}$ at 1010 K. An abrupt increase of over three orders of magnitude in the ionic conductivity of EuCl_2 takes place at the solid–solid phase transition temperature from the orthorhombic to the cubic fluorite-type crystal structure ($0.070 \text{ S} \cdot \text{m}^{-1}$ at 1013 K vs. $58.17 \text{ S} \cdot \text{m}^{-1}$ at 1020 K).

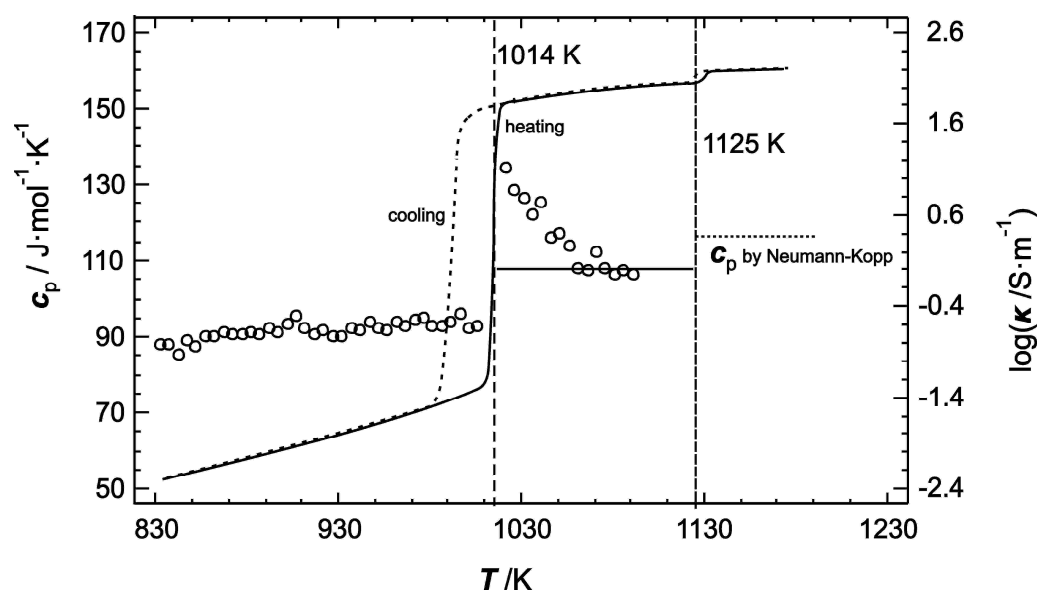


Figure 1. Temperature dependence of electrical conductivity and heat capacity of EuCl_2 .

The conductivity of the high-temperature modification further increases with temperature up to $117.6 \text{ S}\cdot\text{m}^{-1}$ at 1124 K . This value is only slightly lower than that found above the melting point ($124.1 \text{ S}\cdot\text{m}^{-1}$ at 1129 K). Similar behavior has been described in the literature for BaCl_2 [14], where the transition from the low-temperature modification (cotunnite-structured, orthorhombic Pnma space group) to the high-temperature modification (cubic fluorite-type structure) resulted in a similar jump in electrical conductivity, caused by significant disordering of the anionic sublattice.

Given that EuCl_2 is isostructural with BaCl_2 , a similar disordering of the anionic sublattice is to be expected for EuCl_2 . According to the O’Keeffe and Hyde classification [15], EuCl_2 belongs to class II compounds, i.e., compounds whose conductivity changes abruptly at the phase transition temperature, becoming comparable to the conductivity of the liquid phase. This phenomenon results from the loss of long-range order in the anionic sublattice. At a sufficiently high temperature, these anions can be statistically dispersed throughout the structure without being assigned to specific lattice sites. This state can be described as “structural disorder”, and the anion sublattice that has undergone this loss of order can be considered quasi-liquid [16].

Heat capacity measurements (Figure 1) confirm this disordering process and indicate that a state of complete disorder occurs at a temperature approximately $50\text{--}60 \text{ K}$ above the phase transition. Further confirmation of this structural disorder can be found in the literature [7,17], where structural observations obtained by Raman spectroscopy and neutron diffraction suggested a liquid-like behavior of EuCl_2 above 1017 K , i.e., immediately above the phase transition temperature.

The electrical conductivity of EuCl_2 was plotted against temperature as $\ln(\kappa) = f(1/T)$. This dependence deviates from linearity for both solid phases of europium(II) chloride (Figure 2). A linear dependence was observed only for the liquid phase.

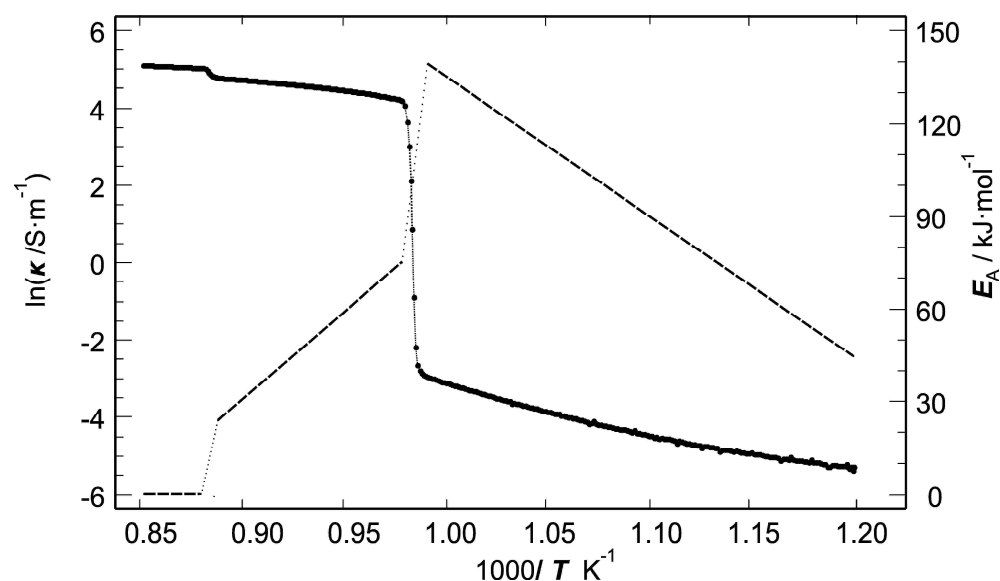


Figure 2. $\ln(\kappa)$ (left axis—black points) and activation energy (right axis—dashed and dotted lines) for electrical conductivity as a function of $(1000/T)$.

The experimental conductivity data were well represented by the polynomial equation

$$\ln(\kappa) = A_0 + A_1 \cdot (1/T) + A_2 \cdot (1/T)^2 \quad (1)$$

where A_0 , A_1 and A_2 , are coefficients determined by the least-squares method. The activation energy (E_A), evaluated via the Arrhenius equation, was defined as:

$$E_A(T) = -R \cdot [d \ln(\kappa)] / (d(1/T)) \quad (2)$$

where R is the gas constant. It becomes:

$$E_A(T) = -R \cdot [A_1 + 2 \cdot A_2 \cdot (1/T)] \quad (3)$$

All A_i coefficients are listed in Table 1, while the temperature dependence of E_A is presented in Figure 2.

Table 1. Coefficients of the equation $\ln(\kappa) \pm \Delta \ln(\kappa) = A_0 + A_1 \cdot (1/T) + A_2 \cdot (1/T)^2$ (κ in $\text{S} \cdot \text{m}^{-1}$).

Temperature Range/K	A_0	A_1	A_2	$\Delta \ln \kappa$
832–1008 low temp. modification	40.27 ± 0.48	$-70,460 \pm 870$	$27,080,000 \pm 397,000$	0.01880
1020–1125 high temp. modification	19.48 ± 0.83	$57,440 \pm 1780$	$-33,950,000 \pm 953,000$	0.005432
1135–1174 liquid	503.71 ± 1.11	$-399,800 \pm 1300$	0	0.0008713

$\Delta \ln(\kappa)$ is the standard error of estimation.

The activation energy for electrical conductivity is strongly influenced by defects in the material's structure, with higher defect concentrations often leading to lower activation energies. Defects can create new energy levels within the band gap, facilitating the movement of charge carriers and thereby reducing the energy barrier required for conduction. Because defects provide easier pathways for charge carriers, the activation energy decreases. This

leads to increased conductivity, especially at lower temperatures where thermal energy alone is insufficient to overcome the larger intrinsic band gap energy.

The above mechanism is in perfect agreement with the conductivity behavior observed for the high-temperature modification of the EuCl_2 compound. As the temperature increases, the defect concentration in the anionic sublattice increases, which results in a decrease in the activation energy and a corresponding increase in electrical conductivity. However, the conductivity behavior of the low-temperature modification is anomalous: the activation energy of electrical conductivity increases instead of decreasing with increasing temperature. It appears that this effect results from perturbations in the concentrations of various types of defects, such as short-lived clusters comprising anion Frenkel interstitials, anion vacancies and surrounding relaxed anions. However, it must be pointed out that it is only tentative hypothesis based on literature information [18].

3.2. LiCl-EuCl_2 Phase Diagram

The DSC investigation performed on samples with ten different compositions yielded both the temperatures and the fusion enthalpies of the respective mixtures. Due to a frequent supercooling effect [12], all experimental temperatures and enthalpies were determined from the heating curves. Representative DSC heating thermograms are presented in Figure 3.

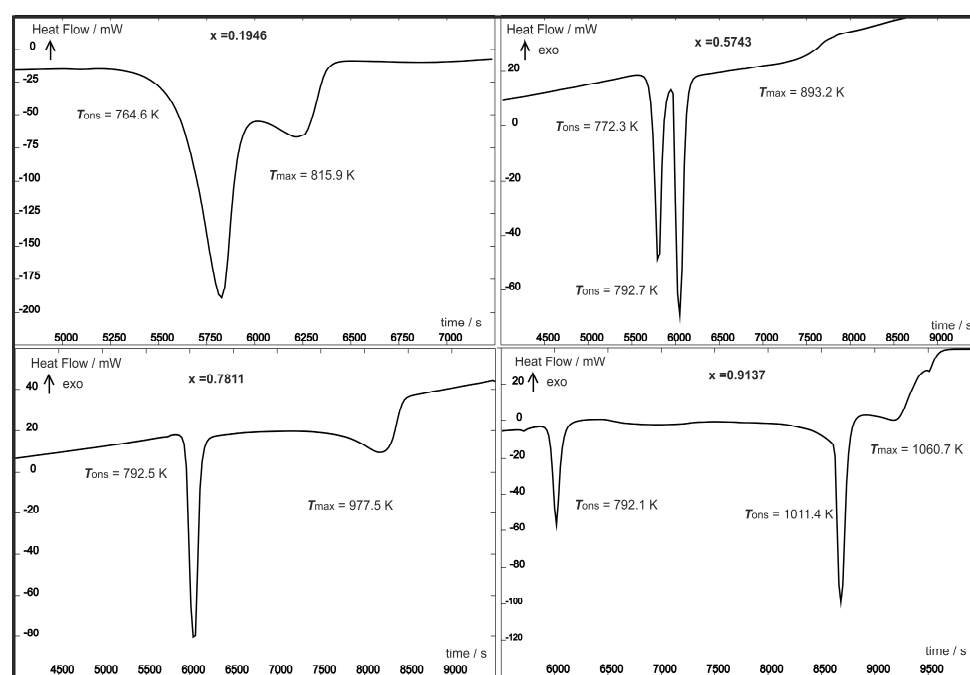


Figure 3. DSC heating curves for the LiCl-EuCl_2 system recorded at a heating rate of 5 K min^{-1} (the arrows indicate the direction of the exothermic effect).

Depending on the sample composition, either two or three thermal effects were observed on the recorded thermograms (Figure 3). The effect occurring at the highest temperature was ascribed to the liquidus, and its temperature was determined from the peak maximum. The other two effects were assigned to the incongruently melting LiEu_3Cl_7 compound ($792 \pm 3 \text{ K}$), the $\text{LiCl-LiEu}_3\text{Cl}_7$ eutectic ($772 \pm 3 \text{ K}$), or the phase transition of EuCl_2 (1014 K), respectively. Their temperatures were determined as the onset temperature (Tonset). All gathered data are summarized in Table 2.

Table 2. Results of the DSC experiments performed on the LiCl–EuCl₂ system: indexes 1, 2 and 3 correspond to the LiCl–LiEu₃Cl₇ eutectic, incongruent melting of LiEu₃Cl₇ compound and solid–solid phase transition of EuCl₂, respectively.

$x(\text{EuCl}_2)$	T_1 K	ΔH_1 J·mol ^{−1}	T_2 K	ΔH_2 J·mol ^{−1}	T_3 K	ΔH_3 J·mol ^{−1}	T_{liq} K
0.0000		0.00					883.0
0.1001	773.0	5.46					847.7
0.1946	764.5	11.57					815.9
0.3021	774.3	17.65					789.0
0.3979	772.4	13.95					794.8
0.4864	774.4	11.03	793.4	2.48			855.4
0.5743	772.4	7.00	792.8	4.37			893.3
0.6930	773.3	3.74	792.6	6.11			938.9
0.7811			791.8	6.78			977.5
0.9137			792.4	2.40	1011	30.19	1060.7
1.0000				0.00	1014	11.50	1125.0
-	772		795		1014		

The phase diagram presented in Figure 4A was found to be of the eutectic type featuring one incongruently melting compound, LiEu₃Cl₇. The mole fraction of EuCl₂ determined from the Tammann plot (Figure 4B) was 0.753 ± 0.014 , which is in excellent agreement with the stoichiometry of the aforementioned compound (0.750). Its enthalpy of melting was determined to be $29.7 \text{ kJ} \cdot \text{mol}^{-1}$.

The LiCl–LiEu₃Cl₇ eutectic composition was determined to be $x(\text{EuCl}_2) = 0.302 \pm 0.011$ using the Tammann construction presented in Figure 4B. The enthalpy of fusion for the eutectic composition is $17.30 \pm 0.46 \text{ kJ} \cdot \text{mol}^{-1}$. In constructing the Tammann diagram, it was assumed that there was no solid-state solubility. Thus, the straight line intersects the composition axis exactly at $x(\text{EuCl}_2) = 0$. The construction of the Tammann diagrams also allowed for the addition of data points locating additional liquidus temperatures (represented by the black points in Figure 4A).

3.3. Calculation of LiCl–EuCl₂ Phase Diagram

The CALPHAD modeling was performed using the PANDAT EDUCATION 2022 software. The thermodynamic properties of the pure system components are described by the following equation:

$$\{G^0(T) - H^{\text{SER}}(298.15 \text{ K})\}_i = A_i + B_i \cdot T + C_i \cdot T \cdot \ln T + D_i \cdot T^2 + E_i \cdot T^{-1} \quad (4)$$

where A_i to E_i are the coefficients describing the temperature dependence of the Gibbs free energy $G^0(T)$, and $H^{\text{SER}}(298.15 \text{ K})$ is the enthalpy in the standard element reference state. The thermodynamic data for LiCl and EuCl₂ were taken from Kubaschewski et al. [19] and Da Silva et al. [8], respectively. The value of $S(298 \text{ K})$ was estimated using the Latimer method [20]. The coefficients for Equation (4) are listed in Table 3. The calculated phase diagram is shown as solid lines in Figure 4A.

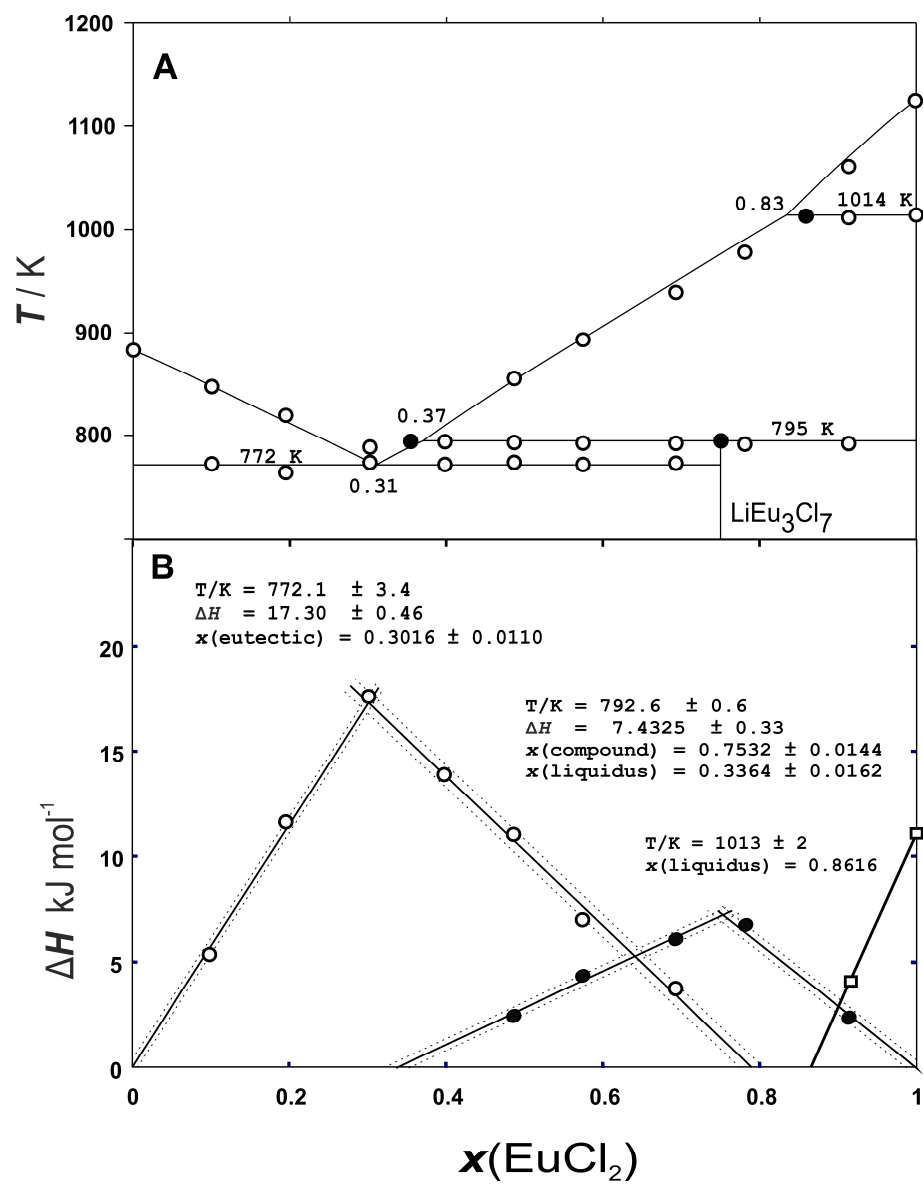


Figure 4. The phase diagram of the LiCl–EuCl₂ system (A) and Tammann diagrams for the LiCl–LiEu₃Cl₇ eutectic (open circles), LiEu₃Cl₇ compound (black circles) and EuCl₂ phase transition (open squares) (B). The phase diagram contains numerical data obtained using the CALPHAD calculations. The black points in phase diagram were deduced from Tammann diagrams.

Table 3. Coefficients of Equation (4) used for CALPHAD calculation with {*G*⁰(*T*) – *H*^{SER}(298.15 K)} expressed in kJ·mol^{−1}.

Compound	A _i	B _i × 10 ^{−2}	C _i	D _i	E _i	Temperature Range/K	Ref.
LiCl (solid)	−16,631.1428	230.570038	−41.42	−0.01795	0	298–883	[19]
LiCl (liquid)	−14,872.4883	435.644474	−73.39	0	0	883–	[19]
EuCl ₂ (solid 1)	−20,879.5115	331.6108	−66.41	−0.01389	−23,200	298–1014	[8]
EuCl ₂ (solid 2)	−40,037.86411	643.02404	−110.71	0	0	1014–1125	[8]
EuCl ₂ (liquid)	−28,099.49380	674.49735	−116.7	0	0	1125–1200	*

* heat capacity estimated by Neumann–Kopp rule.

The Gibbs energy of mixing for the liquid phase was expressed using the Redlich–Kister polynomial:

$$G^{\text{MIX}} = -R \cdot T \cdot [x_{\text{LiCl}} \cdot \ln(x_{\text{LiCl}}) + x_{\text{EuCl}_2} \cdot \ln(x_{\text{EuCl}_2})] + x_{\text{LiCl}} \cdot x_{\text{EuCl}_2} \cdot \sum L_i \cdot (x_{\text{LiCl}} - x_{\text{EuCl}_2})^i \quad (5)$$

where $L_i = H_i + S_i \cdot T$ (with i starting from 0), H_i and S_i are the optimized parameters, and x_{LiCl} and x_{EuCl_2} are the mole fractions of LiCl and EuCl_2 , respectively. The interaction coefficients describing the liquid phase are given in Table 4.

Table 4. The coefficients of $L_i = H_i + S_i \cdot T$ obtained from the CALPHAD calculation.

$H_i/\text{J mol}^{-1}$	$S_i/\text{J mol}^{-1} \text{K}^{-1}$
−700	0
1700	1.5
−1800	−2.5

The accuracy of the obtained thermodynamic data is limited due to the scarcity of experimental data for the mixed liquid phase, meaning that the temperature dependence of the enthalpy and entropy of mixing was omitted. However, the composition dependence of the mixing enthalpy and excess entropy at 1125 K on the mole fraction (Figure 5) reveals relatively small negative values and a distinct asymmetry, with a minimum located near the stoichiometry of the solid intermediate compound. This asymmetry also highlights the liquid solution composition that is thermodynamically most favorable for a possible associate formation reaction.

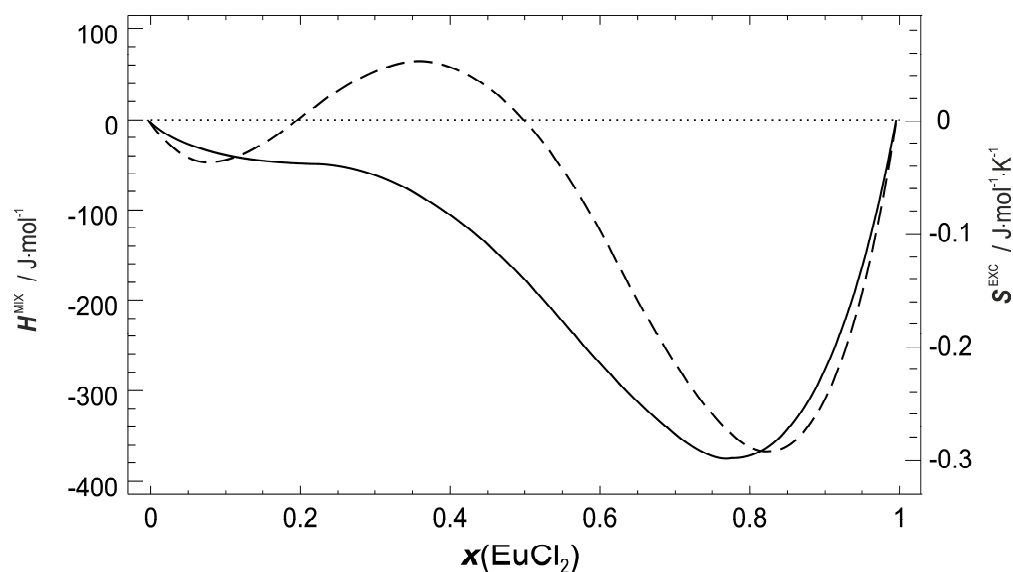


Figure 5. Estimated dependence of the mixing enthalpy (solid line, left axis) and excess entropy (dashed line, right axis) on the mole fraction of EuCl_2 at 1125 K.

The solid phase compound LiEu_3Cl_7 decomposes at 795 K. The Gibbs free energy of its formation from the pure system components, determined via the CALPHAD procedure at the decomposition temperature, was estimated to be $G^{\text{FORM}}(\text{LiEu}_3\text{Cl}_7, 795 \text{ K}) = -950 \text{ J} \cdot \text{mol}^{-1}$.

4. Conclusions

The electrical conductivity of the low-temperature modification of EuCl_2 (cotunnite-structured, orthorhombic Pnma space group) increases monotonically with increasing

temperature. An abrupt increase of over three orders of magnitude in the ionic conductivity of EuCl_2 at the solid–solid phase transition temperature (from the orthorhombic to the cubic fluorite-type crystal structure) implies the presence of significant disorder in the anionic sublattice within the high-temperature phase. As the temperature increases, the defect concentration in the anionic sublattice increases further, resulting in a decrease in the activation energy and a corresponding increase in electrical conductivity.

Conversely, the conductivity behavior of the low-temperature modification is anomalous: its activation energy increases instead of decreasing with increasing temperature. This effect appears to result from perturbations in the defect concentration.

The LiCl – EuCl_2 phase diagram was found to be of a simple eutectic type featuring one incongruently melting compound, LiEu_3Cl_7 . Its CALPHAD optimization demonstrated that the thermodynamic excess functions of the liquid phase deviate only slightly from ideal behavior. The incongruently melting LiEu_3Cl_7 compound decomposes at 795 K with an enthalpy of $29.7 \text{ kJ}\cdot\text{mol}^{-1}$.

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