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Thermodynamic Investigations of The Erbium Tellurides by EMF Method

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The work presents the results of a thermodynamic study of the Er-Te system by the method of electromotive forces (EMF) in the temperature range of 300 - 450 K. From the EMF measurements of the concentration cells relative to the Er and ErTe electrodes, the partial thermodynamic functions of ErTe and Er in the alloys are determined, based on which the standard thermodynamic formation functions and the standard entropies of the intermediate compounds ErTe_3 , Er_2Te_3 , and ErTe are calculated. A comparative analysis of the obtained data with the literature is carried out.

Keywords: phase diagram, erbium tellurides, EMF method, thermodynamic functions.

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Introduction

The rare-earth chalcogenides are a promising functional class of materials due to their unusual electronic [1-3], thermoelectric [3-5], magnetic [6-8], optical and magneto-optical properties [9-11], and have found their practical application in spintronics and high-performance computing [12, 13].

In order to create and optimize modern methods for controlled synthesis of novel complex materials based on rare earth chalcogenides, it is necessary to investigate phase equilibria and thermodynamic properties of the respective systems [14-17].

Er-Te system is characterized by the formation of ErTe, Er_2Te_3 and ErTe_3 [18, 19]. ErTe and Er_2Te_3 compounds melt congruently at 1773 and 1486 K, while ErTe_3 melts at 957 K with decomposition according to the peritectic reaction $\text{Er}_2\text{Te}_3 + \text{L} \leftrightarrow \text{ErTe}_3$. ErTe and Er_2Te_3 have a cubic, ErTe_3 - rhombic structure.

The thermodynamic properties of erbium tellurides are poorly understood and fragmented. The heat of the formation of Er_2Te_3 compound was determined by the authors of [22] using the calorimetric measurements. In a later work [23], the standard thermodynamic functions of the formation of Er_2Te_3 and $\text{ErTe}_{2.85}$ were calculated by using the electromotive force (EMF) method in the

temperature interval 573-723 K. The estimated data on the standard enthalpy of formation and entropy of ErTe and Er_2Te_3 are given in the handbook [24].

The author [25] obtained the thermodynamic functions of the formation of a number of Ln_2Te_3 (Ln - rare earth elements) compounds. They used a combination of the EMF method with low-temperature heat capacity measurements and the tetrad-effect phenomenon. The results obtained showed that they can be used for estimation of the thermodynamic functions of lanthanide tellurides. In a little later paper [26], the same authors using the above combination method presented thermodynamic data for a number of rare-earth chalcogenides.

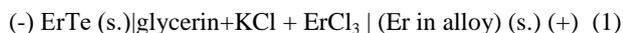
In a recently published work [27], based on the data on the standard heat of formation of Er_2Te_3 [22], its standard thermodynamic functions were calculated, which were then used to calculate the standard thermodynamic functions of ErTe by the EMF method.

This paper presents the results of the study of the Er-Te system in the compositions area > 50 at% Te by the EMF method with glycerol electrolyte

This modification of the EMF method was successfully used for thermodynamic studies of a number of binary and ternary chalcogenide systems [28-34].

I. Experimental

For the thermodynamic investigations of the intermediate phases of the Er-Te system, the following concentration cell was assembled:



and their EMF were measured in the temperature range 300 - 450 K.

In cells of type (1), a saturated glycerol solution of KCl with the addition of 0.1 mass% ErCl_3 was used as an electrolyte. Since the electrolyte should not contain moisture and oxygen, glycerol (analytical grade) was thoroughly dried and degassed by evacuation at ~ 450 K, and anhydrous, chemically pure KCl and ErCl_3 were used. Erbium monotelluride with a slight excess of tellurium ($\text{ErTe}_{1.01}$ composition) was used as the reference electrode (cathode), while equilibrated alloys of the Er-Te system were used as right electrodes (anodes).

The detailed descriptions of the methods for the preparation of the electrodes and electrolyte and assembly of the electrochemical cell were described in [34-37].

ErTe and alloys - the right electrodes were synthesized by the ceramic method by melting of elementary components of high purity in evacuated (10^{-2} Pa) silica ampoules at 1000 K. To prevent the interaction of erbium with the inner walls of quartz, the synthesis was carried out in graphitized ampoules. To achieve a state as close to equilibrium as possible, after melting the intermediate samples were ground into powder, mixed, pressed into cylindrical tablets, and then annealed at 550 K (1000 h), and at 450 K (300 h). The phase composition was controlled by powder X-ray

diffraction analysis (PXRD) (Fig. 1). For example, Fig. 1 shows a powder X-ray diffraction pattern of an alloy from $\text{Er}_2\text{Te}_3 + \text{ErTe}_3$ two-phase area.

In order to prepare the electrodes, the annealed alloys (erbium monotelluride) were ground into a fine powder and were pressed into cylindrical pellets (8 mm in diameter and 3 - 4 mm in thickness) under the pressure of ~ 0.1 GPa in a molybdenum current leads (thickness ~ 0.5 mm).

The EMF measurements were accomplished by the Keithley Model 193 digital voltmeter with an internal resistance of 10^{14} Ohm and accuracy ± 0.1 mV. The measurements were carried out by heating and cooling the cell in steps of no more than 10 K. The temperature of the electrochemical cell was measured with a chromel-alumel thermocouple and a mercury thermometer with an accuracy of 0.5 K. The chromel-alumel thermocouple was pre-calibrated in the temperature range 300 - 510 K based on the melting points of elemental gallium (302.9 K), sulfur (386 K), indium (429.7 K) and tin (505 K).

The first equilibrium EMF values were obtained after holding the cell at ~ 350 K for 40 - 60 h, while the subsequent EMF values were obtained after 3 - 4 h when reaching the desired temperature. The system was considered to be in equilibrium if the EMF measurements were constant or their variations were not significant (< 0.3 mV) regardless of the direction of the temperature change at repeated measurements at a given temperature. The reversibility of the assembled cell and the reproducibility of the results were controlled by checking the mass and PXRD analysis of the selected electrodes before and after the measurements.

In order to control the reproducibility of the cell, for each sample, EMF was measured 2 - 3 times at two constant temperatures within the experiment. Table 1

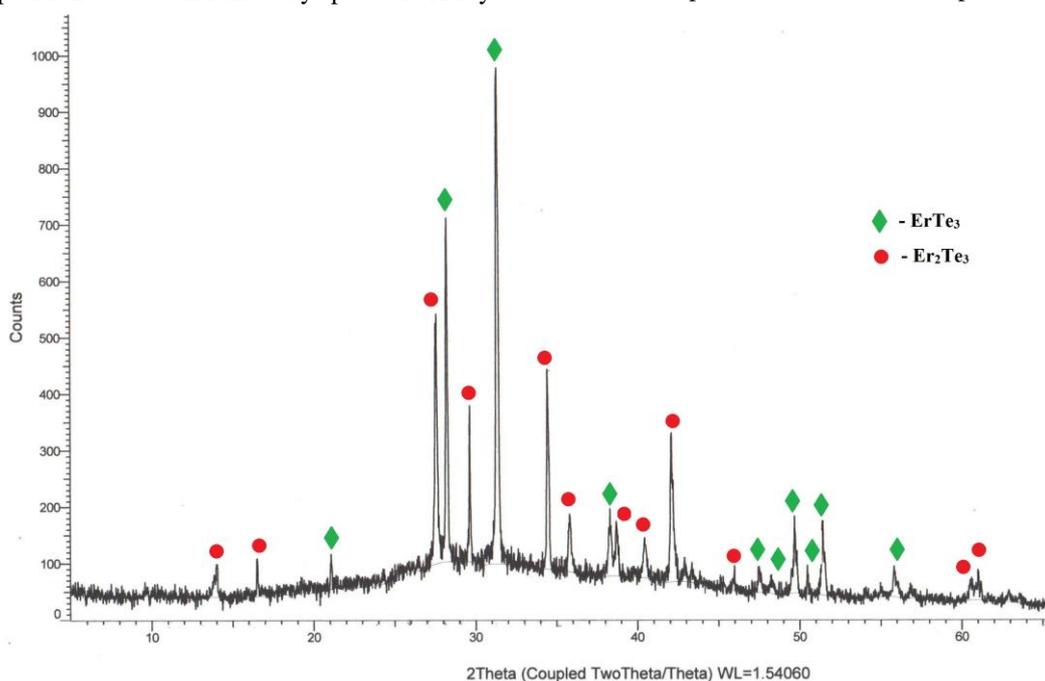


Fig.1. The powder X-ray diffraction pattern of an alloy from $\text{Er}_2\text{Te}_3 + \text{ErTe}_3$ two-phase area.

Table 1

Experimentally obtained data for temperature (T_i) and EMF (E_i) and data associated with the calculation steps for the ErTe_3+Te phase region of the Er-Te system

T_i , K	E_i , mB	$T_i - \bar{T}$	$E_i(T_i - \bar{T})$	$(T_i - \bar{T})^2$	\bar{E}	$E_i - \bar{E}$	$(E_i - \bar{E})^2$
302.3	459.46	-68.32	-31390.31	4667.62	459.52	-0.06	0.00
306.4	459.59	-64.22	-29514.87	4124.21	459.76	-0.17	0.03
311.2	459.53	-59.42	-27305.27	3530.74	460.05	-0.52	0.27
316.3	459.62	-54.32	-24966.56	2950.66	460.36	-0.74	0.54
319.3	461.07	-51.32	-23662.11	2633.74	460.53	0.54	0.29
324.8	460.11	-45.82	-21082.24	2099.47	460.86	-0.75	0.57
329	462.45	-41.62	-19247.17	1732.22	461.11	1.34	1.78
333.1	461.92	-37.52	-17331.24	1407.75	461.36	0.56	0.31
335.9	462.07	-34.72	-16043.07	1205.48	461.53	0.54	0.30
338.2	462.82	-32.42	-15004.62	1051.06	461.66	1.16	1.34
342.9	460.87	-27.72	-12775.32	768.40	461.94	-1.07	1.15
347.1	463.12	-23.52	-10892.58	553.19	462.20	0.92	0.86
352.2	463.41	-18.42	-8536.01	339.30	462.50	0.91	0.83
356.6	463.51	-14.02	-6498.27	196.56	462.76	0.74	0.54
362.1	462.69	-8.52	-3942.12	72.59	463.09	-0.40	0.16
369.4	462.38	-1.22	-564.10	1.49	463.53	-1.15	1.32
375.9	463.19	5.28	2445.64	27.88	463.92	-0.73	0.53
380.2	463.26	9.58	4438.03	91.78	464.17	-0.91	0.83
387.5	464.55	16.88	7841.60	284.93	464.61	-0.06	0.00
392.2	464.96	21.58	10033.84	465.70	464.89	0.07	0.01
398.1	464.35	27.48	12760.34	755.15	465.24	-0.89	0.79
404.6	464.53	33.98	15784.73	1154.64	465.63	-1.10	1.21
410.2	464.77	39.58	18395.60	1566.58	465.96	-1.19	1.43
415.7	466.72	45.08	21039.74	2032.21	466.29	0.43	0.18
420.9	466.99	50.28	23480.26	2528.08	466.60	0.39	0.15
425.1	467.28	54.48	25457.41	2968.07	466.85	0.43	0.18
432.7	468.11	62.08	29060.27	3853.93	467.31	0.80	0.64
437.3	468.22	66.68	31220.91	4446.22	467.58	0.64	0.41
442.8	468.21	72.18	33795.40	5209.95	467.91	0.30	0.09
448.6	468.25	77.98	36514.14	6080.88	468.26	-0.01	0.00
$\bar{T} = 370.62$	$\bar{E} = 463.60$						

presents their averaged values. Experimentally obtained data for temperature (T_i) and EMF (E_i) and data associated with the calculation steps for the ErTe_3+Te phase area are listed in Table 1.

II. Results and discussion

The analysis of EMF measurements showed their reproducibility. For two studied samples from each phase region, the measurement data were practically identical while their temperature dependences were linear (Fig. 2).

This indicates the constancy of the phase compositions of electrodes in the temperature range of EMF measurements and allows us to make thermodynamic calculations [37].

The EMF measurements were processed by the least-squares method [45] on the assumption of a linear relationship of the EMF vs temperature and were expressed by the equation

$$E = a + bT \pm t \left[\frac{S_E^2}{n} + \frac{S_E^2(T - \bar{T})^2}{\sum (T_i - \bar{T})^2} \right]^{1/2}, \quad (2)$$

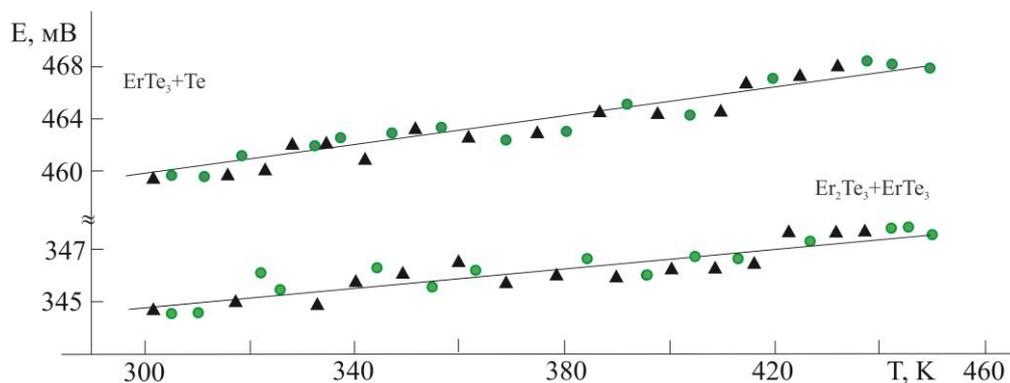


Fig. 2. EMF dependencies upon temperature for alloys of the ErTe_3+Te and $\text{Er}_2\text{Te}_3+\text{ErTe}_3$ phase areas of the Er-Te system. Green circles and black triangles indicate alloys of various compositions in a given phase region.

Table 2

Relations between the EMF and the temperature for type (1) cells in some phase regions of the Er-Te system in the 300–450 K temperature interval

Phase region	$E, \text{mV} = a + bT \pm 2[\delta_E^2/n + \delta_b^2(T - \bar{T})]^{1/2}$
ErTe_3+Te	$441.4 + 0.0597T \pm 2 \left[\frac{0.60}{30} + 1.02 \cdot 10^{-6} (T - 370.62)^2 \right]^{1/2}$
$\text{Er}_2\text{Te}_3+\text{ErTe}_3$	$338.9 + 0.0193T \pm 2 \left[\frac{0.44}{30} + 6.86 \cdot 10^{-6} (T - 377.14)^2 \right]^{1/2}$

Table 3

Relative partial functions of erbium monotelluride in the alloys of the Er-Te system at $T = 298 \text{ K}$

Phase region	$-\Delta\bar{G}_{\text{ErTe}}$	$-\Delta\bar{H}_{\text{ErTe}}$	$\Delta\bar{S}_{\text{ErTe}}$
	$\text{kJ}\cdot\text{mol}^{-1}$		$\text{kJ}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$
ErTe_3+Te	132.94 ± 0.16	127.79 ± 0.69	17.29 ± 1.85
$\text{Er}_2\text{Te}_3+\text{ErTe}_3$	99.78 ± 0.14	98.11 ± 0.58	5.6 ± 1.52

where a and b are empirical constants; n is the number of pairs of T and E values; S_E is the error variance of the EMF measurements; T is the average of the absolute temperature; t is the Student's test. At the confidence level of 95% and $n=30$, the Student's test is $t \leq 2$.

The obtained equations of the type (2) are listed in Table 2.

From obtained relations by using the thermodynamic expressions [36, 37]:

$$\Delta\bar{G}_{\text{ErTe}} = -zFE \quad (3)$$

$$\Delta\bar{S}_{\text{ErTe}} = zF \left(\frac{\partial E}{\partial T} \right)_P = zFb, \quad (4)$$

$$\Delta\bar{H}_{\text{ErTe}} = -zF \left[E - T \left(\frac{\partial E}{\partial T} \right)_P \right] = -zFa, \quad (5)$$

the partial molar Gibbs free energy, enthalpy, and entropy of erbium monotelluride in alloys were calculated (Table 3). Here, z is the number of electrons, participating in the electrochemical reaction, in our case $z = 3$; F is the Faraday constant ($F = 96485.34 \text{ C/mol}$).

The data presented in Table 3 are in accordance with

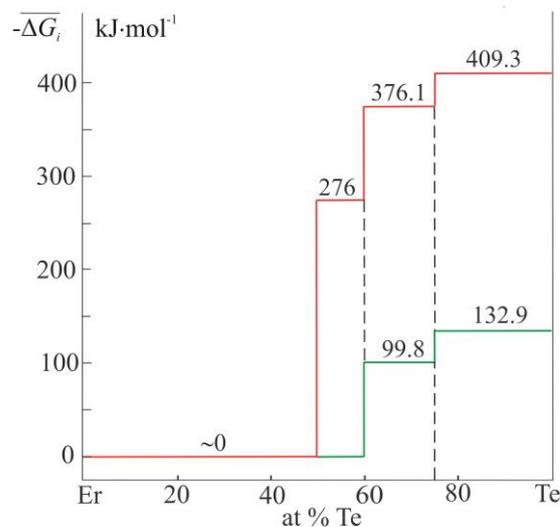


Fig. 3. The dependencies of the partial free Gibbs energies of ErTe (green line) and Er (red line) on the composition in Er-Te alloys at 298 K.

the phase diagram of the Er-Te system [18]. As can be seen from Fig. 3, at a constant temperature, the partial Gibbs free energy of ErTe is constant within the two-phase region and changes stepwise upon transition from one region to another.

These values are the differences between the corresponding partial molar functions of erbium for the right and left electrodes of cells (1). For example,

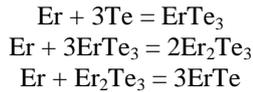
$$\Delta \bar{Z}_{\text{ErTe}} = \Delta \bar{Z}_{\text{Er}} (\text{Er in Te rich alloy}) - \Delta \bar{Z}_{\text{Er}} (\text{ErTe})$$

here $Z \equiv G$ or H . Therefore, the partial thermodynamic functions of erbium in studied phase areas, can be calculated using the relation:

$$\Delta \bar{Z} (\text{Er in alloys}) = \Delta \bar{Z}_{\text{Er}} (\text{ErTe}) + \Delta \bar{Z}_{\text{ErTe}} \quad (6)$$

In order to calculate the partial thermodynamic functions of erbium in alloys ErTe_3+Te and $\text{Er}_2\text{Te}_3+\text{ErTe}_3$, we summarized the data of Table 3 with the corresponding partial molar functions of Er in ErTe [27] (Table 4). The results are shown in Table 4.

According to the phase diagram [18], the partial molar thermodynamic values of erbium in compounds are the thermodynamic characteristics of the following potential formation reactions (the substances in the crystalline state):



Thus, the standard thermodynamic functions of formation of erbium tellurides were calculated by using the relations (7)-(9):

$$\Delta_f Z^0 (\text{ErTe}_3) = \Delta \bar{Z}_{\text{Er}} \quad (7)$$

$$2\Delta_f Z^0 (\text{Er}_2\text{Te}_3) = \Delta \bar{Z}_{\text{Er}} + \Delta Z^0 (\text{ErTe}_3) \quad (8)$$

$$3\Delta_f Z^0 (\text{ErTe}) = \Delta \bar{Z}_{\text{Er}} + \Delta Z^0 (\text{Er}_2\text{Te}_3) \quad (9)$$

while the standard entropies were calculated as:

$$S^0 (\text{ErTe}_3) = [\Delta \bar{S}_{\text{Er}} + S^0 (\text{Er})] + 3S^0 (\text{Te}) \quad (10)$$

$$2S^0 (\text{Er}_2\text{Te}_3) = [\Delta \bar{S}_{\text{Er}} + S^0 (\text{Er})] + 3S^0 (\text{Te}) \quad (11)$$

$$3S^0 (\text{ErTe}) = [\Delta \bar{S}_{\text{Er}} + S^0 (\text{Er})] + S^0 (\text{Te}) \quad (12)$$

For the thermodynamic calculations, in addition to own experimental data (Table 3), we used the literature data [21] on the standard entropies of elementary erbium ($73.14 \pm 0.63 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$) and tellurium ($49.5 \pm 0.2 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$). These values are close to the values given in other modern handbooks [20, 39, 40]. The obtained sets of standard integral thermodynamic functions of erbium tellurides are summarized in Table 5. In all cases, the estimated standard uncertainties were calculated by accumulating the errors.

A comparison of our results for Er_2Te_3 with the data of [23], carried out by the EMF method in the temperature range 580 - 720 K, shows their satisfactory compliance. If the results of [23] for $\text{ErTe}_{2.85}$ are converted to the formula unit ErTe_3 , then we obtain the values ($-\Delta_f G^0(298\text{K}) = 414.3$, $-\Delta_f H^0(298\text{K}) = 412.3 \text{ kJ}\cdot\text{mol}^{-1}$) closer to our data. The calorimetric data on the standard heat of formation of Er_2Te_3 [22] are significantly higher than all other available data, including the estimated values given in the handbook [24]. For Er_2Te_3 , our data practically coincide with the calculated data [25]. Despite the same values of the partial molar values of erbium, some discrepancies of our results for ErTe and data of [27] should be noted. This is because the author [27], for calculations of the integral thermodynamic functions for ErTe, used the results of [22] for the Er_2Te_3 compound, while we used our data (Table 5).

Table 4

Relative partial functions of erbium in the alloys of the Er-Te system at $T = 298 \text{ K}$

Phase region	$-\Delta \bar{G}_{\text{Er}}$	$-\Delta \bar{H}_{\text{Er}}$	$\Delta \bar{S}_{\text{Er}}$
	$\text{kJ}\cdot\text{mol}^{-1}$		$\text{kJ}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$
ErTe ₃ +Te	409.3±0.28	411.38±1.29	-6.97±2.51
Er ₂ Te ₃ +ErTe ₃	376.14±0.26	381.7±1.18	-18.65±2.18
ErTe ₃ +ErTe [27]	276.36±0.12	283.59±0.60	-24.25±0.66

Table 5

Standard integral thermodynamic functions of erbium tellurides at temperature $T = 298 \text{ K}$

Compound	$-\Delta_f G^0(298\text{K})$	$-\Delta_f H^0(298\text{K})$	S_{298}^0
	$\text{kJ}\cdot\text{mol}^{-1}$		$\text{kJ}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$
ErTe ₃	409.3±0.3	411.4±1.3	214.7±3.7
ErTe _{2.85}	392.4±0.5 [23]	391.7±4.8 [23]	-
Er ₂ Te ₃	784.4±0.5	793.1±2.5	269.2±6.5
	797.2±1 [23]	822.2±16.7 [23]	-
	782.5±10 [25]	795.5±10 [25]	256.1±10 [25, 26]
		856±25 [22]	-
		816±120 [24]	262 [24]
ErTe	353.6±0.3	358.9±0.6	106.0±2.6
	357±0.5 [27]	379.6± 6.0 [27]	86.7±9.3 [27]
		314±60 [24]	97±12 [24]

Conclusions

Based on the EMF measurements in the temperature range 300 – 450 K, we obtained new data set on the partial molar functions of erbium and erbium telluride in Er-Te system. By combining these data, the standard thermodynamic functions of formation and the standard entropies of ErTe, Er₂Te₃, and ErTe₃ compounds are calculated. A comparative analysis of obtained mutually consistent data sets on standard integral thermodynamic functions with the available mismatch literary data is done. This is the first mutually agreed dataset for each of the compounds studied and for the Er-Te system as a whole.

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Дослідження термодинаміки телуридів ербію методом електрорушійних сил (ЕРС)

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У роботі подано результати термодинамічного дослідження системи Ер-Те методом електрорушійних сил (ЕРС) в інтервалі температур 300 - 450 К. З ЕРС-вимірювань концентрації комірок відносно електродів Ер та ЕрТе, визначено часткові термодинамічні функції ЕрТе та Ер у сплавах, на основі яких обчислюються стандартні термодинамічні функції утворення та стандартні ентропії проміжних сполук ЕрТе₃, Ер₂Те₃ та ЕрТе. Проведено порівняльний аналіз отриманих даних із літературними.

Ключові слова: фазова діаграма, телуриди ербію, метод ЕРС, термодинамічні функції.