

## SYNTHESIS AND PHYSICOCHEMICAL PROPERTIES OF NEW TRIPLE LUTETIUM TELLURITES

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### ABSTRACT

New triple lutetium tellurites  $\text{LuMeFeTeO}_6$  (Me - Sr, Ba) are synthesized using ceramic technology from oxides  $\text{Lu}_2\text{O}_3$ ,  $\text{TeO}_2$ ,  $\text{Fe}_2\text{O}_3$ , carbonates  $\text{SrCO}_3$  and  $\text{BaCO}_3$ . Using X-ray phase analysis, we confirmed the individuality, and determined the types of syngony, unit cell parameters, X-ray and pycnometric densities of compounds. It has been confirmed that tellurites crystallize in the tetragonal system with a distorted perovskite structural type. Using dynamic calorimetry in the temperature range 298.15 - 673 K, we studied the isobaric heat capacities of tellurites and derived equations for the  $C_p(T)$  dependence. The standard heat capacity of new tellurites has been determined, establishing a fundamental constant. We have calculated temperature dependences of the thermodynamic functions  $S^0(T)$ ,  $H^0(T)$  -  $H^0(298.15)$ ,  $\Phi^{\text{ex}}(T)$ . The study results are significant for inorganic and physical chemistry of oxide materials, as well as inorganic materials science. They can be utilized for inclusion in reference books and thermodynamic data banks.

**Keywords:** lutetium, ternary tellurites, X-ray phase analysis, heat capacity, thermodynamic functions.

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### INTRODUCTION

The creation of new materials with specified physicochemical properties and structure is a fundamental problem of modern materials science. Therefore, establishing the relationship between the composition, synthesis conditions, and, consequently, the structure, and then the properties of inorganic compounds is one of the main tasks. Mixed oxides with a perovskite-like structure are gaining interest due to their potential applications such as heterogeneous catalysts for oxidation reactions, materials for oxygen-selective membranes, electrodes for high-temperature electrochemical devices including solid oxide fuel cells, as well as their semiconductor and ferroelectric properties [1 - 4]. Among the various systems, oxide systems involving rare earth elements (REE) and tellurium are prominent [5, 6]. As a family of second-order Jahn-Teller "SOJT" cations, the lone pair cation ( $\text{Te}^{4+}$ ) is a key element that can induce macroscopic non-centrosymmetric (NCS) structures [7]. Materials chemists are interested in crystalline materials with NCS structure due to their technologically

important characteristics, such as nonlinear optical (NLO) properties, ferroelectricity, pyroelectricity and piezoelectricity [8]. To date, simple tellurites have already been quite well studied and characterized in detail. Thus, the traditional approach to developing new materials - creating compounds that are more complex in composition is implemented in the study of binary and ternary tellurite systems [9, 10]. In this regard, this work aims to synthesize and study the physicochemical properties of new ternary lutetium tellurites.

### EXPERIMENTAL

The synthesis of compounds is carried out using ceramic technology, taking into account stoichiometric amounts of  $\text{Lu}_2\text{O}_3$  (extra pure grade),  $\text{TeO}_2$  (chemically pure grade), carbonates  $\text{SrCO}_3$  and  $\text{BaCO}_3$  (pure for analysis) in terms of the final formula  $\text{LuMeFeTeO}_6$  (Me - Sr, Ba) in the range temperatures 1073 - 1473 K. X-ray diffraction patterns of the synthesized compounds are obtained using an Empyrean powder diffractometer from PANalytical. The synthesis technique, X-ray phase

analysis of the synthesized compounds, interpretation of the obtained X - ray patterns and identification of phases are like the previous work [9].

The heat capacity of the synthesized tellurites was analyzed by using dynamic calorimetry with a serial device IT - S - 400 within the temperature range of 298.15 - 673 K [11]. The operating principle of the calorimeter is based on the comparative method of a dynamic c-calorimeter with a heat meter.

The experiments are carried out in the mode of monotonic, close to linear heating of the sample with an average speed of  $\sim 0.1 \text{ K s}^{-1}$ . In an experiment, we are determining the temperature dependence of the parameter under study. The device circuit allows temperature measurements to be taken at fixed points every 25 K. This is achieved using a direct current potentiometer and switch built into the device. Volume range is not less than  $1 \times 10^7 \text{ J K}^{-1} \text{ m}^{-3}$ . The time required to carry out measurements over the entire temperature range and process experimental data is no more than 2.5 h. The errors in measuring heat capacity on the IT - S - 400 device do not exceed  $\pm 10 \%$  [11, 12].

The calibration of the device, determination of the

heat capacity of the standard copper sample, calculations to determine the standard deviation ( $\delta$ ,  $\text{J g}^{-1} \text{ K}^{-1}$ ) for the averaged values of specific heat capacities, as well as the random component of the error ( $\Delta$ ,  $\text{J mol}^{-1} \text{ K}^{-1}$ ) for the averaged values of molar heat capacity were carried out according to the method given earlier in the work [13].

Satisfactory agreement with the described from Robie et al. [14], as well as with new literary data reported by Bodryakov et al. [15], the result of determining the standard heat capacity of  $\alpha - \text{Al}_2\text{O}_3$  ( $C_p^0$ , 298.15 = 76  $\text{J mol}^{-1} \text{ K}^{-1}$ ), indicates the reliability of the obtained values of heat capacity of the synthesized new tellurites.

## RESULTS AND DISCUSSION

The powder diffraction pattern is an individual characteristic of a crystalline substance. Diffraction patterns of the synthesized ternary lutetium tellurites are presented in Fig. 1.

The results of indexing the X - ray diffraction patterns of the synthesized ternary lutetium tellurites are given in Table 1.

The reliability and correctness of the indexing results

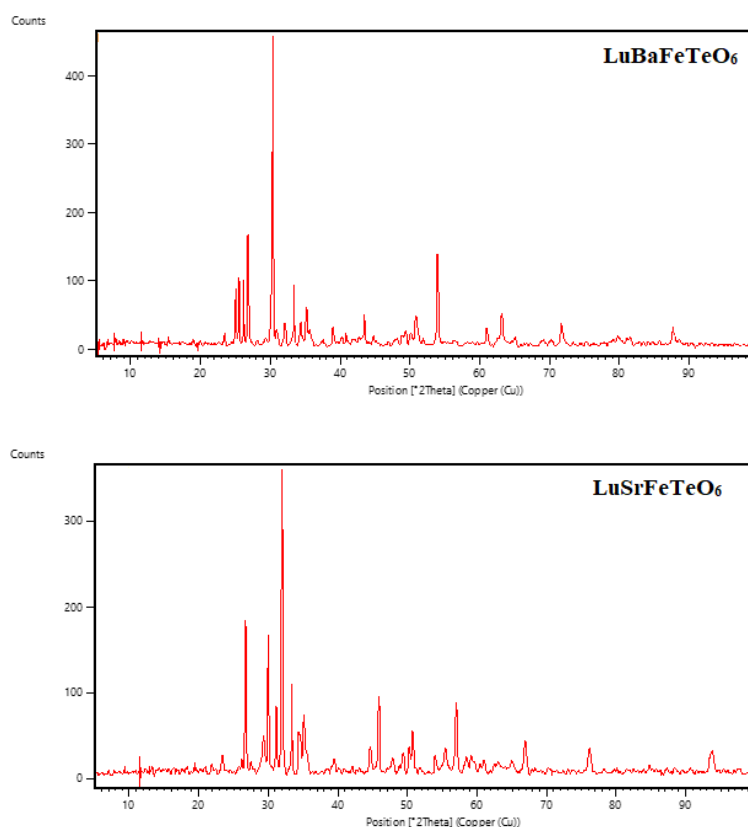


Fig. 1. X - ray diffraction patterns of tellurites  $\text{LuMeFeTeO}_6$  (Me - Sr, Ba).

Table 1. Results of indexing X - ray diffraction patterns of tellurites  $\text{LuMeFeTeO}_6$  (Me - Sr, Ba).

h	k	l	2Th. (c) [°]	2Th. (o) [°]	d-sp. (c) [Å]	d-sp. (o) [Å]	I/I <sub>0</sub>
LuSrFeTeO <sub>6</sub>							
3	1	0	23.3811	23.3823	3.801581	3.801394	5.98
3	2	0	26.7153	26.7486	3.334207	3.330134	47.83
1	1	2	29.2515	29.3112	3.050635	3.044558	12.48
3	2	1	30.0627	29.9975	2.970140	2.976454	43.38
2	0	2	31.1230	31.1624	2.871334	2.867790	21.99
2	1	2	32.0216	31.9983	2.792778	2.794754	100.00
4	2	0	33.3038	33.3766	2.688124	2.682430	28.95
3	3	1	34.4702	34.4462	2.599783	2.601545	13.31
3	0	2	35.4124	35.1059	2.532747	2.554154	19.40
4	3	1	39.9201	39.4577	2.256527	2.281899	3.46
4	4	1	44.8090	44.5595	2.021023	2.031761	7.18
6	1	0	45.8790	45.8009	1.976348	1.979534	22.06
6	2	0	47.8138	47.8232	1.900790	1.900441	4.53
5	2	2	49.4000	49.3597	1.843405	1.844819	6.36
3	2	3	49.9625	50.2171	1.823964	1.815312	8.27
5	4	1	50.5387	50.7149	1.804510	1.798654	13.55
7	1	0	53.8836	53.9342	1.700119	1.698645	5.92
7	0	1	55.2592	55.4330	1.661009	1.656211	8.16
6	4	1	56.9596	56.9645	1.615395	1.615266	23.03
2	0	4	58.4812	58.4622	1.576936	1.577404	2.21
2	1	4	59.0315	59.0574	1.563541	1.562919	0.86
7	0	2	60.8863	61.0040	1.520278	1.517627	3.15
6	0	3	62.9720	62.7818	1.474849	1.478858	0.43
6	2	3	65.0656	65.0398	1.432362	1.432869	2.68
4	2	4	66.9587	66.9541	1.396389	1.396474	9.79
8	5	1	76.0304	76.0467	1.250748	1.250520	7.66
2	2	6	93.7578	93.7699	1.055334	1.055229	6.83
LuBaFeTeO <sub>6</sub>							
0	0	3	25.3432	25.1958	3.511536	3.531743	5.16
2	2	0	26.0411	26.1758	3.418986	3.401697	20.40
2	1	2	26.6483	26.7317	3.342442	3.332200	32.81
3	1	1	30.4122	30.3287	2.936801	2.944695	100.00
3	2	0	33.3811	33.3698	2.682073	2.682958	12.79
3	2	1	34.4788	34.3236	2.599158	2.610556	6.53
1	0	4	35.2921	35.1356	2.541100	2.552058	12.48
2	2	4	43.3323	43.4671	2.086415	2.080254	10.19
2	2	5	50.8652	50.8719	1.793691	1.793469	8.98
5	2	2	53.8987	53.9179	1.699679	1.699118	26.80
1	1	7	63.2147	63.1239	1.469769	1.471664	8.39
0	0	8	71.6012	71.6718	1.316826	1.315703	6.69

2Th. (c) [°] - calculated double Bragg reflection angle in degrees; 2Th. (o) [°] - experimental double Bragg reflection angle in degrees; d-sp. (c) [Å] - calculated interplanar distance; d-sp. (o) [Å] - experimental interplanar distance; [Å] - Angstrom.

are confirmed by the satisfactory agreement between the experimental and calculated values of 2Th. and d - sp (Table 1) and the values of X - ray and pycnometric densities (Table 2).

The theoretical cell volumes of the synthesized tellurites are determined using the data on the cell volumes included in their composition, following a specific scheme:

$$V_{\text{unit cell}}^{\circ} \text{LuMeFeTeO}_6 = 0.5 V_{\text{unit cell}}^{\circ} \text{Lu}_2\text{O}_3 + 0.5 V_{\text{unit cell}}^{\circ} \text{Fe}_2\text{O}_3 + V_{\text{unit cell}}^{\circ} \text{TeO}_2 + V_{\text{unit cell}}^{\circ} \text{MeO} \quad (\text{Me} - \text{Sr, Ba}) \quad (1)$$

The accurate match of the calculated values for the volumes of basic units of tellurites with the sum of the volumes of basic units of the initial oxides of lutetium (+ 3), iron (+ 3), tellurium (+ 4), and strontium (barium) oxides, obtained from the Crystallography Open Database, along with the calculated cell volumes of compounds from X-ray data, further supports the accuracy of the X - ray patterns of the new tellurites [16]. So, for  $\text{LuSrFeTeO}_6$   $V_{\text{unit cell}}^0 = 1029.03 \text{ \AA}^3$  (from the sum of  $V_{\text{unit cell}}^0$  oxides) and  $944.19 \pm 0.02 \text{ \AA}^3$  (from indexing data),  $\text{LuBaFeTeO}_6$   $V_{\text{unit cell}}^0 = 1057.51 \text{ \AA}^3$  (from the sum of  $V_{\text{unit cell}}^0$  oxides) and  $1048.40 \pm 0.02 \text{ \AA}^3$  (from indexing data).

We observe that as the ionic radii increase from Sr to Ba, the unit cell volumes of the synthesized tellurites also increase. Based on the indexing of X - ray diffraction patterns, it was found that  $\text{LuSrFeTeO}_6$  and  $\text{LuBaFeTeO}_6$  crystallize in a tetragonal system with lattice parameters as shown in Table 2.

As in the previous work [9], using the X'Pert HighScore Plus software package of the PANalytical diffractometer, the phase composition of new tellurites that crystallize in the distorted perovskite structural type was confirmed. When assigning compounds to the perovskite structure or to its distorted version, the "tolerance factor"  $t$  is considered [17]. Like presented by Rustembekov et al., the "tolerance factor"  $t$  for the synthesized lutetium tellurites was calculated [9]. The

ionic radii of  $\text{Lu}^{3+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Te}^{4+}$ ,  $\text{O}^{2-}$  according to the classification of Shannon and Prewitt are borrowed [18]. As is known from Welles, for compounds with a perovskite-type structure, the "tolerance factor"  $t$  lies approximately in the range of 0.8 - 1.0 [17]. In our case,  $t$  for  $\text{LuSrFeTeO}_6$  and  $\text{LuBaFeTeO}_6$  are 0.88 and 0.94, respectively, which to a certain extent can be attributed to the perovskite structural type, confirming that these compounds can have semiconductor and ferroelectric properties.

Table 3 and Fig. 2 show the results of a calorimetric study of the heat capacities of synthesized ternary lutetium tellurites.

Equations for the temperature dependence of the heat capacities of compounds by mathematical processing of experimental data were derived. The equation coefficients are given in Table 4.

Since the technical capabilities of the calorimeter do not allow calculating the standard entropies of compounds from experimental data on heat capacities, they are estimated using the ion increment method according to the scheme [19]:

$$S^0(298.15) \text{ LuMeFeTeO}_6 = S'(298.15) \text{ Lu}^{3+} + S'(298.15) \text{ Me}^{2+} + S'(298.15) \text{ Fe}^{3+} + S'(298.15) \text{ Te}^{4+} + 6S'(298.15) \text{ O}^{2-}, \quad (2)$$

where  $S'(298.15)$  - entropy increment of the ion, at 298.15 K;  $\text{Me}^{2+}$  ( $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$ ).

The values of  $S'(298.15) \text{ Lu}^{3+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Te}^{4+}$ ,  $\text{O}^{2-}$  required for the calculation are borrowed from [19].

Based on the experimental data on  $C_p^0(T)$  and the calculated values of  $S^0(298.15)$ , the temperature dependences of the thermodynamic functions were calculated:  $C_p^0(T)$ ,  $S^0(T)$ ,  $H^0(T) - H^0(298.15)$  and  $\Phi^{\text{xx}}(T)$  (Table 5). The thermodynamic functions  $H^0(T) - H^0(298.15)$ ,  $S^0(T)$  and  $\Phi^{\text{xx}}(T)$  were calculated using the following formulas [20]:

$$H^0(T) - H^0(298.15) = \int_{298.15}^T C_p^0 dT, \quad (3)$$

Table 2. Types of syngony and unit cell parameters of triple lutetium tellurites.

Compound	Type of syngony	Lattice parameters, Å		$V_{\text{unit cell}}^0, \text{ \AA}^3$	Z	Density, g cm <sup>-3</sup>	
		a	c			radiog.	pycnom.
$\text{LuSrFeTeO}_6$	tetragonal	$12.021 \pm 0.02$	$6.534 \pm 0.02$	$944.19 \pm 0.02$	3	2.86	$2.84 \pm 0.02$
$\text{LuBaFeTeO}_6$	tetragonal	$10.011 \pm 0.03$	$10.461 \pm 0.03$	$1048.40 \pm 0.02$	4	3.99	$3.97 \pm 0.02$

Table 3. Experimental values of the heat capacities of triple lutetium tellurites.

T, K	$C_p \pm \delta,$ $J g^{-1} K^{-1}$	$C_p^0 \pm \Delta,$ $J mol^{-1} K^{-1}$	T, K	$C_p \pm \delta,$ $J g^{-1} K^{-1}$	$C_p^0 \pm \Delta,$ $J mol^{-1} K^{-1}$
LuSrFeTeO <sub>6</sub>					
298.15	$0.3525 \pm 0.0360$	$191 \pm 10$	498	$0.5107 \pm 0.0273$	$277 \pm 10$
323	$0.3642 \pm 0.0061$	$197 \pm 11$	523	$0.5600 \pm 0.0231$	$303 \pm 11$
348	$0.3997 \pm 0.0147$	$217 \pm 11$	548	$0.5881 \pm 0.0262$	$319 \pm 13$
373	$0.4140 \pm 0.0155$	$224 \pm 9$	573	$0.6107 \pm 0.0259$	$331 \pm 15$
398	$0.4470 \pm 0.0276$	$242 \pm 8$	598	$0.6415 \pm 0.0213$	$348 \pm 13$
423	$0.4545 \pm 0.0285$	$246 \pm 8$	623	$0.6813 \pm 0.0214$	$369 \pm 11$
448	$0.4668 \pm 0.0322$	$253 \pm 8$	648	$0.7066 \pm 0.0203$	$382 \pm 8$
473	$0.4785 \pm 0.0296$	$259 \pm 10$	673	$0.7224 \pm 0.0229$	$392 \pm 11$
LuBaFeTeO <sub>6</sub>					
298.15	$0.3229 \pm 0.0360$	$193 \pm 10$	498	$0.5714 \pm 0.0064$	$338 \pm 10$
323	$0.3418 \pm 0.0061$	$202 \pm 11$	523	$0.6391 \pm 0.0065$	$378 \pm 11$
348	$0.3981 \pm 0.0058$	$235 \pm 11$	548	$0.6739 \pm 0.0180$	$399 \pm 13$
373	$0.4125 \pm 0.0085$	$244 \pm 9$	573	$0.7087 \pm 0.0220$	$419 \pm 15$
398	$0.4601 \pm 0.0081$	$272 \pm 8$	598	$0.7520 \pm 0.0216$	$445 \pm 13$
423	$0.4849 \pm 0.0076$	$287 \pm 8$	623	$0.8042 \pm 0.0247$	$476 \pm 11$
448	$0.5147 \pm 0.0075$	$305 \pm 8$	648	$0.8345 \pm 0.0220$	$494 \pm 8$
473	$0.5295 \pm 0.0070$	$313 \pm 10$	673	$0.8659 \pm 0.0169$	$512 \pm 11$

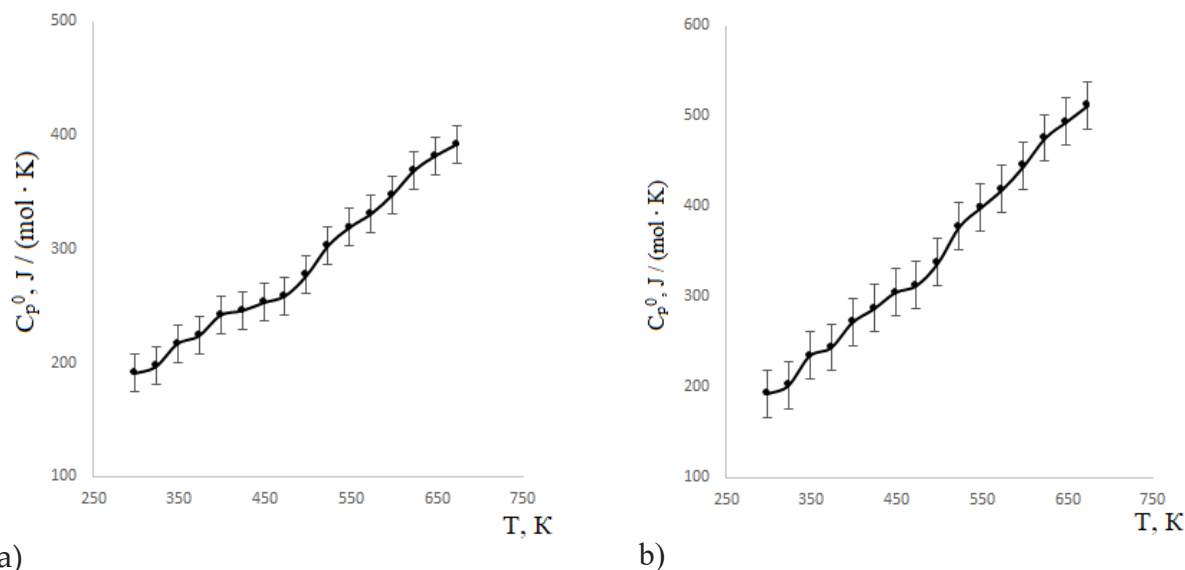
Fig. 2. Dependences of the heat capacities of tellurites on temperature: (a) LuSrFeTeO<sub>6</sub>, (b) LuBaFeTeO<sub>6</sub>.

Table 4. Equations for the temperature dependence of the heat capacities of triple lutetium tellurites.

Compound	Equation coefficients $C_p^0 = a + bT + cT^{-2}, J mol^{-1} K^{-1}$			$\Delta T, K$
	a	$b \times 10^{-3}$	$c \times 10^5$	
LuSrFeTeO <sub>6</sub>	$-(127.59 \pm 3.05)$	$743.21 \pm 2.24$	$86.03 \pm 2.21$	298 - 673
LuBaFeTeO <sub>6</sub>	$-(251.99 \pm 3.11)$	$1454.2 \pm 2.31$	$10.33 \pm 2.27$	298 - 673

$$S^0(T) = S^0(298.15) + \int_{298.15}^T \frac{C_p}{T} dT, \quad (4)$$

$$\Phi^{xx}(T) = S^0(T) - [H^0(T) - H^0(298.15)]/T \quad (5)$$

For all values of heat capacity and enthalpy across the entire temperature range, the average random components of the error are estimated. Additionally, the error estimate includes the accuracy of the entropy calculation ( $\pm 3\%$ ) for the values of entropy and reduced thermodynamic potential.

Table 5. Thermodynamic functions of tellurites in the range 298.15 - 673 K.

T, K	$C_p^0(T) \pm \Delta,$ J mol <sup>-1</sup> K <sup>-1</sup>	$S^0(T) \pm \Delta,$ J mol <sup>-1</sup> K <sup>-1</sup>	$H^0(T) - H^0(298.15) \pm \Delta,$ J mol <sup>-1</sup>	$\Phi^{xx}(T) \pm \Delta,$ J mol <sup>-1</sup> K <sup>-1</sup>
LuSrFeTeO <sub>6</sub>				
298.15	191 ± 9	220 ± 8	-	220 ± 16
323	195 ± 10	235 ± 18	4788 ± 223	221 ± 16
348	202 ± 11	250 ± 19	9743 ± 453	222 ± 16
373	211 ± 11	265 ± 20	14903 ± 693	225 ± 16
398	222 ± 10	279 ± 21	20317 ± 945	228 ± 17
423	235 ± 12	292 ± 22	26025 ± 1210	231 ± 17
448	248 ± 11	306 ± 24	32054 ± 1491	235 ± 18
473	262 ± 13	320 ± 25	38429 ± 1787	239 ± 19
498	277 ± 13	334 ± 26	45166 ± 2101	243 ± 19
523	292 ± 14	348 ± 27	52283 ± 2432	248 ± 19
548	308 ± 14	362 ± 28	59791 ± 2781	253 ± 19
573	324 ± 14	376 ± 29	67699 ± 3149	258 ± 20
598	341 ± 15	390 ± 30	76017 ± 3536	263 ± 20
623	358 ± 15	405 ± 30	84752 ± 3942	269 ± 21
648	375 ± 16	419 ± 31	93909 ± 4368	274 ± 21
673	392 ± 17	434 ± 31	103493 ± 4814	280 ± 22
LuBaFeTeO <sub>6</sub>				
298.15	193 ± 9	230 ± 9	-	230 ± 17
323	233 ± 9	247 ± 18	5311 ± 247	231 ± 17
348	266 ± 10	266 ± 18	11565 ± 538	232 ± 17
373	294 ± 11	285 ± 18	18578 ± 864	235 ± 18
398	318 ± 11	305 ± 19	26235 ± 1220	239 ± 18
423	339 ± 11	325 ± 19	34447 ± 1602	244 ± 19
448	357 ± 12	345 ± 21	43144 ± 2007	249 ± 20
473	373 ± 12	365 ± 21	52274 ± 2431	254 ± 20
498	388 ± 13	384 ± 22	61792 ± 2874	260 ± 21
523	402 ± 14	404 ± 22	71663 ± 3333	267 ± 21
548	414 ± 14	424 ± 22	81859 ± 3807	273 ± 21
573	426 ± 15	442 ± 23	92357 ± 4296	280 ± 21
598	437 ± 15	460 ± 24	103136 ± 4797	287 ± 22
623	447 ± 16	478 ± 25	114180 ± 5310	295 ± 23
648	457 ± 17	496 ± 25	125475 ± 5836	302 ± 24
673	466 ± 17	513 ± 26	137009 ± 6372	310 ± 24

## CONCLUSIONS

New ternary lutetium tellurites of the composition  $\text{LuMeFeTeO}_6$  (Me - Sr, Ba) have been synthesized for the first time. X - ray characteristics (types of syngony, unit cell parameters, X - ray and pycnometric densities) are obtained using an Empyrean powder diffractometer and specialized programs Data Collector version 7.7h and X'Pert HighScore Plus using the Crystallography Open Database and PDF - 2 databases. It has been established that tellurites crystallize in the tetragonal system in the distorted perovskite structural type. Therefore, it can be assumed that these compounds may have semiconductor and ferroelectric properties.

For the first time using the method of dynamic calorimetry in the temperature range 298.15 - 673 K, the isobaric heat capacities of tellurites are studied, based on which the equations for the dependence  $C_p(T)$  are derived. A fundamental constant is determined - the standard heat capacity of new tellurites. The temperature dependences of the thermodynamic functions  $S^0(T)$ ,  $H^0(T) - H^0(298.15)$ ,  $\Phi^{xx}(T)$  are calculated.

The study results are important for inorganic and physical chemistry of oxide materials, as well as inorganic materials science. They can also be utilized for inclusion in reference books and thermodynamic data banks.

The data obtained serve as the basis for further research on the electrophysics of the synthesized new tellurites.

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