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A.A. Toibek[1], K.T. Rustembekov[1*], D.A. Kaikenov[1], M. Stoev[2]

[1]Karagandy University of the name of academician E.A. Buketov, Kazakhstan;
[2]South-West University "Neofit Rilski", Blagoevgrad, Bulgaria
*(*Corresponding author's e-mail: rustembekov_kt@mail.ru)*

Synthesis and properties of double gadolinium tellurites

For the first time, double gadolinium tellurites of the composition $GdM^{II}TeO_{4.5}$ (M^{II} — Sr, Ba) were synthesized by the solid-phase method. The solid-phase synthesis of samples was carried out from decrepitated gadolinium (III) and tellurium (IV) oxides, strontium, and barium carbonates according to the standard ceramic technology. The synthesis was carried out in the temperature range of 800-1100 °C. The samples obtained were confirmed by X-ray phase analysis. X-ray phase analysis was carried out on an Empyrean instrument in the XRDML Pananalitical format. The intensity of the diffraction maxima was estimated on a 100-point scale. X-ray diffraction patterns indexing of the powder of gadolinium tellurites — alkaline earth metals studied were carried out by the homology method. The reliability and correctness of the results of indexing the X-ray diffraction patterns are confirmed by the good agreement between the experimental and calculated values of the interplanar distances (*d*) and the agreement between the values of the X-ray and pycnometric densities. It was found that compounds $GdSrTeO_{4.5}$ and $GdBaTeO_{4.5}$ crystallize in the monoclinic system and have the unit cell parameters, namely $GdSrTeO_{4.5}$ — a = 12.7610, b = 10.4289, c = 8.6235 Å, V° = 1141.83 Å^3, β = 95.77°, Z = 5, $\rho_{rent.}$ = 3.22, $\rho_{pikn.}$ = (3.10±0.09) g/cm^3; $GdBaTeO_{4.5}$ — a = 15.7272, b = 15.8351, c = 7.1393 Å, V° = 1769.72 Å^3, β = 95.53°, Z = 8, ρ_{rent} = 3.71, ρ_{pick} = (3.61±0.10) g/cm^3. Using the Landiya method, the standard heat capacities of the compounds were estimated from the calculated values of the standard entropies, and the temperature dependences of the heat capacities of the gadolinium tellurites synthesized were determined in the temperature range of 298–850 K.

Keywords: double gadolinium tellurites, X-ray phase analysis, crystal system, lattice parameters, heat capacity.

Introduction

It is known that tellurium compounds with metals have semiconducting properties and superconductivity, such as transition and non-transition metals tellurites. On the other hand, chalcogen compounds containing three or more elements tend to polymerize, especially if in addition to tellurium there is an oxygen atom in the composition. These kinds of compounds are used in the non-organic synthesis of composite materials with organic substances.

Tellurium derivatives are characterized by high chemical activity, which determines the prospects of synthetic transformations aimed at obtaining new semiconductor, ferroelectric, and radioluminescent materials of a wide range of implementations. Recently, the attention of scientists has been especially attracted by compounds based on rare earth, alkaline earth oxides, and transition metals in connection with their properties in microelectronics [1]. The investigation of complex oxides of 3d- and 4f- elements with a perovskite structure has great importance for non-organic materials science [2, 3]. In this regard, the purpose of this work was to synthesize and study the properties of new phases — double tellurites of gadolinium with composition $GdM^{II}TeO_{4.5}$ (M^{II} — Sr, Ba).

Experimental

The solid-phase synthesis of samples was carried out according to the standard ceramic technology from decrepitated gadolinium (III) and tellurium (IV) oxides, strontium, and barium carbonates. The stoichiometric amounts of the original materials were thoroughly mixed and ground in an agate mortar. Then they were annealed in alundum crucibles in a SNOL furnace. The following heat treatment mode was used, namely, Stage I for 15 hours at 400 °C, Stage II 20 hours at 800 °C, Stage III 20 hours at 1100 °C, then annealing was carried out at 400 °C for 20 hours in order to obtain stable compounds at low temperatures. After each stage, the mixtures were cooled, mixed, and thoroughly ground.

An X-ray study of the equilibrium compositions of the tellurites synthesized was carried out on an Empyrean device. Empyrean is the only platform that offers many benefits and high data accuracy for all sample types. This instrument is designed for a wide range of applications that include X-ray diffraction and X-ray scattering, as well as X-ray imaging. The Empyrean is designed to operate at 60 kV, which is optimal for X-ray

tubes with an anode of Mo and Ag. The intensity of the diffraction maxima was evaluated on a 100-point scale. The X-ray diffraction patterns of the obtained compounds were indexed by the homology method [4].

The pycnometric density of tellurites was determined by the method [5]. Toluene served as an indifferent liquid. The density of each tellurite was measured 3–5 times and the data was averaged.

Using the Landiya method [6], the standard heat capacities of the compounds were estimated from the calculated values of the standard entropies, and the temperature dependences of the heat capacities of the gadolinium tellurites synthesized were determined in the temperature range of 298–850 K.

Results and Discussion

X-ray phase analysis consists in identifying crystalline phases based on their inherent interplanar distances $d_{(hkl)}$ and the corresponding line intensities $I_{(hkl)}$ of the X-ray spectrum. The individuality and distribution of atoms determine the intensity of the diffracted rays. A powder diffraction pattern is an individual characteristic of a crystalline substance [7].

Figure 1 shows X-ray diffraction patterns of double gadolinium tellurites synthesized.

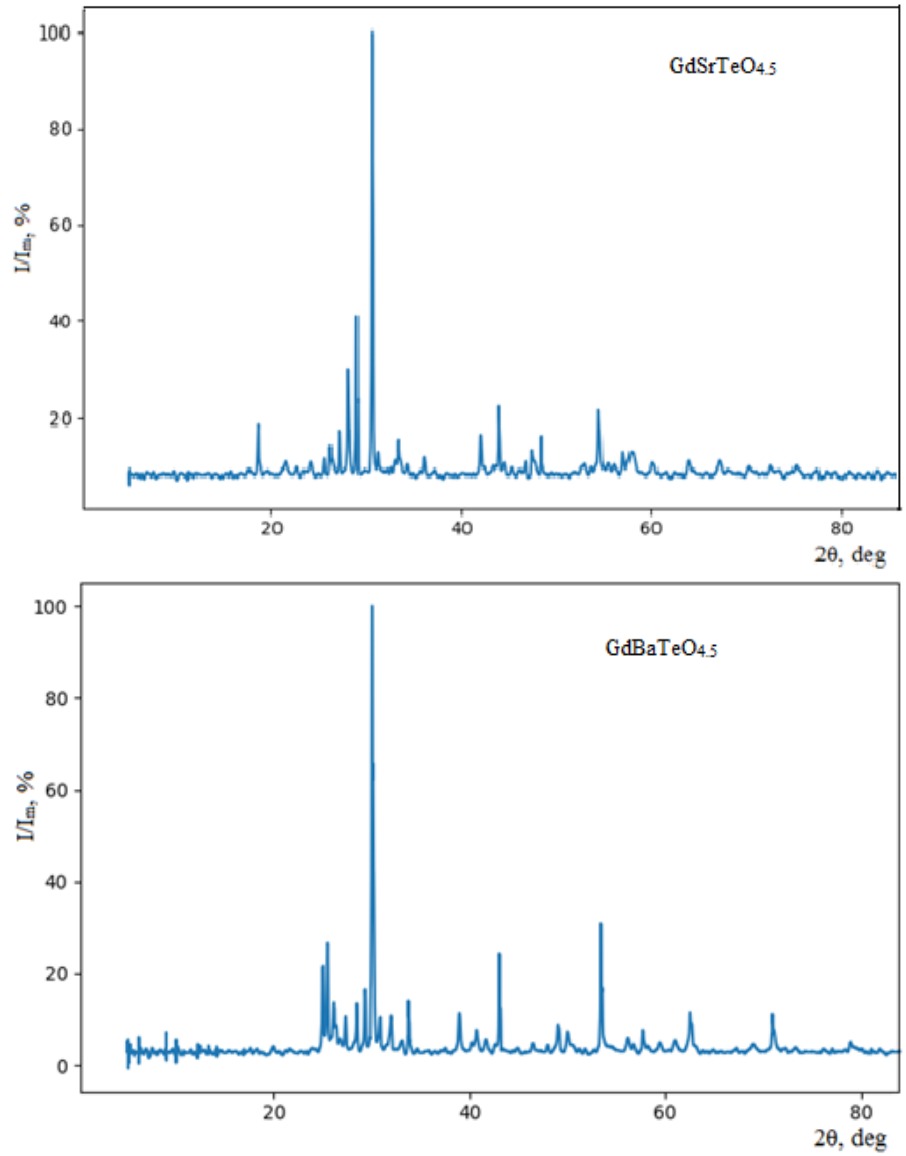


Figure 1. X-ray diffraction of the double-tellurite synthesized

The results of indexing the X-ray diffraction patterns of gadolinium tellurites synthesized are presented in Table 1.

Table 1

Radiographs indexing of tellurite $GdM^{II}TeO_{4,5}$(M^{II} — Sr, Ba) synthesized

h	k	l	$d_{obs.,Å}$	$d_{calc.,Å}$	2 th_{obs}, deg	2 th_{calc}, deg	I/I_{max} (%)
$GdSrTeO_{4.5}$							
2	1	-1	4.76531	4.76827	18.605	18.593	11.56
1	2	1	4.13362	4.13439	21.480	21.476	3.44
3	1	0	3.92007	3.91936	22.665	22.669	1.86
3	1	-1	3.69242	3.69683	24.083	24.053	2.64
0	3	0	3.47500	3.47408	25.614	25.621	4.03
2	0	2	3.39862	3.39690	26.200	26.213	6.06
3	2	0	3.28648	3.28438	27.111	27.128	9.63
3	0	-2	3.17599	3.17418	28.073	28.089	18.58
4	0	0	3.17599	3.17278	28.073	28.102	18.58
4	0	-1	3.07721	3.07777	28.993	28.988	35.75
2	3	-1	2.91623	2.91613	30.632	30.633	100.00
0	0	3	2.85710	2.85733	31.282	31.279	4.29
2	0	-3	2.70951	2.70922	33.033	33.037	1.64
3	2	-2	2.70951	2.71110	33.033	33.013	1.64
4	2	0	2.70951	2.71023	33.033	33.024	1.64
3	3	0	2.68234	2.68506	33.378	33.343	5.52
4	0	-2	2.68234	2.68220	33.378	33.380	5.52
0	4	0	2.60492	2.60591	34.400	34.387	2.28
1	3	2	2.60492	2.60676	34.400	34.375	2.28
3	0	-3	2.48651	2.48655	36.093	36.093	4.16
1	3	3	2.14720	2.14661	42.046	42.059	8.35
6	1	-1	2.06222	2.06207	43.867	43.870	14.88
2	3	3	2.03531	2.03632	44.478	44.454	2.24
5	3	-1	2.03111	2.03126	44.575	44.571	1.72
5	0	-3	2.00034	2.00021	45.298	45.301	1.78
$GdBaTeO_{4.5}$							
0	0	2	3.55333	3.55306	25.040	25.042	16.47
4	1	-1	3.49081	3.48785	25.496	25.518	21.87
1	4	-1	3.40853	3.41004	26.122	26.111	9.03
4	2	-1	3.25760	3.25868	27.356	27.346	7.19
5	0	0	3.13071	3.13077	28.487	28.487	9.72
4	2	1	3.04373	3.04372	29.319	29.319	12.82
5	0	-1	2.97033	2.97194	30.061	30.044	100.00
0	5	1	2.89253	2.89227	30.889	30.892	6.74
4	3	1	2.79599	2.79629	31.984	31.980	7.69
3	5	0	2.70704	2.70698	33.064	33.065	7.69
3	2	2	2.65330	2.65161	33.754	33.776	10.43
4	4	-1	2.55330	2.65330	33.754	33.754	10.43
1	0	3	2.30952	2.30958	38.967	38.966	8.29
7	1	0	2.21459	2.21429	40.709	40.715	3.84
1	3	-3	2.16622	2.16651	41.660	41.654	21.48
5	5	-1	2.16622	2.16697	41.660	41.645	21.48
6	3	1	2.16622	2.16702	41.660	41.644	21.48
4	1	-3	2.09957	2.09956	43.047	43.047	3.78
2	7	-1	2.09470	2.09348	43.152	43.179	3.78
5	4	-2	2.09470	2.09495	43.152	43.147	3.78
5	3	-3	1.85549	1.85589	49.057	49.046	5.75
6	6	0	1.85549	1.85524	49.057	49.064	5.75
3	7	-2	1.82256	1.82241	50.004	50.008	3.70

Notes. hkl — Miller indices; $d_{obs.}$ — experimental interplanar distances; $d_{cal.}$ — calculated interplanar distances; $2th_{obs.}$ — experimental double angle of Bragg reflection; $2th_{calc.}$ — calculated double angle of Bragg reflection; $I/I_{max.}$ — is the relative intensity of the X-ray patterns.

As can be seen from the data in Table 1, the experimental and calculated values of (d) and the X-ray and pycnometric densities values (Table 2) are in satisfactory agreement with each other, which shows the reliability and correctness of indexing results.

Based on indexing radiographs of tellurites investigated, it was found that the compounds $GdSrTeO_{4.5}$ and $GdBaTeO_{4.5}$ crystallize in the monoclinic system and have the unit cell parameters presented in Table 2.

Table 2

Type of syngonies and tellurites unit cell parameters

Compound	Syngony type	The lattice parameters, Å			V^0, Å^3	β, deg.	Z	Density, g/cm^3	
		a	b	c				Radiog.	Pycnom.
$GdSrTeO_{4.5}$	monoclinic	12.7610	10.4289	8.6235	1141.83	95.77	5	3.22	3.10±0.09
$GdBaTeO_{4.5}$	monoclinic	15.7272	15.8351	7.1393	1769.72	95.53	8	3.71	3.61±0.10

According to ASTM card files reference databases [8], tellurites synthesized X-ray diffraction patterns have been compared with X-ray indices [I/I_0, d] of original materials and with possible tellurites of this system. It was revealed that the diffractograms of new tellurites had no analogues. This data additionally confirms that synthesized tellurites are new compounds.

X-ray diffraction data shows that synthesized tellurites crystallize in the structural types of distorted perovskite P_m3_m. It allows supposing that these compounds can have unique electrophysical properties [9–12].

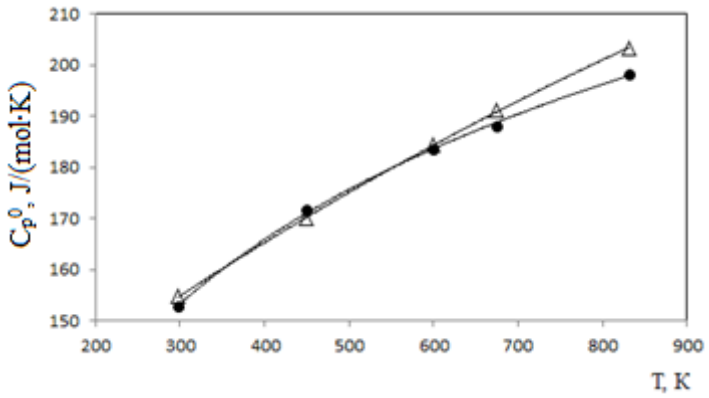
To calculate the temperature dependence of the heat capacities of the gadolinium tellurites synthesized, we chose the Landiya method [6], which is the most reliable of those available in the literature. The standard entropies were calculated using the Kumok ion increment method [13]. Using the Landiya method, the standard heat capacities of tellurites were estimated from the calculated values of the standard entropies, and the temperature dependences of the heat capacity were calculated using the Mayer-Kelly equation. The calculation results are shown in Table 3.

Table 3

Calculated heats capacity of double gadolinium tellurites in the range of 298-831 K

T, K	C_p^0, J/(mol·K)		T, K	C_p^0, J/(mol·K)	
	$GdSrTeO_{4.5}$	$GdBaTeO_{4.5}$		$GdSrTeO_{4.5}$	$GdBaTeO_{4.5}$
298.15	152.84	154.42	575	181.69	177.19
300	153.20	154.37	600	183.47	180.38
325	157.31	154.36	625	185.19	183.65
350	160.86	155.12	650	186.87	186.97
375	163.98	156.46	675	188.52	190.34
400	166.78	158.25	700	190.14	193.75
425	169.33	160.37	725	191.73	197.20
450	171.69	162.76	750	193.30	200.68
475	173.90	165.36	775	194.85	204.18
500	175.98	168.13	780	195.15	204.89
525	177.96	171.04	800	196.38	
550	179.86	174.07	831	198.26	

Figure 2 below depicts graphically the temperature dependences of the heat capacity of the tellurites investigated.



● — $GdSrTeO_{4.5}$, Δ — $GdBaTeO_{4.5}$

Figure 2. The temperature dependence of tellurites heat capacity

Thus, based on the calculated values of S°(298.15) by the Landiya method, for the first time, the estimated values of the standard heat capacity of double gadolinium tellurites are given and their equations for the temperature dependence of the heat capacities are derived. The obtained values of the standard heat capacities of tellurites are equal for $GdSrTeO_{4.5}$ (152.84±9.23) and $GdBaTeO_{4.5}$ (154.42±10.22) J/(mol·K). Equations of the temperature dependence of the heat capacity are as follows:

for $GdSrTeO_{4.5}$ $\quad Cp^0 = 37.16 + 13\cdot10^{-3}T - 4.0\cdot10^5\cdot T^{-2}$ (298–831 K),

for $GdBaTeO_{4.5}$ $\quad Cp^0 = 19.98 + 36\cdot10^{-3}T + 5.5\cdot10^5\cdot T^{-2}$ (298–780 K).

Conclusions

New double gadolinium-strontium $GdSrTeO_{4.5}$ and gadolinium-barium $GdBaTeO_{4.5}$ tellurites were synthesized by the method of ceramic technology. The formation of equilibrium compositions in the compounds synthesized was controlled by X-ray phase analysis. The XRF method was used to determine the system types, unit cell parameters, X-ray, and pycnometric densities. Tellurites crystallize in the structural types of distorted perovskite P_m3_m. It allows supposing that these compounds can have unique electrophysical properties.

Using the Landiya method, the compounds standard heat capacities were estimated from the standard entropies calculated values, and the heat capacities temperature dependence of the tellurites synthesized in the temperature range of 298–850 K was presented. The data obtained is of certain interest for chemistry and non-organic materials science of rare earth elements and chalcogenes complex oxide compounds.

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А.А. Тойбек, К.Т. Рустембеков, Д.А. Кайкенов, М.Стоев

Гадолиний қос теллуриттерінің синтезі және қасиеттері

Алғаш рет қатты фазалық әдіспен $GdM^{II}TeO_{4,5}$ (M^{II} — Sr, Ba) құрамды гадолинийдің қос теллуриттері синтезделді. Үлгілердің қатты фазалық синтезі алдын-ала күйдірілген гадолиний (III) және теллур (IV) оксидтері, стронций және барий карбонаттарынан стандартты керамикалық технология бойынша іске асырылды. Синтез 800–1100 °С температура аралығында жүргізілді. Алынған үлгілер рентгенфазалық анализ әдісімен зерттелді. Рентгенфазалық анализ Empyrean қондырғысында жүргізілді. Дифракциялық максимумдардың қарқындылығы жүз балдық шкаламен бағаланды. Зерттелетін гадолиний — жерсілтілік металдары теллуриттерінің ұнтақтарының рентгенограммаларын индицирлеу гомология әдісімен жүргізілді. Рентгенограммаларды индицирлеу нәтижелерінің дұрыстығын және дәлдігін жазықтықаралық қашықтықтың (d) тәжірибелік және есептелген мәндері мен рентгендік және пикнометрлік тығыздықтарының мәндерінің сәйкестігі дәлелдейді. $GdSrTeO_{4,5}$ және $GdBaTeO_{4,5}$ қосылыстары моноклинді сингонияда кристалданатыны және келесідей элементарлық ұяшық параметрлері табылды: $GdSrTeO_{4,5}$ — a = 12,7610, b = 10,4289, c = 8,6235 Å, V° = 1141,83 Å^3, β = 95,77°, Z = 5, $\rho_{рент.}$ = 3,22, $\rho_{пикн}$ = (3,10±0,09) г/см^3; $GdBaTeO_{4,5}$ — a = 15,7272, b = 15,8351, c = 7,1393 Å, V° = 1769,72 Å^3, β = 95,53°, Z = 8, $\rho_{рент.}$ = 3,71, $\rho_{пикн.}$ = (3,61±10) г/см^3. Ландия әдісімен есептелген стандартты энтропия мәндерінен қосылыстардың стандартты жылу сыйымдылықтары табылды және 298-850 К температура аралығында синтезделген гадолиний теллуриттерінің жылу сыйымдылықтарының температуралық тәуелділіктері анықталды.

Кілт сөздер: гадолиний қос теллуриттері, рентгенфазалық анализ, сингония, тор параметрлері, жылу сыйымдылық.

А.А. Тойбек, К.Т. Рустембеков, Д.А. Кайкенов, М. Стоев

Синтез и свойства двойных теллуритов гадолиния

Твердофазным методом впервые синтезированы двойные теллуриты гадолиния состава $GdM^{II}TeO_{4,5}$ (M^{II} — Sr, Ba). Твердофазный синтез образцов был осуществлен по стандартной керамической технологии из предварительно прокаленных оксидов гадолиния (III) и теллура (IV), карбонатов стронция и бария. Синтез проводился в температурном интервале 800–1100 °С. Полученные образцы были аттестованы методом рентгенофазового анализа. Рентгенофазовый анализ проведен на приборе *Empyrean*. Интенсивность дифракционных максимумов оценивалась по стобалльной шкале. Индицирование рентгенограмм порошка исследуемых теллуритов гадолиния — щелочноземельных металлов проводили методом гомологии. Достоверность и корректность результатов индицирования рентгенограмм подтверждаются хорошим соответствием экспериментальных и расчетных значений межплоскостных расстояний (d) и согласованностью величин рентгеновской и пикнометрической плотностей. Установлено, что соединения $GdSrTeO_{4,5}$ и $GdBaTeO_{4,5}$ кристаллизуются в моноклинной сингонии и имеют параметры элементарных ячеек: $GdSrTeO_{4,5}$ — a = 12,7610, b = 10,4289, c = 8,6235 Å, V° = 1141,83 Å^3, β = 95,77°, Z = 5, $\rho_{рент.}$ = 3,22, $\rho_{пикн.}$ = (3,10±0,09) г/см^3; $GdBaTeO_{4,5}$ — a = 15,7272, b = 15,8351, c = 7,1393 Å, V° = 1769,72 Å^3, β = 95,53°, Z = 8, $\rho_{рент.}$ = 3,71, $\rho_{пикн.}$ = (3,61±0,10) г/см^3. Методом Ландия из вычисленных значений стандартных энтропий рассчитаны стандартные теплоемкости соединений, определены температурные зависимости теплоемкостей синтезированных теллуритов гадолиния в интервале температур 298–850 К.

Ключевые слова: двойные теллуриты гадолиния, рентгенофазовый анализ, сингония, параметры решетки, теплоемкость.

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Information about authors:

Toibek Aitolkyn Ablaikyzy — PhD student Faculty of Chemistry Karagandy University of the name of academician E.A. Buketov, Universitetskaya str., 28, 100024, Karaganda, Kazakhstan; e-mail: aitoka_95@mail.ru; https://orcid.org/0000-0002-8616-5257;

Rustembekov Kenzhebek Tusupovich — Academician of Kazakhstan National Academy of Natural Sciences (KazNaNs), Doctor of chemical sciences, Professor, Karagandy University of the name of academician E.A. Buketov, Universitetskaya str., 28, 100024, Karaganda, Kazakhstan; e-mail: rustembekov_kt@mail.ru; https://orcid.org/0000-0003-0853-523X;

Kaikenov Dauletkhan Asanovich — Leading researcher of the laboratory of engineering profile "Physical and chemical methods of research", Karagandy University of the name of academician E.A. Buketov, Universitetskaya str., 28, 100024, Karaganda, Kazakhstan; e-mail: krg.daykai@mail.ru;

Stoev Mitko — PhD, Associate Professor, South-West University "Neofit Rilski", Blagoevgrad, Bulgaria.