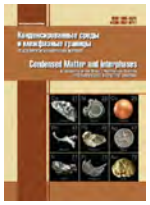

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New compounds $Li_3Ba_2Bi_3(XO_4)_8$ (X = Mo, W): synthesis and properties

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Abstract

New compounds $Li_3Ba_2Bi_3(XO_4)_8$ (X = Mo, W) were obtained by the ceramic technology. Those are the first representatives of the ternary molybdates and tungstates $Li_3Ba_2R_3(XO_4)_8$ family, which contain different from the rare earth elements trivalent metal. The sequence of chemical transformations occurring during the $Li_3Ba_2Bi_3(WO_4)_8$ formation has been established. The primary characterization of the obtained phases was carried out and their ion-conducting properties were studied. The synthesized compounds are shown to melt incongruently, isostructural to the lanthanide-containing analogues (structural type of $BaNd_2(MoO_4)_4$, sp. gr. $C2/c$) and crystallize in the monoclinic crystal system with unit cell parameters a = 5.2798(1), b = 12.8976(4), c = 19.2272(5) Å, β = 90.978(2)° (X = Mo), a = 5.2733(2), b = 12.9032(4), c = 19.2650(6) Å, β = 91.512(3)° (X = W). $Li_3Ba_2Bi_3(XO_4)_8$ are found to undergo the diffuse first-order phase transitions at 441°C (molybdate) and 527°C (tungstate), after that their conductivity reaches values of 10^{-3}–10^{-4} S/cm.

Keywords: triple molybdates and tungstates, solid-state synthesis, powder X-ray diffraction study, thermal properties, ionic conductivity

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1. Introduction

Currently, the class of ternary molybdates includes more than 700 representatives, characterized by a large stoichiometric and structural diversity, and belongs to the most dynamically replenished groups of complex oxide compounds containing a tetrahedral anion and three different cations. Triple molybdates of different valence metals not only have a high material science potential, but also due to the wide possibilities of varying the elemental and quantitative compositions are convenient model objects for establishing genetic relationships in the series of composition – structure – properties of compound – properties of materials. A significant place among them is occupied by the family of triple molybdates obtained for all lanthanides and yttrium with the composition $Li_3Ba_2R_3(MoO_4)_8$, belonging to the structure type $BaNd_2(MoO_4)_4$ (sp. gr. *C*2/*c*, Z = 2) and related to scheelite. These compounds are shown to possess promising luminescent and generation properties [1–3], as well as the properties of solid electrolytes [1, 4]. In particular, the obtained results aimed at study of $Li_3Ba_2R_3(MoO_4)_8$ (*R* = La, Gd, Y) doped with Eu^{3+}, Tb^{3+}, Er^{3+}, Nd^{3+}, indicate the prospects for their use as new photo- and IR-luminophores and laser materials [2, 5–7]. Since 2009, studies on the preparation of ternary tungstates of lithium-barium-lanthanides, isoformular and isostructural to the $Li_3Ba_2Ln_3(MoO_4)_8$ which are, like them, are of great not only scientific but also practical interest started to appear [8–17]. Sizes and quality of grown crystals $Li_3Ba_2Ln_3(WO_4)_8$ (*Ln* = La, Gd, Y) [9–12, 18] doped with Nd^{3+}, Eu^{3+}, Tm^{3+} and other ions, allowed proceeding to a detailed study of the optical-generation characteristics of these new highly efficient laser media. Ceramics $Li_3Ba_2La_3(WO_4)_8$: Eu^{3+} [8] and $Li_3Ba_2Gd_3(WO_4)_8$: Tb^{3+} [15] can be used as red and green luminophores, respectively.

In this study, the first representatives of the considered family of phases containing in their composition a different from the rare earth trivalent element – ternary bismuth – containing molybdate and tungstate of composition $Li_3Ba_2Bi_3(XO_4)_8$ (*X* = Mo, W) were obtained by directed solid state synthesis. The primary characterization of the obtained compounds was carried out and their electrophysical properties were studied.

2. Experimental

Industrial reagents Li_2MoO_4, Li_2WO_4, XO_3 (*X* = Mo, W), Bi_2O_3, $BaMoO_4$, $BaCO_3$ (chemically pure) were used as source components for the synthesis of $Li_3Ba_2Bi_3(XO_4)_8$ (*X* = Mo, W). $BaWO_4$ was obtained by annealing of the stoichiometric mixture of $BaCO_3$ and WO_3 (600–850 °C, 70 h), $Bi_2(MoO_4)_3$ – by the reaction: $Bi_2O_3 + 3MoO_3 = Bi_2(MoO_4)_3$ (450–500 °C, 50 h). Tertiary bismuth tungstate does not exist; it could not be obtained by the solid-state method, as it was shown in the literature data [19] and proved by our unsuccessful attempts to synthesize those. Therefore, in this case the source component was an oxide mixture of Bi_2O_3 and WO_3. AXO_4 (*A* = Ca, Sr, Cd, Pb; *X* = Mo, W), required to study of the possibility of realizing the considered structure in ternary molybdates and bismuth tungstates with complete or partial substitution of barium by another doubly charged cation, were obtained by the interaction of ACO_3 (chemically pure and analytical grade) and XO_3 by the reaction $ACO_3 + XO_3 = AXO_4 + CO_2$. The synthesis conditions were as follows: in case of Ca, Pb – 500–650 ° C, Sr – 500–750 ° C, Cd – 450–500 °C for 50–60 h; tungstates: Ca, Sr – 600–900 °C, Cd - 500–650 ° C, Pb - 500–750 °C for 70–80 h. The single-phase of the synthesized materials was monitored by powder X-ray diffraction analysis. The obtained compounds were identified by comparison with the ICDD PDF-2 database [20].

Powder X-ray diffraction analysis (XRD) was performed using a Bruker D8 ADVANCE diffractometer ($\lambda Cu K_\alpha$, secondary monochromator, scanning step 0.02076°). Unit cell parameters of polycrystalline samples $Li_3Ba_2R_3(XO_4)_8$ (*X* = Mo, W) was calculated by the selection of an isostructural compound. Unit cell parameters were refined by the least squares method using the ICDD software package for preparation of the experimental standards. The Smith–Snyder F_{30} criterion was used as a validation criterion for X-ray patterns indexing [21].

Differential scanning calorimetry studies were carried out using NETZSCH STA 449C synchronous thermal analyser, $V_{heat.(cool.)} = 10°/min$.

For the ion-conducting properties investigation the ceramic discs $Li_3Ba_2Bi_3(XO_4)_8$ (*X* = Mo, W) were prepared by pressing the powder at 1 kbar and annealing at 680 (*X* = Mo) or 730 °C (*X* = W) for 4 hours. The density of the obtained tablets was 90–95 % of the theoretical values. The

disks were in diameter of 10 mm and thickness of 1.8 mm. In order to prepare electrodes, the surfaces of the disks were coated with colloidal platinum, followed by annealing at 660 (X = Mo) or 710 °C (X = W) for 1 hour. The electrical conductivity measurements of the samples were tested using an impedance meter "Z-1500J" at selected frequencies from 1 Hz to 1 MHz in the temperature range of 200–650 °C (X = Mo) and 300–700 °C (X = W) with heating and cooling rates of 2 deg./min.

3. Results and discussion

In the single-phase polycrystalline state, the triple molybdate $Li_3Ba_2Bi_3(MoO_4)_8$ synthesized by annealing of stoichiometric mixtures of Li_2MoO_4, $BaMoO_4$, and $Bi_2(MoO_4)_3$ at 450–550 °C for 150 h, analogous triple tungstate $Li_3Ba_2Bi_3(WO_4)_8$ – 300 hour annealing of Li_2WO_4, $BaWO_4$, Bi_2O_3 and WO_3, taken in a molar ratio of 3: 4: 3: 9, at 550–700 °C (intermediate homogenization was carried out every 15–20 h).

According to powder XRD data (Fig. 1), the sequence of chemical transformations occurring during the formation of $Li_3Ba_2Bi_3(WO_4)_8$ from a stoichiometric mixture of oxides and tertiary tungstates, can be illustrated by the scheme [22]:

$$\left.\begin{matrix} Li_2WO_4 \\ BaWO_4 \\ Bi_2O_3 \\ WO_3 \end{matrix}\right] \xrightarrow[100\ h]{550–610°C,} \begin{matrix} Li_2W_2O_7 \\ BaWO_4 \\ Bi_2WO_6 \end{matrix} \left.\begin{matrix} \\ \\ \\ \end{matrix}\right] \xrightarrow[100\ h]{650–680°C,} \rightarrow \left.\begin{matrix} BaWO_4 \\ LiBi(WO_4)_2 \\ Li_3Ba_2Bi_3(WO_4)_8 \end{matrix}\right] \xrightarrow[100\ h]{700°C,} Li_3Ba_2Bi_3(WO_4)_8$$

The formation of $Li_3Ba_2Bi_3(MoO_4)_8$ most likely also proceeds through the stage of formation of a double lithium-bismuth compound, but due to the close temperature ranges of the formation of intermediate and final products, the appearance of $LiBi(MoO_4)_2$ in the reaction mixture was not recorded.

According to the differential scanning calorimetry (DSC) data, the obtained compounds melt incongruently at 756 (X = Mo) and 786 °C (X = W). In addition to $BaWO_4$ and $LiBi(WO_4)_2$ and Bi_2WO_6 the presence of $BaMoO_4$ and $LiBi(MoO_4)_2$, tungstate was revealed in the cooled melt of molybdate by XRD analysis.

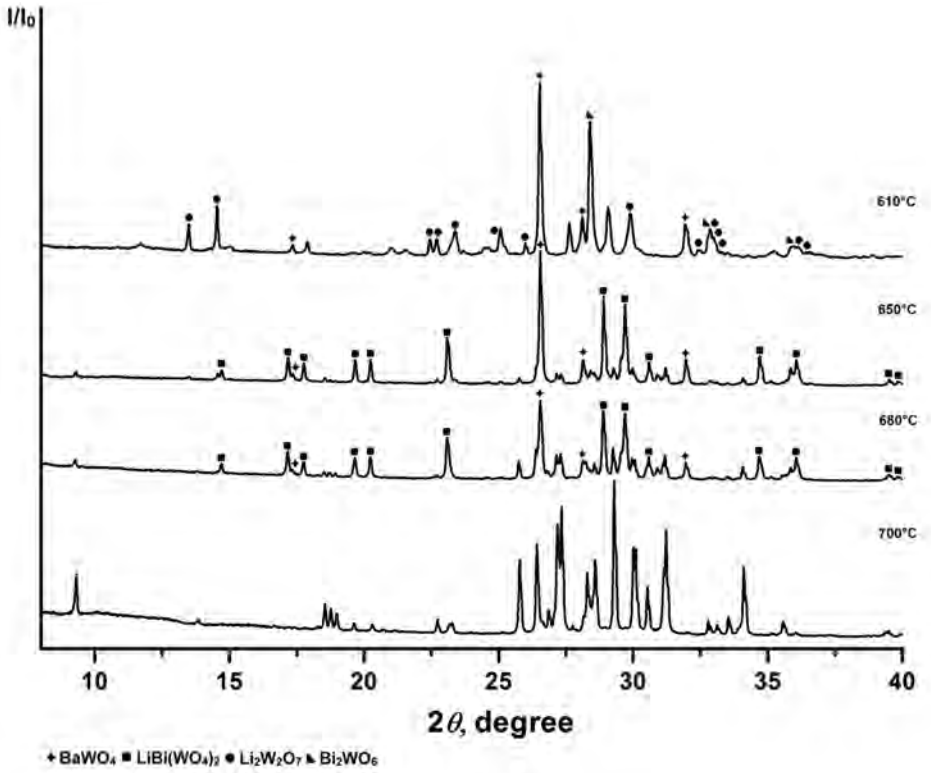


Fig 1. Powder X-ray diffraction patterns of the reaction mixture $Li_2WO_4 + 4BaWO_4 + 3Bi_2O_3 + 9WO_3$, sequentially annealed at different temperatures

Powder XRD patterns of $Li_3Ba_2Bi_3(XO_4)_8$ (X = Mo, W) were indexed satisfactory under the assumption of isostructurality to lanthanide-containing analogues (in the case of molybdate $F(30) = 217.1$ (0.0035; 39), tungstate – $F(30) = 162.3$ (0.0047; 39)). The obtained crystallographic characteristics are shown in Table 1, the results of indexing of $Li_3Ba_2Bi_3(WO_4)_8$ are shown in Table 2.

The possibility of realizing a similar structure in ternary bismuth molybdates and tungstates by replacing barium with another double-charged cation A^{2+} was investigated. However, attempts to synthesize $Li_3A_2Bi_3(XO_4)_8$ (A = Ca, Sr, Cd, Pb) were unsuccessful. The compositions $Li_3Ba_{1.9}A_{0.1}Bi_3(MoO_4)_8$ were obtained by the partial substitution of barium with strontium, cadmium,

Table 1. Crystallographic characteristics of $Li_3Ba_2Bi_3(XO_4)_8$ (X = Mo, W), sp. gr. $C2/c$, Z = 2

Compound	Unit cell parameters				V, Å^3
	a, Å	b, Å	c, Å	β,°	
$Li_3Ba_2Bi_3(MoO_4)_8$	5.2798(1)	12.8976(4)	19.2272(5)	90.978(2)	1309.12
$Li_3Ba_2Bi_3(WO_4)_8$	5.2733(2)	12.9032(4)	19.2650(6)	91.512(3)	1310.38

Table 2. Indexing results of powder X–ray diffraction pattern for $Li_3Ba_2Bi_3(WO_4)_8$

$2\theta_{exp}$,°	I/I_0	d_{exp}, Å	h	k	l	$\Delta = 2\theta_{exp} - 2\theta_{calc}$,°
9.191	31	9.6140	0	0	2	−0.014
13.723	3	6.4475	0	2	0	−0.009
16.539	2	5.3555	0	2	2	−0.013
18.163	2	4.8802	1	1	0	+0.001
18.417	18	4.8134	0	0	4	−0.004
18.637	14	4.7571	−1	1	1	−0.004
18.857	11	4.7021	1	1	1	−0.004
19.501	5	4.5482	0	2	3	−0.010
20.184	5	4.3958	−1	1	2	−0.002
20.592	1	4.3097	1	1	2	−0.005
22.601	10	3.9309	−1	1	3	−0.003
23.032	6	3.8583	0	2	4	−0.002
23.148	8	3.8392	1	1	3	−0.004
25.650	66	3.4701	−1	1	4	+0.002
26.298	57	3.3861	1	1	4	−0.001
26.723	16	3.3332	−1	3	0	0.005
26.943	9	3.3065	0	2	5	−0.005
27.059	86	3.2926	−1	3	1	−0.004
27.219	100	3.2736	1	3	1	−0.009
27.639	8	3.2248	0	4	0	−0.009
28.032	11	3.1804	0	4	1	−0.009
28.172	51	3.1650	−1	3	2	−0.006
28.464	45	3.1331	1	3	2	+0.000
29.167	97	3.0592	−1	1	5	−0.007
29.880	63	2.9878	1	1	5	−0.003
29.982	28	2.9779	−1	3	3	−0.002
30.402	26	2.9377	1	3	3	−0.001
31.001	47	2.8823	0	4	3	−0.001
31.097	61	2.8736	0	2	6	−0.001
32.998	8	2.7123	−1	1	6	+0.000
33.411	14	2.6797	0	4	4	−0.003
33.781	5	2.6512	1	1	6	−0.012
33.985	61	2.6357	2	0	0	+0.000
35.032	1	2.5593	−2	0	2	−0.002
35.293	1	2.5410	−1	3	5	−0.004
35.447	16	2.5303	0	2	7	−0.006
35.527	1	2.5248	2	0	2	−0.008
35.900	2	2.4994	1	3	5	−0.003
36.810	1	2.4397	2	2	0	−0.005
37.084	1	2.4223	−1	1	7	+0.004
37.322	1	2.4074	0	0	8	+0.001
38.601	1	2.3305	−1	3	6	−0.014
38.814	1	2.3182	−1	5	0	+0.007
39.056	1L	2.3044	−1	5	1	+0.000
39.168	2	2.2981	1	5	1	+0.000
39.268	3	2.2924	1	3	6	−0.006
39.382	5	2.2861	2	0	4	−0.012
39.819	2	2.2620	2	2	3	−0.010
39.945	2	2.2551	0	2	8	−0.005
41.030	1	2.1980	−2	2	4	−0.004
41.228	1	2.1879	−1	5	3	−0.006
41.384	2	2.1800	−1	1	8	−0.003
41.554	1L	2.1714	1	5	3	−0.011
41.879	2	2.1553	2	2	4	+0.000
41.975	2	2.1506	0	6	0	+0.002
42.245	1L	2.1375	0	6	1	+0.006
42.946	2	2.1042	1	3	7	−0.005
43.041	1	2.0998	0	6	2	+0.021
43.083	3	2.0979	−1	5	4	−0.002
43.158	1	2.0944	0	4	7	+0.024
43.378	2	2.0843	−2	2	5	−0.001
44.391	25	2.0390	0	6	3	−0.003
44.584	23	2.0306	0	2	9	−0.008
44.740	2	2.0239	2	4	1	−0.033
45.180	12	2.0052	−2	4	2	+0.005
45.395	2	1.9962	−1	5	5	+0.001

End of Table 2

$2\theta_{exp}$,°	I/I_0	d_{exp}, Å	h	k	l	$\Delta = 2\theta_{exp} - 2\theta_{calc}$,°
45.581	15	1.9885	2	4	2	+0.001
45.881	3	1.9762	1	5	5	+0.009
46.136	21	1.9659	−2	2	6	+0.004
46.190	26	1.9637	0	6	4	+0.004
46.363	25	1.9568	−2	4	3	+0.003
46.948	22	1.9338	2	4	3	+0.001
47.077	2	1.9288	0	4	8	−0.014
47.147	9	1.9261	0	0	10	+0.006
47.311	17	1.9198	2	2	6	−0.006
48.018	6	1.8931	−2	4	4	+0.001
48.116	4	1.8895	−1	5	6	+0.000
48.447	3	1.8774	0	6	5	−0.008
48.680	5	1.8689	1	5	6	+0.003
48.771	7	1.8657	2	4	4	+0.004
49.270	11	1.8479	−2	2	7	−0.003
49.347	11	1.8452	0	2	10	−0.004
50.249	1	1.8142	−1	3	9	−0.006
50.481	9	1.8064	−1	1	10	−0.003
50.565	10	1.8036	2	2	7	−0.005
51.066	1	1.7871	1	3	9	−0.001
51.198	25	1.7828	−1	5	7	−0.001
51.391	11	1.7765	1	1	10	−0.003
51.829	24	1.7625	1	5	7	+0.000
52.069	2	1.7550	2	0	8	+0.012
52.558	22	1.7398	1	7	0	−0.007
52.746	8	1.7340	−1	7	1	−0.008
52.826	7	1.7316	1	7	1	+0.000
53.373	4	1.7151	−1	7	2	+0.008
53.558	2	1.7096	1	7	2	−0.001
53.665	2	1.7065	2	4	6	−0.004
54.601	1	1.6794	−1	3	10	−0.007
54.727	1	1.6759	1	7	3	+0.005
55.063	2	1.6664	−2	6	0	+0.005
55.270	7	1.6607	−1	1	11	−0.007
55.526	1	1.6536	0	4	10	0.002
55.627	8	1.6509	−3	1	4	−0.012
55.832	3	1.6453	−2	6	2	−0.045
56.021	2	1.6402	−1	7	4	−0.030
56.133	1	1.6372	2	6	2	−0.005
56.208	5	1.6352	1	1	11	−0.004
56.460	8	1.6285	−2	2	9	−0.006
56.557	2	1.6259	−3	3	0	−0.029
56.627	11	1.6241	3	1	4	+0.009
56.810	4	1.6193	−2	6	3	−0.006
56.869	7	1.6177	3	3	1	+0.006
57.142	6	1.6106	−3	3	2	+0.009
57.309	4	1.6063	2	6	3	+0.000
57.464	8	1.6024	−3	1	5	+0.002
57.651	4	1.5976	3	3	2	+0.003
57.965	5	1.5897	2	2	9	+0.002
58.112	2	1.5860	−3	3	3	−0.001
58.229	7	1.5831	−2	6	4	+0.013
58.287	14	1.5817	−1	5	9	+0.011
58.557	2	1.5750	−2	0	10	+0.006
58.659	4	1.5725	−2	4	8	−0.007
58.727	4	1.5709	3	1	5	−0.013
58.893	13	1.5668	2	6	4	+0.011
59.043	10	1.5632	1	5	9	+0.000
59.148	1	1.5607	−1	3	11	+0.002
59.302	1	1.5570	0	2	12	−0.016
59.704	2	1.5475	−3	1	6	+0.001
59.972	1	1.5412	2	4	8	−0.006
60.070	3	1.5389	0	4	11	0.008
60.210	3	1.5357	−1	1	12	−0.001
60.459	7	1.5300	−2	2	10	+0.000
60.887	1	1.5202	2	6	5	+0.006
61.040	1	1.5168	0	6	9	−0.003
61.181	2	1.5136	1	1	12	+0.001
62.057	7	1.4943	2	2	10	+0.013
62.150	1	1.4923	−2	4	9	+0.005
62.274	1	1.4897	−1	5	10	−0.005
62.368	5	1.4876	0	8	5	−0.004
62.465	1	1.4856	3	3	5	+0.000
63.061	1	1.4729	1	5	10	+0.004
63.220	1L	1.4696	2	6	6	+0.035
63.908	2	1.4555	−1	3	12	+0.000
64.135	1	1.4508	−3	5	1	+0.008
64.475	1L	1.4440	0	2	13	+0.009
64.608	8	1.4414	0	8	6	+0.010
64.832	6	1.4369	0	4	12	+0.003

and lead in the single-phase state. The solubility limit in similar tungstates was less than 5%.

The ion-conducting properties of the obtained compounds were studied. It was established that $Li_3Ba_2Bi_3(XO_4)_8$ undergo phase transformations at 441 (X = Mo) and 527 °C (X = W), which, based on the presence of temperature hysteresis in $\lg(\sigma T)$–$(10^3/T)$ dependences in the heating – cooling cycle can be interpreted as the first order diffuse phase transitions. After the transition, the conductivity $Li_3Ba_2Bi_3(MoO_4)_8$ reached values of 3.5 10^{-3} S/cm (640 °C) at E_a = 1.0 eV, $Li_3Ba_2Bi_3(WO_4)_8$ – 2.7 10^{-4} S/cm (700 °C) at E_a = 0.8 eV. Temperature dependence of electrical conductivity of $Li_3Ba_2Bi_3(MoO_4)_8$ as an example is shown in Fig. 2. The obtained interdependence

of the active and reactive components of the electrical impedance for this compound (at temperatures before and after the phase transition), typical for ionic conductors with blocking electrodes is shown in Fig. 3.

4. Conclusions

Thus, the first compounds of $Li_3Ba_2R_3(XO_4)_8$ (X = Mo, W) family were obtained with the structure of $BaNd_2(MoO_4)_4$ (sp. gr. $C2/c$, Z = 2), Z = 2), containing different from the rare earth elements a trivalent metal. The sequence of chemical transformations occurring during the synthesis of ternary tungstate of lithium, barium, bismuth from a stoichiometric mixture of tertiary tungstates and oxides was established. Crystallographic and thermal characteristics of $Li_3Ba_2Bi_3(XO_4)_8$ (X = Mo, W) were determined and their ion-conducting properties were studied. It was shown that triple molybdates and tungstates

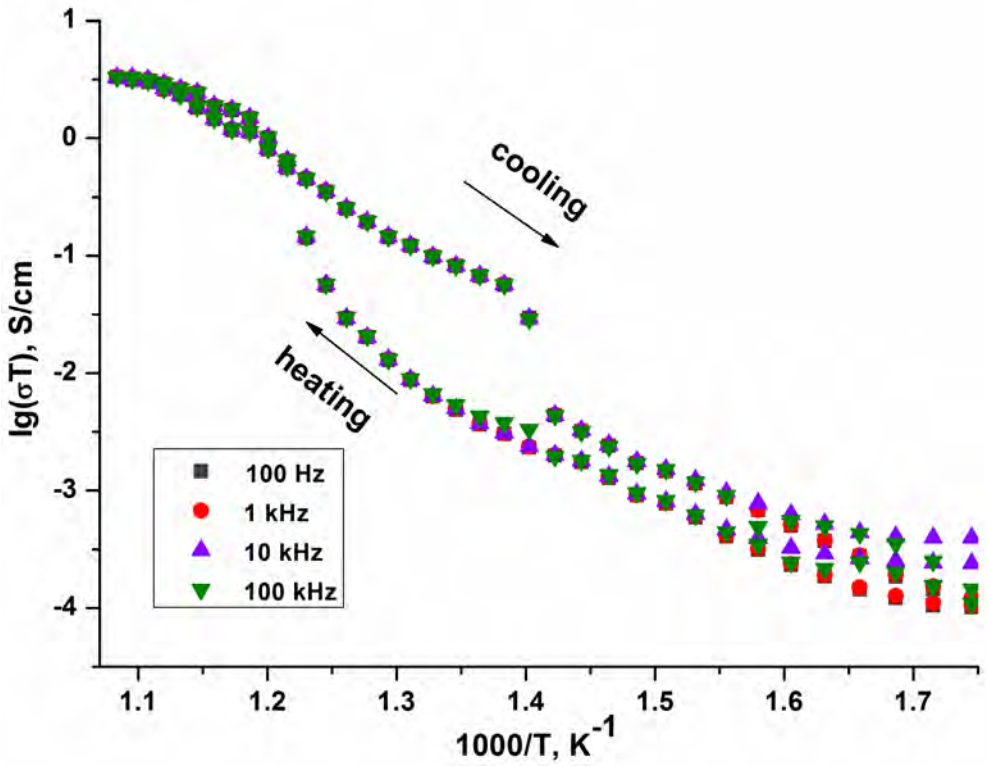
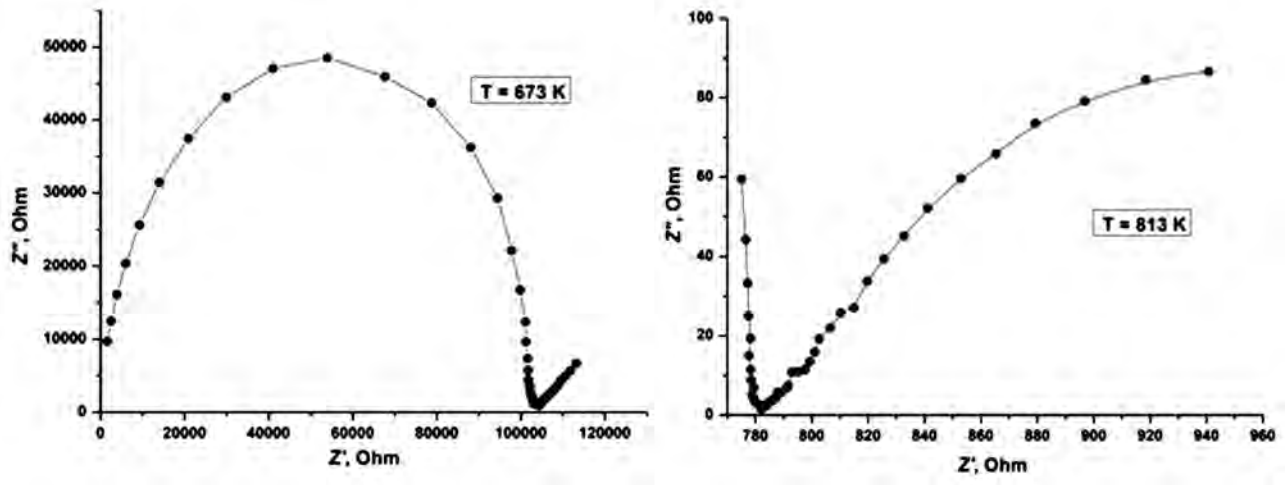


Fig. 2. The temperature dependence of the electrical conductivity for $Li_3Ba_2Bi_3(MoO_4)_8$



a *b*

Fig. 3. Nyquist plot for $Li_3Ba_2Bi_3(MoO_4)_8$ at 673 K (a) and 813 K (b)

$Li_3A_2Bi_3(XO_4)_8$ (A = Ca, Sr, Cd, Pb; X = Mo, W) with the structure $BaNd_2(MoO_4)_4$ are not formed.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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