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Synthesis, X-ray and thermodynamic studies of compounds $Yb_2M^I{}_3Fe_5O_{12}$ (M^I — Li, Na, K)

The new compounds $Yb_2M^I{}_3Fe_5O_{12}$ (M^I — Li, Na, K) have been synthesized by solid phase method from oxides of lithium, sodium, potassium, iron and lanthanum. By means of XRD analysis it was determined that the compounds crystallize in a tetragonal crystal system. The compounds' heat capacities have been measured in the range of temperature 298.15 ... 673 K and have been found λ-shaped jumps on the dependence diagrams $C_P^0 \sim f(T)$ which caused, probably by phase transitions of the second type.

Key words: solid-phase synthesis, ferrites, crystal lattice parameters of the crystal lattice, the temperature dependence of the specific heat, the temperature dependence of the thermodynamic functions, phase transformations.

Systems consisting of oxides of REE, alkaline metals and iron (III) are important for the inorganic materials chemistry and their new ferrites may have simultaneously many original and unique properties. In order to obtain of new ferrites with such properties it is necessary to develop conditions of synthesis, conduct structural investigations and study their thermodynamic and electrical properties.

The obtaining of new compounds as ferrites of the above oxides and study their thermodynamic properties would make a significant contribution to the physical chemistry of oxide compounds of transition (3*d*-, 4*f*-), alkali and alkaline earth metals and in the materials science. The purpose of this work is the synthesis and study of X-ray diffraction, the thermodynamic structure of the novel compounds of next composition $Yb_2M^I{}_3Fe_5O_{12}$ (M^I — Li, Na, K).

The experimental part

For high-temperature solid-phase synthesis were used the following starting materials: ytterbium oxide of «high purity», iron (III), carbonates of alkali metals of «chemically pure». Stoichiometric amounts of initial substances, weighed to the fourth decimal place, calculated taking into account the final composition: $Yb_2M^I{}_3Fe_5O_{12}$ (M^I — Li, Na, K). Prepared samples were thoroughly mixed, crushed in an agate mortar, placed in a corundum crucible and held at a temperature 800 ºC in the furnace «SNOL» within 10 hours. For obtain of stable equilibrium phases at low temperature has been carried out calcination at temperature 400 ºC within 20 hours. During this process the mixture thoroughly was mixed. X-ray exposure of the sample was carried out on diffractometer DRON-2.0 (CuKα-radiation).

Powder diffractograms of novel compounds have been indicated by the homology method [1] and are given in the Table 1.

Table 1

Indexing of powder's roentgenograms $Yb_2M^I{}_3Fe_5O_{12}$ (M^I — Li, Na, K)

I/I_0, %	D, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{calc.}$	I/I_0, %	D, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{calc.}$
$Yb_2Li_3Fe_5O_{12}$					16	3.3922	869.0	311	881.9
25	5.4397	337.9	112	324.8	30	3.2990	923.9	302	914.8
11	5.1547	376.4	201	376.2	28	3.1149	1030.7	223	1025.9
16	5.0807	387.4	201	376.2	34	3.0048	1107.5	320	1095.6
30	4.8113	432.0	210	421.4	10	2.8811	1204.7	313	1194.4
7	4.5562	481.7	202	493.4	20	2.7775	1296.3	224	1299.4
12	4.4515	504.6	202	493.4	100	2.7286	1343.1	400	1348.5
6	4.1264	587.3	212	577.7	50	2.6815	1390.7	401	1387.5
19	3.8343	680.2	220	674.2	56	2.6171	1460.0	411	1471.8
20	3.7930	695.1	203	688.7	40	2.5003	1599.6	412	1589.0
68	3.6239	761.5	300	758.5	10	2.3915	1748.5	206	1743.6

I/I_0, %	D, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{calc.}$
32	2.2321	2007.2	107	1998.7
12	2.1502	2162.9	306	2165.0
8	2.1180	2229.2	511	2230.3
23	2.0792	2313.2	424	2310.7
6	2.0419	2398.5	415	2409.5
6	2.0187	2453.8	520	2444.1
24	1.9784	2554.9	513	2542.9
10	1.9224	2705.9	440	2696.9
21	1.8850	2814.3	514	2816.4
8	1.8462	2934.0	336	2923.5
18	1.8066	3063.9	601	3073.1
18	1.7584	3234.2	109	3248.9
8	1.7190	3384.1	620	3371.2
43	1.6901	3500.9	209	3501.8
16	1.6614	3622.9	542	3611.7
26	1.6319	3755.3	614	3743.5
18	1.5606	4106.0	517	4105.7
11	1.5490	4167.7	701	4168.8
18	1.5210	4322.6	2.1.10	4328.4
16	1.4623	4676.4	3.0.10	4665.5
5	1.4426	4805.2	1.0.11	4811.7
7	1.4316	4879.3	370	4888.2
3	1.4072	5050.0	732	5044.5
4	1.3672	5349.5	4.1.10	5339.7
7	1.3616	5393.9	800	5393.9
5	1.3562	5436.9	801	5432.9
18	1.3311	5644.1	812	5634.4
4	1.3047	5874.6	726	5873.3
5	1.2748	6153.4	830	6152.4
5	1.2507	6392.8	752	6392.9
8	1.1996	6949.1	911	6950.0
4	1.1191	7984.8	667	7982.5
$Yb_2Na_3Fe_5O_{12}$				
6	5.0218	396.5	201	384.0
6	5.0061	399.0	201	384.0
10	4.1717	574.6	004	560.3
37	3.8011	692.1	220	698.0
26	3.4030	863.5	310	872.5
4	3.2673	936.7	302	925.4
6	3.0692	1061.6	115	1050.0
28	2.7662	1306.9	215	1311.8
26	2.7382	1333.7	304	1345.6
100	2.6815	1390.7	400	1396.0
49	2.5873	1493.8	410	1483.3
8	2.5265	1566.6	330	1570.5
10	2.4977	1602.9	331	1605.6
10	2.2523	1971.2	226	1958.7
11	2.2358	2000.5	325	2009.8
14	2.1486	2166.2	217	2152.2
12	2.1023	2262.6	510	2268.6
4	1.9887	2528.5	520	2530.3
15	1.9043	2757.6	416	2744.0
15	1.8942	2787.1	440	2792.1
10	1.8685	2864.4	327	2850.3
15	1.8463	2933.6	442	2932.2
3	1.7647	3211.1	417	3199.3
28	1.6986	3465.9	427	3461.0

I/I_0, %	D, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{calc.}$
5	1.6632	3615.0	541	3612.4
5	1.6357	3737.4	418	3724.6
5	1.6217	3802.4	623	3805.3
5	1.5843	3984.1	517	3984.5
9	1.5606	4106.0	615	4103.8
15	1.5302	4270.7	700	4275.4
26	1.5199	4328.8	1.0.11	4324.7
12	1.4341	4862.3	607	4857.1
5	1.3644	5371.8	3.2.11	5371.7
6	1.3382	5584.2	800	5584.2
3	1.3039	5881.8	654	5882.7
6	1.2507	6392.8	831	6404.5
5	1.2102	6827.9	664	6842.5
6	1.1951	7001.5	841	7015.2
6	1.1851	7120.2	842	7120.3
5	1.1815	7163.6	665	7157.7
3	1.1685	7323.9	755	7332.2
3	1.1651	7366.7	649	7373.8
$Yb_2K_3Fe_5O_{12}$				
5	5.0925	385.6	201	376.5
3	4.5935	473.9	211	463.4
29	3.8066	690.1	213	694.2
22	3.4030	863.5	310	869.1
4	3.2177	965.9	223	955.0
3	3.0615	1066.9	205	1068.9
4	2.9655	1137.1	106	1125.5
29	2.8202	1257.3	322	1245.3
24	2.8253	1252.7	304	1243.8
100	2.6908	1381.1	400	1390.6
38	2.6108	1467.1	410	1477.5
5	2.4977	1602.9	412	1592.9
1	2.4103	1721.3	226	1733.9
9	2.2630	1952.6	414	1939.1
10	2.2403	1992.5	423	1997.9
14	2.1511	2161.1	500	2172.9
10	2.1023	2262.6	510	2259.8
3	2.0470	2386.6	512	2375.2
3	1.9852	2537.4	228	2541.7
18	1.9004	2768.9	440	2781.3
19	1.8917	2794.4	407	2804.3
11	1.8573	2899.0	0.0.10	2885.0
17	1.8361	2966.4	530	2955.1
4	1.7636	3215.1	533	3214.7
2	1.7301	3340.7	612	3331.2
30	1.6986	3465.9	329	3466.7
5	1.6669	3598.9	541	3592.3
5	1.6499	3673.8	1.1.11	3664.7
5	1.6304	3761.8	3.1.10	3754.1
6	1.5847	3982.0	356	3993.7
8	1.5606	4106.0	518	4106.2
14	1.5339	4250.2	1.0.12	4241.3
17	1.5302	4270.7	3.0.11	4273.1
23	1.5217	4318.6	1.1.12	4328.2
3	1.4771	4583.3	2.1.12	4589.0
12	1.4347	4858.2	2.2.12	4849.7
3	1.4042	5071.6	731	5069.9
5	1.3688	5337.5	651	5330.6

I/I_0, %	D, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{calc.}$
9	1.3408	5562.5	800	5562.5
3	1.3305	5649.0	810	5649.4
4	1.3072	5852.0	5.3.10	5840.1

I/I_0, %	D, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{calc.}$
7	1.2514	6386.1	831	6373.6
6	1.2111	6817.7	834	6806.4
8	1.1970	6979.3	6.2.11	6967.4

Further, using the data presented in Table 1 were determined types of syngony, the unit cell parameters of studied ferrites, values are given in Table 2 below.

Table 2

Crystallochemical characteristics of the compounds synthesized

Compound	Type of syngony	The lattice parameters, Å		V^0, Å^3	V^0., Å^3	Z	Density, g/cm^3	
		a	c				X-ray	pycn.
$Yb_2Li_3Fe_5O_{12}$	Tetragonal	10.89	16.00	1898.27	237.28	8	5.86	5.87
$Yb_2Na_3Fe_5O_{12}$	Tetragonal	10.71	16.90	1936.71	242.09	8	6.08	6.18
$Yb_2K_3Fe_5O_{12}$	Tetragonal	10.73	18.62	2142.08	2142.08	8	5.79	6.01

The correctness of indexing is confirmed by good correlation between the experimental and calculated values of the inverse square of the interplanar distance $10^4/d^2$ (Table 1) and by the consistency of the X-ray (ρ_{rent}) and pycnometric (ρ_{pikn}) densities. The ferrites' density has been measured 5 times by method described in [2] in 1 ml glass pycnometer. As an indifferent fluid is chosen tetrabromoethane. The results of X-ray diffraction analysis show that the synthesized ferrites have space group of distorted perovskite *Pm*3*m*.

The heat capacity of ferrites with an accuracy of ±10 % [3, 4] has been studied in the temperature interval 298.15–673 K on the calorimeter IT-400, which works by the method of periodic heat input. The values, experimental conditions and calibration of the calorimeter operation correspond to [5].

There are results of calorimetric investigations of the heat capacity in the Table 3, below.

Table 3

The experimental values of heat capacities $Yb_2M^I_3Fe_5O_{12}$ (M^I — Li, Na, K).

T, K	C_p±δ	$C°_p$±Δ	T, K	C_p±δ	$C°p$±Δ
$Yb_2Li_3Fe_5O_{12}$					
298	0.47±0.01	393±6	498	0.65±0.02	543±18
323	0.73±0.02	616±19	523	0.69±0.03	576±24
348	0.77±0.02	644±19	548	0.73±0.03	614±22
373	0.73±0.02	615±17	573	0.68±0.03	573±24
398	0.56±0.01	470±13	598	0.62±0.02	516±20
423	0.59±0.02	492±15	623	0.57±0.03	479±21
448	0.61±0.02	512±13	648	0.53±0.02	441±20
473	0.62±0.02	517±16	673	0.45±0.03	377±26
$Yb_2Na_3Fe_5O_{12}$					
298	0.24±0.01	213±7	498	0.58±0.03	514±29
323	0.54±0.04	475±32	523	0.55±0.03	487±25
348	0.58±0.04	513±32	548	0.53±0.04	470±32
373	0.61±0.03	537±30	573	0.50±0.03	441±25
398	0.56±0.03	492±28	598	0.43±0.03	382±30
423	0.61±0.04	545±31	623	0.38±0.02	340±20
448	0.61±0.04	545±35	648	0.33±0.07	290±59
473	0.59±0.04	525±33	673	0.31±0.02	279±16
$Yb_2K_3Fe_5O_{12}$					
298	0.75±0.02	632±15	498	0.62±0.04	520±37
323	1.03±0.04	859±38	523	0.68±0.04	574±34
348	1.07±0.05	895±40	548	0.74±0.04	621±34
373	1.04±0.07	868±61	573	0.68±0.03	572±23
398	0.85±0.04	711±34	598	0.50±0.02	417±18
423	0.75±0.03	629±25	623	0.46±0.02	382±16
448	0.67±0.03	557±22	648	0.44±0.02	370±16
473	0.57±0.02	477±18	673	0.44±0.02	365±20

The data in Table 3 show that the measurement errors of the heat capacity are placed within the permissible accuracy of the calorimeter. During the study of the heat capacity is found that a number of ferrites have sharp λ-shaped jump of heat capacity at the curve $C_p° \sim f(T)$, which is probably related to the presence of II-kind phase transitions [6, 7].

Result discussion

The equations of the temperature dependence on the heat capacity have been derived for the temperature interval 298.15–673 K (Table 4) and taking into account the experimental data on C°(T) was plotted the dependence of heat capacity on temperature (Figures 1–3).

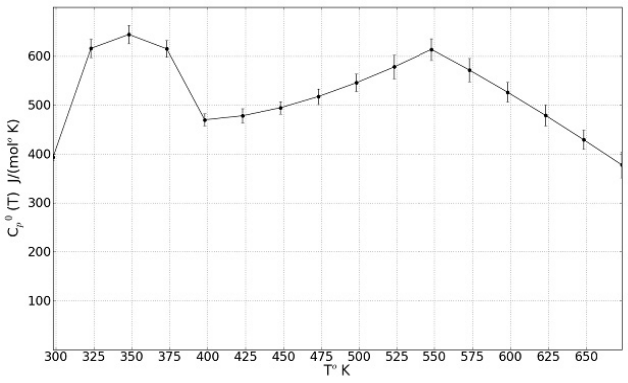

Figure 1. The dependence of the heat capacity on temperature $Yb_2Li_3Fe_5O_{12}$

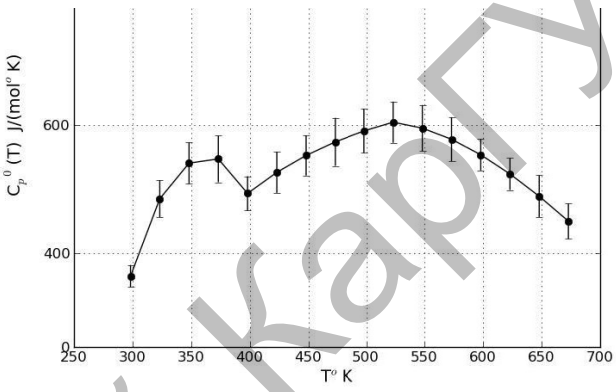

Figure 2. The dependence of the heat capacity on temperature $Yb_2Na_3Fe_5O_{12}$

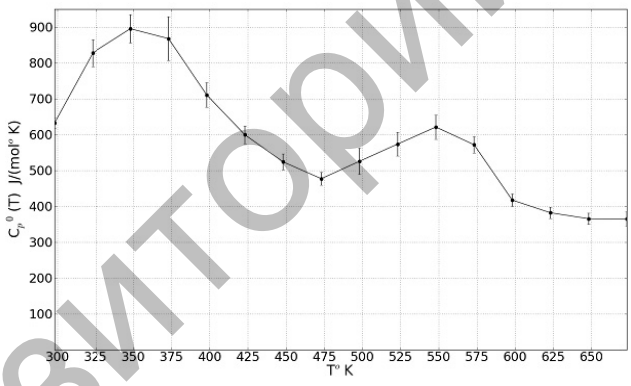

Figure 3. The dependence of the heat capacity on temperature $Yb_2K_3Fe_5O_{12}$

Table 4

Equations of the temperature dependence on heat capacities of ferrites composition $Yb_2M^I{}_3Fe_5O_{12}$ (M^I –Li, Na, K)

Compounds	Coefficients of the equation $C^0_p = a + b\cdot T + c\cdot T^{-2}$, J/(mol· K)			ΔT, K
	a	b	$c\cdot 10^{-5}$	
1	2	3	4	5
$Yb_2Li_3Fe_5O_{12}$	15313.52	–28.78	–5635.03	298.15–348.0
	14767.07	–26.53	–5924.00	348.0–398.0
	–899.62	2.34	693.92	398.0–548.0
	2324.33	–2.62	–818.38	548.0–673.0
$Yb_2Na_3Fe_5O_{12}$	6927.46	–11.51	–2917.77	298.15–373.0
	–11845.97	20.77	6448.45	373.0–423.0
	2290.05	–2.69	–1090.58	423.0–648.0
	580.64	–0.45	0.00	648.0–673.0

1	2	3	4	5
$Yb_2K_3Fe_5O_{12}$	9856.19	–16.96	–3704.68	298.15–373.0
	–2950.48	4.37	3045.118	373.0–473.0
	–314.80	1.77	–100.709	473.0–548.0
	30486.09	–36.20	–30105.5	548.0–598.0
	–4766.40	5.17	7480.219	598.0–673.0

The entropy of studied ferrites has been evaluated using the method of ion entropy increments [8]. Further, from the experimental data on $C_p°\sim f(T)$ and the calculated values of $S°(298,15)$ were derived the temperature dependence of thermodynamic functions $S°(T)$, $H°(T) - H°$ (298,15) of ferrites by the formulas [9–11]:

$$H^0(\mathrm{T}) - H^0(298.15) = \int_{298.15}^{T} C_p^0 dT ;$$

$$S^0(T) = S^0(298.17) + \int_{298.15}^{T} \frac{C_p}{T} dT ;$$

$$F^{xx}(T) = S^0(T) - \frac{H^0(T) - H^0(298.15)}{T}.$$

Error of the temperature dependence of the thermodynamic functions has been calculated based on the average random error of the heat capacity and entropy calculation accuracy (3.0 %) (Table 5).

Table 5

Thermodynamic functions of $Yb_2M^I_3Fe_5O_{12}$ (M^I –Li, Na, K)
[$C^0_p(T)$, $S^0(T)$, $\Phi^{xx}(T)$, $H^0(T) - H^0(298,15)$]

T, K	$C^0_p(T)$	$S^0(T)$	$\Phi^{xx}(T)$	$H^0(T) - H^0(298.15)$
1	2	3	4	5
$Yb_2Li_3Fe_5O_{12}$				
298.15	393±6	424.9 ± 42	424.9 ± 42	
323	616±19	466.7 ± 47	426.4 ± 43	12996.9 ± 818
348	644±19	514.6 ± 51	431.0 ± 43	29092.1 ± 1830
373	615±17	559.1 ± 56	438.2 ± 44	45103.3 ± 2837
398	470±13	594.8 ± 59	446.9 ± 45	58868.9 ± 3703
423	492±15	623.7 ± 62	456.5 ± 46	70695.4 ± 4447
448	512±13	651.5 ± 65	466.6 ± 47	82834.2 ± 5210
473	517±16	679.0 ± 68	477.1 ± 48	95467.7 ± 6005
498	543±18	706.3 ± 71	488.0 ± 49	108741.6 ± 6840
523	576±24	733.8 ± 73	499.1 ± 50	122773.9 ± 7722
548	614±22	761.6 ± 76	510.4 ± 51	137660.8 ± 8659
573	573±24	788.1 ± 79	521.9 ± 52	152479.9 ± 9591
598	516±20	811.5 ± 81	533.6 ± 53	166203.7 ± 10454
623	479±21	832.1 ± 83	545.1 ± 55	178766.4 ± 11244
648	441±20	850.0 ± 85	556.6 ± 56	190112.7 ± 11958
673	377±26	865.2 ± 87	567.8 ± 57	200195.3 ± 12592
$Yb_2Na_3Fe_5O_{12}$				
298.15	213±7	485.2 ± 49	485.2 ± 49	
323	475±32	510.9 ± 51	486.1 ± 49	8024.9 ± 505
348	513±32	545.9 ± 55	489.1 ± 49	19776.3 ± 1244
373	537±30	582.7 ± 58	494.2 ± 49	33033.0 ± 2078
398	492±28	615.5 ± 62	500.8 ± 50	45672.3 ± 2873
423	545±31	646.7 ± 65	508.5 ± 51	58458.3 ± 3677
448	545±35	678.0 ± 68	517.1 ± 52	72087.5 ± 4534
473	525±33	707.2 ± 71	526.4 ± 53	85559.3 ± 5382
498	514±29	734.2 ± 73	536.1 ± 54	98644.7 ± 6205
523	487±25	758.7 ± 76	546.2 ± 55	111158.4 ± 6992
548	470±32	780.8 ± 78	556.4 ± 56	122949.0 ± 7733
573	441±25	800.3 ± 80	566.6 ± 57	133891.5 ± 8422
598	382±30	817.4 ± 82	576.8 ± 58	143881.8 ± 9050

1	2	3	4	5
623	340±20	832.0 ± 83	586.7 ± 59	152832.4 ± 9613
648	290±59	844.4 ± 84	596.4 ± 60	160669.4 ± 10106
673	279±16	855.1 ± 86	605.8 ± 61	167787.9 ± 10554
$Yb_2K_3Fe_5O_{12}$				
298.15	632±15	424.9 ± 42	424.9 ± 42	
323	859±38	484.2 ± 48	427.1 ± 43	18444.6 ± 1160
348	895±40	549.1 ± 55	433.5 ± 43	40209.4 ± 2529
373	868±61	610.7 ± 61	443.4 ± 44	62420.1 ± 3926
398	711±34	661.7 ± 66	455.6 ± 46	82041.5 ± 5160
423	629±25	701.4 ± 70	469.0 ± 47	98331.8 ± 6185
448	557±22	733.5 ± 73	482.9 ± 48	112305.8 ± 7064
473	477±18	760.6 ± 76	496.8 ± 50	124763.7 ± 7848
498	520±37	786.4 ± 79	510.7 ± 51	137297.3 ± 8636
523	574±34	813.3 ± 81	524.6 ± 52	151038.7 ± 9500
548	621±34	841.2 ± 84	538.4 ± 54	165974.0 ± 10440
573	572±23	868.2 ± 87	552.1 ± 55	181123.2 ± 11393
598	417±18	889.7 ± 89	565.8 ± 57	193685.7 ± 12183
623	382±16	857.4 ± 86	575.1 ± 58	175922.6 ± 11066
648	370±16	872.1 ± 87	586.2 ± 59	185229.5 ± 11651
673	365±20	885.9 ± 89	597.1 ± 60	194326.4 ± 12223

Thus, for the first time three ferrites have been synthesized, identified types of syngony, unit cell parameters, the X-ray and pycnometry densities. For the first time in the temperature interval of 298.15–673 K have been investigated the temperature dependences of the heat capacity of ferrites $Yb_2Li_3Fe_5O_{12}$. $Yb_2Na_3Fe_5O_{12}$, $Yb_2K_3Fe_5O_{12}$. The equations of the temperature dependence of the heat capacity of ferrites have been derived their standard entropy and the temperature dependence of thermodynamic functions $C_p°(T)$, $S°(T)$, $H°(T) - H°(298.15)$ have been calculated in the interval of 298.15–673 K. During the study of the heat capacity is found that a number of ferrites have sharp λ-shaped jump of heat capacity at the curve $C_p° \sim f(T)$, which is probably related to the presence of II-kind phase transitions and no doubt is of particular interest for inorganic materials science.

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$Yb_2M^I_3Fe_5O_{12}$ (M^I — Li, Na, K) қосылыстарының синтезі, рентгенографиялық және термодинамикалық зерттеуі

Литий, натрий, калий, лантан, темір оксидтерінен қатты фазалы әдіс арқылы жаңа қосылыстар $Yb_2M^I_3Fe_5O_{12}$ (M^I — Li, Na, K) синтезделіп алынды. Рентгенфазалық талдау әдісі арқылы қосылыстардың тетрагоналды сингонияда кристалданатыны анықталды. Қосылыстардың жылу сыйымдылығы 298,15–673 К температуралар аралығында өлшеніп, $C_p^0 \sim f(T)$ тәуелділік графиктерінде λ-тәріздес секірулердің болатыны байқалған. Бұл жағдай екінші реттік фазалық ауысуларға байланысты болуы мүмкін деген тұжырым жасалған.

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Синтез, рентгенографическое и термодинамическое исследования соединений $Yb_2M^I_3Fe_5O_{12}$(M^I — Li, Na, K)

Твердофазным способом из оксидов лития, натрия, калия, лантана и железа синтезированы новые соединения $Yb_2M^I_3Fe_5O_{12}$ (M^I — Li, Na, K). Методом рентгенофазового анализа определено, что соединение кристаллизуется в тетрагональной сингонии. В интервале 298,15–673 К измерена теплоемкость соединений и обнаружено, что на графиках зависимостей $C_p^0 \sim f(T)$ наблюдаются λ-образные скачки, связанные, вероятно, с фазовыми переходами второго рода.