

Original papers

SOME FINDINGS ON BLUE-EMITTING
LUMINOPHORE BASED ON (Ba, Eu²⁺)Mg ALUMINATE

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The currently used blue-emitting luminophore was proved not to contain the (Ba, Eu)Mg₂Al₁₆O₂₇ aluminate as is often stated in the literature. An initial mixture, corresponding by its composition to the formula given above, yields a mixture of α -Al₂O₃ and the aluminate Ba_{0.72(1-x)}Eu_{0.72x}O_{0.28}Mg_{1.44}Al_{10.08}O₁₇(Ba_{1-x}Eu_xMg₂Al₁₄O₂₄) with a structure of β -Al₂O₃. The structure was found to contain arranged O_{Ba} atoms and was compared to the structure of similar aluminates. The luminophore is conveniently prepared by heating the coprecipitated hydroxides or hydrated oxides at 800–1000 °C and subsequent igniting in a mildly reducing atmosphere (N₂ + H₂) at high temperatures. In excess of atmospheric oxygen, the Eu³⁺ containing aluminate being formed is stabilized by additional O atoms built into its structure. The luminophore exhibits maximum emission in the blue region at an Eu²⁺ content of $x = 0.11$.

INTRODUCTION

Luminophores emitting white light consist of three coloured components (red, green and blue) transforming radiation of 253.7 nm wavelength, generated by electric discharge in mercury vapours, to light within very narrow bands of visible light wavelengths [1]. The component emitting in the blue region and containing Eu²⁺ is an aluminate whose composition is mostly given as (Ba, Eu)Mg₂Al₁₆O₂₇ [2–5]. The advantages of the substance include a suitable wavelength of excitation, emission at 452 nm and attainment of up to 100 % theoretical efficiency [3]. The emission stability of Eu²⁺ also allows small-diameter fluorescence tubes to be manufactured.

However, according to Stevels [6–8], an aluminate of the given composition with a hexagonal magnetoplumbite structure [9] and with a coordination of 12 oxygen atoms around the larger atom in position 2d cannot exist owing to the size of Ba²⁺, and all barium aluminates from the composition region considered therefore crystallize in the deformed structure of β -Al₂O₃. The author sets forth BaMgAl₁₀O₁₇ as an ideal representative of this structure of barium aluminates. In the case of non-stoichiometry, the structures of the β -Al₂O₃ type with coordination of 10 oxygen atoms around the larger atom in position 2d can be achieved so that some of the O_{Ba} oxygen atoms take up the position of barium at 2d while another oxygen atom is placed in position 2c. These layers are separated by spinel blocks which are identical with those of the magnetoplumbite structure [9]. The maximum content of O_{Ba} corresponds to the formula Ba_{0.72}O_{0.28}Mg_{1.44}Al_{10.08}O₁₇ (BaMg₂Al₁₄O₂₄), which is in agreement with the data given in [3, 4], namely

that diffractions of α - Al_2O_3 were found in diffraction patterns of products obtained in the preparation of aluminates with the assumed composition $\text{BaMg}_2\text{Al}_{16}\text{O}_{27}$.

According to Stevels [8], however, the presence of α - Al_2O_3 in the products does not reduce the intensity of Eu^{2+} emission, which is made use of in some procedures [2, 3, 10, 11] for the preparation of luminophores containing the aluminate in question. The process involved is mostly a dry one [2—14], based on heating a mechanical mixture of the starting components at 1200—1600 °C in a mildly reducing atmosphere.

Several practical values of the Eu^{2+} content in the host lattice of this aluminate are given, for example $\text{Ba}_{0.9}\text{Eu}_{0.1}\text{Mg}_2\text{Al}_{16}\text{O}_{27}$ [2, 4] and $\text{Ba}_{0.86}\text{Eu}_{0.14}\text{Mg}_2\text{Al}_{16}\text{O}_{27}$ [3].

The above survey indicates that some data on this luminophore, which is used extensively in practice, are rather ambiguous, and this is why the present authors undertook to deal with the preparation of the aluminate and the composition and structure of the products obtained. In the preparation, use was made of the procedure described quantitatively for the aluminate $\text{Ce}_{0.67}\text{Tb}_{0.33}\text{MgAl}_{11}\text{O}_{19}$ [3], according to which the co-precipitated hydroxides are first heated at 700 °C and only then at higher temperatures. Since also the information on the optimum content of Eu^{2+} in the host lattice of the aluminate is not explicit with respect to the maximum blue emission, the problem was also dealt with.

EXPERIMENTAL

$\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and Eu_2O_3 were the initial substances used in the preparation. The reagents of AR purity were additionally repurified electrolytically. The hydrated oxides or hydroxides of the composition required were coprecipitated from the mixture of solutions by an ammonia solution. The specimens were heated in Marsh's tubular furnace (Elektrodružstvo Prague).

The diffraction patterns were made by the Geigerflex diffractometer (Rigaku Denki, Japan), Cu anode, Ni filter, GM detector. The Powder program implemented by the authors was used to precision the lattice parameters.

The brightness was measured in the laboratory of Concern Corporation Tesla Holešovice with the use of the PR 1980 detector (Photo Research, USA). Comparisons were made with commercial luminophores Silvania 246 Blue and Silvania 2340 Red. No details about their manufacturing process could be obtained; the same applies to the L 47 luminophore of Soviet make.

DISCUSSION

Preparation of (Ba, Eu)—Mg aluminate specimens

The precipitate of hydroxides or hydrated oxides, whose composition corresponded to the formula for $\text{Ba}_{0.9}\text{Eu}_{0.1}\text{Mg}_2\text{Al}_{16}\text{O}_{27} = \frac{1}{0.72}\text{Ba}_{0.65}\text{Eu}_{0.07}\text{O}_{0.28}\text{Mg}_{1.44} \cdot \text{Al}_{10.08}\text{O}_{17} + \text{Al}_2\text{O}_3$, was always amorphous in character after heating at 800—1000 °C, as shown by the diffraction patterns. Heating at 1200—1500 °C in a mildly reducing atmosphere ($\text{N}_2 + \text{H}_2$) yielded substances with identical diffraction patterns, and contained diffractions of aluminate with the structure of β - Al_2O_3 , and diffractions of α - Al_2O_3 .

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This method allows the product which, according to [4], was prepared by igniting a mechanical mixture at 1400 °C for 2 hours, to be produced within 15 minutes by igniting at only 1200 °C. According to [2], heating of the mechanical mixture at 1200 °C yielded products which showed diffractions of MgAl₂O₄.

Determination of the relative intensities of Eu²⁺ emission in the structure of the aluminate prepared showed, in terms of increasing temperature and time of heating, first an increase and later on a decrease, which can be explained by structural changes in the host lattice of the aluminate having the structure of β-Al₂O₃, as no patterns of any possible products of subsequent reactions were found (Table I).

Table I

Relative emission intensity of Eu²⁺ in the host lattice of Ba_{0.65}Eu_{0.07}O_{0.28}Mg_{1.44}Al_{10.08}O₁₇ vs. the time and temperature of ignition, compared to the Sylvania 246 Blue luminophore

Temperature [°C]	Time of ignition [h]					
	1/4	2/4	3/4	4/4	5/4	6/4
1250	71.8	79.2	82.3	82.0	83.0	82.0
1300	80.3	85.0	85.3	88.0	90.8	86.0
1350	80.1	85.3	85.0	86.2	88.9	90.0
1400	79.0	90.9	98.2	88.0	81.0	84.7
1450	77.5	94.2	88.7	77.7	77.0	76.2

The effect of reaction temperature on the aluminate structure

The samples prepared in the same way but by ignition at 1200—1400 °C in air (Table II) exhibited a faintly pink emission of Eu³⁺. The diffraction pattern of a sample heated at 1300 °C was identical with that of samples obtained by ignition in a mildly reducing atmosphere, and included diffractions of aluminate with a structure of β-Al₂O₃, as well as diffractions of α-Al₂O₃.

Table II

Relative pink emission intensity of Eu³⁺ in the host lattice of Ba_{0.65}Eu_{0.07}O_{0.28}Mg_{1.44}Al_{10.08}O₁₇ vs. the temperature and time of ignition, compared to the Sylvania 2340 Red luminophore. The sample were prepared in access of air

Temperature [°C]	Time of ignition [h]				
	1/4	2/4	3/4	4/4	5/4
1000	—	39.4	—	42.1	—
1100	39.8	39.8	—	—	—
1200	41.8	40.3	—	—	—
1200	43.2	42.3	41.5	42.1	40.7
1300	39.6	37.6	38.5	37.6	—
1300	38.7	37.1	36.5	35.9	—
1400	35.8	—	—	—	—

For the sake of comparison, luminophore L 47 was heated in air at 1500 °C, a pink-emitting product was also obtained. The diffraction patterns indicated to changes in the structure due to the heating.

In the case of products prepared by heating in air, reheating in a mildly reducing atmosphere failed the Eu^{3+} to be reduced back to Eu^{2+} .

The effect of the reducing atmosphere on the quality of europium emission in the host lattice of the aluminate is determined by its structure in which the Ba^{2+} atom is surrounded by ten oxygen atoms. According to [6], this coordination is due to the large diameter of Ba^{2+} , 0.152 Å [16]. The smaller value of the ionic radius of Eu, 0.135 Å, [16] therefore allows for a change in coordination to take place in its neighbourhood, together with the building of additional O atoms into the structure of this aluminate, similarly to the case of the magnetoplumbite-type structure. Similar changes have already been described [14, 15]. During the preparation of the aluminate in access of air oxygen, additional oxygen atoms must therefore be built into the structure of $\beta\text{-Al}_2\text{O}_3$ and stabilized by the change in the oxidation number of Eu^{2+} to Eu^{3+} .

x	Intensity of Eu^{2+} emission [%]
0.07	74.0
0.08	75.3
0.09	76.5
0.10	78.1
0.11	81.7
0.12	78.1
0.13	74.0
0.15	71.0
0.17	66.7

Table III

Relative emission intensity of Eu^{2+} in the host lattice of aluminate vs. the content of Eu^{2+} activator, compared to the Sylvania 246 Blue luminophore. The samples of $\text{Ba}_{0.72(1-x)}\text{Eu}_{0.72x}\text{O}_{0.28}\text{Mg}_{1.44}\text{Al}_{10.08}\text{O}_{17}$ were prepared by ignition at 1350 °C for 15 minutes

Emission intensity of Eu^{2+} in the host lattice of the aluminate in terms of Eu^{2+} content

Table III indicates that the maximum emission of Eu^{2+} takes place at the ratio $\text{Ba} : \text{Eu} = 8 : 1$, which corresponds to the composition $\text{Ba}_{0.64}\text{Eu}_{0.08}\text{O}_{0.28}\text{Mg}_{1.44} \cdot \text{Al}_{10.08}\text{O}_{17}$. This ratio is in agreement with the hexagonal symmetry of this aluminate, and with the published [8] maximum content of $\text{O}_{\text{Ba}} \text{Ba} : \text{O}_{\text{Ba}} = 72 : 28$.

Structure of the aluminate samples prepared

The diffraction patterns of all the reaction products were identical and contained only the diffraction of aluminate with the structure of $\beta\text{-Al}_2\text{O}_3$ and diffractions of $\alpha\text{-Al}_2\text{O}_3$. Table IV shows that the diffractions of this aluminate respect the systematic extinctions in space group $\text{P}6_3/\text{mmc} : h h l, l = 2n + 1$. The diffraction at 9.60θ remained unindexed, but using the lattice parameters $a' = a$ and $c' = 2c$, can be furnished with diffraction indices 1 0 3 with $\theta_{\text{calc}} = 9.59^\circ$, which is indicative of an arrangement of O_{Ba} atoms in the structure. A similar diffraction was observed in the pattern of luminophore L 47 where, however, the 1 1 3 and 1 1 7 diffractions of the aluminate do not respect the systematic extinctions in

Table IV

Precisioned values of lattice parameters of our sample of (Ba, Eu)Mg aluminate with a structure of β -Al₂O₃. Use was made of the Powder program (written at the Institute of Chemical Technology Prague). Hexagonal system, $a = 0.5612 \pm 0.0008$, $c = 2.2542 \pm 0.0032$ nm, radiation CuK α_1 , $\lambda = 0.154051$ nm. The samples also exhibited diffractions of α -Al₂O₃. The diffraction at 9.60° can be indexed with diffraction indices 103 with $\theta = 9.58^\circ$ using lattice parameters $a' = a$, $c' = 2c$

			Structure of β -Al ₂ O ₃			Structure of α -Al ₂ O ₃		
$\theta_{\text{cont.}} [^\circ]$	I/I_0	$d [\text{Å}]$	hkl	$\theta_{\text{calc.}} [^\circ]$	diff	hkl	$\theta_{\text{tab}} [^\circ]$	I/I_0
9.10	20	4.870	100	9.12	-0.02			
9.30	25	4.766	101	9.33	-0.03			
9.60	25	4.619	103	9.58	0.02	$a' = a$	$c' = 2c$	
9.93	55	4.467	102	9.94	-0.01			
10.85	10	4.092	103	10.88	-0.03			
12.05	10	3.690	104	12.08	-0.03			
12.80	70	3.477				102	12.79	75
13.45	10	3.312	105	13.48	-0.03			
15.87	60	2.817	110	15.93	-0.06			
16.45	10	2.720	112	16.43	0.02			
16.70	55	2.680	107	16.67	0.03			
17.60	100	2.547				104	17.57	90
17.83	45	2.516	114	17.86	-0.03			
18.55	60	2.421	201	18.59	-0.04			
18.90	45	2.274				110	18.89	40
19.45	15	2.313	203	19.46	-0.01			
20.20	5	2.231	{ 204 109	{ 20.19 20.24	{ 0.01 -0.04			
21.10	20	2.140	205	21.11	-0.01			
21.70	100	2.083				113	21.68	100
22.17	25	2.041	206	22.18	-0.01			

space group P6₃/mmc (Table V), which implies a different arrangement of O_{Ba} atoms and an influence of the preparation procedure on the structure in the proximity of the mirror plane determining the quality of Eu²⁺ emission in the host lattice of this aluminate.

Table VI gives a survey of the lattice parameters of Ba—Mg aluminates having the structure of β -Al₂O₃. These aluminates constitute two groups differing in the values of lattice parameters and the cell volumes.

The first group comprises aluminates prepared by the dry method, whose lattice parameters and cell volumes are larger than those of the representative of the structural type BaMgAl₁₀O₁₇ [6]. The other group includes aluminates with lattice parameters and cell volumes smaller than those of the β -Al₂O₃ structural representative BaMgAl₁₀O₁₇, which is in agreement with the arrangement of O_{Ba} atoms, proved on the aluminates contained in the L 47 luminophore and in the samples prepared within the framework of the present study. This arrangement can be compared with that in the structure of the BaAl₁₂O₁₉ aluminate, established by diffraction on monocrystals [6], where the basic cell has a three-fold volume, two of the subcells have the ideal structure of β -Al₂O₃, and substitution of Ba atoms by O_{Ba} atoms takes place in the third layer.

Table V

Precisioned values of lattice parameters of (Ba, Eu) Mg aluminate in the L 47 luminophore, obtained with the use of the Powder program. Hexagonal system, $a = 0.5604 \pm 0.0005$, $c = 2.2597 \pm 0.0018$ nm, $a' = 1.1209 \pm 0.0011$, $c' = 4.5218 \pm 0.0037$ nm. $\text{Cu}_{K\alpha}$ radiation

$\theta_{\text{cont}} [^\circ]$	I/I_0	$d [\text{Å}]$	hkl	$\theta_{\text{calc}} [^\circ]$	diff	hkl	$\theta_{\text{calc}} [^\circ]$	diff
3.90	15	11.32	002	3.91	-0.01	004	3.91	-0.01
7.85	5	5.640	004	7.84	0.01	008	7.83	0.02
9.13	25	4.854	100	9.13	-0.00	200	9.13	-0.00
9.35	60	4.741	101	9.34	0.01	202	9.34	0.01
9.60	15	4.619				203	9.60	0.00
9.95	100	4.458	102	9.95	0.00	204	9.94	0.01
10.90	20	4.073	103	10.88	0.02	206	10.88	0.02
11.80	< 5	3.776	006	11.80	-0.03	0012	11.79	-0.02
12.07	25	3.684	104	12.08	-0.01	208	12.07	-0.00
12.80	< 5	3.477				{ 214	12.75	0.05
						{ 209	12.75	0.05
13.45	20	3.312	105	13.47	-0.02	2010	13.46	-0.01
15.00	< 5	2.976	106	15.00	-0.00	2012	15.00	0.00
15.85	< 5	2.820	008	15.82	0.03	0016	15.82	0.03
15.95	70	2.803	110	15.96	-0.01	220	15.95	-0.00
16.45	25	2.720	112	16.45	-0.00	224	16.45	-0.00
16.65	95	2.688	107	16.65	-0.00	2014	16.65	0.00
17.10	25	2.620	113	17.06	0.04	226	17.05	0.05
17.85	80	2.513	114	17.87	-0.02	228	17.87	-0.02
18.10	25	2.291				317	18.06	0.04
18.60	35	2.415	201	18.62	-0.02	402	18.61	-0.01
19.45	20	2.313	203	19.48	-0.03	406	19.48	-0.03
19.65	10	2.291				{ 1118	19.63	0.02
						{ 3014	19.69	-0.04
19.90	10	2.263	0010	19.93	-0.03	0020	19.92	-0.02
			{ 109	20.21	-0.01	{ 2018	20.20	0.00
20.20	15	2.231	{ 204	20.21	-0.01	{ 408	20.21	-0.01
21.10	40	2.140	205	21.12	-0.02	4010	21.11	-0.01
21.40	15	2.111	117	21.35	0.05	2214	21.34	0.06
22.20	40	2.039	206	22.19	0.01	4012	22.18	0.02

CONCLUSION

In agreement with the conclusion reached by Stevels, it has been proved that the aluminate $\text{BaMg}_2\text{Al}_{16}\text{O}_{27}$ cannot exist, and that the reaction product prepared from an initial co-precipitate of hydrated oxides and hydroxides corresponding to this composition, is in fact a mixture of $\alpha\text{-Al}_2\text{O}_3$ and the aluminate $\text{Ba}_{0.72(1-x)} \cdot \text{Eu}_{0.72x}\text{O}_{0.28}\text{Mg}_{1.44}\text{Al}_{10.08}\text{O}_{17}(\text{Ba}_{(1-x)}\text{Eu}_x\text{Mg}_2\text{Al}_{14}\text{O}_{24})$, in the structure of which the O_{Ba} atoms were found to be arranged.

It was shown that the aluminate cannot be prepared in access of atmospherical oxygen. The use of a mildly reducing atmosphere has structural reasons, because treatment in access of air brings about irreversible building of oxygen atoms into the structure, which is stabilized by the change of the oxidation state of Eu^{2+} to Eu^{3+} .

The optimum content of the Eu^{2+} ion in the host lattice of the aluminate from the standpoint of emission intensity was precisioned at $\text{Ba}_{0.64}\text{Eu}_{0.08}\text{Mg}_{1.44}\text{Al}_{10.08}\text{O}_{17}$.

Table VI

Lattice parameters of the BaMg aluminates under discussion, having the β -Al₂O₃ structure

	a [nm]	c [nm]	V [nm]	
1	0.562 24 ± ± 0.000 01	2.262 68 ± ± 0.000 06	0.619 44 ± ± 0.000 02	BaMgAl ₁₀ O ₁₇ , ref. [6], a, c values
	0.562 6 ± ± 0.0003	2.266 3 ± ± 0.001 1	0.621 22 ± ± 0.000 56	ref. [2], values of d, I/I ₀ containing diffractions of MgAl ₂ O ₄ , preparation from mechanical mixture, arrangement of atoms not found
	0.562 8 ± ± 0.000 5	2.268 0 ± ± 0.000 5	0.622 13 ± ± 0.000 79	ref. [4], values of a, c, contains diffractions of α -Al ₂ O ₃ , preparation from mechanical mixture, arrangement of O _{Ba} atoms not found
	0.560 6 ± ± 0.001 1	2.261 7 ± ± 0.003 6	0.615 6 ± ± 0.002 0	our measurements of commercial Sylvania 246 Blue, contains diffractions of α -Al ₂ O ₃ , arrangement of O _{Ba} not found
2	0.560 4 ± ± 0.000 5	2.259 7 ± ± 0.001 8 2×	0.614 6 ± ± 0.000 9 2×	our measurements of commercial L 47 luminophore, arrangement of O _{Ba} atoms
	0.561 2 ± ± 0.000 8	2.254 2 ± ± 0.003 2 2×	0.614 8 ± ± 0.001 5 2×	our samples, arrangement of O _{Ba} atoms, contains diffractions of α -Al ₂ O ₃ , preparation from coprecipitated hydrated oxides

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НЭКТЕРЭ ПОЗНАТКЫ О МОДРЕ ЭМИТУЈІЦІМ ЛУМИНОФОРУ
НА БАЗІ (Ba, Eu²⁺)-Mg-HLINITANU

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Bylo studováno složení produktu vzniklého zřídáním výchozí směsi hydratovaných hydroxidů a oxidů, jejíž složení odpovídalo vzorci $Ba_{1-x}Eu_xMg_2Al_{16}O_{27}$ a který nalézá uplatnění jako modře emitující složka luminoforu pro nízkotlaké rtuťové lampy. Bylo prokázáno, že v soulase s pracemi Stevelse, podle nichž neexistuje hlinitan o složení $BaMg_2Al_{16}O_{27}$, je reakční produkt směsi $\alpha-Al_2O_3$ a hlinitanu $Ba_{0,72(1-x)}Eu_{0,72x}O_{0,28}Mg_{1,44}Al_{10,08}O_{17}$ ($Ba_{1-x}Eu_xMg_2Al_{16}O_{24}$). Ve struktuře tohoto hlinitanu byla rentgenograficky prokázána uspořádanost těch atomů kyslíku, které ve struktuře zastupují atomy baria. Tento hlinitan nelze připravovat za přístupu vzdušného kyslíku. Použití slabě redukční atmosféry při přípravě má strukturální důvody, neboť při přípravě za přístupu vzduchu dochází k nevratnému zabudování dalších atomů kyslíku do struktury, které je umožněno menším iontovým poloměrem Eu^{3+} zastupujícím barium ve struktuře a které je stabilizováno změnou oxidačního stavu Eu^{2+} na Eu^{3+} . Byl upřesněn optimální obsah iontu Eu^{2+} v hostitelské mřížce tohoto hlinitanu z hlediska maximální intenzity emise jako $Ba_{0,64}Eu_{0,08}Mg_{1,44}Al_{10,08}O_{17}$.

НЕКОТОРЫЕ ДАННЫЕ ОТНОСИТЕЛЬНО ЛЮМИНОФОРА
НА БАЗЕ (Ba, Eu²⁺) — Mg-АЛЮМИНАТА,
ИСПУСКАЮЩЕГО СИНИЕ ЛУЧИ

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Исследовали состав продукта, полученного обжигом исходной смеси гидратированных гидроксидов и оксидов, состав которой отвечал формуле $Ba_{1-x}Eu_xMg_2Al_{16}O_{27}$. Данный продукт находит широкое применение в качестве составной части люминофора, испускающего синие лучи. Такой люминофор предназначен для производства ртутных ламп низкого давления. Было доказано, что в соответствии с работами Стевельса, согласно которым не существует алюминат составом $BaMg_2Al_{16}O_{27}$, продукт реакции является смесью $\alpha-Al_2O_3$ и алюмината $Ba_{0,72(1-x)}Eu_{0,72x}O_{0,28}Mg_{1,44}Al_{10,08}O_{17}$ ($Ba_{1-x}Eu_xMg_2Al_{16}O_{24}$). В структуре данного алюмината было рентгенографически доказано упорядочение тех атомов кислорода, которые в структуре замещают атомы бария. Упомянутый алюминат нельзя получать при доступе кислорода воздуха. Использование слабо восстановительной среды при получении имеет структурные причины, так как при получении при доступе воздуха происходит необратимое встраивание дальнейших атомов кислорода в структуру. Возможность встраивания обеспечивается меньшим ионным радиусом Eu^{3+} , замещающим барий в структуре и стабилизируется изменением степени окисления Eu^{2+} в Eu^{3+} . Кроме того уточняется оптимальное содержание ионов Eu^{2+} в решетке приводимого алюмината с точки зрения максимальной интенсивности эмиссии как $Ba_{0,64}Eu_{0,08}Mg_{1,44}Al_{10,08}O_{17}$.