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CRYSTAL STRUCTURE AND MAGNETIC PROPERTIES OF $Gd_{1-x}La_xMO_3$ (M = Sc, In, Ga) SOLID SOLUTIONS

$Gd_{1-x}La_xScO_3$, $Gd_{1-x}La_xInO_3$ and $Gd_{1-x}La_xGaO_3$ solid solutions were synthesized by the ceramic method, their crystal structure and magnetic properties were investigated. It has been established that the range of solid solutions $Gd_{1-x}La_xScO_3$ ($0.0 < x \le 1.0$), $Gd_{1-x}La_xInO_3$ ($0.2 \le x \le 1.0$) and $Gd_{1-x}La_xGaO_3$ ($0.5 \le x \le 1.0$) with the structure of orthorhombically distorted perovskite was formed. The effective magnetic moment of the Gd^{3+} ion ($\mu_{ef,\,Gd^{3+}}$) for $Gd_{1-x}La_xScO_3$ solid solutions with $x = 0.10$; 0.50 is equal to 7.76 μ_B; 7.61 μ_B, respectively and it is slightly lower than the theoretical value $\mu_{ef.\ theor,\,Gd^{3+}} = 7.94\ \mu_B$, and for $x = 0.75$; 0.90 it is practically equal to the theoretical value. For $Gd_{1-x}La_xGaO_3$ solid solutions with $x = 0.8$; 0.9 the effective magnetic moment $\mu_{ef,\,Gd^{3+}}$ is 7.90 μ_B; 7.99 μ_B, respectively, and it is close to the theoretical value 7.94 μ_B. For $Gd_{1-x}La_xInO_3$ solid solutions the effective magnetic moment $\mu_{ef,\,Gd^{3+}}$ varies without certain dependence in the interval from 6.91 μ_B to 7.54 μ_B and is lower than theoretical value $\mu_{эф.\ теор,\,Gd^{3+}}$.

Introduction. Compounds of rare earth and other metals oxides with perovskite structure occupy a special place among modern and promising materials widely used in electronics and chemical industry [1–3].

In particular, lanthanum and other rare earth elements (Ln) scandates, gallates, indates, aluminates and their solid solutions with perovskite structure ($LnMO_3$, M = Al, Sc, Ga, In) are promising materials for of active laser elements [4–6].

Indium and gallium compounds containing rare-earth ions are also good photo- and cathodoluminescent phosphors [7, 8], which can be used for white LEDs.

The use of $LaScO_3$ as phosphor matrix when doped by other rare-earth ions was found to be perspective, since $LaScO_3$ has no intrinsic absorption in the visible region of the spectrum [9].

If paramagnetic ions of rare earth elements in $LnAlO_3$, $LnGaO_3$, $LnInO_3$ are partially substituted by isovalent diamagnetic La^{3+} ions there will be observed magnetic dilution of rare-earth ions, resulting in decrease in their interaction and in growing influence of the crystal field on the spin-orbital interaction. That leads to a so-called "partial freezing" of rare earth ions orbital moment. As far as we know such studies are absent in literature despite their great scientific and practical importance.

The aim of this work is to establish patterns of influence of the magnetic dilution of paramagnetic Gd^{3+} ions by diamagnetic La^{3+} ions on the magnetic susceptibility and the effective magnetic moment of Gd^{3+}ions in $Gd_{1-x}La_xScO_3$, $Gd_{1-x}La_xInO_3$, $Gd_{1-x}La_xGaO_3$ solid solutions as promising new materials for electronics and chemical industries.

Experimental technique Polycrystalline samples of $Gd_{1-x}La_xScO_3$ ($x = 0.0$–1.0), $Gd_{1-x}La_xInO_3$ ($x = 0.0$–1.0) $Gd_{1-x}La_xGaO_3$ ($x = 0.3$–1.0) solid solutions being the subject of study, were prepared by ceramic method from oxides Gd_2O_3, La_2O_3, Sc_2O_3, In_2O_3, Ga_2O_3.

Hygroscopic lanthanum oxide had been calcined at 1,173 K for 1 hour before weighting. Stoichiometric amounts of starting materials were mixed and milled in a planetary mill Pulverizette 6 with addition of ethanol.

The resulting mixture was pressed under a pressure of 50–75 MPa into tablets of 25 mm diameter and 5.7 mm height, and then sintered in air in an electric resistance furnace. After the first sintering the tablets were crushed, milled, pressed into bars of 30 mm length and 5×5 mm^2 cross section. Bars were sintered in air in an electric resistance furnace. Parameters of synthesis are shown in Table. 1.

Table 1

Procedures of synthesis of $Gd_{1-x}La_xScO_3$, $Gd_{1-x}La_xInO_3$ and $Gd_{1-x}La_xGaO_3$ solid solutions

System	1-st sintering	2-d sintering	3-rd sintering
$Gd_{1-x}La_xScO_3$	1,523 K, 4 h	1,623 K, 1 h	1,523 K, 4 h
$Gd_{1-x}La_xGaO_3$	1,523 K, 2 h	1,523 K, 2 h	–
$Gd_{1-x}La_xInO_3$ $0 \le x \le 0.7$	1,523 K, 5 h	1,523 K, 5 h	1,773 K, 2 h
$Gd_{1-x}La_xInO_3$ $0.8 \le x \le 1.0$	1,523 K, 5 h	1,523 K, 5 h	–

X-ray diffraction patterns were obtained on a D8 ADVANCE diffractometer using CuK_α-radiation in the range of 2Θ angles 20–80°.

Crystal structure parameters of the investigated compounds were determined using RTP program and data of International Centre for Diffraction Data (ICDD JCPDS) [10–18].

Specific magnetization (σ_{sp}) in magnetic fields up to 14 T and specific magnetic susceptibility (χ_{sp}) in the temperature range 6–300 K in magnetic field of 0.8 T of $Gd_{1-x}La_xInO_3$ samples were measured by vibrational method with universal high-field measuring system (Cryogenic Ltd, London, 4IS) in SSPA "Scientific and Practical Materials Research Centre of NAS of Belarus"; those of $Gd_{1-x}La_xScO_3$ and $Gd_{1-x}La_xGaO_3$ samples – by Faraday method in 77–950 K temperature range in magnetic field of 0.86 T with equipment of the laboratory of magnetic materials physics also SSPA "Scientific and Practical Materials Research Centre of NAS of Belarus".

Main part. X-ray analysis showed that in (1–*x*) $GdScO_3$–*x* $LaScO_3$ and (1–*x*) $GdInO_3$–*x* $LaInO_3$ systems with values $0.2 \le x \le 1.0$ continuous series of $Gd_{1-x}La_xScO_3$ and $Gd_{1-x}La_xInO_3$ perovskite-type solid solutions respectively are formed. All these solid solutions have crystal structure of orthorhombically distorted perovskite.

Increase in substitution degree *x* leads to a gradual increase in the crystal lattice parameters *a*, *b*, *c* of orthorhombically distorted perovskite structure.

$GdScO_3$, $GdInO_3$,and $Gd_{0,9}La_{0,1}InO_3$ samples contained unreacted Gd_2O_3.

X-ray analysis showed that at 1,573 K synthesis temperature the limit of La^{3+} ions substitution with paramagnetic Gd^{3+} ions is 50 mol. %, and it correlates well with published data [19, 20].

$Gd_{1-x}La_xGaO_3$ samples containing more than 30 mol. % gadolinium, in addition to main perovskite phase also contained $Gd_3Ga_5O_{19}$ and $Gd_4Ga_2O_9$ phases.

$Gd_4Ga_2O_9$ is isostructural to $Eu_4Al_2O_9$ and has monoclinic syngony. Gallium garnet $Gd_3Ga_5O_{19}$ has cubic structure.

Crystal structure parameters of individual compounds $GdScO_3$, $LaScO_3$, $LaInO_3$ and $LaGaO_3$ correlate well with literature data [10, 14, 17, 18].

$Gd_{1-x}La_xScO_3$ samples specific magnetization temperature dependences and reciprocal values of specific magnetic susceptibility temperature dependences in the magnetic field of 0.86 T over the temperature range 77–1000K are shown in Fig. 1.

Table 2

Crystal lattice parameters *a*, *b*, *c* of $Gd_{1-x}La_xMO_3$ (*M* = Sc, In, Ga) solid solutions depending on the substitution degree *x*

Substitution degree *x*	$Gd_{1-x}La_xScO_3$ system			$Gd_{1-x}La_xInO_3$ system			$Gd_{1-x}La_xGaO_3$ system		
	a, nm	*b*, nm	*c*, nm	*a*, nm	*b*, nm	*c*, nm	*a*, nm	*b*, nm	*c*, nm
0.00	0.55303	0.57741	0.79379	–	–	–	–	–	–
0.10	0.55498	0.57772	0.79764	–	–	–	–	–	–
0.20	–	–	–	0.55831	0.58735	0.81064	–	–	–
0.30	–	–	–	0.55994	0.58757	0.81211	–	–	–
0.40	–	–	–	0.56250	0.58863	0.81363	–	–	–
0.50	0.56121	0.57791	0.80318	0.56428	0.58894	0.81489	0.54937	0.54857	0.77511
0.60	–	–	–	0.56655	0.59032	0.81811	0.57984	0.57862	0.77555
0.70	–	–	–	0.56612	0.58977	0.81747	0.55123	0.54841	0.77631
0.75	0.56552	0.57846	0.80695	–	–	–	–	–	–
0.80	–	–	–	0.56929	0.59161	0.81855	0.55131	0.54897	0.77623
0.90	0.56732	0.57848	0.80782	0.57108	0.59270	0.82095	0.55183	0.54902	0.77698
1.00	0.56788	0.57848	0.80852	0.57181	0.59249	0.82122	0.55186	0.54925	0.77818

Reciprocals of specific magnetic susceptibility temperature dependences analysis (Fig. 1*b*) showed that for the $Gd_{1-x}La_xScO_3$ samples studied there can be distinguished different areas in which Curie – Weiss law is obeyed (linear dependence of $1/\chi_{sp}$ on *T*).

For temperature ranges where magnetic susceptibility obeys Curie – Weiss law were defined by the least squares method equations of linear dependence $1/\chi_{sp}$ on *T* ($1/\chi_{sp} = a + bT$). Specific Curie constants ($C_{sp} = 1/b$) and Weiss constants ($\Theta = -a/b$) were calculated using *a* and *b* coefficients. Molar Curie constant (C_m) values were determined by multiplying C_{sp} on molar mass of the $Gd_{1-x}La_xScO_3$ compound appropriate.

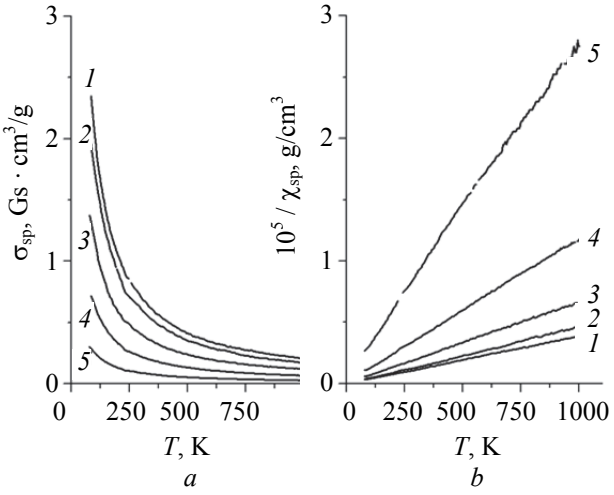

Fig. 1. Specific magnetization (*a*) and reciprocals of specific magnetic susceptibility (*b*) temperature dependences of $Gd_{1-x}La_xScO_3$ samples for different values of *x*: *1* – 0.10; *2* – 0.00; *3* – 0.50; *4* – 0.75; *5* – 0.90

Gadolinium ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) was calculated with the following formula:

$$\mu_{ef,\,Gd^{3+}} = \sqrt{\frac{7.997\,C_m}{1-x}}, \qquad (1)$$

where $7.997 = 3k / N_A\ \mu_B^2$, k – Boltzmann constant; N_A – Avogadro constant; μ_B – Bohr magneton.

Values of Weiss constant and effective magnetic moment of Gd^{3+} ions are presented in Table. 3. Analysis of the Table 3 data shows that calculated values of gadolinium ions effective magnetic moment $\mu_{ef,\,Gd^{3+}}$ in single-phase samples of $Gd_{1-x}La_xScO_3$ solid solutions above 260 K rise gradually with increasing substitution degree x of paramagnetic Gd^{3+} ions with diamagnetic La^{3+} ions and that they are close to theoretical values of the spin magnetic moment for free Gd^{3+} ion $\mu_{ef\,theor,\,Gd^{3+}} = 7.94\ \mu_B$.

Table 3

Weiss constant (Θ) and Gd^{3+} ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) in temperature range of Curie – Weiss law implementation for the $Gd_{1-x}La_xScO_3$ samples

x	$\mu_{ef,\,Gd^{3+}}$, μ_B		Θ, K	
	below 230 K	above 260 K	below 230 K	above 260 K
0.10	7.76 (77–230 K)	7.54 (300–1,080 K)	–8.18 (77–230 K)	–4.88 (300–1,080 K)
0.50	7.61 (77–220 K)	7.62 (270–1,000 K)	–2.86 (77–220 K)	–2.94 (270–1,000 K)
0.75	8.00 (77–1,000 K)		–5.91 (77–1,000 K)	
0.90	8.06 (77–220 K)	8.19 (260–680 K)	–8.83 (77–220 K)	–23.85 (260–680 K)

Note. The temperature ranges in which Curie – Weiss law is obeyed are in parentheses.

The specific magnetic susceptibility and its reciprocals temperature dependences in magnetic field of 0.8T in 7–300 K temperature range for $Gd_{1-x}La_xInO_3$ samples are shown in Fig. 2.

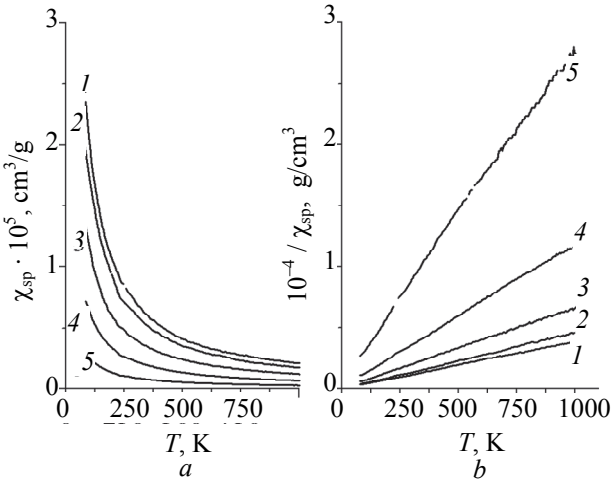

Fig. 2. Specific magnetization (*a*) and reciprocals of specific magnetic susceptibility (*b*) temperature dependences of $Gd_{1-x}La_xInO_3$ samples for different values of *x*: *1* – 0.4; *2* – 0.6; *3* – 0.7; *4* – 0.8; *5* – 0.9

Reciprocals of specific magnetic susceptibility temperature dependences analysis (Fig. 2*b*) showed that for the $Gd_{1-x}La_xInO_3$ samples studied there can be distinguished different areas in which Curie – Weiss law is obeyed (linear dependence of $1/\chi_{sp}$ on *T*).

Mathematical processing for temperature ranges where samples magnetic susceptibility obeys Curie – Weiss law was conducted by the method described above. The results are shown in Table. 4. It was found that calculated values of gadolinium ions effective magnetic moment $\mu_{ef,\,Gd^{3+}}$ in single-phase $Gd_{1-x}La_xInO_3$ samples are significantly below the theoretical value $\mu_{ef\,theor,\,Gd^{3+}} = 7.94\ \mu_B$ for free Gd^{3+} ions up to $x = 0.9$, and $\mu_{ef,\,Gd^{3+}}$ values change without a certain dependence on substitution degree x of paramagnetic Gd^{3+} ions with diamagnetic La^{3+} ions.

Table 4

Weiss constant (Θ) and Gd^{3+} ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) in temperature range of Curie – Weiss law implementationfor the $Gd_{1-x}La_xInO_3$ samples

x	$\mu_{ef,\,Gd^{3+}}$, μ_B	Θ, K	temperature range, K
0.4	7.30	–1.54	7–298
0.6	7.02	2.28	7–180
	7.01	–1.93	215–280
0.7	7.21	–0.63	7–100
	7.17	–0.01	7–230
0.8	6.98	–1.14	7–40
	6.91	5.10	100–270
0.9	7.54	–0.73	5–60

Field dependences of magnetization of $Gd_{1-x}La_xInO_3$ compounds at 5 and 298 K in magnetic fields up to 14 T are shown in Fig. 3.

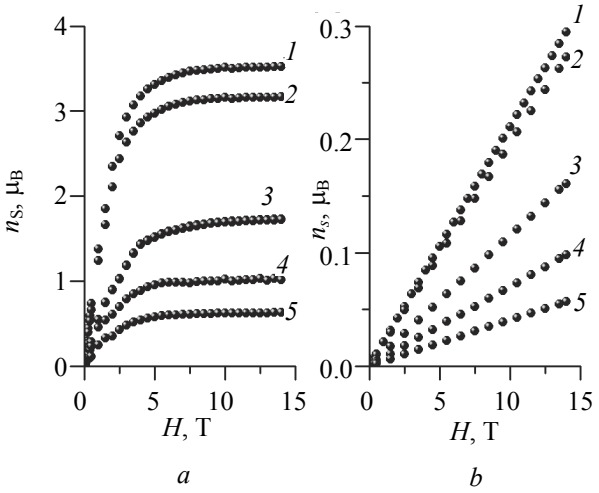

Fig. 3. Field dependences of magnetization of $Gd_{1-x}La_xInO_3$ samples at 5 K (*a*) and 298 K (*b*) in magnetic fields up to 14 T for different values of *x*: *1* – 0.4; *2* – 0.5; *3* – 0.7; *4* – 0.8; *5* – 0.9

At 298 K magnetization rises linearly with magnetic field up to 14 T (Fig. 3*a*) and at 5 K (Fig. 3*b*) in magnetic field above ≈3 T the approach to saturation is observed.

Saturation magnetization n_s of $Gd_{1-x}La_xInO_3$ compounds expressed in μ_B at 5 K is calculated per 1 mole of Gd^{3+} ions according to the next formula

$$n_s = \frac{\sigma_{sp} M}{(1-x)\cdot 5{,}585}, \qquad (2)$$

where σ_{sp} – specific magnetization, $Gs \cdot cm^3/g$; M – molar mass of the corresponding compound, g/mol; 5,585 – a muptiply of the Bohr magneton value ($9.274\cdot10^{-21}$ erg/Gs) by Avogadro constant ($6.021\cdot10^{-23}$ mol^{-1}).

Saturation magnetization n_s calculated values are given in Table 5.

Table 5

Saturation magnetization of $Gd_{1-x}La_xInO_3$ one formula unit (n_s) per 1 mol of Gd^{3}+ ions at 5 K

x	n_s, μ_B per 1 mol of Gd^{3+} ions at 14 T
0.4	5.87
0.6	5.58
0.7	5.76
0.8	5.05
0.9	6.28

Analysis of the Fig. 3 allows us to conclude that in $Gd_{1-x}La_xInO_3$ solid solutions saturation magnetic field decreases with increase in magnetic dilution of paramagnetic Gd^{3+} ions with diamagnetic La^{3+} ions and solid solutions with $x = 0.8$; 0.9 at 5 K in fields of 14 T are magnetized almost to saturation.

Saturation magnetization n_s values calculated per 1 mol of Gd^{3+} ions are significantly lower than the value of 7 μ_B (Table 5) for spin magnetic moment of Gd^{3+} ions. A similar situation has been described for the Gd^{3+} ion in $GdAlO_3$, where the Gd^{3+} ferromagnetic moment is equal to 6,57 μ_B at the exit of magnetization curve to saturation [21].

The specific magnetic susceptibility and its reciprocals temperature dependences in magnetic field of 0.86 T in 77–1000 K temperature range for $Gd_{1-x}La_xGaO_3$samples are shown in Fig. 4.

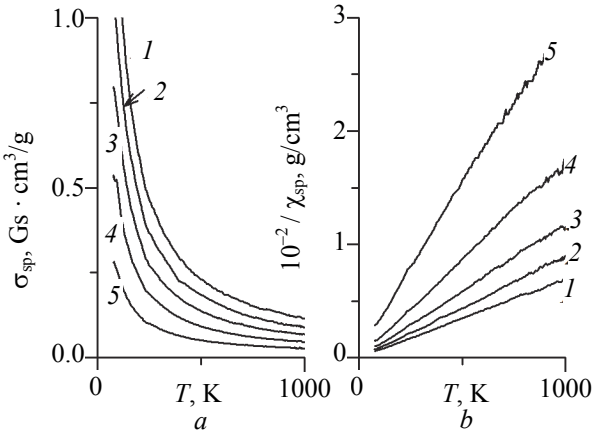

Fig. 4. Specific magnetization (*a*) and reciprocals of specific magnetic susceptibility (*b*) temperature dependences of $Gd_{1-x}La_xGaO_3$ samples for different values of *x*: *1* – 0.5; *2* – 0.6; *3* – 0.7; *4* – 0.8; *5* – 0.9

Reciprocals of specific magnetic susceptibility temperature dependences analysis (Fig. 4*b*) showed that for the $Gd_{1-x}La_xGaO_3$ samples studied there can be distinguished different areas in which Curie – Weiss law is obeyed (linear dependence of $1/\chi_{sp}$ on T).

Mathematical processing for temperature ranges where samples magnetic susceptibility obeys Curie – Weiss law was conducted by the method described above. The results are shown in Table 6.

It was found that the values of Gd^{3+} ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) in single-phase solid solutions $Gd_{1-x}La_xGaO_3$ with $x = 0.8$; 0.9 in temperature range up to 240 K are equal to 7.90 μ_B and 7.99 μ_B respectively and differ slightly from the theoretical value of spin magnetic moment for free Gd^{3+} ions $\mu_{ef\,theor,\,Gd^{3+}} = 7.94$ μ_B. The values of Gd^{3+} ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) in $Gd_{1-x}La_xGaO_3$ solid solutions with $0.5 \le x \le 0.7$ at temperatures above 240 K vary in the range of (7.55–7.77) μ_B at that is slightly lower than theoretical value of $\mu_{ef\,theor,\,Gd^{3+}} = 7.94$ μ_B.

Table 6

Weiss constant (Θ) and Gd^{3+} ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) in temperature range of Curie – Weiss law implementation for the $Gd_{1-x}La_xGaO_3$ samples

x	$\mu_{ef,\,Gd^{3+}}$, μ_B		Θ, K	
	below 240 K	above 240 K	below 240 K	above 240 K
0.5	7.81 (80–1,000 K)		4.14 (80–1,000 K)	
0.6	7.87 (120–240 K)	7.55 (490–750 K)	0.22 (120–240 K)	3.05 (490–750 K)
0.7	7.85 (120–220 K)	7.56 (320–600 K)	1.03 (120–220 K)	23.29 (320–600 K)
0.8	7.90 (120–200 K)	7.57 (320–720 K)	–1.07 (120–200 K)	23.18 (320–720 K)
0.9	7.99 (120–220 K)	–	1.95 (120–220 K)	–

Note. The temperature ranges in which Curie – Weiss law is obeyed are in parentheses.

Conclusion. A continuous series of solid solutions $Gd_{1-x}La_xScO_3$ ($0.0 \le x < 1.0$), $Gd_{1-x}La_xInO_3$ ($0.2 \le x \le 1.0$) $Gd_{1-x}La_xGaO_3$ ($0.5 \le x \le 1.0$) with crystal structure of orthorhombically distorted perovskite was synthesized by ceramic method.

Increasing the substitution degree x of Gd^{3+} ions with La^{3+} ions for $Gd_{1-x}La_xScO_3$ and $Gd_{1-x}La_xInO_3$ solid solutions leads to a gradual increase in crystal lattice parameters a, b, c of orthorhombically distorted perovskite structure.

It was found that Gd^{3+} ions effective magnetic moment ($\mu_{ef,\,Gd^{3+}}$) in $Gd_{1-x}La_xScO_3$ solid solutions with $x = 0.10$; 0.50 is equal to 7.76 μ_B; 7.61 μ_B respectively and that is slightly lower than the theoretical value $\mu_{ef\,theor,\,Gd^{3+}} = 7.94$ μ_B; for $x = 0.75$; 0.90 it is practically equal to the theoretical value.

For $Gd_{1-x}La_xGaO_3$ solid solutions with $x = 0.8$; 0.9 $\mu_{ef,\,Gd^{3+}}$is equal to7.90 μ_B; 7.99 μ_B respectively. For $Gd_{1-x}La_xInO_3$ solid solutions $\mu_{ef,\,Gd^{3+}}$ varies without any dependence in (6.91–7.54) μ_B interval which is below the theoretical value $\mu_{ef\,theor,\,Gd^{3+}}$.

With magnetic field increasing up to 14T magnetization of $Gd_{1-x}La_xInO_3$ solid solutions rises linearly at of 298 K and at 5 K in the fields above ≈3 T the exit to saturation was observed, but the theoretical value of 7 μB for the spin magnetic moment of Gd^{3+} ions was not achieved.

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