



Scientific article

UDC 54-386:546.654:615.213

DOI: 10.52957/2782-1900-2026-7-2-102-111

LANTHANUM(III) WITH GABAPENTIN COORDINATION COMPOUND: METHOD OF PREPARATION AND THERAPEUTIC POTENTIAL

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Keywords: gabapentin, IR spectroscopy, electronic spectroscopy, lanthanum, coordination number, complex stability, PASS-online, antitumor activity.

Abstract. The research examines the coordination of lanthanum(III) compound with 8-oxyquinoline. The article analyses the synthesis method resulted in the formation of a yellow fine-crystalline precipitate. The structure and nature of ligand coordination with the metal ion were confirmed by physicochemical methods (electron and vibrational spectroscopy) and semi-empirical quantum chemical calculations (MOPAC//PM7). Electronic spectroscopy records a significant bathochromic shift ($\Delta\lambda=72$ nm). It indicates the formation of a stable complex. The research reveals the dependence of the complex stability on the pH of the medium, and identifies the its optimal pH range of 7.0–8.0. The theoretical part of the research includes quantum chemical modeling to evaluate the redistribution of electron density, and visualise the optimised geometry of the molecule. Moreover, computer analysis of biological activity and pharmacokinetic parameters (ADME-Tox) indicates the potential value of the synthesized complex as a compound with a modified and improved biological activity profile.

For citation:

Shubina A.A., Orlova T.N. Lanthanum(III) with gabapentin coordination compound: method of preparation and therapeutic potential // From Chemistry towards Technology Step-by-Step. 2026. Vol. 7, Iss. 2. P. 102-111. URL: <https://chemintech.ru/en/nauka/issue/7273/view>

Introduction

In recent decades, the coordination chemistry of rare earth elements, in particular lanthanum, has attracted the interest of researchers due to the wide range of unique biological and physicochemical properties of these elements complexes [1]. Lanthanides, including La^{3+} , possess high coordination ability and can form stable chelates with organic ligands of various natures [2]. They are promising as functional materials for biomedicine, including anticancer activity, and the potential for their further use as drug delivery agents, the ability to influence biologically active compounds, and processes occurring in living organisms [3].

Gabapentin (1-(aminomethyl)cyclohexaneacetic acid) is an amino acid-like drug (ampholytic compound) that is used in clinical practice as an agent for the treatment of neuropathic pain, epilepsy, and chronic pain associated with trigeminal and sciatic nerve lesions as well as herpes zoster [4, 5]. Recently, in clinical practice, this drug has also been used to reduce the severity of symptoms of depression, anxiety, pain syndromes, and for cognitive functions improving [5]. Its structure contains functional groups capable to coordinate with metal ions [6].



Lanthanum traditionally forms complexes through heteroatoms of various natures. According to the HSAB concept, oxygen donors, including carboxyl groups, are the most preferable ones [7]. Studies of lanthanum coordination compounds with various organic ligands (e.g., with nicotinic acid) show a diversity of structural forms and possible applications in chemistry, medicine, materials science, etc. [8].

Metal complexes are widely used in anticancer therapy [9]. The classic example is platinum-based compounds (e.g., cisplatin). Indeed, currently there is active search for new compounds to overcome the problem of resistance to traditional anticancer drugs [10].

Lanthanum complexes are considered by researchers as promising antitumor agents: they are capable of interacting with DNA, inducing oxidative stress, and triggering apoptosis of cancer cells. It demonstrates the potential for their application in oncological diseases of various origins [11]. The therapeutic properties against many types of tumors are explained by the following mechanism of action: La^{3+} affects cellular signaling pathways and induces oxidative stress; inhibition of certain enzymes and an increase in the concentration of certain reactive oxygen species may occur [12]. Traditionally, stable rare earth complexes are found among β -diketonates, phenanthrolines, Schiff bases, and there are reports of metal-organic frameworks containing the aforementioned ions [13]. Gabapentin contains both amine and carboxyl groups, which provides its potential as a nitrogen- and oxygen-donor ligand, analogous to those already studied in the literature. A possible enhancement of the biological activity of gabapentin, due to the influence of the metal on cellular processes, is hypothesized.

Purpose of the paper

The purpose of this study is the synthesis and investigation of the spectral characteristics of the lanthanum(III) complex with gabapentin and an *in silico* comparative assessment of obtained compound and the original ligand therapeutic potential.

Experimental part

We used gabapentin in the form of capsules 'Katena' (BELUPO, Croatia); potassium hydroxide, lanthanum chloride heptahydrate. One gabapentin capsule contains: gabapentin 300 mg and auxiliary components: corn starch, lactose monohydrate, talc; capsule shell: gelatin, titanium dioxide, yellow iron oxide, red iron oxide. To record the electronic and IR spectra, a PE 5400-UV instrument (ECROSKHIM LLC, Russia) and a Spectrum 65 instrument (Perkin Elmer, USA) with an ATR attachment were used, respectively; for elemental analysis, an EA 1112 CHN modification instrument (Thermo Finnigan Italia S.p.A., Italy) was used. The lanthanum content in the complex was determined gravimetrically according to a standard procedure using alkali as the precipitant [14]; chlorine was determined argentometrically [15].

Procedure for extracting the active substance from the drug matrix. The powder was extracted from two capsules, poured into a beaker; 20 mL of cold distilled water was added. The resulting mixture was stirred for 5 minutes on a magnetic stirrer and filtered. The auxiliary

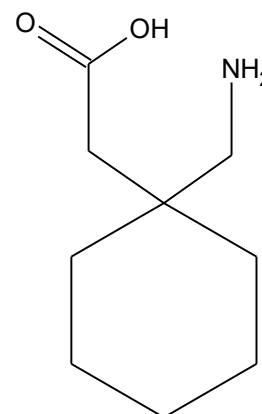
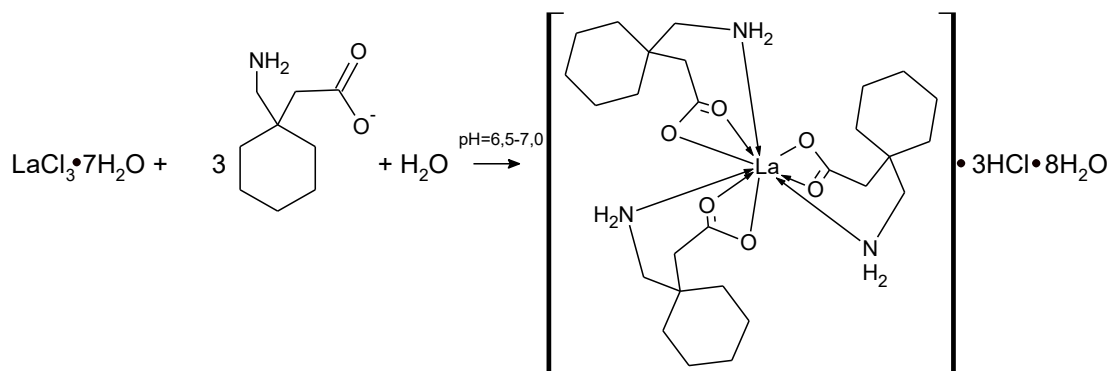


Fig. 1. Structural formula of gabapentin (1-(aminomethyl)cyclohexaneacetic acid)



components remaining on the filter were discarded; the filtrate was left to evaporate in air. To remove residues of lactose monohydrate, the resulting dry residue was recrystallized from ethyl alcohol, after which the ligand was observed to form in crystalline form, ready for further synthetic transformations.

Procedure for the synthesis of the lanthanum(III) complex with gabapentin (GBP). The ligand isolated from the drug matrix (0.52 g; 0.003 mol) was dissolved in 5 mL of distilled water; if necessary, the pH was adjusted (to 6.5 ± 0.5) by dropwise addition of 0.1 M potassium hydroxide solution to convert the carboxyl group to the carboxylate form. A weighed amount of $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ (0.37 g; 0.001 mol) dissolved in a minimal amount of distilled water was added to the ligand solution with constant stirring; the formation of a homogeneous solution was observed. The mixture was kept for 1 hour at 50 ± 5 °C, avoiding overheating, and then placed in a refrigerator. The product that precipitated after 24 hours was isolated by centrifugation, washed with water and ethanol, and dried in a desiccator over anhydrous CaCl_2 to constant weight for 5–6 days (the complex becomes hydrated when dried in air). If residues of unreacted ligand are detected by IR spectroscopy, recrystallization from water–alcohol mixtures or slow solvent diffusion is recommended to obtain a high-quality fine crystalline powder, which will allow the complex to be isolated in pure form. After recrystallization, the compound is a fine crystalline powder of beige color. Yield is 75% (substance mass is 0.68 g). Calculated, %: C 35.90; H 7.48; N 4.65; O 24.80; Cl 11.78; La 15.38. Found, %: C 35.96; H 7.44; N 4.66; O 24.86; Cl 11.65; La 15.42. Molar weight: 903.10 g/mol IR-spectrum, ν , cm^{-1} : 2925 ($\nu_{\text{as}} \text{CH}_2$), 2855 ($\nu_{\text{s}} \text{CH}_2$), 1550 ($\nu_{\text{as}} \text{COO}^-$), 1399 ($\nu_{\text{s}} \text{COO}^-$), 1306 (C-N), 1193 (C-O), 1113, 1038, 929 (δCH_2), 825, 795 ($\text{La}^{3+}\text{-O}$). $\lambda_{\text{max}}=302$ nm. Scheme 1 shows the formation of the complex.



Scheme 1. Formation of the lanthanum(III) complex with gabapentin

The obtained compound is readily soluble in water, alcohols, acetone, DMSO, and DMF, and poorly soluble in acetonitrile, diethyl ether, hexane, and other nonpolar solvents [16].

To determine the instability constant, a 100 mg sample of the complex was dissolved in 5 mL of distilled water; the solution was stirred for 3 hours to establish a concentration value as close to equilibrium as possible. The supersaturated solution was filtered, yielding a saturated solution of the complex compound at room temperature (under the experimental conditions, 21.6 °C – reading of the laboratory thermometer).

Main body

Analysis of the electronic spectra of the ligand relative to the complex showed a bathochromic shift from 204 nm for the ligand to 302 nm for the complex (see Fig. 2).

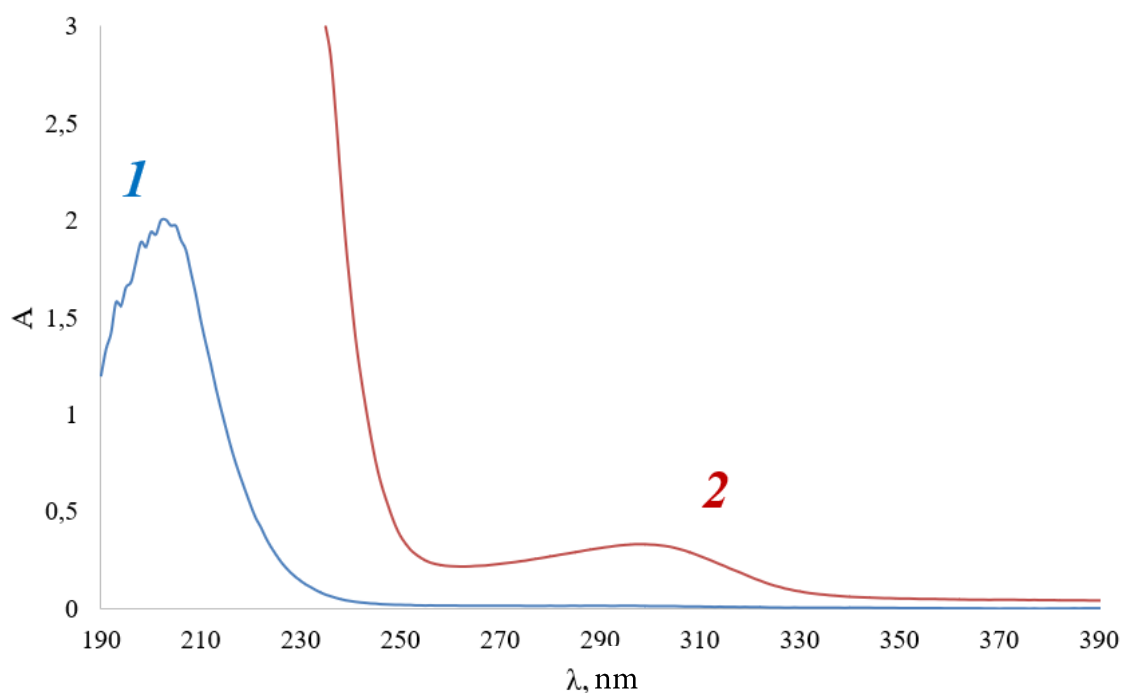


Fig. 2. Electronic spectra (reference solution – distilled water): 1 – gabapentin (pH = 6.50; $C = 9.5 \cdot 10^{-4}$ M), 2 – La(III)-gabapentin complex ($\epsilon = 344.97$ L·mol $^{-1}$ ·cm $^{-1}$; $C = 9.5 \cdot 10^{-4}$ M).

Gabapentin does not contain aromatic rings or conjugated double bond systems in its structure that could involve the carboxyl group. The synthesis is conducted at pH = 6.50; the amino group of gabapentin is predominantly protonated ($-\text{NH}_3^+$); the lone pair of electrons on the nitrogen atom does not participate in $n \rightarrow \pi^*$ transitions. Thus, a $\pi \rightarrow \pi^*$ transition ($\lambda_{\text{max}1} = 204$ nm) occurs within the delocalized π -system of the carboxylate anion ($\text{pK}_a = 3.68$); there is an influence of solvation of the ligand's carboxylate anion by distilled water. Complex formation with the metal is indicated by the LMCT (ligand-to-metal charge transfer) electronic transition at $\lambda_{\text{max}2} = 302$ nm; it leads to the formation of a low-energy chromophore – a charge-transfer complex.

The obtained complex has a high molar extinction coefficient: $\epsilon = 344.97$ L·mol $^{-1}$ ·cm $^{-1}$, which is confirmed by absorption measurements at different concentration values (see Fig. 3, Table 1).

Table 1. Determination of the molar extinction coefficient (ϵ) of the La(III) complex with gabapentin.

C, mol/L	A
$9.0 \cdot 10^{-5}$	0.031
$1.5 \cdot 10^{-4}$	0.056
$2.5 \cdot 10^{-4}$	0.083
$3.5 \cdot 10^{-4}$	0.121
$4.5 \cdot 10^{-4}$	0.148
$6.0 \cdot 10^{-4}$	0.202
$7.0 \cdot 10^{-4}$	0.247
$8.0 \cdot 10^{-4}$	0.286
$9.5 \cdot 10^{-4}$	0.331
$1.0 \cdot 10^{-3}$	0.352
$1.5 \cdot 10^{-3}$	0.507

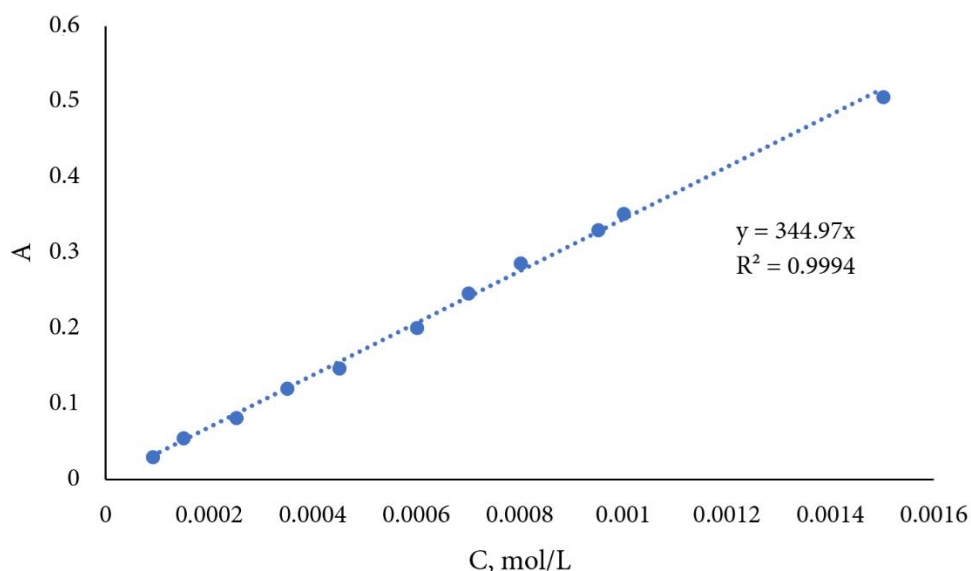


Fig. 3. Determination of the molar extinction coefficient (ϵ) of the La(III) complex with gabapentin using the linear regression equation.

The complexation of gabapentin with lanthanum(III) has also been proven by IR spectroscopy. Figs. 4 and 5 present the vibrational spectra of the ligand and the coordination compound.

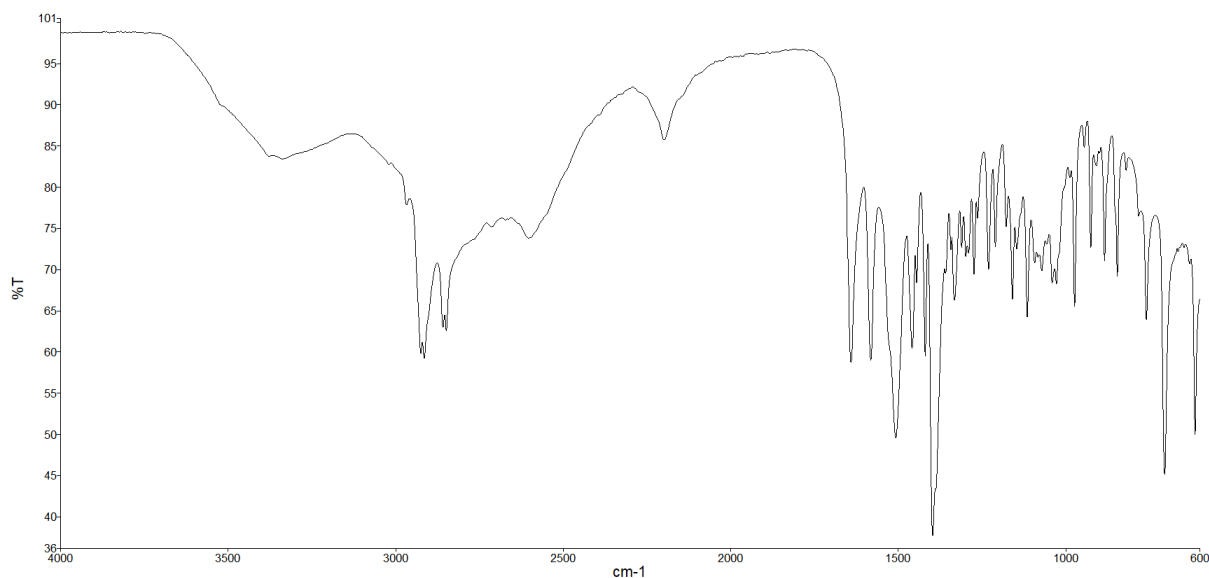


Fig. 4. IR spectrum of the ligand gabapentin.

The IR spectrum of the initial ligand in the solid state presents the bands corresponding to both the protonated carboxyl group ($\nu(\text{C}=\text{O})$) and the carboxylate anion ($\nu_{\text{as}}(\text{COO}^-)$, $\nu_{\text{s}}(\text{COO}^-)$). In the spectrum of the complex with La^{3+} , the $\nu(\text{C}=\text{O})$ band is absent, while the COO^- bands persist and shift, indicating complete deprotonation of the carboxyl group and its participation in coordination with the lanthanum ion.

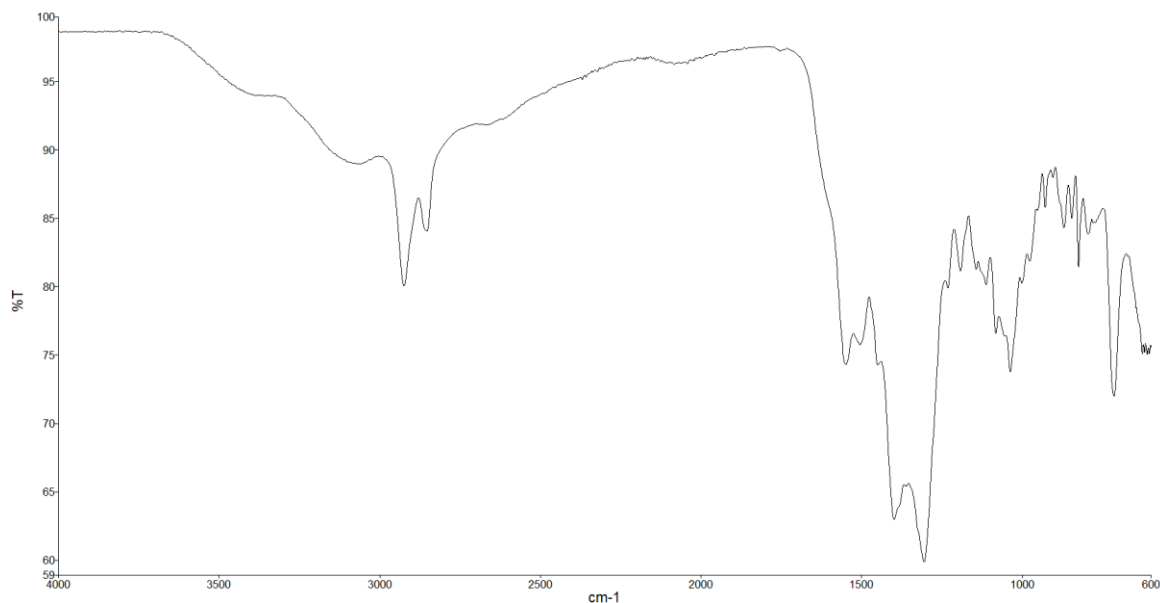


Fig. 5. IR spectrum of La(III) complex with gabapentin.

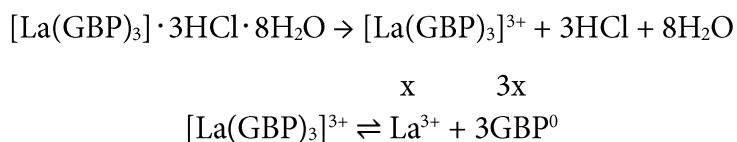
The disappearance of the free -OH group peak (3338 cm^{-1}) is observed. The appearance of new bands ($825, 795\text{ cm}^{-1}$) is characteristic of metal–oxygen vibrations of $\text{La}^{3+}\text{-O}$. Despite the fact that the stretching vibrations of C-H and $\text{-CH}_2\text{-}$ groups in the region of $3000\text{-}2800\text{ cm}^{-1}$ do not change their position upon complexation, the deformation vibrations of methylene groups (scissoring, wagging, rocking, twisting) in the range of $1500\text{-}700\text{ cm}^{-1}$ shift and undergo intensity redistributions. It indicates a decrease in the mobility of the carbon skeleton upon introduction of the metal cation into the structure of the original molecule. Thus, bidentate ($\Delta\nu(\text{COO}^-) = 150\text{ cm}^{-1}$) O,O-coordination through the carboxylate anion is observed. Table 2 provides a detailed comparative interpretation of the peaks.

Table 2. Comparative interpretation of the peaks in the IR spectra of gabapentin and its complex with La(III)

ν, cm^{-1} (%T), ligand	ν, cm^{-1} (%T), complex	Interpretation
3338.76 (83.45)	-	O-H, stretching vibrations
2924.92 (59.88)	2924.53 (80.08)	$\nu_{\text{as}}(\text{-CH}_2\text{-})$, aliphatic vibrations of the carbon skeleton
2858.96 (59.88)	2854.59 (84.08)	$\nu_{\text{s}}(\text{-CH}_2\text{-})$, aliphatic vibrations of the carbon skeleton
1640.91 (58.77)	-	C=O, stretching vibrations
1581.99 (59.03)	1549.76 (74.33)	$\nu_{\text{as}}(\text{COO}^-)$
1507.26 (49.59); 1458.88 (60.43); 1445.25 (68.44)	-	scissoring deformation vibrations of $\text{-CH}_2\text{-}$
1397.47 (37.74)	1399.19 (62.99)	$\nu_{\text{s}}(\text{COO}^-)$
1342.88 (72.42)	1306.23 (59.91)	$\nu(\text{C-N})$
1177.78 (75.23)	1192.53 (81.17)	$\nu(\text{C-O})$
1159.23 (66.44); 1146.49 (72.55)	-	rocking vibrations of $\text{-CH}_2\text{-}$
1115.02 (64.30); 1071.89 (69.89)	1113.20 (80.16); 1037.76 (73.78); 928.91 (85.79)	twisting vibrations of $\text{-CH}_2\text{-}$
973.76 (65.55); 944.91 (84.83); 925.45 (72.67)	928.91 (85.79)	$\delta(\text{CH}_2)$
-	825.09 (81.42); 795.40 (83.86)	$\text{La}^{3+}\text{-O}$



The instability constant of the La(III) complex with gabapentin was determined by the spectrophotometric method using saturation curves. Using elemental analysis, it was established that the metal-to-ligand ratio is 1:3; the gross formula of the studied compound is known. The inner sphere of the complex, being a sparingly soluble electrolyte, dissociates according to the equation:



The equation for the solubility product (SP) has the form of $27x^4$. From the calibration graph in Fig. 3, the concentration of the saturated solution was determined to be $1.44 \cdot 10^{-3}$ mol/L.

$$K_{\text{inst}} = \text{SP} = x \cdot (3x)^3 = 27x^4 = 27 \cdot (1.44 \cdot 10^{-3})^4 = 1.16 \cdot 10^{-10}$$

The effect of complexation of gabapentin with lanthanum on the pharmacokinetic properties of the obtained compounds was examined. It considers an alternative possible coordination numbers (1 and 2) with inner spheres compositions $[\text{La}(\text{GBP})]^{3+}$ and $[\text{La}(\text{GBP})_2]^{3+}$. The *in-silico* analysis was performed using the online services ADMET (<http://qsar.chem.msu.ru/admet/>) and PASS Online (<https://way2drug.com/PassOnline/predict.php>).

According to Table 3, log BB (the ability of a substance to cross the blood–brain barrier) for gabapentin has a negative value – in contrast to the corresponding complexes readily penetrating into the brain. The complexes with coordination numbers 2 and 3 are effective for oral administration (HIA – human intestinal absorption – percentage of absorption in the intestine) – in contrast to the compound with coordination number 1, for which parenteral administration is preferable. All three complexes demonstrate higher affinity for the biological target (higher pK_i and pIC_{50} values) than gabapentin. Therefore, they should have high affinity and, theoretically, provide greater efficacy at lower dosages.

Table 3. Comparative analysis of pharmacokinetic and pharmacodynamic parameters using the ADMET online resource

Compound	log BB	HIA, %	pK_i	pIC_{50}
GBP	-0.63	81.15	3.87	3.72
$[\text{La}(\text{GBP})]^{3+}$	+0.33	30.84	4.47	4.25
$[\text{La}(\text{GBP})_2]^{3+}$	+0.44	83.05	4.82	3.63
$[\text{La}(\text{GBP})_3]^{3+}$	+0.41	74.03	5.06	3.33

Gabapentin demonstrates several key types of biological activity, as shown in Table 4.

Table 4. Probability of pharmacological activity manifestation of gabapentin (PASS Online)

Probability of presence of biological activity P_a	Probability of absence of biological activity P_i	Effect
0.894	0.005	CYP2J substrate
0.811	0.002	GABA-C receptor agonist
0.745	0.008	anticonvulsant agent
0.708	0.009	analgesic
0.855	0.015	treatment of phobic disorders
0.799	0.012	NADPH oxidase inhibitor
0.744	0.010	superoxide dismutase inhibitor



The most studied and pharmacologically significant mechanism is agonism of GABA-C receptors ($P_a = 0.811$) – an effect on the synthesis and release of γ -aminobutyric acid, which is possible for the drug structure similar to this molecule. This provides both anticonvulsant ($P_a = 0.745$) and analgesic ($P_a = 0.708$) effects. Another important indicator is the treatment of phobic disorders ($P_a = 0.855$). The ability of gabapentin to act as an inhibitor of NADPH oxidase ($P_a = 0.799$) and superoxide dismutase ($P_a = 0.744$) contributes to both neuroprotective and adverse effects, reflecting an influence on oxidative stress. Theoretically, gabapentin may also act as a substrate for the CYP2J2 enzyme of the cytochrome P450 system. However, its activity has no clinical significance, since the drug is practically not metabolized and is excreted via the kidneys in an essentially unchanged form [17].

Importantly, gabapentin has 8 theoretically possible adverse effects (with $P_a > 0.7$; see Table 5). Those are characterized by high and moderate severity: vomiting with blood ($P_a = 0.801$), occult bleeding ($P_a = 0.747$), anemia ($P_a = 0.773$), gastrointestinal bleeding ($P_a = 0.754$), and others.

Table 5. Probability of adverse effects of gabapentin (PASS Online)

Probability of presence of adverse effects P_a	Probability of absence of adverse effects P_i	Effect
0.801	0.013	vomiting with blood
0.733	0.005	anemia
0.798	0.038	chills
0.754	0.013	gastrointestinal bleeding
0.747	0.017	occult bleeding
0.736	0.013	hypercholesterolemia
0.737	0.035	pure red cell aplasia
0.710	0.040	aphthous ulcer

In contrast to the ligand gabapentin, the La(III) complexes with the coordination numbers 2 and 3 (which are of greatest interest from the standpoint of bioavailability criteria) exhibit new types of potential biological activity – see Table 6.

Table 6. Probability of pharmacological activity manifestation of lanthanum complexes with gabapentin (PASS Online)

Effect	Probability of pharmacological activity manifestation for the complex with CN = 2 composition $[\text{La}(\text{GBP})_2]^{3+}$; $P_a(P_i)$	Probability of pharmacological activity manifestation for the complex with CN = 3 composition $[\text{La}(\text{GBP})_3]^{3+}$; $P_a(P_i)$
Analgesic	0.748 (0.007)	0.904 (0.004)
Antitumor (multiple myeloma)	-	0.837 (0.002)
Treatment of phobic disorders	0.835 (0.022)	0.812 (0.029)
Protection of mucous membranes	0.795 (0.020)	0.767 (0.028)
Antiseborrheic	0.704 (0.037)	-
GABA-C receptor agonist	0.741 (0.037)	-
Inhibitor of ubiquinol-cytochrome C reductase	0.749 (0.051)	0.714 (0.063)



Thus, the complex with coordination number (CN) 2 is characterized by the manifestation of antiseborrheic properties ($P_a = 0.704$) and retains GABA-C receptor antagonism ($P_a = 0.741$). The most promising compound for further biomedical research is the one with CN = 3, as it has a high probability of manifesting analgesic ($P_a = 0.904$) and antitumor ($P_a = 0.837$) effects against multiple myeloma.

The manifestation of new types of biological activity is associated with a fundamental change in the physicochemical and coordination properties of the molecule upon formation of the complex compound.

Among the adverse effects of the considered metal complexes, it is important to note neurological and systemic adverse effects (euphoria, weight loss, etc.) – as their P_a values decrease upon transition to CN = 3, indicating an improvement in the safety profile compared to CN = 2 (see Table 7).

Table 7. Probability of adverse effects of lanthanum complexes with gabapentin (PASS Online)

Adverse effect	Probability of an adverse effect for the complex with CN = 2 composition [La(GBP) ₂] ³⁺ ; $P_a(P_i)$	Probability of an adverse effect for the complex with CN = 3 composition [La(GBP) ₃] ³⁺ ; $P_a(P_i)$
Euphoria	0.957 (0.004)	0.948 (0.004)
Weight loss	0.913 (0.003)	0.875 (0.003)
Hyperhidrosis	0.890 (0.007)	0.861 (0.009)
Gastrotoxicity	0.754 (0.034)	0.798 (0.026)
Dependence	0.788 (0.007)	0.763 (0.009)

Conclusions

A method has been developed for the synthesis of a water-soluble complex of lanthanum(III) with gabapentin. The efficiency is confirmed by a high yield (75%); the identity of the compound has been confirmed by IR and UV-Vis spectroscopy. The spectral characteristics are as follows: 2925 (ν_{as} CH₂), 2855 (ν_s CH₂), 1550 (ν_{as} COO⁻), 1399 (ν_s COO⁻), 1306 (C-N), 1193 (C-O), 1113, 1038, 929 (δ CH₂), 825, 795 (La³⁺-O); λ_{max} =302 nm. This complexation preserves the prospects of biological activity inherent in the original ligand and provides the possibility of imparting fundamentally new therapeutic properties. The data obtained justify the potential of further preclinical studies of the complex [La(GBP)₃]³⁺ as a multifunctional agent for the treatment of neuropathic pain ($P_a = 0.904$), phobic disorders ($P_a = 0.812$), and cancer ($P_a = 0.837$). The complex, however, requires the development of special dosage forms to minimize the predicted gastrointestinal toxicity ($P_a = 0.798$).

Conflict of interest

The authors declare no conflict of interest.

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Received 26.03.2026

Approved after reviewing 23.06.2026

Accepted 12.05.2026