UDC 541.49:661.693:547.587.26
https://doi.org/10.18524/2304-0947.2026.1(91).367436

M. A. Martyniuk[1], O. E. Martsynko[1*], O. A. Finik[2], O. V. Snurnikova[2]
[1] Odesa I. I. Mechnikov National University, Department of Inorganic Chemistry and Chemical Education,
12 Universytetska St, Odesa, 65082, Ukraine;
e-mail: lborn@ukr.net
[2] BUREAU VERITAS COMMODITIES UKRAINE LLC,
1 Udilnyi Ln, Odesa, 65044, Ukraine

SYNTHESIS AND STRUCTURE OF ONIUM GALLATOGERMANATES(IV) WITH NITROGEN-CONTAINING ORGANIC CATIONS

Optimal methods of synthesis of germanium(IV) complexes with gallic acid (H_4Gal) and exo-ligands benzimidazole (Bim), niacin (Nic), and niamide (Nad) in aqueous solution were developed. The composition and structure of the synthesized complexes were determined using elemental analysis, IR spectroscopy, and mass spectrometry. Thermal analysis established the hydrated composition of the complexes and the nature of their thermal decomposition. It was shown that the obtained compounds belong to the cation-anion type. They contain the tris(galato)germanate anion and organic molecules protonated at the nitrogen atom of the heterocycle as the cation. The following formulas of the synthesized compounds were proposed: $(HBim)_2[Ge(H_2Gal)_3]\cdot 4H_2O$ (1), $(HNic)_2[Ge(H_2Gal)_3]\cdot 3H_2O$ (2), $(HNad)_2[Ge(H_2Gal)_3]\cdot 4H_2O$ (3).

Keywords: germanium, gallic acid, coordination compounds, IR spectroscopy, thermogravimetric analysis, mass spectrometry.

Gallic acid (3,4,5-trihydroxybenzoic acid, H_4Gal) is a naturally occurring phenolic acid that is widely distributed in plants, fruits, tea, and various medicinal herbs. Owing to the presence of one carboxyl group and three phenolic hydroxyl groups, gallic acid exhibits remarkable chemical versatility and possesses a high ability to interact with metal ions. The relatively large distances between the phenolic oxygen atoms facilitate different coordination modes, making gallic acid an attractive organic ligand for the design of coordination compounds and metal-organic frameworks [1].

In addition to its coordination ability, gallic acid is well known for its broad spectrum of biological activities, including antioxidant, anti-inflammatory, antimicrobial, anticancer, and antimutagenic properties [2–9]. These beneficial effects are mainly associated with its strong radical-scavenging ability and reducing properties. Historically, gallic acid has been used in the production of iron gallate inks and dyes because of the intense coloration arising from complex formation with iron ions. It has also found applications in photographic developers, food preservation, and pharmaceutical formulations, further demonstrating its chemical and technological importance [10].

The combination of versatile coordination behavior and valuable biological activity has stimulated extensive research on metal complexes of gallic acid. Depending on the metal ion, pH, and synthesis conditions, gallic acid may coordinate in neutral,

partially deprotonated, or fully deprotonated forms, giving rise to mononuclear complexes, coordination polymers, and metal-organic frameworks with diverse structural architectures. Such compounds often exhibit enhanced thermal stability, modified redox properties, catalytic activity, or improved biological performance compared with the free ligand [1, 10, 11].

Among the most remarkable examples are the calcium-based three-dimensional coordination polymers MIL-155 and MIL-156, which were synthesized under hydrothermal conditions using naturally occurring gallic acid. MIL-155 consists of infinite chains of edge-sharing CaO_7 polyhedra interconnected by partially deprotonated gallate ligands (H_2Gal^{2-}), whereas MIL-156 contains ribbon-like inorganic subunits built from six- and eight-coordinate Ca^{2+} ions linked by fully deprotonated gallate ligands (Gal^{4-}). Biological investigations have demonstrated that both materials are biocompatible [11]. These findings highlight the potential of gallate-based coordination compounds as promising candidates for biomedical and pharmaceutical applications, particularly antioxidant materials.

As well as forming alkaline-earth metal compounds, gallic acid forms stable coordination complexes with p-block elements. An example is the mixed-ligand complex $[Ge(H_2Gal)_2(phen)]$ (phen is 1,10-phenanthroline) synthesised by authors [12]. Single-crystal X-ray diffraction analysis showed that the Ge(IV) center adopts a distorted octahedral coordination geometry formed by four oxygen atoms belonging to two doubly deprotonated gallic acid ligands and two nitrogen atoms of the 1,10-phenanthroline molecule. A complex acid $[Ge(H_2Gal)(H_3Gal)_2]\times H_2O$ was also obtained, in which the composition Ge:ligand = 1:3 is realized [13].

Overall, gallic acid's ability to coordinate a wide range of metal ions, combined with its intrinsic biological activity and multiple coordination modes, makes it an exceptionally attractive ligand for the development of new coordination compounds and metal-organic materials.

This work continues these studies. The goal of this work is to synthesise coordination compounds of Germanium(IV) with gallic acid and organic bioactive molecules (benzimidazole, niacin, niamide) and to research their composition, structure, and properties. The incorporation of such nitrogen-donor ligands into the complex may influence its physicochemical and biological properties.

MATERIALS AND METHODS

The starting reagents (all were purchased from Sigma Aldrich) for the synthesis – Germanium(IV) oxide (GeO_2, CAS 1310-53-8, 99.99%), gallic acid (H_4Gal, CAS 149-91-7), benzimidazole ($C_7H_6N_2$, CAS 51-17-2, ≥99%), niacin (nicotinic acid, $C_6H_5NO_2$, CAS 59-67-6, 99%), niamide (nicotinamide, $C_6H_6N_2O$, CAS 98-92-0, 99.5%).

Synthesis. A portion of GeO_2 (9 mmol, 0.9414 g) was dissolved in 500 ml of distilled water (90 °C), then 27 mmol (4.60 g) of gallic acid was added, stirred, slowly evaporated at 80 °C to a volume of 150 mL, and divided into three parts of 50 mL

each. After cooling to 25 °C (pH = 2.5–3), 20 mmol of benzimidazole (Bim), niacin (Nic) or niamide (Nad) was added and stirred till the reagent was completely dissolved. Precipitates of coordination compounds were formed within a day. They were separated on a Schott glass filter and dried in air at 20–25 °C. Yield: 72–77%.

Elemental analysis was performed in the C,N,H-analyzer Elemental Analyzer CE-440. Germanium content was determined using inductively coupled plasma atomic emission spectroscopy with an Optima 8000 (PerkinElmer).

The elemental analysis results:

Calculated for $C_{35}H_{34}GeN_4O_{19}$ (**1**, M_r = 886,6) (%): C — 47.37, H — 3.83, Ge — 8.19, N — 6.32. Found (%): C — 47.58, H — 3.75, Ge — 8.30, N — 6.42.

Calculated for $C_{33}H_{30}GeN_2O_{22}$ (**2**, M_r = 878.6) (%): C — 45.07, H — 3.41, Ge — 8.26, N — 3.19. Found (%): C — 45.00, H — 3.31, Ge — 8.45, N — 3.30.

Calculated for $C_{33}H_{34}GeN_4O_{21}$ (**3**, M_r = 894.6) (%): C — 44.27, H — 3.80, Ge — 8.12, N — 6.26. Found (%): C — 44.19, H — 4.00, Ge — 8.25, N — 6.35.

Thermogravimetric analysis was performed on a Q-1500D device with a heating rate of 10 °C/min in an air atmosphere in the temperature range of 20–1,000 °C.

The IR spectra in the range of 4,000–400 cm^{-1} were recorded as potassium bromide pellets on a Frontier spectrometer (PerkinElmer). IR spectra were interpreted based on literature data on the characteristic absorption bands of organic molecules [14, 15] and complex compounds of germanium(IV) and other metals [11, 12, 16, 17].

The ESI mass spectra were taken on TSQ Fortis Triple Quadrupole Mass Spectrometer (ThermoFisher Scientific, USA). All spectra were measured in methanol-water-DMSO (1/1/0.5, v/v/v) solutions with complexes at about 20–50 μg/mL concentrations. Samples were infused using a Chemyx Fusion 100T2 syringe pump with a 500 μL Hamilton gas-tight syringe (no. 81265, 1750RNR) at a flow rate of 20 μL/min. The electric potential to initiate ESI was 3.0 to 3.5 kV in positive ionisation mode and 2.0 to 2.5 kV in negative ionisation mode. Mass spectra were collected in centroid mode in the m/z range of 50–500 Da using Q1 full-scan mode, with FWHM resolution set to 0.4 Da and a scan rate of 250 Da/sec.

RESULTS AND DISCUSSION

The complexes **1–3** are white, homogeneous substances stable in air. The elemental analysis revealed that the complexes have a molar ratio of Ge:Gal:Bim(Nic, Nad) = 1:3:2.

The thermodecomposition of compounds **1–3** is similar (Fig. 1): in the temperature range of 60–180 °C, an endothermic effect with a maximum at ~100 °C is observed (Fig. 1). This effect is accompanied by a decrease in mass by 8.00% (Δm_{theor} = 8.12%) for complex **1**; 6.00% (Δm_{theor} = 6.15%) for **2**; 8.00% (Δm_{theor} = 8.00%) for **3**; which corresponds to 4 (for **1**, **3**) and 3 (for **2**) molecules of crystallization water. The final product at 1,000 °C is GeO_2 (Δm_{TG} = 88.00%, Δm_{theor} = 88.20% (**1**), Δm_{TG} = 88.00%, Δm_{theor} = 88.10% (**2**), Δm_{TG} = 85.00%, Δm_{theor} = 88.30% (**3**)).

When analyzing the IR spectra of **1–3** (Figs. 2, 3), it was noted that the existence of bound OH groups in the molecules of the complexes is unambiguously determined by the presence of a broad band at ~3.400 cm^{-1}, as well as the Ge-O valence vibrations at 869 (for **1**, **3**) and 872 cm^{-1} (for **2**).

Coordination of the oxygen atoms of the phenoxy groups is accompanied by a shift in the C_{ph}-O-H vibration band from 1.266 cm^{-1} in gallic acid to 1.219, 1.223 and 1.224 cm^{-1} in complexes **1**, **2**, and **3**, respectively, rather than a splitting to 1.268 and 1.238 cm^{-1}, as in the previously studied complex germanium(IV) with gallic acid [13]. That is, all hydroxyl groups of the ligand are deprotonated. The carboxyl group of gallic acid is uncoordinated, but it shifts slightly to the low-frequency region to ν(COOH) = 1.679, 1.674 and 1.689 cm^{-1} in complexes **1**, **2**, and **3**, respectively. This can be explained by the participation of carboxyl groups in a branched hydrogen-bond network.

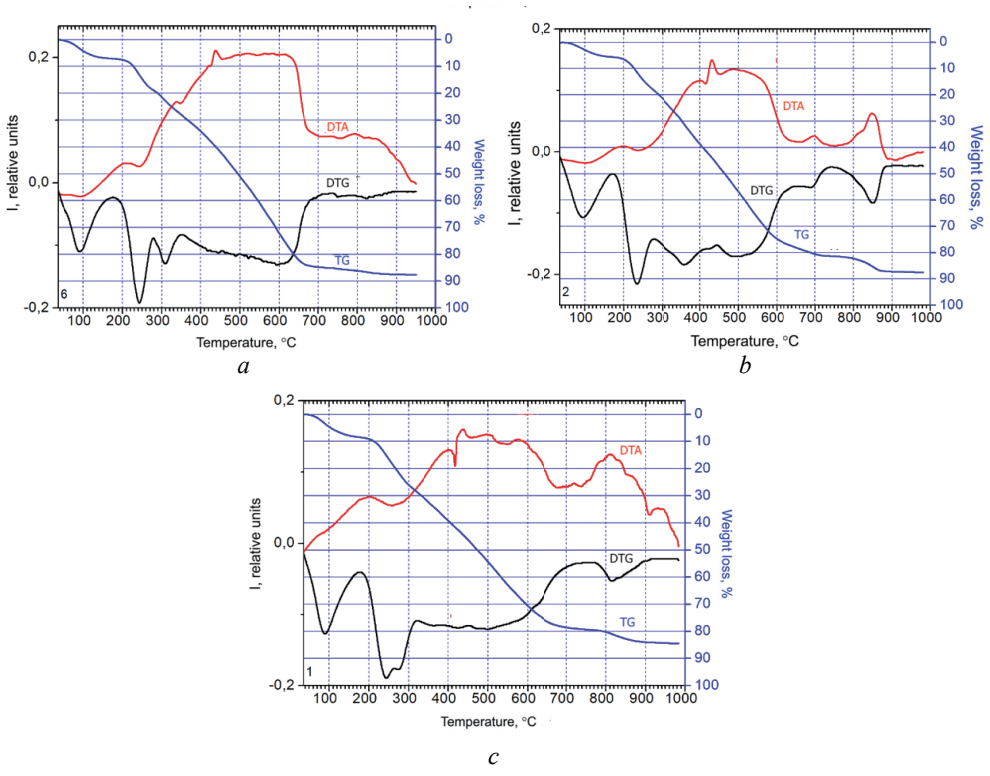


Fig. 1. Thermogravigrams of complexes **1** (*a*), **2** (*b*) and 3 (c)

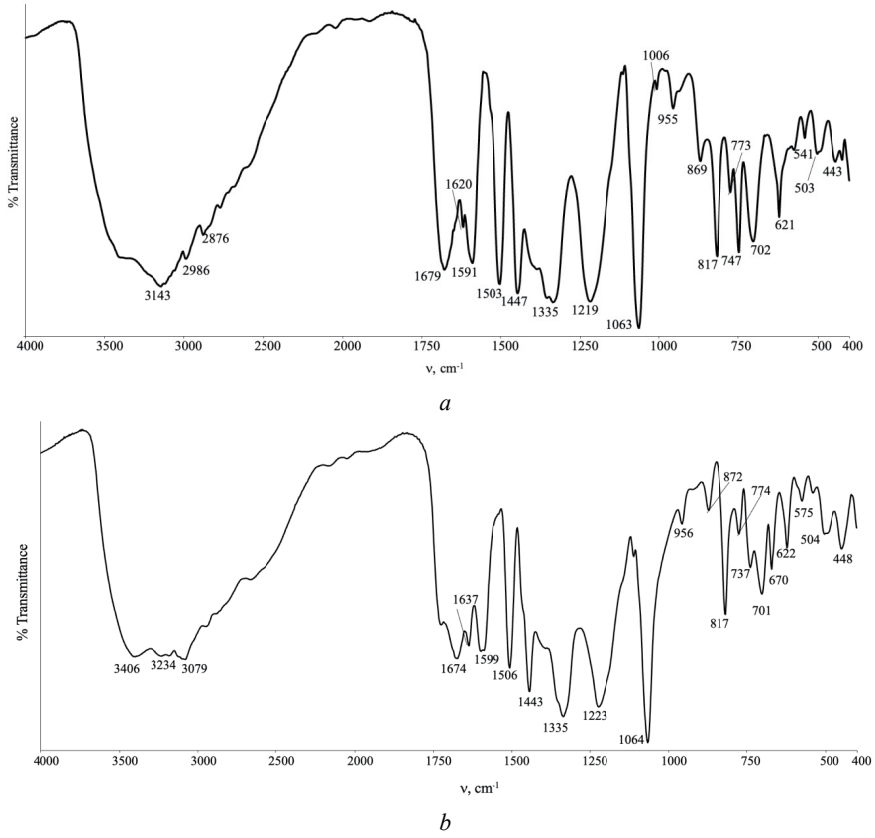


Fig. 2. IR spectra of complexes **1** (*a*) and **2** (*b*)

Comparative analysis of the IR spectra of complexes **1–3** and the initial nitrogen-containing molecules Bim, Nic, and Nad showed that onium compounds are formed due to protonation of the nitrogen atom of the heterocyclic ring. An increase in the frequency of its vibrations ν(CN) by 19–29 cm^{-1} is observed (1.567, 1.570 and 1580 cm^{-1} for Bim, Nic and Nad, and 1.591, 1.599 and 1599 cm^{-1} for **1**, **2** and **3**, respectively).

The behavior of cations and anions in solution was studied by mass spectrometry. ESI(-) mass spectrum of compounds **1–3** (Fig. 4) in negative polarity contains the signal of the gallic acid anion H_3Gal^- (m/z = 169), two small signals of the complex anions $[Ge(H_2Gal)_3]^{2-}$ (m/z = 289), $[Ge(H_3Gal)(H_2Gal)_2]^-$ (m/z = 579) and several signals that can be formed during fragmentation of the complex anion, e.g. m/z = 437–441.

The mass spectra of the ESI(+) compounds in positive polarity contain signals of protonated benzimidazole (m/z = 119), niacin (m/z = 124), and niamide (m/z = 123), as well as several peaks of solvents and unidentified fragments.

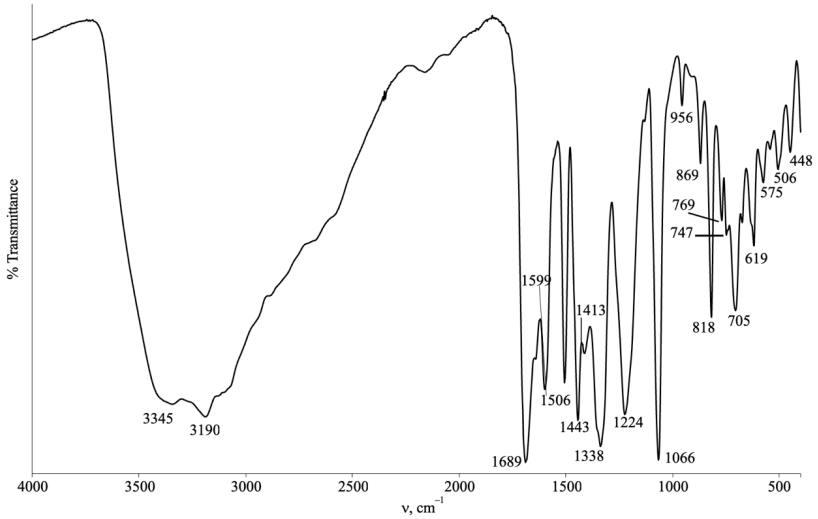
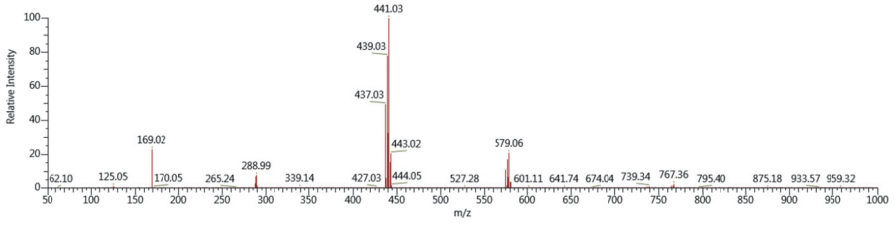
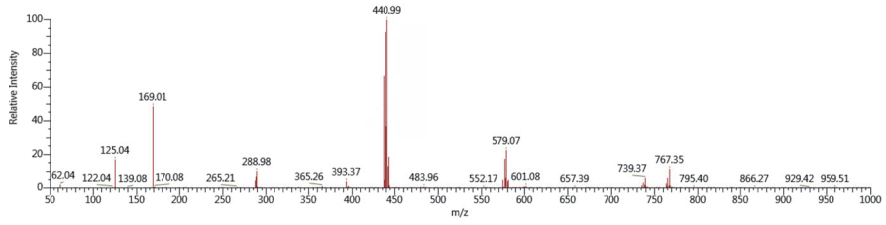


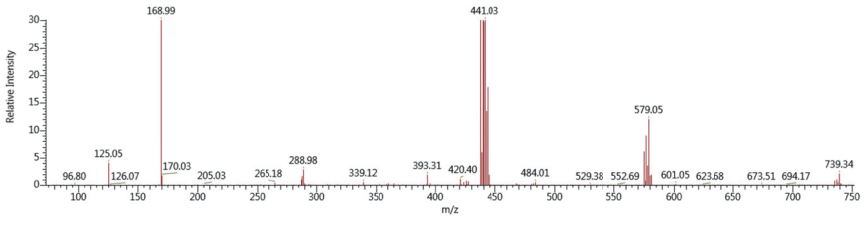
Fig. 3. IR spectrum of complex **3**



a



b



c

Fig. 4. ESI(-)-mass spectra of the solution of complexes **1** (*a*), **2** (*b*), and **3** (*c*)

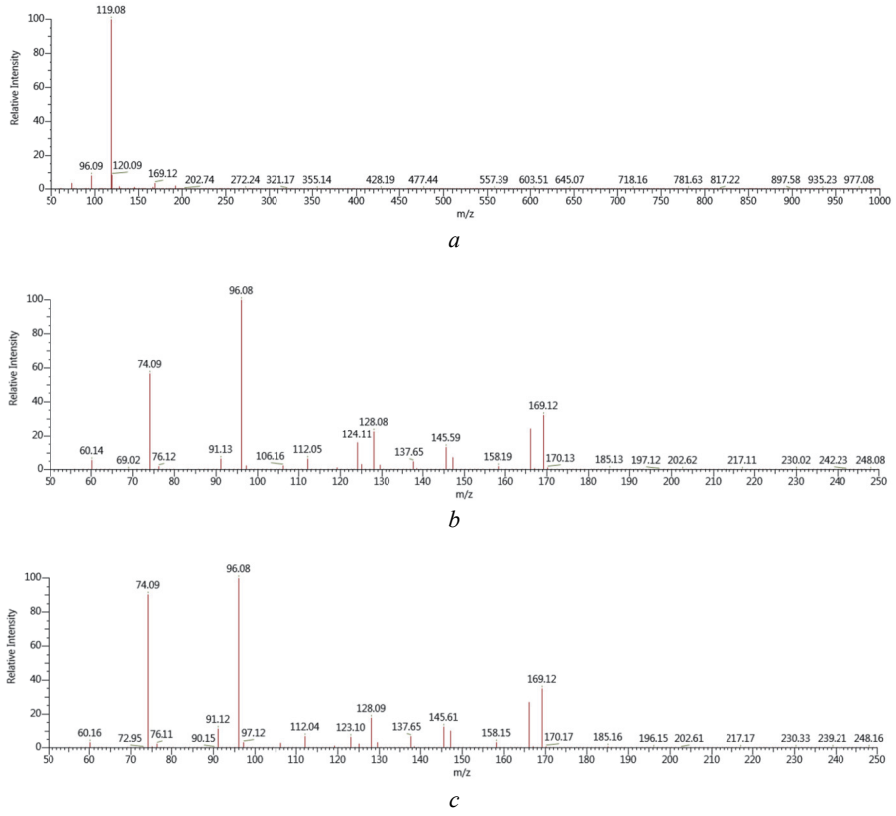


Fig. 5. ESI(+)-mass spectra of the solution of complexes **1** (*a*), **2** (*b*), and **3** (*c*)

Based on the combined results of elemental analysis, thermogravimetry, infrared spectroscopy, and mass spectrometry, considering the coordination number 6 characteristic of Ge(IV), it can be concluded that the obtained onium salts belong to the cation-anion type. They contain the tris(galato)germanate anion and organic molecules protonated at the nitrogen atom of the heterocycle as the cation. The structure schemes for the synthesised complexes $(HBim)_2[Ge(H_2Gal)_3]\cdot 4H_2O$ (**1**), $(HNic)_2[Ge(H_2Gal)_3]\cdot 3H_2O$ (**2**), $(HNad)_2[Ge(H_2Gal)_3]\cdot 4H_2O$ (**3**) were proposed (Fig. 6).

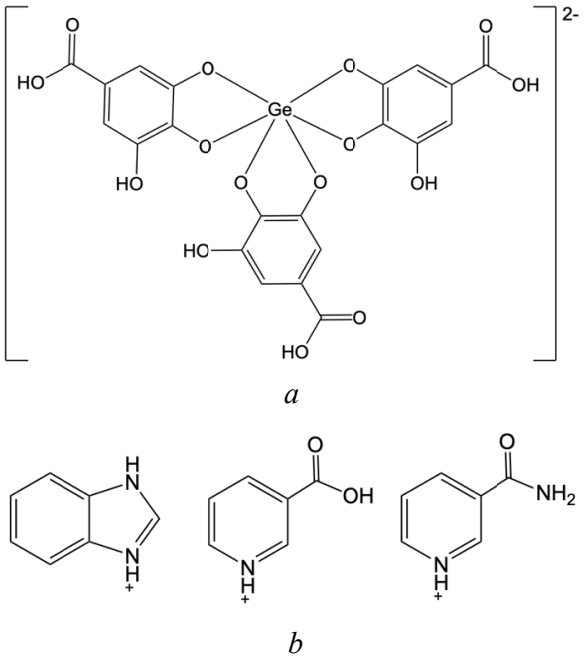


Fig. 6. Schemes of the structures of the complex anion (*a*) and protonated cations (*b*) in compounds **1-3**

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Стаття надійшла до редакції 15.04.2026
Стаття прийнята до друку після рецензування 06.05.2026
Стаття оприлюднена на вебсайті журналу 18.09.2026

М. А. Мартинюк[1], **О. Е. Марцинко**[1], **О. А. Фінік**[2], **О. В. Снурнікова**[2]
[1] Одеський національний університет імені І. І. Мечникова,
кафедра неорганічної хімії та хімічної освіти,
вул. Університетська 12, м. Одеса, 65082, Україна;
e-mail: lborn@ukr.net
[2] ТОВ «БЮРО ВЕРІТАС КОММОДІТІЗ УКРАЇНА»,
пров. Удільний, 1, м. Одеса, 65044, Україна

СИНТЕЗ ТА БУДОВА ОНІЄВИХ ГАЛАТОГЕРМАНАТІВ(IV) З НІТРОГЕНВМІСНИМИ ОРГАНІЧНИМИ КАТІОНАМИ

Розроблено оптимальні методи синтезу комплексів германію(IV) з галовою кислотою (H_4Gal) та екзолігандами бензімідазолом (Bim), нікотиновою кислотою (Nic) і нікотинамілом (Nad) у водному розчині. Склад і будову синтезованих комплексів визначено за допомогою елементного аналізу, ІЧ-спектроскопії та мас-спектрометрії. Показано, що в сполуках реалізується мольне співвідношення Германій : галова кислота : екзо-ліганд = 1:3:2. Запропоновано такі формули синтезованих сполук: $(HBim)_2[Ge(H_2Gal)_3]\cdot 4H_2O$ (**1**), $(HNic)_2[Ge(H_2Gal)_3]\cdot 3H_2O$ (**2**), $(HNad)_2[Ge(H_2Gal)_3]\cdot 4H_2O$ (**3**). Терморозпад сполук є подібним, в інтервалі температур 60–180 °C спостерігається ендотермічний ефект, який супроводжується зменшенням маси, що відповідає 4 (для комплексів **1** та **3**) і 3 (для комплексу **2**) молекулам

кристалізаційної води. При аналізі ІЧ-спектрів комплексів доведена координація депротонованих гідроксогруп галової кислоти до Германію, що визначається як смугою валентних коливань ν(Ge-O), так і зміщенням смуги деформаційних коливань $\delta(C_{ph}\text{-O-H})$ = 1266 см$^{-1}$ у галовій кислоті до ~ 1 220 см$^{-1}$ у комплексах. Мас-спектри ESI(-) сполук містять сигнали аніону галової кислоти H_3Gal^- (m/z = 169), два сигнали комплексних аніонів $[Ge(H_2Gal)_3]^{2-}$ (m/z = 289), $[Ge(H_3Gal)(H_2Gal)_2]^-$ (m/z = 579) та кілька сигналів, які можуть формуватися під час фрагментації комплексного аніону. Показано, що у сполуках в координації з Германієм(IV) беруть участь дві гідроксильні групи від кожного ліганду, утворюючи октаедричний координаційний поліедр. Отримані онієві солі належать до катіон-аніонного типу. Вони містять трис(галато)германатний аніон та протоновані за атомом Нітрогену гетероциклу органічні молекули в якості катіонів.

Ключові слова: германій, галова кислота, координаційні сполуки, ІЧ-спектроскопія, термогравіметричний аналіз, мас-спектрометрія.

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ORCID iDs

M. A. Martyniuk https://orcid.org/0009-0001-2730-6291
O. E. Martsynko https://orcid.org/0000-0002-3374-5987
O. A. Finik https://orcid.org/0000-0002-3770-615X
O. V. Snurnikova https://orcid.org/0009-0004-6530-5427