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OPTIMISATION METHOD FOR OBTAINING A BIOLOGICALLY ACTIVE SUBSTANCES FROM THE PLANT *PETROSIMONIA* *BRACHIATA*

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Abstract. Kazakhstan is rich in medicinal plants, so it needs to be studied. Ten species of the *Petrosimonia* belonging to the *Chenopodiaceae* family are growing in Kazakhstan. These plants have been adapted to harsh climatic conditions in the country's salty, saline, and desert areas. *Petrosimonia* plants are a new species that has not been fully studied. Chinese researchers studied *Petrosimonia sibirica* species and found that the plant contains alkaloids, steroids, terpenes, flavonoids, and phenolic acids and has antibacterial activity (Wen et al, 2015; Ying et al, 2016). The qualitative and quantitative compositions of biologically active substances in *P. sibirica*, *P. Glaucescens*, *P. triandra*, and *P. brachiata* growing in Kazakhstan were studied. To prepare phytopreparations from *P. Sibirica*, *P. Glaucescens* and *P. triandra* plants were extracted using the classical maceration method, and individual representatives of biologically active compounds were isolated (Toktarbek et al, 2021). In this study, a phytochemical study of *P. brachiata* was conducted. The method for obtaining biologically active complexes with high efficiency was optimised by using supercritical fluid and ultrasonic extraction methods. These methods have a short extraction time, can be carried out at room temperature, are cost effective, and are a modern green chemical approach. Using supercritical fluid CO₂ extraction (180 bar, 2 hours), the plant was purified from lipophilic substances. Further, a biologically active complex was obtained by pouring 70% ethanol-water solvent into the plant raw material and performing ultrasound extraction. The obtained complex was analysed by thin-layer chromatography using different organic solvent

systems. Based on our analysis, steroids, terpenes, phenolic acids, and flavonoid glycosides were found in the extract. Hexane and ethyl acetate fractions were obtained as a result of liquid-liquid extraction of the extract using organic solvents. Stigmasterol 3-O- β -D-galactopyranoside, isovanillic acid, quercetin 3-O- β -D-glucopyranoside, and isorhamnetin 3-O- α -L-rhamnopyranoside were isolated by washing the hexane and ethyl acetate fractions in a silica gel column with an organic solvent system. The isolated compounds will be tested for biological activity.

Keywords: *Chenopodiaceae* family, *Petrosimonia brachiata*, ultrasonic extraction, supercritical fluid extraction, chromatography.

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PETROSIMONIA BRACHIATA ӨСІМДІГІНЕН БИОЛОГИЯЛЫҚ БЕЛСЕНДІ ЗАТТАРДЫ АЛУ ӘДІСІН ОҢТАЙЛАНДЫРУ

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Аннотация. Қазақстан аумағы дәрілік өсімдіктерге өте бай және олар зерттеуді қажет етеді. Еліміз аумағында Алабұта (*Chenopodiaceae*) туысына жататын *Petrosimonia* тұқымдасының 10 түрі өседі. Бұл өсімдіктер еліміздегі тұзды, сортаң және шөлейтті жерлерде қатаң климат жағдайына бейімделген. *Petrosimonia* өсімдіктері толық зерттелмеген жаңа нысан болып табылады. Қытай ғалымдары *Petrosimonia sibirica* түрін зерттеп, өсімдік құрамында алкалоидтар, стероидтар, терпендер, флавоноидтар және фенол қышқылдары қатарлы екіншілік метаболиттердің бар екенін анықтап, бактерияға қарсы белсенділік көрсеткенін дәлелдеген (Wen et al, 2015; Ying et al, 2016). Қазақстанда өсетін *P. Sibirica*, *P. Glaucescens*, *P. triandra* және *P. brachiata* түрлерінің құрамындағы биологиялық белсенді заттардың сапалық және сандық құрамы зерттелінген. *P. Sibirica*, *P. Glaucescens* және *P. triandra* өсімдіктерінен фитопрепарат алу үшін класикалық мацерация-экстракцияланып, биологиялық белсенді заттардың жеке өкілдерін бөліп алған (Toktarbek et al, 2021). Бұл зерттеуде *P. brachiata* өсімдігіне фитохимиялық

зерттеу жүргізілді. Жоғары критикалық флюидті және ультрадыбысты экстракция әдістерін пайдалану арқылы тиімділігі жоғары биологиялық белсенді кешен алудың жолы оңтайландырылды. Аталған әдістер экстракция уақыты қысқа, бөлме температура жағдайында жүргізілетін, артық шығынсыз және заманауи жасыл химиялық бағыт болды. Жоғары критикалық флюидті CO₂ экстракцияны (180 бар, 2 сағат) қолданып өсімдік құрамы липофильді заттардадан тазартылды. Ары қарай өсімдік шикізатына 70% этанол-су еріткішін құйып, ультрадыбыспен экстракция жасау арқылы биологиялық белсенді кешен алынды. Алынған кешенге жұқа қабатты хроматографияда, әр түрлі органикалық еріткіштер жүйесі көмегімен сараптама жасалынды. Сараптаудың негізінде экстракт құрамында сетроидтар, терпендер, фенол қышқылдары және флавоноид гликозидтерінің бары анықталды. Экстрактіге органикалық еріткіштерімен сұйық-сұйықтық экстракциясын жүргізу нәтижесінде гексан және этилацетат фракциялары алынды. Гексан және этилацетат фракцияларын силикагель бағанасында органикалық еріткіштер жүйесі арқылы жуу нәтижесінде стигмастеролдың 3-О-β-D-галактопиранозиді, изованил қышқылы, кверцетиннің 3-О-β-D-глюкопиранозиді және изорамнетиннің 3-О-α-L-рамнопиранозиді бөлінді. Бөлінген жеке заттардың биологиялық белсенділігін тексеру жоспарлануда.

Түйін сөздер: *Chenopodiaceae* тұқымдасы, *Petrosimonia brachiata*, ультрадыбыстық экстракция, жоғары критикалық флюидті экстракция, хроматография.

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СПОСОБ ОПТИМИЗАЦИИ ПОЛУЧЕНИЯ БИОЛОГИЧЕСКИ АКТИВНЫХ ВЕЩЕСТВ ИЗ РАСТЕНИЯ *PETROSIMONIA BRACHIATA*

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Аннотация. Казахстан богат лекарственными растениями, поэтому его необходимо изучать. В Казахстане произрастают десять видов *Petrosimonia* семейства маревых. Эти растения адаптированы к суровым климатическим условиям засоленных и пустынных территорий страны. Растения *Petrosimonia* – новый вид, до конца не изученный. Китайские ученые изучили виды *Petrosimonia sibirica* и обнаружили, что растение содержит алкалоиды, стероиды, терпены, флавоноиды и фенольные кислоты и обладает антибактериальной активностью

(Wang et al, 2015; Ying et al, 2016). Изучен качественный и количественный состав биологически активных веществ *P. sibirica*, *P. Glaucescens*, *P. triandra* и *P. brachiata*, произрастающих в Казахстане. Для приготовления фитопрепаратов из растений *P. Sibirica*, *P. Glaucescens* и *P. triandra* экстрагировали классическим методом мацерации и выделяли отдельные представители биологически активных соединений (Toktarbek et al, 2021). В данном исследовании было проведено фитохимическое исследование *P. brachiata*. Оптимизирован метод получения биологически активных комплексов с высокой эффективностью за счет использования методов сверхкритической флюидной и ультразвуковой экстракции. Эти методы имеют короткое время экстракции, могут проводиться при комнатной температуре, являются экономически эффективными и представляют собой современный экологически чистый химический подход. С помощью сверхкритической флюидной CO₂-экстракции (180 бар, 2 часа) установка была очищена от липофильных веществ. Далее биологически активный комплекс получали путем заливки в растительное сырье 70% растворителя этанол-вода и проведения ультразвуковой экстракции. Полученный комплекс анализировали методом тонкослойной хроматографии с использованием различных систем органических растворителей. На основании нашего анализа в экстракте были обнаружены стероиды, терпены, фенольные кислоты и флавоноидные гликозиды. Гексановую и этилацетатную фракции получали в результате жидкостной экстракции экстракта органическими растворителями. Стилмастерол 3-О-β-D-галактопиранозид, изованилиновая кислота, кверцетин 3-О-β-D-глюкопиранозид и изорамнетин 3-О-α-L-рамнопиранозид выделены промывкой гексановой и этилацетатной фракций на силикагеле. Колонка с системой органических растворителей. Выделенные соединения будут проверены на биологическую активность.

Ключевые слова: семейство *Chenopodiaceae*, *Petrosimonia brachiata*, ультразвуковая экстракция, сверхкритическая флюидная экстракция, хроматография

Introduction

Medicinal products of plant origin have been used since ancient times, and even ancient times, civilisations were famous for using plants for healing. The oldest records date back to approximately 5,000 years when plants were used medicinally. The Greeks developed medicinal plants and herbs. Theophrastus (ca. 300 BC) wrote «Historia Plantarum», or the «History of Plants», one of the most important books on plant pharmacology and ancient natural history, in which he discussed the anatomy of plants and their pharmacological uses (Atanasov et al, 2021).

Much of the knowledge of Greco-Roman medicine was lost during the Middle Ages, but Arab and Islamic scholars from Andalusia and the Middle East were able to preserve and update the practises of that era. This progress in medicine occurred between the 9th and 12th centuries, after which the Renaissance began, and science began to be studied and developed more and more in the Western world. For thousands of years, plant extracts have been used to treat diseases. The past 200 years have witnessed

the discovery, isolation, and determination of the structures of thousands of natural compounds.

The new drug discovery approach has not hindered the study of natural compounds. A study of all newly approved drugs from 1981 to 2014 found that a total of 1211 new drugs were approved worldwide. Among them, 320 were natural compounds or their derivatives. These drugs comprise 32.7% of all authorised drugs in the world in the last 30 years (Li et al, 2018).





Compounds obtained from natural sources are not only medicinal products but also important tools for the discovery of new aspects of physiology. Currently, there is an interest in systematic research on low-molecular-weight inhibitors of the main steps of biochemical processes. Because many assays involve phenotyping, changes in living cells are likely to provide natural products that are useful as probes in such studies.

Our research objective: Plants belonging to the *Chenopodiaceae* family that grow in saline environments, are viable, and have high physiological capacity. They are widely used in folk medicine for the treatment of digestive, respiratory, genitourinary, and blood vessel disorders (Boneva et al, 2022). There are 11 species of *Petrosimonia* plants belonging to the *Chenopodiaceae* family worldwide, and 10 species grow in the desert and the desert regions of Kazakhstan. These species are: *P. monandra*, *P. triandra*, *P. litwinowii*, *P. squarrosa*, *P. hirsutissima*, *P. crassifolia*, *P. glaucescens*, *P. brachyphylla*, *P. glauca*, *P. brachiata*, and *P. sibirica*. The chemical compositions of all types of *Petrosimonia* plants have not been fully studied. The qualitative and quantitative compositions of biologically active substances in *P. sibirica*, *P. Glaucescens*, *P. triandra*, and *P. brachiata* growing in Kazakhstan were studied. According to the methods of the first edition of the State Pharmacopeia of the Republic of Kazakhstan, qualitative and quantitative analysis of biological active substances was performed, and the authenticity of plant raw materials was determined. The results of the study are shown in table 1 and table 2 (Seitimova et al, 2022).

Table 1. Quantification and authenticity of the main biological active groups of *P. Sibirica*, *P. Glaucescens*, *P. triandra*, and *P. Brachiata*

Plant names	<i>P. triandra</i>	<i>P. glaucescens</i>	<i>P. brachiata</i>	<i>P. sibirica</i>
Quality indicators of plant raw materials, (%)±SEM				
Humidity	8.09±0.03	5.90±0.04	10.22±0.04	7.81±0.03
Ash content	17.52±0.07	24.50±0.06	20.45±0.15	24.71±0.06
Extractives (80% ethanol-water)	42.70±0.04	46.10±0.02	46.90±0.06	52.90±0.04
Quantification of the main groups of biologically active substances, (%)±SEM				
Saponins	4.53±0.03	8.6±0.03	1.92±0.03	0.6±0.02
Flavonoids	2.55±0.04	4.1±0.03	4.53±0.04	1.92±0.04
Taninns	1.75±0.03	1.1±0.02	0.1±0.01	0.1±0.01
Alkaloids	1.56±0.04	0.27±0.03	0.53±0.04	0.4±0.03
Polysaccharides	1.78±0.01	1.4±0.01	5.14±0.02	4.2±0.04
Free organic acids	0.52±0.03	0.6±0.04	5.51±0.04	3.5±0.04
Coumarins	0.18±0.02	0.9±0.03	0.13±0.03	0.3±0.02

Table 2. Comparative conditions for extracting plant raw materials

Plant Names	<i>Petrosimonia triandra</i>	<i>Petrosimonia glaucescens</i>	<i>Petrosimonia brahiata</i>	<i>Petrosimonia sibirica</i>
Plant pictures				
Extraction type	First, lipophilic substances were removed using super critical fluid extraction, followed by extraction using the maceration method.	Maceration method	First, lipophilic substances were removed using super critical fluid extraction, followed by extraction using ultrasonic method.	Maceration method
Solvent	80% ethanol-water	80% ethanol-water	80% ethanol-water	80% ethanol-water
Extraction time	For super critical fluid extraction 2 hours and for maceration 72 hours	72 hours	For super critical fluid extraction 2 hours and for ultrasonic extraction 30 minutes	72 hours
Extraction temperature	Room temperature	Room temperature	40 °C	Room temperature
Biological activity of the obtained extracts	Antibacterial activity	No activity	Anti-inflammatory activity	No activity

It is important to use modern advanced technologies to obtain ecologically clean products and reduce factors that have an indirect effect on the extraction and distribution of biologically active substances from plant raw materials, especially secondary metabolites. The potential of CO₂ supercritical fluids and ultrasonic extraction to solve these problems is enormous (Nguyen et al, 2023; Shi et al, 2023; Herrero, 2024).

Materials and methods

Materials

The plant raw material was as follow:

The research object was the above-ground parts of *P.brachiata* plant belonging to the *Chenopodiaceae* family. This plant was collected in September 2023 from the saline land of the Enbekshikazakh district of the Almaty region. *Petrosimonia's* specie plant was identified with the help of leading specialists at the Institute of Botany and phytroduction, Almaty. Collected plant was dried at room temperature and protected from light. The dried plant raw material was ground to a diameter of 4 mm using sieves.

Solvents used:

1) Common solvents:

Ethanol and water were used as extractants for extracting plant raw materials.

2) Solvents for NMR spectroscopy:

Dimethylsulfoxide DMSO-d₆ (Cambridge Isotope Laboratories, Inc.), Methanol CD₃OD-d₄ (Cambridge Isotope Laboratories, Inc.), and Acetone C₃D₆O-d₆ (Cambridge Isotope Laboratories, Inc.).

Chromatography materials

Normal phase thin-layer chromatography (Aluminum Silica gel 60 F254) Merck KgaA; Reversed phase thin-layer chromatography (Glass Silica gel 60 RP-18 F254S) Merck Millipore; Silica gel 60, 0.04 – 0.063 mm (230 – 400 μm) Merck; Silica gel 60, 0.063 – 0.200 mm (70 – 230 μm) Merck, and Column.

Methods*Research methods*

For extraction procedure: Supercritical fluid CO₂ extraction and ultrasonic extraction; Isolation and purification procedure: chromatography; Structural elucidation procedure: ¹H-NMR - AVANCE NEO-400, at 400 MHz and ¹³C-NMR, BB, DEPT - AVANCE NEO-400 at 100, 125 and 150 MHz; two-dimensional: NMR ¹H-¹³C HSQC, HMBC, ¹H-¹H COSY-45 °C, NOESY, IR -spectroscopy (Bruker Vector 22, Japan), EI-MS (JEOL 600H-1, Inlet: Direct Probe), FAB-MS (JEOL 600H-2, Inlet: Direct Probe), and melting points were determined by Buchi M-560 apparatus.

Preparation of extracts

The plant raw material was treated to eliminate lipophilic substances using supercritical fluid CO₂ extraction at 180 bar, followed by extraction with 80% ethanol-water at a ratio of 1:8 for 2 hours at room temperature twice by an ultrasonic extractor. The prepared extracts were combined and concentrated under vacuum at a temperature of 45-50 °C. The suspension was prepared by adding 500 ml of distilled water to 177 g of dry extract. Sequential liquid-liquid extraction was performed by adding hexane and ethyl acetate to the suspension according to polarity, and all extracts were desolvated in a rotary evaporator at temperatures not exceeding 50 °C.

The hexane extract was qualitatively analyzed by TLC (solvent system n-Hexane: ethyl acetate 9:1 → 1:9), a solution of Ce(SO₄)₂ in 15% H₂SO₄ was used as a reagent. The presence of lipophilic substances such as steroids and terpenes was determined in this study.

General experimental procedure for isolation of compounds

Silica gel column chromatography was used to separate 10 g of hexane extract into fractions and isolate individual compounds. The column was first washed with 100% hexane solvent, and then chromatography was performed by increasing the concentration of the polar solvent (ethyl acetate, methanol) according to the hexane:ethyl acetate 10:1 → 1:10, 100% ethyl acetate, and ethyl acetate:methanol 10:1 → 7:3. The fractions were combined with similar R_f values and spot colors in TLC to obtain 10 (H1-H10) fractions. Each fraction was concentrated under mild conditions using a rotary evaporator. Fraction H10 was isolated as a pure compound, corresponding to **1** (35 mg).

The ethyl acetate fraction was qualitatively analyzed with the help of TLC (solvent system n-Hexane:ethyl acetate:acetone 8:1:1), as well as a solution of the determining reagent $\text{Ce}(\text{SO}_4)_2$ in 15% H_2SO_4 was sprinkled, and as a result of analyzing the color of the spots formed under UV light (254 and 366 nm), the extract contained phenolic acids, flavonoid aglycones, flavonoid glycosides, and alkaloidal compounds were detected (Li, 2024).

5 g of dried and powdered ethyl acetate extract was chromatographed in SG-60 column chromatography with increasing polarity: 100% hexane \rightarrow hexane-ethyl acetate (in different ratios) \rightarrow 100% ethyl acetate \rightarrow ethyl acetate-methanol (in different ratios) \rightarrow 100% methanol, resulting in 11 fractions (E1-E10) were obtained. Substance **2** (29 mg) was separated from E2 fraction. Pure compound **3** (42 mg) and subfraction E8S1 were separated from fraction E8. The subfraction E8S1 was purified by chromatography on a Sephadex LH-20 column with 100% methanol, and compound **4** (7 mg) was isolated.

The structures of isolated compounds were determined by melting point, IR, FAV-MS, and NMR data analyses.

Results and Discussions

Compounds **1-4** were isolated for the first time from the *Petrosimonia brachiata* species.

Compound **1** is a white crystalline powder, molecular formula $\text{C}_{35}\text{H}_{58}\text{O}_6$, molecular weight m/z 574, melting point 264-266 °C, a single spot in thin-layer chromatography (solvent system dichloromethane:methanol 9:1) after treatment with 15% H_2SO_4 showed a deep green.

The $^1\text{H-NMR}$ spectrum (DMSO, 500MHz) of compound **1** showed six methyl groups: δ_{H} 0.65 (3H, c, H-18), 0.79 (3H, д, $J = 8.1$, H-29), 0.80 (3H, д, $J = 6.9$, H-27), 0.84 (3H, d, $J = 6.3$, H-26), 0.95 (3H, d, $J = 6.3$, H-21), 0.99 (3H, s, H-19); proton occupying one olefinic position δ_{H} 5.15 (1H, br.d, $J = 4.8$, H-6); two protons 4.83 (1H, dd, $J = 8.4, 15$, H-23) and 4.97 (1H, dd, $J = 8.4; 15.0$, H-22); and one anomeric proton showed chemical shift values of 4.19 (1H, d, $J = 7.8$, H-1').

By analyzing the $^{13}\text{C-NMR}$ spectrum, this compound was found to contain 35 carbon signals. A chemical shift of δ_{C} 100.76 indicates the presence of a monosaccharide molecule with an anomeric carbon C-1', whereas chemical shift values of δ_{C} 69.87, 73.22, 75.54, and 76.15 indicate four methanes and methylene at δ_{C} 61.43 chemical shifts C-2', C-3', C-4', C-5', and C. According to the C-6' carbons, the product was found to be β -D-galactopyranose.

The δ_{C} 78.72 shift region corresponds to the C-3 carbon atom bonded to the alcohol hydroxyl group. Chemical shifts δ_{C} 121.71 (C-6), δ_{C} 137.92 (C-23), δ_{C} 128.79 (C-22), and δ_{C} 139.97 (C-5) represent olefinic carbons in the sterol molecule. The value of $J = 7.8$ Hz for the anomeric proton H-1' shows that it is in an axial position to the proton H-2', which indicates that this galactopyranoside fragment is bound to the sterol fragment in the β position.

Based on $^1\text{H-}$ and $^{13}\text{C-NMR}$ chemical shift values and physical data, it was proved that compound **1** is stigmasterol 3-O- β -D-galactopyranoside.

Compound **2** was isolated from the ethyl acetate extract. They were subjected to a qualitative reaction with TLC (Hexane:Ethyl acetate 5:1) using 15% H₂SO₄ and a brown spot formed. These compounds exhibited purple under UV light.

Compound **2** is a white crystalline compound with a melting point of 208–210 °C. The mass spectrum of the isolated compound EI-MS *m/z* 168.1 [M]⁺; molecular formula corresponds to C₈H₈O₄. The valence vibration band in the IR spectrum defines the C=O group at 1682 cm⁻¹ and the OH group at 3484 cm⁻¹. Absorption at 1598 and 1523 cm⁻¹ indicates C=C groups in the aromatic ring, and the band at 1206 cm⁻¹ indicates a C-O bond.

In the ¹H-NMR (CD₃OH, 500MHz) spectrum of the compound **2**, the chemical shift signal at δ_H 3.83 indicates the presence of an OCH₃ group in the molecular structure of the compound. The high-field signals δ_H chemical shifts at 7.61, 7.44, and 6.86 identify the H-2, H-6, and H-5 protons of the aromatic ring, respectively.

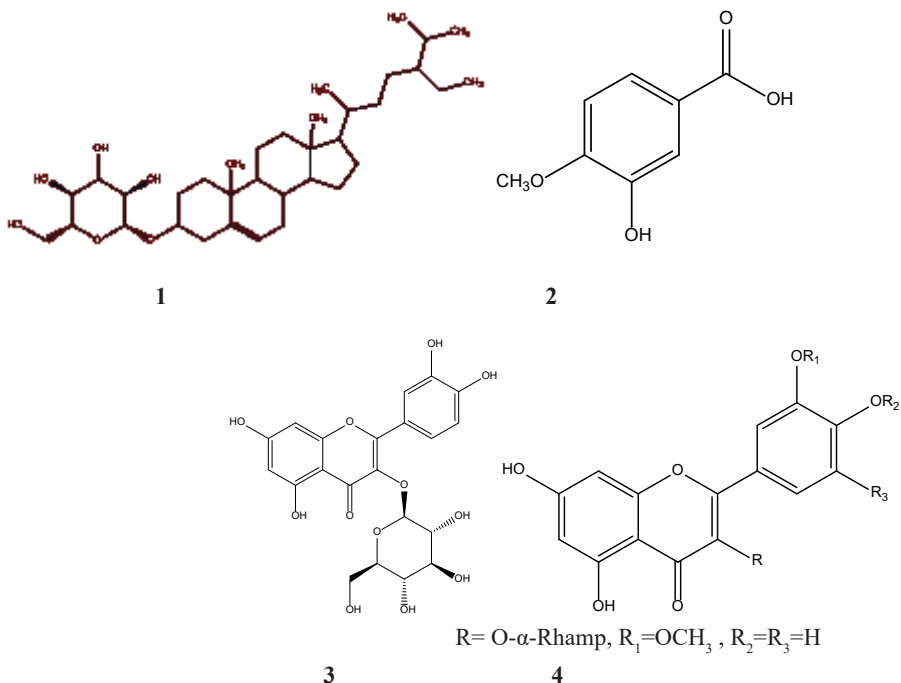
The ¹³C-NMR spectrum of the compound showed 8 signals with 3 methine, 4 quaternary and 1 methyl carbons. The δ_C 55.5 signal is the chemical shift characteristic of the carbon atom in the methoxy group. And the δ_C 167.2 signal region indicates the C=O group of the acidic fragment of the molecular structure. The high-field signals at δ_C 149.1 and 151.2 indicate the shift characteristics of the C-3 and C-4 carbons, respectively, in the aromatic ring. ¹H-NMR, ¹³C-NMR spectral data, etc. based on its physical properties, compound **2** was identified as isovanillic acid.

Compound **3** was isolated as a yellow powder with a melting point of 231–232 °C. FAB-MS corresponding to the molecular formula C₂₁H₂₀O₁₂ showed a molecular ion peak [M+H]⁺ at *m/z* 464.14. ¹H and ¹³C NMR spectra in CD₃OH solvent showed that the aromatic protons were resolved as a single ABX system (B ring). The δ_H 7.849 (d, *J* = 2.3 Hz, H-2'), δ_C 117.90 for δ_H 6.871 (d, *J* = 8.3 Hz); δ_C 116.3 and δ_H 7.586 for H-5' (dd, *J* = 8.3, 2.3 Hz) δ_C 123.05 for H-6'. Another ABX-based (A ring) system δ_H 6.204 (brs), δ_C 100.02 and δ_H 6.407 (brs), δ_C 94.85 assigned to H-6 and H-8 protons, respectively. The ¹³C values of C-3' and C-4' were 150.08, which were assigned 145.94 respectively. The anomeric sugar protons δ_H appeared at 5.16 (d, *J* = 7.53 Hz); δ_C is 103.95. An anomeric proton coupling constant *J* = 7.53 Hz confirmed the β-linkage of the sugar. Based on ¹H- and ¹³C-NMR chemical shift values and physical data, it was proved that compound **3** is quercetin 3-O-β-D-glucopyranoside.

Compound **4** was isolated as a yellow amorphous powder with an FAB-MS 462.40 at an *m/z* [M]⁺ molecular formula corresponding to C₂₂H₂₂O₁₁.

In the ¹H NMR (C₃D₆O) spectrum of compound **4**, three protons of the methoxy group exhibited a singlet signal in the region of δ_H = 3.94. Aromatic protons of ring B show doublet signals at 6.97 (1H, d, *J*=8.45, H-3') and 8.05 (1H, d, *J*=2, H-6'), and 7.66 (1H, dd, *J*=8.5, *J*= 2.05, H-2') proton was in the doublet doublet displacement level. In addition, we observed the aromatic H-6 and H-8 protons of ring A at the 6.28 (1H, t) and 6.53 (1H, t) triplet signal shifts, respectively. The anomeric H-1'' proton of the sugar moieties gave a doublet signal in the chemical shift region of 5.02 (1H, d, *J*= 2.05).

Using ¹³C NMR and HMBC spectra, the binding sites of the sugars were determined. By comparing the results of the physicochemical analysis with the literature data, compound **4** was identified as isorhamnetin 3-O-α-L-rhamnopyranoside (Bojilov et al, 2023).



Conclusion

First, a phytochemical study was conducted on *P. brachiata*. Plant raw materials were extracted using modern methods, including supercritical fluid extraction and ultrasound extraction. The obtained extract was subjected to liquid-liquid extraction using hexane and ethyl acetate solvents. The qualitative composition of the hexane and ethyl acetate fractions was analysed by chromatography, and the presence of steroids, terpene substances, and flavonoid classes was determined. For further purification of the hexane and ethyl acetate fractions, silica gel column chromatography was performed; stigmasterol 3-O- β -D-galactopyranoside, isovanillic acid, quercetin 3-O- β -D-glucopyranoside, and isorhamnetin 3-O- α -L-rhamnopyranoside compounds were isolated, and modern physicochemical methods, including ¹H-NMR, ¹³C-NMR, 2D NMR, EI-MS, FAB-MS, and IR spectroscopy were used to determine their structures. The isolated compounds will be tested for biological activity.

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SYNTHESIS AND RADIOGRAPHY OF NEW ZIRCON-MANGANITES OF LANTHANUM AND ALKALINE EARTH METALS AND CALCULATION OF THEIR THERMODYNAMIC PROPERTIES

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Abstract. The synthesis of $\text{LaMe}^{\text{II}}\text{ZrMnO}_6$ compounds ($\text{Me}^{\text{II}} - \text{Mg, Ca, Sr, Ba}$) was carried out using ceramic technology in the range of 800-1200°C by the interaction of lanthanum (III) oxides of the "extra clean" qualification, zirconium (IV), manganese (III) and magnesium, calcium, strontium and barium carbonates of the "clean for analysis" brand". The weights of the starting materials were weighed with an accuracy of up to the fourth decimal place. Stoichiometric amounts of the starting materials were carefully ground in an agate mortar, then poured into alund crucibles and subjected to

heat treatment for solid-phase interaction in air in the "SNOL" furnace.

X-ray methods have established that all synthesized zircon-manganites crystallize in cubic symmetry with the following lattice parameters: $\text{LaMgZrMnO}_6 - a = 13,46 \pm 0,02 \text{ \AA}$; $V^0 = 2437,86 \pm 0,05 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 609,47 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 4,42 \text{ g/cm}^3$; $\rho_{\text{picn}} = 4,35 \pm 0,07 \text{ g/cm}^3$; $\text{LaCaZrMnO}_6 - a = 14,50 \pm 0,02 \text{ \AA}$; $V^0 = 3048,63 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 762,16 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 3,67 \text{ g/cm}^3$; $\rho_{\text{picn}} = 3,62 \pm 0,05 \text{ g/cm}^3$; $\text{LaSrZrMnO}_6 - a = 14,56 \pm 0,02 \text{ \AA}$; $V^0 = 3087,52 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 771,88 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 4,03 \text{ g/cm}^3$; $\rho_{\text{picn}} = 3,99 \pm 0,04 \text{ g/cm}^3$; $\text{LaBaZrMnO}_6 - a = 14,79 \pm 0,02 \text{ \AA}$; $V^0 = 3233,45 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 808,36 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 4,25 \text{ g/cm}^3$; $\rho_{\text{picn}} = 4,19 \pm 0,05 \text{ g/cm}^3$. It was found that with an increase in the ionic radii in the $\text{Mg} \rightarrow \text{Ca} \rightarrow \text{Sr} \rightarrow \text{Ba}$ series, the values of the parameter "a", the volumes of lattices and elementary cells of zircon-manganites increase. The calculation of the temperature dependence of the heat capacity and the standard heat capacity by indirect methods is carried out. The equations of temperature dependences of the investigated zircon-manganites of lanthanum and alkaline earth metals are derived.

Keywords: lanthanum, zircon-manganite, alkaline earth metals, synthesis, radiography, heat capacity.

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ЖАҢА ЛАНТАН ЖӘНЕ СІЛТІЛІ-ЖЕР МЕТАЛДАРЫ ЦИРКОН МАНГАНИТТЕРІНІҢ СИНТЕЗІ МЕН РЕНТГЕНОГРАФИЯСЫ ЖӘНЕ ОЛАРДЫҢ ТЕРМОДИНАМИКАЛЫҚ ҚАСИЕТТЕРІН ЕСЕПТЕУ

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Аннотация. $\text{LaMe}^{\text{II}}\text{ZrMnO}_6$ ($\text{Me}^{\text{II}} - \text{Mg, Ca, Sr, Ba}$) қосылыстарының синтезі керамикалық технология бойынша $800\text{-}1200^\circ\text{C}$ аралықтарында тазалығы «аса таза» лантан (III) тотығы, «талдау үшін таза» маркалы цирконий (IV), марганец (III) тотықтары мен магний, кальций, стронций және барий карбонаттарының өзара әрекеттесуімен жүргізілді. Бастапқы заттардың өлшемдері үтірден кейінгі төртінші орынға дейін өлшеніп алынды. Бастапқы заттардың стехиометриялық мөлшері ағат келіде мұқият ұнтақталған, содан кейін алунд тигельдеріге салынып, «SNOL» пешінде ауадағы қатты фазалық өзара әрекеттесу үшін термиялық өңдеуден өткен.

Рентгендік дифракция әдістерін қолдана отырып, барлық синтезделген циркон-манганиттер келесі тор параметрлері бар текшелік жүйеде кристалданатыны анықталды: $\text{LaMgZrMnO}_6 - a = 13,46 \pm 0,02 \text{ \AA}$; $V^0 = 2437,86 \pm 0,05 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.ұяш}} = 609,47 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 4,42 \text{ г/см}^3$; $\rho_{\text{никн.}} = 4,35 \pm 0,07 \text{ г/см}^3$; $\text{LaCaZrMnO}_6 - a = 14,50 \pm 0,02 \text{ \AA}$; $V^0 = 3048,63 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.ұяш}} = 762,16 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 3,67 \text{ г/см}^3$; $\rho_{\text{никн.}} = 3,62 \pm 0,05 \text{ г/см}^3$; $\text{LaSrZrMnO}_6 - a = 14,56 \pm 0,02 \text{ \AA}$; $V^0 = 3087,52 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.ұяш}} = 771,88 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 4,03 \text{ г/см}^3$; $\rho_{\text{никн.}} = 3,99 \pm 0,04 \text{ г/см}^3$; $\text{LaBaZrMnO}_6 - a = 14,79 \pm 0,02 \text{ \AA}$; $V^0 = 3233,45 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.ұяш}} = 808,36 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 4,25 \text{ г/см}^3$; $\rho_{\text{никн.}} = 4,19 \pm 0,05 \text{ г/см}^3$. $\text{Mg} \rightarrow \text{Ca} \rightarrow \text{Sr} \rightarrow \text{Ba}$ қатарындағы иондық радиустардың ұлғаюымен циркон-манганиттердің «а» параметрінің, тор көлемдерінің және бірлік ұяшықтарының мәндері өсетіні анықталды. Жылу сыйымдылығы мен стандартты жылу сыйымдылығының температураға тәуелділігі жанама әдістермен есептелді. Зерттеліп отырған лантан және сілтілі-жер металдарының циркон-манганиттерінің температураға тәуелділік теңдеулері шығарылды.

Түйін сөздер: лантан, циркон-манганит, сілтілі-жер металдары, синтез, рентгенография, жылу сыйымдылығы.

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СИНТЕЗ И РЕНТГЕНОГРАФИЯ НОВЫХ ЦИРКОНО-МАНГАНИТОВ ЛАНТАНА И ЩЕЛОЧНОЗЕМЕЛЬНЫХ МЕТАЛЛОВ И РАСЧЕТ ИХ ТЕРМОДИНАМИЧЕСКИХ СВОЙСТВ

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Аннотация. Синтез соединений $\text{LaMe}^{\text{II}}\text{ZrMnO}_6$ ($\text{Me}^{\text{II}} - \text{Mg, Ca, Sr, Ba}$) проводили по керамической технологии в интервале 800-1200°C взаимодействием оксидов лантана (III) квалификации «ос.ч.», циркония (IV), марганца (III) и карбонатов магния, кальция, стронция и бария марки «ч.д.а.». Навески исходных веществ, взвешивались с точностью до четвертого знака после запятой. Стехиометрические количества исходных веществ, тщательно перетирали в агатовой ступке, затем пересыпались в алундовые тигли и подвергались термообработке для твердофазного взаимодействия на воздухе в печи «SNOL».

Методами рентгенографии установлено, что все синтезированные цирконо-манганиты кристаллизуются в кубической сингонии со следующими параметрами решетки: $\text{LaMgZrMnO}_6 - a = 13,46 \pm 0,02 \text{ \AA}$; $V^0 = 2437,86 \pm 0,05 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.яч}} = 609,47 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 4,42 \text{ г/см}^3$; $\rho_{\text{пикн.}} = 4,35 \pm 0,07 \text{ г/см}^3$; $\text{LaCaZrMnO}_6 - a = 14,50 \pm 0,02 \text{ \AA}$; $V^0 = 3048,63 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.яч}} = 762,16 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 3,67$

z/cm^3 ; $\rho_{\text{никн.}} = 3,62 \pm 0,05 z/cm^3$; $LaSrZrMnO_6 - a = 14,56 \pm 0,02 \text{ \AA}$; $V^0 = 3087,52 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.яч}} = 771,88 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 4,03 z/cm^3$; $\rho_{\text{никн.}} = 3,99 \pm 0,04 z/cm^3$, $LaBaZrMnO_6 - a = 14,79 \pm 0,02 \text{ \AA}$; $V^0 = 3233,45 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{эл.яч}} = 808,36 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{рент.}} = 4,25 z/cm^3$; $\rho_{\text{никн.}} = 4,19 \pm 0,05 z/cm^3$. Установлено, что с повышением ионных радиусов в ряду $Mg \rightarrow Ca \rightarrow Sr \rightarrow Ba$ увеличиваются величины параметра «а», объемов решеток и элементарных ячеек цирконо-манганитов. Проведен расчет температурной зависимости теплоемкости и стандартной теплоемкости по косвенными методами. Выведены уравнения температурных зависимостей исследуемых цирконо-манганитов лантана и щелочноземельных металлов.

Ключевые слова: лантан, цирконо-манганит, щелочноземельные металлы, синтез, рентгенография, теплоемкость

Introduction

Zirconium dioxide (ZrO_2) is interesting as a ceramic material with valuable optical, electrical, thermal, strength and other characteristics, and phase transitions in it and impurity stabilization are the subject of many experimental and theoretical studies (Zimichev, et al, 2014; Zavodinsky, 2005). Zirconium dioxide is in third place among the available raw materials in terms of relative wear resistance after graphite and silicon carbide, which is confirmed by the practice of their operation in extreme conditions. Among the available highly refractory oxides, ZrO_2 occupies a leading position.

Oxide materials based on zirconates of rare earth elements (REE) are widely used in various industries. Thus, such materials are used to create thermal barrier coatings, oxygen-conducting solid electrolytes, as well as to immobilize nuclear waste (Mazilin, et al, 2013). Lanthanide zirconates $Ln_2Zr_2O_7$ have high melting points. These compounds exhibit a number of properties that suggest the high practical importance of developing new functional materials based on them. In particular, they are considered as new solid electrolytes with (Mazilin, et al, 2013) high oxygen-ion conductivity (Fergus, 2014). They exhibit dielectric, piezo- or ferroelectric, as well as fluorescent and phosphorescent properties, can be used as materials of electronic technology, as well as as catalysts (Díaz-Guillen, et al, 2015; Wang, et al, 2023; Shlyakhtina, et al, 2005). REE zirconates have chemical and radiation resistance, high radionuclides and are considered promising highly active materials for nuclear energy, and are also of interest as luminescent materials (Sohn, et al, 2002; Popov, et al, 2013; Erdogan, et al, 2017; Chena, et al, 2009; Zhong, et al, 2014; Mahade, et al, 2016; Gagarin, 2018; Merkushev, et al, 2010).

Lanthanum manganites are a unique system in which their electronic, magnetic and lattice properties are interconnected, which can be regulated by changing the chemical composition and the degree of doping. The search for new polyoxo compounds of zircon and manganese with rare earth elements doped with alkali and alkaline earth metal oxides and the study of their properties is of particular interest both for fundamental research and from the point of view of their practical use.

The aim of the work is to obtain new materials from lanthanum (III) oxides, alkaline earth metals, zirconium (IV) and manganese (III) in the form of zircono-manganites

LaMe^{II}ZrMnO₆ (Me^{II} – Mg, Ca, Sr, Ba), their X-ray studies and calculation of their thermodynamic properties.

Materials and basic methods

Solid-phase synthesis of LaMe^{II}ZrMnO₆ compounds (Me^{II} – Mg, Ca, Sr, Ba) was carried out using ceramic technology from lanthanum (III) oxides of the «extra clean» qualification, zirconium (IV), manganese (III) and magnesium, calcium, strontium and barium carbonates of the «clean for analysis» brand. The stoichiometric amounts of the starting substances, previously dehydrated at 400°C, were thoroughly mixed and ground in an agate mortar. Then they were annealed in alund crucibles in the "SNOL" furnace at first at 600°C for 10 hours, 800°C for 10 hours, 1000°C with and 1200°C for 20 hours. At each temperature, the mixtures were cooled to room temperature with repeated mixing and grinding processes and reheated. To obtain equilibrium phases at low temperatures, low-temperature annealing was performed at 400°C for 10 hours, followed by repetitions of mixing and grinding.

X-ray phase analysis (DRON-2.0 device) confirmed the production of synthesized zircono-manganites with a phase composition of LaMe^{II}ZrMnO₆ (Me^{II} – Mg, Ca, Sr, Ba). The intensity of diffraction maxima was estimated on a one-hundred-point scale.

The X-ray images were indexed by the analytical method (Cowba, et al, 1976) (Table 1).

The basic function in thermodynamic modeling is the temperature dependence of the heat capacity, knowledge of which allows us to calculate the change in enthalpy, entropy, and Gibbs energy, i.e. to obtain a wide range of thermodynamic properties of the substance under study (Chumilina, 2016). In fact, the heat capacity is a parameter that characterizes the ability of substances to accumulate and store thermal energy when temperature changes. The study of heat capacity makes it possible not only to calculate thermodynamic functions over a wide temperature range, but also to study various ordering processes that determine, for example, magnetic, ferroelectric or superconductivity properties, as well as the formation of point defects. In connection with the above, we further calculated the temperature dependence of the heat capacity of the studied zircono-manganites of lanthanum and alkaline earth metals.

Based on the dependence of the isochoric heat capacity on temperature according to Debye's theory, Landia (Veritin, et al, 1965) proposed a scheme for calculating the isobaric heat capacity of crystalline inorganic substances. According to Landius, the transition from isochoric to isobaric heat capacity is carried out according to the Magnus–Lindemann equation ($C_p = C_v + \alpha^{3/2}T$). We will carry out a complete calculation scheme borrowed from (Veritin, et al, 1965).

Initial data:

- 1) melting, dissociation and sublimation temperature (T, K);
- 2) standard entropy;
- 3) temperatures of polymorphic (ferromagnetic) transformations.

It should be noted that in his calculations, Landia used a calorie as a thermal unit. We also leave it unchanged.

Designations:

n - is the number of atoms in the compound;

h - is the number of hydrogen atoms in the compounds;

m- is the number of water molecules in crystallohydrates;

$C_v^{a.s.} = (6,6 - a/298) (n - 3m)$ is the heat capacity at V = const and T = 298 To anhydrous salt (in crystallohydrates), and "a" is calculated according to this scheme depending on the type of anhydrous salt and in its standard entropy;

$S^{at} = S^{298}/n$ - is the atomic entropy.

n - is the number of atoms in the compound;

S^{298} - is calculated from the ionic entropy increments of Kumok (Kumok, 1987).

When T is unknown, T' is taken, it is found from the formula $T' = 5070 / S^{at}$

Determine the coefficients a, B and K necessary for the calculation formulas (1-4) below, $a=507+1070 / S^{at}$, $\epsilon=0,8a$, $K=0,34$, $T_{metl} = T'$

Calculation formulas

C'_p is calculated as the sum of atomic heat capacities, which for individual elements are assumed to be equal in our case La, Mg, Ca, Sr, Ba, Zr, Mn=6,2,for O=4 cal/mol. degree

$$C''_p = n \left[6,6 - \frac{a}{298} + \frac{6,38}{T_{metl}} \left(6,6 - \frac{a}{298} \right)^2 \right], \quad (1)$$

$$C'''_p = n \left[6,6 - \frac{14,76}{S^{at}} \cdot \frac{n_1^2 + n_2^2}{n^2} + \frac{6,38}{T_{metl}} \left(6,6 - \frac{14,76}{S^{at}} \cdot \frac{n_1^2 + n_2^2}{n^2} \right)^2 \right], \quad (2)$$

where n_1, n_2 is the number of cation and anion atoms in the compound, represented as a salt of oxygen acid.

$$C_{p,T} = n \left[6,6 + \frac{1,24}{T_{Heating}} \left(6,6 - \frac{a}{298} \right)^2 T^{3/2} 10^{-3} \right], \quad (3)$$

$$C_{p,T} = n \left[6,6 - \frac{a}{\epsilon + K(T - \epsilon)} + \frac{1,24}{T_{Heating}} \left(6,6 - \frac{a}{298} \right)^2 T^{3/2} 10^{-3} \right], \quad (4)$$

(up to $T \ll \epsilon$, $K = 1$).

Results

Based on the indexing of radiographs, it was found that all synthesized zircon-manganites crystallize in cubic symmetry. The main parameters of the X-ray and pycnometric density gratings are determined (Table 2).

Table 1 – Indexing of radiographs of LaMe^{II}ZrMnO₆ powders (Me^{II} – Mg, Ca, Sr, Ba)

l/l_0	$d, \text{Å}$	$10^4/d^2_{exp}$	hkl	$10^4/d^2_{calc.}$
1	2	3	4	5
LaMgZrMnO ₆				
23	15.4449	647.5	422	647.5
8	13.9129	718.8	511	728.4
42	10.1124	988.9	610	998.2
30	8.1796	1223	630	1214
100	7.6729	1303	444	1295
8	6.9169	1446	633	1457
8	6.5025	1538	722	1538
23	5.0625	1975	661	1970
9	4.7961	2085	832	2077
8	4.4521	2246	753	2239
39	3.8025	2630	770	2644
8	3.4225	2922	10.2.2	2914
9	3.0625	3265	11.0.0	3265
8	2.8900	3460	880	3453
6	2.7889	3586	964	3588
32	2.5281	3956	11.5.1	3966
8	2.3716	4217	12.3.2	4236
8	2.1904	4585	11.7.0	4586
11	1.9044	5251	11.7.5	5261
10	1.5129	6610	12.10.1	6610
LaCaZrMnO ₆				
11	16.2409	615.7	511	615.7
13	15.4449	647.5	520	661.3
16	9.7344	1027	630	1026
12	8.8209	1134	710	1140
31	8.1225	1231	633	1231
100	7.6176	1313	730	1323
12	5.0176	1993	664	2007
11	4.0401	2475	10.3.0	2486
31	3.8025	2630	953	2623
10	3.3124	3019	10.4.4	3010
6	3.0276	3303	12.1.0	3307
11	2.6569	3764	10.8.1	3763
27	2.4964	4006	12.4.4	4014
4	2.4025	4162	10.9.1	4150
13	1.8769	5328	15.3.0	5336
8	1.5129	6610	17.1.0	6613
LaSrZrMnO ₆				
8	15.3664	650.8	511	650.8
20	9.8596	1014	541	1012
100	8.5264	1173	700	1181
69	7.6176	1313	633	1302

12	5.0625	1975	833	1977
29	4.2436	2356	770	2362
25	3.8025	2630	10.3.0	2627
8	3.6864	2713	10.3.2	2724
31	2.8224	3543	11.5.1	3543
12	2.1025	4756	12.7.2	4748
7	1.8769	5328	14.5.0	5327
11	1.6950	5917	11.10.5	5930
6	1.5129	6610	15.7.0	6604
LaBaZrMnO ₆				
9	15.4449	647.5	510	647.5
6	9.9225	1008	540	1021
100	8.8209	1134	631	1146
11	5.8564	1708	821	1718
14	5.1076	1958	725	1943
34	4.4100	2268	931	2266
22	3.8416	2603	10.2.1	2615
5	3.0625	3265	955	3262
37	2.9241	3420	883	3412
4	2.6896	3718	10.7.0	3711
21	2.5600	3906	11.6.0	3910
16	2.1904	4565	12.6.2	4582
7	1.9044	5251	997	5255
14	1.7689	5653	11.9.5	5653
8	1.5376	6504	12.9.6	6500
6	1.4641	6830	12.11.3	6824

The X-ray density (ρ_{x-ray}) of the investigated zircono-manganites was determined by the formula (Kumok, 1987).

$$\rho_{x-ray} = \frac{1,66 \cdot Mr \cdot Z}{V^0}, \quad (5)$$

where

Mr - is the molecular weight of the compound,

Z - is the number of formula units in the lattice,

V⁰ - is the volume of the unit cell.

The experimental values of the densities of the studied compounds were determined by pycnometry in toluene (V=1 ml) (Kiwilis, 1959). In this case, the following operations were performed sequentially: determination of the mass of an empty pycnometer (M_0); then a pycnometer filled with distilled water (M_1); a pycnometer filled with toluene (tetrabromoethane) (M_2); then the test substance was placed in the pycnometer and its mass was determined with a dry substance (M_3); finally, the powder was poured with a pycnometric liquid and determined weight (M_4). The density of the test sample is determined by the formula:

$$\rho_{p\text{icn}} = \frac{M_3 - M_0}{\frac{M_1 - M_0}{\rho_1} + \frac{M_2 - M_0}{\rho_2}} \quad (6)$$

where

ρ_1 - is the density of water at 20⁰ (0.9971 g/cm³);

ρ_2 - is the density of the pycnometric liquid, determined by the formula:

$$\rho_2 = \frac{M_2 - M_0}{M_1 - M_0} \cdot \rho_1 \quad (7)$$

The density of each compound was measured 3 times and the data were averaged.

Table 2 shows the parameters of the elementary cells, X-ray and pycnometric densities of the obtained new zircono-manganites of lanthanum and alkaline earth metals (alkaline earth metals).

Table 2 – Lattice parameters of lanthanum zircono-manganites and alkaline earth metals

Zircon-manganite	a, Å	V ^o , Å ³	Z	V ^o _{elem cell} , Å ³	(ρ), g/cm ³	
					ρ _{x-ray}	ρ _{picn}
LaMgZrMnO ₆	13,46 ± 0,02	2437,86 ± 0,05	4	609,47 ± 0,02	4,42	4,35 ± 0,07
LaCaZrMnO ₆	14,50 ± 0,02	3048,63 ± 0,06	4	762,16 ± 0,02	3,67	3,62 ± 0,05
LaSrZrMnO ₆	14,56 ± 0,02	3087,52 ± 0,06	4	771,88 ± 0,02	4,03	3,99 ± 0,04
LaBaZrMnO ₆	14,79 ± 0,02	3233,45 ± 0,06	4	808,36 ± 0,02	4,25	4,19 ± 0,05

Next, we will calculate the heat capacity using the Landius method for zircono-manganites of lanthanum and alkaline earth metals using the example of LaMgZrMnO₆.

$$S_{298}^{\circ} = 185,4 \text{ J/mol K}, S_{298}^{\circ} = \frac{185,4}{4,184} = 44,3117, S^{\text{at}} = \frac{S_{298}^{\circ}}{n} = \frac{44,3117}{10} = 4,4312, T' = \frac{5070}{4,4312} = 1144,17, K = 0,34,$$

$$a = 507 + \frac{1070}{4,4312} = 748,47, B = 0,8 \cdot a = 598,7757, K = 0,34.$$

$$C^{\text{p}} = 6,2 \cdot 4 + 6 \cdot 4 = 48,8 \text{ cal/mol deg},$$

$$C^{\text{p}'} = 10 \left[6,6 - \frac{748,47}{298} + \frac{6,38}{1144,17} \left(6,6 - \frac{748,47}{298} \right)^2 \right] = 41,816 \text{ cal/mol deg},$$

$$C^{\text{p}''} = 10 \left[6,6 - \frac{14,76}{4,4312} \cdot \frac{3^2 + 7^2}{10^2} + \frac{6,38}{1144,17} \left(6,6 - \frac{14,76}{4,4312} \cdot \frac{3^2 + 7^2}{10^2} \right)^2 \right] = 47,896 \text{ cal/mol deg}.$$

Next, go to section 3 of the scheme (Veritin, et al, 1965) and select the item "non-polymorphic transformations", the sub-item "complex oxygen compounds" and the column "consisting of solid oxides" with its subsection "other cases". Then, in section 4 of the scheme (Veritin, et al, 1965), we stop at point V with the wording "calculate C_p at 298 K according to formula (3)". The subparagraph indicates the calculation of C_p at 673 K and T_{dens} according to the formula (4).

$$C_p = \frac{48,8 + 41,816 + 47,896}{3} = 46,1707 \text{ cal/mol deg},$$

$$C_p^{\circ} = 10 \left[6,6 - \frac{748,47}{598,7757 + 0,34(673 - 598,7757)} + \frac{1,24}{673} \left(6,6 - \frac{748,47}{298} \right)^2 \cdot 673^{3/2} \cdot 10^{-3} \right] = 59,383 \text{ cal/mol deg,}$$

$$C_p^{\circ} = 10 \left[6,6 - \frac{748,47}{598,7757 + 0,34(1144,17 - 598,7757)} + \frac{1,24}{1144,17} \cdot 6,6 - \frac{748,47}{298} \right]^2 \cdot 1144,17^{3/2} \cdot 10^{-3} = 63,467 \text{ cal/mol deg}$$

$$1144,17^{3/2} = 38702,22.$$

Based on the values of C_p at 298.15, 673 and 1144 K, we calculate the coefficients of the temperature dependence of the heat capacity, the conclusion of which we gave in the previous sections, and find the equation in the form of the following polynomial:

$$C_p^{\circ} = 58,93 + 4,805 \cdot 10^{-3} T - 12,604 \cdot 10^5 T^{-2} \text{ cal/mol deg} \quad (8)$$

$$C_p^{\circ} = 246,56 + 20,083 \cdot 10^{-3} T - 52,72 \cdot 10^5 T^{-2} \text{ J/mol K (298.15-1144K)}. \quad (9)$$

The heat capacities for lanthanum and calcium, strontium and barium zirconomanganites are calculated in a similar way, summarizing the temperature dependence equations are given below in Table 3.

Table 3 – Temperature dependence $\text{LaMe}^{\text{II}}\text{ZrMnO}_6$ heat capacity according to Landium

Content	Equations of temperature dependence, J/(mol K)			$\Delta T, K$	$C_p^{\circ}(298,15)$ by Land, J/(mol.K)	$C_p^{\circ}(298,15)$ according to Kumok, J/(mol.K)
	a	$b \cdot 10^{-3}$	$-c \cdot 10^5$			
LaMgZrMnO_6	246,6	20,08	52,72	298,15-1144	193,2	199,6
LaCaZrMnO_6	246,6	5,0	50,0	298,15-1056	196,5	204,7
LaSrZrMnO_6	246,6	21,34	48,28	298,15-1001	198,7	206,7
LaBaZrMnO_6	247,9	20,04	47,43	298,15-953	200,54	205,8

To compare the standard heat capacity of zircono-manganites calculated using the Landius method, they were also calculated with the independent method of ion increments using (Kiwilis, 1959) according to the scheme:

$$C_p^{\circ}(298,15)\text{LaMe}^{\text{II}}\text{ZrMnO}_6 = C_p^{\circ}(298,15)\text{La}^{3+} + C_p^{\circ}(298,15)\text{Me}^{2+} + C_p^{\circ}(298,15)\text{Zr}^{4+} + C_p^{\circ}(298,15)\text{Mn}^{3+} + 6C_p^{\circ}(298,15)\text{O}^{2-}, \quad (10)$$

where $C_p^{\circ}(298,15)$ are the increments of the heat capacity of ions which (J/(molK) $\text{La}^{3+}(29,3)$, $\text{Mg}^{2+}(22,2)$, $\text{Ca}^{2+}(27,3)$, $\text{Sr}^{2+}(29,3)$, $\text{Ba}^{2+}(28,4)$, $\text{Zr}^{4+}(22,9)$, $\text{Mn}^{3+}(25,0)$, $\text{O}^{2-}(16,7)$. As can be seen from the data in Table 3, the $C_p^{\circ}(298,15)$ calculated using the Landius and Kumoku methods are in satisfactory agreement with each other within the error limits of the calculation methods.

Discussion

The reliability, correctness and reliability of the results of indexing and determination of lattice parameters are confirmed by a satisfactory agreement of experimental and calculated values of $10^4/d^2$, X-ray and pycnometric densities. Based on the conducted studies, it was found that the obtained zirconate-manganites crystallize in cubic symmetry and they can be attributed to the spatial group of perovskite $\text{Pm}\bar{3}\text{m}$. It was

revealed that with an increase in ionic radii in the range from Mg to Ba, the values of parameter "a" and the volumes of lattices and elementary cells of synthesized zirconate-manganites increase.

The temperature dependences of the heat capacity of the investigated zircono-manganites were calculated using Landium method. The standard heat capacities of their values are also calculated along with the Landius method by the independent Kumoku ion increment method, the results of which are in satisfactory agreement with each other, which shows the reliability and correctness of the data obtained for the values of standard heat capacities.

Conclusion

1. For the first time using ceramic technology, zirconium-manganites of $\text{LaMe}^{\text{II}}\text{ZrMnO}_6$ ($\text{Me}^{\text{II}} - \text{Mg, Ca, Sr, Ba}$) compositions were obtained from lanthanum (III), zirconium (IV) manganese (III) oxides and magnesium, calcium, strontium and barium carbonates.

2. The types of syngony and the parameters of their gratings were determined by X-ray method. It has been established that all zircono-manganites crystallize in cubic symmetry: $\text{LaMgZrMnO}_6 - a = 13,46 \pm 0,02 \text{ \AA}$; $V^0 = 2437,86 \pm 0,05 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 609,47 \pm 0,015 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 4,42 \text{ g/cm}^3$; $\rho_{\text{picn}} = 4,35 \pm 0,07 \text{ g/cm}^3$; $\text{LaCaZrMnO}_6 - a = 14,50 \pm 0,02 \text{ \AA}$; $V^0 = 3048,63 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 762,16 \pm 0,02 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 3,67 \text{ g/cm}^3$; $\rho_{\text{picn}} = 3,62 \pm 0,05 \text{ g/cm}^3$; $\text{LaSrZrMnO}_6 - a = 14,56 \pm 0,02 \text{ \AA}$; $V^0 = 3087,52 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 771,88 \pm 0,015 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 4,03 \text{ g/cm}^3$; $\rho_{\text{picn}} = 3,99 \pm 0,04 \text{ g/cm}^3$; $\text{LaBaZrMnO}_6 - a = 14,79 \pm 0,02 \text{ \AA}$; $V^0 = 3233,45 \pm 0,06 \text{ \AA}^3$; $Z = 4$; $V^{\circ}_{\text{elem cell}} = 808,36 \pm 0,015 \text{ \AA}^3$; $\rho_{\text{x-ray}} = 4,25 \text{ g/cm}^3$; $\rho_{\text{picn}} = 4,19 \pm 0,05 \text{ g/cm}^3$.

It was revealed that the lattice parameters of zirconate-manganites change symbatically with an increase in ionic radii in the $\text{Mg} \rightarrow \text{Ca} \rightarrow \text{Sr} \rightarrow \text{Ba}$ series.

3. Using the Landius and Kumok methods, the temperature dependences of the heat capacity and the standard heat capacities of the investigated zircono-manganites were calculated. The results of the research can later be used for thermodynamic justification of reactions involving the above and similar compounds.

4. The results obtained are of interest for the directed synthesis of similar compounds in inorganic materials science and chemical informatics as new in terms of radiographic and thermodynamic characteristics of previously unexplored compounds.

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СИНТЕЗ И РЕНТГЕНОГРАФИЯ НОВЫХ ЦИРКОНО-МАНГАНИТОВ ЛАНТАНА И ЩЕЛОЧНОЗЕМЕЛЬНЫХ МЕТАЛЛОВ И РАСЧЕТ ИХ ТЕРМОДИНАМИЧЕСКИХ СВОЙСТВ.....186

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