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SYNTHESIS AND BIOLOGICAL ACTIVITY OF COMPLEX COMPOUNDS OF MANGANESE (II) WITH AMINO ACIDS

Abstract. Complex compounds based on manganese (II) chloride crystal hydrate $MnCl_2 \cdot 4H_2O$ with amino acids of different classes, such as aliphatic – glycine and heterocyclic – histidine and tryptophan have been synthesized and identified with IR spectroscopy, differenti-al thermal analysis, electron microscopy and elemental analysis. Using elemental analysis and electron microscopy we determined chemical composition of the complex as $[NH_2CH_2COO]_2Mn \cdot 2H_2O$. The catalytic oxidative activity of $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ comples has been tested in the model reaction of aerobic oxidation of isopropyl-benzene (cumene). $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ complex demostrated high catalytic activity and accelerated the reaction by 10 times compared to the control sample. The tests for biological activity demonstrate antibacterial, antiviral and psychotropic properties of synthesized complexes.

Keywords: glycine, tryptophan, histidine, manganese (II) chloride crystal hydrate, complex compounds, aerobic oxidation, isopropylbenzene (cumene), biological activity

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СИНТЕЗ И БИОЛОГИЧЕСКАЯ АКТИВНОСТЬ КОМПЛЕКСНЫХ СОЕДИНЕНИЙ МАРГАНЦА (II) С АМИНОКИСЛОТАМИ

Аннотация. Синтезированы комплексные соединения на основе кристаллогидрата хлорида марганца (II) $MnCl_2 \cdot 4H_2O$ с аминокислотами различного класса: алифатическими – глицином и гетероциклическими − гистидином и триптофаном с соответствующими выходами. Идентификацию полученных соединений проводили методами ИК-спектроскопии, дифференциально-термического анализа, электронной микроскопии и элементного анализа. На основе химического анализа и электронной микроскопии установлен количественный состав комплекса $[NH_2CH_2COO]_2Mn \cdot 2H_2O$. Исследована каталитическая окислительная активность $[NH_2CH_2COO]_2Mn \cdot 2H_2O$, использованного в качестве активной добавки в модельной реакции аэробного окисления изопропилбензола (кумола). Установлено, что соединение обладает высокой каталитической активностью и ускоряет реакцию в 10 раз по сравнению с контрольным образцом. Синтезированные комплексы были апробированы на биологическую активность. Выявлено, что они обладают антибактериальными, антивирусными и психотропными свойствами.

Ключевые слова: глицин, триптофан, гистидин, кристаллогидрат хлорида марганца (II), комплексные соединения, аэробное окисление, изопропилбензол (кумол), биологическая активность

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Introduction. In recent decades, the demand for significant amounts of α-amino acids has been steadily increasing due to their wide use in biochemistry, microbiology and in the study of plant and animal tissues [1–4]. In addition, proteinogenic amino acids have found wide application as additives to natural and processed food and nutrition products [1, 5–7].

Obtaining biologically active metal-containing compounds by the interaction of transition metals with natural organic compounds in the liquid phase is one of the promising directions in biochemistry [8]. Complexes of metals and amino acids are components of metal-containing enzymes, vitamins, hormones, pigments, and other substances whose biosynthesis occurs with the participation of 3d-metals. Atoms of these metals being in complexes with amino acids, proteins and other natural compounds, for example, iron - in the composition of hemoglobin, cytochromes, transferrins, copper – in the composition of hemocyanin, tyrosinase, lysyl oxidase, manganese – in the composition of carboxylase, arginase, provide the normal course of biochemical processes and stimulate metabolism [9]. Therefore, the study of the interaction of 3d-metals with amino acids as the simplest models for the formation of their complexes with proteins will contribute to a more complete understanding of the mechanisms of biochemical reactions [10–13]. The possibility of such reactions is indicated by the processes of leaching (dissolution) of some transition metals in the presence of amino acids contained in the metabolic products of microorganisms such as bacteria and fungi [14–18].

Thus, it should be noted that the study and investigation of the behavior of metal complex compounds with natural proteinogenic amino acids in biochemical processes occurring in living organisms and plant cells represent a very important, promising and relevant scientific direction of modern biochemical and biomolecular science.

One of the interesting applications of amino acid salts is their potent ability to catalyze the oxidation processes of organic substances. In this sense, manganese salts of amino acids for the catalysis of hydrocarbon reactions are of particular interest. The previous practice of using manganese salts of organic acids has shown that they are quite effective for the processes of interphase aerobic oxidation of hydrocarbons [19].

The main purpose of the work is to study the role of metals and their compounds in living organisms and in the environment, to study the reactivity of metal ions and their compounds in relation to biological substrates, to synthesize biologically active compounds and to create biomaterials.

Experimental part. The present work presents the synthesis of complex compounds of manganese (II) with a number of aliphatic and heterocyclic amino acids according to the corresponding methods [20–22]. The reactions of manganese (II) chloride $MnCl_2 \cdot 4H_2O$ with glycine, histidine and tryptophan have been studied.

Synthesis of manganese complex compounds. Manganese glycinate dihydrate $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ [**I**] was synthesized by interaction of aminoacetic acid (glycine) with manganese tetrahydrate $MnCl_2 \cdot 4H_2O$.

$MnCl_2 \cdot 4H_2O$ solution in the amount of 9.9 g (0.05 mol) has been dissolved in 50 mL of water (pH 3) and heated at 45–50 °C for 30–40 minutes. To the homogeneous solution 7.5 g (0.1 mol) of glycine were added in small portions and kept under continuous stirring for 40-45 minutes at a temperature of 45–50 °C (pH of the reaction solution was 6–6.5). The ratio of initial components in the system was 2 : 1. Then the reaction mixture was cooled down to 10 °C, the crystalline product was filtered, washed thoroughly with ethanol and dried at room temperature. The yield of the target product manganese (II) glycinate dihydrate, $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ was 76 % (9.1 g). Manganese glycinate (II) is a powdery substance of white color with a slight pinkish tint, readily soluble in water, but insoluble in ethanol and acetone.

The complex Mn^{2+} with histidine was obtained in a water-methanol medium at the ratio of the initial components equal to 1 : 2 for 3 hours at a temperature of 35–40 °C with a yield of the target product of 68 %. Crystals of complex compound of manganese (II) obtained in this process are dark purple in color. Synthesized complex $[C_6H_8N_3O_2]_2Mn \cdot 2H_2O$ [**II**] is insoluble in alcohols, benzene, toluene and acetone, but readily soluble in water at room temperature.

Accordingly, the complex of manganese (II) with tryptophan was obtained at the ratio of initial components equal to 1:2 for 3 hours with continuous stirring and at a temperature of 37–40 °C. At the end of the reaction the obtained solution was filtered, washed several times with water followed by recrystallization, evaporated at 40 °C and dried with anhydrous $CaCl_2$ for 24 hours. Precipitated crystals of the complex $[C_{11}H_{11}N_2O_2]_2Mn \cdot 2H_2O$ [**III**] are dark purple in color, insoluble in alcohols, aromatic hydrocarbons, and organic solvents, but readily soluble in water at room temperature. The yield of the reaction product was 71.0 %. The synthesized complexes are sufficiently stable to light and air.

Characterization of the synthesized manganese (II) compounds. Methods of IR spectroscopy, differential thermal analysis, as well as electron microscopy and elemental analysis were used to determine the composition, structure and stability of the obtained products.

Infrared spectra of the samples were recorded using a Thermo Scientific Nicolet iS10 spectrometer (USA). During the formation of the manganese (II) glycinate complex, the absorption bands of NH stretching vibrations of the NH_3^+-group are observed in two cases: νNH = 3 018.98 cm^{-1} and νNH = 3 069 cm^{-1}. According to the IR spectrum of the synthesized complex, the characteristic absorption bands for the carboxyl group fragments are observed in four cases: 1 629 cm^{-1}, 1 570.75 cm^{-1}, 1 408.97 cm^{-1} and 1 343.66 cm^{-1}. The above confirms the formation of manganese (II) glycinate dihydrate based on the reaction of aminoacetic acid with $MnCl_2 \cdot 4H_2O$.

Figure 1 shows the IR spectrum of the synthesized complex compound $[NH_2CH_2COO]_2Mn \cdot 2H_2O$.

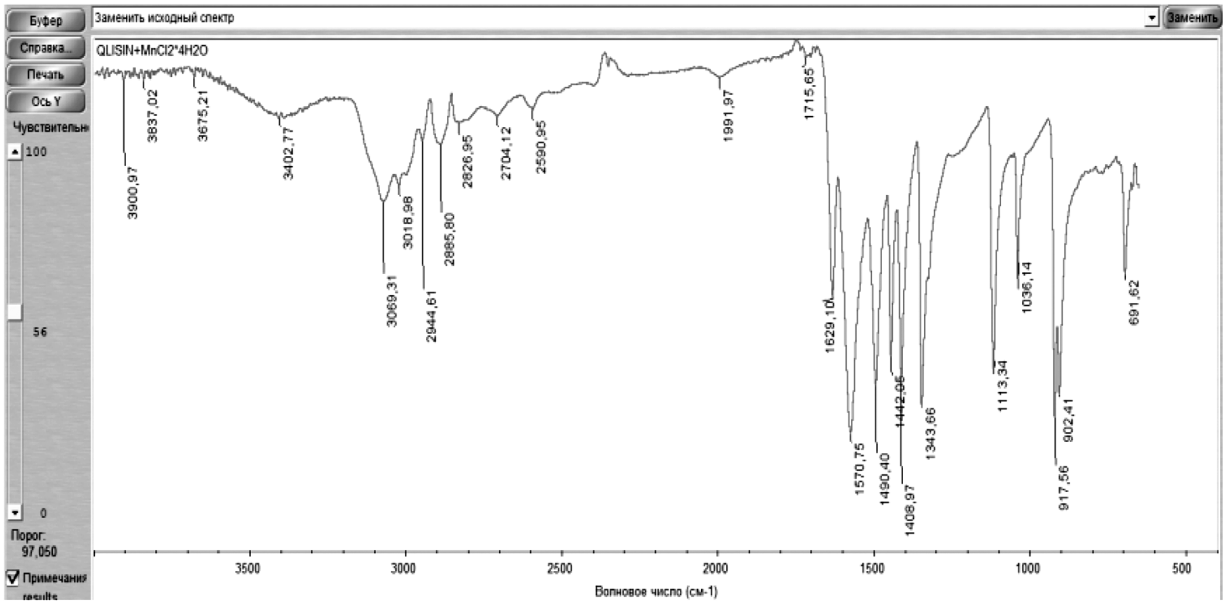
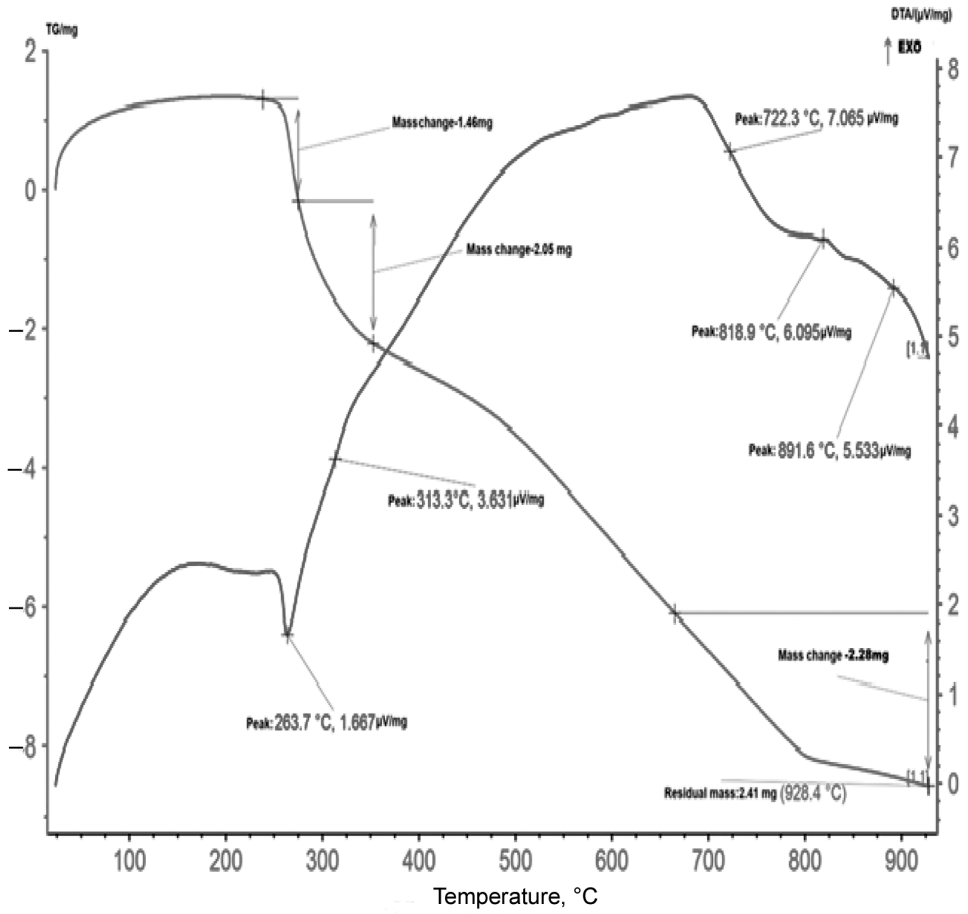


Fig. 1. IR spectrum of the synthesized complex $[NH_2CH_2COO]_2Mn \cdot 2H_2O$

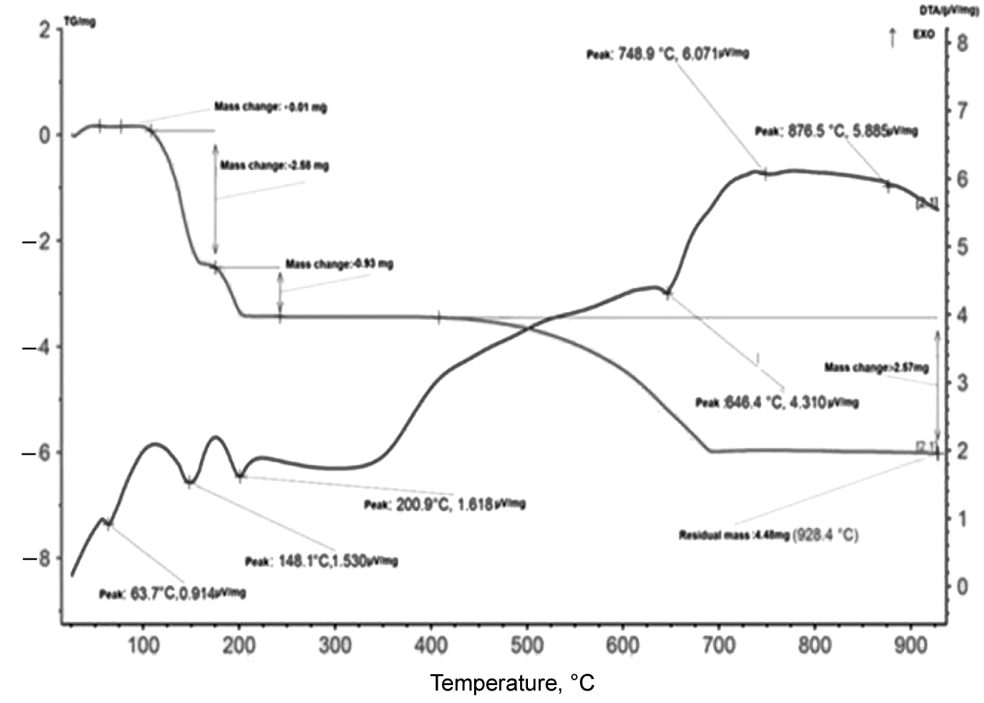
Differential thermal analysis (DTA) was carried out on a NETZSCH Proteus 449 F3 thermal analyzer in heating mode at 10 °C per minute from 25 to 900 °C in nitrogen atmosphere.

Figure 2 shows the TG/DTA curves of the initial tetrahydrate of manganese (II) chloride $MnCl_2 \cdot 4H_2O$ (Fig. 2, *a*) and the synthesized complex compound of manganese (II) glycinate dihydrate, $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ (Fig. 2, *b*).

As can be seen from Fig. 2, *a*, the compound $MnCl_2 \cdot 4H_2O$ is stable only up to 263.7 °C. At this temperature, an endothermic peak is observed on the DTA curve. On the TG curve, this thermal event marks the beginning of the compound's decomposition; in other words, the onset of degradation is endothermic. Then, there is a gradual decrease in the mass of the sample up to the almost complete decomposition of the compound at temperatures of 800–900 °C. According to Fig. 2, *b*, the synthesized compound has a completely different profile from $MnCl_2 \cdot 4H_2O$ in terms of its DTA and TG curves. It should be noted that the main processes of partial decomposition of the synthesized complex $[NH_2CH_2COO]_2Mn \cdot 2H_2O$, such as the rearrangement or detachment of molecular fragments, are observed in the temperature range of 100–200 °C. In this temperature range, two endothermic and two exothermic events are observed on the DTA curves. Obviously, the processes of heat absorption and release are not only alternate but also overlap. According to the thermal analysis, stepwise dehydration occurs at temperatures of 148.1 °C and 200.9 °C, respectively; the amount of evaporated water was calculated from the corresponding peak areas. The TG curve shows a sharp, stepwise mass loss up to a temperature of 200 °C, after which the structure of the remaining part of the molecule remains stable up to 500 °C. With a further increase in temperature, there is a gradual drop in the mass of the sample up to a temperature of 646.4 °C. After this mass loss, there are no further processes of heat absorption



a



b

Fig. 2. DTA results. TG/DTA curves of: *a* – the initial crystal hydrate of manganese chloride $MnCl_2 \cdot 4H_2O$; *b* – synthesized complex $[NH_2CH_2COO]_2Mn \cdot 2H_2O$

and release in the system. It should be noted, that at a temperature of 646.4 °C an endothermic peak indicates the beginning of the separation of manganese from chlorine, followed by the decomposition of the synthesized product at 876.5 °C.

The composition and scanning electron micrographs of the synthesized sample were studied using a Hitachi S-3400N scanning electron microscope (Japan) equipped with an Oxford Instruments energy-dispersive X-ray spectrometer. The melting temperature was determined using a Boëtius optical microheating stage microscope (Germany). As a result of the studies, it was found that the melting onset temperature of the synthesized complex $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ is 230 °C. Figure 3 shows the scanning electron micrographs of the synthesized sample. It should be noted that crystal hydrates under the influence of vacuum change their composition, losing some amount of water.

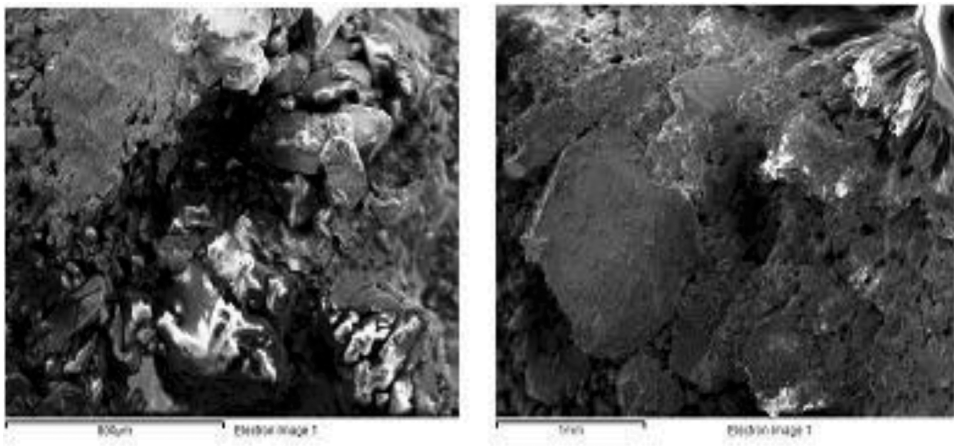


Fig. 3. Scanning electron micrographs of the synthesized sample $[NH_2CH_2COO]_2Mn \cdot 2H_2O$

On the basis of the chemical analysis and electron microscopy, the proposed structure of the synthesized compound consists of the following number of atoms: Mn-1; O-6; C-4; N-2 ; H-12 (Fig. 4).

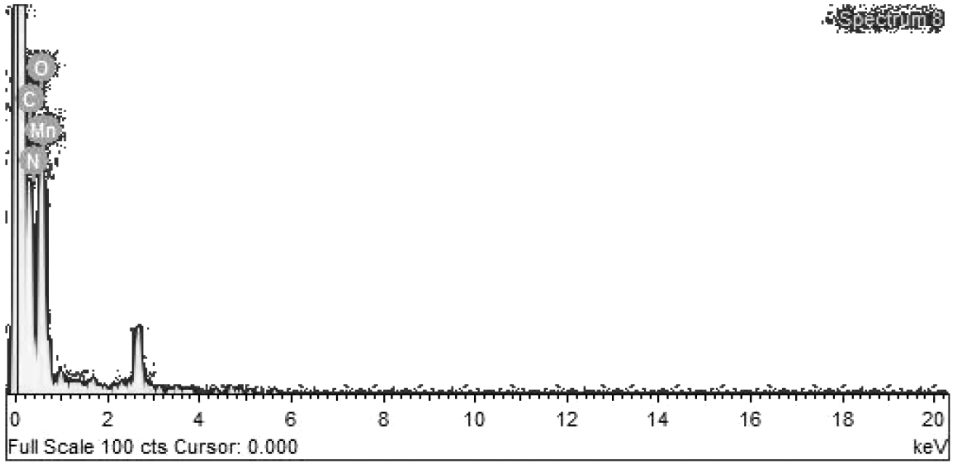


Fig. 4. Atomic spectrum of synthesized manganeseglycinate $[NH_2CH_2COO]_2Mn \cdot 2H_2O$

Oxidation of isopropylbenzene. Figure 5 shows the kinetic curves of oxygen uptake during the aerobic oxidation of isopropylbenzene (cumene) at 100 °C in the presence of $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ additive.

The results show that the complex displays high catalytic activity in the system. From the oxygen uptake kinetics, it is seen that the manganese complex has significant catalytic activity and accelerates the reaction by a factor of 10 (Fig. 5, *2*) compared to the control sample of isopropylbenzene containing no additives (Fig. 5, *1*).

Evaluation of synthesized complexes of Mn^{2+} with glycine, histidine and tryptophan for biological activity. Synthesized complex compounds of manganese (II) with the above-mentioned amino

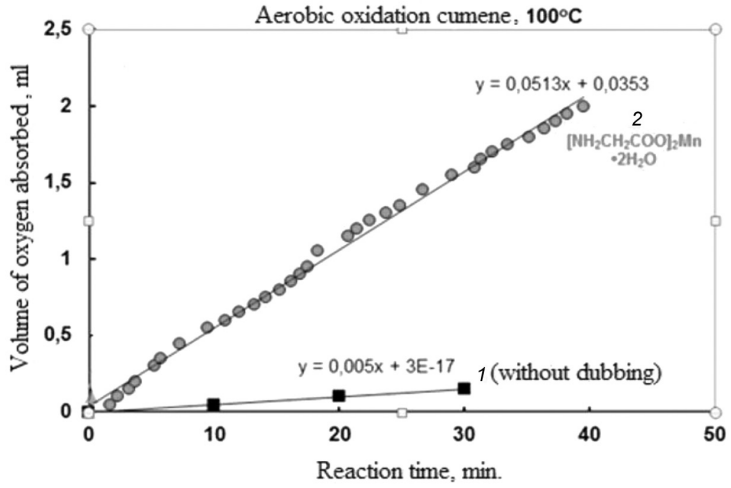


Fig. 5. Profiles of kinetic curves of oxygen uptake during the aerobic oxidation of cumene in the absence (*1*) and presence of glycinate manganese (II) complex $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ (*2*). The volume of the reaction mixture is 10 ml, and the temperature is 100 °C. Amount of additives: *1* – 0; *2* – $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ = 2 g/l

acids were evaluated for biological activity. Primary tests showed that the obtained complex compounds have antibacterial, antimicrobial and antiviral properties, as well as sedative and antidepressant effects (psychotropic activity).

Results of initial tests of complex compounds of manganese (II) with glycine [I], histidine [II] and tryptophan [III]

№	Biological activity		
	Antiviral	Antibacterial	Psychotropic
[**I**]	Not detected	Weak activity against *Staphylococcus aureus* (100 mg/ml)	Not detected
[**II**]	Weak activity against rotavirus	Weak activity against *Staphylococcus aureus* (100 mg/ml)	Sedative sedative effect at a dose of (15–20 mg/kg)
[**III**]	Activity against rotavirus	Not detected	Antidepressant, sedative effect (20–25 mg/kg)

Thus, the synthesized complex compounds can be further used as intermediates for the subsequent production of drugs necessary for human life activity.

Conclusions. Complex compounds based on $MnCI_2 \cdot 4H_2O$ with glycine, histidine and tryptophan have been synthesized. The composition and structure of the obtained products were determined on the basis of IR spectroscopy, differential thermal analysis, electron microscopy, and elemental analysis.

When used as an active additive in the model reaction of aerobic oxidation of isopropyl benzene (cumene), the complex $[NH_2CH_2COO]_2Mn \cdot 2H_2O$ exhibited high catalytic activity and accelerated the reaction by a factor of 10.

The synthesized complex compounds were tested for biological activity and found to possess antibacterial, antiviral and psychotropic properties.

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