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COMPLEX COMPOUNDS OF PALLADIUM (II) WITH  $\gamma$  – GLUTAMIC ACID AMIDE<sup>1,2</sup>Kh.I. Gasanov, <sup>1</sup>A.N. Azizova, <sup>2</sup>N.M. Kuliyeva, <sup>1</sup>Sh.G. Gasimov<sup>1</sup>Azerbaijan Medical University, Scientific Research Center<sup>2</sup>Western Caspian University

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**Abstract:** This study examined the formation of palladium (II) complex compounds with  $\gamma$ -glutamic acid amide in aqueous solutions and calculated the complex stability constants, also known as formation constants. After the complexes were separated from one another, each compound's structure and characteristics were studied individually. Based on data from NMR, IR, and UV spectroscopy it was established, that two ligand molecules coordinate in a monodentate manner along the donor nitrogen atoms of the amino group and in a bidentate manner along the nitrogen atoms of the amino group and oxygen. A planar square internal coordination sphere is formed in the trans- structure, respectively, in the complexes  $[Pd_2N_{amine}2Cl]$  ( $[PdL_2Cl_2]$ ),  $[Pd_2N_{amine}2O_{carb}]$  ( $[Pd(HL)_2]$ ).

**Keywords:** palladium (II), glutamine, ligand, dentation, coordination

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## Introduction

A systematic study of the composition-structure relationship and biological activity of palladium compounds revealed some abilities of its complexes to stabilize the cell membrane, which served as one of the prerequisites for searching among palladium complexes with radioprotective properties [1]. At the same time, the literature also contains information about the high antitumor activity of palladium complexes with organic ligands [1]. Low toxicity is one of the requirements for biologically active compounds. Palladium compounds are characterized by significantly lower toxicity compared to platinum complexes.

Despite the huge number of known and thoroughly characterized coordination compounds of noble metals, the interest of researchers in their synthesis and study does not

decrease. Recently, complexes of which ligands are bioactive natural substances containing amino acids and, in particular, glutamine, have become attractive. Information regarding glutamine's anticancer activity can be found in Ref. [2]. It is of scientific and practical interest that a coordination compound containing the metal complexing agent palladium (II) ion and the polydentate ligand glutamine has been formed. This combination has not been reported in any literature and shows promise as an anticancer medication.

The effort involved studying the interaction between palladium (II) and the  $\gamma$ -amide of L-glutamic acid, as well as establishing the structures of the produced coordination compounds.

## Experimental part

Palladium dichloride ( $\text{PdCl}_2$ ), potassium tetrachloropalladate ( $\text{K}_2[\text{PdCl}_4]$ ) of chemically pure grade and  $\gamma$  - L-glutamic acid amide (L-glutamine, Qln) of reagent grade were used as starting compounds.

IR spectra of the complexes and the ligand were recorded on Thermoscientific, Nikoletis 10 and Bruker IFS-113V spectrometers in vaseline or the suspension of fluorinated oils, as well as in the form of tablets with KBr; and electronic absorption spectra were taken on a Shimadzu UV-Vis-240 Evolution-60S device. NMR spectra were recorded on a Bruker NMR 400 MHz NMR spectrometer. Elemental analysis of non-metals was performed on a CHNS-O EMA 502 analyzer. Palladium analysis was carried

out using our own method.

The thermal behavior of the complexes was studied on a STA 449 F3 Jupiter NETZSCH derivatograph at a heating rate of 10 degree/min up to 800° C.

Complex compounds were synthesized at room temperature in an aqueous solution at a ratio of  $\text{Pd}^{2+} : \text{L}^- = 1 : 2$ . After two days, plate-like light yellow crystals precipitated from the solution, which were then washed with water, ethanol, ether and dried at 40–50° C. Since the routes for carrying out syntheses are not so different, it is more appropriate to describe them briefly. The results of elemental analysis are listed in Table 1.

## Results and discussion

In amino acid amides, the molecule composition contains three potential donor atoms - the nitrogen of the amino and amido groups and the oxygen of the carboxyl group. The donor properties of nitrogen (electronic state  $\text{sp}^3$ ) of the amino group are appeared in a greater degree than of the amido groups (electronic state  $\text{sp}^2$ ), since the free electron pair

of the nitrogen atom in the first case is more mobile than in the second, where the same pair of electrons is located in the p-orbital. This causes it to be moved in the direction of the carbon atom, making the nitrogen atom of the amide group less able to make a bond through the donor-acceptor mechanism [2].

**Table 1.** The result of the elemental analysis for the synthesized complex compounds

| Coordination compounds      | Pd    |       | N     |       | Cl    |       | C     |       | H     |       |
|-----------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|                             | Found | Calc. | Found | Calc. | Found | Calc. | Found | Calc. | Found | Calc. |
| $[\text{PdL}_2\text{Cl}_2]$ | 22.47 | 22.66 | 11.74 | 11.93 | 15.32 | 15.10 | 25.41 | 25.56 | 4.47  | 4.26  |
| $[\text{Pd}(\text{HL})_2]$  | 26.71 | 26.56 | 14.16 | 13.98 |       |       | 30.15 | 29.98 | 5.66  | 5.49  |

To establish the purity and individuality of the resulting complexes, their X-ray photographs were taken. The uniqueness and purity of the ligand are confirmed by the stark differences in its X-ray diffraction patterns from those of the produced complexes (Fig. 1).

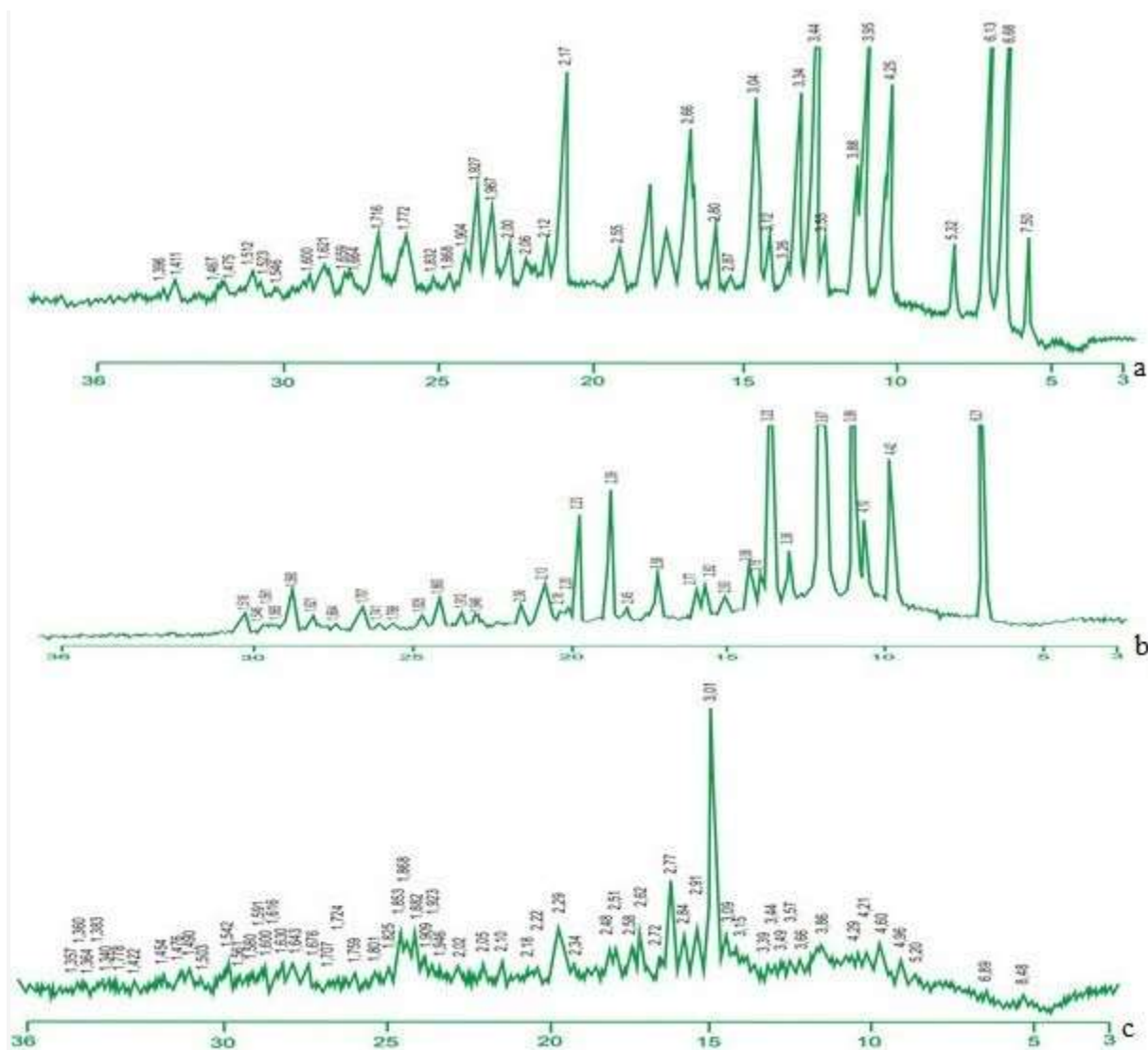
Depending on the synthesis conditions, coordination compounds with mono- (nitrogen atom of the amino group) or bidentately coordinated (nitrogen and oxygen atoms of the amino and carboxyl groups) glutamine molecules ( $\text{HQln}$ ) might be expected to occur in the palladium (II)  $\gamma$  -glutamine-water system.

Consequently, the internal coordination sphere can be supplemented with both water molecules and chlorine ions.

Individual amino acids and metal complexes with their participation are prone to the so-called glass transition [3], which we observed during the synthesis process and made it very difficult to isolate them in crystalline form for X-ray diffraction analysis. Therefore, the conclusion about the structure of the complexes we synthesized was made on the basis of NMR data, vibrations physical spectroscopy and electronic absorption spectra.

NMR spectra for the starting amino acid (pH 5.65) and the synthesized complexes -  $[\text{PdL}_2\text{Cl}_2]$ ,  $[\text{Pd}(\text{HL})_2]$  (pH 6.04 and 6.71, respectively) were recorded in deuterated water ( $\text{D}_2\text{O}$ ). In the spectrum of the amino acid, two multiplets are visible, corresponding to the CH and  $\text{CH}_2$  groups; the  $\text{NH}_2$  group is quickly deuterated, and therefore it is not detected by a separate signal. The data we obtained is as

follows: for the  $\alpha$ -carbon atom, the signal is presented as a doublet of a doublet with  $\delta$  equal to, ppm: 4.109; 4.082; 4.079 and 4.066, and for the  $\gamma$ -atom – as a multiplet with  $\delta$  equal to, ppm: 3.065; 3.056; 3.008; 2.091; 2.951; 2.937; 2.929 and 2.909. The presence of a multiplet in the signal corresponding to the  $\text{CH}_2$  group can be explained by the influence of the neighboring amide group.



glutamine concentration of  $1 \times 10^{-2} \text{ mol l}^{-1}$  in an excess of ligand  $\text{Pd}^{2+} : \text{L}^{-} = 1 : 4$  in weakly acidic solutions (pH 6.71 and 5.84, respectively) were recorded. Prepared solutions of complexes with a concentration of 10–3 M for determining electrical conductivity are not subject to hydrolysis.

A predominantly square planar structure of the coordination site is typical for palladium(II) complexes [4]. In the electronic absorption spectra of palladium(II) complexes with a square planar structure, three spin-allowed d–d transitions are possible, the energies of which are close, which determines the presence of one broadband in the absorption spectra, the location of the latter depends on the donor atoms of the ligand environment of the central atom [5–8]. In the case of the synthesized complexes, specifically such absorption broadband was recorded in the

region 31120–3140  $\text{cm}^{-1}$  confirming the square planar structure of the coordination site.

The conclusion about the composition of the ligand environment of the central atom was made by comparing the values of the experimentally determined frequency of the maximum absorption band with those theoretically calculated for various chromophores using the formula  $\nu_{\text{max}} = \sum n_i \cdot \nu_i$ , where  $n_i$  is the number of donor atoms of each type;  $\nu_i$  is the increment value for donor atoms of each type (Table 2). Taking into account the synthesis conditions for the theoretically most possible chromophores, the coordination site of palladium (II) of various compositions, including with mono- and bidentately coordinated glutamine, was calculated using known increment values  $\nu_{\text{max}}$ : Cl– 5165;  $\text{O}_{\text{H}_2\text{O}}$ – 6570;  $\text{O}_{\text{carbox}}$ – 6780;  $\text{N}_{\text{amin}}$ – 8450 [9].

**Table 2.** The composition of the chromophore and the value of the maximum absorption band of a number of palladium (II) complexes\*

| Composition of the complex particles   | Chromophore                                                                     | $\nu_{\text{max calc.}}, \text{cm}^{-1}$ |
|----------------------------------------|---------------------------------------------------------------------------------|------------------------------------------|
| $[\text{PdL}(\text{H}_2\text{O})_2]$   | $\text{PdN}_{\text{NH}_2}\text{O}_{\text{carbox}}\text{O}_{\text{H}_2\text{O}}$ | 28380                                    |
| $[\text{PdL}_2(\text{H}_2\text{O})_2]$ | $\text{Pd}_2\text{N}_{\text{NH}_2}2\text{O}_{\text{H}_2\text{O}}$               | 30070                                    |
| $[\text{Pd}(\text{HL})_2]$             | $\text{Pd}_2\text{N}_{\text{NH}_2}2\text{O}_{\text{carbox}}$                    | 30450                                    |
| $[\text{PdL}_4]$                       | $\text{Pd}_4\text{N}_{\text{NH}_2}$                                             | 33830                                    |
| $[\text{PdL}_2\text{Cl}_2]$            | $\text{Pd}_2\text{N}_{\text{NH}_2}2\text{Cl}$                                   | 27250                                    |
| $[\text{PdL}_2\text{ClH}_2\text{O}]$   | $\text{Pd}_2\text{N}_{\text{NH}_2}\text{ClO}_{\text{H}_2\text{O}}$              | 28660                                    |
| $[\text{PdLCl}_2]$                     | $\text{PdN}_{\text{NH}_2}2\text{O}_{\text{carbox}}2\text{Cl}$                   | 25570                                    |

\*The charges of complex particles are not given.

As can be seen from Table 2's data, the most satisfactory experimental value for the complex  $[\text{Pd}(\text{HL})_2]$ , of which  $\nu_{\text{max}}$  is equal to 31112  $\text{cm}^{-1}$ , corresponds to the calculated value of 30452  $\text{cm}^{-1}$ , and for the  $[\text{PdL}_2\text{Cl}_2]$  complex, of which  $\nu_{\text{max}}$  is equal to 27165  $\text{cm}^{-1}$ , corresponds to the calculated value 27250 for the chromophores  $\text{Pd}_2\text{N}_{\text{NH}_2}2\text{O}_{\text{carbox}}$  and  $\text{Pd}_2\text{N}_{\text{NH}_2}2\text{Cl}$ , respectively. These data may indicate the synthesis of a planar-square structure of the coordination compound of palladium (II), in which two glutamine molecules, in the first case, are bidentately coordinated with the nitrogen atom of the amino group and the oxygen atom of the carboxyl group, and in the second case – monodentate via the nitrogen atom of the amino group.

IR spectra were recorded in a wide range of wave numbers from 4000 to 200  $\text{cm}^{-1}$ . The shape of the obtained spectra satisfactorily coincides with the characteristic vibrational frequencies of the spectra of amino acids given in the literature [10–12]. There are no IR spectroscopic data for the palladium (II) complex with glutamine in the literature. In the IR spectra, maxima are visible that are responsible for the structure of the ligand. In the high-frequency region of the amino acid spectrum, two stretching vibrations (symmetric  $\nu_s$  and asymmetric  $\nu_{\text{as}}$ ) of free NH bonds of the amide group, which correspond to absorption bands of 3456 and 3410  $\text{cm}^{-1}$ , and of the amine group, 3112 and 2932  $\text{cm}^{-1}$ , are observed. Deformation ( $\delta$ ) vibrations of the NH bond of

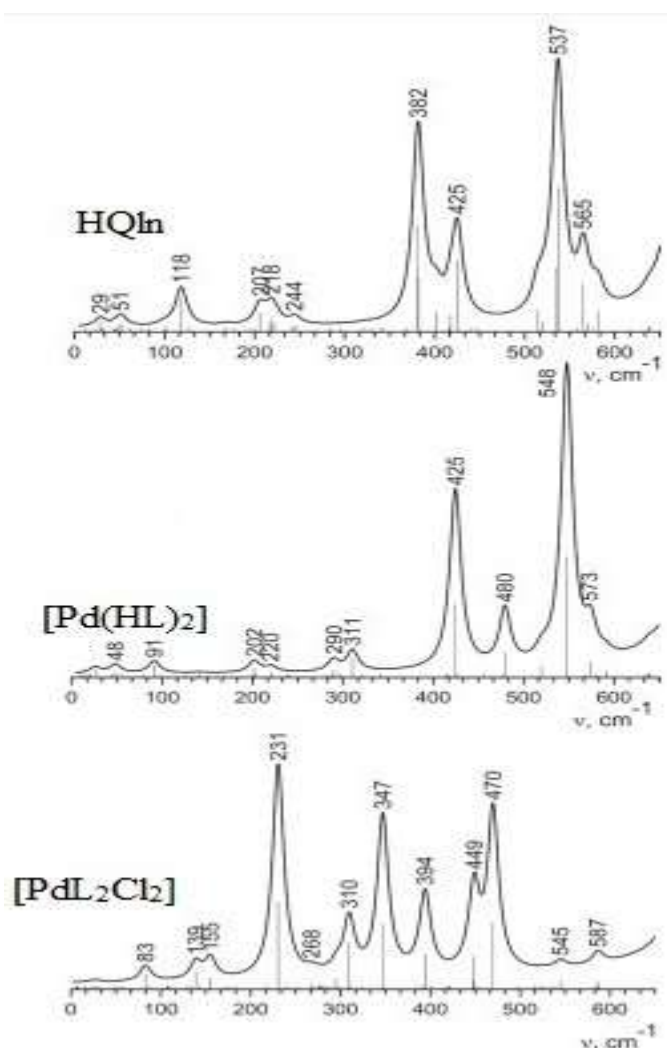
the amide group appear in the spectrum in the region of  $1638\text{ cm}^{-1}$  and  $1420\text{ cm}^{-1}$ . Stretching vibrations of the carbonyl group ( $\text{C}=\text{O}$ ) of the amide were detected in the region of  $1680\text{ cm}^{-1}$ . The absorption maximum of  $1584\text{ cm}^{-1}$  observed in the spectrum of glutamine corresponds to bending vibrations ( $\text{NH}^{+3}$ ) for amino acids, which confirms the zwitterionic form of glutamine [12–14].

The spectrum of palladium (II) complexes with glutamine  $[\text{Pd}(\text{HL})_2]$ ,  $[\text{PdL}_2\text{Cl}_2]$  shows absorption bands with maxima at 3456, 3430 and 3460,  $3420\text{ cm}^{-1}$ , respectively, which correspond to symmetric and asymmetric stretching vibrations ( $\text{NH}$ ) of the amide group. The frequencies  $3310.3072$  and  $3305.3050\text{ cm}^{-1}$  correspond to the stretching vibrations ( $\nu_s$  and  $\nu_{as}$ ) of the amine  $\text{NH}$  group. Bending vibrations

of the amide  $\text{NH}$  group are visible at  $1638.1640$  and  $1426.1430$ . In the IR spectrum of the  $[\text{Pd}(\text{HL})_2]$  complex, stretching vibrations of the carbonyl group ( $\text{C}=\text{O}$ ) are observed at  $1686\text{ cm}^{-1}$ , and the maximum at  $1650\text{ cm}^{-1}$  that appears in the spectrum of the palladium (II) complex with glutamine confirms the fact that the ligand is in zwitterionic form.

We did not identify the assignment of all absorption bands in the obtained spectra; the characteristic bands for the starting amino acid and the synthesized complex were only considered (Table 2).

It is known [11, 15] that  $\text{Pd-N}$  and  $\text{Pd-O}$  bonds correspond to band maxima in the low-frequency region of the spectrum, which are often difficult to recognize due to mixing with other skeletal vibrations.



**Fig.2.** IR spectra of HQln and complexes  $[\text{Pd}(\text{HL})_2]$  and  $[\text{PdL}_2\text{Cl}_2]$

However, after comparing the absorption bands of the amino acid and the palladium

complex in the spectra, maxima are visible at  $470$  and  $449\text{ cm}^{-1}$ , which are absent in the

spectrum of the starting amino acid, which we referred to the stretching vibrations of Pd-N and Pd-O bonds, respectively, which satisfactorily coincides with the data for the palladium (II) complex with amino acids [15]. One clear absorption band at  $347\text{ cm}^{-1}$  observed in the IR spectrum of the complex  $[\text{PdL}_2\text{Cl}_2]$  was

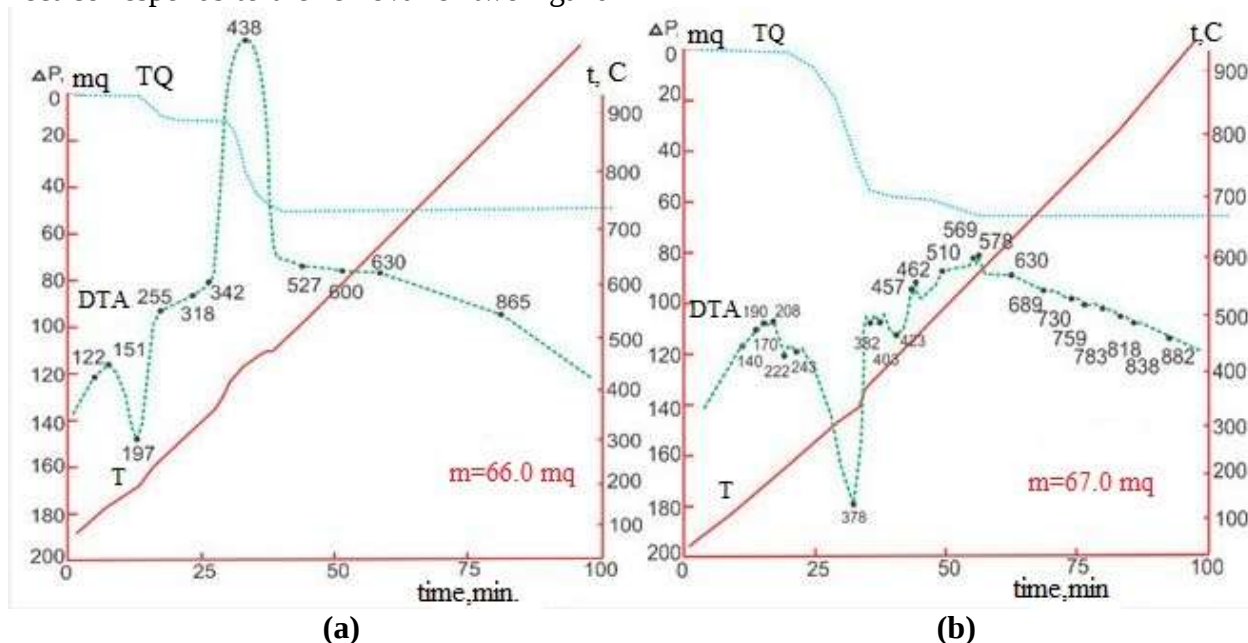
attributed to Pd-Cl valence bonds. The presence of one band for each Pd-N, Pd-O and Pd-Cl bonds in the spectrum indirectly indicates the *trans*-isomeric form of the synthesized complexes [15]. The qualitative thiocarbamide reaction of N.S. Kurnakov was used to prove this judgment.

**Table 3.** Some absorption bands of the IR spectra of glutamine and diglutamine complex of palladium (II)

| HL            | $[\text{PdL}_2\text{Cl}_2]$ | $[\text{Pd}(\text{HL})_2]$ | Absorption band assignment                                             |
|---------------|-----------------------------|----------------------------|------------------------------------------------------------------------|
| 3456 and 3410 | 3460 and 3420               | 3456 and 3430              | $\nu_s$ and $\nu_{as}\text{NH}$ in the amide group                     |
| 3112 and 2932 | 3305 and 3050               | 3310 and 3072              | $\nu_s$ and $\nu_{as}\text{NH}$ in the amine group                     |
| 1680          | 1686                        | 1688                       | $\nu(\text{C}=\text{O})$                                               |
|               | 346                         |                            | $\nu(\text{Pd}-\text{Cl})$                                             |
| 1638          | 1640                        | 1638                       | $\delta(\text{NH})$                                                    |
| 1584          | 1686                        | 1650                       | $\delta(\text{NH}^{+3})$ for aminoacids containing $\text{NH}_2$ group |
| 1420          | 1430                        | 1420                       | $\delta(\text{NH})$                                                    |
| -             | -                           | 476                        | $\nu(\text{Pd}-\text{N})$                                              |
| -             | -                           | 439                        | $\nu(\text{Pd}-\text{O})$                                              |

On the heating curve of the  $[\text{PdL}_2\text{Cl}_2]$  compound, endothermic effects are observed at temperatures of 122, 197 and  $342^\circ\text{C}$ , as well as many exothermic effects. The first endothermic effect corresponds to the removal of two ligand

molecules and a chlorine atom. The DTA curve of the compound  $[\text{Pd}(\text{HL})_2]$  shows only two endothermic effects at 190 and  $208^\circ\text{C}$ , as well as many exothermic effects.



**Fig. 3.** Derivatograms  $[\text{PdL}_2\text{Cl}_2]$  (a) and  $[\text{Pd}(\text{HL})_2]$  (b)

The appearance of the first endothermic effect is consistent with the removal of two ligand molecules. Data from thermal analysis of the studied compounds showed that the complexes decompose without melting and the final product of thermolysis is metallic

palladium.

Thus, physicochemical methods confirmed the formation of a coordination compound of palladium (II) with glutamic acid in the composition  $[\text{Pd}(\text{HL})_2]$  and  $[\text{PdL}_2\text{Cl}_2]$ .

### Conclusion

1. A coordination compound of palladium (II) with glutamine with composition  $[\text{Pd}(\text{HL})_2]$  is formed, in which two ligand molecules are coordinated bidentately with the donor nitrogen atoms of the amino group and the oxygen of the hydroxyl group.
2. In another obtaining complex  $[\text{PdL}_2\text{Cl}_2]$ , monodentate coordination of two ligand molecules is carried out at the nitrogen atom of the amino group.
3. In both complexes, the ligands are coordinated in a *trans*-position relative to each other.

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## PALLADIUMUN (II) $\gamma$ -AMİD QLUTAMİN TURŞUSU İLƏ KOMPLEKS BİRLƏŞMƏLƏRİ

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**Xülasə:** Palladiumun (II) sulu məhlullarda  $\gamma$  – amid qlutamin turşusu ilə kompleks birləşmələrinin alınması və əmələgəlmə sabiti öyrənilmişdir. Məhluldan komplekslər fərdi qaydada ayrılmış, quruluş və tərkibləri öyrənilmişdir.

Sintez edilmiş komplekslərdə NMR, İQ- və UB- spektroskopiya tədqiqatları ilə müəyyən edilmişdir ki, komplekslərdə iki molekul liqand azot atomları ilə monodentatlı, digər komplekslərə isə azot və oksigen atomları ilə bidentatlı koordinasiya edirlər. Müvafiq olaraq neytral tipli komplekslərdə  $[Pd_2Namin_2Cl]$  ( $[PdL_2Cl_2]$ ),  $[Pd_2Namin_2Okarb]$  ( $[Pd(HL)_2]$ ) daxili sferada trans-vəziyyətdə yastı kvadrat müstəvisi formalaşır.

**Açar sözlər:** palladium, qlutamin, liqand, dentatlılıq, koordinasiya

## КОМПЛЕКСНЫЕ СОЕДИНЕНИЯ ПАЛЛАДИЯ (II) С $\gamma$ -АМИДОМ ГЛУТАМИНОВОЙ КИСЛОТЫ

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**Аннотация:** В данной работе изучено образование комплексных соединений палладия (II) в водных растворах с  $\gamma$ -амидом глутаминовой кислоты и определены их константы образования. Комплексы выделены в индивидуальном виде и изучены их строение и свойства.

По данным ЯМР, ИК- и УФ- спектроскопии установлено, что происходит монодентатная координация двух молекул лиганда по донорным атомам азота аминогруппы и бидентатная - по атомам азота аминогруппы и кислорода. Формируется плоскоквадратная внутренняя координационная сфера в транс-строении, соответственно в комплексах  $[Pd_2Namin_2Cl]$  ( $[PdL_2Cl_2]$ ) и  $[Pd_2Namin_2Okarb]$  ( $[Pd(HL)_2]$ ).

**Ключевые слова:** палладий, глутамин, лиганд, дентатность, координация