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СИНТЕЗ И СТРОЕНИЕ ТРИХЛОРО(ДИМЕТИЛСУЛЬФОКСИДО)ПЛАТИНАТА (МЕТОКСИМЕТИЛ)ТРИФЕНИЛФОСФОНИЯ

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Взаимодействием гексахлороплатиноводородной кислоты с хлоридом (метоксиметил)трифенилфосфония в диметилсульфоксиде синтезирован и структурно охарактеризован трихлоро(диметилсульфоксидо)платинат (метоксиметил)трифенилфосфония $[Ph_3PCH_2OCH_3][PtCl_3(dmso\text{-}S)]$. В ходе реакции наблюдалось восстановление Pt(IV) до Pt(II). Реакция сопровождалась лигандным обменом, в ходе которой происходит замещение одного из атомов хлора на молекулу S-координированного диметилсульфоксида в анионе. Медленное испарение растворителя привело к образованию крупных оранжевых кристаллов. Структура полученного соединения идентифицирована методом рентгеноструктурного анализа. Структура расшифрована прямым методом. Позиции и температурные параметры неводородных атомов уточнены в изотропном, а затем в анизотропном приближении полноматричным МНК. РСА кристалла I проведен на дифрактометре D8 QUEST фирмы Bruker. По данным РСА, кристаллографические параметры элементарной ячейки соединения I [моноклинная сингония, пространственная группа $P2_1/c$, M 686,90, a 14,48(2), b 14,48(2), c 19,99(3) Å]. Тетраэдрическая конфигурация катиона (метоксиметил)трифенилфосфония приближается к идеальному значению: величины валентных углов CPC равны 108,5(2)°-110,3(2)°, длины связей P-C незначительно различаются между собой. Атом платины в анионах I имеет плоско-квадратную координацию (транс-расположенные углы ClPtCl и SPtCl равны 177,65(5)° и 178,88(6)°, соответственно; цис-углы приближаются к значению 90°). Расстояния Pt–Cl и Pt–S составляют 2,290(3)–2,314(3) Å и 2,205(3) Å. Атом платины в анионе координирован тремя атомами хлора и молекулой диметилсульфоксида. Молекула диметилсульфоксида координируется на атом платины через атом серы. Геометрия координированного диметилсульфоксидного лиганда (углы OSC и CSC, длина связи S−O) отличается от геометрии свободной молекулы диметилсульфоксида.

Ключевые слова: трихлоро(диметилсульфоксидо)платинат, хлорид (метоксиметил)трифенилфосфония, диметилсульфоксид, рентгеноструктурный анализ

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SYNTHESIS AND STRUCTURE OF (METHOXYMETHYL)TRIPHENYLPHOSPHONIIUM TRICHLORO(DIMETHYLSULFOXIDO)PLATINATE

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(Methoxymethyl)triphenylphosphonium trichloro(dimethylsulfoxido)platinate [$Ph_3PCH_2OCH_3$][$PtCl_3$(dmso-S)] was synthesized by the reaction of hexachloroplatinic acid with (methoxymethyl)triphenylphosphonium chloride in dimethyl sulfoxide. During the reaction, Pt (IV) was reduced to Pt (II). The reactions are accompanied by the ligand exchange in anions with substitution of the S-coordinated dimethyl sulfoxide molecule for one of chlorine atoms. Slow evaporation of the solvent led to the formation of large orange crystals. The product structure was determined by XRDA. The structures were interpreted by the direct method. Positions and temperature parameters of non-hydrogen atoms were refined in isotropic and then in anisotropic approximations by the full-matrix LSM. The X-ray diffraction pattern of crystal I was carried out on a Bruker D8 QUEST diffractometer. According to the data of X-ray analysis the unit cell crystallographic parameters of compound I are [crystal system monoclinic, space group $P2_1/c$, M 686.90, a 14.48(2), b 14.48(2), c 19.99(3) Å]. The tetrahedral configuration of the (methoxymethyl)triphenylphosphonium cation approaches the ideal values (CPC angles are 108.5(2)°-110.3(2)°, bond lengths P-C are slightly differ from each other). Platinum atoms in anions have square coordination (trans-arranged angles ClPtCl are 177.65(5)°, SPtCl are 178.88(6)°, cis-angles approach 90°). The bond lengths Pt–Cl are equal to 2.290(3)–2.314(3) Å, Pt–S are equal to 2.205(3) Å. The platinum atoms are coordinated by three chlorine atoms and dimethyl sulfoxide molecule in the anion. Dimethyl sulfoxide molecule is coordinated to the platinum atoms by sulfur atoms. The geometry of the coordinated dimethyl sulfoxide ligand [OSC and CSC angles, S−O bond] differs from the geometry of a free dimethyl sulfoxide molecule.

Key words: trichloro(dimethylsulfoxido)platinate, (methoxymethyl)triphenylphosphonium chloride, dimethyl sulfoxide, X-ray diffraction analysis

INTRODUCTION

The study of new platinum coordination compounds is an important trend in chemistry. Platinum(II) complexes are thermodynamically and kinetically stable compounds and they are used to study various types of isomerism and the nature of trans- and cis-effects. From this viewpoint, the reactions that result in the introduction of new ligands into the coordination sphere of the platinum atoms are of great interest [1]. Drugs synthesized on the basis of platinum coordination compounds have been widely used for the treatment of a number of tumors [2]. Though, they are widely used, these pharmacological compounds have significant side effects. A selection of the nature of the leaving group and ligands may improve the pharmacological properties of the drugs and decrease unwanted effects [3]. Platinum complexes with sulfur-containing ligands are less toxic and can prevent inactivation of the cisplatin [4, 5]. Therefore, the study of platinum dimethyl sulfoxide complexes is of particular interest.

To continue the studies of the synthesis and structure features of platinum complexes [6-15], we carried out the reaction of (methoxymethyl)-triphenylphosphonium chloride with hexachloroplatinic acid. The structure of the obtained complex was determined by X-ray diffraction analysis (XRDA).

EXPERIMENTAL PART

Synthesis of [$Ph_3PCH_2OCH_3$][$PtCl_3$(dmso-S)] (I). A solution of 50 mg (0.1 mmol) hexachloroplatinic acid and 35 mg (0.1 mmol) (methoxymethyl)triphenylphosphonium chloride were dissolved with stirring in 3 mL of dimethyl sulfoxide. The solution was concentrated, the crystals formed were filtered off and dried. The orange crystals I (49 mg (63%), Mp = 143-145 °C) were obtained. Anal. found, %: C 38.41; H 3.82. Anal. calc. for $C_{22}H_{26}O_2PCl_3PtS$ %: C 38.45; H 3.79.

The X-ray diffraction pattern of crystal **I** was carried out on a Bruker D8 QUEST diffractometer (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). A glass thread was used as the sample holder. At one end of the glass thread, a single-crystal was glued, and the second end of the thread was fixed in a standard goniometric head of the diffractometer. The data were collected, edited and parameterized by the SMART and SAINT-Plus [16]. All calculations for the determination and refinement of the structure were performed by the programs SHELXL/PC [17], OLEX2 [18]. The structure was determined by a direct method and refined by the method of least squares in the anisotropic approximation for non-hydrogen atoms. The crystallographic data and the results of structure refinement are given in Table. Completed tables of atomic coordinates, bond lengths and valence angles are deposited in the Cambridge structural data bank (№1912245; deposit@cc-dc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Table

Crystallographic date, experimental parameters and structure refinements for compound I

***Таблица*. Кристаллографические данные, параметры эксперимента и уточнения структуры I**

Parameter	
Formula weight	686.90
Crystal system	Monoclinic
T, K	293(2)
Space group	$P2_1/c$
a, Å	14.48(2)
b, Å	9.640(15)
c, Å	19.99(3)
α, °	90
β, °.	111.15(5)
γ, °	90
V, Å^3	2603(7)
ρ_{calc}, g/cm^3	1.753
Crystal size, mm	0.51×0.39×0.34
Index ranges	$-21 \le h \le 21$, $-14 \le k \le 14$, $-30 \le l \le 30$

RESULTS AND DISCUSSION

The complex **I** was obtained by interaction of hexachloroplatinic acid with (methoxymethyl)triphenylphosphonium chloride following the equation

$$[Ph_3PCH_2OCH_3]Cl + H_2PtCl_6 + SO(CH_3)_2 \rightarrow$$
$$\rightarrow [Ph_3PCH_2OCH_3][PtCl_3(SO(CH_3)_2)] + 2HCl + Cl_2 \quad \mathbf{I}$$

Slow evaporation of the solvent leads to the formation of orange crystals of (methoxymethyl)triphenylphosphonium trichloro(dimethylsulfoxido)platinate.

The interaction of hexachloroplatinic acid with chloride (methoxymethyl)triphenylphosphonium is accompanied by the redox reaction Pt(IV) → Pt(II). Reduction transformations of Pt(IV) complexes to Pt(II) are characteristic of complexes with coordinated dimethyl sulfoxide molecules in acetone, nitromethane, acetonitrile, dimethyl sulfoxide [19]. The reactions are accompanied by the ligand exchange in anions with substitution of the S-coordinated dimethyl sulfoxide molecule for one of chlorine atoms.

According to the X-ray analysis, complex **I** includes (methoxymethyl)triphenylphosphonium cations and trichloro(dimethylsulfoxido)platinate square anions (Fig. 1). The valence angles of CPC approach the ideal tetrahedral value (108.5(2)°–110.3(2)°). The P–C bonds differ slightly from each other and equal (1.769(5)–1.799(5) Å), and approximately equal the lengths of the similar bonds in tetraorganylphosphnium cations. In square $[PtCl_3(DMSO-S)]^-$ anions of complexes I the trans-ClPtCl and the trans-SPtCl equal 177.65(5)° and 178.88(6)°, respectively. cis-Angles ClPtCl and SPtCl are close to ideal value 90° (88.42(13)°, 89.61(13)° and 89.29(13)°, 92.67(13)°, respectively). The bonds of Pt–Cl and Pt–S equal 2.290(3)–2.314(3) Å and 2.205(3) Å, and are close to sums of the covalent radii of the specified atoms [20].

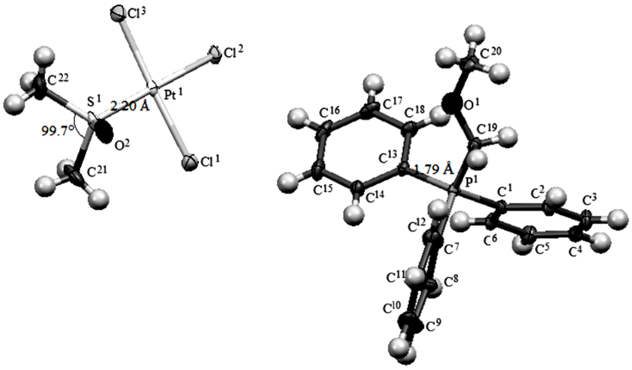

Fig. 1. General view of a complex I molecule
Рис. 1. Общий вид молекулы комплекса I

The geometry of the coordinated dimethyl sulfoxide ligand (OSC angles 107.4(3)°, 108.3(4)°) differs from the geometry of a free dimethyl sulfoxide molecule, where similar angles are 106.7(4), 106.8(4)° [21]. The CSC angle 99.7(4)° also differs from this angle in a free dimethyl sulfoxide molecule (97.4°). The S−C bond lengths (1.777(7) Å, 1.785(8) Å) are comparable with the values 1.771(8), 1.805(11) Å [12]. The S−O bond length of coordinated molecules is significantly shorter 1.466(5) than that in the free dimethyl sulfoxide molecule (1.531 Å [21]), which is consistent with the published data on the increase of the S–O bond order in case of coordination of dimethyl sulfoxide molecule to a metal through the sulfur atom [22].

Cations and anions in crystal of **I** complexes are bound by hydrogen bonds Cl···H 2.84–2.92 Å. In crystal **I** anions are bonded to each other by four hydrogen bonds O···H 2.52–2.63 Å (Fig. 2), cations are linked to each other by two hydrogen bonds H(Ar)···H(Ar) 2.884 Å.

CONCLUSIONS

Thus, complex **I** is an ion compound of platinum. The complex **I** consists of mononuclear tetrahedral cations (methoxymethyl)triphenylphosphonium and trichloro(dimethylsulfoxide)platinate square planar anions, in which dimethyl sulfoxide molecules are coordinated to the central atoms by sulfur.

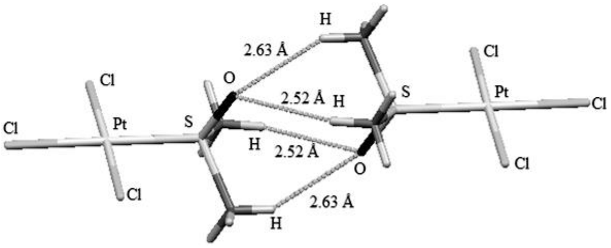

Fig. 2. Hydrogen bonds O···H between dimethyl sulfoxide ligands in the complex I crystal
Рис. 2. Водородные связи O···H между диметилсульфоксидными лигандами в кристалле комплекса I

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