

СИНТЕЗ И СТРОЕНИЕ ГЕКСАТИОЦИАНАТОПЛАТИНАТА(IV) КАЛИЙТЕТРАЭТИЛАММОНИЯ

В.В. Шарутин, О.К. Шарутина, А.Р. Ткачева

Владимир Викторович Шарутин

Управление научной и инновационной деятельности, НИУ Южно-Уральский государственный университет, проспект В.И. Ленина, 76, Челябинск, Российская Федерация, 454080

E-mail: vvsharutin@rambler.ru

Ольга Константиновна Шарутина, Алена Романовна Ткачева

Кафедра теоретической и прикладной химии, НИУ Южно-Уральский государственный университет, проспект В.И. Ленина, 76, Челябинск, Российская Федерация, 454080

E-mail: sharutinao@mail.ru, aesya@mail.ru

Взаимодействием гексакис(изоцианато)платината(IV) калия с хлоридом тетраэтиламмония в водном растворе ацетонитрила синтезирован и структурно охарактеризован гексакис(изоцианато)платинат(IV) калийтетраэтиламмония $[(C_2H_5)_4N][K][Pt(CNS)_6]$. Медленное испарение растворителя привело к образованию крупных красно-коричневых кристаллов. Структура полученного соединения идентифицирована методом РСА. РСА кристалла I проведен на дифрактометре D8 QUEST фирмы Bruker (MoK α -излучение, $l = 0,71073 \text{ \AA}$, графитовый монохроматор). $[C_{14}H_{20}N_7KPtS_6, M = 712,92, \text{ Сингония моноклинная, пространственная группа } C 2/c, a = 10,432(8), b = 14,767(13), c = 16,300(13) \text{ \AA}, V = 2510(4) \text{ \AA}^3, Z = 4, \mu = 6,272 \text{ мм}^{-1}, F(000) = 1384, \text{ размер кристалла } 0,86 \times 0,66 \times 0,50 \text{ мм}].$ Тетраэдрическая конфигурация тетраэтиламмонийного катиона несколько искажена (углы CNC $105,5(5)^\circ$ – $111,8(4)^\circ$, длины связей N-C $1,503(5)$ – $1,519(5) \text{ \AA}$). Ионы платины в анионах I имеют октаэдрическую координацию (транс-углы SPtS составляют 180°), величины цис-углов при атоме платины SPtS приближаются к значению 90° ($88,47(4)^\circ$ – $91,53(4)^\circ$). Длины связей Pt-S в группах Pt(CNS) $_6$ близки между собой и составляют $2,373(2)$ – $2,37(2) \text{ \AA}$. Центросимметричные октаэдрические гексакис(изоцианато)платинатные анионы связаны в единое целое посредством мостиковых изоцианатных лигандов и катионов калия, который гексакоординирован шестью атомами азота изоцианатных групп (N-K $2,828(4)$ – $2,896(4) \text{ \AA}$), однако транс-углы NKN ($128,44(15)^\circ$ – $146,9(2)^\circ$) далеки от идеальных значений для октаэдра. Мостиковые тиоцианатные лиганды связывают катионы платины и калия. Амбидентатные тиоцианатные лиганды одновременно координируются с катионами K $^+$ атомами азота. С помощью мостиковых тиоцианатных лигандов образуется трехмерный координационный полимер. Полученная структура представляет собой трехмерную сетку, в ячейках которой расположены катионы тетраэтиламмония $(Et_4N)^+$.

Ключевые слова: гексатиоцианатоплатинат(IV) калийтетраэтиламмония, синтез, рентгеноструктурный анализ

SYNTHESIS AND STRUCTURE OF POTASSIUM TETRAETHYLAMMONIUM HEXATHIOCYANATOPLATINATE(IV)

V.V. Sharutin, O.K. Sharutina, A.R. Tkacheva

Vladimir V. Sharutin

Department of Science and Innovation, South Ural State University, Lenin ave., 76, Chelyabinsk, 454080, Russia

E-mail: vvsharutin@rambler.ru

Olga K. Sharutina, Alena R. Tkacheva

Department of Theoretical and Applied Chemistry, South Ural State University, Lenin ave., 76, Chelyabinsk, 454080, Russia

E-mail: sharutinao@mail.ru, aesya@mail.ru

Potassium tetraethylammonium hexathiocyanatoplatinate(IV) (Et_4N)(K)[Pt(SCN) $_6$] (I) was synthesized by the reaction of potassium hexathiocyanatoplatinate(IV) with tetraethylammonium chloride in acetonitrile aqueous solution. Slow evaporation of the solvent led to the formation of large red-brown crystals. The product structure was determined by XRDA. The X-ray diffraction pattern of crystal I was carried out on a Bruker D8 QUEST diffractometer (MoK $_{\alpha}$ radiation, $\lambda = 0.71073 \text{ \AA}$, graphite monochromator). [$C_{14}H_{20}N_7KPtS_6$, $M = 712.92$, Crystal system monoclinic, space group C 2/c, $a = 10.432(8)$, $b = 14.767(13)$, $c = 16.300(13) \text{ \AA}$, $V = 2510(4) \text{ \AA}^3$, $Z = 4$, $\mu = 6.272 \text{ mm}^{-1}$, $F(000) = 1384$, crystal size $0.86 \times 0.66 \times 0.50 \text{ mm}$]. The tetrahedral configuration of the tetraethylammonium cation is slightly distorted (CNC angles are $105.5(5)^\circ$ - $111.8(4)^\circ$, bond lengths N-C are $1.503(5)$ - $1.519(5) \text{ \AA}$). Platinum ions in anions have octahedral coordination (trans-angles SPtS are 180° , cis-angles SPtS are $(88.47(4)^\circ)$ - $91.53(4)^\circ$). The bond lengths Pt-S are equal to $2.373(2)$ - $2.37(2) \text{ \AA}$. Potassium cations are coordinated by six nitrogen atoms of thiocyanate groups (distances N K $(2.828(4)$ - $2.896(4) \text{ \AA}$). Trans-angles NKN $(128.44(15)^\circ)$ - $146.9(2)^\circ$ are far from ideal values for the octahedron. Bridged thiocyanate ligands are bonded cations of the platinum and potassium. Ambidentate thiocyanate ligands are simultaneously coordinated to the K $^+$ cation by nitrogen atoms. By means of the bridged thiocyanate ligands a three-dimensional coordination polymer is formed. The resulting structure is a three-dimensional grid, in the cells of which the cations of tetraethylammonium (Et_4N) $^+$ are located.

Key words: potassium tetraethylammonium hexathiocyanatoplatinate(IV), synthesis, X-ray diffraction analysis

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INTRODUCTION

It is known that complex compounds of platinum are effective catalysts for many chemical processes and convenient objects for the study of ligand exchange reactions. For example, in the presence of platinum complexes alkynes are oxidized by the air oxygen through a triple bond to dicarbonyl compounds [1]. Ligand exchange reactions are observed in tetrachloro- and tetraorganophosphonium hexachloroplatinate solutions in dimethylsulfoxide [2]. Among various structurally characterized complexes

of platinum the complexes with thiocyanate ligands are practically not studied [3].

In continuation of the studies of the synthesis and structure features of platinum complexes [4-16], we carried out the reaction of tetraethylammonium chloride with potassium hexathiocyanatoplatinate(IV). The structure of the obtained complex was determined by X-ray diffraction analysis (XRDA).

EXPERIMENTAL

The mixing of water-acetonitrile solutions of equimolar amounts of tetraethylammonium chloride

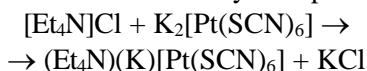
and potassium hexathiocyanatoplatinate(IV) is accompanied by a change in the color of the reaction mixture to red-brown.

Synthesis of $(Et_4N)(K)[Pt(SCN)_6]$ (I). A solution of 130 mg (0.785 mmol) of tetraethylammonium chloride in 60 mL of a 1: 1 mixture of water-acetonitrile was poured into a solution of 488 mg (0.785 mmol) of potassium hexathiocyanatoplatinate(IV) in 60 mL of water-acetonitrile (1: 1). The solution was concentrated, the crystals formed were filtered and dried. The red-brown crystals I (399 mg (56%), $T_m = 187^\circ C$) were obtained. Anal. found, %: C 23.55; H 2.84. Anal. calc. for $C_{14}H_{20}N_7KPtS_6$ %: C 23.59; H 2.81.

The X-ray diffraction pattern of crystal I was carried out on a Bruker D8 QUEST diffractometer (MoK_α radiation, $\lambda = 0.71073 \text{ \AA}$, 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 [17]. All calculations for the determination and refinement of the structure were performed by the programs SHELXL / PC [18], OLEX2 [19]. The structure is 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. Complete tables of atomic coordinates, bond lengths and valence angles are deposited in the Cambridge structural data bank (N1577805; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

Slow evaporation of the solvent leads to the formation of large red-brown crystals of potassium tetraethylammonium hexathiocyanatoplatinate(IV):



Platinum complexes with hexathiocyanatoplatinate anions and ammonium cations [20] or metal-containing cations of complex structure [21] were described in the literature.

According to X-ray diffraction data, complex I includes tetraethylammonium, potassium, and hexathiocyanatoplatinate anions. The tetrahedral configuration of the $(Et_4N)^+$ cation is slightly distorted: the valence angles of CNC vary in the interval $105.5(5)^\circ$ – $111.8(4)^\circ$. The lengths of NNC bonds differ insignificantly ($1.503(5)$ – $1.519(5) \text{ \AA}$), and their values are approximately equal to the lengths of the

similar bonds in tetraorganammonium cations [14]. Thiocyanate ligands in the $[Pt(SCN)_6]^{2-}$ anion are coordinated to the platinum ion, as expected, by sulfur atoms. The octahedral coordination of the platinum ion is slightly distorted: the SPtS trans-angles are 180° , the SPtS cis angles are approximately equal to the ideal value of 90° ($88.47(4)^\circ$ – $91.53(4)^\circ$). The Pt–S bonds are close to each other and equal $2.375(2)$ – $2.380(2) \text{ \AA}$. The PtSC angles (103.2° – 105.7°) differ slightly from the tetrahedral angle. The SCN⁻ ligands have almost linear structure (SCN angles are $175.3(5)^\circ$ – $178.1(4)^\circ$).

Ambidentate thiocyanate ligands are simultaneously coordinated to the K^+ cation by nitrogen atoms. Each potassium cation is coordinated by six ligands (Fig. 1).

Table
Crystallographic data, experimental parameters and structure refinements for compound I

Таблица. Кристаллографические данные, параметры эксперимента и уточнения структуры I

Parameter	
Formula weight	712.92
Crystal system	Monoclinic
<i>T</i> , K	293(2)
Space group	C 2/c
<i>a</i> , Å	10.432(8)
<i>b</i> , Å	14.767(13)
<i>c</i> , Å	16.300(13)
α , °	90
β , °	91.96(3)
γ , °	90
<i>V</i> , Å ³	2510(4)
<i>Z</i>	4
ρ_{calc} , g/cm ³	1.887
μ_{Mo} , mm ⁻¹	6.272
<i>F</i> (000)	1384
Crystal size, mm	0.86×0.66×0.50
θ , grad.	3.029 – 26.37
Index ranges	$-13 \leq h \leq 13$, $-18 \leq k \leq 18$, $-18 \leq l \leq 20$
Measured reflections	20196
Independent reflections	2571
Refinement variables	138
<i>GOOF</i>	1.059
Final <i>R</i> indexes $F^2 > 2\sigma(F^2)$	$R_1 = 0.0252$ $wR_2 = 0.0642$
Final <i>R</i> indexes [all date]	$R_1 = 0.0301$ $wR_2 = 0.0692$
Largest diff. peak/hole / <i>e</i> /Å ³	-1.515 / 1.319

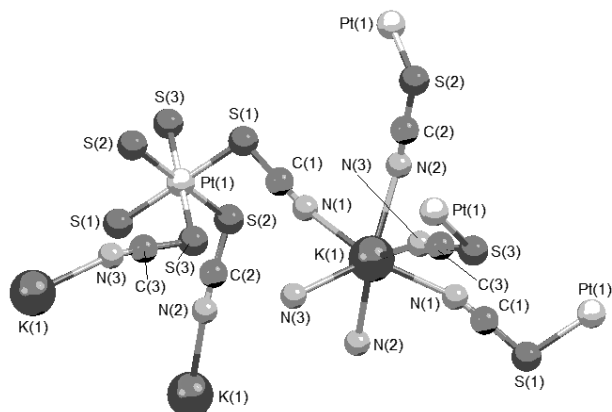


Fig. 1. A fragment of coordination polymer I with thiocyanate bridges

Рис. 1. Фрагмент координационного полимера I с тиоцианатными мостиками

The N–K distances (2.828(4)–2.896(4) Å) are close to the sum of the covalent radii of these atoms (2.66 Å). However, the octahedral coordination of potassium is highly distorted, since the NKN trans-angles (128.44(15)°–146.9(2)°) are far from the ideal value of 180°. The KNC angles are 154.0°–168.0°.

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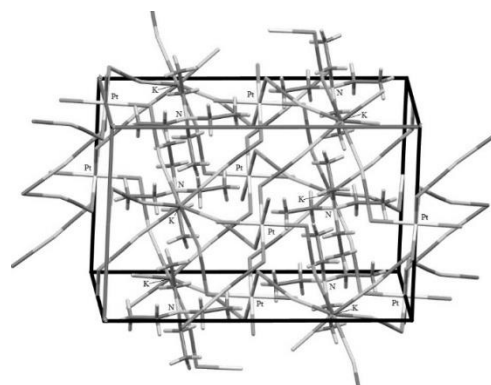


Fig. 2. A fragment of coordination polymer I in the crystal cell
Рис. 2. Фрагмент координационного полимера I в ячейке кристалла

CONCLUSIONS

Thus, complex I is a coordination polymer in which the bridged SCN^- ligands bind two metal cations (Pt and K). The resulting structure is a three-dimensional grid, in the cells of which the cations of tetraethylammonium (Et_4N^+) are located (Fig. 2).

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