

**СИНТЕЗ И СТРОЕНИЕ ДИЦИАНОАРГЕНТАТОВ ОРГАНИЛТРИФЕНИЛФОСФОНИЯ:
[Ph₃P(CH₂)₃PPh₃]²⁺ [Ag(CN)₂]⁻ [Br]⁻ И [Ph₃PR]⁺ [Ag(CN)₂]⁻, R = CH₂CH₂OH, CH₂C₆H₄F-4**

В.В. Шарутин, О.К. Шарутина, М.А. Попкова, Е.И. Данилина

Владимир Викторович Шарутин, Ольга Константиновна Шарутина, Марина Аркадьевна Попкова*,
Елена Ивановна Данилина

Кафедра теоретической и прикладной химии, кафедра экологии и химической технологии, Химический факультет, Южно-Уральский государственный университет, просп. Ленина, 76, Челябинск, Российская Федерация, 454080

E-mail: vvsharutin@rambler.ru, sharutinao@mail.ru, m.popckowa2014@yandex.ru*, danilinaei@susu.ru

По реакции дидицианоаргентаата калия с бромидами органилтрифенилфосфония в воде синтезированы комплексы [Ph₃P(CH₂)₃PPh₃]²⁺[Ag(CN)₂]⁻[Br]⁻ (1), [Ph₃PR]⁺[Ag(CN)₂]⁻, R = CH₂CH₂OH (2), CH₂C₆H₄F-4 (3), строение которых установлено методом рентгеноструктурного анализа. Известно, что в ИК-спектрах органических и неорганических соединений область поглощения циано-групп находится в достаточно узком интервале значений, 2200–2000 см⁻¹, что указывает на отсутствие сильного влияния окружения на колебания этих связей. Полосы поглощения в области 1450–1435 см⁻¹, характерные для связей P–Ph, присутствующие в спектрах соединений 1, 2, 3, проявляют себя в области 1437–1439 см⁻¹. По данным РСА, проведенного при 293 К на автоматическом четырехкружном дифрактометре D8 QuestBruker, кристаллы 1 (C₄₁H₃₆AgBrN₂P₂, M 806,44, сингония триклинная, размер кристалла 0,72 × 0,37 × 0,3 мм), 2 (C₂₂H₂₀AgN₂OP, M467,24, сингония триклинная, размер кристалла 0,8 × 0,52 × 0,35 мм), 3 (C₂₇H₂₁AgFN₂P, M531,30, сингония моноклинная, размер кристалла 0,28 × 0,23 × 0,22 мм). По данным РСА, в кристаллах 1, 2, 3 присутствуют катионы органилтрифенилфосфония и линейные дидицианоаргентатные анионы. В кристалле 1 наблюдаются еще и бром-анионы. Особенность комплексов с анионами [Ag(CN)₂]⁻ состоит в том, что они являются перспективными в плане создания новых нано- и жидкокристаллических систем. Катионы органилтрифенилфосфония имеют искаженную тетраэдрическую конфигурацию (углы СРС изменяются в интервале 106,70(8)°–113,37(9)°, в анионах [Ag(CN)₂]⁻ углы САС равны 171,75(10)° - 178,37(8)°.

Ключевые слова: дидицианоаргентат калия, бромиды органилтрифенилфосфония, рентгеноструктурные исследования, дидицианоаргентаты алкилтрифенилфосфония

**SYNTHESIS AND STRUCTURE
OF ORGANYLTRIPHENYLPHOSPHONIUM DICYANOARGENTATES:
[Ph₃P(CH₂)₃PPh₃]²⁺ [Ag(CN)₂]⁻ [Br]⁻ AND [Ph₃PR]⁺ [Ag(CN)₂]⁻, R = CH₂CH₂OH, CH₂C₆H₄F-4**

V.V. Sharutin, O.K. Sharutina, M.A. Popkova, E.I. Danilina

Vladimir V. Sharutin, Olga K. Sharutina, Marina A. Popkova*, Elena I. Danilina

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

E-mail: vvsharutin@rambler.ru, sharutinao@mail.ru, m.popckowa2014@yandex.ru*, danilinaei@susu.ru

The reaction of potassium dicyanoargentate with organyltriphenylphosphonium bromides in an aqueous solution has led to synthesis of complexes [Ph₃P(CH₂)₃PPh₃]²⁺[Ag(CN)₂]⁻[Br]⁻ (1), [Ph₃PR]⁺[Ag(CN)₂]⁻, R = CH₂CH₂OH (2), CH₂C₆H₄F-4 (3), whose structures have been characterized by X-ray diffraction analysis (XRD). It is known that the absorption range of cyano groups in IR spectra of organic and inorganic compounds is in the form of a narrow interval of 2200–2000 cm⁻¹, which points at the absence of strong surroundings influence on vibrations of these bonds. The absorption bands in the range 1450–1435 cm⁻¹ peculiar to the P–Ph bands that are present in the

spectra of compounds 1, 2, 3 are apparent in the range 1437-1439 cm⁻¹. The XRD data have been obtained at 293 K on a Bruker D8 QUEST automatic four-circle diffractometer: crystals 1 (C₄₁H₃₆AgBrN₂P₂, M 806.44, triclinic syngony, crystal size 0.72 × 0.37 × 0.3 mm), 2 (C₂₂H₂₀AgN₂OP, M 467.24, triclinic syngony, crystal size 0.8 × 0.52 × 0.35 mm), 3 (C₂₇H₂₁AgFN₂P, M 531.30, monoclinic syngony, crystal size 0.28 × 0.23 × 0.22 mm). According to the XRD data, crystals 1, 2, 3 contain organyltriphenylphosphonium cations and linear dicyanoargentate anions. Besides, bromide anions are observed in crystal 1. A characteristic feature of complexes with the [Ag(CN)₂]⁻ anions is their perspective utility for development of new nano- and liquid crystal systems. The organyltriphenylphosphonium cations have a distorted tetrahedral configuration: the CPC angles vary within the range 106.70(8)° - 113.37(9)°. In the [Ag(CN)₂]⁻ anions the CAgC angles equal 171.75(10)° - 178.37(8)°.

Key words: potassium dicyanoargentate, organyltriphenylphosphonium bromide, X-ray diffraction analysis, alkyltriphenylphosphonium dicyanoargentates

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INTRODUCTION

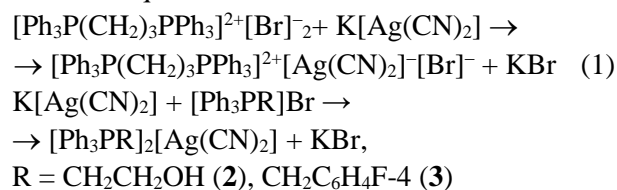
At the present time complexes of metal dicyanides attract researchers' interest in the context of their use for production of semiconductors and optically active materials, which take on various properties [1-6]. Thus, the peculiar structure of dicyanoargentate anions favoring oligomer formation leads to appearance of magnetic activity [7]. Complexes with the [Ag(CN)₂]⁻ anions show great promise for development of new nano- and liquid crystal systems [8, 9].

From the standpoint of a search for structures with valuable physicochemical properties, variation of a cation part of the dicyanoargentate complexes looks promising [10], as the dicyanoargentate anions in crystals, depending on the cation structure, can appear as monomers [2, 11, 12], branched polymers [13,14], perform the bridging function for cations and anions [15], form "dimers" with short Ag...Ag distances [16]. It is pertinent to note that the [Ag(CN)₂]⁻ complexes with organic and organoelement cations are practically unstudied. In the literature the examples of such systems are few in number: with imidazolium cations [11, 17, 18], bis(triphenylphosphine)iminium [19] and triphenyltellurium [20].

EXPERIMENTAL PART

In the present study organyltriphenylphosphonium dicyanoargentate complexes were synthesized and their structures were characterized, namely: [Ph₃P(CH₂)₃PPh₃]²⁺[Ag(CN)₂]⁻[Br]⁻ (**1**) and [Ph₃PR]⁺[Ag(CN)₂]⁻, R = CH₂CH₂OH (**2**), CH₂C₆H₄F-4 (**3**).

Complexes **1–3** were synthesized from potassium dicyanoargentate and organyltriphenylphosphonium bromides in aqueous solutions:



After evaporation of water, drying of the solid precipitate and its recrystallization from acetonitrile, the colorless crystals, stable in air, were obtained. It is known that in IR spectra of organic and inorganic compounds the range of the cyano groups absorption is in the fairly narrow range of 2200-2000 cm⁻¹, which means that the surroundings do not influence the vibrations of these bonds significantly [21]. Consequently, cyanides are easily identified on the strength of their absorption in the stated range. Thus, in the spectra of compounds **1, 2, 3** the intensive bands have been observed at 2133, 2135, 2131 cm⁻¹, respectively.

The absorption bands existing in the spectra of compounds **1, 2, 3** that characterize the P–Ph bonds [21] are observed in the range 1437-1439 cm⁻¹. Besides, spectrum **2** contains the absorption band at 3283 cm⁻¹ that characterizes the hydroxyl group vibrations [21].

According to the XRD data, crystals **1, 2, 3** consist of organyltriphenylphosphonium cations and monomer dicyanoargentate anions, and in crystal **1** bromide anions are also observed (Fig. 1-3).

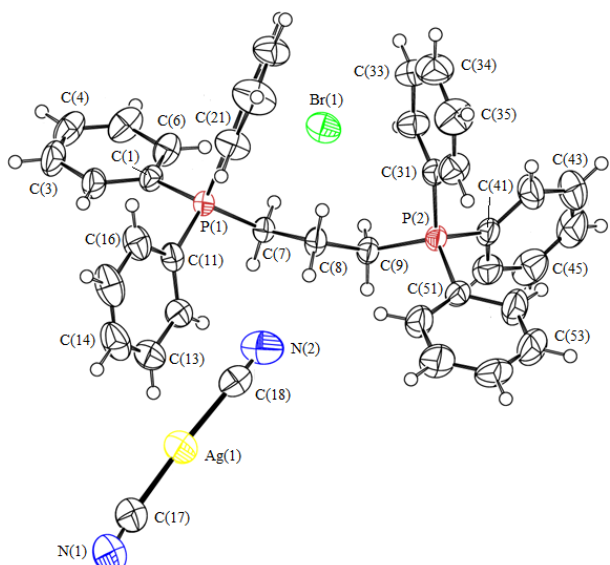


Fig. 1. The structure of complex 1 (hydrogen atoms are not shown)
Рис. 1. Структура комплекса 1 (атомы водорода не показаны)

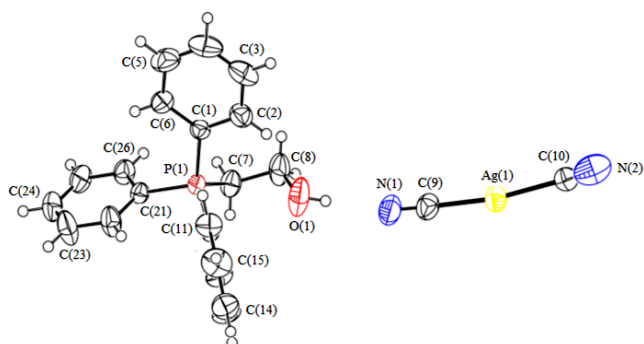


Fig. 2. The structure of complex 2
Рис. 2. Структура комплекса 2

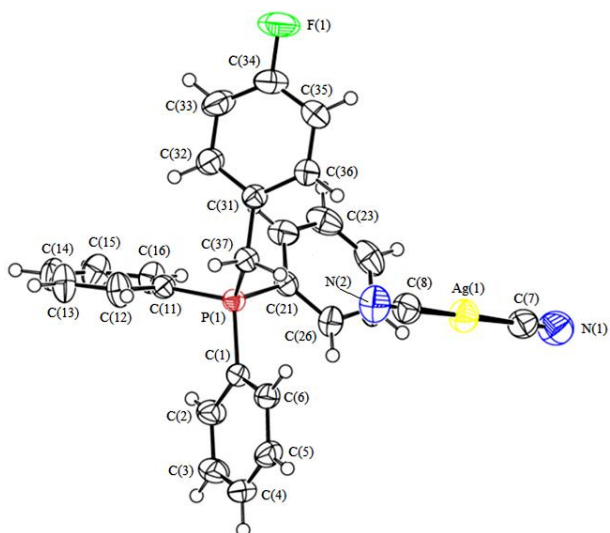


Fig. 3. The structure of complex 3
Рис. 3. Структура комплекса 3

Tetrahedral coordination of the phosphorus atoms in cations is somewhat distorted: the CPC angles deviate from the theoretical value and amount to

106.70(8)°-113.37(9)° (**1**), 106.75(10)°-112.67(10)° (**2**), 106.88(7)°-111.09(7)° (**3**); the lengths of the P-C_{Ph} bonds (1.7875(19)-1.8049(18), 1.7925(19)-1.7983(19) Å (**1**), 1.7926(19)-1.798(2) Å (**2**), 1.7977(16)-1.8009(17) Å (**3**) are close to each other, but they are shorter than the P-C_{alk} distances (1.7987(17), 1.8112(18) Å (**1**), 1.807(2) Å (**2**), 1.8123(16) Å (**3**). The [Ag(CN)₂]⁻ anions deviate from the linear configuration, the CAgC angles equal 176.10(11)° (**1**), 171.75(10)° (**2**), 178.37(8)° (**3**). The Ag-C distances in anions **1-3** are significantly different: 2.063(3) and 2.089(3) Å in **1**, 2.054(3) and 2.186(3) in **2**, 2.069(2) and 2.062(2) Å in **3**. The C≡N triple bonds in the cyanide ligands of these anions equal 1.035(3), 1.091(4) Å (**1**); 0.841(4), 1.134(4) Å (**2**), 1.114(3), 1.130(3) Å (**3**). Structural organization of the crystals is caused by weak hydrogen bonds N...H-C between cations and anions (2.74–2.58 Å (**1**), 2.54–2.68 Å (**2**), 2.48–2.73 Å (**3**)), as well as C...H-C (2.77 Å (**1**), 2.81–2.90 Å (**2**)). In crystals **1** and **2** close contacts H...Br (2.69–2.99 Å) and N...H-O (1.98 Å) are present, respectively.

Thus, the silver complexes, obtained from organyltriphenylphosphonium bromides and potassium dicyanoargentates in water, have ionic structure with monomer dicyanoargentate anions.

RESULTS AND DISCUSSION

The IR spectra of complexes **1-3** were recorded on a Fourier transform infrared spectrophotometer Shimadzu IR Affinity-1S in a KBr pellet.

The XRD analysis of crystals **1-3** was performed on a Bruker D8 QUEST automatic four-circle diffractometer (two-coordinate CCD detector, Mo K α emission, $\lambda = 0.71073$ Å, graphite monochromator) at 293 K. The data were collected and analyzed, the unit cell parameters were refined, and the absorption correction was applied using the SMART and SAINT-Plus programs [22]. All calculations for structure determination and refinement were performed using the SHELXL/PC [23] and OLEX2 [24] programs. The structures were determined by the direct method and refined by the least-squares method in the anisotropic approximation for non-hydrogen atoms. The main crystallographic data and refinement results for structures **1-3** are listed in Table.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CCDC 1897387 (**1**), 1897370 (**2**), 1897369 (**3**); deposit@ccdc.cam.ac.uk; <http://www.ccdc.cam.ac.uk>).

Crystallographical data and refinement results for structures 1–3
Таблица. Кристаллографические данные и результаты структур 1-3

Parameter	Value		
	1	2	3
<i>M</i>	806.44	467.24	531.30
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P\bar{1}$	$P2_1/c$
<i>a</i> , Å	9.577(4)	7.808(3)	9.872(4)
<i>b</i> , Å	10.527(5)	9.392(4)	15.100(6)
<i>c</i> , Å	18.830(10)	15.704(9)	15.551(6)
α , deg	88.80(3)	99.71(3)	90.00
β , deg	86.52(2)	100.328(18)	90.987(14)
γ , deg	73.978(18)	108.276(17)	90.00
<i>V</i> , Å ³	1821.4(16)	1044.1(9)	2317.9(15)
<i>Z</i>	2	2	4
ρ (calcd), g/cm ³	1.470	1.486	1.523
μ , mm ⁻¹	1.769	1.055	0.964
<i>F</i> (000)	816.0	472.0	1072.0
Crystal size, mm	0.72 × 0.37 × 0.3	0.8 × 0.52 × 0.35	0.28 × 0.23 × 0.22
Range of data collection 2 θ , deg	5.92–63.18	5.94–81.08	6.62–60.1
Range of refraction indices	-14 ≤ <i>h</i> ≤ 14, -15 ≤ <i>k</i> ≤ 15, -27 ≤ <i>l</i> ≤ 27	-14 ≤ <i>h</i> ≤ 14, -17 ≤ <i>k</i> ≤ 17, -28 ≤ <i>l</i> ≤ 28	-13 ≤ <i>h</i> ≤ 13, -21 ≤ <i>k</i> ≤ 21, -21 ≤ <i>l</i> ≤ 18
Measured reflections	116476	87190	69762
Independent reflections	12125 (<i>R</i> _{int} = 0.0336)	13311 (<i>R</i> _{int} = 0.0425)	6773 (<i>R</i> _{int} = 0.0371)
<i>R</i> _{int} > 2 σ (<i>I</i>)	8543	6976	5032
Refinement variables	424	245	289
<i>GOOF</i>	1.008	1.014	1.031
<i>R</i> -factors for <i>F</i> ² > 2 σ (<i>F</i> ²)	<i>R</i> ₁ = 0.0360, <i>wR</i> ₂ = 0.0829	<i>R</i> ₁ = 0.0580, <i>wR</i> ₂ = 0.1266	<i>R</i> ₁ = 0.0320, <i>wR</i> ₂ = 0.0750
<i>R</i> -factors for all reflections	<i>R</i> ₁ = 0.0628, <i>wR</i> ₂ = 0.0944	<i>R</i> ₁ = 0.1292, <i>wR</i> ₂ = 0.1523	<i>R</i> ₁ = 0.0536, <i>wR</i> ₂ = 0.0831
Residual electron density (max/min), e/Å ³	0.38/-0.80	0.92/-0.55	0.25/-0.70

Synthesis of trimethylene-bis(triphenylphosphonium) dicyanoargentate bromide [Ph₃PCH₂CH₂CH₂PPh₃]²⁺[Ag(CN)₂]⁻[Br]⁻ (1). To the solution of 181 mg (0.25 mmol) trimethylene-bis(triphenylphosphonium) dibromide in 10 mL of water the solution of 50 mg (0.25 mmol) potassium dicyanoargentate in 4 mL of water was added. The mixture was kept at 20 °C for 14 h. Water was removed, the residue was extracted into acetonitrile (4 mL twice). During slow evaporation of the solvent formation of colorless crystals was observed. The yield was 185 mg (92%) of complex **1**, m.p. 139.6 °C.

The IR spectrum (ν , cm⁻¹): 3446, 3170, 3163, 3086, 3057, 3047, 3010, 2993, 2945, 2916, 2877, 2808, 2690, 2250, 2214, 2137, 2129, 2090, 2002, 1973, 1901, 1816, 1778, 1631, 1587, 1487, 1438, 1415, 1338, 1319, 1290, 1251, 1195, 1163, 1112, 1028, 997, 962, 929, 846, 825, 767, 742, 732, 723, 700, 688, 669, 655, 617, 538, 526, 513, 495, 480, 445, 435, 418.

Found, %: C 59.75, H 4.53.

ForC₄₁H₃₆AgBrN₂P₂

calculated, %: C 61.01, H 4.46.

Compounds **2**, **3** were synthesized along similar lines.

(**2**) (91%), m.p. 153.0 °C. The IR spectrum (ν , cm⁻¹): 3282, 3086, 3059, 3028, 2947, 2899, 2881, 2850, 2833, 2750, 2216, 2135, 1975, 1905, 1826, 1782, 1670, 1610, 1587, 1483, 1436, 1394, 1340, 1315, 1296, 1213, 1192, 1163, 1112, 1078, 1026, 1008, 997, 948, 854, 812, 754, 748, 731, 717, 694, 686, 661, 543, 499, 491, 462, 439.

Found, %: C 56.39, H 4.35.

ForC₂₂H₂₀AgN₂OP

calculated, %: C 56.53, H 4.28.

(**3**) (93%), m.p. 214.0 °C. The IR spectrum (ν , cm⁻¹): 3441, 3167, 3095, 3072, 3057, 3024, 2993, 2972, 2935, 2895, 2848, 2804, 2686, 2586, 2208, 2131,

2086, 1978, 1913, 1894, 1824, 1770, 1666, 1600, 1585, 1510, 1489, 1481, 1435, 1425, 1409, 1338, 1319, 1294, 1274, 1255, 1236, 1228, 1199, 1182, 1163, 1155, 1107, 1072, 1020, 995, 979, 972, 956, 939, 931, 856, 831, 786, 759, 750, 740, 717, 702, 688, 669, 634, 615, 551, 509, 501, 489, 476, 445, 437, 414.

Found, %: C 60.57, H 4.07.

For $C_{27}H_{21}AgFN_2P$

calculated, %: C 60.98, H 3.95.

CONCLUSION

Thus, the interaction of potassium dicyanoargentate with organyltriphenylphosphonium bromides in water was used to synthesize new complexes of organyltriphenylphosphonium dicyanoargentates $[Ph_3P(CH_2)_3PPh_3]^{2+} [Ag(CN)_2]^- [Br]^-$ and $[Ph_3PR]^{+} [Ag(CN)_2]^-$, R = CH_2CH_2OH , $CH_2C_6H_4F-4$, whose structure was characterized by X-ray diffraction and IR spectroscopy.

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