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СИНТЕЗ И СТРОЕНИЕ АРЕНСУЛЬФОНАТОВ ТЕТРАФЕНИЛФОСФОНИЯ

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Взаимодействием пентафенилфосфора (Ph_5P) с аренсульфоновыми кислотами в растворе бензола синтезированы аренсульфонаты тетрафенилфосфония с общей формулой $[Ph_4P]^+[O_3SAr]^-$ (Ar = Ph (I), C_6H_4Me-4 (II), $C_6H_3(Me_2$-2,5) (III) (кристаллогидрат на 1,5 молекулы воды) с выходами 92, 93 и 95 % соответственно. По данным РСА, проведенного при 293 К на автоматическом четырехкружном дифрактометре D8 Quest Bruker (двухкоординатный CCD – детектор, Mo K_α-излучение, $\lambda = 0{,}71073$ Å, графитовый монохроматор) кристаллы I ($C_{30}H_{25}O_3PS$, M 496,53 г/моль, сингония моноклинная, группа симметрии P21/n, размер кристалла 0,25 × 0,2 × 0,15 мм), II($C_{31}H_{27}O_3PS$, M 510,56 г/моль, сингония ромбическая, группа симметрии Pna21, размер кристалла 0,48 × 0,18 × 0,12 мм), III ($C_{32}H_{32}O_{4.5}PS$, M 1097,16 г/моль, сингония моноклинная, группа симметрии P2/c, размер кристалла 0,43 × 0,34 × 0,22 мм) включают тетраэдрические катионы (связи P-C 1,797(2)-1,799(2), 1,652(2)-1,999(3), 1,785(8)-1,815(7) Å; углы CPC 109,29(9)°-110,86(9)°, 104,04(13)°-115,14(12)°, 107,1(4)°-113,3(4)° в I, II, III соответствено) и аренсульфонатные анионы (связи S-O 1,4355(18)-1,4446(17), 1,313(3)-1,597(3), 1,431(6)-1,457(7) Å; углы OSO 113,07(11)°-113,30(11)°, 107,5(2)°-117,2(2)°, 112,3(4)°-114,2(4)° в I, II, III соответствено). Молекулы воды в кристаллогидрате III посредством межмолекулярных водородных связей O···H связывают катионы и анионы в пространственную сетку. В сольвате $[Ph_4P]Br\cdot PhH$ (IV) ($C_{30}H_{26}BrP$, M 497,39 г/моль, сингония триклинная, группа симметрии P1, размер кристалла 0,45 × 0,28 × 0,26 мм), полученном взаимодействием пентафенилфосфора с бромоводородной кислотой с выходом 97 %, связи P-C (1,7941(19)-1,803(2) Å) и углы CPC (107,93(9)°-112,96(9)°) близки к аналогичным значениям в аренсульфонатах тетрафенилфосфония.

Ключевые слова: пентафенилфосфор, аренсульфоновые кислоты, аренсульфонаты тетрафенилфосфония, рентгеноструктурный анализ

SYNTHESIS AND STRUCTURE OF TETRAPHENYLPHOSPHONIUM ARENESULFONATES

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Interaction between pentaphenylphosphorus and arenesulfonic acids (mole ratio 1:1) in the benzene solution of tetraphenylphosphonium arenesulfonates has led to $[Ph_4P]^+[O_3SAr]^-$, Ar = Ph (I), C_6H_4Me-4 (II), $C_6H_3(Me_2$-2,5) (III) (hydrate with 1.5 molecules of water). According to X-ray analysis, which was performed in the automatic four-circle Bruker D8 Quest diffractometer (Mo K_{α}-radiation, λ = 0.71073 Å, graphite monochromator), crystals I ($C_{30}H_{25}O_3PS$, M 496.53 g/mol, crystal system moloclinic, space group P21/n, crystal size 0.25 × 0.2 × 0.15 mm), II($C_{31}H_{27}O_3PS$, M 510.56 g/mol, crystal system rhombic, space group Pna21, crystal size 0.48 × 0.18 × 0.12 mm) and III ($C_{32}H_{32}O_{4.5}PS$, M 1097.16 g/mol, crystal system moloclinic, space group P2/c, crystal size 0.43 × 0.34 × 0.22 mm) include the tetrahedral cations (bond length equal 1.797(2)-1.799(2), 1.652(2)-1.999(3), 1.785(8)-1.815(7) Å; angles CPC equal 109.29(9)°-110.86(9)°, 104.04(13)°-115.14(12)°, 107.1(4)°-113.3(4)° in I, II and III respectively) and arenesulfonate anions (bond length equal S-O 1.4355(18)-1.4446(17), 1.313(3)-1.597(3), 1.431(6)-1.457(7) Å; angles OSO 113.07(11)°-113.30(11)°, 107.5(2)°-117.2(2)°, 112.3(4)°-114.2(4)° in I, II and III respectively). In hydrate III the water molecules associate the cations and anions into the spatial grid through the intermolecular hydrogen bonds. The solvate [Ph₄P]Br·PhH (IV) has been obtained by the interaction of pentaphenylphosphorus with the hydrogen bromide. In IV the P-C bonds (1.7941(19)-1.803(2) Å) and angles CPC (107.93(9)°-112.96(9)°) are close to the similar values in the tetraphenylphosphonium arenesulfonates.

Key words: pentaphenylphosphorus, arenesulfonic acids, tetraphenylphosphonium arenesulfonates, X-ray analysis

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INTRODUCTION

The methods of obtaining and structural features of pentavalent phosphorus organic compounds are described in a literature [1-10]. The reaction ability of the such compounds is much less studied. For example, the reactions between pentaphenylphosphorus and alcohols, non-metallic oxides and acids lead to elimination of the phenyl ligands from the phosphorus atom [10-12].

Interaction between pentaphenylphosphorus and arenesulfonic acids has been described in the present work, the structure of the products of the reaction has been determined.

EXPERIMENTAL

Synthesis of tetraphenylphosphonium benzenesulfonate (**I**) 100 mg of pentaphenylphosphonium in 20 ml of benzene was added to 38 mg of benzenesulfonic acid hydrate in 20 ml of benzene. Then the mixture was heated at 80 °C for a 10 min. The solution was concentrated to a volume of 1 ml. A 110 mg of colourless cryslals were obtained, m.p. 205 °C, IR-spectrum (*ν*, cm^{-1}): 3472, 3063, 1482, 1436, 1238, 1215, 1196, 1141, 1107, 997, 760, 723, 689, 610, 527.

Found, %: C 72.29; H 5.08.

For $C_{30}H_{25}O_3PS$

Calculated, %: C 72.58; H 5.04.

Complex **II**−**IV** was obtained in the same way of **I**

II: The yield of the colourless crystals 93 %, *m.p* = 240 °C. IR-spectrum (*ν*, cm^{-1}): 3474, 2924, 1585, 1483, 1441, 1248, 150, 1141, 1107, 1055, 997, 829, 758, 723, 692, 528.

Found, %: C 72.68; H 5.36.

For $C_{31}H_{27}O_3PS$

Calculated, %: C 72.94; H 5.29.

III: The yield of the colourless crystals 95 %, *m.p* = 170 °C. IR-spectrum (*ν*, cm^{-1}): 3479, 1484, 1437, 1259, 1227, 1188, 1107, 1020, 995, 823,776,721, 691, 627, 528.

Found %: C 69.69; H 6.06.

For $C_{32}H_{32}O_{4.5}PS$

Calculated, %: C 69.66; H 5.86.

IV: The yield of the colourless crystals 97 %, *m.p* = 178 °C. IR-spectrum (*ν*, cm^{-1}): 3502, 3473, 3427, 3277, 3082, 3061, 2692, 2206, 1984, 1913, 1836, 1788, 1653, 1598, 1585, 1571, 1527, 1483, 1436, 1365, 1350, 1315, 1255, 1184, 1166, 1107, 1074, 1028, 997, 954, 937, 858, 819, 781, 759, 723, 692, 646, 615, 582, 528, 455, 412.

Found, %: C 72.35; H 7.54.

For $C_{30}H_{26}BrP$

Calculated, %: C 72.43; H 7.22.

IR spectra were recorded in KBr pellets on the IR-spectrometer Shimadzu IRAffinity-1S in the area of 4000-400 cm^{-1}.

The X-ray diffraction analyses of crystal **I** was performed on the Bruker D8 Quest diffractometer (Mo K_α-radiation, λ = 0.71073 Å, graphite monochromator). The data collection and editing as well as the refinement of unit cell parameters and the absorption accounting were carried out using SMART and SAINT Plus program packages [13]. All calculations for the structure determination and refinement were carried out using the SHELXTL/PC [14] and OLEX2 [15] programs packages. The structures were determined by the direct method and refined by least-squares method calculations in anisotropic approximation for non-hydrogen atoms. Selected crystallographic data and structure refinement results for compounds I and II are given in Table 1, and selected bond lengths and bond angles are listed in Table 2.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (N 1822534 (I), N 1822533 (II), № 1822532 (III), N 1812206 (IV)); deposit@ccdc.cam.ac.uk;http://www.ccdc.cam.ac.uk).

Table 1

Crystallographic data, the experimental parameters and structure refinement parameters for compound I−IV

Таблица 1. **Кристаллографические данные, параметры эксперимента и уточнения структур I–IV**

Параметр	Value			
	I	**II**	**III**	**IV**
M	496.53	510.56	1097.16	497.39
Crystal system	Monoclinic	Rhombic	Monoclinic	Triclinic
Space group	$P2_1/n$	$Pna2_1$	$P2/c$	$P\bar{1}$
a, Å	10.917(9)	13.078(7)	13.556(9)	10.074(7)
b, Å	14.001(10)	13.672(8)	22.109(14)	10.375(7)
c, Å	16.843(12)	14.805(9)	18.763(12)	13.077(8)
α, deg.	90.00	90.00	90.00	72.02(3)
β, deg.	107.17(3)	90.00	99.31(4)	74.09(3)
γ, deg.	90.00	90.00	90.00	86.56(4)
V, $Å^3$	2460(3)	2647(3)	5550(6)	1249.9(14)
Z	4	4	4	2
$\rho_{(calc.)}$, g/cm^3	1.341	1.281	1.313	1.322
μ, mm^{-1}	0.228	0.213	0.213	1.724
F(000)	1040.0	1072.0	2304.0	512.0
Crystal size, mm	0.25 × 0.2 × 0.15	0.48 × 0.18 × 0.12	0.43 × 0.34 × 0.22	0.45 × 0.28 × 0.26
2θ Range of data collection, deg	6 – 45.02	5.5 – 53.44	5.74 – 47.24	6.08 – 62.04
Range of refraction indices	$-11 \leq h \leq 11$, $-15 \leq k \leq 15$, $-18 \leq l \leq 18$	$-16 \leq h \leq 16$, $-15 \leq k \leq 15$, $-14 \leq l \leq 14$	$-15 \leq h \leq 15$, $-24 \leq k \leq 24$, $-21 \leq l \leq 20$	$-14 \leq h \leq 14$, $-15 \leq k \leq 15$, $-18 \leq l \leq 18$
Measured reflections	22329	28048	58452	77260
Independent reflections	3197 (R_{int} = 0.0236)	4083 (R_{int} = 0.0334)	8257 (R_{int} = 0.1016)	7937 (R_{int} = 0.0295)
Refinement variables	316	326	712	290
GOOF	1.068	1.089	0.987	1.045
R factors for $F^2 > 2\sigma(F^2)$	R_1 = 0.0320, wR_2 = 0.0805	R_1 = 0.0392, wR_2 = 0.0996	R_1 = 0.0507, wR_2 = 0.1374	R_1 = 0.0359, wR_2 = 0.0986
R factors for all reflections	R_1 = 0.0373, wR_2 = 0.0846	R_1 = 0.0427, wR_2 = 0.1025	R_1 = 0.1357, wR_2 = 0.1941	R_1 = 0.0558, wR_2 = 0.1083
Residual electron density (min/max), $e/Å^3$	0.17/−0.32	0.26/−0.36	0.46/−0.42	0.59/−0.52

Table 2

Main bond lengths (d) and valency angles in the structure of compounds I–IV

***Таблица 2.* Основные длины связей (d) и валентные углы в структурах I–IV**

Bond	*d*, Å	Angle	ω, °
I			
S(1)–O(1)	1.4355(19)	O(1)S(1)O(2)	113.30(11)
S(1)–O(2)	1.4446(17)	O(1)S(1)O(3)	113.17(11)
S(1)–O(3)	1.4441(18)	O(1)S(1)C(41)	106.27(11)
S(1)–C(41)	1.785(2)	O(2)S(1)C(41)	104.60(11)
P(1)–C(1)	1.797(2)	O(3)S(1)O(2)	113.07(11)
P(1)–C(11)	1.801(2)	O(3)S(1)C(41)	105.47(11)
P(1)–C(21)	1.797(2)	C(21)P(1)C31	108.29(9)
P(1)–C(31)	1.799(2)	C(1)P(1)C31	110.86(9)
II			
P(1)–C(11)	1.999(3)	C(21)P(1)C(1)	104.04(13)
P(1)–C(1)	1.844(3)	C(21)P(1)C(11)	115.14(12)
P(1)–C(31)	1.652(2)	O(1)S(1)C(41)	105.72(14)
P(1)–C(21)	1.710(3)	O(1)S(1)O(3)	117.2(2)
S(1)–O(1)	1.411(3)	O(2)S(1)O(1)	107.5(2)
S(1)–O(2)	1.313(3)	O(2)S(1)C(41)	98.71(18)
S(1)–C(41)	1.785(3)	O(2)S(1)O(3)	116.0(2)
S(1)–O(3)	1.597(3)	O(3)S(1)C(41)	109.70(15)
III			
S(1)–O(1)	1.457(7)	O(1)S(1)C(81)	105.6(4)
S(1)–O(2)	1.443(6)	O(2)S(1)O(1)	111.8(4)
S(1)–O(3)	1.443(6)	O(2)S(1)C(81)	105.0(4)
S(1)–C(81)	1.785(9)	O(3)S(1)O(1)	112.8(4)
S(2)–O(4)	1.441(6)	O(3)S(1)O(2)	114.2(4)
S(2)–O(5)	1.431(6)	O(3)S(1)C(81)	106.6(4)
S(2)–O(6)	1.447(6)	O(4)S(2)O(6)	113.3(4)
S(2)–C(91)	1.802(8)	O(4)S(2)C(91)	105.7(3)
P(1)–C(21)	1.802(8)	O(5)S(2)O(4)	113.0(4)
P(1)–C(1)	1.806(7)	O(5)S(2)O(6)	112.3(4)
P(1)–C(31)	1.809(7)	O(5)S(2)C(91)	106.0(4)
P(1)–C(11)	1.791(8)	O(6)S(2)C(91)	105.8(4)
P(2)–C(61)	1.815(7)	C(21)P(1)C(1)	113.3(4)
P(2)–C(51)	1.789(7)	C(21)P(1)C(11)	107.1(4)
P(2)–C(41)	1.800(8)	C(51)P(2)C(41)	112.2(4)
P(2)–C(71)	1.785(8)	C(41)P(2)C(61)	107.3(4)
IV			
P(1)–C(1)	1.7984(18)	C(1)P(1)C(11)	108.88(9)
P(1)–C(31)	1.7994(19)	C(31)P(1)C(11)	112.96(9)
P(1)–C(21)	1.7941(19)	C(21)P(1)C(31)	107.93(9)
P(1)–C(11)	1.803(2)	C(21)P(1)C(11)	108.34(9)

RESULTS AND DISCUSSION

It had been found that the reaction of dephenylation between pentaphenylphosphorus and arenesulfonic acids (mole ratio 1:1, benzene) leads to quantitative formation of tetraphenylposphonium arenesulfonates. These compounds are colourless crystals, well soluble in aromatic hydrocarbons, tetrahydrofurane, dioxane and ethanol.

$$Ph_5P + HOSO_2Ar \rightarrow [Ph_4P]^+ [O_3SAr]^- + PhH$$

Ar = Ph (**I**), C_6H_4Me-4 (**II**), $C_6H_3(Me_2$-2,5) (**III**).

Interaction of hydrogen bromide with pentaphenylphosphorus leads to similar results, at that tetraphenylphosphonium bromide benzene solvate is crystallized from the reaction mixture.

$$Ph_5P + HBr \xrightarrow{PhH} [Ph_4P]^+Br^- \cdot PhH + PhH$$

IV

According to X-ray analysis, tetraphenylphosphonium arenesulfonates (**I−III**) and tetraphenylphosphonium bromide (**IV**) are ionic compounds. Tetraphenylphosphonium cations have a slightly distorted tetrahedral coordination (Fig. 1-4).

The valence angles in the cations of complexes **I−III** vary in the ranges 109.29(9)°–110.86(9)° (**I**), 104.04(13)°–115.14(12)° (**II**), 107.1(4)°–113.3(4)° (**III**). The P−C distances equal 1.797(2)–1.799(2), 1.652(2)–1.999(3), 1.785(8)–1.815(7) Å respectively. Their average values practically don't differ from the similar values in cation **IV.**

Two types of crystallographically independent molecules are present in hydrate **III**. The hydrate water molecules take part on the formation of the structure, forming weak hydrogen bonds with the arenesulfates anions, which have short contacts $O\cdots H_{Ph}$ with the cations.

It is known that the IR-spectra of sulfonic acids include the typical absorption band: 1260-1150 cm^{-1} (strong intensity band) and 1080–1010 cm^{-1} (middle intencity band) which are attributed to the asymmetric and symmetric stretching vibrations of sulfonate groups [16, 17].

It has been noted that the first band is splitted, but the position of the band in the range 1080-1010 cm^{-1} depends on the structure of the acid, but little. IR-spectra of tetraphenylphosphonium arenesulfates also include the typical absorption bands of sulfonic groups: 1227-1238 cm^{-1}, 1141-1188 cm^{-1} и ~1000 cm^{-1}. It is obviously that the displacement of the last band toward the lower vibrations involves the lengthening of the S=O bonds and alignment of the three bonds in the SO_3 group unlike SO_4 anion containing only one band [18]. In the arenesulfonate anions the bond length S−O equal 1.436(2), 1.444(2), 1.445(2) Å (**I**); 1.313(3), 1.411(3), 1.597(3) Å (**II**); 1.431(6)-1.457(7) Å (**III**)). It should be noted that the same distribution of the electron density is observed in tetraphenylantimony and tetraphenylbismuth 2,5-dimethylsulfonates, the S−O bond lengths equal 1.383(14), 1.440(11), 1.450(2) и 1.439(3), 1.443(3), 1.457(3) Å respectively [19, 20].

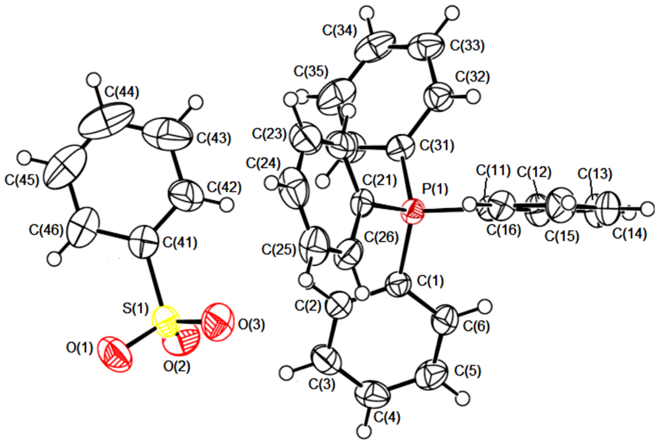

Fig.1. The structure of complex **I**
Рис. 1. Строение комплекса **I**

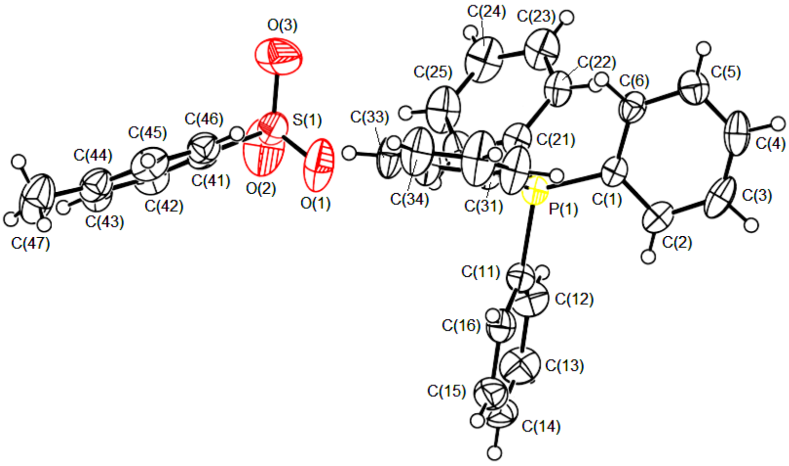

Fig. 2. The structure of complex **II**
Рис. 2. Строение комплекса **II**

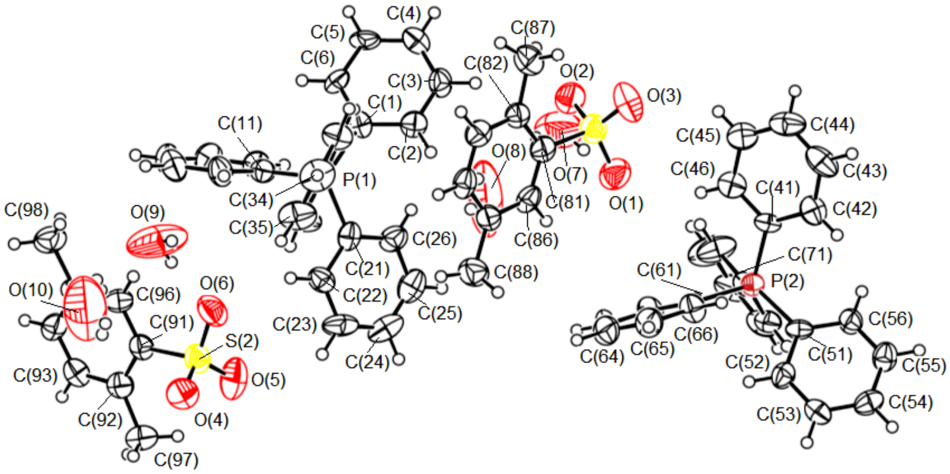

Fig. 3. The structure of hydrate of complex **III**
Рис. 3. Строение гидрата комплекса **III**

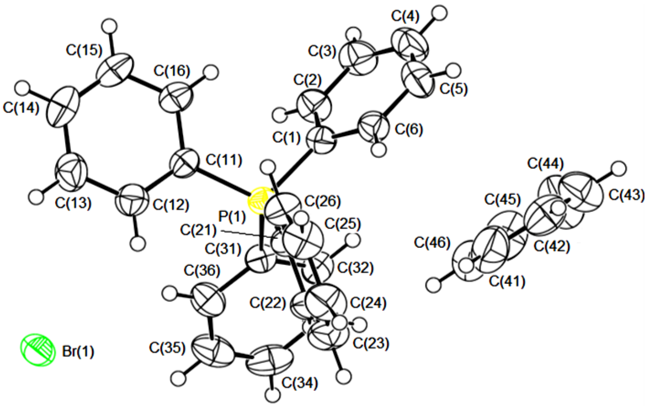

Fig 4. The solvate structure of complex **IV** with benzene
Рис. 4. Строение сольвата комплекса **IV** с бензолом

The reaction between tetraphenylphosphonium arenesulfonates with potassium iodide indicates the ionic nature of the bond. The interaction leads to the instant formation of tetraphenylphosphonium iodide, which is precipitated from the solution in the loose precipitate form.

$$[Ph_4P][O_3SAr'] + KI \rightarrow Ph_4PI + KO_3SAr'$$

CONCLUSION

It has been found that the dephenylation reaction between pentaphenylphosphorus and arenesulfonic acids can be considered as an effective on-stage method of obtaining tetraphenylphosphorus arenesulfonates.

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