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Synthesis and Structures of Organyltriphenylphosphonium Organosulfonates $[Ph_3PR][OSO_2R']$ (R = Ph, R' = $C_6H_3Cl_2$-2,5; R = C_6H_{11}-*cyclo*, R' = $C_6H_3Cl_2$-2,5; R = CH_2OMe, R' = $C_6H_3(NO_2)_2$-2,4; R = CH_2OMe, R' = C_6H_4(COOH-2))

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Abstract—The reactions of equimolar amounts of tetraorganylphosphonium chloride with 2,5-dichlorobenzenesulfonic, 2,4-dinitrobenzenesulfonic, and 2-carboxybenzenesulfonic acids in water afford organyltriphenylphosphonium organosulfonates $[Ph_3PR][OSO_2R']$ (R = Ph, R' = $C_6H_3Cl_2$-2,5 (**I**); R = C_6H_{11}-*cyclo*, R' = $C_6H_3Cl_2$-2,5 (**II**); R = CH_2OMe, R' = $C_6H_3(NO_2)_2$-2,4 (**III**); R = CH_2OMe, R' = C_6H_4(COOH-2) (**IV**)). According to the X-ray diffraction (XRD) data (CIF files CCDC nos. 2142598 (**I**), 2144330 (**II**), 2144708 (**III**), and 2145604 (**IV**)), the complexes are ionic. The phosphorus atoms in the cations of complexes **I**–**IV** are characterized by the tetrahedral coordination, and the organosulfonate anions have the usual geometry with the tetrahedral sulfur atom. The P–C bond lengths are 1.7665(18)–1.836(2) Å, and the CPC bond angles vary in a range of 103.81(6)°–113.43(7)°. The structural organization in the crystals of complexes **I**–**IV** is formed by numerous weak hydrogen bonds between the cations and anions, such as $S{=}O\cdots H{-}C_{Ar}$, $N{=}O\cdots H{-}C_{Ar}$, $C{=}O\cdots H{-}C_{Ar}$, etc. The arenesulfonate anions in complex **I** are structured into dimers by molecules of water of crystallization.

Keywords: tetraorganylphosphonium organosulfonate, synthesis, structure, XRD studies

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INTRODUCTION

Interest in organophosphorus compounds is mainly due to a potential of their use in very diverse fields of practical activities: as insecticides, fungicides, herbicides, plasticizers, ion-exchange resins, and additives to gasolines and lubricants [1]. The phosphorus-containing organic compounds also found use in synthetic organic chemistry, for instance, in the Wittig synthesis of olefins [2] or synthesis of organoelement compounds [3]. Prospects of the practical use of some organophosphorus compounds as catalysts of the hydrofunctionalization of unsaturated substrates [4] and reagents for the *trans*-metallation [5] and metathesis of σ bonds [6] were also reported. Among all pentavalent phosphorus derivatives, the tetraphenylphosphonium salts are studied in most detail. They are usually synthesized via oxidative addition from triphenylphosphine and haloarene in the presence of aluminum chloride [1] or by the reaction of pentaphenylphosphorus with the acids [7–10]. The synthesis of other structurally characterized tetraphenylphosphonium organosulfonates was described [11–17], but examples for the synthesis of organyltriphenylphosphonium arenesulfonates are unknown.

In this work, organyltriphenylphosphonium arenesulfonates $[Ph_3PR][OSO_2R']$ (R = Ph, R' = $C_6H_3Cl_2$-2,5 (**I**); R = C_6H_{11}-*cyclo*, R' = $C_6H_3Cl_2$-2,5 (**II**); R = CH_2OMe, R' = $C_6H_3(NO_2)_2$-2,4 (**III**); R = CH_2OMe, R' = C_6H_4(COOH-2) (**IV**)) were synthesized and structurally characterized for the first time.

EXPERIMENTAL

Organyltriphenylphosphonium chlorides and arenesulfonic acids (Alfa Aesar) were used.

Tetraphenylphosphonium 2,5-dichlorobenzenesulfonate hydrate $[Ph_4P][OSO_2C_6H_3Cl_2\text{-}2,5]\cdot 2H_2O$ (I) was synthesized from equimolar amounts of tetraphenylphosphonium chloride and 2,5-dichlorobenzenesulfonic acid in water. Colorless crystals with T_m = 168°C (94%) were obtained after the recrystallization of the target product from water. IR (ν, cm^{-1}): 3549, 3483, 3063, 1782, 1651, 1585, 1483, 1439, 1375, 1317, 1217, 1165, 1148, 1107, 1065, 1016, 995, 893, 827, 814, 768, 723, 692, 621, 528, 434.

For $C_{30}H_{27}O_5PSCl_2$		
Anal. calcd., %	C, 59.01	H, 4.52
Found, %	C, 58.77	H, 4.56

Cyclohexyltriphenylphosphonium 2,5-dichlorobenzenesulfonate $[Ph_3PC_6H_{11}$-*cyclo*$][OSO_2C_6H_3Cl_2$-2,5] (II) is presented by colorless crystals with T_m = 195°C (95%). IR (ν, cm^{-1}): 3078, 3042, 2930, 2884, 2853, 1585, 1485, 1441, 1371, 1346, 1327, 1238, 1219, 1165, 1146, 1107, 1090, 1061, 1015, 997, 901, 885, 853, 829, 804, 743, 721, 692, 621, 528, 513, 471, 417.

For $C_{30}H_{29}O_3PSCl_2$		
Anal. calcd., %	C, 63.05	H, 5.11
Found, %	C, 62.86	H, 5.14

Methoxymethyltriphenylphosphonium 2,4-dinitrobenzenesulfonate $[Ph_3PCH_2OMe][OSO_2C_6H_3(NO_2)_2$-2,4] (III) represent colorless crystals with T_m = 145°C (97%). IR (ν, cm^{-1}): 3088, 3019, 2893, 2833, 1600, 1587, 1545, 1528, 1485, 1437, 1352, 1236, 1165, 1130, 1115, 1097, 1063, 1028, 997, 943, 903, 851, 833, 797, 746, 727, 691, 635, 557, 532, 507, 494, 467, 447.

For $C_{26}H_{23}N_2O_8PS$		
Anal. calcd., %	C, 56.32	H, 4.18
Found, %	C, 56.17	H, 4.36

Methoxymethyltriphenylphosphonium 2-carboxybenzenesulfonate $[Ph_3PCH_2OMe][OSO_2C_6H_4$(COOH-2)] (IV) represent colorless crystals with T_m = 134°C (97%). IR (ν, cm^{-1}): 3460, 3065, 3024, 2934, 2833, 1715, 1589, 1566, 1487, 1439, 1292, 1248, 1171, 1140, 1115, 1094, 1020, 997, 872, 798, 760, 719, 691, 619, 569, 528, 503, 446.

For $C_{27}H_{25}O_6PS$		
Anal. calcd., %	C, 63.77	H, 4.95
Found, %	C, 63.63	H, 4.99

IR spectra were recorded on a Shimadzu FT-IR spectrometer in KBr pellets. Melting points were measured on a Netzsch 449C Jupiter simultaneous thermal analyzer. Elemental analysis was carried out on a Euro EA3028-HT analyzer. XRD was conducted on a D8 QUEST diffractometer (Bruker) at 293 K. Data collection and structure determination and refinement were performed using the available software [18–20].

The crystallographic data and structure refinement results are given in Table 1. The bond lengths and bond angles are listed in Table 2.

The full tables of atomic coordinates, bond lengths, and bond angles for the structures studied were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2142598 (**I**), 2144330 (**II**), 2144708 (**III**), and 2145604 (**IV**); deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Complexes **I**–**IV** were synthesized by mixing aqueous solutions of organyltriphenylphosphonium chlorides and the corresponding arenesulfonic acid. The slow evaporation of water resulted in the formation of stable in air transparent crystals highly soluble in chloroform, arenes, and dichloromethane and poorly soluble in water and acetonitrile at room temperature.

$$[Ph_3PR]Cl + HOSO_2R' \rightarrow [Ph_3PR][OSO_2R'] + HCl,$$

$$R = Ph,\ R' = C_6H_3Cl_2\text{-}2,5\ (\mathbf{I});\quad R = C_6H_{11}\text{-}cyclo,\ \ R' = C_6H_3Cl_2\text{-}2,5\ (\mathbf{II});$$

$$R = CH_2OMe,\ \ R' = C_6H_3(NO_2)_2\text{-}2,4\ (\mathbf{III});\quad R = CH_2OMe,\ \ R' = C_6H_4(COOH\text{-}2)\ (\mathbf{IV}).$$

Note that complex **I** was also synthesized from pentaphenylphosphorus and 2,5-dichlorobenzenesulfonic acid in benzene and was isolated after recrystallization from water as hydrate in a yield of 93%.

$$Ph_5P + HOSO_2C_6H_3Cl_2\text{-}2,5$$
$$\rightarrow [Ph_4P][OSO_2C_6H_3Cl_2\text{-}2,5] + PhH.$$

The stretching vibration bands of the carbon skeleton of the aromatic fragments appear in the IR spectra of compounds **I**–**IV** in a range of 1600–1483 cm^{-1}. The medium-intensity absorption bands corresponding to stretching and bending vibrations of the C_{Ar}–H bonds are observed at 3088–3019 and 1165–1130 cm^{-1}, and out-of-plane bending vibrations are characterized by the bands at 872–797 cm^{-1}. The absorption bands at 1248–1217 and 1116–1090 cm^{-1} are attributed to asymmetric and symmetric stretching vibrations of the sulfonate group SO_3. The intense absorption bands at 692–691 and 532–528 cm^{-1} can be assigned to stretching vibrations of the S–O and C–S groups. The stretching and bending vibrations of the C_{Ar}–P bonds are characterized by absorption bands at 1441–1437 and 1028–995 cm^{-1}, respectively [21].

The high-intensity bands at 723 and 721 cm^{-1} correspond to vibrations of the C_{Ar}–Cl bonds in the spectra of compounds **I** and **II**. In the IR spectra of complex **III**, the intense absorption bands at 1545, 1528,

Table 1. Crystallographic data and experimental and structure refinement parameters for compounds **I–IV**

Parameter	Value			
	I	**II**	**III**	**IV**
Formula	$C_{30}H_{27}O_5SCl_2P$	$C_{30}H_{29}O_3SCl_2P$	$C_{26}H_{23}N_2O_8SP$	$C_{27}H_{25}O_6SP$
FW	601.45	571.46	554.49	508.50
Crystal system	Triclinic	Monoclinic	Triclinic	Triclinic
Space group	$P\bar{1}$	*Cc*	$P\bar{1}$	$P\bar{1}$
a, Å	10.998(5)	9.089(4)	9.437(6)	8.263(11)
b, Å	11.358(6)	17.082(7)	11.424(10)	12.085(17)
c, Å	12.923(10)	17.647(7)	13.685(12)	12.987(14)
α, deg	85.54(3)	90.00	65.34(3)	84.17(5)
β, deg	67.47(2)	97.22(2)	84.66(3)	86.78(4)
γ, deg	76.81(2)	90.00	72.68(3)	83.51(6)
V, Å^3	1451.5(15)	2718.0(19)	1279.3(17)	1281(3)
Z	2	4	2	2
ρ_{calc}, g/cm^3	1.376	1.397	1.439	1.319
μ, mm^{-1}	0.389	0.406	0.243	0.229
F(000)	624.0	1192.0	576.0	532.0
Crystal size, mm	0.4 × 0.25 × 0.12	0.5 × 0.3 × 0.19	0.32 × 0.23 × 0.17	0.43 × 0.35 × 0.17
Range of data collection over θ, deg	6.14–56.7	5.84–66.34	6.14–72.92	6–57
Ranges of reflection indices	$-14 \le h \le 14$, $-15 \le k \le 15$, $-17 \le l \le 17$	$-13 \le h \le 13$, $-26 \le k \le 26$, $-27 \le l \le 26$	$-15 \le h \le 15$, $-18 \le k \le 18$, $-22 \le l \le 22$	$-11 \le h \le 11$, $-16 \le k \le 16$, $-17 \le l \le 17$
Measured reflections	49272	49456	76725	52567
Independent reflections	7189	10159	11319	6475
R_{int}	0.0393	0.0241	0.0395	0.0423
Refinement variables	358	334	344	339
GOOF	1.034	1.037	1.033	1.024
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0409$, $wR_2 = 0.1015$	$R_1 = 0.0317$, $wR_2 = 0.0773$	$R_1 = 0.0521$, $wR_2 = 0.1302$	$R_1 = 0.0414$, $wR_2 = 0.0999$
R factors for all reflections	$R_1 = 0.0592$, $wR_2 = 0.1116$	$R_1 = 0.0413$, $wR_2 = 0.0814$	$R_1 = 0.0856$, $wR_2 = 0.1455$	$R_1 = 0.0623$, $wR_2 = 0.1104$
Residual electron density (min/max), e/A^3	0.47/–0.65	0.22/–0.27	0.37/–0.62	0.27/–0.30

and 1352 cm^{-1} correspond to asymmetric and symmetric vibrations of the NO_2– group. The IR spectrum of compound **III** contains (at 2833 cm^{-1}) an absorption band corresponding to stretching vibrations of the Ar–O–CH_3 bonds. The presence of water molecules in the crystal of tetraphenylphosphonium 2,5-dichlorobenzenesulfonate hydrate is confirmed by the bands at 3549 and 3483 cm^{-1} in the IR spectrum. The IR spectrum of complex **IV** exhibits an intense stretching vibration band of the carbonyl group (at 1715 cm^{-1}) and a broad band of stretching vibrations of the hydroxy group (3460 cm^{-1}) [22] indicating the presence of the free carboxy group.

According to the XRD data, the crystal of compound **I** consists of tetraphenylphosphonium cations (P–C 1.7981(19)–1.8036(19) Å, CPC 107.44(9)°–112.23(9)°), 2,5-dichlorobenzenesulfonate anions (S–O 1.4498(18)–1.4535(18) Å, OSO 112.83(12)°–113.78(11)°), and water molecules (Fig. 1).

The water molecules link two 2,5-dichlorobenzenesulfonate anions into a dimer by hydrogen bonds (H···O 2.07–2.17 Å). In addition, intramolecular contacts are observed between the *ortho*-chlorine atom and one of the oxygen atoms of the sulfonate group (Cl···O 3.193(5) Å) (Fig. 2).

Similar structuring of arenesulfonate anions by water molecules was reported [23, 24].

The XRD data for compounds **II–IV** indicate that their crystals contain alkyltriphenylphosphonium cations and arenesulfonate anions (Figs. 3–5).

Table 2. Bond lengths and bond angles in the structures of compounds **I**–**IV**

Bond	*d*, Å	Angle	ω, deg
		I	
S(1)–O(1)	1.4498(18)	O(1)S(1)O(2)	112.92(12)
S(1)–O(2)	1.4499(18)	O(1)S(1)O(3)	113.78(11)
S(1)–O(3)	1.4535(18)	O(2)S(1)O(3)	112.83(12)
S(1)–C(41)	1.7852(19)	O(1)S(1)C(41)	106.54(9)
P(1)–C(1)	1.8036(19)	C(1)P(1)C(11)	107.44(9)
P(1)–C(11)	1.7981(19)	C(11)P(1)C(21)	108.72(8)
P(1)–C(21)	1.7984(19)	C(21)P(1)C(31)	107.53(9)
P(1)–C(31)	1.7999(19)	C(1)P(1)C(31)	110.24(8)
Cl(1)–C(42)	1.735(2)	Cl(1)C(42)C(43)	117.98(14)
Cl(2)–C(45)	1.738(2)	Cl(2)C(45)C(46)	118.59(16)
		II	
S(1)–O(1)	1.4497(14)	O(1)S(1)O(2)	113.32(9)
S(1)–O(2)	1.4502(15)	O(1)S(1)O(3)	113.43(10)
S(1)–O(3)	1.4365(13)	O(2)S(1)O(3)	114.84(10)
S(1)–C(41)	1.8063(16)	O(1)S(1)C(41)	104.53(7)
P(1)–C(1)	1.7897(13)	C(1)P(1)C(11)	110.56(5)
P(1)–C(11)	1.7972(13)	C(11)P(1)C(21)	104.95(5)
P(1)–C(21)	1.7984(13)	C(21)P(1)C(31)	111.14(6)
P(1)–C(31)	1.8173(13)	C(1)P(1)C(31)	109.81(6)
Cl(1)–C(42)	1.7386(15)	Cl(1)C(42)C(43)	116.31(12)
Cl(2)–C(45)	1.7391(14)	Cl(2)C(45)C(46)	118.81(12)
		III	
S(1)–O(2)	1.4620(16)	O(2)S(1)O(3)	111.67(10)
S(1)–O(3)	1.3968(18)	O(2)S(1)O(4)	115.86(10)
S(1)–O(4)	1.4146(17)	O(3)S(1)O(4)	113.03(11)
S(1)–C(31)	1.8422(18)	O(2)S(1)C(31)	107.06(9)
P(1)–C(1)	1.8085(17)	C(1)P(1)C(7)	111.80(8)
P(1)–C(7)	1.8081(18)	C(7)P(1)C(11)	103.81(6)
P(1)–C(11)	1.7875(16)	C(11)P(1)C(21)	111.47(7)
P(1)–C(21)	1.7665(18)	C(1)P(1)C(21)	105.91(8)
N(1)–O(5)	1.252(3)	O(5)N(1)O(6)	128.42(19)
N(1)–O(6)	1.224(2)	O(7)N(2)O(8)	123.3(2)
N(2)–O(7)	1.202(3)	C(7)O(1)C(8)	108.73(13)
N(2)–O(8)	1.211(4)	P(1)C(7)O(1)	104.14(9)
O(1)–C(7)	1.3975(18)		
		IV	
S(1)–O(2)	1.465(2)	O(2)S(1)O(3)	111.65(12)
S(1)–O(3)	1.440(2)	O(2)S(1)O(4)	112.50(12)
S(1)–O(4)	1.464(2)	O(3)S(1)O(4)	114.63(11)
S(1)–C(31)	1.791(2)	O(2)S(1)C(31)	104.47(11)
P(1)–C(1)	1.793(2)	C(1)P(1)C(7)	107.96(11)
P(1)–C(7)	1.836(2)	C(7)P(1)C(11)	112.08(11)
P(1)–C(11)	1.807(3)	C(11)P(1)C(21)	108.91(12)
P(1)–C(21)	1.799(2)	C(1)P(1)C(21)	112.67(11)
O(1)–C(7)	1.409(2)	C(7)O(1)C(8)	114.92(18)
O(5A)–C(37)	1.184(9)	O(5A)C(37)O(6A)	115.4(6)
O(5B)–C(37)	1.190(6)	O(5B)C(37)O(6B)	121.2(3)
O(6A)–C(37)	1.371(5)	C(32)C(37)O(5A)	124.1(5)
O(6B)–C(37)	1.347(4)	C(32)C(37)O(6A)	118.6(3)

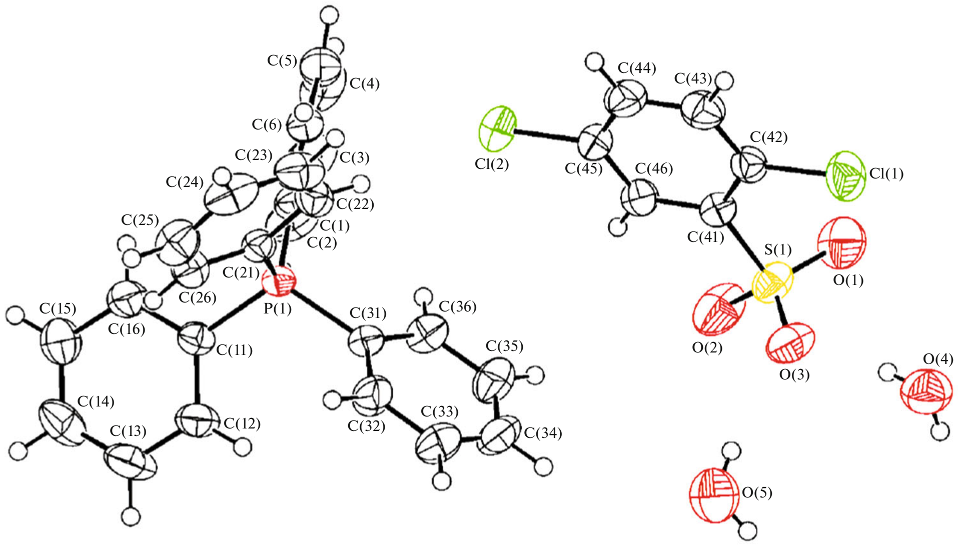

Fig. 1. General view of compound **I**.

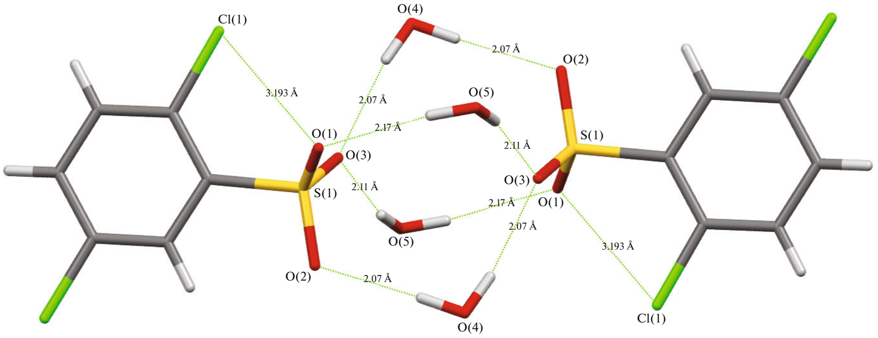

Fig. 2. Dimeric organization of the arenesulfonate anions in the crystal of compound **I**.

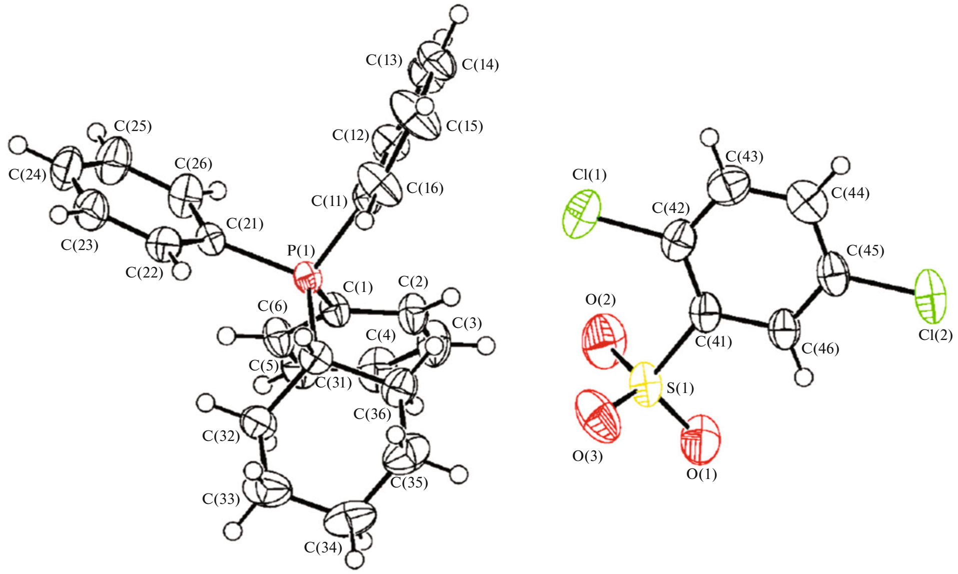

Fig. 3. General view of compound **II**.

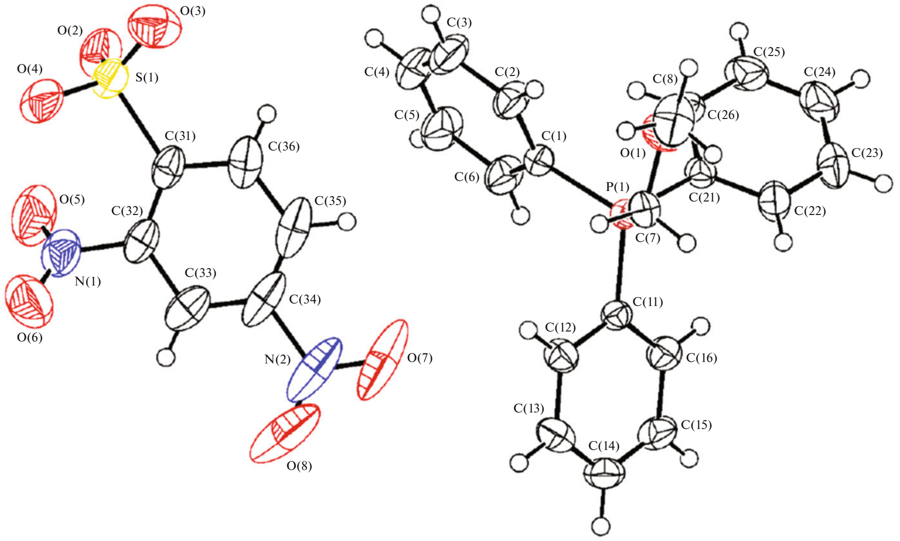

Fig. 4. General view of compound **III**.

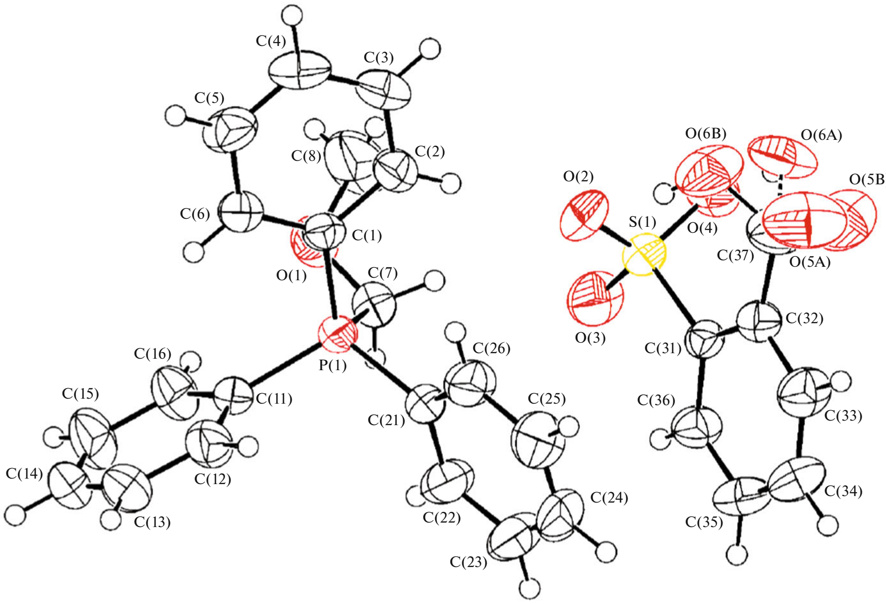

Fig. 5. General view of compound **IV**.

The phosphorus atoms in the cations of compounds **II**, **III**, and **IV** have a tetrahedral coordination (CPC angles lie in ranges of 104.95(5)°–111.14(6)° for **II**, 103.81(6)°–113.43(7)° for **III**, and 105.90(12)°–112.67(11)° for **IV**). The P–C bond lengths are 1.7897(13)–1.8173(13), 1.7665(18)– 1.8085(17), and 1.793(2)–1.836(2) Å, respectively, which is somewhat less than the sum of their covalent radii (1.88 Å [25]). The S–O distances in the arenesulfonate anions differ insignificantly (1.4365(13)– 1.4502(15), 1.3968(18)–1.4620(16), and 1.440(2)– 1.465(2) Å). However, in the first approximation we can speak about the uniform electron density distribution in the SO_3 groups. The S–C bond lengths are 1.8063(16), 1.8422(18), and 1.791(2) Å, which is comparable with a similar value in compound **I** (1.791(2) Å).

The structural organization of crystals of compounds **I**–**IV** is formed due to numerous weak hydrogen bonds between the cations and anions, for example, $S=O\cdots H-C_{Ar}$, $N=O\cdots H-C_{Ar}$, $C=O\cdots H-C_{Ar}$, etc.

The substitution reactions of available organyltriphenylphosphonium chlorides with arenesulfonic acids in water were found to be an efficient method for the synthesis of organyltriphenylphosphonium arenesulfonates and can be considered as a method for their synthesis alternative to the synthesis from pentaorganylphosphorus and sulfonic acids.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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