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Crystal structure of tetraphenylphosphonium diaquaoxonium dichloride dichloromethane solvate, $[P(C_6H_5)_4H_7O_3]Cl_2 \cdot CH_2Cl_2$

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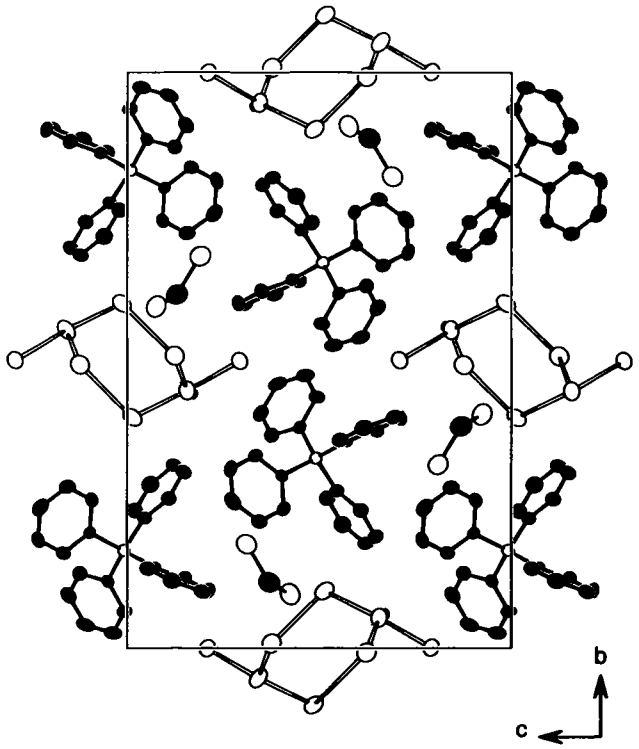

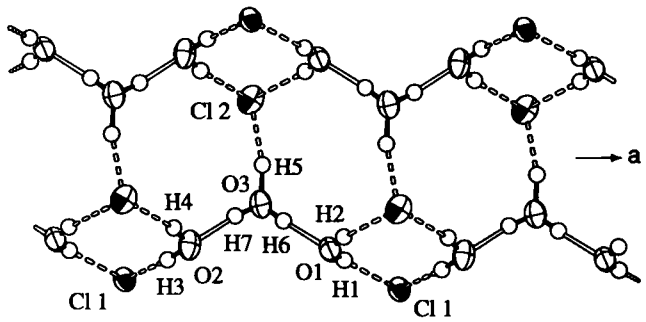

Abstract

$C_{25}H_{29}Cl_4O_3P$, monoclinic, $P12_1/c1$ (No. 14), $a = 7.737(2)$ Å, $b = 22.899(5)$ Å, $c = 15.348(4)$ Å, $\beta = 90.97(5)°$, $V = 2718.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.043$, $wR_{ref}(F^2) = 0.102$, $T = 193$ K.

Source of material

Crystals were obtained as a minor product of a reaction of chlorine with insufficiently dried indium selenide and tetraphenylphosphonium chloride in dichloromethane and subsequent crystallization at 253 K.

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Discussion

As in many compounds with this kind of cations, the $P(C_6H_5)_4^+$ ions are stacked to columns parallel to a [1]. The columns have a pseudo-hexagonal arrangement similar to the one in the structure of $[P(C_6H_5)_4]_2[Mo_2(S_2)_2Cl_8]$ [2]. All O atoms and Cl^- ions are connected via hydrogen bonds within double strands of $H_7O_3^+$ and Cl^- ions. The structure is not isotypic with that of $P(C_6H_5^+)_4H_5O_2^+2Cl^- \cdot H_2O$ [3]. The $H_7O_3^+$ ions are comparable to those in other compounds with this ion [e.g. 4—7]. The hydrogen bonded distances d(O···O) are 2.43 Å and 2.50 Å, d(O···Cl$^-$) are in the range from 2.99 Å to 3.09 Å.

Table 1. Data collection and handling.

Crystal:	colourless needle, size 0.19 × 0.27 × 0.47 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	5.19 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	43.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2863, 2863
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1905
$N(param)_{refined}$:	326
Program:	SHELXL-97 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(12)	4e	−0.3132	0.2834	0.5216	0.041
H(13)	4e	−0.4604	0.2070	0.4560	0.047
H(14)	4e	−0.3298	0.1517	0.3527	0.049
H(15)	4e	−0.0530	0.1715	0.3106	0.045
H(16)	4e	0.0962	0.2486	0.3737	0.038
H(22)	4e	−0.1854	0.3354	0.6530	0.041
H(23)	4e	−0.3952	0.3990	0.7016	0.046
H(24)	4e	−0.4502	0.4847	0.6267	0.045
H(25)	4e	−0.2894	0.5088	0.5078	0.044
H(26)	4e	−0.0774	0.4461	0.4577	0.034
H(32)	4e	0.1792	0.2188	0.5453	0.035
H(33)	4e	0.3654	0.1824	0.6518	0.045
H(34)	4e	0.4450	0.2403	0.7690	0.048
H(35)	4e	0.3479	0.3350	0.7787	0.042
H(36)	4e	0.1707	0.3733	0.6691	0.037
H(42)	4e	−0.0064	0.3784	0.3391	0.038
H(43)	4e	0.1775	0.4136	0.2356	0.043
H(44)	4e	0.4725	0.4161	0.2632	0.042
H(45)	4e	0.5813	0.3843	0.3943	0.044
H(46)	4e	0.3996	0.3494	0.5006	0.040
H(8)	4e	0.8727	0.4161	0.1696	0.077

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(9)	4e	0.9266	0.3793	0.0881	0.077
H(1)	4e	0.587(8)	−0.043(3)	0.688(5)	0.06(3)
H(2)	4e	0.584(8)	−0.058(3)	0.616(5)	0.06(3)
H(3)	4e	−0.051(6)	−0.038(2)	0.707(4)	0.07(2)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(4)	4e	−0.034(6)	−0.056(2)	0.617(4)	0.05(2)
H(5)	4e	0.265(7)	0.036(3)	0.571(5)	0.09(2)
H(6)	4e	0.37(1)	−0.021(4)	0.632(6)	0.12(3)
H(7)	4e	0.18(1)	−0.017(3)	0.624(6)	0.11(3)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	4e	0.7920(1)	−0.00069(5)	0.79169(8)	0.0386(7)	0.0470(7)	0.0393(8)	−0.0032(5)	0.0041(5)	−0.0053(6)
Cl(2)	4e	0.2190(2)	0.09901(6)	0.48645(9)	0.0531(7)	0.0489(8)	0.056(1)	0.0102(6)	−0.0038(6)	−0.0148(7)
O(1)	4e	0.5368(5)	−0.0531(2)	0.6555(4)	0.037(2)	0.057(3)	0.040(3)	−0.008(2)	0.002(2)	−0.001(2)
O(2)	4e	0.0256(5)	−0.0522(2)	0.6583(3)	0.038(2)	0.058(3)	0.053(3)	0.005(2)	0.003(2)	−0.021(2)
O(3)	4e	0.2746(5)	0.0077(2)	0.6234(3)	0.039(2)	0.056(3)	0.056(3)	0.003(2)	0.009(2)	−0.004(2)
P	4e	0.0324(1)	0.33011(4)	0.50856(7)	0.0245(6)	0.0216(6)	0.0219(7)	0.0023(4)	0.0014(4)	−0.0003(5)
C(11)	4e	−0.0901(5)	0.2736(2)	0.4549(3)	0.024(2)	0.025(2)	0.030(3)	0.003(2)	0.001(2)	−0.002(2)
C(12)	4e	−0.2599(5)	0.2610(2)	0.4792(3)	0.030(3)	0.036(3)	0.037(3)	0.005(2)	0.002(2)	−0.003(2)
C(13)	4e	−0.3476(5)	0.2154(2)	0.4401(3)	0.029(2)	0.043(3)	0.045(3)	−0.009(2)	−0.005(2)	0.002(3)
C(14)	4e	−0.2698(6)	0.1826(2)	0.3783(3)	0.055(3)	0.027(3)	0.040(4)	−0.009(2)	−0.012(2)	−0.003(2)
C(15)	4e	−0.1048(6)	0.1942(2)	0.3530(3)	0.044(3)	0.032(3)	0.038(3)	0.001(2)	0.002(2)	−0.007(2)
C(16)	4e	−0.0155(5)	0.2402(2)	0.3913(3)	0.029(2)	0.031(3)	0.037(3)	−0.004(2)	0.006(2)	0.000(2)
C(21)	4e	−0.1117(4)	0.3837(2)	0.5502(3)	0.022(2)	0.025(3)	0.022(3)	0.000(2)	0.002(2)	−0.004(2)
C(22)	4e	−0.2064(5)	0.3702(2)	0.6233(3)	0.037(3)	0.033(3)	0.031(3)	0.005(2)	0.004(2)	−0.001(2)
C(23)	4e	−0.3322(5)	0.4081(2)	0.6523(3)	0.036(3)	0.041(3)	0.037(3)	0.000(2)	0.010(2)	−0.010(3)
C(24)	4e	−0.3640(5)	0.4595(2)	0.6081(3)	0.030(3)	0.029(3)	0.054(4)	0.010(2)	0.006(2)	−0.012(2)
C(25)	4e	−0.2686(5)	0.4736(2)	0.5365(3)	0.032(3)	0.025(3)	0.052(4)	0.007(2)	0.001(2)	−0.004(2)
C(26)	4e	−0.1414(5)	0.4363(2)	0.5063(3)	0.024(2)	0.025(3)	0.036(3)	−0.004(2)	0.001(2)	0.002(2)
C(31)	4e	0.1586(5)	0.2997(2)	0.5967(3)	0.026(2)	0.022(2)	0.022(3)	−0.000(2)	0.003(2)	0.003(2)
C(32)	4e	0.2149(5)	0.2423(2)	0.5916(3)	0.031(2)	0.031(3)	0.026(3)	0.004(2)	0.000(2)	−0.004(2)
C(33)	4e	0.3244(5)	0.2204(2)	0.6556(3)	0.040(3)	0.029(3)	0.043(3)	0.007(2)	−0.002(2)	0.002(2)
C(34)	4e	0.3727(5)	0.2554(2)	0.7255(3)	0.032(3)	0.047(3)	0.041(3)	0.004(2)	−0.004(2)	0.013(3)
C(35)	4e	0.3153(5)	0.3120(2)	0.7312(3)	0.034(3)	0.044(3)	0.027(3)	0.000(2)	−0.001(2)	−0.007(2)
C(36)	4e	0.2084(5)	0.3348(2)	0.6661(3)	0.031(2)	0.028(3)	0.033(3)	−0.000(2)	0.005(2)	−0.002(2)
C(41)	4e	0.1768(5)	0.3599(2)	0.4299(3)	0.028(2)	0.021(2)	0.027(3)	0.001(2)	0.005(2)	0.000(2)
C(42)	4e	0.1120(5)	0.3795(2)	0.3506(3)	0.027(2)	0.034(3)	0.033(3)	−0.001(2)	0.001(2)	0.003(2)
C(43)	4e	0.2217(5)	0.4005(2)	0.2888(3)	0.045(3)	0.038(3)	0.025(3)	−0.003(2)	0.007(2)	0.005(2)
C(44)	4e	0.3982(5)	0.4021(2)	0.3053(3)	0.036(3)	0.029(3)	0.040(4)	−0.006(2)	0.017(2)	−0.006(2)
C(45)	4e	0.4627(5)	0.3831(2)	0.3835(4)	0.021(2)	0.038(3)	0.050(4)	−0.004(2)	0.009(2)	−0.005(2)
C(46)	4e	0.3546(5)	0.3620(2)	0.4472(3)	0.030(2)	0.031(3)	0.038(3)	0.001(2)	−0.001(2)	−0.003(2)
Cl(3)	4e	0.8013(2)	0.31936(6)	0.1898(1)	0.0638(9)	0.061(1)	0.065(1)	−0.0015(7)	−0.0098(7)	−0.0068(8)
Cl(4)	4e	0.6451(2)	0.40638(7)	0.0755(1)	0.081(1)	0.073(1)	0.061(1)	0.0206(8)	−0.0048(7)	−0.0096(8)
C(1)	4e	0.8359(7)	0.3854(2)	0.1300(4)	0.057(3)	0.069(4)	0.067(5)	−0.004(3)	−0.011(3)	−0.016(3)

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