Crystal structure of tetraphenylphosphonium hexachloroantimonate(V), $P(C_6H_5)_4SbCl_6$

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Abstract

$C_{24}H_{20}Cl_6PSb$, tetragonal, $P4/n$ (No. 85), $a = 13.095(2)$ Å, $c = 7.780(2)$ Å, $V = 1334.1$ Å³, $Z = 2$, $R_g(F) = 0.038$, $wR_{ref}(F^2) = 0.074$, $T = 299$ K.

Source of material

$P(C_6H_5)_4Cl$, dried in vacuo at 373 K for 20 h and then dissolved in dry dichloromethane, and distilled $SbCl_5$ were mixed in a molar ratio of 1:1 under anhydrous conditions. Dry CCl_4 was slowly added until beginning precipitation. Crystals of $P(C_6H_5)_4SbCl_6$ resulted by slow partial evaporation of the solvent at reduced pressure.

Discussion

As in many compounds with this kind of cations, the $P(C_6H_5)_4^+$ ions are stacked to columns parallel to c [1]. There are many isotypic compounds. Like in all of them, the $SbCl_6^-$ ions in the title compound take positions on the fourfold rotation axes, but are slightly tilted, with the Sb atom and especially Cl(2) being situated sideways from this axis in four positions with one quarter occupation. This explains the disk-like appearance of the ellipsoid of Cl(2) and the inclination of the ellipsoid of Cl(3). As a consequence, the bond Sb—Cl(2) appears to be shorter than true. This has been thoroughly studied for $P(C_6H_5)_4WCl_6$ [2] and $P(C_6H_5)_4PbCl_6$ [3].

Table 1. Data collection and handling.

Crystal:	colourless tetragonal prism, size $0.26 \times 0.31 \times 0.47$ mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ :	17.07 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	53.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3042, 1457
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1020
$N(param)_{refined}$:	75
Programs:	SHELX-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(2)	8g	0.1288	0.5667	0.0439	0.054
H(3)	8g	0.0022	0.5195	0.2361	0.070
H(4)	8g	-0.0519	0.6305	0.4433	0.075
H(5)	8g	0.0194	0.7900	0.4654	0.077
H(6)	8g	0.1453	0.8397	0.2769	0.065

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Sb	2c	1/4	1/4	0.15946(7)	0.0418(2)	U ₁₁	0.0255(2)	0	0	0
Cl(1)	2c	1/4	1/4	0.4599(3)	0.079(1)	U ₁₁	0.029(1)	0	0	0
Cl(2)	2c	1/4	1/4	-0.1366(3)	0.163(2)	U ₁₁	0.028(1)	0	0	0
Cl(3)	8g	0.1942(1)	0.07808(8)	0.1642(2)	0.0731(8)	0.0463(6)	0.108(1)	-0.0097(5)	0.0015(8)	-0.0202(7)
P	2a	1/4	3/4	0	0.0354(7)	U ₁₁	0.036(1)	0	0	0
C(1)	8g	0.1489(3)	0.7091(3)	0.1393(5)	0.037(2)	0.042(2)	0.038(2)	0.002(2)	0.000(2)	0.001(2)
C(2)	8g	0.1067(3)	0.6121(3)	0.1278(5)	0.044(2)	0.041(2)	0.050(2)	0.002(2)	-0.001(2)	0.001(2)
C(3)	8g	0.0313(3)	0.5842(4)	0.2432(6)	0.048(3)	0.053(3)	0.075(3)	-0.006(2)	0.008(2)	0.011(2)
C(4)	8g	-0.0009(3)	0.6502(4)	0.3671(6)	0.052(3)	0.067(3)	0.069(3)	0.003(2)	0.019(2)	0.013(3)
C(5)	8g	0.0414(4)	0.7454(4)	0.3801(6)	0.071(3)	0.064(3)	0.057(3)	0.009(3)	0.023(2)	-0.004(2)
C(6)	8g	0.1163(3)	0.7751(3)	0.2674(5)	0.063(3)	0.045(2)	0.054(2)	-0.004(2)	0.014(2)	-0.006(2)

References

1. Müller, U.: Strukturverwandtschaften unter den EPh₄⁺-Salzen. *Acta Crystallogr. B* **36** (1980) 1075–1081.
2. Müller, U.; El-Kholi, A.: Tetraphenylphosphonium-hexachlorowolframat(V) und die Fehlordnung in tetragonalen EPh₄⁺-Salzen mit oktaedrischen Anionen. *Acta Crystallogr. C* **45** (1989) 1727–1730.
3. Gruber, H.; Müller, U.: Crystal structure of tetraphenylphosphonium-hexachlorophosphate, P(C₆H₅)₄SbCl₆. *Z. Kristallogr. NCS* **213** (1998) 546–547.
4. Sheldrick, G. M.: SHELX-97. A program package for crystal structure solution and refinement, University of Göttingen, Germany 1997.