Crystallographic report

Bis(triphenylphosphoranylidene)ammonium phenyltetrachlorotellurate

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Unlike most previously described organotetrachlorotellurate complexes, neither secondary Te···Cl interactions nor significant cation–anion interaction are observed in the molecular structure of [Ph₃PNPPh₃][PhTeCl₄]. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; tellurium

COMMENT

Owing to their inherent Lewis acidity, organotellurium trihalides RTeX₃ undergo complexation upon addition of halides X⁻ to give organotetrahalotellurate anions, RTeX₄⁻. Until recently only [Me₃Te][MeTeCl₄], Bu₄N[PhTeCl₃I], and Et₂NH₂[p-PhOC₆H₄TeCl₄] had been investigated by X-ray crystallography, but during the course of this work Lang *et al.* reported a series of phenyltetrahalotellurates featuring different countercations. All of these compounds possess secondary Te···X interactions and significant cation–anion interactions. The title compound of this study, [Ph₃PNPPh₃][PhTeCl₄], lacks such interactions (Fig. 1).

EXPERIMENTAL

The title compound was prepared by reacting PhTeCl₃³ (0.50 g, 1.61 mmol) with [Ph₃PNPPh₃]Cl (0.92 g, 1.61 mmol) in chloroform (50 ml) and crystallizing from a concentrated solution at room temperature. Yield: 93%, m.p. 200–204 °C. ¹H NMR (299.98 MHz, CDCl₃): δ = 7.20–7.80 (m, Ph); ¹³C{¹H} NMR (75.44 MHz, CDCl₃): δ = 126.86 [$^{1}I_{1}^{(13}C_{-}^{31}P)$ = 108], 127.94, 129.30, 129.55 (m), 132.03 (m), 133.56, 133.84; 125 Te{¹H} NMR (94.78 MHz, CDCl₃): δ = 120.8. Intensity data were collected at 293 K on a Bruker SMART Apex CCD diffractometer for a colourless rod 0.10 × 0.15 × 0.50 mm³. C₄₂H₃₅Cl₄NP₂Te, M = 885.05, orthorhombic, Fdd2, a = 32.8648(18), b = 53.156(3), c = 9.1057(5) Å, V = 15907.3(15) Å³, Z = 16, 8942 unique data (θ _{max} = 27.5°), R = 0.054 (7129 [I > $2\sigma(I)$] reflections), wR = 0.098 (all data), Flack parameter 0.005(17), ρ _{max} = 1.20 e Å⁻³

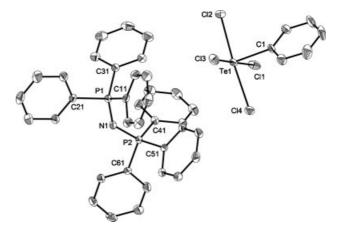


Figure 1. Molecular structure of [Ph₃PNPPh₃][PhTeCl₄] with hydrogen atoms omitted. Key geometric parameters: Te1-Cl1 2.497(2), Te1-Cl2 2.504(2), Te1-Cl3 2.528(1), Te1-Cl4 2.539(2), Te1-Cl 2.130(6) Å; Cl1-Te1-Cl2 89.12(6), Cl1-Te1-Cl3 177.70(6), Cl1-Te1-Cl4 88.72(5), Cl2-Te1-Cl3 91.41(6), Cl2-Te1-Cl4 176.28(5), Cl3-Te1-Cl4 90.63(6), Cl1-Te1-Cl 89.03(15), Cl2-Te1-Cl 89.15(17), Cl3-Te1-Cl 88.74(15), Cl4-Te1-Cl 87.78(17)°.

(near tellurium). Programs used: SAINT, SADABS, SHELX-97, WinGX 2002, and DIAMOND. CCDC deposition number: 246615.

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