Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Redetermination of a cyclic triphosphenium hexachlorostannate salt at 173 K

Bobby D. Ellis and Charles L. B. Macdonald*

Department of Chemistry and Biochemistry, University of Windsor, Windsor, Ontario, Canada N9B 3P4

Correspondence e-mail: cmacd@uwindsor.ca

Received 3 May 2006
Accepted 3 May 2006

Key indicators

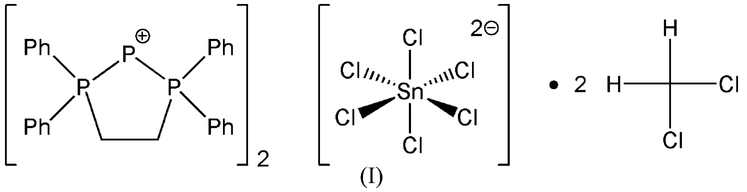
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.008 Å
R factor = 0.054
wR factor = 0.112
Data-to-parameter ratio = 20.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

At 173 K, the structure of the cyclic 'triphosphenium' salt, bis[1,1,3,3-tetraphenyl-1,3,2-triphospholenylium] hexachlorostannate(IV) dichloromethane solvate, $(C_{26}H_{24}P_3)_2[SnCl_6]\cdot 2CH_2Cl_2$, contains one cation, one solvent molecule and half of a centrosymmetric hexachlorostannate dianion in the asymmetric unit. There are no unusually short anion–cation distances and the structures of the component ions are comparable to those that have been reported previously.

Comment

The title compound, (I), was isolated in high yield from the reaction of PCl_3, $SnCl_2$ and 1,2-bis(diphenylphosphino)ethane (dppe) in dichloromethane following the method outlined by Schmidpeter *et al.* (1982). While some information regarding the crystal structure of (I) obtained at room temperature was provided in the initial report, geometrical parameters and other structural details regarding this compound (refcode BEHMEG) are not contained in the Cambridge Structural Database (CSD; Version 5.27; Allen, 2002). Given the importance of this seminal coordination complex of P^I (Ellis & Macdonald 2006), we report here the crystal structure of (I) obtained at low temperature to remedy this omission.


(I)

The asymmetric unit of (I) consists of a complete 1,1,3,3-tetraphenyl-1,3,2-triphospholenylium cation, a molecule of CH_2Cl_2 and one half of a $[SnCl_6]$ dianion. The Sn atom is located on an inversion center at (1/2, 1/2, 1/2) and the other half of a hexachlorostannate dianion is generated by that symmetry element. The contents of the asymmetric unit (including the complete symmetry-generated dianion) are depicted in Fig. 1.

The dianion in (I) is slightly distorted from perfect octahedral symmetry on the bases of both the variations in the Sn–Cl distances and the Cl–Sn–Cl angles (Table 1). The cation in (I) adopts an 'envelope' conformation (C5 is the flap atom) with an acute P–P–P fragment [88.56 (6)°] that exhibits P—P distances [2.1269 (15) and 2.1290 (15) Å] that are considerably shorter than those reported for single P—P bonds. These metrical parameters are similar to those listed in the original report of the room-temperature structure and they are consistent with those that have been reported previously for

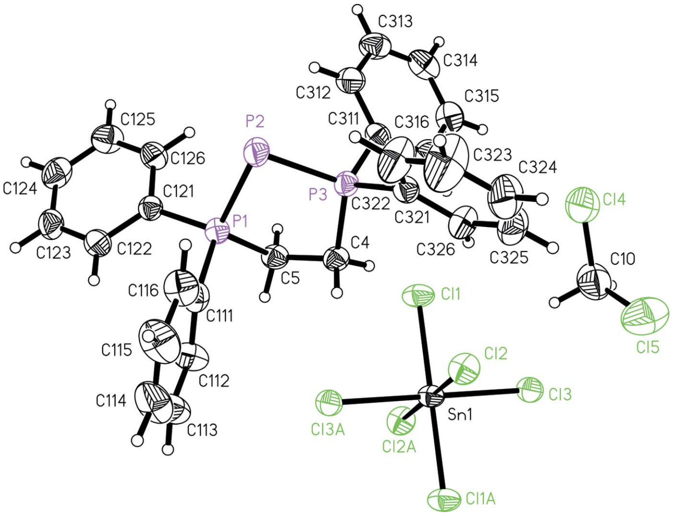


Figure 1
View of the asymmetric unit of (I), with the full hexachlorostannate dianion completed by inversion symmetry. Displacement ellipsoids are depicted at the 50% probability level. [Symmetry code: (A) $-x$, $-y$, $-z$.]

the two other salts that contain the 1,1,3,3-tetraphenyl-1,3,2-triphospholenylium cation (Ellis *et al.*, 2003).

Although they are not unusually short, the closest cation–anion contacts observed in the crystal structure of (I) are listed in Table 2.

Experimental

The title salt, (I), was synthesized according to the method described by Schmidpeter *et al.* (1982). Suitable crystals were obtained by the slow evaporation of a dichloromethane solution of the salt in a nitrogen-filled glove-box. The crystal used for analysis was coated in mineral oil, mounted and placed in the cold stream (Kryoflex) on a Bruker *APEX* CCD diffractometer.

Crystal data

$(C_{26}H_{24}P_3)_2[SnCl_6]\cdot 2CH_2Cl_2$
$M_r = 1359.99$
Triclinic, $P\bar{1}$
$a = 10.8589$ (4) Å
$b = 11.5303$ (5) Å
$c = 13.0841$ (5) Å
$\alpha = 68.777$ (1)°
$\beta = 79.774$ (1)°
$\gamma = 81.332$ (1)°
$V = 1496.16$ (10) Å^3
$Z = 1$
$D_x = 1.509$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.07$ mm^{-1}
$T = 173$ (2) K
Prism, colourless
0.2 × 0.2 × 0.2 mm

Data collection

Bruker APEX-CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.612$, $T_{\max} = 0.807$
13358 measured reflections
6663 independent reflections
5978 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.112$
$S = 1.23$
6663 reflections
322 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 2.7128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.99$ e Å^{-3}
$\Delta\rho_{\min} = -1.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—C5	1.533 (6)	C321—P3	1.809 (4)
C4—P3	1.809 (4)	P1—P2	2.1269 (15)
C5—P1	1.815 (4)	P2—P3	2.1290 (15)
C111—P1	1.801 (4)	Cl1—Sn1	2.4110 (11)
C121—P1	1.801 (4)	Cl2—Sn1	2.4398 (11)
C311—P3	1.812 (4)	Cl3—Sn1	2.4438 (10)
C5—C4—P3	106.8 (3)	Cl1—Sn1—Cl2	89.61 (4)
C4—C5—P1	106.4 (3)	Cl1—Sn1—Cl2$^{\rm i}$	90.39 (4)
C111—P1—C121	108.44 (19)	Cl1—Sn1—Cl3	89.05 (4)
C5—P1—P2	106.54 (14)	Cl2—Sn1—Cl3	90.70 (4)
P1—P2—P3	88.56 (6)	Cl1—Sn1—Cl3$^{\rm i}$	90.95 (4)
C321—P3—C311	107.15 (19)	Cl2—Sn1—Cl3$^{\rm i}$	89.30 (4)
C4—P3—P2	108.29 (14)		
P3—C4—C5—P1	−50.9 (3)	C5—C4—P3—P2	36.9 (3)
C4—C5—P1—P2	44.9 (3)	P1—P2—P3—C4	−7.71 (16)
C5—P1—P2—P3	−17.98 (16)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···Cl1	0.99	2.64	3.585 (4)	159
C10—H10B···Cl2	0.99	2.67	3.583 (6)	154
C313—H313···Cl2$^{\rm ii}$	0.95	2.82	3.663 (5)	148

Symmetry code: (ii) $x+1, y, z$.

H atoms were placed in calculated positions, with phenyl C—H = 0.95 Å or methylene C—H = 0.99 Å. These atoms were included in the refinement using the riding-model approximation, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(phenyl C) or $1.5U_{\rm eq}$(methylene C). The deepest hole in the final difference Fourier map is located 0.76 Å from atom Sn1.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT-Plus* (Bruker, 1997); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*, *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 1999).

The funding that has enabled this work has been provided by the Natural Sciences and Engineering Research Council (Canada), the Canada Foundation for Innovation, the Ontario Innovation Trust and the Ontario Research and Development Challenge Fund (University of Windsor Centre for Catalysis and Materials Research).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (1997). *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ellis, B. D., Carlesimo, M. & Macdonald, C. L. B. (2003). *Chem. Commun.* pp. 1946–1947.
Ellis, B. D. & Macdonald, C. L. B. (2006). *Am. Chem. Soc. Symp. Ser.* **917**, 108–122.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Schmidpeter, A., Lochschmidt, S. & Sheldrick, W. S. (1982). *Angew. Chem. Int. Ed. Engl.* **21**, 63–64.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.