Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[2,2-Bis(chloromethyl)propylene]bis(diphenylstibine)

Simon J. Coles,* Michael B. Hursthouse, Jon S. Joy and Michael A. Paver

Department of Chemistry, University of Southampton, Southampton SO17 1BJ, England

Correspondence e-mail: s.j.coles@soton.ac.uk

Received 16 March 2001
Accepted 18 April 2001
Online 31 May 2001

The title compound, $[Sb_2(C_6H_5)_4(C_5H_8Cl_2)]$, is the product of a substitution reaction of 2,2-bis(chloromethyl)-1,3-dichloropropane with sodium diphenylstibide. Adjacent phenyl groups embrace each other in a parallel fashion in order to minimize interaction. The molecule has crystallographic twofold rotation symmetry.

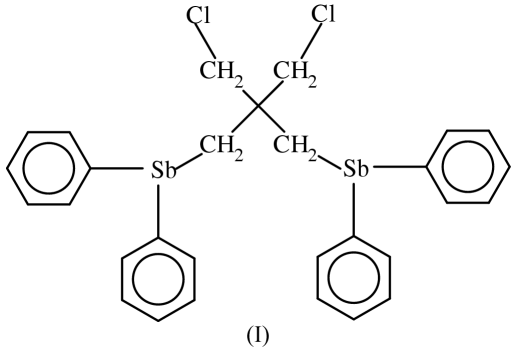
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.025
wR factor = 0.063
Data-to-parameter ratio = 18.1

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

Comment

The coordination chemistry of neutral polydentate phosphine, arsine and stibine ligands has been widely studied, especially with respect to their abilities to stabilize a range of transition metal species (Champness & Levason, 1994). The title compound, (I), was prepared as a precursor to a range of mixed-donor systems, $(Ph_2SbCH_2)_2C(CH_2EPh_x)_2$ (E = Se or Te, x = 1; E = P or As, x = 2) whose organometallic and coordination chemistry will be investigated.



(I)

The structure is composed of a tetrahedral C atom central to two diphenylstibine and two CH_2Cl substituents. Bond lengths and atomic geometries are in accordance with expected values (Orpen *et al.*, 1992). The phenyl rings in adjacent stibine moieties are arranged in a parallel fashion [ring-centroid separation = 4.086 (2) Å] in order to minimize interaction.

There are no classic hydrogen bonds formed and hence no supramolecular assembly; however, there are two weak interactions present. An intramolecular C15—H15···Cl1^{i} interaction occurs with a donor–acceptor distance of 3.188 (3) Å [symmetry code: (i) $1-x, y, \frac{1}{2}-z$]. In addition, there is a minor H···π intermolecular C3—H3···$Cg1$ interaction ($Cg1$ is the centroid of the C7–C12 ring) where C3···$Cg1^{ii}$ = 2.952 (4) Å [symmetry code: (ii) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$].

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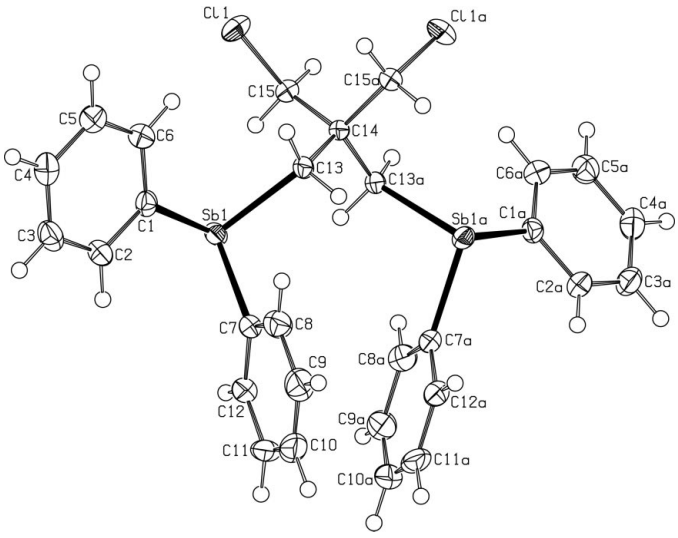

Figure 1
View of (I) (50% probability displacement ellipsoids).

Experimental

Triphenylantimony (9.79 g, 27.8 mmol) and sodium (1.28 g, 55.6 mmol) were stirred under a nitrogen atmosphere at 195 K in liquid ammonia for 5 h. After addition of ammonium chloride (1.48 g, 27.7 mmol), the mixture was stirred for a further 2 h. A solution of pentaerythrityl tetrachloride (2.92 g, 13.9 mmol) in dry diethyl ether was added dropwise and the mixture stirred overnight until decolourization was complete. After evaporation of the ammonia, water (200 ml) and toluene (200 ml) were added and work-up of the organic phase produced an oil which crystallized upon standing.

Crystal data

$[Sb_2(C_6H_5)_4(C_5H_8Cl_2)]$
M_r = 690.91
Monoclinic, $C2/c$
a = 14.578 (3) Å
b = 12.009 (2) Å
c = 15.849 (3) Å
β = 103.67 (3)°
V = 2696.2 (9) Å^3
Z = 4
D_x = 1.702 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6922 reflections
θ = 2.9–26.4°
μ = 2.22 mm^{-1}
T = 150 (2) K
Needle, colourless
0.22 × 0.08 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scans (*SORTAV*; Blessing, 1997)
T_{min} = 0.641, T_{max} = 0.878
9556 measured reflections
2739 independent reflections
2458 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 26.4°
$h = -18 \rightarrow 17$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.063
S = 1.07
2739 reflections
151 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 1.7560P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00078 (11)

Table 1
Selected geometric parameters (Å, °).

C1—Sb1	2.151 (3)	C13—Sb1	2.186 (2)
C7—Sb1	2.146 (3)		
C7—Sb1—C1	94.27 (10)	C1—Sb1—C13	97.34 (10)
C7—Sb1—C13	97.43 (9)		

Cell refinement: *DENZO* (Hooft, 1998) and *COLLECT* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990).

The authors would like to thank EPSRC for funding of the crystallographic facilities.

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