Table 1. *Selected geometric parameters (Å, °)*

Co—N9	2.104 (2)	P1···P2	2.6992 (10)
Co—N5	2.113 (2)	P2—N3	1.546 (2)
Co—N2	2.199 (2)	P2—N2	1.617 (2)
Co—Cl1	2.2788 (12)	P2—N10	1.693 (2)
Co—Cl2	2.2893 (12)	P2—N8	1.701 (2)
P1—N1	1.548 (2)	P3—N3	1.615 (2)
P1—N2	1.614 (2)	P3—N13	1.618 (2)
P1—N4	1.688 (2)	P3—N12	1.620 (2)
P1—N6	1.698 (2)	P3—N1	1.634 (2)
N9—Co—N5	159.31 (8)	N1—P1—N2	118.04 (10)
N9—Co—N2	79.66 (7)	N4—P1—N6	102.30 (10)
N5—Co—N2	80.69 (7)	N3—P2—N2	118.80 (10)
N9—Co—Cl1	98.86 (6)	N10—P2—N8	101.25 (10)
N5—Co—Cl1	96.40 (7)	N13—P3—N12	105.71 (12)
N2—Co—Cl1	119.44 (6)	N3—P3—N1	110.90 (10)
N9—Co—Cl2	92.30 (6)	P1—N1—P3	119.60 (11)
N5—Co—Cl2	93.11 (6)	P1—N2—P2	113.32 (11)
N2—Co—Cl2	123.44 (6)	P2—N3—P3	125.74 (12)
Cl1—Co—Cl2	117.12 (4)		

Most of the H atoms were located from a difference Fourier map and the remainder were fixed at calculated positions. All H atoms were refined isotropically. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined on F^2 by full-matrix least-squares methods using *SHELXL*93 (Sheldrick, 1993).

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *HELENA* (Spek, 1990*b*). Molecular graphics: *PLATON* (Spek, 1990*a*). Software used to prepare material for publication: *SHELXL*93.

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cis-Bis[(2-diphenylphosphinoethyl)dimethylstannyl]platinum(II)

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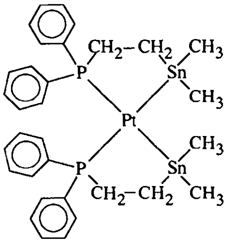
Abstract

In the spirocyclic title complex, $[PtSn_2(CH_3)_4(C_{14}H_{14}P)_2]$, the Pt atom is situated at the centre of a slightly distorted square-planar coordination polyhedron. The Pt—P [2.283 (1) and 2.286 (1) Å] and Pt—Sn [2.5973 (5) and 2.6047 (5) Å] distances agree well with the corresponding distances in related compounds.

Comment

Bis(stannyl)bis(phosphine)palladium(II) and -platinum(II) complexes are of interest as intermediates in the metal-catalysed double stannylation of 1,3-dienes, alkynes and allenes; the oxidative addition of organodistannanes to palladium(0) and platinum(0) complexes, *e.g.* $[M^0(CH_2{=}CH_2)(PPh_3)_2]$ and $[M^0(PPh_3)_4]$ (M = Pd, Pt) is believed to be a crucial step in the catalytic cycle of this stannylation reaction. At present, there are only two fully characterized examples of this type of complex, namely *cis*-$[Pt(SnMe_3)_2\{P(C_6H_4Me\text{-}4)_3\}_2]$ (Obora *et al.*, 1996) and *cis*-$[Pt\{1,1'\text{-}(SnMe_2)_2Fc\}(PPh_3)_2]$ (Fc = $FeC_{10}H_8$) (Herberhold *et al.*, 1997). We report here the crystal structure of the related complex *cis*-bis[(2-diphenylphosphinoethyl)dimethylstannyl]platinum(II), (I). Its synthesis and structural characterization by NMR studies have been published previously (Weichmann, 1982).



(I)

The main feature of the structure of (I) is the anchoring of the two $Ph_2PCH_2CH_2SnMe_2$ ligands through P→Pt coordination and σ-Sn—Pt bonds to the central atom, thus forming a spirocyclic system. The Pt atom is situated at the centre of a slightly distorted square-planar coordination sphere with a *cis* arrangement of the Sn and P atoms. The dihedral angle between the PtP_2 and the $PtSn_2$ planes is only 5.64 (5)°. This deviation from exact planarity is much smaller than in the two complexes mentioned above (34.6 and 20.7°, respectively), obviously as a consequence of the chelate nature of the ligand. The two Pt—Sn and two Pt—P bond lengths in the title compound are in nearly exact agreement with each other and barely differ from the corresponding values in the other two complexes. The molecules of the title compound have C_2 symmetry to a good approximation, with the twofold axis passing through the Pt atom and the midpoints of the lines Sn1···Sn2 and P1···P2. The conformations of the two five-membered chelate rings are similar but highly irregular and far from envelope or half-chair conformations.

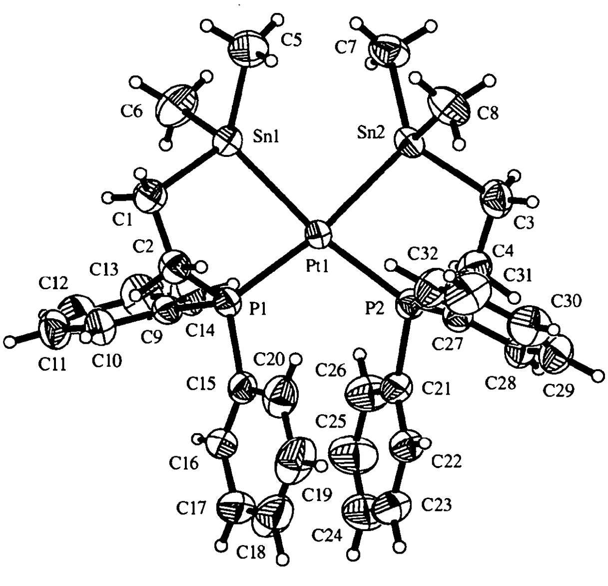


Fig. 1. The molecular structure of the title compound. The displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by small circles of arbitrary radii.

Experimental

The title compound was prepared by a procedure described previously (Weichmann, 1982). In solution, it always occurs in a 4:1 mixture with the *trans* isomer. Separation of the pure isomers can be achieved by fractional crystallization from acetone. In this way, the *cis* isomer was isolated in the form of colourless single crystals suitable for X-ray investigations.

Crystal data

$[PtSn_2(CH_3)_4(C_{14}H_{14}P)_2]$
$M_r = 919.05$
Monoclinic
$P2_1/n$
$a = 11.801\ (2)$ Å
$b = 13.206\ (3)$ Å
$c = 21.697\ (4)$ Å
$\beta = 103.144\ (15)°$
$V = 3292.8\ (11)$ Å^3
$Z = 4$
$D_x = 1.854$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 76 reflections
$\theta = 10.1–17.1°$
$\mu = 5.859$ mm^{-1}
$T = 293\ (2)$ K
Irregular
0.53 × 0.36 × 0.34 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.102$, $T_{max} = 0.136$
17 011 measured reflections
7945 independent reflections
6514 reflections with $I > 2\sigma(I)$

$R_{int} = 0.021$
$\theta_{max} = 28.0°$
$h = -15 \rightarrow 15$
$k = -17 \rightarrow 17$
$l = -28 \rightarrow 28$
3 standard reflections
frequency: 60 min
intensity decay: 5.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.071$
$S = 1.085$
7945 reflections
335 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2 + 2.0278P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 1.239$ e Å^{-3}
$\Delta\rho_{min} = -1.022$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00023 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Pt1—P2	2.2833 (10)	Pt1—Sn2	2.5973 (5)
Pt1—P1	2.2865 (10)	Pt1—Sn1	2.6047 (5)
P2—Pt1—P1	106.33 (4)	P1—Pt1—Sn1	82.49 (3)
P2—Pt1—Sn2	83.16 (3)	Sn2—Pt1—Sn1	87.88 (2)

The quadrants $\pm h{+}k{+}l$ and $\pm h{-}k{-}l$ were registered. H-atom positions were calculated by geometrical considerations and treated according to a riding model. The lowest (-1.022 e Å^{-3}) and highest (1.239 e Å^{-3}) peaks in the final difference Fourier map are located close to the Pt atom at distances of 1.600 and 0.926 Å, respectively.

Data collection: *DIF*4 (Stoe, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP*/*PC* (Siemens, 1990). Software used to prepare material for publication: *SHELXL*93.

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Chloro(dibenzylamine-*N*)dimethylaluminium(III)

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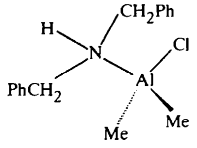
Abstract

The addition of dibenzylamine to Me_2AlCl gives the title alane adduct, $[Me_2ClAl(NHBz_2)]$ or $[AlCl(CH_3)_2(C_{14}H_{15}N)]$. The coordination geometry about the Al atom is distorted tetrahedral in both the crystalline phase and in solution.

Comment

We recently reported (Armstrong *et al.*, 1996) that the reaction of the simple alane amine adduct $[Me_3Al(NHBz_2)]$, (II), with the lithium amide $LiNBz_2$ affords the novel heterobimetallic adduct $[Me_3Al(Bz_2NLi)(NHBz_2)]$, which still retains coordinated amine. In a follow-up study (Armstrong *et al.*, 1998), we showed that the presence of this amine could be used to advantage to promote a subsequent intramolecular methane elimination/amine insertion process that generates the lithium alkylaluminium secondary amide $[Me_2Al(Bz_2N)_2Li(thf)]$ (thf is tetrahydrofuran). As a prerequisite to investigating variations of this reaction sequence, we have been synthesizing and characterizing dibenzylamine adducts of a number of different alanes. In the course of this work, we have crystallized the chloro derivative of (II), $[Me_2ClAl(NHBz_2)]$, (I), whose structure is reported here.



(I)

The crystal structure consists of discrete molecular units, with no intermolecular contacts of significantly less than the sum of van der Waals radii. The coordination geometry about the Al atom is distorted tetrahedral, the largest deviation from ideal geometry being the wide C1—Al1—C2 angle of 122.8 (1)° (Table 1). A similar steric widening of the Me—Al—Me angle is found in the two other known structures containing the Me_2ClAlN moiety (Atwood & Jegier, 1996; Sun *et al.*, 1995). That the tetrahedral arrangement persists in pyridine solution is confirmed by ^{27}Al NMR spectroscopy [see *Experimental* and Akitt (1989)]. Comparing the Al—N distance in (I) [2.020 (2) Å] with those in these two complexes shows that of (I) to be a longer bond than that of the primary amine analogue $[Me_2ClAl(NH_2{}^tBu)]$, [1.993 (4) Å] and that a better match is found with that in $[Me_2ClAl(5,6\text{-benzoquinoline})]$ [2.016 (3) Å].

The greater electronegativity of the Cl ligand and the subsequent lower electron density on the metal centre results in the Al—N bond length of (I) being slightly shorter than that of 2.058 (2) Å in the closely related complex (II). The molecular conformation of (I) is broadly similar to that of (II). Examination of the torsion angles shows that the Me_2ClAl fragment is *anti* with respect to the phenyl ring of one benzyl group [Al1—N1—C10—C11 −179.4 (2)°] and that the orientation about the Al—N bond is such that the other benzyl group lies *anti* to the Cl atom [Cl1—Al1—N1—C3 −171.6 (1)°].