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Tetramethylammonium dichlorodiphenylbismuthate(III)

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Key indicators

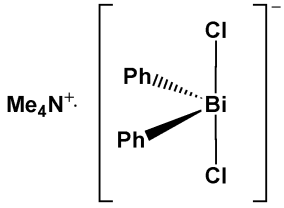
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
R factor = 0.022
wR factor = 0.052
Data-to-parameter ratio = 17.1

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

The crystal structure of the title compound, $(C_4H_{12}N)[BiCl_2(C_6H_5)_2]$, contains a $[BiCl_2Ph_2]^-$ anion with an equatorially vacant trigonal-bipyramidal geometry.

Comment

A variety of anions of formula $[ECl_2Ph_2]^-$ are known for arsenic (Grewe *et al.*, 1998) and antimony (Sheldrick & Martin, 1992; Hall & Sowerby, 1988; Calderazzo *et al.*, 1991; Stark *et al.*, 1999). For bismuth, the $[BiBr_2Ph_2]^-$ (Stark *et al.*, 1999; Clegg *et al.*, 1992) and $[BiI_2Ph_2]^-$ (Clegg *et al.*, 1993) ions have been structurally characterized. This paper reports the structure of the $[BiCl_2Ph_2]^-$ anion as the tetramethylammonium salt, (I).



(I)

The structure of the anion in (I) is similar to that found for other diaryldihalobismuthate(III) anions. The geometry is based on a trigonal bipyramid in which the formal lone pair and phenyl groups occupy equatorial positions and the halides reside in axial sites. For bismuth, the C—Bi—C angle between the equatorial phenyl groups approaches 90°, which is typical of inter-bond angles in Bi^{III} structures (Clegg *et al.*, 1992, 1993).

Experimental

Crystals of the title compound were obtained from a reaction between $BiCl_2Ph$ and $[NMe_4]Cl$ in tetrahydrofuran overlaid with hexane.

Crystal data

$(C_4H_{12}N)[BiCl_2(C_6H_5)_2]$
M_r = 508.23
Monoclinic, $P2_1/n$
a = 12.162 (3) Å
b = 11.1693 (15) Å
c = 14.241 (3) Å
β = 110.642 (14)°
V = 1810.3 (7) Å^3
Z = 4
D_x = 1.865 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 181 reflections
θ = 2–20°
μ = 10.03 mm^{-1}
T = 173 (2) K
Block, colourless
0.30 × 0.10 × 0.05 mm

Data collection

Bruker CCD area-detector diffractometer	3185 independent reflections
0.3° ω scans	2787 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.026$
$T_{min} = 0.305$, $T_{max} = 0.605$	$\theta_{max} = 25.0°$
9238 measured reflections	$h = -11 \rightarrow 14$
	$k = -13 \rightarrow 13$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.022$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.052$	$\Delta\rho_{max} = 1.59$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -1.28$ e Å^{-3}
3185 reflections	Extinction correction: *SHELXL*97
186 parameters	Extinction coefficient: 0.00272 (13)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Bi1—C7	2.236 (5)	Bi1—Cl2	2.7310 (11)
Bi1—C1	2.252 (4)	Bi1—Cl1	2.7348 (11)
C7—Bi1—C1	94.74 (16)	C7—Bi1—Cl1	88.04 (10)
C7—Bi1—Cl2	87.37 (10)	C1—Bi1—Cl1	87.37 (10)
C1—Bi1—Cl2	89.82 (11)	Cl2—Bi1—Cl1	174.40 (4)

Methyl H atoms were positioned using a rotating-group refinement, with isotropic displacement parameters 1.5 times that of their adjacent C atom. The phenyl H atoms were constrained to ideal geometries and assigned isotropic displacement parameters 1.2 times that of their adjacent C atom. The two highest residual electron-density peaks (1.59 and 1.50 e Å^{-3}) are found 0.97 and 0.98 Å from the Bi atom. All other residual electron-density peaks have values less than 1 e Å^{-3}.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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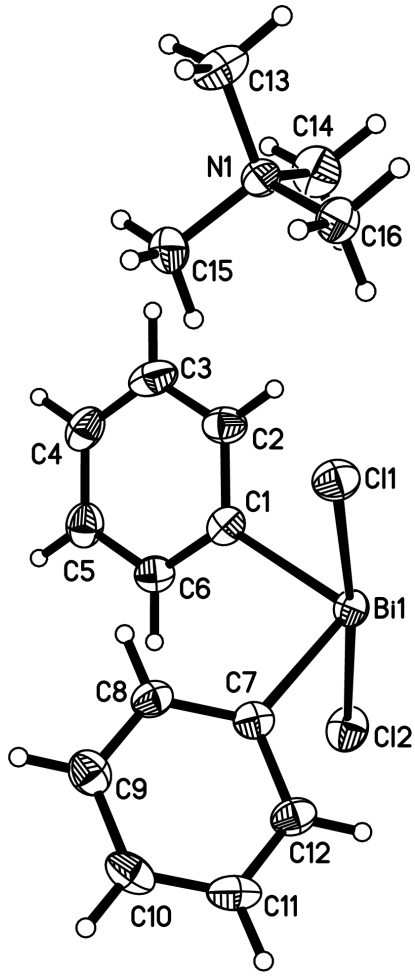

Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

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