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Methyltriphenylstibonium tetrafluoroborate

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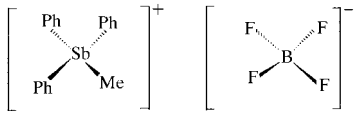
In the title compound, $[Sb(CH_3)(C_6H_5)_3]BF_4$, there are four independent cations and anions in the asymmetric unit. The geometry around the Sb atom is distorted tetrahedral, with Sb—C distances in the range 2.077 (4)–2.099 (10) Å and angles at the Sb atom in the range 103.3 (3)–119.0 (4)°.

Comment

Examination of the structure of the title compound, (I), with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids in the crystal lattice.



(I)

Experimental

The title compound was prepared from Ph_3Sb and $[Me_3O][BF_4]$, following a published procedure (Henry & Wittig, 1960). The material was recrystallized from aqueous EtOH (m.p. 408–409 K).

Crystal data

$[Sb(CH_3)(C_6H_5)_3]BF_4$
M_r = 454.90
Triclinic, $P\overline{1}$
a = 12.874 (4) Å
b = 13.760 (5) Å
c = 24.974 (7) Å
α = 98.82 (3)°
β = 93.97 (2)°
γ = 117.80 (3)°
V = 3817 (2) Å^3
Z = 8
D_x = 1.583 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.53–11.92°
μ = 1.480 mm^{-1}
T = 293 (2) K
Block, colourless
0.42 × 0.28 × 0.26 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian (*NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.689, T_{max} = 0.715
14 181 measured reflections
14 181 independent reflections
8121 reflections with $I > 2\sigma(I)$
θ_{max} = 25.42°
$h = -15 \rightarrow 13$
$k = 0 \rightarrow 16$
$l = -30 \rightarrow 29$
3 standard reflections
frequency: 120 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.181
S = 1.028
14 181 reflections
722 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0943P)^2 + 4.9573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 1.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

Compound (I) crystallized in the triclinic system; space group $P\overline{1}$ was assumed and confirmed by the analysis. The BF_4 anions and the phenyl rings showed large librational effects and were accordingly refined as rigid groups with anisotropic displacement parameters. The F-atom positions of the BF_4 anions were restrained using the *DFIX* option of *SHELXL*97 (Sheldrick, 1997), based on difference-map peaks. All H atoms were placed in calculated positions. In the case of the methyl H atoms, these were placed as six half–H atoms at the corners of a regular hexagon, and refined as riding atoms. The C—H distances were 0.93 and 0.96 Å. The largest peaks on the difference map were adjacent to B3, 1.25–1.46 Å, and its adjacent F atoms, and to the Sb atoms, 1.17–1.28 Å.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992); cell refinement: *SET*4 and *CELDIM* in *CAD*-4-*PC Software;* data reduction: *DATRD*2 in *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *NRCVAX, SHELXL*97 and *WordPerfect* macro *PREP*8 (Ferguson, 1998).

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