Crystal structure of tris(perfluorophenyl)stibine, $C_{18}F_{15}Sb$

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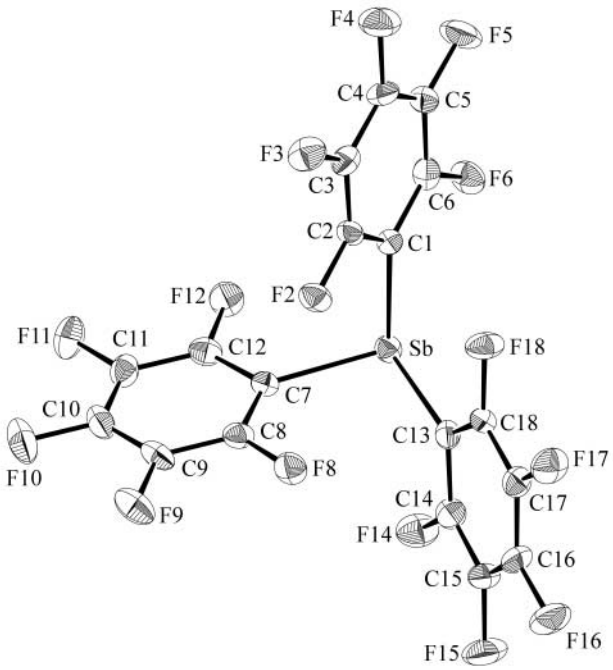

Abstract

$C_{18}F_{15}Sb$, monoclinic, $P12_1/n1$ (No. 14), a = 5.9402(8) Å, b = 20.390(6) Å, c = 15.024(3) Å, β = 93.00(1)°, V = 1817.2 Å^3, Z = 4, $R_{gt(F)}$ = 0.029, $wR_{ref(F^2)}$ = 0.071, T = 173 K.

Source of material

The compound was prepared in accord with the literature procedure [1]. Colorless crystals (mp 349 K) were obtained from the cooling of the mother liquor (benzene/hexane) to 273 K.

Discussion

In $Sb(C_6F_5)_3$, the d(Sb—C) are experimentally equivalent with an average value of 2.170(2) Å and the angles subtended at the trigonal pyramidal antimony atom range from 95.3(1)° to 100.1(1)°. The dihedral angles between the three aromatic rings are 87.9(2)°, 60.6(2)° and 76.1(2)°, respectively, indicating significant deviations from three-fold symmetry.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.11 × 0.18 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	16.70 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5655, 4183
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2685
$N(param)_{refined}$:	307
Programs:	DIRDIF92 [2], teXsan [3], SHELXL-97 [4], ORTEPII [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sb	4*e*	0.10201(4)	0.05779(1)	0.20467(2)	0.0210(1)	0.0221(1)	0.0213(1)	0.0014(1)	0.00111(8)	–0.0014(1)
F(2)	4*e*	–0.3771(4)	–0.0146(1)	0.2674(2)	0.023(1)	0.027(1)	0.031(1)	0.007(1)	0.0015(9)	–0.004(1)
F(3)	4*e*	–0.4978(4)	–0.1413(1)	0.2720(2)	0.030(1)	0.034(1)	0.044(2)	–0.002(1)	0.008(1)	0.007(1)
F(4)	4*e*	–0.2338(5)	–0.2344(1)	0.2019(2)	0.057(2)	0.021(1)	0.054(2)	–0.004(1)	0.011(1)	–0.000(1)
F(5)	4*e*	0.1559(5)	–0.2001(1)	0.1270(2)	0.056(2)	0.026(1)	0.054(2)	0.011(1)	0.018(1)	–0.008(1)
F(6)	4*e*	0.2807(4)	–0.0734(1)	0.1238(2)	0.028(1)	0.034(2)	0.037(1)	0.004(1)	0.012(1)	–0.004(1)
F(8)	4*e*	–0.3705(4)	0.1276(1)	0.2949(2)	0.027(1)	0.034(1)	0.031(1)	0.009(1)	0.001(1)	–0.003(1)
F(9)	4*e*	–0.4507(4)	0.1551(1)	0.4633(2)	0.033(1)	0.039(2)	0.048(2)	0.003(1)	0.011(1)	–0.016(1)
F(10)	4*e*	–0.1510(5)	0.1208(1)	0.5979(2)	0.056(2)	0.057(2)	0.028(1)	–0.006(1)	0.012(1)	–0.013(1)
F(11)	4*e*	0.2388(4)	0.0594(1)	0.5605(2)	0.046(2)	0.054(2)	0.025(1)	0.001(1)	–0.010(1)	0.006(1)
F(12)	4*e*	0.3256(4)	0.0319(1)	0.3906(2)	0.028(1)	0.039(1)	0.034(1)	0.008(1)	–0.003(1)	–0.001(1)
F(14)	4*e*	0.0547(4)	0.2052(1)	0.1710(2)	0.039(2)	0.028(1)	0.052(2)	–0.008(1)	–0.004(1)	0.000(1)
F(15)	4*e*	–0.2481(5)	0.2860(1)	0.0860(2)	0.060(2)	0.019(1)	0.059(2)	–0.001(1)	–0.003(1)	0.008(1)
F(16)	4*e*	–0.6216(4)	0.2346(1)	0.0002(2)	0.054(2)	0.034(2)	0.047(2)	0.014(1)	–0.012(1)	0.011(1)

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Table 2. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(17)	4*e*	–0.6892(4)	0.1034(1)	–0.0016(2)	0.038(1)	0.038(2)	0.038(1)	0.002(1)	–0.015(1)	0.002(1)
F(18)	4*e*	–0.3896(4)	0.0233(1)	0.0808(2)	0.040(1)	0.019(1)	0.042(2)	–0.003(1)	–0.015(1)	–0.001(1)
C(1)	4*e*	–0.0411(6)	–0.0400(2)	0.1979(2)	0.026(2)	0.020(2)	0.019(2)	0.004(1)	0.000(2)	0.000(1)
C(2)	4*e*	–0.2380(6)	–0.0597(2)	0.2341(2)	0.023(2)	0.021(2)	0.021(2)	0.007(2)	–0.001(1)	–0.003(2)
C(3)	4*e*	–0.3057(6)	–0.1240(2)	0.2365(3)	0.026(2)	0.025(2)	0.024(2)	–0.003(2)	0.001(2)	0.004(2)
C(4)	4*e*	–0.1709(7)	–0.1712(2)	0.2005(3)	0.040(2)	0.016(2)	0.030(2)	0.000(2)	–0.001(2)	0.004(2)
C(5)	4*e*	0.0268(7)	–0.1539(2)	0.1627(3)	0.036(2)	0.024(2)	0.024(2)	0.010(2)	0.001(2)	–0.001(2)
C(6)	4*e*	0.0877(6)	–0.0888(2)	0.1621(3)	0.025(2)	0.032(2)	0.022(2)	0.002(2)	0.001(2)	0.000(2)
C(7)	4*e*	–0.0155(6)	0.0802(2)	0.3360(2)	0.027(2)	0.018(2)	0.023(2)	0.001(2)	0.003(2)	–0.002(2)
C(8)	4*e*	–0.2127(6)	0.1107(2)	0.3579(3)	0.027(2)	0.018(2)	0.029(2)	–0.002(2)	–0.002(2)	–0.002(2)
C(9)	4*e*	–0.2592(6)	0.1248(2)	0.4449(3)	0.025(2)	0.023(2)	0.035(2)	–0.002(2)	0.010(2)	–0.010(2)
C(10)	4*e*	–0.1058(7)	0.1074(2)	0.5132(3)	0.037(2)	0.029(2)	0.024(2)	–0.008(2)	0.009(2)	–0.008(2)
C(11)	4*e*	0.0887(7)	0.0770(2)	0.4942(3)	0.034(2)	0.028(2)	0.022(2)	–0.007(2)	–0.004(2)	0.001(2)
C(12)	4*e*	0.1312(6)	0.0632(2)	0.4068(3)	0.023(2)	0.023(2)	0.033(2)	0.001(2)	0.001(2)	–0.002(2)
C(13)	4*e*	–0.1588(6)	0.1119(2)	0.1299(2)	0.025(2)	0.025(2)	0.018(2)	0.001(2)	0.001(2)	–0.001(2)
C(14)	4*e*	–0.1292(6)	0.1792(2)	0.1274(3)	0.028(2)	0.025(2)	0.027(2)	–0.004(2)	0.005(2)	0.000(2)
C(15)	4*e*	–0.2808(7)	0.2212(2)	0.0854(3)	0.040(2)	0.019(2)	0.032(2)	0.003(2)	0.006(2)	0.003(2)
C(16)	4*e*	–0.4691(7)	0.1952(2)	0.0426(3)	0.038(2)	0.025(2)	0.028(2)	0.010(2)	0.001(2)	0.008(2)
C(17)	4*e*	–0.5043(7)	0.1286(2)	0.0410(3)	0.028(2)	0.030(2)	0.023(2)	0.005(2)	–0.004(2)	0.001(2)
C(18)	4*e*	–0.3474(7)	0.0880(2)	0.0841(3)	0.032(2)	0.018(2)	0.021(2)	0.002(2)	0.005(2)	–0.002(2)

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