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Structure of Triphenylstibine

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Abstract. $C_{18}H_{15}Sb$, $M_r = 353·08$, triclinic, $P\bar{1}$, $a = 10·909\ (7)$, $b = 11·728\ (6)$, $c = 13·737\ (6)$ Å, $\alpha = 102·61\ (4)$, $\beta = 104·42\ (4)$, $\gamma = 107·94\ (4)°$, $V = 1534\ (1)$ Å^3, $Z = 4$, $D_x = 1·53$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 17·92$ cm^{-1}, $F(000) = 696$, $T = 294$ K, $R = 0·0373$ for 2421 observed reflections. The two independent molecules in the asymmetric unit differ in phenyl ring conformation; dihedral angles between phenyl ring planes C(1)–C(6), C(7)–C(12) and C(13)–C(18) and the basal plane defined by atoms C(1), C(7) and C(13) for molecule (1) are 65·1, 93·2 and 37·2°, respectively; corresponding values for molecule (2) [phenyl rings: C(19)–C(24), C(25)–C(30) and C(31)–C(36)] with the basal plane defined by atoms C(19), C(25) and C(31) are 64·9, 118·4 and 58·7°, respectively. Average Sb—C distance is 2·155 (9) Å, C—Sb—C angles range from 95·1 (3) to 98·0 (3)°.

Experimental. Colorless parallelepiped crystal (m.p. 326·5 K), obtained by slow evaporation of an acetone/water solution, dimensions 0·20 × 0·30 × 0·45 mm. Data collected at room temperature, graphite-monochromated Mo $K\alpha$ radiation, Nicolet $R3m$/V diffractometer, $\omega/2\theta$ scans of 2–15°min^{-1}, $2\theta_{max} = 45°$, $h = 0$ to 11, $k = -12$ to 12, $l = -14$ to 14, 4299 measurements, 4042 unique ($R_{int} = 0·030$), 2421 observed [$I > 3\sigma(I)$]. Unit-cell dimensions determined by least-squares fit to settings for 29 reflections ($15 < 2\theta < 30°$). Empirical absorption correction ($\mu = 17·92$ cm^{-1}), transmission factors 0·78–1·00; three standards monitored (± 1%), 40·2 h of X-ray exposure.

Solved by direct methods; full-matrix least-squares refinement on F, $R = 0·0373$, $wR = 0·0432$, $S = 0·95$,

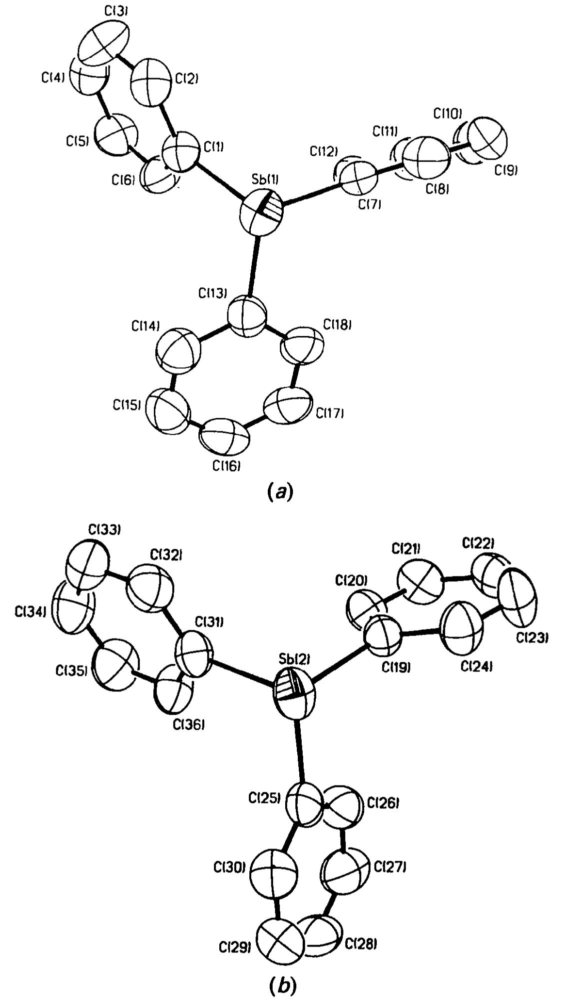

Fig. 1. Thermal-ellipsoid plots (50% probability); H atoms omitted. Molecules are viewed normal to their basal planes. (*a*) Molecule (1). (*b*) Molecule (2).

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
Sb(1)	0·3275 (1)	0·8439 (1)	0·4352 (1)	0·060 (1)
Sb(2)	0·1139 (1)	0·3745 (1)	0·1447 (1)	0·058 (1)
C(1)	0·1233 (7)	0·7035 (6)	0·3601 (6)	0·050 (4)
C(2)	0·0446 (8)	0·7013 (7)	0·2623 (6)	0·061 (4)
C(3)	−0·0858 (9)	0·6122 (8)	0·2077 (7)	0·081 (5)
C(4)	−0·1405 (9)	0·5208 (8)	0·2489 (7)	0·068 (4)
C(5)	−0·0687 (8)	0·5201 (8)	0·3451 (6)	0·068 (4)
C(6)	0·0621 (8)	0·6101 (7)	0·3999 (6)	0·061 (4)
C(7)	0·4358 (8)	0·7258 (8)	0·3938 (5)	0·057 (4)
C(8)	0·5434 (9)	0·7704 (9)	0·3601 (6)	0·081 (5)
C(9)	0·6199 (10)	0·7000 (12)	0·3350 (7)	0·094 (6)
C(10)	0·5828 (11)	0·5800 (11)	0·3463 (7)	0·093 (6)
C(11)	0·4743 (10)	0·5330 (9)	0·3753 (7)	0·082 (5)
C(12)	0·3987 (9)	0·6038 (8)	0·4001 (6)	0·068 (4)
C(13)	0·3489 (8)	0·8278 (7)	0·5910 (6)	0·057 (4)
C(14)	0·2622 (9)	0·8582 (8)	0·6412 (7)	0·074 (5)
C(15)	0·2698 (10)	0·8480 (8)	0·7407 (8)	0·084 (5)
C(16)	0·3669 (10)	0·8081 (8)	0·7927 (7)	0·076 (5)
C(17)	0·4539 (9)	0·7809 (7)	0·7454 (7)	0·075 (5)
C(18)	0·4466 (8)	0·7898 (7)	0·6441 (6)	0·069 (4)
C(19)	−0·0921 (7)	0·2616 (7)	0·0373 (5)	0·051 (3)
C(20)	−0·1601 (8)	0·1356 (7)	0·0238 (6)	0·057 (4)
C(21)	−0·2899 (8)	0·0685 (8)	−0·0489 (6)	0·069 (4)
C(22)	−0·3537 (8)	0·1250 (8)	−0·1123 (6)	0·069 (4)
C(23)	−0·2879 (9)	0·2502 (9)	−0·0997 (7)	0·083 (5)
C(24)	−0·1579 (8)	0·3190 (7)	−0·0247 (6)	0·067 (4)
C(25)	0·2121 (8)	0·3014 (8)	0·0411 (7)	0·065 (4)
C(26)	0·1429 (9)	0·1935 (8)	−0·0463 (7)	0·072 (5)
C(27)	0·2098 (11)	0·1516 (9)	−0·1099 (8)	0·087 (6)
C(28)	0·3453 (12)	0·2179 (13)	−0·0883 (10)	0·109 (9)
C(29)	0·4156 (11)	0·3258 (14)	−0·0033 (10)	0·117 (8)
C(30)	0·3480 (10)	0·3684 (10)	0·0614 (8)	0·090 (6)
C(31)	0·1282 (7)	0·2519 (6)	0·2401 (6)	0·053 (4)
C(32)	0·1046 (8)	0·2810 (8)	0·3351 (6)	0·069 (4)
C(33)	0·1168 (9)	0·2076 (9)	0·4013 (7)	0·083 (5)
C(34)	0·1526 (10)	0·1068 (9)	0·3731 (7)	0·084 (5)
C(35)	0·1747 (9)	0·0761 (8)	0·2791 (7)	0·082 (5)
C(36)	0·1626 (8)	0·1501 (7)	0·2139 (6)	0·065 (4)

Table 2. *Bond distances* (Å) *and angles* (°)

Sb(1)—C(1)	2·143 (6)	Sb(1)—C(7)	2·152 (10)
Sb(1)—C(13)	2·151 (9)	Sb(2)—C(19)	2·154 (6)
Sb(2)—C(25)	2·169 (10)	Sb(2)—C(31)	2·161 (9)
C(1)—C(2)	1·392 (11)	C(1)—C(6)	1·392 (12)
C(2)—C(3)	1·374 (10)	C(3)—C(4)	1·367 (14)
C(4)—C(5)	1·360 (12)	C(5)—C(6)	1·381 (9)
C(7)—C(8)	1·366 (13)	C(7)—C(12)	1·391 (13)
C(8)—C(9)	1·392 (18)	C(9)—C(10)	1·394 (19)
C(10)—C(11)	1·339 (16)	C(11)—C(12)	1·388 (16)
C(13)—C(14)	1·386 (14)	C(13)—C(18)	1·383 (13)
C(14)—C(15)	1·383 (15)	C(15)—C(16)	1·388 (15)
C(16)—C(17)	1·352 (16)	C(17)—C(18)	1·403 (13)
C(19)—C(20)	1·380 (10)	C(19)—C(24)	1·392 (13)
C(20)—C(21)	1·374 (9)	C(21)—C(22)	1·385 (14)
C(22)—C(23)	1·370 (13)	C(23)—C(24)	1·389 (10)
C(25)—C(26)	1·381 (10)	C(25)—C(30)	1·369 (13)
C(26)—C(27)	1·375 (17)	C(27)—C(28)	1·361 (16)
C(28)—C(29)	1·365 (16)	C(29)—C(30)	1·393 (19)
C(31)—C(32)	1·382 (12)	C(31)—C(36)	1·361 (13)
C(32)—C(33)	1·393 (15)	C(33)—C(34)	1·364 (16)
C(34)—C(35)	1·364 (15)	C(35)—C(36)	1·387 (14)
C(1)—Sb(1)—C(7)	98·0 (3)	C(1)—Sb(1)—C(13)	95·7 (3)
C(7)—Sb(1)—C(13)	96·0 (3)	C(19)—Sb(2)—C(25)	95·1 (3)
C(19)—Sb(2)—C(31)	97·5 (3)	C(25)—Sb(2)—C(31)	95·5 (4)
Sb(1)—C(1)—C(2)	118·7 (6)	Sb(1)—C(1)—C(6)	125·2 (5)
C(2)—C(1)—C(6)	116·1 (6)	C(1)—C(2)—C(3)	122·3 (8)
C(2)—C(3)—C(4)	119·5 (8)	C(3)—C(4)—C(5)	120·5 (7)
C(4)—C(5)—C(6)	119·8 (8)	C(1)—C(6)—C)5)	121·8 (7)
Sb(1)—C(7)—C(8)	119·9 (7)	Sb(1)—C(7)—C(12)	121·6 (7)
C(8)—C(7)—C(12)	118·5 (10)	C(7)—C(8)—C(9)	122·7 (10)
C(8)—C(9)—C(10)	117·1 (10)	C(9)—C(10)—C(11)	121·0 (13)
C(10)—C(11)—C(12)	121·4 (11)	C(7)—C(12)—C(11)	119·2 (9)
Sb(1)—C(13)—C(14)	118·7 (7)	Sb(1)—C(13)—C(18)	122·9 (7)
C(14)—C(13)—C(18)	118·4 (8)	C(13)—C(14)—C(15)	121·4 (9)
C(14)—C(15)—C(16)	119·8 (10)	C(15)—C(16)—C(17)	119·3 (9)
C(16)—C(17)—C(18)	121·5 (9)	C(13)—C(18)—C(17)	119·6 (9)
Sb(2)—C(19)—C(20)	124·1 (6)	Sb(2)—C(19)—C(24)	117·4 (5)
C(20)—C(19)—C(24)	118·4 (6)	C(19)—C(20)—C(21)	120·8 (8)
C(20)—C(21)—C(22)	120·6 (7)	C(21)—C(22)—C(23)	119·4 (7)
C(22)—C(23)—C(24)	120·2 (9)	C(19)—C(24)—C(23)	120·6 (8)
Sb(2)—C(25)—C(26)	123·1 (7)	Sb(2)—C(25)—C(30)	118·1 (6)
C(26)—C(25)—C(30)	118·8 (10)	C(25)—C(26)—C(27)	120·8 (8)
C(26)—C(27)—C(28)	120·0 (9)	C(27)—C(28)—C(29)	120·3 (13)
C(28)—C(29)—C(30)	119·8 (11)	C(25)—C(30)—C(29)	120·3 (8)
Sb(2)—C(31)—C(32)	117·8 (6)	Sb(2)—C(31)—C(36)	124·0 (6)
C(32)—C(31)—C(36)	118·2 (8)	C(31)—C(32)—C(33)	120·2 (9)
C(32)—C(33)—C(34)	120·2 (9)	C(33)—C(34)—C(35)	120·3 (10)
C(34)—C(35)—C(36)	119·0 (10)	C(31)—C(36)—C(35)	122·2 (9)

344 variables including positional and anisotropic thermal parameters for non-H atoms, H atoms in idealized positions ($d_{\mathrm{C—H}} = 0·95$ Å) with isotropic group thermal parameter [$U_{\mathrm{iso}} = 0·084\,(5)$ Å^2], $(\Delta/\sigma)_{\mathrm{max}} = 0·01$, function minimized was $\sum w(|F_o|-|F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0·0005|F_o|^2]$. Final difference map peaks ranged from $-0·34$ to 0·42 e Å^{-3}. Computer programs: Sheldrick (1986), scattering factors: Cromer & Waber (1974), real and imaginary anomalous-dispersion corrections: Cromer (1974).

Final atomic coordinates are listed in Table 1,* bonding distances and angles are given in Table 2. Fig. 1 gives the atom-numbering scheme and displays the differences in phenyl ring conformation for the two molecules.

Related literature. For related triaryl antimony derivatives see Sobolev, Romm, Belsky & Guryanova (1979), Sobolev, Romm, Belsky, Syutkina & Guryanova (1981). For other triphenyl Group V compounds see Daly (1964), Brock & Ibers (1973), Sobolev, Belsky, Chernikova & Akhamadulina (1983), Mazhar-ul-Haque, Tayim, Ahmed & Horne (1985).

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* Tables of anisotropic thermal parameters, H-atom coordinates, least-squares planes and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52445 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of Three Related Biphenyl Compounds: 4,4′-Biphenyldiol, 3,3′,5,5′-Tetra-*tert*-butyl-4,4′-biphenyldiol, and 3,3′,5,5′-Tetra-*tert*-butyl-1,1′-bicyclohexa-2,5-dienylidene-4,4′-dione

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Abstract. 4,4′-Biphenyldiol (I), $C_{12}H_{10}O_2$, M_r = 186·2, monoclinic, $P2_1/c$, a = 10·5512 (9), b = 5·359 (2), c = 7·9939 (8) Å, β = 95·736 (8)°, V = 449·7 (6) Å^3, Z = 2, D_x = 1·375 g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, μ = 7·13 cm^{-1}, F(000) = 196, T = 299 (1) K, R = 0·037 for 738 reflections with $I > 3\sigma(I)$ (919 unique observations). This is a redetermination; the original study was based on visually estimated film data, with R = 0·20 for 240 observations [Akhmed, Farag & Amin (1971). *J. Struct. Chem.* (*USSR*), **12**, 676–677; *Zh. Strukt. Khim.* **12**, 738–739]. The molecule is centrosymmetric, and the biphenyl ring system is planar, with average deviation 0·002 (1) Å. The O atom lies 0·013 (1) Å out of this plane, and the hydroxy H atom lies 0·26 (2) Å out of the plane. The central C—C bond has length 1·4954 (11) Å, and the C—O bond 1·3845 (12) Å. Chains of hydrogen bonds along the b direction exist, with O···O 2·8940 (8) Å and a 167 (2)° angle at H. 3,3′,5,5′-Tetra-*tert*-butyl-4,4′-biphenyldiol (II), $C_{28}H_{42}O_2$, M_r = 410·6, orthorhombic, $Fdd2$, a = 20·719 (3), b = 19·905 (3), c = 12·836 (2) Å, V = 5293·7 (14) Å^3, Z = 8, D_x = 1·030 g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, μ = 4·49 cm^{-1}, F(000) = 1808, T = 298 (1) K, R = 0·032 for 1031 reflections with $I > 3\sigma(I)$ (1457 unique observations). The long axis of the molecule lies along a twofold axis. The two phenyl rings are twisted slightly with respect to each other, forming a dihedral angle of 159·4 (2)°. One of the rings exhibits out-of-plane deviations of 0·010 (2) Å for its off-axis atoms, while these deviations are 0·001 (2) Å for the other ring. The central C—C bond of the molecule has length 1·486 (4) Å, with C—O bond lengths 1·385 (4) and 1·391 (3) Å. The C—C bonds of the *tert*-butyl groups are 1·527 (3)–1·545 (4) Å in length. Each of the hydroxy H atoms is disordered into two equally populated positions. 3,3′,5,5′-Tetra-*tert*-butyl-1,1′-bicyclohexa-2,5-dienylidene-4,4′-dione (III), $C_{28}H_{40}O_2$, M_r = 408·6, triclinic, $P\overline{1}$, a = 6·1004 (10), b = 10·4197 (11), c = 10·5260 (14) Å, α = 81·490 (8), β = 75·991 (11), γ = 81·582 (14)°, V = 637·8 (2) Å^3, Z = 1, D_x = 1·064 g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, μ = 4·65 cm^{-1}, F(000) = 224, T = 295 (1) K, R = 0·045 for 2199 reflections with $I > 3\sigma(I)$ (2628 unique observations). This determination confirms the recent determination of Khan, Osman & Tuck [*Acta Cryst.* (1986), C**42**, 1399–1402], which was based on 1131 observed data with Mo $K\alpha$ radiation. Although the present results represent a doubling of the precision, the agreement between the two determinations is excellent, with the largest single difference in a bond distance being only 0·008 Å (2σ).

Experimental. Intensity data for all three structures were obtained on a Enraf–Nonius CAD-4 diffractometer with a graphite incident-beam monochromator using Cu $K\alpha$ radiation. The ω–2θ scans were made at variable scan rates. Absorption corrections were based on ψ scans. All structures were solved by direct methods using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The structures were refined by full-matrix least squares with Enraf–Nonius *SDP*

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