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syn-2-Benzyl-1,3-dithiane 1-Oxide

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Abstract. $C_{11}H_{14}OS_2$, $M_r = 226.35$, orthorhombic, $P2_12_12_1$, $a = 7.598$ (3), $b = 25.635$ (2), $c = 5.774$ (3) Å, $V = 1124.6$ Å^3, $Z = 4$, $D_x = 1.337$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 4.20$ cm^{-1}, $F(000) = 480$, room temperature, $R = 0.033$ for 913 reflections with $I > 3\sigma(I)$. Bond lengths and angles are normal. Structure determination confirmed the constitution of a material prepared with 94% enantiomeric excess, by the sequential asymmetric oxidation and deacylation of 2-acetyl-2-benzyl-1,3-dithiane [Page & Namwindwa (1991). *Synth. Lett.* pp. 80–83].

Experimental. A yellow crystal, 0.15 × 0.35 × 0.45 mm, was analyzed using a Rigaku AFC-6*S* diffractometer with $\omega/2\theta$ scans. Unit-cell parameters were determined from 18 reflections with $20 < 2\theta < 33°$. No absorption correction was applied. $2\theta_{\max} = 50°$; $0 < h < 9$, $0 < k < 30$, $0 < l < 6$. Three standard reflections showed no significant change. 1209 reflections were measured (one unique set), of which 912 with $I > 3\sigma(I)$ were used for refinement. Structure solution was by direct methods (*SHELXS*86; Sheldrick 1986) and refinement on *F*, all within the *TEXSAN* system (Molecular Structure Corporation, 1985); anisotropic vibration parameters for non-H atoms, H atoms located in a difference map and refined with isotropic *U* values. For 183 parameters

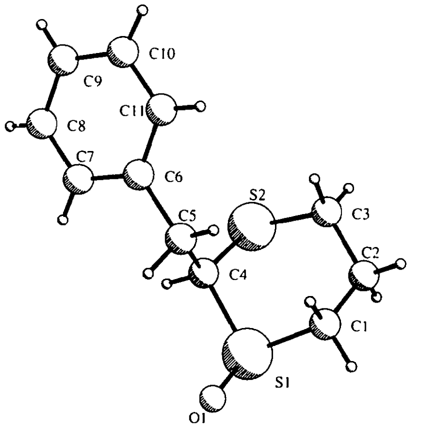

Fig. 1. One molecule of the title compound, illustrating the structure and the atom numbering (*PLUTO*; Motherwell & Clegg, 1978).

Table 1. *Fractional atomic coordinates and equivalent isotropic vibration parameters* (Å^2)

$$B_{eq} = 8\pi^2U_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
S(1)	0.5197 (1)	0.01780 (4)	0.7820 (2)	3.55 (5)
S(2)	0.4527 (2)	0.12721 (5)	0.8792 (2)	4.35 (6)
O(1)	0.6573 (4)	−0.0218 (1)	0.7247 (5)	4.5 (1)
C(1)	0.4237 (6)	0.0344 (2)	0.5054 (9)	3.9 (2)
C(2)	0.2885 (8)	0.0778 (2)	0.511 (1)	4.9 (3)
C(3)	0.3646 (7)	0.1292 (2)	0.588 (1)	4.9 (3)
C(4)	0.6274 (6)	0.0801 (2)	0.8456 (8)	3.3 (2)
C(5)	0.7776 (7)	0.0936 (2)	0.686 (1)	3.9 (2)
C(6)	0.8733 (5)	0.1429 (1)	0.7567 (8)	3.3 (2)
C(7)	0.9636 (7)	0.1459 (2)	0.961 (1)	4.4 (2)
C(8)	1.0527 (8)	0.1901 (2)	1.024 (1)	5.7 (3)
C(9)	1.0533 (8)	0.2328 (2)	0.882 (1)	6.6 (4)
C(10)	0.965 (1)	0.2308 (2)	0.676 (1)	7.0 (4)
C(11)	0.8745 (7)	0.1861 (2)	0.615 (1)	5.1 (3)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

S(1)—O(1)	1.495 (3)	C(5)—C(6)	1.514 (6)
S(1)—C(1)	1.807 (5)	C(6)—C(7)	1.370 (7)
S(1)—C(4)	1.832 (4)	C(6)—C(11)	1.378 (7)
S(2)—C(3)	1.813 (7)	C(7)—C(8)	1.368 (8)
S(2)—C(4)	1.805 (5)	C(8)—C(9)	1.37 (1)
C(1)—C(2)	1.514 (7)	C(9)—C(10)	1.37 (1)
C(2)—C(3)	1.506 (8)	C(10)—C(11)	1.382 (8)
C(4)—C(5)	1.508 (7)		

O(1)—S(1)—C(1)	104.3 (2)	C(4)—C(5)—C(6)	112.9 (4)
O(1)—S(1)—C(4)	108.9 (2)	C(5)—C(6)—C(7)	121.5 (4)
C(1)—S(1)—C(4)	98.7 (2)	C(5)—C(6)—C(11)	120.8 (4)
C(3)—S(2)—C(4)	101.0 (2)	C(7)—C(6)—C(11)	117.7 (4)
S(1)—C(1)—C(2)	115.3 (4)	C(6)—C(7)—C(8)	121.6 (5)
C(1)—C(2)—C(3)	112.8 (5)	C(7)—C(8)—C(9)	120.3 (6)
S(2)—C(3)—C(2)	112.9 (4)	C(8)—C(9)—C(10)	119.3 (6)
S(1)—C(4)—S(2)	106.0 (2)	C(9)—C(10)—C(11)	119.9 (6)
S(1)—C(4)—C(5)	114.5 (3)	C(6)—C(11)—C(10)	121.2 (6)
S(2)—C(4)—C(5)	118.0 (3)		

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and $w^{-1} = \sigma^2(F) + 0.0009F^2$, $R = 0.033$, $wR = 0.036$, $S = 1.25$, maximum shift/e.s.d. = 0.01, maximum and minimum peaks in the final difference map 0.49 and -0.43 e Å^{-3}, respectively. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A and 2.3.1). Coordinates for non-H atoms are recorded in Table 1* and selected bond distances and angles in Table 2. Fig. 1 shows the atom-numbering scheme.

Related literature. Other structures of substituted 1,3-dithiane 1-oxides have been reported (Bryan, Carey, Hernandez & Taylor, 1978; McPhail, Onan & Koskimies, 1976; Dhaneshwar, Menon, Tavale & Guru Row, 1989; Page, Prodger, Hursthouse & Mazid, 1990; Carey, Smith, Maher & Bryan, 1977).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55059 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0121]

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Structure of the 1:1 Complex Formed by Triphenylphosphine Oxide and 4-Nitrophenol

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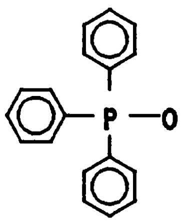
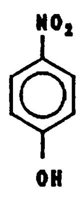
Abstract. $C_{18}H_{15}OP.C_6H_5NO_3$, $M_r = 417.38$, triclinic, $P\bar{1}$, $a = 8.633$ (1), $b = 9.966$ (2), $c = 13.317$ (4) Å, $\alpha = 92.01$ (2), $\beta = 102.90$ (2), $\gamma = 106.25$ (1)°, $V = 1066.4$ (5) Å^3, $Z = 2$, $D_m = 1.29$, $D_x = 1.300$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.15$ mm^{-1}, $F(000) = 436$, $T = 295$ K, $R = 0.0489$ for 1760 observed reflections. The P—O group of the triphenylphosphine oxide molecule is hydrogen bonded to the hydroxyl group of the 4-nitrophenol molecule with an O···O distance of 2.630 (4) Å. The packing of the system is such that the 4-nitrophenol molecules are distributed in layers having *ab* as the mean plane, alternating with layers of triphenylphosphine oxide molecules. In the latter layers, the P—O groups belonging to centrosymmetrically related triphenylphosphine oxide molecules point alternately up and down and make hydrogen bonds with 4-nitrophenol molecules belonging to consecutive layers.

Experimental. Solutions of triphenylphosphine oxide (TPPO) and 4-nitrophenol (NP) in chloroform were combined in a 1:1 molecular ratio of the solutes and left to evaporate slowly. Plate-shaped colorless crystals were obtained; m.p. = 376 (1) K; D_m measured by flotation in aqueous KI solution. A summary of data collection and structural refinement is given in Table 1. Lorentz and polarization corrections were applied but no absorption correction was made ($\mu = 0.15$ mm^{-1}). The structure was solved by super-



TPPO



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