

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  $-0.43 \text{ e } \text{Å}^{-3}$ , 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 &

\* 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]

Guru Row, 1989; Page, Prodger, Hursthouse & Mazid, 1990; Carey, Smith, Maher & Bryan, 1977).

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*Acta Cryst.* (1992). **C48**, 1690–1692

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

BY R. MORENO FUQUEN\* AND J. R. LECHAT

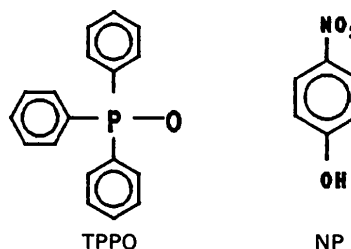
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**Abstract.**  $\text{C}_{18}\text{H}_{15}\text{OP}\cdot\text{C}_6\text{H}_5\text{NO}_2$ ,  $M_r = 417.38$ , triclinic,  $P\bar{1}$ ,  $a = 8.633$  (1),  $b = 9.966$  (2),  $c = 13.317$  (4) Å,  $\alpha = 92.01$  (2),  $\beta = 102.90$  (2),  $\gamma = 106.25$  (1)°,  $V = 1066.4$  (5) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.29$ ,  $D_x = 1.300 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.15 \text{ mm}^{-1}$ ,  $F(000) = 436$ ,  $T = 295 \text{ K}$ ,  $R = 0.0489$  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.

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**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 crys-



tals 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 ( $\mu = 0.15 \text{ mm}^{-1}$ ). The structure was solved by super-

position of the Patterson function using *SHELXS86* (Sheldrick, 1986). The structure was refined by blocked full-matrix least squares based on amplitudes by minimizing  $\sum w(|F_o| - |F_c|)^2$  where weighting  $w^{-1} = \sigma^2(F_o) + 0.00016|F_o|^2$  for observed and  $w = 0$  for unobserved reflections (*SHELX76*; Sheldrick, 1976). Positional and anisotropic thermal parameters for non-H atoms were refined. H atoms in the rings were introduced at calculated positions and the hydroxyl H(O2) was located by difference Fourier synthesis (C—H = 1.08 Å, H-atom temperature factor = 6 Å<sup>2</sup>). Atomic scattering factors for the non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970).

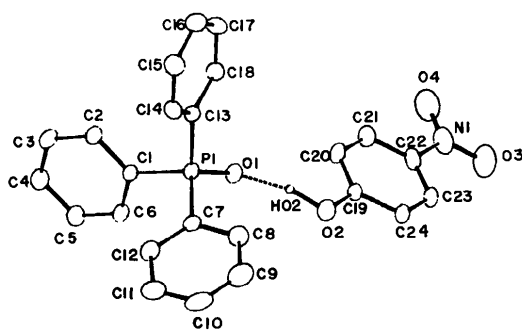


Fig. 1. ORTEP representation (Johnson, 1965) of the hydrogen-bonded complex, giving atomic numbering.

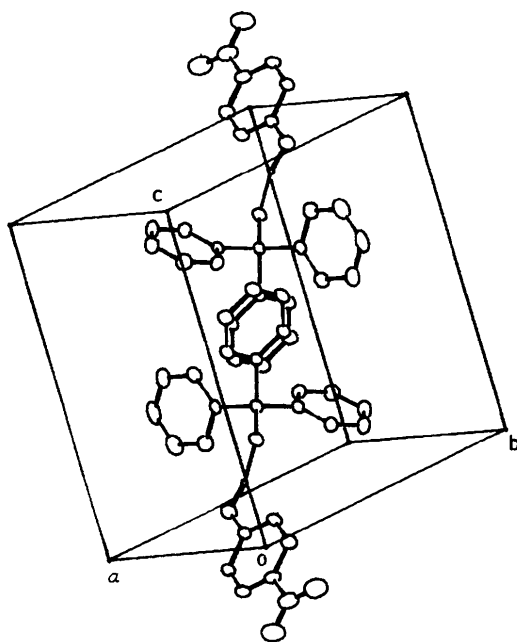


Fig. 2. ORTEP representation (Johnson, 1965) of the unit-cell contents.

Table 1. Crystallographic data

Crystal dimensions (mm)	0.30 × 0.20 × 0.25
Diffractometer used	Enraf-Nonius CAD-4 four-circle diffractometer; $\omega$ - $2\theta$
Number and $2\theta$ range (°) of reflections used for measuring lattice parameters	25, $16 \leq 2\theta \leq 35$
Check reflections and intensity variations (%) throughout experiment	$\bar{4}16$ , $\bar{4}21$ , 2.9, 3.2
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurements (Å <sup>-1</sup> )	0.548
Range of $hkl$ , minimum; maximum	-9, -10, 0; 9, 10, 14
Number of reflections measured	3167
Number of unique reflections	2930
Number of observed reflections	1760
Acceptance criterion for observed reflections	$I \geq 3\sigma(I)$
Parameters refined*	271
Values of $R_{int}$ , $R$ , $wR$	0.0279, 0.0489, 0.0509
Maximum and minimum residual densities (e Å <sup>-3</sup> ) in final difference map	0.306, -0.282
$(\Delta/\sigma)_{max}$	0.05
Goodness of fit, $S$	2.28
Computer programs used	<i>SHELX76</i> (Sheldrick, 1976), <i>SHELXS86</i> (Sheldrick, 1986), <i>ORTEP</i> (Johnson, 1965)

\* Refinement was performed in blocks, with parameters of TPPO and NP in alternate cycles.

Table 2. Fractional atomic coordinates and equivalent isotropic temperature factors (Å<sup>2</sup>) with e.s.d.'s in parentheses

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$B_{eq}$
P(1)	0.2134 (1)	0.2418 (1)	0.3433 (1)	3.46 (3)
O(1)	0.0738 (3)	0.1240 (3)	0.2802 (2)	4.41 (8)
O(2)	0.0514 (4)	-0.0666 (3)	0.1317 (2)	5.8 (1)
O(3)	-0.7120 (5)	-0.2812 (6)	-0.0550 (4)	10.4 (2)
O(4)	-0.6349 (5)	-0.4393 (5)	-0.1291 (4)	10.1 (2)
N(1)	-0.6054 (7)	-0.3328 (6)	-0.0698 (4)	7.6 (2)
C(1)	0.4110 (5)	0.2073 (4)	0.3612 (3)	3.8 (1)
C(2)	0.5256 (5)	0.2316 (5)	0.4532 (4)	5.6 (2)
C(3)	0.6768 (6)	0.2037 (6)	0.4598 (5)	7.0 (2)
C(4)	0.7110 (7)	0.1523 (6)	0.3747 (6)	7.0 (2)
C(5)	0.5965 (8)	0.1263 (7)	0.2836 (5)	8.3 (2)
C(6)	0.4455 (7)	0.1513 (6)	0.2743 (4)	6.5 (2)
C(7)	0.1854 (4)	0.2748 (4)	0.4709 (3)	3.3 (1)
C(8)	0.1804 (5)	0.4031 (5)	0.5102 (3)	4.2 (1)
C(9)	0.1544 (6)	0.4198 (5)	0.6088 (4)	5.4 (2)
C(10)	0.1321 (6)	0.3081 (6)	0.6660 (4)	5.3 (2)
C(11)	0.1344 (6)	0.1798 (5)	0.6274 (4)	5.4 (2)
C(12)	0.1614 (6)	0.1628 (5)	0.5303 (3)	4.8 (1)
C(13)	0.2322 (5)	0.4033 (4)	0.2859 (3)	3.5 (1)
C(14)	0.0884 (5)	0.4202 (5)	0.2220 (3)	4.5 (1)
C(15)	0.0956 (6)	0.5430 (6)	0.1765 (4)	5.6 (2)
C(16)	0.2427 (7)	0.6483 (6)	0.1929 (4)	5.9 (2)
C(17)	0.3851 (6)	0.6314 (5)	0.2537 (4)	5.7 (2)
C(18)	0.3789 (5)	0.5083 (5)	0.2993 (3)	4.6 (1)
C(19)	-0.1089 (5)	-0.1280 (4)	0.0820 (3)	4.3 (1)
C(20)	-0.1469 (6)	-0.2478 (5)	0.0124 (3)	5.0 (1)
C(21)	-0.3092 (7)	-0.3148 (5)	-0.0366 (3)	5.5 (1)
C(22)	-0.4320 (6)	-0.2626 (5)	-0.0170 (3)	5.2 (1)
C(23)	-0.3973 (6)	-0.1440 (5)	0.0491 (3)	5.6 (1)
C(24)	-0.2343 (6)	-0.0767 (5)	0.0993 (3)	5.1 (1)

Table 3. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses

P(1)—C(1)	1.799 (5)	P(1)—C(7)	1.801 (4)
P(1)—C(13)	1.788 (4)	P(1)—O(1)	1.492 (2)
C(19)—C(20)	1.400 (6)	C(20)—C(21)	1.368 (6)
C(21)—C(22)	1.373 (8)	C(22)—C(23)	1.368 (7)
C(23)—C(24)	1.377 (6)	C(19)—O(2)	1.348 (5)
C(22)—N(1)	1.461 (6)	N(1)—O(3)	1.220 (9)
N(1)—O(4)	1.231 (8)	C(19)—C(24)	1.378 (6)
O(1)—P(1)—C(1)	112.4 (2)	O(1)—P(1)—C(7)	111.9 (2)
O(1)—P(1)—C(13)	112.2 (2)	C(1)—P(1)—C(7)	106.4 (2)
C(1)—P(1)—C(13)	107.3 (2)	C(7)—P(1)—C(13)	106.3 (3)

**Related literature.** A view of the complex with the atomic numbering scheme is depicted in Fig. 1. The final atomic fractional coordinates\* with their standard deviations and equivalent isotropic thermal parameters (Hamilton, 1959) are given in Table 2. Selected bond lengths and bond angles are given in Table 3. Fig. 2 shows the contents of the cell. The packing differs from that observed for the complex formed by triphenylarsine oxide and 4-nitrophenol (Lariucci, de Almeida Santos & Lechat, 1986) in that it consists of alternate layers of NP and TPPO molecules parallel to the *ab* plane. This difference is attributed to the solvent used for crystallization and is reflected in the plate shape of the crystals, whereas complexes grown in less-polar or non-polar solvents usually have more uniform dimensions (Lariucci, de Almeida Santos & Lechat, 1986; Etter & Baures, 1988).

The molecular parameters of TPPO and NP in the complex are comparable to those reported for the

\* List of structure factors, anisotropic thermal parameters, bond distances and angles, and fractional H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55092 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0076]

free molecules (Bandoli, Bartolozzo, Clemente, Croatto & Panattoni, 1970; Spek, 1987).

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## Structure of 2,5:3,4-Dianhydro-D-altritol

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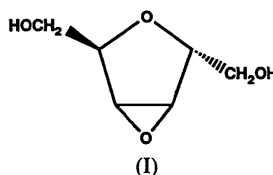
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(Received 23 October 1991; accepted 21 January 1992)

**Abstract.** 3,6-Dioxabicyclo[3.1.0]hexane-2,4-dimethanol, C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>, *M<sub>r</sub>* = 146.1, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>, *a* = 7.6209 (2), *b* = 9.1292 (3), *c* = 9.6135 (5) Å, *V* = 668.8 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.451 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 1.15 cm<sup>-1</sup>, *F*(000) = 312, *T* = 298 K, *R* = 0.029 for 1280 observations with *I* > 3σ(*I*) (of 1695 unique data). The tetrahydrofuran ring has the envelope conformation, <sup>o</sup>*E*, with *P* of 94.3° and τ<sub>*m*</sub> = 24.0°. C atoms deviate from their best plane by ± 0.006 (1) to 0.010 (1) Å, and the O atom lies 0.331 (1) Å from that plane. The epoxide O atom is *syn* to the tetrahydrofuran O atom. Each hydroxy group is involved in intermolecular hydrogen bond-

ing both as donor and acceptor. The two hydrogen bonds have O...O distances of 2.743 (1) and 2.729 (1) Å, and angles about H of 166.3 (12) and 172 (2)°, respectively.

**Experimental.** The title compound (I) was prepared as described in the literature (Guthrie, Jenkins, Waters, Wright & Yamasaki, 1982) from 2,5-anhydro-D-mannitol.



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