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Structure of a Second Monoclinic Polymorph of Triphenylphosphine Oxide

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Abstract. $C_{18}H_{15}OP$, $M_r = 278{\cdot}29$, monoclinic, $P2_1/c$, $a = 15{\cdot}066\ (1)$, $b = 9{\cdot}037\ (2)$, $c = 11{\cdot}296\ (3)$ Å, $\beta = 98{\cdot}47\ (1)°$, $U = 1521{\cdot}2\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}215$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}7$ cm^{-1}, $F(000) = 584$, $T = 295$ K, $R = 0{\cdot}045$ for 3128 reflections with $I > 2{\cdot}5\sigma(I)$. The torsion angles about the three Ph—P bonds in the second monoclinic polymorph of triphenylphosphine oxide deviate only slightly from those reported for the orthorhombic polymorph and significantly from those in the other monoclinic polymorph.

Experimental. A small batch of crystals of the title compound was obtained as the result of an attempt to crystallize a Pd-containing compound. The true composition of the crystals was established *via* an X-ray structure determination. X-ray data for a yellowish transparent crystal (0·25 × 0·62 × 0·85 mm) mounted on top of a glass fibre were collected on an Enraf–Nonius CAD-4F diffractometer using Zr-filtered Mo $K\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the setting angles of 15 reflections ($13 < \theta < 19°$). The space group was determined from the observed systematic absences. A total of 4687 unique reflections ($1{\cdot}37 < \theta < 30°$; $\omega/2\theta$-scan; $-21 \leq h \leq 20$, $-12 \leq k \leq 0$, $0 \leq l \leq 15$) was scanned. Three reference reflections (400, 0$\bar{2}$0, 002) showed variations less than 1·5% about the mean and no decay during the 76 h of X-ray exposure time. The data were corrected for Lp but not absorption. The 3128 reflections with $I > 2{\cdot}5\sigma(I)$ were used in the subsequent analysis. The structure was solved by Patterson methods and refined by full-matrix least squares on F (*SHELX*76; Sheldrick, 1976). H atoms were obtained from a difference map. Convergence with anisotropic thermal parameters for the non-hydrogen atoms and individual isotropic parameters for the hydrogen atoms was reached at $R = 0{\cdot}045$ [$wR = 0{\cdot}042$, $w = 1/\sigma^2(F)$, 3128 reflections, 242 parameters, $S = 0{\cdot}51$]. $(\Delta/\sigma)_{\max} = 0{\cdot}5$. A final difference Fourier map did not show features outside the range of $-0{\cdot}33$ and $0{\cdot}39$ e Å^{-3}. Scattering factors of Cromer & Mann (1968) were used. Calculations of the geometry and illustrations by programs of the *EUCLID* package (Spek, 1982). Fig. 1 shows the molecule with adopted numbering and Fig. 2 a projection of the unit-cell contents down the monoclinic b axis. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Data on the geometry are assembled in Table 2.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43763 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
P	0·23407 (3)	0·13743 (5)	0·26350 (4)	0·0431 (1)
O	0·22786 (8)	0·0818 (1)	0·38565 (9)	0·0571 (3)
C(1)	0·1364 (1)	0·2393 (2)	0·1987 (1)	0·0467 (4)
C(2)	0·1103 (1)	0·3600 (2)	0·2582 (2)	0·0840 (5)
C(3)	0·0318 (2)	0·4338 (3)	0·2162 (2)	0·1019 (5)
C(4)	−0·0213 (1)	0·3901 (3)	0·1159 (2)	0·0856 (4)
C(5)	0·0041 (1)	0·2727 (3)	0·0555 (2)	0·0896 (5)
C(6)	0·0823 (1)	0·1969 (2)	0·0957 (2)	0·0712 (4)
C(7)	0·3287 (1)	0·2597 (2)	0·2630 (1)	0·0434 (3)
C(8)	0·3351 (1)	0·3631 (2)	0·1743 (2)	0·0540 (4)
C(9)	0·4100 (1)	0·4519 (2)	0·1805 (2)	0·0748 (5)
C(10)	0·4780 (1)	0·4381 (2)	0·2747 (2)	0·0872 (5)
C(11)	0·4723 (1)	0·3377 (2)	0·3633 (2)	0·0797 (4)
C(12)	0·3982 (1)	0·2475 (2)	0·3589 (2)	0·0580 (4)
C(13)	0·2474 (1)	−0·0107 (2)	0·1607 (1)	0·0457 (3)
C(14)	0·2841 (1)	0·0087 (2)	0·0562 (2)	0·0603 (4)
C(15)	0·2918 (1)	−0·1103 (2)	−0·0185 (2)	0·0731 (4)
C(16)	0·2645 (1)	−0·2484 (2)	0·0106 (2)	0·0754 (4)
C(17)	0·2274 (1)	−0·2679 (2)	0·1125 (2)	0·0763 (4)
C(18)	0·2188 (1)	−0·1503 (2)	0·1875 (2)	0·0609 (4)
H(2)	0·1477 (7)	0·3940 (9)	0·3242 (8)	0·0778 (9)
H(3)	0·0258 (7)	0·5190 (9)	0·2600 (8)	0·0821 (9)
H(4)	−0·0768 (7)	0·4438 (8)	0·0897 (8)	0·0755 (9)
H(5)	−0·0296 (7)	0·2430 (9)	−0·0197 (8)	0·0816 (9)
H(6)	0·0977 (7)	0·1079 (8)	0·0557 (8)	0·0742 (9)
H(8)	0·2872 (7)	0·3756 (8)	0·1089 (8)	0·0684 (9)
H(9)	0·4159 (7)	0·5179 (9)	0·1183 (8)	0·0739 (9)
H(10)	0·5247 (7)	0·4974 (9)	0·2823 (8)	0·0799 (9)
H(11)	0·5157 (7)	0·3228 (8)	0·4263 (8)	0·0741 (9)
H(12)	0·3916 (7)	0·1714 (8)	0·4188 (8)	0·0687 (9)
H(14)	0·3038 (7)	0·1029 (9)	0·0372 (8)	0·0703 (9)
H(15)	0·3183 (7)	−0·0879 (8)	−0·0834 (8)	0·0731 (9)
H(16)	0·2693 (7)	−0·3324 (8)	−0·0389 (8)	0·0728 (9)
H(17)	0·2090 (7)	−0·3641 (8)	0·1377 (8)	0·0757 (9)
H(18)	0·1909 (7)	−0·1674 (8)	0·2515 (8)	0·0740 (9)

Table 2. *Data on the geometry of triphenylphosphine oxide: bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

P–O	1·484 (1)	C(16)–C(17)	1·363 (3)
P–C(1)	1·798 (2)	C(17)–C(18)	1·377 (3)
P–C(7)	1·804 (2)	C(13)–C(18)	1·381 (3)
P–C(13)	1·803 (2)	C(2)–H(2)	0·92 (1)
C(1)–C(2)	1·369 (3)	C(3)–H(3)	0·927 (9)
C(2)–C(3)	1·380 (3)	C(4)–H(4)	0·97 (1)
C(3)–C(4)	1·346 (3)	C(5)–H(5)	0·96 (1)
C(4)–C(5)	1·347 (4)	C(6)–H(6)	0·967 (8)
C(5)–C(6)	1·381 (2)	C(8)–H(8)	0·96 (1)
C(1)–C(6)	1·373 (2)	C(9)–H(9)	0·936 (9)
C(7)–C(8)	1·384 (3)	C(10)–H(10)	0·88 (1)
C(8)–C(9)	1·378 (2)	C(11)–H(11)	0·90 (1)
C(9)–C(10)	1·370 (3)	C(12)–H(12)	0·980 (9)
C(10)–C(11)	1·363 (3)	C(14)–H(14)	0·937 (9)
C(11)–C(12)	1·377 (2)	C(15)–H(15)	0·91 (1)
C(7)–C(12)	1·396 (2)	C(16)–H(16)	0·952 (8)
C(13)–C(14)	1·386 (2)	C(17)–H(17)	0·968 (8)
C(14)–C(15)	1·382 (3)	C(18)–H(18)	0·90 (1)
C(15)–C(16)	1·369 (3)		
O–P–C(1)	113·08 (6)	C(4)–C(5)–C(6)	121·1 (2)
O–P–C(7)	111·61 (6)	C(5)–C(6)–C(1)	120·8 (2)
O–P–C(13)	112·06 (6)	C(6)–C(1)–C(2)	117·4 (2)
C(1)–P–C(7)	106·55 (8)	C(7)–C(8)–C(9)	120·0 (2)
C(1)–P–C(13)	106·27 (7)	C(8)–C(9)–C(10)	120·0 (2)
C(7)–P–C(13)	106·84 (7)	C(9)–C(10)–C(11)	120·7 (2)
P–C(1)–C(2)	119·0 (1)	C(10)–C(11)–C(12)	120·4 (2)
P–C(1)–C(6)	123·4 (1)	C(11)–C(12)–C(7)	119·5 (2)
P–C(7)–C(8)	123·7 (1)	C(12)–C(7)–C(8)	119·4 (2)
P–C(7)–C(12)	116·9 (1)	C(13)–C(14)–C(15)	120·1 (2)
P–C(13)–C(14)	123·4 (1)	C(14)–C(15)–C(16)	120·5 (2)
P–C(13)–C(18)	118·0 (1)	C(15)–C(16)–C(17)	119·7 (2)
C(1)–C(2)–C(3)	120·7 (2)	C(16)–C(17)–C(18)	120·5 (2)
C(2)–C(3)–C(4)	121·4 (2)	C(17)–C(18)–C(13)	120·6 (2)
C(3)–C(4)–C(5)	118·6 (2)	C(18)–C(13)–C(14)	118·6 (2)
O–P–C(1)–C(2)	56·9 (2)	O–P–C(13)–C(18)	22·9 (1)
O–P–C(7)–C(12)	21·4 (2)		

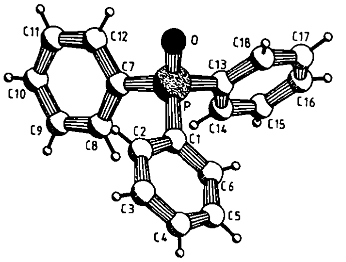

Fig. 1. View of the molecule with adopted numbering.

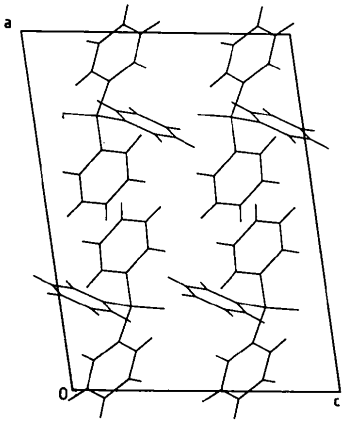

Fig. 2. Projection of the unit-cell contents down the monoclinic *b* axis.

Related literature. The crystal structures of an orthorhombic and a monoclinic modification of triphenylphosphine oxide have been published previously by Bandoli, Bortolozzo, Clemente, Croatto & Panattoni (1970) and Ruban & Zabel (1976) respectively. The present crystal structure concerns a second monoclinic modification and is of interest in view of chemical reaction path studies, such as the stereoisomerization path for triphenylphosphine oxide (Bye, Schweizer & Dunitz, 1982) and the related analysis of their internal molecular motion (Brock, Schweizer & Dunitz, 1985).

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Structure of Diazapolycyclic Compounds. X. 1,4-Dimethyl-1,4-dihydrobenzo[*g*]pyridazino[1,2-*b*]phthalazine-6,13-dione

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Abstract. $C_{18}H_{16}N_2O_2$, $M_r = 292{\cdot}3$, monoclinic, $P2_1/m$, $a = 9{\cdot}8193\ (4)$, $b = 13{\cdot}4046\ (6)$, $c = 5{\cdot}6336\ (1)$ Å, $\beta = 105{\cdot}083\ (2)°$, $U = 715{\cdot}97\ (5)$ Å^3, $Z = 2$, $D_x = 1{\cdot}355$ Mg m^{-3}, graphite-monochromatized Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}682$ mm^{-1}, $F(000) = 308$, $T = 293$ K, $R = 0{\cdot}057$ for 829 observed reflexions $[I > 3\sigma(I)]$. The pyridazine ring displays a boat conformation with the methyl groups in pseudoaxial positions. The bond angles around the N atoms sum to 360·0 (2)° confirming the sp^2 hybridization. The diazoquinone also has a boat conformation, less puckered than the pyridazine one.

Experimental. Yellow crystal, 0·47 × 0·17 × 0·05 mm, used for data collection and determination of lattice constants (Cu $K\alpha$, 63 reflexions, $2 < \theta < 45°$). Philips PW 1100 diffractometer, Cu $K\alpha$, graphite monochromator, bisecting geometry, $\omega/2\theta$ scan mode, 1278 unique reflexions up to $\theta = 65°$, in the *hkl* range −12,12; 0,16; 0,7. Two standard reflexions monitored every 90 minutes, no decay observed. The structure was solved in space group $P2_1$ by means of the *ORIENT* program (*DIRDIF* system, Beurskens *et al.*, 1984) using a three-ring-system model taken from *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain & Woolfson, 1980). Isotropic and anisotropic refinements were carried out in both space groups, $P2_1/m$ and $P2_1$, showing high correlation-matrix elements and unreasonable molecular geometry for the latter. H atoms, from difference synthesis, included isotropically in last cycles of refinement. Empirical weights so as to give no trends in $\langle w\Delta^2\rangle$ versus $\langle |F_o| \rangle$ and $\langle(\sin\theta)/\lambda\rangle$. $R(F) = 0{\cdot}057$, $wR = 0{\cdot}070$, $S = 1{\cdot}01$. Max. and average $\Delta/\sigma = 0{\cdot}23$ and $0{\cdot}03$. Final $\Delta\rho = \pm 0{\cdot}20$ e Å^{-3}. No extinction correction. Computing with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Scattering factors from *International Tables for X-ray Crystallography* (1974). Final atomic coordinates for the non-H atoms and the geometrical parameters are given in Tables 1 and 2,* according to the numbering scheme given in Fig. 1.

Related literature. 1-Methylated compound (Apreda, Foces-Foces, Cano & García-Blanco, 1978), unmethylated and 1,4-diphenyl compounds (Apreda, Foces-Foces, Cano & García-Blanco, 1982). The conformation of the pyridazine ring is shown in Fig. 2 for comparison.

We thank Dr F. Contreras for suggesting the problem and providing the material and Professor S. Garcia-Blanco for the facilities provided during this work.

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j\cos(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	$U_{\rm eq}$(Å^2 × 10^4)
C(1)	−0·0224 (3)	0·1418 (2)	−0·4847 (5)	476 (10)
C(2)	−0·0623 (3)	0·2012 (2)	−0·7147 (5)	529 (10)
C(10)	0·5633 (4)	0·1977 (5)	0·9260 (8)	1190 (28)
C(11)	0·4878 (4)	0·1452 (6)	0·7310 (8)	962 (23)
C(11a)	0·4064 (3)	0·1972 (3)	0·5174 (5)	707 (12)
C(12)	0·3263 (3)	0·1459 (3)	0·3122 (6)	649 (13)
C(12a)	0·2471 (3)	0·1973 (2)	0·1128 (5)	498 (9)
C(13)	0·1596 (3)	0·1426 (2)	−0·0978 (5)	516 (10)
N(14)	0·0739 (2)	0·1974 (2)	−0·2805 (4)	461 (8)
O(16)	0·1588 (2)	0·0507 (2)	−0·1096 (4)	702 (9)
C(17)	−0·1513 (4)	0·1051 (3)	−0·4058 (7)	607 (12)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43668 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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