

Table 1;* the bond distances, bond angles and relevant torsion angles are presented in Table 2. Atomic numbering is shown in Fig. 1, and the packing diagram is presented in Fig. 2.

Related literature. The torsion angles around the hexahydropyrimidine ring alternate in sign and range from 53.2 (2) to 62.4 (2)° (Table 2) with a mean value of 57.9 (2)°. This value is the same as that found for the tetrahydro-1,3-oxazine ring (Shoja & Saba, 1991). A mean value of 55.9° was observed for cyclohexane (Clark & McKerver, 1979). The cyanomethyl groups are perpendicular to the mean plane of the heterocyclic ring with an average dihedral angle of 88.9 (1)°.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55459 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0398]

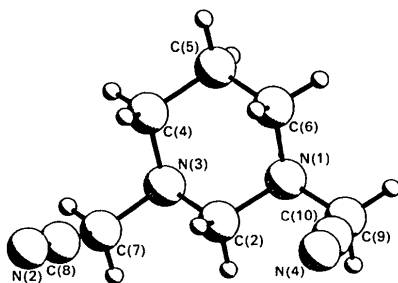


Fig. 1. Numbering of atoms and conformation of the molecule.

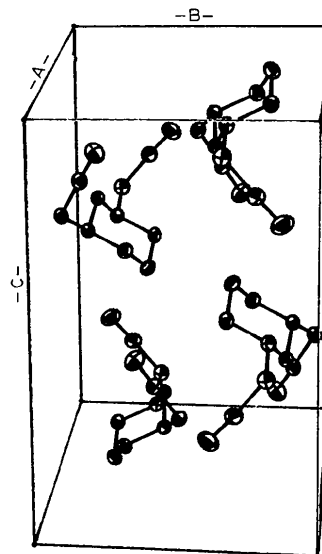


Fig. 2. Molecular packing diagram.

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Acta Cryst. (1993). C49, 355–357

Structure of Orthorhombic Triphenylphosphine Oxide: a Redetermination at Room Temperature*

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(Received 17 March 1992; accepted 8 June 1992)

Abstract. C₁₈H₁₅OP, *M_r* = 278.3, orthorhombic, *Pbca*, *a* = 29.085 (15), *b* = 9.149 (4), *c* = 11.266 (5) Å, *V* = 2997.9 Å³, *Z* = 8, *D_x* = 1.233 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.170 cm⁻¹, *F*(000) = 1168, *T* =

295 K, *R* = 0.048 for 1156 observed reflections. The P—O and three P—C bond lengths are 1.487 (3), and 1.795 (5), 1.799 (5) and 1.804 (5) Å, respectively. The valence angles at P fall into two categories: the three O—P—C angles, 111.8 (2), 112.0 (2) and 113.3 (2)°, and the three C—P—C angles, each significantly smaller at 106.4 (2)°.

* Contribution from the Crystallography Unit, Universities of Aston and Birmingham, England.

Table 1. Fractional atomic coordinates ($\times 10^4$, $P \times 10^5$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

$U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} tensor).				
	x	y	z	U_{eq}
P(1)	11874 (5)	13767 (14)	21413 (12)	46
O(1)	1163 (1)	825 (3)	3381 (3)	62
C(1)	1262 (2)	-89 (5)	1087 (5)	45
C(2)	1445 (2)	88 (8)	-27 (6)	64
C(3)	1500 (3)	-1093 (8)	-762 (6)	81
C(4)	1374 (2)	-2473 (8)	-409 (7)	75
C(5)	1187 (3)	-2652 (7)	674 (6)	74
C(6)	1129 (2)	-1489 (6)	1434 (5)	61
C(7)	680 (2)	2356 (5)	1690 (4)	45
C(8)	402 (2)	1941 (7)	777 (6)	73
C(9)	0 (2)	2693 (10)	531 (7)	100
C(10)	-122 (2)	3887 (9)	1190 (8)	90
C(11)	151 (3)	4332 (8)	2078 (8)	98
C(12)	556 (2)	3586 (9)	2317 (7)	93
C(13)	1662 (2)	2602 (5)	1927 (4)	43
C(14)	1691 (2)	3578 (7)	980 (5)	57
C(15)	2079 (3)	4413 (7)	829 (7)	83
C(16)	2440 (3)	4274 (9)	1586 (9)	100
C(17)	2418 (2)	3342 (10)	2514 (7)	89
C(18)	2029 (2)	2475 (7)	2697 (6)	65

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

P(1)—O(1)	1.487 (3)	C(7)—C(12)	1.377 (8)
P(1)—C(1)	1.804 (5)	C(8)—C(9)	1.384 (8)
P(1)—C(7)	1.799 (5)	C(9)—C(10)	1.368 (10)
P(1)—C(13)	1.795 (5)	C(10)—C(11)	1.341 (9)
C(1)—C(2)	1.372 (7)	C(11)—C(12)	1.388 (8)
C(1)—C(6)	1.395 (7)	C(13)—C(14)	1.394 (6)
C(2)—C(3)	1.371 (8)	C(13)—C(18)	1.381 (7)
C(3)—C(4)	1.373 (9)	C(14)—C(15)	1.373 (8)
C(4)—C(5)	1.346 (8)	C(15)—C(16)	1.359 (10)
C(5)—C(6)	1.376 (8)	C(16)—C(17)	1.351 (10)
C(7)—C(8)	1.363 (7)	C(17)—C(18)	1.398 (8)
O(1)—P(1)—C(1)	111.8 (2)	P(1)—C(7)—C(12)	118.4 (4)
O(1)—P(1)—C(7)	113.3 (2)	C(8)—C(7)—C(12)	117.3 (5)
C(1)—P(1)—C(7)	106.4 (2)	C(7)—C(8)—C(9)	121.0 (7)
O(1)—P(1)—C(13)	112.0 (2)	C(8)—C(9)—C(10)	120.5 (7)
C(1)—P(1)—C(13)	106.4 (2)	C(9)—C(10)—C(11)	119.5 (7)
C(7)—P(1)—C(13)	106.4 (2)	C(10)—C(11)—C(12)	119.9 (7)
P(1)—C(1)—C(2)	124.1 (4)	C(7)—C(12)—C(11)	121.6 (7)
P(1)—C(1)—C(6)	117.7 (4)	P(1)—C(13)—C(14)	123.3 (4)
C(2)—C(1)—C(6)	118.2 (5)	P(1)—C(13)—C(18)	117.3 (5)
C(1)—C(2)—C(3)	120.3 (7)	C(14)—C(13)—C(18)	119.3 (5)
C(2)—C(3)—C(4)	121.3 (7)	C(13)—C(14)—C(15)	120.0 (6)
C(3)—C(4)—C(5)	118.8 (7)	C(14)—C(15)—C(16)	120.4 (7)
C(4)—C(5)—C(6)	121.3 (7)	C(15)—C(16)—C(17)	120.5 (7)
C(1)—C(6)—C(5)	120.1 (6)	C(16)—C(17)—C(18)	120.7 (7)
P(1)—C(7)—C(8)	124.2 (5)	C(13)—C(18)—C(17)	119.1 (7)

Experimental. Crystals were grown from dichloromethane/hexane. A crystal of size $0.15 \times 0.5 \times 0.5$ mm was mounted on an Enraf-Nonius CAD-4 diffractometer and cell dimensions determined from the setting angles of 24 reflections in the range $9.5 < \theta < 13^\circ$. Intensity data were measured with graphite-monochromated Mo $K\alpha$ radiation. 4382 reflections were collected by $\omega/2\theta$ scans up to $\theta = 27.5^\circ$, with index range $h - 1$ to 37, $k - 1$ to 11, $l - 1$ to 14. 3434 reflections were unique ($R_{int} = 0.050$), of which 1156 with $I > 2.5\sigma(I)$ were considered observed and used in the analysis. Three standard reflections measured every 2 h showed no significant variation in intensity.

The structure was refined by least squares using anisotropic thermal parameters for the heavier atoms. H atoms were located from a difference map and refined isotropically. Weights $w = 1/[\sigma^2(F)]$, which resulted in a satisfactory weighting analysis, were used in the least-squares refinement. The refinement was carried out on F values and converged to $R = 0.048$, $wR = 0.040$ with maximum shift/e.s.d. ratio < 0.05 . The residual electron density in a final difference map was within $\pm 0.27 e \text{\AA}^{-3}$. An empirical absorption correction, using *DIFABS* (Walker & Stuart, 1983), was applied to the data (transmission factors in the range 0.577–0.717). Two large low-angle reflections which were affected by extinction ($400 F_o = 167.8$, $F_c = 179.5$, and $800 F_o = 261.0$, $F_c = 275.2$) were removed from the final cycles of refinement.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were carried out largely on the University of Birmingham IBM 3090 computer with the *SHELX76* program (Sheldrick, 1976). The diagram was drawn with *PLUTO* (Motherwell & Clegg, 1978) as implemented at the Manchester Computing Centre.

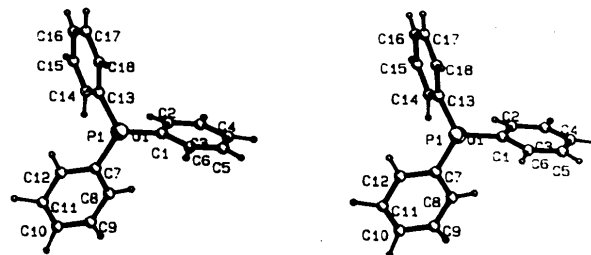


Fig. 1. Stereoscopic view of the molecule along the O—P bond direction showing the atom-numbering scheme.

The molecular structure and atom numbering are shown in Fig. 1. Atomic coordinates are listed in Table 1,* and bond lengths and angles in Table 2.

Related literature. The P—C and P—O bond lengths are longer than the anomalously short 1.75 (1), 1.77 (1) and 1.77 (1) \AA lengths for P—C and 1.46 (1) \AA for P—O measured in the original relatively low-resolution ($d_{min} 0.977 \text{\AA}$) determination (Bandoli, Bortolozzo, Clemente, Croatto & Panattoni, 1970). Our values for these lengths are in good agreement with the results of low-temperature studies (Brock, Schweizer & Dunitz, 1985) and afford a valid room-temperature comparison with the low-temperature work. P—C and P—O lengths similar to our values have also been found in 2-diphenylphosphinoyloxaziridines (Boyd, Malone, McGuckin, Jennings, Rutherford & Sacket, 1988; Cook, Hamor, Jennings, Tebbutt, Watson & Boyd, 1991). However,

* 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 55461 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1009]

for trivalent phosphorus in 2-[Ph₂P(CH₂)₃N=CH]C₆H₄OH the comparable P—C(phenyl) bonds are longer at 1.828 (7) and 1.842 (6) Å (Banbery, Hussain, Hamor, Jones & McCleverty, 1990). The difference between the C—P—C angles (mean 106.4°) and the O—P—C angles [mean 112.4 (5)°] is presumably a result of the repulsive effect of the short electron-rich P—O double bond. The phenyl C—C bonds adjacent to the P—C bond tend to be longer than those away from the centre of the molecule. A similar, but smaller effect was noted by Brock *et al.* (1985) and was considered to be a genuine molecular phenomenon, rather than an artefact caused by molecular libration. Our results would, however, indicate that thermal libration is probably the prime cause for the apparent bond-length variation.

Acta Cryst. (1993). **C49**, 357–359

5,7-Dihydroxy-2-(2,3,4-trimethoxyphenyl)-4H-1-benzopyran-4-one (5,7-Dihydroxy-2',3',4'-trimethoxyflavone)

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(Received 4 March 1992; accepted 23 June 1992)

Abstract. C₁₈H₁₆O₇, *M*_r = 344.32, orthorhombic, *Pna*2₁, *a* = 7.768 (1), *b* = 26.621 (3), *c* = 7.467 (1) Å, *V* = 1544.1 (6) Å³, *Z* = 4, *D*_x = 1.481 g cm⁻³, λ(Cu *K*α) = 1.54184 Å, μ = 9.26 cm⁻¹, *F*(000) = 720, *T* = 291 K, *R* = 0.044 for 1551 observed reflections. The molecule is not planar, the dihedral angle between the phenyl ring and the γ -benzopyrone portion is O(1)—C(2)—C(1')—C(6') = -34.5 (5)°. There is an intramolecular hydrogen bond between the carbonyl O atom and the O atom of the 5-hydroxy group, O(4)⋯O(3) = 2.586 Å. This carbonyl O atom can form a second intermolecular hydrogen bond with the O atom of the 7-hydroxy group O(2)⋯O(4) = 2.759 (5) Å. The two *ortho*-substituted methoxy groups have an out-of-plane conforma-

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tion C(3')—C(2')—O(5)—C(15) = 70.6 (5)°, C(4')—C(3')—O(6)—C(14) = 73.4 (5)°. The third methoxy group is in the plane of the phenyl ring, C(5')—C(4')—O(7)—C(13) = 4.8 (6)°.

Experimental. The title compound (m.p. 428 K) was synthesized from 2,3,4-trimethoxybenzoic acid and 2,4,6-trihydroxyacetophenone (Gaydou & Bianchini, 1978). Good-quality crystals were obtained by slow evaporation from ethanol solution. *D*_m not measured. Parallelepiped crystal with approximate dimensions 0.2 × 0.3 × 0.3 mm. Lattice parameters refined using 25 reflections in the range 25.35 ≤ θ ≤ 33.55°. Enraf-Nonius CAD-4 diffractometer, Ni-filtered Cu *K*α radiation, ω/2θ scan. 3329 *hkl* measured reflections with sinθ/λ ≤ 0.61 Å⁻¹, 0 ≤ *h* ≤ 9, 0 ≤ *k* ≤ 32, 0 ≤ *l* ≤ 9 and *I* ≥ 3σ(*I*). Five reflections checked every 300 reflections, no significant deviation. Structure solved by

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