Related literature. For the preparation and characterization of the compound see Mancera, Rodríguez, Roffe & Galbis (1988). The title compound is an intermediate product in the synthesis of pyrazoles with the sugar nitro-olefins as dipolarophiles. Great difficulty is encountered in the crystallization of these compounds and crystals are poor in quality. Recently the structure of a related compound has been reported (Conde & Conde, 1989).

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Structure of Triphenylphosphine Oxide Hemihydrate

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Abstract. $C_{18}H_{15}OP.\frac{1}{2}H_2O$, $M_r = 287.30$, orthorhombic, $Fdd2$, $a = 19.794 (18)$, $b = 32.540 (12)$, $c = 9.459 (6)$ Å, $V = 6092.5 (22)$ Å^3, $Z = 16$, $D_x = 1.253$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.76$ cm^{-1}, $F(000) = 2416$, $T = 294$ K, $R = 0.037$ for 1677 reflections with $I > 3\sigma(I)$. The water O atoms sit on a crystallographic twofold axis and are linked by hydrogen bonds to two of the oxide molecules with O···OW distances of 2.856 (3) Å. Considerable conformational flexibility is conferred on the molecule by the long P—C bonds (around 1.8 Å each).

Experimental. Two independent experiments intended to produce cocrystallization of triphenylphosphine oxide (TPPO) with a catechol-related compound (carried out by PWB) and racemic phenylalanine (carried out by JVS) gave crystals different from those of the reactants. In the latter case, mass spectroscopy of crystals of the hemihydrate obtained from 1:1 dimethylformamide/water indicated a molecular ion (*m/e*: 278 kg C^{-1}) corresponding to TPPO. Since the unit-cell dimensions

had not been reported previously, crystal-structure determinations were undertaken independently.† Essentially identical results were obtained. PWB used Mo $K\alpha$ X-radiation and his determination involved more measurements and has lower e.s.d.'s than that of JVS who used Cu $K\alpha$ X-radiation. Since there are no inexplicable differences, the results to be described here are those of PWB. A poorly formed block measuring $0.80 \times 0.70 \times 0.50$ mm was carefully carved away from a mass of crystals and was mounted on a glass fiber using an epoxy resin. X-ray data for this crystal were collected on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the setting angles of 25 reflections ($30 < 2\theta < 37°$). The space group was determined from the systematic absences (*hkl*: $h + k$, $h + l$, $k + l = 2n$; 0*kl*: $k + l = 4n$, $k,l = 2n$; *h0l*: $h + l = 4n$, h, $l = 2n$; *hk0*: $h,k = 2n$). 2037 unique reflections ($2 < 2\theta < 56°$; $0 < h < 26$, $0 < k < 43$, $0 <$

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† The structure of TPPO hemihydrate had been solved in the maximal non-isomorphous subgroup *Cc* by Israel Goldberg with unpublished results (Etter, 1989).

Table 1. Final positional parameters with e.s.d.'s in parentheses

	x	y	z	$B^*_{eq}(\text{Å}^2)$
P	0.62005 (3)	0.18591 (2)	0.0252 (1)	3.72 (1)
OW	0.500	0.000	0.500	9.8 (1)
O	0.6613 (1)	0.21966 (6)	−0.0358 (3)	5.95 (5)
C1	0.6298 (1)	0.13899 (8)	−0.0732 (3)	3.77 (5)
C2	0.5779 (2)	0.11031 (8)	−0.0929 (3)	4.60 (6)
C3	0.5908 (2)	0.07394 (9)	−0.1656 (4)	5.40 (7)
C4	0.6535 (2)	0.0657 (1)	−0.2160 (4)	5.83 (7)
C5	0.7059 (2)	0.0932 (1)	−0.1969 (4)	6.02 (7)
C6	0.6935 (1)	0.1305 (1)	−0.1264 (3)	4.81 (6)
C7	0.6447 (1)	0.17427 (9)	0.2046 (3)	4.51 (5)
C8	0.6218 (2)	0.1395 (1)	0.2738 (4)	5.96 (7)
C9	0.6404 (2)	0.1321 (1)	0.4122 (5)	8.7 (1)
C10	0.6832 (2)	0.1597 (2)	0.4802 (5)	11.2 (1)
C11	0.7056 (2)	0.1935 (2)	0.4121 (5)	10.6 (1)
C12	0.6877 (2)	0.2019 (1)	0.2711 (5)	6.85 (8)
C13	0.5318 (1)	0.19863 (7)	0.0339 (3)	3.52 (4)
C14	0.4933 (2)	0.19950 (9)	−0.0884 (3)	4.73 (6)
C15	0.4272 (2)	0.2131 (1)	−0.0844 (4)	5.88 (7)
C16	0.3996 (2)	0.2265 (1)	0.0411 (4)	5.64 (7)
C17	0.4371 (2)	0.2262 (1)	0.1638 (4)	5.19 (7)
C18	0.5032 (2)	0.21210 (9)	0.1604 (3)	4.54 (6)

*Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33}]$.

Table 2. Geometries of the TPPO fragment: bond distances (Å) and angles (°) with e.s.d.'s in parentheses

P—O	1.487 (2)	C8—C9	1.381 (6)
P—C1	1.797 (3)	C9—C10	1.392 (7)
P—C7	1.806 (3)	C10—C11	1.350 (7)
P—C13	1.796 (2)	C11—C12	1.407 (6)
C1—C2	1.401 (4)	C13—C14	1.385 (4)
C1—C6	1.384 (4)	C13—C18	1.394 (4)
C2—C3	1.391 (4)	C14—C15	1.381 (5)
C3—C4	1.356 (5)	C15—C16	1.377 (5)
C4—C5	1.382 (5)	C16—C17	1.377 (5)
C5—C6	1.406 (5)	C17—C18	1.386 (4)
C7—C8	1.384 (5)		
C7—C12	1.387 (5)		
O—P—C1	111.6 (1)	P—C7—C12	117.1 (3)
O—P—C7	111.8 (1)	C8—C7—C12	121.1 (3)
O—P—C13	112.5 (1)	C7—C8—C9	120.3 (3)
C1—P—C7	106.2 (1)	C8—C9—C10	119.1 (4)
C1—P—C13	108.8 (1)	C9—C10—C11	120.4 (4)
C7—P—C13	105.6 (1)	C10—C11—C12	121.8 (4)
P—C1—C2	123.8 (2)	C7—C12—C11	117.3 (3)
P—C1—C6	117.0 (2)	P—C13—C14	120.1 (2)
C2—C1—C6	119.1 (3)	P—C13—C18	120.4 (2)
C1—C2—C3	119.8 (3)	C14—C13—C18	119.1 (2)
C2—C3—C4	120.6 (3)	C13—C14—C15	120.3 (3)
C3—C4—C5	120.8 (3)	C14—C15—C16	120.0 (3)
C4—C5—C6	119.4 (3)	C15—C16—C17	120.6 (3)
C1—C6—C5	120.2 (3)	C16—C17—C18	119.4 (3)
P—C7—C8	121.9 (2)	C13—C18—C17	120.5 (3)

$l < 12$) were collected using variable speed ω–2θ scans. Three reflections ($\overline{11},15,\overline{3}$, $14,\overline{2},\overline{4}$, $8,10,6$) showed a 6.8% decrease in intensity over 25.5 h of exposure time. The data were corrected for this decay using the linear *DECAY* program with a minimum correction of 1.000 and a maximum correction of 1.036 (unless mentioned otherwise, all programs used were from a locally modified version of the Enraf–Nonius *SDP* package). The structure was solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and

refined by full-matrix least-squares refinement on *F*. The 1677 reflections with $I > 3\sigma(I)$ were used in the subsequent analysis. H atoms were fixed at calculated positions (C—H 1.0 Å) and assigned isotropic thermal parameters equal to 1.3 times that of their non-H-atom counterparts except for the water H atom which was located in a difference Fourier map and fixed at this location with an isotropic thermal parameter of 1.3 times that of the water O atom.

An empirical absorption correction was applied using the *DIFABS* program with a θ dependent correction (Walker & Stuart, 1983). The minimum correction was 0.613 and the maximum correction was 1.145. An empirical extinction correction was applied and refined to a value of 3.7×10^{-7}. Convergence with anisotropic thermal parameters for the non-H atoms was achieved at $R = 0.037$, $wR = 0.051$,
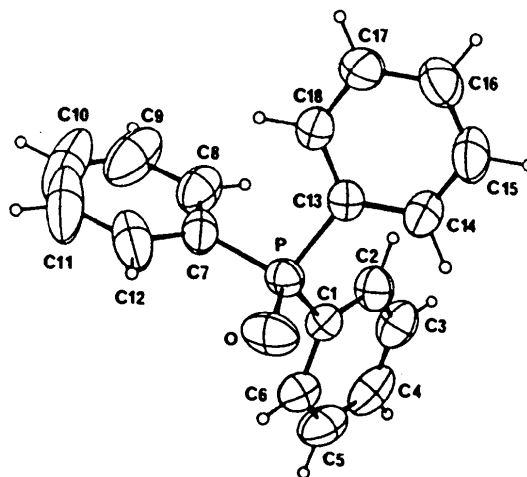
Fig. 1 An *ORTEP* drawing of the TPPO molecule. Thermal ellipsoids are drawn at the 50% level. The water of hydration has been omitted.
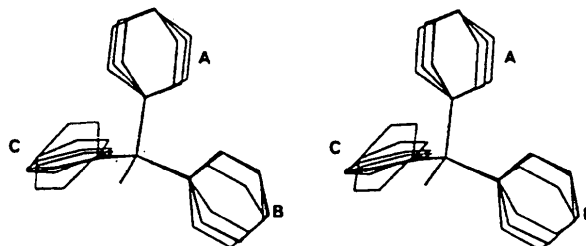
Fig. 2. Stereoview of the TPPO fragment from this structure overlayed with the three TPPO polymorphs by matching the largest torsion angle in each structure. The dihedral angles between the planes of the rings and the plane which is formed from the *ipso* C atom to the P and the O atoms are as follows:

	A	B	C	Reference
Pbca	59.3	21.1	24.7	Bandoli et al. (1970)
P2₁/a	76.9	11.4	18.7	Ruban & Zabel (1976)
P2₁/c	56.9	21.4	22.9	Spek (1987)
Fdd2	77.3	36.3	11.9	Present work

$w = 1/\sigma^2(F)$ with $\sigma^2(F)$ given by the expression $[\sigma^2(I_c) + (0.04F)^2]^{1/2}$, 1677 reflections, 186 variables, $S = 1.847$, $(\Delta/\sigma)_{max} < 0.0$. A final difference Fourier map showed no features outside the range of -0.17 and 0.20 e Å^{-3}. Values of the neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the molecule and Fig. 2 is a stereoview of an overlay between this TPPO fragment and the three known TPPO polymorphs. The long P—C bonds allow considerable conformational flexibility and, in the present case, two independent rings are forced into near parallelism by the hydrogen bonding.

Related literature. TPPO has been shown to be useful for the formation of cocrystalline solids which are often better formed and of higher quality than the substrates themselves (Etter & Baures, 1988). There are three known polymorphs of TPPO, an orthorhombic form (space group *Pbca*) reported by Bandoli, Bortolozzo, Clemente, Croatto & Panattoni (1970), a monoclinic modification (space group

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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 52439 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$P2_1/a$) by Ruban & Zabel (1976) and a second monoclinic modification (space group $P2_1/c$) by Spek (1987). The rotation angles for the three phenyl rings fall within the range for the stereoisomerism of triphenylphosphine oxide fragments and related compounds (Bye, Schweizer & Dunitz, 1982).

PWB thanks Dr Drake Eggleston (Smith Kline and French Laboratories) for his assistance in the structure determination and JVS thanks Mr William Comstock (NHLBI-LC) for mass spectroscopy.

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Structure of a New Form of Betaine Hydrochloride

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Abstract. 1-Carboxy-N,N,N-trimethylmethanaminium chloride, $C_5H_{12}NO_2^+.Cl^-$, $M_r = 153.63$, monoclinic, Cm, $a = 10.578$ (1), $b = 6.9383$ (8), $c = 5.7052$ (8) Å, $\beta = 111.45$ (1)°, $V = 389.72$ (8) Å^3, $Z = 2$, $D_m = 1.357$, $D_x = 1.309$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 4.24$ cm^{-1}, $F(000) = 164$, $T = 294$ K, $R_F = 0.048$ for 820 independent reflections. The packing mode in this new form (I) is different from that in the known form (II) which belongs to space group $P2_1/c$ with $V = 776$ Å^3 and $Z = 4$ [Fischer,

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Templeton & Zalkin (1970). *Acta Cryst.* B26, 1392–1397]. Although the molecule, which contains an O—H⋯Cl hydrogen bond, occupies a site of symmetry m in (I) and has only approximate mirror symmetry in (II), the measured bond lengths and angles in both crystalline forms agree with one another within experimental error.

Experimental. In an attempt to prepare a tin complex of betaine (1-carboxy-N,N,N-trimethylmethanaminium hydroxide inner salt), $(CH_3)_3NCH_2COO$, $SnCl_4.5H_2O$ (0.8972 g, 2.56 mmol) and betaine