C(1)—C(4) and C(1′)—C(4′) being inclined to the inter-ring axis C(1)—C(1′) at 0·8 (9) and 2·6 (9)° respectively, with C(4) and C(4′) *cis*. A similar distortion was found in 2*H*,2′*H*-octafluorobiphenyl (Bowen Jones & Brown, 1982), and in both molecules the atoms C(4), C(1), C(1′) and C(4′) are coplanar, none being displaced by more than 0·005 (4) Å from their least-squares plane.

This analysis has shown that 2-substitution of perfluorobiphenyl with bromine increases the dihedral angle from 59·6° (Gleason & Britton, 1976) to 79·6 (10)°. This is a slightly larger increase than that produced by 2,2′-disubstitution with bromine (Hamor & Hamor, 1980). In contrast, neither of these substitutions has a significant effect on the interannular distance, which has been found to be virtually invariant and independent of dihedral angle in a number of biphenyl systems (Goodhand, Hamor & Hamor, 1978; Bowen Jones & Brown, 1982). The structural analysis of 2-bromononafluorobiphenyl corroborates these findings.

We thank our colleague, Dr A. G. Massey, for suggesting this investigative programme and for providing a sample of the title compound.

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Structure of the 1:1 Adduct Formed by Diphenylmethanol with Triphenylphospine Oxide

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Abstract. $C_{13}H_{12}O.(C_6H_5)_3PO$, $M_r = 462{\cdot}53$, monoclinic, $P2_1/n$, $a = 11{\cdot}074$ (4), $b = 18{\cdot}081$ (2), $c = 12{\cdot}887$ (4) Å, $\beta = 95{\cdot}13$ (3)°, $V = 2570$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}202$ (2) (flotation), $D_x = 1{\cdot}195$ (2) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}95$ cm^{-1}, $F(000) = 976$, room temperature. Direct methods, final $R = 0{\cdot}070$ for 2060 observed reflections. Phenyl rings of diphenylmethanol are disordered. The adduct is formed by hydrogen bonding between the O atoms of the phosphine oxide and alcohol, with an O···O distance of 2·672 (5) Å. The P=O distance [1·482 (4) Å] in triphenylphosphine oxide is significantly longer than in the free molecule [1·46 (1) Å].

Introduction. It was shown by Lechat (1984) that the hydrogen-bond distance in adducts formed by strong and medium hydrogen bonds, where the hydrogen-bond donor and acceptor atoms are O atoms, may be correlated with the values of the difference of pK_a of the acids and bases involved in the adducts. This crystal-structure determination has been undertaken in order to gather structural information for an adduct presenting a unique hydrogen bond of medium strength.

Experimental. Transparent colourless crystals were obtained by slowly evaporating a solution of the title compound in benzene. Technique as well as reagents were provided by Tomita (1972). Crystal of dimensions 0·6 × 0·5 × 0·3 mm used for data collection on an Enraf–Nonius CAD-4 diffractometer. Cell parameters refined from setting angles of 25 reflections in θ range 11·0–18·5°. Three-dimensional intensity data collected using graphite-monochromated Mo $K\alpha$ radiation. ω–2θ scanning mode with varying interval used up to $2\theta = 46°$. Reflections collected in the range $-12 \rightarrow 12$ for h, $0 \rightarrow 19$ for k and $0 \rightarrow 14$ for l. 3570 recorded independent reflections, only 2060 considered above background $[I > 3\sigma(I)$, where $\sigma(I)$ based on counting statistics]. Three standard reflections were measured every 1800 s and presented a maximum 4% variation throughout experiment. Data were reduced to structure factors without absorption correction. Structure was solved by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All non-H atoms appeared clearly in E map based on set of phases giving highest combined figure of merit. H atoms, except hydroxyl H atom, placed at their calculated positions.

Table 1. *Fractional atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* (*Hamilton,* 1959), *with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}(Å²)
P	2446 (1)	1850 (1)	4812 (1)	4·43 (5)
O(1)	2564 (3)	2581 (2)	5332 (3)	6·0 (1)
C(1)	1136 (4)	1801 (3)	3886 (4)	4·1 (2)
C(2)	97 (5)	2157 (3)	4141 (4)	5·6 (2)
C(3)	−942 (6)	2172 (3)	3480 (5)	6·2 (2)
C(4)	−952 (5)	1824 (3)	2527 (4)	5·3 (2)
C(5)	77 (6)	1462 (3)	2257 (4)	5·8 (2)
C(6)	1129 (5)	1451 (3)	2933 (4)	5·0 (2)
C(7)	2268 (5)	1144 (3)	5749 (4)	4·5 (2)
C(8)	1636 (5)	490 (3)	5524 (4)	5·3 (2)
C(9)	1509 (5)	−30 (3)	6311 (5)	5·7 (2)
C(10)	2002 (5)	97 (3)	7304 (5)	6·0 (2)
C(11)	2648 (6)	743 (3)	7541 (5)	6·5 (2)
C(12)	2777 (5)	1249 (3)	6764 (4)	5·4 (2)
C(13)	3732 (5)	1623 (3)	4142 (4)	4·9 (2)
C(14)	4187 (6)	900 (3)	4072 (5)	6·5 (2)
C(15)	5198 (7)	768 (4)	3559 (6)	7·7 (3)
C(16)	5774 (7)	1323 (5)	3099 (7)	9·0 (4)
C(17)	5340 (8)	2032 (5)	3157 (7)	9·1 (4)
C(18)	4320 (6)	2180 (3)	3658 (6)	7·2 (3)
C	2218 (6)	4344 (3)	4406 (4)	6·6 (2)
O(2)	3062 (5)	4015 (2)	5105 (3)	9·1 (2)
C(19)	2336 (9)	4140 (7)	3302 (4)	6·24 (5)
C(20)	3348 (9)	4272 (7)	2758 (4)	6·24 (5)
C(21)	3331 (9)	4072 (7)	1711 (4)	6·24 (5)
C(22)	2302 (9)	3740 (7)	1209 (4)	6·24 (5)
C(23)	1290 (9)	3608 (7)	1753 (4)	6·24 (5)
C(24)	1307 (9)	3808 (7)	2800 (4)	6·24 (5)
C′(19)	2350 (9)	4105 (8)	3267 (6)	6·24 (5)
C′(20)	3523 (10)	4172 (8)	2966 (6)	6·24 (5)
C′(21)	3760 (9)	3987 (8)	1954 (6)	6·24 (5)
C′(22)	2830 (9)	3735 (8)	1243 (6)	6·24 (5)
C′(23)	1660 (9)	3667 (8)	1544 (6)	6·24 (5)
C′(24)	1420 (9)	3852 (8)	2556 (6)	6·24 (5)
C(25)	2141 (8)	5135 (3)	4525 (8)	6·24 (5)
C(26)	3190 (8)	5539 (3)	4816 (8)	6·24 (5)
C(27)	3149 (8)	6310 (3)	4829 (8)	6·24 (5)
C(28)	2060 (8)	6677 (3)	4551 (8)	6·24 (5)
C(29)	1011 (8)	6273 (3)	4260 (8)	6·24 (5)
C(30)	1052 (8)	5501 (3)	4247 (8)	6·24 (5)
C′(25)	2441 (7)	5202 (3)	4571 (7)	6·24 (5)
C′(26)	3524 (7)	5472 (3)	5064 (7)	6·24 (5)
C′(27)	3711 (7)	6233 (3)	5158 (7)	6·24 (5)
C′(28)	2814 (7)	6724 (3)	4758 (7)	6·24 (5)
C′(29)	1731 (7)	6453 (3)	4265 (7)	6·24 (5)
C′(30)	1544 (7)	5692 (3)	4171 (7)	6·24 (5)

The C atoms from C(19) to C′(30) belong to the disordered rings of diphenylmethanol. They were refined as rigid rings and presented the following final occupation factors: from C(19) to C(24), 0·55; from C′(19) to C′(24), 0·45; from C(25) to C(30), 0·47 and from C′(25) to C′(30), 0·53.

Refinement carried out by full-matrix least-squares calculations with anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms. Function minimized was $\sum w_i(k|F_o| - |F_c|)^2$ with $w_i^{-1} = \sigma(F)^2 + 0{\cdot}001F^2$ for observed and $w_i = 0$ for unobserved reflections, until convergence with $R = 0{\cdot}12$. Difference Fourier calculations revealed clear duplication of phenyl rings of diphenylmethanol. To take care of this disorder, two additional phenyl rings were included in the model. Refinement of occupation factors of the four phenyl rings associated with diphenylmethanol and treated as rigid groups (C—C, 1·395 Å and C—C—C, 120°). H-atom coordinates recalculated after each refinement cycle. Hydroxyl H atom located by Fourier difference synthesis and its coordinates fixed. Refinement carried out until max. least-squares shift/e.s.d. <1. Final R and wR, omitting unobserved reflections, were 0·070 and 0·079. Reflections 200, 040, $\bar{2}21$ and 041, affected by non-systematic error, were omitted in the last cycle. Difference Fourier calculations after last refinement cycle revealed max. positive and negative electron density 0·327 and −0·262 e Å⁻³ respectively. Atomic scattering factors used were those of Cromer & Waber (1974).

Final atomic parameters for the non-H atoms are given in Table 1.*

Discussion. Angles and bond lengths in the title compound are given in Fig. 1. Diphenylmethanol bonds to triphenylphosphine oxide by hydrogen bonding between O(1) and O(2), as predicted. The O(1)···O(2) distance is 2·672 (5) Å and the O(1)···H(1)—O(2) angle 146 (4)°. The hydrogen bond is asymmetric and may be classified as medium (Novak, 1974).

The distances and angles in the diphenylmethanol molecule are mostly as expected. The four phenyl rings introduced as rigid groups in order to take care of the disorder have occupation factors close to 0·5 as can be seen from Table 1. The angles between the mean planes through atoms C(19) to C(24) and C′(19) to C′(24) and through C(25) to C(30) and C′(25) to C′(30) which correspond to the disordered rings are respectively equal to 10·1 and 11·3 (3)°. Mean positions for the disordered rings have been calculated in order to

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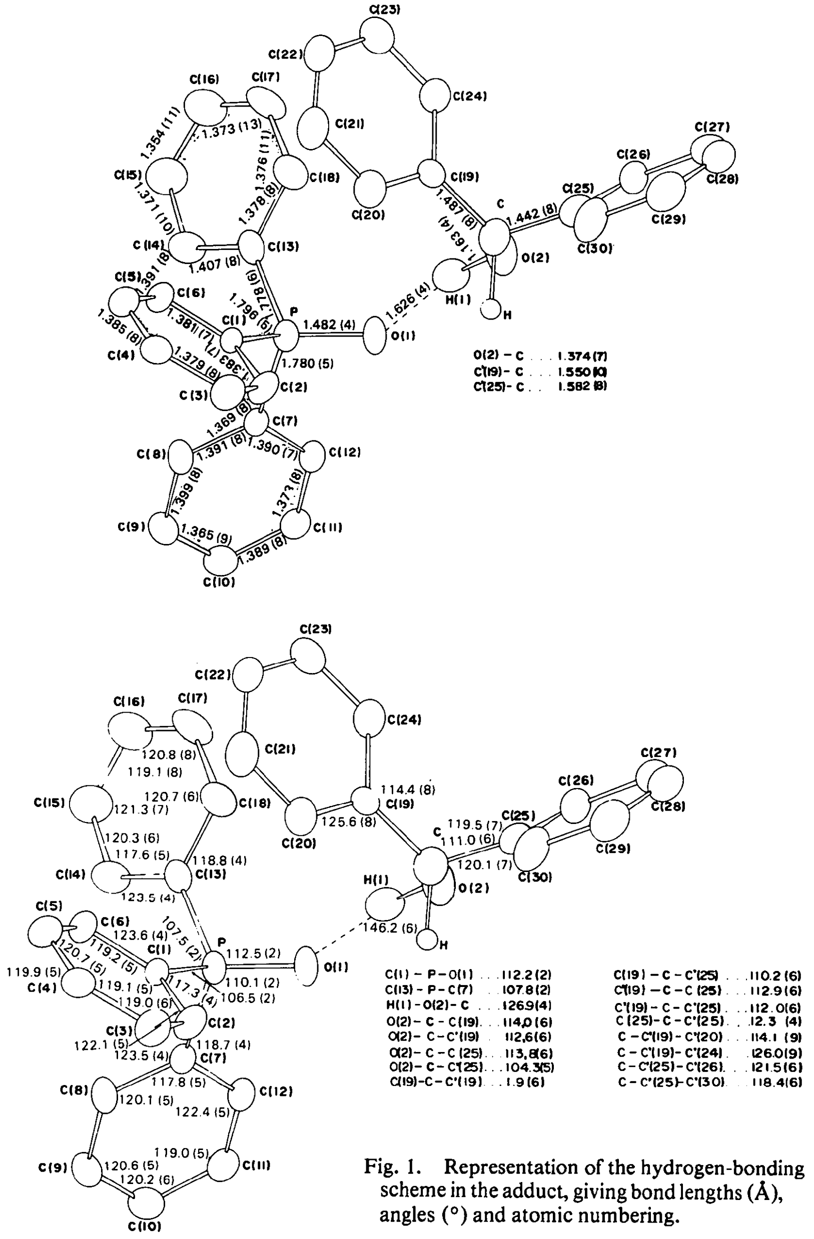
* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths, angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43214 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

simplify representation (Figs. 1 and 2). The structure of triphenylphosphine oxide is essentially the same as that of the free molecule (Bandoli, Bortolozzo, Clemente, Croatto & Panattoni, 1970) except for the P=O distance which is significantly longer [1·482 (4) Å] when compared with the corresponding distance in the free molecule [1·46 (1) Å]. It is interesting to notice that higher values have been reported for the P=O distance in adducts of triphenylphosphine oxide with copper [1·492 (4) Å; Bertrand & Kalyanaraman (1971)] and zinc [1·496 (5) Å; Rose, Lalancette, Potenza & Schugar (1980)] for instance.

The lengthening of the P=O bond is explained by the Gutmann first bond-length variation rule (Gutmann, 1978) and indicates that the intermolecular donor–acceptor interaction in this adduct occurs to a smaller degree than in the adducts with transition metals.

Crystal packing as well as hydrogen bonds are shown in Fig. 2.

All calculations, unless otherwise mentioned in the text, were performed in the Instituto de Física e Química de São Carlos, USP, on PDP 11/45 and VAX 780 computers using the *SHELX*76 (Sheldrick, 1976) crystallographic programs.



O(2) – C	1.374 (7)
C'(19) – C	1.550 (10)
C'(25) – C	1.582 (8)

C(1) – P – O(1)	112.2 (2)	C(19) – C – C'(25)	110.2 (6)
C(13) – P – C(7)	107.8 (2)	C'(19) – C – C(25)	112.9 (6)
H(1) – O(2) – C	126.9 (4)	C'(19) – C – C'(25)	112.0 (6)
O(2) – C – C(19)	114.0 (6)	C(25) – C – C'(25)	12.3 (4)
O(2) – C – C'(19)	112.6 (6)	C – C'(19) – C'(20)	114.1 (9)
O(2) – C – C(25)	113.8 (6)	C – C'(19) – C'(24)	126.0 (9)
O(2) – C – C'(25)	104.3 (5)	C – C'(25) – C'(26)	121.5 (6)
C(19) – C – C'(19)	1.9 (6)	C – C'(25) – C'(30)	118.4 (6)

Fig. 1. Representation of the hydrogen-bonding scheme in the adduct, giving bond lengths (Å), angles (°) and atomic numbering.

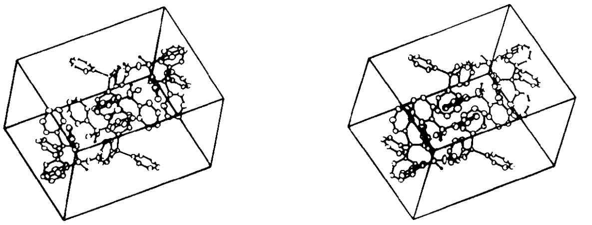


Fig. 2. Stereoview showing molecular packing for the title compound.

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Structure of Methyl 4-(2-Oxo-5-phenyl-2,3-dihydro-3-pyrrolylidene)butyrate*

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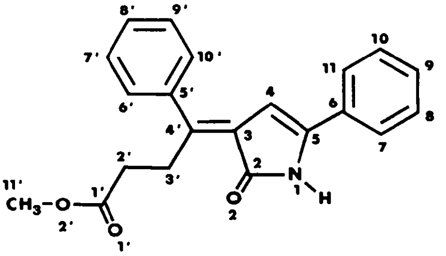
Abstract. $C_{21}H_{19}NO_3$, $M_r = 333{\cdot}4$, monoclinic, $P2_1/n$, $a = 8{\cdot}251$ (2), $b = 21{\cdot}640$ (7), $c = 10{\cdot}201$ (2) Å, $\beta = 105{\cdot}49$ (2)°, $V = 1755$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}26$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}079$ mm^{-1}, $F(000) = 704$, $T = 293$ K, final $R = 0{\cdot}049$ for 1714 reflections. The X-ray study confirms that in the solid state the structure of the title compound is as inferred from spectroscopic evidence. The pyrrolidone and phenyl rings are planar. The molecules in the crystal are connected by N—H···O hydrogen bonds, forming a dimer-like unit across a centre of inversion. There are also two weak C—H···C and C—H···N intramolecular interactions that stabilize the conformation of the 5-phenyl ring. The dimer-like units are held in the crystal by van der Waals forces.

Introduction. As part of our investigation of conformational requirements for respiratory stimulant and antidepressant activities (Lednicer & Mitscher, 1980) in highly-substituted pyrrolidone derivatives, the title compound (1) has been synthesized. The empirical formula $C_{21}H_{19}NO_3$ was established by mass spectrometry and microanalysis, and the functional groups present were characterized by NMR, IR and UV spectroscopy (Xocoyotl, 1986). The structure determination was undertaken to elucidate the stereochemistry of (1).



(1)

Experimental. The title compound (1) was obtained from the reaction of methyl 4-aceto-4-phenylbutyrate (prepared as described by Somerville & Allen, 1943; de Boer & Backer, 1963) with liquid NH_3 in an atmosphere of NH_3 at 373 K for 7 hours. The precipitate from the reacting system was separated and chromatographed over silica gel and eluted with hexane–ethyl acetate

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