numbering scheme used. The structure of the two molecules confirms the reaction path of the scheme. It is worth noting that the dienophilic attack occurs in the *endo* position. For molecule (I*a*), the dienophile is the unsubstituted double bond. Comparison of the 'common' part of (I*a*) and (II*b*) shows no substantial difference in geometrical terms. Molecule (II*b*) is loosely packed: no relevant intermolecular contact is found. In (I*a*) there is one short intermolecular contact, namely N(15)···H(7) 2·47 (3) Å with C(7)—H(7)···N(15) 155 (2)°.

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Structure of 2-Phenyladamantan-2-ol

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(*Received* 9 *June* 1986; *accepted* 5 *September* 1986)

Abstract. 2-Phenyltricyclo[3.3.1.1^{3,7}]decan-2-ol, $C_{16}H_{20}O$, $M_r = 228{\cdot}33$, tetragonal, $I4_1/a$, $a = 22{\cdot}794$ (5), $c = 10{\cdot}004$ (2) Å, $V = 5198$ (2) Å^3, $Z = 16$, $D_x = 1{\cdot}167$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54183$ Å, $\mu(\mathrm{Cu}\,K\alpha) = 5{\cdot}1$ cm^{-1}, $F(000) = 1984$, room temperature, $R = 0{\cdot}062$ for 2174 unique reflections. Four symmetry-related molecules are linked by intermolecular hydrogen bonds forming a tetramer. The tetramers are held together by van der Waals forces. The adamantane skeleton fixes the phenyl ring in such a position that the C—O bond rotates out of the phenyl plane by 82°. The position of the hydroxyl H atom suggests intramolecular OH···π interaction.

Introduction. An infrared study has demonstrated that intramolecular OH···π interaction in benzyl alcohols and anthracylmethanols depends on the position of the phenyl ring relative to the C—O axis (Visser & Van der Maas, 1986). It appears that OH···π interaction occurs if the C—O bond is twisted out of the phenyl plane by 90°. Ahmed & Huber (1981) have investigated the structure of 1-phenylcyclohexanol by means of X-rays. This compound also has a phenyl ring and a hydroxyl group substituted at the same C atom. However, the torsion angles involving the phenyl rings and the C—O bonds are small (between −11·7 and 27·3°). The X-ray analysis of the title compound was undertaken to determine the molecular stereochemistry, especially the position of the phenyl ring in connection with possible OH···π interaction.

Experimental. Data were measured on a crystal of dimensions 0·8 × 1·1 × 0·4 mm on an Enraf–Nonius CAD-4F diffractometer with Ni-filtered Cu $K\alpha$ radiation. Lattice constants from eight reflections in the range $62 < 2\theta < 64°$, ω–2θ mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}15\tan\theta)°$. The crystal is tetragonal with space group $I4_1/a$. 2460 unique reflections were measured up to $\theta = 70°$, $0 \le h \le 27$, $0 \le k \le 27$, $0 \le l \le 12$, 2178 of these were considered observed [$I > 1{\cdot}5\sigma(I)$] and used for structure determination and refinement. Three periodically measured standard reflections (600, 060, 004) showed intensity variations less then 6%; Lp correction, no correction for absorption or secondary extinction. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$ where $P(=0{\cdot}014)$ is the instability constant (McCandlish, Stout & Andrews, 1975). The structure was solved with *SHELXS*84 (Sheldrick, 1984); the best E map gave all non-H atoms. H atoms were placed on calculated positions except the hydrogen H atom which was located on a difference map. The temperature parameters of H atoms were set equal to the equivalent isotropic temperature factors of the carrier atoms. All refinements were full-matrix least-squares refinements using *SHELX*76 (Sheldrick, 1976). Hydrogen parameters were kept fixed except for the coordinates of the hydroxyl H atom. The reflections 040, 020, $\bar{2}40$ and 240 were omitted [$\Delta F/\sigma(F) > 10$] and the refinement on F converged at $R = 0{\cdot}0624$, $wR = 0{\cdot}0789$ where $w = 1/\sigma^2(F)$, and $S = 4{\cdot}3$. $|\Delta/\sigma| = 0{\cdot}03$ (av.) and 0·09 (max.) for non-H-atom parameters; final electron density showed a maximum and minimum of 0·13 and

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$-0{\cdot}22\,e\,Å^{-3}$. Scattering factors were taken from Cromer & Mann (1968) for C and O atoms and from Stewart, Davidson & Simpson (1965) for H atoms. Calculations were carried out on the Cyber 180–855 of the University of Utrecht Computer Center. The program package *EUCLID* (Spek, 1982) is used for the calculation of geometric data and preparation of illustrations.

Table 1. *Positional and equivalent isotropic thermal parameters* ($Å^2$) *for non-*H *atoms and the hydroxyl* H *atom with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
O	0·0758 (1)	0·2176 (1)	0·6602 (2)	0·0663 (5)
H(1)	0·0481 (9)	0·1939 (9)	0·636 (2)	0·0595
C(1)	0·1558 (1)	0·2162 (1)	0·8145 (2)	0·0587 (6)
C(2)	0·1179 (1)	0·1783 (1)	0·7204 (2)	0·0479 (5)
C(3)	0·0863 (1)	0·1328 (1)	0·8083 (2)	0·0541 (6)
C(4)	0·1309 (1)	0·0939 (1)	0·8807 (2)	0·0690 (6)
C(5)	0·1691 (1)	0·1318 (1)	0·9709 (2)	0·0777 (8)
C(6)	0·1309 (1)	0·1629 (1)	1·0756 (2)	0·096 (1)
C(7)	0·0859 (1)	0·2012 (1)	1·0030 (3)	0·088 (1)
C(8)	0·1176 (1)	0·2472 (1)	0·9175 (2)	0·0800 (8)
C(9)	0·0480 (1)	0·1635 (1)	0·9128 (2)	0·0767 (8)
C(10)	0·2004 (1)	0·1774 (1)	0·8860 (2)	0·0700 (6)
C(11)	0·1527 (1)	0·1516 (1)	0·6043 (2)	0·0495 (6)
C(12)	0·1400 (1)	0·0974 (1)	0·5526 (2)	0·0823 (6)
C(13)	0·1692 (1)	0·0748 (1)	0·4422 (3)	0·105 (1)
C(14)	0·2119 (1)	0·1063 (1)	0·3809 (3)	0·092 (1)
C(15)	0·2250 (1)	0·1607 (1)	0·4300 (3)	0·091 (1)
C(16)	0·1956 (1)	0·1834 (1)	0·5393 (2)	0·0730 (6)

Table 2. *Bond distances* (Å), *bond angles* (°) *and a selection of torsion angles* (°) *for non-*H *atoms and the hydroxyl* H *atom with e.s.d.'s in parentheses*

O–H	0·87 (2)	C(5)–C(10)	1·520 (3)
O–C(2)	1·444 (3)	C(6)–C(7)	1·530 (3)
C(1)–C(10)	1·526 (3)	C(7)–C(9)	1·516 (3)
C(1)–C(2)	1·542 (3)	C(7)–C(8)	1·534 (3)
C(1)–C(8)	1·523 (3)	C(11)–C(16)	1·380 (3)
C(2)–C(11)	1·533 (3)	C(11)–C(12)	1·370 (3)
C(2)–C(3)	1·539 (3)	C(12)–C(13)	1·389 (3)
C(3)–C(4)	1·531 (3)	C(13)–C(14)	1·356 (3)
C(3)–C(9)	1·531 (3)	C(14)–C(15)	1·367 (3)
C(4)–C(5)	1·523 (3)	C(15)–C(16)	1·383 (3)
C(5)–C(6)	1·536 (3)		

C(2)–O–H	102 (1)	C(4)–C(5)–C(10)	109·0 (2)
C(2)–C(1)–C(8)	110·6 (2)	C(5)–C(6)–C(7)	108·6 (2)
C(2)–C(1)–C(10)	109·6 (2)	C(6)–C(7)–C(8)	109·8 (2)
C(8)–C(1)–C(10)	109·4 (2)	C(6)–C(7)–C(9)	109·9 (2)
O–C(2)–C(1)	106·2 (2)	C(8)–C(7)–C(9)	108·9 (2)
C(1)–C(2)–C(3)	106·9 (2)	C(1)–C(8)–C(7)	109·2 (2)
C(3)–C(2)–C(11)	114·1 (2)	C(3)–C(9)–C(7)	109·9 (2)
O–C(2)–C(11)	105·9 (2)	C(1)–C(10)–C(5)	110·2 (2)
O–C(2)–C(3)	110·2 (2)	C(2)–C(11)–C(12)	122·3 (2)
C(1)–C(2)–C(11)	113·3 (2)	C(2)–C(11)–C(16)	121·0 (2)
C(2)–C(3)–C(9)	110·4 (2)	C(12)–C(11)–C(16)	116·4 (2)
C(2)–C(3)–C(4)	110·5 (2)	C(11)–C(12)–C(13)	122·2 (2)
C(4)–C(3)–C(9)	108·7 (2)	C(12)–C(13)–C(14)	120·5 (2)
C(3)–C(4)–C(5)	109·4 (2)	C(13)–C(14)–C(15)	118·3 (3)
C(4)–C(5)–C(6)	110·0 (2)	C(14)–C(15)–C(16)	121·2 (2)
C(6)–C(5)–C(10)	109·4 (2)	C(11)–C(16)–C(15)	121·3 (2)

H–O–C(2)–C(11)	81 (1)
O–C(2)–C(11)–C(12)	−98·1 (2)

Discussion. The final atomic parameters are given in Table 1.* The conformation of an alcohol molecule and the atom numbering are illustrated in Fig. 1. Bond distances and angles are tabulated in Table 2. The asymmetric unit contains one molecule with bond distances which are in the normal range. The bond angle C(12)C(11)C(16) deviates from the other bond angles in the phenyl ring but is in accordance with 1-phenylcyclohexanol (Ahmed & Huber, 1981). The bond angles involving the C(2) atom deviate from the regular tetrahedral value. This is partly due to intermolecular hydrogen bonds (comparable effects were found in 1-phenylcyclohexanol), and partly to steric effects as will be discussed below.

The molecular packing is illustrated in Fig. 2. As in 1-phenylcyclohexanol, four molecules are linked by hydrogen bonds producing a tetramer. The molecules in the tetramer are related by a $\bar{4}$ axis, whereas the tetramers are related by a 4_1 axis.

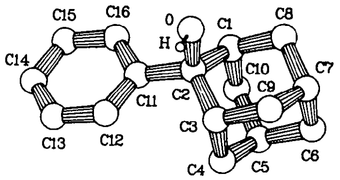

Fig. 1. Molecular structure with atom numbering of the title compound.

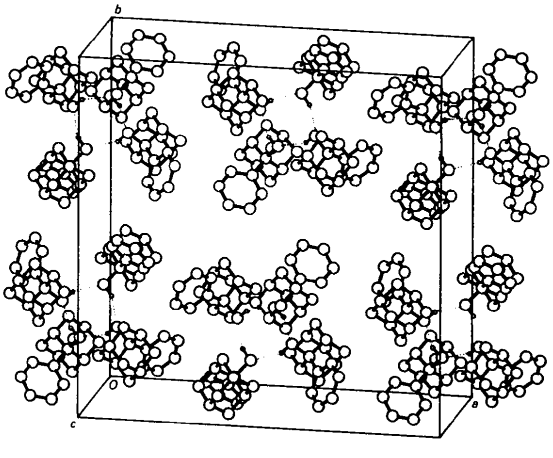

Fig. 2. Packing diagram viewed down c. Dotted lines indicate hydrogen bonds.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43379 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

There are no intermolecular C—C distances less then 3·6 Å and the shortest H—H contact is 2·182 (3) Å. The distance between the O atoms involved in a hydrogen bond [2·749 (2) Å] as well as the O—H···O angle [154 (2)°] are in the normal range.

The position of the phenyl ring [OC(2)C(11)C(12) = −98·1 (2)°] causes short intramolecular distances between the hydrogen atoms at C(1) and C(16) [1·984 (3) Å] and between the hydrogen atoms at C(3) and C(12) [1·972 (3) Å]. The deviations of the angles C(1)C(2)C(11), C(1)C(2)C(3) and C(3)C(2)C(11) can be ascribed, therefore, to the substitution of the phenyl ring at C(2). As can be expected, this effect is not found in 1-phenylcyclohexanol, where the steric hindrance can be relieved by a more or less planar arrangement of the O atom and the phenyl group. In our compound this conformation would be impossible owing to further steric hindrance. Thus in contradistinction to 1-phenylcyclohexanol, the adamantane skeleton fixes the phenyl ring in a position where OH···π interaction is possible.

The X-ray data were kindly collected by Dr A. J. M. Duisenberg. We are grateful to Dr A. L. Spek for helpful discussions.

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Acta Cryst. (1987). C**43**, 311–313

Structure of 4-Hydroxymonophenylbutazone

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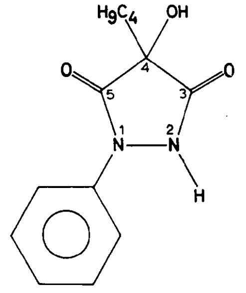
(*Received* 22 *May* 1986; *accepted* 21 *August* 1986)

Abstract. 4-Butyl-4-hydroxy-1-phenyl-3,5-pyrazolidinedione, $C_{13}H_{16}N_2O_3$, $M_r = 248\cdot3$, monoclinic, $P2_1/n$, $a = 22\cdot357$ (5), $b = 5\cdot014$ (2), $c = 11\cdot350$ (4) Å, $\beta = 91\cdot88$ (3)°, $V = 1272$ (1) Å^3, $Z = 4$, $D_m = 1\cdot296$ (3), $D_x = 1\cdot297$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0\cdot777$ mm^{-1}, $F(000) = 528$, $T = 293$ K. Final $R = 0\cdot059$ for 1668 observed reflections. The hetero nitrogen which carries the six-membered ring is planar in the structure while the other unsubstituted one is pyramidal. The five- and six-membered rings are almost coplanar. The crystal is made up of infinite columns of hydrogen-bonded molecules.

Introduction. Non-steroidal anti-inflammatory antipyretic analgesics, in spite of their diverse chemical structures, are believed to act through the inhibition of prostaglandin biosynthesis (Flower, 1974). With the objective of understanding their molecular geometry, the nature of their interactions, and the geometric and electronic consequences of such interactions, we have analysed the structures of several analgetic pyrazole derivatives, fenamates and their complexes (Dhanaraj & Vijayan, 1983; Vijayan, 1983, and the references therein). Monophenylbutazone is a drug belonging to the family of pyrazolidinedione derivatives. The crystal structures of the other important members of this family, phenylbutazone and oxyphenbutazone, have already been reported (Singh & Vijayan, 1977; Krishna Murthy & Vijayan, 1981). During attempts to crystallize monophenylbutazone from a commercial sample, a hydroxy derivative of the compound, present as an impurity, readily crystallized. The crystal structure of this derivative (I) is reported here.



(I)