1-Isoquinolyl(phenyl)methanol

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Abstract. $C_{16}H_{13}NO$, $M_r = 235 \cdot 3$, monoclinic, $P2_1/c$, $a = 7 \cdot 875$ (1), $b = 9 \cdot 770$ (1), $c = 15 \cdot 883$ (2) Å, $\beta = 100 \cdot 42$ (1)°, $V = 1201 \cdot 9$ Å^3, $D_x = 1 \cdot 30$ Mg m^{-3}, $Z = 4$, $F(000) = 496$, μ(Cu $K\alpha$) $= 0 \cdot 65$ mm^{-1}. The structure was solved by direct methods and refined with 1287 observed Cu $K\alpha$ diffractometer data to $R = 0 \cdot 04$. Two molecules of 1-isoquinolyl(phenyl)methanol are linked by bifurcated hydrogen bonds (intra- and intermolecular) forming dimers about centers of symmetry $(0,\frac{1}{2},0; 0,0,\frac{1}{2})$ in the crystal. The N$\cdots$O distances are $2 \cdot 682$ (2) and $2 \cdot 974$ (2) Å for the intra- and intermolecular interactions respectively. The mean H$\cdots$N distance is $2 \cdot 23$ (4) Å.

Introduction. This work is a continuation of our study of 1-isoquinolylcarbinols:
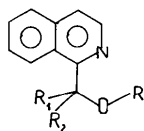
		R	R_1	R_2
(I*a*)*		COOC$_2$H$_5$	C$_6$H$_5$	H
(I*b*)		COC$_6$H$_5$	C$_6$H$_5$	H
(I*c*)		COC$_6$H$_5$	CH$_3$	CH$_3$
(I*h*)		H	C$_6$H$_5$	H

* Symbols (I*a*), (I*b*), (I*c*) and (I*h*) are in accordance with the notation used in our previous paper (Skrzypczak-Jankun, Hoser, Grzesiak & Kałuski, 1979).

Crystals of the title compound (I*h*) suitable for X-ray analysis were obtained from ethanol solution. Reflections were collected on a Syntex $P2_1$ diffractometer with graphite-monochromatized Cu $K\alpha$ radiation within the range $2\theta < 116°$, in a 2θ–ω scan mode, at a scanning rate of 2° min^{-1}. The background and integrated intensity for each reflection were evaluated by a profile-analysis procedure (Lehmann & Larsen, 1974). The intensities were corrected for Lorentz and polarization effects and for absorption from empirical φ-scan data. Of the 1641 independent reflections measured 1287 had $F_o \geq 3 \cdot 92\sigma(F_o)$ and were used in the subsequent calculations. The structure was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971) and refined by the full-matrix least-squares method. A difference Fourier synthesis revealed all the H atoms, including that of the hydroxyl group, which corresponded to the lowest peak on this map. Positional and isotropic temperature factors of all H atoms were included in the last cycles of refinement and allowed to vary. The final discrepancy factors are: $R = 0 \cdot 040$ and $R_w = 0 \cdot 043$. The function minimized was $\sum w|F_o - F_c|^2$, where $w = (F_o/4 \cdot 75)^2$ for $F_o < 4 \cdot 75$, $w = 1$ for $4 \cdot 75 \leq F_o \leq 12 \cdot 25$, and $w = (12 \cdot 25/F_o)^2$ for $F_o > 12 \cdot 25$. In the final cycle of

Table 1. *Positional* ($\times 10^4$; $\times 10^3$ *for* H *atoms*) *and thermal parameters with their e.s.d.'s*

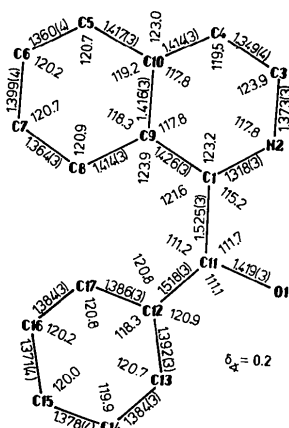$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} \, a_i^* \, a_j^* \, \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)		x	y	z	B_{iso} (Å^2)	C–H ($\sigma = 0 \cdot 03$ Å)
C(1)	8773 (2)	2560 (2)	4864 (1)	3·2 (1)						
N(2)	9548 (2)	1695 (2)	5445 (1)	3·8 (1)						
C(3)	9771 (3)	2084 (3)	6288 (2)	4·4 (2)	H(3)	1039 (3)	139 (2)	667 (2)	5·6 (6)	0·98 Å
C(4)	9229 (3)	3291 (3)	6556 (2)	4·2 (2)	H(4)	941 (3)	354 (2)	714 (2)	5·3 (5)	0·95
C(5)	7788 (3)	5528 (3)	6174 (2)	4·4 (2)	H(5)	798 (3)	576 (2)	678 (2)	5·4 (5)	0·97
C(6)	7008 (3)	6412 (3)	5563 (2)	4·9 (2)	H(6)	657 (3)	731 (3)	572 (2)	6·1 (6)	0·99
C(7)	6767 (3)	6046 (3)	4698 (2)	4·5 (2)	H(7)	619 (3)	667 (3)	428 (2)	5·9 (6)	0·96
C(8)	7312 (3)	4806 (2)	4456 (2)	3·7 (1)	H(8)	715 (3)	457 (2)	387 (1)	3·9 (5)	0·95
C(9)	8143 (2)	3864 (2)	5071 (1)	3·0 (1)						
C(10)	8380 (3)	4235 (2)	5947 (1)	3·7 (1)						
C(11)	8549 (3)	2060 (2)	3941 (1)	3·4 (1)	H(11)	876 (2)	285 (2)	355 (1)	2·9 (4)	1·02
O(1)	9850 (2)	1107 (2)	3829 (1)	4·1 (1)	H(O)	1000 (4)	54 (4)	430 (2)	10·7 (1·2)	0·92
C(12)	6760 (3)	1466 (2)	3643 (1)	3·0 (1)						
C(13)	6214 (3)	319 (2)	4040 (2)	3·7 (1)	H(13)	696 (3)	−10 (2)	452 (2)	5·3 (5)	0·97
C(14)	4578 (3)	−216 (3)	3771 (2)	4·6 (2)	H(14)	417 (3)	−105 (3)	407 (2)	5·8 (6)	1·02
C(15)	3475 (3)	385 (3)	3100 (2)	4·8 (2)	H(15)	232 (3)	4 (2)	293 (2)	5·8 (6)	0·96
C(16)	4006 (3)	1501 (3)	2692 (2)	4·5 (2)	H(16)	323 (3)	194 (2)	223 (2)	5·8 (6)	0·97
C(17)	5641 (3)	2038 (3)	2960 (1)	4·0 (2)	H(17)	603 (3)	281 (2)	267 (1)	4·9 (5)	0·96

least-squares refinement only the y coordinate of H(O) had a Δ/σ value different from zero (0·02). The final difference function was flat and featureless.

All calculations were performed on a NOVA 1200 computer with the program *PRAN* (Jaskólski, 1979) for profile analysis and the programs from *XTL/ E-XTL Structure Determination System* (Syntex, 1976). Final positional and thermal parameters are given in Table 1.*

Discussion. The atom-numbering system, bond distances and angles are given in Fig. 1. There is nothing abnormal about the bond lengths in the ring systems. The atoms of the isoquinolyl system are nearly coplanar, though with small but significant deviations (data for the best least-squares planes have been deposited).* The molecule is slightly bent around the

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Fig. 1. Bond distances (Å) and angles (°) with their e.s.d.'s.

Fig. 2. Unit-cell projection along a. All H atoms except that from the hydroxyl group have been omitted for clarity. H···N contacts are marked as dashed lines.

Fig. 3. Diagram showing the hydrogen-bonded dimer with the distances (Å) and angles (°) describing the hydrogen bonds.

C(9)–C(10) bond; the dihedral angle between planes defined by the pyridine and benzene rings from the isoquinoline is 0·7°. The molecular geometry of 1-isoquinolyl(phenyl)methanol (Ih) is very similar to those observed for the ethyl carbonate of 1-iso-quinolyl(phenyl)methanol (Ia; Skrzypczak-Jankun & Kałuski, 1977) and 1-isoquinolyl(phenyl)methyl benzoate (Ib; Skrzypczak-Jankun et al., 1979). However, the torsion angle N(2)–C(1)–C(11)–O(1), −27·60 (2)°, differs from the same angles obtained for structures (Ia) and (Ib) (+12·8 and +9·0° respectively), which have bulky substituents (R) at O(1). This is probably due to interactions between the neighboring molecules. In contrast to the structures (Ia), (Ib) and (Ic), this isoquinoline derivative (Ih) has basic properties ($pKa = 4·1$; Rozwadowska, 1977) and possesses a hydroxyl group, which could act as a proton donor. The higher e.s.d.'s in the positional parameters, the large shifts in the y direction during the refinement process, a relatively high B_{iso} for the H(O) atom and the O–H distance of 0·92 (4) Å, as well as the short contacts O···N and O···N′ (−x,−y,−z), suggested that the H atom from the hydroxyl group is shared between the N, N′ and O atoms.

A projection of the structure along a, illustrating the packing of the molecules, is shown in Fig. 2. It can be seen that the principal interaction is the formation of centrosymmetric dimers through hydrogen bonds involving N(2) from the isoquinoline moiety and the hydroxyl groups. Fig. 3 gives a detailed description of the hydrogen-bonded dimer. A similar situation was found in 1-chloro-3-isoquinolinol, where the two molecules are linked together into a centrosymmetric dimer by hydrogen bonds, N···O = 2·83, N···H = 2·03 Å, ∠NHO = 171° (Ammon & Wheeler, 1974). In 1-isoquinolyl(phenyl)methanol, apart from the inter-molecular distance N···O = 2·974 (2) Å, there is a short intra-annular contact, N···O = 2·682 (2) Å. The five-membered ring formed in this way prevents the interaction being as strong as in 5-hydroxy-1-(3-methyl-1-isoquinolyl)-3-phenylpyrazole, where the intra-annular bond through the six-membered ring has an N···O distance of 2·51 Å (King & Reimlinger,

1971). Nevertheless, the intra- and intermolecular H···N distances noted here, 2·21 (4) and 2·24 (4) Å, are equal within the range of the experimental errors and are considerably shorter than the sum of the van der Waals radii. So, we can state that the interaction

O—H:$\overset{\cdots\text{N}'\cdots}{\underset{\cdots\text{N}\cdots}{\cdots}}$:H—O' observed in the crystal structure of 1-isoquinolyl(phenyl)methanol is an interesting example of a bifurcated hydrogen bond.

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The Structure of 2-(2-Aminobenzoyl)benzoic Acid

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Abstract. $C_{14}H_{11}NO_3$, orthorhombic, $Pca2_1$, $a = 13·860$ (1), $b = 10·135$ (1), $c = 8·286$ (1) Å, $Z = 4$, $V = 1163·9$ Å^3, $D_x = 1·38$, $D_m = 1·37$ Mg m^{-3}, $F(000) = 504$. The crystal structure was solved from diffractometer data by direct methods and refined by full-matrix least-squares procedures to an R of 0·040 for 844 observed reflexions. The packing of the molecules in the crystals is stabilized by hydrogen bonds: $O(15)—H(11)\cdots O(17) = 2·663$ (21) and $N(18)—H(2)\cdots O(16) = 3·194$ (13) Å. An intramolecular hydrogen bond, $N(18)—H(1)\cdots O(17) = 2·690$ (8) Å, is present.

Introduction. This determination of the crystal and molecular structure of 2-(2-aminobenzoyl)benzoic acid is a continuation of our studies on substituted 2-aroylbenzoic acids. Suitable crystals for X-ray analysis were kindly provided by Professor J. Gronowska of the Organic Chemistry Department of this University.

Crystal symmetry and approximate cell parameters were determined from rotation and Weissenberg photographs. Space group $Pca2_1$ or $Pcam$ was deduced from systematic absences. On the basis of the distribution of normalized structure factors the noncentrosymmetric space group $Pca2_1$ was selected.

Accurate cell dimensions and intensities were measured on an automated Syntex $P2_1$ four-circle diffractometer, with Cu $K\alpha$ radiation and a graphite monochromator. Reflexions were collected up to $2\theta_{max} = 116°$. 844 reflexions from the data set had $I >$

Table 1. *Fractional atomic coordinates* ($\times 10^4$, *for* H $\times 10^3$), *with estimated standard deviations, and* B_{eq} (Å^2)

$$B_{eq} = \tfrac{4}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \, \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq}
C(1)	−503 (3)	8104 (4)	10607 (34)	2·1
C(2)	−788 (3)	9337 (5)	11288 (35)	2·5
C(3)	−1600 (3)	9986 (5)	10630 (35)	2·8
C(4)	−2086 (3)	9482 (5)	9348 (35)	3·0
C(5)	−1808 (4)	8275 (5)	8652 (34)	2·9
C(6)	−1037 (3)	7609 (5)	9275 (35)	2·4
C(7)	331 (3)	7384 (5)	11182 (34)	2·3
C(8)	517 (3)	5990 (4)	10602 (35)	2·3
C(9)	1376 (3)	5645 (4)	9822 (35)	2·2
C(10)	1592 (3)	4328 (5)	9543 (35)	3·0
C(11)	950 (4)	3355 (5)	9972 (35)	3·3
C(12)	76 (4)	3684 (5)	10691 (35)	3·3
C(13)	−134 (4)	5001 (6)	11036 (35)	2·8
C(14)	2000 (3)	6694 (5)	9100 (35)	2·5
O(15)	2897 (2)	6323 (4)	8885 (34)	4·1
O(16)	1696 (2)	7765 (3)	8718 (34)	3·6
O(17)	894 (2)	7838 (3)	12210 (34)	3·3
N(18)	−323 (4)	9885 (5)	12569 (35)	3·8
H(1)	38 (5)	959 (7)	1311 (9)	
H(2)	−50 (5)	1088 (6)	1293 (9)	
H(11)	313 (5)	699 (7)	836 (10)	