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The Ethyl Carbonate of 1-Isoquinolyl(phenyl)methanol

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Abstract. $C_{19}H_{17}NO_3$, monoclinic, $P2_1/c$, a = 9·233 (1), b = 10·869 (1), c = 18·066 (2) Å, β = 117·81 (1)°, V = 1603·6 Å^3, Z = 4, d_x = 1·27 g cm^{-3}, μ(Cu $K\alpha$, λ = 1·54178 Å) = 7·1 cm^{-1}. The structure was solved by direct methods and refined by full-matrix least-squares calculations to an R of 0·061 for 1673 observed reflexions. There is a short intramolecular contact: N(2)···O(1) 2·64 Å.

Introduction. Cell dimensions were determined by a least-squares fit to the settings for 14 reflexions, 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 $2\theta_{max}$ = 114°. After the application of an Lp correction (but no absorption correction) equivalent reflexions were merged to give 2163 unique reflexions, of which 1673 with $I > 1{\cdot}96\sigma(I)$ were employed for structure refinement.

171 reflexions with $E > 1{\cdot}8$ were used to run *MULTAN*. The E map computed from the solution with the highest ABSFOM (1·13) and COMBINED FOM (2·54), and the lowest RESID (57·7) revealed the positions of all 23 non-hydrogen atoms (R = 0·40). Three cycles of full-matrix least-squares refinement reduced the R value to 0·31 and showed that two C atoms from the ethyl group were poorly located. They were removed and the new positional parameters were found from a Fourier synthesis. After the next few cycles of refinement first with isotropic and then anisotropic thermal parameters the R factor decreased to 0·11. The subsequent difference maps revealed all but two H atoms. The positional and thermal parameters (B_j = 5·5 Å^2) were included in the calculations and allowed to vary. The positions of the missing H atoms were generated on the assumption of a regular tetrahedron with C–H constrained to 1·08 Å. The parameters for the CH_3 group were fixed. Refinement converged to R = 0·061 with a corresponding R_w ($= \sum w^{1/2}|F_o - F_c|/\sum w^{1/2}|F_o|$) of 0·076 for 1673 observed reflexions.* The function minimized was $\sum w|F_o - F_c|^2$, where $w = 1/[\sigma^2(F_o) + (0{\cdot}049F_o)^2]$; the error of fit was 1·40. The final positional parameters for all atoms are given in Table 1.

All calculations were performed on a NOVA 1200 computer with programs from the Syntex XTL/E–XTL structure determination system (Syntex, 1976).

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32809 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional atomic coordinates* (×10^4, *for* H ×10^3) *with their e.s.d.'s*

	x	y	z
C(1)	92 (4)	2494 (3)	1543 (2)
N(2)	942 (4)	3504 (3)	1219 (2)
C(3)	1127 (5)	3888 (4)	459 (3)
C(4)	485 (5)	3301 (4)	25 (2)
C(5)	1189 (5)	1568 (4)	−54 (2)
C(6)	2070 (5)	537 (4)	293 (2)
C(7)	2252 (4)	105 (4)	1055 (2)
C(8)	1561 (4)	709 (3)	1474 (2)
C(9)	645 (4)	1797 (3)	1141 (2)
C(10)	452 (4)	2231 (3)	359 (2)
C(11)	94 (4)	2079 (3)	2386 (2)
C(12)	1756 (4)	2334 (3)	3098 (2)
C(13)	2402 (4)	3513 (3)	3247 (2)
C(14)	3912 (4)	3746 (4)	3915 (2)
C(15)	4807 (5)	2832 (4)	4445 (3)
C(16)	4190 (5)	1656 (4)	4301 (3)
C(17)	2678 (5)	1399 (4)	3637 (2)
O(1)	1036 (3)	2751 (2)	2586 (2)
C(18)	2612 (4)	2466 (4)	2115 (3)
O(2)	3137 (3)	1622 (3)	1644 (2)
O(3)	3483 (3)	3307 (3)	2268 (2)
C(19)	5286 (6)	3175 (7)	1820 (7)
C(20)	6004	4356	1766
H(1)	184 (5)	464 (4)	23 (2)
H(2)	60 (4)	358 (3)	−51 (2)
H(3)	102 (5)	188 (3)	−53 (2)
H(4)	256 (5)	6 (3)	1 (2)
H(5)	286 (4)	−58 (3)	128 (2)
H(6)	164 (3)	36 (3)	196 (2)
H(7)	22 (4)	122 (3)	237 (2)
H(8)	171 (4)	414 (3)	290 (2)
H(9)	427 (4)	454 (4)	402 (2)
H(10)	582 (6)	305 (4)	496 (3)
H(11)	478 (5)	106 (4)	465 (3)
H(12)	225 (5)	63 (4)	353 (2)
H(13)	463 (10)	265 (8)	221 (5)
H(14)	445 (9)	269 (6)	142 (5)
H(15)	498	473	171
H(16)	602	473	232
H(17)	713	459	122

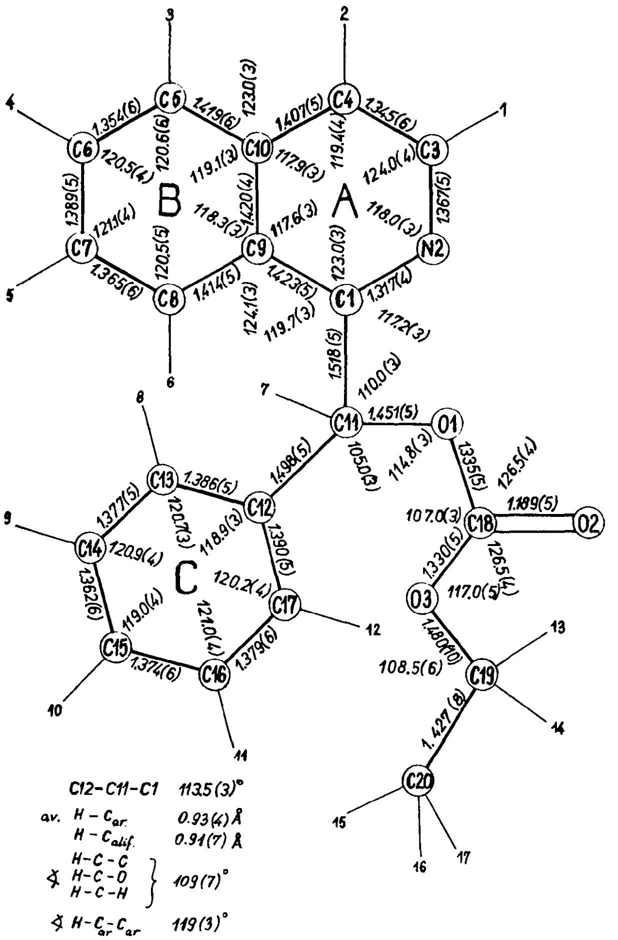


Fig. 1. Bond distances (Å) and valency angles (°) with their e.s.d.'s.

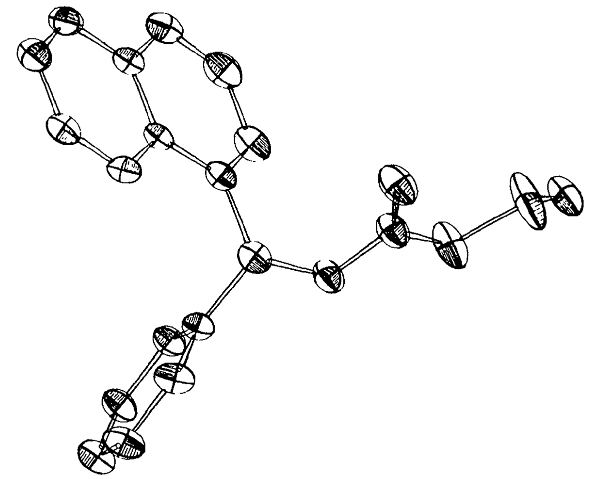


Fig. 2. Molecular conformation. Thermal ellipsoids represent 40% probability.

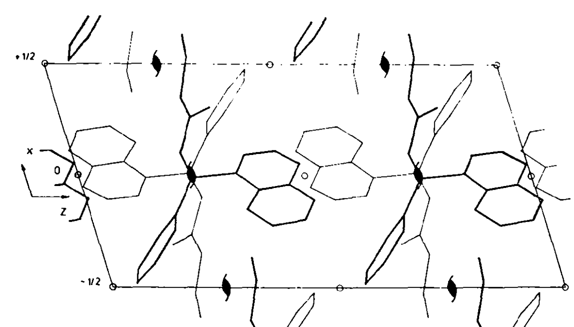
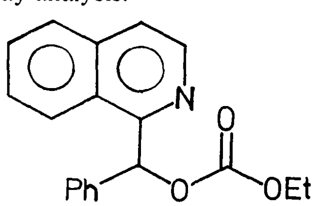


Fig. 3. Molecular packing in the unit cell projected down **b**.

Discussion. The isoquinoline derivative $C_{19}H_{17}NO_3$ was synthesized by Rozwadowska (1977).

From the chemical and spectroscopic investigations the following configuration was suggested and confirmed by X-ray analysis.



The atom-numbering system, and bond distances and angles are shown in Fig. 1. Bond lengths and valency angles in the isoquinoline fragment are comparable with the values cited for other isoquinoline derivatives with an unprotonated N(2) atom, *i.e.* 1-chloro-3-hydroxy-isoquinoline (Ammon & Wheeler, 1974), 6-cyano-7-(2-aminophenyl)isoquinoline (Chiaroni, Doucerain & Riche, 1976), 5-hydroxy-3-phenyl-1-(3-methyl-1-isoquinoline (Ammon & Wheeler, 1974), 6-cyano-7-(2-aminophenyl)isoquinoline (Chiaroni, Doucerain Kálmán & Djurić, 1974). The remaining bond distances and angles agree well with their expected values. The only exception is the very short C(19)–C(20) at 1·427 Å. This may result from the constrained refinement (*i.e.* fixed CH_3 parameters with C–H = 1·08 Å) and from the large thermal motion of these two atoms in the terminal ethyl group.

The conformation of the molecule is shown in Fig. 2. The isoquinoline fragment is approximately planar, although χ^2 for this set of atoms is rather high (Table 2). The angle between planes *A* and *B* (Fig. 1) is 0·8°.

There is a short N(2)···O(1) intramolecular contact of 2·64 Å. This value is close to the extreme limit of minimum contact between non-bonded N···O atoms of 2·6 Å (Ramachandran & Sasisekharan, 1968) and much shorter than the sum of the van der Waals radii (3·12 Å) (Kitaigorodsky, 1973). The torsion angle N(2)–C(1)–C(11)–O(1) is −12·8°.

Table 2. *Best least-squares planes*

Deviations of atoms from the planes (Å)

Plane	C(1)	N(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)	C(10)	C(11)†	χ^2
1(*A* + *B*)	0·005	0·010	0·005	0·017	−0·008	−0·014	0·001	0·012	0·005	−0·001	−0·018	54·54
2(*A*)	0·001	0·005	0·003	0·008					0·005	−0·008	−0·002	14·12
3(*B*)					0·003	−0·004	0·000	0·003	−0·003	0·001		3·04

Plane	C(11)†	C(12)	C(13)	C(14)	C(15)	C(16)	C(17)	χ^2
4(*C*)	0·037	0·003	0·003	0·001	−0·005	0·005	0·001	3·12

Plane	O(1)	C(18)	O(2)	O(3)	χ^2
5	0·000	0·002	0·000	0·000	0·20

Equations to the planes [x,y,z are orthogonal coordinates (in Å), z is along z^*]

$$1 \quad -0{\cdot}6923x - 0{\cdot}5483y - 0{\cdot}4691z + 1{\cdot}6778 = 0$$
$$2 \quad -0{\cdot}6957x - 0{\cdot}5484y - 0{\cdot}4641z + 1{\cdot}6675 = 0$$
$$3 \quad 0{\cdot}6879x - 0{\cdot}5484y - 0{\cdot}4755z + 1{\cdot}6829 = 0$$
$$4 \quad 0{\cdot}7960x - 0{\cdot}1816y - 0{\cdot}5775z + 4{\cdot}1053 = 0$$
$$5 \quad 0{\cdot}3614x + 0{\cdot}5689y - 0{\cdot}7387z + 2{\cdot}4856 = 0$$

Angles between the planes

1 to 4	100·4°
2 to 3	0·8

† Excluded from the calculation.

A projection of the structure along **b**, illustrating the packing of the molecules, is shown in Fig. 3.

References

AMMON, H. L. & WHEELER, G. L. (1974). *Acta Cryst.* B**30**, 1146–1154.

CHIARONI, A., DOUCERAIN, H. & RICHE, C. (1976). *Acta Cryst.* B**32**, 1920–1922.

KING, G. S. D. & REIMLINGER, H. (1971). *Chem. Ber.* **104**, 2694–2701.

KITAIGORODSKY, A. I. (1973). *Molecular Crystals and Molecules*. New York, London: Academic Press.

RAMACHANDRAN, G. N. & SASISEKHARAN, V. (1968). *Advanc. Protein Chem.* **23**, 283.

RIBÁR, B., DIVJAKOVIĆ, V., JANIĆ, J., ARGAY, GY., KÁLMÁN, A. & DJURIĆ, S. (1974). *Cryst. Struct. Commun.* **3**, 323–325.

ROZWADOWSKA, M. D. (1977). *Can. J. Chem.* **55**, 164–170.

Syntex (1976). The Syntex XTL/E–XTL structure determination system. Syntex Analytical Instruments, Inc., 10040 Bubb Road, Cupertino, California 95014.

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1-(2-Pyridyl)-5-methylimidazole

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Abstract. $C_9H_9N_3$, triclinic, $P\bar{1}$, $a = 7{\cdot}271$ (2), $b = 6{\cdot}533$ (2), $c = 9{\cdot}685$ (3) Å, $\alpha = 114{\cdot}12$ (3), $\beta = 100{\cdot}93$ (2), $\gamma = 93{\cdot}42$ (3)° ($\lambda = 1{\cdot}5418$ Å), $U = 407{\cdot}5$ Å^3, $Z = 2$, $D_x = 1{\cdot}30$, $D_m = 1{\cdot}30$ g cm^{-3}. The structure was solved by direct methods, and least-squares refinement gave $R = 0{\cdot}047$ for 1068 unique significant reflections whose intensities were measured by counter diffractometry. The analysis identified the molecule as the title compound. The 5-methyl substituent is co-

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