

Fig. 3. Molecular packing viewed down *c*.

91–61°]. This type of intramolecular interaction is quite likely between the C(3)—H and ether oxygen, and is favoured sterically in the formation of a stable five-membered ring. There are no abnormal intermolecular contacts and the structure is stabilized by van der Waals interactions.

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Structure of Ethyl 1-(2-Cyanoethyl)-5-methoxy-3-methylindole-2-carboxylate

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Abstract. $C_{16}H_{18}N_2O_3$, $M_r = 286.34$, triclinic, $P\bar{1}$, $a = 9.120$ (5), $b = 11.954$ (2), $c = 14.877$ (2) Å, $\alpha = 105.65$ (1), $\beta = 102.16$ (2), $\gamma = 78.84$ (2)°, $Z = 4$, $V = 1510.5$ Å³, $D_x = 1.259$, $D_m = 1.25$ (5) Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.05$ mm⁻¹, $F(000) = 608$, $T = 300$ K, final $R = 0.065$ for 2459 observed reflections. There are two independent molecules per asymmetric unit. The indole ring system is planar. The ethyl carboxylate group makes an angle of 13.7 (6)° with the mean plane of the indole rings in molecule *A* and 18.5 (6)° in molecule *B*. The cyanoethyl group is inclined at an angle of 89° to the mean plane of the indole rings in both molecules.

Introduction. Indole derivatives exhibit important pharmacological properties, such as being a CNS depressant, and have muscle relaxant properties (Harris & Uhle, 1960; Wei & Stanley, 1970; Reynolds & Carson, 1970; Houlihah, 1973; White & Black, 1976; Ho, Haegman & Prisco, 1986). Reduction of the title compound in the presence of lithium aluminium hydride and cyclization of the resulting amino compound yields a novel compound, 9-methoxy-11-methyl-1,2,3,4-tetrahydro-1*H*-1,4-diazepino[1,2-*a*]indole which exhibits a marked tranquilizing property (Archer & Sternbach, 1968). The crystallographic study of the title compound was

undertaken to establish the structure unambiguously since very little information is available on non-steroidal indole derivatives (Chakraborty & Talapatra, 1986). The present work forms part of the investigations of some pharmacologically important indoles and their precursors. The compound was synthesized by treating ethyl 5-methoxy-3-methyl-indole-2-carboxylate with acrylonitrile in the presence of the basic catalyst Triton B.

Experimental. Colorless transparent crystals grown by diffusion of hexane into benzene solution, crystal 0.2 × 0.2 × 0.2 mm mounted on an Enraf-Nonius CAD-4 diffractometer, $\omega/2\theta$ scan; cell dimensions from least-squares refinement of 25 centred reflections in the range $7 < \theta < 12^\circ$, density by flotation technique, intensity data using Mo $K\alpha$ radiation, to a maximum $2\theta = 60^\circ$; $0 \leq h \leq 9$, $-12 \leq k \leq 12$, $-15 \leq l \leq 15$; three standard reflections ($12\bar{6}$, $1\bar{2}6$, 225) monitored after every 2 h, a total of 3711 independent reflections of which 2459 reflections with $I \geq 3\sigma(I)$ were considered observed; data corrected for Lorentz-polarization effects, no absorption correction. The structure was solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), hydrogens attached to the ring C atoms located from successive difference Fourier maps, other hydrogens fixed by stereochemical considerations, full-matrix least-squares refinement with anisotropic thermal parameters for non-H atoms and isotropic for H atoms using *SHELX76* (Sheldrick, 1976) with unit weights. The final $R = 0.065$, $wR = 0.065$. The refinement was terminated when the shift/e.s.d. for the non-H atoms was less than 0.2, the minimum and maximum residual electron densities in the final difference Fourier map were -0.26 and $0.23 \text{ e } \text{\AA}^{-3}$, respectively.* Scattering factors for C, N, O and H were those incorporated in *SHELX76*.

Discussion. The final positional parameters of non-H atoms and U_{eq} are given in Table 1. The bond lengths and angles of molecules *A* and *B* are given in Table 2. An *ORTEP* (Johnson, 1965) plot of one of the molecules with 50% probability thermal ellipsoids and the atomic numbering is shown in Fig. 1.

The indole ring system is planar in both the molecules with a maximum deviation of 0.019 (6) Å in molecule *A* and 0.018 (5) Å in molecule *B*. The mean plane of the ethyl carboxylate group is inclined to the indole rings at an angle of 13.7 (6)° in molecule *A* and 18.5 (6)° in molecule *B*. The ester moiety is *syn*

* 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 53090 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$) for non-H atoms, with e.s.d.'s in parentheses

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
N(1)	8088 (5)	2888 (4)	7134 (3)	631 (19)
C(2)	7086 (6)	3937 (4)	7333 (3)	514 (20)
C(3)	6660 (5)	4073 (4)	8190 (3)	468 (19)
C(4)	7398 (6)	2747 (4)	9369 (3)	539 (21)
C(5)	8252 (6)	1710 (4)	9503 (3)	568 (22)
C(6)	9126 (6)	1011 (4)	8832 (4)	680 (25)
C(7)	9132 (7)	1306 (4)	8007 (4)	687 (25)
C(8)	8289 (6)	2374 (4)	7881 (3)	522 (20)
C(9)	7405 (5)	3085 (4)	8541 (3)	451 (19)
C(10)	5584 (6)	5034 (4)	8693 (4)	649 (23)
O(11)	8369 (5)	1270 (3)	10279 (3)	758 (18)
C(12)	7503 (8)	1909 (5)	10973 (4)	859 (30)
C(13)	9034 (8)	2480 (6)	6350 (5)	961 (32)
C(14)	8097 (8)	1739 (6)	5646 (6)	1055 (38)
C(15)	9088 (9)	1254 (6)	4777 (5)	1112 (37)
N(16)	9575 (6)	820 (4)	4142 (4)	925 (26)
C(17)	6609 (6)	4652 (5)	6638 (4)	606 (24)
O(18)	6860 (5)	4376 (3)	5847 (3)	836 (19)
O(19)	5814 (4)	5679 (3)	7000 (2)	707 (16)
C(20)	5087 (7)	6414 (5)	6351 (4)	733 (26)
C(21)	4052 (8)	7367 (6)	6869 (5)	973 (33)
N(1)	6524 (5)	7562 (3)	2534 (3)	638 (19)
C(2)	7089 (6)	7367 (4)	1699 (3)	533 (21)
C(3)	8081 (5)	6335 (4)	1582 (3)	517 (21)
C(4)	9018 (6)	4880 (4)	2668 (4)	602 (23)
C(5)	8845 (6)	4696 (5)	3499 (4)	664 (25)
C(6)	7844 (7)	5486 (5)	4069 (4)	765 (28)
C(7)	7029 (7)	6456 (5)	3814 (4)	700 (26)
C(8)	7197 (6)	6658 (4)	2962 (3)	554 (21)
C(9)	8164 (5)	5881 (4)	2382 (3)	510 (20)
C(10)	8999 (6)	5768 (5)	807 (4)	714 (25)
O(11)	9590 (5)	3757 (4)	3868 (3)	927 (21)
C(12)	642 (8)	2953 (6)	3352 (6)	1060 (37)
C(13)	5360 (7)	8491 (5)	2910 (4)	731 (26)
C(14)	6121 (7)	9482 (5)	3573 (4)	808 (29)
C(15)	4931 (9)	10417 (5)	3994 (5)	917 (33)
N(16)	4084 (8)	11123 (5)	4330 (4)	1145 (32)
C(17)	6686 (6)	8246 (5)	1134 (4)	641 (25)
O(18)	6104 (6)	9240 (4)	1397 (3)	997 (22)
O(19)	7090 (5)	7820 (3)	283 (2)	741 (17)
C(20)	6901 (8)	8652 (6)	-299 (4)	901 (32)
C(21)	7666 (9)	8045 (7)	-1124 (5)	1092 (38)

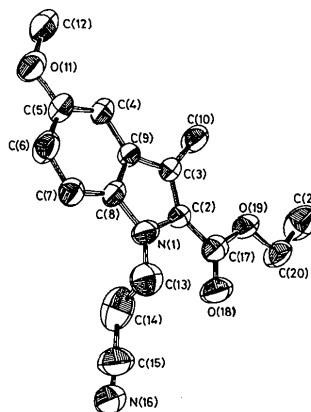


Fig. 1. *ORTEP* plot of one of the molecules with 50% probability thermal ellipsoids and the atomic numbering scheme.

to C=O about the C—O bond; the torsion angle C(20)—O(19)—C(17)—O(18) is 7.5 (8)° in molecule *A* and 4.7 (8)° in molecule *B*.

The bond lengths and angles in the indole rings are normal. The two C—N bonds in molecule *B* are

Table 2. Bond lengths (Å) and angles (°) for molecules *A* and *B*, with e.s.d.'s in parentheses

N(1)—C(2)	1.402 (6)	C(6)—C(7)	1.367 (9)
	1.389 (7)		1.351 (8)
N(1)—C(8)	1.372 (7)	C(7)—C(8)	1.395 (7)
	1.378 (7)		1.396 (9)
N(1)—C(13)	1.516 (9)	C(8)—C(9)	1.399 (6)
	1.460 (7)		1.399 (7)
C(2)—C(3)	1.369 (7)	O(11)—C(12)	1.399 (8)
	1.375 (6)		1.410 (8)
C(2)—C(17)	1.460 (8)	C(13)—C(14)	1.424 (9)
	1.464 (8)		1.502 (8)
C(3)—C(9)	1.414 (6)	C(14)—C(15)	1.645 (11)
	1.420 (8)		1.503 (9)
C(3)—C(10)	1.501 (6)	C(15)—N(16)	1.087 (9)
	1.501 (8)		1.114 (9)
C(4)—C(5)	1.371 (7)	C(17)—O(18)	1.192 (7)
	1.358 (9)		1.199 (7)
C(4)—C(9)	1.397 (8)	C(17)—O(19)	1.337 (6)
	1.411 (7)		1.332 (7)
C(5)—C(6)	1.399 (7)	O(19)—C(20)	1.452 (7)
	1.416 (8)		1.450 (9)
C(5)—O(11)	1.368 (7)	C(20)—C(21)	1.483 (8)
	1.379 (7)		1.485 (10)
C(2)—N(1)—C(8)	107.6 (4)	C(7)—C(8)—C(9)	121.7 (5)
	108.0 (4)		121.5 (5)
C(2)—N(1)—C(13)	127.5 (5)	C(7)—C(8)—N(1)	129.8 (5)
	128.8 (5)		130.3 (5)
C(8)—N(1)—C(13)	123.9 (4)	C(9)—C(8)—N(1)	108.4 (4)
	123.1 (5)		108.2 (5)
N(1)—C(2)—C(3)	109.4 (4)	C(8)—C(9)—C(3)	107.7 (4)
	109.5 (5)		107.5 (4)
N(1)—C(2)—C(17)	119.7 (4)	C(8)—C(9)—C(4)	119.7 (4)
	120.3 (4)		119.7 (5)
C(3)—C(2)—C(17)	130.8 (4)	C(3)—C(9)—C(4)	132.6 (4)
	130.0 (5)		132.7 (5)
C(2)—C(3)—C(9)	107.0 (4)	C(5)—O(11)—C(12)	117.4 (4)
	106.8 (4)		117.0 (6)
C(2)—C(3)—C(10)	129.2 (4)	N(1)—C(13)—C(14)	101.0 (6)
	129.5 (5)		108.7 (5)
C(9)—C(3)—C(10)	123.8 (5)	C(13)—C(14)—C(15)	103.1 (6)
	123.7 (4)		108.8 (5)
C(9)—C(4)—C(5)	118.5 (4)	C(14)—C(15)—N(16)	168.7 (8)
	118.2 (5)		177.8 (7)
C(4)—C(5)—C(6)	120.8 (5)	C(2)—C(17)—O(18)	126.0 (5)
	121.2 (5)		125.4 (5)
C(4)—C(5)—O(11)	125.2 (5)	C(2)—C(17)—O(19)	110.8 (5)
	124.9 (5)		111.6 (4)
C(6)—C(5)—O(11)	114.0 (4)	O(18)—C(17)—O(19)	123.2 (5)
	113.9 (6)		123.0 (6)
C(5)—C(6)—C(7)	122.0 (5)	C(17)—O(19)—C(20)	116.9 (4)
	121.6 (6)		116.3 (4)
C(6)—C(7)—C(8)	117.1 (5)	O(19)—C(20)—C(21)	106.3 (5)
	117.8 (5)		106.8 (5)

almost equal having an average value of 1.383 (7) Å but differ by 0.03 Å in molecule *A*. The average bond length of C(3)—C(9) = 1.417 (7) Å is slightly longer than the standard aromatic C—C bond of 1.395 Å but is smaller than the values observed in a number of non-steroidal indole derivatives (Falkenberg & Carlstrom, 1971; Vijayalakshmi & Srinivasan, 1975; Sakaki, Sogo, Wakahara, Kanai, Fujiwara & Tomita, 1976; Chandrashekar & Pattabhi, 1980; Shoja, 1988). The bond length C(2)—C(3) is shorter than the normal aromatic C—C bond and is in agreement with the values observed in other indole derivatives. The bond angle C(4)—C(5)—O(11) is larger than C(6)—C(5)—O(11) by 11.1° and this may be due to the *cis* orientation of C(4)—C(5) and O(11)—C(12) about the C(5)—O(11) bond causing the bond angle to be larger as a result of the repulsion between C(4) and C(12) (Sakaki *et al.*, 1976).

Fig. 2. Stereoview of the unit-cell contents along *b*.

The C=O distances are normal. The apparent change in the bond lengths of the ethyl groups might be due to strong thermal vibrations. The bond lengths and angles of the cyanoethyl group in molecule *B* are in agreement with the values observed in other structures (Waite & Sim, 1971; Grigg, Jordan, Tangthongkum, Einstein & Jones, 1984); however, the corresponding values in molecule *A* have larger variations compared to the reported values and this may be due to the relatively high temperature factors associated with the atoms of the cyanoethyl group of molecule *A*.

A stereoview of the unit-cell contents along *b* is shown in Fig. 2. There are a few short contacts less than 3.4 Å. There are no intermolecular hydrogen bonds and the molecules are held by van der Waals interactions.

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Methyl 1,6-Dihydro-1-(dimethylcarbamoyl)-4-methyl-6-oxo-2-phenyl-3-pyridinecarboxylate

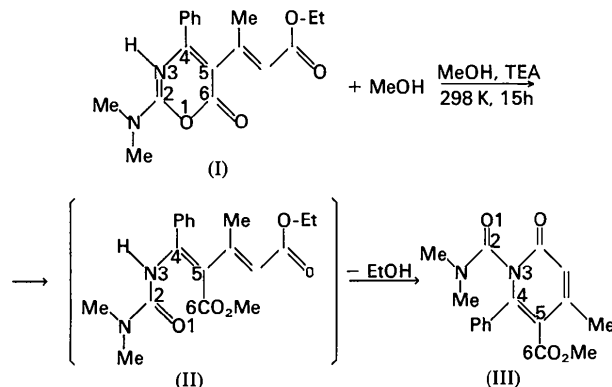
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Abstract. C₁₇H₁₈N₂O₄, $M_r = 314.34$, monoclinic, $P2_1/n$, $a = 8.494$ (1), $b = 18.303$ (3), $c = 10.364$ (2) Å, $\beta = 101.51$ (1)°, $V = 1578.8$ (4) Å³, $Z = 4$, $D_x = 1.322$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 8.9$ mm⁻¹, $F(000) = 664$, room temperature, final $R = 0.041$ for 2051 observed reflections with $I > \sigma(I)$. Crystal grown by evaporation from ethyl ether and *n*-hexane. The title compound is an unexpected product of the alcoholysis of a substituted 6*H*-1,3-oxazin-6-one. The heterocyclic ring has an envelope conformation with C(2) out of the plane of the other atoms. The deviation from planarity is due to intermolecular interactions.

Introduction. As part of a continuing investigation on the structure and reactivity of 1,3-oxazin-6-ones and of their reaction products (Becalli, Benincori & Marchesini, 1988; Becalli & Marchesini, 1987; Becalli, Marchesini & Pilati, 1989; Pilati, 1988, 1989), we report the crystal and molecular structure of the title compound (III), obtained by Marchesini (1989), from the substituted 1,3-oxazin-6-one (I), according to the following scheme.



The title compound was completely unexpected; in fact, under the same conditions, the reaction of other 4,5-substituted 2-(*N,N*-dimethylamino)-1,3-oxazin-6-ones gives acyclic compounds like (II), or products in which the dimethylamino group is shifted from position 2 to position 6. This X-ray crystal analysis was undertaken to confirm the structure of this new product.

Experimental. IR data (Nujol): 1716, 1668, 1602 cm⁻¹; ¹H NMR data (CHCl₃): 7.4 (5*H*,*m*), 6.4 (1*H*,*s*), 3.43 (3*H*,*s*), 2.85 (3*H*,*s*), 2.80 (3*H*,*s*), 2.3 (3*H*,*s*); melting point 396–397 K. Crystal size 0.35 × 0.25 × 0.075 mm. Nonius CAD-4 diffractometer with graphite monochromator; lattice parameters determined using 25 reflections in the θ range 13–15°; data collection $0 < \theta < 25^\circ$; hkl range: 0→10, 0→21, -12→12; three standard reflections, no significant variation of intensity. Of 2765 independent data collected, 2051 with $I > \sigma(I)$ were considered observed. Data were corrected for Lorentz and polarization coefficients, not for absorption. The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). All heavy atoms were obtained from the 'best' *E* map. H atoms placed in calculated positions, refined isotropically, non-H anisotropically. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 4I_o/[\sigma^2(I_o) + 0.0001I_o^2]$ and I_o is the intensity on an absolute scale. A secondary-extinction parameter g [Larson (1967), equation (3)] was refined [final value $6.0(7) \times 10^{-6}$]. The range of the final difference Fourier map was $\pm 0.2 \text{ e \AA}^{-3}$; in the last cycle, $(\Delta/\sigma)_{\text{max}}$ was 0.05; $S = 1.90$, $R = 0.041$ and $wR = 0.034$ for 2051 observed reflections. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).