

Triclinic

*P*1̄*a* = 9.296 (2) Å*b* = 10.524 (3) Å*c* = 11.044 (3) Å α = 113.89 (2)° β = 100.15 (2)° γ = 109.75 (2)°*V* = 866.8 (4) Å³*Z* = 2*D_x* = 1.376 Mg m⁻³*D_m* not measured

Data collection

Enraf–Nonius CAD-4

diffractometer

 $\omega/2\theta$ scans

Absorption correction: none

3245 measured reflections

3039 independent reflections

2060 reflections with

I > 2 σ (*I*)

Refinement

Refinement on *F*² $R[F^2 > 2\sigma(F^2)] = 0.067$ $wR(F^2) = 0.150$ *S* = 1.232

3039 reflections

281 parameters

All H atoms refined

 $w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.9770P]$ where $P = (F_o^2 + 2F_c^2)/3$

Cell parameters from 25

reflections

 $\theta = 5-12^\circ$ $\mu = 0.382 \text{ mm}^{-1}$ *T* = 293 (2) K

Plate

0.6 × 0.3 × 0.1 mm

Pale yellow

*R*_{int} = 0.023 $\theta_{\text{max}} = 24.98^\circ$ *h* = 0 → 11*k* = -12 → 11*l* = -13 → 12

2 standard reflections

every 200 reflections

intensity decay: none

 $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.239 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.298 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1102). Services for accessing these data are described at the back of the journal.

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Ethyl 1-(2-cyanoethyl)-3,5-dimethyl-1*H*-indole-2-carboxylate

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Abstract

The title compound, C₁₆H₁₈N₂O₂, is an important precursor in the synthesis of 1,2,3,4-tetrahydropyrazinoindoles, which show excellent antihistamine, antihypertensive and central nervous system depressant properties. The carbethoxy group attached to C2 and the planar cyanoethyl group attached to N1 make dihedral angles of 11.0 (4) and 75.0 (3)°, respectively, with the mean plane of the indole ring. The C—C=N chain is linear with a bond angle of 179.3 (4)°.

Comment

The title compound belongs to the family of indole derivatives. Interest in these compounds is increasing with the realisation of their role in obtaining phar-

Table 3. Selected torsion angles (°)

N1—C3—C14—C15	−158.3 (4)	N1—C3—C14—C19	21.9 (5)
C2—C3—C14—C15	87.0 (5)	C2—C3—C14—C19	−92.8 (5)

Table 4. Hydrogen-bonding geometry (Å, °) for (II)

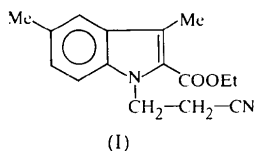
D—H...A	D—H	H...A	D...A	D—H...A
C3—H3...C11	0.95 (5)	2.68 (3)	3.032 (4)	102.9 (3)
C7—H7...C12	0.93 (4)	2.62 (5)	3.046 (5)	109.0 (3)

Two standard reflections (12 $\bar{4}$ and 21 $\bar{2}$) were used to monitor crystal decay in (II). All H atoms were unambiguously located in difference Fourier calculations and they were included in the refinement with isotropic displacement parameters.

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structures: *SHELXS97* (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *PLUTO* (Motherwell & Clegg, 1978); software used to prepare material for publication: *SHELXL97*.

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macologically active compounds of the type 1,2,3,4-tetrahydropiperazino[1,2-*a*]indoles and diazepino[1,2-*a*]indoles which are found to possess central nervous system depressant (Yamamoto *et al.*, 1974), antihypertensive (Merck, 1973) and muscle-relaxant (Hendi & Basangoudar, 1981) properties. As a continuation of our work on substituted 2-carbethoxy indoles and the effect of cyanoethyl and cyanomethyl groups on the nature of the crystal structure, the present paper reports the solid-state conformation of the title compound. In the case of 5-methoxy-3-phenyl derivatives, substitution on the indole-N atom by a cyanomethyl group results in a change in space group from monoclinic *C2/c* to orthorhombic *Pbca*. In the case of 5-methoxy-3-methyl derivatives, substitution by a cyanoethyl group brings about a remarkable change in crystal packing, but no change in the space group (*P1*). In light of the above observations, the crystal structure of the title compound, (I), has been determined.



The two methyl groups attached to the indole moiety at C3 and C5 are in the plane of the indole rings with deviations -0.021 (5) and 0.016 (4) Å, respectively. The planar cyanoethyl group attached to N1 is inclined to the indole plane at an angle 75.0 (3)°. The carbethoxy group attached to C2 is almost planar and the mean plane through this group makes a dihedral angle of 11.0 (4)° with the plane of the indole ring.

The β -cyanoethyl group is twisted towards the fused benzene ring. The methylene-C atom (C15) attached to the indole-N atom and the carbonyl-O atom (O11) of the ester moiety bear a *peri* relationship to each other.

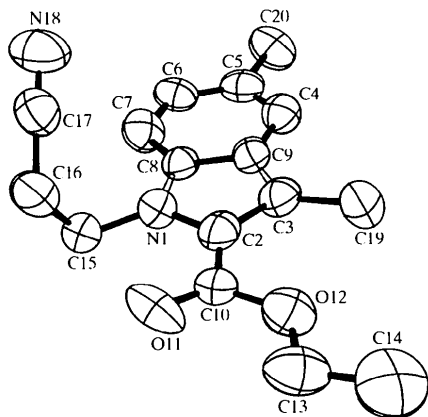


Fig. 1. ORTEP (Johnson, 1965) plot of the molecule drawn with 60% probability displacement ellipsoids.

The aromatic carbon and methyl group bond distances C3—C19 = 1.483 (4) and C5—C20 = 1.509 (5) Å may indicate a greater delocalization of the nitrogen lone pair in the pyrrole moiety than in the benzene ring, or (perhaps more likely) the difference may be due to extended conjugation within the five-membered ring and the carboxylate group. The bond distance 1.526 (4) Å for C15—C16 falls in the range of Csp^3 — Csp^3 bond lengths and the C16—C17 distance of 1.456 (4) Å is shortened due to the electron-withdrawing cyano group.

The angles around the indole-C atoms C2 and C3, *i.e.* C3—C2—C10 = 130.0 (3) and C2—C3—C19 = 128.4 (3)°, are greater than usual to keep the steric repulsions to a minimum, which is also observed in other indocarboxylates (Chandrakantha, Nirmala *et al.*, 1990; Chandrakantha, Puttaraja & Pattabhi, 1990, 1991; Chandrakantha, Puttaraja & Nethaji, 1990).

In the five-membered ring, the two C—N bonds are unequal and similar observations are made in other derivatives investigated (Chandrakantha, Puttaraja & Pattabhi, 1990, 1991). The C—C=N chain is linear and the bond angle C16—C17—N18 is 179.3 (4)°.

Experimental

The title compound was synthesized in two steps. In the first step, the indole derivative was synthesized by the reaction of ethyl 2-oxo-butanoate of *p*-tolylphenylhydrazine with dry HCl gas. This compound, when made to react with β -chloroethylcyanide and sodium hydride, yielded the title compound.

Crystal data

$C_{16}H_{18}N_2O_2$

$M_r = 270.32$

Triclinic

P1

$a = 5.925$ (2) Å

$b = 8.321$ (1) Å

$c = 16.126$ (3) Å

$\alpha = 103.41$ (1)°

$\beta = 98.85$ (2)°

$\gamma = 99.51$ (2)°

$V = 747.3$ (3) Å³

$Z = 2$

$D_x = 1.201$ Mg m⁻³

$D_m = 1.190$ Mg m⁻³

D_m measured by flotation
in potassium iodide and
water

Cu $K\alpha$ radiation

$\lambda = 1.5418$ Å

Cell parameters from 25
reflections

$\theta = 10$ – 50 °

$\mu = 0.643$ mm⁻¹

$T = 293$ (2) K

Needle

$0.30 \times 0.25 \times 0.25$ mm

Yellow

Data collection

Enraf–Nonius CAD-4
diffractometer

ω - 2θ scans

Absorption correction: none

2103 measured reflections

2103 independent reflections

1721 reflections with

$F^2 > 2\sigma(F^2)$

$\theta_{\max} = 74.82$ °

$h = -7 \rightarrow 7$

$k = 0 \rightarrow 10$

$l = -20 \rightarrow 19$

3 standard reflections

every 100 reflections

intensity decay: none

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.064$ $wR(F^2) = 0.182$ $S = 1.101$

2051 reflections

201 parameters

H atoms: see text

 $w = 1/[\sigma^2(F_o^2) + (0.1048P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = -0.001$ $\Delta\rho_{\max} = 0.158 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.160 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Scattering factors from

*International Tables for
Crystallography* (Vol. C)Table 1. Selected geometric parameters ($\text{\AA}$, $^\circ$)

N1—C8	1.378 (4)	C5—C20	1.509 (5)
N1—C2	1.392 (3)	C15—C16	1.526 (4)
N1—C15	1.463 (3)	C16—C17	1.456 (4)
C3—C19	1.486 (4)		
C3—C2—C10	130.0 (3)	N18—C17—C16	179.3 (4)
C2—C3—C19	128.4 (3)		

All the non-H atoms were refined using *SHELXL93* (Sheldrick, 1993) with anisotropic displacement parameters. H atoms were revealed in difference Fourier maps, but only those attached to C13 and C15 were refined, and all others were treated with a riding model.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993). Data reduction: *DATRD2* in *NRCVAX* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1055). Services for accessing these data are described at the back of the journal.

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Dimethyl oleana-13(18),15(16)-diene-3 β ,28-diacetate

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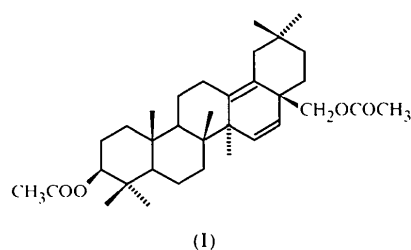
(Received 3 November 1998; accepted 4 February 1999)

Abstract

The title compound, C₃₄H₅₂O₄, consists of five six-membered rings. All of the rings except the one with two double bonds exist in chair form. The ring with two double bonds is almost planar with a mean deviation from planarity of only 0.03 Å.

Comment

The title compound, (I), has been isolated from the leaves of the plant *Jacquinia Ruscifolia* which is found in Mexico. The leaves of the plant contains saponins which are generally used as fish poison. The compound has been studied chemically and NMR spectroscopic studies have also been carried out (Mahato & Kundu, 1994). The present X-ray analysis was initiated in order to resolve the details of the molecular structure derived initially from detailed spectral analyses.



All five rings in the common oleanane-type triterpenes are known to exist mainly in the chair form. In the case of the present compound, rings A, B, C and E are in the chair conformation. However, due to the presence of the two double bonds, ring D is almost planar. The mean deviation from planarity of ring D is 0.0315 Å. The acetate group at C3 is almost planar, with a torsion angle C32—C31—O1—C3 = 178.1 (5)°. The dihedral angle between the mean plane formed by the acetate group and ring A is 121.8 (4)° which indicates