Triclinic
$P\bar{1}$
a = 8.900 (2) Å
b = 11.508 (2) Å
c = 7.7675 (10) Å
α = 94.713 (14)°
β = 107.873 (13)°
γ = 67.784 (14)°
V = 700.4 (2) Å^3
Z = 2
D_x = 1.495 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 30–40°
μ = 0.398 mm^{-1}
T = 293 (2) K
Block
0.30 × 0.25 × 0.20 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6290 measured reflections
3203 independent reflections
2489 reflections with $I > 2\sigma(I)$

R_{int} = 0.018
θ_{max} = 27.51°
$h = -10 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 9$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.099
S = 1.026
3200 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.1658P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.267 e Å^{-3}
$\Delta\rho_{min}$ = −0.292 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—O1	1.436 (2)	S1—C11	1.756 (2)
S1—N2	1.564 (2)	S1—C21	1.761 (2)
O1—S1—N2	120.21 (11)	O1—S1—C21	110.17 (10)
O1—S1—C11	109.07 (9)	N2—S1—C21	102.92 (10)
N2—S1—C11	103.61 (10)	C11—S1—C21	110.44 (9)

The hydrogen sulfate anion was found to be disordered. Three sets of O atoms were refined with equivalent displacement parameters and idealized tetrahedral geometries. Aromatic H atoms were constrained with a riding model [$U_H = 1.2U_{iso}$(C)]. Both N—H and hydrogen sulfate H atoms (major component only) were located in a difference map and their coordinates refined with a fixed displacement parameter [$U_H = 1.2U_{iso}$(N) and $U_H = 0.08 \times 10^3$ Å^2, respectively].

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1214). Services for accessing these data are described at the back of the journal.

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Rotundifoline, an Oxoindole Alkaloid

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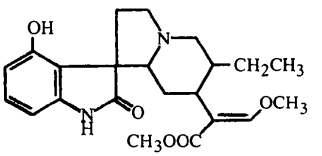
Abstract

In the title compound, methyl 2-{6′-ethyl-2′,3′,5′,6′,-7′,8′-hexahydro-4-hydroxy-2-oxo-spiro[1*H*-indole-3(2*H*),1′(8a′*H*)-indolizin]-7′-yl}-3-methoxyacrylate, $C_{22}H_{28}N_2O_5$, the indole molecule is not planar. The planarity of the atom group C13—N1—C2═O1 of the indole moiety and the short N1—C2 bond of 1.363 (11) Å are due to delocalization of the benzoid electrons, which extend over the atoms N1, C2 and O1. The five-membered ring of the indolizine moiety is puckered and the six-membered ring fused to it has a normal chair conformation. The methoxycarbonyl and the methoxy groups have a *trans* configuration about the C16═C17 bond in the acrylate moiety. The structure is stabilized by intramolecular hydrogen bonding of the type O—H···N and intermolecular hydrogen bonding of the type N—H···O.

Comment

Indole alkaloids are known for their interesting chemical and physiological activities. As part of a continuing structural study of indole and oxoindole compounds (Chakraborty *et al.*, 1985, 1991; Chakraborty & Talapatra, 1986), the structural analysis of the title compound, (I), was undertaken to confirm the conclusion of chemical work and to provide further structural data for the indole alkaloid obtained from the natural source.



(I)

The title compound was obtained from the leaves of *Mitragyna rubro stipulata* (schum), from Havil, Uganda, East Africa (Shellard & Lala, 1978). The genus *Mitragyna* is known to possess opiate, analgesic and sedative properties (Shellard, 1972). The individual bond lengths and angles of the indole ring are in agreement with those of similar indole alkaloids, such as (±)-21-oxoisopterpodine (Lynch *et al.*, 1991) and mitragyanine hydroiodide (Zacharias *et al.*, 1965). The indole system is not planar: its two rings are inclined to each other by 3.5 (3)° along the C8—C13 bond. The atoms comprising the C13—N1—C2═O1 least-squares plane do not deviate from it by more than 0.03 Å. This planarity and the short N1—C2 bond of 1.363 (11) Å are due to the delocalized π-electron system, which extends over N1, C2 and O1 (James & Williams, 1972). The five-membered pyrrole ring is slightly puckered, the puckering parameter q_2 (Cremer & Pople, 1975) being 0.492 (8) Å.

The methoxy and methoxycarbonyl groups are attached mutually *trans* about the C16═C17 double bond of the acrylate moiety.

The puckering parameters for the piperidine ring have been calculated using the method of Cremer & Pople (1975) and are φ_2 = 62 (9)°, q_2 = 0.046 (8) Å, q_3 = 0.600 (8) Å, θ = 4.4 (8)° and Q_T = 0.602 (8) Å. The puckering parameters indicate a slightly distorted chair conformation; the Q_T value lies close to the value of 0.63 Å for an ideal cyclohexane chair (Cremer & Pople, 1975). The five-membered ring of the indolizine system, which is connected *via* the spiro atom C7 to the pyrrole ring of the indole moiety is puckered with φ_2 = 151 (7)° and q_2 = 0.064 (8) Å.

The NH group of the pyrrole ring is engaged in an intermolecular hydrogen bond with atom O5 of the hydroxy group in a neighbouring molecule, having N1···O5^{i} 2.805 (9), H1N···O5^{i} 2.10 Å and N1—H1N···O5^{i} 140° [symmetry code: (i) $-x$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$]. The hydroxy moiety also forms a rather strong intramolecular hydrogen bond with the N4 atom of the indolizine moiety, with O5···N4 2.613 (10), H5O···N4 1.81 (8) Å and O5—H5O···N4 168 (4)°.

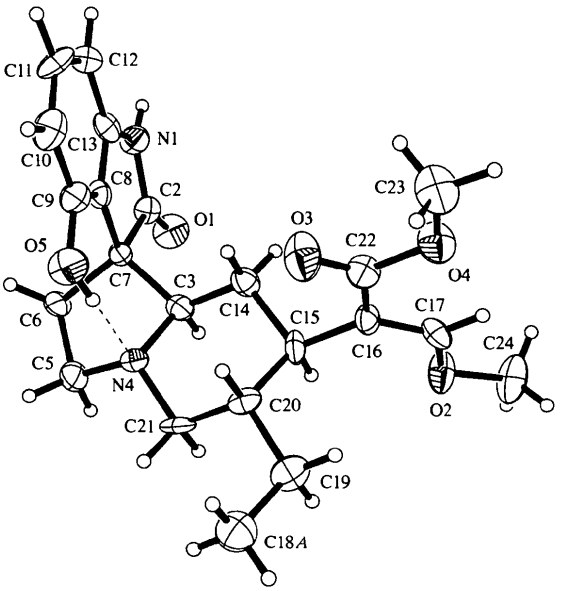


Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the numbering of the non-H atoms. H atoms have been assigned small arbitrary radii for clarity. Only the major [0.71 (6)] component of the disorder affecting C18 is shown.

Experimental

The title compound was obtained from a solution of the compound in absolute alcohol, by slow evaporation at room temperature.

Crystal data

$C_{22}H_{28}N_2O_5$	Mo $K\alpha$ radiation
M_r = 400.46	λ = 0.71073 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 10–15°
a = 8.313 (1) Å	μ = 0.090 mm^{-1}
b = 13.367 (1) Å	T = 293 (2) K
c = 18.981 (1) Å	Plate
V = 2109.2 (3) Å^3	0.26 × 0.24 × 0.10 mm
Z = 4	Colourless
D_x = 1.261 Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 25.1°
ω–2θ scans	h = 0 → 9
Absorption correction: none	k = 0 → 15
2141 measured reflections	l = 0 → 22
2141 independent reflections	1 standard reflection
1029 reflections with $I > 2\sigma(I)$	frequency: 30 min
	intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.082$
$wR(F^2) = 0.245$
$S = 1.058$
2141 reflections
264 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.12P)^2 + 1.51P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.013$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.013 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

One atom (C18) was found to be affected by disorder: this was modelled in terms of two unequal sites of occupancies 0.71 (6) and 0.29 (6). Residual disorder is thought to be largely responsible for the high conventional R values. The distances between these isotropic components and C19 were restrained to be 1.52 (1) Å. H atoms were included at geometrically calculated positions, except for the hydroxy H atom (H5O) on O5 which was found from a circular Fourier synthesis. They were then allowed to ride on their parent atoms with $U_{iso} = xU_{eq}$(parent), where $x = 1.5$ for methyl and hydroxy H atoms and $x = 1.2$ for all others. Standard uncertainties on C—C distances range from 0.009 to 0.014 Å.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *MULTAN*78 (Main *et al.*, 1978). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983).

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6-Allyl-8,8-dimethyl-3-oxo-2-(1-phenylethyl)-2-azabicyclo[4.3.0]non-1(9)-ene-5-carboxylic Acid, a Key Compound in the Asymmetric Synthesis of Quadrone

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Abstract

The X-ray structure analysis of the title compound, $C_{22}H_{27}NO_3$, establishes unambiguously the relative and absolute configurations of the two asymmetric C atoms, C5 (*S*) and C6 (*S*), based on the known *R* absolute configuration of the C10 atom, and provides essential information on the transition state of the Michael reaction leading to its formation.

Comment

The enantioselective synthesis of quaternary carbon centres through the Michael addition of chiral imines to electrophilic alkenes under neutral conditions (d'Angelo *et al.*, 1992) has been used for the asymmetric synthesis of a large number of biologically active compounds (d'Angelo *et al.*, 1993). For our part, we were interested in the asymmetric synthesis of (−)-quadrone, (1), an antitumour compound isolated from the fungus