

Experimental

The title compound was prepared by oxidation of the corresponding sulfide or sulfoxide with excess hydrogen peroxide in acetic acid. Single crystals were obtained by slow evaporation of an ethanol solution (Chandrasekaran, 1991).

Crystal data

$C_{14}H_{13}ClO_2S$

$M_r = 280.75$

Monoclinic

$C2/c$

$a = 20.239 (5) \text{ \AA}$

$b = 10.911 (2) \text{ \AA}$

$c = 15.197 (6) \text{ \AA}$

$\beta = 126.580 (11)^\circ$

$V = 2694.8 (14) \text{ \AA}^3$

$Z = 8$

$D_x = 1.384 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 14\text{--}18^\circ$

$\mu = 0.429 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Rectangular block

$0.20 \times 0.15 \times 0.15 \text{ mm}$

Transparent

Data collection

Rigaku AFC-7S diffractometer

$\omega/2\theta$ scans

Absorption correction: none

2934 measured reflections

2367 independent reflections

1744 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 25.02^\circ$

$h = 0 \rightarrow 24$

$k = 0 \rightarrow 12$

$l = -18 \rightarrow 14$

3 standard reflections

every 100 reflections

frequency: 60 min

intensity decay: $<1\%$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.153$

$S = 1.056$

2367 reflections

215 parameters

H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 4.2184P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.067$

$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters ($\text{\AA}$, $^\circ$)

S1—O1	1.431 (2)	S1—C1	1.779 (3)
S1—O2	1.437 (2)	Cl—C4'	1.734 (3)
S1—Cl'	1.775 (2)		
O1—S1—O2	116.76 (12)	C6—C1—S1	120.6 (2)
O1—S1—Cl'	108.61 (11)	C2—C1—S1	117.6 (2)
O2—S1—Cl'	106.69 (11)	C6'—C1'—S1	119.2 (2)
O1—S1—C1	110.04 (11)	C2'—C1'—S1	120.2 (2)
O2—S1—C1	110.56 (12)	C3'—C4'—Cl	120.1 (2)
Cl'—S1—C1	103.24 (12)	C5'—C4'—Cl	118.6 (2)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *MSC/AFC Diffractometer Control Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP92* (Vicković, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1313). Services for accessing these data are described at the back of the journal.

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3-Benzoyl-2-methylindole

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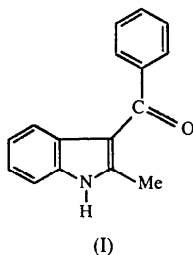
Abstract

The crystal and molecular structure of $C_{16}H_{13}NO$ has been determined. Bond lengths and angles are within normal ranges and molecules are held together in the crystal by intermolecular $N\text{—}H \cdots O$ hydrogen bonding.

Comment

The specific alkylation or acylation of the indole ring is an important question for the preparation of derivatives on position 3 or 1 and the appropriate alkyl or acyl derivatives serve for the preparation of aspidosperm alkaloids in which we are interested (Rodríguez & Urrutia, 1996). Reactions of methylindolylmagnesium salts with benzoyl chloride give 3-benzoyl-2-methylindole, (I), in all cases; this product claimed our attention because of the orange colour of the crystals and

the anomalous IR, proton NMR and mass spectral data, and thus, for structural elucidation, the X-ray diffraction analysis was undertaken.



The structural analysis establishes that the indole ring is substituted by methyl (position 2) and by benzoyl (position 3) groups. Both the indole and benzene rings are individually planar and the dihedral angle between them is 62.83(7)° (Fig. 1). In the indole ring, the maximum deviations from the least-squares planes are −0.020(1) Å for C2, −0.021(2) Å for C6 and 0.025(2) Å for C4. In the benzene ring of the benzoyl group, the maximum deviation is 0.011(1) Å for C17. The O atom lies only 0.337(3) Å from the plane of the indole ring and 0.897(4) Å from the plane of the benzene ring.

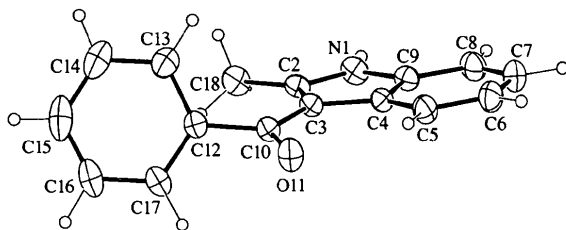


Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids. H atoms are drawn as small circles of arbitrary radii.

Bond lengths and angles are in good agreement with those found in indole (Kaneda & Tanaka, 1976) and other analogous compounds (Cameron, Prout, Denton, Spagna & White, 1975; Rodriguez, Temprano, Esteban-Calderon & Martinez-Ripoll, 1989; Schollmeyer, Fisher & Pindur, 1995; Rodriguez, del Valle, Esteban-Calderon & Martinez-Ripoll, 1995).

Molecules are linked to form chains *via* intermolecular hydrogen bonding between N1 and O11(*x*, −*y*, *z* − ½) [N1⋯O11 2.836(3), H1⋯O11 2.00 Å and N1—H1⋯O11 164°].

The packing is directed by the hydrogen bonds forming columns along the *c* axis. The columns are related by a system of T-contacts between the phenyl rings, formed by two independent ones, with distances between centroids of 4.881(3) and 5.123(4) Å. Moreover, these columns appear related by H⋯H phenyl con-

tacts with a distance between centroids of 5.895(3) Å; there is also an Me(18)—phenyl-centroid (C4—C9) distance of 3.429(3) Å, and a centroid—centroid distance of 3.730(2) Å between pyrrole rings along the *b* axis, all of which describes the whole crystal (Albert & Cano, 1991).

Experimental

The title compound was prepared by the reaction of 2-methylindoylmagnesium iodide, which was prepared *in situ* (Rodriguez & San Andres, 1991), and benzoyl chloride (molar ratio 1:2) in diethyl ether solution for 6 h at 298 K. Crystals were grown from ethyl acetate solution.

Crystal data

C₁₆H₁₃NO
 $M_r = 235.27$
 Monoclinic
*C*2/*c*
 $a = 22.822(3)$ Å
 $b = 7.667(1)$ Å
 $c = 14.697(1)$ Å
 $\beta = 106.31(1)^\circ$
 $V = 2468.1(5)$ Å³
 $Z = 8$
 $D_x = 1.266$ Mg m^{−3}
 D_m not measured

Cu $K\alpha$ radiation
 $\lambda = 1.54180$ Å
 Cell parameters from 60 reflections
 $\theta = 6-35^\circ$
 $\mu = 0.624$ mm^{−1}
 $T = 293(2)$ K
 Prism
 0.66 × 0.33 × 0.13 mm
 Orange

Data collection

Seifert XRD 3000S diffractometer
 ω –2 θ scans
 Absorption correction: none
 1965 measured reflections
 1965 independent reflections
 1660 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 64.85^\circ$
 $h = 0 \rightarrow 26$
 $k = 0 \rightarrow 8$
 $l = -15 \rightarrow 15$
 2 standard reflections
 frequency: 90 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.155$
 $S = 1.130$
 1965 reflections
 164 parameters
 H atoms: riding model
 $w = 1/[\sigma^2(F_o^2) + (0.1059P)^2 + 0.4338P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
 $\Delta\rho_{\max} = 0.325$ e Å^{−3}
 $\Delta\rho_{\min} = -0.425$ e Å^{−3}
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: Seifert XRD 3000S. Cell refinement: *LSUCRE* (Appleman, 1971). Data reduction: *XRAY80* (Stewart, Kundell & Baldwin, 1980). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *Xtal3.0* (Hall & Stewart, 1990). 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: FG1244). Services for accessing these data are described at the back of the journal.

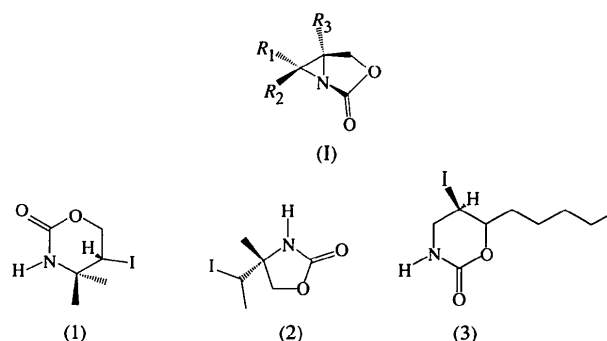
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cyclic aziridinocarbamates. The structures obtained confirm the stereochemistries deduced from spectroscopic data. Compounds (1) and (2) exist in the solid state as centrosymmetric N—H...O hydrogen-bonded dimers: for (1), O1...N' = 2.901 (8), and for (2), N...O2' = 2.902 (4) Å. Compound (3) crystallizes with two independent molecules in the asymmetric unit which are hydrogen-bonded to form centrosymmetric tetramers: O2...N2' = 2.970 (6) and O4...N1' = 2.819 (6) Å.

Comment

Recently, a convenient synthesis of bicyclic aziridinocarbamates, (I), has been developed (Ensley & Ling, 1997) involving addition of iodine isocyanate to allylic alcohols. Despite the exclusive formation of the five-membered oxazolidinone ring in the final products, spectroscopic data on the cyclic iodide precursors to (I) suggested that some of these contained six-membered oxazinone rings. To confirm this, the structures of representative examples of these intermediates [(1)–(3)] were determined.



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Intermediates for the Convenient Synthesis of Bicyclic Aziridinocarbamates

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Abstract

3,4,5,6-Tetrahydro-5-iodo-4,4-dimethyl-2*H*-1,3-oxazin-2-one, C₆H₁₀INO₂, (1), 4,5-dihydro-4-(1-iodoethyl)-4-methyl-3*H*-1,3-oxazol-2-one, C₆H₁₀INO₂, (2), and 3,4,5,6-tetrahydro-5-iodo-6-(*n*-pentyl)-2*H*-1,3-oxazin-2-one, C₉H₁₆INO₂, (3), are examples of a series of readily prepared intermediates in a convenient synthesis of bi-

Compound (2) exists in the solid state as pairs of hydrogen-bonded molecules with N—H1*n*...O2' and N'—H1*n*'...O2 pairwise interactions: H1*n*...O2' = 2.03 (2) Å and N—H1*n*...O2' = 163 (1)° (primed atoms in the molecule at $-x, -y, -z$). Although the H atom bonded to N in (1) could not be located, it is likely that an analogous pairwise hydrogen bonding interaction occurs here as well based on the O1...N' distance of 2.901 (8) Å (primed atom in the molecule at $-1-x, 2-y, -z$) which is virtually identical to the N...O2' distance [2.902 (4) Å] seen in (2). In (3), hydrogen bonding occurs between the two independent molecules to give tetramers disposed about inversion centers (Fig. 4) with the aliphatic side chains disposed on the outer surfaces to generate hydrophobic channels between them. The hydrogen bonding involves O2—H2*n*'...N2' [O2...H2*n*' = 1.99 (2) Å and O2...H2*n*'—N2' = 166 (1)°; primed atoms in the molecule at $1-x, -y, 1-z$] and O4...H1*n*'—N1' [O4...H1*n*' = 1.98 (2) Å and O4...H1*n*'—N1' = 144 (1)°; primed