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Stereochemistry of Rings. XVI.* Indole Derivatives. 1. 2,3-Dimethyl-5-nitroindole

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(Received 20 August 1988; accepted 13 January 1989)

Abstract. C₁₀H₁₀N₂O₂, $M_r = 190.2$, monoclinic, $P2_1/n$, $a = 14.345$ (2), $b = 8.518$ (2), $c = 7.741$ (2) Å, $\beta = 95.23$ (3)°, $V = 941.9$ Å³, $Z = 4$, $D_x = 1.34$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 7.50$ cm⁻¹, $F(000) = 400$, room temperature. The structure was solved by direct methods and refined by full-matrix least squares to a final $R = 0.048$ for 1519 observed reflections. The molecule as a whole is nearly planar and the methyl groups are both equatorial. A partial π delocalization is observed.

Introduction. As a continuation of our systematic conformational studies on the deformation of rings with different substituents, we report in this paper a structural investigation of an indole derivative. The greatly increasing importance of a number of indole-based biological compounds suggests further conformational analysis on this kind of molecule.

Experimental. Red prismatic crystal (0.31 × 0.42 × 0.64 mm), obtained by slow evaporation of an ethanolic solution, room temperature, Siemens AED single-crystal diffractometer controlled by an IBM PS/2 M30 computer. Cell parameters obtained from setting angles of 27 reflections ($12 \leq \theta \leq 36^\circ$) automatically well centered on the diffractometer. Intensities collected with Cu K α radiation in the $3 \leq \theta \leq 70^\circ$ range, $h -17 \rightarrow 17$, $k 0 \rightarrow 10$, $l 0 \rightarrow 9$, with a modified version (Belletti, Cantoni & Pasquinelli, 1988) of the Lehmann & Larsen (1974) procedure, 1794 independent reflections ($R_{\text{int}} = 0.007$), 1519 with $I \geq 2\sigma(I)$ classed as observed, one check reflection monitored every 50

reflections, its variation was within counting statistics, Lorentz–polarization corrections applied but none for absorption.

The structure was solved by direct methods using *SHELX76* (Sheldrick, 1976). All the non-H atoms were located from the best E map. A few cycles of isotropic refinement converged to $R = 15.8\%$, those performed with anisotropic thermal parameters gave $R = 6.86\%$, all H atoms localized from Fourier difference map and refined freely giving a final R value of 0.048 and $wR = 0.063$ with $w = 1.0/(\sigma^2 F + 0.0283F^2)$. The function minimized during refinement was $\sum w(\Delta F)^2$, 167 parameters refined. At the end of the refinement the maximum shift-to-e.s.d. ratio was 0.24 for the $z(\text{O}1)$ variable, while the maximum peak in the final ΔF map was 0.26 e Å⁻³.

Scattering factors for heavy atoms from *International Tables for X-ray Crystallography* (1974) and from Stewart, Davidson & Simpson (1965) for H atoms. Positional parameters of atoms are in Table 1.†

All the calculations were performed on an IBM PS/2 M80 computer with the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreotti, 1987).

Discussion. The geometrical features of the molecule, depicted in Fig. 1, are detailed in Table 2.

The indole skeleton is nearly planar, the dihedral angle formed by the aromatic and pyrrole mean ring

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51811 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Part XV: Bocelli & Grenier-Loustalot (1984).

Table 1. Atomic positional parameters ($\times 10^4$ for O, N and C, $\times 10^3$ for H atoms) with U_{eq} ($\times 10^4$) for O, N and C and U_{iso} ($\times 10^2$) for H atoms

	x	y	z	$U_{eq}^*/U_{iso}(\text{\AA}^2)$
O1	2261 (1)	1943 (2)	4929 (3)	915 (7)
O2	1505 (1)	4091 (2)	5154 (2)	790 (6)
N1	-1510 (1)	-255 (2)	1319 (2)	541 (4)
N2	1545 (1)	2703 (2)	4679 (2)	601 (5)
C1	-1138 (1)	-1740 (2)	1073 (2)	522 (5)
C2	-230 (1)	-1770 (2)	1731 (2)	491 (5)
C3	-18 (1)	-237 (2)	2434 (2)	448 (5)
C4	779 (1)	430 (2)	3282 (2)	476 (5)
C5	719 (1)	1979 (2)	3801 (2)	494 (5)
C6	-92 (1)	2881 (2)	3533 (2)	505 (5)
C7	-891 (1)	2237 (2)	2690 (2)	510 (5)
C8	-846 (1)	681 (2)	2153 (2)	462 (5)
C9	-1745 (2)	-2985 (3)	192 (3)	711 (8)
C10	447 (2)	-3112 (2)	1742 (3)	654 (7)
HN1	-212 (2)	-8 (3)	99 (2)	65 (6)
H4	136 (2)	-13 (3)	346 (3)	60 (5)
H6	-7 (2)	393 (3)	391 (2)	54 (5)
H7	-140 (2)	282 (3)	250 (3)	60 (5)
H91	-138 (3)	-403 (6)	-14 (6)	159 (14)
H92	-219 (2)	-330 (4)	98 (4)	99 (9)
H93	-207 (2)	-271 (4)	-92 (4)	104 (9)
H101	105 (2)	-268 (4)	115 (4)	103 (8)
H102	17 (2)	-409 (4)	126 (4)	98 (9)
H103	79 (2)	-328 (4)	295 (5)	110 (10)

* Hamilton (1959).

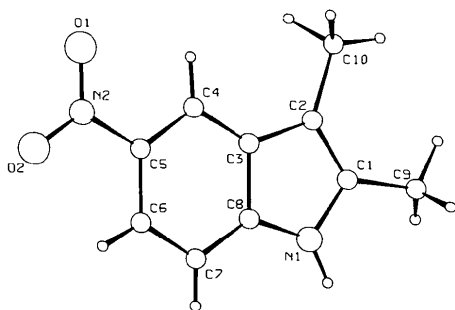


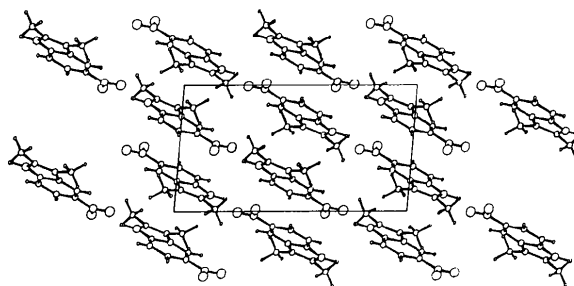
Fig. 1. Projection of the molecule.

planes being $0.6(1)^\circ$. The largest displacements of atoms from the mean plane are $-0.008(2)$ for C1, $0.007(2)$ for C3 and $-0.007(2)$ \AA for C6. The six C—C bonds and C—C—C internal angles in the benzene ring average $1.391(7)$ \AA and $120.0(9)^\circ$. The shortening of the N1—C8 [$1.359(2)$ \AA] bond length with respect to that of N1—C1 [$1.393(2)$ \AA] is due to the delocalized π electrons of the aromatic ring extending over the N1—C8 bond. A partial delocalization of π electrons of the C1—C2 double bond over C1—C9 [$1.497(3)$ \AA] and C2—C10 [$1.500(3)$ \AA] causes a shortening of these bond distances with respect to that reported for a C—C single-bond distance (1.544 \AA ; Mossel & Romers, 1964).

The two methyl substituents are both in equatorial positions, although C9 lies $-0.026(2)$ \AA out of the indole plane, and the *p*-nitro group, which shows normal geometrical parameters, lies practically in the plane of the indole skeleton, the tilt around the N2—C5

Table 2. Bond distances (\AA) and bond angles ($^\circ$)

O1—N2	1.215 (2)	C2—C10	1.500 (2)
O2—N2	1.241 (2)	C3—C4	1.387 (2)
N1—C1	1.393 (2)	C3—C8	1.422 (2)
N1—C8	1.359 (2)	C4—C5	1.384 (2)
N2—C5	1.449 (2)	C5—C6	1.394 (2)
C1—C2	1.355 (2)	C6—C7	1.380 (2)
C1—C9	1.497 (3)	C7—C8	1.392 (2)
C2—C3	1.437 (2)		
N1—HN1	0.90 (2)	C9—H92	0.96 (3)
C4—H4	0.95 (2)	C9—H93	0.97 (3)
C6—H6	0.94 (2)	C10—H101	1.08 (3)
C7—H7	0.88 (2)	C10—H102	0.99 (3)
C9—H91	1.08 (5)	C10—H103	1.02 (3)
C1—N1—C8	109.7 (2)	C2—C3—C4	133.5 (2)
O1—N2—O2	121.5 (2)	C4—C3—C8	119.2 (2)
O2—N2—C5	119.1 (2)	C3—C4—C5	117.2 (2)
O1—N2—C5	119.4 (2)	N2—C5—C4	118.2 (1)
N1—C1—C9	119.5 (2)	C4—C5—C6	123.7 (2)
N1—C1—C2	109.5 (2)	N2—C5—C6	118.1 (2)
C2—C1—C9	131.1 (2)	C5—C6—C7	119.8 (2)
C1—C2—C10	128.1 (2)	C6—C7—C8	117.4 (2)
C1—C2—C3	106.8 (1)	C3—C8—C7	122.6 (2)
C3—C2—C10	125.2 (2)	N1—C8—C7	130.6 (2)
C2—C3—C8	107.3 (1)	N1—C8—C3	106.8 (2)

Fig. 2. Packing of the molecules along *y*.

bond of this group with respect to the phenyl ring being only $0.5(1)^\circ$.

In the crystal the molecules are joined together by a hydrogen bond (Fig. 2): $\text{HN1}\cdots\text{O2}^i = 2.19(2)$, $\text{N1}\cdots\text{O2}^i = 3.072(2)$ \AA , $\text{N1—HN1}\cdots\text{O2}^i = 167(2)^\circ$ ($i = x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$).

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