

C(15)	-1.1479 (4)	-0.4039 (3)	-0.6970 (2)	2.1 (2)
C(16)	-1.1618 (4)	-0.3321 (3)	-0.7698 (2)	2.2 (2)
C(17)	-1.0543 (4)	-0.2794 (3)	-0.8094 (2)	2.0 (2)
C(18)	-0.9306 (4)	-0.2998 (3)	-0.7717 (2)	1.6 (1)
C(19)	-0.7373 (4)	-0.3841 (3)	-0.5716 (2)	1.5 (2)
C(20)	-0.6525 (4)	-0.1907 (3)	-0.5646 (2)	2.1 (2)
C(21)	-0.7166 (4)	-0.1688 (3)	-0.6585 (2)	1.7 (2)
C(22)	-0.5970 (4)	-0.4377 (3)	-0.7671 (2)	1.8 (2)
O(22)	-0.5373 (3)	-0.4843 (2)	-0.8266 (2)	2.4 (1)

Table 2. Geometric parameters (Å, °)

C(1)—N(2)	1.502 (5)	C(7)—C(8)	1.539 (6)
C(1)—C(3)	1.556 (5)	C(8)—N(9)	1.471 (5)
C(1)—C(12)	1.556 (5)	N(9)—C(10)	1.473 (5)
C(1)—C(21)	1.536 (5)	N(9)—C(19)	1.458 (5)
N(2)—C(1')	1.364 (5)	C(10)—C(11)	1.547 (5)
N(2)—C(18)	1.440 (5)	C(11)—C(12)	1.572 (5)
C(1')—O(1')	1.216 (5)	C(11)—C(22)	1.518 (5)
C(1')—O(2')	1.344 (5)	C(12)—C(13)	1.492 (5)
O(2')—C(3')	1.457 (5)	C(12)—C(19)	1.530 (5)
C(3)—O(3)	1.402 (5)	C(13)—C(14)	1.367 (5)
C(3)—C(4)	1.581 (5)	C(13)—C(18)	1.394 (5)
C(3)—C(22)	1.521 (5)	C(14)—C(15)	1.391 (6)
C(4)—C(5)	1.550 (5)	C(15)—C(16)	1.386 (5)
C(5)—C(6)	1.538 (5)	C(16)—C(17)	1.393 (6)
C(5)—C(19)	1.537 (5)	C(17)—C(18)	1.401 (6)
C(5)—C(20)	1.527 (5)	C(20)—C(21)	1.559 (5)
C(6)—C(7)	1.531 (6)	C(22)—O(22)	1.207 (5)
N(2)—C(1)—C(3)	118.4 (3)	C(8)—N(9)—C(19)	112.9 (3)
N(2)—C(1)—C(12)	101.1 (3)	C(10)—N(9)—C(19)	108.4 (3)
N(2)—C(1)—C(21)	109.4 (3)	N(9)—C(10)—C(11)	104.0 (3)
C(3)—C(1)—C(12)	100.5 (3)	C(10)—C(11)—C(12)	106.0 (3)
C(3)—C(1)—C(21)	112.6 (3)	C(10)—C(11)—C(22)	113.4 (3)
C(12)—C(1)—C(21)	114.1 (3)	C(12)—C(11)—C(22)	104.1 (3)
C(1)—N(2)—C(1')	122.8 (3)	C(1)—C(12)—C(11)	106.7 (3)
C(1)—N(2)—C(18)	107.2 (3)	C(1)—C(12)—C(13)	103.5 (3)
C(1')—N(2)—C(18)	127.8 (3)	C(1)—C(12)—C(19)	109.8 (3)
N(2)—C(1')—O(1')	125.1 (4)	C(11)—C(12)—C(13)	112.3 (3)
N(2)—C(1')—O(2')	112.2 (3)	C(11)—C(12)—C(19)	103.2 (3)
O(1')—C(1')—O(2')	122.7 (3)	C(13)—C(12)—C(19)	120.7 (3)
C(1')—O(2')—C(3')	114.4 (3)	C(12)—C(13)—C(14)	128.4 (3)
C(1)—C(3)—O(3)	120.0 (3)	C(12)—C(13)—C(18)	109.0 (3)
C(1)—C(3)—C(4)	104.7 (3)	C(14)—C(13)—C(18)	122.5 (3)
C(1)—C(3)—C(22)	102.7 (3)	C(13)—C(14)—C(15)	118.1 (3)
O(3)—C(3)—C(4)	107.3 (3)	C(14)—C(15)—C(16)	120.3 (4)
O(3)—C(3)—C(22)	114.4 (3)	C(15)—C(16)—C(17)	121.8 (4)
C(4)—C(3)—C(22)	106.7 (3)	C(16)—C(17)—C(18)	117.6 (3)
C(3)—C(4)—C(5)	113.7 (3)	N(2)—C(18)—C(13)	109.5 (3)
C(4)—C(5)—C(6)	111.2 (3)	N(2)—C(18)—C(17)	130.9 (3)
C(4)—C(5)—C(19)	112.1 (3)	C(13)—C(18)—C(17)	119.6 (3)
C(4)—C(5)—C(20)	105.0 (3)	C(5)—C(19)—N(9)	117.6 (3)
C(6)—C(5)—C(19)	109.0 (3)	C(5)—C(19)—C(12)	109.2 (3)
C(6)—C(5)—C(20)	111.8 (3)	N(9)—C(19)—C(12)	103.6 (3)
C(19)—C(5)—C(20)	107.6 (3)	C(5)—C(20)—C(21)	109.2 (3)
C(5)—C(6)—C(7)	112.3 (3)	C(1)—C(21)—C(20)	110.8 (3)
C(6)—C(7)—C(8)	109.9 (3)	C(3)—C(22)—C(11)	107.1 (3)
C(7)—C(8)—N(9)	114.1 (3)	C(3)—C(22)—O(22)	126.2 (3)
C(8)—N(9)—C(10)	114.6 (3)	C(11)—C(22)—O(22)	126.7 (3)

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55613 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1025]

References

- Camerman, A., Camerman, N. & Trotter, J. (1965). *Acta Cryst.* **19**, 314–320.
 Chiaroni, A., Langlois, N. & Riche, C. (1977). *Acta Cryst.* **B33**, 3410–3414.
 Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* **A24**, 390–397.

- Duchamp, D. J. (1977). *Am. Chem. Soc. Symp. Ser.* No.46, 98–121.
 Duchamp, D. J. (1984). *CRYM. A System of Crystallographic Programs*. The Upjohn Company, Kalamazoo, MI, USA.
 Guggisberg, A., Gorman, A. A., Bycroft, B. W. & Schmid, H. (1969). *Helv. Chim. Acta*, **52**, 76–89.
 Hendrickson, J. B. (1967). *J. Am. Chem. Soc.* **89**, 7036–7043.
 Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 Mills, J. F. D. & Nyburg, S. C. (1960). *J. Chem. Soc.* pp. 1458–1463.
 Ramakrishnan, C. (1980). In *Computing in Crystallography*, edited by R. Diamond, S. Ramaseshan & K. Venkatesan. Bangalore: Indian Academy of Sciences.
 Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 Toffoli, P., Rodier, N., Lewin, G. & Poisson, J. (1986). *Acta Cryst.* **C42**, 1606–1610.

Acta Cryst. (1993). **C49**, 173–175

Structure of 3,3-Dichloro-1*H*-indol-2(3*H*)-one

J. ZUKERMAN-SCHPECTOR

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 São Carlos, SP, Brazil

ANGELO DA C. PINTO, J. F. M. DA SILVA AND
M. T. F. C. BARCELLOS

Instituto de Química, Universidade Federal de Rio de Janeiro, Cidade Universitária, 21910 Rio de Janeiro, RJ, Brazil

(Received 12 May 1992; accepted 29 July 1992)

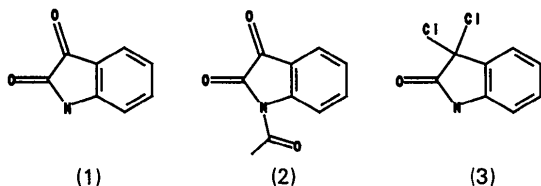
Abstract

The indol-2-one moiety is essentially planar with a C(2)—C(3) distance of 1.556 (6) Å. Centrosymmetrically related pairs of molecules are linked through hydrogen bonds forming dimers.

Comment

The study of the structural features of isatin (1*H*-indole-2,3-dione) (1) (Palenik, Koziol, Katritzky & Fan, 1990) and its *N*-acetyl derivative, 1-acetylindole-2,3-dione (2) (Zukerman-Schpector, Castellano, Pinto, da Silva & Barcellos, 1992), has led to the observation that in both cases the C(2)—C(3) bond length is significantly longer than the expected value (1.48 Å) for a C(sp²)—C(sp²) single bond; this was ascribed to non-bonded lone pair-

lone pair repulsions. In the present structure (3), the C(2)—C(3) distance of 1.556 (6) Å is longer than expected, the characteristic value for a C(sp²)—C(sp²) bond being 1.50 Å. This may well be due to repulsions between the outer-electron clouds of the O and Cl atoms, showing that the effect on the C(2)—C(3) bond length of a voluminous atom bonded at the C(3) position is similar to that caused by the C(3) carbonyl O atom in (1) and (2).



The 1H-indol-2-one moiety is essentially planar, $\sigma_{av} = 0.011$ Å [$\sigma_{av} = (\sum_i d_i^2 / N - 3)^{1/2}$]. The molecules are linked in pairs across a symmetry centre through N—H...O hydrogen bonds [N—H(N) = 1.00 (3), O...N = 2.867 (5), O...H(N) = 1.88 (3) Å; N—H(N)...O = 172 (1)°] forming isolated dimers.

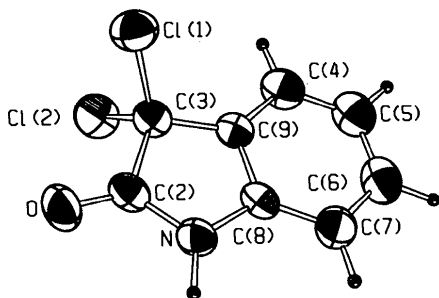


Fig. 1. The molecular structure of C₈H₅Cl₂NO showing the atom labelling. 50% thermal ellipsoids are shown for non-H atoms.

Experimental

Crystal data

C₈H₅Cl₂NO

M_r = 202.04

Monoclinic

*P*2₁/*n*

a = 11.224 (2) Å

b = 6.182 (2) Å

c = 12.6485 (2) Å

β = 103.16 (2)°

V = 854.6 (6) Å³

Z = 4

D_s = 1.570 Mg m⁻³

Mo K α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 9–19°

μ = 0.71 mm⁻¹

T = 292 K

Irregular

0.45 × 0.18 × 0.05 mm

Pale yellow

Crystal source: from acetic anhydride

Data collection

Enraf–Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

1020 observed reflections

[*I* > 3 σ (*I*)]

*R*_{int} = 0.034

Absorption correction:

empirical (Walker & Stuart, 1983)

*T*_{min} = 0.080, *T*_{max} = 1.26

2060 measured reflections

1709 independent reflections

θ_{max} = 28°

h = -14 → 14

k = 0 → 8

l = 0 → 16

2 standard reflections

frequency: 30 min

intensity variation: ±1.5%

Refinement

Refinement on *F*

Final *R* = 0.045

wR = 0.046

S = 1.44

1020 reflections

129 parameters

$w = 1/[\sigma^2(|F_o|) + 0.0003|F_o|^2]$

(Δ/σ)_{max} = 0.001

$\Delta\rho_{max}$ = 0.23 e Å⁻³

$\Delta\rho_{min}$ = -0.22 e Å⁻³

Atomic scattering factors from SHELX76 (Sheldrick, 1976)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

$$B_{eq} = \frac{4}{3} \sum_i \sum_j B_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Cl(1)	0.3905 (1)	0.1975 (2)	0.0193 (1)	4.62 (4)
Cl(2)	0.2891 (1)	0.4636 (2)	0.1646 (1)	4.82 (4)
O	0.1508 (3)	0.0273 (5)	0.0881 (2)	4.5 (1)
N	0.0631 (3)	0.2399 (6)	-0.0572 (3)	3.5 (1)
C(2)	0.1514 (3)	0.1764 (8)	0.0271 (3)	3.3 (1)
C(3)	0.2576 (3)	0.3420 (7)	0.0355 (3)	3.2 (1)
C(4)	0.2629 (4)	0.6747 (8)	-0.0903 (4)	4.1 (1)
C(5)	0.1974 (5)	0.7865 (9)	-0.1779 (4)	4.6 (2)
C(6)	0.0825 (5)	0.7185 (8)	-0.2304 (4)	4.5 (2)
C(7)	0.0286 (4)	0.5371 (9)	-0.1970 (3)	4.1 (2)
C(8)	0.0953 (3)	0.4272 (7)	-0.1089 (3)	3.2 (1)
C(9)	0.2121 (3)	0.4931 (7)	-0.0559 (3)	3.0 (1)

Table 2. Geometric parameters (Å, °)

Cl(1)—C(3)	1.790 (4)	Cl(2)—C(3)	1.759 (4)
O—C(2)	1.203 (5)	N—C(2)	1.339 (5)
N—C(8)	1.417 (5)	C(2)—C(3)	1.556 (6)
C(3)—C(9)	1.484 (6)	C(4)—C(5)	1.369 (7)
C(4)—C(9)	1.374 (6)	C(5)—C(6)	1.375 (7)
C(6)—C(7)	1.385 (7)	C(7)—C(8)	1.373 (6)
C(8)—C(9)	1.391 (5)		
C(2)—N—C(8)	112.8 (3)	O—C(2)—N	128.6 (4)
O—C(2)—C(3)	125.2 (4)	N—C(2)—C(3)	106.1 (3)
Cl(1)—C(3)—Cl(2)	108.8 (2)	Cl(1)—C(3)—C(2)	108.0 (3)
Cl(1)—C(3)—C(9)	111.9 (3)	Cl(2)—C(3)—C(2)	109.5 (3)
Cl(2)—C(3)—C(9)	114.5 (3)	C(2)—C(3)—C(9)	103.8 (3)
C(5)—C(4)—C(9)	118.9 (4)	C(4)—C(5)—C(6)	120.5 (5)
C(5)—C(6)—C(7)	121.8 (5)	C(6)—C(7)—C(8)	116.9 (4)
N—C(8)—C(7)	129.1 (4)	N—C(8)—C(9)	109.2 (3)
C(7)—C(8)—C(9)	121.8 (4)	C(3)—C(9)—C(4)	131.9 (4)
C(3)—C(9)—C(8)	108.1 (3)	C(4)—C(9)—C(8)	120.0 (4)

Data were corrected for Lorentz, polarization and absorption effects. The structure was solved by direct methods and refined by full-matrix least squares. H atoms were found by a difference synthesis and refined isotropically.

The programs used were SHELXS86 (Sheldrick, 1985), SHELX76 and ORTEP (Johnson, 1965). Most of the calculations were performed on a VAX 6420 computer at the Instituto de Física e Química de São Carlos.

This work has received partial support from FAPESP, CNPq, CAPES and FINEP. One of us (JZ-S) would like to thank the Associação de Amigos do Instituto Weizmann de São Paulo for a scholarship.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55375 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1016]

References

- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Palenik, G. J., Koziol, A. E., Katritzky, A. R. & Fan, W.-Q. (1990). *J. Chem. Soc. Chem. Commun.* pp. 715–716.
- Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
- Zukerman-Schpector, J., Castellano, E. E., Pinto, A. Da C., da Silva, J. F. M. & Barcellos, M. T. F. C. (1992). *Acta Cryst.* **C48**, 760–762.

Acta Cryst. (1993). **C49**, 175–177

Structure of Hydrocotarnine Hydrobromide†

J. SEETHARAMAN, S. S. RAJAN* AND R. SRINIVASAN

*Department of Crystallography and Biophysics,
University of Madras, Madras-25, India*

(Received 10 April 1992; accepted 10 August 1992)

Abstract

The heterocyclic ring of the isoquinoline adopts a half-chair conformation and the dioxole ring an envelope conformation. The methyl group is rotated from the plane of the benzene ring attached to it by $104.2(3)^\circ$. The N atom of the heterocyclic ring is displaced from the plane of the ring by $0.606(3) \text{ \AA}$. The structure is stabilized by N—H \cdots Br hydrogen bonds.

Comment

As part of our studies on cough suppressants, the structure determination of the compound hydrocotarnine (5,6,7,8-tetrahydro-4-methoxy-6-methyl-1,3-dioxolo[4,5-g]isoquinoline) hydrobromide (1) was undertaken. The heterocyclic ring of the isoquinoline is in a half-chair conformation with an asymmetry parameter $\Delta C_2 = 5.4(1)^\circ$ (Duax, Weeks & Rohrer, 1976) as in similar isoquinoline structures (Ahmed, 1978; Bernath, Kobor, Fulop, Sohar, Argay &

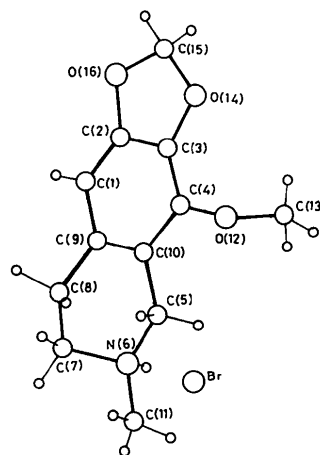


Fig. 1. *PLUTO* diagram of the molecule showing the atom-numbering scheme.

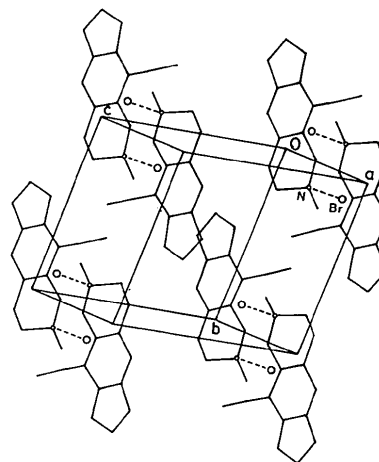


Fig. 2. View of the unit-cell packing viewed down the *c* axis. The H atoms are omitted for clarity. The hydrogen bonding is shown by dotted lines.

Kalman, 1986) and the dioxole ring is in an envelope conformation. C(15) is displaced from the least-squares plane of O(16), C(2), C(3) and O(14) by $0.059(5) \text{ \AA}$. The methoxy group is rotated from the plane of the benzene ring by $104.2(3)^\circ$. The bond lengths of the heterocyclic ring of the isoquinoline are in good agreement with similar isoquinoline rings of 1-phenyl-3-methylisoquinoline hydrobromide (Rychlewska, Palenik & Kosturkiewicz, 1975), 1-chloro-3-hydroxyisoquinoline (Ammon & Wheeler, 1974), 5-hydroxy-3-phenyl-1-(3-methyl-1-isoquinolyl)pyrazole (King & Reimlinger, 1971) and 1-(1-naphthyl)isoquinoline (Ljungstrom, Lindqvist & Overbeek, 1978). A *PLUTO* diagram (Motherwell, 1976) of the molecule with atom-numbering scheme is shown in Fig. 1 and a view of the unit-cell packing in Fig. 2. The molecules in the unit cell are stabilized by a hydrogen-bonding network involving N and Br, with an N—H \cdots Br angle of $175.9(4)^\circ$, and N \cdots Br

† DCB Contribution No. 798.