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1- α -Chloroacetylisatin

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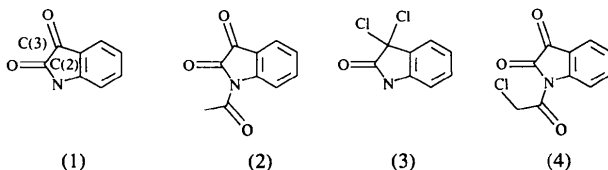
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Abstract

The structure of 1-chloroacetylisatin, $C_{10}H_6ClNO_3$, has been determined. The heterocyclic ring is essentially planar making a dihedral angle of $8.3(1)^\circ$ with the chloroacetyl moiety. The C(2)—C(3) distance is $1.542(6)$ Å. The molecules are linked through Cl \cdots H—C interactions.

Comment

The study of the structural features of isatin, (1), (Palenik, Koziol, Katritzky & Fan, 1990), and some of its derivatives such as (2) and (3) (Zukerman-Schpector, Castellano, Pinto, Da Silva & Barcellos, 1992; Zukerman-Schpector, Pinto, Da Silva & Barcellos, 1993), has led to the observation that the C(2)—C(3) bond length is, in these cases, significantly longer than the values of 1.48 and 1.50 Å expected for C(sp^2)—C(sp^2) and C(sp^2)—C(sp^3) single bonds, respectively. As part of our continuing interest in the structural features of this type of molecule, the title compound (4) was obtained from the reaction of isatin with α -chloroacetyl chloride and its crystal structure was determined.



As in the related structures, the C(2)—C(3) bond [$1.542(6)$ Å] is significantly longer than the expected value. The N—C(10) distance is the same, to within experimental error, as that found in compound (2). The heterocyclic ring is essentially planar, $\sigma_{av} = 0.013$ Å [$\sigma_{av} = (\sum d_i^2 / N - 3)^{1/2}$]. The packing of the

molecules in the crystal is determined by the following interaction: Cl \cdots C(6)ⁱ = $3.970(5)$, Cl \cdots H(C6)ⁱ = $2.909(1)$ Å; C(6)ⁱ—H(C6)ⁱ \cdots Cl = $168.9(2)^\circ$ [symmetry code: (i) $2 - x, -1 - y, 1 - z$].

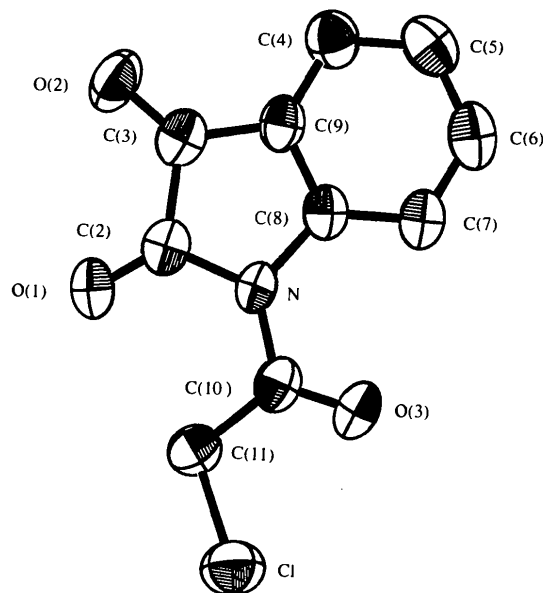


Fig. 1. The molecular structure of $C_{10}H_6ClNO_3$ showing the atom labelling. 50% displacement ellipsoids are shown for non-H atoms.

Experimental

Crystal data

$C_{10}H_6ClNO_3$
 $M_r = 223.62$
Monoclinic
 $P2_1/c$
 $a = 9.735(1)$ Å
 $b = 4.9966(9)$ Å
 $c = 19.185(3)$ Å
 $\beta = 94.61(1)^\circ$
 $V = 930.2(4)$ Å³
 $Z = 4$
 $D_x = 1.60$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
 $\theta = 8-18^\circ$
 $\mu = 0.389$ mm⁻¹
 $T = 292$ K
Irregular
 0.20×0.15 mm
(max. and min.)
Yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
1544 measured reflections
1417 independent reflections

906 observed reflections
[$I > 3\sigma(I)$]
 $R_{int} = 0.020$
 $\theta_{max} = 25^\circ$
 $h = -11 \rightarrow 11$
 $k = 0 \rightarrow 5$
 $l = 0 \rightarrow 22$
2 standard reflections
frequency: 30 min
intensity decay: 0.8%

Refinement

Refinement on F
 $R = 0.045$

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

wR = 0.047

S = 1.49

906 reflections

137 parameters

H atoms were refined with
one overall U_{iso} $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.17 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -0.27 \text{ e } \text{Å}^{-3}$

Extinction correction: none

Atomic scattering factors
from SHELX76
(Sheldrick, 1976)

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: LI1118). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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7,10-Dimethoxybenzo[*b*]carbazole†

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Abstract

The asymmetric unit of the title compound, C₁₈H₁₅NO₂, contains two crystallographically independent molecules with similar conformations. Each molecule consists of a carbazole ring system and a fused substituted benzene ring. The molecules are highly planar and their conformations are similar to that of ellipticine [Courseille, Busetta & Hospital (1974). *Acta Cryst.* **B30**, 2628–2631], which is a DNA-intercalating molecule.

Comment

Preliminary studies of carbazole derivatives have shown that the presence of oxygenated substituents increases their biological activity (Hewlins, Oliveira-Campos & Shannon, 1984). Their structures are analogous to that of ellipticine (Courseille, Busetta & Hospital, 1974), a plant alkaloid having pronounced anti-tumour activity (Hartwell & Abbott, 1969), and they have been found to have DNA-intercalating properties (Neidle, 1979; Gale, Cundliffe, Reynolds,

† DCB contribution No. 837.

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

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

| | x | y | z | B _{eq} |
|-------|------------|-------------|------------|-----------------|
| Cl | 0.6410 (1) | 0.0435 (3) | 0.5583 (1) | 6.04 (5) |
| N | 1.0358 (3) | -0.0072 (6) | 0.6319 (2) | 3.3 (1) |
| O(1) | 0.9848 (3) | 0.3551 (6) | 0.7022 (2) | 4.4 (1) |
| O(2) | 1.2610 (3) | 0.2545 (7) | 0.7665 (2) | 5.5 (1) |
| O(3) | 0.9000 (3) | -0.2222 (7) | 0.5490 (2) | 5.9 (1) |
| C(2) | 1.0612 (4) | 0.1833 (9) | 0.6855 (2) | 3.6 (1) |
| C(3) | 1.2090 (4) | 0.1287 (9) | 0.7174 (2) | 4.0 (1) |
| C(4) | 1.3891 (5) | -0.206 (1) | 0.6820 (2) | 4.7 (2) |
| C(5) | 1.4137 (5) | -0.410 (1) | 0.6353 (3) | 5.0 (2) |
| C(6) | 1.3092 (5) | -0.4888 (9) | 0.5857 (2) | 4.8 (2) |
| C(7) | 1.1803 (4) | -0.3712 (8) | 0.5802 (2) | 4.1 (2) |
| C(8) | 1.1576 (4) | -0.1680 (8) | 0.6270 (2) | 3.5 (1) |
| C(9) | 1.2607 (4) | -0.0892 (8) | 0.6768 (2) | 3.7 (1) |
| C(10) | 0.9112 (4) | -0.0469 (9) | 0.5917 (2) | 3.8 (1) |
| C(11) | 0.7953 (4) | 0.1402 (9) | 0.6066 (2) | 4.2 (1) |

Table 2. Selected geometric parameters (Å, °)

| | | | |
|----------------|-----------|------------------|-----------|
| Cl—C(11) | 1.768 (4) | N—C(2) | 1.408 (5) |
| N—C(8) | 1.442 (5) | N—C(10) | 1.398 (5) |
| O(1)—C(2) | 1.196 (5) | O(2)—C(3) | 1.209 (5) |
| O(3)—C(10) | 1.198 (5) | C(2)—C(3) | 1.542 (6) |
| C(3)—C(9) | 1.452 (6) | C(4)—C(5) | 1.390 (7) |
| C(4)—C(9) | 1.376 (6) | C(5)—C(6) | 1.393 (7) |
| C(6)—C(7) | 1.382 (6) | C(7)—C(8) | 1.385 (6) |
| C(8)—C(9) | 1.386 (6) | C(10)—C(11) | 1.510 (6) |
| C(2)—N—C(8) | 109.1 (3) | C(6)—C(7)—C(8) | 117.0 (4) |
| C(2)—N—C(10) | 126.3 (3) | N—C(8)—C(7) | 128.7 (4) |
| C(8)—N—C(10) | 124.5 (3) | N—C(8)—C(9) | 110.5 (3) |
| N—C(2)—O(1) | 127.2 (4) | C(7)—C(8)—C(9) | 120.7 (4) |
| N—C(2)—C(3) | 105.9 (3) | C(3)—C(9)—C(4) | 129.1 (4) |
| O(1)—C(2)—C(3) | 126.9 (4) | C(3)—C(9)—C(8) | 108.6 (4) |
| O(2)—C(3)—C(2) | 122.4 (4) | C(4)—C(9)—C(8) | 122.2 (4) |
| O(2)—C(3)—C(9) | 131.8 (4) | N—C(10)—O(3) | 120.5 (4) |
| C(2)—C(3)—C(9) | 105.8 (3) | N—C(10)—C(11) | 115.8 (3) |
| C(5)—C(4)—C(9) | 117.8 (4) | O(3)—C(10)—C(11) | 123.6 (4) |
| C(4)—C(5)—C(6) | 119.6 (4) | Cl—C(11)—C(10) | 110.4 (3) |
| C(5)—C(6)—C(7) | 122.7 (4) | | |

Data were corrected for Lorentz, polarization and absorption effects. The structure was solved by direct methods. H atoms were found in a difference synthesis and included as fixed contributors with an overall isotropic displacement factor that refined to U_{iso} = 0.081 (6) Å². Programs used were: *SHELXS86* (Sheldrick, 1985), *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). Most of the calculations were performed on a MicroVAX 3600 computer at the Weizmann Institute of Science, Israel.

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