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5-Chloroindoline-2,3-dione

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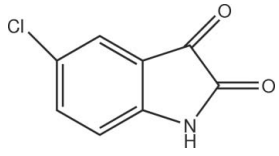
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.102; data-to-parameter ratio = 16.0.

The title compound, $C_8H_4ClNO_2$, is almost planar (r.m.s. deviation for the non-H atoms = 0.023 Å). In the crystal, N—H···O hydrogen bonds link the molecules into $C(4)$ chains propagating in [001] and C—H···O interactions cross-link the chains.

Related literature

For further synthetic details, see: Silva *et al.* (2001). For reference bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_4ClNO_2$
M_r = 181.57
Orthorhombic, $Pna2_1$
a = 24.706 (5) Å
b = 5.6890 (11) Å
c = 5.209 (1) Å
V = 732.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 293 K
0.10 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.955, T_{max} = 0.977
1746 measured reflections
884 independent reflections
734 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.102
S = 1.00
884 reflections
109 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983), 862 Friedel pairs
Flack parameter: 0.11 (16)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O1^{i}	0.86	2.04	2.893 (4)	172
C7—H7A···O2^{ii}	0.93	2.39	3.301 (5)	166

Symmetry codes: (i) $-x+1, -y, z-\frac{1}{2}$; (ii) $x, y-1, z-1$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5688).

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supporting information

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5-Chloroindoline-2,3-dione

Wen-Bin Wei, Shuo Tian, Hao Zhou, Jie Sun and Hai-Bo Wang

S1. Comment

5-Chloroindoline-2,3-dione is an important pharmaceutical intermediate for synthesizing 5-chlorooxindole and tenidap which was evaluated as novel nonsteroidal anti-inflammatory agents. We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (N/C1—C3/C8) and B (C3—C8) are nearly coplanar, and they are oriented at dihedral angles of A/B = 0.30 (3).

In the crystal structure, intermolecular N—H···O interaction may be effective in the stabilization of the structure.

S2. Experimental

For the preparation of the title compound, the method developed by Sandmeyer is the oldest and the most frequently used. It consists in the reaction of 4-chloroaniline with chloral hydrate and hydroxylamine hydrochloride in aqueous sodium sulfate to form an 4-chloroisonitrosoacetanilide, which after isolation, when treated with concentrated sulfuric acid, furnishes the title compound in 75% overall yield (Silva *et al.*, 2001). Red blocks of (I) were obtained by slow evaporation of a methanol solution (m.p. 520 K).

S3. Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 Å for aromatic, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*N*), where x = 1.5 for NH H and x = 1.2 for all other H atoms.

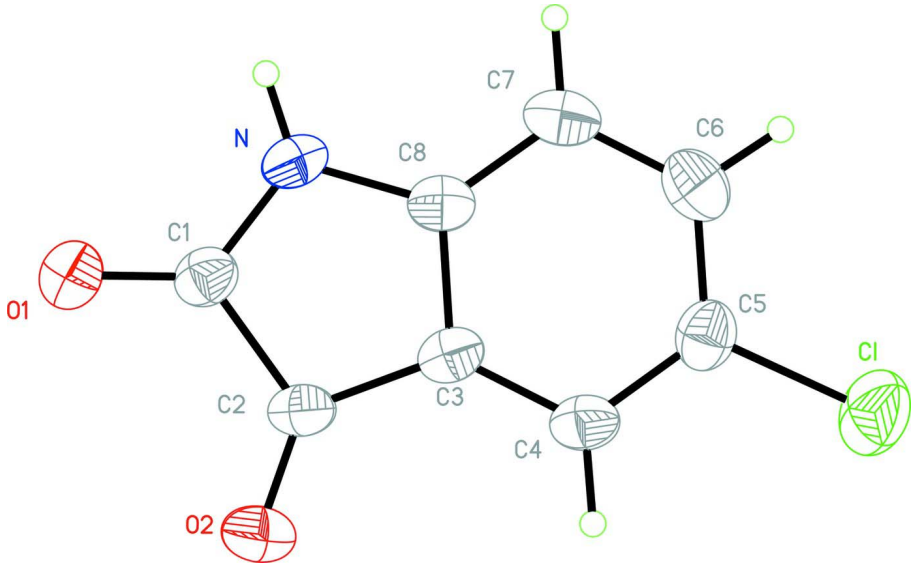

Figure 1

View of the title compound with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

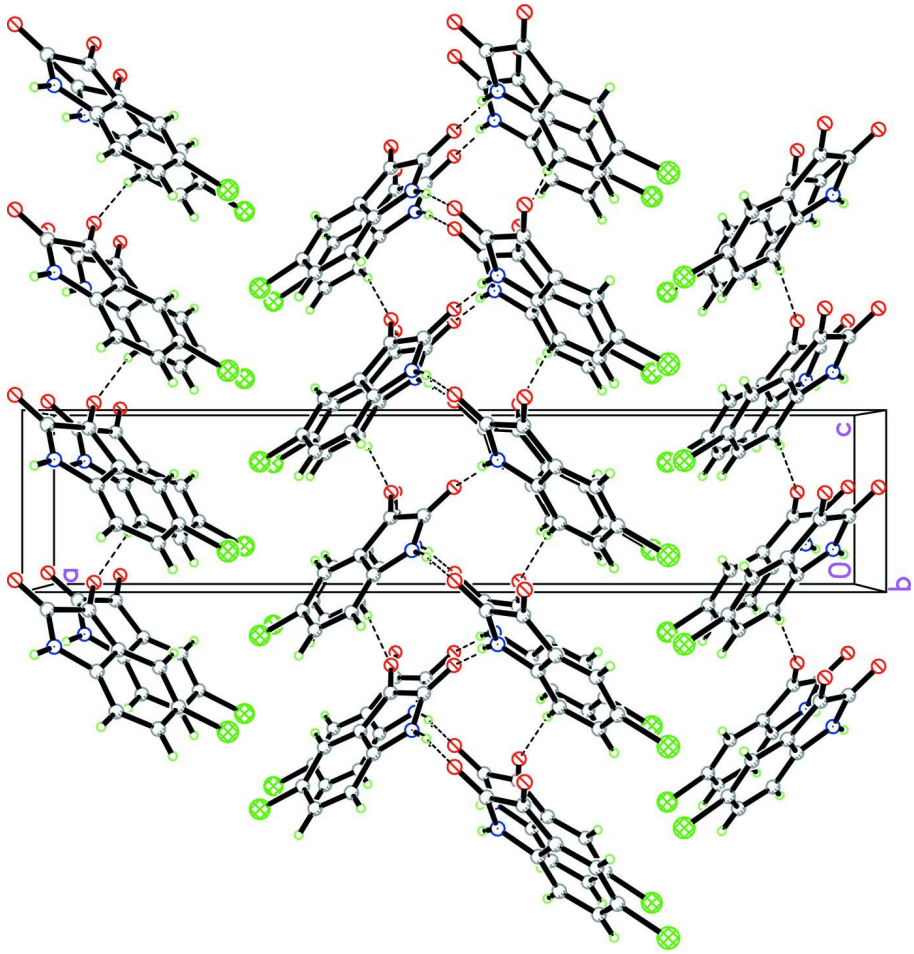

Figure 2

Packing diagram.

5-Chloroindoline-2,3-dione

Crystal data

$C_8H_4ClNO_2$
$M_r = 181.57$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 24.706$ (5) Å
$b = 5.6890$ (11) Å
$c = 5.209$ (1) Å
$V = 732.1$ (2) Å^3
$Z = 4$
$F(000) = 368$
$D_x = 1.647$ Mg m^{-3}
Melting point: 520 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.47$ mm^{-1}
$T = 293$ K
Block, red
0.10 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.955$, $T_{max} = 0.977$
1746 measured reflections
884 independent reflections
734 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.7°$
$h = -31\rightarrow31$
$k = -7\rightarrow0$
$l = 0\rightarrow6$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.102$
$S = 1.00$
884 reflections
109 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Absolute structure: Flack (1983), 862 Friedel pairs
Absolute structure parameter: 0.11 (16)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	0.26410 (4)	0.6028 (2)	0.2215 (3)	0.0588 (4)
N	0.45038 (11)	0.1571 (5)	0.7251 (9)	0.0378 (8)
H0A	0.4676	0.0291	0.6924	0.045*
O1	0.49964 (12)	0.2818 (5)	1.0752 (7)	0.0441 (7)
C1	0.46351 (13)	0.3075 (6)	0.9153 (9)	0.0335 (8)
O2	0.42293 (11)	0.6781 (5)	1.0449 (7)	0.0446 (7)
C2	0.42335 (13)	0.5098 (6)	0.9018 (8)	0.0332 (8)
C3	0.38713 (13)	0.4525 (6)	0.6867 (8)	0.0321 (8)
C4	0.34278 (14)	0.5650 (7)	0.5820 (9)	0.0351 (9)
H4A	0.3300	0.7058	0.6497	0.042*
C5	0.31808 (13)	0.4609 (7)	0.3733 (8)	0.0373 (9)
C6	0.33568 (15)	0.2468 (7)	0.2760 (8)	0.0409 (10)
H6A	0.3175	0.1793	0.1381	0.049*
C7	0.37976 (16)	0.1329 (6)	0.3811 (10)	0.0390 (9)
H7A	0.3920	−0.0093	0.3148	0.047*
C8	0.40480 (13)	0.2366 (6)	0.5864 (9)	0.0333 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0493 (6)	0.0757 (8)	0.0512 (6)	0.0147 (5)	−0.0117 (6)	0.0048 (8)
N	0.0425 (16)	0.0279 (14)	0.0430 (19)	0.0100 (12)	−0.0008 (19)	−0.002 (2)
O1	0.0477 (13)	0.0415 (14)	0.0431 (17)	0.0070 (13)	−0.0070 (15)	0.0018 (16)
C1	0.0351 (18)	0.0309 (18)	0.034 (2)	0.0025 (15)	0.0050 (19)	0.0035 (19)
O2	0.0524 (16)	0.0387 (14)	0.0427 (17)	0.0069 (13)	0.0030 (15)	−0.0108 (15)
C2	0.0398 (18)	0.0263 (15)	0.033 (2)	0.0039 (15)	0.0081 (18)	−0.0007 (18)
C3	0.0341 (16)	0.0284 (15)	0.034 (2)	0.0043 (14)	0.0069 (18)	0.0027 (17)
C4	0.0402 (18)	0.0330 (17)	0.032 (2)	0.0046 (15)	0.0074 (18)	−0.0012 (18)
C5	0.0326 (16)	0.045 (2)	0.034 (2)	0.0015 (16)	0.0002 (18)	0.006 (2)
C6	0.0439 (19)	0.045 (2)	0.033 (2)	−0.0096 (18)	0.0013 (18)	−0.0013 (19)
C7	0.049 (2)	0.0302 (17)	0.038 (2)	−0.0020 (16)	0.009 (2)	−0.0053 (18)
C8	0.0352 (17)	0.0294 (17)	0.035 (2)	−0.0004 (15)	0.0059 (18)	−0.0018 (18)

Geometric parameters (Å, º)

Cl—C5	1.748 (4)	C3—C8	1.404 (5)
N—C1	1.349 (6)	C4—C5	1.381 (6)
N—C8	1.412 (5)	C4—H4A	0.9300
N—H0A	0.8600	C5—C6	1.389 (6)
O1—C1	1.229 (5)	C6—C7	1.380 (6)
C1—C2	1.521 (4)	C6—H6A	0.9300
O2—C2	1.213 (4)	C7—C8	1.369 (6)
C2—C3	1.471 (5)	C7—H7A	0.9300
C3—C4	1.381 (5)		

C1—N—C8	111.4 (3)	C3—C4—H4A	121.2
C1—N—H0A	124.3	C4—C5—C6	121.7 (4)
C8—N—H0A	124.3	C4—C5—Cl	119.7 (3)
O1—C1—N	126.7 (3)	C6—C5—Cl	118.6 (3)
O1—C1—C2	126.5 (4)	C7—C6—C5	120.9 (4)
N—C1—C2	106.8 (3)	C7—C6—H6A	119.5
O2—C2—C3	129.6 (3)	C5—C6—H6A	119.5
O2—C2—C1	125.1 (4)	C8—C7—C6	117.6 (4)
C3—C2—C1	105.3 (3)	C8—C7—H7A	121.2
C4—C3—C8	120.3 (4)	C6—C7—H7A	121.2
C4—C3—C2	132.9 (3)	C7—C8—C3	121.8 (4)
C8—C3—C2	106.7 (3)	C7—C8—N	128.5 (3)
C5—C4—C3	117.6 (4)	C3—C8—N	109.7 (4)
C5—C4—H4A	121.2		

C8—N—C1—O1	176.7 (4)	C3—C4—C5—Cl	−176.5 (3)
C8—N—C1—C2	−0.8 (4)	C4—C5—C6—C7	−1.8 (6)
O1—C1—C2—O2	2.4 (7)	Cl—C5—C6—C7	176.9 (3)
N—C1—C2—O2	179.9 (4)	C5—C6—C7—C8	1.0 (6)
O1—C1—C2—C3	−177.1 (4)	C6—C7—C8—C3	−0.7 (6)
N—C1—C2—C3	0.4 (4)	C6—C7—C8—N	−179.5 (4)
O2—C2—C3—C4	−0.2 (7)	C4—C3—C8—C7	1.2 (6)
C1—C2—C3—C4	179.2 (4)	C2—C3—C8—C7	−179.6 (4)
O2—C2—C3—C8	−179.3 (4)	C4—C3—C8—N	−179.8 (4)
C1—C2—C3—C8	0.2 (4)	C2—C3—C8—N	−0.6 (4)
C8—C3—C4—C5	−1.9 (6)	C1—N—C8—C7	179.8 (4)
C2—C3—C4—C5	179.2 (4)	C1—N—C8—C3	0.9 (5)
C3—C4—C5—C6	2.2 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O1^{i}	0.86	2.04	2.893 (4)	172
C7—H7*A*···O2^{ii}	0.93	2.39	3.301 (5)	166

Symmetry codes: (i) −*x*+1, −*y*, *z*−1/2; (ii) *x*, *y*−1, *z*−1.