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6-Chloro-1-methylindoline-2,3-dione

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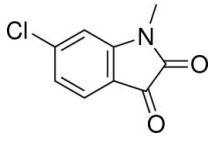
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.118; data-to-parameter ratio = 13.1.

The title molecule, $C_9H_6ClNO_2$, is essentially planar: the maximum deviation from the mean plane of the indoline ring is 0.020 (2) Å and the substituents do not deviate by more than 0.053 (2) Å from this plane. C—H···O hydrogen bonds help to consolidate the crystal structure.

Related literature

The title compound is a halogenated derivative of isatin. For the cytotoxic and antineoplastic activity of halogenated isatin derivatives, see: Vine *et al.* (2007); Matesic *et al.* (2008). For the preparation of the title compound, see: Bouhfid *et al.* (2005). For a related structure, see: Wu *et al.* (2011).



Experimental

Crystal data

$C_9H_6ClNO_2$
M_r = 195.60
Monoclinic, $C2/c$
a = 13.077 (3) Å
b = 7.9390 (16) Å
c = 16.673 (3) Å
β = 101.95 (3)°
V = 1693.5 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.887, T_{max} = 0.960
3124 measured reflections
1557 independent reflections
1250 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.118
S = 1.00
1557 reflections
119 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O1^{i}	0.93	2.50	3.419 (2)	168

Symmetry code: (i) $x-\frac{1}{2}, y-\frac{1}{2}, z$.

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: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2487).

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supporting information

Acta Cryst. (2012). E**68**, o14 [doi:10.1107/S1600536811051294]

6-Chloro-1-methylindoline-2,3-dione

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S1. Comment

Halogenated derivatives of isatin have been reported to exhibit cytotoxic and antineoplastic activities (Vine *et al.*, 2007; Matesic *et al.*, 2008). As a part of our studies on the synthesis of isatin derivatives, the title compound was synthesized (Bouhfid *et al.* (2005)). We report herein the crystal structure of the title compound.

The title molecule (Fig. 1) is essentially planar with the maximum deviation of C4 atom from the mean-plane of indoline ring (N,C1–C8) is 0.020 (2) Å and the substituents do not deviate more than 0.053 (2) Å from this plane. In the crystal structure, intermolecular and intramolecular C—H···O hydrogen bonds helps to consolidate the crystal packing (Fig. 2 & Tab. 1).

S2. Experimental

6-Chloroisatin (1.81 g, 0.01 mol) was reacted with iodomethane (0.02 mol) in the presence of K_2CO_3 (2.76 g, 0.02 mol) and tetrabutylammonium bromide (0.32 g, 0.001 mol) in DMF (60 ml). After 12 h stirring at room temperature, the precipitate was removed by filtration and purified by recrystallization from ethanol (m.p. 450–451 K; yield 67%). Yellow crystals of the title compound were obtained by slow evaporation from ethanol at room temperature.

S3. Refinement

All H atoms were placed geometrically at the distances C—H = 0.93 and 0.96 Å for aryl and methyl type H-atoms and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

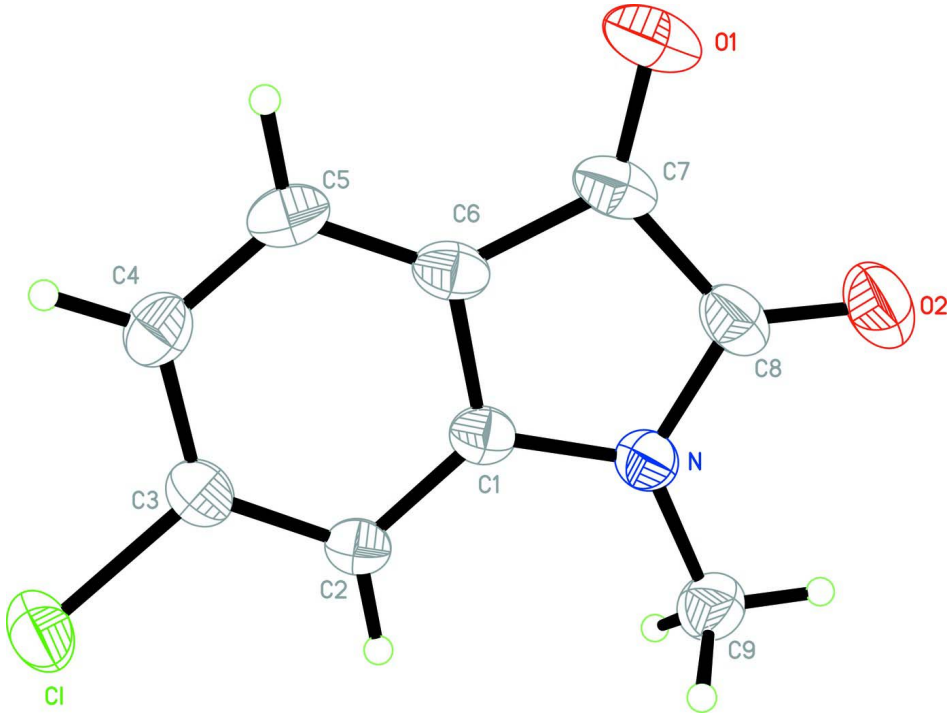

Figure 1

The molecular structure of the title molecule showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

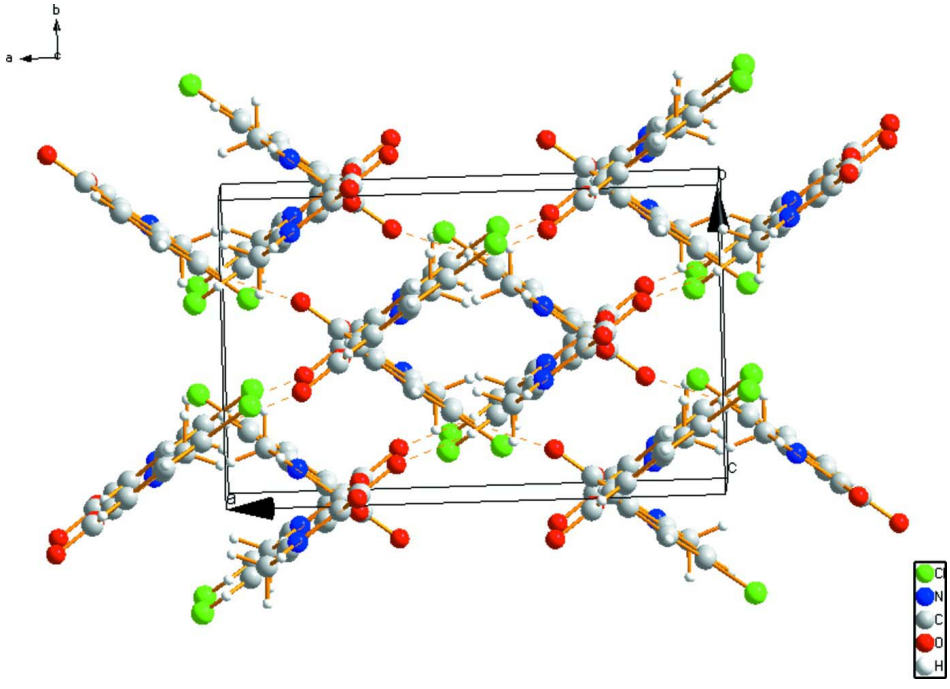

Figure 2

A packing diagram of the title compound. The intermolecular hydrogen bonds are shown as dashed lines.

6-Chloro-1-methylindoline-2,3-dione

Crystal data

$C_9H_6ClNO_2$
$M_r = 195.60$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 13.077$ (3) Å
$b = 7.9390$ (16) Å
$c = 16.673$ (3) Å
$\beta = 101.95$ (3)°
$V = 1693.5$ (6) Å^3
$Z = 8$
$F(000) = 800$
$D_x = 1.534$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.41$ mm^{-1}
$T = 293$ K
Block, yellow
0.30 × 0.20 × 0.10 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)
$T_{min} = 0.887$, $T_{max} = 0.960$
3124 measured reflections
1557 independent reflections
1250 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.5°$
$h = 0 \rightarrow 15$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 20$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.118$
$S = 1.00$
1557 reflections
119 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.080P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl	0.44670 (4)	0.16058 (8)	0.34958 (3)	0.0700 (3)
N	0.64277 (11)	0.39456 (19)	0.62790 (9)	0.0490 (4)
C1	0.62506 (13)	0.3717 (2)	0.54308 (11)	0.0427 (4)

O1	0.84333 (11)	0.63253 (19)	0.57603 (13)	0.0790 (5)
C2	0.54622 (13)	0.2784 (2)	0.49497 (11)	0.0441 (4)
H2A	0.4961	0.2225	0.5172	0.053*
O2	0.76431 (12)	0.5344 (2)	0.72436 (10)	0.0801 (5)
C3	0.54593 (14)	0.2728 (2)	0.41247 (12)	0.0492 (5)
C4	0.62028 (16)	0.3525 (2)	0.37645 (13)	0.0552 (5)
H4A	0.6180	0.3424	0.3205	0.066*
C5	0.69747 (15)	0.4469 (2)	0.42582 (13)	0.0554 (5)
H5A	0.7472	0.5033	0.4033	0.067*
C6	0.69983 (13)	0.4564 (2)	0.50844 (12)	0.0484 (5)
C7	0.76899 (14)	0.5415 (2)	0.57711 (15)	0.0579 (5)
C8	0.72891 (14)	0.4939 (2)	0.65333 (14)	0.0579 (5)
C9	0.58013 (17)	0.3220 (3)	0.68150 (14)	0.0608 (5)
H9A	0.6096	0.3521	0.7373	0.091*
H9B	0.5794	0.2016	0.6761	0.091*
H9C	0.5100	0.3643	0.6666	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0696 (4)	0.0753 (4)	0.0577 (4)	−0.0114 (3)	−0.0042 (3)	−0.0050 (2)
N	0.0434 (9)	0.0508 (9)	0.0522 (9)	−0.0023 (7)	0.0088 (7)	−0.0046 (7)
C1	0.0372 (9)	0.0385 (8)	0.0526 (10)	0.0041 (7)	0.0095 (7)	0.0015 (7)
O1	0.0522 (9)	0.0643 (9)	0.1197 (15)	−0.0190 (8)	0.0158 (9)	−0.0066 (9)
C2	0.0394 (9)	0.0423 (9)	0.0509 (10)	−0.0021 (7)	0.0100 (7)	0.0035 (7)
O2	0.0701 (10)	0.0856 (11)	0.0755 (12)	−0.0061 (8)	−0.0060 (8)	−0.0214 (9)
C3	0.0455 (10)	0.0446 (10)	0.0544 (11)	0.0033 (8)	0.0031 (8)	0.0022 (8)
C4	0.0631 (12)	0.0558 (12)	0.0481 (11)	0.0060 (9)	0.0143 (9)	0.0089 (8)
C5	0.0535 (11)	0.0511 (11)	0.0673 (13)	0.0018 (9)	0.0253 (9)	0.0131 (9)
C6	0.0384 (9)	0.0385 (9)	0.0690 (13)	0.0007 (7)	0.0126 (8)	0.0029 (8)
C7	0.0377 (10)	0.0444 (10)	0.0900 (16)	−0.0023 (8)	0.0098 (9)	−0.0042 (9)
C8	0.0433 (10)	0.0534 (11)	0.0715 (14)	0.0020 (8)	−0.0009 (9)	−0.0132 (9)
C9	0.0596 (12)	0.0710 (14)	0.0530 (12)	0.0004 (10)	0.0141 (9)	0.0037 (10)

Geometric parameters (Å, º)

Cl—C3	1.7361 (19)	C3—C4	1.396 (3)
N—C8	1.369 (2)	C4—C5	1.383 (3)
N—C1	1.397 (2)	C4—H4A	0.9300
N—C9	1.451 (3)	C5—C6	1.373 (3)
C1—C2	1.383 (2)	C5—H5A	0.9300
C1—C6	1.406 (2)	C6—C7	1.468 (3)
O1—C7	1.215 (2)	C7—C8	1.519 (3)
C2—C3	1.376 (3)	C9—H9A	0.9600
C2—H2A	0.9300	C9—H9B	0.9600
O2—C8	1.222 (3)	C9—H9C	0.9600
C8—N—C1	109.98 (16)	C4—C5—H5A	120.4

C8—N—C9	124.86 (18)	C5—C6—C1	120.85 (18)
C1—N—C9	125.16 (15)	C5—C6—C7	133.59 (17)
C2—C1—N	127.17 (16)	C1—C6—C7	105.55 (17)
C2—C1—C6	121.09 (17)	O1—C7—C6	128.9 (2)
N—C1—C6	111.74 (16)	O1—C7—C8	125.2 (2)
C3—C2—C1	116.40 (16)	C6—C7—C8	105.93 (16)
C3—C2—H2A	121.8	O2—C8—N	125.0 (2)
C1—C2—H2A	121.8	O2—C8—C7	128.2 (2)
C2—C3—C4	123.89 (18)	N—C8—C7	106.77 (17)
C2—C3—Cl	117.88 (14)	N—C9—H9A	109.5
C4—C3—Cl	118.23 (15)	N—C9—H9B	109.5
C5—C4—C3	118.50 (19)	H9A—C9—H9B	109.5
C5—C4—H4A	120.8	N—C9—H9C	109.5
C3—C4—H4A	120.8	H9A—C9—H9C	109.5
C6—C5—C4	119.24 (17)	H9B—C9—H9C	109.5
C6—C5—H5A	120.4		

C8—N—C1—C2	179.45 (16)	C2—C1—C6—C7	179.49 (15)
C9—N—C1—C2	0.2 (3)	N—C1—C6—C7	−0.97 (19)
C8—N—C1—C6	0.0 (2)	C5—C6—C7—O1	2.2 (4)
C9—N—C1—C6	−179.27 (17)	C1—C6—C7—O1	−178.19 (19)
N—C1—C2—C3	−179.06 (16)	C5—C6—C7—C8	−178.04 (19)
C6—C1—C2—C3	0.4 (2)	C1—C6—C7—C8	1.52 (19)
C1—C2—C3—C4	1.0 (3)	C1—N—C8—O2	−179.21 (19)
C1—C2—C3—Cl	−178.72 (13)	C9—N—C8—O2	0.0 (3)
C2—C3—C4—C5	−1.9 (3)	C1—N—C8—C7	1.0 (2)
Cl—C3—C4—C5	177.82 (14)	C9—N—C8—C7	−179.76 (16)
C3—C4—C5—C6	1.3 (3)	O1—C7—C8—O2	−1.6 (3)
C4—C5—C6—C1	0.0 (3)	C6—C7—C8—O2	178.7 (2)
C4—C5—C6—C7	179.48 (19)	O1—C7—C8—N	178.14 (18)
C2—C1—C6—C5	−0.9 (3)	C6—C7—C8—N	−1.6 (2)
N—C1—C6—C5	178.65 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···O1^{i}	0.93	2.50	3.419 (2)	168
C9—H9*A*···O2	0.96	2.53	2.906 (3)	103

Symmetry code: (i) *x*−1/2, *y*−1/2, *z*.