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(3*Z*)-5-Chloro-3-(hydroxyimino)indolin-2-one

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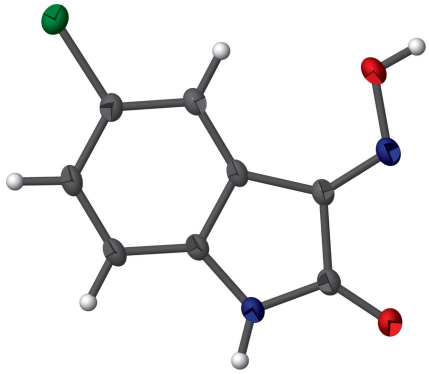
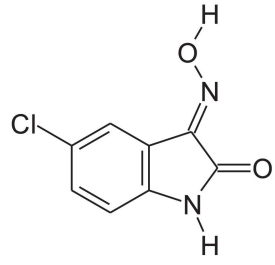
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_8H_5ClN_2O_2$ (common name: 5-chloroisatin 3-oxime), the molecular structure deviates slightly from the ideal planarity, with a maximum deviation of 0.0478 (8) Å for the non-H atoms. In the crystal, molecules are linked by N—H···O interactions, building centrosymmetric dimers with graph-set motif $R_2^2(8)$. Additionally, the molecules are connected by pairs of O—H···O interactions into chains along [100] with a $C(6)$ motif. The hydrogen-bonded dimers and chains build a two-dimensional network parallel to (100). The packing also features π–π stacking interactions between benzene rings [centroid–centroid distance = 3.748 (2) Å].

3D view



Chemical scheme



Structure description

As part of our study on the structural chemistry of isatin derivatives we report herein the crystal structure of 5-chloroisatin-3-oxime (for the asymmetric unit, see Fig. 1). The title compound is almost planar with an r.m.s. maximum deviation for the non-H atoms of 0.0478 (8) Å for O2. In the solid state, the molecules are connected into dimers *via* pairs of N1—H1···O1 interactions and a graph-set motif $R_2^2(8)$ is observed. In addition, the molecules are connected into chains with a $C(6)$ graph-set motif by O2—H5···O1 hydrogen bonds (Fig. 2 and Table 1). The O—H···O interactions connect the centrosymmetric dimers building a two-dimensional network, a tape structure, parallel to (100). As the outstanding feature, the ketone oxygen atom, O1, accepts two hydrogen bonds. As the difference between the two H···O distances is less than 0.2 Å, the hydrogen bonds presumably have roughly equal strength and the arrangement may be described as symmetric. The packing also features π–π stacking interactions between benzene rings [centroid–centroid distance = 3.748 (2) A °].

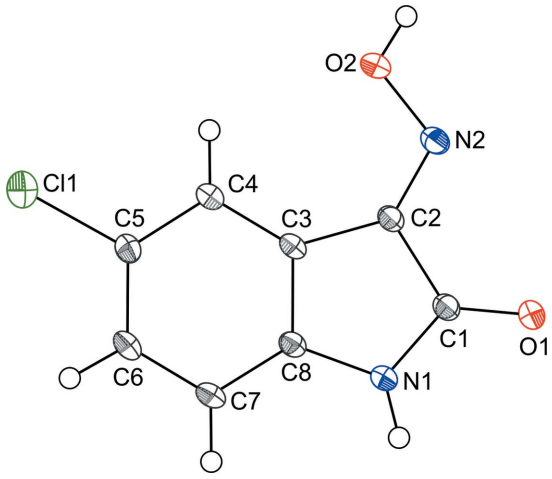


Figure 1
The molecular structure of the title compound, with labeling and displacement ellipsoids drawn at the 40% probability level.

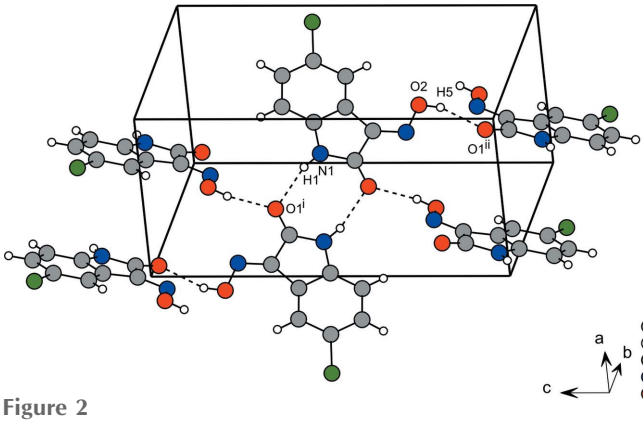


Figure 2
Partial view of the crystal structure of the title compound. Hydrogen bonds are shown as dashed lines; see Table 2 for details.

Synthesis and crystallization

The glacial acetic acid catalyzed reaction of 5-chloroisatin (3 mmol) and hydroxylamine hydrochloride (3 mmol) in ethanol (50 ml) was stirred and refluxed for 6 h. After cooling and filtering, single crystals suitable for X-ray diffraction were obtained from the ethanolic solution by solvent evaporation. For an alternative synthesis of 5-chloroisatin-3-oxime, see: Kearney *et al.*, 1992.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88	2.00	2.8476 (13)	162
O2—H5···O1^{ii}	0.84	1.94	2.7235 (12)	155

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_5ClN_2O_2$
M_r	196.59
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	200
a, b, c (Å)	7.4296 (3), 7.5187 (3), 14.3206 (7)
β (°)	94.184 (1)
V (Å^3)	797.83 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.44
Crystal size (mm)	0.24 × 0.20 × 0.16
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.902, 0.933
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10472, 2451, 2182
R_{int}	0.014
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.716
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.095, 1.05
No. of reflections	2451
No. of parameters	119
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.28

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 2006), *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

References

Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2012). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kearney, T., Harris, P. A., Jackson, A. & Joule, J. A. (1992). *Synthesis*, **1992**, 769–772.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2016). **1**, x161506 [https://doi.org/10.1107/S2414314616015066]

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(3*Z*)-5-Chloro-3-(hydroxyimino)indolin-2-one

Crystal data

$C_8H_5ClN_2O_2$
M_r = 196.59
Monoclinic, $P2_1/c$
a = 7.4296 (3) Å
b = 7.5187 (3) Å
c = 14.3206 (7) Å
β = 94.184 (1)°
V = 797.83 (6) Å^3
Z = 4
$F(000)$ = 400
D_x = 1.637 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5902 reflections
θ = 2.8–30.6°
μ = 0.44 mm^{-1}
T = 200 K
Prism, orange
0.24 × 0.20 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube, Bruker APEX2
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.902, T_{max} = 0.933
10472 measured reflections
2451 independent reflections
2182 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 30.6°, θ_{min} = 2.8°
h = −10→10
k = −10→10
l = −19→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.095
S = 1.05
2451 reflections
119 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2884P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.58311 (15)	0.21274 (15)	0.43029 (7)	0.0205 (2)
C2	0.64041 (15)	0.39353 (14)	0.39942 (7)	0.0192 (2)
C3	0.72801 (14)	0.48058 (14)	0.48207 (7)	0.0182 (2)
C4	0.80538 (14)	0.64698 (15)	0.49770 (7)	0.0202 (2)
H2	0.8097	0.7320	0.4488	0.024*
C5	0.87646 (15)	0.68356 (15)	0.58850 (7)	0.0215 (2)
C6	0.87171 (16)	0.56117 (17)	0.66104 (8)	0.0246 (2)
H3	0.9214	0.5918	0.7219	0.030*
C7	0.79446 (16)	0.39372 (16)	0.64513 (8)	0.0237 (2)
H4	0.7913	0.3086	0.6940	0.028*
C8	0.72266 (14)	0.35647 (14)	0.55542 (7)	0.0193 (2)
Cl1	0.97441 (4)	0.89044 (4)	0.61183 (2)	0.03057 (11)
N1	0.63574 (13)	0.20035 (13)	0.52268 (6)	0.02202 (19)
H1	0.6178	0.1066	0.5576	0.026*
N2	0.59837 (14)	0.43956 (13)	0.31406 (6)	0.0231 (2)
O1	0.50045 (13)	0.09832 (11)	0.38255 (6)	0.02689 (19)
O2	0.64795 (14)	0.61138 (12)	0.29692 (6)	0.0291 (2)
H5	0.6115	0.6396	0.2420	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0238 (5)	0.0208 (5)	0.0166 (4)	0.0020 (4)	−0.0008 (4)	0.0000 (4)
C2	0.0217 (5)	0.0208 (5)	0.0147 (4)	0.0018 (4)	−0.0004 (3)	−0.0002 (3)
C3	0.0192 (4)	0.0223 (5)	0.0130 (4)	0.0027 (4)	−0.0005 (3)	0.0006 (3)
C4	0.0217 (5)	0.0233 (5)	0.0153 (4)	0.0008 (4)	−0.0002 (4)	0.0012 (4)
C5	0.0220 (5)	0.0238 (5)	0.0183 (5)	−0.0009 (4)	−0.0021 (4)	−0.0014 (4)
C6	0.0273 (5)	0.0299 (6)	0.0157 (4)	−0.0004 (4)	−0.0049 (4)	0.0002 (4)
C7	0.0282 (5)	0.0269 (5)	0.0154 (5)	0.0000 (4)	−0.0033 (4)	0.0040 (4)
C8	0.0204 (4)	0.0212 (5)	0.0160 (4)	0.0017 (4)	−0.0010 (3)	0.0014 (4)
Cl1	0.03654 (18)	0.02817 (16)	0.02608 (16)	−0.00784 (11)	−0.00398 (12)	−0.00205 (10)
N1	0.0285 (5)	0.0207 (4)	0.0162 (4)	−0.0008 (3)	−0.0029 (3)	0.0021 (3)
N2	0.0282 (5)	0.0244 (4)	0.0166 (4)	0.0011 (4)	−0.0003 (3)	0.0010 (3)
O1	0.0375 (5)	0.0234 (4)	0.0190 (4)	−0.0039 (3)	−0.0029 (3)	−0.0013 (3)
O2	0.0416 (5)	0.0271 (4)	0.0176 (4)	−0.0048 (4)	−0.0044 (3)	0.0054 (3)

Geometric parameters (Å, º)

C1—O1	1.2343 (13)	C5—Cl1	1.7393 (11)
C1—N1	1.3551 (13)	C6—C7	1.3955 (16)
C1—C2	1.5006 (15)	C6—H3	0.9500
C2—N2	1.2869 (13)	C7—C8	1.3833 (14)
C2—C3	1.4629 (14)	C7—H4	0.9500
C3—C4	1.3882 (15)	C8—N1	1.4038 (14)
C3—C8	1.4078 (14)	N1—H1	0.8800
C4—C5	1.3947 (14)	N2—O2	1.3704 (13)
C4—H2	0.9500	O2—H5	0.8400
C5—C6	1.3902 (16)		
O1—C1—N1	126.11 (10)	C5—C6—C7	120.52 (10)
O1—C1—C2	127.48 (10)	C5—C6—H3	119.7
N1—C1—C2	106.38 (9)	C7—C6—H3	119.7
N2—C2—C3	135.31 (10)	C8—C7—C6	117.41 (10)
N2—C2—C1	117.97 (10)	C8—C7—H4	121.3
C3—C2—C1	106.62 (9)	C6—C7—H4	121.3
C4—C3—C8	120.73 (9)	C7—C8—N1	128.05 (10)
C4—C3—C2	133.49 (9)	C7—C8—C3	121.93 (10)
C8—C3—C2	105.78 (9)	N1—C8—C3	110.02 (9)
C3—C4—C5	116.89 (10)	C1—N1—C8	111.19 (9)
C3—C4—H2	121.6	C1—N1—H1	124.4
C5—C4—H2	121.6	C8—N1—H1	124.4
C6—C5—C4	122.52 (10)	C2—N2—O2	111.96 (9)
C6—C5—Cl1	118.78 (8)	N2—O2—H5	109.5
C4—C5—Cl1	118.69 (9)		
O1—C1—C2—N2	1.07 (18)	C5—C6—C7—C8	−0.52 (18)
N1—C1—C2—N2	−177.28 (10)	C6—C7—C8—N1	−178.76 (11)
O1—C1—C2—C3	178.05 (11)	C6—C7—C8—C3	0.66 (17)
N1—C1—C2—C3	−0.30 (12)	C4—C3—C8—C7	−0.45 (16)
N2—C2—C3—C4	−2.8 (2)	C2—C3—C8—C7	179.89 (10)
C1—C2—C3—C4	−179.06 (11)	C4—C3—C8—N1	179.07 (10)
N2—C2—C3—C8	176.75 (13)	C2—C3—C8—N1	−0.59 (12)
C1—C2—C3—C8	0.54 (11)	O1—C1—N1—C8	−178.44 (11)
C8—C3—C4—C5	0.08 (15)	C2—C1—N1—C8	−0.07 (12)
C2—C3—C4—C5	179.63 (11)	C7—C8—N1—C1	179.91 (11)
C3—C4—C5—C6	0.05 (17)	C3—C8—N1—C1	0.43 (13)
C3—C4—C5—Cl1	−179.83 (8)	C3—C2—N2—O2	0.03 (19)
C4—C5—C6—C7	0.18 (18)	C1—C2—N2—O2	175.92 (9)
Cl1—C5—C6—C7	−179.94 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88	2.00	2.8476 (13)	162

O2—H5···O1^{ii}	0.84	1.94	2.7235 (12)	155

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