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1-Allyl-5-chloroindoline-2,3-dione

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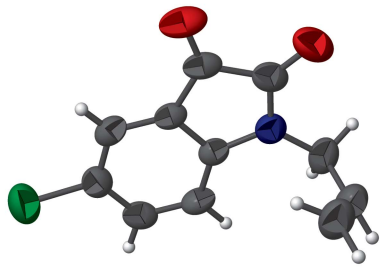
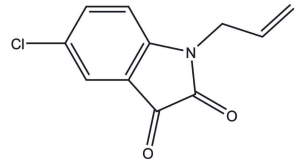
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_8ClNO_2$, the allyl side chain is almost perpendicular to the 5-chloroindoline-2,3-dione ring system, with a dihedral angle of 88.0 (3)°. In the crystal, C—H···O interactions link the molecules into layers lying parallel to the *bc* plane.

3D view



Chemical scheme



Structure description

As part of our ongoing studies of 5-chloroisatin derivatives (Kharbach *et al.*, 2016), we now describe the structure of the title compound, $C_{11}H_8ClNO_2$ (Fig. 1), which was obtained by reaction of 5-chloroisatin with allyl bromide in phase-transfer catalysis conditions.

The allyl group is almost perpendicular to the 5-chloroindoline-2,3-dione ring system (r.m.s. deviation = 0.034 Å) with a dihedral angle of 88.0 (3)°: the N1—C9—C10—C11 torsion angle is −5.7 (5)°. In the crystal, C—H···O interactions (Fig. 2, Table 1) link molecules into layers running parallel to the *bc* plane (Fig. 3).

Synthesis and crystallization

To a solution of 5-chloro-1*H*-indole-2,3-dione (0.4 g, 2,20 mmol) in DMF (25 ml) was added K_2CO_3 (0.5 g, 3,30 mmol) as a base, tetra-*n*-butylammoium bromide (0.1 g, 0,3 mmol) as catalyst and then 3-bromoprop-1-ene (0.34 ml, 2.97 mmol). The reaction mixture was stirred for 48 h at room temperature; once the reaction was complete, the solvent was evaporated *in vacuo*. The title compound obtained was recrystallized from ethanol solution to afford red crystals (yield: 89%, m.p.: 415 K).

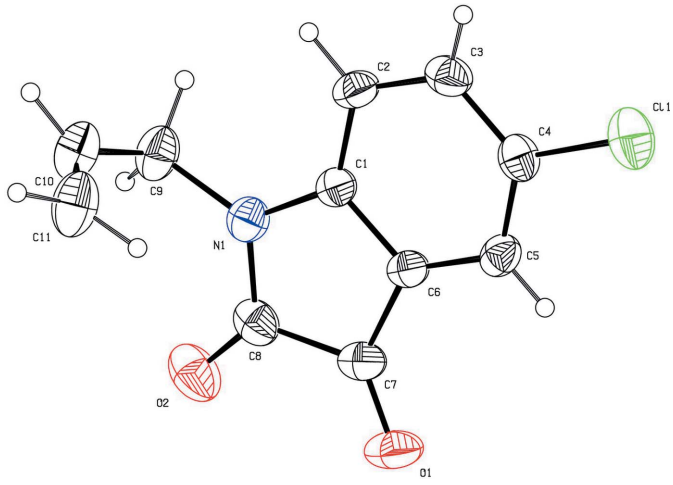


Figure 1
The molecular structure of the title molecule, showing displacement ellipsoids drawn at the 30% probability level.

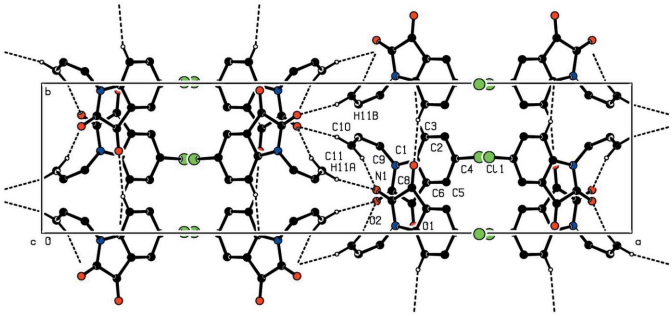


Figure 2
The crystal structure of the title compound, viewed along the *c* axis, showing layers parallel to the *bc* plane linked by C—H···O hydrogen bonds (dashed lines). For the sake of clarity, H atoms not involved in the hydrogen bonds have been omitted.

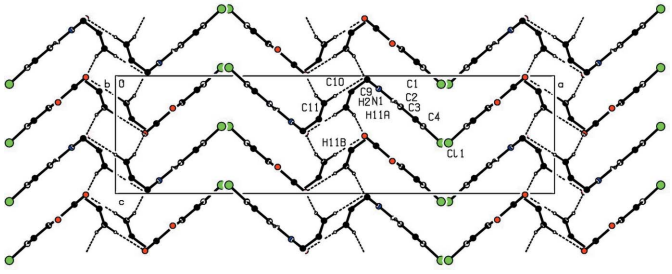


Figure 3
The crystal structure of the title compound, viewed along the *b* axis, showing chains parallel to the *bc* plane linked by C—H···O hydrogen bonds (dashed lines). For the sake of clarity, H atoms not involved in the hydrogen bonds have been omitted.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1^{i}	0.96 (2)	2.42 (2)	3.283 (2)	151.0 (19)
C11—H11A···O2^{ii}	1.02 (4)	2.39 (3)	3.377 (3)	165 (3)
C11—H11B···O2^{iii}	1.04 (3)	2.55 (3)	3.572 (3)	167 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_8ClNO_2$
M_r	221.63
Crystal system, space group	Orthorhombic, *Pccn*
Temperature (K)	296
a, b, c (Å)	31.2222 (6), 7.9107 (2), 8.3373 (2)
V (Å^3)	2059.23 (8)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.35
Crystal size (mm)	0.53 × 0.30 × 0.28
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*, Bruker, 2015)
T_{min}, T_{max}	0.705, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33452, 2554, 1889
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.666
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.146, 1.02
No. of reflections	2554
No. of parameters	168
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.20

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Refinement

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

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full crystallographic data

IUCrData (2016). **1**, x160862 [doi:10.1107/S2414314616008622]

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Crystal data

$C_{11}H_8ClNO_2$
M_r = 221.63
Orthorhombic, *Pccn*
a = 31.2222 (6) Å
b = 7.9107 (2) Å
c = 8.3373 (2) Å
V = 2059.23 (8) Å^3
Z = 8
F(000) = 912
D_x = 1.430 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 9899 reflections
θ = 2.7–26.7°
μ = 0.35 mm^{-1}
T = 296 K
Block, red
0.53 × 0.30 × 0.28 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS, Bruker, 2015)
T_{min} = 0.705, T_{max} = 0.746
33452 measured reflections
2554 independent reflections
1889 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.3°, θ_{min} = 2.6°
h = −41→41
k = −10→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.146
S = 1.02
2554 reflections
168 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2 + 0.7243P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Experimental. SADABS-2014/5 (Bruker,2014/5) was used for absorption correction. wR2(int) was 0.0610 before and 0.0436 after correction. The Ratio of minimum to maximum transmission is 0.9447. The $\lambda/2$ correction factor is Not present.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.74155 (2)	0.50698 (9)	0.57033 (8)	0.0830 (3)
O1	0.63056 (6)	0.04791 (17)	0.2249 (2)	0.0740 (5)
N1	0.59967 (5)	0.4519 (2)	0.10974 (19)	0.0534 (4)
O2	0.56737 (5)	0.2127 (2)	0.0112 (2)	0.0819 (5)
C6	0.65189 (5)	0.3371 (2)	0.2739 (2)	0.0442 (4)
C1	0.63365 (6)	0.4861 (2)	0.2156 (2)	0.0442 (4)
C7	0.62735 (6)	0.1975 (2)	0.2048 (2)	0.0527 (5)
C5	0.68569 (6)	0.3403 (2)	0.3809 (2)	0.0503 (4)
C2	0.64914 (7)	0.6414 (2)	0.2625 (2)	0.0536 (5)
C8	0.59347 (6)	0.2826 (3)	0.0950 (2)	0.0573 (5)
C4	0.70061 (6)	0.4964 (3)	0.4293 (2)	0.0545 (5)
C3	0.68305 (7)	0.6445 (3)	0.3703 (3)	0.0568 (5)
C9	0.57319 (8)	0.5799 (4)	0.0313 (3)	0.0660 (6)
C10	0.53684 (8)	0.6399 (4)	0.1317 (3)	0.0751 (7)
C11	0.52504 (9)	0.5846 (4)	0.2675 (3)	0.0843 (8)
H2	0.6377 (7)	0.744 (3)	0.220 (3)	0.066 (6)*
H5	0.6966 (7)	0.247 (3)	0.423 (2)	0.058 (6)*
H3	0.6932 (7)	0.743 (3)	0.409 (3)	0.072 (7)*
H9A	0.5617 (9)	0.529 (3)	−0.069 (3)	0.086 (8)*
H11A	0.5430 (13)	0.501 (4)	0.331 (4)	0.116 (11)*
H10	0.5239 (13)	0.753 (6)	0.133 (5)	0.162 (15)*
H11B	0.5002 (10)	0.641 (4)	0.330 (4)	0.102 (9)*
H9B	0.5925 (9)	0.678 (4)	0.002 (4)	0.095 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0629 (4)	0.1088 (6)	0.0774 (4)	−0.0004 (3)	−0.0143 (3)	−0.0100 (3)
O1	0.0905 (11)	0.0387 (7)	0.0928 (11)	−0.0027 (7)	0.0278 (9)	−0.0023 (7)
N1	0.0521 (8)	0.0554 (9)	0.0527 (8)	0.0046 (7)	0.0032 (7)	−0.0018 (7)
O2	0.0649 (9)	0.0938 (12)	0.0869 (11)	−0.0134 (8)	0.0027 (8)	−0.0329 (10)
C6	0.0468 (9)	0.0383 (8)	0.0474 (9)	0.0017 (6)	0.0135 (7)	0.0007 (7)
C1	0.0446 (8)	0.0434 (8)	0.0447 (8)	0.0027 (7)	0.0098 (7)	0.0011 (7)
C7	0.0584 (10)	0.0418 (9)	0.0578 (10)	−0.0020 (7)	0.0238 (9)	−0.0051 (8)
C5	0.0493 (9)	0.0487 (10)	0.0529 (10)	0.0093 (8)	0.0106 (8)	0.0057 (8)
C2	0.0616 (11)	0.0372 (9)	0.0620 (11)	0.0050 (8)	0.0098 (9)	0.0021 (8)
C8	0.0499 (9)	0.0625 (12)	0.0595 (11)	−0.0044 (9)	0.0136 (9)	−0.0139 (9)
C4	0.0457 (9)	0.0652 (12)	0.0525 (10)	−0.0001 (8)	0.0050 (8)	−0.0041 (9)
C3	0.0572 (11)	0.0482 (10)	0.0651 (12)	−0.0076 (8)	0.0097 (9)	−0.0097 (9)
C9	0.0627 (13)	0.0810 (15)	0.0543 (11)	0.0172 (11)	−0.0011 (10)	0.0087 (11)
C10	0.0705 (14)	0.0852 (17)	0.0695 (14)	0.0262 (12)	−0.0011 (11)	0.0013 (12)
C11	0.0718 (15)	0.111 (2)	0.0702 (16)	0.0296 (15)	0.0040 (13)	−0.0046 (15)

Geometric parameters (Å, º)

Cl1—C4	1.739 (2)	C5—H5	0.89 (2)
O1—C7	1.200 (2)	C2—C3	1.389 (3)
N1—C1	1.406 (2)	C2—H2	0.95 (2)
N1—C8	1.359 (3)	C4—C3	1.384 (3)
N1—C9	1.461 (3)	C3—H3	0.91 (3)
O2—C8	1.208 (2)	C9—C10	1.487 (3)
C6—C1	1.396 (2)	C9—H9A	1.00 (3)
C6—C7	1.462 (3)	C9—H9B	1.01 (3)
C6—C5	1.382 (3)	C10—C11	1.269 (4)
C1—C2	1.377 (3)	C10—H10	0.98 (5)
C7—C8	1.552 (3)	C11—H11A	1.02 (4)
C5—C4	1.380 (3)	C11—H11B	1.04 (3)
C1—N1—C9	125.07 (18)	O2—C8—C7	127.1 (2)
C8—N1—C1	110.69 (16)	C5—C4—Cl1	119.25 (16)
C8—N1—C9	124.2 (2)	C5—C4—C3	121.34 (19)
C1—C6—C7	106.63 (16)	C3—C4—Cl1	119.40 (16)
C5—C6—C1	121.35 (16)	C2—C3—H3	121.0 (15)
C5—C6—C7	131.98 (16)	C4—C3—C2	121.12 (18)
C6—C1—N1	111.36 (15)	C4—C3—H3	117.8 (15)
C2—C1—N1	127.91 (17)	N1—C9—C10	113.65 (19)
C2—C1—C6	120.73 (17)	N1—C9—H9A	107.4 (16)
O1—C7—C6	130.2 (2)	N1—C9—H9B	107.7 (17)
O1—C7—C8	124.55 (19)	C10—C9—H9A	109.1 (16)
C6—C7—C8	105.22 (15)	C10—C9—H9B	110.3 (17)
C6—C5—H5	122.0 (14)	H9A—C9—H9B	109 (2)
C4—C5—C6	117.58 (17)	C9—C10—H10	128 (3)
C4—C5—H5	120.3 (14)	C11—C10—C9	127.8 (2)
C1—C2—C3	117.86 (18)	C11—C10—H10	101 (3)
C1—C2—H2	121.4 (14)	C10—C11—H11A	122 (2)
C3—C2—H2	120.7 (14)	C10—C11—H11B	121.3 (16)
N1—C8—C7	106.07 (16)	H11A—C11—H11B	116 (3)
O2—C8—N1	126.8 (2)		
Cl1—C4—C3—C2	−177.33 (15)	C1—C2—C3—C4	−0.3 (3)
O1—C7—C8—N1	177.91 (18)	C7—C6—C1—N1	−1.66 (18)
O1—C7—C8—O2	−3.3 (3)	C7—C6—C1—C2	178.41 (16)
N1—C1—C2—C3	179.47 (17)	C7—C6—C5—C4	−176.80 (17)
N1—C9—C10—C11	−5.7 (5)	C5—C6—C1—N1	−179.56 (15)
C6—C1—C2—C3	−0.6 (3)	C5—C6—C1—C2	0.5 (3)
C6—C7—C8—N1	−1.16 (18)	C5—C6—C7—O1	0.3 (3)
C6—C7—C8—O2	177.59 (18)	C5—C6—C7—C8	179.26 (17)
C6—C5—C4—Cl1	177.25 (13)	C5—C4—C3—C2	1.3 (3)
C6—C5—C4—C3	−1.4 (3)	C8—N1—C1—C6	0.9 (2)
C1—N1—C8—O2	−178.56 (18)	C8—N1—C1—C2	−179.15 (18)
C1—N1—C8—C7	0.19 (19)	C8—N1—C9—C10	92.4 (3)

C1—N1—C9—C10	−84.7 (3)	C9—N1—C1—C6	178.33 (17)
C1—C6—C7—O1	−177.32 (19)	C9—N1—C1—C2	−1.7 (3)
C1—C6—C7—C8	1.67 (17)	C9—N1—C8—O2	4.0 (3)
C1—C6—C5—C4	0.5 (3)	C9—N1—C8—C7	−177.24 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{i}	0.96 (2)	2.42 (2)	3.283 (2)	151.0 (19)
C11—H11*A*···O2^{ii}	1.02 (4)	2.39 (3)	3.377 (3)	165 (3)
C11—H11*B*···O2^{iii}	1.04 (3)	2.55 (3)	3.572 (3)	167 (2)

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