1-Benzyl-3,3-dichloro-1*H*-indol-2(3*H*)-one

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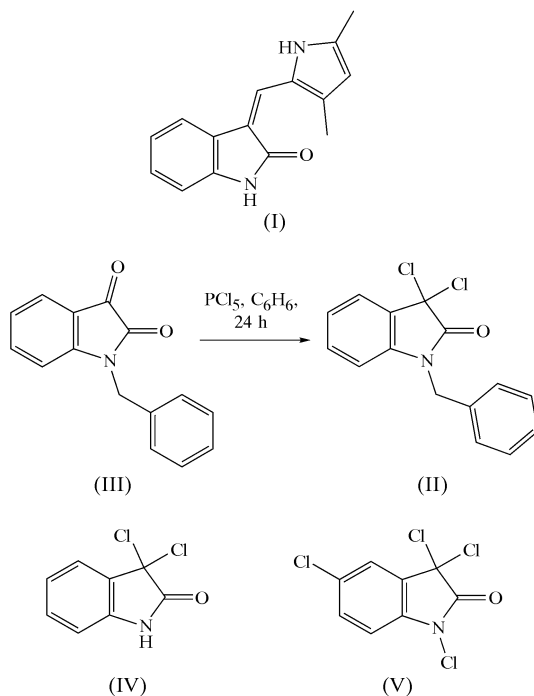Correspondence e-mail: dekoning@chem.wits.ac.za

The title compound, $C_{15}H_{11}Cl_2NO$, was synthesized from *N*-benzylisatin. The compound crystallizes as stacks of molecules running down the *c* axis. Molecules within each of these stacks interact with each other through $\pi-\pi$ and $C-H\cdots\pi$ interactions, and interact with neighbouring stacks through $C-H\cdots O$ interactions.

Comment

Oxindoles occur commonly as subunits of biologically active compounds. For example, compound (I) (see scheme) has been found to be a potential inhibitor of the kinase insert

As part of our research programme, we have been interested in the synthesis of substituted heterocycles, such as carbazoles (de Koning *et al.*, 2003; Pelly *et al.*, 2005; Pathak *et al.*, 2006) and fused indole systems (de Koning *et al.*, 2004). During the course of our work on the synthesis of potential kinase inhibitors (Fabbro *et al.*, 2002; Geyer *et al.*, 2005; Noble *et al.*, 2005), we had reason to synthesize the simple oxindole
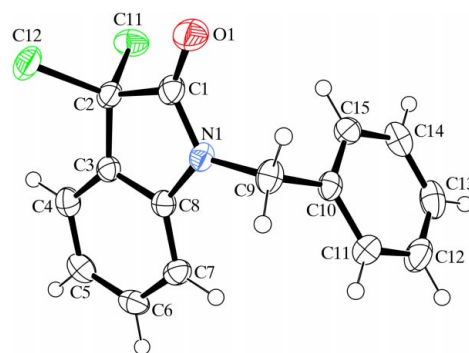

Figure 1
A view of the title molecule, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown with an arbitrary radius.
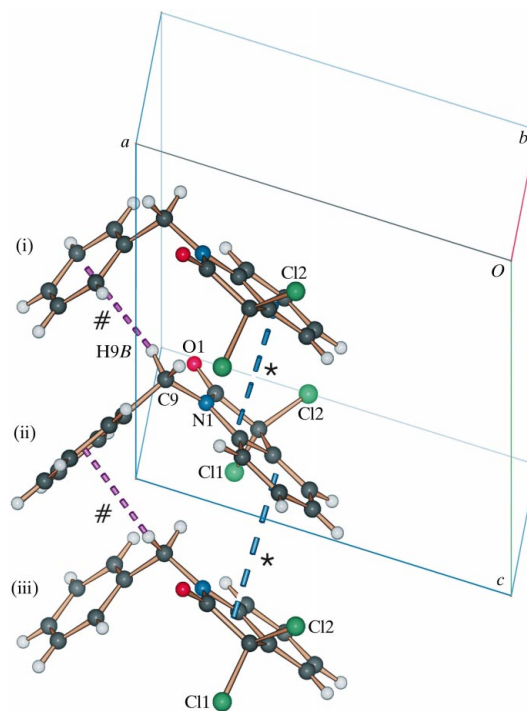

Figure 2
Weak interactions between molecules related by the *c*-glide plane producing a stack of molecules running down the *c* axis. Indicated with a star (★) on the diagram are the $\pi-\pi$ interactions between the N/C1–C3/C8 and C3–C8 rings. Indicated with a hash (#) are the $C-H\cdots\pi$ interactions between the C9–H9*B* group and the C10–C15 ring. Molecules (i), (ii) and (iii) are at the symmetry positions (x, y, z), $(x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(x, y, 1 + z)$, respectively.

domain-containing receptor (KDR), alternatively referred to as VEGFR-2, a receptor for vascular endothelial growth factors (Bouérat *et al.*, 2005). In essence, this compound is believed to function as a key regulator of angiogenesis.

organic compounds

derivative (II) from *N*-benzylisatin, (III), where the carbonyl group at the 3-position of isatin is replaced by two Cl atoms. The structure of the product, (II), was confirmed by single-crystal X-ray crystallography (Fig. 1).

The bond lengths and angles for (II) were found to be typical for compounds of this type. Bond lengths and angles for the nitrogen-containing ring of the indol-2-one system are given in Table 1. A search of the Cambridge Structural Database (CSD; Version 5.27; Allen, 2002) for indol-2-one compounds with dichloro substitution on C2 (or IUPAC position 3; Fig. 1) yielded only two structures, *viz.* 3,3-dichloro-1*H*-indol-2(3*H*)-one, (IV) (CSD refcode KUNMUB; Zukerman-Schpector *et al.*, 1993), and 1,3,3,5-tetrachloro-1,3-dihydroindol-2-one, (V) (QASXEO; Meketa *et al.*, 2005). Comparison of the bond angles around the N-containing five-membered ring indicates that the N1—C1 bond length, as well as the C8—N1—C1 and N1—C1—C2 angles, are most affected by the atom type bonded to the N atom (Table 3). Comparing geometric parameters around the rest of the five-

membered ring for all three structures leads to differences of less than 0.01 Å and 1° for the bond lengths and angles, respectively.

The title compound is capable of rotation around the C9—N1 and C9—C10 bonds (Fig. 1). The conformation adopted by the molecule is one in which the indol-2-one system is rotated such that it is almost perpendicular [82.03 (3)°] to the phenyl ring (Fig. 2). This conformation allows for simultaneous C—H···π and π–π interactions between molecules related by the *c*-glide plane. The C—H···π interaction occurs between the C9—H9*B* group and the C10–C15 ring of a neighbouring molecule (Table 2 and Fig. 2).

The π–π interaction occurs between the N1/C1–C3/C8 ring (the five-membered ring of the indol-2-one system) and the C3–C8 ring (the six-membered ring of the indol-2-one system) of a neighbouring molecule (Fig. 2). In this interaction, the two rings are slipped by 30.2° relative to their ring perpendiculars (the average interplanar distance is 3.484 Å), the ring-centroid-to-ring-centroid distance being 4.0329 (9) Å. Admittedly, the five-membered ring of the indol-2-one system is only partly aromatic, but a significant part of the ring (O1, C1 and N1) is involved in conjugation with the aromatic six-membered ring, as shown by the bond lengths between these atoms (Table 1). The π–π interaction is therefore really between these atoms and those of the neighbouring ring, and if this were taken into account the centroid-to-centroid distance would be even shorter. A consequence of the C—H···π and π–π interactions is the creation of stacks of molecules running down the *c* axis (Fig. 3).

Finally, acting between the stacks of molecules just described are C—H···O and C—H···Cl interactions (Table 2 and Fig. 4).
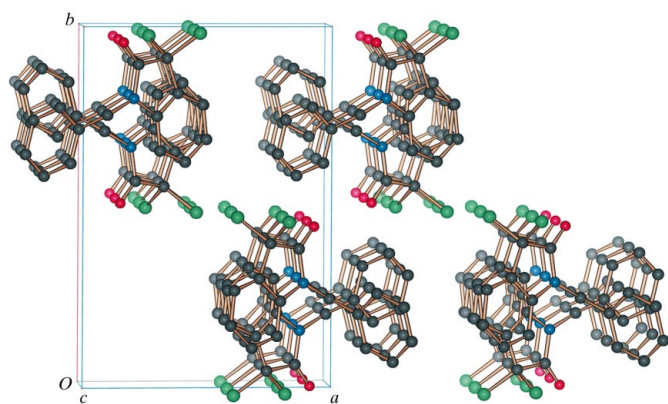
Figure 3
The crystal packing in (II), showing the stacks of molecules running down the *c* axis.
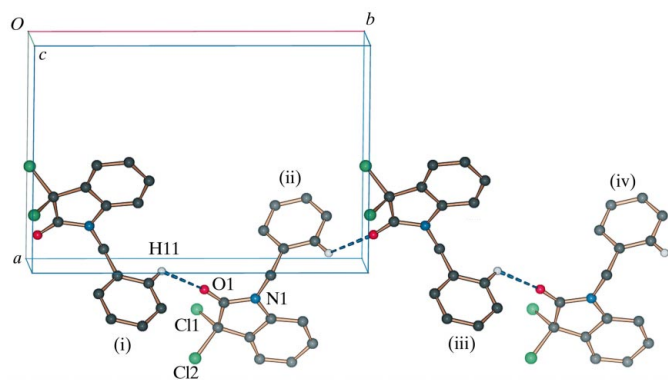
Figure 4
C—H···O interactions between molecules related by the screw symmetry running down the *b* axis. These interactions act between the stacks shown in Fig. 2. Molecules (i), (ii), (iii) and (iv) are at the symmetry positions (x, y, z), ($2 - x, \frac{1}{2} + y, \frac{1}{2} - z$), ($x, 1 + y, z$) and ($2 - x, \frac{3}{2} + y, \frac{1}{2} - z$), respectively.

Experimental

The dione (III) (6.40 g, 27.00 mmol) was dissolved in C_6H_6 (70 ml) in a round-bottomed flask and cooled to 273 K. PCl_5 (12.80 g, 62.10 mmol, 2.3 equivalents) was added and the solution was warmed to 298 K for 24 h. The solvent was removed *in vacuo* to obtain a yellow–brown residue, which was further purified by column chromatography (5% EtOAc/hexane) to afford (II) as a light-yellow oil. On addition of EtOH, (II) precipitated as a colourless solid [yield 6.37 g, 81%; m.p. 398–399 K (literature m.p. 399–400 K; Palazzo & Rosnati, 1953)]. ^{1}H NMR (300 MHz, CDCl$_3$, Me$_4$Si): δ_H 4.94 (2H, *s*, PhCH$_2$N), 6.72 (1H, *d*, *J* = 7.9 Hz, ArH), 7.14 (1H, *t*, *J* = 7.6 Hz, ArH), 7.22–7.39 (6H, *m*, 6 × ArH) and 7.64 (1H, *d*, *J* = 7.5 Hz, ArH). ^{13}C NMR (75 MHz, CDCl$_3$): δ_C 44.5 (PhCH$_2$N), 110.1 (ArCCl$_2$), 124.2 (CH), 124.9 (CH), 127.1 (2 × CH), 128.1 (CH), 129.0 (2 × CH), 129.3 (C), 131.8 (CH), 134.4 (C), 139.8 (C) and 169.2 (C═O) (one CH not observed in spectrum).

Crystal data

$C_{15}H_{11}Cl_2NO$	$Z = 4$
$M_r = 292.15$	$D_x = 1.453$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.4847$ (13) Å	$\mu = 0.48$ mm^{-1}
$b = 14.4641$ (18) Å	$T = 173$ (2) K
$c = 9.2203$ (11) Å	Irregular, colourless
$\beta = 107.244$ (2)°	$0.48 \times 0.30 \times 0.26$ mm
$V = 1335.4$ (3) Å^3	

Bruker SMART CCD area-detector diffractometer	3213 independent reflections
	2603 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.028$
8835 measured reflections	$\theta_{max} = 28.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.032$	$+ 0.2606P]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
3213 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—O	1.2107 (17)	C2—C3	1.4945 (19)
C1—N	1.3637 (18)	C3—C8	1.3966 (18)
C1—C2	1.5555 (19)	C8—N	1.4109 (17)
N—C1—C2	106.23 (11)	C3—C8—N	110.30 (11)
C3—C2—C1	103.98 (11)	C1—N—C8	111.85 (11)
C8—C3—C2	107.49 (11)		

Table 2
Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C10–C15 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C11—H11$\cdots$O^i	0.95	2.64	3.325 (2)	129
C9—H9$B\cdots Cg^{ii}$	0.99	2.93	3.660 (2)	132
C6—H6$\cdots$Cl2^{iii}	0.95	2.98	3.4461 (15)	112
C13—H13$\cdots$Cl1^{iv}	0.95	3.01	3.4815 (18)	112

Symmetry codes: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y - \frac{1}{2}, z - \frac{3}{2}$; (iii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iv) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$.

Table 3
Comparison of selected bond lengths and angles (Å, °) for compound (II), KUNMUB and QASXEO.

Structure/CSD refcode	N—C1	C8—N—C1	N—C1—C2
(II)	1.364 (2)	111.8 (1)	106.2 (1)
KUNMUB^a	1.339 (5)	112.8 (3)	106.1 (3)
QASXEO^b	1.37	114	105

Notes: (a) Zukerman-Schpector et $al.$ (1993); (b) Meketa et $al.$ (2005).

H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with C—H bond lengths of 0.95

(aromatic CH) or 0.99 Å (CH$_2$) and isotropic displacement parameters equal to 1.2 times U_{eq} of the parent atom.

Data collection: $SMART$-NT (Bruker, 1998); cell refinement: $SAINT$-$Plus$ (Bruker, 1999); data reduction: $SAINT$-$Plus$; program(s) used to solve structure: $SHELXTL$ (Bruker, 1999); program(s) used to refine structure: $SHELXTL$; molecular graphics: $ORTEP$-3 (Farrugia, 1997) and $SCHAKAL99$ (Keller, 1999); software used to prepare material for publication: $PLATON$ (Spek, 2003) and $SHELXTL$.

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