organic papers

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Key indicators

Single-crystal X-ray study
$T = 292$ K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.129
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

2-(5-Benzyloxy-2,4-dichlorophenyl)-4,5,6,7-tetrahydroisoindole-1,3-dione

The crystal structure of the title compound, $C_{21}H_{17}Cl_2NO_3$, shows that there are no intra- or intermolecular π–π stacking interactions. The structure is stabilized by C—H$\cdots$O and C—H$\cdots$Cl hydrogen bonds, which form a nine-membered ring.

Comment

It has been found that some cyclic imides show strong herbicidal activity at small doses against a wide variety of weeds including Gramineae weeds, Cyperaceae weeds and broadleaved weeds in upland fields, as well as weeds in paddy fields, and these compounds do not produce any material phytotoxicity on agricultural crops such as corn, soybean, wheat, peanut and rice plants (Nagano *et al.*, 1982). We are interested in exploring and developing novel strategies for synthesizing cyclic imides. The title compound, (I), was synthesized directly from the hydroxy analog.


(I)
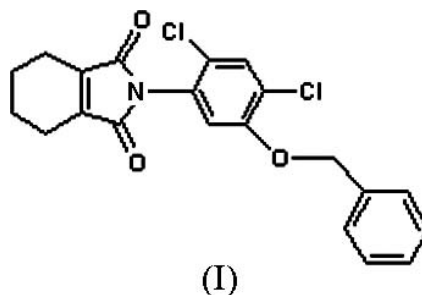
In the molecule (Fig. 1), the dihedral angle between the phenyl (C1–C6) and the benzene (C8–C13) rings is 101.6 (1)°. The dihedral angle between the phenyl ring and the five-membered heterocyclic ring (C14–C16/C21/N1) is 37.9 (1)°. The dihedral angle between the benzene and heterocyclic rings is 85.6 (1)°. There are intermolecular C—H$\cdots$O and C—H$\cdots$Cl hydrogen bonds (Table 1), which form a nine-membered ring, as shown in Fig. 2.

Experimental

5-Benzyloxy-2,4-dichlorophenylamine (0.14 g) and 3,4,5,6-tetra-hydrophthalic anhydride (0.28 g) were dissolved in acetic acid (10 ml) and refluxed for 4 h. The resultant mixture was allowed to cool to room temperature and poured into water, followed by extraction with diethyl ether. This extract was washed with water, dried over anhydrous sodium sulfate and subjected to filtration. The filtrate was concentrated under reduced pressure and the residue was purified by silica gel chromatography to obtain (I) (0.30 g). Single crystals suitable for X-ray diffraction were grown from an ethanol solution at 277 K.
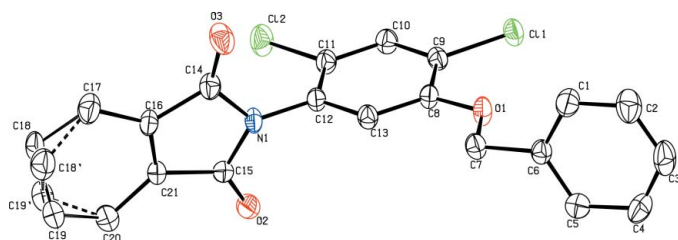
Figure 1
A view of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Dashed lines denote the minor component of the disorder.

Crystal data

$C_{21}H_{17}Cl_2NO_3$
$M_r = 402.26$
Monoclinic, $P2_1/c$
$a = 12.1705 (17)$ Å
$b = 11.0375 (15)$ Å
$c = 15.078 (2)$ Å
$\beta = 107.232 (3)°$
$V = 1934.6 (5)$ Å^3
$Z = 4$

$D_x = 1.381$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1743
    reflections
$\theta = 2.3–20.4°$
$\mu = 0.36$ mm^{-1}
$T = 292 (2)$ K
Plate, colorless
$0.30 \times 0.20 \times 0.08$ mm

Data collection

Bruker SMART CCD area-detector
    diffractometer
φ and ω scans
Absorption correction: none
9530 measured reflections
3386 independent reflections

2169 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 25.0°$
$h = -14 \rightarrow 9$
$k = -10 \rightarrow 13$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.129$
$S = 1.05$
3386 reflections
263 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.1448P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C20—H20A···Cl1^i	0.97	2.81	3.745 (4)	163
C10—H10···O2^{ii}	0.93	2.51	3.393 (4)	159

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

H atoms were placed at calculated positions and treated as riding atoms, with C—H = 0.93 or 0.97 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$. Atoms C18 and C19 were described by two disorder components each, with separately refined displacement parameters. The site-occupancy factors of the disordered atoms refined to 0.571 (15) and 0.429 (15).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine
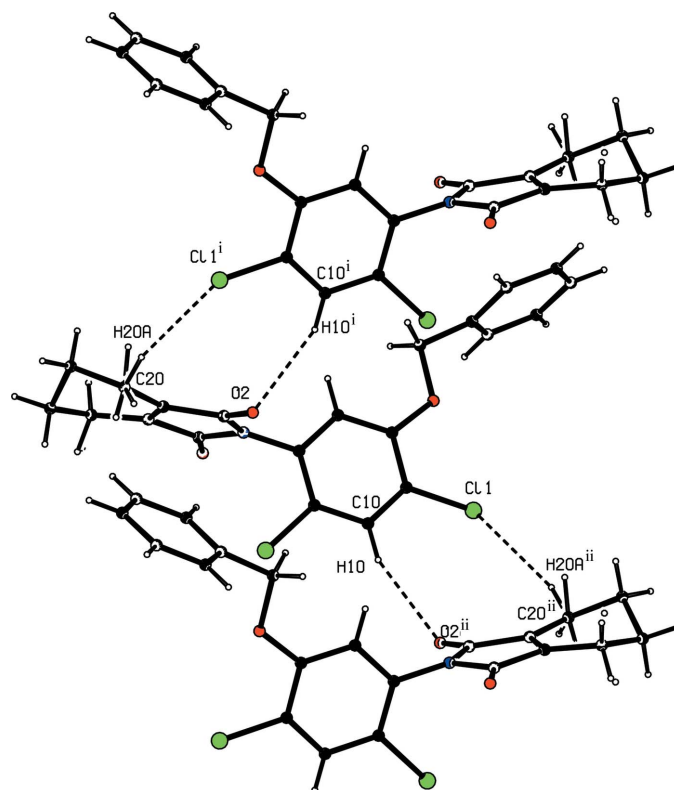
Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$].

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

References

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