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# 2-(5-Benzyloxy-2,4-dichlorophenyl)perhydroisoindole-1,3-dione monohydrate

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**Key indicators**

Single-crystal X-ray study
*T* = 292 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.053
*wR* factor = 0.135
Data-to-parameter ratio = 16.8

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

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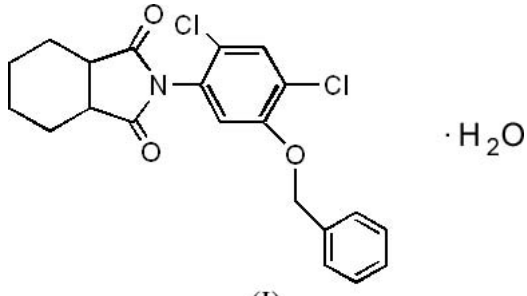
The crystal structure of the title compound, $C_{21}H_{19}Cl_2NO_3\cdot H_2O$, shows that there are no intra- or intermolecular $\pi-\pi$ stacking interactions. The structure is stabilized by O—H···O hydrogen bonds involving the carbonyl group and the solvent water molecule.

## Comment

It has been found that some cyclic imides show strong herbicidal activity at small doses against a wide variety of weeds, including Granineae weeds, Cyperaceae weeds and broad-leaved weeds, and they do not produce any material phytotoxicity in various agricultural crops (Nagano *et al.*, 1982). Thus, we are interested in exploring and developing novel strategies for synthesizing cyclic imides. The title compound, (I), was synthesized directly from the hydroxy analogue.




(I)

In the molecule of (I), the dihedral angle between the benzene ring (C9–C14) and the phenyl ring (C16–C21) is 62.2 (2)°. The six-membered ring (C1–C6) adopts a boat conformation (Fig. 1). The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence C1–C6 are $Q$ = 0.704 (3) Å, $\varphi_2$ = 185.7 (3)° and $\theta_2$ = 90.9 (3)°. The five-

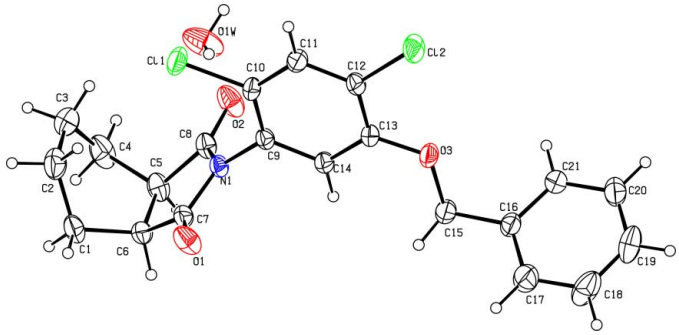



**Figure 1**
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

membered heterocyclic ring (N1/C5–C8) adopts an almost planar conformation.

The crystal structure of (I) shows that there are no intra- or intermolecular $\pi$–$\pi$ stacking interactions. As shown in Fig. 2, the structure is stabilized by O—H···O hydrogen bonds involving the carbonyl group and the solvent water molecule (Table 1).

## Experimental

To a solution of 2-(2,4-dichloro-5-hydroxyphenyl)hexahydroisoindole-1,3-dione (1 g) in dimethylformamide (10 ml), anhydrous potassium carbonate (0.44 g) and then chloromethylbenzene (0.44 g) were added. The resultant mixture was stirred at 343–353 K for 3 h. Water (20 ml) was then added, followed by extraction with diethyl ether. The ether layer was washed with water, dried and concentrated. The residue was purified by silica-gel chromatography to obtain 2-(5-benzyloxy-2,4-dichlorophenyl)hexahydroisoindole-1,3-dione monohydrate (yield 0.96 g, 75%). Single crystals of (I) suitable for X-ray diffraction were grown from an ethanol solution at 277 K.

*Crystal data*

| | |
|---|---|
| $C_{21}H_{19}Cl_2NO_3\cdot H_2O$ | $D_x$ = 1.391 Mg m$^{-3}$ |
| $M_r$ = 422.29 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 2919 reflections |
| $a$ = 9.2694 (10) Å | $\theta$ = 2.2–27.4° |
| $b$ = 9.3192 (10) Å | $\mu$ = 0.35 mm$^{-1}$ |
| $c$ = 23.448 (3) Å | $T$ = 292 (2) K |
| $\beta$ = 95.234 (2)° | Thick plate, colourless |
| $V$ = 2017.1 (4) Å$^3$ | 0.30 × 0.20 × 0.10 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | $R_{int}$ = 0.031 |
| $\varphi$ and $\omega$ scans | $\theta_{max}$ = 27.0° |
| 11507 measured reflections | $h = -11 \rightarrow 11$ |
| 4395 independent reflections | $k = -11 \rightarrow 9$ |
| 3383 reflections with $I > 2\sigma(I)$ | $l = -29 \rightarrow 29$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.592P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.053 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $wR(F^2)$ = 0.135 | $\Delta\rho_{max}$ = 0.28 e Å$^{-3}$ |
| $S$ = 1.07 | $\Delta\rho_{min}$ = −0.23 e Å$^{-3}$ |
| 4395 reflections | |
| 261 parameters | |
| H atoms treated by a mixture of independent and constrained refinement | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$WA$···O1$^{i}$ | 0.84 (1) | 2.06 (1) | 2.892 (3) | 170 (4) |
| O1$W$—H1$WB$···O2 | 0.84 (1) | 2.03 (1) | 2.852 (3) | 166 (4) |

Symmetry code: (i) $x, y+1, z$.

The water H atoms were located and refined with distance restraints O—H = 0.84 (1) Å. H atoms attached to C atoms were placed in calculated positions and treated as riding atoms, with C—H = 0.93–0.98 Å and with $U_{iso}$(H) = 1.2$U_{eq}$(C).

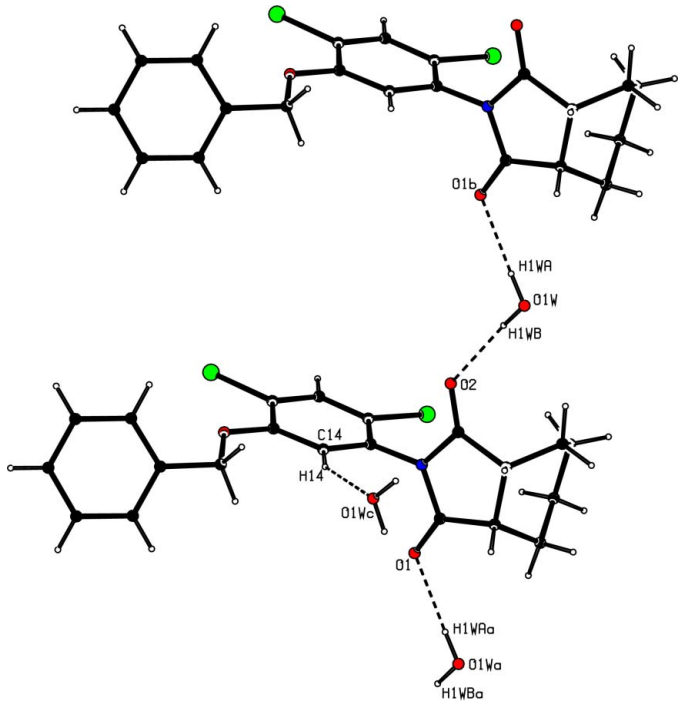



**Figure 2**
The hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (*a*) $x, y-1, z$; (*b*) $x, y+1, z$; (*c*) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$.]

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

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## References

Bruker (1997). *SMART*. Version 5.054. Bruker AXS Inc., Madison,Wisconsin, USA.
Bruker (1999). *SAINT*. Version 6.01. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Nagano, E., Hashimo, S., Yoshida, R., Matsumoto, H., Oshio, H. & Kamo, S. (1982). *EP Patent No.* 0061741.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.