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3-Benzyl-4,5,6,7-tetrachloro-3-hydroxy-2-(3-hydroxypropyl)isoindolin-1-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.084
Data-to-parameter ratio = 22.9

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

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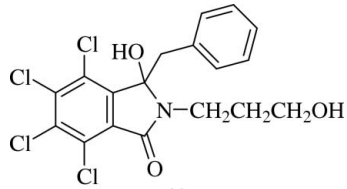
In the title compound, $C_{18}H_{15}Cl_4NO_3$, the isoindole fragment is slightly twisted and exhibits a propeller-like conformation. The dihedral angle between the isoindoline mean plane and phenyl ring is 59.47 (7)°. Intermolecular O—H···O hydrogen bonds link the molecules into chains running along the *b* axis. The crystal packing is further stabilized by weak C—H···O interactions.

Comment

Photoinduced electron-transfer reaction of phthalimides with alkenes has been an active research area in organic photochemistry (Griesbeck *et al.*, 1996; Kanaoka, 1978). In a continuation of our recent work on photoinduced reactions of 4,5,6,7-tetrachlorophthalimide with alkenes (Xue *et al.*, 2000), we investigated the photoreaction of 4,5,6,7-tetrachloro-*N*-(2-hydroxyethyl)phthalimide with 1-phenylcyclohexene, in which the title compound, (I), was obtained as one of the unexpected products.



(I)

In (I) (Fig. 1), all bond lengths and angles are in normal ranges (Allen *et al.*, 1987). The isoindoline fragment (C1–C8/N1) is slightly twisted and exhibits a propeller-like conformation, with the largest deviation from the mean plane (*M*) of 0.063 (1) Å for atom N3. The dihedral angle between *M* and phenyl ring C10–C15 is 59.47 (7)°. The hydroxypropyl (C16–C18/O3) substituent is (−)-anticlinally attached at atom N1, with a C8—N1—C16—C17 torsion angle of −112.09 (14)°, while the benzyl (C9–C15) group is (−)-synclinally attached at atom C8, with a C8—C9—C10—C11 torsion angle of −88.07 (15).

Intermolecular O—H···O hydrogen bonds (Table 1) link the molecules into chains running along the *b* axis. The crystal packing (Fig. 2) is further stabilized by weak C—H···O interactions (Table 1).

Experimental

The title compound, (I), was synthesized by a photoinduced reaction between 4,5,6,7-tetrachloro-*N*-(2-hydroxyethyl)phthalimide (2 mmol) and an excess amount of 1-phenylcyclohexene (10 mmol)

in a benzene solution (80 ml). It was isolated, as a minor product, using silica-gel column chromatography. Colourless block-shaped single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a petroleum ether/chloroform solution (1:1 *v*/*v*) (m.p. 488–489 K).

Crystal data

$C_{18}H_{15}Cl_4NO_3$
M_r = 435.11
Monoclinic, $P2_1/c$
a = 10.8345 (2) Å
b = 9.9173 (2) Å
c = 18.7629 (3) Å
β = 113.983 (1)°
V = 1842.00 (6) Å^3
Z = 4
D_x = 1.569 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 100.0 (1) K
Block, colourless
0.37 × 0.31 × 0.28 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.792, T_{max} = 0.835
36202 measured reflections
5374 independent reflections
4723 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 30.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.084
S = 1.06
5374 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.9751P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.82	1.94	2.7569 (15)	175
O3—H3A···O1	0.82	2.20	2.9284 (15)	149
C9—H9A···O3^{ii}	0.97	2.51	3.4113 (17)	155
C16—H16B···O3^{ii}	0.97	2.56	3.511 (2)	168

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

Hydroxyl H atoms were located in a difference map and refined as riding, with O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O). The remaining H-atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aromatic and 0.97 Å for CH_2 H atoms, and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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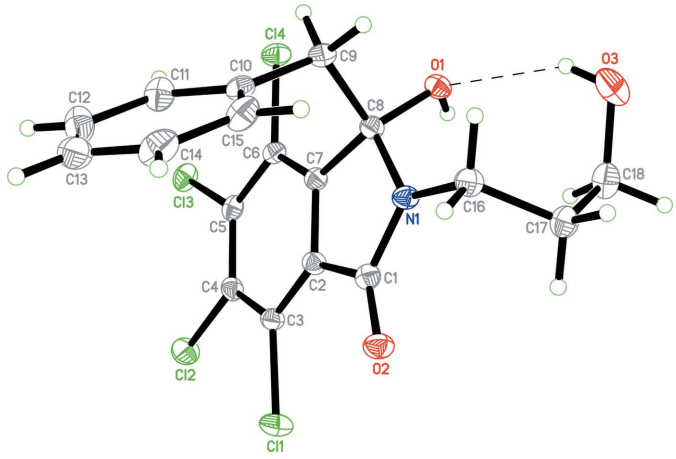

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed line indicates a hydrogen bond.

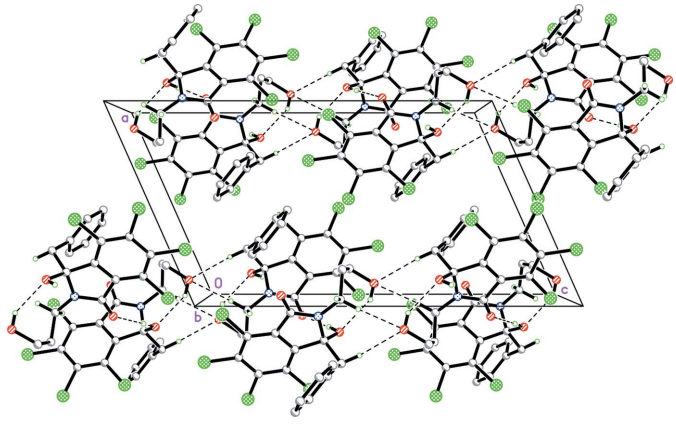

Figure 2
The crystal packing, viewed down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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