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9,10,11,12-Tetrachloro-12b-hydroxy-12c-phenyl-1,2,3,3a,5,6,12b,12c-octahydro-8*H*-cyclopenta[6,7][1,4]oxazepino[5,4-*a*]isoindol-8-one

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Key indicators

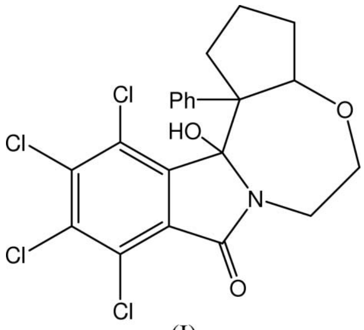
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.007 Å
R factor = 0.070
wR factor = 0.223
Data-to-parameter ratio = 18.2

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

The medium-sized heterocyclic title compound, $C_{21}H_{17}Cl_4NO_3$, crystallizes with two crystallographically independent molecules in the asymmetric unit. The cyclopentane rings adopt half-chair conformations. Symmetry-related molecules are linked *via* O—H···O hydrogen bonds to form dimers, which are arranged into one-dimensional chains along the [100] direction. C—H···π and π–π interactions were also observed in the crystal structure.

Comment

The construction of medium and large heterocyclic ring systems is an important task in organic synthesis (Evans & Holmes, 1991; Griesbeck *et al.*, 2001; Illuminati & Mandolini, 1981). In a continuation of our recent work on photoinduced cyclization reactions (Xue *et al.*, 2000), we prepared the title compound, (I), by the photochemical reaction of *N*-(2-hydroxyethyl)-4,5,6,7-tetrachlorophthalimide with 1-phenylcyclopentane and we report its structure here.



(I)

The asymmetric unit of (I) contains two molecules, *A* and *B*, that differ little in their conformations (Fig. 1). Their bond lengths and angles are similar and show normal values (Allen *et al.*, 1987) that are comparable to related structures (Xue *et al.*, 2000). In both molecules, the cyclopentane rings adopt half-chair conformations, with puckering parameters (Cremer & Pople 1975) Q = 0.426 (6) Å and φ = 95.9 (7)° in molecule *A* [Q = 0.449 (5) Å and φ = 141.8 (7)° in molecule *B*]. The conformation of the seven-membered ring is twist-chair. The dihedral angles between the C16–C21 benzene rings and the mean planes of the C1–C8/N1 units are 31.9 (2)° in molecule *A* and 39.6 (2)° in molecule *B*.

The crystal packing of (I) (Fig. 2) shows that adjacent molecules are linked into dimers by O3*A*—H3*AA*···O2*B* hydrogen bonds (Table 1) and these dimers are arranged into one-dimensional chains along the [100] direction. The molecular structure is further stabilized by C—H···π interactions (Table 1). π–π Interactions are also observed with centroid–

centroid distances of $Cg1\cdots Cg5$ = 3.444 (3) Å, $Cg3\cdots Cg5$ = 3.594 (3) Å, $Cg2\cdots Cg6$ = 3.839 (3) Å and $Cg4\cdots Cg6$ = 3.725 (3) Å, where *Cg*1 is the centroid of the C1*A*/C2*A*/C7*A*/C8*A*/N1*A* ring, *Cg*2 that of C1*B*/C2*B*/C7*B*/C8*B*/N1*B*; *Cg*3, *Cg*4, *Cg*5 and *Cg*6 those of the C2*A*–C7*A*, C2*B*–C7*B*, C16*A*–C21*A* and C16*B*–C21*B* benzene rings, respectively.

Experimental

Compound (I) was synthesized by a photoinduced reaction between *N*-(2-hydroxyethyl)-4,5,6,7-tetrachlorophthalimide (3 mmol) and an excess of 1-phenylcyclopentene (15 mmol) in a benzene solution (120 ml), using a medium-pressure mercury lamp in a glass cooling water jacket as the light source. It was isolated by column chromatography on silica gel using petroleum ether and ethyl acetate as eluants. Colorless block-shaped single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the solvents from a petroleum ether/chloroform solution (1:1 *v*/*v*), m.p. 538–540 K.

Crystal data

$C_{21}H_{17}Cl_4NO_3$
M_r = 473.16
Triclinic, $P\overline{1}$
a = 9.0183 (4) Å
b = 12.5563 (5) Å
c = 18.3224 (7) Å
α = 75.391 (2)°
β = 88.559 (2)°
γ = 88.083 (2)°
V = 2006.26 (14) $Å^3$
Z = 4
D_x = 1.566 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 100.0 (1) K
Block, colorless
0.59 × 0.47 × 0.24 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.712, T_{max} = 0.867
29592 measured reflections
9537 independent reflections
8288 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.223
S = 1.15
9537 reflections
523 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2 + 15.8392P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.44 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.68 e $Å^{-3}$

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O3A-H3AA\cdots O2B$	0.82	1.97	2.763 (5)	162
$O3B-H3BA\cdots O2A^{i}$	0.82	1.94	2.751 (5)	173
$C9A-H9AA\cdots O2A$	0.97	2.45	2.852 (5)	104
$C9B-H9BA\cdots O2B$	0.97	2.48	2.870 (5)	104
$C9B-H9BB\cdots O3B$	0.97	2.52	2.875 (6)	102
$C9B-H9BB\cdots O1B^{ii}$	0.97	2.59	3.357 (5)	136
$C10A-H10A\cdots O1A^{iii}$	0.97	2.59	3.096 (6)	113
$C14A-H14A\cdots Cl1A$	0.97	2.68	3.292 (5)	121
$C14A-H14B\cdots O3A$	0.97	2.37	2.796 (6)	106
$C14B-H14D\cdots O3B$	0.97	2.32	2.675 (6)	101
$C9A-H9AA\cdots Cg4^{iv}$	0.97	3.25	3.938 (5)	130

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $x+1, y, z$.

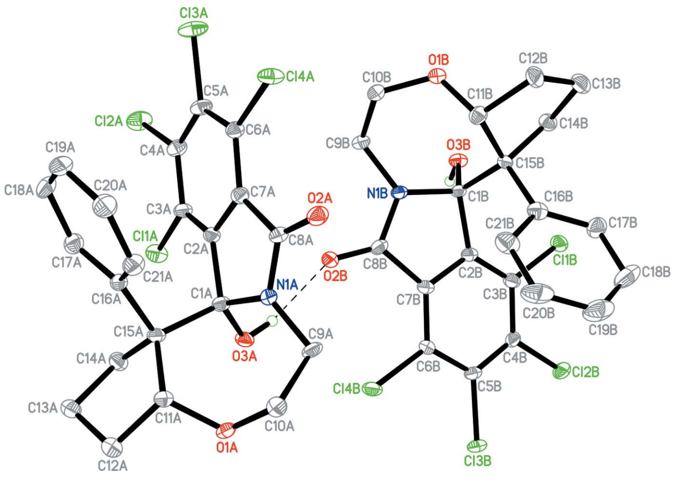


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. For clarity, only hydroxyl H atoms are shown, with the hydrogen bond drawn as a dashed line.

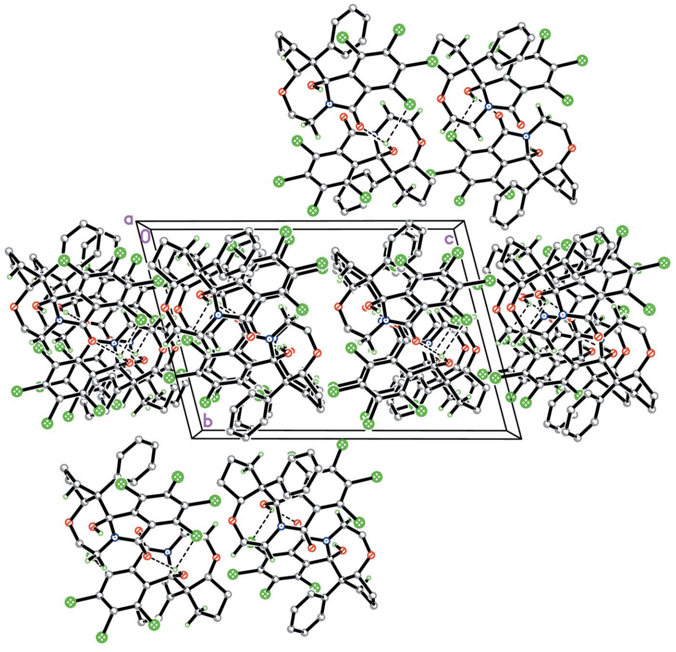


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are drawn as dashed lines. H atoms not involved in these interactions have been omitted.

All H-atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å for aromatic, 0.98 Å for CH and 0.97 Å for CH_2, and O—H = 0.82 Å; $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$. The highest residual electron density peak is located 0.92 Å from Cl1*A*.

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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