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7,8,9,10-Tetrachloro-10b-(2-hydroxy-1-phenylethyl)-3,4-dihydro-2*H*-1,3-oxazino[2,3-*a*]isoindol-6(10b*H*)-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.159
Data-to-parameter ratio = 21.9

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

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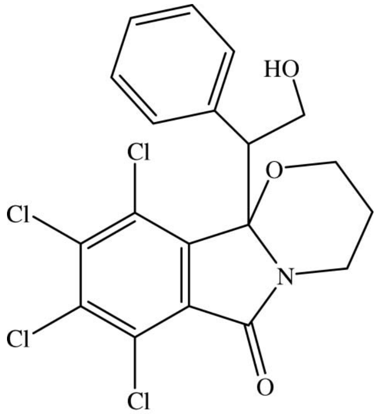
In the title compound, $C_{19}H_{15}Cl_4NO_3$, the isoindole unit is planar and the oxazino ring adopts a twist-boat conformation. The dihedral angle between the isoindole unit and the phenyl ring is 50.46 (13)°. The molecules are linked *via* intermolecular O—H⋯O hydrogen bonds and weak C—H⋯O and C—H⋯Cl interactions, forming a three-dimensional network.

Comment

The construction of medium and large heterocyclic ring systems is an important task in organic synthesis (Evans & Holmes, 1991; Griesbeck *et al.*, 1996; Illuminati & Mandolini, 1981). As a continuation of our recent work on photo-induced cyclization reactions (Xue *et al.*, 2000; Fun, Chantrapromma *et al.*, 2007; Fun, Shen *et al.*, 2007), the title compound, (I), was obtained as one of the products of the reaction of photo-excited *N*-substituted 4,5,6,7-tetrachloro-*N*-(2-hydroxyethyl)phthalimide (TCP) with styrene and its structure is reported here (Fig. 1).



(I)

In (I), the isoindole unit (atoms C1–C8/N1) is essentially planar, with a maximum deviation of 0.065 (3) Å for N1. The oxazino ring (C8/O1/C9–C11/N1) adopts a twist-boat conformation, with Cremer & Pople (1975) puckering parameters Q = 0.660 (3) Å, θ = 99.8 (3) and φ = 131.9 (3)°. The 2-hydroxy-1-phenylethyl substituent and the oxazino ring lie on opposite sides of the isoindole plane. The mean plane of the hydroxyethyl unit (C12/C19/O3) makes a dihedral angle of 61.7 (2)° with the five-membered isoindole ring. The dihedral angle between the phenyl and isoindole rings is 50.46 (13)°. All bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

The crystal packing (Fig. 2) shows intermolecular O—H⋯O hydrogen bonds and weak C—H⋯O and C—H⋯Cl

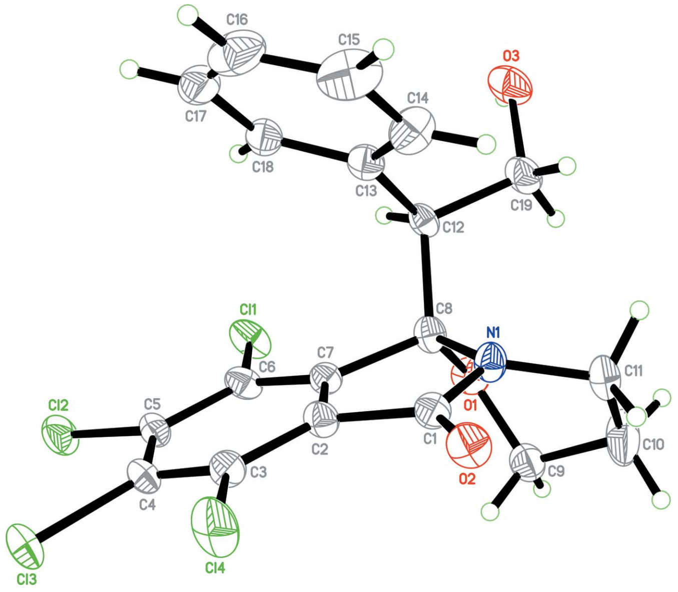


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

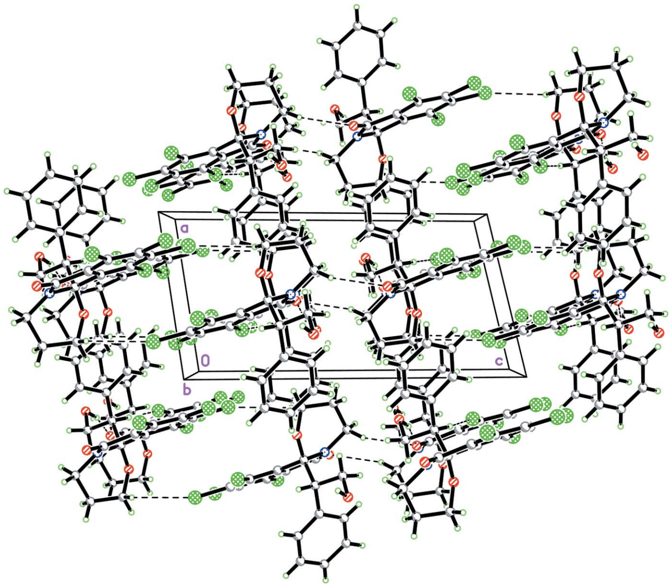


Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

interactions, linking the molecules into a three-dimensional network.

Experimental

The title compound, (I), was synthesized by a photo-induced reaction between 4,5,6,7-tetrachloro-*N*-(2-hydroxyethyl)phthalimide (0.025 *M*) and an excess of styrene in benzene (120 ml). Compound (I) was isolated by silica-gel column chromatography using petroleum ether–ethyl acetate as eluents via gradient elution. Single crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of a chloroform–petroleum ether (1:4 *v*/*v*) solution (m.p. 472–474 K).

Crystal data

$C_{19}H_{15}Cl_4NO_3$
$M_r = 447.12$
Triclinic, $P\overline{1}$
$a = 8.3221\ (3)$ Å
$b = 8.7849\ (2)$ Å
$c = 14.9376\ (4)$ Å
$\alpha = 100.189\ (2)^\circ$
$\beta = 95.084\ (2)^\circ$
$\gamma = 117.496\ (2)^\circ$
$V = 935.12\ (5)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.65$ mm^{-1}
$T = 100.0\ (1)$ K
0.25 × 0.20 × 0.11 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min} = 0.852$, $T_{\max} = 0.931$
17582 measured reflections
5427 independent reflections
3846 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.159$
$S = 1.07$
5427 reflections
248 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 1.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1*O*3···O2$^{\rm i}$	0.88 (5)	2.11 (6)	2.955 (3)	162 (4)
C11—H11*A*···O2	0.97	2.51	2.890 (3)	103
C11—H11*B*···O2$^{\rm ii}$	0.97	2.49	3.276 (3)	139
C12—H12*A*···Cl1	0.98	2.78	3.465 (2)	127
C12—H12*A*···Cl4$^{\rm i}$	0.98	2.68	3.612 (3)	158
C19—H19*B*···O1	0.97	2.49	2.900 (4)	105

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

The hydroxy H atom was located in a difference map and refined isotropically. The remaining 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$_2$ groups, and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) or $1.5U_{\rm eq}$(O). The highest residual electron density peak is located 1.46 Å from Cl1.

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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