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4,5,6,7-Tetrachloro-2-(2-hydroxyphenyl)-1*H*-isoindole-1,3(2*H*)-dione

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The title compound, $C_{14}H_5Cl_4NO_3$, crystallizes with two crystallographically independent molecules in the asymmetric unit. Each independent molecule exists as part of an O/C—H···O hydrogen-bonded centrosymmetric dimer. The crystal packing is further stabilized by intermolecular C—H···Cl and π–π interactions.

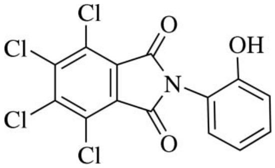
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.067
Data-to-parameter ratio = 20.8

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

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 cyclizations *via* the photoinduced electron transfer reactions of ω-hydroxyalkylphthalimides with alkenes (Xue *et al.*, 2000), the title compound, (I), was prepared as a reagent. We report here the crystal structure of (I).



(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). In both molecules, the isoindole unit (atoms N1/C1–C8) is planar, with maximum deviations of 0.027 (1) and 0.050 (1) Å for atoms C1*A* and N1*B*, respectively. The dihedral angle between the isoindole unit and the hydroxyphenyl ring is 63.78 (5)° in molecule *A* and 60.86 (5)° in *B*.

The crystal packing of (I) (Fig. 2) shows that each independent molecule exists as part of an O—H···O hydrogen-bonded centrosymmetric $R_2^2(14)$ dimer (Bernstein *et al.*, 1995); these dimers are further strengthened by weak C—H···O interactions (Table 1). The *A*- and *B*-type dimers are cross-linked *via* C—H···Cl interactions. π–π Interactions are also observed, with centroid–centroid distances of $Cg1\cdots Cg1^{iv}$ = 3.6547 (7) Å and $Cg2\cdots Cg2^{v}$ = 3.6355 (7) Å, where *Cg*1 and *Cg*2 are the centroids of the C2*A*–C7*A* and C2*B*–C7*B* benzene rings, respectively [symmetry codes: (iv) $1 - x, 2 - y, 1 - z$; (v) $2 - x, 1 - y, 1 - z$].

Experimental

Compound (I) was synthesized by the reaction of 4,5,6,7-tetrachloroisobenzofuran-1,3-dione (0.5 mol) with 2-aminophenol

(0.6 mol) in refluxing acetic acid (100 ml). Yellow block-shaped single crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of an acetone solution (m.p. 563–565 K).

Crystal data

$C_{14}H_5Cl_4NO_3$	$Z = 8$
$M_r = 376.99$	$D_x = 1.774$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.2779$ (2) Å	$\mu = 0.85$ mm^{-1}
$b = 14.0350$ (2) Å	$T = 100.0$ (1) K
$c = 19.9624$ (3) Å	Block, yellow
$\beta = 124.834$ (1)°	0.50 × 0.43 × 0.39 mm
$V = 2823.53$ (8) Å^3	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	77565 measured reflections
ω scans	8247 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	7498 reflections with $I > 2\sigma(I)$
$T_{min} = 0.675$, $T_{max} = 0.734$	$R_{int} = 0.031$
	$\theta_{max} = 30.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 1.7606P]$
$R[F^2 > 2\sigma(F^2)] = 0.026$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.48$ e Å^{-3}
8247 reflections	$\Delta\rho_{min} = -0.23$ e Å^{-3}
397 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O2A—H2AA···O3A^{i}	0.82	2.01	2.8259 (16)	172
O2B—H2BA···O3B^{ii}	0.82	2.09	2.8959 (16)	168
C11A—H11A···O3A^{i}	0.93	2.59	3.2588 (16)	129
C11B—H11B···O3B^{ii}	0.93	2.44	3.1711 (16)	136
C12B—H12B···Cl2A^{iii}	0.93	2.82	3.5240 (16)	134

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

O-bound H atoms were located in a difference map, idealized and then refined as riding, with O—H = 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$. C-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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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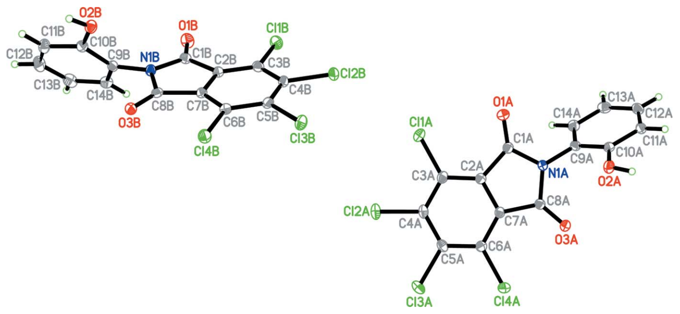

Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

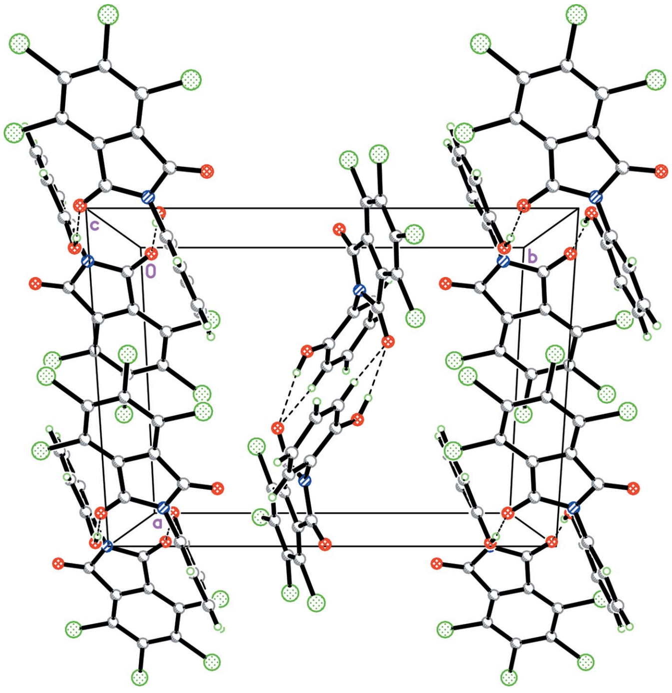

Figure 2
The crystal packing of (I), viewed down the *c* axis, showing hydrogen-bonded (dashed lines) dimers.

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