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4,5,7-Trichloro-6-(2,4-dimethylthiazol-5-yl)-2-(3-hydroxypropyl)isoindole-1,3-dione

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Key indicators

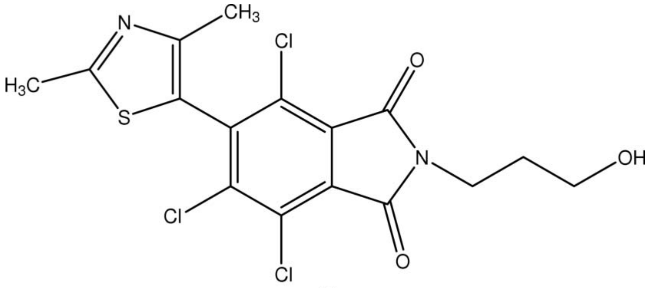
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.101
Data-to-parameter ratio = 16.7

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

In the title compound, $C_{16}H_{13}Cl_3N_2O_3S$, the 1,3-thiazole ring is almost orthogonal to the isoindoline plane, making a dihedral angle of 89.74 (9)°. Intermolecular O—H···N and C—H···O interactions and S···O short contacts link the molecules into a three-dimensional framework. The crystal structure is further stabilized by weak intermolecular C—H···π interactions.

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 reactions of 4,5,6,7-tetrachlorophthalimide with alkenes (Xue *et al.*, 2000), the title compound, (I), was obtained by the reaction of photo-excited *N*-substituted 4,5,6,7-tetrachloro-*N*-(2-hydroxyethyl)phthalimide (TCP) with 2,4-dimethylthiazole and its structure is reported here, (I) (Fig. 1).



(I)

Bond lengths and angles in (I) are comparable with those observed in a related structure (Liu *et al.*, 2005) and display normal values (Allen *et al.*, 1987). The 1,3-thiazole (S1/C1/N1/C2–C3) ring at C4 is twisted away from the isoindoline plane (C4–C8/N2/C9–C11) [torsion angle C2═C3—C4—C11 = 90.2 (3)°], while the dihedral angle between the 1,3-thiazole (S1/C1/N1/C2-C3) ring and the isoindoline plane (C4–C8/N2/C9–C11) is 89.74 (9)°. The methyl groups at C1 and C2 are twisted slightly in opposite directions from the S1/C1/N1/C2–C3 ring plane, with out-of-plane distances for atoms C12 and C13 of −0.037 (3) and 0.018 (3) Å, respectively.

Intramolecular C14—H14*A*···O2 and C14—H14*A*···O3 interactions generate *S*(5) ring motifs (Bernstein *et al.*, 1995) (Table 1 and Fig. 1). In the crystal structure, molecules are interconnected by intermolecular O3—H3*A*···N1^{i}, C12—H12*B*···O3^{ii}, C13—H13*A*···O2^{iii} and C14—H14*B*···O1^{iv} interactions (Table 1) which, together with a short S1···O2($1 - x, 1 - y, 1 - z$) contact [3.165 (2) Å], generate a three-dimensional framework (Fig. 2). In addition, the crystal structure is further stabilized by C—H$\cdots\pi$ interactions involving the S1/C1/N1/C2–C3 ring at ($-x + 1$, $-y$, $-z + 1$) (Table 1).

Experimental

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

Crystal data

$C_{16}H_{13}Cl_3N_2O_3S$
$M_r = 419.69$
Triclinic, $P\bar{1}$
$a = 7.2134$ (3) Å
$b = 10.2368$ (5) Å
$c = 12.5457$ (5) Å
$\alpha = 101.869$ (3)°
$\beta = 98.002$ (3)°
$\gamma = 103.406$ (3)°
$V = 864.73$ (7) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.67$ mm^{-1}
$T = 100.0$ (1) K
0.30 × 0.16 × 0.07 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.838$, $T_{max} = 0.955$
11106 measured reflections
3884 independent reflections
3113 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.101$
$S = 1.06$
3884 reflections
232 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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*
C14—H14*A*$\cdots$O2	0.97	2.56	2.908 (3)	101
C14—H14*A*$\cdots$O3	0.97	2.54	2.938 (3)	105
O3—H3*A*$\cdots$N1^{i}	0.95 (4)	1.90 (4)	2.846 (3)	171 (4)
C12—H12*B*$\cdots$O3^{ii}	0.96	2.46	3.415 (4)	174
C13—H13*A*$\cdots$O2^{iii}	0.96	2.54	3.374 (3)	145
C14—H14*B*$\cdots$O1^{iv}	0.97	2.56	3.447 (3)	152
C12—H12*C*$\cdots$*Cg*1^{v}	0.96	2.67	3.616 (2)	167

Symmetry codes: (i) $x, y + 1, z + 1$; (ii) $x - 1, y - 1, z - 1$; (iii) $-x + 2, -y + 1, -z + 2$; (iv) $-x + 1, -y + 2, -z + 2$; (v) $-x + 1, -y, -z + 1$. *Cg*1 is the centroid of the S1/C1/N1/C2–C3 ring.

The O-bound H atom was located in a difference map and refined isotropically. The remaining H atoms were positional geometrically and refined as riding, with C—H distances in the range 0.95–0.97 Å. The U_{iso} values were set at $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

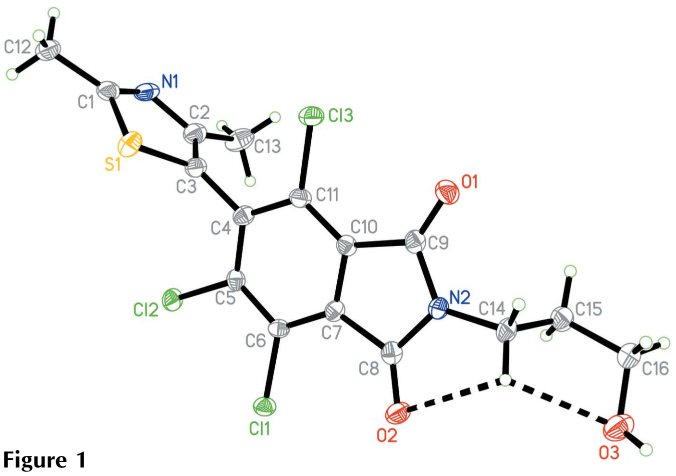


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed lines indicate intramolecular hydrogen bonds.

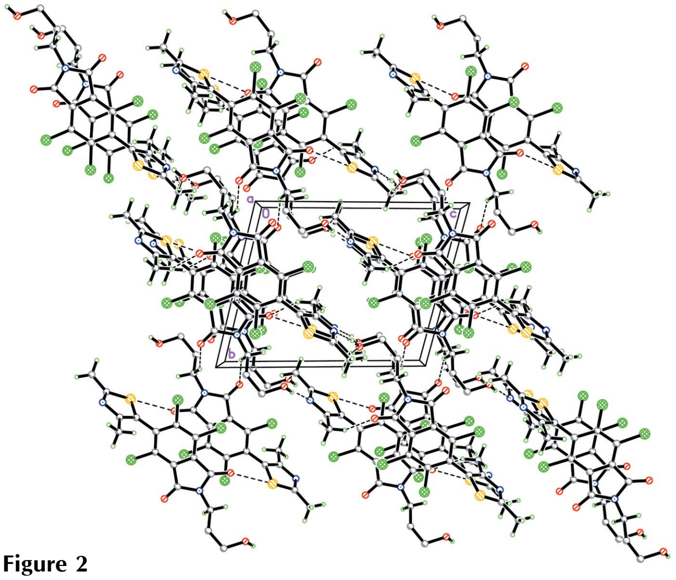


Figure 2
The crystal packing of (I), viewed down the *a* axis. Intermolecular O—H$\cdots$N and C—H$\cdots$O interactions and short S$\cdots$O contacts are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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