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9,10,11,12-Tetrachloro-12b-hydroxy-2,12c-dimethyl-3a,5,6,12c-tetrahydrothiazalo[4,5-c][1,4]-oxazepino[5,4-*a*]isoindol-8-one monohydrate

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Key indicators

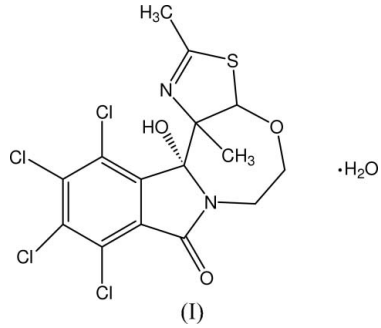
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.023
wR factor = 0.064
Data-to-parameter ratio = 24.8

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

The crystal structure of the title compound, $C_{15}H_{14}Cl_4N_2O_4S$, is stabilized by O—H···O, O—H···N, C—H···Cl and C—H···O interactions, together with short Cl···O and Cl···S contacts.

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). In a continuation of our recent work on photo-induced reactions of phthalimide with alkenes for medium and large ring construction (Xue *et al.*, 2000), we prepared the title compound, (I), by the reaction of photo-excited *N*-(2-hydroxyethyl)-4,5,6,7-tetrachlorophthalimide (TCP) with 2,4-dimethylthiazole, and its structure is reported here (Fig. 1).



(I)

Bond lengths and angles in (I) have normal values (Allen *et al.*, 1987). The thiazole ring, C1/C6–C7/N1/C12, adopts an envelope conformation, with C12 deviating from the mean plane of the other four atoms by 0.207 (1) Å. The puckering parameters are $Q(2)$ = 0.127 (1) Å and $\varphi(2)$ = 135.8 (4)° (Cremer & Pople, 1975).

Intramolecular C8—H8*B*···O2 and C9—H9*A*···S1, C13—H13*C*···S1, and C13—H13*B*···Cl1 hydrogen bonds generate *S*(5), *S*(6) and *S*(7) ring motifs, respectively (Table 1 and Fig. 1) (Bernstein *et al.*, 1995). The crystal structure is stabilized by intermolecular O—H···O, O—H···N, C—H···Cl and C—H···O interactions (Table 1 and Fig. 2). The relatively short distances Cl2···O2^{i} [3.110 (1) Å] and Cl3···S1^{ii} [3.381 (1) Å; symmetry codes: (i) *x*, 1 + *y*, *z*; (ii) *x*, *y*, 1 + *z*] indicate the presence of intermolecular interactions, which contribute to the further stabilization of the crystal structure.

Experimental

Compound (I) was synthesized by the photo-induced reaction between *N*-(2-hydroxyethyl)-4,5,6,7-tetrachlorophthalimide (0.025

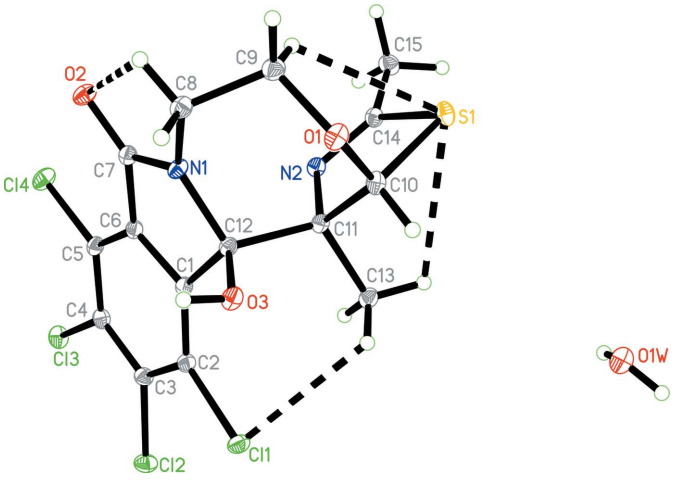


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atom numbering. Dashed lines indicate the intramolecular hydrogen bonds.

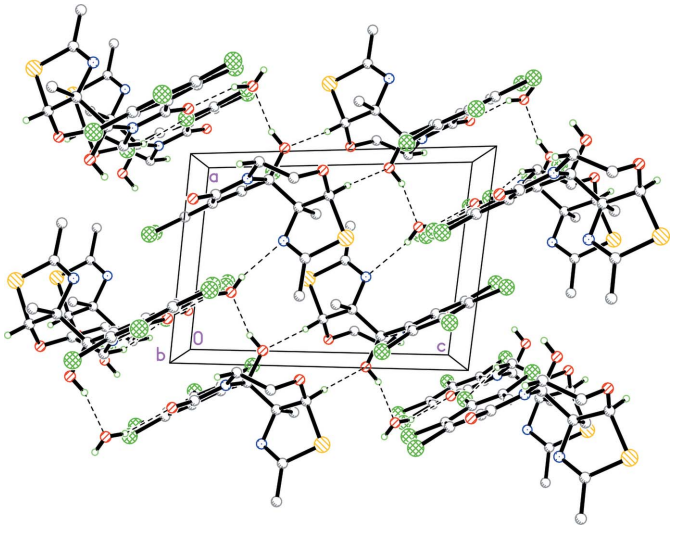


Figure 2
The crystal packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in the hydrogen bonds have been omitted for clarity.

M) and an excess amount of 2,4-dimethylthiazole in a benzene (120 ml) solution. The title compound was isolated using silica gel column chromatography with petroleum ether (b.p. 333–363 K)–ethyl acetate as eluant for gradient elution. Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the solvents from a petroleum ether–chloroform solution (2:1 *v*/*v*).

Crystal data

$C_{15}H_{14}Cl_4N_2O_4S$
M_r = 460.14
Triclinic, $P\overline{1}$
a = 8.0229 (1) Å
b = 9.9314 (2) Å
c = 11.8275 (2) Å
α = 68.407 (1)°
β = 80.964 (1)°
γ = 81.615 (1)°
V = 861.40 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 100.0 (1) K
0.46 × 0.45 × 0.43 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.701, T_{max} = 0.713
18330 measured reflections
6168 independent reflections
5896 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.064
S = 1.13
6168 reflections
249 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W1···O2^{i}	0.81 (2)	2.09 (2)	2.862 (1)	159 (2)
O1W—H2W1···N2^{ii}	0.77 (2)	2.32 (2)	3.071 (1)	166 (2)
O3—H1$O3$···O1W^{iii}	0.81 (2)	1.95 (2)	2.738 (1)	163 (2)
C8—H8B···Cl2^{iv}	0.97	2.72	3.650 (1)	161
C8—H8B···O2	0.97	2.44	2.852 (1)	105
C9—H9A···S1	0.97	2.76	3.221 (1)	110
C10—H10A···O3^{iii}	0.98	2.57	3.537 (1)	169
C13—H13B···Cl1	0.96	2.76	3.197 (1)	108
C13—H13C···S1	0.96	2.88	3.288 (1)	107

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

The water and hydroxyl H atoms were located in a difference Fourier map and were refined with isotropic displacement parameters. Other H atoms were positioned geometrically and treated as riding, with C—H = 0.96 or 0.97 Å, and U_{iso}(H)= 1.2U_{eq}(C) or 1.5$_{eq}$(methyl 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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