Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,5,6,7-Tetrachloro-2-hydroxyisoindoline-1,3-dione *N*,*N*-dimethylformamide solvate

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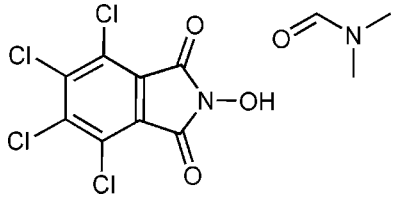
Received 22 June 2007; accepted 23 June 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.095; data-to-parameter ratio = 13.2.

In the title compound, $C_8HCl_4NO_3\cdot C_3H_7NO$, the crystal packing is consolidated by O—H···O hydrogen bonds.

Related literature

For related structures, see: Liang *et al.* (2006, 2007). For background, see: Lima *et al.* (2002).



Experimental

Crystal data

$C_8HCl_4NO_3\cdot C_3H_7NO$
M_r = 373.99
Orthorhombic, *Pbca*
a = 16.885 (2) Å
b = 7.7823 (11) Å
c = 21.974 (3) Å
V = 2887.5 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 298 (2) K
0.45 × 0.38 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.705, T_{max} = 0.892
11352 measured reflections
2550 independent reflections
2127 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.095
S = 1.03
2550 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4^{i}	0.82	1.76	2.562 (3)	167

Symmetry code: (i) $-x+\frac{1}{2}, -y, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Natural Science Foundation of Weifang University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2456).

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supplementary materials

Acta Cryst. (2007). E**63**, o3333 [**doi:10.1107/S1600536807030656**]

4,5,6,7-Tetrachloro-2-hydroxyisoindoline-1,3-dione *N*,*N*-dimethylformamide solvate

J. Li

Comment

Phthalimides and N-substituted phthalimides are an important class of compounds because of their interesting biological activities (Lima *et al.*, 2002). In this paper, the structure of the title compound, (I), is reported. The asymmetric unit of (I) contains one 4,5,6,7-Tetrachloro-2-hydroxylisoindoline-1,3-dione molecule and one DMF molecule (Fig. 1). The bond lengths and angles agree with those in those similar compounds 4-phthalimidobenzoic acid *N*,*N*-dimethylformamide solvate (Liang *et al.*, 2006) and 4-(5-bromo-1,3-dioxoisoindolin-2-yl)benzoic acid *N*,*N*-dimethylformamide solvate (Liang *et al.*, 2007). 4,5,6,7-tetrachloro-2-hydroxylisoindoline-1,3-dione molecule and DMF molecule are planar, within 0.024 (2) Å and 0.019 (2) Å for all non-H atoms, respectively. The dihedral angle between them is 70.6 (2) °. The crystal structure is stabilized by an O—H···O hydrogen bond which connects the benzoic acid and DMF molecules (Fig. 2 and Table 1).

Experimental

A mixture of 4,5,6,7-tetrachloroisobenzofuran-1,3-dione (0.01 mol) and hydroxyamine hydrochloride (0.01 mol) in acetic acid (10 ml) was refluxed for 1 h. After cooling, filtration and drying, 4,5,6,7-tetrachloro-2-hydroxylisoindoline-1,3-dione was obtained. 10 mg of this compound were dissolved in DMF (5 ml), and the solution was kept at room temperature for 10 d. Natural evaporation gave colourless slabls of (I).

Refinement

The H atoms were initially located from difference maps, then relocated in idealized locations (C—H = 0.93–0.96 Å, O—H = 0.82 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(O, methyl-C).

Figures

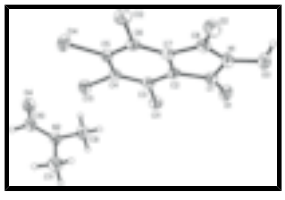

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms).

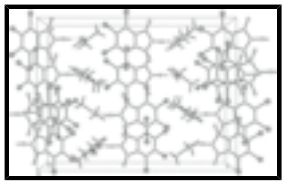

Fig. 2. The crystal packing of (I), viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines.

4,5,6,7-Tetrachloro-2-hydroxyisoindoline-1,3-dione *N*,*N*-dimethylformamide solvate

Crystal data

$C_8HCl_4NO_3 \cdot C_3H_7NO$	$F_{000} = 1504$
$M_r = 373.99$	$D_x = 1.721$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 4163 reflections
a = 16.885 (2) Å	θ = 2.4–27.1º
b = 7.7823 (11) Å	μ = 0.84 mm^{-1}
c = 21.974 (3) Å	*T* = 298 (2) K
V = 2887.5 (7) Å^3	Slab, colourless
Z = 8	0.45 × 0.38 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer	2550 independent reflections
Radiation source: fine-focus sealed tube	2127 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
T = 298(2) K	$\theta_{max} = 25.0$º
ω scans	$\theta_{min} = 2.2$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	*h* = −20→15
$T_{min} = 0.705$, $T_{max} = 0.892$	*k* = −9→8
11352 measured reflections	*l* = −19→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.936P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} = 0.001$
2550 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
193 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.20622 (14)	0.3474 (3)	0.45063 (10)	0.0411 (5)
C2	0.17868 (11)	0.2593 (3)	0.50706 (9)	0.0347 (5)
C3	0.22109 (12)	0.1868 (2)	0.55376 (9)	0.0350 (5)
C4	0.17926 (12)	0.1107 (3)	0.60146 (9)	0.0348 (5)
C5	0.09689 (12)	0.1052 (3)	0.60049 (9)	0.0361 (5)
C6	0.05419 (12)	0.1804 (3)	0.55290 (9)	0.0366 (5)
C7	0.09632 (11)	0.2569 (3)	0.50683 (9)	0.0360 (5)
C8	0.06792 (14)	0.3430 (3)	0.45026 (10)	0.0431 (5)
C9	0.42878 (16)	0.0147 (4)	0.70928 (12)	0.0624 (7)
H9A	0.4798	0.0639	0.7176	0.094*
H9B	0.4053	0.0722	0.6751	0.094*
H9C	0.4347	−0.1052	0.7002	0.094*
C10	0.35766 (14)	−0.0968 (3)	0.79531 (12)	0.0533 (6)
H10	0.3267	−0.0758	0.8295	0.064*
C11	0.35773 (17)	0.2081 (4)	0.78049 (15)	0.0706 (8)
H11A	0.3245	0.2040	0.8159	0.106*
H11B	0.3300	0.2644	0.7480	0.106*
H11C	0.4052	0.2708	0.7897	0.106*
N1	0.13743 (11)	0.3839 (3)	0.41940 (9)	0.0481 (5)
N2	0.37816 (11)	0.0348 (3)	0.76212 (9)	0.0452 (5)
O1	0.27226 (10)	0.3821 (2)	0.43417 (8)	0.0577 (5)
O2	0.00221 (10)	0.3717 (2)	0.43250 (8)	0.0612 (5)
O3	0.13795 (11)	0.4787 (2)	0.36673 (8)	0.0597 (5)
H3	0.1379	0.4138	0.3373	0.090*
O4	0.37631 (13)	−0.2469 (3)	0.78468 (9)	0.0749 (6)
Cl1	0.32275 (3)	0.18521 (7)	0.55363 (3)	0.04350 (18)
Cl2	−0.04705 (3)	0.17228 (8)	0.55248 (3)	0.05155 (19)
Cl3	0.22967 (3)	0.02190 (8)	0.66120 (3)	0.04968 (19)
Cl4	0.04709 (4)	0.00212 (8)	0.65760 (3)	0.0554 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0459 (14)	0.0381 (12)	0.0393 (12)	−0.0018 (10)	0.0023 (10)	−0.0028 (9)
C2	0.0319 (11)	0.0330 (10)	0.0391 (12)	−0.0011 (9)	0.0023 (9)	−0.0040 (9)
C3	0.0298 (11)	0.0361 (11)	0.0390 (11)	−0.0012 (8)	0.0002 (9)	−0.0076 (9)
C4	0.0335 (11)	0.0358 (11)	0.0351 (11)	0.0005 (9)	−0.0016 (9)	−0.0030 (9)

C5	0.0333 (11)	0.0383 (11)	0.0367 (11)	−0.0031 (9)	0.0045 (9)	−0.0037 (9)
C6	0.0281 (11)	0.0392 (12)	0.0424 (12)	−0.0004 (8)	−0.0002 (9)	−0.0069 (9)
C7	0.0327 (11)	0.0349 (11)	0.0404 (12)	0.0000 (9)	−0.0035 (9)	−0.0061 (9)
C8	0.0454 (13)	0.0417 (12)	0.0422 (13)	0.0006 (10)	−0.0081 (10)	−0.0021 (10)
C9	0.0547 (16)	0.0831 (19)	0.0494 (15)	0.0002 (13)	0.0027 (12)	0.0048 (14)
C10	0.0554 (16)	0.0638 (17)	0.0408 (14)	−0.0016 (13)	−0.0023 (11)	−0.0020 (13)
C11	0.088 (2)	0.0539 (16)	0.0695 (19)	0.0068 (14)	−0.0037 (16)	−0.0019 (15)
N1	0.0547 (13)	0.0513 (12)	0.0381 (11)	−0.0014 (9)	−0.0047 (9)	0.0080 (9)
N2	0.0448 (11)	0.0507 (11)	0.0402 (11)	0.0010 (9)	−0.0034 (8)	0.0011 (9)
O1	0.0479 (10)	0.0712 (11)	0.0539 (10)	−0.0073 (9)	0.0119 (8)	0.0101 (9)
O2	0.0481 (10)	0.0721 (12)	0.0634 (11)	0.0025 (9)	−0.0182 (8)	0.0087 (9)
O3	0.0874 (15)	0.0487 (10)	0.0429 (10)	−0.0019 (9)	−0.0054 (9)	0.0113 (8)
O4	0.1157 (17)	0.0529 (11)	0.0562 (12)	0.0021 (11)	0.0095 (11)	0.0013 (10)
Cl1	0.0267 (3)	0.0512 (3)	0.0525 (4)	−0.0005 (2)	0.0024 (2)	−0.0021 (2)
Cl2	0.0267 (3)	0.0645 (4)	0.0635 (4)	−0.0001 (2)	−0.0016 (2)	−0.0001 (3)
Cl3	0.0416 (3)	0.0637 (4)	0.0437 (3)	−0.0001 (3)	−0.0072 (2)	0.0096 (3)
Cl4	0.0419 (3)	0.0724 (4)	0.0517 (4)	−0.0062 (3)	0.0107 (3)	0.0128 (3)

Geometric parameters (Å, °)

C1—O1	1.203 (3)	C8—N1	1.392 (3)
C1—N1	1.379 (3)	C9—N2	1.450 (3)
C1—C2	1.491 (3)	C9—H9A	0.9600
C2—C3	1.372 (3)	C9—H9B	0.9600
C2—C7	1.391 (3)	C9—H9C	0.9600
C3—C4	1.396 (3)	C10—O4	1.232 (3)
C3—Cl1	1.717 (2)	C10—N2	1.304 (3)
C4—C5	1.392 (3)	C10—H10	0.9300
C4—Cl3	1.710 (2)	C11—N2	1.450 (3)
C5—C6	1.398 (3)	C11—H11A	0.9600
C5—Cl4	1.710 (2)	C11—H11B	0.9600
C6—C7	1.373 (3)	C11—H11C	0.9600
C6—Cl2	1.711 (2)	N1—O3	1.372 (3)
C7—C8	1.492 (3)	O3—H3	0.8200
C8—O2	1.197 (3)		
?···?	?		
O1—C1—N1	125.8 (2)	N1—C8—C7	103.75 (18)
O1—C1—C2	130.0 (2)	N2—C9—H9A	109.5
N1—C1—C2	104.23 (19)	N2—C9—H9B	109.5
C3—C2—C7	121.26 (19)	H9A—C9—H9B	109.5
C3—C2—C1	130.38 (19)	N2—C9—H9C	109.5
C7—C2—C1	108.36 (18)	H9A—C9—H9C	109.5
C2—C3—C4	118.15 (19)	H9B—C9—H9C	109.5
C2—C3—Cl1	121.57 (16)	O4—C10—N2	124.8 (2)
C4—C3—Cl1	120.27 (16)	O4—C10—H10	117.6
C5—C4—C3	120.49 (18)	N2—C10—H10	117.6
C5—C4—Cl3	119.77 (15)	N2—C11—H11A	109.5
C3—C4—Cl3	119.74 (15)	N2—C11—H11B	109.5
C4—C5—C6	120.92 (18)	H11A—C11—H11B	109.5

C4—C5—Cl4	119.65 (16)	N2—C11—H11C	109.5
C6—C5—Cl4	119.43 (16)	H11A—C11—H11C	109.5
C7—C6—C5	117.75 (18)	H11B—C11—H11C	109.5
C7—C6—Cl2	121.98 (16)	O3—N1—C1	121.67 (19)
C5—C6—Cl2	120.26 (16)	O3—N1—C8	122.61 (18)
C6—C7—C2	121.41 (19)	C1—N1—C8	114.88 (19)
C6—C7—C8	130.01 (19)	C10—N2—C11	120.8 (2)
C2—C7—C8	108.57 (19)	C10—N2—C9	121.3 (2)
O2—C8—N1	125.4 (2)	C11—N2—C9	117.6 (2)
O2—C8—C7	130.8 (2)	N1—O3—H3	109.5
O1—C1—C2—C3	−3.6 (4)	Cl2—C6—C7—C2	−178.59 (15)
N1—C1—C2—C3	176.6 (2)	C5—C6—C7—C8	178.6 (2)
O1—C1—C2—C7	177.0 (2)	Cl2—C6—C7—C8	−0.3 (3)
N1—C1—C2—C7	−2.8 (2)	C3—C2—C7—C6	−0.7 (3)
C7—C2—C3—C4	−0.3 (3)	C1—C2—C7—C6	178.70 (19)
C1—C2—C3—C4	−179.5 (2)	C3—C2—C7—C8	−179.34 (19)
C7—C2—C3—Cl1	178.59 (15)	C1—C2—C7—C8	0.1 (2)
C1—C2—C3—Cl1	−0.7 (3)	C6—C7—C8—O2	3.1 (4)
C2—C3—C4—C5	1.6 (3)	C2—C7—C8—O2	−178.4 (2)
Cl1—C3—C4—C5	−177.22 (15)	C6—C7—C8—N1	−175.9 (2)
C2—C3—C4—Cl3	−178.96 (15)	C2—C7—C8—N1	2.6 (2)
Cl1—C3—C4—Cl3	2.2 (2)	O1—C1—N1—O3	−5.3 (4)
C3—C4—C5—C6	−2.1 (3)	C2—C1—N1—O3	174.53 (19)
Cl3—C4—C5—C6	178.50 (16)	O1—C1—N1—C8	−175.0 (2)
C3—C4—C5—Cl4	176.83 (16)	C2—C1—N1—C8	4.8 (3)
Cl3—C4—C5—Cl4	−2.6 (2)	O2—C8—N1—O3	6.6 (4)
C4—C5—C6—C7	1.1 (3)	C7—C8—N1—O3	−174.36 (19)
Cl4—C5—C6—C7	−177.83 (16)	O2—C8—N1—C1	176.3 (2)
C4—C5—C6—Cl2	180.00 (15)	C7—C8—N1—C1	−4.7 (3)
Cl4—C5—C6—Cl2	1.1 (2)	O4—C10—N2—C11	−175.9 (3)
C5—C6—C7—C2	0.3 (3)	O4—C10—N2—C9	−2.3 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4^{i}	0.82	1.76	2.562 (3)	167

Symmetry codes: (i) −*x*+1/2, −*y*, *z*−1/2.

Fig. 1

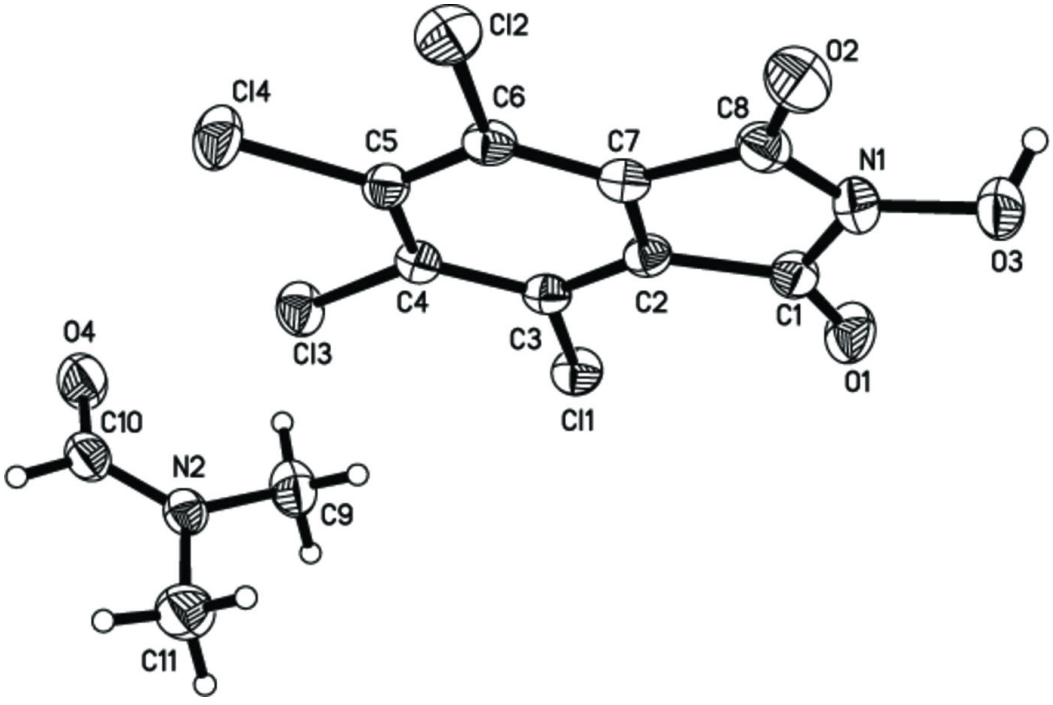

Fig. 2