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Tetrachloroisoindoline-1,3-dione *N*,*N*-dimethylformamide solvate

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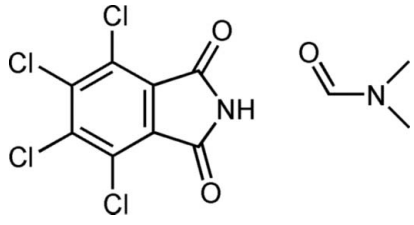
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.046; wR factor = 0.132; data-to-parameter ratio = 13.5.

The crystal structure of the title compound, $C_8HCl_4NO_2\cdot C_3H_7NO$, comprises tetrachloroisoindoline-1,3-dione and *N*,*N*-dimethylformamide (DMF) solvent molecules, which are held together by N—H···O, C—H···O and C—H···Cl hydrogen bonds. The tetrachloroisoindoline-1,3-dione molecule is essentially planar.

Related literature

For the structure of the related *N*-methylphthalimide, see Liang & Li (2006).



Experimental

Crystal data

$C_8HCl_4NO_2\cdot C_3H_7NO$
M_r = 357.99
Triclinic, $P\overline{1}$
a = 5.6583 (16) Å
b = 11.268 (3) Å
c = 11.817 (3) Å
α = 73.244 (4)°
β = 82.518 (5)°
γ = 80.991 (5)°
V = 709.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 294 (2) K
0.26 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.811, T_{max} = 0.906
3691 measured reflections
2488 independent reflections
1776 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 1.04
2488 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.98 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	1.90	2.752 (4)	170
C9—H9···O2^{ii}	0.93	2.53	3.452 (6)	175

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

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*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2366).

References

Bruker (1997). *SADABS* (Version 2.01), *SMART* (Version 5.044), *SAINT* (Version 5.01) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Liang, Z.-P. & Li, J. (2006). *Acta Cryst.* E**62**, o4274–o4275.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

Acta Cryst. (2007). E**63**, o2460 [**doi:10.1107/S1600536807017333**]

Tetrachloroisoindoline-1,3-dione *N*,*N*-dimethylformamide solvate

Z.-P. Liang, J. Li, Y.-C. Zhang and X.-S. Tai

Comment

Phthalimides and N-substituted phthalimides are an important class of compounds because of their interesting biological activities. Phthalimides have also served as starting materials and intermediates for the syntheses of alkaloids. In this paper, the structure of the title compound, (I), is reported.

The asymmetric unit of (I) (Fig. 1) consists one tetrachloroisoindoline-1,3-dione molecule and one DMF solvent molecule. The tetrachloroisoindoline-1,3-dione molecule is essentially planar, with an r.m.s.

deviation of 0.022 (2) Å from the mean plane for the non-H atoms. The DMF molecule is also planar, with an r.m.s. deviation of 0.007 (3) Å for the non-H

atoms (Fig. 2). The dihedral angle between the two molecules is 113.8 (2)°. The

geometric parameters of tetrachloroisoindoline-1,3-dione are similar to those in the compound *N*-methylphthalimide (Liang & Li, 2006).

The crystal structure of (I) is stabilized by N—H···O, C—H···O and C—H···Cl hydrogen bonds (Fig.2 and Table 1).

Experimental

A mixture of tetrachloroisobenzofuran-1,3-dione (0.01 mol) and urea (0.01 mol) in acetic acid (10 ml) was refluxed for 5 h. After cooling, filtration and drying, the compound tetrachloroisoindoline-1,3-dione was obtained. This compound (10 mg) was dissolved in DMF (5 ml) and the solution was allowed to evaporate at room temperature. Colourless single crystals of (I) were formed after 20 d.

Refinement

The H atoms were positioned geometrically, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(methyl C).

Figures

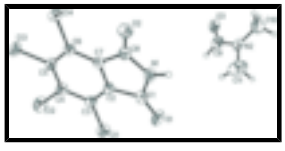
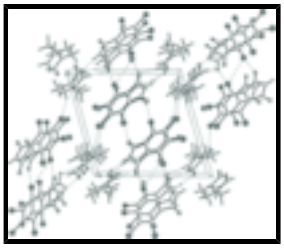


Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms).
Fig. 2. The crystal packing of (I), viewed along the a axis. Hydrogen bonds are indicated by dashed lines.



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Crystal data

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$M_r = 357.99$	$F_{000} = 360$
Triclinic, $P\overline{1}$	$D_x = 1.675$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.6583$ (16) Å	Cell parameters from 1410 reflections
$b = 11.268$ (3) Å	$\theta = 3.6$–24.8º
$c = 11.817$ (3) Å	$\mu = 0.84$ mm^{-1}
$\alpha = 73.244$ (4)º	$T = 294$ (2) K
$\beta = 82.518$ (5)º	Block, colourless
$\gamma = 80.991$ (5)º	0.26 × 0.20 × 0.12 mm
$V = 709.6$ (3) Å^3	

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Bruker SMART CCD area-detector diffractometer	2488 independent reflections
Radiation source: fine-focus sealed tube	1776 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 294$(2) K	$\theta_{max} = 25.0$º
ω scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -5 \rightarrow 6$
$T_{min} = 0.811$, $T_{max} = 0.906$	$k = -9 \rightarrow 13$
3691 measured reflections	$l = -14 \rightarrow 13$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.5593P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.132$	$\Delta\rho_{max} = 0.98$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.29$ e Å^{-3}

2488 reflections

184 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.031 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
Cl1	1.31478 (18)	0.50354 (9)	0.38085 (9)	0.0521 (3)
Cl2	1.0432 (2)	0.53339 (11)	0.15763 (10)	0.0718 (4)
Cl3	0.6824 (2)	0.35207 (11)	0.16242 (9)	0.0616 (4)
Cl4	0.59312 (17)	0.13136 (9)	0.38830 (9)	0.0509 (3)
O1	1.3399 (6)	0.3021 (3)	0.6299 (3)	0.0657 (8)
O2	0.8175 (5)	0.0305 (3)	0.6327 (2)	0.0588 (8)
N1	1.0840 (6)	0.1529 (3)	0.6599 (3)	0.0494 (8)
H1	1.1143	0.1118	0.7310	0.059*
C1	1.1895 (7)	0.2577 (4)	0.5967 (3)	0.0458 (9)
C2	1.0819 (6)	0.3003 (3)	0.4800 (3)	0.0364 (8)
C3	1.1208 (6)	0.4000 (3)	0.3818 (3)	0.0402 (8)
C4	0.9965 (7)	0.4132 (3)	0.2833 (3)	0.0431 (9)
C5	0.8359 (6)	0.3311 (3)	0.2847 (3)	0.0411 (8)
C6	0.7955 (6)	0.2318 (3)	0.3850 (3)	0.0377 (8)
C7	0.9221 (6)	0.2174 (3)	0.4818 (3)	0.0352 (8)
C8	0.9253 (7)	0.1202 (3)	0.5982 (3)	0.0442 (9)
O3	0.2152 (6)	1.0015 (3)	0.8760 (3)	0.0703 (9)
N2	0.5006 (6)	0.8550 (3)	0.9699 (3)	0.0575 (9)
C9	0.4085 (11)	0.9480 (5)	0.8766 (5)	0.0736 (14)
H9	0.5104	0.9702	0.8076	0.088*
C10	0.3708 (13)	0.8099 (6)	1.0762 (5)	0.113 (2)
H10A	0.2469	0.8740	1.0905	0.170*
H10B	0.4755	0.7853	1.1390	0.170*
H10C	0.2993	0.7390	1.0736	0.170*
C11	0.7448 (11)	0.8044 (6)	0.9431 (7)	0.114 (2)

H11A	0.7946	0.8409	0.8611	0.171*
H11B	0.7545	0.7155	0.9579	0.171*
H11C	0.8480	0.8231	0.9923	0.171*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0567 (6)	0.0435 (6)	0.0610 (6)	−0.0187 (5)	−0.0079 (5)	−0.0144 (5)
Cl2	0.0916 (9)	0.0616 (7)	0.0538 (6)	−0.0304 (6)	−0.0212 (6)	0.0149 (5)
Cl3	0.0682 (7)	0.0733 (8)	0.0461 (6)	−0.0149 (6)	−0.0246 (5)	−0.0093 (5)
Cl4	0.0471 (6)	0.0533 (6)	0.0588 (6)	−0.0178 (4)	−0.0076 (4)	−0.0180 (5)
O1	0.076 (2)	0.070 (2)	0.0603 (18)	−0.0251 (16)	−0.0292 (15)	−0.0129 (15)
O2	0.0666 (19)	0.0518 (17)	0.0534 (17)	−0.0236 (15)	−0.0044 (14)	0.0008 (13)
N1	0.061 (2)	0.0493 (19)	0.0348 (16)	−0.0105 (16)	−0.0143 (15)	−0.0010 (14)
C1	0.050 (2)	0.044 (2)	0.045 (2)	−0.0022 (18)	−0.0114 (17)	−0.0134 (18)
C2	0.0383 (19)	0.0362 (19)	0.0366 (18)	−0.0049 (15)	−0.0053 (14)	−0.0119 (15)
C3	0.0393 (19)	0.0371 (19)	0.046 (2)	−0.0078 (15)	−0.0055 (16)	−0.0126 (16)
C4	0.049 (2)	0.038 (2)	0.0397 (19)	−0.0062 (17)	−0.0082 (16)	−0.0039 (16)
C5	0.043 (2)	0.044 (2)	0.0376 (19)	−0.0048 (16)	−0.0119 (16)	−0.0098 (16)
C6	0.0347 (18)	0.0374 (19)	0.0434 (19)	−0.0056 (15)	−0.0043 (15)	−0.0139 (16)
C7	0.0366 (19)	0.0325 (18)	0.0356 (18)	−0.0022 (15)	−0.0041 (14)	−0.0088 (14)
C8	0.048 (2)	0.039 (2)	0.043 (2)	−0.0036 (18)	−0.0070 (17)	−0.0070 (17)
O3	0.070 (2)	0.076 (2)	0.0573 (19)	0.0034 (18)	−0.0231 (16)	−0.0056 (16)
N2	0.058 (2)	0.052 (2)	0.067 (2)	−0.0059 (17)	−0.0295 (18)	−0.0125 (18)
C9	0.085 (4)	0.080 (4)	0.068 (3)	−0.028 (3)	−0.012 (3)	−0.029 (3)
C10	0.147 (6)	0.119 (5)	0.082 (4)	−0.084 (5)	−0.019 (4)	−0.001 (4)
C11	0.081 (4)	0.102 (5)	0.175 (7)	0.001 (4)	−0.023 (4)	−0.067 (5)

Geometric parameters (Å, °)

Cl1—C3	1.719 (4)	C5—C6	1.398 (5)
Cl2—C4	1.720 (4)	C6—C7	1.385 (5)
Cl3—C5	1.722 (3)	C7—C8	1.491 (5)
Cl4—C6	1.721 (4)	O3—C9	1.163 (6)
O1—C1	1.208 (5)	N2—C10	1.373 (7)
O2—C8	1.205 (5)	N2—C9	1.378 (6)
N1—C8	1.379 (5)	N2—C11	1.441 (6)
N1—C1	1.380 (5)	C9—H9	0.9300
N1—H1	0.8600	C10—H10A	0.9600
C1—C2	1.500 (5)	C10—H10B	0.9600
C2—C3	1.385 (5)	C10—H10C	0.9600
C2—C7	1.392 (5)	C11—H11A	0.9600
C3—C4	1.397 (5)	C11—H11B	0.9600
C4—C5	1.390 (5)	C11—H11C	0.9600
C8—N1—C1	113.8 (3)	C2—C7—C8	108.2 (3)
C8—N1—H1	123.1	O2—C8—N1	126.2 (3)
C1—N1—H1	123.1	O2—C8—C7	128.6 (3)
O1—C1—N1	126.2 (3)	N1—C8—C7	105.2 (3)

O1—C1—C2	128.6 (4)	C10—N2—C9	123.8 (5)
N1—C1—C2	105.2 (3)	C10—N2—C11	122.8 (5)
C3—C2—C7	121.5 (3)	C9—N2—C11	113.3 (5)
C3—C2—C1	130.9 (3)	O3—C9—N2	126.1 (5)
C7—C2—C1	107.5 (3)	O3—C9—H9	116.9
C2—C3—C4	117.4 (3)	N2—C9—H9	116.9
C2—C3—Cl1	121.3 (3)	N2—C10—H10A	109.5
C4—C3—Cl1	121.2 (3)	N2—C10—H10B	109.5
C5—C4—C3	121.3 (3)	H10A—C10—H10B	109.5
C5—C4—Cl2	119.3 (3)	N2—C10—H10C	109.5
C3—C4—Cl2	119.4 (3)	H10A—C10—H10C	109.5
C4—C5—C6	120.7 (3)	H10B—C10—H10C	109.5
C4—C5—Cl3	119.9 (3)	N2—C11—H11A	109.5
C6—C5—Cl3	119.3 (3)	N2—C11—H11B	109.5
C7—C6—C5	117.9 (3)	H11A—C11—H11B	109.5
C7—C6—Cl4	121.2 (3)	N2—C11—H11C	109.5
C5—C6—Cl4	121.0 (3)	H11A—C11—H11C	109.5
C6—C7—C2	121.1 (3)	H11B—C11—H11C	109.5
C6—C7—C8	130.7 (3)		
C8—N1—C1—O1	−177.3 (4)	Cl3—C5—C6—C7	179.9 (3)
C8—N1—C1—C2	1.4 (4)	C4—C5—C6—Cl4	178.9 (3)
O1—C1—C2—C3	−1.1 (7)	Cl3—C5—C6—Cl4	−0.6 (4)
N1—C1—C2—C3	−179.7 (3)	C5—C6—C7—C2	0.9 (5)
O1—C1—C2—C7	178.2 (4)	Cl4—C6—C7—C2	−178.6 (3)
N1—C1—C2—C7	−0.5 (4)	C5—C6—C7—C8	−177.7 (3)
C7—C2—C3—C4	−1.1 (5)	Cl4—C6—C7—C8	2.8 (5)
C1—C2—C3—C4	178.0 (4)	C3—C2—C7—C6	0.0 (5)
C7—C2—C3—Cl1	179.0 (3)	C1—C2—C7—C6	−179.3 (3)
C1—C2—C3—Cl1	−1.9 (5)	C3—C2—C7—C8	178.9 (3)
C2—C3—C4—C5	1.4 (5)	C1—C2—C7—C8	−0.5 (4)
Cl1—C3—C4—C5	−178.7 (3)	C1—N1—C8—O2	177.4 (4)
C2—C3—C4—Cl2	−178.5 (3)	C1—N1—C8—C7	−1.6 (4)
Cl1—C3—C4—Cl2	1.4 (4)	C6—C7—C8—O2	1.0 (7)
C3—C4—C5—C6	−0.5 (6)	C2—C7—C8—O2	−177.7 (4)
Cl2—C4—C5—C6	179.4 (3)	C6—C7—C8—N1	180.0 (3)
C3—C4—C5—Cl3	178.9 (3)	C2—C7—C8—N1	1.2 (4)
Cl2—C4—C5—Cl3	−1.2 (5)	C10—N2—C9—O3	1.7 (7)
C4—C5—C6—C7	−0.6 (5)	C11—N2—C9—O3	178.7 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.86	1.90	2.752 (4)	170
C9—H9···O2^{ii}	0.93	2.53	3.452 (6)	175

Symmetry codes: (i) *x*+1, *y*−1, *z*; (ii) *x*, *y*+1, *z*.

Fig. 1

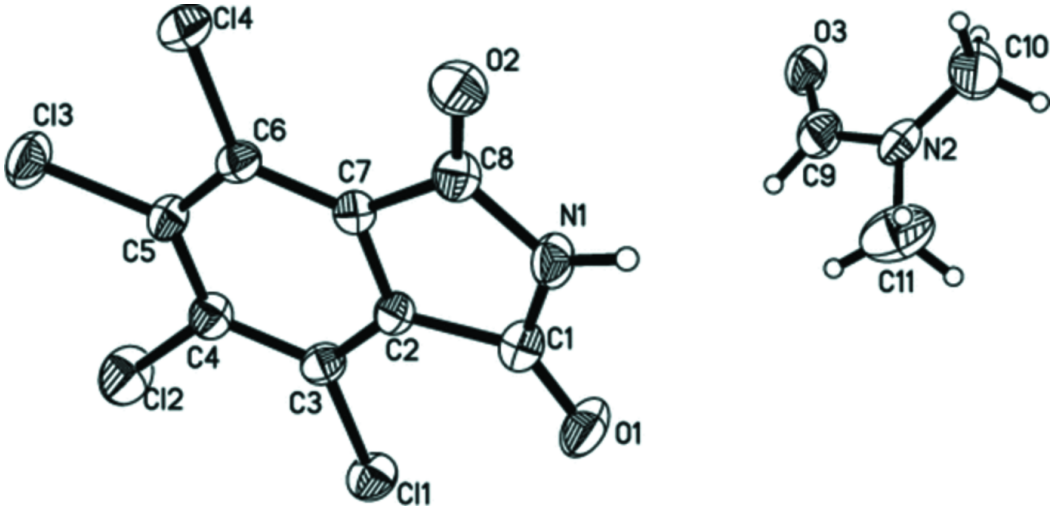

Fig. 2