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4,5,6,7-Tetrachloro-2-(4-fluorophenyl)-isoindoline-1,3-dione

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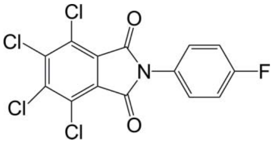
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.005 Å; R factor = 0.050; wR factor = 0.204; data-to-parameter ratio = 11.4.

The title compound, $C_{14}H_4Cl_4FNO_2$, has crystallographic twofold symmetry with the N and F atoms and two C atoms of the benzene ring located on a twofold rotation axis. The isoindoledione ring system is almost planar [maximum atomic deviation = 0.036 (3) Å], and is twisted with respect to the florobenzene ring, making a dihedral angle of 58.56 (16)°. Weak intermolecular C—H⋯Cl hydrogen bonding is present in the crystal structure.

Related literature

The title compound is an intermediate in the synthesis of organic electro-luminescent materials, see: Han & Kay (2005). For a related structure, see: Xu *et al.* (2006).



Experimental

Crystal data

$C_{14}H_4Cl_4FNO_2$
M_r = 378.98
Orthorhombic, *Pcca*
a = 7.9400 (16) Å
b = 5.6744 (11) Å
c = 29.461 (6) Å
V = 1327.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.91 mm^{-1}
T = 113 K
0.20 × 0.18 × 0.12 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2001)
$T_{\min}$ = 0.839, $T_{\max}$ = 0.899
6423 measured reflections
1174 independent reflections
1053 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.204
S = 1.04
1174 reflections
103 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.84 e Å^{-3}
$\Delta\rho_{\min}$ = −0.74 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7⋯Cl2^{i}	0.95	2.80	3.690 (4)	157

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

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2780).

References

Han, K. J. & Kay, K. Y. (2005). *J. Korean Chem. Soc.* **49**, 233–238.
Rigaku/MSC (2001). *CrystalClear*. Rigaku/MSC, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xu, D., Shi, Y.-Q., Chen, B., Cheng, Y.-H. & Gao, X. (2006). *Acta Cryst.* E**62**, o408–o409.

supplementary materials

Acta Cryst. (2010). E**66**, o1744 [**doi:10.1107/S1600536810023032**]

4,5,6,7-Tetrachloro-2-(4-fluorophenyl)isoindoline-1,3-dione

X.-S. Fu, **X.-P. Yu**, **W.-M. Wang** and **F. Lin**

Comment

The title compound is a key intermediate in the synthesis of organic electro-luminescent materials. The emission of light by organic molecules exposed to an electric field has been wide investigated in both an academic and industrial context (Han & Kay, 2005).

The molecular structure of the title compound is illustrated in Fig. 1. In the title compound, the dihedral angle between the benzene ring and the indole ring system is 58.56 (16)°, which is similar to 59.95 (4)° found in a related compound *N*-(2-fluorophenyl)phthalimide (Xu *et al.*, 2006). Weak intermolecular C—H···Cl hydrogen bonding is present in the crystal structure (Table 1).

Experimental

An acetic acid solution of tetrachlorophthalic anhydride (28.6 g, 100 mmol) and 4-fluoroaniline (9.45 ml, 100 mmol) was refluxed overnight, and then filtered. The crude produce was recrystallized from ethyl acetate.

Refinement

H atoms were positioned geometrically and refined as riding with C—H = 0.95 Å, and U_{iso}(H) = 1.2U_{eq}(C).

Figures

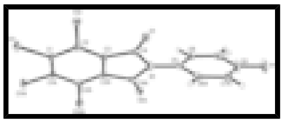


Fig. 1. View of the molecule of showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

4,5,6,7-Tetrachloro-2-(4-fluorophenyl)isoindoline-1,3-dione

Crystal data

$C_{14}H_4Cl_4FNO_2$	F(000) = 752
M_r = 378.98	D_x = 1.896 Mg m^{-3}
Orthorhombic, *Pcca*	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2a 2ac	Cell parameters from 3007 reflections
a = 7.9400 (16) Å	θ = 2.8–27.9°
b = 5.6744 (11) Å	μ = 0.91 mm^{-1}
c = 29.461 (6) Å	T = 113 K
V = 1327.4 (5) Å^3	Prism, colorless

$Z = 4$

$0.20 \times 0.18 \times 0.12$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer

Radiation source: rotating anode

confocal

Detector resolution: 7.31 pixels mm^{-1}

ω and φ scans

Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2001)

$T_{\text{min}} = 0.839$, $T_{\text{max}} = 0.899$

6423 measured reflections

1174 independent reflections

1053 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\text{max}} = 25.0°$, $\theta_{\text{min}} = 2.8°$

$h = -9\rightarrow9$

$k = -6\rightarrow6$

$l = -35\rightarrow30$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.204$

$S = 1.03$

1174 reflections

103 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1623P)^2 + 2.0637P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.84$ e Å^{-3}

$\Delta\rho_{\text{min}} = -0.74$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $\text{Fc}^* = \text{kFc}[1+0.001\text{xFc}^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.039 (8)

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 $F^2 > \sigma(F^2)$ 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	0.14081 (12)	0.27246 (16)	−0.00242 (3)	0.0266 (5)
Cl2	0.01306 (11)	0.05859 (15)	0.08874 (3)	0.0246 (5)

F1	0.2500	0.5000	0.39104 (10)	0.0445 (10)
O1	0.0906 (3)	0.1621 (5)	0.19160 (8)	0.0236 (7)
N1	0.2500	0.5000	0.20376 (13)	0.0224 (10)
C1	0.1999 (4)	0.4011 (6)	0.04757 (10)	0.0199 (8)
C2	0.1431 (4)	0.3007 (7)	0.08894 (12)	0.0209 (9)
C3	0.1986 (4)	0.4021 (6)	0.12895 (12)	0.0184 (8)
C4	0.1677 (4)	0.3289 (6)	0.17733 (11)	0.0183 (8)
C5	0.2500	0.5000	0.25202 (14)	0.0173 (11)
C6	0.1893 (4)	0.6957 (6)	0.27533 (11)	0.0206 (8)
H6	0.1499	0.8293	0.2591	0.025*
C7	0.1864 (5)	0.6948 (7)	0.32247 (12)	0.0264 (9)
H7	0.1419	0.8248	0.3389	0.032*
C8	0.2500	0.5000	0.34501 (17)	0.0338 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0358 (8)	0.0274 (8)	0.0165 (7)	0.0042 (4)	−0.0031 (3)	−0.0059 (3)
Cl2	0.0301 (7)	0.0219 (8)	0.0219 (7)	−0.0043 (3)	−0.0010 (3)	−0.0042 (3)
F1	0.063 (2)	0.054 (2)	0.0166 (17)	−0.027 (2)	0.000	0.000
O1	0.0269 (14)	0.0221 (14)	0.0218 (13)	−0.0051 (10)	0.0023 (10)	0.0039 (10)
N1	0.032 (2)	0.017 (2)	0.017 (2)	−0.0042 (18)	0.000	0.000
C1	0.0228 (18)	0.0250 (18)	0.0119 (17)	0.0079 (13)	−0.0016 (11)	−0.0046 (12)
C2	0.0182 (16)	0.0226 (19)	0.022 (2)	0.0011 (13)	0.0021 (11)	0.0000 (14)
C3	0.0179 (16)	0.0204 (16)	0.0168 (17)	0.0031 (13)	−0.0014 (12)	−0.0001 (13)
C4	0.0159 (14)	0.0220 (19)	0.0171 (18)	0.0024 (14)	−0.0009 (12)	0.0013 (13)
C5	0.014 (2)	0.020 (2)	0.018 (2)	−0.0002 (18)	0.000	0.000
C6	0.0195 (16)	0.0208 (18)	0.0213 (18)	−0.0038 (13)	0.0008 (12)	−0.0016 (14)
C7	0.0243 (18)	0.030 (2)	0.0252 (19)	−0.0066 (15)	0.0024 (14)	−0.0089 (15)
C8	0.050 (3)	0.036 (3)	0.015 (2)	−0.025 (3)	0.000	0.000

Geometric parameters (Å, °)

Cl1—C1	1.709 (3)	C3—C3^{i}	1.379 (7)
Cl2—C2	1.719 (4)	C3—C4	1.504 (5)
F1—C8	1.356 (6)	C5—C6	1.392 (4)
O1—C4	1.203 (5)	C5—C6^{i}	1.392 (4)
N1—C4	1.406 (4)	C6—C7	1.389 (5)
N1—C4^{i}	1.406 (4)	C6—H6	0.9500
N1—C5	1.422 (5)	C7—C8	1.385 (5)
C1—C1^{i}	1.376 (7)	C7—H7	0.9500
C1—C2	1.419 (5)	C8—C7^{i}	1.385 (5)
C2—C3	1.384 (5)		
C4—N1—C4^{i}	112.7 (4)	N1—C4—C3	105.0 (3)
C4—N1—C5	123.7 (2)	C6—C5—C6^{i}	120.9 (4)
C4^{i}—N1—C5	123.7 (2)	C6—C5—N1	119.6 (2)
C1^{i}—C1—C2	120.7 (2)	C6^{i}—C5—N1	119.6 (2)

C1^{i}—C1—Cl1	120.48 (13)	C7—C6—C5	119.7 (3)
C2—C1—Cl1	118.8 (3)	C7—C6—H6	120.1
C3—C2—C1	117.6 (3)	C5—C6—H6	120.1
C3—C2—Cl2	121.8 (3)	C8—C7—C6	118.4 (4)
C1—C2—Cl2	120.6 (3)	C8—C7—H7	120.8
C3^{i}—C3—C2	121.6 (2)	C6—C7—H7	120.8
C3^{i}—C3—C4	108.58 (19)	F1—C8—C7^{i}	118.6 (2)
C2—C3—C4	129.8 (3)	F1—C8—C7	118.6 (2)
O1—C4—N1	125.9 (3)	C7^{i}—C8—C7	122.7 (5)
O1—C4—C3	129.1 (3)		

C1^{i}—C1—C2—C3	−3.0 (6)	C2—C3—C4—O1	1.9 (6)
Cl1—C1—C2—C3	177.3 (2)	C3^{i}—C3—C4—N1	3.1 (4)
C1^{i}—C1—C2—Cl2	178.1 (3)	C2—C3—C4—N1	−178.2 (3)
Cl1—C1—C2—Cl2	−1.6 (4)	C4—N1—C5—C6	−122.6 (2)
C1—C2—C3—C3^{i}	1.9 (6)	C4^{i}—N1—C5—C6	57.4 (2)
Cl2—C2—C3—C3^{i}	−179.2 (3)	C4—N1—C5—C6^{i}	57.4 (2)
C1—C2—C3—C4	−176.6 (3)	C4^{i}—N1—C5—C6^{i}	−122.6 (2)
Cl2—C2—C3—C4	2.3 (5)	C6^{i}—C5—C6—C7	−1.1 (2)
C4^{i}—N1—C4—O1	178.8 (4)	N1—C5—C6—C7	178.9 (2)
C5—N1—C4—O1	−1.2 (4)	C5—C6—C7—C8	2.1 (4)
C4^{i}—N1—C4—C3	−1.12 (15)	C6—C7—C8—F1	178.9 (2)
C5—N1—C4—C3	178.88 (15)	C6—C7—C8—C7^{i}	−1.1 (2)
C3^{i}—C3—C4—O1	−176.8 (4)		

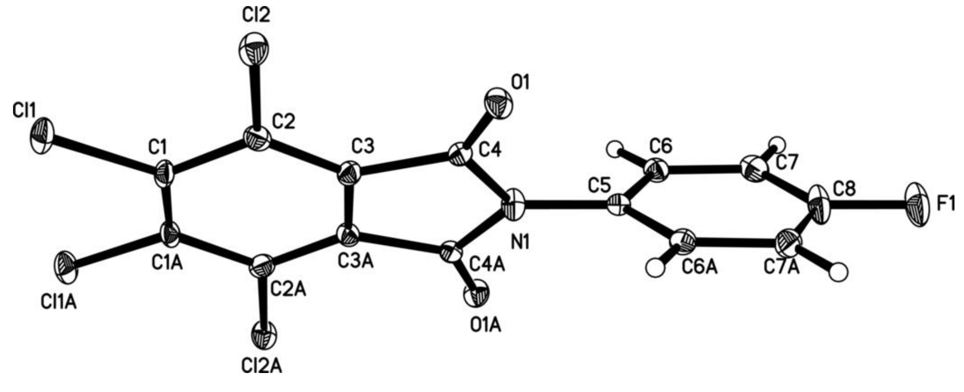
Symmetry codes: (i) $-x+1/2, -y+1, z$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···Cl2^{ii}	0.95	2.80	3.690 (4)	157

Symmetry codes: (ii) $-x, y+1, -z+1/2$.

Fig. 1


Cl2
Cl1
C2
O1
C1
C3
C4
C6
C7
C5
C8
F1
N1
C1A
C2A
C3A
C4A
C6A
C7A
Cl1A
O1A
Cl2A