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4,5,6,7-Tetrachloro-2-(1-naphthyl)-isoindole-1,3-dione acetone solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.105
Data-to-parameter ratio = 13.5

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

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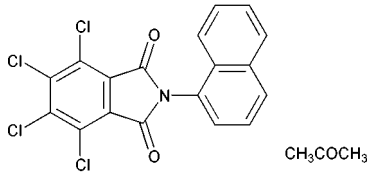
The phthalimide and naphthalene moieties of the title compound, $C_{18}H_7Cl_4NO_2\cdot C_3H_6O$, are oriented almost perpendicular to each other. The crystal structure is stabilized by weak π–π, C—H···π and van der Waals interactions.

Comment

N-Phthaloyl derivatization is one of the most frequently used methods of protection in synthesis involving compounds with primary amino groups (Falck *et al.*, 1995). Phthalimides have also served as starting materials and intermediates for the syntheses of alkaloids (Couture *et al.*, 1998) and pharmacophores (Couture *et al.*, 1997). They have cytotoxicity (Hall *et al.*, 1995) and anti-HIV activity (van Derpoorten *et al.*, 1997). In this paper, we report the structure of the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The isoindole moiety is planar, with a maximum deviation of 0.038 (2) Å for N1 , and the naphthalene ring system is planar within ±0.012 (3) Å. The dihedral angle between these two planes [84.2 (1)°] shows that they are orthogonal. The bond lengths and angles in the phthalimide moiety are comparable to those reported for phthalimide (Ng, 1992). The C—Cl bond lengths are in the range 1.713 (2)–1.716 (2) Å and are in agreement with the values reported in the literature (Busetti *et al.*, 1980). In the solid state, the naphthalene ring system of the inversion-related (x, $1-y$, $-z$) molecules are stacked with partial overlap, such that the centroids of the benzene ring (C9–C13/C18) and its inversion equivalent are separated by 3.693 (2) Å. The crystal structure is further stabilized by weak C—H···π interactions (Table 2) involving the solvent molecule, Cl···O [Cl2···O3 = 3.152 (4) Å and Cl4···O1($x-1$, $y-1$, z) = 3.250 (2) Å] short contacts and van der Waals forces (Fig. 2). In Table 2, *Cg*1, *Cg*2 and *Cg*3 denote the centroids of the benzene rings *A* (C2–C7), *B* (C9–C13/C18) and *C* (C13–C18), respectively.

Experimental

A mixture of tetrachlorophthalic anhydride (10 mmol) and 1-naphthylamine (10 mmol) were refluxed in acetic acid (40 ml) until the

disappearance of the starting materials as evidenced by thin-layer chromatography. After the reaction was complete, the yellow precipitate was filtered off and washed with a minimum amount of water to give the title compound (I). M.p. 551–552 K; IR (KBr): 1719.3 cm^{-1} (C═O); ^1H NMR ($CDCl_3$, p.p.m.): 6.55–7.66 (*m*, 7H); 20 mg of (I) was dissolved in 15 ml of an acetone–ether mixed solvent and the solution was kept at room temperature for 10 d to give colorless single crystals of (I) by slow evaporation.

Crystal data

$C_{18}H_7Cl_4NO_2\cdot C_3H_6O$
M_r = 469.12
Triclinic, $P\overline{1}$
a = 7.706 (3) Å
b = 9.007 (3) Å
c = 16.213 (6) Å
α = 94.985 (6)°
β = 91.165 (6)°
γ = 114.879 (5)°
V = 1015.0 (6) $Å^3$
Z = 2
D_x = 1.535 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 899 reflections
θ = 2.5–26.2°
μ = 0.61 mm^{-1}
T = 293 (2) K
Plate, colorless
0.40 × 0.35 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.784, T_{max} = 0.941
4239 measured reflections
3567 independent reflections
2748 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.0°
$h = -9 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -19 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.04
3567 reflections
264 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.33 e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.716 (3)	N1—C1	1.394 (3)
Cl2—C4	1.715 (2)	N1—C8	1.396 (3)
Cl3—C5	1.713 (2)	N1—C9	1.441 (3)
Cl4—C6	1.715 (3)	O3—C20	1.206 (4)
O1—C1	1.190 (3)	C19—C20	1.474 (5)
O2—C8	1.192 (3)	C20—C21	1.478 (5)
C1—N1—C8	112.9 (2)	C3—C2—C1	129.8 (2)
C1—N1—C9	122.8 (2)	C6—C7—C8	130.4 (2)
C8—N1—C9	124.2 (2)	O2—C8—N1	125.5 (2)
O1—C1—N1	125.6 (2)	O2—C8—C7	129.4 (2)
O1—C1—C2	129.5 (2)		
C1—N1—C9—C10	81.4 (3)	C1—N1—C9—C18	−98.4 (3)
C8—N1—C9—C10	−94.8 (3)	C8—N1—C9—C18	85.5 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19*A*···*Cg*1^{i}	0.96	3.21	4.101 (4)	156
C21—H21*B*···*Cg*2^{ii}	0.96	3.12	3.831 (5)	132
C21—H21*B*···*Cg*3^{ii}	0.96	3.05	3.913 (6)	151

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, 2-y, 1-z$.

H atoms were positioned geometrically and were treated as riding atoms on the parent C atoms with aromatic C—H = 0.93 Å and methyl C—H = 0.96 Å. The U_{iso}(H) values were set equal to $1.2U_{eq}$ of the carrier atom for the aromatic H atoms and at $1.5U_{eq}$ for the methyl H atoms.

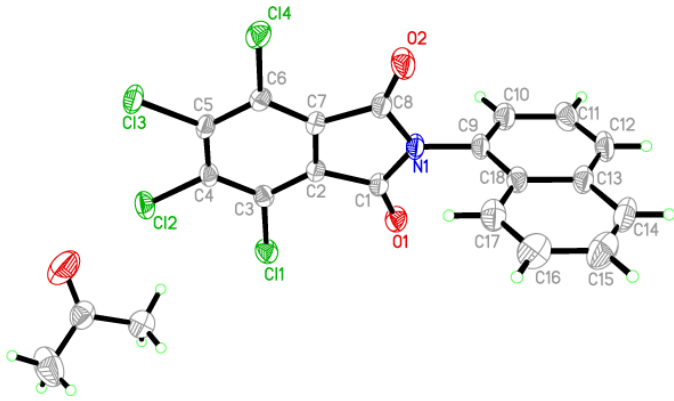


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

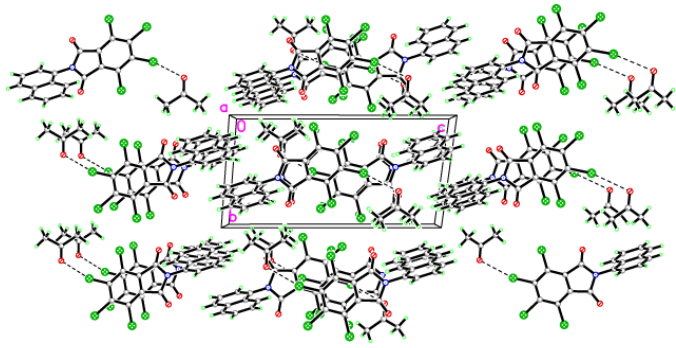


Figure 2
The molecular packing of (I), viewed down the *a* axis.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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