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Tetrafluoro-*N*-phenylphthalimide

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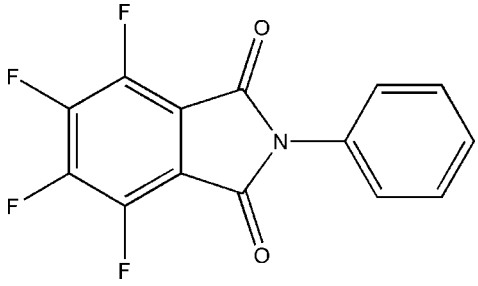
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.035; wR factor = 0.090; data-to-parameter ratio = 7.3.

In the molecule of the title compound, $C_{14}H_5F_4NO_2$, the dihedral angles between the planar rings *A* (phenyl), *B* (five-membered) and *C* (fused benzene) are *A*/*B* = 50.13 (3)°, *A*/*C* = 48.34 (3)° and *B*/*C* = 3.80 (2)°.

Related literature

For general background, see: Cai *et al.* (2006). For bond-length data, see: Allen *et al.* (1987). For related literature, see: Nowak & Lin (1991).



Experimental

Crystal data

$C_{14}H_5F_4NO_2$
M_r = 295.19
Orthorhombic, $P2_12_12_1$
a = 7.0960 (14) Å
b = 8.4060 (17) Å
c = 20.068 (4) Å
V = 1197.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 298 (2) K
0.30 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.955, T_{max} = 0.985
2348 measured reflections
1388 independent reflections
1079 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.03
1388 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: HK2269).

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supplementary materials

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Tetrafluoro-*N*-phenylphthalimide

F. Wen, R.-J. Ni and C. Yao

Comment

The title compound, (I), can be utilized to synthsize 2,3,4,5-tetrafluoro- benzoic acid, it is an important intermediate of fluoroquinolone antibiotics (Cai *et al.*, 2006). We report herein the crystal structure of the title compound, (I).

In the molecule of the title compound, (I), (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angles between the planar rings A (C1—C6), B (N/C7—C10) and C (C9—C14) are A/B = 50.13 (3)°, A/C = 48.34 (3)° and B/C = 3.80 (2)°.

Experimental

The title compound, (I), was prepared by adding tetrachloro-*N*-phenylphthalimide (100 g, 0.28 mol) to 413 g sulfolane solution of KF (193 g, 3.3 mol) at 433 K (Nowak & Lin, 1991). After stirring for 3 h, the mixture was filtered hot, the product was separated from the sulfolane by the addition of cold water (1900 ml). The yellow solid, was recrystallized by the addition of acetic acid. Then, the crystals of compound (I) were obtained by evaporating the solvent ethyl acetate slowly at room temperature for about 7 d.

Refinement

H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H atoms, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C),

Figures

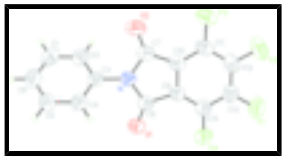

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Tetrafluoro-*N*-phenylphthalimide

Crystal data

$C_{14}H_5F_4NO_2$	F_{000} = 592
M_r = 295.19	D_x = 1.638 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 7.0960 (14) Å	θ = 10–13º

b = 8.4060 (17) Å	μ = 0.15 mm^{-1}
c = 20.068 (4) Å	T = 298 (2) K
V = 1197.0 (4) Å^3	Block, colorless
Z = 4	0.30 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.032
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.0º
T = 298(2) K	h = −8→8
ω/2θ scans	k = 0→10
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→24
T_{min} = 0.955, T_{max} = 0.985	3 standard reflections
2348 measured reflections	every 120 min
1388 independent reflections	intensity decay: none
1079 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.035	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.090	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1388 reflections	Extinction correction: none
190 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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}
F1	0.7900 (3)	1.1852 (2)	−0.18876 (8)	0.0824 (6)
F2	0.4777 (3)	1.2338 (2)	−0.11603 (10)	0.0814 (7)
F3	0.4567 (2)	1.1333 (2)	0.01142 (9)	0.0673 (5)
F4	1.0865 (3)	1.0230 (2)	−0.13876 (8)	0.0734 (6)
O1	1.2119 (3)	0.8510 (2)	−0.01010 (9)	0.0576 (5)
O2	0.6970 (3)	0.9697 (3)	0.11857 (9)	0.0606 (5)
N	0.9795 (3)	0.8960 (3)	0.06903 (10)	0.0454 (5)
C1	1.0590 (6)	0.6500 (4)	0.21979 (15)	0.0730 (10)
H1A	0.9942	0.5770	0.2460	0.088*
C2	0.9740 (5)	0.7132 (4)	0.16367 (15)	0.0605 (8)
H2A	0.8535	0.6809	0.1514	0.073*
C3	1.0692 (4)	0.8240 (3)	0.12618 (12)	0.0470 (6)
C4	1.2493 (4)	0.8701 (4)	0.14324 (14)	0.0566 (8)
H4A	1.3134	0.9445	0.1175	0.068*
C5	1.3328 (5)	0.8057 (4)	0.19844 (14)	0.0682 (9)
H5A	1.4542	0.8366	0.2102	0.082*
C6	1.2381 (6)	0.6949 (5)	0.23682 (15)	0.0754 (10)
H6A	1.2959	0.6509	0.2741	0.090*
C7	0.7977 (4)	0.9623 (3)	0.07029 (13)	0.0478 (6)
C8	1.0603 (4)	0.9040 (3)	0.00558 (13)	0.0461 (6)
C9	0.9221 (4)	0.9915 (3)	−0.03620 (13)	0.0450 (6)
C10	0.7641 (4)	1.0220 (3)	0.00212 (12)	0.0457 (6)
C11	0.6128 (4)	1.1016 (3)	−0.02440 (15)	0.0511 (7)
C12	0.6241 (4)	1.1549 (3)	−0.08918 (15)	0.0574 (8)
C13	0.7831 (5)	1.1282 (4)	−0.12682 (14)	0.0582 (8)
C14	0.9322 (4)	1.0455 (3)	−0.10040 (14)	0.0536 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0990 (15)	0.0909 (13)	0.0573 (10)	0.0218 (13)	−0.0001 (11)	0.0167 (10)
F2	0.0773 (14)	0.0777 (12)	0.0893 (14)	0.0330 (12)	−0.0168 (11)	0.0018 (11)
F3	0.0498 (9)	0.0643 (11)	0.0878 (12)	0.0148 (9)	0.0115 (9)	−0.0076 (9)
F4	0.0708 (11)	0.0915 (13)	0.0578 (9)	0.0197 (12)	0.0179 (9)	0.0110 (9)
O1	0.0524 (11)	0.0644 (12)	0.0560 (11)	0.0151 (11)	0.0074 (10)	−0.0006 (10)
O2	0.0502 (11)	0.0740 (14)	0.0576 (11)	−0.0024 (12)	0.0118 (10)	−0.0086 (10)
N	0.0443 (12)	0.0505 (12)	0.0413 (10)	−0.0034 (11)	0.0016 (10)	−0.0022 (10)
C1	0.088 (3)	0.075 (2)	0.0565 (18)	−0.005 (2)	0.0140 (17)	0.0132 (16)
C2	0.0644 (19)	0.0591 (17)	0.0579 (16)	−0.0104 (17)	0.0043 (16)	0.0007 (15)
C3	0.0492 (15)	0.0501 (15)	0.0415 (13)	−0.0017 (14)	0.0028 (12)	−0.0029 (12)
C4	0.0528 (17)	0.068 (2)	0.0487 (15)	−0.0076 (16)	0.0000 (13)	0.0009 (14)
C5	0.0546 (18)	0.097 (2)	0.0527 (16)	−0.0034 (19)	−0.0084 (15)	0.0002 (18)
C6	0.083 (2)	0.095 (3)	0.0487 (16)	0.013 (2)	0.0006 (17)	0.0082 (18)
C7	0.0450 (14)	0.0479 (15)	0.0507 (14)	−0.0069 (14)	0.0029 (13)	−0.0112 (12)

C8	0.0462 (14)	0.0445 (14)	0.0477 (14)	0.0008 (13)	0.0036 (13)	−0.0069 (12)
C9	0.0450 (14)	0.0401 (13)	0.0498 (14)	0.0021 (12)	0.0018 (12)	−0.0047 (11)
C10	0.0468 (14)	0.0376 (13)	0.0525 (14)	−0.0007 (12)	0.0042 (12)	−0.0081 (12)
C11	0.0446 (14)	0.0431 (14)	0.0656 (17)	0.0031 (14)	0.0037 (14)	−0.0120 (13)
C12	0.0617 (18)	0.0432 (16)	0.0672 (18)	0.0119 (15)	−0.0094 (15)	−0.0059 (14)
C13	0.070 (2)	0.0550 (17)	0.0494 (14)	0.0080 (17)	0.0001 (15)	−0.0001 (14)
C14	0.0577 (17)	0.0534 (16)	0.0497 (14)	0.0091 (15)	0.0065 (14)	−0.0033 (13)

Geometric parameters (Å, °)

F1—C13	1.333 (3)	C3—C4	1.379 (4)
F2—C12	1.345 (3)	C4—C5	1.368 (4)
F3—C11	1.347 (3)	C4—H4A	0.9300
F4—C14	1.352 (3)	C5—C6	1.383 (5)
O1—C8	1.206 (3)	C5—H5A	0.9300
O2—C7	1.206 (3)	C6—H6A	0.9300
N—C8	1.398 (3)	C7—C10	1.477 (4)
N—C7	1.405 (4)	C8—C9	1.485 (4)
N—C3	1.445 (3)	C9—C14	1.368 (4)
C1—C6	1.369 (5)	C9—C10	1.384 (3)
C1—C2	1.383 (5)	C10—C11	1.373 (4)
C1—H1A	0.9300	C11—C12	1.377 (4)
C2—C3	1.375 (4)	C12—C13	1.376 (4)
C2—H2A	0.9300	C13—C14	1.373 (4)
C8—N—C7	112.0 (2)	N—C7—C10	105.5 (2)
C8—N—C3	124.2 (2)	O1—C8—N	125.9 (3)
C7—N—C3	123.8 (2)	O1—C8—C9	128.6 (3)
C6—C1—C2	120.1 (3)	N—C8—C9	105.5 (2)
C6—C1—H1A	119.9	C14—C9—C10	120.3 (3)
C2—C1—H1A	119.9	C14—C9—C8	131.4 (3)
C3—C2—C1	119.4 (3)	C10—C9—C8	108.2 (2)
C3—C2—H2A	120.3	C11—C10—C9	120.6 (2)
C1—C2—H2A	120.3	C11—C10—C7	130.6 (2)
C2—C3—C4	120.7 (3)	C9—C10—C7	108.7 (2)
C2—C3—N	120.1 (3)	F3—C11—C10	122.2 (3)
C4—C3—N	119.2 (3)	F3—C11—C12	119.2 (3)
C5—C4—C3	119.4 (3)	C10—C11—C12	118.6 (3)
C5—C4—H4A	120.3	F2—C12—C13	119.6 (3)
C3—C4—H4A	120.3	F2—C12—C11	119.6 (3)
C4—C5—C6	120.5 (3)	C13—C12—C11	120.9 (3)
C4—C5—H5A	119.8	F1—C13—C14	120.9 (3)
C6—C5—H5A	119.8	F1—C13—C12	118.9 (3)
C1—C6—C5	119.9 (3)	C14—C13—C12	120.2 (3)
C1—C6—H6A	120.1	F4—C14—C9	122.2 (3)
C5—C6—H6A	120.1	F4—C14—C13	118.4 (2)
O2—C7—N	125.4 (3)	C9—C14—C13	119.4 (3)
O2—C7—C10	129.1 (3)		
C6—C1—C2—C3	−1.6 (5)	C14—C9—C10—C7	−175.3 (2)
C1—C2—C3—C4	1.3 (4)	C8—C9—C10—C7	2.5 (3)

C1—C2—C3—N	−177.4 (3)	O2—C7—C10—C11	0.5 (5)
C8—N—C3—C2	−129.0 (3)	N—C7—C10—C11	−177.8 (3)
C7—N—C3—C2	49.0 (4)	O2—C7—C10—C9	177.9 (3)
C8—N—C3—C4	52.3 (4)	N—C7—C10—C9	−0.4 (3)
C7—N—C3—C4	−129.7 (3)	C9—C10—C11—F3	180.0 (2)
C2—C3—C4—C5	−0.6 (4)	C7—C10—C11—F3	−2.9 (5)
N—C3—C4—C5	178.1 (3)	C9—C10—C11—C12	−2.1 (4)
C3—C4—C5—C6	0.2 (5)	C7—C10—C11—C12	175.1 (3)
C2—C1—C6—C5	1.2 (6)	F3—C11—C12—F2	−1.7 (4)
C4—C5—C6—C1	−0.5 (5)	C10—C11—C12—F2	−179.7 (3)
C8—N—C7—O2	179.6 (3)	F3—C11—C12—C13	178.4 (3)
C3—N—C7—O2	1.4 (4)	C10—C11—C12—C13	0.4 (4)
C8—N—C7—C10	−2.1 (3)	F2—C12—C13—F1	1.8 (4)
C3—N—C7—C10	179.7 (2)	C11—C12—C13—F1	−178.4 (3)
C7—N—C8—O1	−177.2 (3)	F2—C12—C13—C14	−178.9 (3)
C3—N—C8—O1	1.0 (5)	C11—C12—C13—C14	1.0 (5)
C7—N—C8—C9	3.5 (3)	C10—C9—C14—F4	177.7 (3)
C3—N—C8—C9	−178.3 (2)	C8—C9—C14—F4	0.5 (5)
O1—C8—C9—C14	−5.5 (5)	C10—C9—C14—C13	−1.0 (4)
N—C8—C9—C14	173.8 (3)	C8—C9—C14—C13	−178.2 (3)
O1—C8—C9—C10	177.0 (3)	F1—C13—C14—F4	−0.1 (5)
N—C8—C9—C10	−3.7 (3)	C12—C13—C14—F4	−179.4 (3)
C14—C9—C10—C11	2.4 (4)	F1—C13—C14—C9	178.6 (3)
C8—C9—C10—C11	−179.8 (2)	C12—C13—C14—C9	−0.7 (5)

Fig. 1