organic compounds

4,5,6,7-Tetrafluoro-2-(2-hydroxyphenyl)-isoindoline-1,3-dione

Shou-Xin Wang and Lai-Jin Tian*

Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China
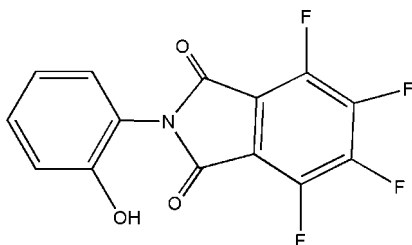
Correspondence e-mail: laijintian@163.com

The crystal structure of the title compound, $C_{14}H_5F_4NO_3$, which was synthesized by the condensation of tetrafluorophthalic anhydride with 2-aminophenol, is stabilized by an intermolecular O—H⋯O hydrogen bond. The dihedral angle between the phthalimide and hydroxyphenyl planes is 56.88 (6)°.

Related literature

For related literature, see: Allen *et al.* (1987); Cai *et al.* (2006); Collin *et al.* (2001); Fun *et al.* (2007); Li *et al.* (2007); Miyachi *et al.* (1997); Niwayama *et al.* (1996).

Experimental

Crystal data

$C_{14}H_5F_4NO_3$	V = 1231.6 (4) Å^3
M_r = 311.19	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
a = 7.1741 (14) Å	μ = 0.16 mm^{-1}
b = 8.5862 (17) Å	T = 295 (2) K
c = 19.994 (4) Å	0.36 × 0.20 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer	9661 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	1421 independent reflections
T_{min} = 0.945, T_{max} = 0.970	1357 reflections with $I > 2\sigma(I)$
	R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029	200 parameters
$wR(F^2)$ = 0.078	H-atom parameters constrained
S = 1.09	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
1421 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3⋯O1^i	0.82	1.99	2.804 (2)	175

Symmetry code: (i) $x - 1, y, z$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2541).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2002). *SADABS, SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cai, X. W., Feng, Y. Q., Qi, X. & Zhang, C. Y. (2006). *Chem. Ind. Eng.* **23**, 320–322.

Collin, X., Robert, J.-M., Wielgosz, G., Baut, G. L., Dubigeon, C. B., Grimaud, N. & Petit, J.-Y. (2001). *Eur. J. Med. Chem.* **36**, 639–649.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Fun, H.-K., Chantrapromma, S., Li, Z., Shen, Y.-M. & Xu, J.-H. (2007). *Acta Cryst.* E**63**, o946–o947.

Li, J., Liang, Z.-P. & Wang, H.-Q. (2007). *Acta Cryst.* E**63**, o627–o628.

Miyachi, H., Azuma, A., Ogasawara, A., Uchimura, E., Watanabe, N., Kobayashi, Y., Kato, F., Kato, M. & Hashimoto, H. (1997). *J. Med. Chem.* **40**, 2858–2865.

Niwayama, S., Turk, B. & Liu, J. (1996). *J. Med. Chem.* **39**, 3044–3045.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

4,5,6,7-Tetrafluoro-2-(2-hydroxyphenyl)isoindoline-1,3-dione

S.-X. Wang and L.-J. Tian

Comment

N-Substituted 2,3,4,5-tetrafluorophthalimides are an important class of compounds because of their interesting biological activities (Collin *et al.*, 2001; Miyachi *et al.*, 1997; Niwayama *et al.*, 1996). They are intermediates for the synthesis of fluoroquinolone antibiotics (Cai *et al.*, 2006).

   The bond lengths and angles of the title compound are within normal ranges (Allen *et al.*, 1987). The isoindole unit (atoms N1/C1—C8) is essentially planar, with maximum deviation of 0.0493 (21) Å for atom C8. The dihedral angle between the isoindole unit and the hydroxyphenyl ring is 56.88 (6)°, which is less than that found in 2-(2-hydroxyphenyl)isoindoline-1,3-dione [71.8 (2)°] (Li *et al.*, 2007) and 4,5,6,7-tetrachloro-2-(2-hydroxyphenyl)isoindoline-1,3-dione [63.78 (5)°] (Fun *et al.*, 2007). The crystal structure is stabilized by an intermolecular O—H⋯O hydrogen bond (Table 1).

Experimental

The title compound was prepared by the reaction of tetrafluorophthalic anhydride (1.10 g, 5 mmol) with 2-aminophenol (0.55 g, 5 mmol) in the presence of 4-methylbenzenesulfonic acid in refluxing acetic acid (30 ml). Pale yellow block-shaped single crystals were obtained by slow evaporation of an acetone solution at room temperature (yield 79.7%, m.p. 526–527 K).

Refinement

H atoms were placed at calculated positions and refined in the riding-model approximation, with C—H = 0.93 Å and $U_{iso}(H)$ = 1.2Ueq(C) for aromatic H atoms, O—H = 0.82 Å and $U_{iso}(H)$ = 1.5Ueq(O) for the hydroxyl H atom. In the absence of significant anomalous scattering effects, Friedel pairs were merged.
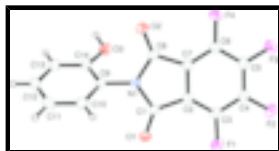
Figures

Fig. 1. The structure of (I), with displacement ellipsoids drawn at the 30% probability level.

4,5,6,7-Tetrafluoro-2-(2-hydroxyphenyl)isoindoline-1,3-dione

Crystal data

$C_{14}H_5F_4NO_3$	$F_{000} = 624$
$M_r = 311.19$	$D_x = 1.678$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation

supplementary materials

λ = 0.71073 Å

Hall symbol: P 2ac 2ab

Cell parameters from 5027 reflections

a = 7.1741 (14) Å

θ = 2.6–27.9º

b = 8.5862 (17) Å

μ = 0.16 mm^{-1}

c = 19.994 (4) Å

T = 295 (2) K

V = 1231.6 (4) Å^3

Block, pale yellow

Z = 4

0.36 × 0.20 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer

1421 independent reflections

Radiation source: fine-focus sealed tube

1357 reflections with $I > 2σ(I)$

Monochromator: graphite

R_{int} = 0.022

T = 295(2) K

$θ_{max}$ = 26.0º

φ and ω scans

$θ_{min}$ = 2.0º

Absorption correction: multi-scan (SADABS; Bruker, 2002)

h = −8→8

T_{min} = 0.945, T_{max} = 0.970

k = −10→10

9661 measured reflections

l = −24→24

Refinement

Refinement on F^2

H-atom parameters constrained

Least-squares matrix: full

$w = 1/[σ^2(F_o^2) + (0.0461P)^2 + 0.1954P]$
where $P = (F_o^2 + 2F_c^2)/3$

$R[F^2 > 2σ(F^2)] = 0.029$

$(Δ/σ)_{max}$ = 0.001

$wR(F^2)$ = 0.078

$Δρ_{max}$ = 0.15 e Å^{-3}

S = 1.09

$Δρ_{min}$ = −0.16 e Å^{-3}

1421 reflections

Extinction correction: none

200 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 > 2sigma(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 (Å²)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
O2	−0.1913 (2)	0.8342 (2)	1.01130 (8)	0.0506 (4)
C1	0.2077 (3)	0.9716 (2)	0.93266 (10)	0.0323 (4)
O1	0.30879 (19)	0.9848 (2)	0.88482 (7)	0.0418 (4)
C2	0.2369 (3)	1.0283 (2)	1.00242 (10)	0.0331 (4)
C3	0.3795 (3)	1.1116 (3)	1.02994 (11)	0.0376 (5)
C4	0.3656 (3)	1.1581 (3)	1.09631 (12)	0.0433 (5)
C5	0.2105 (4)	1.1218 (3)	1.13324 (11)	0.0456 (5)
C6	0.0671 (3)	1.0354 (3)	1.10543 (11)	0.0436 (5)
C7	0.0816 (3)	0.9886 (2)	1.04006 (10)	0.0361 (4)
C8	−0.0482 (3)	0.8983 (2)	0.99679 (10)	0.0362 (4)
C9	−0.0515 (3)	0.8314 (3)	0.87496 (10)	0.0347 (4)
C10	0.0392 (3)	0.7145 (3)	0.84035 (11)	0.0465 (5)
H10	0.1556	0.6800	0.8544	0.056*
C11	−0.0441 (4)	0.6494 (3)	0.78494 (12)	0.0581 (7)
H11	0.0171	0.5718	0.7610	0.070*
C12	−0.2181 (4)	0.6990 (3)	0.76485 (12)	0.0573 (7)
H12	−0.2748	0.6536	0.7278	0.069*
C13	−0.3084 (4)	0.8156 (3)	0.79935 (11)	0.0504 (6)
H13	−0.4260	0.8482	0.7857	0.061*
C14	−0.2247 (3)	0.8843 (3)	0.85420 (10)	0.0370 (5)
F1	0.53099 (17)	1.15288 (17)	0.99578 (7)	0.0536 (4)
F2	0.5037 (2)	1.23938 (19)	1.12395 (8)	0.0605 (4)
F3	0.1976 (3)	1.17079 (19)	1.19626 (7)	0.0649 (4)
F4	−0.0821 (2)	1.0039 (2)	1.14282 (7)	0.0665 (5)
N1	0.0331 (2)	0.9009 (2)	0.93265 (8)	0.0327 (4)
O3	−0.3012 (2)	1.0052 (2)	0.88828 (9)	0.0508 (4)
H3	−0.4151	1.0014	0.8851	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0456 (9)	0.0615 (10)	0.0448 (8)	−0.0229 (9)	0.0081 (8)	−0.0005 (8)
C1	0.0269 (9)	0.0343 (10)	0.0357 (9)	0.0038 (9)	−0.0016 (8)	0.0047 (8)
O1	0.0277 (7)	0.0606 (9)	0.0373 (7)	−0.0004 (8)	0.0044 (6)	0.0036 (7)
C2	0.0316 (9)	0.0315 (9)	0.0363 (9)	0.0013 (9)	0.0001 (8)	0.0026 (8)
C3	0.0326 (10)	0.0373 (11)	0.0431 (11)	−0.0019 (9)	0.0018 (9)	0.0028 (9)
C4	0.0407 (12)	0.0434 (12)	0.0459 (12)	−0.0069 (10)	−0.0082 (10)	−0.0049 (10)
C5	0.0536 (14)	0.0483 (12)	0.0348 (10)	−0.0058 (12)	−0.0011 (10)	−0.0049 (10)
C6	0.0447 (11)	0.0485 (12)	0.0375 (11)	−0.0113 (11)	0.0070 (9)	−0.0007 (10)
C7	0.0357 (10)	0.0351 (10)	0.0375 (10)	−0.0048 (9)	0.0012 (8)	0.0024 (9)
C8	0.0356 (10)	0.0365 (10)	0.0364 (10)	−0.0050 (9)	0.0026 (9)	0.0027 (9)
C9	0.0317 (10)	0.0408 (10)	0.0317 (9)	−0.0042 (9)	0.0008 (8)	0.0008 (8)
C10	0.0433 (12)	0.0507 (12)	0.0454 (11)	0.0071 (11)	0.0029 (10)	−0.0024 (10)
C11	0.0693 (17)	0.0600 (15)	0.0450 (13)	−0.0025 (14)	0.0124 (12)	−0.0147 (12)

C12	0.0640 (16)	0.0733 (17)	0.0347 (10)	−0.0183 (15)	−0.0031 (12)	−0.0105 (12)
C13	0.0398 (12)	0.0715 (15)	0.0400 (11)	−0.0061 (13)	−0.0083 (11)	0.0012 (12)
C14	0.0302 (10)	0.0450 (11)	0.0357 (10)	−0.0034 (9)	0.0029 (8)	0.0006 (9)
F1	0.0371 (7)	0.0655 (9)	0.0582 (8)	−0.0169 (7)	0.0095 (6)	−0.0085 (7)
F2	0.0520 (8)	0.0712 (9)	0.0582 (8)	−0.0209 (8)	−0.0087 (7)	−0.0143 (8)
F3	0.0724 (10)	0.0821 (10)	0.0402 (7)	−0.0192 (10)	0.0055 (8)	−0.0178 (7)
F4	0.0600 (9)	0.0929 (12)	0.0465 (7)	−0.0322 (9)	0.0211 (7)	−0.0149 (8)
N1	0.0252 (8)	0.0406 (9)	0.0322 (8)	0.0001 (7)	−0.0006 (6)	−0.0006 (7)
O3	0.0272 (7)	0.0599 (10)	0.0653 (10)	0.0069 (8)	−0.0039 (8)	−0.0126 (9)

Geometric parameters (Å, °)

O2—C8	1.200 (3)	C8—N1	1.409 (3)
C1—O1	1.206 (2)	C9—C10	1.382 (3)
C1—N1	1.391 (3)	C9—C14	1.386 (3)
C1—C2	1.492 (3)	C9—N1	1.434 (3)
C2—C3	1.364 (3)	C10—C11	1.377 (3)
C2—C7	1.387 (3)	C10—H10	0.9300
C3—F1	1.332 (2)	C11—C12	1.379 (4)
C3—C4	1.389 (3)	C11—H11	0.9300
C4—F2	1.332 (2)	C12—C13	1.377 (4)
C4—C5	1.371 (3)	C12—H12	0.9300
C5—F3	1.332 (3)	C13—C14	1.382 (3)
C5—C6	1.385 (3)	C13—H13	0.9300
C6—F4	1.333 (3)	C14—O3	1.358 (3)
C6—C7	1.371 (3)	O3—H3	0.8200
C7—C8	1.489 (3)		
O1—C1—N1	125.62 (19)	N1—C8—C7	105.14 (16)
O1—C1—C2	128.77 (18)	C10—C9—C14	120.6 (2)
N1—C1—C2	105.60 (16)	C10—C9—N1	120.38 (19)
C3—C2—C7	120.82 (19)	C14—C9—N1	118.98 (19)
C3—C2—C1	130.82 (19)	C11—C10—C9	119.6 (2)
C7—C2—C1	108.30 (17)	C11—C10—H10	120.2
F1—C3—C2	123.0 (2)	C9—C10—H10	120.2
F1—C3—C4	118.1 (2)	C10—C11—C12	120.1 (3)
C2—C3—C4	118.8 (2)	C10—C11—H11	120.0
F2—C4—C5	119.9 (2)	C12—C11—H11	120.0
F2—C4—C3	119.6 (2)	C13—C12—C11	120.3 (2)
C5—C4—C3	120.5 (2)	C13—C12—H12	119.9
F3—C5—C4	119.6 (2)	C11—C12—H12	119.9
F3—C5—C6	119.8 (2)	C12—C13—C14	120.2 (2)
C4—C5—C6	120.5 (2)	C12—C13—H13	119.9
F4—C6—C7	122.4 (2)	C14—C13—H13	119.9
F4—C6—C5	118.7 (2)	O3—C14—C13	123.3 (2)
C7—C6—C5	118.9 (2)	O3—C14—C9	117.53 (19)
C6—C7—C2	120.4 (2)	C13—C14—C9	119.2 (2)
C6—C7—C8	131.2 (2)	C1—N1—C8	112.31 (16)
C2—C7—C8	108.38 (18)	C1—N1—C9	124.25 (16)
O2—C8—N1	125.6 (2)	C8—N1—C9	123.37 (17)

O2—C8—C7	129.3 (2)	C14—O3—H3	109.5
O1—C1—C2—C3	1.8 (4)	C6—C7—C8—O2	−6.8 (4)
N1—C1—C2—C3	−176.8 (2)	C2—C7—C8—O2	174.6 (2)
O1—C1—C2—C7	179.0 (2)	C6—C7—C8—N1	173.8 (2)
N1—C1—C2—C7	0.4 (2)	C2—C7—C8—N1	−4.8 (2)
C7—C2—C3—F1	−179.78 (19)	C14—C9—C10—C11	−0.4 (3)
C1—C2—C3—F1	−3.0 (4)	N1—C9—C10—C11	−179.8 (2)
C7—C2—C3—C4	−0.9 (3)	C9—C10—C11—C12	−1.0 (4)
C1—C2—C3—C4	175.9 (2)	C10—C11—C12—C13	1.1 (4)
F1—C3—C4—F2	−1.1 (3)	C11—C12—C13—C14	0.3 (4)
C2—C3—C4—F2	180.0 (2)	C12—C13—C14—O3	176.4 (2)
F1—C3—C4—C5	178.5 (2)	C12—C13—C14—C9	−1.7 (3)
C2—C3—C4—C5	−0.5 (3)	C10—C9—C14—O3	−176.4 (2)
F2—C4—C5—F3	1.3 (4)	N1—C9—C14—O3	3.0 (3)
C3—C4—C5—F3	−178.3 (2)	C10—C9—C14—C13	1.7 (3)
F2—C4—C5—C6	−179.0 (2)	N1—C9—C14—C13	−178.8 (2)
C3—C4—C5—C6	1.4 (4)	O1—C1—N1—C8	177.7 (2)
F3—C5—C6—F4	0.3 (4)	C2—C1—N1—C8	−3.6 (2)
C4—C5—C6—F4	−179.3 (2)	O1—C1—N1—C9	0.6 (3)
F3—C5—C6—C7	178.8 (2)	C2—C1—N1—C9	179.30 (18)
C4—C5—C6—C7	−0.9 (4)	O2—C8—N1—C1	−174.2 (2)
F4—C6—C7—C2	177.9 (2)	C7—C8—N1—C1	5.2 (2)
C5—C6—C7—C2	−0.5 (3)	O2—C8—N1—C9	2.9 (3)
F4—C6—C7—C8	−0.6 (4)	C7—C8—N1—C9	−177.65 (18)
C5—C6—C7—C8	−179.0 (2)	C10—C9—N1—C1	56.7 (3)
C3—C2—C7—C6	1.4 (3)	C14—C9—N1—C1	−122.8 (2)
C1—C2—C7—C6	−176.1 (2)	C10—C9—N1—C8	−120.0 (2)
C3—C2—C7—C8	−179.76 (19)	C14—C9—N1—C8	60.5 (3)
C1—C2—C7—C8	2.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3⋯O1[i]	0.82	1.99	2.804 (2)	175

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1