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N-(3,4-Difluorophenyl)phthalimide

Xian-Shu Fu, Xiao-Ping Yu, Wei-Min Wang* and Fang Lin

College of Life Sciences, China Jiliang University, Hangzhou 310018, People's Republic of China

Correspondence e-mail: clshangzhou@yahoo.com.cn

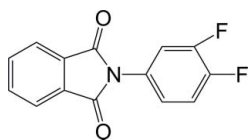
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 Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.115; data-to-parameter ratio = 11.1.

In the title compound, $\text{C}_{14}\text{H}_7\text{F}_2\text{NO}_2$, the phthalimide ring system is nearly planar [maximum atomic deviation = 0.028 (1) Å] and it is twisted with respect to the attached benzene ring, making a dihedral angle of 55.70 (6)°. Weak intermolecular $\text{C}-\text{H}\cdots\text{F}$ hydrogen bonds are 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 the synthesis, see: Valkonen *et al.* (2007); Barchin *et al.* (2002). For a related structure, see: Xu *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_7\text{F}_2\text{NO}_2$
 $M_r = 259.21$

 Orthorhombic, *Pbca*
 $a = 15.101$ (3) Å

 $b = 5.8093$ (12) Å

 $c = 24.866$ (5) Å

 $V = 2181.4$ (8) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.13$ mm⁻¹
 $T = 113$ K

 $0.20 \times 0.10 \times 0.08$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer

 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)

 $T_{\min} = 0.975$, $T_{\max} = 0.990$

14468 measured reflections

1920 independent reflections

 1780 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.115$
 $S = 1.03$

1920 reflections

173 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.06$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4}\cdots\text{F2}^i$	0.95	2.47	3.317 (2)	149
$\text{C5}-\text{H5}\cdots\text{F2}^{ii}$	0.95	2.54	3.321 (2)	139

 Symmetry codes: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2786).

References

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supporting information

Acta Cryst. (2010). E66, o1809 [doi:10.1107/S1600536810024189]

N-(3,4-Difluorophenyl)phthalimide

Xian-Shu Fu, Xiao-Ping Yu, Wei-Min Wang and Fang Lin

S1. 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 phthalimide ring system is nearly planar [maximum atomic deviation 0.028 (1) Å for N1 atom] and the dihedral angle between the benzene ring and the phthalimide plane is 55.70 (6)°, which is similar to 59.95 (4)° found in a related compound *N*-(2-fluorophenyl)phthalimide (Xu *et al.*, 2006). Weak intermolecular C—H...F hydrogen bonding is present in the crystal structure (Table 1).

S2. Experimental

An acetic acid solution of phthalic anhydride (14.8 g, 100 mmol) and 3,4-difluoroaniline (9.91 ml, 100 mmol) was refluxed overnight, and then filtered. The crude product was recrystallized from ethyl acetate.

S3. Refinement

H atoms were positioned geometrically and refined as riding with C—H = 0.95 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The (002) and (102) reflections were omitted in the refinement.

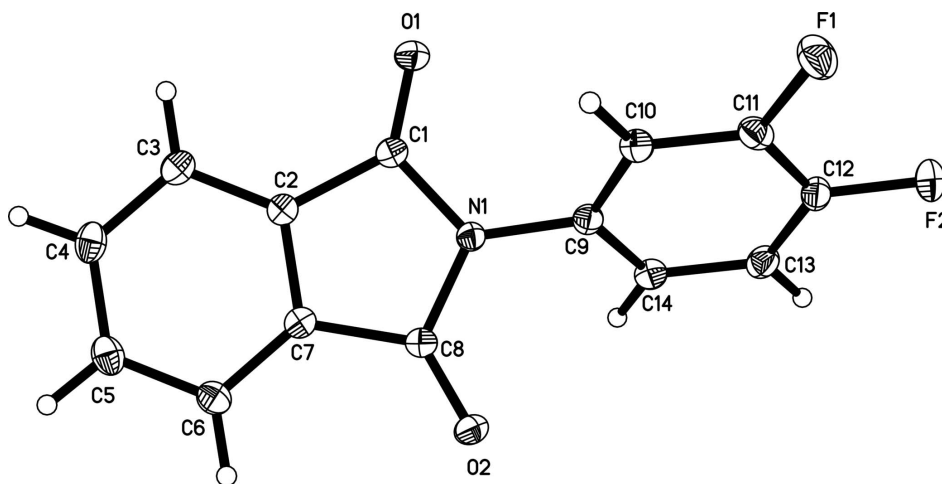


Figure 1

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

N*-(3,4-Difluorophenyl)phthalimideCrystal data*C₁₄H₇F₂NO₂ $M_r = 259.21$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 15.101 (3) \text{ \AA}$ $b = 5.8093 (12) \text{ \AA}$ $c = 24.866 (5) \text{ \AA}$ $V = 2181.4 (8) \text{ \AA}^3$ $Z = 8$ $F(000) = 1056$ $D_x = 1.579 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5269 reflections

 $\theta = 1.6\text{--}27.9^\circ$ $\mu = 0.13 \text{ mm}^{-1}$ $T = 113 \text{ K}$

Prism, colorless

 $0.20 \times 0.10 \times 0.08 \text{ mm}$ *Data collection*Rigaku Saturn CCD area-detector
diffractometer

Radiation source: rotating anode

Confocal monochromator

Detector resolution: 7.31 pixels mm^{-1} ω and ϕ scans

Absorption correction: multi-scan

(CrystalClear; Rigaku, 2005) $T_{\min} = 0.975$, $T_{\max} = 0.990$

14468 measured reflections

1920 independent reflections

1780 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.040$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.6^\circ$ $h = -17 \rightarrow 17$ $k = -6 \rightarrow 6$ $l = -29 \rightarrow 20$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.040$ $wR(F^2) = 0.115$ $S = 1.03$

1920 reflections

173 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.7186P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.06 \text{ e \AA}^{-3}$ Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.046 (4)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.18581 (6)	0.83152 (17)	0.21976 (4)	0.0398 (3)
F2	0.12250 (7)	0.45784 (18)	0.17125 (4)	0.0409 (3)
O1	0.04193 (7)	0.85824 (18)	0.40561 (4)	0.0289 (3)

O2	0.19778 (7)	0.17995 (18)	0.40802 (4)	0.0282 (3)
N1	0.11557 (7)	0.5119 (2)	0.39257 (5)	0.0217 (3)
C1	0.08090 (9)	0.6931 (2)	0.42336 (6)	0.0221 (4)
C2	0.10111 (9)	0.6332 (3)	0.48041 (6)	0.0223 (4)
C3	0.08065 (10)	0.7479 (3)	0.52755 (6)	0.0277 (4)
H3	0.0499	0.8905	0.5271	0.033*
C4	0.10706 (10)	0.6458 (3)	0.57568 (6)	0.0315 (4)
H4	0.0942	0.7206	0.6087	0.038*
C5	0.15184 (10)	0.4367 (3)	0.57625 (6)	0.0314 (4)
H5	0.1688	0.3708	0.6097	0.038*
C6	0.17221 (10)	0.3224 (3)	0.52856 (6)	0.0276 (4)
H6	0.2028	0.1794	0.5287	0.033*
C7	0.14620 (9)	0.4251 (3)	0.48107 (6)	0.0225 (4)
C8	0.15903 (9)	0.3464 (2)	0.42484 (6)	0.0218 (4)
C9	0.11494 (9)	0.5016 (3)	0.33528 (6)	0.0232 (4)
C10	0.15100 (9)	0.6824 (3)	0.30585 (6)	0.0251 (4)
H10	0.1744	0.8147	0.3232	0.030*
C11	0.15153 (9)	0.6626 (3)	0.25060 (6)	0.0276 (4)
C12	0.11839 (10)	0.4696 (3)	0.22544 (6)	0.0284 (4)
C13	0.08237 (10)	0.2919 (3)	0.25441 (6)	0.0297 (4)
H13	0.0591	0.1601	0.2367	0.036*
C14	0.08046 (9)	0.3082 (3)	0.31032 (6)	0.0262 (4)
H14	0.0557	0.1874	0.3312	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0481 (6)	0.0388 (7)	0.0325 (6)	-0.0077 (5)	0.0028 (4)	0.0079 (4)
F2	0.0557 (7)	0.0463 (7)	0.0206 (5)	0.0059 (5)	-0.0013 (4)	-0.0034 (4)
O1	0.0296 (6)	0.0255 (6)	0.0315 (6)	0.0052 (4)	-0.0016 (4)	0.0006 (4)
O2	0.0294 (6)	0.0246 (6)	0.0305 (6)	0.0059 (4)	-0.0002 (4)	-0.0033 (4)
N1	0.0215 (6)	0.0225 (7)	0.0211 (7)	0.0022 (5)	-0.0005 (4)	-0.0009 (5)
C1	0.0179 (7)	0.0223 (8)	0.0262 (8)	-0.0003 (6)	0.0012 (5)	-0.0009 (6)
C2	0.0189 (7)	0.0235 (8)	0.0247 (8)	-0.0018 (6)	0.0008 (6)	-0.0007 (6)
C3	0.0241 (7)	0.0277 (9)	0.0313 (8)	-0.0013 (6)	0.0036 (6)	-0.0033 (7)
C4	0.0322 (8)	0.0381 (10)	0.0242 (8)	-0.0065 (7)	0.0071 (6)	-0.0039 (7)
C5	0.0334 (8)	0.0366 (10)	0.0242 (8)	-0.0064 (7)	0.0018 (6)	0.0054 (7)
C6	0.0273 (8)	0.0272 (8)	0.0282 (8)	-0.0022 (7)	0.0012 (6)	0.0048 (6)
C7	0.0198 (7)	0.0217 (8)	0.0260 (8)	-0.0027 (6)	0.0018 (6)	-0.0007 (6)
C8	0.0188 (7)	0.0208 (8)	0.0257 (8)	-0.0012 (5)	-0.0009 (6)	0.0003 (6)
C9	0.0194 (7)	0.0265 (8)	0.0236 (8)	0.0034 (6)	-0.0015 (5)	-0.0016 (6)
C10	0.0235 (7)	0.0249 (8)	0.0270 (8)	-0.0010 (6)	-0.0021 (6)	-0.0007 (6)
C11	0.0254 (7)	0.0289 (9)	0.0287 (8)	0.0021 (6)	0.0025 (6)	0.0061 (7)
C12	0.0293 (8)	0.0344 (9)	0.0217 (8)	0.0080 (7)	-0.0011 (6)	-0.0024 (6)
C13	0.0301 (8)	0.0289 (9)	0.0300 (8)	0.0032 (7)	-0.0042 (6)	-0.0070 (7)
C14	0.0248 (7)	0.0248 (8)	0.0289 (8)	0.0006 (6)	-0.0001 (6)	-0.0007 (6)

Geometric parameters (Å, °)

F1—C11	1.3486 (18)	C5—C6	1.394 (2)
F2—C12	1.3507 (17)	C5—H5	0.9500
O1—C1	1.2088 (17)	C6—C7	1.380 (2)
O2—C8	1.2051 (18)	C6—H6	0.9500
N1—C1	1.4032 (18)	C7—C8	1.484 (2)
N1—C8	1.4139 (18)	C9—C14	1.385 (2)
N1—C9	1.4257 (19)	C9—C10	1.391 (2)
C1—C2	1.4923 (19)	C10—C11	1.379 (2)
C2—C3	1.383 (2)	C10—H10	0.9500
C2—C7	1.388 (2)	C11—C12	1.378 (2)
C3—C4	1.394 (2)	C12—C13	1.371 (2)
C3—H3	0.9500	C13—C14	1.394 (2)
C4—C5	1.390 (3)	C13—H13	0.9500
C4—H4	0.9500	C14—H14	0.9500
C1—N1—C8	111.94 (12)	C2—C7—C8	108.76 (12)
C1—N1—C9	125.04 (12)	O2—C8—N1	125.03 (13)
C8—N1—C9	122.80 (12)	O2—C8—C7	129.63 (13)
O1—C1—N1	125.29 (13)	N1—C8—C7	105.34 (12)
O1—C1—C2	129.18 (13)	C14—C9—C10	121.58 (15)
N1—C1—C2	105.51 (12)	C14—C9—N1	118.96 (14)
C3—C2—C7	121.30 (14)	C10—C9—N1	119.44 (13)
C3—C2—C1	130.35 (14)	C11—C10—C9	117.63 (14)
C7—C2—C1	108.34 (12)	C11—C10—H10	121.2
C2—C3—C4	117.29 (15)	C9—C10—H10	121.2
C2—C3—H3	121.4	F1—C11—C10	120.55 (14)
C4—C3—H3	121.4	F1—C11—C12	118.25 (14)
C5—C4—C3	121.32 (14)	C10—C11—C12	121.19 (14)
C5—C4—H4	119.3	F2—C12—C13	120.28 (14)
C3—C4—H4	119.3	F2—C12—C11	118.50 (14)
C4—C5—C6	121.00 (15)	C13—C12—C11	121.21 (14)
C4—C5—H5	119.5	C12—C13—C14	118.75 (14)
C6—C5—H5	119.5	C12—C13—H13	120.6
C7—C6—C5	117.33 (15)	C14—C13—H13	120.6
C7—C6—H6	121.3	C9—C14—C13	119.63 (14)
C5—C6—H6	121.3	C9—C14—H14	120.2
C6—C7—C2	121.76 (14)	C13—C14—H14	120.2
C6—C7—C8	129.48 (14)		
C8—N1—C1—O1	178.83 (13)	C9—N1—C8—C7	178.07 (12)
C9—N1—C1—O1	4.1 (2)	C6—C7—C8—O2	-3.1 (3)
C8—N1—C1—C2	-2.26 (14)	C2—C7—C8—O2	176.76 (14)
C9—N1—C1—C2	-177.01 (12)	C6—C7—C8—N1	177.27 (14)
O1—C1—C2—C3	0.4 (3)	C2—C7—C8—N1	-2.88 (15)
N1—C1—C2—C3	-178.49 (14)	C1—N1—C9—C14	-127.38 (15)
O1—C1—C2—C7	179.19 (14)	C8—N1—C9—C14	58.42 (17)

N1—C1—C2—C7	0.34 (15)	C1—N1—C9—C10	54.38 (18)
C7—C2—C3—C4	-0.1 (2)	C8—N1—C9—C10	-119.82 (15)
C1—C2—C3—C4	178.61 (14)	C14—C9—C10—C11	-0.1 (2)
C2—C3—C4—C5	-0.2 (2)	N1—C9—C10—C11	178.06 (12)
C3—C4—C5—C6	0.3 (2)	C9—C10—C11—F1	-179.79 (12)
C4—C5—C6—C7	0.0 (2)	C9—C10—C11—C12	-0.7 (2)
C5—C6—C7—C2	-0.4 (2)	F1—C11—C12—F2	0.6 (2)
C5—C6—C7—C8	179.47 (14)	C10—C11—C12—F2	-178.55 (13)
C3—C2—C7—C6	0.4 (2)	F1—C11—C12—C13	-179.79 (13)
C1—C2—C7—C6	-178.56 (13)	C10—C11—C12—C13	1.1 (2)
C3—C2—C7—C8	-179.47 (13)	F2—C12—C13—C14	179.01 (13)
C1—C2—C7—C8	1.58 (16)	C11—C12—C13—C14	-0.6 (2)
C1—N1—C8—O2	-176.48 (13)	C10—C9—C14—C13	0.6 (2)
C9—N1—C8—O2	-1.6 (2)	N1—C9—C14—C13	-177.63 (12)
C1—N1—C8—C7	3.18 (14)	C12—C13—C14—C9	-0.2 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C4—H4...F2 ⁱ	0.95	2.47	3.317 (2)	149
C5—H5...F2 ⁱⁱ	0.95	2.54	3.321 (2)	139

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