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N′-(2-Fluorobenzoyl)benzohydrazide

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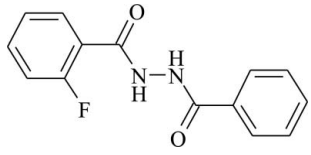
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.034; wR factor = 0.095; data-to-parameter ratio = 10.2.

In the crystal structure of the title compound, $C_{14}H_{11}FN_2O_2$, the molecule is centrosymmetric. The F atom is disordered over four positions, on the two ortho positions of each ring, with occupancies of 0.287:0.213 (5). In the crystal structure, molecules are linked by intermolecular N—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Silva *et al.* (2006); Chopra *et al.* (2006); de Souza *et al.* (2007); Ahmad *et al.* (2001); Al-Soud, *et al.* (2004); Al-Talib *et al.* (1990); El-Emam *et al.* (2004); Yousif *et al.* (1986); Zareef & Iqbal (2007); Zheng *et al.* (2003).



Experimental

Crystal data

$C_{14}H_{11}FN_2O_2$
M_r = 258.25
Monoclinic, $P2_1/c$
a = 4.7698 (10) Å
b = 5.2435 (10) Å
c = 23.913 (5) Å
β = 100.89 (3)°
V = 587.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 90.0 (1) K
0.25 × 0.20 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: none
3819 measured reflections
1205 independent reflections
1060 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.07
1205 reflections
118 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.84 (2)	2.05 (2)	2.8549 (16)	160.4 (16)
N1—H1···O3^{ii}	0.84 (2)	2.325 (16)	2.6302 (14)	101.8 (14)
C8—H8···O3^{iii}	0.945 (17)	2.416 (16)	3.2687 (17)	150.0 (12)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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*.

The authors are grateful to the late Professor Dr Rashid Iqbal for his great contribution to this research project.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2073).

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supplementary materials

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N'-(2-Fluorobenzoyl)benzohydrazide

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Comment

N,*N*'-Diacylhydrazines are very important intermediates especially for the synthesis of various biological active five member heterocyclic compounds such as 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In view of the versatility of these compounds, we have synthesized the title compound, (I), using a literature method (Zareef *et al.*, 2007) and reported its crystal structure. The geometry of (I) is normal and (Table 1) compares well with those found in other crystal structures (Silva *et al.*, 2006; Chopra *et al.*, 2006; Souza *et al.*, 2007). The title molecule, $C_{14}H_{11}N_2O_2F$, is non-planar. The dihedral angle between the benzene rings and CONHNHCO group is 34.5 (5) °. The disorder of the title molecule is realised by the presents of two positions for F atom with occupancy factors of 0.3 for F10 and 0.2 for F10'. The molecules are linked into a three-dimensional framework by a combination of two N–H···O and one weak C–H···O hydrogen bonds.

Experimental

For the synthesis of title compound (I), benzoyl chloride (5.1 mmol) was added in portions to a suspension of 2-fluorobenzoic hydrazide (5.0 mmol) in dry acetonitrile (50 ml), and the reaction mixture was stirred for 9 h at 296 K. Then, the resulting mixture was concentrated, and the solid product filtered and recrystallized from aqueous ethanol to afford the title compound (yield; 87%). Suitable crystals were grown from a solution of (I) in ethanol by slow evaporation at room temperature.

Refinement

The occupancy factors for the disordered fluorine and hydrogen (H5 and H9) atoms were refined using free variables. The H5 nad H9 were included in the refinement at geometrically idealized positions with C-H distances 0.96 A and their parameters are not refinement. The remaining H atoms were located in a difference map and freely refined.

Figures

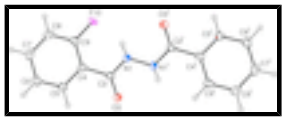

Fig. 1. View of the title molecule with anisotropic displacement parameters shown at the 50% probability level. [Symmetry code: (i) -x, -y, -z].

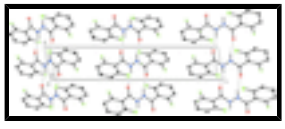

Fig. 2. The packing diagram of the title compound. Dashed lines indicate hydrogen bonds.

N'-(2-Fluorobenzoyl)benzohydrazide

Crystal data

$C_{14}H_{11}FN_2O_2$	$F(000) = 268$
$M_r = 258.25$	$D_x = 1.460$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1205 reflections
$a = 4.7698$ (10) Å	$\theta = 3.5$–$26.5°$
$b = 5.2435$ (10) Å	$\mu = 0.11$ mm^{-1}
$c = 23.913$ (5) Å	$T = 90$ K
$\beta = 100.89$ (3)°	Plate, colourless
$V = 587.3$ (2) Å^3	0.25 × 0.20 × 0.10 mm
$Z = 2$	

Data collection

Oxford Diffraction Xcalibur diffractometer	1060 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.044$
graphite	$\theta_{max} = 26.5°$, $\theta_{min} = 3.5°$
Detector resolution: 1024 x 1024 with blocks 2 x 2 pixels mm^{-1}	$h = -5 \rightarrow 5$
ω scans	$k = -4 \rightarrow 6$
3819 measured reflections	$l = -30 \rightarrow 30$
1205 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.094$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 1.578P]$ where $P = (F_o^2 + 2F_c^2)/3$
1205 reflections	$(\Delta/\sigma)_{max} < 0.001$
118 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds

in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}	Occ. (<1)
N1	0.4363 (2)	0.5857 (2)	0.47949 (4)	0.0183 (3)	
C2	0.5853 (2)	0.6622 (2)	0.44055 (5)	0.0166 (3)	
O3	0.83154 (17)	0.58840 (19)	0.44180 (4)	0.0255 (3)	
C4	0.4404 (2)	0.8420 (2)	0.39624 (5)	0.0163 (3)	
C5	0.5167 (3)	0.8362 (2)	0.34288 (5)	0.0193 (3)	
H5	0.6561	0.7149	0.3356	0.023*	0.77 (6)
C6	0.3958 (3)	1.0022 (3)	0.30033 (5)	0.0228 (3)	
C7	0.1997 (3)	1.1812 (2)	0.31099 (5)	0.0237 (3)	
C8	0.1226 (3)	1.1921 (2)	0.36398 (6)	0.0235 (3)	
C9	0.2420 (2)	1.0224 (2)	0.40602 (5)	0.0189 (3)	
H9	0.1862	1.0290	0.4425	0.023*	0.73 (6)
F10	0.1593 (5)	1.0332 (5)	0.45796 (13)	0.0224 (10)	0.287 (5)
F10'	0.6956 (8)	0.6688 (8)	0.33028 (14)	0.0240 (13)	0.213 (5)
H1	0.258 (4)	0.605 (3)	0.4759 (7)	0.037 (5)*	
H6	0.456 (3)	0.995 (3)	0.2630 (7)	0.030 (4)*	
H7	0.114 (3)	1.296 (3)	0.2808 (7)	0.034 (4)*	
H8	−0.007 (3)	1.315 (3)	0.3730 (6)	0.030 (4)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0108 (5)	0.0275 (6)	0.0168 (5)	0.0041 (4)	0.0032 (4)	0.0053 (4)
C2	0.0118 (6)	0.0233 (6)	0.0149 (5)	0.0003 (4)	0.0029 (4)	−0.0014 (4)
O3	0.0128 (5)	0.0416 (6)	0.0234 (5)	0.0072 (4)	0.0066 (3)	0.0104 (4)
C4	0.0112 (5)	0.0201 (6)	0.0170 (6)	−0.0023 (4)	0.0011 (4)	−0.0004 (4)
C5	0.0167 (6)	0.0225 (6)	0.0185 (6)	0.0016 (5)	0.0030 (5)	−0.0002 (5)
C6	0.0233 (7)	0.0274 (7)	0.0171 (6)	−0.0013 (5)	0.0028 (5)	0.0017 (5)
C7	0.0205 (6)	0.0231 (6)	0.0251 (6)	−0.0002 (5)	−0.0019 (5)	0.0073 (5)
C8	0.0192 (6)	0.0191 (6)	0.0324 (7)	0.0032 (5)	0.0052 (5)	0.0007 (5)
C9	0.0171 (6)	0.0202 (6)	0.0200 (6)	−0.0013 (5)	0.0050 (5)	−0.0017 (4)
F10	0.0267 (15)	0.0234 (14)	0.0195 (16)	0.0052 (10)	0.0104 (10)	−0.0014 (10)
F10'	0.026 (2)	0.029 (2)	0.0184 (18)	0.0116 (15)	0.0068 (14)	0.0018 (13)

Geometric parameters (Å, °)

N1—C2	1.3354 (16)	C6—C7	1.3824 (19)
N1—N1^{i}	1.384 (2)	C6—H6	0.989 (15)

N1—H1	0.84 (2)	C7—C8	1.3856 (19)
C2—O3	1.2315 (14)	C7—H7	0.971 (17)
C2—C4	1.4875 (16)	C8—C9	1.3822 (18)
C4—C9	1.3890 (17)	C8—H8	0.945 (17)
C4—C5	1.3916 (17)	C9—F10	1.373 (4)
C5—F10'	1.299 (5)	C9—H9	0.9600
C5—C6	1.3797 (18)	F10—H9	0.4136
C5—H5	0.9600	F10'—H5	0.3448
C2—N1—N1^{i}	117.97 (12)	C5—C6—C7	119.69 (12)
C2—N1—H1	123.5 (11)	C5—C6—H6	119.3 (10)
N1^{i}—N1—H1	116.7 (12)	C7—C6—H6	121.0 (10)
O3—C2—N1	121.31 (11)	C6—C7—C8	120.16 (12)
O3—C2—C4	121.94 (11)	C6—C7—H7	119.4 (10)
N1—C2—C4	116.75 (10)	C8—C7—H7	120.4 (10)
C9—C4—C5	118.28 (11)	C9—C8—C7	119.64 (12)
C9—C4—C2	123.46 (11)	C9—C8—H8	118.0 (9)
C5—C4—C2	118.20 (10)	C7—C8—H8	122.3 (9)
F10'—C5—C6	117.16 (18)	F10—C9—C8	118.83 (14)
F10'—C5—C4	121.64 (17)	F10—C9—C4	120.10 (14)
C6—C5—C4	121.15 (11)	C8—C9—C4	121.07 (11)
C6—C5—H5	119.4	C8—C9—H9	119.4
C4—C5—H5	119.5	C4—C9—H9	119.5
N1^{i}—N1—C2—O3	−1.9 (2)	F10'—C5—C6—C7	178.7 (2)
N1^{i}—N1—C2—C4	178.67 (12)	C4—C5—C6—C7	1.29 (19)
O3—C2—C4—C9	−147.25 (13)	C5—C6—C7—C8	−0.64 (19)
N1—C2—C4—C9	32.16 (16)	C6—C7—C8—C9	−0.29 (19)
O3—C2—C4—C5	29.97 (17)	C7—C8—C9—F10	−178.88 (16)
N1—C2—C4—C5	−150.62 (11)	C7—C8—C9—C4	0.61 (19)
C9—C4—C5—F10'	−178.3 (2)	C5—C4—C9—F10	179.49 (16)
C2—C4—C5—F10'	4.3 (3)	C2—C4—C9—F10	−3.3 (2)
C9—C4—C5—C6	−0.97 (18)	C5—C4—C9—C8	0.01 (18)
C2—C4—C5—C6	−178.33 (11)	C2—C4—C9—C8	177.23 (11)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{ii}	0.84 (2)	2.05 (2)	2.8549 (16)	160.4 (16)
N1—H1···O3^{i}	0.84 (2)	2.325 (16)	2.6302 (14)	101.8 (14)
C8—H8···O3^{iii}	0.945 (17)	2.416 (16)	3.2687 (17)	150.0 (12)

Symmetry codes: (ii) *x*−1, *y*, *z*; (i) −*x*+1, −*y*+1, −*z*+1; (iii) *x*−1, *y*+1, *z*.

Fig. 1

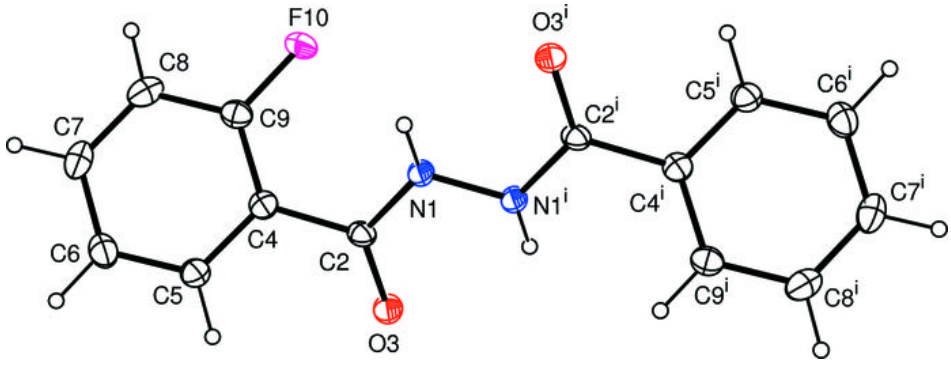

Fig. 2