Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-*N*′-Hydroxy-4-(trifluoromethyl)-benzimidamide

Fei Liu,[a]* Fang Zhang,[a] Qifan Chen[b] and Mingdong Dong[b]

[a]College of Chemical Engineering & Materials, Eastern Liaoning University, No. 325 Wenhua Road, Yuanbao District, Dandong City, Liaoning Province 118003, People's Republic of China, and [b]Experiment Center, Eastern Liaoning University, No. 325 Wenhua Road, Yuanbao District, Dandong City, Liaoning Province 118003, People's Republic of China
Correspondence e-mail: berylliu8090@sina.com

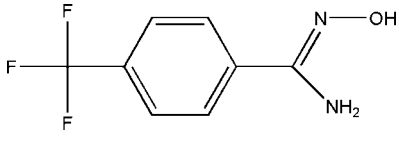
Received 10 February 2011; accepted 10 February 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.056; wR factor = 0.183; data-to-parameter ratio = 12.5.

In the title compound, $C_8H_7F_3N_2O$, the OH and NH_2 substituents adopt a Z configuration with respect to the C=N bond. The hydroxyimidamide unit is almost planar (r.m.s. deviation = 0.007 Å) and subtends an angle of 26.25 (13)° with the benzene ring. The F atoms of the trifluoromethyl substituent are disordered over two sets of sites with an occupancy ratio of 0.783 (15):0.217 (15). In the crystal, O—H···N hydrogen bonds form centrosymmetric dimers. Additional N—H···O hydrogen bonds link the dimers into zigzag chains along the b axis. Weak intermolecular F···F contacts of 2.714 (5) Å are also observed.

Related literature

For the preparation of the title compound, see: Rai *et al.* (2010). For the use of oxime derivatives in crystal engineering, see: Aakeröy *et al.* (2000). For a related structure, see: Orama & Saarinen (1996).



Experimental

Crystal data

$C_8H_7F_3N_2O$
M_r = 204.16
Monoclinic, $P2_1/c$
a = 9.8706 (8) Å
b = 11.2540 (12) Å
c = 8.4033 (7) Å
β = 104.61 (2)°
V = 903.29 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.32 × 0.24 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.946, T_{max} = 0.972
8605 measured reflections
2058 independent reflections
1324 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.183
S = 1.07
2058 reflections
164 parameters
42 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.84 (3)	2.36 (3)	3.165 (3)	161 (3)
O1—H1*O*···N2^{ii}	0.86 (3)	1.98 (3)	2.766 (2)	152 (3)

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

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors gratefully acknowledge financial support from the Education Department of Liaoning Province (2009 A 265) and Liaoning University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5105).

References

Aakeröy, C. B., Beatty, A. M. & Leinen, D. S. (2000). *CrystEngComm*, **2**, 145–150.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Orama, M. & Saarinen, H. (1996). *Acta Chem. Scand.* **50**, 1087–1091.
Rai, N. P., Narayanaswamy, V. K., Govender, T., Manuprasad, B. K., Shashikanth, S. & Arunachalam, P. N. (2010). *Eur. J. Med. Chem.* **45**, 2677–2682.
Rigaku (2006). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2007). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan, and Rigaku Americas, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o859 [doi:10.1107/S1600536811005022]

(*Z*)-*N*′-Hydroxy-4-(trifluoromethyl)benzimidamide

Fei Liu, Fang Zhang, Qifan Chen and Mingdong Dong

S1. Comment

The oxime functionality is well known in organic synthesis, analytical chemistry, and coordination chemistry, yet it has remained relatively unexplored as an intermolecular connector in crystal engineering (Aakeröy *et al.*, 2000).

In the title compound, the oxime also carries an amine substituent and assumes a *Z* configuration with respect to the C8═N2 bond (Fig. 1). Atoms F1A:F3B, F2A:F1B, F3A: F2B are disordered over two positions and with site occupancies of 0.5:0.5. The C8,N1,N2,O1 hydroxyimidamide unit is almost planar (r.m.s. deviation 0.007 Å) and subtends an angle of 26.25 (13)° to the C2···C7 benzene ring. The torsion angle O1—N2—C8—C5 between the oxime unit and the ring system is -177.71 (15)°. In the crystal O1–H1O···N1 hydrogen bonds form centrosymmetric dimers. An additional N1–H1N···O1 hydrogen bond links these dimers into zigzag chains along *b*. Weak intermolecular F2A···F2A^{iii} contacts, 2.714 (5) Å, (^{iii} = -*x*, 1-*y*, -*z*) are also observed (Fig. 2).

S2. Experimental

The compound was prepared by a reported procedure (Rai *et al.*, 2010) To a solution of 4-(trifluoromethyl)benzonitrile (0.2 mol) in ethanol (20 mL) was added hydroxylamine hydrochloride (0.4 mol) in water (40 mL). Then anhydrous sodium carbonate(0.4 mol) in water (120 mL) was slowly added to the resulting solution and the mixture was stirred at 358k for 5 h and then concentrated under vacuum to evaporate some water. The resulting suspension was filtered, the solid that formed was washed with cold water and dried under vacuum. Block-shaped crystals suitable for X-ray diffraction were obtained from methanol.

S3. Refinement

H atoms bound to N and O were located in difference Fourier maps and refined isotropically with U_{iso}(H) = 1.2U_{eq}(N) [1.5U_{eq}(O)]. H atoms attached to C were added at their calculated positions and included in the structure factor calculations, with C—H = 0.93Å (aromatic) and 0.97 Å (methylene), and with U_{iso}(H) = 1.2U_{eq}(C). The F atoms of the CF_3 group were disordered over two positions. Occupancy was fixed at 0.5 for each component in the final refinement cycles.

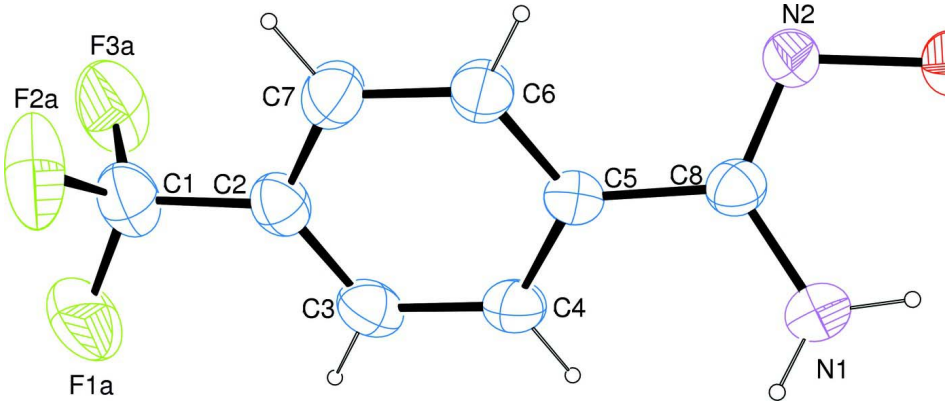


Figure 1

Structure of the title compound with 50% probability displacement ellipsoids. For clarity, only one of the two equivalent disorder components is shown.

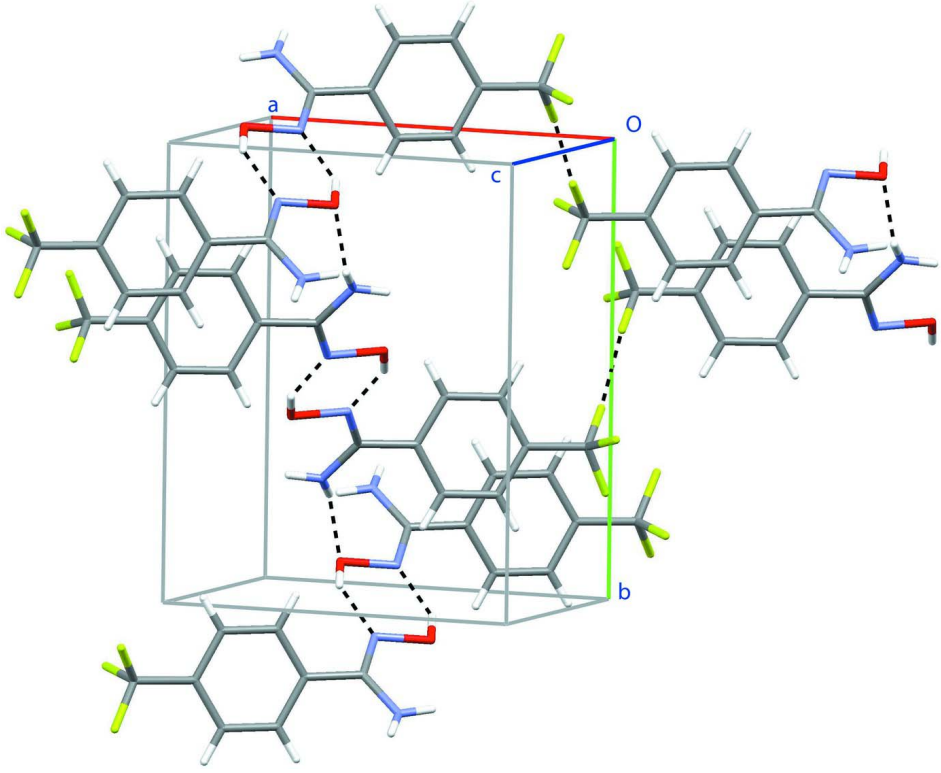


Figure 2

Crystal packing of the title compound viewed down the *c* axis. Hydrogen bonds and F···F contacts are drawn as dashed lines.

N′-hydroxy-4-(trifluoromethyl)benzene-1-carboximidamide

Crystal data

$C_8H_7F_3N_2O$
M_r = 204.16
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.8706 (8) Å
b = 11.2540 (12) Å

c = 8.4033 (7) Å
β = 104.61 (2)°
V = 903.29 (16) Å^3
Z = 4
F(000) = 416
D_x = 1.501 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 4741 reflections
θ = 3.1–27.4°
μ = 0.14 mm^{-1}
T = 293 K
Irregular block, colorless
0.32 × 0.24 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.946, T_{max} = 0.972

8605 measured reflections
2058 independent reflections
1324 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 27.4°, θ_{min} = 3.1°
h = −12→12
k = −14→14
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.183
S = 1.07
2058 reflections
164 parameters
42 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.101P)^2 + 0.058P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	−0.0030 (2)	0.6553 (2)	0.2394 (3)	0.0759 (7)	
F1A	−0.0355 (6)	0.7584 (3)	0.1633 (6)	0.1154 (16)	0.783 (15)
F2A	−0.0071 (5)	0.5782 (6)	0.1205 (6)	0.1246 (19)	0.783 (15)
F3A	−0.1063 (3)	0.6322 (8)	0.3082 (5)	0.1140 (18)	0.783 (15)
F2B	−0.082 (2)	0.564 (2)	0.260 (3)	0.133 (8)	0.217 (15)
F3B	−0.0726 (17)	0.7557 (13)	0.244 (4)	0.133 (8)	0.217 (15)
F1B	−0.0132 (15)	0.629 (2)	0.0833 (8)	0.119 (6)	0.217 (15)
C2	0.1369 (2)	0.65458 (19)	0.3615 (3)	0.0581 (6)	

C3	0.2008 (2)	0.7593 (2)	0.4227 (3)	0.0620 (6)
H3	0.1607	0.8316	0.3828	0.074*
C4	0.3252 (2)	0.75674 (18)	0.5442 (3)	0.0575 (6)
H4	0.3685	0.8276	0.5857	0.069*
C5	0.3858 (2)	0.64908 (16)	0.6046 (2)	0.0484 (5)
C8	0.5128 (2)	0.64594 (16)	0.7435 (2)	0.0503 (5)
N1	0.6164 (2)	0.72612 (19)	0.7499 (3)	0.0731 (6)
H1N	0.620 (3)	0.764 (3)	0.665 (4)	0.088*
H2N	0.689 (3)	0.717 (2)	0.830 (4)	0.088*
N2	0.51210 (16)	0.56917 (14)	0.85708 (19)	0.0515 (5)
O1	0.63766 (15)	0.57836 (13)	0.98654 (18)	0.0622 (5)
H1O	0.618 (3)	0.532 (2)	1.059 (3)	0.093*
C6	0.3222 (2)	0.54448 (19)	0.5390 (3)	0.0622 (6)
H6	0.3633	0.4720	0.5765	0.075*
C7	0.1980 (2)	0.5470 (2)	0.4181 (3)	0.0679 (7)
H7	0.1554	0.4763	0.3747	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0610 (14)	0.104 (2)	0.0610 (14)	0.0069 (14)	0.0127 (11)	0.0105 (14)
F1A	0.099 (2)	0.128 (3)	0.101 (3)	0.0203 (16)	−0.0095 (18)	0.0460 (19)
F2A	0.105 (2)	0.156 (4)	0.085 (2)	0.026 (2)	−0.027 (2)	−0.034 (2)
F3A	0.0503 (12)	0.200 (5)	0.0912 (19)	0.0029 (18)	0.0178 (11)	0.032 (2)
F2B	0.071 (8)	0.173 (13)	0.131 (12)	−0.053 (8)	−0.019 (7)	0.066 (9)
F3B	0.075 (7)	0.144 (11)	0.154 (15)	0.039 (7)	−0.020 (8)	−0.008 (9)
F1B	0.061 (5)	0.216 (15)	0.075 (6)	0.026 (7)	0.010 (4)	0.049 (8)
C2	0.0521 (11)	0.0711 (14)	0.0523 (11)	0.0048 (10)	0.0152 (9)	0.0074 (10)
C3	0.0615 (13)	0.0589 (13)	0.0664 (13)	0.0101 (10)	0.0177 (11)	0.0179 (10)
C4	0.0622 (12)	0.0453 (11)	0.0645 (12)	−0.0022 (9)	0.0149 (10)	0.0077 (9)
C5	0.0508 (11)	0.0461 (11)	0.0500 (10)	0.0000 (8)	0.0161 (8)	0.0033 (8)
C8	0.0513 (11)	0.0430 (10)	0.0567 (11)	0.0002 (8)	0.0136 (9)	−0.0008 (8)
N1	0.0646 (12)	0.0725 (13)	0.0765 (14)	−0.0214 (10)	0.0071 (10)	0.0161 (11)
N2	0.0519 (9)	0.0474 (9)	0.0516 (9)	0.0000 (7)	0.0062 (7)	0.0021 (7)
O1	0.0586 (9)	0.0609 (10)	0.0585 (9)	−0.0033 (7)	−0.0009 (7)	0.0033 (7)
C6	0.0634 (13)	0.0451 (11)	0.0709 (14)	0.0038 (9)	0.0035 (11)	0.0016 (9)
C7	0.0663 (14)	0.0583 (13)	0.0718 (14)	−0.0050 (10)	0.0042 (11)	−0.0059 (10)

Geometric parameters (Å, º)

C1—F2A	1.316 (3)	C4—H4	0.9300
C1—F3A	1.318 (3)	C5—C6	1.381 (3)
C1—F1B	1.323 (3)	C5—C8	1.483 (3)
C1—F1A	1.324 (3)	C8—N2	1.289 (2)
C1—F3B	1.328 (3)	C8—N1	1.354 (3)
C1—F2B	1.330 (3)	N1—H1N	0.84 (3)
C1—C2	1.498 (3)	N1—H2N	0.86 (3)
C2—C3	1.374 (3)	N2—O1	1.432 (2)

C2—C7	1.383 (3)	O1—H1O	0.86 (3)
C3—C4	1.385 (3)	C6—C7	1.382 (3)
C3—H3	0.9300	C6—H6	0.9300
C4—C5	1.389 (3)	C7—H7	0.9300

F2A—C1—F3A	108.9 (3)	C2—C3—C4	119.77 (18)
F2A—C1—F1B	28.5 (8)	C2—C3—H3	120.1
F3A—C1—F1B	121.1 (7)	C4—C3—H3	120.1
F2A—C1—F1A	104.7 (3)	C3—C4—C5	120.46 (18)
F3A—C1—F1A	105.3 (3)	C3—C4—H4	119.8
F1B—C1—F1A	76.5 (9)	C5—C4—H4	119.8
F2A—C1—F3B	131.8 (9)	C6—C5—C4	119.17 (19)
F3A—C1—F3B	72.1 (12)	C6—C5—C8	120.16 (17)
F1B—C1—F3B	107.9 (10)	C4—C5—C8	120.59 (17)
F1A—C1—F3B	37.4 (13)	N2—C8—N1	124.23 (19)
F2A—C1—F2B	71.5 (14)	N2—C8—C5	115.93 (16)
F3A—C1—F2B	41.1 (14)	N1—C8—C5	119.69 (18)
F1B—C1—F2B	93.3 (11)	C8—N1—H1N	120 (2)
F1A—C1—F2B	131.5 (9)	C8—N1—H2N	115.4 (18)
F3B—C1—F2B	109.3 (11)	H1N—N1—H2N	121 (3)
F2A—C1—C2	111.3 (3)	C8—N2—O1	110.41 (15)
F3A—C1—C2	112.3 (2)	N2—O1—H1O	100.9 (19)
F1B—C1—C2	120.3 (6)	C5—C6—C7	120.37 (19)
F1A—C1—C2	113.9 (2)	C5—C6—H6	119.8
F3B—C1—C2	112.2 (7)	C7—C6—H6	119.8
F2B—C1—C2	112.1 (6)	C6—C7—C2	120.0 (2)
C3—C2—C7	120.2 (2)	C6—C7—H7	120.0
C3—C2—C1	120.58 (19)	C2—C7—H7	120.0
C7—C2—C1	119.1 (2)		

F2A—C1—C2—C3	137.2 (4)	C2—C3—C4—C5	0.0 (3)
F3A—C1—C2—C3	−100.5 (5)	C3—C4—C5—C6	1.6 (3)
F1B—C1—C2—C3	107.0 (12)	C3—C4—C5—C8	−175.20 (18)
F1A—C1—C2—C3	19.2 (4)	C6—C5—C8—N2	−41.5 (3)
F3B—C1—C2—C3	−21.5 (17)	C4—C5—C8—N2	135.3 (2)
F2B—C1—C2—C3	−145.0 (19)	C6—C5—C8—N1	142.6 (2)
F2A—C1—C2—C7	−46.0 (5)	C4—C5—C8—N1	−40.6 (3)
F3A—C1—C2—C7	76.4 (5)	N1—C8—N2—O1	−2.0 (3)
F1B—C1—C2—C7	−76.1 (12)	C5—C8—N2—O1	−177.71 (15)
F1A—C1—C2—C7	−164.0 (4)	C4—C5—C6—C7	−1.7 (3)
F3B—C1—C2—C7	155.3 (17)	C8—C5—C6—C7	175.06 (19)
F2B—C1—C2—C7	31.9 (19)	C5—C6—C7—C2	0.3 (4)
C7—C2—C3—C4	−1.5 (3)	C3—C2—C7—C6	1.3 (4)
C1—C2—C3—C4	175.34 (19)	C1—C2—C7—C6	−175.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.84 (3)	2.36 (3)	3.165 (3)	161 (3)
O1—H1*O*···N2^{ii}	0.86 (3)	1.98 (3)	2.766 (2)	152 (3)

Symmetry codes: (i) x, $-y+3/2$, $z-1/2$; (ii) $-x+1$, $-y+1$, $-z+2$.