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3,4-Difluorobenzohydroxamic acid

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.006 Å
R factor = 0.056
wR factor = 0.157
Data-to-parameter ratio = 7.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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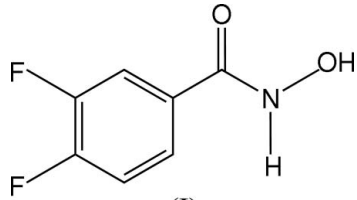
The title compound, $C_7H_5F_2NO_2$, was prepared by the reaction of methyl 3,4-difluorobenzoate with excess NH_2OH in basic solution. In the crystal structure, the molecules are linked into a three-dimensional extended network by N—H···O, O—H···O, C—H···O and C—H···F hydrogen-bond interactions.

Comment

Taking the pharmacological potential of hydroxamic acid derivatives into account (Barbaric *et al.*, 2005), we have synthesized some new types of halo-substituted benzohydroxamic acids. The crystal structure of one of the chloro-substituted benzohydroxamic acids was reported recently by our group (Shang *et al.*, 2005). Here, we describe the structure of the title fluoro-substituted benzohydroxamic acid, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The mean deviation from the plane of atoms O2/N1/O1/C7 is 0.0365 Å, and the angle between the mean O2/N1/O1/C7 and C1/C2/C3/C4/C5/C6 planes is 36.4 (1)°.

In the crystal structure, the molecules of (I) are linked *via* N—H···O, O—H···O, C—H···O and C—H···F intermolecular hydrogen bonds (Table 1). These two- or three-centre interactions form a three-dimensional extended network, illustrated in Fig. 2.

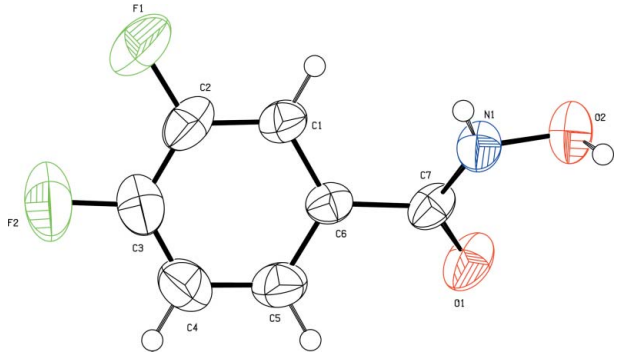


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Compound (I) was prepared by adding methyl 3,4-difluorobenzoate (1.72 g, 10 mmol) to a solution of NH_2OH (16 mmol) in methanol (30 ml) under N_2. The system was stirred at room temperature overnight. Under ice cooling, the pH of the solution was adjusted to *ca* 7 with concentrated HCl. A white precipitate formed and was filtered off. Single crystals of (I), suitable for X-ray analysis, were obtained by recrystallization of this white precipitate from methanol.

Crystal data

$C_7H_5F_2NO_2$
$M_r = 173.12$
Orthorhombic, $P2_12_12_1$
$a = 4.9983$ (11) Å
$b = 5.5158$ (12) Å
$c = 26.418$ (6) Å
$V = 728.3$ (3) Å^3
$Z = 4$
$D_x = 1.579$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1242 reflections
$\theta = 3.8$–$21.2°$
$\mu = 0.15$ mm^{-1}
$T = 292$ (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
4166 measured reflections
886 independent reflections
752 reflections with $I > 2\sigma(I)$
$R_{int} = 0.085$
$\theta_{max} = 26.0°$
$h = -5 \rightarrow 6$
$k = -6 \rightarrow 3$
$l = -30 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.157$
$S = 1.06$
886 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2 + 0.2027P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.80 (1)	2.04 (2)	2.808 (4)	161 (5)
O2—H2···O1^{ii}	0.82	1.87	2.630 (4)	154
C1—H1···O2^{iii}	0.93	2.51	3.433 (5)	170
C5—H5···F1^{iv}	0.93	2.47	3.391 (5)	172

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

The H atoms bonded to the benzene ring and to O2 were placed in calculated positions and treated as riding atoms, with C—H = 0.93 Å and O—H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$. The H atom associated with atom N1 was located in a difference map and refined with a restraint of 0.80 (1) Å. In the absence of significant anomalous dispersion effects, Friedel pairs were merged and the $\delta f''$ term set to zero.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

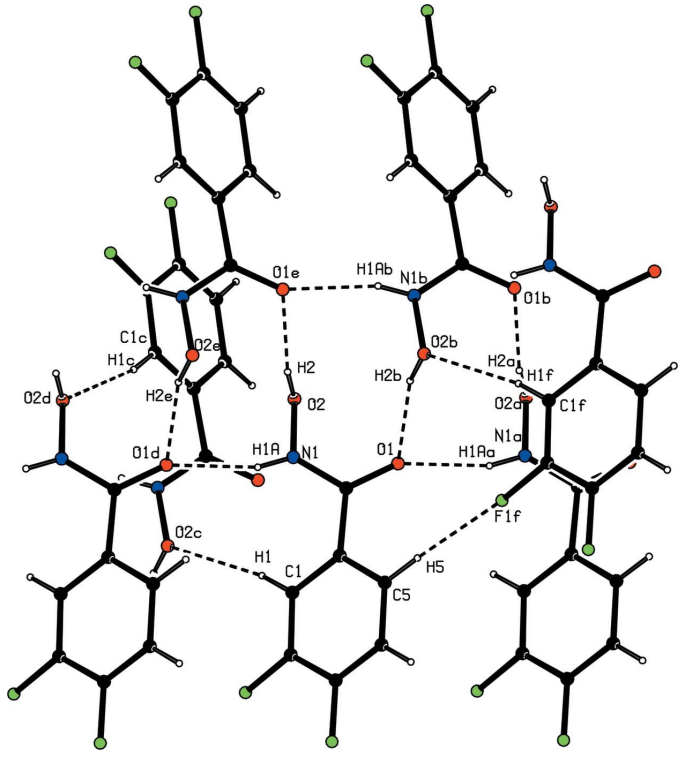


Figure 2
A packing diagram for compound (I), showing the hydrogen-bond interactions as dashed lines [symmetry codes: (i) $-\frac{1}{2}+x, \frac{1}{2}-y, 2-z$; (ii) $-\frac{1}{2}+x, \frac{3}{2}-y, 2-z$; (iii) $-1+x, y, z$; (iv) $1+x, -1+y, z$].

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