

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-Fluoro-N-(4-methoxyphenyl)benzamide

Aamer Saeed,^{a*} Rasheed Ahmad Khera,^a Madiyah Arfan,^a Jim Simpson^b and Roderick G. Stanley^b^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan,and ^bDepartment of Chemistry, University of Otago, PO Box 56, Dunedin, New Zealand

Correspondence e-mail: aamersaeed@yahoo.com

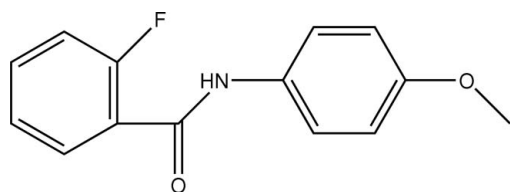
Received 3 March 2009; accepted 11 March 2009

Key indicators: single-crystal X-ray study; $T = 89$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 13.0.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{FNO}_2$, the fluorobenzene and methoxybenzene rings are inclined at 27.06 (7) and 23.86 (7)°, respectively, to the amide portion of the molecule and at 3.46 (9)° to one another. The methoxy substituent lies close to the methoxybenzene ring plane, with a maximum deviation of 0.152 (3) Å for the methyl C atom. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules into rows along a . Weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{F}$ interactions further stabilize the packing, forming corrugated sheets in the bc plane.

Related literature

For the biological activity of benzamides and related compounds and their use in organic synthesis, see: Vega-Noverola *et al.* (1989); Yoo *et al.* (2005); Saeed *et al.* (2008). For related structures, see: Saeed *et al.* (2008); Chopra & Guru Row (2008); Donnelly *et al.* (2008); Cockroft *et al.* (2007); Spitaleri *et al.* (2004).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{12}\text{FNO}_2$ $M_r = 245.25$ Orthorhombic, $P2_12_12_1$ $a = 5.2901$ (2) Å $b = 6.6435$ (3) Å $c = 31.7823$ (11) Å $V = 1116.98$ (8) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.11$ mm⁻¹ $T = 89$ K $0.30 \times 0.23 \times 0.04$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2006)

 $T_{\min} = 0.853$, $T_{\max} = 0.996$

19727 measured reflections

2185 independent reflections

1980 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.049$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$ $wR(F^2) = 0.096$ $S = 1.06$

2185 reflections

168 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.37$ e Å⁻³ $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.84 (2)	2.34 (2)	3.1366 (17)	158.5 (17)
$\text{C4}-\text{H4}\cdots\text{F1}^{\text{ii}}$	0.95	2.44	3.2472 (17)	143
$\text{C14}-\text{H14B}\cdots\text{O2}^{\text{iii}}$	0.98	2.61	3.3753 (18)	135

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x + \frac{1}{2}, -y + \frac{5}{2}, -z$. Cg2 is the centroid of the C3–C8 benzene ring.

Data collection: APEX2 (Bruker 2006); cell refinement: APEX2 and SAINT (Bruker 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008) and TITAN2000 (Hunter & Simpson, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and TITAN2000; molecular graphics: SHELXTL (Sheldrick, 2008) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97, enCIFer (Allen *et al.*, 2004), PLATON (Spek, 2009) and publCIF (Westrip, 2009).

MA gratefully acknowledges a research scholarship from the HEC, Islamabad, under the HEC Indigenous PhD Scholarship 5000 Scheme. We also thank the University of Otago for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2159).

References

- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Bruker (2006). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chopra, D. & Guru Row, T. N. (2008). *CrystEngComm*, **10**, 54–67.
- Cockroft, S. L., Perkins, J., Zonta, C., Adams, H., Spey, S. E., Low, C. M. R., Vinter, J. G., Lawson, K. R., Urch, C. J. & Hunter, C. A. (2007). *Org. Biomol. Chem.* **5**, 1062–1080.
- Donnelly, K., Gallagher, J. F. & Lough, A. J. (2008). *Acta Cryst.* **C64**, o335–o340.
- Hunter, K. A. & Simpson, J. (1999). TITAN2000. University of Otago, New Zealand.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Saeed, A., Khera, R. A., Abbas, N., Simpson, J. & Stanley, R. G. (2008). *Acta Cryst.* **E64**, o2322–o2323.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

Spitaleri, A., Hunter, C. A., McCabe, J. F., Packer, M. J. & Cockroft, S. L. (2004). *CrystEngComm*, **6**, 489–493.

Vega-Noverola, A. P., Soto, J. M., Noguera, F. P., Mauri, J. M. & Spickett, G. W. R. (1989). US Patent No. 4 877 780.

Westrip, S. P. (2009). *pubCIF*. In preparation.

Yoo, C. L., Fettinger, J. C. & Kurth, M. J. (2005). *J. Org. Chem.* **70**, 6941–6943.

supporting information

Acta Cryst. (2009). E65, o802–o803 [doi:10.1107/S1600536809008927]

2-Fluoro-*N*-(4-methoxyphenyl)benzamide

Aamer Saeed, Rasheed Ahmad Khera, Madiah Arfan, Jim Simpson and Roderick G. Stanley

S1. Comment

The benzanilide core is present in compounds with a wide range of biological activity and benzanilides and benzamides are also used extensively in organic synthesis (Saeed *et al.*, 2008). Various *N*-substituted benzamides exhibit potent antiemetic activity (Vega-Noverola *et al.*, 1989). A one-pot conversion of 2-nitro-*n*-arylbenzamides to 2,3-dihydro-1*H*-quinazoline-4-ones has also been reported (Yoo *et al.*, 2005).

As part of our work on the structure of benzanilides and related compounds, we report here the structure of the title 2-fluorobenzamide derivative, I, Fig 1. In the structure of (I), the C2...C7 and C8...C13 rings are inclined at 27.06 (7)° and 23.86 (7)° respectively to the C2/C1/O1/N1/C8 amide portion of the molecule and at 3.46 (9)° to one another. The methoxy substituent lies close to the methoxybenzene ring plane with a maximum deviation of 0.152 (3) Å for C14. *N*-aryl 2-fluorobenzamide derivatives are reasonably common and bond distances and angles in the present molecule agree well with those in similar structures (see for example Saeed *et al.* 2008, Chopra & Guru Row, 2008, Donnelly *et al.* 2008, Cockroft *et al.* (2007), Spitaleri *et al.* 2004).

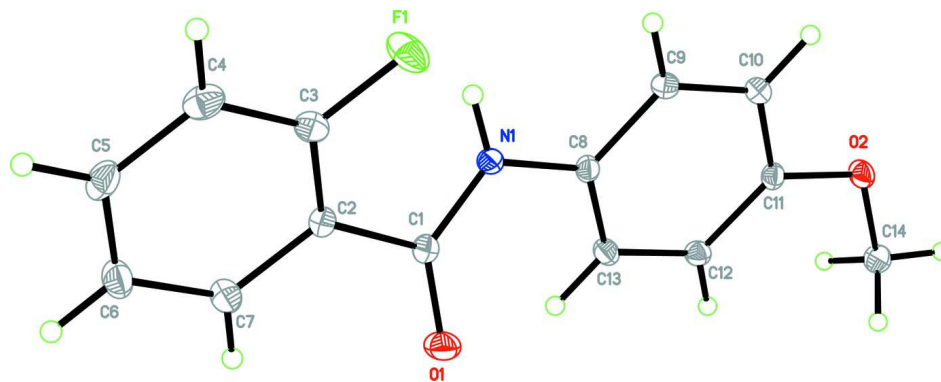
In the crystal structure intermolecular N1—H1N...O1 hydrogen bonds link molecules into rows along *a*. Weak C—H...O and C—H...F interactions further stabilize the packing, forming corrugated sheets in the *bc* plane (Table 1 and Fig.2).

S2. Experimental

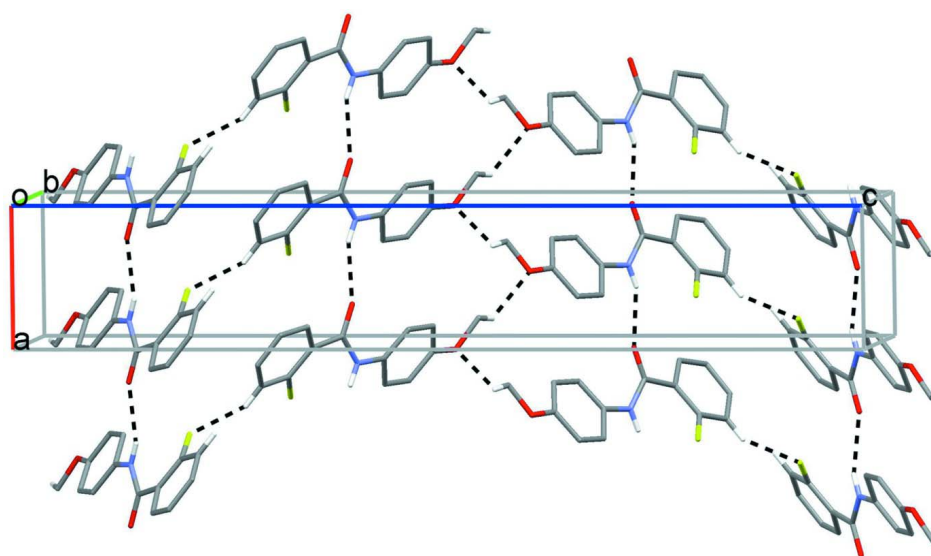
2-Fluorobenzoyl chloride (5.4 mmol) in CHCl₃ was treated with 4-methoxyaniline (21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with CHCl₃ and washed consecutively with aq 1 M HCl and saturated aq NaHCO₃. The organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Crystallization of the residue from CHCl₃ afforded the title compound (81%) as white needles: Anal. calcd. for C₁₄H₁₂FNO₂: C 68.56, H 4.93, N 5.71%; found: C 68.49, H 4.97, N 5.63%

S3. Refinement

The H atom bound to N1 was located in a difference map and refined isotropically. All other H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.95 Å, $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C) for aromatic and 0.98 Å, $U_{\text{iso}} = 1.5U_{\text{eq}}$ (C) for CH₃ hydrogen atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged and a Flack parameter is not reported.

**Figure 1**

The structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level.

**Figure 2**

Crystal packing for (I) viewed down the *b* axis with hydrogen bonds drawn as dashed lines and H atoms not involved in hydrogen bonding omitted.

2-Fluoro-*N*-(4-methoxyphenyl)benzamide

Crystal data

$C_{14}H_{12}FNO_2$

$M_r = 245.25$

Orthorhombic, $P2_12_12_1$

Hall symbol: $P\ 2ac\ 2ab$

$a = 5.2901\ (2)\ \text{\AA}$

$b = 6.6435\ (3)\ \text{\AA}$

$c = 31.7823\ (11)\ \text{\AA}$

$V = 1116.98\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 512$

$D_x = 1.458\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5036 reflections

$\theta = 3.1\text{--}31.4^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 89\ \text{K}$

Rectangular, colourless

$0.30 \times 0.23 \times 0.04\ \text{mm}$

Data collection

Bruker APEXII CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2006)
 $T_{\min} = 0.853$, $T_{\max} = 0.996$

19727 measured reflections
2185 independent reflections
1980 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\max} = 31.5^\circ$, $\theta_{\min} = 1.3^\circ$
 $h = -7 \rightarrow 7$
 $k = -8 \rightarrow 9$
 $l = -46 \rightarrow 45$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.096$
 $S = 1.06$
2185 reflections
168 parameters
0 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
 $w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.1314P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.8287 (2)	0.30490 (19)	0.12515 (4)	0.0120 (2)
H1N	0.681 (4)	0.274 (3)	0.1327 (6)	0.018 (5)*
C1	1.0253 (3)	0.1910 (2)	0.13830 (4)	0.0122 (3)
O1	1.2460 (2)	0.21863 (18)	0.12733 (3)	0.0195 (2)
C2	0.9628 (3)	0.0195 (2)	0.16750 (4)	0.0113 (2)
C3	0.7617 (3)	0.0129 (2)	0.19568 (4)	0.0138 (3)
F1	0.60670 (19)	0.17461 (15)	0.19944 (3)	0.0211 (2)
C4	0.7144 (3)	-0.1504 (3)	0.22153 (4)	0.0172 (3)
H4	0.5746	-0.1494	0.2403	0.021*
C5	0.8740 (3)	-0.3153 (2)	0.21964 (5)	0.0175 (3)
H5	0.8427	-0.4295	0.2369	0.021*
C6	1.0809 (3)	-0.3137 (2)	0.19238 (5)	0.0176 (3)
H6	1.1921	-0.4259	0.1912	0.021*
C7	1.1234 (3)	-0.1472 (2)	0.16695 (4)	0.0151 (3)
H7	1.2656	-0.1467	0.1487	0.018*

C8	0.8450 (3)	0.4821 (2)	0.10044 (4)	0.0109 (2)
C9	0.6527 (3)	0.6247 (2)	0.10514 (4)	0.0124 (3)
H9	0.5154	0.5977	0.1236	0.015*
C10	0.6600 (3)	0.8048 (2)	0.08330 (4)	0.0127 (3)
H10	0.5290	0.9010	0.0869	0.015*
C11	0.8609 (3)	0.8447 (2)	0.05591 (4)	0.0113 (3)
C12	1.0490 (3)	0.7011 (2)	0.05019 (4)	0.0120 (3)
H12	1.1830	0.7261	0.0310	0.014*
C13	1.0418 (3)	0.5203 (2)	0.07255 (4)	0.0123 (3)
H13	1.1716	0.4232	0.0687	0.015*
O2	0.8549 (2)	1.02823 (15)	0.03604 (3)	0.0144 (2)
C14	1.0690 (3)	1.0759 (2)	0.01050 (5)	0.0159 (3)
H14A	1.2233	1.0662	0.0274	0.024*
H14B	1.0517	1.2132	-0.0004	0.024*
H14C	1.0788	0.9811	-0.0131	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0097 (5)	0.0108 (5)	0.0155 (5)	0.0003 (5)	0.0019 (4)	0.0030 (4)
C1	0.0124 (6)	0.0103 (6)	0.0138 (6)	-0.0004 (5)	0.0005 (5)	0.0013 (5)
O1	0.0117 (5)	0.0212 (6)	0.0255 (5)	0.0005 (4)	0.0013 (4)	0.0099 (5)
C2	0.0125 (6)	0.0102 (6)	0.0112 (5)	-0.0008 (5)	-0.0003 (5)	0.0000 (5)
C3	0.0124 (6)	0.0157 (6)	0.0132 (6)	0.0011 (5)	-0.0009 (5)	-0.0002 (5)
F1	0.0214 (5)	0.0231 (5)	0.0189 (4)	0.0094 (4)	0.0071 (4)	0.0027 (4)
C4	0.0154 (6)	0.0228 (8)	0.0134 (6)	-0.0033 (6)	0.0003 (5)	0.0036 (6)
C5	0.0194 (7)	0.0161 (7)	0.0169 (6)	-0.0054 (6)	-0.0028 (5)	0.0056 (6)
C6	0.0198 (7)	0.0132 (7)	0.0200 (6)	0.0019 (6)	-0.0024 (6)	0.0027 (6)
C7	0.0159 (6)	0.0139 (6)	0.0156 (6)	0.0020 (6)	0.0005 (5)	0.0006 (5)
C8	0.0117 (6)	0.0087 (5)	0.0122 (5)	0.0001 (5)	-0.0013 (5)	0.0005 (5)
C9	0.0108 (6)	0.0125 (6)	0.0140 (6)	0.0000 (5)	0.0022 (5)	-0.0004 (5)
C10	0.0116 (6)	0.0114 (6)	0.0150 (6)	0.0013 (5)	0.0006 (5)	-0.0002 (5)
C11	0.0124 (6)	0.0091 (6)	0.0123 (5)	-0.0005 (5)	-0.0018 (5)	0.0008 (5)
C12	0.0110 (5)	0.0123 (6)	0.0127 (5)	0.0003 (5)	0.0010 (5)	0.0009 (5)
C13	0.0119 (6)	0.0112 (6)	0.0139 (6)	0.0020 (5)	0.0013 (5)	0.0001 (5)
O2	0.0145 (5)	0.0108 (5)	0.0178 (5)	0.0019 (4)	0.0029 (4)	0.0042 (4)
C14	0.0146 (6)	0.0133 (6)	0.0199 (6)	-0.0009 (6)	0.0029 (6)	0.0047 (5)

Geometric parameters (Å, °)

N1—C1	1.3522 (19)	C8—C13	1.3909 (18)
N1—C8	1.4177 (17)	C8—C9	1.3980 (19)
N1—H1N	0.84 (2)	C9—C10	1.3835 (19)
C1—O1	1.2317 (17)	C9—H9	0.9500
C1—C2	1.5066 (19)	C10—C11	1.3992 (19)
C2—C3	1.3914 (19)	C10—H10	0.9500
C2—C7	1.396 (2)	C11—O2	1.3734 (16)
C3—F1	1.3567 (17)	C11—C12	1.3902 (19)

C3—C4	1.384 (2)	C12—C13	1.3959 (19)
C4—C5	1.384 (2)	C12—H12	0.9500
C4—H4	0.9500	C13—H13	0.9500
C5—C6	1.396 (2)	O2—C14	1.4289 (18)
C5—H5	0.9500	C14—H14A	0.9800
C6—C7	1.388 (2)	C14—H14B	0.9800
C6—H6	0.9500	C14—H14C	0.9800
C7—H7	0.9500		
C1—N1—C8	126.09 (12)	C13—C8—N1	123.38 (12)
C1—N1—H1N	119.3 (14)	C9—C8—N1	117.36 (12)
C8—N1—H1N	114.6 (14)	C10—C9—C8	120.82 (13)
O1—C1—N1	123.93 (13)	C10—C9—H9	119.6
O1—C1—C2	119.66 (13)	C8—C9—H9	119.6
N1—C1—C2	116.40 (12)	C9—C10—C11	119.81 (13)
C3—C2—C7	116.65 (13)	C9—C10—H10	120.1
C3—C2—C1	126.02 (13)	C11—C10—H10	120.1
C7—C2—C1	117.31 (12)	O2—C11—C12	124.45 (12)
F1—C3—C4	117.34 (13)	O2—C11—C10	115.90 (12)
F1—C3—C2	119.61 (13)	C12—C11—C10	119.66 (12)
C4—C3—C2	123.02 (14)	C11—C12—C13	120.29 (13)
C3—C4—C5	118.98 (14)	C11—C12—H12	119.9
C3—C4—H4	120.5	C13—C12—H12	119.9
C5—C4—H4	120.5	C8—C13—C12	120.13 (13)
C4—C5—C6	119.95 (14)	C8—C13—H13	119.9
C4—C5—H5	120.0	C12—C13—H13	119.9
C6—C5—H5	120.0	C11—O2—C14	116.09 (11)
C7—C6—C5	119.60 (14)	O2—C14—H14A	109.5
C7—C6—H6	120.2	O2—C14—H14B	109.5
C5—C6—H6	120.2	H14A—C14—H14B	109.5
C6—C7—C2	121.77 (14)	O2—C14—H14C	109.5
C6—C7—H7	119.1	H14A—C14—H14C	109.5
C2—C7—H7	119.1	H14B—C14—H14C	109.5
C13—C8—C9	119.26 (13)		

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>
N1—H1N...O1 ⁱ	0.84 (2)	2.34 (2)	3.1366 (17)	158.5 (17)
C4—H4...F1 ⁱⁱ	0.95	2.44	3.2472 (17)	143
C14—H14B...O2 ⁱⁱⁱ	0.98	2.61	3.3753 (18)	135

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, y-1/2, -z+1/2$; (iii) $x+1/2, -y+5/2, -z$.