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Crystal structures of 3-fluoro-*N*-[2-(trifluoromethyl)phenyl]benzamide, 3-bromo-*N*-[2-(trifluoromethyl)phenyl]benzamide and 3-iodo-*N*-[2-(trifluoromethyl)phenyl]benzamide

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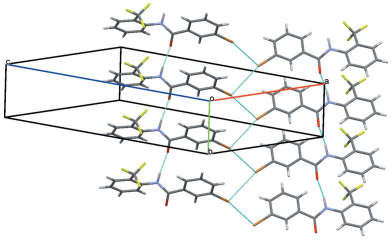
CCDC references: 1479657; 1479656; 1479655

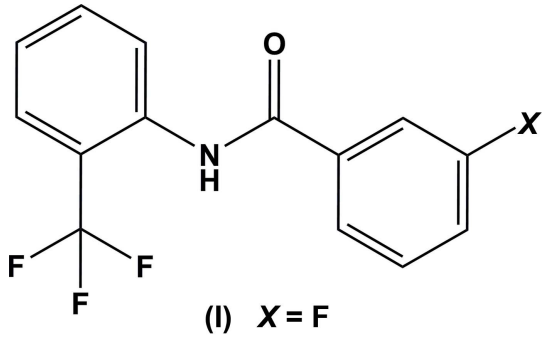
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compounds, $C_{14}H_9F_4NO$, (I), $C_{14}H_9BrF_3NO$, (II), and $C_{14}H_9F_3INO$, (III), the two benzene rings are inclined to one another by 43.94 (8)° in molecule *A* and 55.66 (7)° in molecule *B* of compound (I), which crystallizes with two independent molecules in the asymmetric unit, but by only 10.40 (12)° in compound (II) and 12.5 (2)° in compound (III). In the crystals of all three compounds, N—H···O hydrogen bonds link the molecules to form chains propagating along the *a*-axis direction for (I), and along the *b*-axis direction for (II) and (III). In the crystal of (I), *–A–B–A–B–* chains are linked by C—H···O hydrogen bonds, forming layers parallel to (010). Within the layers there are weak offset π–π interactions present [intercentroid distances = 3.868 (1) and 3.855 (1) Å]. In the crystals of (II) and (III), the chains are linked *via* short halogen–halogen contacts [Br···Br = 3.6141 (4) Å in (II) and I···I = 3.7797 (5) Å in (III)], resulting in the formation of ribbons propagating along the *b*-axis direction.

1. Chemical context

Amides are very common in nature, and are easily synthesized and provide structural rigidity to various molecules (Gowda *et al.*, 2003). Furthermore, *N*-arylamides show a broad spectrum of pharmacological properties, including antibacterial (Manojkumar *et al.*, 2013*a*), antitumor (Abdou *et al.*, 2004), antioxidant, analgesic and antiviral activity (Manojkumar *et al.*, 2013*b*). In view of their importance, the title *N*-(2-trifluoromethylphenyl)benzamides (I)–(III) were synthesized and we report herein on their crystal structures.





(I) *X* = F
(II) *X* = Br
(III) *X* = I

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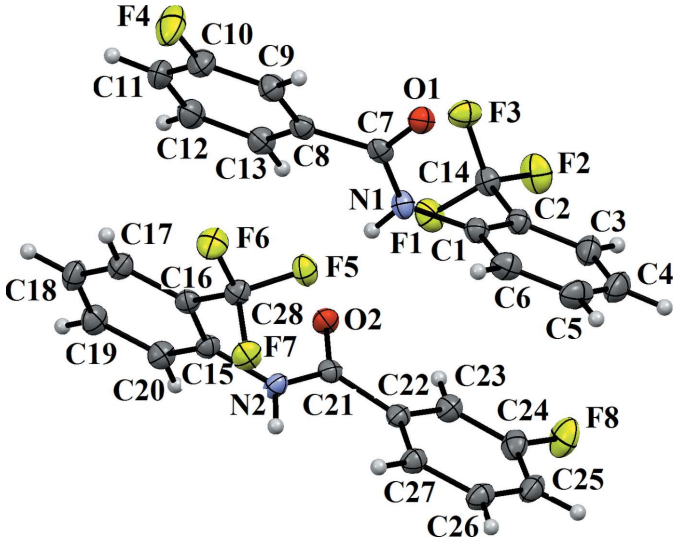

Figure 1
A view of the molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

The molecular structure of compound (I) is illustrated in Fig. 1. It crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit, which slightly differ in their molecular conformations, as shown in the *AutoMolFit* diagram (Fig. 2; Spek, 2009). In both molecules, the 3-fluoro substituent on the benzoic acid ring and the 2-CF_3 substituent on the aniline ring are *anti* to one another, and the 3-fluoro substituent is *anti* to the N—H bond in the central –C_{ar}—C(=O)—N—C_{ar}– (ar = aromatic) segment of the molecules. The dihedral angle between the two benzene rings is 43.94 (8)° in molecule *A*, while in molecule *B* it is larger, being 55.66 (7)°. The torsion angle of the central –C_{ar}—C(=O)—N—C_{ar}– segment is 176.74 (12)° in molecule *A* and −179.58 (12)° in molecule *B*.

The molecular structures of compounds (II) and (III) are illustrated in Figs. 3 and 4, respectively. Here, the 3-bromo and 3-iodo substituents on the benzoic acid ring and the 2-CF_3 substitution on the aniline ring are *anti* to one another, and the 3-bromo and 3-iodo substituents are *anti* to the N—H bond in the central –C_{ar}—C(=O)—N—C_{ar}– segment of the molecules, similar to situation observed in (I). The dihedral angle between the two benzene rings is 10.40 (12)° in (II) and 12.5 (2)° in (III), which is much less than observed for molecules *A* and *B* of compound (I). The torsion angle of the central –C_{ar}—C(=O)—N—C_{ar}– segment is −175.5 (2)° in (II) and 174.8 (3)° in (III), again similar to that in molecules *A* and *B* of compound (I).

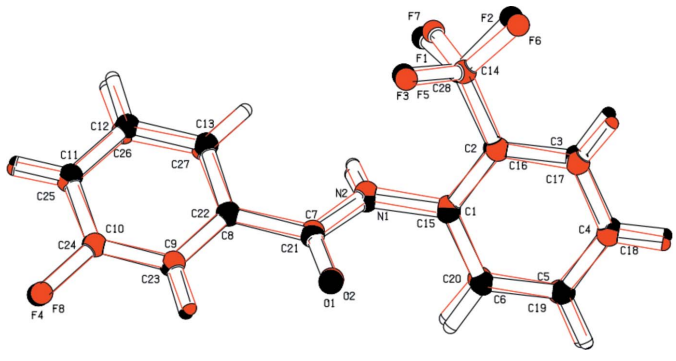

Figure 2
A view of the molecular fit of molecules *A* (black) and *B* (red) of compound (I).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.87 (2)	2.01 (2)	2.8239 (16)	157 (1)
N2—H2···O1^{i}	0.89 (2)	1.99 (2)	2.8303 (16)	158 (1)
C5—H5···O2^{ii}	0.95	2.35	3.2861 (18)	167
C12—H12···O1^{iii}	0.95	2.45	3.3172 (17)	152

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

3. Supramolecular features

In the crystal of (I), strong N1—H1···O2 and N2—H2···O1 hydrogen bonds link the molecules to form –*A*–*B*–*A*–*B*– *C*(4) chains running along the *a*-axis direction (Table 1 and Fig. 5).

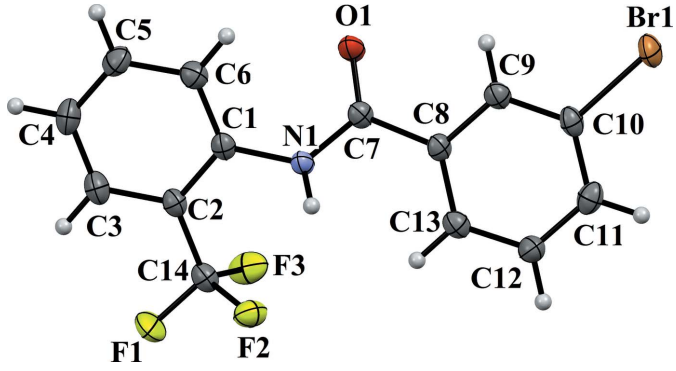

Figure 3
A view of the molecular structure of compound (II), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

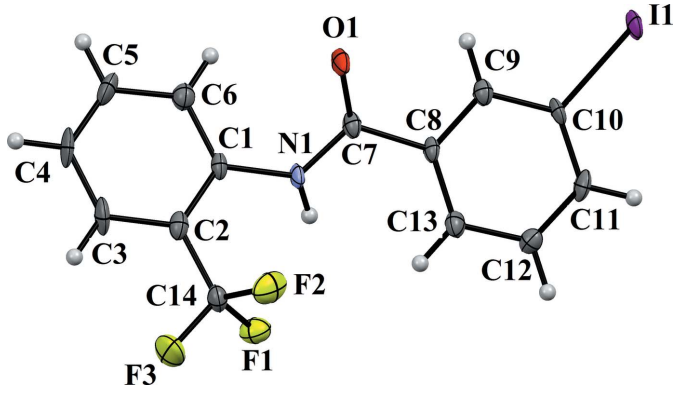

Figure 4
A view of the molecular structure of compound (III), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

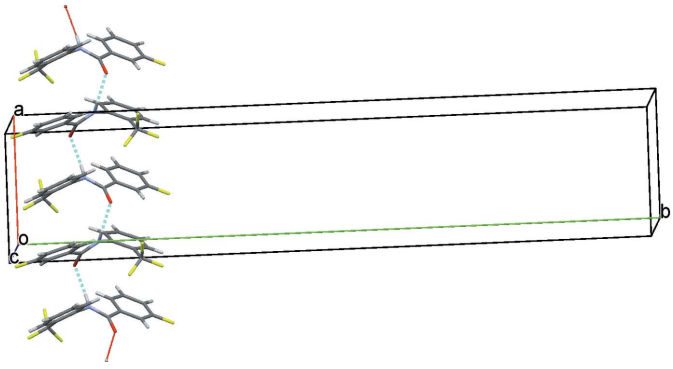

Figure 5
A view along the *c* axis of the crystal packing of compound (I). The N—H···O hydrogen bonds are shown as dashed lines (see Table 1).

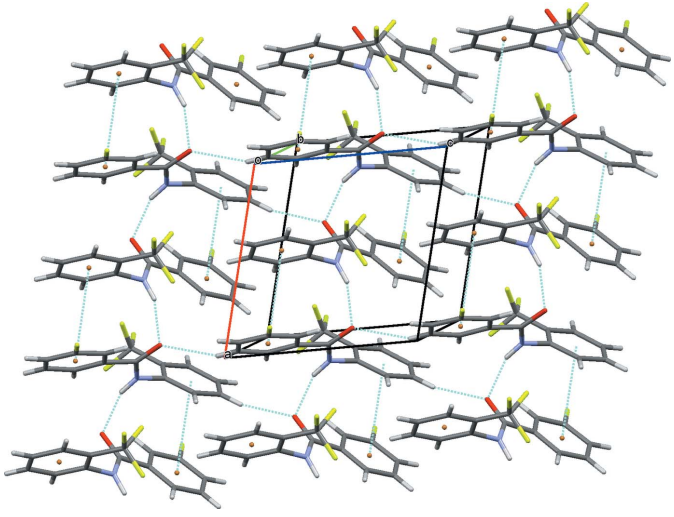

Figure 6
A view along the *b* axis of the crystal packing of compound (I). The C—H···O (see Table 1) and π–π interactions are shown as dashed lines.

Neighbouring chains are linked *via* C5—H5···O2 and C12—H12···O1 hydrogen bonds (Table 1), forming layers lying parallel to the *ac* plane (Fig. 6). Within the layers there are weak offset π–π interactions present involving the aniline and benzoic acid rings [$Cg1\cdots Cg4$ = 3.8682 (9) Å and $Cg2\cdots Cg3^{i}$ = 3.8553 (9) Å; $Cg1$ and $Cg3$ are the centroids of the aniline rings C1–C6 and C15–C20, respectively; $Cg2$ and $Cg4$ are the centroids of the benzoic acid rings C8–C13 and C22–C27, respectively; symmetry code (i) $x-1, y, z$]. The crystal structure does not feature any C—H···F or F···F interactions (Fig. 6).

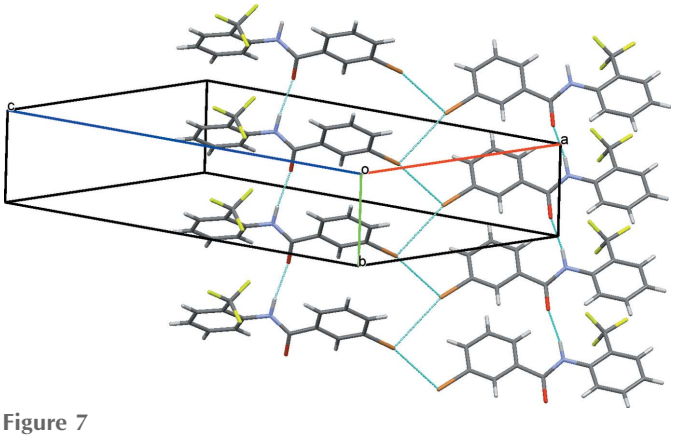

Figure 7
A view along the *b* axis of the crystal packing of compound (II). The N—H···O hydrogen bonds (see Table 2) and the Br···Br contacts are shown as dashed lines.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.89 (2)	2.00 (2)	2.835 (2)	156 (3)

Symmetry code: (i) $x, y-1, z$.

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.89 (3)	1.99 (4)	2.826 (5)	156 (5)

Symmetry code: (i) $x, y+1, z$.

The crystal structure of (II), features strong N1—H1···O1 hydrogen bonds (Fig. 7 and Table 2) similar to those observed in (I), linking the molecules into $C(4)$ chains running parallel to the *b* axis (Fig. 7). Adjacent chains are connected *via* short Br···Br contacts [3.6141 (4) Å], forming ribbons along [010]; see Fig. 7.

The crystal structure of (III), features similar characteristics to that of (II). Strong N1—H1···O1 hydrogen bonds link the molecules into $C(4)$ chains running parallel to the *b* axis (Table 3 and Fig. 8). Adjacent chains are linked *via* short I···I contacts [3.7797 (5) Å], forming ribbons along [010]; see Fig. 8.

From the above observations, it can be concluded that the bromo and iodo substitutions on the *meta* position of the benzoic acid ring have a similar effect on the molecular conformations and the supramolecular architectures exhibited by this class of compounds, whereas the fluoro substitution has a very different influence. For instance, there are two molecules in the asymmetric unit of (I) compared to one molecules in those of (II) and (III). Also, the dihedral angle between the two benzene rings is much larger in the two

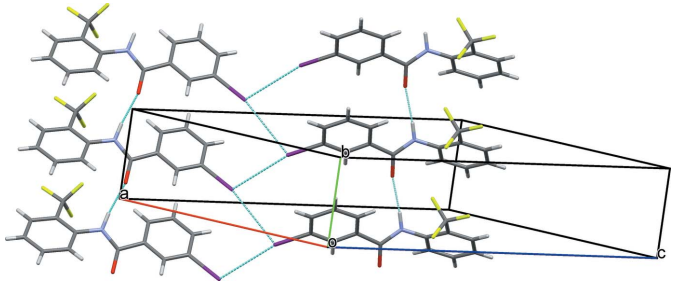

Figure 8
A view along the *b* axis of the crystal packing of compound (III). The N—H···O hydrogen bonds (see Table 3) and the I···I contacts are shown as dashed lines.

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{14}H_9F_4NO$	$C_{14}H_9BrF_3NO$	$C_{14}H_9F_3INO$
M_r	283.22	344.13	391.12
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
Temperature (K)	173	173	173
a, b, c (Å)	8.0258 (2), 39.7598 (12), 7.8932 (2)	12.9456 (6), 4.7377 (2), 21.9025 (10)	13.3358 (6), 4.7471 (2), 22.3558 (10)
β (°)	103.937 (1)	104.770 (2)	105.848 (2)
V (Å^3)	2444.60 (11)	1298.94 (10)	1361.47 (10)
Z	8	4	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	1.22	4.63	18.78
Crystal size (mm)	0.29 × 0.22 × 0.19	0.28 × 0.24 × 0.20	0.27 × 0.22 × 0.18
Data collection			
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)	Multi-scan (*SADABS*; Bruker, 2009)	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.760, 0.793	0.315, 0.396	0.081, 0.133
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13874, 3997, 3816	8466, 2114, 1986	7120, 2223, 2124
R_{int}	0.034	0.039	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.584	0.585	0.584
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.091, 1.06	0.034, 0.090, 1.05	0.043, 0.109, 1.09
No. of reflections	3997	2114	2223
No. of parameters	369	185	185
No. of restraints	2	1	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.17	0.62, −0.34	1.84, −1.41

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

molecules (*A* and *B*) of (I), compared to the values observed in (II) and (III). Furthermore, the crystal structures of both (II) and (III) feature short halogen···halogen contacts, in addition to the N—H···O hydrogen bonds, resulting in one-dimensional structures, whereas in (I), in the absence of F···F contacts, C—H···O hydrogen bonds and π–π interactions are observed, in addition to the strong N—H···O hydrogen bonds, resulting in a two-dimensional architecture.

4. Database survey

A search of the Cambridge Structural Database (CSD; Version 5.37, update February 2016; Groom *et al.*, 2016) for similar compounds *viz.* *N*-(2-(trifluoromethyl)phenyl)arylamides, gave four hits. They include *N*-(2-(trifluoromethyl)-phenyl)benzamide, for which there are three reports: JOZFUB and JOZFUB01 in space group $P4_3$ (Hathwar *et al.*, 2014) and LASHOE in space group $P4_1$ (Panini & Chopra, 2012), and 2-(trifluoromethyl)-*N*-(2-(trifluoromethyl)phenyl)-benzamide (LASKAT; Panini & Chopra, 2012). In compounds LASHOE and LASKAT, the 2-CF_3 group in the aniline ring is nearly *syn* to the N—H bond in the central amide segment of the molecule, as observed in the title compounds. In LASHOE (Panini & Chopra, 2012), the dihedral angle between the two benzene rings is 41.3 (1)°, and the torsion angle of the central –C_{ar}—N—C(=O)—C_{ar}– segment is 175.1 (5)°, which is very close to the values observed for the two independent molecules in compound (I). This shows that introducing a fluorine atom into the *meta* position of the benzoyl ring, as in compound (I), has little effect on the molecular conformation of this class of compounds.

5. Synthesis and crystallization

The different substituted benzoic acids (3 mmol) were dissolved in phosphorous oxychloride taken in a 250 ml round-bottomed flask. The mixtures were refluxed for an hour and later cooled to 273 K. An equimolar amount of 2-(trifluoromethyl)aniline was added dropwise to these mixtures with continuous stirring. After completion of the addition, the reaction mixtures were brought to room temperature and stirring was continued for 1 h. The reaction mixtures were poured into ice-cold water. The solids that separated were washed thoroughly with water, followed by washing with dilute hydrochloric acid, water, aqueous sodium hydrogen carbonate solution and again with water. The compounds were filtered under suction, dried and recrystallized from aqueous ethanol to constant melting points. Prismatic colourless single crystals of all three compounds were obtained by slow

evaporation of solutions in methanol, with a few drops of water.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. In all three compounds the NH H atoms were located in difference Fourier maps and refined with a distance restraint: N—H = 0.90 (4) Å. The C-bound H atoms were positioned with idealized geometry and refined using a riding model: C—H = 0.95 Å, with $U_{iso} = 1.2U_{eq}$(C). In the final cycles of refinement of compound (III), a bad reflection ($\bar{4}$ 2 2) was omitted, which lead to an improvement in the values of *R*1, *wR*2, and GOF.

Acknowledgements

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supporting information

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Crystal structures of 3-fluoro-*N*-[2-(trifluoromethyl)phenyl]benzamide, 3-bromo-*N*-[2-(trifluoromethyl)phenyl]benzamide and 3-iodo-*N*-[2-(trifluoromethyl)phenyl]benzamide

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Computing details

For all compounds, data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

(I) 3-Fluoro-*N*-[2-(trifluoromethyl)phenyl]benzamide

Crystal data

$C_{14}H_9F_4NO$
M_r = 283.22
Monoclinic, $P2_1/c$
a = 8.0258 (2) Å
b = 39.7598 (12) Å
c = 7.8932 (2) Å
β = 103.937 (1)°
V = 2444.60 (11) Å^3
Z = 8
$F(000)$ = 1152
Prism
D_x = 1.539 Mg m^{-3}
Melting point: 377 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 143 reflections
θ = 2.2–64.2°
μ = 1.22 mm^{-1}
T = 173 K
Prism, colourless
0.29 × 0.22 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.760, T_{max} = 0.793
13874 measured reflections
3997 independent reflections
3816 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 64.2°, θ_{min} = 2.2°
h = −9→7
k = −44→45
l = −6→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.06
3997 reflections
369 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.8967P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
H2	0.667 (2)	0.1103 (4)	0.558 (2)	0.034 (5)*
H1	0.140 (2)	0.1376 (4)	0.456 (2)	0.029 (4)*
F5	0.33100 (11)	0.07085 (2)	0.60352 (10)	0.0292 (2)
F6	0.35108 (12)	0.01935 (2)	0.52831 (11)	0.0329 (2)
F7	0.57809 (11)	0.04694 (2)	0.65368 (10)	0.0292 (2)
O2	0.35341 (12)	0.15177 (2)	0.38529 (12)	0.0247 (2)
N2	0.56532 (16)	0.11452 (3)	0.48712 (15)	0.0222 (3)
F8	0.45852 (14)	0.24815 (2)	0.79593 (13)	0.0436 (3)
C21	0.48174 (17)	0.14343 (3)	0.49780 (17)	0.0198 (3)
C22	0.54636 (17)	0.16483 (3)	0.65650 (17)	0.0210 (3)
C28	0.42663 (18)	0.04946 (3)	0.53435 (18)	0.0234 (3)
C15	0.51404 (17)	0.09248 (3)	0.34014 (17)	0.0209 (3)
C20	0.53464 (19)	0.10260 (4)	0.17839 (19)	0.0254 (3)
H20	0.5775	0.1244	0.1644	0.030*
C23	0.47773 (19)	0.19705 (4)	0.65248 (18)	0.0252 (3)
H23	0.3996	0.2052	0.5506	0.030*
C27	0.66387 (18)	0.15357 (4)	0.80616 (18)	0.0238 (3)
H27	0.7132	0.1318	0.8083	0.029*
C24	0.5257 (2)	0.21680 (4)	0.7997 (2)	0.0291 (3)
C26	0.70845 (19)	0.17425 (4)	0.95187 (19)	0.0280 (3)
H26	0.7879	0.1664	1.0537	0.034*
C25	0.6385 (2)	0.20607 (4)	0.95048 (19)	0.0291 (3)
H25	0.6675	0.2201	1.0506	0.035*
C19	0.49280 (19)	0.08093 (4)	0.03697 (18)	0.0280 (3)
H19	0.5084	0.0878	−0.0734	0.034*
C18	0.4285 (2)	0.04928 (4)	0.05599 (19)	0.0283 (3)
H18	0.3997	0.0345	−0.0413	0.034*
C17	0.40585 (19)	0.03907 (4)	0.21705 (18)	0.0254 (3)
H17	0.3613	0.0173	0.2300	0.030*
C16	0.44841 (17)	0.06066 (4)	0.35957 (18)	0.0212 (3)
F1	0.01785 (11)	0.19821 (2)	0.34114 (10)	0.0316 (2)

F3	−0.21044 (11)	0.17739 (2)	0.39516 (11)	0.0316 (2)
F2	−0.14591 (13)	0.22917 (2)	0.45373 (12)	0.0392 (2)
O1	−0.08868 (12)	0.09581 (2)	0.62751 (12)	0.0247 (2)
F4	−0.13792 (16)	−0.00092 (3)	0.21276 (14)	0.0509 (3)
N1	0.07498 (16)	0.13350 (3)	0.52692 (15)	0.0227 (3)
C7	−0.01510 (17)	0.10475 (3)	0.51462 (17)	0.0211 (3)
C13	−0.00037 (19)	0.09805 (4)	0.19810 (19)	0.0263 (3)
H13	0.0340	0.1209	0.1952	0.032*
C8	−0.02734 (17)	0.08437 (4)	0.35245 (18)	0.0223 (3)
C14	−0.07845 (19)	0.19824 (4)	0.45914 (18)	0.0243 (3)
C1	0.09082 (18)	0.15641 (4)	0.66916 (18)	0.0221 (3)
C2	0.02149 (18)	0.18867 (4)	0.63846 (18)	0.0223 (3)
C6	0.17972 (19)	0.14752 (4)	0.83585 (19)	0.0291 (3)
H6	0.2254	0.1255	0.8577	0.035*
C9	−0.07529 (19)	0.05080 (4)	0.3572 (2)	0.0275 (3)
H9	−0.0945	0.0411	0.4610	0.033*
C12	−0.0237 (2)	0.07837 (4)	0.04937 (19)	0.0310 (3)
H12	−0.0068	0.0879	−0.0557	0.037*
C3	0.0453 (2)	0.21184 (4)	0.7738 (2)	0.0300 (3)
H3	−0.0009	0.2339	0.7528	0.036*
C10	−0.0941 (2)	0.03200 (4)	0.2076 (2)	0.0325 (4)
C11	−0.0716 (2)	0.04501 (4)	0.0527 (2)	0.0334 (4)
H11	−0.0886	0.0314	−0.0490	0.040*
C5	0.2025 (2)	0.17060 (5)	0.9713 (2)	0.0357 (4)
H5	0.2633	0.1643	1.0856	0.043*
C4	0.1367 (2)	0.20270 (4)	0.9394 (2)	0.0364 (4)
H4	0.1543	0.2186	1.0318	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F5	0.0319 (5)	0.0342 (5)	0.0230 (4)	0.0058 (4)	0.0097 (3)	−0.0001 (3)
F6	0.0415 (5)	0.0273 (5)	0.0320 (5)	−0.0069 (4)	0.0132 (4)	0.0028 (4)
F7	0.0296 (5)	0.0343 (5)	0.0208 (4)	0.0041 (4)	0.0002 (3)	0.0055 (3)
O2	0.0234 (5)	0.0309 (6)	0.0180 (5)	0.0025 (4)	0.0015 (4)	0.0013 (4)
N2	0.0225 (6)	0.0228 (6)	0.0182 (6)	−0.0006 (5)	−0.0009 (5)	−0.0026 (5)
F8	0.0592 (7)	0.0246 (5)	0.0447 (6)	0.0047 (4)	0.0078 (5)	−0.0076 (4)
C21	0.0200 (7)	0.0234 (7)	0.0170 (6)	−0.0028 (5)	0.0061 (5)	0.0024 (5)
C22	0.0216 (7)	0.0234 (7)	0.0190 (7)	−0.0043 (5)	0.0070 (5)	0.0002 (5)
C28	0.0243 (7)	0.0229 (7)	0.0223 (7)	0.0008 (6)	0.0042 (6)	0.0006 (5)
C15	0.0197 (7)	0.0230 (7)	0.0182 (7)	0.0010 (5)	0.0011 (5)	−0.0014 (5)
C20	0.0272 (8)	0.0250 (7)	0.0236 (7)	−0.0005 (6)	0.0055 (6)	0.0022 (6)
C23	0.0275 (7)	0.0247 (7)	0.0230 (7)	−0.0017 (6)	0.0057 (6)	0.0013 (6)
C27	0.0246 (7)	0.0258 (7)	0.0204 (7)	−0.0018 (6)	0.0041 (6)	0.0001 (6)
C24	0.0357 (8)	0.0209 (7)	0.0328 (8)	−0.0026 (6)	0.0124 (7)	−0.0031 (6)
C26	0.0298 (8)	0.0331 (8)	0.0196 (7)	−0.0053 (6)	0.0032 (6)	−0.0007 (6)
C25	0.0345 (8)	0.0308 (8)	0.0227 (7)	−0.0105 (6)	0.0081 (6)	−0.0072 (6)
C19	0.0309 (8)	0.0346 (8)	0.0180 (7)	0.0021 (6)	0.0053 (6)	0.0020 (6)

C18	0.0327 (8)	0.0294 (8)	0.0210 (7)	0.0032 (6)	0.0029 (6)	−0.0055 (6)
C17	0.0267 (8)	0.0238 (7)	0.0241 (7)	0.0002 (6)	0.0030 (6)	−0.0027 (6)
C16	0.0199 (7)	0.0235 (7)	0.0193 (7)	0.0023 (5)	0.0028 (5)	0.0008 (6)
F1	0.0357 (5)	0.0376 (5)	0.0238 (4)	0.0038 (4)	0.0115 (4)	0.0090 (4)
F3	0.0284 (5)	0.0391 (5)	0.0241 (4)	−0.0029 (4)	0.0002 (3)	0.0043 (4)
F2	0.0486 (6)	0.0281 (5)	0.0412 (5)	0.0161 (4)	0.0112 (4)	0.0085 (4)
O1	0.0253 (5)	0.0299 (5)	0.0187 (5)	−0.0009 (4)	0.0051 (4)	0.0028 (4)
F4	0.0739 (8)	0.0355 (6)	0.0510 (6)	−0.0214 (5)	0.0300 (6)	−0.0176 (5)
N1	0.0272 (6)	0.0217 (6)	0.0213 (6)	0.0022 (5)	0.0096 (5)	0.0012 (5)
C7	0.0187 (7)	0.0241 (7)	0.0194 (7)	0.0062 (5)	0.0025 (5)	0.0046 (5)
C13	0.0262 (7)	0.0295 (8)	0.0230 (7)	0.0023 (6)	0.0054 (6)	0.0037 (6)
C8	0.0186 (7)	0.0265 (7)	0.0213 (7)	0.0027 (5)	0.0038 (5)	0.0014 (6)
C14	0.0271 (7)	0.0221 (7)	0.0246 (7)	0.0033 (6)	0.0081 (6)	0.0025 (6)
C1	0.0219 (7)	0.0251 (7)	0.0197 (7)	−0.0002 (6)	0.0061 (5)	0.0009 (5)
C2	0.0234 (7)	0.0232 (7)	0.0214 (7)	−0.0005 (6)	0.0072 (5)	0.0005 (6)
C6	0.0267 (8)	0.0348 (8)	0.0247 (8)	0.0019 (6)	0.0040 (6)	0.0072 (6)
C9	0.0284 (8)	0.0295 (8)	0.0258 (7)	−0.0019 (6)	0.0088 (6)	−0.0003 (6)
C12	0.0288 (8)	0.0437 (9)	0.0203 (7)	0.0016 (7)	0.0057 (6)	0.0014 (6)
C3	0.0359 (9)	0.0274 (8)	0.0287 (8)	−0.0034 (6)	0.0115 (7)	−0.0060 (6)
C10	0.0325 (8)	0.0307 (8)	0.0357 (9)	−0.0069 (7)	0.0112 (7)	−0.0077 (7)
C11	0.0298 (8)	0.0444 (10)	0.0264 (8)	−0.0021 (7)	0.0074 (6)	−0.0112 (7)
C5	0.0318 (8)	0.0542 (11)	0.0191 (7)	−0.0077 (8)	0.0022 (6)	0.0032 (7)
C4	0.0410 (9)	0.0450 (10)	0.0244 (8)	−0.0117 (8)	0.0100 (7)	−0.0115 (7)

Geometric parameters (Å, º)

F5—C28	1.3462 (16)	F1—C14	1.3459 (17)
F6—C28	1.3377 (16)	F3—C14	1.3440 (17)
F7—C28	1.3503 (17)	F2—C14	1.3403 (17)
O2—C21	1.2324 (17)	O1—C7	1.2338 (17)
N2—H2	0.889 (18)	F4—C10	1.3583 (19)
N2—C21	1.3438 (18)	N1—H1	0.868 (18)
N2—C15	1.4327 (18)	N1—C7	1.3437 (19)
F8—C24	1.3557 (18)	N1—C1	1.4273 (18)
C21—C22	1.4998 (19)	C7—C8	1.498 (2)
C22—C23	1.392 (2)	C13—H13	0.9500
C22—C27	1.396 (2)	C13—C8	1.398 (2)
C28—C16	1.4993 (19)	C13—C12	1.385 (2)
C15—C20	1.386 (2)	C8—C9	1.392 (2)
C15—C16	1.393 (2)	C14—C2	1.498 (2)
C20—H20	0.9500	C1—C2	1.396 (2)
C20—C19	1.386 (2)	C1—C6	1.383 (2)
C23—H23	0.9500	C2—C3	1.388 (2)
C23—C24	1.378 (2)	C6—H6	0.9500
C27—H27	0.9500	C6—C5	1.387 (2)
C27—C26	1.389 (2)	C9—H9	0.9500
C24—C25	1.378 (2)	C9—C10	1.375 (2)
C26—H26	0.9500	C12—H12	0.9500

C26—C25	1.383 (2)	C12—C11	1.383 (2)
C25—H25	0.9500	C3—H3	0.9500
C19—H19	0.9500	C3—C4	1.385 (2)
C19—C18	1.382 (2)	C10—C11	1.378 (2)
C18—H18	0.9500	C11—H11	0.9500
C18—C17	1.387 (2)	C5—H5	0.9500
C17—H17	0.9500	C5—C4	1.381 (3)
C17—C16	1.391 (2)	C4—H4	0.9500

C21—N2—H2	121.3 (12)	C7—N1—H1	120.6 (11)
C21—N2—C15	121.57 (12)	C7—N1—C1	122.97 (12)
C15—N2—H2	115.8 (12)	C1—N1—H1	115.8 (11)
O2—C21—N2	121.88 (12)	O1—C7—N1	122.38 (13)
O2—C21—C22	120.67 (12)	O1—C7—C8	121.14 (13)
N2—C21—C22	117.41 (12)	N1—C7—C8	116.45 (12)
C23—C22—C21	116.60 (12)	C8—C13—H13	120.0
C23—C22—C27	119.84 (13)	C12—C13—H13	120.0
C27—C22—C21	123.51 (13)	C12—C13—C8	120.07 (14)
F5—C28—F7	105.63 (11)	C13—C8—C7	122.83 (13)
F5—C28—C16	112.95 (11)	C9—C8—C7	117.21 (12)
F6—C28—F5	106.38 (11)	C9—C8—C13	119.90 (13)
F6—C28—F7	106.41 (11)	F1—C14—C2	112.80 (12)
F6—C28—C16	112.65 (11)	F3—C14—F1	105.76 (11)
F7—C28—C16	112.27 (11)	F3—C14—C2	113.12 (11)
C20—C15—N2	119.58 (12)	F2—C14—F1	105.87 (11)
C20—C15—C16	119.81 (13)	F2—C14—F3	106.14 (11)
C16—C15—N2	120.58 (12)	F2—C14—C2	112.54 (12)
C15—C20—H20	119.9	C2—C1—N1	119.57 (12)
C19—C20—C15	120.13 (13)	C6—C1—N1	120.87 (13)
C19—C20—H20	119.9	C6—C1—C2	119.52 (13)
C22—C23—H23	120.8	C1—C2—C14	119.84 (12)
C24—C23—C22	118.46 (13)	C3—C2—C14	120.09 (13)
C24—C23—H23	120.8	C3—C2—C1	120.06 (13)
C22—C27—H27	120.1	C1—C6—H6	119.8
C26—C27—C22	119.85 (14)	C1—C6—C5	120.40 (15)
C26—C27—H27	120.1	C5—C6—H6	119.8
F8—C24—C23	118.46 (14)	C8—C9—H9	120.9
F8—C24—C25	118.60 (13)	C10—C9—C8	118.16 (14)
C25—C24—C23	122.93 (14)	C10—C9—H9	120.9
C27—C26—H26	119.6	C13—C12—H12	119.8
C25—C26—C27	120.80 (14)	C11—C12—C13	120.45 (14)
C25—C26—H26	119.6	C11—C12—H12	119.8
C24—C25—C26	118.10 (13)	C2—C3—H3	120.1
C24—C25—H25	121.0	C4—C3—C2	119.71 (15)
C26—C25—H25	121.0	C4—C3—H3	120.1
C20—C19—H19	119.9	F4—C10—C9	118.27 (14)
C18—C19—C20	120.20 (13)	F4—C10—C11	118.59 (14)
C18—C19—H19	119.9	C9—C10—C11	123.14 (15)

C19—C18—H18	120.0	C12—C11—H11	120.9
C19—C18—C17	120.04 (13)	C10—C11—C12	118.26 (14)
C17—C18—H18	120.0	C10—C11—H11	120.9
C18—C17—H17	120.0	C6—C5—H5	120.1
C18—C17—C16	119.99 (14)	C4—C5—C6	119.84 (14)
C16—C17—H17	120.0	C4—C5—H5	120.1
C15—C16—C28	120.12 (12)	C3—C4—H4	119.8
C17—C16—C28	120.05 (13)	C5—C4—C3	120.45 (14)
C17—C16—C15	119.82 (13)	C5—C4—H4	119.8

F5—C28—C16—C15	54.98 (17)	F1—C14—C2—C1	−64.46 (17)
F5—C28—C16—C17	−126.11 (14)	F1—C14—C2—C3	115.45 (14)
F6—C28—C16—C15	175.54 (12)	F3—C14—C2—C1	55.52 (17)
F6—C28—C16—C17	−5.54 (18)	F3—C14—C2—C3	−124.56 (14)
F7—C28—C16—C15	−64.33 (16)	F2—C14—C2—C1	175.83 (12)
F7—C28—C16—C17	114.58 (14)	F2—C14—C2—C3	−4.26 (19)
O2—C21—C22—C23	−12.78 (18)	O1—C7—C8—C13	157.12 (13)
O2—C21—C22—C27	164.63 (13)	O1—C7—C8—C9	−19.98 (19)
N2—C21—C22—C23	169.55 (12)	F4—C10—C11—C12	−178.75 (14)
N2—C21—C22—C27	−13.04 (19)	N1—C7—C8—C13	−20.83 (19)
N2—C15—C20—C19	177.07 (13)	N1—C7—C8—C9	162.08 (13)
N2—C15—C16—C28	1.5 (2)	N1—C1—C2—C14	3.9 (2)
N2—C15—C16—C17	−177.37 (13)	N1—C1—C2—C3	−176.01 (13)
F8—C24—C25—C26	−178.96 (13)	N1—C1—C6—C5	176.39 (14)
C21—N2—C15—C20	68.06 (18)	C7—N1—C1—C2	−115.64 (15)
C21—N2—C15—C16	−113.80 (15)	C7—N1—C1—C6	66.89 (19)
C21—C22—C23—C24	176.07 (13)	C7—C8—C9—C10	177.40 (13)
C21—C22—C27—C26	−175.71 (13)	C13—C8—C9—C10	0.2 (2)
C22—C23—C24—F8	−179.84 (13)	C13—C12—C11—C10	−0.4 (2)
C22—C23—C24—C25	0.0 (2)	C8—C13—C12—C11	−0.8 (2)
C22—C27—C26—C25	−0.4 (2)	C8—C9—C10—F4	178.85 (14)
C15—N2—C21—O2	2.8 (2)	C8—C9—C10—C11	−1.5 (2)
C15—N2—C21—C22	−179.58 (12)	C14—C2—C3—C4	179.50 (14)
C15—C20—C19—C18	0.8 (2)	C1—N1—C7—O1	−1.2 (2)
C20—C15—C16—C28	179.68 (13)	C1—N1—C7—C8	176.74 (12)
C20—C15—C16—C17	0.8 (2)	C1—C2—C3—C4	−0.6 (2)
C20—C19—C18—C17	−0.1 (2)	C1—C6—C5—C4	−0.3 (2)
C23—C22—C27—C26	1.6 (2)	C2—C1—C6—C5	−1.1 (2)
C23—C24—C25—C26	1.2 (2)	C2—C3—C4—C5	−0.8 (2)
C27—C22—C23—C24	−1.4 (2)	C6—C1—C2—C14	−178.59 (13)
C27—C26—C25—C24	−1.0 (2)	C6—C1—C2—C3	1.5 (2)
C19—C18—C17—C16	−0.2 (2)	C6—C5—C4—C3	1.2 (2)
C18—C17—C16—C28	−179.05 (13)	C9—C10—C11—C12	1.6 (3)
C18—C17—C16—C15	−0.1 (2)	C12—C13—C8—C7	−176.08 (13)
C16—C15—C20—C19	−1.1 (2)	C12—C13—C8—C9	0.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.87 (2)	2.01 (2)	2.8239 (16)	157 (1)
N2—H2···O1^{i}	0.89 (2)	1.99 (2)	2.8303 (16)	158 (1)
C5—H5···O2^{ii}	0.95	2.35	3.2861 (18)	167
C12—H12···O1^{iii}	0.95	2.45	3.3172 (17)	152

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

(II) 3-Bromo-*N*-[2-(trifluoromethyl)phenyl]benzamide

Crystal data

$C_{14}H_9BrF_3NO$
M_r = 344.13
Monoclinic, $P2_1/n$
a = 12.9456 (6) Å
b = 4.7377 (2) Å
c = 21.9025 (10) Å
β = 104.770 (2)°
V = 1298.94 (10) Å^3
Z = 4
$F(000)$ = 680

Prism
D_x = 1.760 Mg m^{-3}
Melting point: 369 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 132 reflections
θ = 6.4–64.4°
μ = 4.63 mm^{-1}
T = 173 K
Prism, colourless
0.28 × 0.24 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.315, T_{max} = 0.396

8466 measured reflections
2114 independent reflections
1986 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 64.4°, θ_{min} = 6.4°
h = −14→15
k = −5→4
l = −24→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.090
S = 1.05
2114 reflections
185 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 0.3564P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
H1	1.167 (2)	0.262 (5)	0.0800 (14)	0.022 (7)*
Br1	0.85044 (2)	0.78177 (6)	0.240898 (12)	0.02952 (16)
F3	1.32385 (11)	0.0139 (3)	0.09354 (7)	0.0309 (4)
F1	1.46295 (12)	0.0991 (4)	0.06107 (8)	0.0414 (4)
O1	1.10042 (14)	0.8797 (4)	0.07865 (8)	0.0262 (4)
C12	1.1116 (2)	0.2628 (5)	0.25054 (12)	0.0254 (6)
H12	1.1500	0.1231	0.2784	0.031*
F2	1.41294 (13)	0.3932 (3)	0.12181 (7)	0.0379 (4)
C14	1.3768 (2)	0.2193 (5)	0.07210 (13)	0.0249 (6)
C13	1.1429 (2)	0.3313 (5)	0.19641 (12)	0.0240 (5)
H13	1.2016	0.2364	0.1869	0.029*
C3	1.3518 (2)	0.4176 (6)	−0.03571 (12)	0.0285 (6)
H3	1.4176	0.3321	−0.0371	0.034*
N1	1.16903 (15)	0.4434 (4)	0.07018 (9)	0.0210 (4)
C9	1.0021 (2)	0.6762 (5)	0.17009 (12)	0.0232 (5)
H9	0.9649	0.8205	0.1431	0.028*
C8	1.08811 (17)	0.5397 (5)	0.15593 (11)	0.0199 (5)
C1	1.21188 (18)	0.4948 (5)	0.01743 (11)	0.0207 (5)
C11	1.0248 (2)	0.3955 (5)	0.26468 (11)	0.0267 (5)
H11	1.0030	0.3468	0.3016	0.032*
C6	1.1575 (2)	0.6606 (5)	−0.03303 (12)	0.0254 (5)
H6	1.0908	0.7434	−0.0328	0.030*
C10	0.97086 (18)	0.6007 (5)	0.22362 (11)	0.0232 (5)
C5	1.2015 (2)	0.7035 (6)	−0.08358 (13)	0.0297 (6)
H5	1.1646	0.8180	−0.1178	0.036*
C2	1.31040 (18)	0.3751 (5)	0.01629 (11)	0.0217 (5)
C4	1.2976 (2)	0.5838 (6)	−0.08530 (12)	0.0317 (6)
H4	1.3264	0.6155	−0.1205	0.038*
C7	1.11912 (18)	0.6373 (5)	0.09788 (11)	0.0201 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0248 (2)	0.0363 (2)	0.0329 (2)	0.00046 (9)	0.01727 (14)	−0.00145 (10)
F3	0.0284 (7)	0.0269 (8)	0.0370 (8)	0.0009 (6)	0.0073 (6)	0.0084 (6)
F1	0.0305 (8)	0.0536 (11)	0.0446 (9)	0.0194 (8)	0.0177 (7)	0.0089 (8)
O1	0.0314 (9)	0.0173 (9)	0.0350 (10)	0.0019 (7)	0.0179 (7)	0.0024 (7)
C12	0.0288 (15)	0.0243 (13)	0.0234 (14)	0.0018 (10)	0.0071 (11)	0.0019 (9)
F2	0.0466 (9)	0.0294 (8)	0.0297 (8)	−0.0022 (7)	−0.0048 (7)	−0.0037 (6)
C14	0.0235 (13)	0.0246 (13)	0.0281 (14)	0.0012 (10)	0.0093 (11)	−0.0042 (9)

C13	0.0218 (12)	0.0228 (12)	0.0286 (13)	−0.0004 (10)	0.0085 (10)	−0.0024 (10)
C3	0.0260 (12)	0.0333 (15)	0.0295 (13)	−0.0006 (11)	0.0134 (10)	−0.0044 (11)
N1	0.0236 (10)	0.0167 (10)	0.0262 (10)	0.0021 (8)	0.0130 (8)	0.0024 (8)
C9	0.0239 (12)	0.0191 (11)	0.0283 (13)	−0.0015 (9)	0.0100 (10)	−0.0013 (9)
C8	0.0180 (11)	0.0183 (11)	0.0250 (12)	−0.0041 (9)	0.0085 (9)	−0.0012 (9)
C1	0.0223 (11)	0.0181 (11)	0.0234 (11)	−0.0035 (9)	0.0093 (9)	−0.0035 (9)
C11	0.0305 (13)	0.0291 (14)	0.0207 (12)	−0.0075 (11)	0.0066 (10)	−0.0018 (10)
C6	0.0247 (12)	0.0238 (12)	0.0278 (13)	0.0004 (10)	0.0069 (10)	−0.0005 (10)
C10	0.0204 (11)	0.0241 (13)	0.0281 (12)	−0.0037 (10)	0.0117 (10)	−0.0042 (10)
C5	0.0359 (16)	0.0322 (14)	0.0207 (14)	−0.0005 (11)	0.0066 (11)	0.0026 (10)
C2	0.0211 (11)	0.0214 (12)	0.0235 (12)	−0.0008 (10)	0.0076 (9)	−0.0037 (9)
C4	0.0349 (14)	0.0394 (15)	0.0249 (13)	−0.0041 (12)	0.0151 (11)	−0.0005 (11)
C7	0.0176 (11)	0.0176 (12)	0.0264 (12)	−0.0039 (9)	0.0077 (9)	−0.0016 (9)

Geometric parameters (Å, º)

Br1—C10	1.900 (2)	N1—C1	1.424 (3)
F3—C14	1.342 (3)	N1—H1	0.89 (3)
F1—C14	1.328 (3)	C9—C10	1.381 (3)
O1—C7	1.226 (3)	C9—C8	1.390 (3)
C12—C13	1.386 (4)	C9—H9	0.9500
C12—C11	1.389 (4)	C8—C7	1.500 (3)
C12—H12	0.9500	C1—C6	1.392 (3)
F2—C14	1.350 (3)	C1—C2	1.402 (3)
C14—C2	1.497 (4)	C11—C10	1.386 (4)
C13—C8	1.394 (3)	C11—H11	0.9500
C13—H13	0.9500	C6—C5	1.383 (4)
C3—C4	1.380 (4)	C6—H6	0.9500
C3—C2	1.392 (3)	C5—C4	1.376 (4)
C3—H3	0.9500	C5—H5	0.9500
N1—C7	1.353 (3)	C4—H4	0.9500
C13—C12—C11	121.0 (2)	C6—C1—C2	119.5 (2)
C13—C12—H12	119.5	C6—C1—N1	121.2 (2)
C11—C12—H12	119.5	C2—C1—N1	119.3 (2)
F1—C14—F3	106.3 (2)	C10—C11—C12	118.4 (2)
F1—C14—F2	105.9 (2)	C10—C11—H11	120.8
F3—C14—F2	105.3 (2)	C12—C11—H11	120.8
F1—C14—C2	113.4 (2)	C5—C6—C1	119.4 (2)
F3—C14—C2	113.9 (2)	C5—C6—H6	120.3
F2—C14—C2	111.5 (2)	C1—C6—H6	120.3
C12—C13—C8	119.8 (2)	C9—C10—C11	121.6 (2)
C12—C13—H13	120.1	C9—C10—Br1	118.99 (19)
C8—C13—H13	120.1	C11—C10—Br1	119.40 (18)
C4—C3—C2	120.1 (2)	C4—C5—C6	121.4 (2)
C4—C3—H3	119.9	C4—C5—H5	119.3
C2—C3—H3	119.9	C6—C5—H5	119.3
C7—N1—C1	125.2 (2)	C3—C2—C1	119.9 (2)

C7—N1—H1	120 (2)	C3—C2—C14	118.6 (2)
C1—N1—H1	114 (2)	C1—C2—C14	121.4 (2)
C10—C9—C8	119.6 (2)	C5—C4—C3	119.7 (2)
C10—C9—H9	120.2	C5—C4—H4	120.1
C8—C9—H9	120.2	C3—C4—H4	120.1
C9—C8—C13	119.6 (2)	O1—C7—N1	123.9 (2)
C9—C8—C7	116.6 (2)	O1—C7—C8	120.5 (2)
C13—C8—C7	123.7 (2)	N1—C7—C8	115.6 (2)
C11—C12—C13—C8	1.2 (4)	N1—C1—C2—C3	178.3 (2)
C10—C9—C8—C13	−1.0 (3)	C6—C1—C2—C14	173.9 (2)
C10—C9—C8—C7	−178.6 (2)	N1—C1—C2—C14	−6.4 (3)
C12—C13—C8—C9	−0.3 (4)	F1—C14—C2—C3	−10.1 (3)
C12—C13—C8—C7	177.1 (2)	F3—C14—C2—C3	−131.8 (2)
C7—N1—C1—C6	−40.8 (3)	F2—C14—C2—C3	109.3 (3)
C7—N1—C1—C2	139.5 (2)	F1—C14—C2—C1	174.5 (2)
C13—C12—C11—C10	−0.7 (4)	F3—C14—C2—C1	52.8 (3)
C2—C1—C6—C5	0.2 (4)	F2—C14—C2—C1	−66.1 (3)
N1—C1—C6—C5	−179.5 (2)	C6—C5—C4—C3	−0.1 (4)
C8—C9—C10—C11	1.5 (4)	C2—C3—C4—C5	−1.1 (4)
C8—C9—C10—Br1	−178.14 (17)	C1—N1—C7—O1	3.6 (4)
C12—C11—C10—C9	−0.6 (4)	C1—N1—C7—C8	−175.5 (2)
C12—C11—C10—Br1	178.98 (18)	C9—C8—C7—O1	27.0 (3)
C1—C6—C5—C4	0.5 (4)	C13—C8—C7—O1	−150.5 (2)
C4—C3—C2—C1	1.8 (4)	C9—C8—C7—N1	−153.9 (2)
C4—C3—C2—C14	−173.6 (2)	C13—C8—C7—N1	28.5 (3)
C6—C1—C2—C3	−1.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89 (2)	2.00 (2)	2.835 (2)	156 (3)

Symmetry code: (i) *x*, *y*−1, *z*.

(III) 3-Iodo-*N*-[2-(trifluoromethyl)phenyl]benzamide

Crystal data

$C_{14}H_9F_3INO$
M_r = 391.12
Monoclinic, $P2_1/n$
a = 13.3358 (6) Å
b = 4.7471 (2) Å
c = 22.3558 (10) Å
β = 105.848 (2)°
V = 1361.47 (10) Å^3
Z = 4
F(000) = 752

Prism
D_x = 1.908 Mg m^{-3}
Melting point: 393 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 131 reflections
θ = 6.2–64.3°
μ = 18.78 mm^{-1}
T = 173 K
Prism, colourless
0.27 × 0.22 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.081, T_{max} = 0.133
7120 measured reflections
2223 independent reflections
2124 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 64.3°, θ_{min} = 6.2°
h = −15→14
k = −5→5
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.109
S = 1.09
2223 reflections
185 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.84 e Å^{-3}
$\Delta\rho_{min}$ = −1.41 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C12	1.1208 (4)	0.7461 (9)	0.2483 (2)	0.0187 (10)
H12	1.1593	0.8842	0.2760	0.022*
H1	1.167 (4)	0.751 (7)	0.081 (2)	0.017 (13)*
I1	0.85495 (2)	0.21117 (7)	0.240621 (11)	0.02136 (18)
F1	1.32578 (18)	0.9967 (6)	0.09091 (11)	0.0280 (6)
F3	1.4613 (2)	0.8794 (9)	0.06351 (14)	0.0448 (8)
C13	1.1487 (4)	0.6826 (9)	0.19429 (19)	0.0196 (9)
H13	1.2047	0.7794	0.1847	0.024*
O1	1.1058 (2)	0.1341 (7)	0.07951 (13)	0.0225 (7)
C3	1.3480 (3)	0.5671 (12)	−0.03282 (19)	0.0270 (11)
H3	1.4136	0.6425	−0.0335	0.032*
F2	1.4039 (2)	0.6087 (7)	0.12348 (11)	0.0360 (6)
C9	1.0116 (3)	0.3381 (9)	0.16847 (18)	0.0177 (8)
H9	0.9745	0.1950	0.1417	0.021*
C8	1.0943 (3)	0.4774 (9)	0.15463 (16)	0.0147 (8)

C14	1.3742 (4)	0.7780 (9)	0.0729 (2)	0.0202 (10)
N1	1.1697 (2)	0.5723 (8)	0.06981 (14)	0.0165 (7)
C11	1.0390 (3)	0.6134 (10)	0.26236 (17)	0.0204 (9)
H11	1.0203	0.6594	0.2992	0.025*
C6	1.1542 (3)	0.3508 (10)	−0.03154 (19)	0.0216 (9)
H6	1.0877	0.2776	−0.0320	0.026*
C7	1.1231 (3)	0.3789 (10)	0.09730 (18)	0.0165 (9)
C10	0.9841 (3)	0.4115 (9)	0.22197 (17)	0.0167 (8)
C1	1.2104 (3)	0.5142 (9)	0.01811 (16)	0.0154 (8)
C5	1.1976 (5)	0.2967 (10)	−0.0806 (2)	0.0277 (11)
H5	1.1602	0.1826	−0.1143	0.033*
C2	1.3078 (3)	0.6222 (10)	0.01769 (17)	0.0184 (8)
C4	1.2923 (4)	0.4035 (12)	−0.08166 (18)	0.0302 (11)
H4	1.3197	0.3651	−0.1158	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C12	0.016 (2)	0.022 (3)	0.017 (2)	0.0021 (16)	0.0033 (19)	−0.0030 (16)
I1	0.0206 (2)	0.0284 (3)	0.0216 (2)	0.00032 (9)	0.01687 (16)	0.00060 (9)
F1	0.0282 (13)	0.0237 (15)	0.0317 (12)	−0.0011 (11)	0.0074 (11)	−0.0056 (11)
F3	0.0302 (15)	0.067 (2)	0.0431 (16)	−0.0210 (16)	0.0205 (13)	−0.0115 (17)
C13	0.027 (2)	0.018 (3)	0.017 (2)	0.0013 (18)	0.0107 (18)	0.0035 (16)
O1	0.0341 (17)	0.0156 (17)	0.0265 (15)	−0.0004 (14)	0.0233 (13)	−0.0033 (13)
C3	0.025 (2)	0.044 (3)	0.0185 (19)	0.008 (2)	0.0166 (17)	0.006 (2)
F2	0.0484 (16)	0.0305 (17)	0.0216 (12)	0.0002 (13)	−0.0034 (11)	0.0045 (12)
C9	0.019 (2)	0.019 (2)	0.0179 (19)	0.0030 (17)	0.0096 (16)	0.0005 (16)
C8	0.0173 (17)	0.016 (2)	0.0145 (16)	0.0041 (16)	0.0104 (14)	0.0004 (15)
C14	0.020 (3)	0.026 (3)	0.017 (2)	−0.0005 (17)	0.0094 (19)	0.0018 (16)
N1	0.0217 (16)	0.017 (2)	0.0167 (15)	−0.0002 (14)	0.0150 (13)	−0.0016 (14)
C11	0.022 (2)	0.028 (3)	0.0143 (18)	0.0093 (19)	0.0097 (16)	−0.0019 (18)
C6	0.024 (2)	0.024 (3)	0.0190 (19)	0.0005 (19)	0.0092 (17)	−0.0009 (18)
C7	0.0177 (19)	0.017 (2)	0.0181 (19)	0.0047 (18)	0.0108 (15)	0.0015 (18)
C10	0.0164 (17)	0.020 (2)	0.0187 (18)	0.0016 (17)	0.0133 (15)	0.0041 (17)
C1	0.0194 (18)	0.017 (2)	0.0130 (16)	0.0061 (16)	0.0099 (15)	0.0036 (15)
C5	0.040 (3)	0.033 (3)	0.011 (2)	0.004 (2)	0.009 (2)	−0.0045 (17)
C2	0.024 (2)	0.019 (2)	0.0157 (18)	0.0059 (18)	0.0097 (16)	0.0016 (17)
C4	0.037 (2)	0.044 (3)	0.0166 (19)	0.010 (2)	0.0203 (18)	0.002 (2)

Geometric parameters (Å, º)

C12—H12	0.9500	C9—C10	1.387 (5)
C12—C13	1.390 (6)	C8—C7	1.509 (5)
C12—C11	1.368 (7)	C14—C2	1.502 (6)
I1—C10	2.106 (4)	N1—H1	0.89 (3)
F1—C14	1.342 (5)	N1—C7	1.348 (6)
F3—C14	1.326 (6)	N1—C1	1.431 (4)
C13—H13	0.9500	C11—H11	0.9500

C13—C8	1.382 (6)	C11—C10	1.382 (6)
O1—C7	1.230 (6)	C6—H6	0.9500
C3—H3	0.9500	C6—C1	1.393 (6)
C3—C2	1.400 (5)	C6—C5	1.397 (6)
C3—C4	1.380 (7)	C1—C2	1.399 (6)
F2—C14	1.356 (5)	C5—H5	0.9500
C9—H9	0.9500	C5—C4	1.368 (8)
C9—C8	1.391 (6)	C4—H4	0.9500
C13—C12—H12	119.4	C12—C11—H11	120.6
C11—C12—H12	119.4	C12—C11—C10	118.9 (3)
C11—C12—C13	121.2 (5)	C10—C11—H11	120.6
C12—C13—H13	120.3	C1—C6—H6	120.7
C8—C13—C12	119.5 (4)	C1—C6—C5	118.7 (4)
C8—C13—H13	120.3	C5—C6—H6	120.7
C2—C3—H3	119.9	O1—C7—C8	119.9 (4)
C4—C3—H3	119.9	O1—C7—N1	124.4 (3)
C4—C3—C2	120.1 (4)	N1—C7—C8	115.6 (4)
C8—C9—H9	120.5	C9—C10—I1	118.7 (3)
C10—C9—H9	120.5	C11—C10—I1	120.0 (2)
C10—C9—C8	119.0 (4)	C11—C10—C9	121.3 (4)
C13—C8—C9	120.1 (3)	C6—C1—N1	120.7 (3)
C13—C8—C7	123.5 (4)	C6—C1—C2	119.8 (3)
C9—C8—C7	116.3 (4)	C2—C1—N1	119.5 (3)
F1—C14—F2	105.2 (3)	C6—C5—H5	119.1
F1—C14—C2	113.8 (4)	C4—C5—C6	121.9 (4)
F3—C14—F1	106.3 (4)	C4—C5—H5	119.1
F3—C14—F2	106.2 (4)	C3—C2—C14	119.0 (4)
F3—C14—C2	113.2 (4)	C1—C2—C3	119.8 (4)
F2—C14—C2	111.5 (4)	C1—C2—C14	121.0 (3)
C7—N1—H1	117 (3)	C3—C4—H4	120.2
C7—N1—C1	124.1 (4)	C5—C4—C3	119.7 (4)
C1—N1—H1	118 (3)	C5—C4—H4	120.2
C12—C13—C8—C9	0.7 (6)	N1—C1—C2—C14	5.1 (6)
C12—C13—C8—C7	−175.6 (4)	C11—C12—C13—C8	−1.4 (7)
C12—C11—C10—I1	−178.2 (3)	C6—C1—C2—C3	0.4 (6)
C12—C11—C10—C9	1.2 (6)	C6—C1—C2—C14	−174.7 (4)
F1—C14—C2—C3	129.8 (4)	C6—C5—C4—C3	0.7 (8)
F1—C14—C2—C1	−55.0 (5)	C7—N1—C1—C6	43.4 (6)
F3—C14—C2—C3	8.3 (6)	C7—N1—C1—C2	−136.4 (4)
F3—C14—C2—C1	−176.5 (4)	C10—C9—C8—C13	0.9 (6)
C13—C12—C11—C10	0.5 (7)	C10—C9—C8—C7	177.4 (4)
C13—C8—C7—O1	148.7 (4)	C1—N1—C7—O1	−3.5 (6)
C13—C8—C7—N1	−29.7 (6)	C1—N1—C7—C8	174.8 (3)
F2—C14—C2—C3	−111.3 (5)	C1—C6—C5—C4	−1.1 (7)
F2—C14—C2—C1	63.9 (5)	C5—C6—C1—N1	−179.2 (4)
C9—C8—C7—O1	−27.7 (5)	C5—C6—C1—C2	0.6 (6)

C9—C8—C7—N1	153.9 (4)	C2—C3—C4—C5	0.4 (8)
C8—C9—C10—I1	177.5 (3)	C4—C3—C2—C14	174.4 (4)
C8—C9—C10—C11	−1.9 (6)	C4—C3—C2—C1	−0.9 (7)
N1—C1—C2—C3	−179.8 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89 (3)	1.99 (4)	2.826 (5)	156 (5)

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