

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# 1-[4-Bromo-2-(trifluoromethoxy)phenyl]-3-methyl-1*H*-1,2,4-triazole

C Sandeep,<sup>a</sup> Basavaraj Padmashali,<sup>a,b\*</sup> P. A. Suchetan<sup>c</sup> and Rashmi S. Kulkarni<sup>d</sup>

<sup>a</sup>Department of Chemistry, Sahyadri Science College (Autonomous), Shimoga 577 203, Karnataka, India, <sup>b</sup>Department of Studies and Research in Chemistry, School of Basic Sciences, Rani Channamma University, Belagavi 591 156, Karnataka, India, <sup>c</sup>Department of Studies in Chemistry, U.C.S., Tumkur University, Tumkur, Karnataka 572 103, India, and <sup>d</sup>PG Department of Chemistry, Jain University, Bangalore 560 019, Karnataka, India  
Correspondence e-mail: basavarajpadmashali@yahoo.com

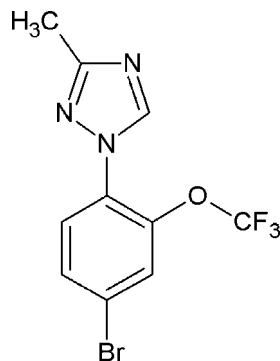
Received 23 April 2014; accepted 5 May 2014

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.135; data-to-parameter ratio = 21.2.

In the title compound,  $\text{C}_{10}\text{H}_7\text{BrF}_3\text{N}_3\text{O}$ , the dihedral angle between the benzene and triazole rings is  $23.17(12)^\circ$  and the C atom of the  $-\text{CF}_3$  group deviates from its attached ring plane by  $1.147(3)$  Å. In the crystal, molecules are linked by  $\text{C}-\text{H}\cdots\text{N}$  interactions, generating  $C(7)$  chains running along  $[010]$ .

## Related literature

For the antibacterial activity of 1,2,4-triazoles, see: Gabriela *et al.* (2009); Palekar *et al.* (2009). For their antiviral activity, see: Upmanyu *et al.* (2006). For antimicrobial agents, see: Badr & Barwa (2011), and for antimycotic activity such as voriconazole, see: Haber (2001).



## Experimental

### Crystal data

$\text{C}_{10}\text{H}_7\text{BrF}_3\text{N}_3\text{O}$   
 $M_r = 322.10$   
 Monoclinic,  $P2_1/n$   
 $a = 5.2389(3)$  Å  
 $b = 16.1548(8)$  Å  
 $c = 14.0315(7)$  Å  
 $\beta = 92.673(3)^\circ$   
 $V = 1186.24(11)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 3.50$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.33 \times 0.21 \times 0.14$  mm

### Data collection

Bruker APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2009)  
 $T_{\min} = 0.419$ ,  $T_{\max} = 0.613$   
 37092 measured reflections  
 3499 independent reflections  
 2373 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.051$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.135$   
 $S = 0.99$   
 3499 reflections  
 165 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.41$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.68$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2}\cdots\text{N3}^i$	0.93	2.59	3.511 (3)	169

Symmetry code: (i)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ .

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT-Plus (Bruker, 2009); data reduction: SAINT-Plus; 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.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7221).

## References

- Badr, S. M. I. & Barwa, R. M. (2011). *Bioorg. Med. Chem.* **19**, 4506–4512.  
 Bruker (2009). APEX2, SADABS and SAINT-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Gabriela, L. A., Stefania, F. B., Almagan, E., Draghici, C. & Saramet, G. (2009). *Eur. J. Med. Chem.* **44**, 3083–3089.  
 Haber, J. (2001). *Cas. Lek. Cesk.* **140**, 596–604.  
 Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.  
 Palekar, V. S., Damle, A. J. & Shukla, S. R. (2009). *Eur. J. Med. Chem.* **44**, 5112–5116.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Upmanyu, N., Garg, G., Dolly, A., Gupta, B. N. & Mishra, P. (2006). *Pharmainfo*, **4**, 3.

## supplementary materials

*Acta Cryst.* (2014). E70, o654 [doi:10.1107/S1600536814010083]

**1-[4-Bromo-2-(trifluoromethoxy)phenyl]-3-methyl-1H-1,2,4-triazole**

**C Sandeep, Basavaraj Padmashali, P. A. Suchetan and Rashmi S. Kulkarni**

**1. Introduction**

1,2,4-triazole containing ring system have been incorporated into a wide variety of therapeutically interesting drug candidates including anti-inflammatory, CNS stimulants sedatives, antibacterial (Gabriela *et al.*, 2009, Palekar *et al.*, 2009), antiviral (Upmanyu *et al.*, 2006), antimicrobial agents (Badr *et al.*, 2011) and antimycotic activity such as fluconazole, intraconazole and voriconazole (Haber *et al.*, 2001). The search for new agent is one of the most challenging tasks to a medicinal chemist. The synthesis of high nitrogen containing heterocyclic systems has been attracting increasing interest over the past decade because of their utility in various applications. In recent years, the chemistry of triazoles and their fused heterocyclic derivatives has received considerable attention owing to their synthetic and effective biological importance. The presence of three nitrogen hetero-atoms in five membered ring system defines an interesting class of compounds. Keeping this in mind, we synthesized the title compound to study its crystal structure.

**2. Experimental****2.1. Synthesis and crystallization**

To a stirred solution of 4-bromo-1-iodo-2-(trifluoromethoxy)benzene (1 g, 2.73mmol) in N,N-Dimethyl Formamide (10 mL), was added potassium carbonate (0.41g, 3.0 mmol), followed by 3-methyl-1H-1,2,4-triazole (0.23g 2.73 mmol). The mixture was stirred at room temperature for 30 minutes. Completion of the reaction was monitored by TLC. The reaction mixture was poured to 100g of crushed ice and the separated solid was filtered off and dried under vacuum.

Single crystals of the title compound were obtained from hexane-ethyl acetate (1:1 v/v) solvent system.

**2.2. Refinement**

Crystal data, data collection and structure refinement details are summarized in Table 1.

**3. Results and discussion**

In the title compound, C<sub>10</sub>H<sub>7</sub>BrF<sub>3</sub>N<sub>3</sub>O, the dihedral angle between the two planes defined by the benzene ring and the triazole ring is 23.17 (12)°. In the crystal structure, the molecules are linked to one another through C2—H2···N3 interactions generating zig zag C(7) chains running along [010].

**4. Refinement**

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å. The isotropic displacement parameters for all H atoms were set to 1.2–1.5 times  $U_{eq}$  (Carbon).

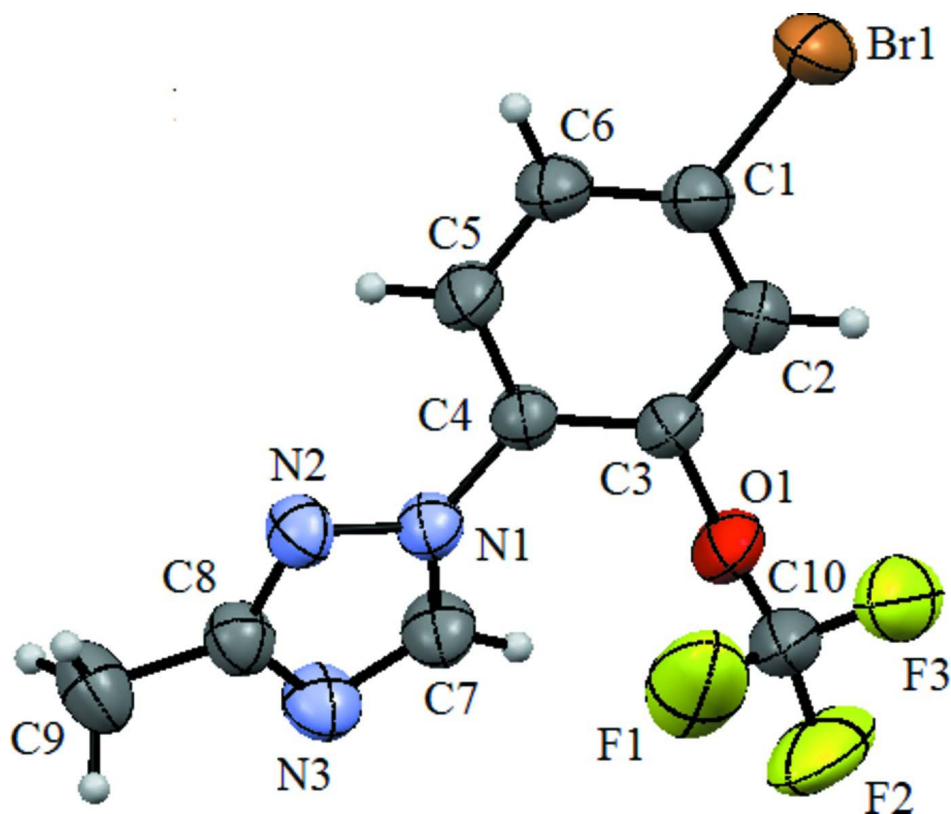


Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

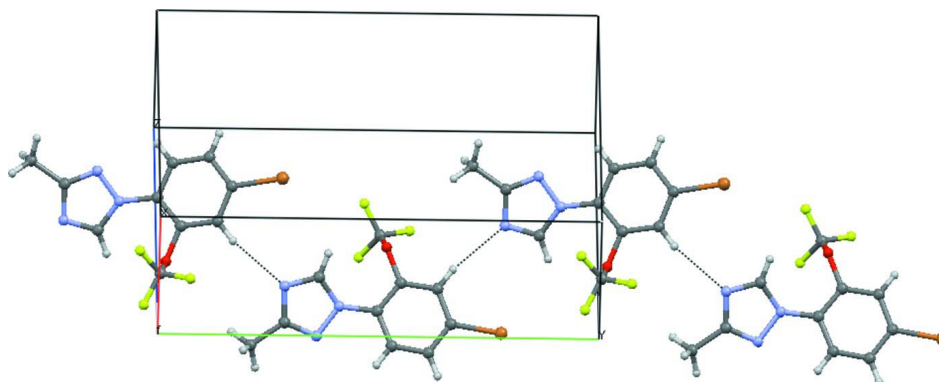


Figure 2

Linking of molecules in the crystal structure through C—H...N interactions into C(7) chains.

### 1-[4-Bromo-2-(trifluoromethoxy)phenyl]-3-methyl-1*H*-1,2,4-triazole

#### Crystal data

$C_{10}H_7BrF_3N_3O$

$M_r = 322.10$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 5.2389\ (3)\ \text{\AA}$

$b = 16.1548\ (8)\ \text{\AA}$

$c = 14.0315\ (7)\ \text{\AA}$

$\beta = 92.673\ (3)^\circ$

$V = 1186.24\ (11)\ \text{\AA}^3$

$Z = 4$

$F(000) = 632$

Prism

$D_x = 1.804 \text{ Mg m}^{-3}$   
 Melting point: 453 K  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 1.9\text{--}30.1^\circ$

$\mu = 3.50 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Prism, colourless  
 $0.33 \times 0.21 \times 0.14 \text{ mm}$

*Data collection*

Bruker APEXII CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2009)  
 $T_{\min} = 0.419$ ,  $T_{\max} = 0.613$

37092 measured reflections  
 3499 independent reflections  
 2373 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.051$   
 $\theta_{\max} = 30.1^\circ$ ,  $\theta_{\min} = 1.9^\circ$   
 $h = -7 \rightarrow 7$   
 $k = -22 \rightarrow 22$   
 $l = -19 \rightarrow 19$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.135$   
 $S = 0.99$   
 3499 reflections  
 165 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-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0871P)^2 + 0.1014P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.41 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.68 \text{ e \AA}^{-3}$   
 Extinction correction: SHELXL97 (Sheldrick,  
 2008),  $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.0045 (16)

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.1456 (5)	0.66662 (15)	0.14761 (15)	0.0523 (5)
C2	0.0451 (4)	0.63544 (14)	0.20903 (15)	0.0524 (5)
H2	0.1392	0.6703	0.2500	0.063*
C3	0.0922 (4)	0.55182 (13)	0.20808 (14)	0.0453 (4)
C4	-0.0376 (4)	0.49886 (12)	0.14480 (14)	0.0427 (4)
C5	-0.2245 (4)	0.53249 (14)	0.08273 (16)	0.0524 (5)
H5	-0.3122	0.4982	0.0392	0.063*
C6	-0.2820 (5)	0.61578 (15)	0.08462 (17)	0.0559 (5)
H6	-0.4107	0.6374	0.0441	0.067*
N1	0.0066 (3)	0.41252 (11)	0.14056 (12)	0.0446 (4)

C7	0.2131 (4)	0.36565 (15)	0.16360 (18)	0.0541 (5)
H7	0.3662	0.3859	0.1905	0.065*
C8	-0.0743 (4)	0.28860 (13)	0.10549 (17)	0.0508 (5)
C9	-0.2132 (6)	0.21249 (15)	0.0729 (3)	0.0708 (8)
H9A	-0.3924	0.2244	0.0646	0.106*
H9B	-0.1874	0.1697	0.1198	0.106*
H9C	-0.1494	0.1944	0.0134	0.106*
C10	0.2286 (6)	0.50572 (18)	0.36008 (19)	0.0688 (7)
N2	-0.1818 (3)	0.36208 (11)	0.10201 (14)	0.0492 (4)
N3	0.1693 (4)	0.28792 (12)	0.14296 (18)	0.0582 (5)
O1	0.2873 (3)	0.51928 (11)	0.26983 (13)	0.0592 (4)
F1	0.0294 (6)	0.45837 (17)	0.36571 (16)	0.1342 (11)
F2	0.4236 (6)	0.46877 (15)	0.40292 (16)	0.1310 (10)
F3	0.1867 (5)	0.57307 (12)	0.40782 (12)	0.0998 (6)
Br1	-0.22139 (7)	0.780974 (16)	0.14867 (2)	0.07657 (17)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0645 (12)	0.0470 (11)	0.0453 (11)	0.0065 (9)	0.0010 (9)	0.0023 (8)
C2	0.0605 (12)	0.0480 (11)	0.0478 (11)	-0.0036 (9)	-0.0062 (9)	-0.0017 (8)
C3	0.0458 (10)	0.0467 (10)	0.0425 (10)	0.0017 (8)	-0.0066 (8)	0.0012 (8)
C4	0.0415 (9)	0.0416 (10)	0.0447 (10)	0.0022 (7)	-0.0012 (7)	-0.0007 (8)
C5	0.0536 (11)	0.0507 (11)	0.0514 (12)	0.0059 (9)	-0.0128 (9)	-0.0030 (9)
C6	0.0634 (13)	0.0511 (12)	0.0520 (12)	0.0118 (10)	-0.0099 (10)	0.0037 (9)
N1	0.0415 (8)	0.0438 (9)	0.0479 (9)	0.0036 (6)	-0.0040 (6)	-0.0028 (7)
C7	0.0411 (10)	0.0539 (12)	0.0664 (14)	0.0093 (9)	-0.0077 (9)	-0.0052 (10)
C8	0.0507 (11)	0.0471 (12)	0.0547 (12)	0.0030 (8)	0.0024 (9)	-0.0046 (9)
C9	0.0684 (16)	0.0487 (14)	0.095 (2)	-0.0032 (10)	0.0046 (14)	-0.0127 (12)
C10	0.0948 (19)	0.0510 (13)	0.0577 (15)	0.0047 (12)	-0.0265 (13)	0.0011 (10)
N2	0.0410 (8)	0.0462 (9)	0.0598 (10)	-0.0013 (7)	-0.0046 (7)	-0.0023 (8)
N3	0.0511 (10)	0.0518 (11)	0.0713 (14)	0.0114 (8)	-0.0016 (9)	-0.0039 (8)
O1	0.0567 (8)	0.0600 (9)	0.0589 (9)	0.0055 (7)	-0.0205 (7)	-0.0029 (7)
F1	0.181 (2)	0.139 (2)	0.0812 (14)	-0.080 (2)	-0.0096 (15)	0.0274 (13)
F2	0.177 (2)	0.1229 (19)	0.0866 (14)	0.0713 (17)	-0.0627 (15)	-0.0062 (13)
F3	0.1628 (19)	0.0694 (11)	0.0658 (10)	0.0264 (12)	-0.0112 (11)	-0.0113 (8)
Br1	0.1162 (3)	0.04531 (19)	0.0671 (2)	0.01567 (12)	-0.00788 (17)	0.00117 (10)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—C6	1.381 (3)	N1—N2	1.372 (2)
C1—C2	1.383 (3)	C7—N3	1.307 (3)
C1—Br1	1.890 (2)	C7—H7	0.9300
C2—C3	1.373 (3)	C8—N2	1.314 (3)
C2—H2	0.9300	C8—N3	1.358 (3)
C3—C4	1.388 (3)	C8—C9	1.490 (3)
C3—O1	1.410 (2)	C9—H9A	0.9600
C4—C5	1.390 (3)	C9—H9B	0.9600
C4—N1	1.416 (3)	C9—H9C	0.9600
C5—C6	1.379 (3)	C10—F1	1.299 (4)

C5—H5	0.9300	C10—F2	1.306 (3)
C6—H6	0.9300	C10—F3	1.302 (3)
N1—C7	1.347 (3)	C10—O1	1.335 (3)
C6—C1—C2	121.3 (2)	N3—C7—N1	110.9 (2)
C6—C1—Br1	118.93 (17)	N3—C7—H7	124.6
C2—C1—Br1	119.80 (17)	N1—C7—H7	124.6
C3—C2—C1	118.5 (2)	N2—C8—N3	114.54 (19)
C3—C2—H2	120.8	N2—C8—C9	122.1 (2)
C1—C2—H2	120.8	N3—C8—C9	123.3 (2)
C2—C3—C4	121.97 (18)	C8—C9—H9A	109.5
C2—C3—O1	119.08 (18)	C8—C9—H9B	109.5
C4—C3—O1	118.86 (18)	H9A—C9—H9B	109.5
C5—C4—C3	118.01 (19)	C8—C9—H9C	109.5
C5—C4—N1	118.07 (18)	H9A—C9—H9C	109.5
C3—C4—N1	123.91 (18)	H9B—C9—H9C	109.5
C4—C5—C6	121.1 (2)	F1—C10—F2	108.4 (3)
C4—C5—H5	119.4	F1—C10—F3	107.8 (3)
C6—C5—H5	119.4	F2—C10—F3	107.0 (2)
C1—C6—C5	119.0 (2)	F1—C10—O1	112.0 (2)
C1—C6—H6	120.5	F2—C10—O1	107.6 (3)
C5—C6—H6	120.5	F3—C10—O1	113.8 (2)
C7—N1—N2	108.42 (17)	C8—N2—N1	102.87 (16)
C7—N1—C4	132.43 (18)	C7—N3—C8	103.32 (18)
N2—N1—C4	119.06 (16)	C10—O1—C3	116.85 (18)
C6—C1—C2—C3	-1.7 (3)	C3—C4—N1—N2	157.37 (19)
Br1—C1—C2—C3	178.63 (17)	N2—N1—C7—N3	-0.5 (3)
C1—C2—C3—C4	2.8 (3)	C4—N1—C7—N3	-176.8 (2)
C1—C2—C3—O1	179.54 (19)	N3—C8—N2—N1	-0.3 (3)
C2—C3—C4—C5	-1.6 (3)	C9—C8—N2—N1	178.3 (2)
O1—C3—C4—C5	-178.29 (19)	C7—N1—N2—C8	0.4 (2)
C2—C3—C4—N1	179.3 (2)	C4—N1—N2—C8	177.29 (19)
O1—C3—C4—N1	2.6 (3)	N1—C7—N3—C8	0.3 (3)
C3—C4—C5—C6	-0.8 (3)	N2—C8—N3—C7	0.0 (3)
N1—C4—C5—C6	178.4 (2)	C9—C8—N3—C7	-178.5 (3)
C2—C1—C6—C5	-0.6 (4)	F1—C10—O1—C3	55.1 (3)
Br1—C1—C6—C5	179.06 (18)	F2—C10—O1—C3	174.1 (2)
C4—C5—C6—C1	1.9 (4)	F3—C10—O1—C3	-67.6 (3)
C5—C4—N1—C7	154.2 (2)	C2—C3—O1—C10	81.4 (3)
C3—C4—N1—C7	-26.6 (3)	C4—C3—O1—C10	-101.8 (2)
C5—C4—N1—N2	-21.8 (3)		

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>
C2—H2...N3 <sup>i</sup>	0.93	2.59	3.511 (3)	169

Symmetry code: (i)  $-x+1/2, y+1/2, -z+1/2$ .