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

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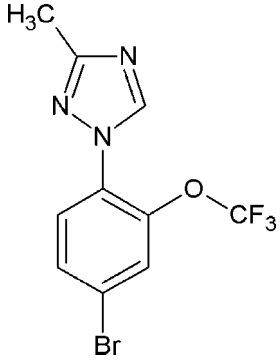
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.135; data-to-parameter ratio = 21.2.

In the title compound, $C_{10}H_7BrF_3N_3O$, the dihedral angle between the benzene and triazole rings is 23.17 (12)° and the C atom of the –CF_3 group deviates from its attached ring plane by 1.147 (3) Å. In the crystal, molecules are linked by C—H···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

$C_{10}H_7BrF_3N_3O$
M_r = 322.10
Monoclinic, $P2_1/n$
a = 5.2389 (3) Å
b = 16.1548 (8) Å
c = 14.0315 (7) Å
β = 92.673 (3)°
V = 1186.24 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.50 mm^{-1}
T = 293 K
0.33 × 0.21 × 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_{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^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···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).

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supporting information

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

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

S1. 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.

S2. Experimental

S2.1. 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 vaccum.

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

S2.2. Refinement

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

S3. Results and discussion

In the title compound, $C_{10}H_7BrF_3N_3O$, 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].

S4. 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).

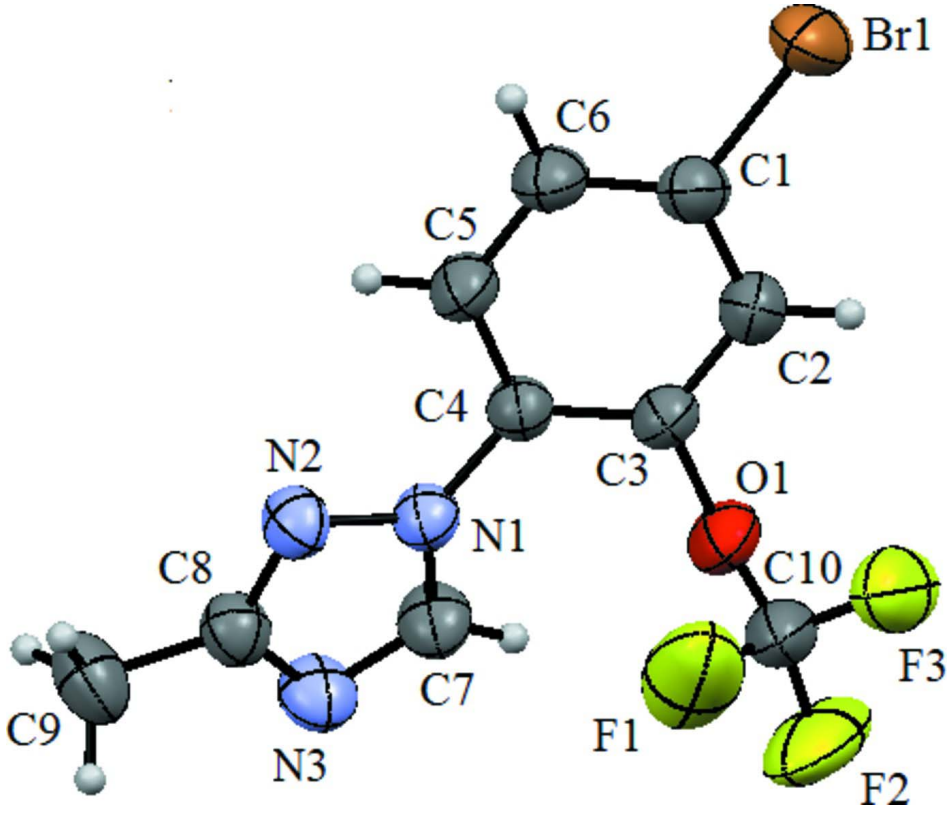

Figure 1

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

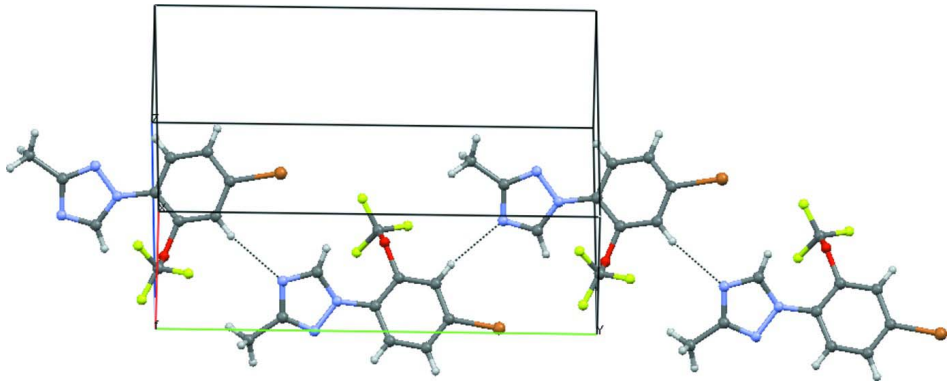

Figure 2

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

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Crystal data

$C_{10}H_7BrF_3N_3O$
M_r = 322.10
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 5.2389 (3) Å
b = 16.1548 (8) Å
c = 14.0315 (7) Å
β = 92.673 (3)°
V = 1186.24 (11) Å^3
Z = 4
F(000) = 632
Prism

D_x = 1.804 Mg m^{-3}
Melting point: 453 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 1.9–30.1°

μ = 3.50 mm^{-1}
T = 293 K
Prism, colourless
0.33 × 0.21 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω 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_{int} = 0.051
θ_{max} = 30.1°, θ_{min} = 1.9°
h = −7→7
k = −22→22
l = −19→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 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 (Å^2)

	x	y	z	U_{iso}*/U_{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 (Å²)

	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 (Å, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N3^{i}	0.93	2.59	3.511 (3)	169

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+1/2.