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2-Bromo-2-(5-bromo-1*H*-1,2,4-triazol-1-yl)-1-(2,4-difluorophenyl)ethanone

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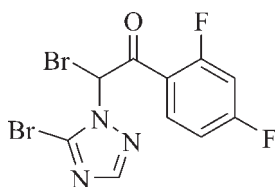
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.039; wR factor = 0.100; data-to-parameter ratio = 13.5.

In the title compound, $\text{C}_{10}\text{H}_5\text{Br}_2\text{F}_2\text{N}_3\text{O}$, the mean planes of the benzene and triazole rings form a dihedral angle of $84.86(2)^\circ$. In the crystal structure, weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules into extended chains propagating along the c axis.

Related literature

For general properties of 1,2,4-triazole derivatives, see: Garfunkle *et al.* (2008); Yu *et al.* (2009). For their antimicrobial activity, see: Luo *et al.* (2009); Zhang *et al.* (2010).



Experimental

Crystal data

$\text{C}_{10}\text{H}_5\text{Br}_2\text{F}_2\text{N}_3\text{O}$
 $M_r = 380.99$
 Monoclinic, $P2_1/c$
 $a = 9.273(2)$ Å
 $b = 9.375(2)$ Å

$c = 14.982(3)$ Å
 $\beta = 104.916(3)^\circ$
 $V = 1258.5(5)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 6.46$ mm⁻¹
 $T = 298$ K

0.26 × 0.25 × 0.25 mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.199$, $T_{\max} = 0.203$

6096 measured reflections
 2206 independent reflections
 1577 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.100$
 $S = 1.04$
 2206 reflections

163 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.61$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C7}-\text{H7}\cdots\text{O1}^i$	0.93	2.55	3.229 (5)	130

Symmetry code: (i) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5016).

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supporting information

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2-Bromo-2-(5-bromo-1*H*-1,2,4-triazol-1-yl)-1-(2,4-difluorophenyl)ethanone**Kun Wan, Bo Fang, Guang-Zhou Wang and Cheng-He Zhou****S1. Comment**

1,2,4-Triazole derivatives are important types of nitrogen-containing aromatic heterocyclic compounds with excellent safety profiles, favorable pharmacokinetic characteristics and a wide range of biological activities (Garfunkle *et al.*, 2008; Yu *et al.*, 2009). Our attention has been focused on the discovery of novel 1,2,4-triazole compounds as antimicrobial agents, and we found that some reported 1,2,4-triazole compounds display significant antimicrobial activities (Luo *et al.*, 2009; Zhang *et al.*, 2010). As part of our research, we report herein structure of the title compound (I).

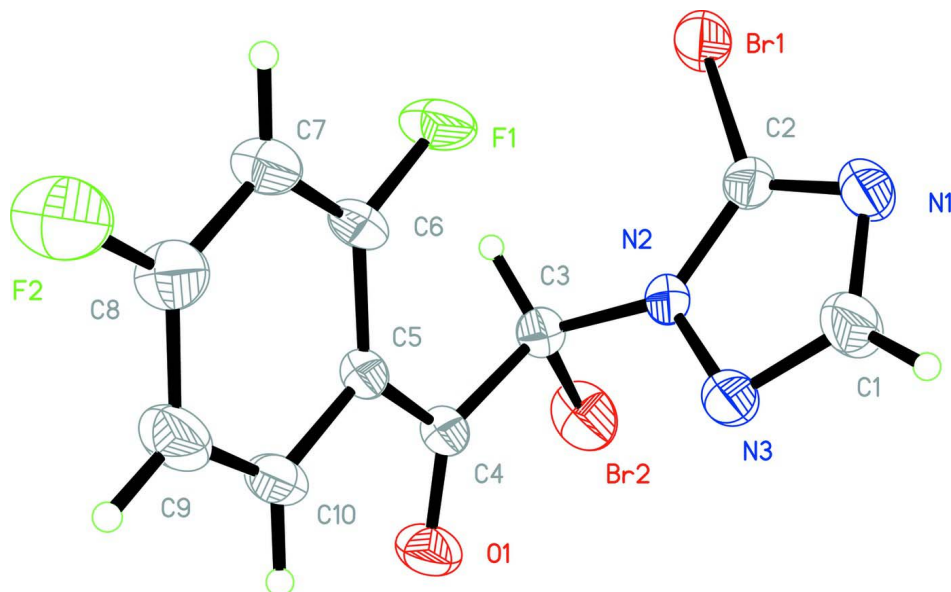
The molecular structure of the title compound is shown in Fig. 1. The mean planes of the benzene and triazole rings form a dihedral angle of 84.86 (2)°. In the crystal structure weak intermolecular C—H···O hydrogen bonds link molecules into extended chains along the *c* axis.

S2. Experimental

To a solution of 1-(2,4-difluorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone (1.0 g, 4.4 mmol), sodium acetate (1.4 g, 4.4 mmol) and acetic acid (4 ml) was added the mixture of Br₂ (0.45 ml) and acetic acid (2.5 ml) dropwise, and stirred at 338–348 K. The progress of the reaction was monitored by TLC. Upon completion, the reaction was extracted with chloroform (15 ml × 3). The filtrate was concentrated and then directly purified by chromatographic column (chloroform) to afford the title compound (I). A crystal suitable for X-ray analysis was grown from a solution of (I) in a mixture of petroleum and chloroform by slow evaporation at room temperature.

S3. Refinement

Hydrogen atoms were placed in calculated positions with C—H = 0.93 Å and 0.98 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$

**Figure 1**

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

2-Bromo-2-(5-bromo-1*H*-1,2,4-triazol-1-yl)-1-(2,4-difluorophenyl)ethanone

Crystal data

$C_{10}H_5Br_2F_2N_3O$

$M_r = 380.99$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 9.273\ (2)\ \text{\AA}$

$b = 9.375\ (2)\ \text{\AA}$

$c = 14.982\ (3)\ \text{\AA}$

$\beta = 104.916\ (3)^\circ$

$V = 1258.5\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 728$

$D_x = 2.011\ \text{Mg m}^{-3}$

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

Cell parameters from 1838 reflections

$\theta = 2.3\text{--}22.5^\circ$

$\mu = 6.46\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, colourless

$0.26 \times 0.25 \times 0.25\ \text{mm}$

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.199$, $T_{\max} = 0.203$

6096 measured reflections

2206 independent reflections

1577 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -10 \rightarrow 11$

$k = -10 \rightarrow 11$

$l = -15 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.100$

$S = 1.04$

2206 reflections

163 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

$$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.5764P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.61 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.49 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.88429 (6)	0.61973 (5)	0.21078 (5)	0.0741 (2)
Br2	0.65968 (6)	0.75021 (7)	0.40152 (3)	0.0777 (2)
C1	0.7324 (6)	1.0030 (5)	0.1617 (4)	0.0783 (17)
H1	0.7343	1.0949	0.1387	0.094*
C2	0.7921 (5)	0.7951 (5)	0.1945 (3)	0.0512 (11)
C3	0.5841 (4)	0.7348 (5)	0.2679 (3)	0.0426 (10)
H3	0.5927	0.6355	0.2496	0.051*
C4	0.4187 (5)	0.7796 (4)	0.2384 (3)	0.0426 (10)
C5	0.3310 (4)	0.7498 (4)	0.1425 (3)	0.0375 (9)
C6	0.3760 (5)	0.6720 (4)	0.0765 (3)	0.0424 (10)
C7	0.2865 (5)	0.6440 (5)	-0.0092 (3)	0.0502 (11)
H7	0.3205	0.5910	-0.0521	0.060*
C8	0.1455 (6)	0.6970 (5)	-0.0291 (3)	0.0576 (12)
C9	0.0924 (5)	0.7769 (6)	0.0313 (4)	0.0656 (14)
H9	-0.0044	0.8127	0.0152	0.079*
C10	0.1867 (5)	0.8030 (5)	0.1170 (3)	0.0553 (12)
H10	0.1525	0.8579	0.1591	0.066*
F1	0.5154 (3)	0.6180 (3)	0.09542 (17)	0.0645 (8)
F2	0.0553 (3)	0.6703 (4)	-0.11300 (19)	0.0911 (10)
N1	0.8368 (5)	0.9052 (4)	0.1564 (3)	0.0719 (13)
N2	0.6690 (4)	0.8238 (4)	0.2223 (2)	0.0450 (9)
N3	0.6279 (4)	0.9620 (4)	0.2011 (3)	0.0602 (10)
O1	0.3620 (4)	0.8357 (4)	0.2925 (2)	0.0640 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0500 (3)	0.0474 (3)	0.1299 (5)	0.0074 (2)	0.0324 (3)	-0.0047 (3)
Br2	0.0597 (4)	0.1177 (5)	0.0494 (3)	-0.0031 (3)	0.0025 (2)	-0.0060 (3)
C1	0.075 (4)	0.046 (3)	0.133 (5)	0.008 (3)	0.063 (4)	0.018 (3)
C2	0.039 (3)	0.041 (3)	0.075 (3)	-0.001 (2)	0.016 (2)	-0.010 (2)

C3	0.039 (2)	0.044 (2)	0.045 (2)	-0.0020 (19)	0.0114 (19)	-0.0029 (19)
C4	0.039 (2)	0.042 (2)	0.051 (2)	-0.0024 (19)	0.019 (2)	-0.0017 (19)
C5	0.034 (2)	0.038 (2)	0.044 (2)	-0.0016 (18)	0.0159 (17)	-0.0005 (18)
C6	0.034 (2)	0.047 (2)	0.049 (2)	0.012 (2)	0.016 (2)	0.004 (2)
C7	0.045 (3)	0.063 (3)	0.042 (2)	0.013 (2)	0.011 (2)	-0.003 (2)
C8	0.055 (3)	0.064 (3)	0.047 (3)	0.008 (3)	0.001 (2)	-0.003 (2)
C9	0.036 (3)	0.088 (4)	0.068 (3)	0.014 (3)	0.005 (2)	-0.013 (3)
C10	0.037 (3)	0.067 (3)	0.063 (3)	0.006 (2)	0.016 (2)	-0.020 (2)
F1	0.0477 (16)	0.085 (2)	0.0583 (16)	0.0246 (14)	0.0088 (12)	-0.0174 (14)
F2	0.067 (2)	0.136 (3)	0.0549 (17)	0.030 (2)	-0.0130 (15)	-0.0225 (18)
N1	0.065 (3)	0.048 (3)	0.120 (4)	0.000 (2)	0.056 (3)	0.006 (2)
N2	0.036 (2)	0.037 (2)	0.064 (2)	-0.0055 (16)	0.0180 (17)	-0.0069 (17)
N3	0.058 (3)	0.038 (2)	0.096 (3)	0.0038 (19)	0.039 (2)	0.006 (2)
O1	0.052 (2)	0.085 (2)	0.058 (2)	0.0053 (18)	0.0201 (16)	-0.0244 (18)

Geometric parameters (Å, °)

Br1—C2	1.840 (4)	C5—C6	1.378 (5)
Br2—C3	1.948 (4)	C5—C10	1.387 (6)
C1—N3	1.314 (6)	C6—F1	1.348 (4)
C1—N1	1.351 (6)	C6—C7	1.362 (6)
C1—H1	0.9300	C7—C8	1.358 (6)
C2—N1	1.297 (6)	C7—H7	0.9300
C2—N2	1.339 (5)	C8—F2	1.342 (5)
C3—N2	1.435 (5)	C8—C9	1.361 (7)
C3—C4	1.541 (6)	C9—C10	1.376 (6)
C3—H3	0.9800	C9—H9	0.9300
C4—O1	1.196 (5)	C10—H10	0.9300
C4—C5	1.483 (6)	N2—N3	1.365 (5)
N3—C1—N1	116.8 (4)	F1—C6—C5	119.9 (4)
N3—C1—H1	121.6	C7—C6—C5	123.7 (4)
N1—C1—H1	121.6	C8—C7—C6	117.1 (4)
N1—C2—N2	111.8 (4)	C8—C7—H7	121.5
N1—C2—Br1	125.4 (3)	C6—C7—H7	121.5
N2—C2—Br1	122.8 (3)	F2—C8—C7	118.1 (4)
N2—C3—C4	109.4 (3)	F2—C8—C9	118.7 (4)
N2—C3—Br2	110.5 (3)	C7—C8—C9	123.2 (4)
C4—C3—Br2	110.1 (3)	C8—C9—C10	117.8 (4)
N2—C3—H3	108.9	C8—C9—H9	121.1
C4—C3—H3	108.9	C10—C9—H9	121.1
Br2—C3—H3	108.9	C9—C10—C5	122.0 (4)
O1—C4—C5	120.8 (4)	C9—C10—H10	119.0
O1—C4—C3	120.2 (4)	C5—C10—H10	119.0
C5—C4—C3	119.0 (3)	C2—N1—C1	101.5 (4)
C6—C5—C10	116.2 (4)	C2—N2—N3	109.1 (4)
C6—C5—C4	127.1 (4)	C2—N2—C3	130.4 (4)
C10—C5—C4	116.7 (4)	N3—N2—C3	120.5 (3)

F1—C6—C7	116.4 (3)	C1—N3—N2	100.8 (4)
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Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C7—H7...O1 ⁱ	0.93	2.55	3.229 (5)	130

Symmetry code: (i) $x, -y+3/2, z-1/2$.