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## Structure Reports

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## 2-Azido-1-(4-fluorophenyl)ethanone

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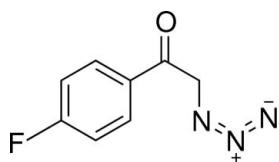
Received 22 March 2012; accepted 28 March 2012

Key indicators: single-crystal X-ray study;  $T = 273$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.033;  $wR$  factor = 0.090; data-to-parameter ratio = 12.5.

The crystal structure of the title compound,  $\text{C}_8\text{H}_6\text{FN}_3\text{O}$ , is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, which link the molecules into chains running parallel to the  $a$  axis.

## Related literature

The title compound is an intermediate obtained during an attempt to synthesize biologically active triazoles. For the biological activity of triazoles, see: Genin *et al.* (2000); Parmee *et al.* (2000); Koble *et al.* (1995); Moltzen *et al.* (1994). For standard bond lengths: Allen *et al.* (1987).



## Experimental

## Crystal data

 $\text{C}_8\text{H}_6\text{FN}_3\text{O}$  $M_r = 179.16$ Orthorhombic,  $Pbca$  $a = 10.7985$  (16) Å $b = 8.3971$  (12) Å $c = 17.485$  (3) Å $V = 1585.5$  (4) Å<sup>3</sup> $Z = 8$ Mo  $K\alpha$  radiation $\mu = 0.12$  mm<sup>-1</sup> $T = 273$  K $0.35 \times 0.28 \times 0.20$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Bruker, 2000) $T_{\min} = 0.959$ ,  $T_{\max} = 0.976$ 

8606 measured reflections

1476 independent reflections

1239 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.034$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.033$  $wR(F^2) = 0.090$  $S = 1.06$ 

1476 reflections

118 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.20$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                           | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C}1-\text{H}1\text{B}\cdots\text{O}1^i$ | 0.93         | 2.45               | 3.3722 (17) | 174                  |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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 PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2730).

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## supporting information

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## 2-Azido-1-(4-fluorophenyl)ethanone

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### S1. Comment

The synthesis of 1,2,3-triazoles *via* click chemistry approach has gained much attention by synthetic chemists due to their immensely known medicinal importance (Genin *et al.*, 2000, Parmee *et al.*, 2000, Koble *et al.*, 1995, Moltzen *et al.*, 1994). In our quest for the synthesis of therapeutically active triazoles starting from commercially available acetophenone derivatives, a number of azides including the title compound, whose crystal structure is reported herein, have been prepared as an intermediate.

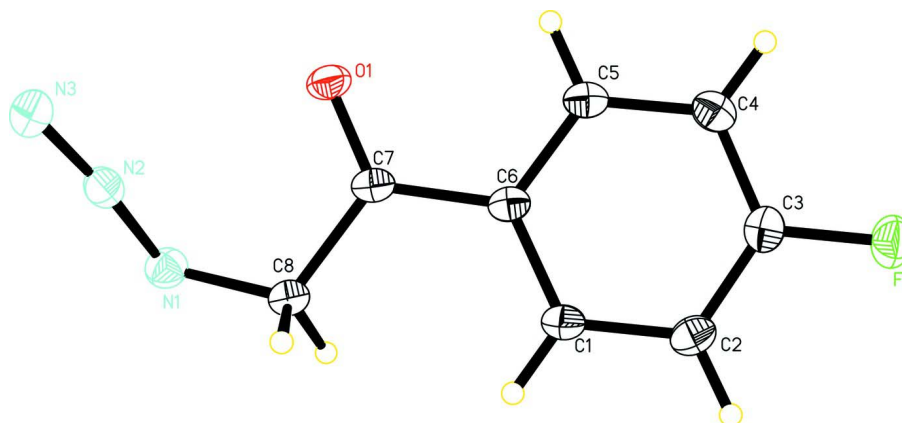
In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The crystal structure (Fig. 2) is stabilized by intermolecular hydrogen bonds (Table 1) linking the molecules to form chains parallel to the *a* axis.

### S2. Experimental

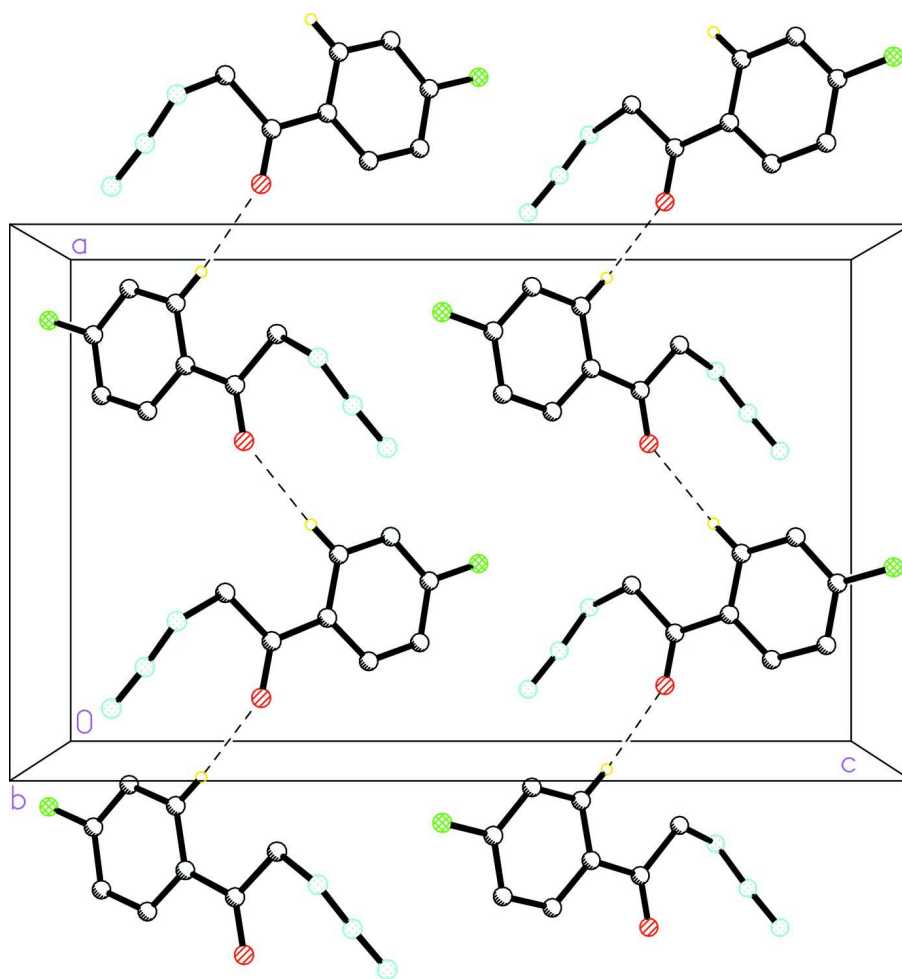
1-(4-Fluorophenyl)ethanone (7.239 mmol, 1.0 equiv.) was dissolved in acetonitrile (18 ml) in a round bottom flask. To the stirred mixture, *p*-toluenesulphonic acid (10.858 mmol, 1.5 equiv.) and *N*-bromosuccinimide (10.134 mmol, 1.4 equiv.) were added, and the solution was heated to reflux for 1 to 1.5 h until completion of the reaction as monitored by TLC analysis. The reaction mixture was cooled to room temperature and sodium azide (21.717 mmol, 3.0 equiv.) was added. After additional stirring for 2 to 3 h, ice cooled water was added to quench the reaction. The reaction mixture was extracted with diethyl ether (2 × 25 ml) and the combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated in vacuum to get the crude product. The crude product was purified by flash silica gel chromatography (EtOAc/hexane, 1:9–3:7 *v/v*) to afford the title compound in 65% yield. Recrystallization by slow evaporation of an ethanol solution afforded crystals suitable for single-crystal X-ray studies. All chemicals were purchased from Sigma-Aldrich.

### S3. Refinement

H atoms were positioned geometrically with C—H = 0.93–0.97 Å, and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ .

**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.

**Figure 2**

Crystal packing of the title compound viewed along the *b* axis. Hydrogen atoms not involved in hydrogen bonds (dashed lines) are omitted.

## 2-Azido-1-(4-fluorophenyl)ethanone

## Crystal data

C<sub>8</sub>H<sub>6</sub>FN<sub>3</sub>O $M_r = 179.16$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 10.7985$  (16) Å $b = 8.3971$  (12) Å $c = 17.485$  (3) Å $V = 1585.5$  (4) Å<sup>3</sup> $Z = 8$  $F(000) = 736$  $D_x = 1.501$  Mg m<sup>-3</sup>Mo *K*α radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2831 reflections

 $\theta = 2.3$ – $27.8^\circ$  $\mu = 0.12$  mm<sup>-1</sup> $T = 273$  K

Block, colourless

 $0.35 \times 0.28 \times 0.20$  mm

## Data collection

Bruker SMART APEX CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  scanAbsorption correction: multi-scan  
(*SADABS*; Bruker, 2000) $T_{\min} = 0.959$ ,  $T_{\max} = 0.976$ 

8606 measured reflections

1476 independent reflections

1239 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.034$  $\theta_{\text{max}} = 25.5^\circ$ ,  $\theta_{\text{min}} = 2.3^\circ$  $h = -10 \rightarrow 13$  $k = -10 \rightarrow 10$  $l = -21 \rightarrow 21$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.033$  $wR(F^2) = 0.090$  $S = 1.06$ 

1476 reflections

118 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.2956P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} < 0.001$  $\Delta\rho_{\text{max}} = 0.20$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

## 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 (Å<sup>2</sup>)

|    | <i>x</i>     | <i>y</i>      | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|--------------|---------------|-------------|----------------------------------|
| O1 | 0.61761 (8)  | 0.03188 (12)  | 0.73973 (5) | 0.0358 (3)                       |
| N1 | 0.77319 (12) | -0.13811 (15) | 0.83252 (7) | 0.0361 (3)                       |
| N2 | 0.68380 (11) | -0.09577 (14) | 0.87237 (7) | 0.0336 (3)                       |
| N3 | 0.60363 (13) | -0.07646 (17) | 0.91283 (7) | 0.0451 (4)                       |
| C1 | 0.89336 (12) | 0.19093 (16)  | 0.64990 (8) | 0.0305 (3)                       |

|     |              |               |             |            |
|-----|--------------|---------------|-------------|------------|
| H1B | 0.9536       | 0.1521        | 0.6830      | 0.037*     |
| C2  | 0.92779 (14) | 0.28254 (17)  | 0.58742 (8) | 0.0348 (4) |
| H2A | 1.0106       | 0.3060        | 0.5782      | 0.042*     |
| C3  | 0.83684 (14) | 0.33759 (17)  | 0.53966 (8) | 0.0342 (3) |
| C4  | 0.71318 (14) | 0.30861 (18)  | 0.55138 (8) | 0.0366 (4) |
| H4A | 0.6536       | 0.3492        | 0.5182      | 0.044*     |
| C5  | 0.67981 (13) | 0.21777 (17)  | 0.61363 (8) | 0.0333 (3) |
| H5A | 0.5965       | 0.1968        | 0.6227      | 0.040*     |
| C6  | 0.76889 (12) | 0.15677 (16)  | 0.66330 (7) | 0.0273 (3) |
| C7  | 0.72692 (12) | 0.05618 (16)  | 0.72799 (7) | 0.0277 (3) |
| C8  | 0.82354 (13) | -0.01705 (17) | 0.78080 (8) | 0.0313 (3) |
| H8A | 0.8882       | -0.0650       | 0.7499      | 0.038*     |
| H8B | 0.8611       | 0.0669        | 0.8110      | 0.038*     |
| F1  | 0.87045 (9)  | 0.42468 (11)  | 0.47751 (5) | 0.0478 (3) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|    | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|----|------------|------------|------------|-------------|-------------|-------------|
| O1 | 0.0225 (6) | 0.0481 (6) | 0.0370 (5) | -0.0033 (4) | -0.0003 (4) | -0.0021 (5) |
| N1 | 0.0337 (7) | 0.0341 (7) | 0.0405 (7) | 0.0037 (5)  | 0.0029 (6)  | 0.0004 (5)  |
| N2 | 0.0346 (7) | 0.0329 (7) | 0.0333 (6) | -0.0019 (5) | -0.0049 (6) | 0.0031 (5)  |
| N3 | 0.0413 (8) | 0.0548 (9) | 0.0392 (7) | 0.0024 (7)  | 0.0065 (6)  | 0.0069 (6)  |
| C1 | 0.0248 (7) | 0.0338 (8) | 0.0328 (7) | 0.0029 (6)  | -0.0019 (6) | -0.0054 (6) |
| C2 | 0.0293 (8) | 0.0374 (8) | 0.0376 (7) | -0.0015 (6) | 0.0049 (6)  | -0.0064 (6) |
| C3 | 0.0414 (9) | 0.0314 (7) | 0.0299 (7) | -0.0013 (6) | 0.0036 (6)  | -0.0031 (6) |
| C4 | 0.0355 (9) | 0.0378 (8) | 0.0366 (8) | 0.0021 (6)  | -0.0068 (6) | -0.0006 (6) |
| C5 | 0.0260 (7) | 0.0343 (8) | 0.0394 (8) | 0.0003 (6)  | -0.0025 (6) | -0.0041 (6) |
| C6 | 0.0241 (7) | 0.0274 (7) | 0.0304 (7) | 0.0011 (6)  | 0.0005 (5)  | -0.0075 (5) |
| C7 | 0.0232 (7) | 0.0287 (7) | 0.0313 (7) | 0.0004 (5)  | -0.0009 (5) | -0.0098 (5) |
| C8 | 0.0254 (7) | 0.0336 (7) | 0.0348 (7) | -0.0004 (6) | 0.0003 (6)  | -0.0012 (6) |
| F1 | 0.0544 (6) | 0.0501 (6) | 0.0389 (5) | -0.0045 (4) | 0.0040 (4)  | 0.0089 (4)  |

*Geometric parameters (Å, °)*

|          |             |           |             |
|----------|-------------|-----------|-------------|
| O1—C7    | 1.2154 (16) | C3—C4     | 1.373 (2)   |
| N1—N2    | 1.2425 (17) | C4—C5     | 1.377 (2)   |
| N1—C8    | 1.4652 (19) | C4—H4A    | 0.9300      |
| N2—N3    | 1.1296 (17) | C5—C6     | 1.3936 (19) |
| C1—C2    | 1.387 (2)   | C5—H5A    | 0.9300      |
| C1—C6    | 1.3942 (19) | C6—C7     | 1.4826 (19) |
| C1—H1B   | 0.9300      | C7—C8     | 1.5229 (19) |
| C2—C3    | 1.370 (2)   | C8—H8A    | 0.9700      |
| C2—H2A   | 0.9300      | C8—H8B    | 0.9700      |
| C3—F1    | 1.3591 (16) |           |             |
| N2—N1—C8 | 115.85 (12) | C4—C5—H5A | 119.5       |
| N3—N2—N1 | 170.98 (14) | C6—C5—H5A | 119.5       |
| C2—C1—C6 | 120.33 (13) | C5—C6—C1  | 119.02 (13) |

|             |              |             |              |
|-------------|--------------|-------------|--------------|
| C2—C1—H1B   | 119.8        | C5—C6—C7    | 118.29 (12)  |
| C6—C1—H1B   | 119.8        | C1—C6—C7    | 122.69 (12)  |
| C3—C2—C1    | 118.38 (13)  | O1—C7—C6    | 121.43 (12)  |
| C3—C2—H2A   | 120.8        | O1—C7—C8    | 119.67 (12)  |
| C1—C2—H2A   | 120.8        | C6—C7—C8    | 118.90 (11)  |
| F1—C3—C2    | 118.53 (13)  | N1—C8—C7    | 113.59 (12)  |
| F1—C3—C4    | 118.33 (13)  | N1—C8—H8A   | 108.8        |
| C2—C3—C4    | 123.14 (14)  | C7—C8—H8A   | 108.8        |
| C3—C4—C5    | 118.09 (14)  | N1—C8—H8B   | 108.8        |
| C3—C4—H4A   | 121.0        | C7—C8—H8B   | 108.8        |
| C5—C4—H4A   | 121.0        | H8A—C8—H8B  | 107.7        |
| C4—C5—C6    | 121.04 (13)  |             |              |
| C6—C1—C2—C3 | -0.2 (2)     | C2—C1—C6—C7 | 178.42 (12)  |
| C1—C2—C3—F1 | -178.68 (12) | C5—C6—C7—O1 | -2.87 (19)   |
| C1—C2—C3—C4 | 1.1 (2)      | C1—C6—C7—O1 | 177.86 (12)  |
| F1—C3—C4—C5 | 178.78 (12)  | C5—C6—C7—C8 | 177.46 (12)  |
| C2—C3—C4—C5 | -1.0 (2)     | C1—C6—C7—C8 | -1.81 (18)   |
| C3—C4—C5—C6 | -0.1 (2)     | N2—N1—C8—C7 | -54.22 (16)  |
| C4—C5—C6—C1 | 1.0 (2)      | O1—C7—C8—N1 | 11.84 (17)   |
| C4—C5—C6—C7 | -178.34 (12) | C6—C7—C8—N1 | -168.49 (11) |
| C2—C1—C6—C5 | -0.84 (19)   |             |              |

*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> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| C1—H1B...O1 <sup>i</sup> | 0.93        | 2.45          | 3.3722 (17)           | 174                     |

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