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2-Azido-1-(4-methylphenyl)ethanone

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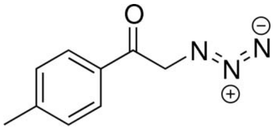
Key indicators: single-crystal X-ray study; $T = 273$ K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.090; data-to-parameter ratio = 13.3.

In the molecule of the title compound, $C_9H_9N_3O$, the angle formed by the least-squares line through the azide group with the normal to the plane of the benzene plane ring is 46.62 (16)°. The crystal structure features C—H⋯O hydrogen bonds, which link the molecules into zigzag chains running parallel to [010].

Related literature

For a related structure, see: Yousuf *et al.* (2012). For the biological activity of triazoles, see: Genin *et al.* (2000); Parmee *et al.* (2000); Koble *et al.* (1995); Moltzen *et al.* (1994).



Experimental

Crystal data

$C_9H_9N_3O$
$M_r = 175.19$
Monoclinic, $P2_1/c$
$a = 7.696$ (3) Å
$b = 9.025$ (3) Å
$c = 14.248$ (4) Å
$\beta = 118.726$ (15)°
$V = 867.8$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 273$ K
0.30 × 0.21 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.973$, $T_{max} = 0.985$
4915 measured reflections
1595 independent reflections
1464 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.090$
$S = 1.07$
1595 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C8—H8A⋯O1^{i}	0.97	2.40	3.2404 (19)	145

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

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: RZ2744).

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2-Azido-1-(4-methylphenyl)ethanone

Muhammad Arshad, Sammer Yousuf, Hafiza Madiha Butt, Sumayya Saeed and Fatima Z. Basha

S1. Comment

Triazoles are considered an important class of compounds due to their therapeutic potential (Genin *et al.*, 2000; Parmee *et al.*, 2000; Koble *et al.*, 1995; Moltzen *et al.*, 1994). The title compound was obtained as an intermediate during our attempt to synthesize biologically active triazoles.

The structure of the title compound (Fig. 1) is similar to that of our recently published compound 2-azido-1-(4-fluorophenyl)ethanone (Yousuf *et al.*, 2012) with the difference that the fluorophenyl ring is replaced by a toluene ring. The bond lengths and angles are similar to those found in the previously reported compound. The azide group is not linear (N3–N2–N1 = 170.84 (11)°) and the least-square line through it forms with the normal to the plane of benzene ring an angle of 46.62 (16)°. The crystal structure is stabilized by C—H···O intermolecular hydrogen bonds (Table 1) forming zig-zag chains parallel to the *b* axis (Fig. 2).

S2. Experimental

1-*p*-Tolylethanone (8.32 mmol, 1.0 equiv.) was dissolved in acetonitrile (20 ml) in a round bottom flask. To the stirred mixture, *p*-toluene sulphonic acid (12.5 mmol, 1.5 equiv.) and *N*-bromosuccinimide (11.6 mmol, 1.4 equiv.) were added, and the mixtyre refluxed for 1 to 1.5 h until TLC analysis showed no starting material present. The reaction mixture was cooled to room temperature, sodium azide (24.9 mmol, 3.0 equiv.) was added and the mixture further stirred for 2 to 3 hrs followed by the addition of ice cooled water to quench the reaction. The reaction mixture was extracted with ethylacetate (25 ml × 2) and the combined organic layer were dried over anhydrous Na_2SO_4, 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 crystalline title compound in 70% yield. The crystals were found to be 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 $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$ for methyl H atoms. A rotating group model was applied to the methyl group.

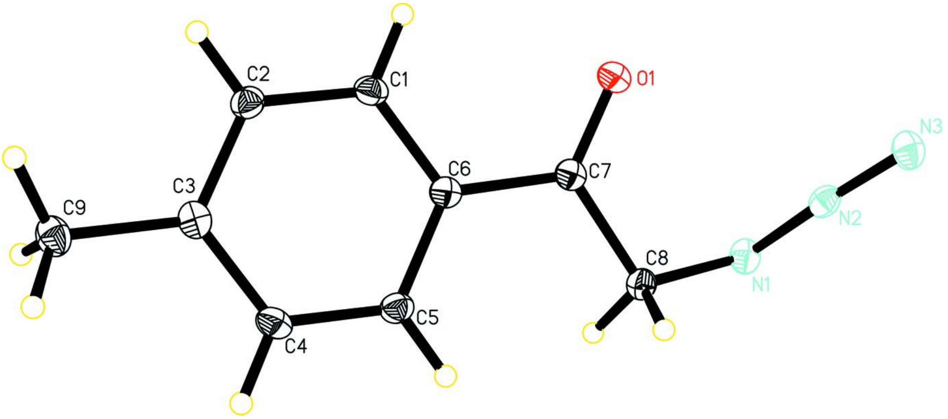

Figure 1

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

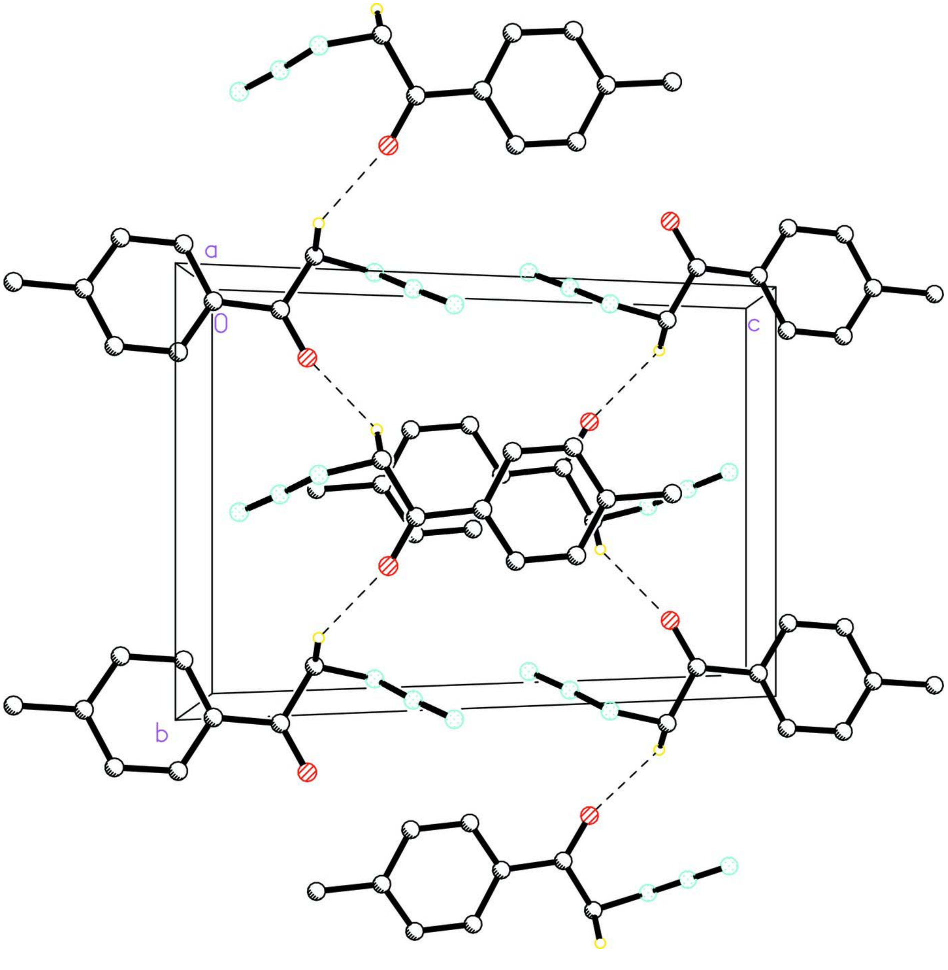

Figure 2

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

2-Azido-1-(4-methylphenyl)ethanone

Crystal data

$C_9H_9N_3O$
M_r = 175.19
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.696 (3) Å
b = 9.025 (3) Å
c = 14.248 (4) Å
β = 118.726 (15)°
V = 867.8 (5) Å^3
Z = 4
F(000) = 368
D_x = 1.341 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3299 reflections
θ = 2.8–25.5°
μ = 0.09 mm^{-1}
T = 273 K
Block, colourless
0.30 × 0.21 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.973, T_{max} = 0.985
4915 measured reflections
1595 independent reflections
1464 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.5°, θ_{min} = 2.8°
h = −9→9
k = −10→10
l = −16→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.090
S = 1.07
1595 reflections
120 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
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.1892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.032 (4)

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^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.37626 (11)	0.32286 (8)	0.67821 (6)	0.0259 (2)
N1	0.30617 (13)	0.53889 (11)	0.79105 (8)	0.0250 (3)
N2	0.46826 (13)	0.49335 (10)	0.86119 (7)	0.0220 (2)

N3	0.60533 (15)	0.45093 (11)	0.93395 (8)	0.0285 (3)
C1	0.27074 (15)	0.33313 (12)	0.45933 (9)	0.0225 (3)
H1B	0.3054	0.2413	0.4929	0.027*
C2	0.22351 (15)	0.34548 (12)	0.35311 (9)	0.0234 (3)
H2A	0.2257	0.2615	0.3159	0.028*
C3	0.17253 (15)	0.48218 (12)	0.30073 (9)	0.0217 (3)
C4	0.17304 (15)	0.60668 (12)	0.35936 (9)	0.0238 (3)
H4B	0.1425	0.6991	0.3264	0.029*
C5	0.21797 (15)	0.59490 (12)	0.46497 (9)	0.0226 (3)
H5A	0.2157	0.6789	0.5022	0.027*
C6	0.26701 (14)	0.45736 (11)	0.51680 (9)	0.0198 (3)
C7	0.31704 (14)	0.43963 (11)	0.63045 (9)	0.0201 (3)
C8	0.29227 (16)	0.57461 (12)	0.68730 (9)	0.0224 (3)
H8A	0.3935	0.6468	0.6981	0.027*
H8B	0.1643	0.6195	0.6418	0.027*
C9	0.11424 (17)	0.49495 (13)	0.18444 (9)	0.0275 (3)
H9A	0.1646	0.5861	0.1723	0.041*
H9B	−0.0276	0.4939	0.1426	0.041*
H9C	0.1684	0.4130	0.1640	0.041*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0310 (4)	0.0219 (4)	0.0239 (4)	0.0038 (3)	0.0125 (4)	0.0038 (3)
N1	0.0204 (5)	0.0313 (5)	0.0230 (5)	0.0014 (4)	0.0103 (4)	−0.0017 (4)
N2	0.0254 (5)	0.0220 (5)	0.0229 (5)	−0.0032 (4)	0.0151 (5)	−0.0038 (4)
N3	0.0296 (5)	0.0333 (6)	0.0225 (5)	0.0008 (4)	0.0125 (5)	0.0017 (4)
C1	0.0214 (5)	0.0199 (5)	0.0245 (6)	0.0039 (4)	0.0097 (4)	0.0022 (4)
C2	0.0226 (5)	0.0233 (6)	0.0239 (6)	0.0029 (4)	0.0109 (4)	−0.0020 (4)
C3	0.0155 (5)	0.0282 (6)	0.0222 (6)	−0.0008 (4)	0.0096 (4)	0.0014 (4)
C4	0.0234 (5)	0.0198 (5)	0.0261 (6)	−0.0005 (4)	0.0104 (5)	0.0045 (4)
C5	0.0227 (5)	0.0187 (5)	0.0246 (6)	−0.0018 (4)	0.0100 (5)	−0.0016 (4)
C6	0.0152 (5)	0.0204 (5)	0.0222 (6)	−0.0010 (4)	0.0076 (4)	−0.0001 (4)
C7	0.0152 (5)	0.0212 (5)	0.0219 (6)	−0.0014 (4)	0.0074 (4)	0.0001 (4)
C8	0.0215 (5)	0.0224 (5)	0.0209 (6)	−0.0002 (4)	0.0083 (4)	−0.0014 (4)
C9	0.0270 (6)	0.0333 (6)	0.0254 (6)	0.0025 (5)	0.0151 (5)	0.0041 (5)

Geometric parameters (Å, º)

O1—C7	1.2180 (13)	C4—C5	1.3765 (17)
N1—N2	1.2350 (14)	C4—H4B	0.9300
N1—C8	1.4659 (15)	C5—C6	1.4003 (16)
N2—N3	1.1327 (14)	C5—H5A	0.9300
C1—C2	1.3806 (16)	C6—C7	1.4821 (16)
C1—C6	1.3968 (16)	C7—C8	1.5247 (15)
C1—H1B	0.9300	C8—H8A	0.9700
C2—C3	1.3970 (16)	C8—H8B	0.9700
C2—H2A	0.9300	C9—H9A	0.9600

C3—C4	1.3991 (16)	C9—H9B	0.9600
C3—C9	1.4984 (17)	C9—H9C	0.9600

N2—N1—C8	116.42 (9)	C1—C6—C7	119.08 (10)
N3—N2—N1	170.84 (11)	C5—C6—C7	122.31 (10)
C2—C1—C6	120.60 (10)	O1—C7—C6	122.33 (10)
C2—C1—H1B	119.7	O1—C7—C8	120.22 (10)
C6—C1—H1B	119.7	C6—C7—C8	117.45 (9)
C1—C2—C3	121.02 (10)	N1—C8—C7	113.13 (9)
C1—C2—H2A	119.5	N1—C8—H8A	109.0
C3—C2—H2A	119.5	C7—C8—H8A	109.0
C2—C3—C4	118.09 (10)	N1—C8—H8B	109.0
C2—C3—C9	121.11 (10)	C7—C8—H8B	109.0
C4—C3—C9	120.79 (10)	H8A—C8—H8B	107.8
C5—C4—C3	121.19 (10)	C3—C9—H9A	109.5
C5—C4—H4B	119.4	C3—C9—H9B	109.5
C3—C4—H4B	119.4	H9A—C9—H9B	109.5
C4—C5—C6	120.49 (10)	C3—C9—H9C	109.5
C4—C5—H5A	119.8	H9A—C9—H9C	109.5
C6—C5—H5A	119.8	H9B—C9—H9C	109.5
C1—C6—C5	118.60 (11)		

C6—C1—C2—C3	0.53 (16)	C4—C5—C6—C7	179.79 (9)
C1—C2—C3—C4	0.77 (16)	C1—C6—C7—O1	5.82 (15)
C1—C2—C3—C9	−177.81 (9)	C5—C6—C7—O1	−173.48 (10)
C2—C3—C4—C5	−1.46 (16)	C1—C6—C7—C8	−174.32 (9)
C9—C3—C4—C5	177.13 (10)	C5—C6—C7—C8	6.38 (14)
C3—C4—C5—C6	0.84 (16)	N2—N1—C8—C7	65.13 (12)
C2—C1—C6—C5	−1.16 (15)	O1—C7—C8—N1	−11.81 (14)
C2—C1—C6—C7	179.51 (9)	C6—C7—C8—N1	168.33 (8)
C4—C5—C6—C1	0.48 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1$^{\rm i}$	0.97	2.40	3.2404 (19)	145

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