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

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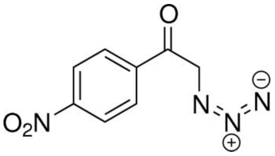
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.074; data-to-parameter ratio = 12.1.

In the title compound, $C_8H_6N_4O_3$, the ketone [C—C(=O)—C] and nitro groups are tilted with respect to the benzene ring by 18.92 (6) and 24.11 (15)°, respectively. In the crystal, molecules are linked into interwoven chains running parallel to the [100] direction by C—H⋯N hydrogen bonds and weak π–π stacking interactions, with centroid–centroid separations of 3.897 (3) Å.

Related literature

For the crystal structure of the related compound 2-azido-1-(4-fluorophenyl)ethanone, see: Yousuf *et al.* (2012). For the biological activities of triazoles, see: Genin *et al.* (2000); Parmee *et al.* (2000); Koble *et al.* (1995); Moltzen *et al.* (1994).



Experimental

Crystal data

$C_8H_6N_4O_3$
M_r = 206.17
Orthorhombic, $Pca2_1$
a = 7.6307 (5) Å
b = 9.5168 (6) Å
c = 12.4097 (8) Å
V = 901.19 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 273 K
0.50 × 0.23 × 0.11 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000) $T_{\min}$ = 0.942, $T_{\max}$ = 0.987
4914 measured reflections
1649 independent reflections
1461 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.074
S = 1.06
1649 reflections
136 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.11 e Å^{-3}
$\Delta\rho_{\min}$ = −0.11 e Å^{-3}
Absolute structure: Flack (1983), 767 Friedel pairs
Flack parameter: 0.2 (14)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C8—H8B⋯N2$^{\rm i}$	0.97	2.48	3.422 (3)	165

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

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: RZ2752).

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supplementary materials

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

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Comment

The title compound was obtained as intermediate during an attempt to synthesize new triazoles, an important class of organic compounds with a wide range of biological activities (Genin *et al.*, 2000; Parmee *et al.*, 2000; Koble *et al.*, 1995; Moltzen *et al.*, 1994).

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 nitrobenzene ring. The benzene ring forms dihedral angles of 18.92 (6) and 24.11 (15)° with the planes through the ketone (O3/C3/C7/C8) and nitro (N1/O1/O2) groups, respectively. The azide group is not linear (N3–N2–N1 = 171.7 (2)°). The bond lengths and angle are similar to those found in the previously reported compound (Yousuf *et al.*, 2012). The crystal structure is stabilized by intermolecular C—H···N (Table 1) hydrogen bonds and π–π stacking interactions (centroid-to-centroid separations of 3.897 (3) Å) forming interwoven chains parallel to the *a* axis (Fig. 2).

Experimental

1-(4-Nitrophenyl)ethanone (6.05 mmoL, 1.0 eq.) was dissolved in acetonitrile (18 ml) in a round bottom flask. To the stirred mixture, *p*-toluene sulphonic acid (9.08 mmoL, 1.5 eq.) and *N*-bromosuccinimide (8.48 mmol, 1.4 eq.) were added, and then heated to reflux for 1 to 1.5 h until TLC analysis showed no starting material present in the mixture. The reaction mixture was cooled to room temperature, sodium azide (18.16 mmoL, 3.0 eq.) was added and further stirred for 2 to 3 hrs followed by the addition of the ice cooled water 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_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 title compound in 70% yield. Recrystallization from ethanol afforded crystals suitable for single-crystal X-ray studies. All chemicals were purchased from Sigma-Aldrich.

Refinement

Methylene and aromatic H atoms were positioned geometrically with C—H = 0.93–0.97 Å, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. 765 Friedel pairs were not merged.

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); 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* (Sheldrick, 2008), *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

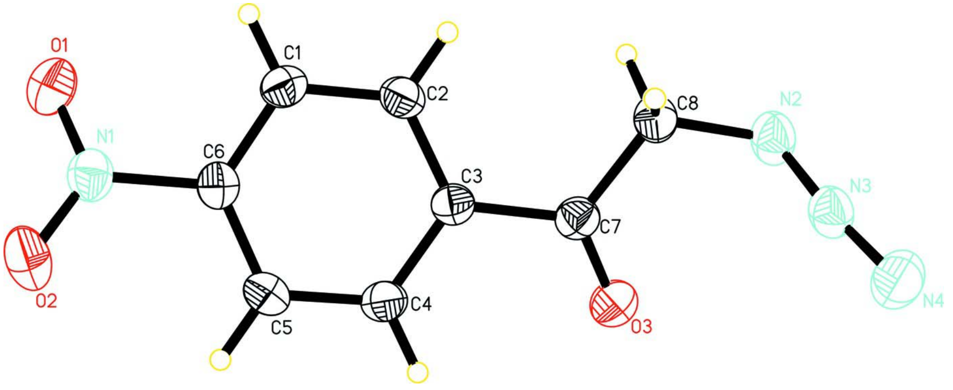

Figure 1

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

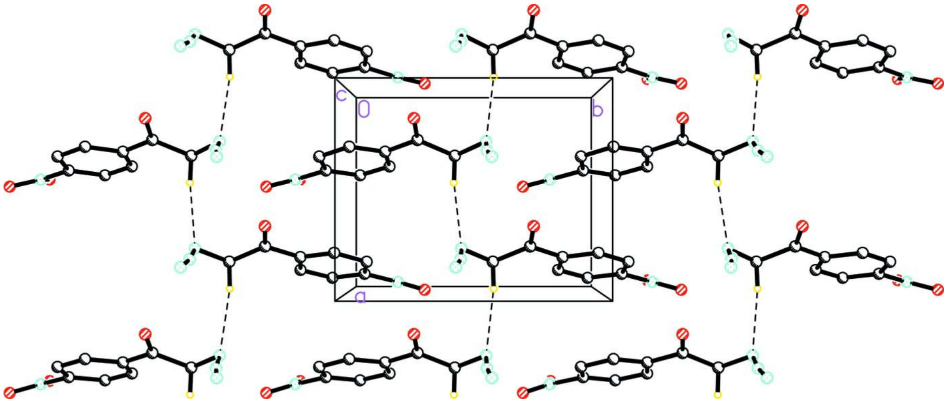

Figure 2

The crystal packing of the title compound. Hydrogen atoms not involved in hydrogen bonding (dashed lines) are omitted for clarity.

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

Crystal data

$C_8H_6N_4O_3$
$M_r = 206.17$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c 2ac
$a = 7.6307$ (5) Å
$b = 9.5168$ (6) Å
$c = 12.4097$ (8) Å
$V = 901.19$ (10) Å^3
$Z = 4$

$F(000) = 424$
$D_x = 1.520$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1557 reflections
$\theta = 3.3$–$24.2°$
$\mu = 0.12$ mm^{-1}
$T = 273$ K
Block, colourles
0.50 × 0.23 × 0.11 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)
$T_{\min} = 0.942$, $T_{\max} = 0.987$
4914 measured reflections
1649 independent reflections
1461 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$
$\theta_{\text{max}} = 25.5°$, $\theta_{\text{min}} = 2.1°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.074$
$S = 1.06$
1649 reflections
136 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 0.0663P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} < 0.001$
$\Delta\rho_{\text{max}} = 0.11$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.11$ e Å^{-3}
Absolute structure: Flack (1983), 767 Friedel pairs
Flack parameter: 0.2 (14)

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	0.9428 (3)	1.19359 (17)	0.40028 (13)	0.0815 (5)
O2	0.9798 (2)	1.30833 (17)	0.54808 (15)	0.0744 (5)
O3	0.6676 (2)	0.72287 (15)	0.81581 (12)	0.0673 (4)
N1	0.9448 (2)	1.20158 (19)	0.49879 (14)	0.0553 (5)
N2	0.7720 (3)	0.45389 (18)	0.76819 (14)	0.0603 (5)
N3	0.8183 (2)	0.45144 (17)	0.86298 (15)	0.0553 (4)
N4	0.8503 (3)	0.4344 (2)	0.95022 (17)	0.0740 (6)
C1	0.9238 (2)	0.9459 (2)	0.51250 (14)	0.0478 (5)
H1A	0.9657	0.9388	0.4423	0.057*
C2	0.8824 (3)	0.8273 (2)	0.57102 (15)	0.0472 (5)
H2B	0.8991	0.7389	0.5407	0.057*
C3	0.8160 (2)	0.83847 (19)	0.67466 (14)	0.0418 (4)
C4	0.7943 (2)	0.9715 (2)	0.71966 (14)	0.0474 (5)
H4A	0.7489	0.9796	0.7889	0.057*
C5	0.8386 (2)	1.0906 (2)	0.66373 (16)	0.0482 (4)
H5A	0.8264	1.1791	0.6946	0.058*
C6	0.9017 (2)	1.0752 (2)	0.56038 (14)	0.0438 (4)
C7	0.7632 (3)	0.7125 (2)	0.73869 (14)	0.0459 (4)
C8	0.8367 (3)	0.57217 (19)	0.70483 (15)	0.0520 (5)
H8A	0.8079	0.5563	0.6297	0.062*
H8B	0.9634	0.5754	0.7107	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1121 (14)	0.0726 (11)	0.0598 (10)	−0.0001 (10)	0.0104 (9)	0.0177 (8)
O2	0.0870 (11)	0.0473 (9)	0.0889 (12)	−0.0090 (8)	−0.0091 (10)	0.0089 (9)
O3	0.0804 (11)	0.0609 (9)	0.0606 (8)	−0.0032 (7)	0.0250 (8)	0.0022 (7)
N1	0.0540 (10)	0.0514 (12)	0.0607 (12)	0.0034 (8)	−0.0015 (9)	0.0086 (9)
N2	0.0725 (12)	0.0476 (10)	0.0607 (10)	−0.0110 (9)	−0.0118 (9)	0.0040 (8)
N3	0.0606 (11)	0.0435 (9)	0.0617 (11)	−0.0068 (8)	−0.0014 (9)	0.0033 (8)
N4	0.0883 (14)	0.0733 (14)	0.0603 (11)	−0.0078 (11)	−0.0024 (11)	0.0088 (10)
C1	0.0507 (10)	0.0538 (13)	0.0390 (9)	0.0025 (9)	0.0020 (8)	−0.0012 (9)
C2	0.0520 (10)	0.0424 (11)	0.0472 (10)	0.0023 (9)	0.0000 (9)	−0.0059 (8)
C3	0.0402 (9)	0.0447 (11)	0.0406 (9)	−0.0012 (8)	−0.0020 (8)	−0.0021 (8)
C4	0.0498 (10)	0.0512 (12)	0.0413 (9)	−0.0013 (9)	0.0033 (8)	−0.0052 (9)
C5	0.0510 (10)	0.0419 (11)	0.0516 (10)	0.0002 (8)	−0.0032 (9)	−0.0080 (9)
C6	0.0399 (9)	0.0439 (11)	0.0475 (9)	−0.0005 (8)	−0.0050 (8)	0.0046 (9)
C7	0.0460 (9)	0.0490 (11)	0.0426 (9)	−0.0047 (9)	−0.0030 (9)	−0.0025 (8)
C8	0.0594 (12)	0.0456 (12)	0.0510 (10)	−0.0008 (10)	−0.0012 (10)	0.0031 (9)

Geometric parameters (Å, º)

O1—N1	1.225 (2)	C2—H2B	0.9300
O2—N1	1.216 (2)	C3—C4	1.393 (3)
O3—C7	1.207 (2)	C3—C7	1.493 (3)
N1—C6	1.462 (2)	C4—C5	1.372 (3)
N2—N3	1.229 (2)	C4—H4A	0.9300
N2—C8	1.459 (2)	C5—C6	1.378 (3)
N3—N4	1.122 (2)	C5—H5A	0.9300
C1—C6	1.377 (3)	C7—C8	1.508 (3)
C1—C2	1.379 (3)	C8—H8A	0.9700
C1—H1A	0.9300	C8—H8B	0.9700
C2—C3	1.386 (2)		
O2—N1—O1	123.83 (18)	C3—C4—H4A	119.4
O2—N1—C6	118.28 (16)	C4—C5—C6	118.00 (18)
O1—N1—C6	117.89 (18)	C4—C5—H5A	121.0
N3—N2—C8	115.67 (17)	C6—C5—H5A	121.0
N4—N3—N2	171.7 (2)	C1—C6—C5	122.64 (18)
C6—C1—C2	118.44 (17)	C1—C6—N1	118.80 (16)
C6—C1—H1A	120.8	C5—C6—N1	118.55 (18)
C2—C1—H1A	120.8	O3—C7—C3	121.29 (19)
C1—C2—C3	120.62 (18)	O3—C7—C8	121.17 (19)
C1—C2—H2B	119.7	C3—C7—C8	117.52 (15)
C3—C2—H2B	119.7	N2—C8—C7	114.03 (16)
C2—C3—C4	119.02 (17)	N2—C8—H8A	108.7
C2—C3—C7	122.04 (17)	C7—C8—H8A	108.7
C4—C3—C7	118.93 (15)	N2—C8—H8B	108.7
C5—C4—C3	121.25 (16)	C7—C8—H8B	108.7
C5—C4—H4A	119.4	H8A—C8—H8B	107.6

C8—N2—N3—N4	−176.5 (17)	O2—N1—C6—C1	−156.72 (18)
C6—C1—C2—C3	1.5 (3)	O1—N1—C6—C1	23.6 (2)
C1—C2—C3—C4	−1.0 (3)	O2—N1—C6—C5	24.0 (2)
C1—C2—C3—C7	177.54 (16)	O1—N1—C6—C5	−155.73 (18)
C2—C3—C4—C5	−0.6 (3)	C2—C3—C7—O3	−161.46 (19)
C7—C3—C4—C5	−179.14 (16)	C4—C3—C7—O3	17.1 (3)
C3—C4—C5—C6	1.5 (3)	C2—C3—C7—C8	19.8 (2)
C2—C1—C6—C5	−0.6 (3)	C4—C3—C7—C8	−161.67 (16)
C2—C1—C6—N1	−179.83 (16)	N3—N2—C8—C7	−67.2 (3)
C4—C5—C6—C1	−0.9 (3)	O3—C7—C8—N2	4.1 (3)
C4—C5—C6—N1	178.33 (16)	C3—C7—C8—N2	−177.17 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···N2$^{\text{i}}$	0.97	2.48	3.422 (3)	165

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