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Methyl 1-benzyl-1*H*-1,2,3-triazole-4-carboxylate

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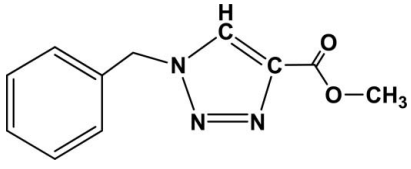
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 12.7.

In the title compound, $C_{11}H_{11}N_3O_2$, prepared by the [3+2] cycloaddition reaction of benzyl azide with methyl propiolate, the dihedral angle between the ring planes is 67.87 (11)°.

Related literature

For catalytic transformations of organic alkynes mediated by ruthenium complexes, see: Naota *et al.* (1998); Bruneau & Dixneuf (1999); Trost *et al.* (2001); Chen *et al.* (2009); Cheng *et al.* (2009). For the synthesis of triazoles, see: Padwa (1976). For applications of triazoles, see: Krivopalov & Shkurko (2005).



Experimental

Crystal data

$C_{11}H_{11}N_3O_2$
M_r = 217.23
Monoclinic, $P2_1/c$
a = 12.0551 (6) Å
b = 5.6285 (3) Å
c = 16.7578 (10) Å
β = 110.664 (3)°
V = 1063.90 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 K
0.55 × 0.40 × 0.35 mm

Data collection

Nonuis KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.949, T_{max} = 0.967
7021 measured reflections
1845 independent reflections
1615 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.01
1845 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2294).

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supplementary materials

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Methyl 1-benzyl-1*H*-1,2,3-triazole-4-carboxylate

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Comment

Catalytic transformations of organic alkynes mediated by ruthenium complexes are well known, and confirmation for the intermediacy of ruthenium(II) acetylide and vinylidene complexes has been provided (Bruneau & Dixneuf, 1999; Cheng *et al.*, 2009; Naota *et al.*, 1998; Trost *et al.*, 2001). Therefore, ruthenium was a logical choice in our search for a new catalyst of click reaction (Chen *et al.*, 2009). Organic azides are synthetically useful reagents. Amongst many reactions, perhaps the most significant are the 1,3-dipolar cycloaddition reactions with alkynes to synthesize triazoles (Padwa, 1976). Triazoles are nitrogen heteroarenes which have found a range of important applications in the pharmaceutical and agricultural industries (Krivopalov & Shkurko, 2005).

A mixture of benzyl azide and methyl propiolate (1:1.5 equiv, respectively) in toluene was refluxed for 24 h in the presence of 5% moles of {(Tp)(PPh_3)$_2$Ru(N_3)}, leading to the title compound [Tp is hydridotris(pyrazolyl)borate]. Single crystals of the title compound suitable for X-ray structure analysis were obtained by recrystallization of the crude product from dichloromethane–ether.

In the title compound (Fig. 1), phenyl and triazole are linked together through a methylene group. Of major interest is the methylene C atom, which presents a C—CH_2—N angle of 112.13 (11)°, larger than the ideal tetrahedral value of 109.47°. The N3—C4, C4—C3, C3—N1, N1—N2, and N2—N3 bond lengths are 1.3367 (17), 1.3722 (17), 1.3621 (16), 1.3092 (15) and 1.3560 (15) Å, respectively, which compare with those found for C═C, N═N and C—N bonds in related compounds.

Experimental

A mixture of benzyl azide, methyl propiolate and {(Tp)(PPh_3)$_2$Ru(N_3)} in toluene was refluxed for 24 h. The solvent was removed under vacuum and the product was purified by silica gel chromatography. The unreacted alkyne and traces of side products were first eluted out with ether. The pure 1,4-disubstituted triazole product was then obtained by elution with CH_2Cl_2.

Refinement

H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.2 U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C).

Figures

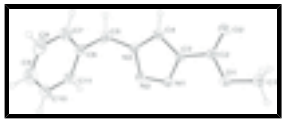

Fig. 1. Molecular structure of the title compound with labelling and displacement ellipsoids drawn at the 30% probability level (H atoms are shown as spheres of arbitrary radius).

Methyl 1-benzyl-1*H*-1,2,3-triazole-4-carboxylate

Crystal data

$C_{11}H_{11}N_3O_2$	$F(000) = 456$
$M_r = 217.23$	$D_x = 1.356$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3680 reflections
$a = 12.0551$ (6) Å	$\theta = 2.6$–$25.0°$
$b = 5.6285$ (3) Å	$\mu = 0.10$ mm^{-1}
$c = 16.7578$ (10) Å	$T = 200$ K
$\beta = 110.664$ (3)°	Prism, colorless
$V = 1063.90$ (10) Å^3	0.55 × 0.40 × 0.35 mm
$Z = 4$	

Data collection

Nonuis KappaCCD diffractometer	1845 independent reflections
Radiation source: fine-focus sealed tube	1615 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.023$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
CCD rotation images, thick slices scans	$h = -14 \rightarrow 14$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	$k = -6 \rightarrow 6$
$T_{min} = 0.949$, $T_{max} = 0.967$	$l = -19 \rightarrow 18$
7021 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.091$	H-atom parameters constrained
$S = 1.01$	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.2782P]$ where $P = (F_o^2 + 2F_c^2)/3$
1845 reflections	$(\Delta/\sigma)_{max} < 0.001$
145 parameters	$\Delta\rho_{max} = 0.15$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.16$ e Å^{-3}
0 constraints	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}

C1	−0.11251 (12)	1.1370 (3)	0.66802 (10)	0.0462 (4)
H1A	−0.1102	1.3069	0.6814	0.069*
H1B	−0.1054	1.0444	0.7191	0.069*
H1C	−0.1878	1.0991	0.6226	0.069*
C2	−0.00887 (11)	0.8505 (2)	0.61959 (8)	0.0359 (3)
C3	0.09145 (10)	0.8061 (2)	0.59130 (8)	0.0344 (3)
C4	0.13667 (11)	0.5915 (2)	0.57855 (8)	0.0379 (3)
H4	0.1102	0.4373	0.5864	0.045*
C5	0.30857 (12)	0.4920 (2)	0.53001 (10)	0.0441 (3)
H5A	0.3067	0.5334	0.4721	0.053*
H5B	0.2820	0.3252	0.5288	0.053*
C6	0.43422 (11)	0.5135 (2)	0.59233 (8)	0.0359 (3)
C7	0.48209 (14)	0.3404 (3)	0.65326 (10)	0.0508 (4)
H7	0.4357	0.2066	0.6564	0.061*
C8	0.59814 (16)	0.3616 (4)	0.71015 (11)	0.0662 (5)
H8	0.6306	0.2417	0.7518	0.079*
C9	0.66570 (14)	0.5532 (4)	0.70663 (11)	0.0647 (5)
H9	0.7448	0.5669	0.7456	0.078*
C10	0.61865 (14)	0.7251 (3)	0.64659 (12)	0.0630 (5)
H10	0.6653	0.8590	0.6442	0.076*
C11	0.50372 (12)	0.7062 (3)	0.58917 (10)	0.0488 (4)
H11	0.4724	0.8262	0.5474	0.059*
N1	0.15518 (9)	0.98335 (18)	0.57253 (7)	0.0379 (3)
N2	0.23722 (9)	0.88701 (19)	0.54855 (8)	0.0404 (3)
N3	0.22627 (9)	0.64798 (18)	0.55266 (7)	0.0380 (3)
O1	−0.01523 (8)	1.07905 (16)	0.63991 (6)	0.0417 (3)
O2	−0.07666 (8)	0.69845 (17)	0.62483 (7)	0.0480 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0442 (8)	0.0438 (8)	0.0558 (9)	−0.0033 (6)	0.0239 (7)	−0.0050 (6)
C2	0.0330 (6)	0.0314 (6)	0.0372 (7)	−0.0041 (5)	0.0047 (5)	0.0008 (5)
C3	0.0298 (6)	0.0293 (6)	0.0373 (7)	−0.0038 (5)	0.0032 (5)	−0.0005 (5)
C4	0.0326 (6)	0.0298 (6)	0.0456 (8)	−0.0047 (5)	0.0067 (6)	−0.0009 (5)
C5	0.0396 (7)	0.0351 (7)	0.0554 (8)	0.0013 (6)	0.0143 (6)	−0.0090 (6)
C6	0.0361 (7)	0.0332 (7)	0.0416 (7)	0.0016 (5)	0.0177 (6)	−0.0016 (5)
C7	0.0564 (9)	0.0444 (8)	0.0557 (9)	0.0048 (7)	0.0248 (7)	0.0087 (7)
C8	0.0688 (11)	0.0781 (13)	0.0459 (9)	0.0277 (10)	0.0131 (8)	0.0094 (8)
C9	0.0395 (8)	0.0891 (14)	0.0590 (10)	0.0112 (9)	0.0091 (7)	−0.0207 (10)
C10	0.0409 (8)	0.0662 (11)	0.0854 (13)	−0.0141 (8)	0.0266 (8)	−0.0157 (9)
C11	0.0439 (8)	0.0435 (8)	0.0618 (9)	−0.0029 (6)	0.0221 (7)	0.0054 (7)
N1	0.0323 (5)	0.0309 (6)	0.0472 (7)	−0.0012 (4)	0.0098 (5)	−0.0001 (5)
N2	0.0358 (6)	0.0300 (6)	0.0533 (7)	−0.0010 (4)	0.0134 (5)	−0.0008 (5)
N3	0.0329 (5)	0.0283 (5)	0.0474 (7)	−0.0015 (4)	0.0076 (5)	−0.0037 (5)
O1	0.0387 (5)	0.0336 (5)	0.0555 (6)	−0.0053 (4)	0.0199 (4)	−0.0067 (4)
O2	0.0437 (5)	0.0365 (5)	0.0641 (7)	−0.0083 (4)	0.0196 (5)	0.0017 (4)

Geometric parameters (Å, °)

C1—O1	1.4469 (16)	C5—H5B	0.9900
C1—H1A	0.9800	C6—C7	1.3817 (19)
C1—H1B	0.9800	C6—C11	1.3830 (19)
C1—H1C	0.9800	C7—C8	1.392 (2)
C2—O2	1.2076 (15)	C7—H7	0.9500
C2—O1	1.3400 (15)	C8—C9	1.365 (3)
C2—C3	1.4678 (19)	C8—H8	0.9500
C3—N1	1.3621 (16)	C9—C10	1.367 (3)
C3—C4	1.3722 (17)	C9—H9	0.9500
C4—N3	1.3367 (17)	C10—C11	1.384 (2)
C4—H4	0.9500	C10—H10	0.9500
C5—N3	1.4713 (17)	C11—H11	0.9500
C5—C6	1.5110 (18)	N1—N2	1.3092 (15)
C5—H5A	0.9900	N2—N3	1.3560 (15)
O1—C1—H1A	109.5	C11—C6—C5	120.62 (12)
O1—C1—H1B	109.5	C6—C7—C8	120.16 (15)
H1A—C1—H1B	109.5	C6—C7—H7	119.9
O1—C1—H1C	109.5	C8—C7—H7	119.9
H1A—C1—H1C	109.5	C9—C8—C7	120.57 (16)
H1B—C1—H1C	109.5	C9—C8—H8	119.7
O2—C2—O1	124.12 (13)	C7—C8—H8	119.7
O2—C2—C3	123.97 (12)	C8—C9—C10	119.48 (15)
O1—C2—C3	111.90 (10)	C8—C9—H9	120.3
N1—C3—C4	108.74 (11)	C10—C9—H9	120.3
N1—C3—C2	123.10 (11)	C9—C10—C11	120.74 (16)
C4—C3—C2	128.16 (11)	C9—C10—H10	119.6
N3—C4—C3	104.61 (11)	C11—C10—H10	119.6
N3—C4—H4	127.7	C6—C11—C10	120.32 (15)
C3—C4—H4	127.7	C6—C11—H11	119.8
N3—C5—C6	112.13 (11)	C10—C11—H11	119.8
N3—C5—H5A	109.2	N2—N1—C3	108.43 (10)
C6—C5—H5A	109.2	N1—N2—N3	107.32 (10)
N3—C5—H5B	109.2	C4—N3—N2	110.89 (11)
C6—C5—H5B	109.2	C4—N3—C5	129.62 (11)
H5A—C5—H5B	107.9	N2—N3—C5	119.49 (11)
C7—C6—C11	118.73 (13)	C2—O1—C1	115.14 (10)
C7—C6—C5	120.65 (12)		

Fig. 1