

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Methyl 2-methyl-2H-1,2,3-triazole-4-carboxylate

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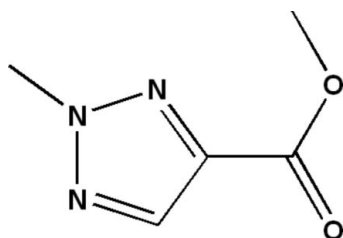
Received 16 June 2009; accepted 27 June 2009

Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.119; data-to-parameter ratio = 13.6.

In the title compound, $\text{C}_5\text{H}_7\text{N}_3\text{O}_2$, all non-H atoms lie in a common plane, with a maximum deviation of 0.061 (2°) for the ester methyl C atom. The structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background to the applications of triazoles and their derivatives, see: Abu-Orabi *et al.* (1989); Fan & Katritzky (1996); Dehne (1994); Wang *et al.* (1998). For a related structure, see: Prabakaran *et al.* (2009).



Experimental

Crystal data

$\text{C}_5\text{H}_7\text{N}_3\text{O}_2$
 $M_r = 141.14$
Monoclinic, $P2_1/c$
 $a = 3.9482$ (10) Å
 $b = 7.9549$ (15) Å
 $c = 21.655$ (4) Å
 $\beta = 92.05$ (2°)

$V = 679.7$ (2) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 290$ K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Oxford Xcalibur Eos(Nova) CCD detector diffractometer
Absorption correction: multi-scan (*CrysAlisPro RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.926$, $T_{\max} = 0.989$

7464 measured reflections
1262 independent reflections
910 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.119$
 $S = 1.07$
1262 reflections

93 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C1}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.93	2.53	3.416 (3)	159

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

Data collection: *CrysAlisPro CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlisPro CCD*; data reduction: *CrysAlisPro RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank the Department of Science and Technology, India, for use of the CCD facility setup under the IRHPA-DST program at IISc. We thank Professor T. N. Guru Row, IISc, Bangalore, for useful crystallographic discussions. FNK thanks the DST for Fast Track Proposal funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2973).

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supporting information

Acta Cryst. (2009). E65, o1752 [doi:10.1107/S1600536809024829]

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S1. Comment

Triazoles and their derivatives find their application in pharmaceuticals, agrochemicals, dyes, photographic materials, and in corrosion inhibition (Fan & Katritzky, 1996; Dehne, 1994; Abu-Orabi *et al.*, 1989). In continuation of our earlier report (Prabakaran *et al.*, 2009), here the crystal structure of the title compound is presented. All non-H atoms lie in a common plane with maximum deviation of 0.061 (2)° for atom C4. The packing is stabilized by C—H···O hydrogen bonds.

S2. Experimental

To Methyl 1H-1,2,3-triazole-4-carboxylate (2 g) in dry DMF (15 ml) maintained at 273 K in nitrogen atmosphere, was added K₂CO₃ (1.3 g), methyl iodide (ml), the mixture was then stirred at 273 K for 1 hr, allowed to warm to room temperature and stirred till completion of reaction, monitored by TLC. The reaction mixture on LCMS analysis showed three isomers well separated with their significant retention time and high purity. Three fractions were identified by mass spectroscopy. The solvent was evaporated under vacuo and the residue was isolated into individual isomers by column chromatography. A portion of the mixture was also analysed by HPLC analysis and also isolated by preparative HPLC techniques. The single crystal of the title compound for X-ray structure analysis was obtained from ether solution by slow evaporation.

S3. Refinement

All the H atoms were positioned geometrically and refined using a riding model with C—H bond lengths of 0.93 Å and 0.96 Å for aromatic and for methyl H atoms respectively and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The methyl groups were allowed to rotate but not to tip.

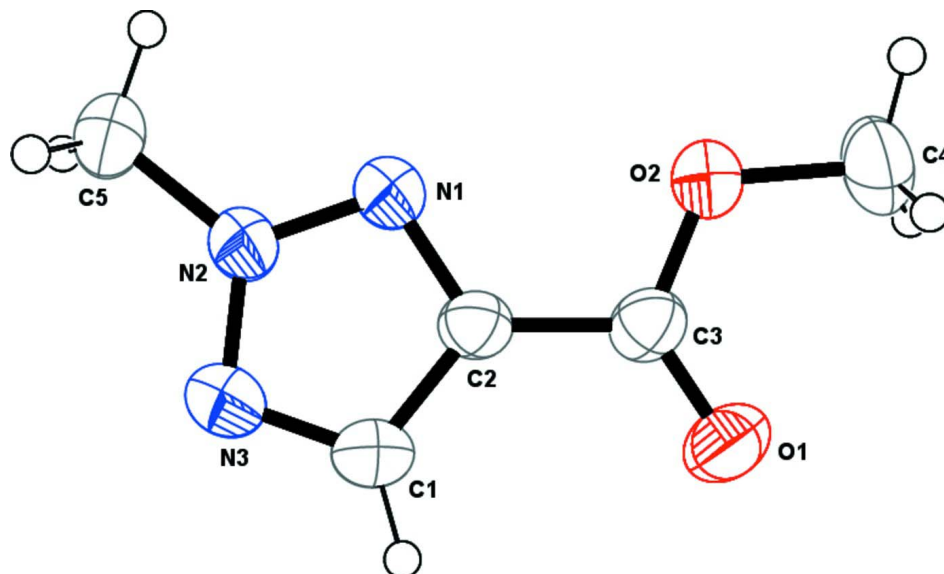


Figure 1

ORTEP diagram of the asymmetric unit of (I) with 50% probability displacement ellipsoids.

Methyl 2-methyl-2H-1,2,3-triazole-4-carboxylate

Crystal data

$C_5H_7N_3O_2$

$M_r = 141.14$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1bc$

$a = 3.9482 (10) \text{ \AA}$

$b = 7.9549 (15) \text{ \AA}$

$c = 21.655 (4) \text{ \AA}$

$\beta = 92.05 (2)^\circ$

$V = 679.7 (2) \text{ \AA}^3$

$Z = 4$

$F(000) = 296$

$D_x = 1.379 \text{ Mg m}^{-3}$

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

Cell parameters from 783 reflections

$\theta = 2.0\text{--}21.4^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 290 \text{ K}$

Plate, colorless

$0.30 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Oxford Xcalibur Eos(Nova) CCD detector
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.926$, $T_{\max} = 0.989$

7464 measured reflections

1262 independent reflections

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

$R_{\text{int}} = 0.043$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 3.2^\circ$

$h = -4 \rightarrow 4$

$k = -9 \rightarrow 9$

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.119$

$S = 1.07$

1262 reflections

93 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

$$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.0659P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$$

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.2453 (4)	0.56849 (18)	0.43005 (7)	0.0442 (4)
N2	0.3338 (4)	0.71794 (19)	0.45090 (7)	0.0449 (4)
N3	0.4803 (5)	0.8185 (2)	0.41047 (7)	0.0562 (5)
O1	0.3755 (4)	0.42664 (18)	0.27655 (6)	0.0620 (5)
O2	0.1319 (4)	0.29948 (17)	0.35597 (6)	0.0539 (4)
C1	0.4849 (6)	0.7258 (2)	0.35967 (9)	0.0547 (6)
H1	0.5707	0.7596	0.3222	0.066*
C2	0.3408 (5)	0.5700 (2)	0.37131 (8)	0.0407 (5)
C3	0.2884 (5)	0.4274 (2)	0.32930 (8)	0.0435 (5)
C4	0.0524 (6)	0.1556 (3)	0.31750 (10)	0.0634 (6)
H4A	-0.0937	0.1895	0.2834	0.095*
H4B	-0.0597	0.0720	0.3414	0.095*
H4C	0.2579	0.1095	0.3022	0.095*
C5	0.2690 (6)	0.7700 (3)	0.51385 (9)	0.0567 (6)
H5A	0.1463	0.6831	0.5342	0.085*
H5B	0.1374	0.8715	0.5130	0.085*
H5C	0.4805	0.7896	0.5359	0.085*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0534 (11)	0.0371 (9)	0.0423 (9)	-0.0034 (7)	0.0041 (7)	0.0000 (7)
N2	0.0581 (11)	0.0348 (9)	0.0419 (9)	-0.0033 (7)	0.0029 (7)	-0.0004 (7)
N3	0.0755 (13)	0.0424 (10)	0.0511 (10)	-0.0100 (9)	0.0069 (9)	0.0050 (8)
O1	0.0904 (12)	0.0553 (9)	0.0413 (8)	0.0058 (8)	0.0164 (7)	0.0007 (6)
O2	0.0715 (10)	0.0457 (8)	0.0450 (8)	-0.0120 (7)	0.0066 (6)	-0.0058 (6)
C1	0.0740 (15)	0.0480 (12)	0.0427 (11)	-0.0060 (10)	0.0109 (10)	0.0065 (9)
C2	0.0454 (11)	0.0381 (10)	0.0388 (10)	0.0017 (8)	0.0034 (8)	0.0050 (8)
C3	0.0502 (12)	0.0422 (11)	0.0382 (10)	0.0072 (9)	0.0018 (8)	0.0038 (8)
C4	0.0752 (16)	0.0449 (12)	0.0699 (15)	-0.0064 (11)	0.0016 (12)	-0.0155 (10)
C5	0.0770 (16)	0.0479 (13)	0.0455 (11)	-0.0022 (10)	0.0067 (10)	-0.0084 (9)

Geometric parameters (\AA , $^\circ$)

N1—N2	1.315 (2)	C1—H1	0.9300
N1—C2	1.340 (2)	C2—C3	1.464 (3)

N2—N3	1.333 (2)	C4—H4A	0.9600
N2—C5	1.456 (2)	C4—H4B	0.9600
N3—C1	1.325 (2)	C4—H4C	0.9600
O1—C3	1.205 (2)	C5—H5A	0.9600
O2—C3	1.333 (2)	C5—H5B	0.9600
O2—C4	1.444 (2)	C5—H5C	0.9600
C1—C2	1.391 (3)		
N2—N1—C2	103.75 (15)	O2—C3—C2	112.31 (16)
N1—N2—N3	115.69 (15)	O2—C4—H4A	109.5
N1—N2—C5	121.67 (15)	O2—C4—H4B	109.5
N3—N2—C5	122.63 (16)	H4A—C4—H4B	109.5
C1—N3—N2	103.33 (16)	O2—C4—H4C	109.5
C3—O2—C4	116.74 (16)	H4A—C4—H4C	109.5
N3—C1—C2	109.13 (17)	H4B—C4—H4C	109.5
N3—C1—H1	125.4	N2—C5—H5A	109.5
C2—C1—H1	125.4	N2—C5—H5B	109.5
N1—C2—C1	108.10 (16)	H5A—C5—H5B	109.5
N1—C2—C3	123.02 (17)	N2—C5—H5C	109.5
C1—C2—C3	128.88 (17)	H5A—C5—H5C	109.5
O1—C3—O2	124.03 (17)	H5B—C5—H5C	109.5
O1—C3—C2	123.65 (18)		
C2—N1—N2—N3	0.1 (2)	N3—C1—C2—C3	179.47 (18)
C2—N1—N2—C5	179.01 (17)	C4—O2—C3—O1	-2.7 (3)
N1—N2—N3—C1	0.2 (2)	C4—O2—C3—C2	176.96 (16)
C5—N2—N3—C1	-178.75 (18)	N1—C2—C3—O1	-179.38 (18)
N2—N3—C1—C2	-0.3 (2)	C1—C2—C3—O1	1.7 (3)
N2—N1—C2—C1	-0.3 (2)	N1—C2—C3—O2	1.0 (3)
N2—N1—C2—C3	-179.42 (17)	C1—C2—C3—O2	-177.96 (19)
N3—C1—C2—N1	0.4 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1 \cdots O1 ⁱ	0.93	2.53	3.416 (3)	159

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