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Crystal structure of 1-methoxy-5-methyl-*N*-phenyl-1,2,3-triazole-4-carboxamide

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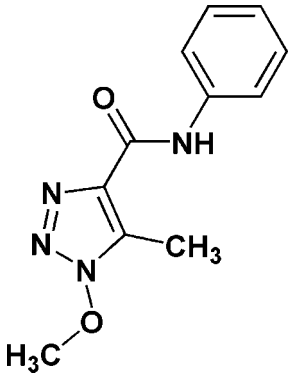
The title compound, $C_{11}H_{12}N_4O_2$,was prepared *via* the transformation of sodium 4-acetyl-1-phenyl-1*H*-[1.2.3]triazolate under the action of methoxyamine hydrochloride. The dihedral angle between the triazole and phenyl rings is 25.12 (16)° and the C atom of the methoxy group deviates from the triazole plane by 0.894 (4)Å. The conformation of the CONH*R*-group is consolodated by an intramolecular N—H···N hydrogen bond to an N-atom of the triazole ring, which closes an *S*(5) ring. In the crystal, weak N—H···N hydrogen bonds link the molecules into *C*(6) [010] chains.

Keywords: crystal structure; 1,2,3-triazole; rearrangements; hydrogen bonding.

CCDC reference: 1426448

1. Related literature

For biological activities of 1.2.3-triazoles, see: Sathish Kumar & Kavitha (2013); Khazhieva *et al.* (2015*a*). For the synthesis, see: Khazhieva *et al.* (2015*b*).



2. Experimental

2.1. Crystal data

$C_{11}H_{12}N_4O_2$
M_r = 232.25
Monoclinic, $P2_1/c$
a = 11.4637 (8) Å
b = 6.4345 (13) Å
c = 15.822 (3) Å
β = 100.367 (12)°
V = 1148.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.21 × 0.16 × 0.09 mm

2.2. Data collection

Agilent Xcalibur S CCD diffractometer
7259 measured reflections
2302 independent reflections
1077 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.147
S = 1.00
2302 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N2	0.86 (2)	2.33 (3)	2.780 (4)	113 (2)
N1—H1···N3^{i}	0.86 (2)	2.41 (2)	3.184 (3)	150 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXS97* (Sheldrick, 2008); molecular graphics: *publCIF* (Westrip, 2010); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7511).

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supporting information

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S1. Synthesis and crystallization

The titled compound was prepared as previously reported (Khazhieva *et al.*, 2015*b*). Crystals were obtained by slow evaporation of a solution in ethanol.

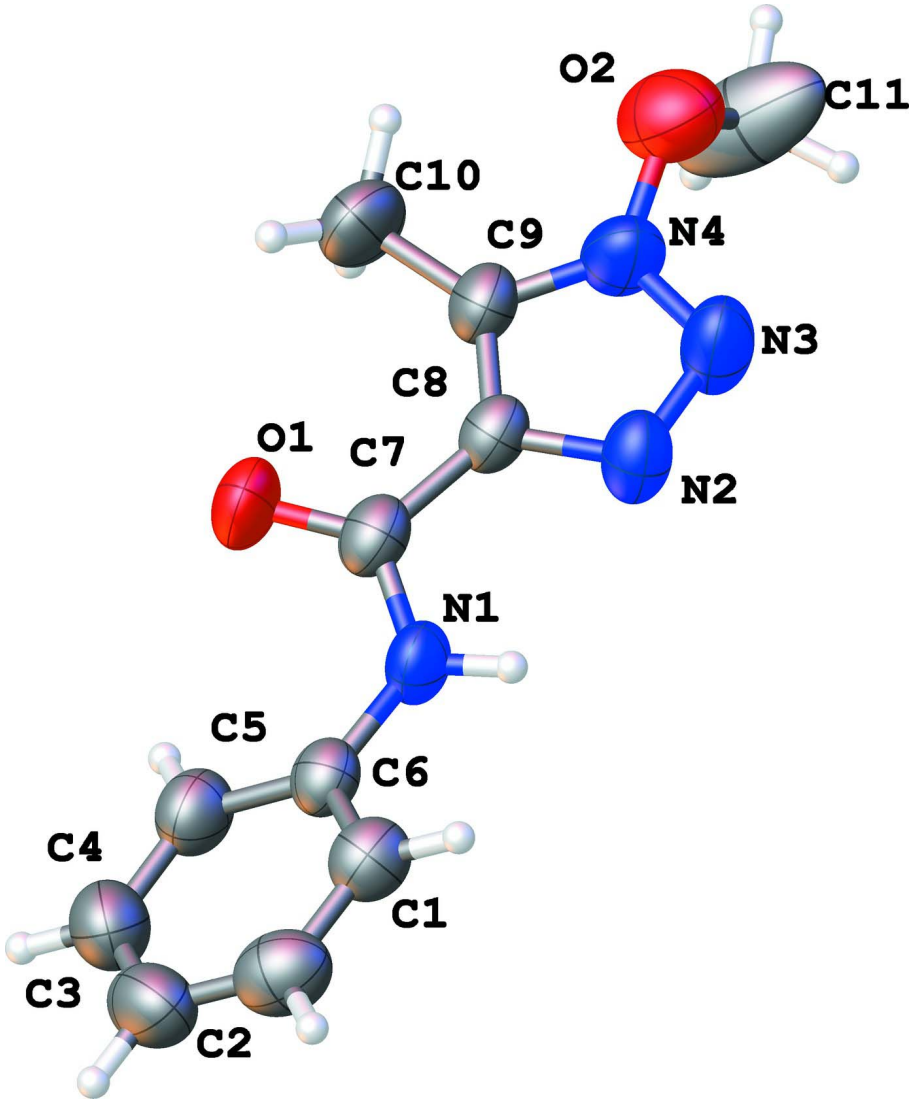

Figure 1

The molecular structure of (I), with 50% probability displacement ellipsoids for non-H atoms.

1-Methoxy-5-methyl-*N*-phenyl-1,2,3-triazole-4-carboxamide

Crystal data

$C_{11}H_{12}N_4O_2$
M_r = 232.25
Monoclinic, $P2_1/c$
a = 11.4637 (8) Å
b = 6.4345 (13) Å
c = 15.822 (3) Å
β = 100.367 (12)°
V = 1148.0 (3) Å^3
Z = 4
F(000) = 488

D_x = 1.344 Mg m^{-3}
Melting point: 310 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1077 reflections
θ = 2.9–26.4°
μ = 0.10 mm^{-1}
T = 295 K
Prism, colorless
0.21 × 0.16 × 0.09 mm

Data collection

Agilent Xcalibur S CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
7259 measured reflections
2302 independent reflections
1077 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.9°$
$h = -7 \rightarrow 14$
$k = -5 \rightarrow 8$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.147$
$S = 1.00$
2302 reflections
160 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-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.

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.61315 (16)	0.0563 (3)	0.15704 (14)	0.0777 (7)
C8	0.7887 (2)	0.1712 (4)	0.24262 (18)	0.0475 (7)
C6	0.7399 (2)	−0.2566 (4)	0.08075 (18)	0.0496 (7)
C7	0.7204 (2)	0.0365 (4)	0.17728 (19)	0.0533 (7)
N2	0.90562 (18)	0.1400 (4)	0.27423 (17)	0.0605 (7)
N4	0.8463 (2)	0.3915 (4)	0.33811 (19)	0.0708 (8)
C9	0.7489 (2)	0.3375 (4)	0.28291 (19)	0.0553 (8)
N1	0.7844 (2)	−0.1083 (4)	0.14353 (16)	0.0530 (6)
N3	0.9416 (2)	0.2771 (4)	0.3343 (2)	0.0759 (8)
O2	0.8515 (2)	0.5302 (4)	0.40535 (18)	0.0956 (8)
C1	0.7975 (2)	−0.4450 (5)	0.0824 (2)	0.0589 (8)
H1A	0.8634	−0.4721	0.1246	0.071*
C5	0.6434 (3)	−0.2172 (5)	0.0169 (2)	0.0641 (8)
H5A	0.6049	−0.0896	0.0148	0.077*
C3	0.6605 (3)	−0.5520 (6)	−0.0411 (2)	0.0809 (10)
H3A	0.6331	−0.6524	−0.0821	0.097*

C2	0.7571 (3)	−0.5924 (5)	0.0214 (2)	0.0739 (9)
H2A	0.7955	−0.7200	0.0225	0.089*
C4	0.6048 (3)	−0.3651 (6)	−0.0430 (2)	0.0769 (10)
H4A	0.5395	−0.3381	−0.0857	0.092*
C11	0.9070 (4)	0.7045 (6)	0.3901 (3)	0.137 (2)
H11A	0.8970	0.8073	0.4321	0.205*
H11B	0.8741	0.7551	0.3337	0.205*
H11C	0.9900	0.6765	0.3933	0.205*
C10	0.6343 (3)	0.4512 (5)	0.2740 (2)	0.0818 (10)
H10A	0.6298	0.5214	0.3268	0.123*
H10B	0.5699	0.3543	0.2609	0.123*
H10C	0.6292	0.5511	0.2284	0.123*
H1	0.858 (2)	−0.109 (4)	0.1666 (17)	0.048 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0365 (12)	0.1021 (17)	0.0930 (17)	0.0057 (10)	0.0076 (11)	−0.0186 (13)
C8	0.0393 (15)	0.0458 (16)	0.0589 (18)	0.0007 (12)	0.0134 (13)	0.0056 (14)
C6	0.0414 (15)	0.0538 (18)	0.0549 (19)	−0.0035 (14)	0.0117 (14)	0.0045 (16)
C7	0.0418 (16)	0.0582 (18)	0.062 (2)	−0.0016 (14)	0.0146 (15)	0.0066 (16)
N2	0.0444 (14)	0.0516 (15)	0.0836 (19)	−0.0032 (11)	0.0062 (13)	−0.0019 (14)
N4	0.0607 (17)	0.0658 (17)	0.089 (2)	−0.0011 (13)	0.0206 (15)	−0.0268 (16)
C9	0.0400 (16)	0.065 (2)	0.0624 (19)	−0.0028 (14)	0.0117 (15)	−0.0013 (16)
N1	0.0354 (13)	0.0558 (15)	0.0661 (17)	0.0045 (11)	0.0041 (12)	−0.0011 (13)
N3	0.0465 (15)	0.0710 (17)	0.107 (2)	0.0004 (13)	0.0059 (14)	−0.0188 (17)
O2	0.0967 (18)	0.0961 (18)	0.102 (2)	−0.0125 (14)	0.0395 (15)	−0.0152 (16)
C1	0.0573 (17)	0.0563 (19)	0.064 (2)	0.0021 (15)	0.0140 (15)	0.0062 (17)
C5	0.0512 (18)	0.073 (2)	0.067 (2)	0.0069 (15)	0.0089 (16)	0.0035 (19)
C3	0.077 (2)	0.093 (3)	0.075 (3)	−0.018 (2)	0.018 (2)	−0.024 (2)
C2	0.081 (2)	0.061 (2)	0.085 (3)	−0.0020 (18)	0.030 (2)	−0.004 (2)
C4	0.061 (2)	0.098 (3)	0.070 (2)	−0.005 (2)	0.0055 (17)	−0.010 (2)
C11	0.171 (4)	0.055 (2)	0.221 (5)	−0.019 (2)	0.133 (4)	−0.005 (3)
C10	0.0566 (19)	0.098 (2)	0.092 (3)	0.0195 (17)	0.0172 (17)	−0.016 (2)

Geometric parameters (Å, º)

O1—C7	1.220 (3)	C1—C2	1.372 (4)
C8—N2	1.359 (3)	C1—H1A	0.9300
C8—C9	1.365 (3)	C5—C4	1.359 (4)
C8—C7	1.463 (4)	C5—H5A	0.9300
C6—C1	1.379 (4)	C3—C4	1.360 (5)
C6—C5	1.381 (4)	C3—C2	1.370 (5)
C6—N1	1.405 (3)	C3—H3A	0.9300
C7—N1	1.354 (3)	C2—H2A	0.9300
N2—N3	1.308 (3)	C4—H4A	0.9300
N4—N3	1.327 (3)	C11—H11A	0.9600
N4—C9	1.334 (3)	C11—H11B	0.9600

N4—O2	1.382 (3)	C11—H11C	0.9600
C9—C10	1.488 (4)	C10—H10A	0.9600
N1—H1	0.85 (3)	C10—H10B	0.9600
O2—C11	1.333 (4)	C10—H10C	0.9600
N2—C8—C9	109.5 (2)	C4—C5—C6	120.0 (3)
N2—C8—C7	122.6 (2)	C4—C5—H5A	120.0
C9—C8—C7	127.8 (2)	C6—C5—H5A	120.0
C1—C6—C5	119.6 (3)	C4—C3—C2	120.0 (3)
C1—C6—N1	118.1 (3)	C4—C3—H3A	120.0
C5—C6—N1	122.3 (3)	C2—C3—H3A	120.0
O1—C7—N1	124.1 (3)	C3—C2—C1	120.2 (3)
O1—C7—C8	120.6 (2)	C3—C2—H2A	119.9
N1—C7—C8	115.3 (2)	C1—C2—H2A	119.9
N3—N2—C8	109.2 (2)	C5—C4—C3	120.7 (3)
N3—N4—C9	115.2 (2)	C5—C4—H4A	119.6
N3—N4—O2	118.1 (3)	C3—C4—H4A	119.6
C9—N4—O2	125.9 (2)	O2—C11—H11A	109.5
N4—C9—C8	101.5 (2)	O2—C11—H11B	109.5
N4—C9—C10	123.7 (3)	H11A—C11—H11B	109.5
C8—C9—C10	134.8 (3)	O2—C11—H11C	109.5
C7—N1—C6	126.3 (3)	H11A—C11—H11C	109.5
C7—N1—H1	113.4 (17)	H11B—C11—H11C	109.5
C6—N1—H1	120.2 (17)	C9—C10—H10A	109.5
N2—N3—N4	104.6 (2)	C9—C10—H10B	109.5
C11—O2—N4	111.1 (3)	H10A—C10—H10B	109.5
C2—C1—C6	119.6 (3)	C9—C10—H10C	109.5
C2—C1—H1A	120.2	H10A—C10—H10C	109.5
C6—C1—H1A	120.2	H10B—C10—H10C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N2	0.86 (2)	2.33 (3)	2.780 (4)	113 (2)
N1—H1···N3^{i}	0.86 (2)	2.41 (2)	3.184 (3)	150 (2)

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