Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Methoxy-4-methyl-1*H*-1,2,4-triazol-5(4*H*)-one monohydrate

Wei Gao,* Xin-Ling Wang, Jing Yang and Xue-Fen Wu

School of Pharmacy, Henan University of Traditional Chinese Medicine, Zhengzhou 450008, People's Republic of China
Correspondence e-mail: xiaojun801115@163.com

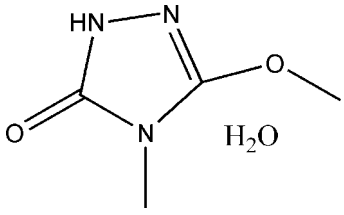
Received 18 May 2012; accepted 22 May 2012

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(O–C) = 0.001 Å; R factor = 0.032; wR factor = 0.090; data-to-parameter ratio = 12.1.

In the title hydrate, $C_4H_7N_3O_2\cdot H_2O$, all the non-H atoms lie on a crystallographic mirror plane. The H atoms of both methyl groups are disordered over two sets of sites. In the crystal, N—H$\cdots$O$_w$ and O$_w$—H$\cdots$O$_k$ (w = water and k = ketone) hydrogen bonds link the components into (010) sheets.

Related literature

For related structures, see: Jin *et al.* (2011); Liu & Liu (2011); Liu *et al.* (2011, 2012); Ustabaş *et al.* (2010). For bioactivity data, see Tan *et al.* (2012).



Experimental

Crystal data

$C_4H_7N_3O_2\cdot H_2O$
M_r = 147.14
Orthorhombic, *Pnma*
a = 6.810 (4) Å
b = 6.506 (4) Å
c = 15.277 (9) Å
V = 676.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 113 K
0.20 × 0.18 × 0.14 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.976, T_{max} = 0.983
6611 measured reflections
873 independent reflections
727 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.090
S = 1.01
873 reflections
72 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
N2—H2$\cdots$O3	0.90 (1)	1.85 (1)	2.7520 (15)	174 (1)
O3—H3$A\cdots$O1^{i}	0.87 (1)	1.89 (1)	2.7518 (18)	174 (1)
O3—H3$B\cdots$O1^{ii}	0.86 (1)	1.94 (1)	2.8024 (18)	179 (1)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *CrystalStructure* (Rigaku/MSC, 2005).

We gratefully acknowledge the financial support from the Doctoral Research Fund of Henan University of Traditional Chinese Medicine.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6806).

References

Jin, D.-C., Zhang, W.-B., Piao, F.-Y. & Han, R.-B. (2011). *Acta Cryst.* E**67**, o1821.
Liu, X.-F. & Liu, X.-H. (2011). *Acta Cryst.* E**67**, o202.
Liu, X. H., Pan, L., Weng, J. Q., Tan, C. X., Li, Y. H., Wang, B. L. & Li, Z. M. (2012). *Mol. Divers.* doi:10,1007/s11030-011-9352-z.
Liu, X. H., Tan, C. X. & Weng, J. Q. (2011). *Phosphorus Sulfur Silicon Relat. Elem.* **186**, 558–564.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tan, C. X., Shi, Y. X., Weng, J. Q., Liu, X. H., Li, B. J. & Zhao, W. G. (2012). *Lett. Drug. Des. Discov.* **9**, 431–435.
Ustabaş, R., Çoruh, U., Ünlüer, D., Hökelek, T. & Ermiş, E. (2010). *Acta Cryst.* E**66**, o2615.

supplementary materials

Acta Cryst. (2012). E**68**, o1925 [doi:10.1107/S1600536812023380]

3-Methoxy-4-methyl-1*H*-1,2,4-triazol-5(4*H*)-one monohydrate

Wei Gao, Xin-Ling Wang, Jing Yang and Xue-Fen Wu

Comment

Sulfur and nitrogen heterocyclic compounds have received considerable attention in recent years because of their medicinal and pesticidal importance, such as 1,3,4-thiadiazoles, pyrimidines, 1,2,4-triazoles (Jin *et al.* 2011; Liu & Liu, 2011; Liu *et al.* 2011; Liu *et al.*, 2012; Tan *et al.*, 2011; Ustabaş *et al.*, 2010).

Single-crystal X-ray diffraction analysis reveals that the title compound crystallizes in the orthorhombic space group Pnma. As shown in Fig. 2, the crystal structure features intermolecular hydrogen bonds O-H···O and N-H···O.

Experimental

The tite compound was available commercially. The crystals were grown from ethanol as colourless prisms

Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Computing details

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear* (Rigaku/MSC, 2005); data reduction: *CrystalClear* (Rigaku/MSC, 2005); 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: *CrystalStructure* (Rigaku/MSC, 2005).

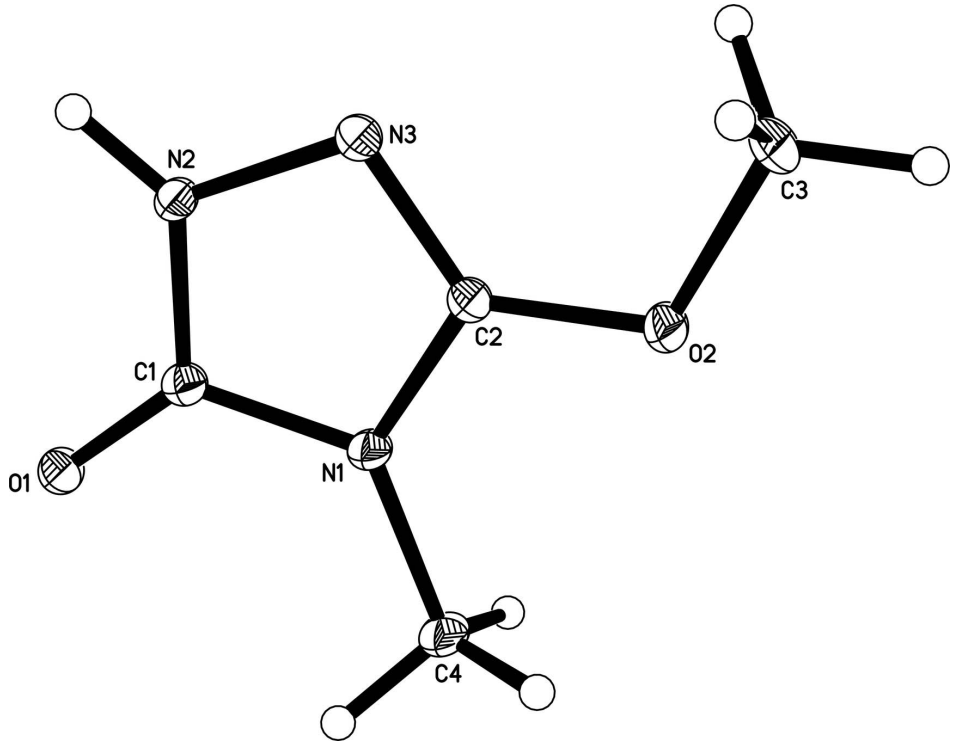


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

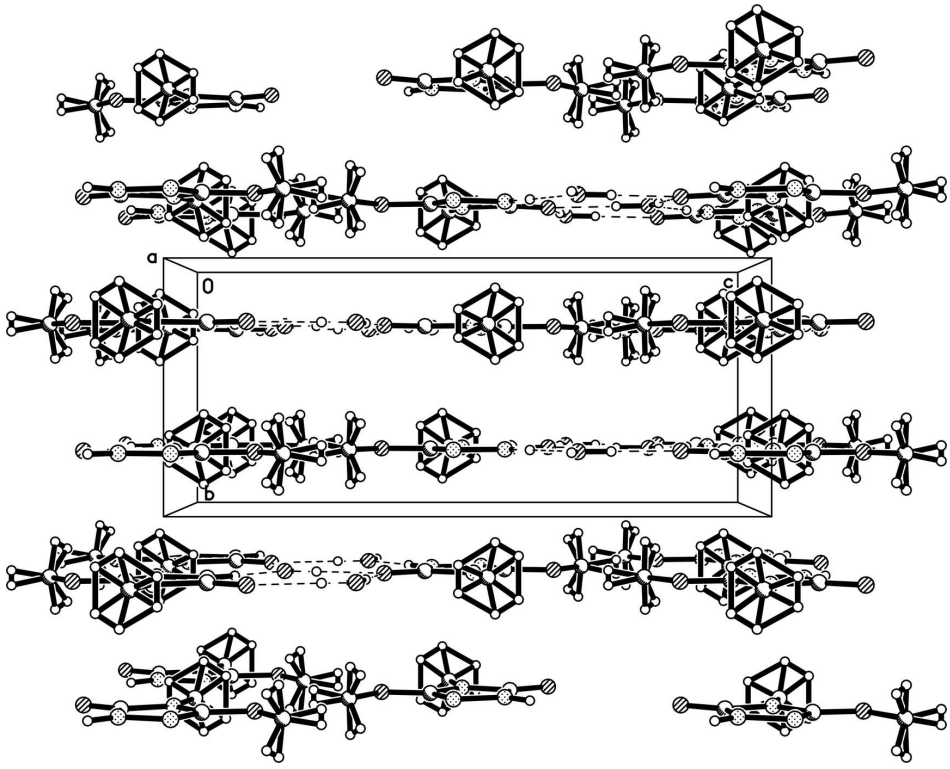


Figure 2

The crystal packing for (I).

3-Methoxy-4-methyl-1*H*-1,2,4-triazol-5(4*H*)-one monohydrate

Crystal data

$C_4H_7N_3O_2·H_2O$
M_r = 147.14
Orthorhombic, *Pnma*
a = 6.810 (4) Å
b = 6.506 (4) Å
c = 15.277 (9) Å
V = 676.9 (7) Å^3
Z = 4
F(000) = 312
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2353 reflections
θ = 3.0–27.8°
μ = 0.12 mm^{-1}
T = 113 K
Prism, colorless
0.20 × 0.18 × 0.14 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.976, T_{max} = 0.983
6611 measured reflections
873 independent reflections
727 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.9°, θ_{min} = 3.3°
h = −8→8
k = −8→7
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.090$
$S = 1.01$
873 reflections
72 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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}	Occ. (<1)
O1	0.64561 (9)	0.2500	0.35699 (4)	0.02165 (15)	
O2	0.49415 (9)	0.2500	0.65038 (4)	0.02269 (16)	
N1	0.62148 (10)	0.2500	0.50977 (4)	0.01766 (17)	
N2	0.35229 (11)	0.2500	0.43609 (4)	0.01895 (17)	
N3	0.29539 (11)	0.2500	0.52418 (5)	0.01921 (18)	
C1	0.54905 (11)	0.2500	0.42578 (6)	0.0172 (2)	
C2	0.46308 (12)	0.2500	0.56483 (6)	0.0169 (2)	
C4	0.82847 (13)	0.2500	0.53342 (6)	0.0235 (2)	
H4A	0.9060	0.1949	0.4847	0.035*	0.50
H4B	0.8481	0.1642	0.5854	0.035*	0.50
H4C	0.8707	0.3909	0.5461	0.035*	0.50
C3	0.31615 (14)	0.2500	0.70293 (6)	0.0257 (2)	
H3D	0.2528	0.1151	0.6990	0.039*	0.50
H3E	0.2262	0.3558	0.6811	0.039*	0.50
H3C	0.3495	0.2791	0.7641	0.039*	0.50
O3	0.04758 (10)	0.2500	0.31776 (5)	0.0449 (2)	
H2	0.2590 (12)	0.2500	0.3942 (6)	0.034 (3)*	
H3A	0.0698 (13)	0.2500	0.2619 (4)	0.047 (4)*	
H3B	−0.0752 (9)	0.2500	0.3306 (6)	0.063 (4)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0167 (3)	0.0331 (3)	0.0152 (3)	0.000	0.0026 (2)	0.000
O2	0.0164 (3)	0.0380 (4)	0.0136 (3)	0.000	0.0014 (2)	0.000

N1	0.0116 (3)	0.0256 (4)	0.0158 (3)	0.000	0.0001 (3)	0.000
N2	0.0134 (3)	0.0301 (4)	0.0133 (3)	0.000	−0.0005 (3)	0.000
N3	0.0152 (3)	0.0273 (4)	0.0151 (3)	0.000	0.0014 (3)	0.000
C1	0.0154 (4)	0.0183 (4)	0.0179 (4)	0.000	−0.0004 (3)	0.000
C2	0.0149 (4)	0.0211 (4)	0.0147 (4)	0.000	0.0011 (3)	0.000
C4	0.0116 (4)	0.0377 (5)	0.0213 (4)	0.000	−0.0012 (3)	0.000
C3	0.0212 (4)	0.0394 (5)	0.0166 (4)	0.000	0.0074 (3)	0.000
O3	0.0155 (3)	0.1026 (7)	0.0165 (3)	0.000	−0.0008 (3)	0.000

Geometric parameters (Å, º)

O1—C1	1.2397 (12)	N3—C2	1.2999 (12)
O2—C2	1.3239 (13)	C4—H4A	0.9800
O2—C3	1.4540 (13)	C4—H4B	0.9800
N1—C2	1.3679 (12)	C4—H4C	0.9800
N1—C1	1.3746 (13)	C3—H3D	0.9800
N1—C4	1.4552 (14)	C3—H3E	0.9800
N2—C1	1.3492 (13)	C3—H3C	0.9800
N2—N3	1.4003 (12)	O3—H3A	0.867 (6)
N2—H2	0.901 (7)	O3—H3B	0.859 (6)
C2—O2—C3	114.32 (7)	N1—C4—H4A	109.5
C2—N1—C1	106.92 (8)	N1—C4—H4B	109.5
C2—N1—C4	127.68 (8)	H4A—C4—H4B	109.5
C1—N1—C4	125.41 (7)	N1—C4—H4C	109.5
C1—N2—N3	112.77 (7)	H4A—C4—H4C	109.5
C1—N2—H2	128.1 (6)	H4B—C4—H4C	109.5
N3—N2—H2	119.1 (6)	O2—C3—H3D	109.5
C2—N3—N2	102.47 (7)	O2—C3—H3E	109.5
O1—C1—N2	128.74 (8)	H3D—C3—H3E	109.5
O1—C1—N1	126.94 (8)	O2—C3—H3C	109.5
N2—C1—N1	104.32 (7)	H3D—C3—H3C	109.5
N3—C2—O2	127.74 (8)	H3E—C3—H3C	109.5
N3—C2—N1	113.52 (9)	H3A—O3—H3B	113.2 (8)
O2—C2—N1	118.75 (8)		
C1—N2—N3—C2	0.0	N2—N3—C2—N1	0.0
N3—N2—C1—O1	180.0	C3—O2—C2—N3	0.0
N3—N2—C1—N1	0.0	C3—O2—C2—N1	180.0
C2—N1—C1—O1	180.0	C1—N1—C2—N3	0.0
C4—N1—C1—O1	0.0	C4—N1—C2—N3	180.0
C2—N1—C1—N2	0.0	C1—N1—C2—O2	180.0
C4—N1—C1—N2	180.0	C4—N1—C2—O2	0.0
N2—N3—C2—O2	180.0		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3	0.90 (1)	1.85 (1)	2.7520 (15)	174 (1)

O3—H3*A*···O1^{i}	0.87 (1)	1.89 (1)	2.7518 (18)	174 (1)
O3—H3*B*···O1^{ii}	0.86 (1)	1.94 (1)	2.8024 (18)	179 (1)

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