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3-Methyl-1-phenyl-4,5-dihydro-1,2,4-triazol-5(1*H*)-one

Heng Jing, Hong-Jun Zhu,* Shan Liu, Wei Zheng and Ya-Ming Wu

Department of Applied Chemistry, College of Science, Nanjing University of Technology, Nanjing 210009, People's Republic of China

Correspondence e-mail: zhuhj@njut.edu.cn

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Key indicators

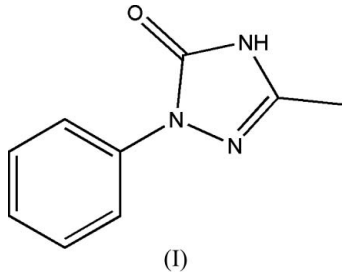
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.146
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the molecule of the title compound, $C_9H_9N_3O$, the dihedral angle between the planar rings is 7.49 (3)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into dimers, which may be effective in the stabilization of the crystal structure.

Comment

1-Aryl-1,2,4-traizolin-5-one derivatives have herbicidal activity (Kajioka & Kurono, 1982) and can be used as herbicides in soybeans (Keifer & Tymonko, 1990) and to prevent or destroy undesired plant growth (Theodoridis, 1991). The title compound, (I), is an important intermediate in the synthesis of triazolinone. We report here its crystal structure.



(I)

In the molecule of the title compound, (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Intramolecular C—H···N and C—H···O [H4*A*···N1 = 2.44 Å, C4···N1 = 2.781 (3) Å and C4—H4*A*···N1 = 101°; H6*A*···O = 2.32 Å, C6···O = 2.955 (3) Å and C6—H6*A*···O = 125°] hydrogen bonds lead to the formation of five- and six-membered rings (Fig. 1). Rings *A* (C1–C6) and *B* (N1–N3/C7/C8) are, of course, planar and the dihedral angle between them is 7.49 (3)°.

As can be seen from the packing diagram (Fig. 2), intermolecular N—H···O [H3*A*···O^{i} = 1.97 Å, N3···O^{i} = 2.815 (3) Å and N3—H3*A*···O^{i} = 169°; symmetry code: (i) $-x$, $1-y$, $1-z$] hydrogen bonds link the molecules into dimers, which may be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

For the preparation of the title compound, 3-methyl-1-phenyl-1,2,4-triazolidin-5-one (20 g, 0.17 mol) was dissolved in aqueous sodium hypochlorite (350 ml, 5%) and stirred at room temperature. After 15 min, the mixture became cloudy, the temperature had risen to 303 K, bubbling took place and an oily solid formed on the surface. Sodium hydroxide (7 g, 0.17 mol) was added to the mixture, which

was then extracted with diethyl ether, after which the aqueous phase was neutralized with HCl. The mixture was collected by filtration and recrystallized from ethyl acetate/heptane (40:15) by slow evaporation (yield 13.5 g; m.p. 434–435 K).

Crystal data

$C_9H_9N_3O$	$Z = 8$
$M_r = 175.19$	$D_x = 1.338$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation
$a = 13.812$ (3) Å	$\mu = 0.09$ mm^{-1}
$b = 7.5400$ (15) Å	$T = 294$ (2) K
$c = 16.708$ (3) Å	Block, colorless
$V = 1740.0$ (6) Å^3	0.30 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1707 independent reflections
$\omega/2\theta$ scans	1014 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North *et al.*, 1968)	$\theta_{max} = 26.0°$
$T_{min} = 0.973$, $T_{max} = 0.982$	3 standard reflections
1707 measured reflections	every 200 reflections
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.34P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.146$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
1707 reflections	Extinction correction: *SHELXL97*
119 parameters	Extinction coefficient: 0.023 (2)
H-atom parameters constrained	

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N)$, where $x = 1.5$ for methyl and $x = 1.2$ for all other H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

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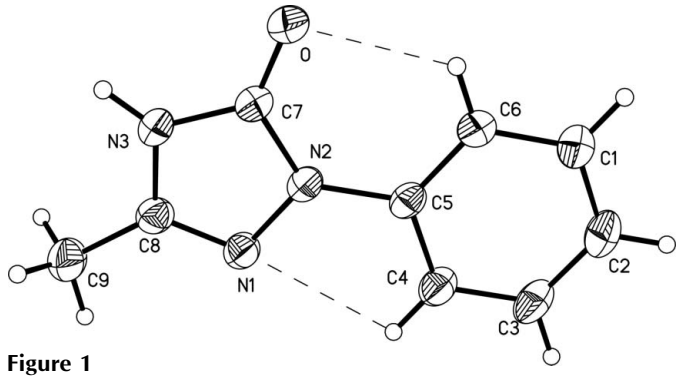

Figure 1
The molecular structure, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines.

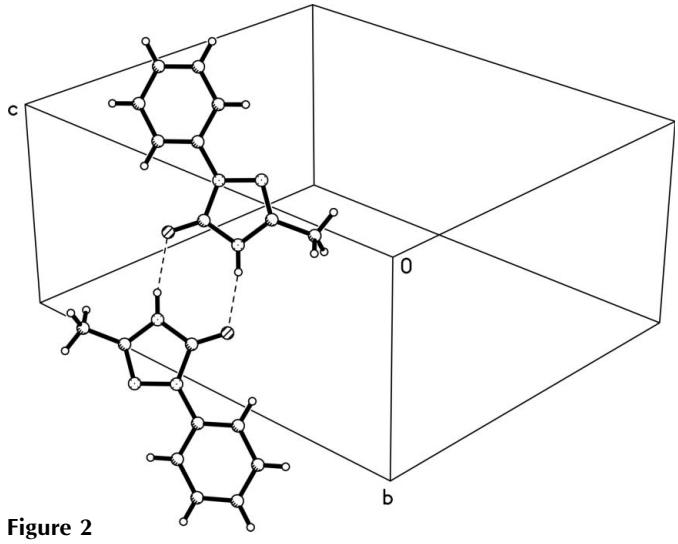

Figure 2
A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

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