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3-Methyl-3-pyrazolin-5-one

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Key indicators

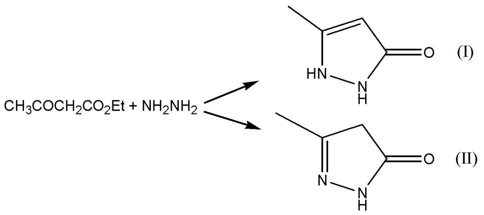
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.115
Data-to-parameter ratio = 7.0

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

The title compound, $C_4H_6N_2O$, is an intermediate in the further synthesis of theterocyclic compounds. The present crystal structure is orthorhombic (space group *Fdd*2), and a monoclinic form ($P2_1/a$) has been reported by De Camp & Stewart [(1971). *Acta Cryst.* B**27**, 1227–1232].

Comment

In the course of our research on nitrogen heterocycles, we recently needed to prepare several pyrazolin-5-one compounds. Synthetic considerations made 3-methyl-3-pyrazolin-5-one, (I), and 3-methyl-2-pyrazolin-5-one, (II), the first logical choices. Both (I) and (II) can be readily obtained by the cyclization reaction of ethyl acetoacetate with hydrazine in water at different pH values, these two isomers are interconvertible. To conduct research on their reaction mechanisms and conformational effects on reactivity, it was necessary for us to complete their X-ray structure analyses.



Unexpectedly, we found polymorphism. In fact, a literature search for (I) showed that its crystal structure was reported early in 1971 (De Camp & Stewart, 1971). The reported structure belongs to the monoclinic space group $P2_1/a$ [a = 10.520 (3), b = 6.499 (2), c = 8.052 (1) Å, β = 114.45 (1)°, Z = 4, and the solvent used for crystallization was dimethylformamide]. We report here the orthorhombic form of the title compound, (I).

In (I), the non-H atoms of the molecule are coplanar, with a mean deviation of 0.022 Å. The bond lengths and angles are in very good agreement with those found in the monoclinic polymorph and the values lie in expected ranges (Allen *et al.*, 1987). There are intermolecular N—H···O hydrogen bonds, forming an infinite three-dimensional network (Fig. 2 and Table 2).

Experimental

The preparation of the title compound (I) was carried out by the reaction of ethyl acetoacetate with hydrazine (molar ratio 1:1) in aqueous media at room temperature, for no more than 20 min. Single crystals of (I) were obtained by cooling of the hot solution of the crude product in ethanol (95%) very slowly over 1–2 weeks (yield: 93%, mp 488 K). ^{1}HNMR (300 MHz, dmso): δ 10.30 (*s*, 2H, 2NH), δ 5.20 (*s*, 1H, CH=), δ 2.06 (*s*, 3H, CH_3).

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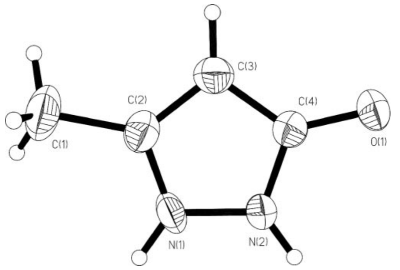

Figure 1
Molecular structure of (I), showing 40% probability displacement ellipsoids.

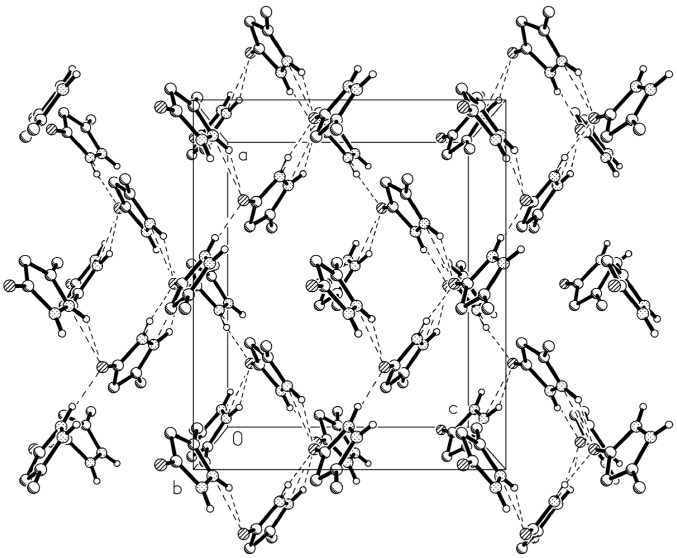

Figure 2
Packing diagram of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Crystal data

$C_4H_6N_2O$
$M_r = 98.11$
Orthorhombic, *Fdd*2
$a = 11.722$ (2) Å
$b = 16.945$ (3) Å
$c = 9.898$ (2) Å
$V = 1966.0$ (7) Å^3
$Z = 16$
$D_x = 1.326$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 867 reflections
$\theta = 5.9$–$44.4°$
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker Smart CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.557$, $T_{\max} = 0.981$
1933 measured reflections
460 independent reflections

439 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 25.0°$
$h = -13 \rightarrow 13$
$k = -20 \rightarrow 9$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.115$
$S = 1.16$
460 reflections
66 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 1.0082P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.011$
$\Delta\rho_{\max} = 0.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0020 (8)

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.275 (3)	N2—C4	1.324 (4)
N1—C2	1.315 (5)	C2—C3	1.368 (5)
N1—N2	1.365 (4)	C3—C4	1.403 (5)
C2—N1—N2	108.7 (3)	N1—C2—C1	120.7 (4)
C4—N2—N1	109.4 (3)	C3—C2—C1	130.7 (4)
N1—C2—C3	108.5 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1$^{\rm i}$	0.86	1.93	2.694 (4)	147.9
N2—H2···O1$^{\rm ii}$	0.86	1.81	2.654 (4)	168.6

Symmetry codes: (i) $\frac{7}{4} - x, y - \frac{1}{4}, \frac{1}{4} + z$; (ii) $x - \frac{1}{4}, \frac{3}{4} - y, \frac{1}{4} + z$.

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged – the Flack (1983) parameter was −1 (3). H atoms were placed in their idealized positions (N—H = 0.86, C—H = 0.93 and 0.96 Å; $U_{\rm iso} = 1.2U_{\rm eq}$ of the parent atom) and allowed to ride on the respective parent atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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