

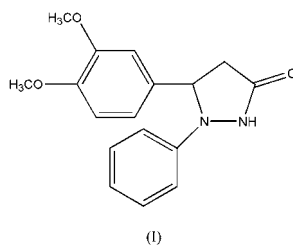
Hong-Jun Zhu,* Ji Ma,
Chang-Mei Wei and Jin-Tang
WangDepartment of Applied Chemistry, College of
Science, Nanjing University of Technology,
Xinmofan Road No. 5 Nanjing, Nanjing
210009, People's Republic of China

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

Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.065
 wR factor = 0.275
Data-to-parameter ratio = 13.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(±)-5-(3,4-Dimethoxyphenyl)-1-phenyl-
pyrazolidin-3-one**The title compound, $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_3$, was synthesized by the
reaction of ethyl 3-(3,4-dimethoxyphenyl)acrylate and phenyl-
hydrazine. There are intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-$
 $\text{H}\cdots\text{O}$ hydrogen bonds, and also $\text{C}-\text{H}\cdots\pi$ interactions.Received 5 January 2004
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Comment

Pyrazolidin-3-one derivatives are of great interest because of
their biological properties, such as antipyretic activity
(Menozzi *et al.*, 1990), lipoxygenase enzyme inhibition
(Brooks *et al.*, 1990) and cholecystokinin (CCK) receptor
antagonist activity (Greenwood *et al.*, 1995). We report here
the crystal structure of the title compound, (I).

The molecular structure of (I) is shown in Fig. 1, and the bond lengths and angles are given in Table 1. The pyrazolidine ring (N1/N2/C3/C2/C1) adopts a twist form (Low *et al.*, 2003). The dihedral angle between the C1/C2/C3 and C1/N1/N2/C3 planes is $18.3(4)^\circ$, while the dihedral angle between the two benzene rings is $87.4(1)^\circ$. In the crystal structure, molecules are linked by $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2), forming a three-dimensional network. The $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds are formed between the C9—H and methoxy groups, and between the C17—H and C1=O1 groups of adjacent molecules (Fig. 2). There is also a $\text{C}-\text{H}\cdots\pi$ interaction in the crystal structure (Fig. 3), where the C2—H group acts as the hydrogen-bond donor and a phenyl ring (C4—C9) acts as the acceptor (Steiner *et al.*, 1995). The $\text{C}-\text{H}\cdots\pi$ distance seems to decrease as the acidity of the C—H atom increases (Takahashi *et al.*, 2000). Because of the

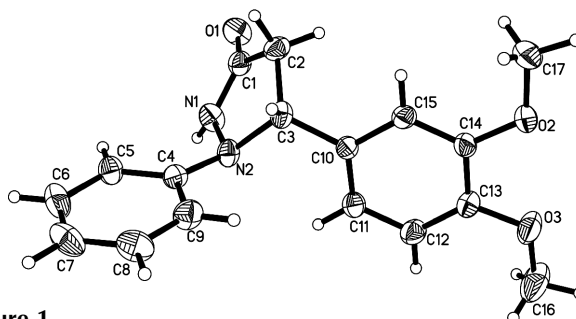


Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

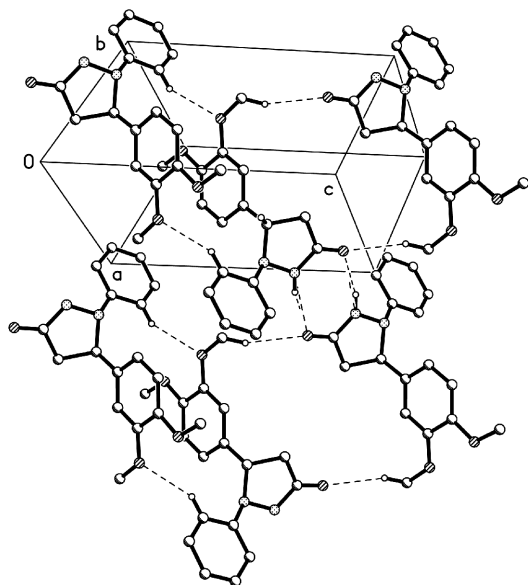


Figure 2
The crystal structure of (I). Dashed lines indicate C—H...O and N—H...O hydrogen bonds. H atoms not involved in the hydrogen bonding have been omitted for clarity.

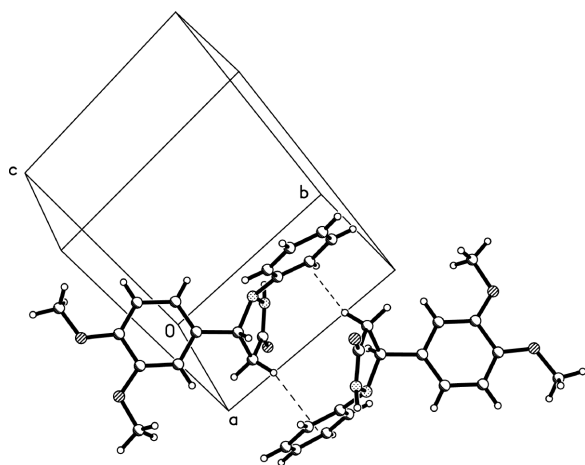


Figure 3
The C—H... π interactions in (I).

presence of the C=O group in the pyrazolidine ring, the acidity of the H atoms on C2 may be increased.

Experimental

To a solution of sodium (40 mmol) in anhydrous methanol (9 ml) was added ethanolamine (4 ml) and *n*-butanol (20 ml). The methanol was removed by distillation and ethyl 3-(3,4-dimethoxyphenyl)acrylate was added. The resulting mixture was refluxed for 1 h at 373 K, after which time phenylhydrazine (4 ml) was added. The reaction mixture was refluxed for a further 6 h, left to cool to room temperature, acidified with 36% acetic acid, allowed to stand, filtered, and the filter cake was crystallized from ethyl acetate to give pure compound (I) (m.p. 434–435 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis, IR (KBr, ν , cm^{-1}): 3300, 3061, 3030, 2900, 2860, 1689, 1595, 1514, 1252; ^1H NMR (CDCl_3 , δ , p.p.m.): 8.4(s, 1H), 6.9–7.3 (*m*, 9H), 4.88 (*d* \times *d*, 1H, $J = 3.6$ Hz and $J = 8.9$ Hz), 3.9 (*s*, 1H), 3.3 (*d* \times *d*, 1H,

$J = 8.9$ Hz and $J = 16.7$ Hz), 2.5 (*d* \times *d*, 1H, $J = 3.6$ Hz and $J = 16.7$ Hz); analysis calculated for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_3$: C 68.43, H 6.09, N 9.39%; found: C 68.30, H 6.08, N 9.36%.

Crystal data

$\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_3$	$Z = 2$
$M_r = 298.33$	$D_x = 1.280 \text{ Mg m}^{-3}$
Triclinic, $P1$	Mo $K\alpha$ radiation
$a = 9.011(2) \text{ \AA}$	Cell parameters from 25 reflections
$b = 9.403(2) \text{ \AA}$	$\theta = 10\text{--}13^\circ$
$c = 11.621(2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 66.49(3)^\circ$	$T = 293(2) \text{ K}$
$\beta = 67.67(3)^\circ$	Block, colourless
$\gamma = 62.05(3)^\circ$	$0.4 \times 0.3 \times 0.2 \text{ mm}$
$V = 774.6(4) \text{ \AA}^3$	

Data collection

Nonius CAD-4/PC diffractometer	$R_{\text{int}} = 0.070$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 10$
$T_{\text{min}} = 0.777$, $T_{\text{max}} = 0.982$	$k = -9 \rightarrow 11$
2902 measured reflections	$l = -12 \rightarrow 13$
2715 independent reflections	3 standard reflections
2090 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: 0.9%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.16P)^2 + 0.4P]$
$R[F^2 > 2\sigma(F^2)] = 0.065$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.275$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.18$	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
2715 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
204 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.058 (17)

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—C1	1.236 (4)	N1—N2	1.417 (4)
O2—C14	1.351 (4)	N2—C4	1.411 (4)
O2—C17	1.419 (5)	N2—C3	1.492 (4)
O3—C13	1.361 (4)	C1—C2	1.496 (5)
O3—C16	1.388 (6)	C2—C3	1.533 (5)
N1—C1	1.324 (5)	C3—C10	1.513 (4)
C14—O2—C17	117.7 (3)	O1—C1—N1	125.9 (3)
C13—O3—C16	118.4 (3)	N1—C1—C2	107.5 (3)
C1—N1—N2	115.0 (3)	C1—C2—C3	104.3 (3)
N1—N2—C3	105.6 (2)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1...O1 ⁱ	0.97 (5)	1.90 (6)	2.807 (5)	155 (5)
C9—H9A...O2 ⁱⁱ	0.93	2.47	3.315 (5)	151
C17—H17C...O1 ⁱⁱⁱ	0.96	2.52	3.432 (5)	159
C2—H2A...Cg1 ^{iv}	0.97	2.66	3.576 (4)	157

Symmetry codes: (i) $-1 - x, 3 - y, -z$; (ii) $-x, 2 - y, 1 - z$; (iii) $-x, 2 - y, -z$; (iv) $-x, 3 - y, -z$. Cg1 is the centroid of the C4–C9 phenyl ring

The H atom on nitrogen was refined isotropically. The H atoms on C were positioned geometrically and distances to these H atoms were set at 0.93–0.98 \AA , with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 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: *SHELXS97*.

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