Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-(4-Methoxyphenyl)-1-phenylpyrazolidin-3-one

Hong Shi, Hong-Jun Zhu, Peng-Wei Yin, Jin-Tang Wang and Xiao-Lan Shi*

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.162
Data-to-parameter ratio = 14.4

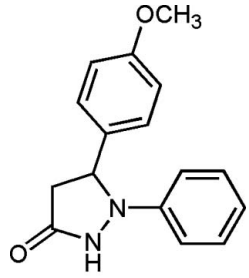
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 May 2005
Accepted 16 June 2005
Online 24 June 2005

The title compound, $C_{16}H_{16}N_2O_2$, was synthesized by the reaction of ethyl 3-(4-methylphenyl)acrylate and phenylhydrazine. There are intermolecular N—H···O hydrogen bonds and C—H···π interactions.

Comment

Pyrazolidin-3-one derivatives are effective medicines used in the treatment of inflammation and related disorders (Reddy & Bell, 2003), as liphoxygenase enzyme inhibitors (Brooks *et al.*, 1990), and in pesticides and herbicides (Tanaka *et al.*, 1999). We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. The pyrazolidine ring (N1/N2/C10/C9/C8) adopts a twisted conformation (Low *et al.*, 2003). The dihedral angle between the C8/C9/C10 and C8/N1/N2/C10 planes is 15.69 (19)°. In the crystal structure, molecules are linked by N—H···O hydrogen bonds and C—H···π interactions (Table 2), forming a three-

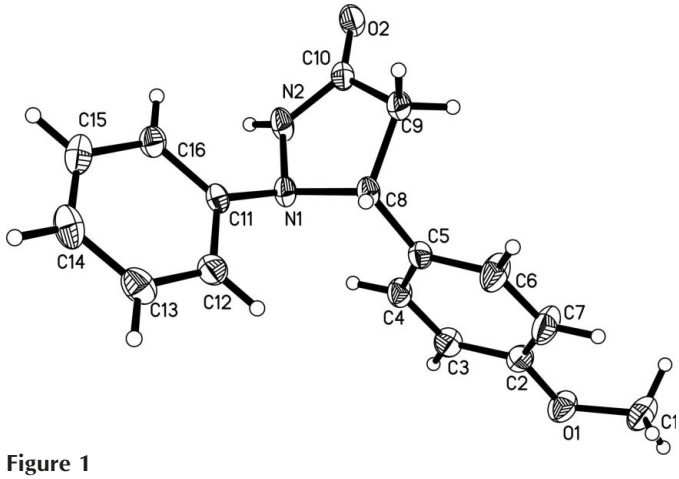


Figure 1
The molecule of (I). Displacement ellipsoids are drawn at the 30% probability level.

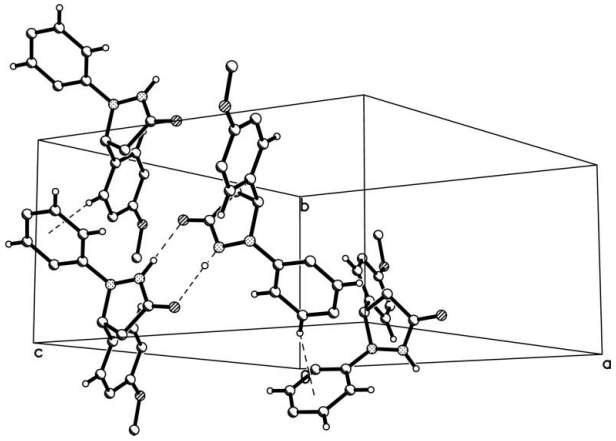


Figure 2
The crystal structure of (I). Dashed lines indicate N—H···O and C—H···π hydrogen bonds. H atoms not involved the hydrogen bonding are omitted for clarity.

dimensional network (Fig. 2). The phenyl ring acts as a double C—H···π hydrogen-bond acceptor, accepting one H···π bond from C6 of a methoxyphenyl group on one side and another H···π bond from C15 of a phenyl group on the other side. The dihedral angle between the two aromatic rings within the molecule is 79.5 (1)°. Owing to the presence of the carbonyl group in the pyrazolidine ring, the acidity of the H atom on C9 may be increased (Zhu *et al.*, 2004).

Experimental

To a solution of sodium (40 mmol) in anhydrous methanol (9 mol) were added ethanolamine (4 ml) and *n*-butanol (20 ml). The methanol was then removed by distillation and 3-(4-methylphenyl)-acrylic acid was added. The mixture was refluxed for 1 h at a temperature above 373 K, after which phenylhydrazine (4 ml) was added. The reactants were refluxed for a further 7 h, left to cool to room temperature, acidified with 36% acetic acid and then allowed to stand. After filtration, the filter cake was crystallized from ethyl acetate to give pure compound (I) (m.p. 435–437 K). The compound was crystallized twice by slow evaporation of an ethyl acetate solution and crystals suitable for X-ray diffraction analysis were obtained.

Crystal data

$C_{16}H_{16}N_2O_2$
M_r = 268.31
Monoclinic, $P2_1/n$
a = 13.151 (3) Å
b = 7.1660 (14) Å
c = 14.700 (3) Å
β = 97.53 (3)°
V = 1373.4 (5) Å^3
Z = 4
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.4 × 0.3 × 0.3 mm

Data collection

Entaf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
2792 measured reflections
2675 independent reflections
1884 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.0°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 8$
$l = -17 \rightarrow 17$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.162
S = 1.06
2675 reflections
186 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.091 (8)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.373 (2)	N1—C11	1.425 (2)
O1—C1	1.424 (3)	N1—C8	1.500 (2)
O2—C10	1.233 (2)	N2—C10	1.332 (3)
N1—N2	1.416 (2)		
C2—O1—C1	117.39 (17)	C10—N2—N1	114.88 (16)
N2—N1—C11	113.72 (15)	C10—N2—H2*A*	123.8 (16)
N2—N1—C8	105.61 (14)	N1—N2—H2*A*	119.2 (15)
C11—N1—C8	116.17 (15)		

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

*Cg*3 is the centroid of the six-membered ring C11–C16.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{i}	0.90 (3)	1.94 (3)	2.825 (2)	169 (2)
C6—H6*A*···*Cg*3^{ii}	0.93	2.65	3.558 (3)	167
C15—H15*A*···*Cg*3^{iii}	0.93	2.86	3.737 (2)	157

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, y-1, z$; (iii) $-x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

The H atom on N was located in a difference electron-density map and refined isotropically. H atoms on C atoms were positioned geometrically and distances to these H atoms were set at 0.93–0.98 Å, with U_{iso} values constrained to be 1.5U_{eq} of the parent 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, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

The authors thank the Centre of Test and Analysis, Nanjing University, for support.

References

Brooks, D. W., Basha, A., Gunn, B. P. & Bhatia, P. A. (1990). US Patent No. 4 970 210.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1996). *XCAD4*. University of Marburg, Germany.
Reddy, M. V. R. & Bell, S. C.(2003). US Patent No. WO 03 024 958.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tanaka, K., Adachi, H. & Yamaguchi, M. (1999). JP Patent No. 11 236 376.
Zhu, H.-J., Ma J., Wei, C.-M. & Wang, J.-T. (2004). *Acta Cryst.* E**60**, o411–o413.