Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-[(2-Hydroxy-3-methoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Chun-Hua Diao, Zhi Fan,* Ming Yu, Xin Chen, Zuo-Liang Jing and Qi-Liang Deng

College of Sciences, Tianjin University of Science and Technology, Tianjin 300222, People's Republic of China

Correspondence e-mail: zhifan@public.tpt.tj.cn

Received 12 July 2005
Accepted 31 August 2005
Online 17 September 2005

Key indicators

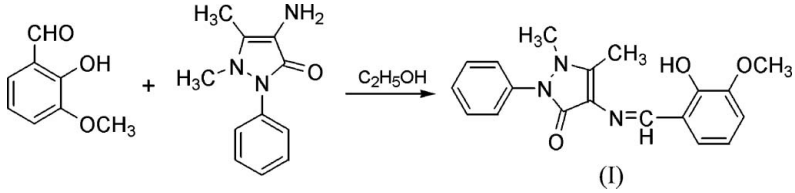
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.128
Data-to-parameter ratio = 14.8

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

The title compound, $C_{19}H_{19}N_3O_3$, was prepared by the reaction of 2-hydroxy-3-methoxybenzaldehyde (*o*-vanillin) and 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one. The crystal structure shows that a strong intramolecular O—H···N hydrogen bond stabilizes the conformation of the molecule, while intermolecular C—H···O hydrogen bonds give rise to a stable structure in the solid state.

Comment

The synthesis of new and designed crystal structures is part of a major strand of modern chemistry. One of the aims of crystal engineering is to establish control over the preparation of crystalline solid materials, so that their architecture and properties are predictable (Parashar *et al.*, 1988; Tynan *et al.*, 2005). In the present study, we report the synthesis and structure of the title compound, (I) (Fig. 1 and Table 1), which will provide useful information on its physical and chemical properties.



(I)

The central system, N1/N2/C7/O1/C9/C10/N3/C8, is planar, with an r.m.s. deviation for fitted atoms of 0.038 Å, and the dihedral angle with the phenyl ring (C1–C6) is 55.17 (5)°. The *o*-vanillin moiety (N3/C12–C19/O2/O3) is planar, with an r.m.s. deviation for fitted atoms of 0.021 Å. The dihedral angle between the central system and the *o*-vanillin moiety is 7.01 (6)°. Intramolecular O—H···N and C—H···O hydrogen bonds (Table 2) stabilize the conformation of the molecule. The molecules are associated *via* weak C—H···O intermolecular hydrogen bonds (Table 2) to form a supramolecular structure (Fig. 2).

Experimental

An anhydrous ethanol solution of 2-hydroxy-3-methoxybenzaldehyde (*o*-vanillin, 1.52 g, 10 mmol) was added to an anhydrous ethanol solution of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (4-aminoantipyrine, 2.03 g, 10 mmol) and the mixture was stirred at 350 K for 5 h under nitrogen, whereupon a yellow precipitate appeared. The product was isolated and recrystallized from ethanol, and then dried *in vacuo* to give pure (I) in 89% yield. Bright yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{19}H_{19}N_3O_3$
M_r = 337.37
Monoclinic, $C2/c$
a = 27.920 (6) Å
b = 7.5547 (15) Å
c = 16.712 (3) Å
β = 105.753 (4)°
V = 3392.6 (12) Å^3
Z = 8
D_x = 1.321 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1997 reflections
θ = 2.8–24.2°
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, yellow
0.26 × 0.24 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.970, T_{max} = 0.986
9165 measured reflections
3449 independent reflections
2024 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 26.3°
$h = -34 \rightarrow 32$
$k = -8 \rightarrow 9$
$l = -20 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.00
3449 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.5212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.232 (2)	N1—C1	1.426 (2)
O2—C18	1.355 (2)	N2—C9	1.374 (2)
O3—C17	1.375 (2)	N2—C11	1.472 (2)
O3—C19	1.423 (2)	N3—C12	1.293 (2)
N1—C7	1.408 (2)	N3—C8	1.392 (2)
N1—N2	1.420 (2)		
C7—N1—N2	108.98 (14)	C9—N2—C11	122.52 (17)
C7—N1—C1	121.24 (15)	N1—N2—C11	114.45 (15)
N2—N1—C1	118.27 (15)	C12—N3—C8	122.63 (16)
C9—N2—N1	106.23 (14)	N3—C12—C13	120.94 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N3	0.99 (3)	1.66 (3)	2.585 (2)	153 (2)
C12—H12···O1	0.93	2.41	3.069 (2)	128
C10—H10B···O1^{i}	0.96	2.40	3.212 (3)	143
C10—H10A···O3^{ii}	0.96	2.60	3.468 (3)	151

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

The H atom of the hydroxy group was found in a difference map and refined with free coordinates and isotropic U parameter. Other H atoms were included in calculated positions and refined using a riding model approximation. Constrained C—H bond lengths and U_{iso} parameters were 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH, and 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3.

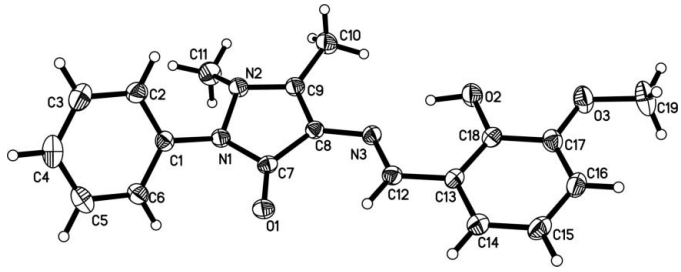


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

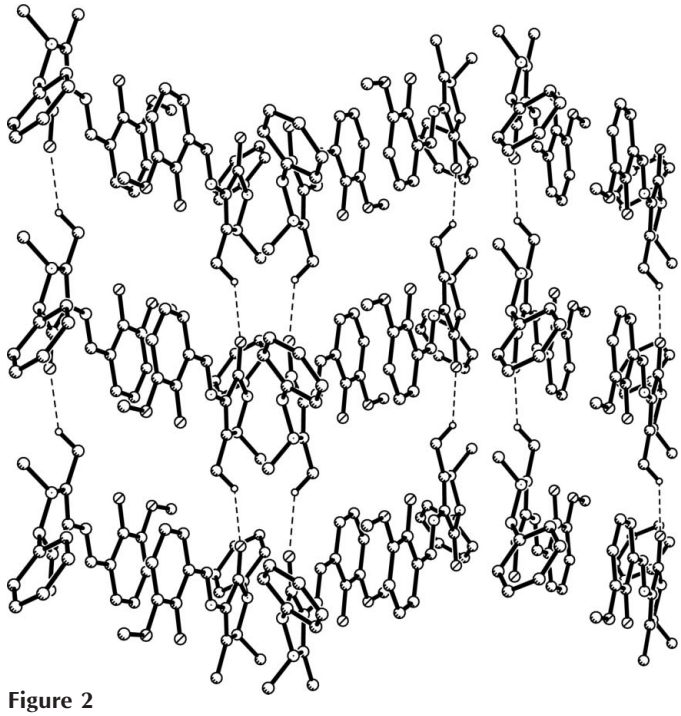


Figure 2
Intermolecular hydrogen-bonding interactions (dashed lines) in the crystal structure of (I). H atoms have been omitted.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1999). *SADABS*, *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Parashar, R. K., Sharma, R. C., Kumar, A. & Mohan, G. (1988). *Inorg. Chim. Acta*, **151**, 201–208.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tynan, E., Jensen, P., Lees, A. C., Moubaraki, B., Murray, K. S. & Kruger, P. E. (2005). *CrystEngComm*, **7**, 90–95.