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(*E*)-4-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yliminomethyl)-2-methoxyphenyl benzenesulfonate

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In the title compound, $C_{25}H_{23}N_3O_5S$, the vanillin group makes dihedral angles of 1.42 (11), 63.76 (10) and 51.00 (10)°, respectively, with the pyrazolone ring, the phenyl ring attached to the sulfonyl group, and the phenyl ring attached to the pyrazolone ring. The crystal packing is governed by C—H···O hydrogen bonds and π–π interactions.

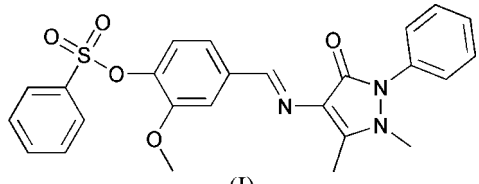
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.130
Data-to-parameter ratio = 13.4

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

Comment

Schiff-base ligands have received a good deal of attention in biology and chemistry (Kahwa *et al.*, 1986). Among the large number of compounds, 4-amino-1,5-dimethyl-2-phenyl-pyrazol-3-one forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some of them, such as 4-[(2-hydroxy-3-methoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Diao *et al.*, 2005) and (*E*)-4-[4-(4-chlorobenzyloxy)-3-ethoxybenzylideneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Zhang *et al.*, 2006) have been reported. We report here the synthesis and structure of the title compound, (I).



(I)

In the title molecule (Fig. 1), bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The pyrazolone ring (C16–C18/N1–N3/O5) is almost planar, with an r.m.s. deviation for fitted atoms of 0.0413 Å. It makes a dihedral angle of 52.25 (10)° with the attached phenyl ring (C20–C25). The vanillin group (C7–C12/C14/O3/O4) is nearly planar, with an r.m.s. deviation for fitted atoms of 0.0427 Å. This group makes dihedral angles of 1.42 (11), 63.76 (10) and 51.00 (10)°, respectively, with the pyrazolone ring (C16–C18/N1–N3/O5), the C1–C6 and C20–C25 phenyl rings.

The crystal packing is stabilized by weak non-classical intermolecular C8—H8···O5^{i} hydrogen bonds (Table 1) that form centrosymmetric dimers (Fig. 2), together with intermolecular C15—H15*C*···O5^{ii} and C13—H13*B*···O1^{ii} hydrogen bonds. In addition, π–π stacking interaction is observed between the pyrazolone ring and the C7–C12 phenyl ring of the molecule at the symmetry position (1 − x, 1 − y, 1 − z); the centroid–centroid distance between the two rings is 3.557 (2) Å.

Experimental

An anhydrous ethanol solution (50 ml) of 4-formyl-2-methoxyphenyl benzenesulfonate (2.92 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture stirred at 350 K for 3 h under N_2, giving a yellow precipitate. The product was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give pure compound (I) in 87% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{25}H_{23}N_3O_5S$
M_r = 477.52
Triclinic, $P\overline{1}$
a = 7.306 (7) Å
b = 12.977 (12) Å
c = 13.162 (12) Å
α = 89.272 (15)°
β = 77.437 (16)°
γ = 78.286 (14)°
V = 1192.0 (19) Å^3
Z = 2
D_x = 1.331 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 294 (2) K
Block, yellow
0.38 × 0.34 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.918, T_{max} = 0.969
6073 measured reflections
4164 independent reflections
2601 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.130
S = 1.05
4164 reflections
310 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.2214P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.039 (3)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O5^{i}	0.93	2.48	3.270 (4)	143
C15—H15C···O5^{ii}	0.96	2.48	3.434 (4)	171
C13—H13B···O1^{ii}	0.96	2.54	3.388 (5)	147

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

The H atoms were included in calculated positions and refined using a riding model approximation: C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH; C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3.

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

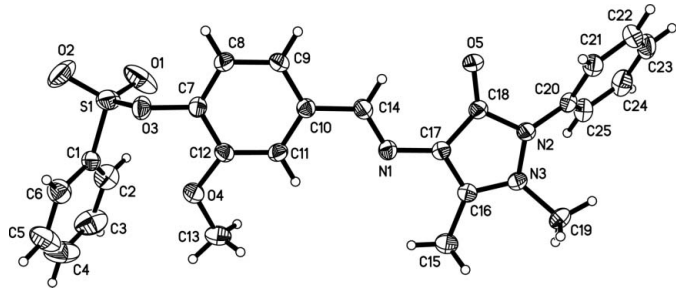


Figure 1
The molecular structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

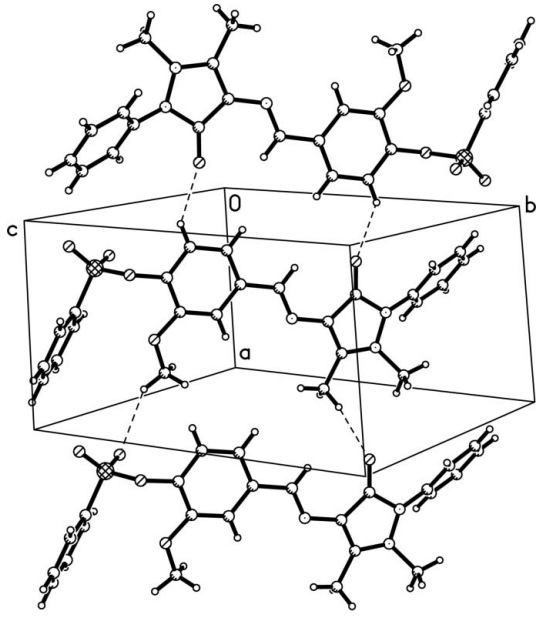


Figure 2
Packing diagram for (I) with hydrogen bonds drawn as dashed lines.

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