(E)-4-Bromo-2-[(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino)methyl]-6-methoxyphenyl 4-methylbenzenesulfonate N,N-dimethylformamide solvate

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Key indicators

Single-crystal X-ray study
$T = 294$ K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.046
wR factor = 0.133
Data-to-parameter ratio = 12.5

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

In the title compound, $C_{26}H_{24}BrN_3O_5S \cdot C_3H_7NO$, the substituted o-vanillin group makes dihedral angles of 24.91 (6), 17.63 (6) and 70.12 (12)°, respectively, with the pyrazolone ring, the methyl-substituted benzene ring and the phenyl ring. The crystal packing is stabilized by C—H···O hydrogen bonds, which form inversion-related dimers.

Comment

Schiff base ligands have played an important role in the development of coordination chemistry since the late 19th century. Consequently, a large number of these species are reported to be superior reagents for biological, pharmacological, clinical and analytical applications (Santos et al., 2001; Wang et al., 2005; Yang et al., 2005). As part of an investigation of the crystal structures of such compounds, which will provide useful information on the coordination properties of Schiff bases functioning as ligands, we report here the synthesis and crystal structure of the title compound, (I).

(I)
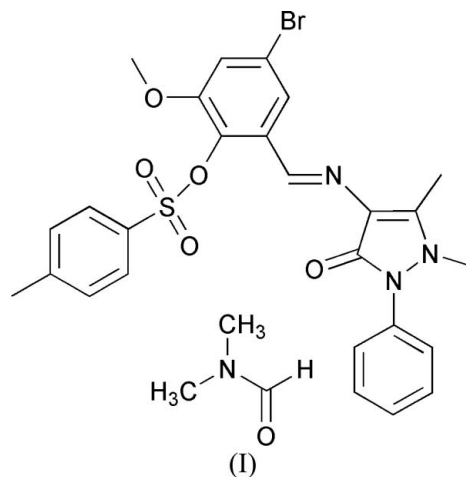
In (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen et al., 1987). The pyrazolone ring (C16/C17/C20/N1/N2/N3/O5) is almost planar, with an r.m.s. deviation for fitted atoms of 0.0295 Å. It makes a dihedral angle of 52.75 (14)° with the attached phenyl ring (C21–C26). The substituted o-vanillin group (C8–C13/C15/O3/O4) is nearly planar, with an r.m.s. deviation for fitted atoms of 0.0197 Å. This group makes dihedral angles of 24.91 (6), 17.63 (6) and 70.12 (12)°, respectively, with the the pyrazolone ring (C16/C17/C20/N1/N2/N3/O5), the C1–C6 benzene ring and the C21–C26 phenyl ring.

The packing is stabilized by weak non-classical intermolecular C—HC···O hydrogen bonds, which form centro-
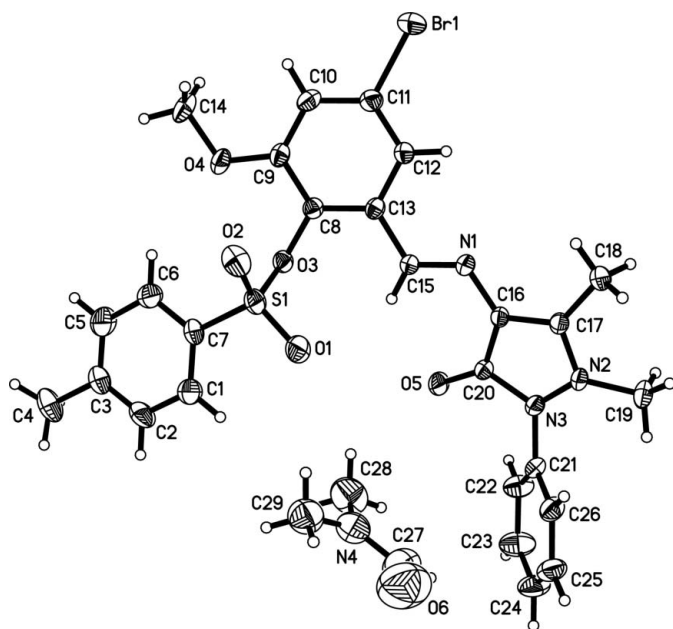
organic papers

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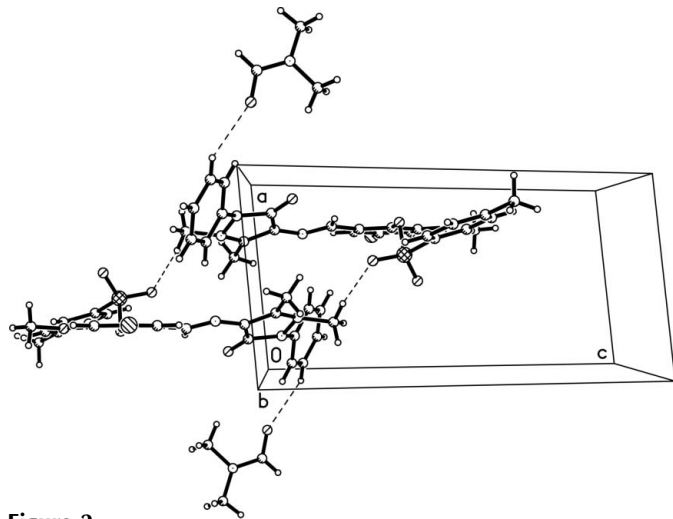

Figure 2
A partial packing diagram for (I), with hydrogen bonds drawn as dashed lines.

symmetric dimers (Table 1 and Fig. 2). In addition, a weak non-classical intermolecular C23—H23···O6 hydrogen bond links the main molecule and the solvent molecule.

Experimental

An anhydrous ethanol solution (50 ml) of 4-bromo-2-formyl-6-methoxyphenyl 4-methylbenzenesulfonate (3.85 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 5 h under nitrogen. The resulting yellow precipitate was isolated, recrystallized from acetonitrile and then dried in a vacuum to give the pure compound in 81% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a dimethylformamide solution.

Crystal data

$C_{26}H_{24}BrN_3O_5S \cdot C_3H_7NO$	$V = 1468.9$ (4) Å^3
$M_r = 643.55$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.455$ Mg m^{-3}
$a = 7.4537$ (11) Å	Mo $K\alpha$ radiation
$b = 13.5585$ (19) Å	$\mu = 1.52$ mm^{-1}
$c = 15.509$ (2) Å	$T = 294$ (2) K
$\alpha = 109.269$ (2)°	Block, yellow
$\beta = 93.852$ (2)°	$0.28 \times 0.26 \times 0.20$ mm
$\gamma = 94.085$ (3)°	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	7501 measured reflections
φ and ω scans	5130 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3305 reflections with $I > 2\sigma(I)$
$T_{min} = 0.621$, $T_{max} = 0.738$	$R_{int} = 0.021$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$+ 0.6477P]$
$wR(F^2) = 0.134$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
5130 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
411 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1	0.93	2.52	2.915 (5)	106
C15—H15···O5	0.93	2.38	3.031 (5)	127
C23—H23···O6^i	0.93	2.56	3.286 (13)	136
C19—H19C···O1^{ii}	0.96	2.52	3.475 (5)	171

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

The solvent molecule is disordered over two positions, with occupancy factors of 0.518 (9) and 0.482 (9). The C=O, N—Csp^3 and N—Csp^2 distances involving the disordered atoms were restrained to 1.22 (1), 1.45 (1) and 1.33 (1) Å, respectively, and the U^{ij} components were restrained to an approximate isotropic behaviour. H atoms were included in calculated positions and refined using a riding-model approximation, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic, and C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms.

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*.

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