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1,5-Dimethyl-4-[2-(methylsulfanyl)benzylideneamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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In the title compound, $C_{19}H_{19}N_3OS$, a Schiff base, there are three different intramolecular hydrogen bonds (C—H···O and C—H···S), which stabilize the structure; this is further extended into one-dimensional chains parallel to the *a* axis *via* intermolecular hydrogen bonds.

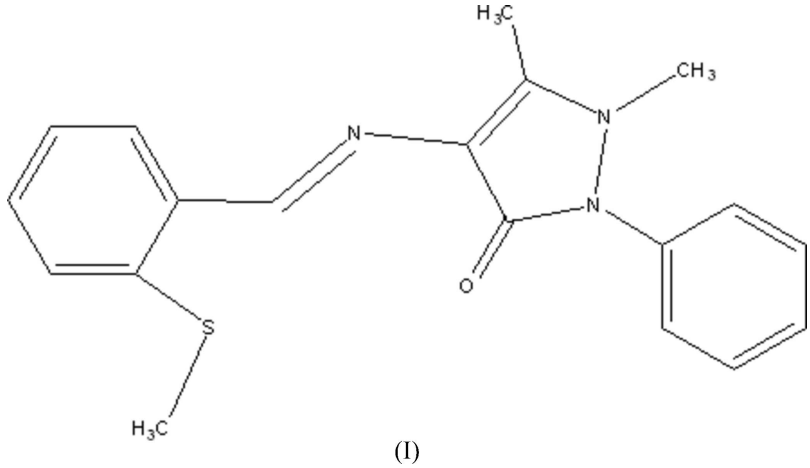
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.050 Å
R factor = 0.040
wR factor = 0.111
Data-to-parameter ratio = 20.6

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

Comment

Schiff base ligands and their metal complexes are interesting. For example, Schiff bases have exhibited solvatochromicity, and are suitable NLO (non-linear optical) materials (Alemi & Shaabani, 2000). They are also useful in asymmetric oxidation of methyl phenyl sulfide and enantioselective epoxidation (Kim & Shin, 1999). In this paper, we report the synthesis and crystal structure of the title Schiff base, (I).



(I)

The molecular structure of (I) (Fig. 1) contains three intramolecular hydrogen bonds (C8—H8···O1 and C8—H8···S1; Table 2). The C8—N1 distance of 1.255 (4) Å is indicative of a normal C=N bond. The other C—N, C—S and C—C distances show no remarkable features.

In addition, intermolecular C—H···O hydrogen bonds (Table 2) between the molecules lead to one-dimensional chains parallel to the *a* axis (Fig. 2).

Experimental

Under nitrogen, a mixture of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (2.03 g, 10 mmol), Na_2SO_4 (3.0 g) and 2-methylthiobenzaldehyde (1.52 g, 10 mmol) in absolute ethanol (20 ml) was refluxed for about 12 h, yielding a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was redissolved in CH_2Cl_2 (100 ml) and washed with water (2 × 10 ml) and brine (10 ml). After drying over Na_2SO_4, the

solvent was removed under vacuum, and a yellow solid was isolated in 92% yield (3.1 g). Yellow single crystals of the Schiff base (I) suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol (4:1) by slow evaporation of the solvents at room temperature over a period of about a week.

Crystal data

$C_{19}H_{19}N_3OS$
M_r = 337.43
Orthorhombic, $P2_12_12_1$
a = 6.9535 (9) Å
b = 12.9078 (17) Å
c = 19.800 (3) Å
V = 1777.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 273 (2) K
0.43 × 0.35 × 0.34 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.922, T_{max} = 0.938
13405 measured reflections
4287 independent reflections
2166 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 0.91
4287 reflections
208 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Absolute structure: Flack (1983), 1759 Friedel pairs
Flack parameter: −0.02 (9)

Table 1
Selected geometric parameters (Å, °).

C18—S1	1.78 (2)	C12—N1	1.26 (4)
C11—O1	1.24 (4)	C19—S1	1.73 (6)
C11—N2	1.40 (4)	N2—N3	1.40 (4)
N1—C10—C11	130 (3)	C19—S1—C18	105 (2)
C12—N1—C10	121 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···S1	0.93	2.62	3.03 (4)	107
C12—H12···O1	0.93	2.41	3.07 (4)	128
C7—H7A···O1^{i}	0.96	2.55	3.37 (5)	142
C8—H8C···O1^{i}	0.96	2.54	3.49 (6)	170

Symmetry code: (i) $x+1, y, z$.

All H atoms were placed in calculated positions [C—H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(aromatic C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

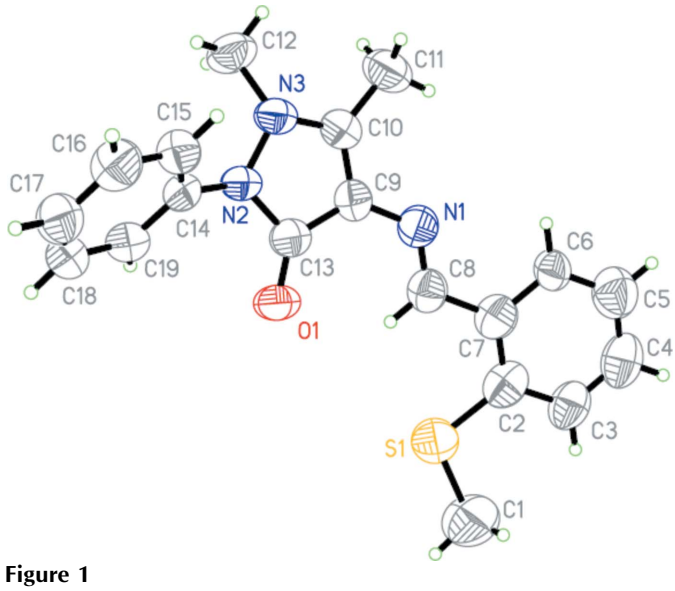


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Non-H atoms are shown as 50% probability displacement ellipsoids.

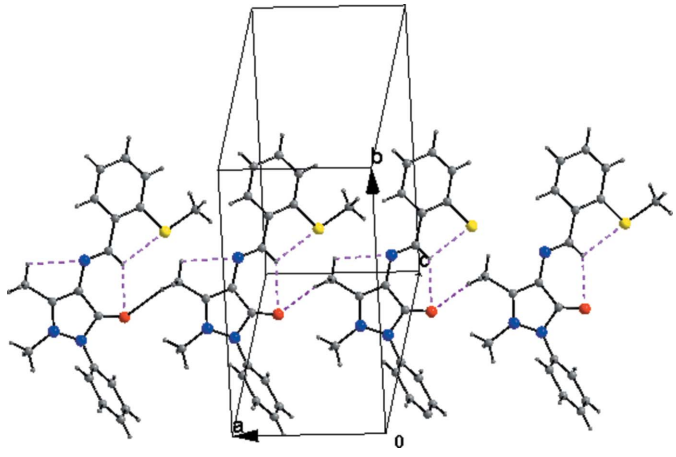


Figure 2
A chain of molecules linked by hydrogen bonds (dashed lines).

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References

Alemi, A. A. & Shaabani, B. (2000). *Acta Chim. Slov.*, **47**, 363–369.
Bruker (1998). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsis, USA.
Bruker (1999). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsis, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Kim, G. J. & Shin, J. H. (1999). *Catal. Lett.* **63**, 83–89.
Sheldrick, G. M. (1997). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.