Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1,5-Dimethyl-2-phenyl-4-(2-thienylmethyleneamino)-1*H*-pyrazol-3(2*H*)-one

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Received 10 December 2006
Accepted 15 December 2006

In the crystal structure of the title Schiff base compound, $C_{16}H_{15}N_3OS$, there are three C—H···O hydrogen bonds which stabilize the molecular and crystal structures.

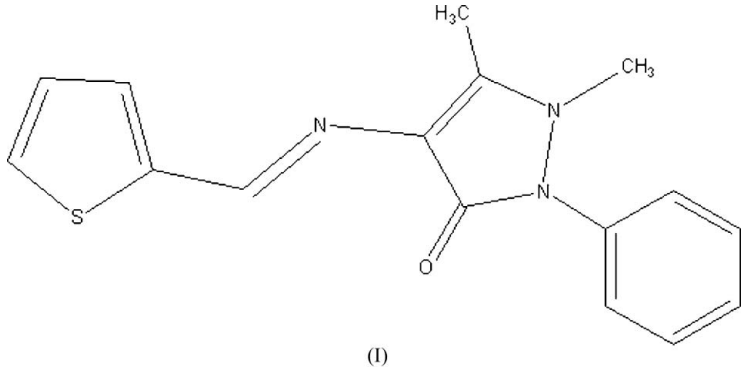
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.123
Data-to-parameter ratio = 13.7

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

Comment

Schiff base ligands have significant importance in chemistry, especially in the development of Schiff base complexes, because Schiff base ligands are potentially capable of forming stable complexes with metal ions (Johnson *et al.*, 1996; Alizadeh *et al.*, 1999). Schiff bases showing solvent-dependent UV/vis spectra (solvatochromicity) can be suitable NLO (nonlinear optical) active materials (Alemi *et al.*, 2000). They are also useful in the asymmetric oxidation of methyl phenyl sulfide and enantioselective (Kim & Shin, 1999). In this paper, we report the synthesis and crystal structure of the title compound, (I) (Fig. 1).



(I)

In the crystal structure of the title compound, there is one intramolecular C—H···O hydrogen bond (Table 1) which stabilizes the molecular structure, while two intermolecular C—H···O hydrogen bonds stabilize the crystal structure, forming a molecular tape running along the *c* axis (Fig. 2).

Experimental

A mixture of 2-thiophenecarboxaldehyde (1.14 g, 10 mmol), Na_2SO_4 (3.0 g) and 4-amino-2,3-dimethyl-1-phenyl-3-pyrazolin-5-one (2.72 g, 20 mmol) in absolute ethanol (20 ml) was refluxed under nitrogen for about 5 h, yielding a yellow precipitate. The product was filtered and washed with ethanol. The crude solid was dissolved in CH_2Cl_2 (100 ml) and washed with water (2 × 10 ml) and brine (10 ml), and dried over Na_2SO_4. The remaining solvent was removed under vacuum (yield 95%, 3.33 g). Colourless single crystals of (I) suitable for X-ray analysis were grown by slow evaporation of a CH_2Cl_2–absolute ethanol (4:1) solution at room temperature over a period of about a week.

Crystal data

$C_{16}H_{15}N_3OS$
M_r = 297.38
Monoclinic, $P2_1/c$
a = 8.727 (6) Å
b = 24.836 (18) Å
c = 6.902 (5) Å
β = 98.932 (13)°
V = 1477.7 (18) Å^3
Z = 4
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 293 (2) K
Block, colourless
0.36 × 0.30 × 0.25 mm

Data collection

Bruker APEX-II area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.925, T_{max} = 0.947
8923 measured reflections
2626 independent reflections
1380 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.123
S = 0.87
2626 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1	0.93	2.33	3.008 (4)	130
C12—H12C···O1^{i}	0.96	2.44	3.395 (4)	175
C14—H14···O1^{ii}	0.93	2.48	3.330 (4)	152

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

H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and refined as a riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1998); 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, 1998); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Natural Science Foundation of Zhejiang Province (No. M203052) and the Research Foundation of Lishui University (No. FC06002) for financial support.

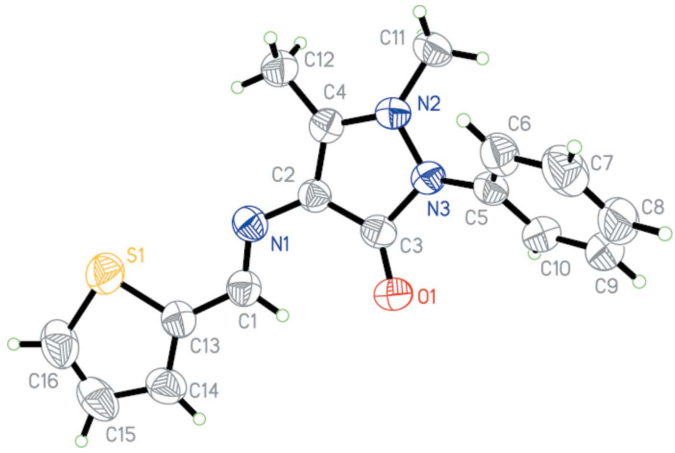


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Non-H atoms are drawn as 50% probability displacement ellipsoids.

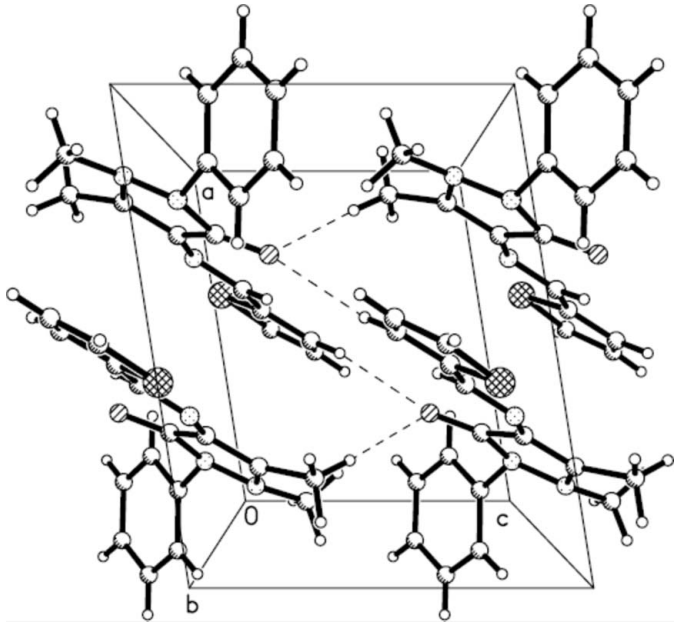


Figure 2
A packing diagram of (I), viewed down the *b* axis, showing the hydrogen-bonded molecular tape. Dashed lines indicate C—H···O hydrogen bonds.

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