Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-4-[(5-Chloro-2-thienyl)methyleneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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Received 31 December 2006
Accepted 18 January 2007

Intermolecular weak C—H···O hydrogen bonding helps to stabilize the crystal structure of the title Schiff base compound, $C_{16}H_{14}ClN_3OS$.

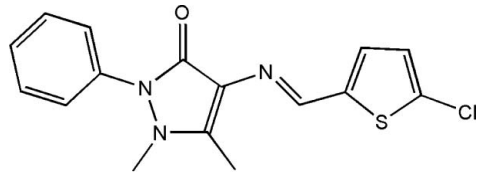
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.103
Data-to-parameter ratio = 14.0

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

Comment

Schiff bases and their complexes are widely used in chemical fields such as asymmetric oxidation (Kim & Shin, 1999) and non-linear optical materials (Alemi & Shaabani, 2000). As a part of an investigation of Schiff base compounds, the crystal structure of the title compound, (I), is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. The C5—N3 bond distance of 1.282 (2) Å shows C═N double-bond character. Intermolecular weak C—H···O hydrogen bonding (Table 1 and Fig. 2) helps to stabilize the crystal structure.

Experimental

Under nitrogen, a mixture of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (2.28 g, 10 mmol), Na_2SO_4 (3.0 g) and 5-chloro-2-thiophenecarboxaldehyde (1.56 g, 10 mmol) in absolute ethanol (20 ml) was refluxed for 12 h, yielding a yellow precipitate. The product was collected by vacuum filtration and then 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). After being dried over Na_2SO_4, the solvent was removed under vacuum, and a yellow solid was isolated in 92% yield (3.1 g). Single crystals of (I) were

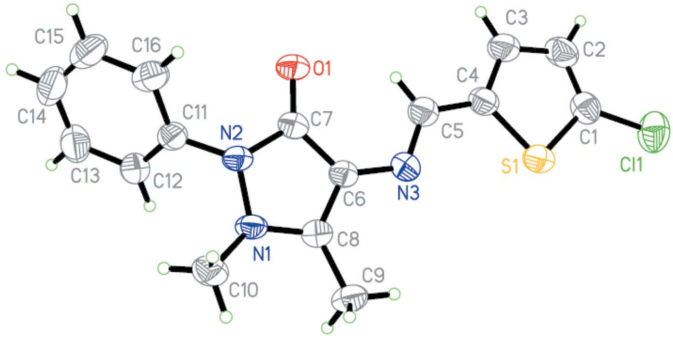


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

obtained from a CH_2Cl_2–ethanol solution (4:1) by slow evaporation of the solvent at room temperature.

Crystal data

$C_{16}H_{14}ClN_3OS$
M_r = 331.81
Monoclinic, $P2_1/n$
a = 9.1582 (5) Å
b = 7.3314 (4) Å
c = 24.1175 (13) Å
β = 100.746 (1)°
V = 1590.91 (15) Å^3
Z = 4
D_x = 1.385 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 296 (2) K
Block, colourless
0.50 × 0.35 × 0.28 mm

Data collection

Bruker APEX-II area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.814, T_{max} = 0.902
9409 measured reflections
2807 independent reflections
2286 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.103
S = 0.92
2807 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.0443P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.54	3.115 (2)	120
C5—H5···O1	0.93	2.25	2.969 (2)	133
C9—H9A···O1^{ii}	0.96	2.51	3.438 (2)	161

Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $x, y+1, z$.

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

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*.

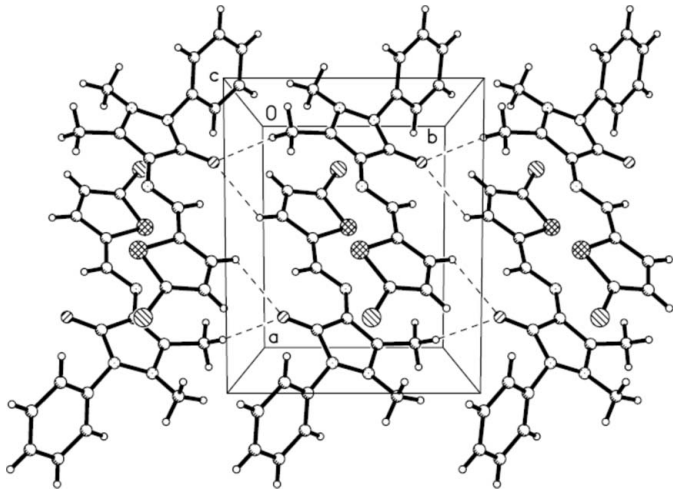


Figure 2
The packing of (I); dashed lines indicate hydrogen bonds.

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.

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