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4-(3-Bromothien-2-yl)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.088
Data-to-parameter ratio = 14.4

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

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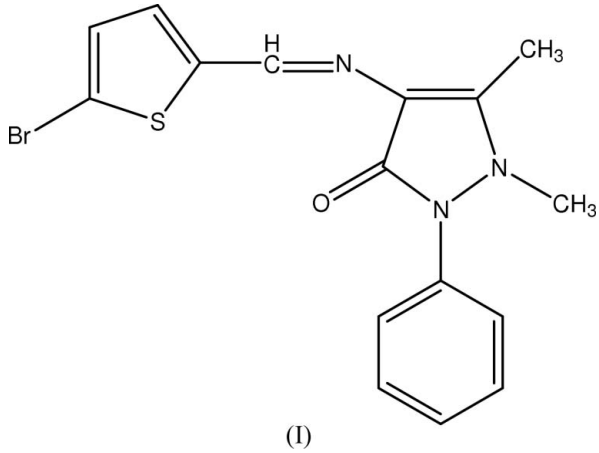
In the title molecule, $C_{15}H_{14}BrN_3OS$, the pyrazole ring is essentially planar and forms dihedral angles of 5.9 (2) and 54.0 (1)° with the substituted thiophene and the phenyl ring, respectively. Bond conjugation is observed for the linear sequence of atoms joining the thiophene ring and pyrazolone rings.

Comment

Schiff bases are important compounds having potential biological activities such as antibacterial and antitumor (Klayman *et al.*, 1979). Among the large number of such compounds, 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of these compounds, *e.g.* (*E*)-4-[4-(4-chlorobenzyloxy)-3-methoxybenzylideneamino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Duan *et al.*, 2006) and 4-[(*E*)-4-(2-{4-[(*E*)-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)iminomethyl]phenoxy}ethoxy)benzylideneamino]-1,5-dimethyl-1-phenyl-1*H*-dihydropyrazol-3(2*H*)-one (Diao & Fan, 2006) have been reported. We report here the crystal structure of the related title compound, (I).



(I)

The bond lengths and angles in the title molecule (Fig. 1) are within normal ranges (Allen *et al.*, 1987). The pyrazole ring (C6–C8/N2/N3) is essentially planar with an r.m.s. deviation of 0.0336 Å for the fitted atoms; this ring makes a dihedral angle of 54.0 (1)° with the phenyl ring (C9–C14). The substituted thiophene ring (C1–C4/S1/Br) is also essentially planar, with an r.m.s. deviation of 0.0020 Å for the fitted atoms; it forms a dihedral angle of 5.9 (2)° with the pyrazole ring. Bond conjugation is observed in the C5/N1/C6 sequence of atoms (Jin *et al.*, 2004).

Experimental

Under a nitrogen atmosphere, a mixture of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (0.42 g, 2 mmol), anhydrous Na_2SO_4 (3.0 g) and 3-bromo-2-thiophenecarboxaldehyde (0.36 g, 2 mmol) in anhydrous ethanol (20 ml) was refluxed for 6 h, yielding a light-yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was recrystallized from CH_2Cl_2 (30 ml) and washed with water (5 ml) and brine (5 ml). After being dried over Na_2SO_4, the solvent was removed under vacuum, and a light-yellow solid was isolated in yield 92% (0.68 g). Yellow single crystals of the compound suitable for X-ray analysis were grown by slow evaporation of a hexane/anhydrous ethanol (4:1) solution of (I) at room temperature over a period of about a week.

Crystal data

$C_{16}H_{14}BrN_3OS$
M_r = 376.27
Monoclinic, $P2_1/c$
a = 9.2439 (12) Å
b = 7.3436 (10) Å
c = 24.218 (3) Å
β = 101.003 (4)°
V = 1613.8 (4) Å^3
Z = 4
D_x = 1.549 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.68 mm^{-1}
T = 298 (2) K
Block, yellow
0.24 × 0.17 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.592, T_{max} = 0.661
8225 measured reflections
2891 independent reflections
2328 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.088
S = 1.02
2891 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.6288P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C), and Csp^3—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C).

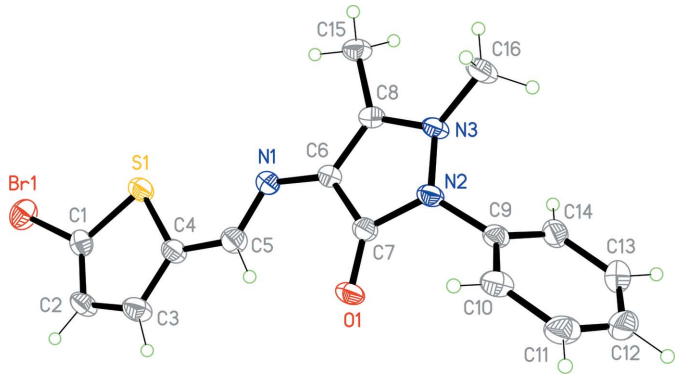


Figure 1
The molecular structure of (I) with the atom numbering, showing displacement ellipsoids drawn at the 30% probability level.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

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