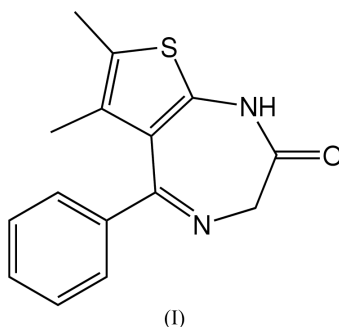


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Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.045
 wR factor = 0.152
Data-to-parameter ratio = 17.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.6,7-Dimethyl-5-phenyl-1*H*-thieno[2,3-*e*]-
[1,4]diazepin-2(3*H*)-oneIn the title molecule, $\text{C}_{15}\text{H}_{14}\text{N}_2\text{OS}$, the seven-membered ring
adopts a boat conformation. The carbonyl, imine and phenyl
groups lie to one side of the molecule, and the thienyl ring and
methylene group to the other.

Comment

Compounds related to the title compound, (I), are known to
enhance agonist binding to the A_1 adenosine receptor (Tran-
berg *et al.*, 2001). In this context, the conformation of the
seven-membered ring is of some interest as this determines the
conformation of the N atoms which, in turn, influences activity.

The structure of (I) is shown in Fig. 1 from which it can be seen that the seven-membered ring adopts a boat conformation. In this description, the N1, N4, C2 and C5 atoms are essentially coplanar, so that the N1–H, C2=O and phenyl groups lie to one side of the molecule, and the thienyl ring and methylene group to the other. Centrosymmetrically related pairs of molecules associate *via* hydrogen bonding between the amide groups: N1–H1 0.88 Å, H1···O2ⁱ 1.95 Å, N1···O2ⁱ 2.824 (3) Å and N1–H1···O2ⁱ 173° [symmetry code: (i) $2 - x, -y, -z$]. Interactions of the type C–H··· π also operate in the structure such that C3–H3b···Cgⁱⁱ is 2.62 Å (Cg is the centroid of the thienyl ring), with the angle subtended at H3b being 162° [symmetry code: (ii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$].

Experimental

Dry ammonia gas was introduced with ice cooling, to a solution of *N*-(3-benzoyl-4,5-dimethyl-thiophen-2-yl)-2-iodoacetamide (3.2 g, 8.0 mmol) in CHCl_3 (dry, 10 ml) and methanol (dry, 1 ml) over a period of 40 min. The mixture was then stirred at room temperature for a further 5 h. After this time, ice and water were added. The organic phase was washed with NaHCO_3 solution (saturated, 10 ml), H_2O ($3 \times 10\text{ ml}$) and dried with MgSO_4 . Subsequent removal of solvent under reduced pressure gave 2.024 g of crude product which was purified with silica chromatography. Hexane–ethyl acetate (1:1) as eluent gave the product (1.158 g, 53%) which was recrystallized from ethyl acetate to give clear crystals, m.p. (decomposition) 511–

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514 K. ^1H NMR (DMSO- d_6) δ 1.50 (s, 3H, CH₃), 2.24 (s, 3H, CH₃), 3.80 (d, 1H, CH₂), 4.50 (d, 1H, CH₂), 7.39–7.45 (m, 5H, C₆H₅), 11.03 (br, s, 1H, NH). ^{13}C NMR (DMSO- d_6) δ 12.43, 14.09, 57.85, 125.02, 125.94, 128.26, 128.34, 128.98, 129.93, 138.48, 142.48, 166.36, 168.29.

Crystal data

C₁₅H₁₄N₂OS

$M_r = 270.34$

Orthorhombic, *Pbca*

$a = 34.115$ (6) Å

$b = 10.4377$ (17) Å

$c = 7.484$ (3) Å

$V = 2664.7$ (13) Å³

$Z = 8$

$D_x = 1.348$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 24 reflections

$\theta = 7.4$ – 10.3°

$\mu = 0.24$ mm⁻¹

$T = 173$ (2) K

Block, colourless

$0.50 \times 0.31 \times 0.18$ mm

Data collection

Rigaku AFC-7R diffractometer

ω scans

5860 measured reflections

3057 independent reflections

2084 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\text{max}} = 27.5^\circ$

$h = 0 \rightarrow 44$

$k = -13 \rightarrow 10$

$l = -8 \rightarrow 9$

3 standard reflections

every 400 reflections

intensity decay: 0.6%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.152$

$S = 1.02$

3057 reflections

173 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.40$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

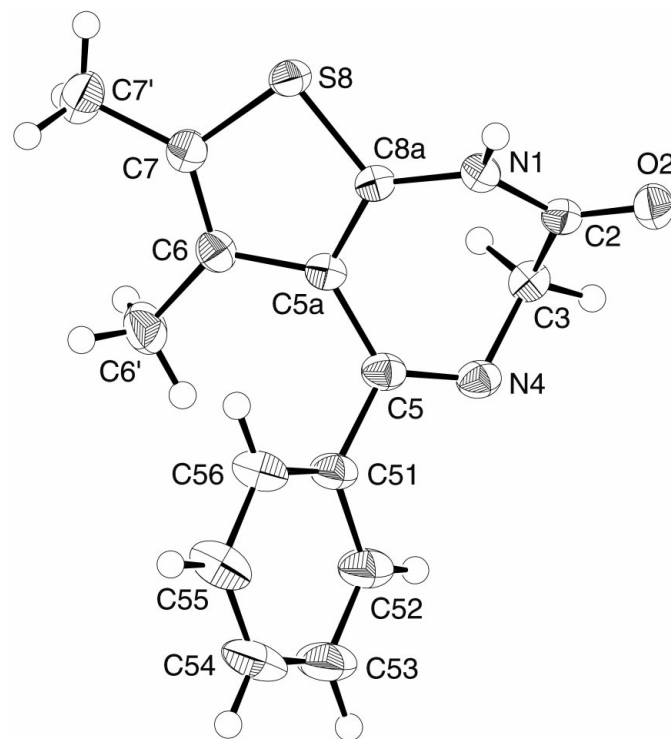


Figure 1

The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (ORTEPII; Johnson, 1976).

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); data reduction: *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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