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4-(*o*-Methoxyphenyl)-1,3,4,4a,5,6,7,8a-octahydro-2*H*-pyrano[2,3-*d*]pyrimidine-2-thione

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Key indicators

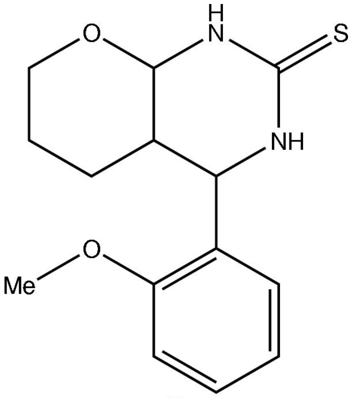
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.093
Data-to-parameter ratio = 17.6

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

The title compound, $C_{14}H_{18}N_2O_2S$, which was obtained from an extended Biginelli reaction, contains a pyran ring with a chair conformation, a pyrimidine ring with a half-chair conformation and a benzene ring. The crystal structure is stabilized by an intermolecular hydrogen-bond network.

Comment

Heterocyclic thiones, and especially heterocyclic molecules containing the thioamide group [—C(S)—NH—], form a class of compounds which are interesting because of the thione-thiol equilibrium. Due to this equilibrium, a variety of heterocyclic thione complexes of different metals, *i.e.* metal complexes of these compounds acting as ligands?]are formed having wide-ranging applications as analytical reagents, metal corrosion inhibitors, and as medically and biologically active molecules (Hussain et al., 1990).



(I)

Recently, the title compound, (I), was synthesized in our laboratory through an extended Biginelli reaction. The Biginelli reaction, one of the classic one-pot three-component reactions, furnishes functionalized dihydropyrimidines, which are increasingly attracting the attention of chemists because of their remarkable pharmacological efficiency (Kappe, 2000). The structure of 4-*p*-tolyl-1,3,4,4a,5,6,7,8a-octahydro-2*H*-pyrano[2,3-d]pyrimidin-2-one, (II), has already been reported (Yan *et al.*, 2005). We report here the structure of an analogue, (I).

The structure of (I), together with the atom-numbering scheme, is shown in Fig. 1. The molecule contains two fused six-membered rings and an arene substituent. The conformations of the rings in compounds (I) and (II) are very similar with, for example, a chair conformation of the pyran ring and a half-chair conformation of the pyrimidine ring. Owing to the S═C double bond, the environment of atom C2 is planar.

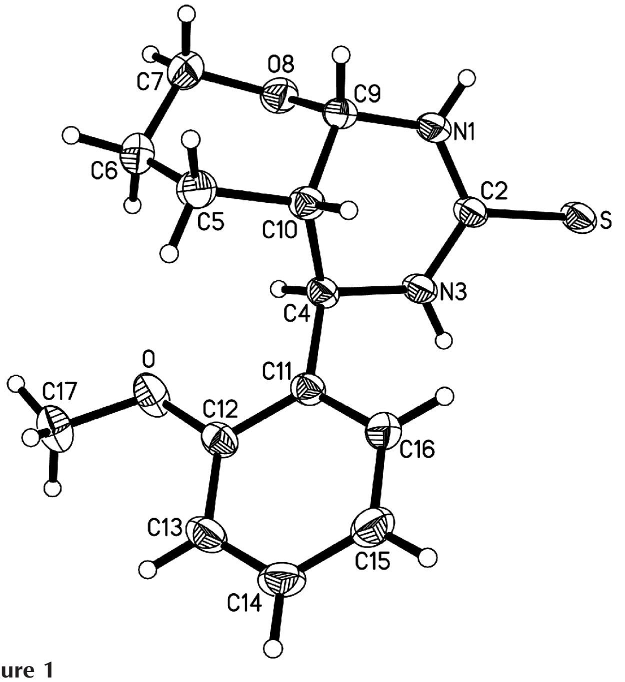


Figure 1
A view of the title molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

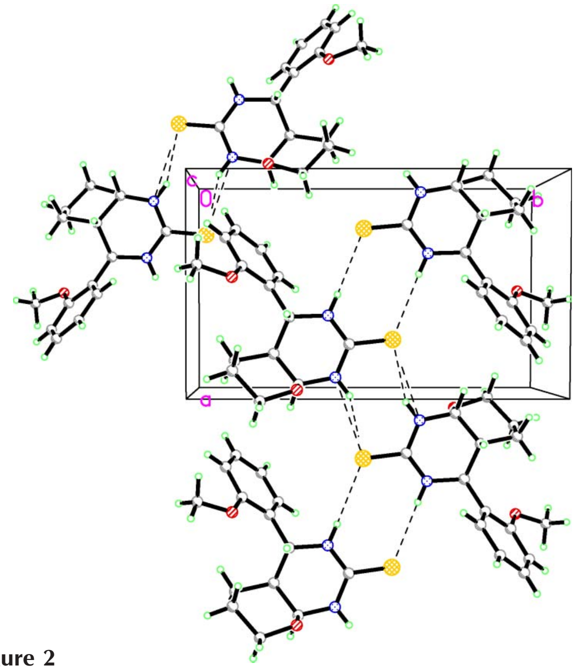


Figure 2
The molecular packing of compound (I), viewed along the *c* axis. H atoms not involved in hydrogen bonding have been omitted for clarity. Hydrogen bonds are indicated by dashed lines.

There are no unusual bond lengths and angles in (I), and the geometry is in good agreement with that found in (II). The O8—C9—C10—C5 torsion angle of −54.86 (14)° confirms a *cis* configuration of the two fused six-membered rings. Also, the C11—C4—C10—C5 torsion angle of −68.08 (14)° indicates a *trans*-C4,C10 configuration. These and related torsion angles are detailed in Table 1. The value of the bond length [1.7018 (12) Å] for S═C is in agreement with those of similar compounds, for example, of dithiobiurea (1.702 Å; Spofford & Amma, 1972). Selected bond distances are also listed in Table 1.

In the pyrimidinone analogue (II), atoms N1 and N3 are hydrogen bonded to the O atom of symmetry-related molecules at (2 − *x*, −*y*, −*z*) and (1 − *x*, −*y*, −*z*), respectively. In the molecule of (I), since the S atom replaces the O atom attached to the pyrimidine ring, there are two N—H⋯S═C instead of N—H⋯O═C intermolecular hydrogen bonds. Each of the hydrogen bonds participates in an eight-membered ring, as shown in Fig. 2. The molecular packing in the crystal structure of (I) is stabilized by these intermolecular hydrogen bonds.

Experimental

Details of the synthesis and purification of (I) will be described elsewhere (Zhu *et al.*, 2005). Crystals suitable for data collection were obtained by slow evaporation of an *N*,*N*-dimethylformamide solution at room temperature over a period of a week.

Crystal data

$C_{14}H_{18}N_2O_2S$
$M_r = 278.36$
Monoclinic, $P2_1/c$
$a = 8.5277$ (8) Å
$b = 14.2285$ (13) Å
$c = 11.5972$ (11) Å
$\beta = 93.590$ (2)°
$V = 1404.4$ (2) Å^3
$Z = 4$

$D_x = 1.317$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3491 reflections
$\theta = 2.3$–28.6°
$\mu = 0.23$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.54 × 0.34 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8142 measured reflections
3056 independent reflections

2486 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\rm max} = 27.0$°
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 18$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.093$
$S = 1.12$
3056 reflections
174 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.1006P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.047 (14)

Table 1
Selected geometric parameters (Å, °).

S—C2	1.7018 (12)	N1—C9	1.4299 (15)
O—C12	1.3655 (15)	N3—C2	1.3251 (14)
N1—C2	1.3409 (15)	N3—C4	1.4733 (15)
C12—O—C17	118.51 (11)	O8—C9—C10	111.65 (10)
C2—N3—C4	125.30 (10)	O—C12—C13	123.37 (11)
N3—C2—N1	118.37 (11)	O—C12—C11	116.55 (11)
O8—C9—C10—C5	−54.86 (14)	C11—C4—C10—C9	169.56 (10)
O8—C9—C10—C4	70.02 (13)	C11—C4—C10—C5	−68.08 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S^{i}	0.86	2.52	3.3061 (11)	153
N3—H3···S^{ii}	0.86	2.62	3.4702 (11)	172

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

All H atoms were positioned geometrically [N—H = 0.86 (1) Å, C—H = 0.93–0.98 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N)] and refined using a riding model.

Data collection: *SMART* (Bruker, 1999); 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*.

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