Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

A carbamoyl-substituted dihydropyrimidine with dimethyl sulfoxide solvent

B. Sridhar* and K. Ravikumar

Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India

Correspondence e-mail: sshiya@yahoo.com

Received 12 April 2005
Accepted 15 April 2005
Online 23 April 2005

Key indicators

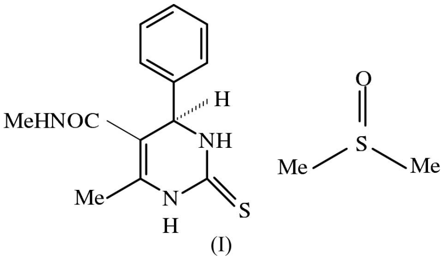
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.163
Data-to-parameter ratio = 19.9

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

6-Methyl-5-(*N*-methylcarbamoyl)-4-phenyl-1,2,3,4-tetrahydropyrimidine-2-thione dimethyl sulfoxide solvate, $C_{13}H_{15}N_3OS{\cdot}C_2H_6OS$, crystallizes with one molecule of each species in the asymmetric unit. The dihydropyridimine group adopts a flattened boat conformation. An N—H···O hydrogen bond exists between the N atom of the carbamoyl side chain and the carbonyl O atom of the solvent molecule. In addition, intermolecular N—H···O and N—H···S hydrogen bonding is observed in the crystal structure.

Comment

Pyrimidines are found in medically important compounds, such as antiviral, antitumor and cardiovascular agents (Atwal *et al.*, 1989). In addition, dihydropyrimidines have been found in some novel alkaloids, isolated from marine organisms, that exhibit biological activity (Fu *et al.*, 2003). Furthermore, dihydropyrimidines (DHPMs) are potential mimics of dihydropyridines (DHPs) as calcium channel blockers (Atwal *et al.*, 1990). Recently, we reported the structure of 6-methyl-5-(*N*-methylcarbamoyl)-4-phenyl-1,2,3,4-tetrahydropyrimidine-2-thione monohydrate (Ravikumar & Sridhar, 2005). In the present study, the crystal structure of the title compound, (I), as a dimethyl sulfoxide solvate, is reported.



(I)

The asymmetric unit contains one molecule of the dihydropyrimidine and one molecule of DMSO solvent. The geometry of (I) is very similar to that of the hydrated structure. An r.m.s overlay (pyrimidine ring r.m.s deviation = 0.019 Å) of the two structures shows significant similarities (Fig. 3). The DHPM ring adopts a more or less flattened boat-like conformation [asymmetry parameter ΔC_s(N1) = 0.048 (1); Nardelli, 1983], with atoms N1 and C4 defining the stern and bow positions, respectively. A similar conformation was also observed in the hydrated structure and in other DHPM compounds (Atwal *et al.*, 1990; Kappe *et al.*, 1997). The DPHM ring exists in the thione form; the C6═S1 = bond length of 1.687 (2) Å indicates double bond character. This distance is slightly longer than the pure double-bond distance

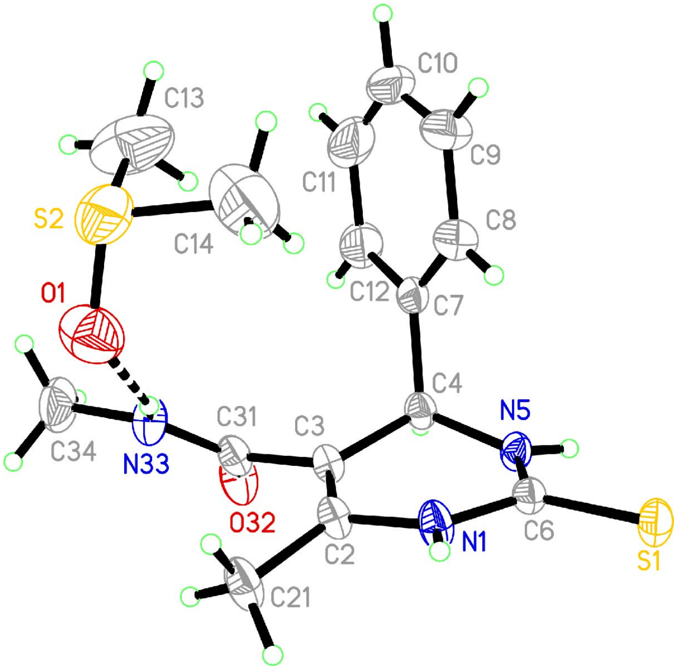


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The hydrogen bond is shown dashed.

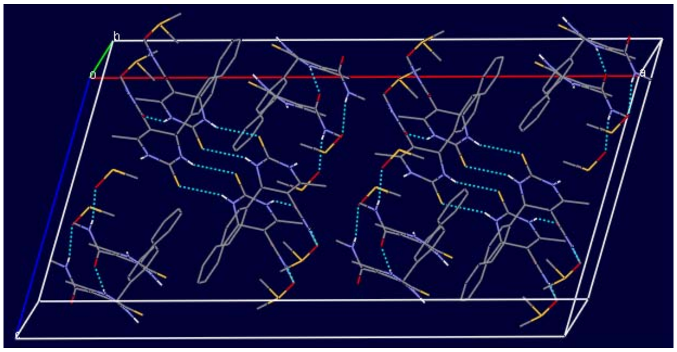


Figure 2
A packing diagram for (I), showing N—H···O and N—H···S hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

(1.61 Å; Pauling, 1960). This lengthening may be due to the participation in hydrogen bonding of the S atom (Tiekink, 1989). The carbamoyl side chain is in an extended conformation.

As observed in other DPHM structures, the phenyl ring is oriented perpendicular to the DPHM ring, with a dihedral angle of 79.3 (1)°. The C2—C3—C4—C7 and C6—N5—C4—C7 torsion angles of −105.5 (2) and 96.72 (19)°, respectively, clearly indicate the pseudo-axial orientation of the phenyl ring.

The crystal structure is stabilized by N—H···O and N—H···S hydrogen bonding (Fig. 2). As observed in the hydrated structure, the dimers form an $R_2^2(8)$ motif (Bernstein *et al.*, 1995). This dimer-forming tendency of the pyrimidine ring is also observed in other dihydropyrimidine structures (Chandra Mohan *et al.*, 2003). In (I), the S atom is hydrogen bonded to N5, while in the hydrated structure, it interacts with atom N1 of the pyrimidine ring. Atom N1 of the pyrimidine ring is hydrogen bonded with the carbonyl atom of the carbamoyl side chain.

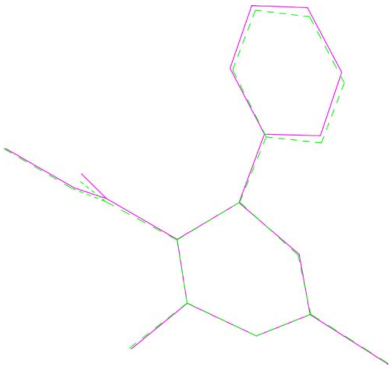


Figure 3
An least-squares overlay of (I) (dashed lines) with the hydrated structure (solid lines), showing the similarities in conformation.

The DMSO solvent forms an N—H···O hydrogen bond with atom N33 of the carbamoyl side chain, while in the hydrated structure, atom N33 is linked to atom O32 of the carbamoyl side chain.

In the crystal structure, the dimers are interconnected through N—H···O hydrogen bonding as infinite chains along the *b* axis.

Experimental

Compound (I) was prepared by known synthetic methods (Sadanandam *et al.*, 1992) and was recrystallized from DMSO.

Crystal data

$C_{13}H_{15}N_3OS\cdot C_2H_6OS$
$M_r = 339.47$
Monoclinic, $C2/c$
$a = 32.2080\ (19)$ Å
$b = 6.8884\ (4)$ Å
$c = 16.6204\ (10)$ Å
$\beta = 109.156\ (1)°$
$V = 3483.2\ (4)$ Å^3
$Z = 8$

$D_x = 1.295$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6813 reflections
$\theta = 2.5$–$28.0°$
$\mu = 0.32$ mm^{-1}
$T = 273\ (2)$ K
Block, colorless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
14 448 measured reflections
4040 independent reflections
3511 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.019$
$\theta_{\max} = 28.0°$
$h = -42 \rightarrow 42$
$k = -8 \rightarrow 8$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.164$
$S = 1.05$
4040 reflections
203 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0953P)^2 + 2.7256P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.74$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

O32—C31	1.229 (2)	N5—C4	1.460 (2)
N1—C6	1.355 (2)	N33—C31	1.331 (3)
N1—C2	1.401 (2)	C2—C3	1.328 (3)
N5—C6	1.324 (2)	C3—C4	1.518 (2)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯O32^{i}	0.86	2.02	2.825 (2)	156
N5—H5⋯S1^{ii}	0.86	2.60	3.415 (1)	158
N33—H33⋯O1	0.86	2.06	2.867 (3)	157

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$.

H atoms were included in calculated positions (C—H = 0.93–0.98 Å and N—H = 0.86 Å) and refined as riding, with U_{iso}(H) values set to 1.2 and 1.5 (CH_3) times the U_{eq} values of the parent atoms. In addition, the methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr M. Meera Shetty for providing the compound and Dr J. S. Yadav, Director, IICT, Hyderabad, for his kind encouragement.

References

Atwal, K. S., Rovnyak, G. C., O'Reilly, B. C. & Schwartz, J. (1989). *J. Org. Chem.* **54**, 5898–5907.

Atwal, K. S., Rovnyak, G. C., Schwartz, J., Malley, M. F. & Floyd, D. M. (1990). *J. Med. Chem.* **33**, 1510–1515.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2001). *SMART* (Version 5.625) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.

Bruno, I., Cole, J. J. C., Edgington, P. R., Kessler, M. K., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.

Chandra Mohan, K., Ravikumar, K., Shetty, M. M & Velmurugan, D. (2003). *Z. Kristallogr.* **218**, 46–55.

Fu, N. Y., Yuan, Y. Q., Pang, M. L. & Wang, J. T. (2003). *Chem. J. Chin. Univ.* **24**, 79–81.

Kappe, C. O., Fabian, W. M. F. & Semones, M. A. (1997). *Tetrahedron*, **53**, 2803–2816.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Pauling, L. (1960). *The Nature of Chemical Bond*, 3rd ed. Ithaca: Cornell University Press.

Ravikumar, K. & Sridhar, B. (2005). *Acta Cryst.* C**61**, o41–o44.

Sadanandam, Y. S., Shetty, M. M. & Diwan, P. V. (1992). *Eur. J. Med. Chem.* **27**, 87–92.

Sheldrick, G. M. (1990). *SHELXTL/PC*. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Tiekink, E. R. T. (1989). *Z. Kristallogr.* **187**, 79–84.