organic compounds

3,5,6-Trimethylthieno[2,3-d]pyrimidin-4(3H)-one

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In the title compound, $C_9H_{10}N_2OS$, the thienopyrimidine ring system is almost planar [greatest deviation from the mean plane = 0.0318 (13) Å for the S atom]. The crystal packing features C—H···O hydrogen bonds and π–π stacking interactions between inversion-related pairs of molecules with a centroid–centroid distance of 3.530 (3) Å.

Related literature

For the synthesis, properties and biological activity of pyrimidinone derivatives, see: Litvinov (2004); Al-Taisan *et al.* (2010). For the crystal and molecular structures of related compounds, see: Tashkhodzhaev *et al.* (2002).
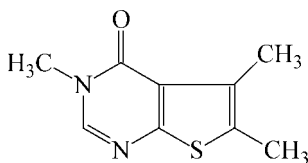
Experimental

Crystal data

$C_9H_{10}N_2OS$

$M_r = 194.25$

Monoclinic, $P2_1/c$

$a = 8.027$ (3) Å

$b = 10.706$ (5) Å

$c = 10.907$ (3) Å

$\beta = 97.333$ (3)°

$V = 929.7$ (6) Å^3

$Z = 4$

Cu $K\alpha$ radiation

$\mu = 2.77$ mm^{-1}

$T = 293$ K

$0.42 \times 0.36 \times 0.28$ mm

Data collection

Oxford Diffraction Xcalibur Ruby diffractometer

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)

$T_{min} = 0.324$, $T_{max} = 1.000$

2983 measured reflections

1588 independent reflections

1208 reflections with $I > 2\sigma(I)$

$R_{int} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.151$

$S = 1.05$

1588 reflections

122 parameters

H-atom parameters constrained

$\Delta\rho_{max} = 0.27$ e Å^{-3}

$\Delta\rho_{min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1[i]	0.93	2.32	3.250 (4)	173

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2064).

References

Al-Taisan Kh, M., Al-Hazimi, H. M. A. & Al-Shihry, Sh. S. (2010). *Molecules*, **15**, 3932–3957.

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Tashkhodzhaev, B., Turgunov, K. K., Usmanova, B., Grintselev-Knyazev, G. V., Antipin, M. Yu. & Shakhidoyatov, Kh. M. (2002). *J. Struct. Chem.* **43**, 872–875.

supporting information

3,5,6-Trimethylthieno[2,3-*d*]pyrimidin-4(3*H*)-one

Khamroqul Khatamov, Fozil Saitqulov, Jamshid Ashurov and Khusnutdin Shakhidoyatov

S1. Comment

The derivatives of thienopyrimidine are of interest because of their interesting pharmacological and biological activities (Litvinov, 2004). The title compound, $C_9H_{10}N_2OS$, may be used for obtaining bioactive molecules. The asymmetric unit of the title compound consists of a single molecule (Fig.1). The thienopyrimidine ring system (N1/C2/N3/C4/C5/C6/C7/C8/S1) is ideal planar with greatest deviation from mean plane 0.0318 (12) Å for the S1). The crystal packing is stabilized by intermolecular hydrogen bonds (Table 1) and π-π stacking interactions between inversion-related pair of molecules with a centroid-centroid (N1/C2/N3/C4/C5/C6/C7/C8/S1) distance of 3.530 (3) Å.

S2. Experimental

To a suspension of 5,6-trimethylthieno(2,3 - d)pyrimidin-4-one (181 mg, 0.1 mmol) in 50 ml e thanol sodium hydride (24 mg, 1 mmol) was added. The mixture was stirred at room temperature for 30 min. Then a solution of methyl iodide(142 mg, 1 mmol) in ethanol was added drop wise. The solution was stirred at 353–363 K for 4 h, then the solution was evaporated under reduced pressure and the residue was treated by distilled water. The precipitate was filtered and dried. Yield of 84% (174 mg). Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from chloroform at room temperature.

S3. Refinement

All H atoms were placed in geometrically idealized positions (C—H 0.96 (methyl) and C—H 0.93 Å (phenyl) and treated as riding on their parent atoms, with U(H) set to 1.2 to 1.5U(C).
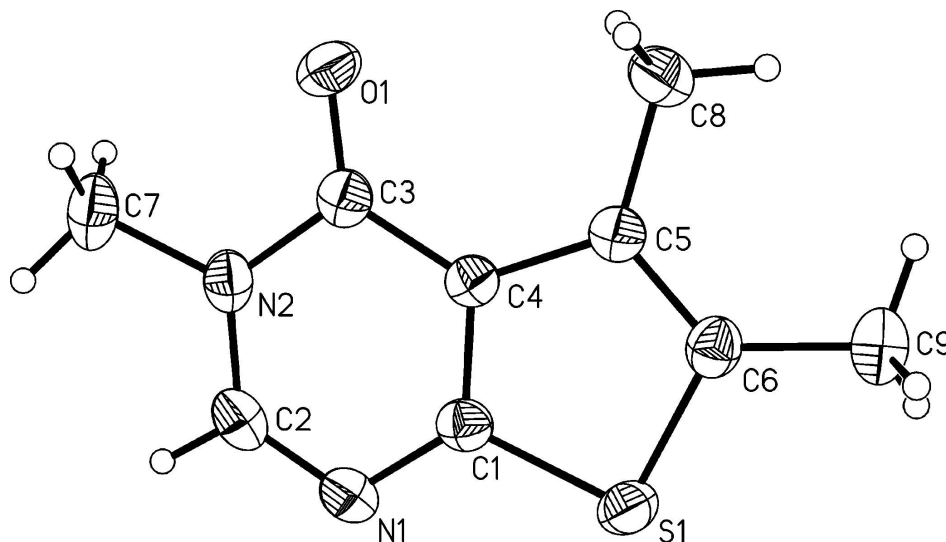
Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom numbering scheme.
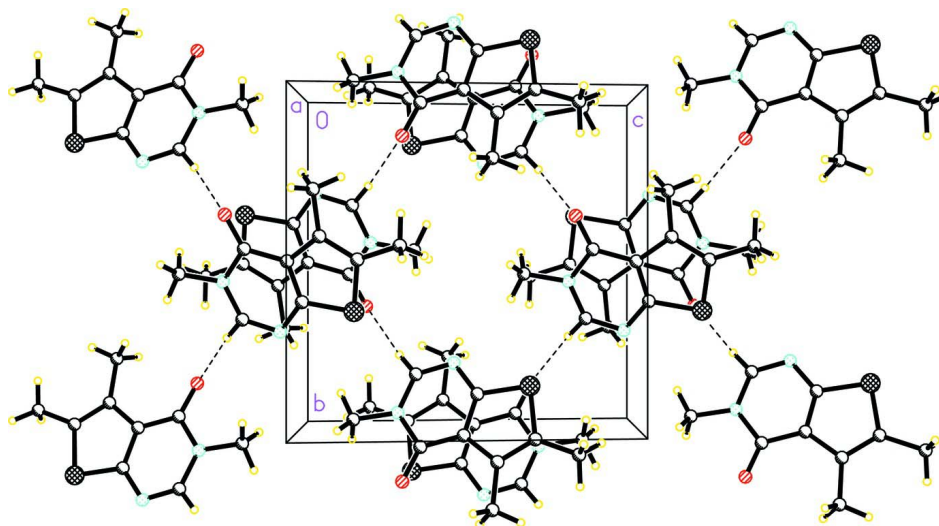
Figure 2

A packing diagram for title compound. C—H···O hydrogen bonds are shown as dashed lines.

3,5,6-Trimethylthieno[2,3-*d*]pyrimidin-4(3*H*)-one

Crystal data

$C_9H_{10}N_2OS$	$V = 929.7\ (6)$ Å^3
$M_r = 194.25$	$Z = 4$
Monoclinic, $P2_1/c$	$F(000) = 408$
Hall symbol: -P 2ybc	$D_x = 1.388$ Mg m^{-3}
$a = 8.027\ (3)$ Å	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$b = 10.706\ (5)$ Å	Cell parameters from 185 reflections
$c = 10.907\ (3)$ Å	$\theta = 4.1–43.7°$
$\beta = 97.333\ (3)°$	$\mu = 2.77$ mm^{-1}

T = 293 K

Block, colourless

0.42 × 0.36 × 0.28 mm

Data collection

Oxford Diffraction Xcalibur Ruby diffractometer	2983 measured reflections
Radiation source: fine-focus sealed tube	1588 independent reflections
Graphite monochromator	1208 reflections with $I > 2\sigma(I)$
Detector resolution: 10.2576 pixels mm^{-1}	R_{int} = 0.029
ω scans	θ_{max} = 67.2°, θ_{min} = 5.6°
Absorption correction: multi-scan	$h = -8 \rightarrow 9$
(*CrysAlis PRO*; Oxford Diffraction, 2009)	$k = -12 \rightarrow 12$
T_{min} = 0.324, T_{max} = 1.000	$l = -13 \rightarrow 11$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.1022P)^2 + 0.0132P]$
$wR(F^2)$ = 0.151	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
1588 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
122 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0070 (14)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.3074 (3)	0.1156 (2)	0.4761 (3)	0.0448 (7)
C2	0.4524 (4)	0.1684 (3)	0.6555 (3)	0.0559 (8)
H2	0.5198	0.2239	0.7052	0.067*
C3	0.3198 (3)	−0.0372 (3)	0.6401 (2)	0.0467 (7)
C4	0.2664 (3)	−0.0020 (2)	0.5153 (2)	0.0396 (6)
C5	0.1683 (3)	−0.0728 (2)	0.4196 (2)	0.0446 (7)
C6	0.1362 (4)	−0.0066 (3)	0.3125 (2)	0.0498 (7)
C7	0.4735 (4)	0.0352 (4)	0.8380 (3)	0.0693 (10)
H7A	0.3808	0.0084	0.8791	0.104*
H7B	0.5195	0.1109	0.8754	0.104*
H7C	0.5584	−0.0285	0.8454	0.104*
C8	0.1125 (4)	−0.2045 (3)	0.4345 (3)	0.0615 (8)

H8A	0.0263	−0.2062	0.4879	0.092*
H8B	0.2062	−0.2539	0.4699	0.092*
H8C	0.0690	−0.2379	0.3551	0.092*
C9	0.0413 (4)	−0.0454 (4)	0.1917 (3)	0.0694 (9)
H9A	−0.0690	−0.0091	0.1831	0.104*
H9B	0.0319	−0.1348	0.1890	0.104*
H9C	0.0998	−0.0173	0.1254	0.104*
N1	0.4025 (3)	0.2028 (2)	0.5443 (2)	0.0537 (6)
N2	0.4153 (3)	0.0576 (2)	0.7075 (2)	0.0505 (6)
O1	0.2922 (3)	−0.13487 (19)	0.69116 (18)	0.0595 (6)
S1	0.22443 (10)	0.14187 (7)	0.32561 (6)	0.0543 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0458 (16)	0.0418 (14)	0.0482 (15)	0.0049 (11)	0.0116 (12)	−0.0006 (11)
C2	0.0553 (19)	0.0501 (17)	0.062 (2)	0.0007 (13)	0.0055 (14)	−0.0163 (14)
C3	0.0443 (16)	0.0495 (16)	0.0468 (15)	0.0075 (12)	0.0074 (12)	−0.0022 (12)
C4	0.0368 (14)	0.0398 (14)	0.0430 (14)	0.0064 (10)	0.0081 (11)	0.0000 (10)
C5	0.0427 (16)	0.0448 (15)	0.0470 (15)	0.0027 (11)	0.0083 (12)	−0.0010 (11)
C6	0.0476 (17)	0.0553 (17)	0.0458 (15)	0.0063 (13)	0.0036 (12)	−0.0013 (12)
C7	0.073 (2)	0.088 (3)	0.0436 (17)	0.0157 (18)	−0.0037 (15)	−0.0115 (15)
C8	0.067 (2)	0.0505 (17)	0.067 (2)	−0.0105 (14)	0.0080 (16)	−0.0035 (14)
C9	0.072 (2)	0.083 (2)	0.0512 (18)	−0.0023 (18)	−0.0023 (15)	−0.0075 (16)
N1	0.0568 (15)	0.0428 (13)	0.0613 (16)	0.0001 (11)	0.0071 (12)	−0.0060 (11)
N2	0.0519 (14)	0.0564 (15)	0.0419 (13)	0.0091 (11)	0.0006 (10)	−0.0089 (10)
O1	0.0688 (15)	0.0553 (13)	0.0531 (12)	0.0013 (10)	0.0025 (10)	0.0164 (9)
S1	0.0631 (6)	0.0502 (5)	0.0493 (5)	0.0040 (3)	0.0068 (3)	0.0099 (3)

Geometric parameters (Å, º)

C1—N1	1.365 (4)	C6—C9	1.494 (4)
C1—C4	1.382 (4)	C6—S1	1.739 (3)
C1—S1	1.714 (3)	C7—N2	1.461 (4)
C2—N1	1.282 (4)	C7—H7A	0.9600
C2—N2	1.364 (4)	C7—H7B	0.9600
C2—H2	0.9300	C7—H7C	0.9600
C3—O1	1.218 (3)	C8—H8A	0.9600
C3—N2	1.419 (4)	C8—H8B	0.9600
C3—C4	1.424 (4)	C8—H8C	0.9600
C4—C5	1.441 (3)	C9—H9A	0.9600
C5—C6	1.362 (4)	C9—H9B	0.9600
C5—C8	1.495 (4)	C9—H9C	0.9600
N1—C1—C4	126.3 (3)	N2—C7—H7C	109.5
N1—C1—S1	122.1 (2)	H7A—C7—H7C	109.5
C4—C1—S1	111.6 (2)	H7B—C7—H7C	109.5
N1—C2—N2	125.8 (3)	C5—C8—H8A	109.5

N1—C2—H2	117.1	C5—C8—H8B	109.5
N2—C2—H2	117.1	H8A—C8—H8B	109.5
O1—C3—N2	119.6 (3)	C5—C8—H8C	109.5
O1—C3—C4	127.9 (3)	H8A—C8—H8C	109.5
N2—C3—C4	112.4 (2)	H8B—C8—H8C	109.5
C1—C4—C3	118.9 (2)	C6—C9—H9A	109.5
C1—C4—C5	112.6 (2)	C6—C9—H9B	109.5
C3—C4—C5	128.5 (2)	H9A—C9—H9B	109.5
C6—C5—C4	112.0 (2)	C6—C9—H9C	109.5
C6—C5—C8	123.8 (3)	H9A—C9—H9C	109.5
C4—C5—C8	124.2 (2)	H9B—C9—H9C	109.5
C5—C6—C9	129.1 (3)	C2—N1—C1	113.8 (3)
C5—C6—S1	112.0 (2)	C2—N2—C3	122.6 (2)
C9—C6—S1	118.9 (2)	C2—N2—C7	119.2 (3)
N2—C7—H7A	109.5	C3—N2—C7	118.2 (3)
N2—C7—H7B	109.5	C1—S1—C6	91.90 (13)
H7A—C7—H7B	109.5		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1[i]	0.93	2.32	3.250 (4)	173

Symmetry code: (i) −x+1, y+1/2, −z+3/2.