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2,4,6-Trimethyl-1,3,5-tris(2-pyrimidinylthiomethyl)benzene chloroform hemisolvate

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Key indicators

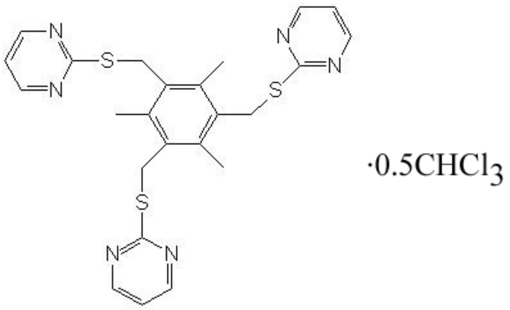
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
Disorder in solvent or counterion
R factor = 0.070
wR factor = 0.180
Data-to-parameter ratio = 14.5

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

The title compound, $C_{24}H_{24}N_6S_3\cdot0.5CHCl_3$ (TPSB·0.5CHCl$_3$), crystallizes with one molecule of TPSB and half a solvent molecule of CHCl$_3$ in the asymmetric unit. The three 2-pyrimidinylthiomethyl groups attached to the central benzene ring adopt a *cis,cis,cis* conformation. The molecule of TPSB is analogous to a 'three-legged stool', and the dihedral angles between each of the three 'legs' (2-pyrimidinyl rings) and the central benzene ring are 87.0 (5), 78.2 (3) and 97.1 (6)°, respectively, while the angles between the three 'legs' are 41.1 (3), 91.3 (5) and 49.5 (7)°, respectively.

Comment

Hitherto, much progress has been achieved in crystal engineering and supramolecular chemistry, aimed at preparing new materials with novel optical, magnetic and electronic functions. A remarkable development in this area may be the possibility to select suitable building blocks and assemble them into structures with specific topologies and properties (Goodgame *et al.*, 1993). Multi-thioether ligands possess unusual potential for structure control in crystal construction, and some interesting crystal structures of complexes with multi-thioether ligands (Bu *et al.*, 2002; Alcock *et al.*, 1978) have been reported. In addition, the ligand containing the pyrimidinyl group has also shown interesting coordination chemistry with transition metal ions (Su *et al.*, 1999). However, only a few examples of these types of ligands have been structurally characterized (Gormley *et al.*, 2000; Li *et al.*, 2002). Here we report the synthesis and structure of a new flexible trithioether ligand, containing pyrimidinyl groups, 2,4,6-trimethyl-1,3,5-tris(2-pyrimidinylthiomethyl)benzene (TPSB), (I). Further work on the reaction of the ligand with transition metal ions, such as Ag^{2+} and Pt^{2+}, is still in progress in our group.



(I)

As shown in Fig. 1, the three 2-pyrimidinylthio groups of TPSB have a *cis,cis,cis* conformation, which makes TPSB look like a 'three-legged stool'. The three 2-pyrimidinylthio groups

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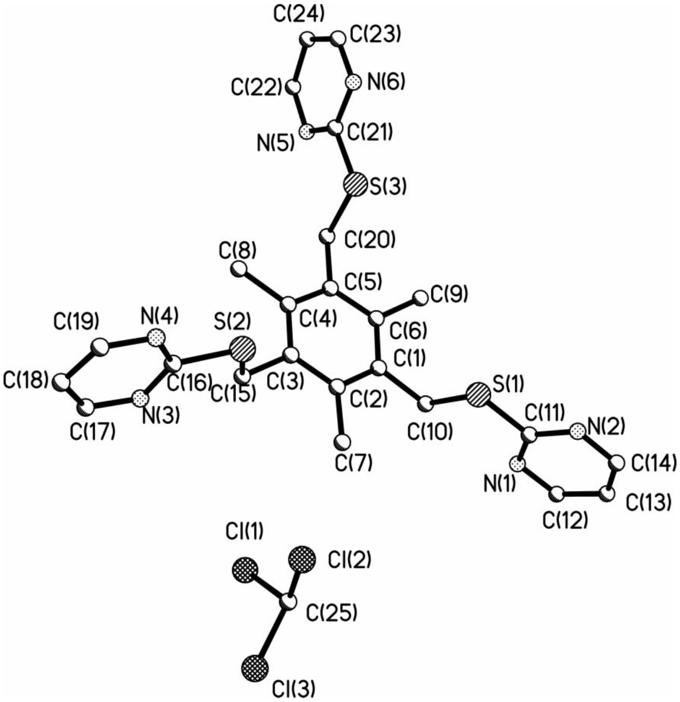

Figure 1
View of the title compound, with 30% probability displacement ellipsoids.

lie on the same side of the plane of the central benzene ring, three methyl groups (C7–C9) and three methene groups (C10, C15 and C20). The plane formed by the three S atoms is almost parallel to the central benzene ring, with a dihedral angle of 2.2 (8)°. The three dihedral angles between each pyrimidinyl group (*viz*. C11–C14/N1/N2, C16–C19/N3/N4 and C21–C24/N5/N6) and the central benzene ring are 87.0 (5), 78.2 (3) and 97.1 (6)°, respectively, while the angles between the three pyrimidinyl planes are 41.1 (3), 91.3 (5) and 49.5 (7)°, respectively. The torsion angles C15—S2—C16—N3, C10—S1—C11—N1 and C20—S3—C21—N5 are 0, −7.6 (6) and 12.2 (3)°, respectively, which indicates that there may be conjugation between atom S2 and the neighboring pyrimidinyl ring.

Experimental

1,3,5-Tris(bromethyl)-2,4,6-trimethylbenzene was prepared according to a reported method (van der Made & van der Made, 1993), and sodium 2-pyrimidinylthiolate was obtained by the reaction of 2-mercaptopyrimidine with EtONa. 1,3,5-Tris(2-pyrimidinylthiomethyl)-2,4,6-trimethylbenzene was synthesized by the reaction of 1,3,5-tris(bromethyl)-2,4,6-trimethylbenzene and sodium 2-pyrimidinylthiolate in EtOH at 353 K (yield: 85%; mp: 468–469 K); IR (KBr): 1565 (*s*), 1548 (*s*), 1381 (*s*), 1254 (*w*), 1207 (*m*), 1183 (*m*), 798 (*w*), 772 (*m*), 749 (*m*), 629 (*w*); ^{1}H NMR (CDCl$_3$): δ 2.53 (3H, *s*), 4.50 (2H, *s*), 7.00 (1H, *t*), 8.56 (2H, *d*); analysis calculated for $C_{24}H_{24}N_6S_3$: C 58.51, H 4.91, N 17.06%; found: C 58.42, H 5.03, N 17.13%. Colorless single crystals of (I) were obtained by recrystallization from chloroform.

Crystal data

$C_{24}H_{24}N_6S_3\cdot0.5CHCl_3$
M_r = 552.36
Monoclinic, $P2_1/c$
a = 18.957 (6) Å
b = 9.961 (3) Å
c = 16.335 (5) Å
β = 110.885 (6)°
V = 2881.9 (16) Å^3
Z = 4

D_x = 1.273 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10845 reflections
θ = 2.4–24.7°
μ = 0.42 mm^{-1}
T = 293 (2) K
Needle, colorless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.884, T_{max} = 0.921
11025 measured reflections

4853 independent reflections
1832 reflections with $I > 2\sigma(I)$
R_{int} = 0.098
θ_{max} = 25.0°
$h = -22 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.180$
S = 0.93
4853 reflections
334 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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