Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,4,6-Tris(2-pyridylsulfanylmethyl)-1,3,5-triazine

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Received 13 December 2006
Accepted 15 December 2006

In the title compound, $C_{21}H_{18}N_6S_3$, the molecules are bound into a three-dimensional array through π–π stacking interactions.

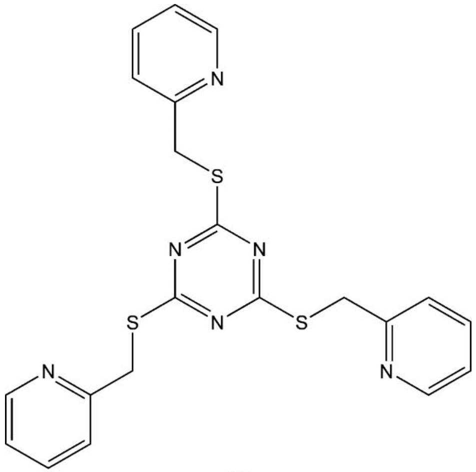
Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.080
Data-to-parameter ratio = 17.1

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

Comment

There is one report in the literature (Ray *et al.*, 1994) of a previous synthesis of the title compound, (I) (2-TPST). This procedure involved the synthesis of 2-TPST in an acetone solution at room temperature from the starting materials 2-mercaptopyridine and cyanuric chloride, followed by column chromatography with $CHCl_3$ as the eluent to give the compound in a 65% yield. However, we have been able to increase this yield by using different starting materials and a simple aqueous addition reaction. The synthesis of flexible thioether-containing ligands, such as the title compound, has received increasing interest in recent years (Amoore *et al.*, 2003, 2005; Awaleh *et al.*, 2006; Li *et al.*, 2003; Bu *et al.*, 2003). This is primarily due to the ability of flexible ligands to be present in a variety of conformations, allowing for a greater diversity of structures to be generated through variations in the remaining components of the crystal structure (Hu *et al.*, 2005; Sun *et al.*, 2005; Awaleh *et al.*, 2005; Plater *et al.*, 2001; Steel, 2005). A crystal structure containing 2-TPST has never been reported. However, the closely related ligand 2,4,6-tri[(4′-pyridyl)sulfanylmethyl]-1,3,5-triazine (4-TPST) has been incorporated into a variety of crystal structures with silver (Hong *et al.*, 2000*a*) and nickel salts (Hong *et al.*, 2000*b*) and also with water (Amoore & Kepert, 2005).



(I)

The crystal structure of the title compound contains one 2-TPST molecule in the asymmetric unit (Fig. 1). The 2-TPST molecule adopts an approximately planar conformation with

doi:10.1107/S1600536806054456

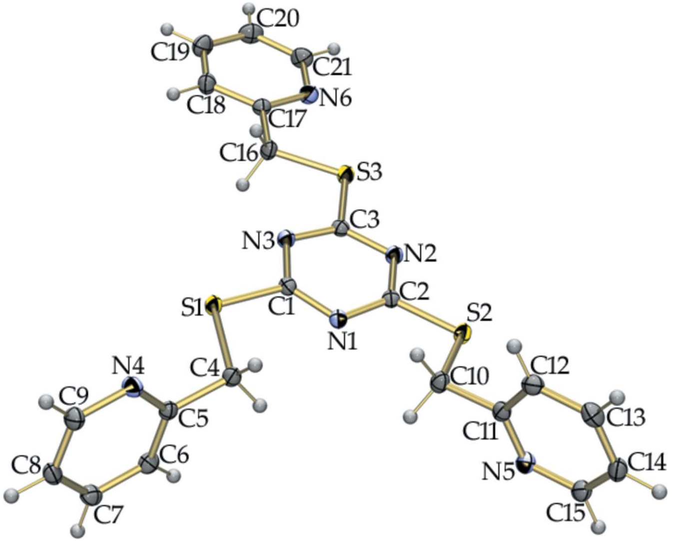


Figure 1
The molecular structure with displacement ellipsoids drawn at the 50% probability level.

the exception of two of the pyridine rings, which have dihedral angles of 85.67 (3) (N1,C11–C16) and 80.73 (4)° (N4,C5–C9) with respect to the central triazine ring. The conformation of the 2-TPST molecule is quite different from that seen in the crystal structures of the related 4-TPST molecule in which the pyridine rings were bent above or below the plane of the central triazine ring in all instances (Hong *et al.*, 2000*a*,*b*; Amoore & Kepert 2005). The 2-TPST molecules are held together in three dimensions through intermolecular π–π stacking interactions. All aromatic rings in the 2-TPST molecule are involved in face-to-face π–π stacking interactions with crystallographically equivalent rings of four separate adjacent molecules. The centroid-to-centroid distance between triazine rings of adjacent molecules is 3.6089 (2) Å. For the π–π stacking interactions between pyridine rings the centroid-to-centroid distances are 3.7515 (2), 3.6038 (2) and 3.5098 (2) Å (Janiak 2000).

Experimental

To a vigorously stirred solution of 2-picolinyl chloride hydrochloride (5.00 g, 30.5 mmol) in water (200 ml), a solution of 1,3,5-triazine-2,4,6-trithiol trisodium salt (15.16 g, 15.0% *w*/*w* in water) and sodium carbonate (4.71 g, 44.5 mmol) in water (50 ml) was added dropwise over 30 minutes, causing the formation of a white viscous precipitate. The mixture was then stirred for 24 h. The reaction mixture was extracted with dichloromethane (3 × 75 ml), the organic portions combined and back-extracted with water (150 ml). The organic portions were then evaporated under vacuum to give a light-brown solid. This solid was purified on a silica gel column eluted with a dichloromethane: ethanol solution (1:3, R_f = 0.70), to give a semi-crystalline tan solid (3.1240 g, 74%). EI-HRMS (dichloromethane/methanol); calc. for $C_{21}H_{18}N_6S_3$ + H = 451.08319 *m*/*z*; found 451.082127 *m*/*z*; error 2.4×10^{-6}. Crystals of the title compound were grown by the slow evaporation of a column fraction containing a 1:3 mixture of dichloromethane and ethanol.

Crystal data

$C_{21}H_{18}N_6S_3$
$M_r = 450.59$
Triclinic, $P\overline{1}$
$a = 8.5801$ (7) Å
$b = 10.1489$ (8) Å
$c = 11.9189$ (9) Å
$\alpha = 97.5154$ (12)°
$\beta = 98.4549$ (13)°
$\gamma = 92.4718$ (13)°
$V = 1015.72$ (14) Å^3
$Z = 2$
$D_x = 1.473$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.39$ mm^{-1}
$T = 123$ (2) K
Rectangular rod, pale yellow
0.41 × 0.24 × 0.22 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.847$, $T_{\max} = 0.92$
9852 measured reflections
4635 independent reflections
4206 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 28.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.02$
4635 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 0.5598P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT-Plus* (Bruker, 1997); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *XCIF* (Bruker, 2001).

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