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1,3,5-Tris(phenylthiomethyl)-2,4,6-trimethylbenzene

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Key indicators

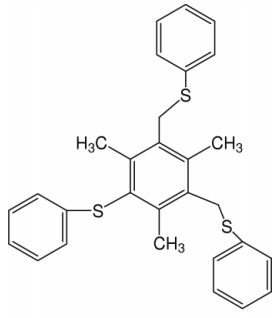
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.160
Data-to-parameter ratio = 15.8

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

The molecular structure of the title compound, $C_{30}H_{30}S_3$, adopts a *cis, trans, trans*-conformation for the three phenylthio groups about the central benzyl ring, which is analogous to a 'soft-shelled crawling turtle'. The dihedral angle between the 'head' phenyl ring and the central phenyl ring is 89.0 (4)°, while the angles between the two 'foot' phenyl rings and the central ring are 18.0 (6) and 6.7 (4)°, respectively.

Comment

Crystal engineering and supramolecular chemistry aimed at developing systems to perform optical, magnetic and electronic functions as well as intercalation systems for ion- or molecule-exchange and catalytic properties are some of the most attractive research areas currently, and much progress has been achieved in both theoretical studies and applications for new materials (Yuge *et al.*, 1996). One of the most remarkable developments in crystal engineering may be the possibility to select suitable building blocks to assemble into structures with specific topologies (Goodgame *et al.*, 1993; Gormley *et al.*, 2000). Multithioether ligands possess unusual potential for structure control in inorganic chemistry, and many unusual crystal structures of complexes with multithioether ligands (Bu *et al.*, 2002; Alcock *et al.*, 1978) have been reported. There are, however, just a few examples of organic ligands of multithioether ligands that have been structurally characterized (Gormley *et al.*, 2000). In recent years, one of our interests has been to study further the complexing abilities and the reactivities of a few types of dipodal or tripodal flexible ligands including disulfoxide, trisulfoxide, dithioether, trithioether, and their derivatives. As part of our structural studies of the trithioether series, we report here the synthesis and structure of a new flexible tripodal ligand, 1,3,5-tris-(phenylthiomethyl)-2,4,6-trimethylbenzene, (I); more work on the reaction with transition metal ions is still in progress.



(I)

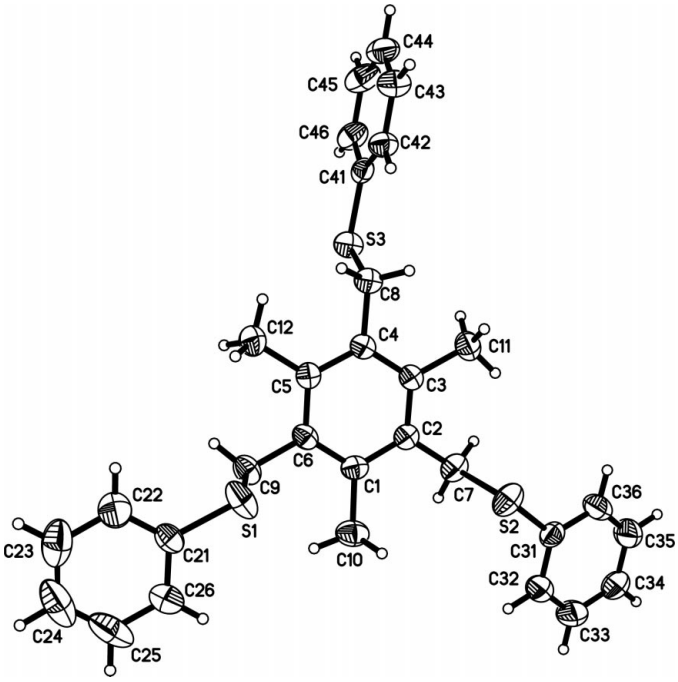


Figure 1
View of the title compound with 30% probability ellipsoids.

The title molecule has a *cis, trans, trans*-conformation (Fig. 1) for the three phenylthio groups about the central benzyl ring, which is analogous to a 'soft-shelled crawling turtle'. Phenylthio 1 (composed of S1 and C21–C26) and 2 (composed of S2 and C31–C36) are on the same side of the central ring; these, together with the two adjacent methyl groups (C11 and C12), form the four feet of this 'soft-shelled turtle'. Phenylthio 3 (S3, C41–C46) is on the other side of the central ring, forming the head, held high. The remaining methyl group (C10) is the tail. The dihedral angle between the head ring (phenyl 3) and the central ring is 89.0 (4)°, while the angles between the other two phenyl rings (1 and 2) and the central ring are 18.0 (6) and 6.7 (4)°, respectively. The dihedral angle between the two rear foot phenyl rings (1 and 2) is 21.3 (3)°.

Experimental

1,3,5-Tris(bromethyl)-2,4,6-trimethylbenzene was prepared according to a reported procedure and was characterized by NMR and IR analysis, giving results consistent with those in the literature (van der Made & van der Made, 1993). Phenylthiopotassium was obtained by the reaction of KOH with thiophenol in warm EtOH. 1,3,5-Tris-(phenylthiomethyl)-2,4,6-trimethylbenzene, (I), was synthesized by the reaction of 1,3,5-tris(bromethyl)-2,4,6-trimethylbenzene and phenylthiopotassium in EtOH at 353 K; yield: 86%; m.p. 381–383 K; IR (KBr): 2957(*w*), 2921(*w*), 1584(*m*), 1572(*m*), 1479(*s*), 1438(*s*), 1226(*m*), 1091(*m*), 1024(*m*), 755(*s*), 689(*s*), 471(*w*) cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.45 (3H, *s*), 4.19 (2H, *s*), 7.22–7.40 (5H, *m*); analysis calculated for $C_{30}H_{30}S_3$: C 73.00, H 6.21%; found: C 73.12, H 6.18%. Colorless single crystals of (I) were obtained by recrystallization from acetonitrile.

Crystal data

$C_{30}H_{30}S_3$	D_x = 1.215 Mg m^{-3}
M_r = 486.72	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2901 reflections
a = 9.0660 (6) Å	θ = 1.7–25.1°
b = 18.5382 (13) Å	μ = 0.30 mm^{-1}
c = 15.9779 (11) Å	T = 293 (2) K
β = 97.785 (2)°	Prism, colorless
V = 2660.6 (3) $Å^3$	0.30 × 0.25 × 0.20 mm
Z = 4	

Data collection

Siemens SMART CCD diffractometer	4703 independent reflections
ω scans	2679 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.033
T_{min} = 0.917, T_{max} = 0.943	θ_{max} = 25.1°
9493 measured reflections	$h = -10 \rightarrow 4$
	$k = -18 \rightarrow 22$
	$l = -18 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.7755P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.054	$(\Delta/\sigma)_{max}$ = 0.005
$wR(F^2)$ = 0.160	$\Delta\rho_{max}$ = 0.22 e $Å^{-3}$
S = 1.02	$\Delta\rho_{min}$ = −0.24 e $Å^{-3}$
4703 reflections	
298 parameters	
H-atom parameters constrained	

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*.

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