

1,2,4,6-Tetrathiacycloheptane

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Key indicators

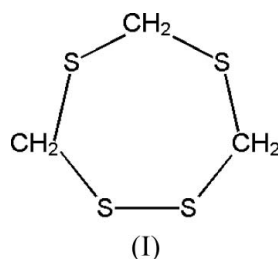
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(S-C) = 0.004$ Å
 R factor = 0.066
 wR factor = 0.138
Data-to-parameter ratio = 31.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $C_3H_6S_4$, is a cyclic polysulfane containing one disulfane S_2 group and two single S atoms bridged by three CH_2 groups, thus forming a seven-membered ring. The ring exhibits a chair conformation.

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Comment

The title compound, (I), was synthesized in the course of studies of metal complexes of polysulfides and related compounds (von Chrzanowski *et al.*, 2005; Steudel, 2002).



The molecule of (I) contains two sulfide and one disulfide groups, separated by CH_2 groups. The $S1-S2$ distance in the disulfide group is 2.028 (1) Å, which agrees well with the $S-S$ distances found for other disulfides, *e.g.* in dibenzylidysulfide (2.02 Å; Lee & Bryant, 1969).

The $S-C$ bonds between the disulfide S atoms and the neighbouring C atoms [$S1-C3 = 1.829$ (4) Å and $S2-C1 = 1.827$ (4) Å] are comparatively long, whereas the bonds between the isolated S atoms and the C atoms connected to the disulfide S atoms [$S3-C1 = 1.798$ (4) Å and $S4-C3 = 1.798$ (4) Å] are short, and the $C-S$ distances for the CH_2 groups bridging the two single S atoms are $S3-C2 = 1.816$ (4) Å and $S4-C2 = 1.823$ (4) Å, which compare well with the $C-S$ distances in thia-crown ethers, *e.g.* 1,4,7,10-tetrathiacyclododecane (1.806–1.825 Å; Robinson & Sango-

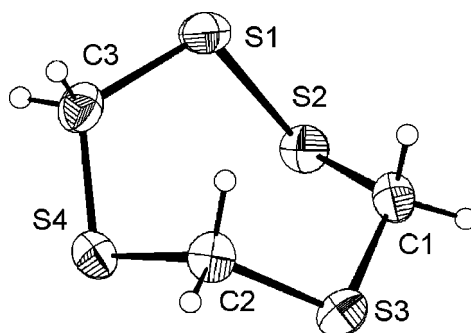


Figure 1
The molecule structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

koya, 1988). The ring has a chair conformation, with atoms S1 and C2 forming the apices.

Experimental

The title compound was formed as a by-product of the synthesis of 1,2,4-trithiacyclopentane by reaction of sodium sulfide (0.83 mol of the nonahydrate) with sulfur (0.16 mol) in water (350 ml), stirring for 7 h, extraction with dichloromethane and evaporation of the solvent. A yellow oil resulted, from which colourless crystals of (I) crystallized within a week.

Crystal data

$C_3H_6S_4$	$Z = 4$
$M_r = 170.32$	$D_x = 1.711 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 11.1285 (4) \text{ \AA}$	$\mu = 1.31 \text{ mm}^{-1}$
$b = 5.2653 (2) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 11.7399 (4) \text{ \AA}$	Prism, colourless
$\beta = 105.963 (1)^\circ$	$0.20 \times 0.18 \times 0.15 \text{ mm}$
$V = 661.37 (4) \text{ \AA}^3$	

Data collection

Siemens SMART CCD area-detector diffractometer	2018 independent reflections
ω scans	1622 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.090$
5831 measured reflections	$\theta_{\text{max}} = 30.6^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2 + 0.65P]$
$R[F^2 > 2\sigma(F^2)] = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.138$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.46$	$\Delta\rho_{\text{max}} = 0.61 \text{ e \AA}^{-3}$
2018 reflections	$\Delta\rho_{\text{min}} = -0.64 \text{ e \AA}^{-3}$
64 parameters	
H-atom parameters constrained	

H atoms were positioned geometrically, with C—H = 0.97 Å, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1996); software used to prepare material for publication: *SHELXL97*.

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