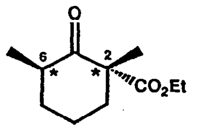
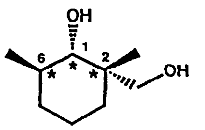
(±)-(1) with $LiAlH_4$ (Sugai, Kakeya, Ohta, Morooka & Ohba, 1989). The relative configuration of C(2) and C(6) revealed in the present study is in agreement with the assignment based on the ^1H NMR spectra (Weiss, 1978). The absolute configuration around C(2) in (−)-(1) was determined to be *R* by converting it to (−)-deoxypodocarpic acid methyl ester (Sugai *et al.*, 1989).



(2*R**,6*R**)-(±)-(1)



(1*S**,2*S**,6*R**)-(±)-(2)

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Structure of a Hexahydrodithia[3.3]paracyclophane Involving *trans* Bonds to the Cyclohexane Ring

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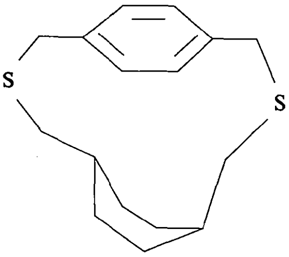
Abstract. 4,5,6,7,8,9-Hexahydro-2,11-dithia[3.3]paracyclophane, $C_{16}H_{22}S_2$, $M_r = 278{\cdot}5$, monoclinic, $P2_1/n$, $a = 8{\cdot}851$ (1), $b = 15{\cdot}398$ (2), $c = 11{\cdot}590$ (2) Å, $\beta = 108{\cdot}86$ (1)°, $V = 1494{\cdot}8$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}24$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 3{\cdot}2$ cm^{-1}, $F(000) = 600$, $T = 293$ K, $R = 0{\cdot}036$ for 1586 reflections with $F_o^2 > 3\sigma(F_o^2)$. The two —CH_2SCH_2— linkages which bond the phenylene group to the cyclohexyl ring have C—S—C angles of 101·7 (2) and 102·8 (2)°. The cyclohexyl ring is in the twisted boat form with four C—C—C—C torsion angles of 23·7 to 36·1° and two of −58·4 and −63·8°. There are four intramolecular contacts between H atoms and the phenylene ring in the range 2·53 to 2·68 Å which contribute to the 'bent benzene' typical for paracyclophanes: in the aromatic ring the C atoms bonded to the CH_2 groups are displaced 0·057 (2) and 0·065 (3) Å from the plane of the other four C atoms, and the C atoms of these CH_2 groups are 0·303 (3) and 0·358 (3) Å from the same plane.

Experimental. Title compound (I) obtained by the slow addition of *trans*-1,4-bis(mercaptomethyl)cyclohexane and dichloroxylene to basic ethanol using high-dilution techniques. The product was obtained after chromatographic separation on a silica-gel column. The colorless data crystal 0·30 × 0·30 × 0·40 mm was mounted with epoxy on a glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell determined from least-squares analysis of



(I)

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Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters*

The thermal parameters are of the form $B = 8\pi^2U$ and $B_{eq} = \frac{8}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B/B_{eq}(Å^2)
S(1)	0·44264 (9)	0·03248 (5)	0·14874 (7)	4·36 (2)
S(2)	0·49127 (9)	0·44309 (5)	0·30920 (7)	4·25 (2)
C(1)	0·4312 (3)	0·0282 (2)	0·3036 (3)	4·17 (7)
C(2)	0·4229 (3)	0·1162 (2)	0·3554 (2)	3·04 (6)
C(3)	0·2938 (3)	0·1709 (2)	0·3046 (2)	3·58 (6)
C(4)	0·3001 (3)	0·2574 (2)	0·3351 (2)	3·55 (6)
C(5)	0·4359 (3)	0·2926 (2)	0·4202 (2)	3·15 (6)
C(6)	0·5579 (3)	0·2362 (2)	0·4792 (2)	3·26 (6)
C(7)	0·5524 (3)	0·1496 (2)	0·4474 (2)	3·21 (6)
C(8)	0·4559 (3)	0·3888 (2)	0·4388 (3)	4·25 (7)
C(9)	0·6591 (3)	0·3821 (2)	0·2946 (2)	3·10 (6)
C(10)	0·6609 (3)	0·3702 (2)	0·1644 (2)	3·15 (6)
C(11)	0·8076 (3)	0·3171 (2)	0·1667 (2)	3·71 (6)
C(12)	0·7986 (3)	0·2233 (2)	0·2072 (2)	3·36 (6)
C(13)	0·6288 (3)	0·1869 (2)	0·1715 (2)	3·34 (6)
C(14)	0·5258 (4)	0·2304 (2)	0·0570 (3)	4·41 (8)
C(15)	0·5096 (3)	0·3273 (2)	0·0777 (3)	3·95 (7)
C(16)	0·6302 (3)	0·0883 (2)	0·1699 (3)	3·92 (7)
H(1*A*)	0·341 (3)	−0·003 (2)	0·293 (2)	4·6 (6)*
H(1*B*)	0·525 (3)	−0·006 (2)	0·352 (2)	5·5 (7)*
H(3)	0·205 (3)	0·148 (1)	0·252 (2)	3·6 (6)*
H(4)	0·219 (2)	0·293 (1)	0·299 (2)	2·9 (5)*
H(6)	0·646 (2)	0·256 (1)	0·534 (2)	2·5 (5)*
H(7)	0·638 (2)	0·113 (1)	0·484 (2)	2·9 (5)*
H(8*A*)	0·551 (3)	0·402 (2)	0·508 (2)	4·3 (6)*
H(8*B*)	0·365 (3)	0·416 (2)	0·444 (2)	5·2 (7)*
H(9*A*)	0·748 (2)	0·411 (1)	0·340 (2)	2·9 (5)*
H(9*B*)	0·657 (2)	0·327 (1)	0·334 (2)	2·1 (5)*
H(10)	0·671 (3)	0·426 (1)	0·134 (2)	3·3 (6)*
H(11*A*)	0·812 (3)	0·315 (2)	0·085 (2)	3·9 (6)*
H(11*B*)	0·904 (3)	0·345 (1)	0·224 (2)	3·4 (6)*
H(12*A*)	0·861 (3)	0·187 (2)	0·177 (2)	3·8 (6)*
H(12*B*)	0·846 (3)	0·215 (2)	0·295 (2)	4·1 (6)*
H(13)	0·577 (3)	0·208 (1)	0·236 (2)	4·0 (6)*
H(14*A*)	0·593 (4)	0·219 (2)	−0·013 (3)	9 (1)*
H(14*B*)	0·418 (3)	0·204 (2)	0·029 (2)	6·0 (8)*
H(15*A*)	0·482 (3)	0·360 (2)	0·002 (2)	5·2 (7)*
H(15*B*)	0·419 (3)	0·335 (2)	0·110 (2)	5·6 (7)*
H(16*A*)	0·704 (3)	0·067 (2)	0·240 (2)	5·1 (7)*
H(16*B*)	0·663 (3)	0·073 (2)	0·104 (2)	6·07 (7)*

* Atoms refined isotropically.

Table 2. *Selected bond distances* (Å), *angles* (°) *and torsion angles* (°) *with e.s.d.'s in parentheses*

S(1)	C(1)	1·831 (3)	C(1)	S(1)	C(16)	102·8 (2)
S(1)	C(16)	1·815 (3)	C(8)	S(2)	C(9)	101·7 (2)
S(2)	C(8)	1·832 (3)	S(1)	C(1)	C(2)	112·8 (2)
S(2)	C(9)	1·811 (3)	S(1)	C(16)	C(13)	117·7 (2)
C(1)	C(2)	1·493 (4)	S(2)	C(8)	C(5)	112·2 (2)
C(2)	C(3)	1·389 (4)	S(2)	C(9)	C(10)	115·2 (2)
C(2)	C(7)	1·387 (4)	C(1)	C(2)	C(3)	121·4 (3)
C(3)	C(4)	1·374 (4)	C(1)	C(2)	C(7)	120·7 (3)
C(4)	C(5)	1·394 (4)	C(3)	C(2)	C(7)	117·5 (3)
C(5)	C(6)	1·382 (4)	C(2)	C(3)	C(4)	121·3 (3)
C(5)	C(8)	1·499 (4)	C(3)	C(4)	C(5)	120·9 (3)
C(6)	C(7)	1·380 (4)	C(4)	C(5)	C(6)	117·6 (3)
C(9)	C(10)	1·526 (4)	C(4)	C(5)	C(8)	121·4 (3)
C(13)	C(16)	1·518 (4)	C(6)	C(5)	C(8)	120·8 (3)
C(Cy)	C(Cy)	1·502 (4)–1·539 (4)	C(5)	C(6)	C(7)	121·4 (3)
			C(2)	C(7)	C(6)	120·9 (3)
C	H	0·89 (3)–1·16 (4)	C(9)	C(10)	C(15)	114·3 (2)
			C(9)	C(10)	C(11)	109·1 (2)
H(9*B*)	C(5)	2·53 (2)	C(12)	C(13)	C(16)	110·9 (2)
H(9*B*)	C(6)	2·56 (2)	C(14)	C(13)	C(16)	116·1 (3)
H(13)	C(2)	2·64 (2)	C(Cy)	C(Cy)	C(Cy)	109·7 (3)–114·6 (3)
H(13)	C(7)	2·68 (3)	S,C,H	C(tetra.)	H	102 (2)–113 (2)
			C	C(trig.)	H	118 (2)–121 (2)

C(10)	C(11)	C(12)	C(13)	+31·4 (5)	C(13)	C(14)	C(15)	C(10)	+36·1 (5)
C(11)	C(12)	C(13)	C(14)	+29·0 (5)	C(14)	C(15)	C(10)	C(11)	+23·7 (5)
C(12)	C(13)	C(14)	C(15)	−63·8 (5)	C(15)	C(10)	C(11)	C(12)	−58·4 (5)

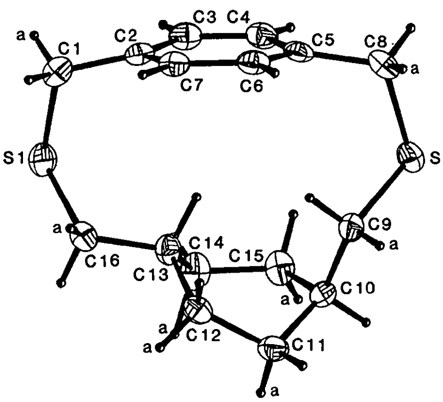

Fig. 1. *ORTEP* (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

angle data for 25 reflections with $16 < 2\theta < 20°$. Absorption correction based on ψ scans varied from 0·98 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $-10 < h < 10$, $0 < k < 18$, $0 < l < 13$. Three standard reflections (071; $\bar{4}22$; 134) indicated crystal decomposition of less than 0·5% over 23·6 h of data collection. 2752 reflections measured, 2621 unique (R_{int} = 0·035), 1034 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma_{cs}^2(I) + (0{\cdot}04I)^2$; $\sigma_{cs}(I)$ is standard deviation of I based on counting statistics. The $10\bar{1}$ reflection was not used in the refinement because of evidence of extinction. S-atom positions were determined by an analysis of the Patterson map. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. H atoms were refined with isotropic thermal parameters and non-H atoms were refined anisotropically for a total of 251 parameters. R = 0·036, wR = 0·042, GOF = 1·3, where non-Poisson $w^{-1} = [\sigma^2(I) + (0{\cdot}04I)^2]/4F^2$. Final $(\Delta/\sigma)_{\max} < 0{\cdot}2$, $\rho_{\max} = 0{\cdot}28\,(5)$ and $\rho_{\min} = -0{\cdot}24\,(5)$ e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atomic coordinates and isotropic thermal parameters and Table 2 gives intramolecular bond distances and angles, H—phenylene contacts, and torsion angles for the cyclohexane ring. Fig. 1 shows the molecule with the numbering scheme.

Related literature. The structure of an isomer of the title compound with *cis* bonds to a cyclohexane ring in the chair form has been reported earlier (Swepston, Lin, Hawkins, Humphrey, Siegel & Cordes,

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* Tables of anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52225 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1990). C**46**, 172–173

Structure of the zeolite theta-1. Redetermination using single-crystal synchrotron-radiation data.

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Abstract

Single-crystal synchrotron-radiation data have been recorded, using an area detector, for a crystal of dimensions 4 × 8 × 125 μm, composition SiO_2, and the structure redetermined. $R = 0{\cdot}116$ for 394 reflections with $F > 6\sigma(F)$. The unit cell, and the framework of SiO_4 tetrahedra, sharing corners, with channels parallel to **c**, previously established from powder diffraction data by Highcock, Smith & Wood [*Acta Cryst.* (1985), C**41**, 1391–1394] is confirmed, but atom positions (*z* parameters) are revised by 0·1–2 Å. $M_r = 60{\cdot}1$, $Cmc2_1$, $a = 13{\cdot}836\,(3)$, $b = 17{\cdot}415\,(4)$, $c = 5{\cdot}042\,(1)$ Å, $V = 1215$ Å^3, $Z = 24$, $\lambda = 0{\cdot}914$ Å.

Introduction

For the zeolite theta-1, large enough crystals for single-crystal data measurement with a conventional X-ray source were not obtainable so structure determination was attempted from X-ray powder diffraction data (Highcock, Smith & Wood, 1985). The high intensity of a synchrotron source (SRS) and an area detector diffractometer have now allowed single-crystal data collection, from which the structure has been redetermined.

Experimental

Needle crystals of theta-1 were of uniform size, *ca* 4 × 8 × 125 μm. The Enraf–Nonius 'FAST' area detector diffractometer on workstation 9.6 at SERC Daresbury Laboratory was used, with the SRS running at 2 GeV, 150 mA. The procedures for data collection and processing using *MADNES* software (Messerschmidt & Pflugrath, 1987) were very similar to those described for piperazine silicate (Andrews *et al.*, 1988). 100° of data were recorded about each of two rotation axes in the crystal, at 45° to each other. The first set alone included 785 observations, which merged to 331 independent reflections with a merging *R* (on intensity) of 0·06; the crystal rocking width was 2°. For the second orientation the rocking width appeared to be 10°; addition of these 463 observations to the earlier set yielded 486 independent reflection intensities with a merging *R* of 0·08, to a resolution of 1 Å.

The primitive monoclinic unit cell found initially with $a = 10{\cdot}944\,(20)$, $b = 10{\cdot}970\,(24)$, $c = 4{\cdot}958\,(10)$ Å, $\alpha = \beta = 90°$, $\gamma = 76{\cdot}615\,(86)°$ and assuming $\lambda = 0{\cdot}90\,(1)$ Å, was converted into an orthorhombic cell; the latter is in agreement with that of Highcock, Smith & Wood (1985), $a = 13{\cdot}836\,(3)$, $b = 17{\cdot}415\,(4)$, $c = 5{\cdot}042\,(1)$ Å, assuming $\lambda = 0{\cdot}914$ Å. Since there was some uncertainty in the precise value of the wavelength used on workstation 9.6, the latter cell dimensions and wavelength were used in the analysis. The systematic absences observed for the orthorhombic cell are also in agreement with space group $Cmc2_1$, 24 SiO_2 per unit cell, chosen by Highcock, Smith & Wood.

The structure was solved with the direct-methods facility of *SHELX*86 (Sheldrick, 1986) and refined (*SHELX*76; Sheldrick, 1976) with anisotropic vibration parameters for

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