Acta Chemica Scandinavica A 29 (1975) 436—442

Structures of Linear Multisulfur Systems. IX. The Crystal and Molecular Structure of 2,6-Bis(*p*-methoxyphenyl-1,2-dithiole-3-ylidene)cyclohexanethione Carbon Disulfide Solvate, $C_{26}H_{22}O_2S_5 \cdot \frac{1}{2}CS_2$

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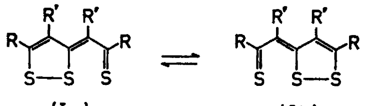
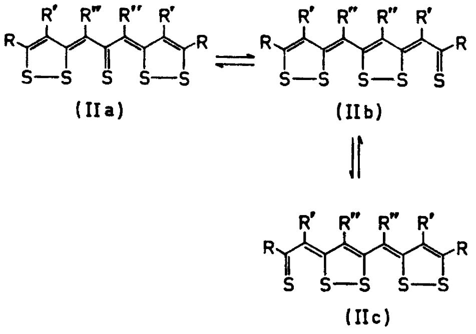
Crystals of the title compound, $C_{26}H_{22}O_2S_5 \cdot \frac{1}{2}CS_2$, are triclinic, space group $P\bar{1}$, with $Z=2$ in a unit cell of dimensions $a=9.871(5)$ Å, $b=11.705(2)$ Å, $c=10.918(1)$ Å, $\alpha=90.84(1)°$, $\beta=94.32(1)°$, $\gamma=90.58(1)°$. 5760 unique reflections were recorded on an off-line four-circle diffractometer. The structure was solved by Patterson synthesis, and refined by full-matrix least-squares technique to an R of 0.049. The sulfur atoms of the 5-sulfur molecule are approximately collinearly arranged. The S–S bonds are all partial bonds with bond lengths in the region between a single bond and van der Waals distance. Although the molecule is symmetrically substituted, the sulfur row shows a pronounced deviation from twofold symmetry with distances: S(1)–S(2) = 2.113 Å, S(2)–S(3) = 2.626 Å, S(3)–S(4) = 2.396 Å, S(4)–S(5) = 2.271 Å. The carbon disulfide molecule is disordered around a centre of symmetry.

In a symmetrically substituted thiathiophthene the S–S bonds are expected to be of equal length according to the simple valence bond resonance.


(Ia) (Ib)

A linear five-sulfur compound may be considered as two fused thiathiophthene molecules. Using the same simple valence bond formalism as above, the two central S–S bond lengths would be expected to be longer than the terminal ones.


(IIa) (IIb) (IIc)

It might be interesting to find out if the overall length of the five-sulfur sequence is equal to twice the chain length in thiathiophthenes, and if any of the three canonical forms are predominant.

Previously the structure of one symmetrically substituted five-sulfur compound has been determined.[1] The sulfur sequence of the molecule is symmetrical within experimental error. A comparison of the S–C bond lengths indicates that IIa is the more dominant resonance form. Furthermore, half the overall chainlength of 4.76 Å is significantly longer than the S···S···S length in any of the thiathiophthene structures reported.

EXPERIMENTAL

The compound was synthesized by Stavaux and Lozac'h.[2,3] Very dark bluish crystals grew from a carbon disulfide solution by slow

Table 1a. Atomic coordinates and anisotropic thermal parameters with the corresponding standard deviations, referring to the last decimal places, listed in parentheses. Values for coordinates are multiplied by 10^5, thermal parameters by 10^4. The anisotropic temperature factors are defined by: $T_i = \exp\ [-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{23}klb^*c^* + 2U_{13}hla^*c^*)]$.

Atom	X/a	Y/b	Z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
S(1)	−6845(7)	70421(6)	4786(6)	392(4)	541(4)	351(3)	146(3)	69(3)	94(3)
S(2)	1567(6)	61541(6)	20211(6)	310(3)	487(4)	314(3)	87(3)	21(3)	76(2)
S(3)	12616(7)	51144(7)	39582(6)	329(3)	637(5)	317(3)	144(3)	25(3)	104(3)
S(4)	22169(6)	42872(6)	58184(6)	306(3)	561(4)	295(3)	64(3)	−7(3)	88(2)
S(5)	31572(7)	35064(7)	75703(6)	328(4)	712(5)	315(3)	105(3)	65(3)	61(3)
O(1)	2225(25)	83394(20)	−52971(18)	841(16)	701(15)	401(11)	86(12)	185(10)	33(11)
O(2)	83465(22)	3081(20)	96249(20)	615(14)	778(16)	616(13)	281(12)	176(12)	−12(11)
C(1)	5914(25)	67070(22)	−4601(22)	335(13)	398(14)	314(12)	−14(11)	−10(10)	52(10)
C(2)	15834(25)	60389(23)	444(22)	320(13)	483(15)	318(12)	32(11)	−3(11)	75(10)
C(3)	15662(24)	56591(22)	12826(21)	304(12)	403(14)	303(12)	10(10)	−27(10)	65(10)
C(4)	25561(24)	49842(22)	18570(21)	296(12)	441(14)	282(12)	28(11)	−34(10)	56(9)
C(5)	25530(24)	46808(22)	31178(21)	283(12)	421(14)	302(12)	12(10)	−38(10)	74(9)
C(6)	36050(24)	40579(21)	37403(21)	295(12)	404(14)	310(12)	30(10)	−21(10)	63(10)
C(7)	35769(24)	38318(21)	49913(21)	303(12)	383(14)	320(12)	23(10)	−23(10)	57(10)
C(8)	46100(25)	32238(23)	56761(22)	334(13)	500(16)	334(13)	79(12)	11(11)	54(10)
C(9)	45516(25)	29911(23)	68874(22)	315(13)	463(15)	349(13)	5(11)	5(11)	40(10)
C(10)	4893(26)	71604(21)	−17145(22)	368(13)	363(14)	343(13)	−17(11)	31(10)	35(10)
C(11)	−6160(28)	77983(27)	−21405(26)	412(15)	668(20)	434(15)	117(14)	110(14)	104(12)
C(12)	−7477(31)	82116(28)	−33277(28)	515(18)	630(20)	488(17)	125(15)	159(14)	2(14)
C(13)	2456(32)	79906(24)	−41092(25)	612(19)	420(16)	361(14)	−25(14)	59(12)	55(13)
C(14)	13746(32)	73708(26)	−36971(25)	585(18)	587(19)	384(15)	108(15)	64(13)	149(13)
C(15)	14869(29)	69546(25)	−25219(25)	468(16)	544(17)	386(14)	119(13)	70(12)	98(12)
C(16)	−10004(40)	88228(32)	−58065(30)	874(27)	749(24)	512(19)	−32(20)	198(17)	−116(18)
C(17)	37283(24)	45817(24)	11461(21)	312(13)	639(18)	277(12)	92(12)	−11(12)	65(10)
C(18)	43610(27)	35118(25)	16753(23)	399(14)	588(17)	344(13)	132(13)	−72(12)	88(11)
C(19)	47786(26)	36534(25)	30350(23)	370(14)	595(17)	323(12)	163(13)	25(12)	94(10)
C(20)	55489(26)	23120(23)	76234(22)	347(13)	450(15)	341(12)	6(11)	9(11)	11(10)
C(21)	52846(29)	19513(26)	87864(25)	411(15)	581(18)	387(14)	92(13)	63(13)	86(12)
C(22)	61828(32)	12751(28)	94876(27)	527(18)	634(20)	407(15)	102(15)	150(14)	63(13)
C(23)	73836(30)	9579(25)	90284(26)	473(16)	461(16)	469(15)	84(13)	35(13)	−30(13)
C(24)	76776(31)	13251(28)	78697(28)	407(16)	689(21)	519(17)	144(15)	69(15)	78(13)
C(25)	67883(29)	19981(28)	71947(26)	395(15)	675(20)	386(14)	88(14)	96(14)	75(12)
C(26)	81662(45)	49(36)	108528(36)	730(26)	540(22)	618(22)	119(20)	170(18)	−80(18)

Table 1b. Coordinates and isotropic thermal parameters for CS_2 and hydrogen atoms with corresponding standard deviations in parentheses. Values for coordinates are multiplied by a factor of 10^4, and thermal parameters by 10^3. Temperature factors are defined by $T_i = \exp[-8\pi^2 U(\sin^2\theta)/\lambda^2]$.

Atom	*X/a*	*Y/b*	*Z/c*	*U*	Atom	*X/a*	*Y/b*	*Z/c*	*U*
S(61)	4372(6)	9549(4)	6440(5)	150(2)	H(172)	4418(27)	5259(23)	1184(23)	44(8)
S(62)	5678(5)	10309(4)	4150(5)	144(2)	H(181)	3726(27)	2843(23)	1506(24)	46(8)
C(27)	5081(14)	9883(11)	5357(9)	79(3)	H(182)	5169(26)	3277(22)	1214(23)	42(7)
H(2)	2285(27)	5788(22)	−380(23)	44(8)	H(191)	5116(26)	2963(22)	3374(23)	40(7)
H(8)	5389(28)	2895(23)	5253(24)	47(8)	H(192)	5533(30)	4337(25)	3199(27)	59(9)
H(11)	−1313(31)	7940(26)	−1620(27)	60(9)	H(21)	4466(31)	2186(25)	9127(26)	49(8)
H(12)	−1563(31)	8675(26)	−3528(27)	63(9)	H(22)	5937(36)	995(30)	10256(33)	78(11)
H(14)	2040(27)	7196(23)	−4256(24)	47(8)	H(24)	8602(32)	1112(26)	7595(27)	53(8)
H(15)	2323(31)	6487(26)	−2312(27)	60(9)	H(25)	7086(30)	2294(25)	6429(28)	54(9)
H(161)	−1222(33)	9472(29)	−5451(30)	74(10)	H(261)	8112(41)	554(36)	11405(37)	87(14)
H(162)	−886(42)	8960(35)	−6634(38)	107(14)	H(262)	8954(42)	−523(35)	11092(35)	99(13)
H(163)	−1832(41)	8352(35)	−5749(36)	105(14)	H(263)	7331(40)	−479(34)	10921(33)	83(13)
H(171)	3425(28)	4456(23)	311(24)	48(8)					

evaporation at 5 °C. The crystal used throughout the data collection had dimensions 0.69 mm × 0.41 mm × 0.16 mm and was mounted along its longest dimension which is parallel to the [101] vector. Weissenberg and precession photographs showed that the space group was triclinic. Unit cell dimensions were derived from diffractometer measurement of 2θ values for 21 reflections using Mo$K\alpha$ radiation ($\lambda_{\alpha 1} = 0.70926$ Å). The density as measured by flotation in a KI-water solution is 1.47 g cm^{-3}. This does not agree with the density calculated for two $C_{26}H_{22}O_2S_5$ molecules (later referred to as "5S" molecules) per unit cell. It is likely that CS_2 has cocrystallized. The density calculated for two "5S" molecules and one CS_2 molecule per unit cell is 1.49 g cm^{-3}.

5760 unique reflections with $2\theta \leq 55°$ were measured according to the experimental procedure described elsewhere.[4] No significant deterioration was detected during the course of data collection. The crystals do, however, disintegrate over a period of approximately six months at room temperature.

947 of the reflections were less than twice the estimated error in measurement, these reflections were given the threshold value of $2\sigma_c$ and were not included in the refinement unless $|F_c| > |F_{\text{threshold}}|$. Standard deviations in intensities were calculated as $\sigma_I = k[\sigma_c^2 + (0.02\ N_{\text{net}})^2]^{\frac{1}{2}}$, where k is the appropriate scale factor, σ_c is the estimated error due to counting statistics and N_{net} is the net count, ($N_{\text{scan}} - N_{\text{background}}$), of the reflection. Standard deviations in structure factors were calculated as $\sigma_F = \sigma_I / 2(I\ Lp)^{\frac{1}{2}}$. The data were corrected for Lorentz and polarization effects according to standard procedures and for absorption using the method of Coppens *et al.*[5]

CRYSTAL DATA

$C_{26}H_{22}O_2S_5 \cdot \frac{1}{2}CS_2$; M.W. = 564.95. Crystal system triclinic, space group $P\bar{1}$ (or $P1$). Cell dimensions: $a = 9.871(5)$ Å, $b = 11.705(2)$ Å, $c = 10.918(1)$ Å, $\alpha = 90.84(1)°$, $\beta = 94.32(1)°$, $\gamma = 90.58(3)°$.

$V = 1258$ Å^3, $Z = 2$.

$D_x = 1.492$ g cm^{-3}, $D_m = 1.47$ g cm^{-3}.

$\mu_{\text{Mo}K\alpha} = 4.9$ cm^{-1}.

Table 2. Bond angles, not involving hydrogen atoms, with the corresponding standard deviations, referring to the last demical places, in parentheses.

Angle	(°)	Angle	(°)
S(2) – S(1) – C(1)	95.63(9)	C(7) – C(8) – C(9)	122.7(2)
S(1) – S(2) – S(3)	177.91(3)	S(5) – C(9) – C(8)	116.1(2)
S(1) – S(2) – C(3)	94.55(8)	S(5) – C(9) – C(20)	118.8(2)
S(3) – S(2) – C(3)	85.17(8)	C(8) – C(9) – C(20)	125.1(2)
S(2) – S(3) – S(4)	175.61(3)	C(1) – C(10) – C(11)	121.1(2)
S(2) – S(3) – C(5)	89.49(8)	C(1) – C(10) – C(15)	121.5(2)
S(4) – S(3) – C(5)	93.95(8)	C(11) – C(10) – C(15)	117.4(2)
S(3) – S(4) – S(5)	179.05(3)	C(10) – C(11) – C(12)	122.0(2)
S(3) – S(4) – C(7)	87.66(8)	C(10) – C(12) – C(13)	119.5(2)
S(5) – S(4) – C(7)	91.50(8)	C(12) – C(13) – C(14)	119.6(2)
S(4) – S(5) – C(9)	94.12(9)	C(12) – C(13) – O(1)	124.9(2)
S(1) – C(1) – C(2)	115.5(2)	C(14) – C(13) – O(1)	115.6(2)
S(1) – C(1) – C(10)	117.7(2)	C(13) – C(14) – C(15)	120.4(2)
C(2) – C(1) – C(10)	126.7(2)	C(10) – C(15) – C(14)	121.1(2)
C(1) – C(2) – C(3)	121.2(2)	C(13) – O(1) – C(16)	117.1(2)
S(2) – C(3) – C(2)	113.1(2)	C(4) – C(17) – C(18)	112.0(2)
S(2) – C(3) – C(4)	122.8(2)	C(17) – C(18) – C(19)	111.8(2)
C(2) – C(3) – C(4)	124.1(2)	C(6) – C(19) – C(18)	111.4(2)
C(3) – C(4) – C(5)	122.6(2)	C(9) – C(20) – C(21)	120.8(2)
C(3) – C(4) – C(17)	119.4(2)	C(9) – C(20) – C(25)	122.3(2)
C(5) – C(4) – C(17)	118.0(2)	C(21) – C(20) – C(25)	117.0(2)
S(3) – C(5) – C(4)	119.8(2)	C(20) – C(21) – C(22)	122.2(2)
S(3) – C(5) – C(6)	116.9(2)	C(21) – C(22) – C(23)	119.4(2)
C(4) – C(5) – C(6)	123.3(2)	C(22) – C(23) – C(24)	119.3(2)
C(5) – C(6) – C(7)	120.7(2)	C(22) – C(23) – O(2)	125.1(2)
C(5) – C(6) – C(19)	119.0(2)	C(24) – C(23) – O(2)	115.7(2)
C(7) – C(6) – C(19)	120.4(2)	C(23) – C(24) – C(25)	120.6(3)
S(4) – C(7) – C(6)	120.9(2)	C(20) – C(25) – C(24)	121.4(2)
S(4) – C(7) – C(8)	115.6(2)	C(23) – O(2) – C(26)	118.2(3)
C(6) – C(7) – C(8)	123.5(2)		

STRUCTURE DETERMINATION AND REFINEMENT

The intensity distribution clearly suggested a centrosymmetric structure, thus the space group was assumed to be $P\bar{1}$ rather than $P1$. The sulfur atoms of the "5S" molecule were localized from a three dimensional Patterson synthesis. Structure factor calculation based on five sulfur atoms gave an R of 0.57 ($R = \sum||F_o| - |F_c||/\sum|F_o|$). The remaining non-hydrogen atoms of the "5S" molecule were found in two subsequent Fourier maps. An elongated region of electron density with centre of gravity in ($\frac{1}{2}$,1,$\frac{1}{2}$) indicated the presence of a three-atomic molecule with the central atom in the centre of symmetry. Tentatively half a CS_2 molecule was included in the asymmetric unit. During the course of refinement these atoms got abnormal temperature parameters. A difference map indicated that a disordered model would be more appropriate. The model arrived at is one where the CS_2 molecule is slightly displaced from the centre of symmetry, and is situated on alternate sides of the centre in different unit cells throughout the crystal. The occupancy factors of the CS_2 atoms were refined; the values arrived at did not deviate significantly from 0.5. The CS_2 atoms were refined with isotropic, and the remaining atoms with anisotropic thermal parameters in the full-matrix least-squares refinement, minimizing the function $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma_F$. Hydrogen atoms were located from a difference map and were included in the refinement with isotropic temperature factors.

At the end of the refinement it was found that a few strong, low order reflections clearly suffered from extinction. A correction for secondary extinction was thus carried out according to the method of Zachariassen.[6] The extinction coefficient arrived at is 0.89×10^{-6}, the maximum correction in F_{obs} amounting to 26 %.

The refinement converged at a conventional R of 0.049, and a weighted R of 0.050. The standard deviation of an observation of unit weight, $[\sum w(|F_o| - |F_c|)^2/(m-n)]^{\frac{1}{2}}$, is 3.88. Atomic scattering factors used were for sulfur, oxygen, and carbon those of Hanson *et al.*[7] and for hydrogen those of Stewart *et al.*[8]

The final atomic positional and thermal parameters with standard deviations as estimated from the inverse least-squares matrix, are listed in Tables 1 and 2. Lists of observed and calculated structure factors may be obtained from the author.

THERMAL MOTION

The thermal parameters were analyzed in terms of rigid body motion according to the method of Schomaker and Trueblood.[9] The thermal motions of the 14 atoms in "rings" A, B, C, D (Fig. 1) are described reasonably well by the r.b.m. approximation; the r.m.s. difference between observed and calculated U_{ij}'s being 0.0020 Å^2. The axis of largest libration is approximately parallel to the row of sulfur atoms, the angle of libration being 5.4°. Bond distances corrected for rigid body motion were calculated according to

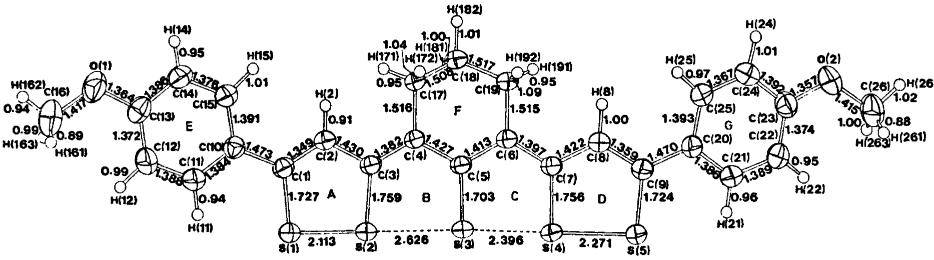


Fig. 1. Bond distances (not corrected for thermal motion) in the „5S" molecule. Standard deviations in S–S, S–C, C–C(O) and C–H bonds are 0.001, 0.003, 0.004, and 0.04 Å, respectively. The thermal ellipsoids are plotted at the 50 % probability level; H atoms are drawn with a fixed radius.

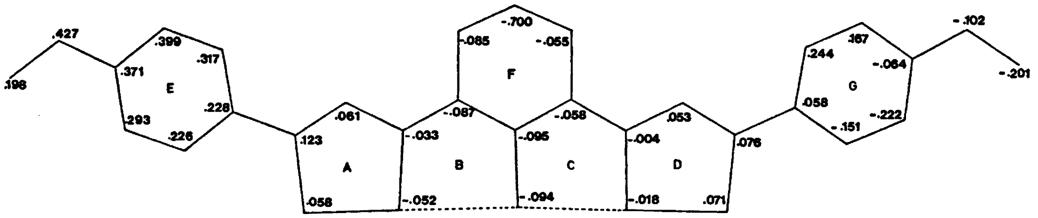


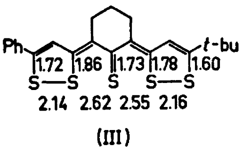
Fig. 2. Atomic deviations from the least-squares plane through rings A+B+C+D.

Cruickshank's method.[10] The corrections in S−S bond lengths amount to only 0.001 Å, and are thus not significant, corrections in C−S bonds are 0.008 Å, while C−C bond length corrections in rings A, B, C, D are in the range 0.001−0.002 Å.

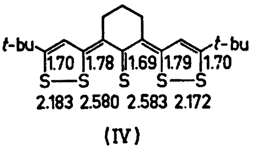
RESULTS AND DISCUSSION

Bond distances are shown in Fig. 1, and the bond angles except those involving hydrogen atoms are listed in Table 2. The five sulfur atoms are almost collinearly arranged, the angles S(1)−S(2)−S(3), S(2)−S(3)−S(4) and S(3)−S(4)−S(5) being 177.91, 175.31, and 179.05°, respectively. Each of the five-membered rings, A, B, C, and D (Fig. 2) is approximately planar. The molecule is, however, slightly but significantly bent around the S(3)−C(5) bond, the angle between the planes through A+B and C+D being 4.4°. A similar geometry has also been observed in the two other five-sulfur compounds studied.[1,11] The four intramolecular sulfur-sulfur distances are all significantly longer than single bonds of 2.10 Å,[12] suggesting partial bonding between all sulfur atoms. The σ-bonding in the sulfur sequence may be formally considered as a 5-centre-6-electron system delocalized across the five atoms. This is quite analogous to the 3-centre-4-electron delocalized σ-bonding proposed for the thiathiophthenes.[13]

In the linear five-sulfur compounds studied previously the feature of partial bonding between the sulfur atoms is also obvious, III, IV.



(III)



(IV)

In the unsymmetrically substituted compound (III) the sulfur sequence shows a small deviation from twofold symmetry; while in the symmetrically substituted compound (IV) the S···S row has twofold symmetry within the experimental error. In the present compound however, the deviation from symmetry in the sulfur sequence is pronounced, although the molecule is symmetrically substituted. There is a sligth difference in conformation between the two halves of the molecule, as the plane of the phenyl group E is twisted 2.5° relative to the plane of ring A; while phenyl group G, is twisted around the C(9)−C(20) bond and bent out of the plane of ring D by 12.0°. It is unlikely, however, that this small difference in twist angles may account for the observed asymmetry in the S···S sequence. On the other hand the intermolecular environment of the two halves of the molecule is quite different. Dithiole ring A is sandwiched between the corresponding ring A′ of a centrosymmetrically related molecule ($-x,1-y,-z$), and the phenyl group G″ of the molecule in position $1-x,1-y,1-z$. The distances between the partially overlapping rings are in the range 3.4−3.5 Å. Dithiole ring D, is partially overlapping the saturated ring F″, the interplanar spacing being 3.7 Å [excluding atom C(18)″]. The central sulfur atom S(3) is situated above the centre of phenylgroup E′ at a distance of 3.65 Å. Furthermore there are intermolecular S···S contacts in the range 3.361 to 3.537 Å between molecules related by a centre of symmetry, $-x,1-y,1-z$. One may notice that while S(1) has only one such contact, S(1)···S(5)‴ = 3.420 Å, S(5) has two contacts, S(5)···S(1)‴ = 3.420 Å and S(5)···S(2)‴ = 3.361 Å.

The mutual interaction between the π-systems of the aromatic rings A, A′, and G″, and between phenyl group E′ and S(3) may produce an overall shift of electron density

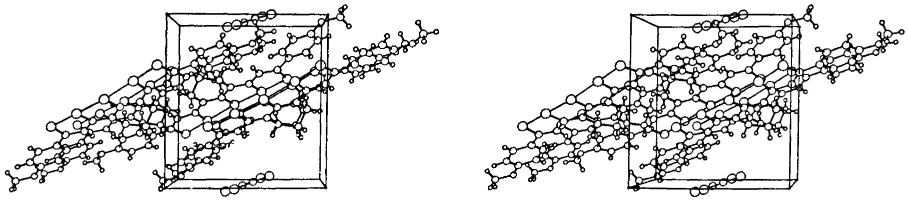


Fig. 3. Stereodrawing showing the packing of molecules in the crystal lattice. CS_2 molecules are drawn at both of the fractional sites. The a^*-axis is pointing towards the viewer, the b-axis is running vertically, top to bottom, and c horisontally, left to right. Figs. 1 and 3 were drawn by use of the ORTEP program.[17]

in the sulfur sequence from S(1) towards S(5). Theoretical calculations have shown that such a redistribution of charge is consistent with a shortening of S(1) – S(2) and a lengthening of S(4) – S(5).[14]

The variation in S – S bond lengths in the molecule is consistent with a predominance of the canonical form IIa. The asymmetry indicates that resonance form IIb is energetically more favourable than IIc due to intra- and intermolecular forces. The overall chain length is significantly shorter in this five-sulfur compound than in the two previous ones (III, IV). However, the shortening is mainly concentrated in the S(3)···S(5) portion of the chain.

There are no short contacts between the "5S" molecule and the cocrystallized CS_2 molecule. Due to the disorder, the dimensions in CS_2 are not accurately determined, C(27) – S(61) = 1.47(2) Å, C(27) – S(62) = 1.57(2) Å, ∠S – C – S = 173(1)°. The average C – S distance agrees reasonably well with the distance of 1.553 Å as determined by IR and microwave spectroscopy.[15,16]

The packing of the molecules in the unit cell is illustrated in the stereodrawing, Fig. 3.

Acknowledgement. The author is indebted to Dr. M. Stavaux for supplying a sample of the compound.

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Received December 6, 1974.