

a real attraction between H(8) and S(1') (which lie almost in the plane of the backbone of molecule 1), it would explain why the side chain in molecule 1 is not in the expected staggered configuration but twisted about the C(2)–C(5) bond.

The hydrogen bonds are listed in Table 6 and the surroundings of each sulphur atom are shown in Fig. 6. It seems that the packing of the molecule is dominated by the tendency to form N–H---S hydrogen bonds. As seen, the H---S distances are 2.47–2.68 Å in an N–H---S hydrogen bond. These values could possibly be shortened by about 0.15 Å if it is assumed that the N–H distance should be nearly 1.00 Å and that the N---S distance has been accurately measured. The H---S distance of C(5)–H(8)---S(1') is only 2.72 Å. One hesitates to postulate a hydrogen bond from a carbon atom, especially to a sulphur atom, but some electrostatic interaction with a slight positive charge on the hydrogen atom seems to be consistent with our results.

This tendency to association, between two molecules of opposite absolute configuration (but not across a center of symmetry), may explain why there are two molecules in the asymmetric unit. It involves molecule 1 (at x, y, z as listed in Table 2), which is associated with molecule 2 (at $-x, y - \frac{1}{2}, -\frac{1}{2} - z$ with respect to parameters for the second molecule listed in Table 2) through the contact C(5)–H(8)---S(1') and through the hydrogen bond N(6)–H(6)---S(1') to form a ten-membered ring (including hydrogen atoms).

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The Crystal Structure of the β -Form of 2,4,6-Trimethyl-1,3,5-Trithiane, 'Trithioacetaldehyde'*

BY G. VALLE, V. BUSETTI AND M. MAMMI

*Centri di Struturistica e di Chimica delle Macromolecole del C.N.R.,
Istituto di Chimica Organica dell'Università, 35100 Padova, Italy.*

AND G. CARAZZOLO

Centro Ricerche Resine, Montecatini-Edison, 21053 Castellanza, Varese, Italy

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The crystal structure of the β -form of 2,4,6-trimethyl-1,3,5-trithiane, *i.e.* trithioacetaldehyde, m.p. 126°C, has been determined by X-ray photographic three-dimensional data and refined by full-matrix least-squares ($R=0.078$). The crystals are orthorhombic, space group $P2_12_12_1$, $a=14.668$, $b=13.438$, $c=4.751$ Å, $Z=4$. The molecule is a six-membered ring in the chair conformation, with three methyl groups in the equatorial positions and with C–S=1.80 Å, C–S–C=98.4°, S–C–S=115.3°.

Introduction

Two isomeric 2,4,6-trimethyl derivatives of 1,3,5-trithiane are described in the literature (Klinger, 1878;

Wörner, 1896): an α form, with melting point 101°C, and a β form, with melting point 126°C. They are reported to differ from each other in having a chair ring with two and three methyl groups respectively in equatorial positions, as indicated by chemical evidence (Chattaway & Kellett, 1930), by electron-diffraction

* Work carried out with CNR aid.

Table 1. Observed and calculated structure factors

Columns are H_c , $|10 \times F_{obs}|$, $|10 \times F_{calc}|$, $10 \times A_{calc}$, $10 \times B_{calc}$. Unobserved reflexions, which were given half the minimum observed intensity, are marked by an asterisk. In parentheses, reflexions not observed for mechanical reasons.

Table with multiple columns containing numerical data for structure factors. The table is organized into a grid with sub-headers for different hkl reflections. Each row contains values for H_c, |10 x F_obs|, |10 x F_calc|, 10 x A_calc, and 10 x B_calc. Asterisks indicate unobserved reflexions.

investigations (Hassel & Viervoll, 1947) and more recently by nuclear magnetic resonance spectroscopy (Campaigne, Chamberlain & Edwards, 1962).

We have carried out the structure analysis of the higher melting point isomer, the β form, of 2,4,6-trimethyl-1,3,5-trithiane, *i.e.* trithioacetaldehyde, $(\text{CH}_3\text{-CHS})_3$, (TTA). The analysis also provides a comparison with the previously determined structures of 1,3,5-trithiane (Valle, Busetti, Mammi & Carazzolo, 1969) and of other cyclic acetals (Mammi, Carazzolo, Valle, & Del Pra, 1968; Busetti, Del Pra & Mammi, 1969), including the structure of the corresponding selenium analogue, triselenoacetaldehyde, whose preliminary data have already been reported (Credali, Russo, Mortillaro, De Checchi, Valle & Mammi, 1967).

The researches on these compounds are also related to the studies on the polymerization processes that some of them undergo topotactically in the crystalline state (Carazzolo & Mammi, 1967; Mammi, Carazzolo & Valle, 1968, and references therein).

Experimental

Colourless needles of nearly square cross-section and elongated along [001] were obtained by evaporation of n-hexane solutions of TTA (m.p. 126°C). The crystals are orthorhombic, space group $P2_12_12_1$ (D_2^7 , No.19) with $a = 14.668 \pm 0.005$, $b = 13.438 \pm 0.005$, $c = 4.751 \pm 0.004$ Å, $V = 936.5$ Å³, $F.W. = 180.4$, $Z = 4$, $D_m = 1.25$ (by flotation), $D_x = 1.28$ g.cm⁻³, $F(000) = 384$, $\mu_{\text{CuK}\alpha} = 65.1$ cm⁻¹.

The hkl reflexions (with $l = 0$ to 4) from a crystal 0.1 mm thick, were collected in a Weissenberg camera with Cu K α Ni-filtered radiation and the equi-inclination multiple-film technique. The intensities of 602 re-

flexions, out of the 1135 possible in the collected layers, were measured by a recording microdensitometer. Corrections were applied for the non-linear behaviour of the X-ray films used (Ilford, Industrial G) (Mammi, Bardi & Bezzi, 1963; Morimoto & Uyeda, 1963) and for the usual Lorentz and polarization factors. Precession photographs from the same crystal were used for inter-layer scaling.

Determination and refinement of the structure

Comparison of experimental data of TTA and of triselenoacetaldehyde clearly showed that the two compounds are isostructural, a not surprising result (Kitai-gorodskii, 1961a; Mortillaro, Credali, Mammi & Valle, 1965).

The first structure-factor calculation was therefore computed with the atomic fractional coordinates of the selenium compound, though not refined. The R index was 0.29. A three-dimensional F_o synthesis confirmed the structure, indicating some shifts of coordinates, and the refinement was continued by ΔF and differential synthesis (Nardelli, Musatti, Domiano & Andreotti, 1965) with the atomic scattering factors by Forsyth & Wells (1959) and keeping the vibrational parameters constant and equal to 3.0 Å². Convergence was reached after ten cycles, with $R = 0.12$.

The least-squares full-matrix program *ORFLS* by Busing, Martin & Levy (1962), as adapted in *X-ray 63 System* (1965), was then used to refine the coordinates on an IBM 7094/7040 DCS computer; the anisotropic vibrational parameters were also refined. Only the observed reflexions were included, with a unitary weighting scheme, and a fixed contribution from the hydrogen atoms. After two cycles the shifts of both

Table 2. Atomic coordinates and anisotropic vibrational parameters ($\times 10^4$) with *e.s.d.*'s

The expression for the temperature factor is: $\exp[-(\beta_{11}h^2 + \dots + 2\beta_{12}hk + \dots)]$.

	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S(1)	0.2240 (3)	0.2034 (3)	0.1765 (13)	46 (2)	61 (3)	515 (33)	6 (2)	-18 (8)	-3 (10)
S(2)	0.4291 (3)	0.2234 (4)	0.1620 (13)	46 (2)	74 (3)	505 (33)	3 (2)	-22 (9)	-14 (11)
S(3)	0.3137 (3)	0.3934 (3)	0.3895 (10)	49 (2)	65 (3)	284 (19)	-4 (2)	-8 (7)	-10 (8)
C(1)	0.2208 (13)	0.3352 (13)	0.1951 (44)	67 (11)	66 (12)	447 (123)	-1 (10)	9 (34)	-42 (34)
C(2)	0.3273 (14)	0.1890 (13)	-0.0221 (32)	69 (11)	82 (13)	243 (75)	6 (11)	16 (29)	-38 (27)
C(3)	0.4075 (12)	0.3554 (13)	0.1731 (49)	54 (10)	83 (13)	466 (127)	-10 (10)	8 (33)	-20 (40)
C(4)	0.1326 (12)	0.3632 (16)	0.3534 (58)	47 (10)	98 (16)	946 (164)	11 (11)	-50 (44)	-110 (53)
C(5)	0.3370 (15)	0.0749 (13)	-0.1019 (48)	100 (14)	65 (12)	622 (126)	-6 (11)	9 (45)	-38 (38)
C(6)	0.4965 (14)	0.4100 (17)	0.2929 (59)	58 (11)	127 (19)	1192 (183)	-36 (13)	-67 (41)	-155 (59)
				B	Bonding atom				
H(1)	0.219	0.363	-0.017	4.9 Å ²	C(1)				
H(2)	0.320	0.233	-0.210	4.7	C(2)				
H(3)	0.396	0.377	-0.042	5.0	C(3)				
H(4)	0.142	0.326	0.552	6.6	C(4)				
H(5)	0.131	0.443	0.365	6.6	C(4)				
H(6)	0.078	0.329	0.239	6.6	C(4)				
H(7)	0.347	0.033	0.088	6.3	C(5)				
H(8)	0.399	0.066	-0.221	6.3	C(5)				
H(9)	0.276	0.054	-0.213	6.3	C(5)				
H(10)	0.502	0.383	0.505	8.3	C(6)				
H(11)	0.484	0.489	0.300	8.3	C(6)				
H(12)	0.553	0.387	0.163	8.3	C(6)				

positional and vibrational parameters were much less (0.2) than the standard deviations, and the refinement was stopped ($R=0.078$). A ΔF synthesis calculated with the final parameters did not show spurious peaks, the electron density fluctuation being about $\pm 0.15 \text{ e.}\text{\AA}^{-3}$.

Observed and calculated structure factors are listed in Table 1 and final atomic and vibrational parameters, with estimated standard deviations are reported in Table 2. The hydrogen atom coordinates have been calculated assuming tetrahedral carbon valence angles, $\text{C-H}=1.075 \text{ \AA}$ and *trans* conformation of the methyl groups, as also indicated by previous ΔF syntheses.

Results and discussion

The present analysis confirms that the molecule of TTA is a six-membered ring in the chair conformation, with the three methyl groups in the equatorial positions. Bond lengths and valence angles with their estimated standard deviations, calculated according to Ahmed & Cruickshank (1953) and Darlow (1960) respectively, are reported in Table 3 and shown in Fig. 1. The observed differences in the ring bond lengths and angles are

within the error, with average values: C-S , 1.80 \AA ; C-S-C , 98.4 ; S-C-S , 115.3° (1.814 \AA , 98.9 and 114.7° respectively, in the unsubstituted 1,3,5-trithiane, Valle *et al.* 1969). The previously reported values for the ring valence angles, obtained by electron diffraction (Hassel & Viervoll, 1947), were C-S-C , 106.5 and S-C-S , 114.5° for both TTA and 1,3,5-trithiane. These results confirm once more that in this type of cyclic molecule the ring valence angle of the carbon atom is larger than the normal tetrahedral value, corresponding to a smaller value at the hetero-atom. The mean internal rotation angle of the ring bonds is 66° (see Table 4) and the molecular thickness (distance between the sulphur atom and the ring carbon atom planes) is 0.66 \AA , as in 1,3,5-trithiane.

Within the e.s.d.'s the TTA ring exhibits the maximum molecular symmetry $3m$, while some deviations are observed in the three methyl groups. The reported bond lengths to the rings of C(5) and C(6), obtained from the least-squares refinement, are rather large, though the difference from the standard value does not seem to be very significant ($\Delta/\sigma=2$). Shortening was not supported by either the R index or the ΔF synthe-

Table 3. Bond lengths and angles (e.s.d.'s in parentheses)

C(1)-S(1)	1.78 (2) \AA	C(1)-S(1)-C(2)	99.0 (0.9) $^\circ$
S(1)-C(2)	1.80 (2)	C(2)-S(2)-C(3)	97.0 (0.9)
C(2)-S(2)	1.79 (2)	C(1)-S(3)-C(3)	99.3 (0.9)
S(2)-C(3)	1.80 (2)	S(1)-C(1)-S(3)	115.7 (1.0)
C(3)-S(3)	1.79 (2)	S(1)-C(2)-S(2)	114.8 (0.9)
S(3)-C(1)	1.82 (2)	S(2)-C(3)-S(3)	115.6 (1.0)
C(1)-C(4)	1.54 (3)	S(1)-C(1)-C(4)	106.9 (1.3)
C(2)-C(5)	1.59 (3)	S(3)-C(1)-C(4)	106.0 (1.4)
C(3)-C(6)	1.60 (3)	S(1)-C(2)-C(5)	107.8 (1.3)
		S(2)-C(2)-C(5)	107.0 (1.4)
		S(2)-C(3)-C(6)	108.5 (1.3)
		S(3)-C(3)-C(6)	107.0 (1.5)
Other intramolecular values			
S(1)-S(2)	3.02 (1)	S(2)-S(1)-S(3)	60.2 (0.2)
S(2)-S(3)	3.04 (1)	S(1)-S(2)-S(3)	60.3 (0.2)
S(3)-S(1)	3.04 (1)	S(1)-S(3)-S(2)	59.5 (0.2)
C(1)-C(2)	2.71 (3)	C(2)-C(1)-C(3)	59.0 (0.7)
C(2)-C(3)	2.69 (3)	C(1)-C(2)-C(3)	61.2 (0.7)
C(3)-C(1)	2.75 (3)	C(1)-C(3)-C(2)	59.8 (0.7)

Table 4. Intramolecular angles and distances involving the planes:

$\pi_1 = \text{S(1)S(2)S(3)}$	$\pi_2 = \text{C(1)C(2)C(3)}$	$\pi_3 = \text{C(4)C(5)C(6)}$
$\pi_4 = \text{C(1)S(1)C(2)}$	$\pi_5 = \text{S(1)C(2)S(2)}$	$\pi_6 = \text{C(2)S(2)C(3)}$
$\pi_7 = \text{S(2)C(3)S(3)}$	$\pi_8 = \text{C(3)S(3)C(1)}$	$\pi_9 = \text{S(3)C(1)S(1)}$

Angle between the planes

$\pi_1\pi_2 = 1.7^\circ$	$\pi_4\pi_5 = 68.7^\circ$	$\pi_7\pi_8 = 65.1^\circ$
$\pi_2\pi_3 = 1.4$	$\pi_5\pi_6 = 69.1$	$\pi_8\pi_9 = 63.1$
$\pi_1\pi_3 = 3.3$	$\pi_6\pi_7 = 67.8$	$\pi_9\pi_4 = 64.3$

Methyl carbon atom values

Distance of	C(4) from	π_1	0.15 \AA	π_8	0.03 \AA	π_4	0.05 \AA		
	C(5)	π_1	0.44	π_4	0.20	π_6	0.23		
	C(6)	π_1	0.36	π_6	0.21	π_8	0.16		
Angle between	C(4)-C(1) and	π_1	17.7 $^\circ$	π_8	1.2 $^\circ$	π_4	1.9 $^\circ$	π_9	57.9 $^\circ$
	C(5)-C(2)	π_1	9.5	π_4	7.4	π_6	8.3	π_5	55.6
	C(6)-C(3)	π_1	11.0	π_6	7.5	π_8	5.8	π_7	55.2

ses. Larger differences, up to ten times the e.s.d.'s, are observed in the other methyl parameters reported in Table 4. The three radial methyl groups lie between the sulphur atom and the carbon atom planes π_1 and π_2 , but C(4) is much nearer to π_1 than the other two carbon atoms and the C(4)–C(1) bond is inclined at a greater angle to this plane (17.7°). It lies almost exactly on each of the two contiguous C–S–C planes, π_4 and π_8 , *trans* to S(1)–C(2) and S(3)–C(3), unlike the bonds from C(5)

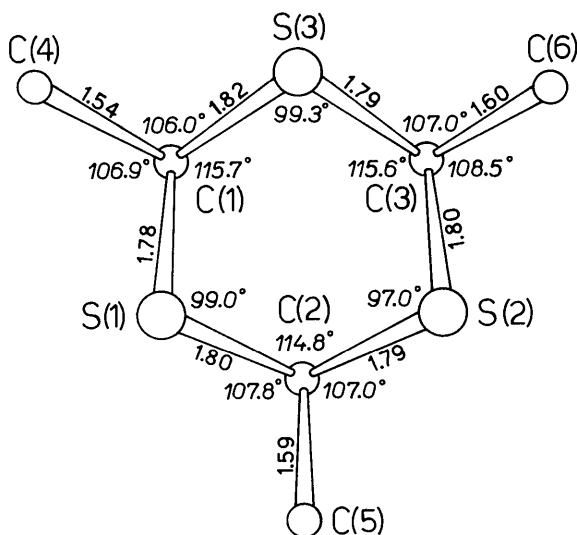


Fig. 1. Bond lengths and angles in 2,4,6-trimethyl-1,3,5-trithiane (TTA).

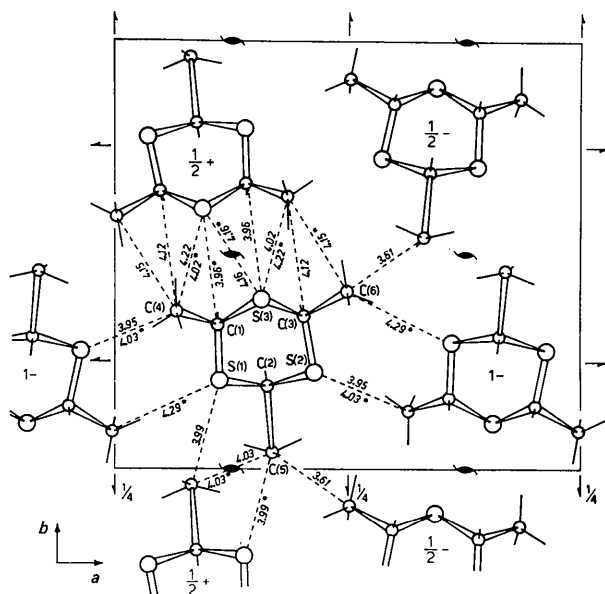


Fig. 2. Molecular packing viewed along the *c* axis, with the intramolecular distances from the molecule at *x, y, z* to the neighbouring molecules, whose *z* coordinates are indicated. The asterisk denotes distances to the same molecule, but shifted by $-c$.

and C(6) which are tilted out of the corresponding C–S–C planes by about 7° and appear to be bent toward the plane of the molecule. This bending is also supported by the values of the external valence angle of the ring carbon atoms, smaller for C(5) and C(6) than for C(4), as estimated from the angle between each C–C bond and the corresponding S–C–S plane (Table 4).

The bending of the C(5)–C(2) and C(6)–C(3) bonds might be caused by the relatively short intermolecular contact of 3.61 \AA , the shortest in the structure, which involves the C(5) and C(6) methyl groups of different molecules, and which otherwise would be further reduced. This molecular approach also includes a C(6)–H(7) contact of 2.9 \AA ; the C(6) atom has its minimum vibration direction along the C(6)–C(5) contact.

All the other non-hydrogen intermolecular distances are larger than 3.9 \AA , as shown in Fig. 2 and Table 5. This indicates a rather loose crystal packing, in agreement with a calculated packing coefficient of 0.68 (Kitaigorodskii, 1961 *b*), and with the values of the vibrational parameters reported in Table 6; the largest values are observed for the methyl carbon atoms, each vibrating mostly in a plane nearly perpendicular to the corresponding C–C bond. It may be noted, however, that some of the reported distances do involve a collinear, or nearly collinear, H atom at the van der Waals distance, as in the case of the S(1)–C(6) and the S(3)–C(2') contacts. The sulphur atom environment consists mainly of H atoms.

Table 5. *Interatomic distances of superimposed molecules*

Only the contacts to the atoms at *x, y, z*–1, indicated by a prime are listed.

C(1)–S(3')	4.14 Å	C(3)–S(3')	4.00
C(2)–S(1')	4.10	C(5)–S(1')	4.18
C(2)–S(2')	4.18	C(5)–S(2')	4.25
C(2)–S(3')	3.92	C(1)–C(4')	4.22

Many intermolecular contacts are observed between superimposed molecules, so that it does not appear impossible, on a geometrical basis, that polymerization along the *c* axis might occur without large steric hindrances by the methyl groups.

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Table 6. Root mean square displacement of atoms along the principal axes of the thermal ellipsoids and angles between these principal axes and the crystallographic directions

	Axis	U_i	θ_{ia}	θ_{ib}	θ_{ic}
S(1)	1	0.2111 Å	32.8°	114.7°	69.9°
	2	0.2369	98.2	141.6	127.2
	3	0.2533	58.5	62.6	135.9
S(2)	1	0.2128	31.8	90.4	58.2
	2	0.2432	63.8	122.4	136.0
	3	0.2667	73.3	32.4	116.9
S(3)	1	0.1777	81.3	82.2	11.7
	2	0.2285	153.3	113.9	78.8
	3	0.2481	115.0	25.3	93.4
C(1)	1	0.2031	93.6	54.8	35.4
	2	0.2618	120.0	136.5	61.7
	3	0.2733	30.2	112.1	70.5
C(2)	1	0.1544	97.7	75.7	16.3
	2	0.2693	140.3	54.2	104.9
	3	0.2857	51.4	39.4	96.4
C(3)	1	0.2271	93.0	76.3	14.0
	2	0.2329	155.9	113.9	87.2
	3	0.2868	113.9	28.0	103.7
C(4)	1	0.2149	14.9	92.1	75.3
	2	0.2498	83.0	143.7	125.4
	3	0.3748	77.0	53.8	140.8
C(5)	1	0.2269	86.6	31.7	58.5
	2	0.2789	100.5	120.4	32.5
	3	0.3321	11.1	98.4	82.8
C(6)	1	0.1609	41.0	58.7	66.5
	2	0.3350	130.9	56.3	59.1
	3	0.4214	92.7	49.6	139.5

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