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The Conformation of Non-Aromatic Ring Compounds XLVI.* The Crystal and Molecular Structure of 2,2′-Dichlorotrimethylene Sulphite

BY JOHANNA W. L. VAN OYEN, R. C. D. E. HASEKAMP, G. C. VERSCHOOR AND C. ROMERS

Organic Chemistry Department, University of Leiden, The Netherlands

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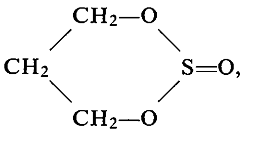
2,2′-Dichlorotrimethylene sulphite crystallizes in the form of monoclinic crystals with space group $P2_1$ and $Z=2$. The unit cell dimensions are $a=6{\cdot}173$, $b=6{\cdot}500$, $c=8{\cdot}986$ Å and $\beta=104{\cdot}7°$, at 20°C. The diffraction data were collected at room temperature with a diffractometer using Mo $K\alpha$ radiation. The structure was solved by the heavy-atom technique and refined by the least-squares method, the final R value being 5·0%. The molecule has the chair conformation with the S=O group pointing towards the axial direction. The conformation features are discussed and compared with those of analogous compounds.

Introduction

During the past decade the conformation of cyclic sulphites, notably trimethylene sulphite



TSU, has been frequently investigated They have been mainly studied in the liquid phase by the application of a number of physical methods such as the infrared technique, nuclear magnetic resonance and electric dipole measurements. The interpretation of the physical properties was, however, difficult and resulted in conflicting conclusions concerning the conformation. The papers of van Woerden & Havinga (1967*a*,*b*) give details and further references.

Altona, Geise & Romers (1966) determined the crystal structure of TSU at −100°C and proved that in the solid state it has the chair conformation with

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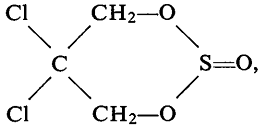
* Part XLV, Altona, Hageman & Havinga, (1968).

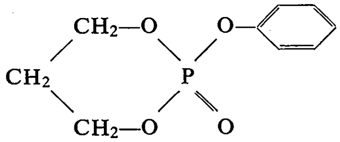
the S=O group pointing towards the axial position. Van Woerden & Havinga (1967*a*) recently published dipole data of fifteen cyclic sulphites and studied their infrared spectra taken in various solvents and in the solid state. They derived a value of 2·5 Debye units for the partial S=O moment and concluded that the molecules are chairs with a preference for an axial S=O group.

In view of the rather low accuracy achieved in the structure determination of TSU (melting point −14 °C) it was deemed necessary to repeat the diffraction measurements with the more suitable compound 2,2′-dichlorotrimethylene sulphite



DCT,

which has a melting point of 63 °C. In the meantime structural data became available for a cyclic ester of phosphoric acid, 2-oxo-2-phenoxy-1,3,2-dioxaphosphorinane (Geise, 1967):



, PPP.

For this reason it was interesting to compare their molecular structures and to investigate to what extent the introduction of several endocyclic hetero atoms deforms the cyclohexane ring system.

Experimental

Unit cell dimensions of DCT (Table 1) were determined from zero-layer Weissenberg photographs taken about [010] and [001] with unfiltered copper radiation ($\lambda=1\cdot5418$ Å) and superposed with aluminum powder lines ($a=4\cdot0489$ Å) for calibration purposes. About 50 high angle reflexions were carefully measured and refined with a least-squares procedure. The cited errors in cell edges are standard deviations. The absent reflexions ($0k0$ when k is odd) indicate $P2_1$ or $P2_1/m$ as possible space groups. The former was chosen on the grounds of the interpretation of the Patterson function.

Table 1. *Crystal data of 2,2′-dichlorotrimethylene sulphite*

Molecular composition $C_3H_4SO_3Cl_2$
Melting point 63 °C
Monoclinic, $P2_1$
$a=6\cdot173\pm0\cdot013$, $b=6\cdot500\pm0\cdot008$, $c=8\cdot986\pm0\cdot004$ Å,
$\beta=104\cdot7\pm0\cdot2°$ at 20 °C
$d_{exp}{}^{20}=1\cdot79$, g.cm^{-3} $d_X{}^{20}=1\cdot82$ g.cm^{-3}
$Z=2$ $F(000)=192$;
$V=348\cdot8$ Å^3 μ (Cu $K\alpha$) $=105$
μ (Mo $K\alpha$) $=11\cdot4$ cm^{-1}

The diffraction intensities of 793 reflexions were measured by means of a non-automatic three-circle diffractometer, applying the ω-scan (moving crystal-stationary counter). Zirconium-filtered Mo $K\alpha$ radiation ($\lambda=0\cdot7107$ Å) was used with a scintillation counter as detector. The chosen crystal was nearly spherical with a diameter of 0·7 mm. The strongest reflexions were measured with attenuation foils and the long-term drift, as well as the loss in intensity by slow evaporation of the crystal, were corrected by reference to a frequently measured standard reflexion.

The intensities were reduced to structure factor moduli after multiplication with Lorentz, polarization and absorption factors. The absorption factor was calculated by assuming an ideal spherical shape for the crystal used, with $\mu R=0\cdot4$ (Bond, 1959). During the final cycles of refinement (see next section) a correction was applied for primary extinction by plotting ln F_o *versus* F_c^2 values.

Refinement

The positions of the heavy atoms Cl(1), Cl(2) and S(4) (see Fig. 1 for the numbering of the atoms) were derived from a three-dimensional Patterson function. The carbon and oxygen atoms C(6), C(7), C(8), O(3), O(5) and O(9) were located in the usual way in a subsequently calculated Fourier synthesis. The model obtained ($R=17\cdot8\%$) was refined according to the method of least squares with a program devised by E. W. M. Rutten (Geise, Romers & Rutten, 1966) in a block-diagonal-

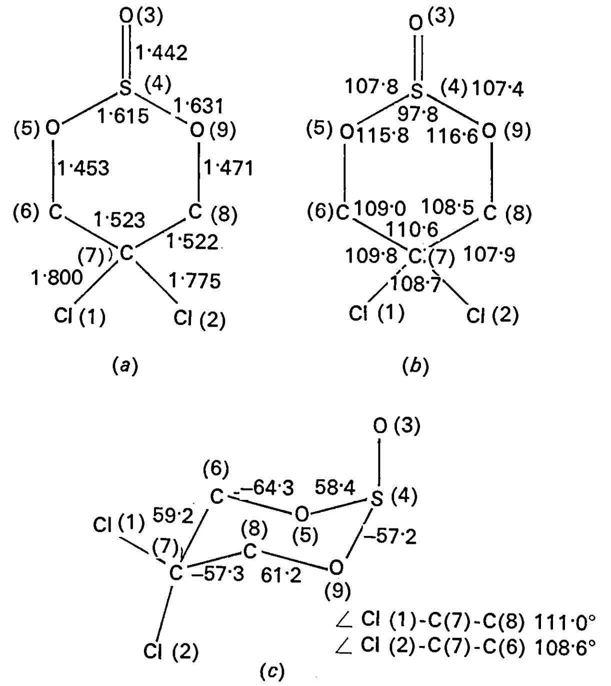


Fig. 1. Schematic drawings of the molecule of 2,2′-dichlorotrimethylene sulphite. (*a*) Interatomic distances, (*b*) valency angles, (*c*) dihedral angles.

Table 2. *Survey of the applied refinement procedures*

Cycle	*R*	Procedure	Description
1	31·3	Structure factors	Cl(1), Cl(2) and S(4) only; overall *B*.
1*a*	–	Fourier synthesis	Location of C(6), C(7), C(8), O(3), O(5) and O(9).
2	17·8	Structure factors least squares	All heavy atoms included; unitary weights; overall *B*.
3–5	12·8	S F L S	All heavy atoms included; unitary weights; overall *B*.
6–10	11·9	S F L S	Isotropic individual *B*'s.
10*a*	–	Difference Fourier	Hydrogen atoms located, but introduced at calculated positions.
11–15	5·66	S F L S	Anisotropic *U*'s for heavy atoms; H's kept at fixed positions with fixed *B*'s = 3·0 Å^2.
16–20	5·28	S F L S	As for 11-15 but with individual weights based upon the counting statistics.
21–23	5·25	S F L S	Heavy atoms kept at fixed positions; refinement of H's with isotropic *B*'s.
20*a*	5·14	S F output	Correction for primary extinction.
24–26	5·01	S F L S	The same as during cycles 16–20, starting with output of cycle 20.

Table 3. *Calculated and observed structure factors for the asymmetric unit*

h k l	Fcalc	Fobs
1 0 0	14.0	12.6
1	73.6	72.9
2	13.6	13.2
3	2.4	2.6
5	6.3	5.9
6	10.5	11.3
7	20.7	20.3
10	3.2	1.8
2 0 0	26.0	26.4
1	50.7	49.6
2	8.3	8.8
3	12.7	12.4
4	12.8	12.3
5	0.7	2.1
6	19.4	18.7
8	13.5	13.4
3 0 0	56.7	58.1
1	2.1	1.4
2	31.1	31.9
3	3.5	3.7
4	17.8	15.5
5	3.6	4.1
6	4.4	4.9
7	3.9	3.3
9	3.9	3.6
4 0 1	13.8	13.9
3	13.7	14.1
4	5.3	3.8
6	8.1	7.6
7	3.5	2.4
5 0 0	13.5	13.4
1	11.8	12.0
3	4.6	4.3
4	5.6	5.9
5	4.3	5.3
6	2.7	2.9
6 0 0	2.7	5.2
1	6.0	5.3
2	7.0	7.1
4	2.9	2.9
5	2.9	2.1
6	3.1	4.1
0 0 2	36.8	36.0
4	18.4	19.1
5	9.4	10.7
6	5.6	6.8
7	16.7	17.1
8	5.6	8.3
9	7.0	7.9
10	1.5	0.8
2 8 1	3.5	3.3
0 10 0	2.8	4.2
4 3 2	8.5	8.0
3	4.3	4.3
4	4.0	3.5
5	2.5	2.5
6	3.0	2.8
8	3.1	3.6
5 3 1	5.5	4.5
2	7.6	7.7
7	3.5	3.5
6 3 1	6.0	6.2
2	3.4	2.6
4	3.9	4.1
7 3 1	7.2	7.3
-1 4 0	5.2	5.6
1	17.9	18.0
2	22.1	21.4
3	30.2	30.5
4	20.5	19.8
5	11.5	11.0
6	8.4	7.7
7	3.2	4.1
8	5.6	5.0
9	6.8	7.3
10	5.4	5.3
11	5.7	5.3
-2 4 0	8.1	8.9
1	6.3	6.4
2	10.4	10.3
3	11.7	11.5
4	11.7	11.5
5	3.3	3.9
6	2.8	2.1
7	6.0	5.9
8	4.6	5.3
9	9.9	10.0
10	8.0	7.4
11	3.5	2.9
-3 4 0	7.3	7.6
1	7.1	6.8
2	12.8	12.9
3	26.3	26.0
4	11.5	11.1
5	8.9	8.7
6	6.8	6.8
7	1.9	3.4
8	6.7	6.7
9	3.3	5.0
11	2.7	3.1
-4 4 0	10.2	11.4
1	10.3	10.5
2	13.7	13.6
3	11.8	11.5
-1 0 1	26.8	25.6
3	2.3	3.2
4	7.6	7.2
5	9.3	8.3
6	48.3	47.3
8	22.2	21.7
10	4.8	4.7
-2 0 1	47.8	47.5
2	16.9	17.3
3	14.1	14.9
4	8.1	8.7
5	32.9	33.1
6	5.6	5.9
7	16.9	17.0
9	6.3	6.0
12	7.4	7.8
-3 0 2	4.6	5.1
3	8.7	8.6
4	15.6	16.2
5	7.6	6.5
6	3.5	3.6
7	20.4	20.4
8	12.2	11.7
10	6.1	5.7
11	3.7	4.2
12	2.2	2.8
13	5.1	5.3
-4 0 1	11.1	10.9
2	20.1	20.7
4	6.9	7.4
6	34.5	34.5
7	4.1	4.3
8	4.8	4.8
9	5.7	5.8
-5 0 2	8.3	8.1
3	7.0	7.6
4	3.5	3.6
5	21.1	20.4
7	6.2	5.3
8	4.1	4.0
9	2.6	3.2
11	3.1	2.6
-6 0 2	4.5	5.1
3	11.1	11.1
4	7.4	7.3
5	4.2	4.3
6	11.3	11.6
7	4.4	4.1
8	4.5	4.8
10	4.5	3.9
1 0 4	14.0	14.5
-1 0 2	6.2	5.8
-4 4 4	8.1	7.8
5	5.9	5.8
6	2.6	3.2
7	5.6	5.3
9	7.9	8.0
-5 4 0	9.4	9.2
1	3.6	3.4
2	3.4	3.3
3	3.2	1.7
4	6.2	6.1
5	4.6	4.4
6	5.8	5.3
7	4.0	4.1
8	6.7	6.4
9	4.4	5.0
-6 4 0	4.0	4.2
3	8.8	8.3
4	5.2	4.4
5	2.7	3.6
6	6.1	5.8
7	2.9	2.3
8	2.9	2.7
-7 4 0	4.2	4.1
2	4.0	3.5
3	3.6	2.8
5	4.0	3.4
-8 4 0	3.6	3.7
0 4 0	10.1	10.5
1	10.8	10.6
2	17.9	17.8
3	27.3	26.7
4	16.9	17.1
5	17.8	16.7
6	8.2	7.9
7	4.5	5.1
8	3.4	4.0
10	2.6	2.5
1 4 1	3.7	5.5
2	13.8	13.3
3	8.4	8.4
5	6.3	6.1
6	7.1	7.0
7	4.5	4.4
10	3.4	3.9
9	7.4	7.4
2 4 1	10.7	10.6
2	10.4	9.9
3	21.7	21.4
4	12.7	12.3
5	8.0	7.2
6	6.7	6.9
7	4.6	5.2
-6 0 11	5.7	6.0
-7 0 1	5.1	3.9
2	7.7	7.6
4	6.5	6.8
5	6.5	6.1
6	5.1	5.4
7	2.1	3.3
-8 0 0	5.0	5.3
3	7.5	7.5
8	4.2	4.2
-1 1 0	20.3	20.8
1	45.0	45.1
2	42.9	42.1
3	41.1	41.5
4	23.5	24.0
5	4.6	4.9
6	5.3	6.7
7	16.2	16.0
8	9.0	10.4
9	7.9	8.1
10	6.2	6.4
11	7.6	7.8
-2 1 0	28.7	29.4
1	27.1	26.5
2	38.9	40.3
3	12.7	13.1
4	28.0	28.4
5	14.5	14.6
6	12.7	12.5
7	16.6	16.5
8	12.5	12.2
9	8.3	8.0
10	7.7	8.1
-3 1 0	13.8	14.3
2	20.8	20.5
3	12.8	13.3
4	20.4	20.9
5	12.4	12.4
6	8.3	8.2
7	15.6	15.7
8	2.9	2.6
9	3.7	4.2
10	8.7	8.6
11	6.1	5.7
-4 1 0	11.3	11.9
1	21.6	21.3
2	17.4	17.7
3	30.3	29.6
4	17.1	17.5
5	7.9	7.7
1 1 0	11.2	11.9
-2 2 0	10.1	10.8
2 4 9	4.0	4.6
3 4 1	9.9	9.9
2	4.5	4.4
3	9.9	10.3
4	8.7	8.7
5	7.9	7.0
6	5.1	4.6
7	2.7	4.3
4 4 1	6.7	7.0
2	3.5	2.8
4	5.0	4.6
5	2.4	2.1
6	3.6	3.6
5 4 1	5.2	5.0
3	6.2	6.2
6 4 1	5.2	4.9
-1 5 0	15.6	16.3
1	7.5	7.1
2	11.8	11.7
3	9.6	9.4
4	11.3	10.3
5	9.9	10.1
6	3.5	2.8
7	11.0	11.1
9	4.1	3.2
10	3.5	4.0
-2 5 0	12.1	12.1
1	10.4	10.3
2	11.6	12.0
4	13.7	13.9
5	4.7	4.9
6	10.2	10.9
7	5.7	5.1
8	3.8	3.5
9	3.1	3.8
10	4.0	3.1
-3 5 0	10.4	10.2
1	7.7	7.8
2	7.1	6.7
3	4.6	4.7
4	7.1	7.0
5	3.8	3.6
6	9.6	9.3
7	3.3	3.1
8	6.0	6.4
9	3.7	2.7
10	2.9	2.1
-4 5 0	4.1	4.8
2	9.6	9.4
4	3.6	4.1
5	12.4	11.8
6	4.8	4.2
-4 1 6	2.4	1.9
7	6.9	6.7
8	7.8	7.6
9	9.1	8.4
10	7.6	7.8
11	2.9	2.7
-5 1 0	3.4	2.9
2	15.9	15.7
3	11.9	14.1
4	9.2	9.2
5	7.7	7.6
7	9.4	9.1
8	11.6	11.2
9	9.2	9.1
10	6.6	6.6
-6 1 0	3.5	3.4
1	6.6	6.7
2	12.5	12.5
3	6.7	6.4
4	9.3	9.0
6	4.5	4.3
7	8.5	7.9
8	6.8	7.0
9	6.2	5.7
-7 1 0	4.7	4.5
1	7.9	7.8
2	5.6	6.6
3	4.7	4.5
4	4.2	3.4
5	4.9	3.8
6	4.0	3.2
7	5.6	5.7
8	4.9	4.6
-8 1 2	3.6	3.4
3	3.6	3.9
4	6.1	5.4
6	4.5	3.3
0 1 1	40.5	38.8
2	39.0	36.2
3	56.9	56.2
4	19.3	18.7
5	23.0	24.1
6	8.2	8.3
7	9.9	10.0
9	6.2	5.8
10	5.9	6.5
11	3.3	3.6
1 1 1	37.2	35.9
3	11.8	11.9
4	36.0	36.2
-6 2 8	4.8	5.2
9	7.4	7.0
10	5.2	5.2
-7 2 0	6.1	5.9
1	3.8	2.6
3	10.2	9.6
4	3.4	3.3
6	4.4	4.2
7	3.0	2.4
8	5.3	4.2
-8 2 3	4.9	5.8
6	5.9	5.7
0 2 0	48.3	48.0
1	29.1	27.2
2	25.2	25.4
3	20.6	20.6
4	3.5	5.2
5	4.0	4.4
6	18.9	18.6
7	3.8	4.8
9	7.7	7.3
10	2.7	2.9
11	3.6	3.9
1 2 1	25.7	25.7
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3	27.3	27.2
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5	8.5	10.0
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7	14.3	14.8
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6	2.6	2.9
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6	5.3	6.4
7	7.6	8.6
8	5.2	6.4
9	3.5	3.8
10	7.4	7.5
2 1 1	22.5	21.8
2	21.1	20.8
3	6.5	8.3
4	21.2	21.6
5	3.7	4.0
6	6.3	5.7
7	5.1	6.6
8	5.0	5.1
10	4.1	4.3
11	5.4	6.2
3 1 1	22.7	23.2
2	16.5	16.6
3	19.0	18.0
4	14.5	14.2
5	21.1	20.8
6	4.8	3.4
7	4.9	5.9
4 1 1	11.5	11.4
2	9.2	8.6
3	8.8	8.7
4	8.0	8.4
5	6.5	5.9
6	3.4	2.8
7	1.9	1.9
5 1 1	3.4	4.0
2	5.3	5.5
3	4.4	3.2
4	10.7	10.4
6 1 0	5.2	5.2
5	5.3	5.1
7 1 1	3.8	2.2
2	4.3	4.7
4	3.9	3.7
-1 2 0	34.9	34.1
1	5.5	6.8
2	20.2	20.4
3	13.9	14.6
4	20.5	20.1
5	20.3	20.8
6	13.2	13.6
7	8.2	9.1
8	10.0	10.3
9	7.5	7.8
10	3.5	3.3
4 2 7	5.1	5.4
5 2 1	15.3	15.5
2	4.8	4.5
3	2.6	2.8
4	3.5	3.3
6 2 3	7.3	6.8
5	4.3	3.4
7 2 2	4.2	4.3
3	4.5	4.7
4	4.1	4.1
-1 3 1	39.2	40.1
2	9.2	9.9
3	7.1	7.6
4	17.7	17.2
5	24.0	23.1
6	4.9	5.9
7	10.5	10.7
8	8.8	8.6
10	1.7	2.7
-2 3 0	20.6	21.5
1	7.2	7.9
2	20.1	20.1
3	5.3	6.0
4	25.5	26.1
5	4.0	4.0
6	11.2	11.1
7	17.6	16.3
8	4.1	1.6
11	5.4	5.4
-3 3 0	22.1	21.8
1	21.6	21.7
2	11.7	11.3
3	4.1	4.8
4	3.3	4.5
5	13.8	13.5
6	13.5	14.0
8	3.0	3.1
9	2.5	2.6
10	8.4	8.8
-4 3 0	13.6	13.9
1	18.3	17.2
2	1.3	2.0
3	11.3	11.1
4	12.8	13.3
5	12.1	11.5
6	9.4	6.9
7	5.7	5.8
8	7.9	7.8
9	6.8	6.3
10	3.3	3.4
11	3.5	3.3
-5 3 0	8.1	8.3
1	5.1	5.2
2	8.7	8.6
3	7.6	7.5
-2 2 1	28.7	29.2
2	9.7	10.0
3	34.2	35.1
4	17.7	18.3
5	11.2	11.1
6	11.6	11.3
7	8.1	9.2
8	8.7	9.3
9	4.7	2.9
10	5.9	5.8
11	4.9	4.9
-3 2 0	16.5	17.6
1	17.9	17.7
2	16.5	16.5
3	11.3	11.5
4	15.7	16.1
5	9.6	9.8
6	9.3	9.3
7	13.5	13.0
8	6.6	6.2
9	11.1	11.0
10	4.3	4.7
11	3.1	3.0
-4 2 0	23.7	24.2
1	9.7	9.1
2	3.9	3.0
3	21.6	20.7
4	10.1	10.0
5	10.6	9.9
6	14.0	14.0
7	9.8	9.8
8	8.2	8.6
12	4.5	4.4
-5 2 1	8.7	8.1
2	15.5	14.9
3	14.7	14.2
4	10.4	10.4
5	7.9	8.4
6	14.4	14.3
7	5.9	6.1
8	4.3	4.0
9	6.1	6.1
10	4.6	4.5
-6 2 0	6.3	6.0
2	5.1	5.2
3	2.7	2.5
4	8.4	7.8
5	11.1	10.8
6	2.4	2.0
7	7.8	7.6
-5 3 4	6.2	6.7
5	11.1	10.6
6	4.9	5.4
7	12.9	12.8
8	4.4	3.5
-6 3 0	8.5	8.0
1	8.0	7.6
2	1.2	1.7
3	3.1	2.5
4	4.7	4.3
5	4.7	4.1
6	9.1	9.2
7	4.5	4.1
8	3.5	3.3
9	2.6	2.2
10	4.4	4.8
-7 3 0	2.9	2.5
2	4.8	4.6
4	4.0	2.1
6	7.2	6.8
7	5.1	5.4
0 3 1	25.6	24.7
2	23.3	22.5
3	19.3	19.9
4	8.2	8.7
6	10.7	10.5
7	11.1	10.7
8	8.1	7.9
9	3 0	3.6
10	4.5	5.2
1 3 1	16.0	16.4
2	17.9	18.1
3	8.8	8.7
4	21.6	21.5
5	4.1	4.9
6	11.0	10.6
7	4.4	5.3
8	4.4	5.7
2 3 1	14.6	14.6
2	3.1	3.1
3	5.1	5.4
4	4.0	3.4
5	12.3	12.6
6	9.8	9.8
7	9.6	9.4
8	3.1	2.6
3 3 1	15.6	15.7
2	10.8	9.9
3	8.7	8.1
4	4.7	5.0
5	7.2	6.5
6	5.4	5.3
7	10.0	10.1
8	4.5	4.3
4 3 1	18.9	18.6

Table 3 (*cont.*)

h k l	Fcalc	Fobs	h k l	Fcalc	Fobs	h k l	Fcalc	Fobs
-4 5 7	6.8	6.6	-1 6 4	6.0	6.6	-1 7 1	6.9	7.1
10	4.0	4.2	5	2.2	3.1	2	2.5	3.2
-5 5 0	2.6	3.8	-2 6 0	5.2	6.5	3	5.9	5.8
1	7.7	7.7	1	5.0	5.5	4	3.8	4.2
2	4.2	4.1	3	5.9	6.8	5	5.7	5.5
4	10.8	10.6	4	6.8	5.6	6	3.4	2.4
5	2.9	3.5	6	9.1	9.4	7	3.8	3.5
6	5.3	5.4	9	4.1	4.6	8	3.0	3.9
8	4.4	4.2	-3 6 1	5.8	5.2	-2 7 0	3.5	3.5
-6 5 0	4.4	4.6	2	8.6	8.9	1	3.2	3.1
3	2.4	2.6	3	10.2	10.2	2	7.8	7.7
7	5.7	5.5	4	3.9	4.3	3	3.0	2.7
-7 5 5	5.1	4.4	5	8.1	8.1	4	6.4	7.1
0 5 1	18.3	18.4	8	2.5	2.5	6	4.6	4.8
2	7.6	8.2	9	3.7	3.4	7	4.2	4.6
3	6.0	6.7	-4 6 0	10.2	10.9	-3 7 1	4.8	5.5
4	13.9	14.1	1	4.6	4.8	5	5.6	5.5
6	6.0	6.5	2	7.4	7.8	7	3.5	3.3
7	3.1	3.1	3	4.0	4.4	-4 7 3	4.3	4.2
8	9.0	9.3	8	4.0	4.0	5	4.2	4.6
10	3.3	3.5	-5 6 0	3.0	3.6	7	3.2	3.0
1 5 1	6.9	7.0	1	5.3	5.5	0 7 1	8.3	8.4
2	18.4	18.0	4	6.4	5.4	2	6.9	6.9
3	3.8	3.4	5	3.7	3.4	5	5.8	6.0
4	9.8	8.6	6	7.6	7.2	5	3.6	3.7
5	6.7	6.6	-6 6 1	2.3	2.5	1 7 1	3.5	3.7
6	6.5	6.5	2	3.2	3.3	2	8.0	7.8
2 5 1	4.6	4.5	3	4.5	3.7	3	4.4	3.8
2	1.8	2.4	5	5.0	4.2	4	6.6	6.8
3	6.2	7.0	-7 6 0	4.0	4.5	2 7 1	7.9	7.2
4	7.0	7.0	0 6 0	2.4	3.0	2	4.1	4.7
5	2.6	2.5	1	3.2	4.6	5	3.5	3.3
6	4.9	4.9	2	5.7	6.0	3 7 1	5.6	5.8
7	7.4	7.8	3	7.6	7.9	2	6.1	6.1
8	4.4	4.7	4	8.0	8.7	3	4.8	5.4
9	2.9	2.5	5	6.7	7.0	4	4.3	3.2
3 5 1	15.4	15.1	6	6.9	7.8	4 7 1	1.9	2.2
2	5.7	6.2	7	3.3	3.7	2	5.7	3.7
3	5.7	4.7	1 6 1	3.4	3.7	4	3.2	3.7
4	6.0	6.2	2	8.0	7.2	-1 8 0	5.1	5.3
7	1.6	2.6	3	6.5	7.0	3	3.3	3.6
8	5.3	5.4	6	4.4	4.4	5	4.0	3.4
4 5 1	4.3	4.2	2 6 2	2 0	2.4	6	4.8	5.0
2	12.9	12.8	3	9.4	8.9	-2 8 1	4.8	4.8
4	4.8	4.3	4	7.6	7.7	5	4.2	4.1
5 5 1	3.3	3.2	5	4.9	4.9	-3 8 1	4.0	3.9
3	2.3	1.9	3 6 4	4.5	4.8	2	4.4	4.5
6 5 1	5.2	4.6	5	2.8	2.8	4	3.5	4.0
3	2.9	3.6	6	6.3	6.6	0 8 0	7.7	8.3
-1 6 0	13.4	13.7	4 6 1	4.1	3.7	2	4.4	4.4
1	5.9	5.7	2	6.1	5.5	3	4.7	4.7
2	7.6	8.1	4	2.5	2.1	1 8 0	3.6	3.6
3	11.9	12.3	5 6 1	5.7	5.6	5	3.6	2.4
-1 7 0	7.3	7.4	3	3.3	2.7			

ized approximation to the matrix of normal equations. Table 2 gives a summary of the procedures applied. The scattering curves for sulphur, chlorine, oxygen, carbon and hydrogen are those published by Dawson (1960) for S and Cl, Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for O and C, and Stewart, Davidson & Simpson (1965) for H.

During cycles 21–23 attempts were made to refine the positional and isotropic temperature parameters of the four hydrogen atoms, keeping the heavy atoms at fixed positions with fixed temperature parameters. The resulting temperature factors, bond lengths C–H and bond angles H–C–H and H–C–C, showed variations between 0·6–2·9 Å^2, 0·97–1·09 Å and 100–120°, respectively. Moreover, the R index and the residual $\Sigma\, w(F_c-F_o)^2$ did not decrease significantly during these cycles. After correction for primary extinction we returned, therefore, to cycle number 20 and continued the refinement of the heavy atoms until R dropped to 5·01% and the largest shifts were about one fourth of the corresponding standard deviations.

The observed and calculated structure factors based upon cycle number 26 are listed in Table 3, the posi-

Table 4. *Final atomic parameters (fractions of cell edges) and their standard deviations* (Å) *for* 2,2′-*dichlorotrimethylene sulphite*

	X	Y	Z	$\sigma(X)$	$\sigma(Y)$	$\sigma(Z)$
Cl(1)	−0·3919	0·2077	0·4178	0·0017	0·0022	0·0016
Cl(2)	−0·3564	0·6011	0·2712	0·0017	0·0021	0·0016
O(3)	0·0840	0·1290	0·0669	0·0040	0·0045	0·0036
S(4)	−0·0034	0·0029	0·1702	0·0015	0·0020	0·0015
O(5)	0·0527	0·1169	0·3353	0·0035	0·0048	0·0034
C(6)	−0·0179	0·3303	0·3335	0·0057	0·0073	0·0051
C(7)	−0·2725	0·3391	0·2811	0·0055	0·0060	0·0052
C(8)	−0·3565	0·2464	0·1213	0·0054	0·0059	0·0054
O(9)	−0·2746	0·0334	0·1252	0·0035	0·0044	0·0036
H(10)	0·054	0·417	0·255	—	—	—
H(11)	0·028	0·385	0·455	—	—	—
H(12)	−0·301	0·335	0·041	—	—	—
H(13)	−0·542	0·235	0·105	—	—	—

Table 5. *Anisotropic thermal parameters* (Å^2) *for* 2,2′-*dichlorotrimethylene sulphite*

The corresponding standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{31}$
Cl(1)	0·0475	0·0660	0·0521	0·0017	0·0331	0·0565
	(0·0009)	(0·0011)	(0·0009)	(0·0022)	(0·0023)	(0·0015)
Cl(2)	0·0785	0·0393	0·0516	0·0282	0·0076	0·0430
	(0·0012)	(0·0008)	(0·0009)	(0·0022)	(0·0020)	(0·0018)
O(3)	0·0614	0·0590	0·0422	0·0076	0·0217	0·0580
	(0·0027)	(0·0031)	(0·0022)	(0·0054)	(0·0052)	(0·0039)
S(4)	0·0395	0·0443	0·0463	0·0086	−0·0066	0·0292
	(0·0008)	(0·0009)	(0·0008)	(0·0022)	(0·0020)	(0·0014)
O(5)	0·0320	0·0570	0·0420	0·0199	0·0044	0·0041
	(0·0021)	(0·0028)	(0·0022)	(0·0048)	(0·0052)	(0·0036)
C(6)	0·0480	0·0528	0·0371	−0·0352	−0·0216	0·0256
	(0·0040)	(0·0040)	(0·0034)	(0·0080)	(0·0076)	(0·0058)
C(7)	0·0454	0·0259	0·0372	−0·0210	−0·0023	0·0403
	(0·0035)	(0·0031)	(0·0031)	(0·0068)	(0·0063)	(0·0055)
C(8)	0·0407	0·0348	0·0460	0·0032	0·0180	0·0308
	(0·0035)	(0·0037)	(0·0032)	(0·0068)	(0·0069)	(0·0056)
O(9)	0·0362	0·0434	0·0513	−0·0113	−0·0146	0·0014
	(0·0021)	(0·0027)	(0·0023)	(0·0050)	(0·0049)	(0·0037)

tional parameters as well as their standard deviations in Table 4 and, in Table 5, the temperature parameters U_{ij}, defined by the expression

$$\exp\,[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33} + 2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})]\,.$$

The machine-calculated standard deviations are, of course, underestimations. For this reason the standard errors in bond lengths and bond angles (see next section) have been multiplied by a factor of 1·40. The standard errors in the unit-cell dimensions are sufficiently small for their influence on the errors in atomic distances and valency angles to be neglected, so they are not taken into account.

Table 6. *Translational and librational components* T_{ij} (Å²) *and* ω_{ij} (*radian*²) *defined with respect to orthogonal axes* a^*, *b and c*

i–j	T_{ij}	$\sigma(T_{ij})$	ω_{ij}	$\sigma(\omega_{ij})$
1–1	0·0256	0·0034	0·0067	0·0009
2–2	0·0308	0·0022	0·0063	0·0011
3–3	0·0328	0·0028	0·0097	0·0010
1–2	−0·0076	0·0024	−0·0047	0·0008
2–3	0·0057	0·0022	0·0034	0·0008
3–1	0·0039	0·0024	−0·0015	0·0007

Table 7. *The vibrational tensor components* (Å²) U_{ij} *and* U'_{ij} *for the atoms* Cl(1) *and* S(4), *the tensor being defined with respect to the orthogonal axes* a^*, *b and c*

i–j	U_{ij}[Cl(1)]	U_{ij}'[Cl(1)]	U_{ij}[S(4)]	U_{ij}'[S(4)]
1–1	0·0475	0·0480	0·0395	0·0439
2–2	0·0660	0·0586	0·0444	0·0472
3–3	0·0436	0·0436	0·0443	0·0400
1–2	0·0086	−0·0013	0·0043	0·0079
2–3	0·0168	0·0128	−0·0045	−0·0096
3–1	0·0171	0·0193	0·0050	0·0016

Table 8. *Positional parameters of* DCT (*fractions of cell edges*) *corrected for angular molecular oscillations*

	X	*Y*	*Z*
Cl(1)	−0·3929	0·2069	0·4185
Cl(2)	−0·3565	0·6027	0·2711
O(3)	0·0852	0·1293	0·0663
S(4)	−0·0028	0·0020	0·1701
O(5)	0·0539	0·1165	0·3359
C(6)	−0·0165	0·3311	0·3340
C(7)	−0·2728	0·3396	0·2811
C(8)	−0·3576	0·2464	0·1205
O(9)	−0·2755	0·0322	0·1247

In order to investigate whether or not the anisotropic thermal parameters are due to rigid-body vibrations the tensors $\mathbf{U}^r$ were analysed in terms of the translational tensor $\mathbf{T}$ and the librational tensor $\boldsymbol{\omega}$ (Cruickshank, 1956). Inspection of Table 6 reveals that the rigid-body model seems quite reasonable in view of the estimated standard deviations. A further check can be made in Table 7 where the least-squares tensors **U** [Cl(1)] and **U** [S(4)] are compared with the corresponding tensors **U**′ [Cl(1)] and **U**′ [S(4)] predicted by the rigid-body hypothesis. In view of these results we have corrected the bond lengths for errors due to angular oscillations (Cruickshank, 1961). The positional atomic parameters corrected for this effect are to be found in Table 8.

Discussion of the structure

The intermolecular atomic distances, valency angles and dihedral angles of DCT, corrected for angular oscillations, are given in Fig. 1. Uncorrected and corrected bond lengths, as well as corrected bond angles are also given in Table 9. It can be seen that the six-membered ring of DCT has the chair conformation. The Newman projection of the molecule along the bonds C(6)–C(7) and S(4)–O(9) is shown in Fig. 2. Fig. 1(*c*) and Fig. 2 clearly demonstrate that the vectors C(7)–Cl(2) and S(4)–O(3) point towards the axial direction and the vector C(7)–Cl(1) towards the equatorial direction. The coordination of sulphur by oxygen atoms is pyramidal: S(4) is 0·66 Å outside the plane of O(5), O(9) and O(3). On the basis of the 1% confidence limit the difference in bond length (0·025 Å) between C(7)–Cl(1) and C(7)–Cl(2) lies in the range of possible significance. Although these bonds are sterically unequivalent there is no logical explanation for this phenomenon and without further evidence this difference must be considered to be doubtful.

Fig. 1 shows that the molecule DCT has the symmetry C_s within the limits of error. The hetero ring

Table 9. *Uncorrected and corrected bond distances* (Å), *corrected bond angles* (*degrees*) *and their standard deviations*

Bond	r_{un}	r_{cor}	$\sigma(r)$	Bond angle	θ	$\sigma(\theta)$
C(6)–C(7)	1·514	1·523	0·012	C(6) –C(7)–C(8)	110·6	0·6
C(7)–C(8)	1·513	1·522	0·011	C(6) –C(7)–Cl(1)	109·8	0·6
C(6)–O(5)	1·446	1·453	0·010	C(6) –C(7)–Cl(2)	108·6	0·6
C(8)–O(9)	1·464	1·471	0·010	C(8) –C(7)–Cl(1)	111·0	0·6
S(4)–O(5)	1·608	1·615	0·006	C(8) –C(7)–Cl(2)	107·9	0·6
S(4)–O(9)	1·623	1·631	0·006	Cl(1)–C(7)–Cl(2)	108·7	0·5
S(4)–O(3)	1·433	1·442	0·006	C(7) –C(6)–O(5)	109·0	0·6
C(7)–Cl(1)	1·789	1·800	0·008	C(7) –C(8)–O(9)	108·5	0·6
C(7)–Cl(2)	1·769	1·775	0·008	C(6) –O(5)–S(4)	115·8	0·5
				C(8) –O(9)–S(4)	116·6	0·5
				O(5) –S(4) –O(9)	97·8	0·3
				O(5) –S(4) –O(3)	107·8	0·3
				O(9) –S(4) –O(3)	107·4	0·3

Average ring bond angle = 109·7

systems of TSU and PPP also possess mirror plane symmetry. For this reason average bond distances and average valency angles of DCT, TSU and PPP are listed in Table 10 for the purpose of comparison. The average C–C distances of DCT do not show significant deviations from the expected value (1·526 Å) for C–C bonds with Sp^3–Sp^3 hybridization (Lide, 1962). The single and double-bonded S–O distances compare favourably with those found in ice-like S_3O_9 (1·64 and 1·40 Å; Westrik & MacGillavry, 1941), in asbestos-like $(SO_3)_n$ (mean values 1·61 and 1·41 Å; Westrik & MacGillavry, 1954), in $HNO_3.2SO_3$ (1·60 and 1·50 Å; Steeman & MacGillavry, 1954) and in $N_2O_5.3SO_3$ (1·64 and 1·40 Å; Eriks & MacGillavry, 1954). The average C–O distances (1·462 Å) are rather high compared with the accepted value for C–O bonds in ethers and alcohols (1·43–1·44 Å), but the deviation is hardly significant. With the exception of the C–O–S angles all valency angles in the ring are normal and do not require comment. The C–O–S angle (116°) are significantly larger than the accepted value (109–110°) and compensate the small O–S–O angle (97·8°). This compensation results in an average ring-bond value of 109·7°, indicating that the ring has about the same puckering as cyclohexane. The exocyclic bond angles O(3)–S(4)–O(5) and O(3)–S(4)–O(9) (mean value 107·2°) have the same values as those found by Shearer (1959) in the α-modification of 1,4-dithiane-1,4-dioxide.

The bond lengths and bond angles of DCT and TSU do not differ significantly, which indicates that the introduction of two chlorine atoms does not produce an extra deformation of the ring system. The same trend is also observed for PPP and the earlier expectation that the C–C lengths in TSU and PPP would be smaller than the normal values is not corroborated by the results obtained in the structure determination of DCT.

The dihedral or torsional angles of the ring system of DCT, TSU and PPP are listed in Table 11. Also included in this list are the average torsional angles ψ and average valency angles θ. The average dihedral angle is in agreement with the value calculated by means of the relation (Altona, 1964)

$$\cos\psi = -\cos\theta/(1+\cos\theta)\,.$$

It can be concluded that PPP is less staggered than DCT and TSU.

Table 11. *Dihedral angles in the heterocyclic ring systems of* DCT, TSU *and* PPP

R–C(*i*)–C(*j*)–R′	DCT	TSU	PPP
C(8)–C(7)	57·2	60·3	59
C(7)–C(6)	59·2	60·6	59
C(6)–O(5)	64·3	64·0	52
O(5)–S,P(4)	58·2	57·0	42
S(4) –O(9)	57·2	54·0	43
O(9)–C(8)	61·2	59·2	54
ψ (average)	59·6	59·2	51·5
θ (average)	109·7	109·8	112·5
ψ (calc)	59·5	59·1	51·7

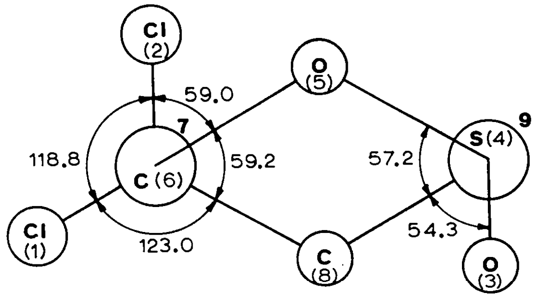

Fig. 2. Newman projection of 2,2′-dichlorotrimethylene sulphite along the bonds C(6)–C(7) and S(4)–O(9). The numbers indicate the values of the torsional angles about the bonds C(6)–C(7) and S(4)–O(9).

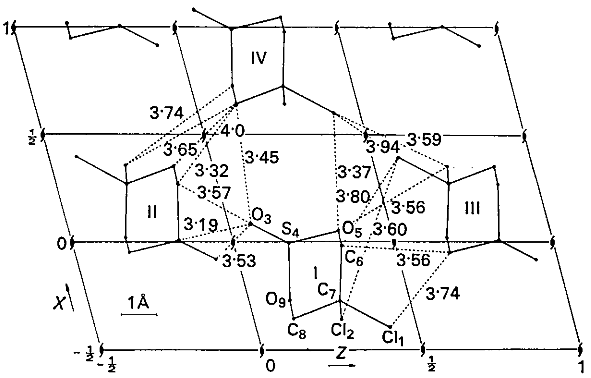

Fig. 3. Projection of the structure along [010]. The Roman numbers are explained in the text.

Table 10 *Average bond lengths* (Å) *and valency angles* (*decimal degrees*) *of* DCT, TSU *and* PPP

Bond, Angle	DCT	σ	TSU	σ	PPP	σ
C–C	1·523	0·008	1·52	0·018	1·49	0·027
C–O	1·462	0·007	1·46	0·015	1·44	0·021
S, P–O	1·622	0·004	1·60	0·008	1·57	0·012
S, P=O	1·442	0·006	1·45	0·011	1·48	0·018
C–C–C	110·6	0·6	110	1·3	110	2·2
C–C–O	108·8	0·4	109	1·2	111	1·5
O–S, P–O	97·8	0·3	100	0·6	107	1·0
O–S, P=O	107·2	0·2	107	0·4	114	0·7
C–O–S, P	116·2	0·3	116	0·7	118	1·0

Fig. 3 shows a projection of the structure of DCT along [010]. Within an upper limit of 4 Å the reference molecule I at x,y,z is surrounded by 12 neighbours at:

$x, \pm 1+y, z$ (not shown in Fig. 3);
$-x, \pm\frac{1}{2}+y, -z$ (II);
$\pm 1+x, y, z$ (III);
$-1-x, \pm\frac{1}{2}+y, -z$;
$-x, \pm\frac{1}{2}+y, 1-z$;
$-1-x, \pm\frac{1}{2}+y, 1-z$.

The shortest intermolecular distances (3·19 Å) are found between O(3) at x,y,z and S(4) at $-x, \frac{1}{2}+y, -z$. All other intermolecular contacts are larger than 3·4 Å.

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Possible Close-Packed Ordered Structures Related to $MoNi_4$

BY PAUL A. BECK

Department of Metallurgy, University of Illinois, Urbana, Illinois 61801, *U.S.A.*

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The geometric conditions of periodic stacking of ordered close-packed layers of the type occurring in $MoNi_4$ are investigated, and a method is described for the systematic generation of such layer structures with a given number of layers in the primitive unit cell.

Several ordered close-packed structures of the AB_3 composition are known to be related to the $AuCu_3$ structure in the sense that they consist of ordered layers identical with those in $AuCu_3$, but stacked in a different way. The best known among these AB_3 structure types are the following: $MgCd_3$ (h.c.p. stacking, $\dot{a}b$, of ordered layers; Dwight & Beck, 1959), $TiNi_3$ (La-stacking, $\dot{a}bcb$, of ordered layers; Laves & Wallbaum, 1939), VCo_3 (stacking of ordered layers, $\dot{a}bcac\dot{b}$; Saito, 1959) and $Ta(Pd,Rh)_3$ (Sm-stacking, $\dot{a}bcbcacab$, of ordered layers; Giessen & Grant, 1959).* General procedures, which may be used to derive all geometrically possible stacking variants based on a given number of stackings of a certain layer in systems with a connectivity of two or four, were described recently (Beck, 1967). The ordered close-packed layer of composition AB_4, of which the $MoNi_4$ structure (Harker, 1944) may be built, was described by Beattie (1967) and it is shown in Fig. 1. Stacking variants based on this layer, and having up to seven-layers, are systematically derived in the following by adapting to this more complicated system the method previously outlined (Beck, 1967).

In Fig. 1 the two-dimensional primitive unit mesh of the close-packed ordered layer is outlined. In the $MoNi_4$ structure both layers adjacent to the one shown in the Figure (the latter is characterized by Mo atoms on sites *a*, *i.e.* by 'stacking position *a*') have Mo atoms placed over (or under) one of the four interstitial sites designated 0, 9, 7 and 2 in Fig. 1. Thus, the nearest

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* Some of the structures mentioned actually are slightly distorted. For the purposes of the present paper this may be disregarded.