Discussion. The final atomic parameters are listed in Table 1.* Molecular structures of (1) and (2) are shown in Fig. 1 with numbering schemes. The stereochemistry of (1) is shown to be 3,5-*trans* as previously thought and that of (2) as 3,5-*cis* unequivocally. The geometries of the isoxazolidine rings in both compounds are similar but a slight difference is observed in some of the bond lengths and angles as shown in Table 2. The conformation of the isoxazolidine ring in the crystal has approximate C_2 symmetry through C(5) in (1) [deviation from O(1)—C(5)—C(4) plane: N(2) −0·21 Å and C(3) +0·34 Å], and C_s symmetry [with N(2) the out-of-plane atom] in (2) [N(2) −0·56 Å]. In both compounds, the configuration of the substituents at the N(2) and C(3) positions is *trans*-diaxial due to the steric repulsion of the two substituents. The torsion angles between the protons on the ring are show in Table 2 together with the coupling constants observed in $CDCl_3$. The angles and the coupling constants are not necessarily correlated; this is apparently due either to the pseudorotation or to equilibrium among several conformers in solution.

The crystal packings are shown in Fig. 2. In (1), both the disordered hydroxyl groups [C(10)—O(11*A*) and C(10)—O(11*B*)] are bonded to the carbonyl O atom [O(7)] of the ester group [O(11*A*)(*x*,*y*,*z*)···O(7)(1 − *x*, −*y*,1 − *z*) = 2·781 (5) Å, C(10)—O(11*A*)···O(7) = 100·9 (3)°; O(11*B*)(*x*,*y*,*z*)···O(7)(1 − *x*, −*y*,1 − *z*) = 2·956 (8) Å, C(10)—O(11*A*)···O(7) = 93·3 (4)°]. Other interatomic contacts are within van der Waals distances; the shortest distance is 3·302 (5) Å for O(1)(*x*,*y*,*z*)···C(17)(−*x*, −*y*, −*z*). On the other hand, only van der Waals contacts are recognized in (2). The shortest distance is 3·348 (7) Å for C(3)(*x*,*y*,*z*)···O(1)(1·5 − *x*,1 − *y*,0·5 + *z*). The somewhat large thermal vibrations observed at C(16) and C(17) in (2) may be due to the presence of some disorder around the ethoxymethyl group.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and bond lengths have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52448 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). C**46**, 643–645

X-ray Structure of the Monoclinic Form of 1,8,15,22-Tetrathia[1.1.1.1]metacyclophane

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(*Received* 11 *May* 1989; *accepted* 27 *June* 1989)

Abstract. 2,8,14,20-Tetrathiapentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene, $C_{24}H_{16}S_4$, M_r = 432·6, monoclinic, $P2_1/n$, a = 15·418 (2), b = 7·776 (1), c = 17·048 (1) Å, β = 93·24 (1)°, V = 2040·5 (3) Å^3, Z = 4, D_x = 1·408 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·46 mm^{-1}, F(000) = 896, T = 293 K, R = 0·052 for 1848 observed reflections. The molecule has distorted *mm*2 symmetry and a nonplanar conformation, close to that observed in the triclinic form. Two of the four benzene rings have rather short contacts of ~3·50 Å with the corresponding and almost parallel rings of the neighbouring molecules, suggesting a weak intermolecular stacking interaction.

Introduction. The conformational flexibility of phenylenesulfides resulting in the existence of different conformers in crystals is of significant importance for their possible application as complexones (Ovchinnikov, Zamaev, Shklover, Palyulin,

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Table 1. *Atomic coordinates for non-*H *atoms* ($\times 10^5$ *for* S, $\times 10^4$ *for* C) *and equivalent isotropic thermal parameters*

$$U_{eq} = (1/24\pi^2)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
S(1)	43032 (7)	11922 (20)	19056 (8)	6·12 (5)
S(2)	6971 (7)	8408 (24)	18207 (8)	7·31 (5)
S(3)	8554 (8)	44004 (21)	−8972 (10)	7·75 (5)
S(4)	44052 (7)	34763 (19)	−10950 (8)	6·08 (5)
C(1)	3297 (2)	904 (5)	2341 (3)	4·1 (1)
C(2)	2500 (2)	996 (6)	1926 (2)	4·6 (1)
C(3)	1746 (2)	772 (6)	2309 (2)	4·2 (1)
C(4)	1784 (3)	503 (6)	3114 (2)	4·3 (1)
C(5)	2574 (3)	468 (6)	3526 (3)	4·9 (1)
C(6)	3324 (3)	645 (6)	3144 (3)	4·6 (1)
C(7)	930 (2)	864 (6)	817 (2)	4·3 (1)
C(8)	870 (2)	2391 (6)	406 (3)	4·1 (1)
C(9)	1016 (2)	2421 (6)	−385 (3)	4·1 (1)
C(10)	1236 (3)	937 (6)	−771 (3)	4·7 (1)
C(11)	1305 (3)	−571 (6)	−355 (3)	5·1 (1)
C(12)	1150 (3)	−613 (6)	428 (3)	5·9 (1)
C(13)	1872 (3)	4881 (5)	−1266 (2)	3·9 (1)
C(14)	2636 (2)	4089 (5)	−1009 (2)	3·1 (1)
C(15)	3405 (3)	4523 (5)	−1328 (2)	3·9 (1)
C(16)	3417 (3)	5809 (6)	−1895 (3)	5·5 (1)
C(17)	2653 (3)	6596 (6)	−2129 (3)	6·0 (1)
C(18)	1884 (3)	6150 (6)	−1841 (3)	5·1 (1)
C(19)	4107 (2)	1845 (6)	−430 (3)	4·2 (1)
C(20)	4245 (2)	2133 (5)	364 (3)	4·0 (1)
C(21)	4045 (2)	858 (6)	893 (3)	4·4 (1)
C(22)	3697 (3)	−687 (7)	615 (3)	5·0 (1)
C(23)	3556 (3)	−953 (6)	−180 (3)	5·5 (1)
C(24)	3764 (3)	310 (6)	−709 (3)	4·9 (1)

Table 2. *Main bond lengths* (Å), *bond and torsion angles* (°)

S(1)—C(1)	1·772 (4)	S(3)—C(9)	1·780 (5)
S(1)—C(21)	1·769 (4)	S(3)—C(13)	1·761 (4)
S(2)—C(3)	1·777 (4)	S(4)—C(15)	1·769 (4)
S(2)—C(7)	1·768 (4)	S(4)—C(19)	1·779 (4)
C(1)S(1)C(21)	103·7 (2)	C(9)S(3)C(13)	104·9 (2)
C(3)S(2)C(7)	103·0 (2)	C(15)S(4)C(19)	102·6 (2)
C(1)S(1)C(21)C(20)	−124·4 (5)	C(8)C(9)S(3)C(13)	−120·2 (5)
C(2)C(1)S(1)C(21)	19·0 (5)	C(9)S(3)C(13)C(14)	15·2 (4)
C(2)C(3)S(2)C(7)	−9·6 (5)	C(14)C(15)S(4)C(19)	−2·0 (4)
C(3)S(2)C(7)C(8)	102·7 (5)	C(15)S(4)C(19)C(20)	98·5 (5)

Struchkov, Astankov, Nedel'kin & Sergeev, 1989). Besides, this flexibility favours the formation of different polymorphic modifications, for instance in the case of the title compound [alternatively named *cyclo*-tetrakis(*m*-phenylenesulfide)]. Its triclinic modification (I) has been described by us earlier (Zamaev, Shklover, Ovchinnikov, Struchkov, Astankov, Nedel'kin & Sergeev, 1989). The present paper reports the X-ray structure of the monoclinic form (II).

Experimental. Colourless crystals of irregular shape. The unit-cell parameters were determined from 12 reflections with $28 < 2\theta < 32°$. 3550 independent reflections were measured ($-20 \leq h \leq 20$, $0 \leq k \leq 10$, $0 \leq l \leq 22$) with an automated Hilger & Watts diffractometer (graphite monochromator, $\theta/2\theta$ scan, $2\theta_{max} = 56°$). No appreciable change in intensities of the two standard reflections (004 and $2\bar{2}0$) was observed. No absorption or secondary-extinction corrections were applied. The structure was solved by direct methods (*MULTAN*; Germain, Main & Woolfson, 1971) and refined by the block-diagonal least-squares technique in the anisotropic approximation for non-H atoms. 1848 reflections with $I > 2\sigma$ were used. The function minimized was $\sum w(|F_o| - |F_c|)^2$; $w = 1/\sigma^2(F)$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) were used. The H-atom positions were calculated and refined isotropically. The final discrepancy factors are $R = 0{\cdot}052$, $wR = 0{\cdot}034$, $S = 2{\cdot}71$, max. $(\Delta/\sigma) = 0{\cdot}5$, the electron-density variation in the final difference synthesis is $\pm 0{\cdot}5$ e Å^{-3}. All calculations were carried out with an Eclipse S/200 computer using the *INEXTL* program package (Gerr, Yanovsky & Struchkov, 1983).

Discussion. Atomic positional and equivalent isotropic thermal parameters are given in Table 1,*

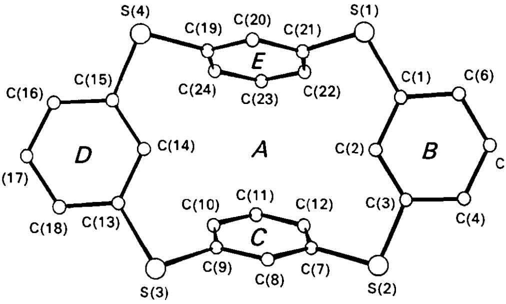

Fig. 1. General view of molecule (II). H atoms are not shown.

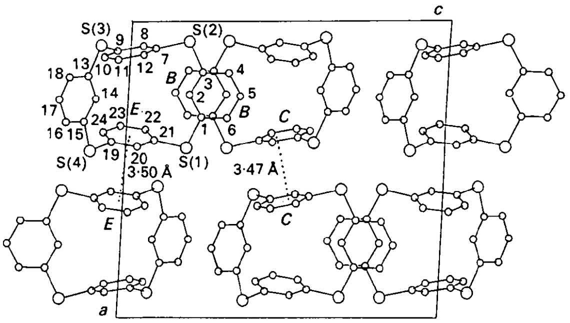

Fig. 2. Projection of crystal structure (II) along the b axis. The distances between the parallel benzene rings of the neighbouring molecules are given.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and bond lengths and bond angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52382 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

main bond lengths, bond and torsion angles are given in Table 2. Fig. 1 represents a general view of molecule (II), with atomic numbering.

The geometry of the molecule in the monoclinic structure (II) does not differ significantly from that in the triclinic form (I): the maximal difference between the corresponding CSCC torsion angles, which fully define molecular conformations in these structures, is equal to 16°. The configuration of the molecule in structure (II) is closer to the ideal *mm*2 symmetry than in (I): the absolute values of the CSCC torsion angles related by the approximate mirror planes normal to the *A* macrocycle and passing through the midpoints of its S···S edges (Fig. 1) differ on average by 16°, while in the triclinic form the corresponding average difference is 26°. The S_4 tetragon in structure (II) has a much more planar conformation than in structure (I), the dihedral angles between the S(1) S(2) S(3) and the S(1) S(4) S(3) planes being 15·6 and 27·0° respectively. The observed higher symmetry of the molecule in the monoclinic form (II) may be a consequence of a rather interesting trend of simultaneous increase in molecular and packing symmetry, observed to a different extent in crystals of other phenylenesulfides (Sergeev, Ovchinnikov, Nedel'kin, Astankov, Andrianova, Shklover, Zamaev, Struchkov, 1988): the centre of the S_4 cycle is located in the vicinity of $\frac{1}{4},\frac{1}{4},0$ (its exact coordinates are 0·257, 0·248, 0·043) and the enantiomers of one type form layers in the *bc* plane (Fig. 2).

According to Kitaigorodsky (1971), an increase in the packing symmetry is accompanied as a rule by a decrease in the density. Nevertheless, crystal (II) has a higher density than crystal (I): 1·408 and 1·368 Mg m^{-3} respectively. This peculiarity of (II) may be caused by its stronger net intermolecular interaction owing to the contribution of the stacking interactions, in which two (*C* and *E*) of the four benzene rings of each molecule take part (Fig. 2). The higher energy of the intermolecular interaction is also reflected in the higher (by 32 K) melting point of the monoclinic form (II).

The average values of S—C bond lengths, 1·772 Å, and CSC bond angles, 103·6°, in structure (II) differ only slightly from those in structure (I) (1·777 Å and 104·2° respectively); the deviations of the S atoms from the planes of the benzene rings bonded to them do not exceed 0·12 Å, as compared to 0·14 Å in (I). Thus the steric-strain energies in both conformers (I) and (II) are quite close.

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Acta Cryst. (1990). C**46**, 645–648

The Structure of T-2 Toxin

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(*Received* 3 *April* 1989; *accepted* 6 *June* 1989)

Abstract. T-2 toxin, 12,13-epoxytrichothec-9-ene-3,4,8,15-tetraol 4,15-diacetate 8-(3-methylbutanoate), $C_{24}H_{34}O_9$, $M_r = 466·53$, orthorhombic, $P2_12_12_1$, $a = 7·808$ (2), $b = 15·800$ (4), $c = 40·891$ (9) Å, $V = 5044$ (2) Å^3, $Z = 8$, $D_x = 1·228$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·74$ mm^{-1}, $F(000) = 2000$, $T = 295$ K, final $R = 0·055$, $wR = 0·063$ for 3434 independent observed reflections. The chemically established structural formula was corroborated. The two molecules in the asymmetric unit differ only in the torsion angles of the ester side chains.

Introduction. T-2 toxin, produced by a mold growing on stored corn, was the first trichothecene to be implicated in animal-feed toxicosis in 1971; since then more than a score have been found which are

0108-2701/90/040645-04$03.00