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Stereochemical Studies of Oligomers. IX.* Structures of 1,4-Butanediyl Bis[*o*-(*m*- and *p*-)chlorobenzoates] (BDDO, BDDM and BDDP), $C_{18}H_{16}Cl_2O_4$

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Abstract. $M_r = 367\cdot2$, room temperature, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1\cdot5418$ Å. BDDO: monoclinic, $P2_1/n$, $a = 8\cdot858$ (1), $b = 12\cdot956$ (2), $c = 7\cdot472$ (1) Å, $\beta = 97\cdot08$ (2)°, $Z = 2$, $V = 850\cdot98$ (20) Å^3, $D_x = 1\cdot44$ g cm^{-3}, $\mu = 36\cdot6$ cm^{-1}, $F(000) = 380$, $R = 0\cdot050$, 1183 observed reflections. BDDM: monoclinic, $P2_1/c$, $a = 5\cdot870$ (3), $b = 19\cdot270$ (2), $c = 7\cdot651$ (2) Å, $\beta = 97\cdot29$ (3)°, $Z = 2$, $V = 858\cdot45$ (50) Å^3, $D_x = 1\cdot42$ g cm^{-3}, $\mu = 36\cdot3$ cm^{-1}, $F(000) = 380$, $R = 0\cdot049$, 1301 observed reflections. BDDP: triclinic, $P1$, $a = 7\cdot609$ (2), $b = 10\cdot318$ (1), $c = 5\cdot924$ (3) Å, $\alpha = 96\cdot31$ (2), $\beta = 98\cdot35$ (3), $\gamma = 111\cdot05$ (2)°, $Z = 1$, $V = 422\cdot79$ (26) Å^3, $D_x = 1\cdot44$ g cm^{-3}, $\mu = 36\cdot8$ cm^{-1}, $F(000) = 190$, $R = 0\cdot081$, 1006 observed reflections. The usual tilt of the carboxylic groups with respect to their phenyl rings is observed and in the case of the *ortho* derivative the steric hindrance increases the value [31·2 (1)°] noticeably. The conformation of the butanediyl chain is *gauche–trans–gauche* in the *ortho* derivative and all-*trans* for the other two compounds.

Introduction. During studies on polymers in the late nineteen forties, it was often found that stereochemistry strongly influences polymerization mechanisms and relationships between molecular structure and physical and mechanical properties of polymers. In particular, a review of experiments on polyester derivatives made over the course of a decade at the University of Montreal (Brisse, Marchessault & Pérez, 1980) shows that substantial progress in understanding their structures was made if their stereochemistry was absolutely certain. For this class of polymers the polymeric chain may be symbolically divided into a number of monomers and the knowledge of the structure of these small 'model compounds' is essential if the correct polymer structure is to be obtained. This approach was employed in the structural determination of a large number of biopolymers (Brant, Tonelli & Flory, 1969; Pérez & Brisse, 1978; Pérez & Marchessault, 1978) or polymers (Benedetti, Pedone & Allegra, 1970) and to confirm the structure of poly(ethylene terephthalate) on the basis of those of ethylene glycol dibenzoate (Pérez & Brisse, 1976*a*) and of its *p*-chloro derivative (Pérez & Brisse, 1975). Moreover, the possible structural conformation of the not yet known poly(trimethylene terephthalate) was hypothesized on the basis of that of related monomers: trimethylene glycol dibenzoate (Pérez & Brisse, 1977) and its *p*-chloro derivative (Pérez & Brisse, 1976*b*). From these examples it is clear that structural data on monomers provide a powerful basis for investigating the structures of macromolecules. Consequently, increasing attention is devoted to the conformations of these types of monomers in order to obtain precise geometrical information which is also useful in the energy calculations.

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* Part VIII: Bocelli & Grenier-Loustalot (1983*c*).

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with* B_{eq} *values* (*Hamilton,* 1959) *for BDDO, BDDM and BDDP*

	x	y	z	B_{eq}(Å^2)
BDDO				
Cl	3553 (1)	935 (1)	8322 (1)	6·53
O(1)	1128 (3)	1500 (2)	5358 (3)	6·96
O(2)	1594 (2)	908 (1)	2679 (3)	4·54
C(1)	3707 (3)	1345 (2)	4722 (3)	3·96
C(2)	4479 (3)	1235 (2)	6470 (4)	4·38
C(3)	6041 (4)	1327 (3)	6765 (5)	5·41
C(4)	6853 (4)	1546 (3)	5366 (6)	6·96
C(5)	6118 (4)	1676 (2)	3646 (5)	4·54
C(6)	4557 (3)	1561 (2)	3321 (4)	3·96
C(7)	2014 (3)	1260 (2)	4346 (4)	4·38
C(8)	−51 (3)	848 (3)	2136 (5)	5·41
C(9)	−285 (3)	530 (2)	162 (4)	5·84
BDDM				
Cl	10722 (1)	1731 (1)	3906 (1)	6·74
O(1)	11835 (3)	4980 (1)	3515 (2)	5·89
O(2)	8921 (3)	4309 (1)	2349 (2)	4·67
C(1)	12107 (3)	3754 (1)	3917 (3)	4·03
C(2)	10995 (4)	3122 (1)	3653 (3)	4·16
C(3)	12106 (4)	2524 (1)	4257 (3)	4·58
C(4)	14317 (5)	2539 (2)	5153 (3)	5·43
C(5)	15410 (5)	3171 (2)	5421 (4)	5·75
C(6)	14332 (4)	3777 (1)	4825 (3)	4·87
C(7)	10989 (3)	4415 (1)	3286 (3)	4·30
C(8)	7683 (4)	4923 (1)	1664 (3)	4·93
C(9)	5708 (4)	4688 (1)	367 (3)	4·31
BDDP				
Cl(1)	8796 (4)	11602 (2)	11567 (4)	5·40
Cl(2)	−9852 (5)	−1306 (4)	−13979 (7)	6·17
O(1)	3035 (20)	5207 (12)	5600 (23)	4·93
O(2)	2581 (21)	6592 (15)	3147 (23)	5·45
O(3)	−4089 (20)	5123 (14)	−8092 (22)	5·18
O(4)	−3617 (15)	3743 (11)	−5564 (19)	3·63
C(1)	4692 (11)	7667 (7)	6654 (13)	3·20
C(2)	5231 (10)	8999 (8)	6031 (15)	3·34
C(3)	6454 (13)	10258 (10)	7466 (15)	4·17
C(4)	7276 (14)	10129 (13)	9648 (17)	4·94
C(5)	6869 (16)	8798 (11)	10429 (19)	4·85
C(6)	5506 (16)	7538 (12)	8859 (17)	4·73
C(7)	3365 (25)	6302 (15)	5129 (23)	6·17
C(8)	1303 (21)	5408 (12)	1620 (24)	5·45
C(9)	224 (27)	5776 (16)	−253 (25)	4·93
C(10)	−1178 (29)	4507 (19)	−1975 (37)	3·63
C(11)	−2153 (34)	5157 (20)	−3936 (36)	5·18
C(12)	−4434 (27)	3928 (23)	−7592 (38)	3·20
C(13)	−5847 (15)	2625 (12)	−9128 (18)	3·34
C(14)	−6671 (15)	2661 (10)	−11293 (16)	4·17
C(15)	−7862 (16)	1480 (14)	−12764 (18)	4·94
C(16)	−8309 (15)	234 (9)	−12062 (19)	4·85
C(17)	−7595 (18)	142 (11)	−9877 (22)	4·73
C(18)	−6123 (25)	1438 (13)	−8339 (19)	2·96

The three crystal structures of monomers reported here constitute a new step in this field of research.

Experimental. For all compounds: Siemens AED single-crystal diffractometer, cell parameters by least-squares fit of 21–25 automatically centred reflections, intensities obtained by a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) notation, scan speed 6–24 s deg^{-1} determined from the intensity of a 'flash' (1 s), scan range from $\Delta_0 = 0{\cdot}55°$ below θ to $\Delta_1 = \{0{\cdot}55+[(\lambda_{\alpha_1}-\lambda_{\alpha_2})/\bar{\lambda}]\tan\theta\}°$ above, one standard reflection every 50 counts, Lp and polarization corrections, absorption ignored.

BDDO: colourless prism recrystallized from a petroleum ether solution, $0{\cdot}17 \times 0{\cdot}26 \times 0{\cdot}38$ mm, 1820 reflections collected, 1630 unique, $R_{\rm int} = 0{\cdot}037$, 1183 observed [$I \geq 2\sigma(I)$], $3 \leq \theta \leq 70°$, no decay in intensity of check reflection, $-10 < h < 10$, $0 < k < 15$, $0 < l < 9$, direct methods with *SHELX* (Sheldrick, 1976), refinement on $\sum w(\Delta F)^2$, non-hydrogen atoms anisotropic, H atoms located in a ΔF map and refined, $R = 0{\cdot}050$, $R_w = 0{\cdot}061$, $w = 1{\cdot}0/(\sigma^2F_o^2 + 0{\cdot}0062\,F_o^2)$, in final cycle 141 parameters refined, one reflection omitted, $\Delta\rho_{\rm max}$ 0·20 e Å^{-3}, $(\Delta/\sigma)_{\rm max}$ 0·145, scattering factors in *SHELX*.

BDDM: colourless prism recrystallized from acetone, $0{\cdot}26 \times 0{\cdot}33 \times 0{\cdot}44$ mm, 1852 reflections collected, 1645 unique, $R_{\rm int} = 0{\cdot}013$, 1301 observed [$I \geq 2\sigma(I)$], $2 \leq \theta \leq 70°$, no decay in intensity of check reflection, $-6 < h < 7$, $0 < k < 23$, $0 < l < 9$, direct methods with *SHELX*, refinement on $\sum w(\Delta F)^2$, non-hydrogen atoms anisotropic, H atoms located in a ΔF map and refined, $R = 0{\cdot}049$, unit weights, in final cycle 141 parameters refined, 7 reflections omitted, $\Delta\rho_{\rm max}$ 0·23 e Å^{-3}, $(\Delta/\sigma)_{\rm max}$ 0·225, scattering factors in *SHELX*.

BDDP: colourless prism recrystallized from acetone, $0{\cdot}09 \times 0{\cdot}11 \times 0{\cdot}23$ mm, 1575 reflections collected, 1308 unique, 1006 observed [$I \geq 2\sigma(I)$], $2 \leq \theta \leq 70°$; intensity of check reflection decayed 17%; data were corrected for this decay but the paucity of data affects the structure results; intensity statistics do not unequivocally indicate whether the structure is centro- or

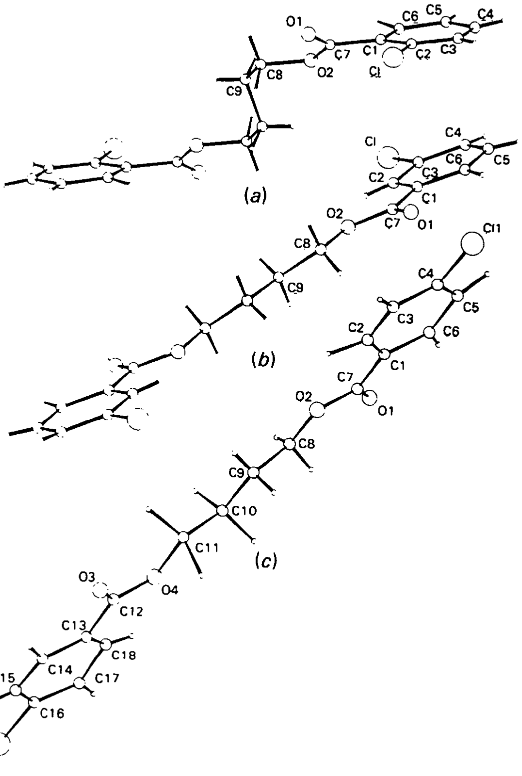

Fig. 1. Projections of (*a*) BDDO, (*b*) BDDM and (*c*) BDDP showing the atom-numbering schemes.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

	BDDO	BDDM	BDDP		BDDP
Cl–C(2)	1·737 (3)				
Cl–C(3)		1·736 (3)			
Cl(1)–C(4)			1·708 (5)	Cl(2)–C(16)	1·752 (5)
C(1)–C(2)	1·405 (4)	1·384 (3)	1·394 (6)	C(13)–C(14)	1·354 (7)
C(1)–C(6)	1·390 (4)	1·400 (3)	1·404 (7)	C(13)–C(18)	1·316 (9)
C(1)–C(7)	1·495 (4)	1·485 (3)	1·501 (7)	C(12)–C(13)	1·483 (10)
C(2)–C(3)	1·380 (4)	1·375 (3)	1·391 (5)	C(14)–C(15)	1·345 (7)
C(3)–C(4)	1·370 (6)	1·389 (4)	1·394 (7)	C(15)–C(16)	1·336 (9)
C(4)–C(5)	1·377 (5)	1·380 (5)	1·439 (9)	C(16)–C(17)	1·356 (8)
C(5)–C(6)	1·382 (4)	1·378 (4)	1·455 (6)	C(17)–C(18)	1·494 (7)
O(1)–C(7)	1·196 (4)	1·200 (3)	1·142 (10)	O(3)–C(12)	1·244 (14)
O(2)–C(7)	1·335 (4)	1·344 (3)	1·351 (11)	O(4)–C(12)	1·336 (12)
O(2)–C(8)	1·466 (3)	1·451 (3)	1·380 (8)	O(4)–C(11)	1·384 (9)
C(8)–C(9)	1·521 (5)	1·497 (3)	1·450 (12)	C(10)–C(11)	1·621 (15)
C(9)–C(9′)	1·493 (4)	1·528 (3)			
C(9)–C(10)			1·510 (10)		
C(7)–O(2)–C(8)	115·4 (2)	116·5 (2)	113·6 (6)	C(11)–O(4)–C(12)	114·3 (7)
C(6)–C(1)–C(7)	119·8 (2)	118·7 (2)	115·0 (5)	C(18)–C(13)–C(12)	116·3 (6)
C(2)–C(1)–C(7)	121·9 (2)	121·8 (2)	126·0 (5)	C(14)–C(13)–C(12)	121·0 (6)
C(2)–C(1)–C(6)	118·3 (2)	119·6 (2)	118·9 (5)	C(14)–C(13)–C(18)	122·5 (6)
C(1)–C(2)–C(3)	120·2 (3)	119·6 (2)	125·5 (4)	C(13)–C(14)–C(15)	121·9 (6)
Cl–C(2)–C(1)	122·7 (2)				
Cl–C(2)–C(3)	117·1 (3)				
Cl–C(3)–C(2)		119·6 (2)			
Cl–C(3)–C(4)		119·0 (2)			
C(2)–C(3)–C(4)	120·4 (3)	121·5 (2)	115·6 (5)	C(14)–C(15)–C(16)	119·7 (5)
Cl(1)–C(4)–C(3)			119·9 (5)	Cl(2)–C(16)–C(15)	119·7 (5)
Cl(1)–C(4)–C(5)			116·9 (4)	Cl(2)–C(16)–C(17)	119·3 (4)
C(3)–C(4)–C(5)	120·4 (3)	118·7 (3)	123·2 (5)	C(15)–C(16)–C(17)	121·0 (5)
C(4)–C(5)–C(6)	119·9 (3)	120·9 (3)	117·7 (5)	C(16)–C(17)–C(18)	118·8 (5)
C(1)–C(6)–C(5)	120·8 (3)	119·9 (2)	119·0 (5)	C(13)–C(18)–C(17)	115·5 (6)
O(2)–C(7)–C(1)	111·0 (2)	112·0 (2)	108·6 (6)	O(4)–C(12)–C(13)	115·4 (8)
O(1)–C(7)–C(1)	125·7 (3)	125·0 (2)	125·5 (7)	O(3)–C(12)–C(13)	122·8 (9)
O(1)–C(7)–O(2)	123·3 (3)	123·0 (2)	125·9 (7)	O(3)–C(12)–O(4)	121·7 (10)
O(2)–C(8)–C(9)	107·2 (2)	107·6 (2)	111·6 (6)	O(4)–C(11)–C(10)	99·9 (6)
C(8)–C(9)–C(9′)	113·4 (3)	110·2 (2)		C(9)–C(10)–C(11)	104·8 (7)
C(8)–C(9)–C(10)			113·1 (7)		
C(2)–C(1)–C(7)–O(1)	31·5 (4)	177·9 (2)	174·3 (7)	C(14)–C(13)–C(12)–O(4)	−175·3 (7)
C(2)–C(1)–C(7)–O(2)	−150·7 (3)	−4·7 (3)	−7·8 (9)	C(14)–C(13)–C(12)–O(3)	8·0 (13)
C(6)–C(1)–C(7)–O(1)	−147·1 (3)	−1·2 (3)	−2·5 (11)	C(18)–C(13)–C(12)–O(4)	−0·7 (12)
C(6)–C(1)–C(7)–O(2)	30·7 (3)	176·3 (2)	175·4 (3)	C(18)–C(13)–C(12)–O(3)	−177·4 (9)
C(1)–C(7)–O(2)–C(8)	−176·9 (2)	−179·8 (2)	−179·0 (6)	C(13)–C(12)–O(4)–C(11)	−178·6 (7)
O(1) C(7)–O(2)–C(8)	1·0 (4)	−2·3 (3)	−1·1 (12)	O(3)–C(12)–O(4)–C(11)	−1·8 (13)
C(7)–O(2)–C(8)–C(9)	174·8 (2)	168·1 (2)	168·7 (7)	C(12)–O(4)–C(11)–C(10)	−169·3 (8)
O(2)–C(8)–C(9)–C(9′)	65·2 (3)	178·4 (2)			
O(2)–C(8)–C(9)–C(10)			179·4 (7)	O(4)–C(11)–C(10)–C(9)	−178·2 (7)
C(8)–C(9)–C(9′)–C(8′)	180·0 (3)	180·0 (2)			
C(8)–C(9)–C(10)–C(11)			−178·0 (7)		

noncentrosymmetric; direct methods with *MULTAN* (Germain, Main & Woolfson, 1974) after many attempts in space groups $P1$ and $P\bar{1}$; although many E maps yielded molecular fragments, the attempt to refine the structure in the centrosymmetric space group failed; refinement on $\sum w(\Delta F)^2$, block-matrix, non-hydrogen atoms anisotropic, most H atoms in ΔF map, four placed in calculated positions (Nardelli, 1982), all refined, $R = 0{\cdot}081$, $R_w = 0{\cdot}077$, $w = 2{\cdot}4515/(\sigma^2F_o^2 + 0{\cdot}71255F_o^2)$, in final cycles 202 and 80 parameters refined, 12 reflections suffering from extinction omitted, $\Delta\rho_{\max}$ 0·85 e Å^{-3}, $(\Delta/\sigma)_{\max}$ 0·832, scattering factors in *SHELX*.

MULTAN calculations carried out on the CDC Cyber 76 computer of the Centro di Calcolo Interuniversitario dell'Italia Nord-Orientale, Casalecchio (Bologna); all others on a Gould-Sel 32/77 computer.

Discussion. Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Views of the molecules are shown in Fig. 1.

Least-squares-planes calculations show that the phenyl rings are planar to within 0·010 and 0·003 Å for BDDO and BDDM respectively, while some deformations are found in the aromatic rings of BDDP. This loss of planarity is probably related to the poor condition of the specimen and, consequently, to the low accuracy of the resulting geometrical data which limits its use as a guide to the discussion. The mean bond

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39039 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

lengths and internal angles of the rings are standard within the errors [1·386 (5), 1·379 (3), 1·410 (11) and 1·374 (26) Å; 119·7 (4), 119·8 (6), 120·6 (15) and 118·5 (14)°]. In accordance with the effects of substituents on the aromatic-ring skeleton, the internal angles at Cl atoms are all close to that expected [121·37 (9)°] (Domenicano, Vaciago & Coulson, 1975) and a narrowing is present in the adjacent ones. The C—Cl bond lengths of BDDO and BDDM [1·737 (3), 1·736 (3) Å] are in agreement with the compilation of Domenicano *et al.* (1975) [mean value 1·7404 (11) Å] and with the mean value [1·736 (2) Å] calculated by Palenik, Donohue & Trueblood (1968).

The sum of the bond angles around C(7) and C(12) is always ~360°, indicating that the carboxylic groups are planar. Moreover, the angles opposite the C=O bonds are invariably significantly smaller than the adjacent ones. This not surprising behaviour is one of the relationships found by Borthwick (1980) for a large number of —COO$^-$ fragments and was also found in a long series of derivatives analogous to those reported here (Pérez & Brisse, 1975, 1976*a,b*, 1977; Brisson & Brisse, 1982; Bocelli & Grenier-Loustalot, 1983*a,b,c,d;* and references cited therein).

Values for C—O bond lengths are 1·142 (10)–1·244 (14) Å if double and 1·335 (3)–1·466 (3) Å if single. These values are in good agreement with the usual findings. All bond distances and angles of the butane chains are normal and a relative shortening of some Csp^3—Csp^3 distances compared with the expected value of 1·537 (5) Å (Sutton, 1965) is not unusual in this type of derivative. It should be pointed out that the C(10)—C(11)—O(4) angle for BDDP [99·9 (6)°]is noticeably the narrowest found in this class of derivatives.

The carboxylic planes are tilted 31·2 (1), 3·3 (1), 4·6 (2) and 5·4 (3)° from the connected aromatic planes for the three compounds respectively. It is of interest to notice that the tilt observed for BDDO is markedly outside the normal range previously observed for these derivatives [0·3–12·9 (3)°] but in good agreement with those observed in 1,6-hexanediyl bis(*o*-chlorobenzoate) (Bocelli & Grenier-Loustalot, 1983*d*) [36·0 (1), 33·8 (1)°]. An increase of the tilt value was also observed in 2,3-butanediyl bis(*o*-chlorobenzoate) (Bocelli & Grenier-Loustalot, 1983*b*) [23·7 (1)°]. All these data clearly show that the tilts of the carboxylic moieties with respect to their respective phenyl rings are greatly influenced by the steric hindrance.

Perhaps the most interesting aspect of these three structures involves the conformations of the aliphatic chains. From a comparison of the selected torsion angles the conformational behaviour of the butanediyl part of the chain is clearly similar for BDDM and BDDP; their torsion angles are near to 180° and the conformation is all-*trans*. For BDDO, however, the O(2)—C(8)—C(9)—C(9′) torsion angle is 65·2 (3)° making the conformation of this part of the molecule *gauche*. Moreover, while the torsion angle C(7)—O(2)—C(8)—C(9) is 174·8 (2)° in BDDO, the departure of this angle from a *trans* conformation is larger for BDDM and BDDP [168·1 (3), 168·7 (7) and 169·3 (8)° respectively]. In conclusion, the conformation of the butanediyl chain which was *gauche–trans–trans* in the unchlorinated derivative (Bocelli & Grenier-Loustalot, 1982) becomes *gauche–trans–gauche* with a Cl atom in the *ortho* position and *trans–trans–trans* with Cl in a *meta* or *para* position.

In the crystals the molecules are linked to each other by van der Waals forces only (Figs. 2–4).

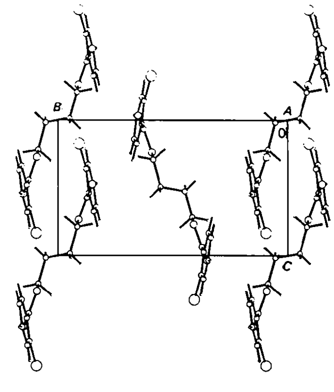

Fig. 2. Packing of BDDO along *x*.

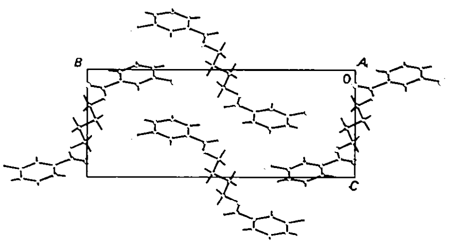

Fig. 3. Packing of BDDM along *x*.

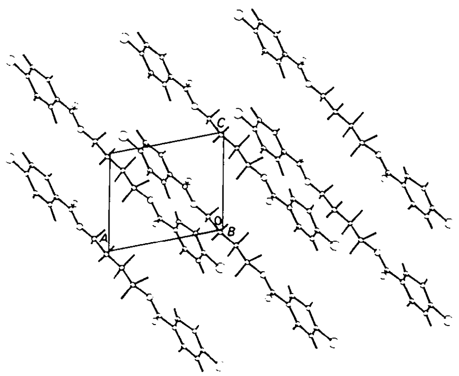

Fig. 4. Packing of BDDP along *y*.

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The Structure of the Five-Membered Cyclic Sultone 9,10-Dihydro-10a-methyl-3a,9-methano-3*H*,10a*H*-benzo[5,6]cyclohept[1,2-*d*][1,2]oxathiol-4(3a*H*)-one 2,2-Dioxide, $C_{14}H_{14}O_4S$

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Abstract. $M_r = 278{\cdot}2$, monoclinic, $P2_1/c$, $a = 11{\cdot}672\,(2)$, $b = 7{\cdot}717\,(1)$, $c = 14{\cdot}489\,(2)$ Å, $\beta = 101{\cdot}7\,(1)^\circ$, $U = 1277{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}446$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}250$ mm^{-1}, $F(000) = 584$, $T = 293$ K, $R = 0{\cdot}074$ for 1467 observed reflections. The structure is the first example of a saturated five-membered cyclic sultone. It is compared with known structures of partly unsaturated sultone rings. Bond lengths of interest are: mean acyclic S—O 1·417 (4), cyclic S—O 1·559 (3) and S—C 1·772 (7) Å.

Introduction. Our preliminary studies of the acid-catalysed reactions of alkyl-substituted homologues of tetrafluoro(1-methoxy)benzobarrelene (1) showed that trifluoroacetic acid gave products derived from simple rearrangements (Hales, Heaney & Ley, 1974). On the other hand, reactions carried out in sulphuric acid (98%) frequently gave products, derived from subsequent rearrangements, with more complex structures (Heaney & Ley, 1974; Hales & Heaney, 1975). The reactions of 1-methoxy-2,6-dimethylbenzobarrelene (2) and its tetrafluorobenzo analogue are such cases. The reactions of these compounds, but not the tetrachloro analogue, gave polar products, the structures of which could not be fully elucidated by a consideration of the available spectral data (Brown, Heaney, Ley, Mason & Singh, 1978). In particular, the stereochemistry of the sultone ring could not be deduced with certainty. It has been established that the ketones (3) and (4) also give the sultone in sulphuric acid and the fact that the tetrachloro compounds fail to react in an analogous manner suggests that a 1,2-acyl shift in the ketone (4) is a key step involved in the formation of the sultone (5).

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