

0.72 (4) Å to an idealized value of 1.03 Å, atom H(3) would be calculated to lie 0.29 (5) Å out of the plane defined above.

Since the structures of the 7- and 8-chloro derivatives appear to be substantially similar, little can be concluded concerning the difference between *in vitro* active molecules which are also active *in vivo* and those which are not. Evidently, 8-chloro substitution hinders absorption or transport without effecting a significant structural change.

The inactivity of the 4,9-dichloro compound is readily rationalized in the light of these observations. Evidently, it is the 4-chloro substituent which introduces the major structural changes, and these changes occur at or near the presumed active site. Although the active site in these molecules is not known, it is reasonable to assume that it involves the chemically active sites N(2), N(3), and/or N(5). As is apparent from Figs. 1 and 2, approach to these sites is relatively unhindered in the 7- and 8-chloro derivatives, as it is in the other analogues which have been studied (Eggleston *et al.*, 1984; Rychlewska *et al.*, 1984). It has been determined that *N,N*-dimethylformamide, a small acceptor molecule, can interact strongly with N(3) of clofazimine in the solid state (Eggleston *et al.*, 1984) and it seems feasible that small donor molecules could easily approach N(2) and/or N(5). Thus, in all of these active (*in vitro*) analogues, approach to the nitrogen atoms is unhindered and the geometry at N(3) is approximately constant. In the inactive 4,9-dichloro analogue, however, approach to N(5) is presumably hindered by the presence of Cl(4) (see Fig. 3), and the geometry and potential hydrogen-bonding capacity of the N(3) site are entirely different than in these other

compounds. Structural and theoretical studies on other active and inactive analogues which are currently in progress should permit us to determine which of these features is of the greater significance.

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(±)-2,3-Dihydroxy-2,3-dimethylbutanedioic Acid [(±)-Dimethyltartaric Acid], C₆H₁₀O₆

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Abstract. $M_r = 178.2$, triclinic, $P\bar{1}$, $a = 8.632$ (4), $b = 10.064$ (3), $c = 10.505$ (3) Å, $\alpha = 114.22$ (2), $\beta = 106.71$ (2), $\gamma = 92.92$ (3)°, $V = 782.1$ (5) Å³, $Z = 4$, $D_x = 1.51$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.13$ mm⁻¹, $F(000) = 376$, $T = 293$ (2) K, final $R = 0.058$ for 2086 observed unique reflections. The poorly staggered conformations are (–)-synclinal and (+)-

synclinal for *R,R* and *S,S* molecules respectively. The torsion-angle deviation from perfect staggering of about 17° is toward a closer contact for the methyl substituents. As also found for the *R,S* (*meso*) modification, the C–C bond lengths are significantly greater than those which have been determined for the parent compound, tartaric acid.

Introduction. 2,3-Dihydroxy-2,3-dimethylbutanedioic acid, $\text{HOCC}(\text{CH}_3)(\text{OH})\text{C}(\text{CH}_3)(\text{OH})\text{COOH}$ (dimethyltartaric acid), exists as an *R,S* (*meso*) isomer and as the *R,R* and *S,S* pair of enantiomers. Recently we reported the structure of the *meso* isomer (Duesler, Mondragon & Tapscott, 1984). Here, as a continuation of studies on the stereochemistry of the tartrates and their derivatives, we report the structure of the racemic modification, (±)-dimethyltartaric acid.

Experimental. Dimethyltartaric acid was prepared and its isomers were separated by literature methods (Leermakers & Vesley, 1963; Tatsumi, Izumi, Imaida, Fukuda & Akabori, 1966). Crystal obtained by evaporation of ethyl acetate/ethanol solution, prism with dimensions $0.12 \times 0.18 \times 0.28$ mm. Syntex P3/F diffractometer, graphite monochromator. Lattice parameters from 22 reflections ($4 < 2\theta < 21^\circ$). Empirical absorption correction based on ψ scans; min. and max. transmission coefficients 0.891 and 0.944. θ - 2θ scan, $(\sin\theta)/\lambda = 0.01$ to 0.59 \AA^{-1} ($1 \leq 2\theta \leq 50^\circ$, $\pm h, \pm k, \pm l$). Three standard reflections (400, 030, 004) every 96 reflections (8% variation). 2899 reflections measured, 2708 unique ($R_{\text{int}} = 0.019$ for merge), 622 unobserved [$I < 2.0\sigma(I)$]. No secondary-extinction correction. The space group could have been either *P1* or *P1*; however, successful refinement indicated that the latter was the correct choice. All computer programs taken from the *SHELXTL* package (Sheldrick, 1981) and scattering-factor data from *International Tables for X-ray Crystallography* (1974). Structure solved by direct methods. H atoms were located from difference maps and their positions were allowed to vary but their isotropic thermal parameters were fixed at 1.1 times the last value of U_{eq} for the atom to which they were attached. Anisotropic refinement on *F* of all non-H atoms with isotropic refinement of H atoms (fixed U_{iso}) converged to $R = 0.058$, $wR = 0.054$ [$w^{-1} = \sigma(F)^2 + |q|F_o^2$ with $q = 0.00104$], $S = 1.14$ for 2086 observations and 277 refined variables. Max. and min. peak heights in final difference map 0.26 and -0.33 e \AA^{-3} . Max. and mean Δ/σ during final cycle of refinement 0.021 and 0.006.

Discussion. The final fractional coordinates for all atoms and the U_{eq} values for the non-H atoms are given in Table 1.*

The asymmetric unit consists of an enantiomeric pair of crystallographically distinct molecules connected by a hydrogen bond from a hydroxyl O atom of one to a carboxyl H of the other (Fig. 1). Like the *meso* isomer

Table 1. Atomic coordinates and equivalent isotropic thermal parameters for (±)- $\text{C}_6\text{H}_{10}\text{O}_6$

Coordinates in fractions of the unit-cell edge. Equivalent isotropic thermal parameters are calculated as $U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ with the U_{ii} 's in an orthogonal coordinate system (Willis & Pryor, 1975) and the original anisotropic thermal parameters defined by $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

	x	y	z	$U_{\text{eq}}(\text{\AA}^2)$
C(1)	0.8481 (3)	0.1766 (3)	0.0470 (3)	0.029 (1)
C(2)	0.8999 (3)	0.1921 (3)	0.2059 (3)	0.026 (1)
C(3)	0.7844 (3)	0.0670 (3)	0.2077 (3)	0.028 (1)
C(4)	0.8772 (3)	0.0382 (3)	0.3390 (3)	0.028 (1)
C(5)	0.9057 (4)	0.3491 (3)	0.3165 (3)	0.034 (1)
C(6)	0.6179 (4)	0.1028 (3)	0.2184 (3)	0.034 (1)
O(1)	0.9280 (3)	0.1316 (3)	-0.0326 (2)	0.049 (1)
O(2)	0.7074 (3)	0.2199 (2)	0.0091 (2)	0.040 (1)
O(3)	1.0641 (2)	0.1662 (2)	0.2419 (2)	0.033 (1)
O(4)	0.7516 (3)	-0.0645 (2)	0.0740 (2)	0.034 (1)
O(5)	0.9283 (3)	-0.0794 (2)	0.3163 (2)	0.042 (1)
O(6)	0.8984 (3)	0.1424 (2)	0.4694 (2)	0.043 (1)
C(7)	0.3906 (3)	0.2210 (3)	-0.4542 (3)	0.030 (1)
C(8)	0.5164 (3)	0.3005 (3)	-0.2977 (3)	0.027 (1)
C(9)	0.6427 (3)	0.4239 (3)	-0.2904 (3)	0.028 (1)
C(10)	0.8017 (3)	0.4575 (3)	-0.1616 (3)	0.030 (1)
C(11)	0.4257 (4)	0.3539 (3)	-0.1866 (3)	0.035 (1)
C(12)	0.5821 (4)	0.5686 (3)	-0.2720 (4)	0.038 (2)
O(7)	0.2930 (2)	0.3066 (2)	-0.4878 (2)	0.042 (1)
O(8)	0.3780 (3)	0.0930 (2)	-0.5351 (2)	0.056 (1)
O(9)	0.6087 (3)	0.1958 (2)	-0.2699 (2)	0.034 (1)
O(10)	0.6788 (2)	0.3678 (2)	-0.4236 (2)	0.039 (1)
O(11)	0.9281 (2)	0.4234 (2)	-0.1837 (2)	0.042 (1)
O(12)	0.7894 (3)	0.5261 (3)	-0.0310 (2)	0.045 (1)
H(5A)	0.943 (4)	0.355 (3)	0.415 (3)	
H(5B)	0.984 (4)	0.418 (3)	0.312 (3)	
H(5C)	0.805 (4)	0.380 (3)	0.291 (3)	
H(6A)	0.630 (3)	0.194 (3)	0.311 (3)	
H(6B)	0.558 (3)	0.120 (3)	0.130 (3)	
H(6C)	0.556 (3)	0.024 (3)	0.215 (3)	
H(11A)	0.499 (3)	0.396 (3)	-0.093 (3)	
H(11B)	0.362 (3)	0.432 (3)	-0.196 (3)	
H(11C)	0.355 (3)	0.269 (3)	-0.198 (3)	
H(12A)	0.484 (4)	0.545 (3)	-0.361 (3)	
H(12B)	0.562 (4)	0.612 (3)	-0.177 (3)	
H(12C)	0.668 (4)	0.638 (3)	-0.263 (3)	
H(2)	0.685 (4)	0.204 (3)	-0.083 (4)	
H(3)	1.071 (3)	0.095 (3)	0.183 (3)	
H(4)	0.821 (4)	-0.103 (3)	0.075 (4)	
H(6)	0.956 (4)	0.124 (3)	0.550 (3)	
H(7)	0.223 (4)	0.257 (3)	-0.577 (3)	
H(9)	0.589 (3)	0.124 (3)	-0.342 (3)	
H(10)	0.738 (4)	0.314 (3)	-0.420 (3)	
H(12)	0.881 (4)	0.543 (4)	0.039 (4)	

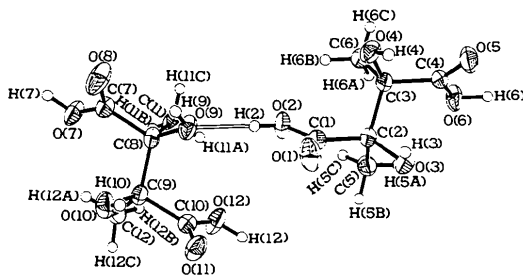


Fig. 1. View of a pair of crystallographically distinct molecules present in the racemic modification of dimethyltartaric acid. The hydrogen bond between the two molecules is shown as an open line. The thermal ellipsoids (except for the atoms which are shown as arbitrarily sized spheres) are shown at the 50% probability level.

* Tables of structure factors, anisotropic thermal parameters and fixed H-atom thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39591 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å), bond angles and torsion angles (°) involving non-H atoms with *e.s.d.*'s in parentheses for (±)-C₆H₁₀O₆

C(1)–C(2)	1.537 (5)	C(1)–O(2)	1.319 (4)
C(2)–C(3)	1.574 (5)	C(2)–O(3)	1.425 (3)
C(2)–C(5)	1.517 (4)	C(3)–O(4)	1.418 (3)
C(3)–C(4)	1.532 (5)	C(4)–O(5)	1.239 (4)
C(3)–C(6)	1.523 (4)	C(4)–O(6)	1.290 (3)
C(7)–C(8)	1.526 (3)	C(7)–O(7)	1.315 (4)
C(8)–C(9)	1.573 (5)	C(7)–O(8)	1.195 (3)
C(8)–C(11)	1.523 (5)	C(8)–O(9)	1.426 (4)
C(9)–C(10)	1.530 (4)	C(9)–O(10)	1.412 (4)
C(9)–C(12)	1.529 (5)	C(10)–O(11)	1.217 (4)
C(1)–O(1)	1.198 (4)	C(10)–O(12)	1.298 (4)
C(3)–C(4)–O(6)	116.2 (3)	C(1)–C(2)–C(3)	108.4 (2)
C(4)–C(3)–O(4)	109.1 (2)	C(1)–C(2)–C(5)	111.1 (3)
C(5)–C(2)–O(3)	106.5 (2)	C(2)–C(3)–C(4)	107.7 (2)
C(6)–C(3)–O(4)	106.7 (2)	C(2)–C(3)–C(6)	114.7 (3)
C(7)–C(8)–O(9)	108.1 (2)	C(3)–C(2)–C(5)	115.1 (3)
C(8)–C(7)–O(7)	112.8 (2)	C(4)–C(3)–C(6)	109.6 (3)
C(8)–C(7)–O(8)	123.5 (3)	C(7)–C(8)–C(9)	109.2 (3)
C(8)–C(9)–O(10)	108.5 (2)	C(7)–C(8)–C(11)	109.1 (2)
C(9)–C(8)–O(9)	106.5 (2)	C(8)–C(9)–C(10)	108.4 (3)
C(9)–C(10)–O(11)	121.2 (3)	C(8)–C(9)–C(12)	114.9 (3)
C(9)–C(10)–O(12)	114.5 (3)	C(9)–C(8)–C(11)	115.7 (2)
C(10)–C(9)–O(10)	108.4 (2)	C(10)–C(9)–C(12)	109.1 (2)
C(11)–C(8)–O(9)	108.0 (3)	C(1)–C(2)–O(3)	107.1 (3)
C(12)–C(9)–O(10)	107.3 (3)	C(2)–C(1)–O(1)	123.4 (3)
O(1)–C(1)–O(2)	124.0 (3)	C(2)–C(1)–O(2)	112.6 (3)
O(5)–C(4)–O(6)	123.9 (3)	C(2)–C(3)–O(4)	108.8 (3)
O(7)–C(7)–O(8)	123.7 (2)	C(3)–C(2)–O(3)	108.3 (3)
O(11)–C(10)–O(12)	124.3 (2)	C(3)–C(4)–O(5)	119.8 (2)
O(1)–C(1)–C(2)–O(3)	8.8 (4)	C(5)–C(2)–C(3)–C(6)	43.2 (3)
O(4)–C(3)–C(4)–O(5)	8.7 (4)	C(11)–C(8)–C(9)–C(12)	–42.9 (3)*
O(8)–C(7)–C(8)–O(9)	–0.6 (5)*	O(3)–C(2)–C(3)–O(4)	–78.3 (3)
O(11)–C(10)–C(9)–O(10)	–6.6 (4)*	O(9)–C(8)–C(9)–O(10)	77.0 (3)*

* These angles are for molecules having an *S,S* absolute configuration. The remaining data are for *R,R* isomers.

of dimethyltartaric acid (Duesler *et al.*, 1984), the C–C bond lengths found for (±)-dimethyltartaric acid (Table 2) are significantly longer than those which have been reported for corresponding parameters in four independent structures of isomers of the non-methyl-substituted parent compound (Okaya, Stemple & Kay, 1966; Bootsma & Schoone, 1967).

The torsion angles observed for the (±)-dimethyltartaric acid molecules (Table 2) are of some interest. Past studies have shown that the small deviations from planarity observed for α-hydroxycarboxylate groups are usually toward a (–)-synperiplanar* conformation (negative O–C–C–O angle) for groups containing a carbinol C atom of *S* absolute configuration (though unsubstituted tartaric acids seem to be an exception) (Tapscott, 1982). We see such a trend here also.

The overall conformations of the dimethyltartaric acid molecules are (–)-synclinal for the *R,R* enantiomers and (+)-synclinal for the *S,S* enantiomers. These are the same conformations found for racemic and active isomers of unsubstituted tartaric acid and its simple salts (Tapscott, 1982); however, unlike the non-methyl-substituted compounds (Tapscott, Belford

& Paul, 1969), the two crystallographically distinct (±)-dimethyltartaric acid molecules are not at all well staggered. Particularly surprising about this is that the torsion-angle deviations from perfect staggering (60°) are toward a closer contact for the presumably bulkier methyl groups (H₃C–C–C–CH₃ torsion angles, 43°) than observed for H atoms of (+)-tartaric acid [53° torsion angle calculated from neutron diffraction data reported by Okaya *et al.* (1966)].

The crystal packing is shown in Fig. 2 and the rather complex hydrogen-bonding scheme proposed is described in Table 3. Three of the proposed interactions, all involving bifurcated bonds [O(4)–H(4)···O(5), O(9)–H(9)···O(8), and O(10)–H(10)···O(11)] have O–H···O angles considerably smaller than those normally found for hydrogen-bonded systems (Hamilton & Ibers, 1968). On the other hand, these three interactions are internal to α-hydroxycarboxylate groupings and very similar associations observed for other α-hydroxycarboxylic acids have been described as hydrogen bonds (Tapscott, 1982). Each crystallographically distinct molecule is paired with a second of the same type by hydrogen bonding of carboxylic

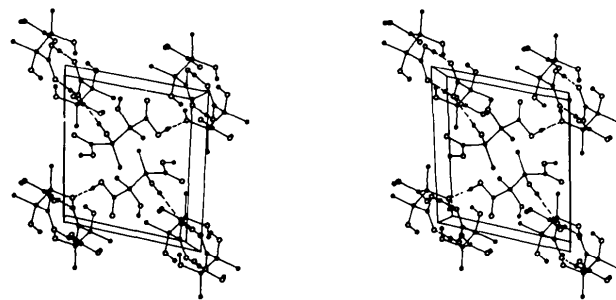


Fig. 2. Stereoview of the crystal packing for (±)-C₆H₁₀O₆ as viewed down the *c* axis. The origin lies at the back of the upper right corner with the *a* axis pointing to the left, the *b* axis pointing to the bottom, and the *c* axis projecting toward the viewer. The atoms are shown as spheres of arbitrary size (C atoms, filled spheres; O atoms, larger open spheres; H atoms, smaller open spheres). For clarity, the methyl-group H atoms are not shown. Dotted lines depict hydrogen bonds.

Table 3. Distances (Å) and angles (°) for hydrogen bonds

	O···O	O–H	H···O	O–H···O
O(2)–H(2)···O(9)	2.708 (3)	0.87 (4)	1.85 (4)	169 (4)
O(3)–H(3)···O(11)	2.919 (4)	0.75 (3)	2.18 (4)	169 (3)
O(4)–H(4)···O(11)	2.989 (4)	0.73 (3)	2.34 (4)	148 (4)
O(4)–H(4)···O(5)	2.633 (3)	0.73 (3)	2.34 (4)	106 (3)
O(6)–H(6)···O(5 ⁱⁱ)	2.653 (3)	0.94 (4)	1.71 (3)	174 (3)
O(7)–H(7)···O(3 ⁱⁱⁱ)	2.676 (3)	0.86 (2)	1.81 (4)	174 (3)
O(9)–H(9)···O(8)	2.652 (3)	0.77 (2)	2.20 (4)	119 (3)
O(9)–H(9)···O(8 ^{iv})	2.818 (3)	0.77 (2)	2.12 (4)	152 (3)
O(10)–H(10)···O(11)	2.626 (3)	0.77 (3)	2.30 (4)	107 (3)
O(10)–H(10)···O(6 ^v)	3.094 (3)	0.77 (3)	2.40 (3)	150 (3)
O(12)–H(12)···O(11)	2.658 (3)	0.86 (3)	1.79 (4)	180 (4)

Symmetry code: (i) 2 – *x*, \bar{y} , \bar{z} ; (ii) 2 – *x*, \bar{y} , 1 – *z*; (iii) *x* – 1, *y*, *z* – 1; (iv) 1 – *x*, \bar{y} , –1 – *z*; (v) *x*, *y*, *z* – 1.

* See Klyne & Prelog (1960) for definitions of the conformation designations.

acid groups about a center of symmetry. These dimers are then joined laterally to other dimers of the same type. Crystallographically different molecules are joined only by the hydrogen bonds O(2)—H(2)···O(9) and O(7)—H(7)···O(3).

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Structure of Bis(phenylsulphonyl) Trisulphide, C₁₂H₁₀O₄S₃

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Abstract. $M_r = 378.52$, tetragonal, $P4_12_2$, $a = 7.758$ (1), $c = 26.204$ (4) Å, $V = 1577.1$ (1) Å³, $Z = 4$, $D_m = 1.56$, $D_x = 1.594$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7093$ Å, $\mu(\text{Mo } K\alpha) = 0.715$ mm⁻¹, $T = 298$ K, $F(000) = 776$, $R = 0.042$ for 1146 observed reflections. The molecule contains a twofold axis. There are two kinds of S—S bond lengths: 2.026 (1) and 2.102 (1) Å, the longer bonds involving the S atoms with the dioxide substituents.

Introduction. The nature of the S—S bond has been of great interest for many decades. The structures of certain polysulphides and sulphonylsulphides were investigated by Dawson, Mathieson & Robertson (1948) and Mathieson & Robertson (1949). The S chain skeleton was confirmed later for various polysulphides of disulphonate (O₃S—S_n—SO₃)²⁻ compounds (Stewart & Szymański, 1979*a,b*) and ϕ -SO_x-S_n-SO_y- ϕ (Kiers & Vos, 1978). In order to compare the conformation of the polysulphide chain and to understand the effect of O substituents on the S—S bond, the title compound was synthesized by the method of

Troeger & Hornung (1899) and characterized by structure determination.

Experimental. Crystal obtained by slow evaporation of acetic acid solution. 390 μm diameter spherical crystal. CAD-4 diffractometer. 16 reflections in 2θ range 26°–31° used in the refinement of the cell parameters. D_m by flotation method (CCl₄H/CBr₃H). Spherical absorption correction applied. $2\theta_{\max} = 70^\circ$, $-12 \leq h \leq 12$, $-42 \leq l \leq 42$, and $h > k$. Three standard reflections monitored every hour, variation less than 3%, 3468 unique reflections, 1146 of which observed (including Friedel pairs) with $I \geq 2\sigma(I)$. $R = 0.042$, $R_w = 0.020$, $w = 4F_o^2/\sigma^2(I)$ where $\sigma^2(I) = (P + t^2B)$. Structure solved by Patterson method. H atoms calculated, and then refined in the least-squares process. All non-hydrogen and H atoms refined anisotropically and isotropically, respectively. In final cycle $(\Delta/\sigma)_{\max} = 0.71$. Peaks on final difference Fourier synthesis between 0.29 and -0.30 e Å⁻³. Secondary-extinction-coefficient value 0.24 (length in μm). Atomic scattering factors calculated by the analytical form using the coefficients given in *International Tables for X-ray Crystallography* (1974); anomalous-dispersion

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