

Table 1. Atomic coordinates and equivalent isotropic temperature factors

	x	y	z	$B_{eq}(\text{\AA}^2)^*$
S(1)	0.6641 (1)	0.7876 (1)	0.50000	3.19 (4)
C(1)	0.6608 (2)	0.5859 (2)	0.5209 (2)	3.16 (8)
C(2)	0.5812 (3)	0.4904 (2)	0.4531 (3)	3.80 (9)
C(3)	0.5874 (3)	0.3332 (3)	0.4767 (4)	5.72 (14)
C(4)	0.6690 (4)	0.2751 (4)	0.5645 (5)	6.38 (18)
C(5)	0.7457 (3)	0.3721 (4)	0.6297 (3)	5.80 (15)
C(6)	0.7426 (3)	0.5292 (3)	0.6088 (3)	4.54 (10)
C(7)	0.4880 (4)	0.5467 (3)	0.3602 (3)	5.16 (12)
O(1)	0.5180 (2)	0.8399 (2)	0.5090 (2)	3.62 (6)
O(2)	0.7188 (2)	0.8164 (2)	0.3883 (2)	4.72 (9)
O(3)	0.7456 (2)	0.8530 (3)	0.5910 (2)	5.57 (9)
O(W1)	0.3881 (2)	0.9559 (2)	0.3327 (2)	4.40 (8)
O(W2)	0.5682 (2)	1.0337 (2)	0.2007 (2)	4.66 (8)

$$*B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i \cdot a_j$$

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

S(1)—C(1)	1.771 (2)	S(1)—O(1)	1.473 (2)
S(1)—O(2)	1.447 (2)	S(1)—O(3)	1.448 (2)
C(1)—C(2)	1.384 (3)	C(1)—C(6)	1.394 (4)
C(2)—C(3)	1.396 (3)	C(2)—C(7)	1.500 (5)
C(3)—C(4)	1.396 (6)	C(4)—C(5)	1.359 (6)
C(5)—C(6)	1.388 (4)		
C(1)—S(1)—O(1)	106.2 (1)	C(1)—S(1)—O(2)	107.8 (1)
C(1)—S(1)—O(3)	107.1 (1)	O(1)—S(1)—O(2)	110.9 (1)
O(1)—S(1)—O(3)	109.6 (1)	O(2)—S(1)—O(3)	114.9 (1)
S(1)—C(1)—C(2)	121.5 (2)	S(1)—C(1)—C(6)	116.4 (2)
C(2)—C(1)—C(6)	122.1 (2)	C(1)—C(2)—C(3)	116.6 (3)
C(1)—C(2)—C(7)	123.9 (2)	C(3)—C(2)—C(7)	119.5 (3)
C(2)—C(3)—C(4)	121.8 (3)	C(3)—C(4)—C(5)	120.1 (3)
C(4)—C(5)—C(6)	119.8 (3)	C(1)—C(6)—C(5)	119.6 (3)

bonding linkage runs through the crystal along the *c* axis. The sulfonate O(1) atom accepts two hydrogen bonds from the water molecules in contrast to the O(2) and O(3) atoms accepting one hydrogen bond.

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## Structure of 1,3,3,5-Tetramethylcyclohexanol

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**Abstract.**  $C_{10}H_{20}O$ ,  $M_r = 156.27$ , tetragonal,  $I\bar{4}$ ,  $a = b = 16.600$  (12),  $c = 7.337$  (3)  $\text{\AA}$ ,  $V = 2022$  (2)  $\text{\AA}^3$ ,  $Z = 8$ ,  $D_m$  (floatation) = 0.96 (2),  $D_x = 1.03$   $\text{g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha_1) = 0.7107$   $\text{\AA}$ ,  $\mu = 0.34$   $\text{cm}^{-1}$ ,  $F(000) = 704$ ,  $T = 184$  K,  $R(F) = 0.062$  for 1288 independent

The lone pair on the sulfonate O(1) atom almost coincides with the hydrogen-bond direction with S—O(1)⋯O angles of 118.9 (2) and 120.5 (2)°. On the other hand, the O(W1)H⋯O(2) hydrogen bond with an S—O(2)⋯O angle of 125.3 (2)° is also satisfactory for the O(2) lone pair, but the O(W2)⋯O(3) hydrogen bond with an S—O(3)⋯O angle of 160.0 (2)° largely deviates from the lone-pair direction of the O(3) atom. Thus the O(W2)H⋯O(3) hydrogen bond is weaker than the others. From such asymmetry, the  $H_5O_2^+$  cation can be assumed to be an aquaoxonium ion with the  $H_3O(W1)^+$  ion bonded to the normal  $H_2O(W2)$  molecule, because the O⋯O distances around O(W1) are shorter than those around O(W2) (Fig. 1b), and the refined H atom between the two water molecules is closer to O(W1).

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Table 1. Fractional positional parameters and  $B_{eq}$  for non-H atoms and  $B_{iso}$  for H atoms with *e.s.d.*'s in parentheses

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$B_{eq}/B_{iso} (\text{\AA}^2)$
C(1)	0.1710 (1)	0.0194 (1)	-0.1687 (3)	1.68
C(2)	0.2461 (1)	-0.0290 (1)	-0.1158 (3)	1.91
C(3)	0.3024 (1)	0.0095 (1)	0.0261 (3)	2.11
C(4)	0.3196 (1)	0.0972 (1)	-0.0276 (4)	2.31
C(5)	0.2440 (1)	0.1467 (1)	-0.0643 (3)	2.00
C(6)	0.1943 (1)	0.1059 (1)	-0.2144 (3)	2.03
C(7)	0.1284 (2)	-0.0200 (2)	-0.3306 (4)	2.52
C(8)	0.3822 (2)	-0.0378 (2)	0.0242 (6)	3.40
C(9)	0.2674 (2)	0.0051 (2)	0.2192 (4)	2.68
C(10)	0.2658 (2)	0.2328 (1)	-0.1181 (4)	2.73
O(1)	0.1154 (1)	0.0240 (1)	-0.0180 (2)	1.89
H(2A)	0.221 (2)	-0.086 (2)	-0.074 (5)	2.5 (6)
H(2B)	0.273 (2)	-0.037 (2)	-0.227 (5)	1.7 (6)
H(4A)	0.349 (3)	0.097 (3)	-0.141 (10)	5.5 (11)
H(4B)	0.354 (2)	0.121 (2)	0.076 (6)	3.6 (8)
H(5A)	0.215 (2)	0.151 (2)	0.052 (5)	1.9 (6)
H(6A)	0.219 (3)	0.110 (3)	-0.327 (9)	5.3 (10)
H(6B)	0.139 (2)	-0.141 (2)	-0.232 (6)	3.1 (6)
H(7A)	0.091 (2)	-0.020 (2)	-0.009 (6)	2.8 (6)
H(7B)	0.079 (2)	0.015 (2)	-0.365 (6)	2.7 (6)
H(7C)	0.157 (2)	-0.020 (2)	-0.437 (5)	2.1 (6)
H(8A)	0.120 (3)	-0.078 (3)	-0.293 (10)	6.6 (12)
H(8B)	0.419 (3)	-0.022 (3)	0.126 (7)	4.2 (9)
H(8C)	0.409 (4)	-0.033 (3)	-0.093 (10)	6.4 (13)
H(8D)	0.373 (3)	-0.095 (2)	0.053 (7)	4.0 (8)
H(9A)	0.257 (3)	-0.051 (3)	0.271 (9)	6.1 (12)
H(9B)	0.220 (2)	0.038 (2)	0.227 (5)	2.4 (6)
H(9C)	0.301 (2)	0.027 (2)	0.310 (6)	2.8 (6)
H(10A)	0.215 (3)	0.271 (3)	-0.133 (7)	4.3 (10)
H(10B)	0.295 (3)	0.231 (3)	-0.219 (8)	5.2 (11)
H(10C)	0.295 (3)	0.257 (3)	-0.024 (7)	4.4 (9)

**Introduction.** This study was undertaken to confirm the results of our NMR lanthanide shift reagent studies on 1,3,3,5-tetramethylcyclohexanol. The synthesis of the compound (Pearson, 1978) created asymmetric centers at C(1) and C(5). The conformation which best fits the NMR results is C(1) and C(5) methyl groups equatorial and OH axial. This study also confirms the predictions of bond and torsional angles made by *MM2* molecular-mechanics calculations.

**Experimental.** The compound was prepared (Pearson, 1978) by hydrogenation of the 5,6 double bond in isophorone and Grignard addition of  $\text{CH}_3\text{MgI}$  across the carbonyl group and subsequent hydrolysis. A colorless crystal,  $0.5 \times 0.6 \times 1.0$  mm, was mounted in a sealed capillary tube and cooled to 184 K. Unit-cell parameters refined from  $\theta$  measurements of 25 reflections. Intensities of 1417 reflections measured using  $\omega/2\theta$  scans with a CAD-4 diffractometer,  $0 \leq \theta \leq 28^\circ$ ,  $0 \leq h \leq 21$ ,  $0 \leq k \leq 21$ ,  $0 \leq l \leq 9$  with three standard reflections showing no significant variations throughout exposure. No absorption or extinction corrections applied.

Structure solution using *MULTAN* (Germain, Main & Woolfson, 1971). Full-matrix least-squares refinements on 1288 independent reflections with  $I > 0$  ( $R_{int} = 0.058$ ) with *SHELX76* (Sheldrick, 1976), eight H atoms attached to C atoms located from an

$F_o - F_c$  synthesis, the rest calculated from ideal geometry. The OH H atom was located from an  $F_o - F_c$  synthesis, in which the O atom, all the C atoms, and the other 19 H atoms were included in the  $F_c$  calculations. H atoms refined isotropically, non-H atoms refined anisotropically.  $R(F) = 0.062$ ,  $wR = 0.078$ ,  $S = 0.98$ ,  $w = 0.0158/[\sigma^2(F) + 0.006F^2]$ ,  $\Delta/\sigma_{max} = 0.062$ ,  $\Delta\rho_{max} = 0.35$ ,  $\Delta\rho_{min} = -0.46 \text{ e \AA}^{-3}$ . Atomic scattering factors for non-H atoms from *International Tables for X-ray Crystallography* (1968) and for H atoms from Stewart, Davidson & Simpson (1965).

**Discussion.** Atomic positions and equivalent isotropic temperature factors are shown in Table 1.\* A drawing of the molecule is given in Fig. 1. The

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52938 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

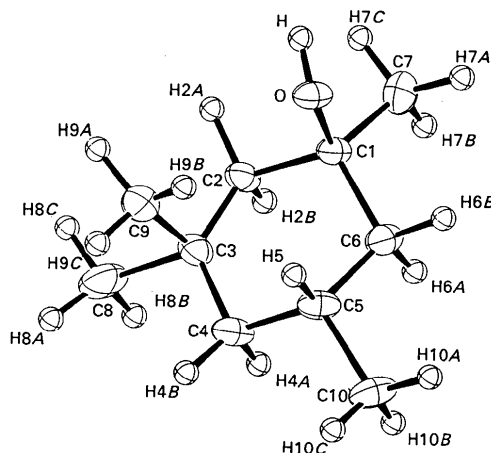


Fig. 1. Thermal ellipsoids in 1,3,3,5-tetramethylcyclohexanol.

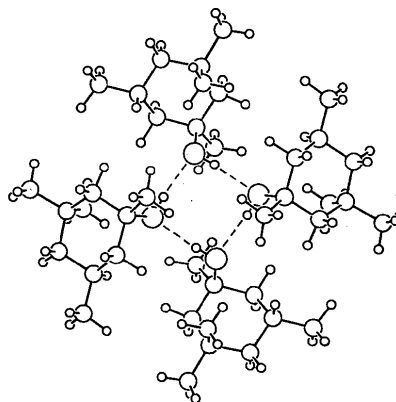


Fig. 2. Hydrogen-bonded tetramers about the  $\bar{4}$  axis.

Table 2. Selected bond distances (Å) and angles (°)

C(1)—C(2)	1.533 (3)	C(1)—C(7)	1.529 (3)
C(2)—C(3)	1.538 (3)	C(3)—C(8)	1.540 (4)
C(3)—C(4)	1.535 (3)	C(3)—C(9)	1.533 (3)
C(4)—C(5)	1.524 (3)	C(5)—C(10)	1.526 (3)
C(5)—C(6)	1.534 (3)	O—H(O)	0.84 (4)
C(6)—C(1)	1.524 (3)	H(O)⋯O'	1.95 (4)
C(1)—O	1.442 (3)	O⋯O'	2.780 (2)
	Experiment		MM2 molecular mechanics
C(6)—C(1)—C(2)	110.1 (3)		110.6
C(1)—C(2)—C(3)	116.6 (3)		116.4
C(2)—C(3)—C(4)	109.6 (3)		109.8
C(3)—C(4)—C(5)	113.8 (3)		113.7
C(4)—C(5)—C(6)	109.4 (3)		110.1
C(5)—C(6)—C(1)	113.2 (3)		112.4
O—C(1)—C(2)	110.7 (3)		
O—C(1)—C(6)	106.3 (3)		
O—C(1)—C(7)	108.8 (3)		
C(7)—C(1)—C(2)	110.4 (3)		
C(7)—C(1)—C(6)	110.4 (3)		
C(8)—C(3)—C(2)	107.7 (3)		
C(8)—C(3)—C(4)	108.7 (3)		
C(8)—C(3)—C(9)	108.1 (4)		
C(9)—C(3)—C(2)	112.1 (4)		
C(9)—C(3)—C(4)	110.7 (3)		
C(10)—C(5)—C(4)	110.8 (3)		
C(10)—C(5)—C(6)	110.8 (3)		
O—H(O)⋯O'	174 (5)		
C(6)—C(1)—C(2)—C(3)	+ 49.1 (4)		+ 49.2
C(1)—C(2)—C(3)—C(4)	- 48.3 (4)		- 48.0
C(2)—C(3)—C(4)—C(5)	+ 52.0 (4)		+ 51.3
C(3)—C(4)—C(5)—C(6)	- 57.2 (4)		- 56.8
C(4)—C(5)—C(6)—C(1)	+ 57.0 (4)		+ 57.0
C(5)—C(6)—C(1)—C(2)	- 52.7 (4)		- 52.7

methyl groups attached to C(1) and C(5) are equatorial, in agreement with previous NMR studies. The molecules are arranged in tetramers about the 4 axis with the O—H groups arranged as four-membered hydrogen-bonded rings. This implies that both enantiomers are present in the crystals. The OH groups in the hydrogen bonds are ordered. No evi-

dence was found for two half-hydrogens between O atoms characteristic of the structure of ice (Peterson & Levy, 1957). Fig. 2 illustrates the hydrogen bonding. The non-hydrogen bond distances, bond angles and torsion angles in the cyclohexanol ring are listed in Table 2. Molecular-mechanics calculations using the MM2 computer program (Allinger & Yuh, 1980) were performed on tetramethylcyclohexanol. The predictions of bond and torsion angles for the minimum-energy conformation are included for comparison in Table 2. The experimental results are in agreement with the deviations of bond angles from the value of 109.5° and torsional angles from the value of 54.9° for cyclohexane.

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## Structure of *N*- $\gamma$ -L-Glutamyl-L-cysteine Ethyl Ester Monohydrate

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**Abstract.** C<sub>10</sub>H<sub>18</sub>N<sub>2</sub>O<sub>5</sub>S.H<sub>2</sub>O,  $M_r = 296.39$ , monoclinic,  $P2_1$ ,  $a = 19.600$  (1),  $b = 7.480$  (1),  $c = 5.061$  (1) Å,  $\beta = 94.74$  (1)°,  $V = 739.445$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.31$ ,  $D_x = 1.331$  g cm<sup>-3</sup>,  $Cu K\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 21.4$  cm<sup>-1</sup>,  $F(000) = 316$ , room temperature,  $R = 0.057$  for 1263 reflections. The molecule adopts an extended form. The molecules are arranged so as to form double molecular sheets parallel to the  $bc$  plane through some hydrogen bonds.

**Introduction.** Glutathione is present in almost all biological tissues as a major intracellular reducing agent and plays important roles in catalysis, metabolism, transport and protection in cells. The title compound is a precursor of  $\gamma$ -L-glu-L-cys, which is known as an intermediate in the biosynthesis of glutathione. This compound has anti-cataractogenic properties (Ohtsu, Kitahara & Fujii, 1990) and has potential for the treatment of kidney and liver diseases. X-ray analysis of this drug has been performed