

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*-γ-L-Glutamyl-L-cysteine Ethyl Ester Monohydrate

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Abstract. C₁₀H₁₈N₂O₅S.H₂O, *M_r* = 296.39, monoclinic, *P*2₁, *a* = 19.600 (1), *b* = 7.480 (1), *c* = 5.061 (1) Å, β = 94.74 (1)°, *V* = 739.445 (1) Å³, *Z* = 2, *D_m* = 1.31, *D_x* = 1.331 g cm⁻³, *Cu Kα*, λ = 1.54178 Å, μ = 21.4 cm⁻¹, *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 γ-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

Table 1. Atomic coordinates and equivalent isotropic temperature factors

$B_{eq} = 8\pi^2(U_1 + U_2 + U_3)/3$, where U_1 , U_2 and U_3 are the principal components of the mean-square displacement matrix U . Values in parentheses are e.s.d.'s and those in angle brackets give the anisotropy, defined by $[\sum(B_{eq} - 8\pi^2U_i)^2/3]^{1/2}$; they refer to the last decimal places.

	x	y	z	$B_{eq}(\text{\AA}^2)$
S	0.2961 (1)	-0.0730	1.2003 (4)	6.53 (107)
O(1)	0.3956 (2)	0.3427 (9)	1.3768 (8)	6.0 (31)
O(2)	0.4435 (2)	0.157 (1)	1.0952 (8)	7.9 (64)
O(3)	0.2340 (2)	0.4093 (7)	0.6384 (6)	4.2 (19)
O(4)	0.0366 (2)	0.6460 (5)	1.2193 (5)	3.2 (13)
O(5)	-0.0203 (2)	0.6485 (7)	0.8217 (6)	4.4 (23)
N(1)	0.2790 (2)	0.3667 (6)	1.0549 (7)	3.3 (13)
N(2)	0.0598 (2)	0.9039 (5)	0.6443 (6)	2.4 (3)
C(1)	0.3331 (2)	0.2555 (9)	0.9715 (9)	3.9 (20)
C(2)	0.3103 (3)	0.065 (1)	0.913 (1)	5.3 (26)
C(3)	0.3933 (2)	0.259 (1)	1.174 (1)	4.6 (22)
C(4)	0.5053 (3)	0.137 (3)	1.282 (1)	13.2 (128)
C(5)	0.5618 (4)	0.187 (2)	1.179 (2)	10.4 (66)
C(6)	0.2320 (2)	0.4362 (8)	0.8784 (8)	3.0 (4)
C(7)	0.1756 (2)	0.5438 (8)	0.9857 (8)	3.4 (12)
C(8)	0.1435 (2)	0.6708 (7)	0.7765 (8)	2.8 (6)
C(9)	0.0876 (2)	0.7933 (6)	0.8729 (7)	2.3 (4)
C(10)	0.0286 (2)	0.6885 (6)	0.9813 (8)	2.7 (4)
O(W)	0.1500 (2)	0.1407 (7)	1.3882 (8)	5.1 (17)

to obtain some information for the interpretation of the mechanism of its pharmacological action.

Experimental. The title compound was synthesized by one of the authors (SK); plate-like crystals from ethanol:water (4:1); D_m by flotation in a mixture of cyclohexane and carbon tetrachloride; crystal size 0.40 × 0.40 × 0.05 mm; Rigaku AFC5R diffractometer; graphite-monochromated Cu $K\alpha$ radiation; unit-cell dimensions determined by least-squares refinement of 20 reflections with $48.5 < 2\theta < 50.9^\circ$; intensities measured for $5 < 2\theta < 126^\circ$; $h - 22 \rightarrow 22$, $k 0 \rightarrow 8$, $l 0 \rightarrow 5$, $2\theta - \omega$ scan mode; scan rate variable within $6-12^\circ \text{ min}^{-1}$ in θ ; scan width 1.3° ; three reference reflections monitored every 100 reflections, no significant intensity deterioration; corrections for Lorentz-polarization and absorption (max., min. transmission factors 0.94, 0.67); 1304 independent reflections, seven reflections with no net intensities designated as zero reflections; standard deviations estimated by $\sigma^2(F_o) = \sigma_P^2(F_o) + q|F_o|^2$ where $\sigma_P(F_o)$ was evaluated by counting statistics and q estimated to be 1.91×10^{-4} from the variation of the monitored reflections. Structure solved by direct methods and refined on F by full-matrix least squares; $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/\sigma^2(F_o)$; zero reflections with $F_c > F_{lim}$ ($F_{lim} = 0.946$) included in the least-squares calculation with the assumption that $F_o = F_{lim}$ and $w = w(F_{lim})$; all H atoms except those of the ethyl ester moiety found on a difference map and included in subsequent refinement; final $R = 0.057$ for 1263 reflections with $F_o > 3\sigma(F_o)$, $wR = 0.074$, $S = 3.71$, max. $\Delta/\sigma = 0.1$ for non-H atoms,

Table 2. Bond distances (\AA), bond angles ($^\circ$) and hydrogen-bond distances (\AA) with e.s.d.'s in parentheses

S—C(2)	1.824 (7)	O(1)—C(3)	1.202 (8)
O(2)—C(3)	1.332 (8)	O(2)—C(4)	1.48 (2)
O(3)—C(6)	1.235 (6)	O(4)—C(10)	1.243 (5)
O(5)—C(10)	1.238 (6)	N(1)—C(1)	1.437 (7)
N(1)—C(6)	1.335 (6)	N(2)—C(9)	1.489 (5)
C(1)—C(2)	1.516 (8)	C(1)—C(3)	1.497 (8)
C(4)—C(5)	1.32 (2)	C(6)—C(7)	1.505 (7)
C(7)—C(8)	1.520 (7)	C(8)—C(9)	1.538 (6)
C(9)—C(10)	1.535 (6)		
C(3)—O(2)—C(4)	116.9 (7)	C(1)—N(1)—C(6)	120.9 (4)
N(1)—C(1)—C(2)	112.9 (5)	N(1)—C(1)—C(3)	110.3 (4)
C(2)—C(1)—C(3)	110.6 (5)	S—C(2)—C(1)	115.9 (4)
O(1)—C(3)—C(1)	124.4 (5)	O(1)—C(3)—O(2)	125.3 (6)
O(2)—C(3)—C(1)	110.3 (5)	O(2)—C(4)—C(5)	113 (1)
O(3)—C(6)—N(1)	121.2 (4)	O(3)—C(6)—C(7)	121.8 (4)
N(1)—C(6)—C(7)	117.0 (4)	C(6)—C(7)—C(8)	111.0 (4)
C(7)—C(8)—C(9)	114.6 (4)	N(2)—C(9)—C(8)	108.0 (3)
N(2)—C(9)—C(10)	108.9 (3)	C(8)—C(9)—C(10)	112.7 (3)
O(4)—C(10)—O(5)	126.8 (4)	O(4)—C(10)—C(9)	116.0 (4)
O(5)—C(10)—C(9)	117.1 (4)		

X—H...Y	X...Y
S—H(S)...O(W)	3.479 (5)
N(1)—H(N1)...O(3) ⁱ	3.168 (6)
N(2)—H(N2A)...O(W) ⁱⁱ	2.885 (6)
N(2)—H(N2B)...O(4) ⁱⁱⁱ	2.745 (5)
N(2)—H(N2C)...O(4) ^{iv}	2.898 (5)
O(W)—H(WA)...O(5) ^v	2.675 (6)
O(W)—H(WB)...O(3) ^j	2.830 (6)

Symmetry code: (i) $x, y, 1+z$; (ii) $x, 1+y, -1+z$; (iii) $-x, \frac{1}{2}+y, 2-z$; (iv) $x, y, -1+z$; (v) $-x, -\frac{1}{2}+y, 2-z$.

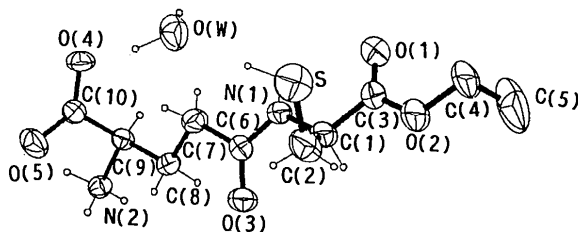
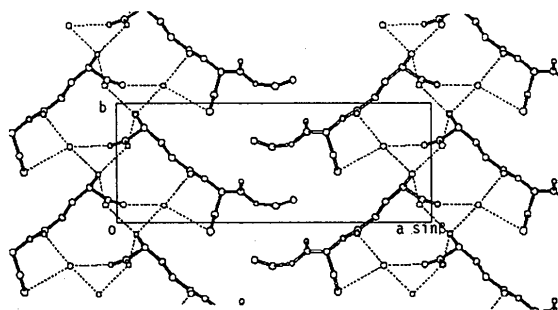


Fig. 1. Thermal-ellipsoid drawing (50% probability) with atomic numbering.

Fig. 2. Crystal structure of the title compound projected along the c axis.

max. $\Delta\rho = 0.33 \text{ e } \text{Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *XSEN* system (Takenaka & Sasada, 1983) and *DABEX* (Ohba, 1986). Final atomic parameters are given in Table 1.*

Discussion. Fig. 1 shows the conformation of the molecule with the atom numbering. Bond distances and angles are in Table 2.

The dimensions of the glutamic acid moiety, the cysteine moiety and peptide moiety are in good agreement with those in related compounds (Wright, 1958; Kerr, Ashmore & Koetzle, 1975; Lehmann & Nunes, 1980; Lehmann, Koetzle & Hamilton, 1972; Takusagawa, Koetzle, Kou & Parthasarathy, 1981). As the thermal factors of the ethyl ester moiety are large due to disorder or large thermal vibrations, the C(4)—C(5) bond distance is seemingly shorter than the usual value. The molecule is in an extended form, and some conformation angles are: N(2)—C(9)—C(8)—C(7) 182.0 (4), C(9)—C(8)—C(7)—C(6) 182.9 (4), C(8)—C(7)—C(6)—N(1) 157.9 (4), C(7)—C(6)—N(1)—C(1) 177.2 (4), C(6)—N(1)—C(1)—C(3) 155.9 (5) and N(1)—C(1)—C(3)—O(2) 180.5 (5)°.

The crystal structure viewed along the *c* axis is shown in Fig. 2 and the hydrogen bonds are listed in

* Lists of structure factors, anisotropic thermal parameters and atomic parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52955 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. The SH group of the cysteine residue is hydrogen bonded to a water molecule, S...O(W) 3.479 (5) and H(S)...OW 2.20 (6) Å. These values are similar to those found in the neutron diffraction study of L-cysteine (Takusagawa, Koetzle, Kou & Parthasarathy, 1981). The molecules are arranged so as to form ribbons along a twofold screw axis through N(2)H...O(4)ⁱⁱⁱ. As seen from Fig. 2, the ribbons are connected to each other through N(2)H...O(4)^{iv} hydrogen bonds along the *c* axis and a double molecular sheet is formed parallel to the *bc* plane. In addition, there are some hydrogen bonds to reinforce the sheet structure. The double sheets are loosely packed, the shortest contact being C(4)...O(1) $(1 - x, -\frac{1}{2} + y, 3 - z) = 3.32 (1) \text{ Å}$.

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SHORT-FORMAT PAPERS

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Structure of Potassium Difluorooctamolybdate Hexahydrate

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Abstract. $\text{K}_6[\text{Mo}_8\text{O}_{26}\text{F}_2] \cdot 6\text{H}_2\text{O}$, $M_r = 1564.18$, triclinic, $P\bar{1}$, $a = 10.497 (2)$, $b = 10.403 (2)$, $c = 8.001 (2) \text{ Å}$, $\alpha = 107.16 (2)$, $\beta = 95.77 (2)$, $\gamma =$

$105.53 (2)^\circ$, $V = 788.9 (3) \text{ Å}^3$, $Z = 1$, $D_x = 3.29 \text{ Mg m}^{-3}$, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0.7107 \text{ Å}$, $\mu(\text{Mo } K\alpha) = 36.63 \text{ cm}^{-1}$,