$\omega/2\theta$ scans; ω-scan width $0.7° + K\alpha$ separation; variable scan rate $2\text{-}20°\ min^{-1}$
Absorption correction: none
1989 measured reflections
1989 independent reflections
$\theta_{max} = 30°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = -11 \rightarrow 10$
3 standard reflections
frequency: 41 min
intensity variation: negligible

Refinement

Refinement on F
Final $R = 0.0351$
$wR = 0.0577$
$S = 1.76$
1779 reflections
73 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -1.59$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Te	0.3195 (1)	0.0295 (1)	−0.0017 (1)	0.049 (1)
Cl	0.1446 (2)	0.1466 (1)	0.2741 (2)	0.071 (1)
N(1)	0.7016 (5)	0.0778 (3)	0.3444 (5)	0.048 (1)
C(1)	0.4984 (5)	0.0873 (3)	0.2734 (5)	0.042 (1)
C(2)	0.4081 (6)	0.1333 (3)	0.3745 (6)	0.045 (1)
C(3)	0.5259 (7)	0.1685 (3)	0.5568 (6)	0.053 (2)
C(4)	0.7371 (6)	0.1575 (3)	0.6294 (6)	0.057 (2)
C(5)	0.8119 (6)	0.1135 (4)	0.5172 (6)	0.055 (2)

Table 2. *Geometric parameters* (Å, °)

Te—C(1)	2.121 (3)	Te—Te*A*	2.725 (1)
Cl—C(2)	1.736 (4)	N(1)—C(1)	1.340 (5)
N(1)—C(5)	1.327 (5)	C(1)—C(2)	1.370 (7)
C(2)—C(3)	1.392 (6)	C(3)—C(4)	1.396 (6)
C(4)—C(5)	1.351 (7)		
C(1)—Te—Te*A*	85.8 (1)	C(1)—N(1)—C(5)	117.5 (4)
Te—C(1)—N(1)	118.0 (3)	Te—C(1)—C(2)	120.7 (2)
N(1)—C(1)—C(2)	121.3 (3)	Cl—C(2)—C(1)	119.5 (3)
Cl—C(2)—C(3)	120.0 (4)	C(1)—C(2)—C(3)	120.5 (4)
C(2)—C(3)—C(4)	117.6 (5)	C(3)—C(4)—C(5)	117.5 (4)
N(1)—C(5)—C(4)	125.6 (4)		

Preliminary examination and data collection were performed by the Molecular Structure Corporation (The Woodlands, Texas, USA) on a sample mounted on a glass fiber with epoxy cement. The structure was solved by Patterson methods and final calculations were carried out with the *SHELXTL-Plus* programs (Sheldrick, 1990). Full-matrix least-squares refinement of coordinates and anisotropic temperature factors for non-H atoms was carried out and H atoms were assigned idealized positions at 0.96 Å from the C atom to which they were attached. The intensities of 1989 reflections were measured, 91 of which were discarded because of systematic extinction.

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Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55816 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1041]

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(3*R*,5*R*)-Tetrahydro-2*H*-1,4-thiazine-3,5-dicarboxylic Acid Monohydrate

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Abstract

(3*R*,5*R*)-Tetrahydro-2*H*-1,4-thiazine-3,5-dicarboxylic acid monohydrate, which is in the zwitterionic form in the crystal, shows a puckered chair conformation of the six-membered ring with S(1) and N(4) out of the C(2), C(3), C(5), C(6) mean plane by 0.972 and 0.648 Å, respectively. The ionized carboxyl group is axially oriented.

Comment

An improved method for the synthesis of the diastereomeric dimethyl diesters of tetrahydro-2*H*-1,4-thiazine-3,5-dicarboxylic acid has been recently reported by reacting methyl (*R*,*S*)-2,3-dibromopropionate with (*R*)-cysteine methyl ester (Paglialunga Paradisi, Pagani Zecchini, Torrini & Lucente, 1990). Taking into account the absolute configuration of

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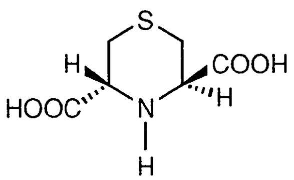
the starting cysteine methyl ester, (*R*,*R*) configuration instead of (*S*,*S*) (Eremeev *et al.*, 1986) was confirmed for the *trans* isomer which showed negative optical rotation (Paglialunga Paradisi, Pagani Zecchini, Torrini & Lucente, 1990).

In view of the large interest raised by the detection of tetrahydro-2*H*-1,4-thiazine-3,5-dicarboxylic acid (Tht) in bovine brain and human urine (Cavallini *et al.*, 1991) and of the possible role of the conformation on the biological properties, the X-ray analysis of the *trans* isomer of Tht (1) was undertaken. The title compound has been prepared by basic hydrolysis of the corresponding dimethyl diester.



(I)

From Fig. 1 it appears that the six-membered ring adopts a puckered chair conformation. It has been observed (Schultz, Kucsman & Hargittai, 1988) that substituting a C atom by a hetero atom into a six-membered ring increases the ring puckering in comparison with the conformation of cyclohexane (Bastiansen, Fernholt, Seip, Kambara & Kuchitsu, 1973). The ring puckering can be characterized by the mean torsional angle, φ, which is equal to 54.6° in cyclohexane (Bastiansen, Fernholt, Seip, Kambara & Kuchitsu, 1973), 57.4° in piperidine (Gundersen & Rankin, 1983) and 58.2° in thiane (Schultz, Kucsman & Hargittai, 1988). In *trans* Tht φ is 60.7°, which corresponds to enhanced puckering because of the double substitution of the carbon skeleton of the six-membered ring by the S and N atoms. S(1) and N(4) have been found out of the C(2), C(3), C(5) and C(6) mean plane by 0.972 and 0.648 Å, respectively.

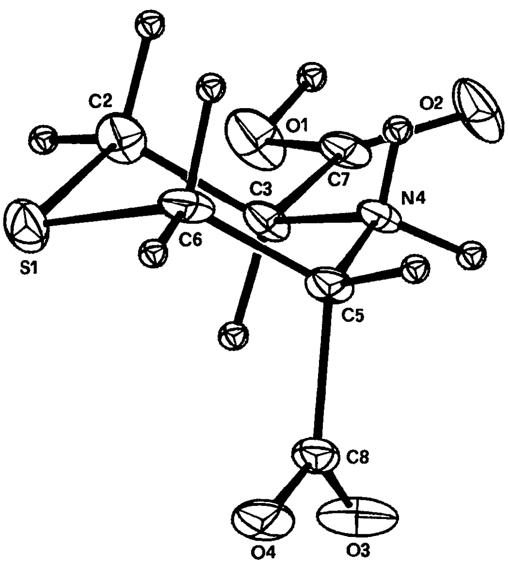


Fig. 1. A drawing of the *trans* Tht molecule in the crystal, showing the anisotropy of the thermal motion. The thermal ellipsoids of the non-H atoms have been scaled to the 50% probability level. The drawing is based on the atomic parameters from the final refinement.

Trans Tht, which occurs as a zwitterion in the crystal, shows the ionized carboxyl group in an axial position. The same axial position for the ionized carboxyl group has been observed for chondrine (Palmer, Lee, Wong & Carson, 1972); furthermore, in the case of cycloalliin (Palmer & Lee, 1966) the unionized carboxyl group is equatorially oriented.

The hydrogen-bonding network includes a very short intermolecular contact between O(1) and O(4) (2.48 Å).

Experimental

Crystal data

$C_6H_{11}NO_5S$
$M_r = 209.22$
Orthorhombic
$P2_12_12_1$
$a = 6.104$ (1) Å
$b = 8.933$ (3) Å
$c = 16.062$ (3) Å
$V = 875.84$ (2) Å^3
$Z = 4$
$D_x = 1.587$ Mg m^{-3}
$D_m = 1.586$ Mg m^{-3}

Cu radiation
$\lambda = 1.54184$ Å
Cell parameters from 18 reflections
$\theta = 42$-$55°$
$\mu = 3.20$ mm^{-1}
$T = 293$ K
Tablets
0.50 × 0.50 × 0.45 mm
Colourless

Data collection

Syntex $P2_1$ diffractometer
$\omega/2\theta$ scans
2450 measured reflections
950 independent reflections
940 observed reflections
[$F > 3\sigma F$]
$R_{int} = 0.043$
$\theta_{max} = 69°$

$h = 0 \rightarrow 7$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 19$
3 standard reflections
monitored every 97 reflections
intensity variation: <3%

Refinement

Refinement on F
Final $R = 0.0511$
$wR = 0.0690$
$S = 0.214$
940 reflections
118 parameters

H-atom parameters not refined
$w = 1/[2.592+F+0.031F^2]$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.1$ e Å^{-3}
$\Delta\rho_{min} = -0.1$ e Å^{-3}

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j b_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
S(1)	0.76827 (18)	0.43809 (11)	0.85525 (6)	1.8 (2)
O(1)	0.19112 (59)	0.40883 (34)	1.03534 (18)	2.1 (1)
O(2)	0.11928 (56)	0.64976 (33)	1.00879 (18)	2.1 (1)

O(3)	0.22743 (58)	0.54841 (38)	0.73892 (18)	2.4 (1)
O(4)	0.53024 (51)	0.59677 (38)	0.66478 (16)	2.1 (1)
O(5)	0.90428 (55)	0.41161 (34)	0.64285 (19)	2.2 (1)
N(4)	0.38349 (58)	0.66905 (31)	0.88055 (19)	1.1 (1)
C(2)	0.58974 (73)	0.46024 (47)	0.94414 (22)	1.6 (1)
C(3)	0.36450 (72)	0.51658 (42)	0.91911 (22)	1.1 (1)
C(5)	0.52170 (69)	0.68028 (44)	0.80361 (22)	1.2 (1)
C(6)	0.75560 (71)	0.63222 (45)	0.82274 (24)	1.5 (1)
C(7)	0.20916 (68)	0.53081 (43)	0.99395 (22)	1.2 (1)
C(8)	0.41309 (69)	0.59969 (43)	0.73035 (24)	1.3 (1)

Table 2. *Geometric parameters* (Å, °)

S(1)—C(2)	1.807 (4)	C(3)—C(7)	1.536 (5)
S(1)—C(6)	1.813 (4)	O(1)—C(7)	1.281 (5)
C(2)—C(3)	1.518 (6)	O(2)—C(7)	1.219 (5)
C(5)—C(6)	1.522 (6)	C(5)—C(8)	1.531 (5)
N(4)—C(3)	1.501 (5)	O(3)—C(8)	1.230 (5)
N(4)—C(5)	1.500 (4)	O(4)—C(8)	1.273 (5)
C(6)—S(1)—C(2)	95.6 (2)	C(3)—C(7)—O(1)	112.9 (3)
S(1)—C(2)—C(3)	111.9 (3)	C(3)—C(7)—O(2)	120.2 (3)
C(2)—C(3)—N(4)	109.9 (3)	O(1)—C(7)—O(2)	126.9 (3)
C(3)—N(4)—C(5)	116.4 (3)	C(6)—C(5)—C(8)	115.4 (3)
N(4)—C(5)—C(6)	110.0 (3)	N(4)—C(5)—C(8)	111.0 (3)
C(5)—C(6)—S(1)	111.6 (3)	C(5)—C(8)—O(3)	119.2 (3)
C(2)—C(3)—C(7)	112.3 (3)	C(5)—C(8)—O(4)	113.7 (4)
N(4)—C(3)—C(7)	107.2 (3)	O(3)—C(8)—O(4)	127.0 (4)

Density was measured by flotation in a $CHCl_3/CHBr_3$ mixture. Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods with *SIR*88 (Burla *et al.*, 1989) and refined by anisotropic full-matrix least squares on *F*. At this stage the water molecule and all the H atoms were clearly located through difference syntheses. In the final refinement the H atoms were modeled at ideal geometry with isotropic thermal parameters fixed to the corresponding value of the non-H atoms to which they are linked, and fixed bond lengths (X—H = 1.03 Å with X = C, N, O). Calculations were carried out on the Data General Eclipse MV/8000 II of the CNR Area (Roma) using mainly *SIRCAOS* (Camalli *et al.*, 1986). Some of the final calculations were performed with *PARST* (Nardelli, 1983). The scattering factors were those of Cromer & Mann (1968) for the non-H atoms and those of Hanson, Herman, Lea & Skillman (1964) for the H atoms.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55749 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE1003]

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Structure of Sinoacutine

BÉLA RIBÁR

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Abstract

In the title alkaloid, $(9\alpha,13\alpha)$-5,6,8,14-tetradehydro-4-hydroxy-3,6-dimethoxy-17-methylmorphinan-7-one, the mean value of three C—N—C angles is 111.0 (4)° indicating sp^3 hybridization of the N atom. The interplanar angle between the aromatic ring *A* and planar ring *D* [maximum deviation 0.051 (5) Å at C14] is 53.8 (1)°. The absolute configuration has been assigned to agree with the known chirality at C3 (*S*) [Southon & Buckingham, (1989). *Dictionary of Alkaloids*, p. 974. London: Chapman and Hall].

Comment

Sinoacutine is an enantiomer of salutaridine. In nature it occurs both in optically active and racemic

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