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The Crystal Structure of *N*-(2-Hydroxyethyl)taurine, $HOCH_2CH_2NHCH_2CH_2SO_3H$

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The crystals of *N*-(2-hydroxyethyl)taurine are orthorhombic; $a = 9.666\ (4)$, $b = 11.681\ (6)$, $c = 12.754\ (8)$ Å; space group is Pbca with eight formula units in the unit cell. A three-dimensional X-ray crystal structure analysis has shown that the compound crystallizes as zwitterion, formula $HOCH_2CH_2NH_2^+CH_2CH_2SO_3^-$. Dihedral angle S—C—C—N = 175.6°, and N—C—C—O = — 59.8°. Zwitterions are connected by hydrogen bonds into a three-dimensional network.

INTRODUCTION

Preparation and crystal data of *N*-(2-hydroxyethyl)taurine, (3-aza-5-hydroxypentansulfonic acid) have been reported recently.[1] The compound is a derivative of taurine, $^+H_3NCH_2CH_2SO_3^-$, whose structure has already been reported[2–4].

Taurine and its derivatives are known as natural compounds or moieties of substances with biological significance[5,6]. They possess, for example, tumor-inhibiting properties[7] or are »high energy« compounds whose hydrolysis produces energy for biochemical reactions[8]. A knowledge of the structure of *N*-(2-hydroxyethyl)taurine is of interest for determining its structural relationship with taurine. This is a part of structural investigation of taurine derivatives which could be of pharmaceutical interest. On the other hand *N*-(2-hydroxyethyl)taurine was used as a model structure for phase determination by direct methods[9].

EXPERIMENTAL

The crystal data were determined from oscillation and Weissenberg photographs using CuKα radiation ($\lambda = 1.5418$ Å) at room temperature. The crystals are orthorhombic:

$a = 9.666(4)$ Å	$b = 11.681(6)$ Å	$c = 12.754(8)$ Å
$U = 1440.0$ Å^3	$Z = 8$	
$D_m = 1.560$ g cm^{-3}	$D_x = 1.563$ g cm^{-3}	

space group Pbca (No. 61).

The density was determined pycnometrically using decalin as liquid. The three-dimensional intensity data were recorded on multiple equiinclination Weissenberg

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photographs with CuKα radiation ($\lambda = 1.5418$ Å) from crystals ground to spheres in order to apply abstorption corrections ($\mu = 36.2$ cm^{-1}). The spheres having μr equal to 0.9, 0.7 and 0.8 were used for collecting intensity data of $0kl$-$4kl$, $h0l$-$h6l$ and $hk0$-$hk6$ layers, respectively. The intensities were measured with an optical densitometer. Calculations were performed on the CAE 90—40 computer using programs written or modified at the Institute »Ruđer Bošković«[9-11].

STRUCTURE DETERMINATION

The structure was solved by Patterson and successive Fourier three-dimensional synthesis using $hk0$-$hk6$ data. Simultaneously the structure was solved by direct method[9]. Positions of nonhydrogen atoms were refined by several cycles of least-squares refinement using $hk0$-$hk6$ data until the error was less than the shift in parameters. Then the scale factors among the various hkl levels of data (obtained from three crystallographic axes) were improved in the course of refinement, the observed structure factors were transformed into absolute scale, the mean values were calculated for reflections whose intensities were determined several times, and the list of 1256 observed reflections was obtained. Weights (w) were assigned as follows: for unobserved reflections $w = 0$; for $F_o < 10$, $w = 0.25$; for $10 < F_o < 115$, $w = 1$, and for $F_o > 115$, $w = 0.5$. Neutral-atom scattering factors were used[12]. The obtained R_{obs}, was 0.143 with isotropic and 0.134 with anisotropic temperature factors. The attempt to locate hydrogen atoms by means of a three-dimensional difference Fourier synthesis was not successful. The inclusion into least squares refinement of hydrogen atom parameters (whose values were calculated using 1.09 Å for C—H and 0.99 Å for N—H distances with tetrahedral angle of 109.47°, and 1.0 Å for O—H distance assuming straight hydrogen bond between neighboring oxygen atoms) gave an R-value of 0.120 (or 0.149 with 273 unobserved reflections included as $F_o = 0$). No extinction corrections were used, but seven reflections (with $\sin\vartheta/\lambda < 0.2$), strongly affected by extinction, were excluded from final cycles of refinement. They are as follows:

hkl	$\lvert F_o \rvert$	$\lvert F_c \rvert$	*hkl*	$\lvert F_o \rvert$	$\lvert F_c \rvert$	*hkl*	$\lvert F_o \rvert$	$\lvert F_c \rvert$
210	86.5	119.6	022	112.7	175.8	212	108.1	177.5
121	73.2	96.9	102	80.4	103.1	123	85.6	115.9
131	115.4	170.3						

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

N-(2-Hydroxyethyl)taurine crystallizes as zwitterions, $HOCH_2CH_2NH_2^+$-$CH_2CH_2SO_3^-$, which are connected to a three-dimensional network by hydrogen bonds. All atoms are in general positions. Their coordinates with standard deviations and anisotropic thermal parameters are listed in Tables I and II. The observed and calculated structure factors are compared in Table III which is deposited in the Journal library.* The interatomic distances and angles with their standard deviations are given in Table IV.

The coordination polyhedron around the sulfur atom is an elongated tetrahedron with sulfur-carbon distance of 1.792(7) Å and three sulfur-oxygen distances of 1.454(5), 1.449(5) and 1.462(5) Å. The average O—S—O and C—S—O angles in the sulfonate group are 112.8° and 105.9°, respectively. These and the other distances and angles including carbon, nitrogen and oxygen atoms are

* Photocopies may be obtained on request referring to Table III in CCA-826

TABLE I

Final atomic coordinates ($\times 10^4$) *and anisotropic thermal parameters* ($\times 10^4$). *Thermal parameters are of the form:* exp $[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. *Standard deviations are in parentheses.*

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	1252(2)	1697(1)	235(1)	28(1)	15(1)	9(1)	—6(1)	—2(1)	—1(1)
O(1)	791(5)	1840(4)	—843(3)	45(6)	36(5)	5(3)	0	0	0
O(2)	2229(5)	770(4)	378(4)	63(8)	15(4)	26(4)	15(4)	—7(4)	3(3)
O(3)	94(5)	1632(5)	970(4)	34(6)	60(6)	25(4)	—25(5)	22(4)	—4(4)
O(4)	5865(5)	5295(4)	2586(4)	13(5)	29(4)	35(4)	—8(4)	4(4)	—1(4)
N	3457(6)	4036(4)	1879(4)	25(6)	11(4)	6(3)	—2(4)	—7(4)	—3(3)
C(1)	2166(7)	2987(5)	545(5)	42(8)	9(5)	11(4)	—4(5)	—1(5)	—5(4)
C(2)	2637(7)	2994(5)	1684(5)	46(8)	5(4)	12(4)	—7(5)	—8(5)	3(4)
C(3)	3814(8)	4193(6)	3009(5)	48(8)	26(5)	3(4)	3(6)	—6(5)	3(4)
C(4)	4604(7)	5290(7)	3181(5)	32(8)	39(6)	17(5)	—0(6)	5(5)	19(5)

TABLE II

Atomic coordinates ($\times 10^4$) *for hydrogen with thermal parameters* $B = 4.0$ Å^2

	x	y	z
H(1)	1470	3704	391
H(2)	3054	3044	16
H(3)	3270	2235	1837
H(4)	1736	2980	2200
H(5)	2915	4709	1643
H(6)	4325	3991	1467
H(7)	4458	3473	3267
H(8)	2868	4208	3474
H(9)	4837	5375	4012
H(10)	3945	6002	2934
H(11)	5517	5787	1992

TABLE IV

Interatomic distances/Å and angles. Small letters indicate symmetry transformations and cell translations:

(a) $-x, -y, -z$; (b) $1/2 + x, 1/2 - y, -z$; (c) $1/2 - x, 1/2 + y, z$; (d) $-x, 1/2 + y, 1/2 - z$; (e) $x, 1/2 - y, 1/2 + z$; (f) $1/2 - x, -y, 1/2 + z$; (g) $1/2 + x, y, 1/2 - z$; (h) $1/2 + x - 1, y, 1/2 - z$; (i) $1/2 + x - 1, 1/2 - y, -z$; (j) $x, y + 1, z$; (k) $x + 1, 1/2 - y, 1/2 + z$; (l) $x + 1, y, z$; (m) $1/2 + x, 1/2 + y - 1, z$.

a) Distances within the S polyhedron

S — O(1)	1.454(5)	S — O(3)	1.462(5)
S — O(2)	1.449(5)	S — C(1)	1.792(7)

b) Other distances within asymmetric unit

C(1) — C(2)	1.521(9)	C(3) — C(4)	1.508(10)
C(2) — N	1.473(8)	C(4) — O(4)	1.436(9)
N — C(3)	1.492(7)	N — O(4)	2.898(7)

c) Distances between neighbouring units shorter than 3.6 Å

O(3c) — O(4)	2.746(8)	O(2c) — O(4h)	3.236(7)
N — O(1b)	2.807(7)	O(1b) — O(4)	3.339(7)
N — O(2c)	2.864(7)	N — O(3c)	3.535(8)
N — O(4h)	2.984(7)		

d) Angles within S polyhedron

O(1)—S—O(2)	113.9(3)°	O(1)—S—C(1)	105.3(3)°
O(1)—S—O(3)	112.1(3)°	O(2)—S—C(1)	106.2(3)°
O(2)—S—O(3)	112.3(3)°	O(3)—S—C(1)	106.3(3)°

e) Other angles within asymmetric unit

C(2)—C(1)—S	111.3(5)°	C(4)—C(3)—N	111.3(5)°
N—C(2)—C(1)	109.0(5)°	O(4)—C(4)—C(3)	110.9(6)°
C(3)—N—C(2)	112.9(5)°		

f) Angles on nitrogen atom

C(2)—N—C(3)	112.9°	O(2c)—N—C(3)	127.7°
O(1b)—N—C(2)	93.0°	O(2c)—N—O(1b)	97.5°
O(1b)—N—C(3)	108.2°	O(4)—N—O(4h)	110.7°
O(2c)—N—C(2)	110.3°		

in good agreement with literature data (Tables V and VI). The main difference between the structures of taurine and *N*-(2-hydroxyethyl)taurine is the position of sulfur and nitrogen atoms regarding the C(1)—C(2) linkage. In taurine they are in *gauche* and in *N*-(hydroxyethyl)taurine in *anti* position with dihedral angle S—C(1)—C(2)—N of 175.6°. But oxygen O(4) from hydroxyethyl group and nitrogen atom, regarding C(3)—C(4) linkage, are in *gauche* position with dihedral angle N—C(3)—C(4)—O(4) of —59.8°.

TABLE V

Interatomic distances/Å in taurine and some similar compounds

Compound	S—O	S—C	C—C	C—N	C—O	Reference
$C_2H_8NO_4P$[a]			1.521(8)	1.491(7)	1.429(7)	13
$C_2H_8NO_3P$[b]			1.514(6)	1.488(5)		14
$C_8H_{20}O_6NP$[c]			1.538(9) 1.553(13) 1.510(8) 1.532(7) 1.498(10) 1.534(8)	1.510(9) 1.480(10) 1.540(8) 1.522(7) 1.487(10) 1.516(11) 1.476(8) 1.529(8)	1.449(8) 1.433(7) 1.390(8) 1.418(9) 1.435(8) 1.405(7) 1.457(7) 1.442(8)	15
$C_2H_7NO_3S$[d]	1.461(2) 1.448(2) 1.465(2)	1.780(2)	1.520(3)	1.484(2)		4
$C_4H_{14}N_2O_2S_2^{2+}$; $2\,Cl^- \cdot H_2O$[e]	1.417(10) 1.439(10)	1.782(10)	not mentioned			16
$[C_2H_4O_6S_2](H_3O)_2$[f]	1.458(3) 1.457(3) 1.457(3)	1.775(3)	1.506(5)			17
$C_2H_7NO_3S_2$[g]	1.48(1) 1.41(1) 1.48(1) 1.48(1) 1.47(1) 1.39(1)	1.88(1) 1.76(1)	1.49(1) 1.55(2)	1.50(1) 1.51(1)		18
$C_5H_{14}NO_6P \cdot H_2O$[h]			1.483(5) 1.508(4) 1.517(5)	1.495(4)	1.418(4) 1.407(4) 1.443(4) 1.433(4)	19
$C_4H_{11}NO_4S$	1.454(5) 1.449(5) 1.462(5)	1.792(7)	1.521(9) 1.508(10)	1.473(8) 1.492(7)	1.436(9)	this work

[a] 2-amino-ethanol phosphate, $NH^+_3—CH_2—CH_2—O—PO_3H^-$
[b] β-ciliatine, 2-aminoethylphosphonic acid, $NH_3^+—CH_2—CH_2—PO_3H^-$
[c] L-α-glycerylphosphorylcholin, $HO—CH_2—CH(OH)—CH_2—O—PO_2^-—O—CH_2—CH_2—N(CH_3)_3^+$
[d] taurine, 2-aminoethylsulfonic acid, $NH_3^+—CH_2—CH_2—SO_3^-$
[e] 2-aminoethyl 2-aminoethanethiosulfonate dihydrochloride $Cl^-, NH_3^+—CH_2—CH_2—S—SO_2—CH_2—CH_2—NH_3^+, Cl$
[f] dioxonium-ethane-1,2-disulfonate, $(H_3O^+)_2{}^-O_3S—CH_2—CH_2—SO_3^-$
[g] 2-aminoethanethiosulfuric acid, $NH_3^+—CH_2—CH_2—S—SO_3^-$
[h] L-α-glycerylphosphorylethanolamine, $HO—CH_2—CH(OH)—CH_2—O—PO_2^-—O—CH_2—CH_2—NH_3^+$

TABLE VI

Valence angles (in degrees) in taurine and some similar compounds

Compound	O—S—O	O—S—C	S—C—C	C—C—N	C—N—C	C—C—O	Reference
$C_2H_8NO_4P$				111.4(3)		108.9(4)	13
$C_2H_8NO_3P$				111.9			14
$C_8H_{20}O_6NP$				115.3(5)	113.1(5)	111.7(6)	15
				115.0(5)	109.9(6)	107.5(5)	
					106.3(5)	107.6(5)	
					111.0(5)	109.1(6)	
					107.7(6)	112.7(7)	
					108.5(5)	107.6(5)	
					113.5(5)	111.5(5)	
					112.9(7)	107.0(5)	
					107.2(6)	108.4(5)	
					107.0(6)	110.8(6)	
					105.6(7)		
					110.5(6)		
$C_2H_7NO_3S$	113.7	105.8	112.9	112.6			4
	110.9	106.9					
	113.0	105.8					
$C_4H_{14}N_2O_2S_2^{2+}$,	121.	106.					16
$2\ Cl^-$, H_2O		108.					
$[C_2H_4O_6S_2](H_3O)_2$	112.4(2)	107.6(2)	111.2(2)				17
	112.6(2)	106.0(2)					
	111.6(2)	106.3(2)					
$C_2H_7NO_3S_2$	110.		114.	113.			18
	116.						
	117.						
	112.		111.	115.			
	115.						
	115.						
$C_5H_{14}NO_6P \cdot H_2O$				112.2(3)		109.4(3)	19
						107.8(2)	
						107.9(2)	
						109.4(3)	
						113.8(3)	
$C_4H_{11}NO_4S$	113.9(3)	105.3(3)	111.3(5)	109.0(5)	112.9(5)	110.9(6)	this work
	112.1(3)	106.2(3)		111.3(5)			

Some O...N and O...O distances between neighbouring zwitterions range from 2.746(8) to 2.984(7) Å. The next shortest distance is 3.236(7) Å. Shorter distances indicate possible hydrogen bonding. Further evidence in selection of hydrogen bond locations is provided by the list of angles on nitrogen atom, also given in Table IV. Three short distances between nitrogen and three oxygen atoms from neighbouring units, O(1b), O(2c) and O(4h) (Table IV), could be explained by one bifurcated hydrogen bond N—H(5)... ...O(2c), O(4h), and by N—H(6)...O(1b) hydrogen bond (Fig. 1). The third

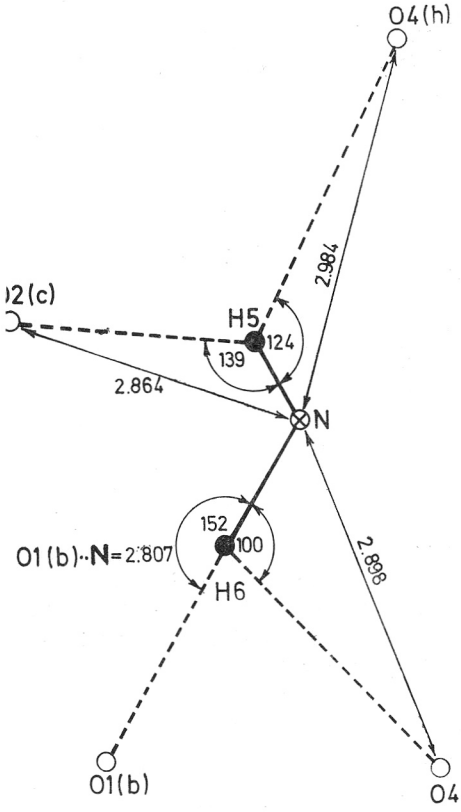

Fig. 1. Possible N—H...O bonds.

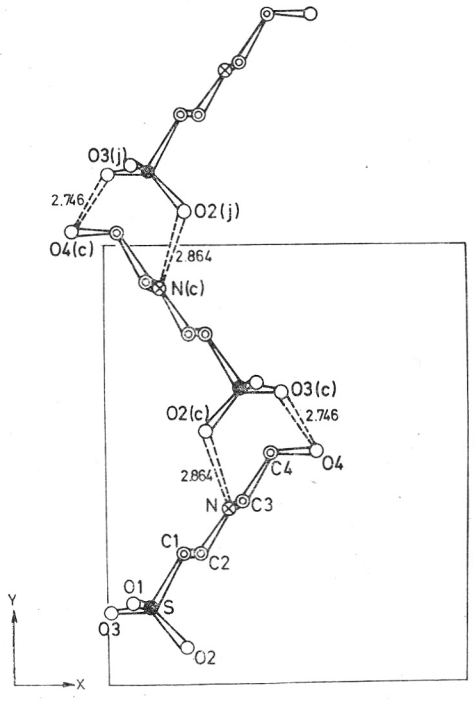

Fig. 2. The projection of the structure along [001].

hydrogen atom, H(11), which is involved in hydrogen bond O(4)—H(11)... ...O(3c), belongs to the hydroxyethyl group (Fig. 2). It is evident that all oxygen atoms from the sulfonate group are involved in hydrogen bonding, one in a relatively strong bond, O(3)...H(11)—O(4) of 2.746(8) Å and two in weaker hydrogen bonds, O(1b)...H(6)...N of 2.807(7) Å and O(2c)...H(5)—N of 2.864(7) Å. The smallest sulfur-oxygen separation of 1.449(5) Å includes oxygen atom O(2), which exhibits the weakest hydrogen bond, and the greatest sulfur-oxygen separation of 1.462(5) Å is with oxygen atom O(3), which exhibits the strongest hydrogen bond. Since hydrogen atom parameters were not experimentally determined, further discussion of their distances and angles is not presented.

The hydrogen bonds connect asymmetric units and build up a three--dimensional network in the following way: O(4)—H(11) ... O(3c) and N—H(5) ... O(2c) hydrogen bonds connect zwitterions infinitely along the b axis direction (Fig. 2). At the points of contact nine-membered rings are formed

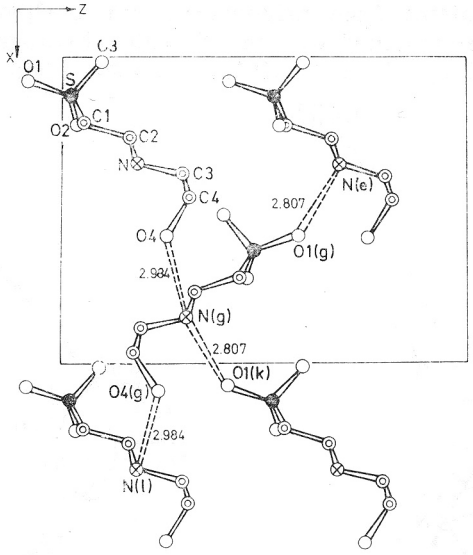

Fig. 3. The projection of the structure along [010].

which include two hydrogen atoms, H(5), H(11) (points of contact), and seven atoms (from adjacent units), N, C(3), C(4), O(4), O(3c), S(c) and O(2c); the remaining two hydrogen bonds, N—H(6) ... O(1b) and N—H(5) ... O(4h), of 2.807(7) and 2.984(7) Å, respectively, complete three-dimensional connections between zwitterions in the crystal structure (Figs. 1 and 3).

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SAŽETAK

Kristalna struktura *N*-(2-hidroksietil)taurina, $HOCH_2CH_2NHCH_2CH_2SO_3H$

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N-(2-Hidroksietil)taurin kristalizira u rompskom sustavu: $a = 9{,}666(4)$, $b = 11{,}681(6)$, $c = 12{,}754(8)$ Å, prostorna grupa je Pbca s osam formulskih jedinica u jediničnoj ćeliji. Trodimenzionalna kristalna struktura, određena metodom rendgenske difrakcije, pokazala je da spoj kristalizira kao »zwitterion« i ima formulu $HOCH_2CH_2NH_2^+CH_2CH_2SO_3^-$. Diedrijski kut S—C—C—N = 175,6°, a N—C—C—O = = — 59,8°. »Zwitterioni« su međusobno povezani vodikovim vezama u trodimenzionalnu mrežu.

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