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The Crystal and Molecular Structure of Taurine

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The structure of taurine, $NH_2[CH_2]_2HSO_3$, has been determined by Patterson superposition and Fourier methods. The crystals are monoclinic with space group $P2_1/c$. The unit-cell dimensions are,

$$a=5{\cdot}30,\ b=11{\cdot}65,\ c=7{,}94\ \text{Å},\ \beta=94°\ 17'.$$

The structure was refined by least-squares methods using three-dimensional data. The final R-factor, 0·11, was obtained using individual isotropic temperature factors for the heavier atoms and an over-all isotropic temperature factor for the hydrogen atoms. The bond lengths, bond angles and inter-molecular contacts were found to be normal.

Introduction

The X-ray investigation of several related compounds is at present being undertaken by the authors, to obtain structural information on the sulphonate and sulphuric radicals, taurine being the first of the series.

Taurine, $NH_2[CH_2]_2HSO_3$, occurs in the combined state in ox-gall and many other animal secretions. In the animal organism taurine is formed from cysteine by decarboxylation followed by oxidation. It can be obtained synthetically by the addition of sodium bisulphite to nitroethylene and the subsequent reduction of the nitroethane sulphonic acid. It forms colourless crystals, and melts and decomposes at about 328 °C; it is readily soluble in water but insoluble in alcohol and is quoted as forming tetragonal crystals (*Handbook of Chemistry and Physics*, 1961).

Experimental

A sample of taurine was recrystallized from water, the form of the resulting crystals depending on the rate of evaporation of the solution. With rapid evaporation, needle-shaped crystals were formed—the length of the needle being parallel to the a-axis—whereas with slow evaporation the crystals adopted a tabular appearance, the greatest length being along the a-axis. Both crystal types possessed identical space groups and unit-cell dimensions.

The space group and unit-cell dimensions were obtained from Weissenberg films using Cu $K\alpha$ radiation; the systematic absences,

$$0k0 \text{ with } k=2n+1 \quad \text{and} \quad h0l \text{ with } l=2n+1$$

uniquely determined the space group as $P2_1/c$; the unit-cell dimensions were:

$$a=5{\cdot}30\pm0{\cdot}01,\ b=11{\cdot}65\pm0{\cdot}01,$$
$$c=7{\cdot}94\pm0{\cdot}01\ \text{Å};\ \beta=94°\ 17'\pm5'.$$

The density measured at 20 °C by the method of flotation, using a benzene-tetrabromoethane mixture, was $1{\cdot}70\pm0{\cdot}02$ g.cm^{-3}. The calculated value, assuming four molecules to the unit cell, was 1·71 g.cm^{-3}.

The intensity data were measured visually from sets of multiple-film equi-inclination Weissenberg photographs (Robertson, 1943) taken with Cu $K\alpha$ radiation. 850 independent reflexions were measured from the $0kl$, $hk0$, $h0l$ and hkh zones and upper layers ($h=1$ to 5) taken with the a-axis as rotation axis. The intensities were corrected for Lorentz and polarization factors, but since the crystals were needle-shaped no attempt was made to apply absorption corrections. A correction for the variation in the area of the reflexions occurring in the upper-level Weissenberg photographs was made by the method of Phillips (1954). It was found that when the Phillips correction factor was applied to the extended reflexions, there was a difference in scale factor between the reflexions on both halves of the film, the scale factor appearing to vary with the Bragg angle, θ. An empirical correction curve was calculated for the contracted reflexions

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on the basis of the variation of the area of the reflexion with sin θ. Since the area is also dependent on the intensity of the reflexion, groups of reflexions of approximately the same intensity were compared. After applying the correction factor, intensities of the reflexions common to both halves of the film were compared and found to agree within the limits of experimental error. As a check on the visual data, the intensities of the $0kl$ spectra were measured with a single-crystal Geiger-counter goniometer using Cu $K\alpha$ radiation and both sets of $0kl$ data were found to agree within the limits of experimental error.

The data were put on an approximately absolute scale using the method of Wilson (1942).

Structure determination

The $0kl$ Patterson synthesis (Fig. 1(*a*)) was examined for possible sulphur-sulphur vectors. It was not possible to identify a rotation peak corresponding to a sulphur–sulphur interaction. In Fig. 1(*a*) the peaks labelled A and B correspond to double-weight reflexion peaks situated on the lines $(y, \frac{1}{2})$ and $(\frac{1}{2}, z)$: the related single-weight rotation peak is labelled C and corresponds to a sulphur oxygen–sulphur oxygen vector, assumed to arise in projection from overlap within the sulphonate group. This vector was used as a basis for producing a minimum-function map $M4(y, z)$ which is shown in Fig. 1(*b*).

The minimum-function map, $M4(y, z)$, showed seven separate maxima which were capable of several interpretations. The positions of the atoms which comprised the molecular 'head' were obtained immediately whereas the remaining three peaks were capable of several interpretations depending on whether the 'tail' lay along b or c. An $0kl$ Fourier synthesis was constructed and this was in agreement with the $M4(y, z)$ map.

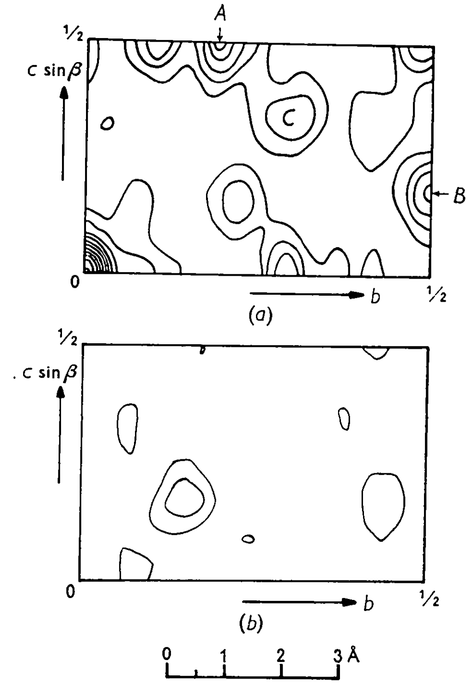

Fig. 1. (*a*) The (100) Patterson synthesis; contours at arbitrary intervals. (*b*) Minimum-function map, $M4(y, z)$.

Two-dimensional least-squares refinement of the various permitted structures eliminated all but the correct structure. Using an over-all isotropic temperature factor of 2·0 Å^2 the y and z atomic co-ordinates were refined until the R-factor was reduced to 0·14. All the crystallographic computations were carried out on a Stantec ZEBRA computer. The least-squares program of Schoone (1960) and the Fourier-synthesis program of Smits (1960) were used.

Patterson syntheses, with the $h0l$ and $hk0$ data gave the x co-ordinate of the sulphur atom. As in the case of the $0kl$ Patterson synthesis the sulphur–sulphur vectors were contained in multiple interaction peaks. The x co-ordinates of the other atoms were calculated from their known y and z parameters and the x, y and z co-ordinates of the sulphur atom by postulating normal bond lengths and bond angles. The resulting co-ordinates gave an R-factor of 0·23 for the combined $h0l$ and $hk0$ data.

Refinement

The x co-ordinates of the atoms were refined using the $h0l$ and $hk0$ data by the method of least squares to give an R-factor of 0·15 with an over-all isotropic temperature factor of 2·0 Å^2.

After several cycles of full three-dimensional least-squares refinement using an over-all isotropic temperature factor an R-factor of 0·15 was obtained and this value dropped to 0·14 when individual isotropic temperature factors were used.

The positional parameters of the four hydrogen atoms attached to C5 and C6 were obtained by assuming a carbon–hydrogen bond length of 1·08 Å and tetrahedral co-ordination. Using a bond-scan program of Rogers (1960), the various intra-molecular and inter-molecular distances were calculated. Several of the inter-molecular distances were of a length consistent with hydrogen bonds. To eliminate some of the possibilities and ascertain the positions of the remaining hydrogen atoms, a three-dimensional difference Fourier synthesis was computed using those reflexions with $\sin\theta/\lambda < 0{\cdot}45$. This synthesis clearly indicated the positions of the hydrogen atoms which showed up as peaks in the electron-density map with peak heights between 0·55 e.Å^{-3} and 0·75 e.Å^{-3}. No other peak greater than 0·35 e.Å^{-3} was found. The hydrogen atoms attached to C5 and C6 were very close to the calculated positions. Two hydrogen atoms were attached to the nitrogen atom and a third to the remaining unbonded oxygen atom. The seven hydrogen atoms were included in the three-dimensional least-squares calculations with an isotropic

Table 1. *The positional parameters, their estimated standard deviations and the thermal parameters of the heavy atoms*

	x/a	$\sigma(x/a)$	y/b	$\sigma(y/b)$	z/c	$\sigma(z/c)$	B
S1	0·2961	0·0007	0·1515	0·0003	0·1488	0·0006	1·31
O2	0·5659	0·0013	0·1636	0·0006	0·2086	0·0011	2·62
O3	0·1564	0·0013	0·2589	0·0006	0·1434	0·0010	2·45
O4	0·2672	0·0012	0·0897	0·0006	−0·0119	0·0010	1·78
C5	0·1569	0·0018	0·0609	0·0009	0·3014	0·0014	2·03
C6	0·2869	0·0018	−0·0545	0·0009	0·3199	0·0014	2·05
N7	0·2359	0·0014	−0·1295	0·0007	0·1674	0·0012	1·73

temperature factor of 2·5 Å^2; neither the hydrogen atom co-ordinates nor their thermal parameters were refined.

After including the hydrogen atoms and removing the six reflexions which apparently were affected by extinction, the three-dimensional least-squares calculations were continued until the R-factor dropped to 0·11, at which stage, since the shifts in the parameters were less than the expected errors arising from random errors in the observational data, it was decided to stop the refinement.

The final co-ordinates of the heavy atoms along with their estimated standard deviations, calculated using the method of Cruickshank (1949), are shown in Table 1.

Table 2. *Hydrogen atom parameters*

	x/a	y/b	z/c	B
H1	0·1920	0·1093	0·4193	2·5
H2	−0·0048	0·0654	0·2464	2·5
H3	0·2172	−0·0957	0·4147	2·5
H4	0·4515	−0·0477	0·3351	2·5
H5	0·3016	−0·2162	0·1898	2·5
H6	0·0850	−0·1327	0·1621	2·5
H7	0·2837	0·2736	0·1975	2·5

The hydrogen-atom parameters, obtained from the three-dimensional difference Fourier synthesis, are given in Table 2.

Final electron-density projections on (100) and (001) are shown in Fig. 2 and Fig. 3. The minimum function map, $M4(y, z)$, shown in Fig. 1(b), compares well with the final $0kl$ electron density map shown in Fig. 2. The final R-factors for the three zones were $R(hk0)=0·12$, $R(h0l)=0·11$ and $R(0kl)=0·11$.

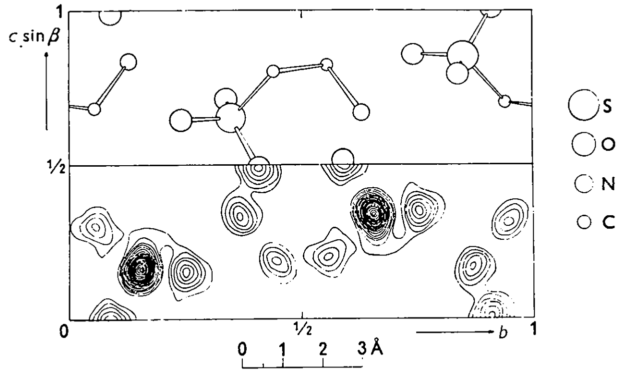

Fig. 2. The electron-density projection on (100). Contours at intervals of 2 e.Å^{-2} starting at ca. 3 e.Å^{-2}.

The observed and final calculated structure factors are compared in Table 4. During the evaluation of the R-factors all the accidentally absent reflexions were excluded. The observed intensities of some of the strongest reflexions, namely the 040, 002, 012, 110, 111 and 200 planes, marked by an asterisk in Table 4 are low, probably because of extinction effects. These six reflexions were omitted from the later cycles of the three-dimensional least-squares refinement and from the three-dimensional difference Fourier synthesis.

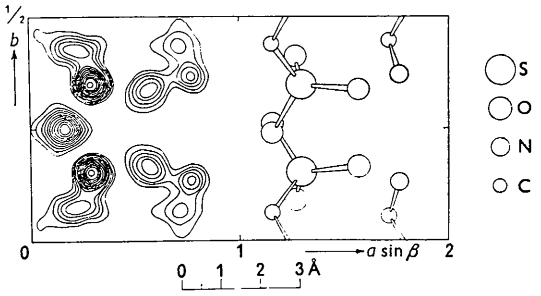

Fig. 3. The electron-density projection on (001). Contours at intervals of 2 e.Å^{-2} starting at ca. 4 e.Å^{-2}.

Discussion

In the crystals of taurine the molecules lie with their greatest length essentially along the b-axis of the unit cell. They are distributed throughout the cell in a close-packed arrangement with the molecular 'tail' occupying a position between the sulphonate groups of neighbouring molecules, as can be seen in Fig. 4.

Bond lengths and bond angles were calculated from the atomic co-ordinates given in Tables 1 and 2:

Table 3. *Bond lengths, bond angles and their estimated standard deviations*

Bond	Distance	σ	Angle	Degrees	σ
S1–O2	1·479 Å	0·008 Å	O2–S1–O3	113·9°	0·9°
S1–O3	1·453	0·008	O2–S1–O4	111·3	0·8
S1–O4	1·463	0·009	O2–S1–C5	105·9	0·9
S1–C5	1·805	0·012	O3–S1–O4	112·3	0·8
C5–C6	1·513	0·014	O3–S1–C5	106·9	0·9
C6–N7	1·501	0·014	O4–S1–C5	106·0	0·8
			S1–C5–C6	112·3	1·0
			C5–C6–N7	112·7	1·3

Table 4. *Final observed and calculated structure factors for taurine*

The column headings are k, l, F_o and F_c

h = 0

2	0	14.3	-17.7
4	0	48.3 *	-67.6
6	0	2.8	4.2
8	0	8.2	6.2
10	0	40.6	-38.1
12	0	16.6	13.6
14	0	13.9	11.7
1	1	33.7	38.8
2	1	36.8	-40.0
3	1	47.0	-50.4
4	1	33.4	33.7
5	1	21.0	-21.3
6	1	13.3	10.8
7	1	25.4	22.6
8	1	33.2	-33.0
9	1	6.8	- 5.6
10	1	19.8	16.0
12	1	14.8	12.5
13	1	12.4	10.8
14	1	14.2	-12.4
0	2	45.8 *	-59.6
1	2	46.0 *	-60.0
2	2	15.5	14.9
3	2	23.4	21.8
4	2	6.9	6.9
5	2	15.3	15.3
6	2	26.5	-25.9
7	2	18.3	-12.9
8	2	2.7	1.6
9	2	22.4	-22.9
10	2	28.6	23.8
11	2	30.0	25.3
13	2	4.2	- 2.6
14	2	5.6	- 4.5
1	3	2.3	- 0.7
2	3	11.2	8.4
3	3	60.2	68.5
4	3	7.4	3.1
5	3	14.0	-12.6
6	3	6.1	7.2
7	3	52.9	-57.1
8	3	6.6	- 6.8
9	3	12.7	13.2
10	3	4.0	- 3.2
11	3	12.5	11.0
12	3	3.4	3.1
13	3	11.9	-11.1
0	4	25.9	-25.9
1	4	28.0	29.8
2	4	27.7	27.3
3	4	2.8	- 2.9
4	4	5.2	2.7
5	4	11.5	-12.6
6	4	18.6	-17.7
7	4	9.6	10.5
8	4	17.8	-16.8
9	4	10.7	8.0
10	4	27.7	23.8
11	4	17.2	-14.6
13	4	5.1	4.6
1	5	6.1	6.7
2	5	16.6	18.1
3	5	21.9	-20.5
4	5	14.6	-12.9
5	5	6.4	- 4.5
6	5	13.0	-13.5
7	5	9.1	7.3
8	5	18.8	17.8
9	5	8.5	6.8
10	5	9.4	- 7.3
11	5	8.0	6.9
12	5	8.5	- 9.2
0	6	48.8	50.7
1	6	15.1	16.6
2	6	4.6	- 3.5
3	6	4.5	4.3
4	6	18.6	-20.8
6	6	4.2	3.2
7	6	3.8	- 4.4
8	6	5.5	5.4
9	6	8.0	7.7
10	6	17.7	-16.5
11	6	9.4	- 9.2
1	7	21.2	20.5
2	7	3.7	2.4
3	7	15.2	-15.3
4	7	9.0	7.9
5	7	9.5	- 9.9
7	7	10.8	12.0
8	7	5.9	- 5.5
9	7	10.6	- 9.2
10	7	3.6	3.0
1	8	13.9	-12.1
2	8	6.9	- 7.1
4	8	3.5	- 3.0
5	8	5.0	6.0
7	8	7.0	- 6.0
8	8	5.5	5.8
9	8	7.2	-10.5
2	9	6.7	- 8.0
3	9	8.9	9.3
4	9	7.9	8.8
6	9	5.3	7.3
0	10	5.3	-10.0
2	10	6.3	9.6

h = 1

0	0	18.1	16.1
1	0	36.6 *	-52.0
2	0	5.2	3.5
4	0	28.7	31.5
6	0	31.5	-31.1
7	0	9.1	8.6
8	0	8.1	5.4
9	0	10.8	- 7.9
10	0	6.9	5.6
11	0	23.3	22.9
12	0	6.1	- 4.7
13	0	7.3	- 5.8
14	0	4.3	- 2.7
1	1	63.1	-71.4
2	1	12.7	-12.2
3	1	35.7	37.2
4	1	41.1	-42.0
5	1	17.7	13.5
6	1	6.9	- 1.1
7	1	24.0	-22.9
8	1	6.3	5.3
9	1	22.1	21.8
10	1	10.2	8.7
11	1	16.7	17.3
12	1	4.1	2.0
13	1	15.8	-13.5
1	-1	49.7	50.0
2	-1	60.6	61.4
3	-1	12.9	-10.1
4	-1	30.7	-24.7
5	-1	19.8	-17.2
6	-1	24.4	-19.2
7	-1	17.0	17.1
8	-1	21.2	18.1
9	-1	16.1	-14.9
10	-1	10.8	-10.4
11	-1	12.9	14.2
12	-1	10.6	- 9.1
13	-1	4.7	4.1
14	-1	17.7	16.4
0	2	38.1	-19.7
1	2	3.3	- 8.0
2	2	38.6	35.4
3	2	3.2	- 4.0
4	2	13.2	12.4
5	2	24.5	-22.1
6	2	5.6	- 4.0
7	2	22.0	22.1
8	2	29.4	-28.2
9	2	11.8	11.9
10	2	16.0	17.1
11	2	12.2	-11.7
13	2	6.7	- 6.2
14	2	9.4	- 8.5
0	-2	59.8	63.4
1	-2	28.2	24.0
2	-2	61.8	-58.3
3	-2	50.6	49.8
4	-2	36.5	-35.7
5	-2	19.8	-18.9
6	-2	40.3	39.6
7	-2	9.6	5.9
8	-2	27.2	26.0
9	-2	6.8	- 7.4
10	-2	25.1	-25.0
12	-2	10.6	9.2
14	-2	8.9	8.4
1	3	11.7	14.0
2	3	8.0	- 8.0
3	3	3.7	- 2.3
4	3	11.1	-13.0
5	3	5.2	1.9
7	3	9.1	- 8.8
8	3	29.4	28.6
9	3	5.5	- 3.1
12	3	16.0	-16.2
13	3	4.5	2.8
1	-3	12.6	11.4
2	-3	6.1	0.8
3	-3	40.4	-38.9
4	-3	32.4	30.9
5	-3	10.3	12.1
6	-3	7.6	5.1
7	-3	23.8	24.1
8	-3	33.4	-34.6
9	-3	12.9	-13.2
10	-3	6.0	5.9
11	-3	4.8	- 4.7
12	-3	10.6	10.8
13	-3	12.1	11.2
14	-3	8.8	- 6.4
0	4	16.8	12.5
1	4	6.6	8.0
2	4	14.2	-10.9
3	4	36.0	-33.1
4	4	27.4	-25.8
5	4	12.8	- 9.9
6	4	15.8	15.9
8	4	15.8	17.0
9	4	16.9	18.6
10	4	7.4	- 7.4
11	4	9.1	- 8.5
0	-4	27.2	-26.8
1	-4	36.6	-37.8
2	-4	33.7	28.8
3	-4	11.3	11.0
4	-4	14.7	12.8
5	-4	29.1	30.8
6	-4	5.1	- 4.3
7	-4	20.2	-20.1
8	-4	22.2	-20.8
9	-4	20.4	-22.5
10	-4	3.9	2.6
11	-4	22.0	21.7
12	-4	5.4	- 5.1
1	5	21.6	21.8
2	5	9.4	- 6.8
3	5	22.7	-22.4
4	5	7.6	- 5.7
5	5	8.8	- 8.7
7	5	21.7	22.0
8	5	4.2	5.2
9	5	8.9	- 9.0
11	5	10.3	-10.5
1	-5	36.6	-39.3
2	-5	8.3	- 6.7
3	-5	35.2	38.2
4	-5	17.3	15.9
5	-5	5.7	6.8
6	-5	7.1	6.7
7	-5	21.2	-24.5
8	-5	11.8	-11.6
9	-5	18.0	19.8
10	-5	5.2	- 4.0
11	-5	9.0	7.5
12	-5	7.7	7.6
0	6	25.6	31.2
1	6	10.9	-11.3
2	6	4.9	- 6.1
5	6	14.0	14.0
9	6	4.8	- 4.7
10	6	10.9	-11.8
11	6	7.4	9.6
0	-6	36.5	-39.6
1	-6	24.0	26.5
4	-6	13.4	16.6
6	-6	8.7	- 9.2
8	-6	4.8	1.2
9	-6	4.8	3.4
10	-6	16.6	16.9
11	-6	14.5	-14.9
1	7	8.4	- 9.5
2	7	18.7	-23.8
3	7	5.5	6.9
6	7	5.5	5.5
7	7	6.7	- 7.1
8	7	9.5	-11.8
9	7	4.7	4.0
10	7	5.8	8.1
1	-7	10.2	10.4
2	-7	23.7	26.9
4	-7	8.7	-10.7
6	-7	10.6	-11.5
8	-7	12.8	15.9
9	-7	8.0	- 7.7
10	-7	6.8	- 7.1
0	8	18.4	-23.9
1	8	8.8	-13.3
2	8	5.5	7.8
3	8	4.3	- 3.6
4	8	9.8	15.1
6	8	9.4	-13.8
8	8	4.5	- 6.5
0	-8	16.3	23.5
1	-8	10.3	12.1
2	-8	10.0	-13.3
3	-8	8.3	8.3
4	-8	8.7	-10.7
5	-8	8.1	- 9.5
6	-8	9.7	14.1
8	-8	6.8	8.2
9	-8	3.3	6.6
2	9	4.7	7.1
3	9	7.6	13.7
3	-9	11.2	-15.4
4	-9	4.4	5.4
5	-9	4.4	3.7
1	-10	4.7	-10.7

h = 2

0	0	68.1 *	-87.4
1	0	16.5	-16.9
2	0	7.6	- 7.4
3	0	10.4	14.3
4	0	33.0	34.2
5	0	34.3	-34.0
6	0	9.7	6.6
7	0	4.2	1.4
8	0	11.5	- 8.5
9	0	6.1	4.1
10	0	18.1	19.0
11	0	4.4	1.6
12	0	4.8	- 3.4
14	0	11.9	-10.3
1	1	30.8	-30.1
2	1	15.2	15.0
3	1	19.5	19.0
4	1	7.3	- 8.1
5	1	20.1	19.9
6	1	11.4	- 8.0
7	1	8.3	- 4.8
8	1	18.1	14.3
9	1	4.2	2.4
10	1	5.2	- 3.5
11	1	8.3	- 6.4
12	1	16.8	-15.2
14	1	9.4	11.2
1	-1	21.1	-23.0
2	-1	29.2	-25.7
3	-1	28.2	30.0
4	-1	8.7	- 9.9
5	-1	13.4	14.3
6	-1	6.3	- 4.7
7	-1	19.4	-18.1
8	-1	9.1	7.4
10	-1	5.5	- 4.3
11	-1	8.7	6.2
13	-1	16.0	-14.6
14	-1	4.5	5.0
0	2	5.1	- 4.9
1	2	28.6	31.2
2	2	9.4	12.2
3	2	4.7	3.0
4	2	16.9	-17.9
5	2	4.2	2.5
6	2	23.1	24.3
7	2	12.7	12.3
9	2	10.4	8.8
10	-2	7.5	- 6.3
11	-2	11.7	-11.8
12	-2	8.9	- 7.7
1	3	8.3	8.0
2	3	8.1	- 5.7
3	3	38.9	-40.3
4	3	12.5	-11.1
5	3	7.9	7.9
6	3	5.2	- 4.6
7	3	38.0	37.2
9	3	11.2	- 9.9
10	3	5.4	- 3.4
11	3	16.6	-14.7
12	3	10.6	8.6
13	3	11.4	11.8
1	-3	13.8	-12.4
2	-3	31.0	30.7
3	-3	17.8	-17.5
4	-3	11.3	-12.4
6	-3	5.3	- 4.1
7	-3	29.0	30.9
8	-3	22.7	21.3
11	-3	12.6	- 9.6
12	-3	19.0	-17.9
0	4	25.1	22.8
1	4	10.8	- 9.8
2	4	21.8	-24.3
3	4	14.7	-14.4
4	4	7.1	- 4.5
5	4	18.5	19.4
6	4	20.1	20.3
7	4	14.7	-15.2
8	4	13.5	10.4
9	4	10.2	- 7.8
10	4	17.4	-16.0
11	4	14.5	13.5
0	-4	22.5	22.9
2	-4	11.3	-10.4
3	-4	10.7	9.8
4	-4	17.6	-18.3
5	-4	23.9	-26.2
6	-4	26.1	25.7
7	-4	11.5	9.0
8	-4	7.3	6.2
10	-4	22.5	-23.1
13	-4	3.6	- 3.6
1	5	12.0	-11.8
2	5	14.3	-15.5
6	-5	8.9	4.9
7	-5	9.1	9.0
8	-5	13.2	-11.2
9	-5	17.6	-16.7
10	-5	9.5	7.3
11	-5	10.5	- 9.5
12	-5	8.9	10.0
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1	6	12.9	-12.6
2	6	4.0	2.8
4	6	23.9	30.7
6	6	16.8	-17.6
8	6	5.2	- 4.0
10	6	12.5	14.4
0	-6	31.1	-31.3
1	-6	20.9	-23.8
3	-6	12.6	-10.9
4	-6	6.9	5.9
5	-6	25.9	29.2
6	-6	10.7	10.5
7	-6	7.1	- 5.8
8	-6	5.5	- 3.8
9	-6	9.9	- 9.8
10	-6	8.9	8.5
11	-6	8.1	11.6
1	7	14.8	-18.5
2	7	5.6	8.0
3	7	14.5	18.2
4	7	14.7	-17.3
5	7	6.7	6.4
6	7	7.9	- 6.6
7	7	9.1	-10.0
8	7	9.1	10.9
9	7	6.0	8.0
1	-7	16.4	-19.4
2	-7	14.6	-19.6
3	-7	17.0	22.3
4	-7	6.7	6.3
5	-7	4.9	3.3
6	-7	7.9	6.9
7	-7	12.6	-15.4
8	-7	4.9	- 5.1
9	-7	9.7	10.5
1	8	10.2	14.5
3	8	5.8	6.8
4	8	3.9	- 4.9
5	8	6.6	-11.7
6	8	6.4	9.1
10	2	17.9	-18.2
11	2	22.9	-19.2
12	2	7.9	- 5.8
0	-2	3.6	2.9
1	-2	6.9	2.4
2	-2	16.0	-15.0
3	-2	12.4	-11.9
4	-2	28.4	26.5
5	-2	18.8	-16.6
6	-2	10.9	- 9.4
7	-2	7.5	6.2
8	-2	11.9	8.7
9	-2	17.8	16.7
3	5	14.7	15.7
4	5	22.8	25.5
5	5	7.9	6.3
6	5	14.8	12.8
7	5	7.9	- 7.1
8	5	17.9	-16.9
9	5	4.8	- 2.9
10	5	6.7	5.1
1	-5	10.7	10.6
2	-5	20.3	-19.0
3	-5	13.0	-14.8
4	-5	4.5	- 0.5
5	-5	14.0	12.8
7	8	7.3	12.8
0	-8	8.5	-11.2
1	-8	6.1	8.1
2	-8	6.3	7.9
3	-8	7.1	- 8.0
4	-8	11.7	15.3
5	-8	6.9	- 8.4
6	-8	13.6	-15.8
1	9	3.9	9.0
2	9	2.9	3.8
3	9	7.7	-17.8
1	-9	4.3	- 3.4
2	-9	10.7	16.2
4	-9	8.9	-12.9

h = 3

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1	0	27.7	22.8
2	0	7.7	7.3
3	0	10.1	8.8
4	0	27.4	-26.3
5	0	26.6	-25.0
6	0	20.6	21.3
7	0	9.2	7.5
10	0	9.2	- 7.9
11	0	14.0	-12.7
12	0	2.6	3.0
13	0	5.5	7.7
1	1	47.2	38.0
2	1	14.6	13.2
3	1	46.9	-44.0
4	1	21.6	23.3
5	1	2.9	- 0.1
6	1	11.1	12.5
7	1	22.5	23.7
8	1	4.4	- 6.0
9	1	16.4	-15.3
10	1	14.2	-12.2
11	1	11.8	-12.0
12	1	2.4	1.7
13	1	12.4	15.7
1	-1	16.3	-14.8
2	-1	38.2	34.4
3	-1	17.6	18.1
4	-1	17.8	-18.8
6	-1	20.4	-17.1
7	-1	14.7	-13.4
8	-1	13.0	11.9
9	-1	8.1	7.5
11	-1	4.2	5.6
12	-1	12.6	-11.1
13	-1	2.0	1.1
0	2	32.8	32.5
1	2	12.3	-10.2
2	2	10.4	- 8.7
3	2	10.2	- 8.6
4	2	21.0	-22.5
5	2	37.2	37.0
6	2	11.8	12.6
7	2	24.8	-24.6
8	2	5.4	5.9
9	2	14.8	-14.0
10	2	8.0	- 8.8
11	2	14.5	14.1
12	2	3.2	3.4
0	-2	19.6	-18.7
1	-2	7.2	8.5
2	-2	12.4	12.7
3	-2	39.2	41.0
4	-2	27.5	27.7
5	-2	7.1	- 6.4
6	-2	27.1	-30.8
7	-2	2.9	2.7
8	-2	8.4	-10.2
10	-2	15.6	15.2
11	-2	4.4	- 5.3
13	-2	8.2	- 8.8
1	3	10.8	-10.5
2	3	19.3	-20.0
3	3	16.4	16.5
4	3	18.5	18.6
5	3	4.9	- 4.6
7	3	8.8	- 8.0
8	3	26.9	-26.3
9	3	8.0	8.1
10	3	8.8	6.0
11	3	7.0	4.8
12	3	16.4	17.5
1	-3	5.8	5.1
2	-3	3.6	3.5
3	-3	16.9	16.1
4	-3	12.3	13.7
5	-3	4.5	- 5.1
7	-3	13.0	-14.4
8	-3	15.3	-15.1
9	-3	7.6	7.9
10	-3	3.3	3.4
11	-3	12.5	11.2
12	-3	3.5	5.3
0	4	28.3	-29.7
1	4	7.0	8.7
2	4	8.1	5.5
3	4	20.6	20.2
4	4	34.4	33.9
5	4	5.9	6.1
6	4	31.9	-29.4
7	4	2.8	- 1.3
8	4	3.1	- 2.9
9	4	3.5	- 4.8
10	4	15.8	15.6
11	4	2.7	- 1.3
0	-4	16.4	14.9
1	-4	7.4	- 7.3
2	-4	8.8	- 9.9
3	-4	11.7	-10.7
4	-4	2.6	- 2.3
5	-4	14.3	16.6
6	-4	6.9	- 6.9
7	-4	15.1	-15.4
8	-4	9.0	8.5
9	-4	11.6	-12.9
10	-4	4.1	4.7
11	-4	12.2	13.1
1	5	12.8	-13.6
2	5	28.9	27.9
3	5	18.1	19.0
4	5	8.9	- 8.6
6	5	5.6	- 5.8
7	5	15.0	-15.1
8	5	10.4	10.0
9	5	5.4	4.9
10	5	5.2	- 5.2
1	-5	16.7	18.9
2	-5	9.2	- 9.1
3	-5	12.3	-12.5
4	-5	19.4	17.0
5	-5	7.7	- 8.6
6	-5	13.7	13.8
7	-5	6.8	6.9
8	-5	11.2	-12.4
9	-5	5.5	- 5.6
10	-5	7.7	- 8.0
11	-5	5.1	- 5.8
0	6	5.1	4.3
1	6	10.3	10.1
3	6	3.2	5.4
4	6	6.2	- 5.7
5	6	25.7	-24.6
6	6	8.2	8.5
7	6	4.8	5.2
9	6	2.6	3.5
0	-6	19.1	17.2
2	-6	2.7	3.4
4	-6	17.5	-18.2
5	-6	11.3	10.4
6	-6	11.1	11.3
7	-6	3.3	- 2.8
9	-6	4.2	- 4.5
10	-6	7.6	-10.2
1	7	9.5	11.9
2	7	7.0	8.3
3	7	16.0	-19.6
4	7	5.1	4.7
7	7	11.1	15.2
3	-7	9.5	-10.9
4	-7	8.2	- 8.9
6	-7	9.8	-10.6
7	-7	4.1	5.8
8	-7	4.7	5.8
0	8	10.5	11.6
2	8	2.9	- 2.4
3	8	3.2	3.1
4	8	8.9	-14.1
5	8	7.6	13.2

h = 4

0	0	21.9	19.7
1	0	20.8	-22.0
2	0	6.4	6.0
3	0	10.0	-10.7
4	0	20.4	-23.0
5	0	33.7	39.8
6	0	7.8	6.7
7	0	10.0	- 8.1
8	0	2.4	2.9
9	0	12.7	-10.8
11	0	11.2	9.6
1	1	3.1	- 1.0
2	1	17.1	-19.1
3	1	14.7	13.9
4	1	14.2	16.0
5	1	13.4	-13.4
6	1	7.7	7.7
7	1	13.8	-13.2
8	1	13.6	-12.2
9	1	13.8	12.7
10	1	7.1	2.2
11	1	6.1	5.3
1	-1	25.9	28.0
2	-1	24.0	26.2
3	-1	30.5	-34.3
4	-1	4.8	6.2
6	-1	3.4	- 3.0
7	-1	20.6	17.3
8	-1	6.8	5.8
9	-1	13.6	-11.5
11	-1	8.	- 6.1
12	-1	7.8	- 7.3
0	2	19.0	-12.9
1	2	6.6	7.3
2	2	7.2	8.0
3	2	10.3	-10.8
4	2	19.7	21.8
5	2	14.0	-12.9
6	2	30.0	-30.7
7	2	5.8	- 5.6
8	2	3.9	- 3.5
9	2	8.3	6.0
10	2	17.7	16.6
11	2	1.8	3.5
0	-2	23.8	22.3
1	-2	7.3	- 6.0
2	-2	1.7	- 1.0
3	-2	2.9	- 3.0
4	-2	19.9	-20.4
5	-2	23.8	24.0
6	-2	14.3	12.9
7	-2	17.5	-15.7
8	-2	4.4	- 4.1
9	-2	9.0	- 6.9
10	-2	11.4	- 7.9
11	-2	9.5	9.0
1	3	5.5	- 5.6
2	3	20.6	22.9
3	3	13.8	14.6
4	3	11.7	-10.8
6	3	5.7	- 6.3
7	3	15.3	-14.3
8	3	18.2	16.2
9	3	3.5	3.6
2	-3	28.6	-32.8
3	-3	11.4	10.8
4	-3	14.1	14.3
5	-3	3.4	- 4.0
6	-3	4.6	4.7
7	-3	12.1	- 9.7
8	-3	24.7	-23.9
11	-3	5.1	5.4
0	4	11.4	10.7
1	4	9.2	8.3
2	4	4.0	- 3.5
3	4	11.0	12.
4	4	5.3	- 4.1
5	4	12.2	-14.0
7	4	14.6	12.2
8	4	3.5	- 1.8
9	4	6.6	6.5
0	-4	17.1	-15.7
1	-4	6.5	- 5.4
2	-4	4.4	4.8
4	-4	18.7	[illegible]
5	-4	17.9	20.9
6	-4	24.2	-25.3
9	-4	12.1	-11.4
10	-4	13.8	14.4
1	6	17.3	17.1
2	5	6.8	- 4.1
3	5	17.7	-15.7
4	5	9.2	- 8.5
5	5	4.4	- 4.8
6	5	5.5	- 7.5
7	5	8.5	8.1
9	5	4.6	- 6.8
1	-5	5.6	- 7.6
2	-5	17.2	15.6
3	-5	15.3	13.9
5	-5	9.5	- 7.5
7	-5	13.8	-12.9
8	-5	5.8	4.9
9	-5	10.7	10.5
10	-5	4.8	- 6.1
0	6	17.9	16.7
1	6	7.9	- 7.6
4	6	18.0	-18.6
5	6	9.4	7.9
6	6	7.2	8.8
7	6	2.4	- 1.3
0	-6	10.1	9.4
1	-6	11.2	12.0
2	-6	3.9	2.0
3	-6	9.0	8.2
4	-6	4.1	- 3.7
5	-6	25.7	-26.8
6	-6	4.4	- 3.9
7	-6	3.2	2.9
1	7	3.3	3.9
2	7	4.6	- 6.4
3	7	4.8	- 5.6
4	7	9.0	11.6
5	7	3.3	- 3.8
1	-7	8.0	9.1
2	-7	15.8	18.1
3	-7	11.4	-12.1
4	-7	4.8	- 2.5
6	-7	7.3	- 8.2
7	-7	5.1	7.7
0	-8	7.8	7.5
1	-8	4.1	3.4
3	-8	6.3	7.4
4	-8	7.8	-11.6

h = 5

0	0	22.5	-21.7
1	0	11.4	10.1
2	0	4.8	0.8
4	0	13.4	14.4
6	0	11.6	-10.6
9	0	7.3	6.3
10	0	11.7	12.7
1	1	6.7	- 7.1
2	1	7.5	8.2
3	1	10.9	13.5
4	1	18.6	-17.4
5	1	4.2	2.6
6	1	14.1	-14.3
7	1	9.6	-10.8
1	-1	9.5	- 8.2
2	-1	17.1	17.5
3	-1	10.2	9.2
4	-1	8.0	- 7.5
6	-1	9.6	- 9.8
7	-1	4.0	- 4.1
8	-1	5.1	6.6
0	2	4.0	1.5
1	2	5.1	6.7
2	2	5.1	- 5.5
4	2	7.0	7.2
5	2	16.8	-17.8
7	2	5.9	7.8
1	-2	23.0	26.6
2	-2	4.4	4.6
3	-2	16.4	18.9
4	-2	7.4	- 8.9
5	-2	15.2	-14.2
6	-2	8.7	8.7
7	-2	7.6	6.3
3	3	13.4	-12.4
6	3	5.4	5.4
7	3	9.7	13.8
1	-3	6.3	6.5
2	-3	11.8	13.2
3	-3	18.7	-18.9
4	-3	7.2	- 5.6
5	-3	7.0	5.9
6	-3	9.4	- 9.8
7	-3	15.2	15.6
8	-3	5.0	4.4
0	4	16.6	15.5
1	4	12.5	8.3
3	4	11.5	- 8.1
4	4	16.5	-14.2
0	-4	10.6	10.2
1	-4	6.4	- 5.8
2	-4	10.6	- 9.7
3	-4	9.2	- 7.9
4	-4	5.7	- 5.2
5	-4	11.8	11.4
6	-4	16.9	16.1
7	-4	11.1	-10.9
1	5	8.3	6.7
2	5	17.1	-17.7
3	5	7.2	- 6.3
4	5	3.0	2.6
2	-5	17.9	-16.5
4	-5	20.5	21.4
5	-5	4.0	3.6
6	-5	15.9	14.8
0	6	11.4	-12.5
1	6	5.4	- 5.8
0	-6	18.0	-15.6

h = 6

0	2	10.8	7.6
0	-2	18.0	-14.0
0	4	6.8	- 8.2
0	-4	4.4	- 3.4
1	0	15.0	15.4
3	0	6.5	5.4
4	0	6.5	5.4
5	0	15.4	-15.0
6	0	4.0	- 1.6

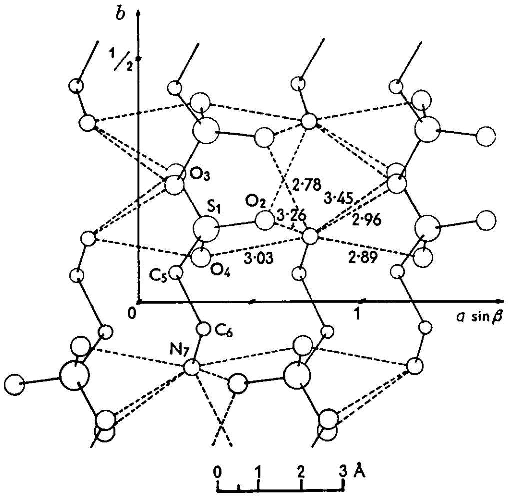

Fig. 4. A view of the structure along the *c*-axis. The inter-molecular distances < 3·5 Å are indicated.

no attempt was made to correct for possible angular oscillations of the molecule (Cruickshank, 1956). From the estimated standard deviations of the atomic co-ordinates the standard deviations of the bond lengths were calculated by the method of Cruickshank & Robertson (1953). Since the standard deviations in the x, y and z atomic co-ordinates were very similar, the standard deviations of the bond angles were calculated, assuming isotropic standard deviations for each atom, by the method of Darlow (1960). The bond lengths, bond angles and their estimated standard deviations are collected in Table 3.

The mean value of the sulphur–oxygen bond lengths is 1·465 Å and the differences between this and the individual bond lengths are not significant. The sulphur–carbon bond length is 1·805 Å and both this value and the values of the sulphur–oxygen bond lengths agree well with the mean values quoted for other compounds containing bonded sulphur, oxygen and carbon atoms. The atom C5 and the three oxygen atoms form a tetrahedral group with the sulphur atom at the centre. The mean oxygen–sulphur–oxygen bond angle is 112·2° which is significantly greater than the regular tetrahedral angle of 109° 48′, while all three oxygen–sulphur–oxygen bond angles are essentially identical. This deviation from the regular tetrahedral angle is possibly due to the short separation distance between the oxygen atoms of 2·43 Å. These results are very similar to those obtained by Hargreaves (1957) for the sulphonate radical in zinc *p*-toluene sulphonate hexahydrate. The carbon–carbon bond length of 1·513 Å is significantly shorter than the accepted value of 1·545 Å of the carbon–carbon single bond. The normal single carbon–nitrogen bond is 1·475 Å and thus the value determined here of 1·501 Å is possibly significantly longer than the accepted value. Hahn (1957), analyzing the more accurate structural determinations made on amino and carboxylic acids, noted that for amino acids the molecule occurs as a 'zwitterion' *i.e.* the amino groups have the form C1–C2–NH_3^+. The mean values quoted for the bond lengths C1–C2 and C2–NH_3^+ were 1·51 Å and 1·50$_5$ Å respectively and the mean C1–C2–NH_3^+ bond angle was 110°. These are very similar to the values found in this determination. There was, however, no evidence in the three-dimensional difference Fourier synthesis for the existence of the 'zwitterion' configuration.

The two hydrogen atoms attached to N7 are involved in hydrogen bonding to oxygen atoms of neighbouring molecules. The N–H · · · O hydrogen bonds were calculated as 2·78 Å and 2·89 Å bonding N7 to O2 $(1-x, -\frac{1}{2}+y, \frac{1}{2}-z)$ and to O4 $(\bar{x}, \bar{y}, \bar{z})$ respectively. These hydrogen bonds are certainly shorter than the accurately determined N–H · · · O distances in urea (2·99 Å and 3·04 Å) by Vaughan & Donohue (1952) but they do agree well with the hydrogen-bond lengths determined in thiourea dioxide of 2·84 Å and 2·85 Å by Sullivan & Hargreaves (1962). The angles C6–N7–O2 and C6–N7–O4 involved in the hydrogen bonding were 100·3° and 110·8° respectively while the angles H–N7–O2 and H–N7–O4 were 7·8° and 34·1° respectively.

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