The Structure of Dimethanesulphonyl Disulphide

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In the present note, some preliminary results are reported of a crystal structure analysis of dimethanesulphonyl disulphide [1]. This compound is the analogue of potassium tetrathionate, for which lattice dimensions, space group and refractive indices are known [2]. No other data on tetrathionic compounds are reported in literature.

The crystals of dimethanesulphonyl disulphide are in most cases found as colourless, transparent needles or plates elongated in the *a*-axis direction. The most frequent faces of the monoclinic crystals are (010), (021), (001) and (011). The crystals seem to have good cleavage parallel to the planes (011) and (01$\bar{1}$) with less good cleavage parallel to the (010) plane.

0.1029 g substance: 0.4325 g $BaSO_4$.
$(CH_3SO_2S)_2$ (222.3)
Calc. S 57.68. Found S 57.72.

Further experiments on aliphatic thiosulphonates and disulphonyl disulphides, including measurements of their equilibria with iodide-iodine, will be described in a later article.

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The dimensions of the unit cell were obtained from rotation and goniometer photographs: $a = 5.52 \pm 0.02$ Å, $b = 15.78 \pm 0.02$ Å, $c = 10.05 \pm 0.02$ Å. $\beta = 97.6°$ and $V = 866$ Å^3. The values for the axial lengths correspond to axial ratios of $a : b : c = 0.349 : 1 : 0.637$. The density of the crystals is 1.71 g/cm^3 and the number of $(CH_3SO_2S)_2$ in the unit cell therefore 4. The following reflexions are absent in the X-ray photographs: $0k0$ when k is odd, $00l$ when l is odd, and $h0l$ when l is odd. The *b*-axis is accordingly a twofold screw axis and the (010) plane is a glide plane of symmetry with translation $c/2$. Laue patterns indicate monoclinic holoedry and no other observations contradict this finding. The space group is therefore $C_{2h}^5 - P\frac{2_1}{c}$.

The molecule could possibly have a plane, a centre or twofold axis of symmetry. The space group C_{2h}^5 possesses only glide planes, screw axis and centres. The possibilities of molecular symmetry are therefore reduced to centre only. It has, however, proved impossible to account for the observed reflexion intensities if the centres of the molecules should be located to the symmetry centres of the space group. The molecules must consequently be placed in general positions, and can thus have no strict symmetry of its own. A general point repeats four times in the space group C_{2h}^5. All the molecules must therefore be crystallographically equivalent to each other, since there are only four molecules in the unit cell.

A preliminary analysis has shown that the molecule has an unbranched sulphur chain structure. The main direction of the sulphur chain is nearly parallel to the [011] or [01$\bar{1}$] direction alternatingly. The approximate centres of the four molecules are located very closely to the positions:

$$x\ \tfrac{1}{8}\ \tfrac{1}{8}\ ;\ x\ \tfrac{\bar{1}}{8}\ \tfrac{\bar{1}}{8}\ \text{and}\ x\ \tfrac{7}{8}\ \tfrac{\bar{7}}{8}\ ;\ x\ \tfrac{\bar{7}}{8}\ \tfrac{7}{8}$$

Studies on Liver Arginase. II. The Separation of a Single Protein with Arginase Activity

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An extensive study of the properties of horse liver arginase revealed that although it is extremely sensitive to pH values lower than 5.0, yet it can withstand unusually high temperatures in the neutral range. It is neither precipitated nor its activity impaired by such treatment. Manganese-treated crude extracts at pH 7.2—7.7, can be heated directly on the flame to a temperature of 80—90° C. A lot of inert proteins closely associated with the enzyme can thus be removed. Moreover, the enzyme activity is unaffected by lead ion in any concentration. The amount of lead ion that can be added to the enzyme extract without causing its precipitation depends upon the concentration of the enzyme protein and previous treatments; such as, addition of manganese and heating. The enzyme is more easily precipitated with lead if previously heated. Unheated liver extracts can be treated with considerably high concentrations of lead (about 8 mg Pb^{++}/ml) without any change in the enzyme which remains in solution. This fact was first mentioned and utilized for the partial purification of arginase by Safwat Mohamed and Greenberg [1]. Their procedure was utilized later by Thompson [2, 3] with some additional steps. The use of the combination of Mn^{++} and phosphate ions as a means of purification of arginase, reported by Thompson, has been tried by the author and was found to entail a considerable loss to the enzyme. The disappearance of the color in Thompson's final extract (K)[3] is due more to dilution than to removal of the coloring matter. Still more loss is to be encountered if the final dilute extract (K) is to be concentrated through the use of acetone or ammonium sulfate.

In the following a brief account is given of the procedure that yields arginase of the highest purity and activity.

a) Three hundred g acetone-dried horse liver, extracted with 3 l distilled water containing 2 g $MnSO_4 \cdot 4H_2O$. Adjust pH to 7.5. Stir continuously for 8 h at room temperature. Centrifuge and discard residue.

b) Supernatent (I) at 3° C is mixed with 0.7 volumes cold acetone with stirring. Let stand at 3° C for 8 h. Centrifuge in cold room. Discard residue.

c) Supernatent, at 3° C, mixed with 0.5 volumes cold acetone with stirring and left at 3° C for 8 h. Centrifuge in cold room and discard supernatent.

d) Precipitate is taken up in distilled water. Extract (II); add 0.5 g $MnSO_4$, adjust pH to 7.5. Heat to 80° C and cool rapidly. Centrifuge and discard precipitate.

e) To supernatent (III) add $Pb(CH_3COO)_2 \cdot 3H_2O$ to give 2.0 mg Pb++/ml. Mix thoroughly and adjust pH to 7.5. Let stand at room temperature for 2 h Centrifuge.

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either of these pairs forming double molecules around the centres of symmetry in (000) and (0 $\frac{1}{2}$ $\frac{1}{2}$) respectively.

This structure is in agreement with interatomic distances, previously reported, with a Patterson synthesis of the 0*kl* data and can account satisfactorily for the relative intensities of the reflexions.

Further data and a more detailed description of this structure will be given in another paper.

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