CRYSTAL STRUCTURE OF THIODIGLYCOLLIC ACID

SUKLA ROY

INDIAN ASSOCIATION FOR THE CULTIVATION OF SCIENCE,
CALCUTTA-32.

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The unit cell and space group of thiodiglycollic acid has already been determined (Roy, 1962). The lattice is orthorhombic and there are 4 molecules per unit cell. The systematic absences observed are consistent with the space group Pnam or $Pna2_1$ with

$$a = 5.03 \pm .03 \text{ Å}$$
$$b = 6.66 \pm .03 \text{ Å}$$
$$c = 17.76 \pm .03 \text{ Å}$$

All the intensity data were obtained from Weissenberg photograph about a and b axis using multiple film technique (Robertson, 1943). Intensities were estimated visually by comparison with a standard intensity strip. After correction for Lorentz and polarisation factors $Fo^2(okl)$, were put on an absolute scale by Wilson's method. No correction for absorption was made at this stage.

The $N(Z)$ statistical test (Howells *et al.*) was applied to 100 zone reflections for which the projection is centric for Pnam and acentric for $Pna2_1$. It was found that the experimental values of $N(Z)$ agreed very well with the theoretical curve for the centrosymmetric case, thereby implying the space group Pnam.

There are 8 general positions for the space group Pnam. Since each unit cell of the crystal contains 4 molecules the sulphur atoms must occupy the following special position,

$$x, y, \tfrac{1}{4}\ ;\quad \bar{x}, \bar{y}, 3/4\ ;\quad \tfrac{1}{2}-x,\ \tfrac{1}{2}+y,\ 3/4\ ;\quad \tfrac{1}{2}+x,\ \tfrac{1}{2}-y,\ 1/4\ ;$$

Using the *okl* data a two-dimensional Patterson was computed to facilitate the determination of approximate y, z parameters of the sulphur atoms.

Approximate coordinates of the sulphur atoms thus obtained were used to calculate the structure factors of the *okl* reflections.

Signs of some of the structure factors thus obtained were then used to compute the (100) electron density projection (Fig. 1) from which an approximate trial structure could be postulated. At this stage the reliability index was 0.42. The overall temperature factor was taken throughout to be 1.34.

Three cycles of Fourier refinement reduced the R value to .34. But no further change was observed. Two cycles of difference fourier were then carried

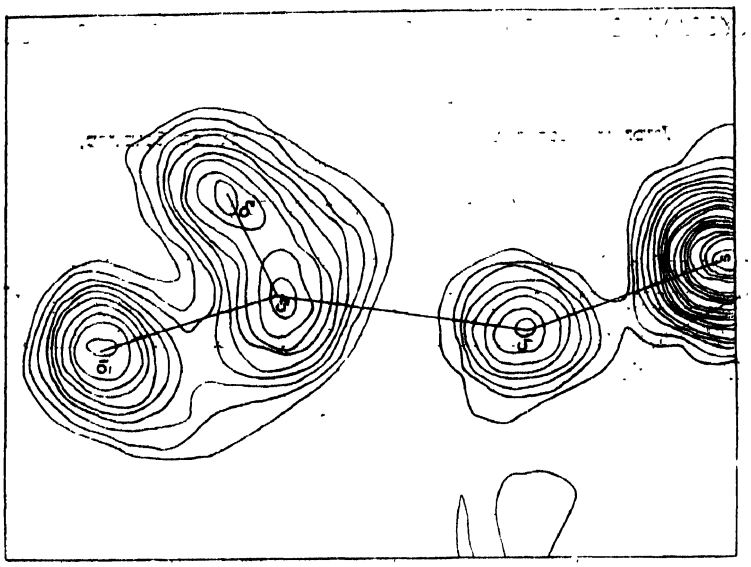

Fig. 1

out and these reduced the R value to 0.26. It was apparent from the difference Fourier that some atoms had a temperature factor higher than 1.34 and the sulphur atom had an anisotropic temperature factor.

Another Patterson projection was computed to determine the third co-ordinate from the 'b' axis 0-layer data. The R value for this zone is now 0.30.

Further refinement is proceeding using difference synthesis and least squares method.

Throughout the analysis of the crystal the scattering factor value of Berghus *et al.* were used for carbon and oxygen while sacttering factors of sulphur were based on values given by C. H. Stam.

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