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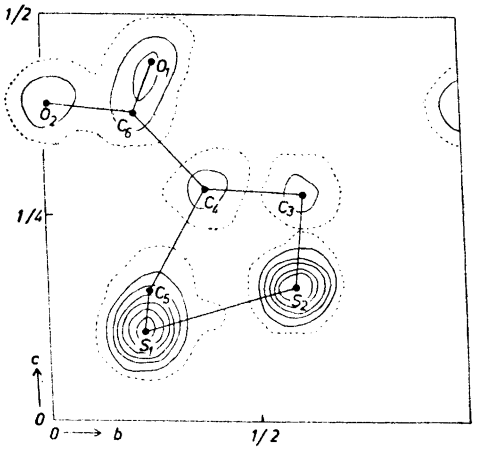

Fig. 1. Electron density projection of 1,2-dithiolane-4-carboxylic acid along the a axis, showing one asymmetric unit (one molecule). Contours at intervals of 3 e · Å^{-2}, beginning with 3 e · Å^{-2} (dashed).

Structure of Crystalline 1,2-Dithiolane-4-carboxylic Acid

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As part of a program for the study of crystal structures of five-, six- and seven-membered cyclic disulphides, with the view of obtaining information about the stereochemistry of the disulphide group in the rings, unit cell and space group determinations have recently been made of some 1,2,4-dithiazolidine derivatives [1] and of some 1,2-dithiolane-, 1,2-dithiane-, 1,2-diselenane- and 1,2-dithiepane-carboxylic acids [2]. Also, the detailed structure of thiuret (3,5-diimino-1,2,4-dithiazolidine) hydroiodide has been worked out [3,4]. The present note gives the preliminary results of a crystal structure determination of 1,2-dithiolane-4-carboxylic acid [5,6].

The crystals are triclinic, with the unit cell dimensions [2], $a = 5.34$ Å, $b = 5.85$ Å, $c = 10.75$ Å, $\alpha = 93\frac{1}{2}°$, $\beta = 89\frac{1}{2}°$, $\gamma = 109\frac{1}{2}°$. The space group is $C_i^1 - P\bar{1}$, and there are two molecules per unit cell. The intensities of the $0kl$ reflections were estimated visually from Weissenberg photographs taken with CuK radiation, and were converted to relative structure factors in the usual way. The positions of the two sulphur atoms were found from a F_0^2 synthesis of the $0kl$ data, and a subsequent F_0 synthesis with signs based on the sulphur contributions showed the outline of the molecule and gave approximate coordinates for the carbon and oxygen atoms. After a few F_0 and $(F_0 - F_c)$ refinements the $0kl$ electron density map is as shown in Fig. 1, and the y and z coordinates as listed in Table 1. The reliability index R is at this stage 15.7 %.

Provided that the chemically equivalent S_1-C_5 and S_2-C_3 bonds and $S_1-S_2-C_3$ and $S_2-S_1-C_5$ bond angles do not have too different dimensions, the a-axis projection shows that the disulphide group of the 1,2-dithiolane ring is not planar. If, pending the determination of the x coordinates through a projection along another axis, the assumption is made that the S—C bond lengths are equal and approximately 1.81 Å, and that the S—S—C bond angles are equal, the y and z coordinates of the sulphur atoms and the C_3 and C_5 atoms lead to a disulphide group with dimensions approximately as follows: S—S bond length = 2.09 Å, S—S—C bond angles = 95°, and CSS/SSC dihedral angle = 20°.

The carboxyl groups of different molecules appear to be arranged around symmetry centres at $z = \frac{1}{2}$, and non-bonded S—S contacts only to occur across the c plane at $z = 0$.

Table 1. Atomic coordinates, in fractions of triclinic cell edges. Origin at a centre of symmetry.

	y	*z*
S_1	0.230	0.108
S_2	0.593	0.164
C_3	0.620	0.280
C_4	0.385	0.286
C_5	0.243	0.161
C_6	0.218	0.379
O_1	0.265	0.443
O_2	0.012	0.390

It is intended to refine the *a*-axis projection further, and to carry out a detailed structure analysis.

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Structure of Crystalline *Racem*-1,2-dithiane-3,6-dicarboxylic Acid

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In two preceding notes [1,2], crystal data on some carboxylic acid derivatives of five-, six- and seven-membered cyclic disulphides, and preliminary results of a crystal structure determination of the five-membered one, 1,2-dithiolane-4-carboxylic acid, have been reported. A corresponding study of a six-membered representative, *racem*-1,2-dithiane-3,6-dicarboxylic acid[3-6], has so far given the following results.

The crystals are monoclinic prismatic, with a four-molecule unit cell based on the space group C_{2h}^6-I2/c, and with the unit cell dimensions [1], $a = 9.62$ Å, $b = 9.26$ Å, $c = 9.70$ Å, $\beta = 102\frac{1}{2}°$. The correctness of the centrosymmetric space group was first assumed, and confirmed through the subsequent work. The intensities of the *h0l* reflections were estimated visually from Weissenberg photographs taken with Cu*K* radiation; 51 reflections out of 57 attainable were recorded with measurable intensities. The position of the one sulphur atom of the asymmetric unit was derived

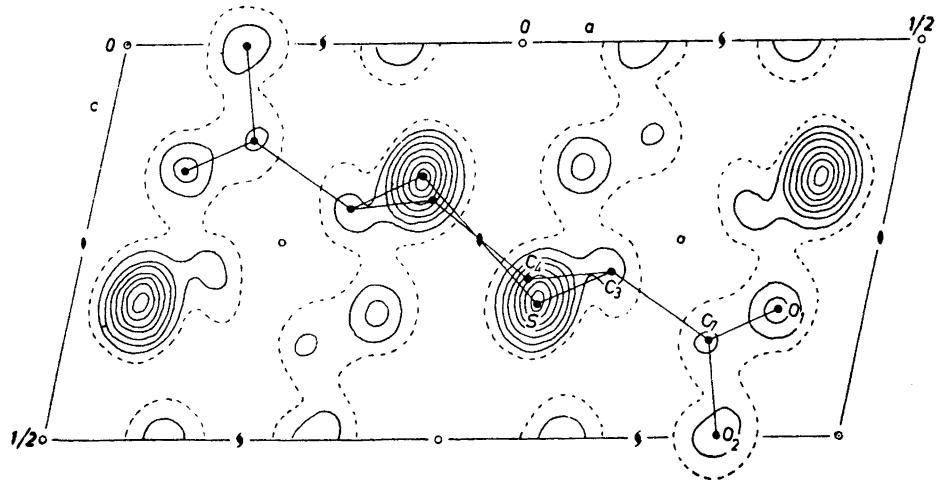

Fig. 1. Electron density projection of *racem*-1,2-dithiane-3,6-dicarboxylic acid along the *b* axis. Four asymmetric units (half a unit cell) are shown, and lines are drawn to indicate the outline of one molecule. Contours at intervals of 3 e · Å^{-2}, beginning with 4 e · Å^{-2} (dashed).