

experiments where  $^{15}\text{NH}_4\text{Cl}$ , ATP,  $\text{MgCl}_2$ , acetyl glutamate and aspartate- $^{14}\text{C}$  were substrates. The influence of increasing amounts of  $^{13}\text{C}$  on the isotopic pattern of the formed USA was studied. The results were in full agreement with the scheme, since the  $^{15}\text{N}$ -content of USA sharply decreased with increasing amounts of CAP, while the  $^{13}\text{C}$ -content increased and finally reached the same level as the  $^{14}\text{C}$ -content. Similar results were obtained with labeled citrulline in place of ammonia.

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## The Preparation of 2,6-Dichlorophenylacetic Acid

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*Ortho* halogen substituted benzoic, phenylacetic and phenoxyacetic acids have in recent years been frequently tested as to their auxin activity, among others by Thimann<sup>1</sup>. The activity of a benzoic acid is greatly increased by di-substitution of halogen atoms in 2,6-position, while the same substitution in a phenoxyacetic acid completely destroys the activity.

The 2,4,6-trichlorophenylacetic acid was recently prepared by Jönsson<sup>2</sup>, but the 2,6-dichlorophenylacetic acid is not earlier described. By a method related by Fierz-David and Blangey<sup>3</sup> 2,6-dichlorotoluene was prepared. This compound was chlorinated to benzyl chloride which was treated with potassium cyanide to benzyl cyanide and hydrolysed to phenylacetic acid as Staedel<sup>4</sup> has described.

For the sake of control a sample of the 2,6-dichlorotoluene was oxidized, yielding pure 2,6-dichlorobenzoic acid with m. p. 140–141°.

*Experimental: 2,6-Dichlorobenzyl chloride.* 16.1 g of 2,6-dichlorotoluene was chlorinated with dry chlorine at 180° in ultra-violet rays until an increase of the weight of 3.5 g had been obtained. The product was fractionated *in vacuo* yielding 12 g (61 %) of colourless oil, boiling at 114–119°/13 mm, melting point 11–12°.

*2,6-Dichlorobenzyl cyanide.* 6.5 g of benzyl chloride was refluxed 5 hours with 2.7 g of potassium cyanide and 30 ml alcohol. The alcohol was distilled off from the solid.

*2,6-Dichlorophenylacetic acid.* The before mentioned cyanide was refluxed with 1 N sodium hydroxide overnight. After extraction with ether the mixture was acidified with 2 N hydrochloric acid. The dichlorophenylacetic acid was obtained as a colourless solid (4.0 g; 60 %) which was repeatedly crystallised from aqueous ethanol. Melting point 158–159°. (Found: Equiv. wt. 205.4; Cl 34.41 %. Calc. for  $\text{C}_8\text{H}_6\text{O}_2\text{Cl}_2$ : Equiv. wt. 205.0; Cl 34.59 %).

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## The Structure of Tellurium Dibenzenethiosulphonate

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This compound<sup>1</sup>,  $\text{Te}(\text{S}_2\text{O}_2\text{C}_6\text{H}_5)_2$ , is an analogue of tellurium dimethanethiosulphonate,  $\text{Te}(\text{S}_2\text{O}_2\text{CH}_3)_2$ , and of salts of telluropentathionic acid,  $\text{Te}(\text{S}_2\text{O}_5\text{OH})_2$ . The crystal structure has been worked out and found to be closely analogous to that of

tellurium dimethanethiosulphonate<sup>2</sup> and of ammonium telluropentathionate<sup>3</sup>.

The crystals of tellurium dibenzenethiosulphonate are orthorhombic bipyramidal, with the axial lengths<sup>2</sup>:  $a = 14.46 \text{ \AA}$ ,  $b = 11.18 \text{ \AA}$ ,  $c = 10.48 \text{ \AA}$ . There are four molecules per unit cell, and the space group is  $D_{2h}^{14}$ - $Pbcn$ . The tellurium atom is located on a twofold axis of symmetry.

The intensities of the  $h0l$  and  $hk0$  reflections were estimated visually from zero layer Weissenberg photographs taken with  $\text{CuK}$  radiation, and the structure was solved through Patterson and Fourier projections along the  $b$  and  $c$  axes. The  $h0l$  Fourier map, with lines drawn to show the outline of a molecule, is reproduced in Fig. 1.

The coordinates of the atoms of the asymmetric unit, in fractions of corresponding cell edges and referring to a centre of symmetry as an origin, are as follows.

With the atomic scattering curves of the *International Tables*, and a temperature factor of  $B = 2.4 \text{ \AA}^2$ , these coordinates give reliability factors of  $R = 0.16$  and  $0.17$ , respectively, for the  $h0l$  and  $hk0$  reflections.

	$x$	$y$	$z$
Te	0	0.043	0.250
S <sub>2</sub>	-0.068	0.186	0.395
S <sub>1</sub>	0.041	0.233	0.514
O <sub>1</sub>	-0.006	0.316	0.598
O <sub>2</sub>	0.083	0.139	0.566
C <sub>1</sub>	0.117	0.312	0.413
C <sub>2</sub>	0.207	0.271	0.396
C <sub>3</sub>	0.268	0.316	0.316
C <sub>4</sub>	0.238	0.435	0.253
C <sub>5</sub>	0.148	0.476	0.270
C <sub>6</sub>	0.087	0.414	0.349

The S-S-Te-S-S chain is unbranched and has a *trans*<sup>4</sup> configuration. The bond lengths and angles are,  $\text{Te}-\text{S}_2 = 2.41 \pm 0.03 \text{ \AA}$ ,  $\text{S}_2-\text{S}_1 = 2.08 \pm 0.03 \text{ \AA}$ ,  $\angle \text{S}_2\text{TeS}_1' = 97 \pm 2^\circ$ ,  $\angle \text{TeS}_2\text{S}_1 = 104 \pm 2^\circ$ , and  $\text{S}_1\text{S}_2\text{Te}/\text{S}_2\text{TeS}_1'$  (dihedral angle) =  $79^\circ$ . These values are in fair agreement with earlier data<sup>2,3</sup> for such chains. The benzenesulphonyl groups have, likewise, normal dimensions.

Details of the work will be published later.

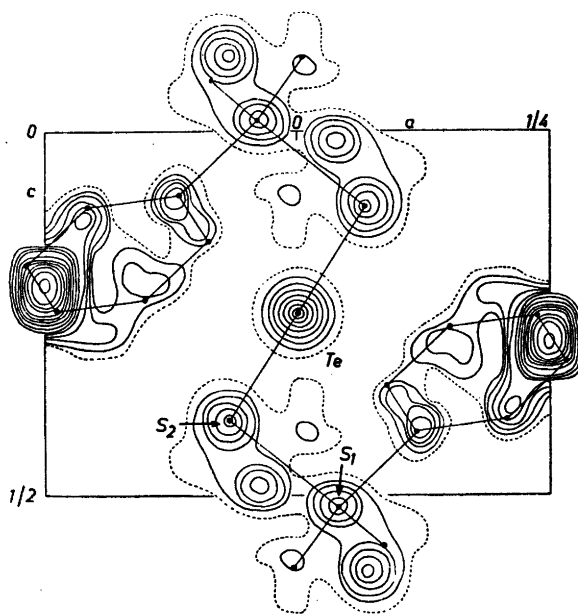


Fig. 1. Electron density projection of  $\text{Te}(\text{S}_2\text{O}_2\text{C}_6\text{H}_5)_2$  along the  $b$  axis. Contour lines are at intervals of  $1 e \cdot \text{\AA}^{-2}$  for carbon atoms,  $5 e \cdot \text{\AA}^{-2}$  for sulphur and oxygen atoms, and  $15 e \cdot \text{\AA}^{-2}$  for the tellurium atom, starting with 6, 10, and  $15 e \cdot \text{\AA}^{-2}$ , respectively. The 5-electron line is dashed.

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## The Structure of Tellurium Di-*p*-toluenethiosulphonate

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In a preceding note<sup>1</sup>, the crystal structure of tellurium dibenzenethiosulphonate,  $\text{Te}(\text{S}_2\text{O}_2\text{C}_6\text{H}_5)_2$ , was described. The crystals are orthorhombic, with a four-molecule unit cell based on the space group  $D_{2h}^{14}-Pbcn$ , and  $a = 14.46 \text{ \AA}$ ,  $b = 11.18 \text{ \AA}$ ,  $c = 10.48 \text{ \AA}$ . A twofold axis of symmetry, parallel to the  $b$  axis, passes through the tellurium atom at  $x = 0$ ,  $z = \frac{1}{2}$ . In the projection along the  $c$  axis, of symmetry  $cnm$ , the tellurium atom lies in an apparent mirror plane.

The six analogous compounds, sulphur<sup>2</sup> and selenium<sup>3</sup> dibenzenethiosulphonate, and sulphur<sup>3</sup>, selenium and tellurium di-*p*-toluenethiosulphonate and triselenium di-*p*-toluenesulphinat<sup>3</sup>, crystallize in the tetragonal space group,  $D_4^4-P4_12_12$  (or the enantiomorph) with four molecules per unit cell. A twofold axis of molecular symmetry is required, as in orthorhombic tellurium dibenzenethiosulphonate. The crystal structure of tetragonal tellurium di-*p*-toluenethiosulphonate,  $\text{Te}(\text{S}_2\text{O}_2\text{C}_6\text{H}_4\text{CH}_3)_2$ , has been solved by means of reference to the known structure of the benzene analogue, as follows.

In the centered setting,  $C4_122_1$ , the twofold rotation axes of the tetragonal unit cell of tellurium di-*p*-toluenethiosulphonate become parallel to the  $a$  and  $b$  axes. The axial lengths then are:  $a = 10.93 \text{ \AA}$ ,  $c = 29.88 \text{ \AA}$ . These axes, and the centered setting instead of the primitive one, are referred to in the following. It is seen that the tetragonal  $a$  axis is of about the same length as the  $b$  and  $c$  axes of orthorhombic tellurium dibenzenethiosulphonate, *viz.*,  $11.18 \text{ \AA}$  and  $10.48 \text{ \AA}$ , respectively, while

the tetragonal  $c$  axis is a little longer than twice the orthorhombic  $a$  axis,  $14.46 \text{ \AA}$ .

In the projection of  $C4_122_1$  along the  $b$  axis, the symmetry corresponds to that of the plane group,  $pgm$ , twice along the  $a$  axis. Considering the projected area,  $a/2 \times c$ , the eight general positions may be described as made up of two fourfold sets, both of symmetry  $pgm$ , with the coordinates,  $x, z$  and  $y, \frac{1}{2} + z$ , respectively, for the first of the four positions of each set. The corresponding coordinates of the special positions on the twofold rotation axes are,  $0,0$  and  $y, \frac{1}{2}$ . A comparison with the orthorhombic space group,  $Pbcn$ , shows that a molecule, belonging to the set,  $x, z$ , in the  $b$  axis projection of  $C4_122_1$ , corresponds to a molecule in the  $b$  axis projection of  $Pbcn$ , and one belonging to the set,  $y, \frac{1}{2} + z$ , corresponds to one in the  $c$  axis projection of  $Pbcn$ . The similarities of tetragonal and orthorhombic cell dimensions made it likely that the projected shapes of thus corresponding molecules were the same, or nearly the same, in the two space groups.

The intensities of the  $h0l$  reflections of tellurium di-*p*-toluenethiosulphonate were estimated visually from Weissenberg photographs taken with  $\text{CuK}$  radiation. A Patterson synthesis based on these data gave the  $y$  coordinate of tellurium, and a Fourier synthesis was performed, using the strongest reflections and signs obtained from the tellurium contributions alone. Peaks appeared in the Fourier map at or close to the expected sulphur positions, but the oxygen and carbon positions were uncertain, also after a second Fourier synthesis based on signs from the tellurium and the sulphur contributions. Using the tellurium positions as reference points, and corresponding axes of the two crystals coinciding or parallel, the sulphur, oxygen and carbon atoms of the asymmetric units of the two fourfold sets discussed above were then placed exactly as found in the  $b$  and  $c$  axis projections, respectively, of orthorhombic tellurium dibenzenethiosulphonate, with *p*-methyl carbons added. This was done by placing the tetragonal Fourier map, drawn on transparent paper, on the orthorhombic maps, of the same scale, and marking the positions. The subsequent structure factor calculation, with appropriate temperature and scale factors, lead to a reliability factor of  $R = 0.33$ . The most notable effect of the inclusion of the oxygen and carbon contributions was to change the sign of the next to strongest