

Borsook *et al.*⁴ reported that they were unable to separate α -aminoadipic acid from glutamic acid by paper chromatography and had to resort to the more cumbersome chromatography on starch when investigating the natural occurrence of this compound.

Due to the possible practical importance to workers in this field we wish to report an observation made in connection with work on lysine requiring mutants of *Ophiostoma*⁶, some of which has been found to grow with α -aminoadipic acid⁷. We have found that this amino acid can be clearly separated from glutamic acid by chromatography on paper with an acid medium similar to that described by Edman⁸.

On Munktells filter paper no. 0B⁹ with a medium consisting of 45 % *n*-butyric, 45 % isovaleric and 10 % water by volume, a mixture of aspartic, glutamic and α -amino adipic acid was separated after 12 hours in well developed spots with R^F values of respectively 0.18, 0.26 and 0.33, the descending solvent front having moved 41 cm.

In more complex amino acid mixtures two dimensional developments with this medium in combination with phenol, butanol or amyl alcohol-pyridine make identification possible.

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3. Dubnoff, J. W., and Borsook, H. *J. Biol. Chem.* **173** (1948) 425.
4. Borsook, H., Deasy, C. L., Haagen-Smit, A. J., Keighley, G., and Lowy, P. H. *J. Biol. Chem.* **173** (1948) 423, **176** (1948) 1383, 1395.
5. Mitchell, H. K., and Houlahan, M. B. *J. Biol. Chem.* **174** (1948) 883.
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8. Edman, P. *Arkiv Kemi, Mineral., Geol.* **A 22** (1945) no. 3.
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Received March 4, 1949.

The Crystal Structure of Tetrachlorocyclohexane of m.p. 174° C

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The structure formula of the compound $C_6H_8Cl_4$ obtained by direct chlorination of cyclohexane¹ and melting at 174° C has not been determined. On treating cyclohexane or chlorocyclohexane with chlorine at room temperature in artificial light and separating the deposited crystals from the liquid, this substance is easily obtained. We also prepared it from 1,2-dichlorocyclohexane using the same procedure, a fact which seems to indicate that at least two of the chlorine atoms occupy 1,2-positions. An electron diffraction investigation of the vapour carried out in our laboratory some time ago led to the assumption that the molecule has the configuration 1 ϵ , 2 ϵ , 4 κ , 5 κ . A measurement of the dipole moment² supported this view, indicating that the tetrachloride has a configuration corresponding to that of the tetrabromocyclohexane of m.p. 185° earlier investigated³.

In order to check the result of the electron diffraction work a complete X-ray crystallographic analysis has been carried out, and an extract of the results will be given here. We intend to bring an account of both the electron diffraction investigation and the crystal analysis with full details shortly in order to demonstrate the close agreement between the results obtained in these two independent investigations.

The crystals of tetrachlorocyclohexane belong to the orthorhombic sphenoidal class. Laue and Weissenberg photographs show that the *c*-axis has pseudo-tetragonal symmetry. The lattice constants are:

$$a = 7.60 \text{ \AA}, \quad b = 7.54 \text{ \AA}, \quad c = 7.72 \text{ \AA}$$

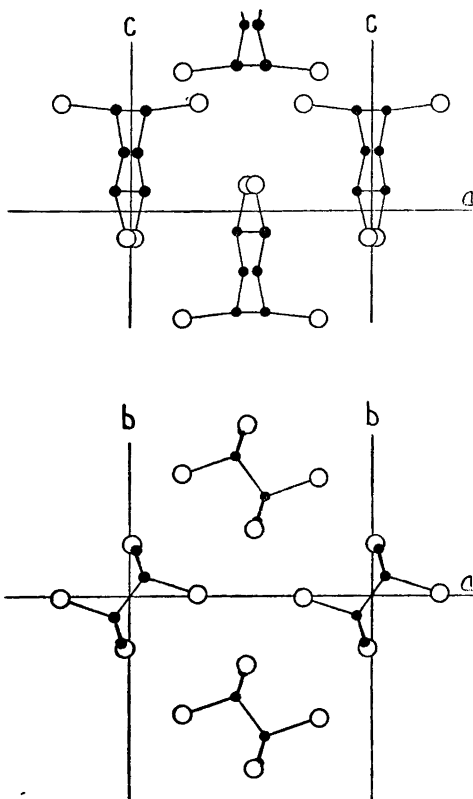


Fig. 1. Projection showing the position of the molecules in the unit cell. The upper part gives the projection in the (010)-plane, the lower part in the (001)-plane. Open circles represent chlorine atoms, filled circles carbon atoms. C—C-distance 1.54 Å, C—Cl distance 1.79 Å.

The unit cell contains *two* molecules. From the fact that the space group is $D_2^3-P2_12_12$ it follows that the molecules are crystallographically equivalent and possess a twofold axis of symmetry running parallel to the *c*-axis. This axis must of course bisect the distance between say the carbon atoms 1 and 2 and the carbon atoms 4 and 5.

Intensity data obtained from Weissenberg photographs by means of the multiple film method served as a basis for the

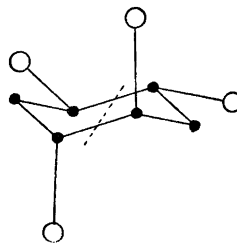


Fig. 2. Shape of the tetrachlorocyclohexane molecule.

two-dimensional Fourier analyses, of which one gave the projection of the electron density in the (001)-plane, a second in the (010) plane. In order to find the correct sign of the structure factors the 1ε , 2ε , 4κ , 5κ structure was assumed to be correct. The Fourier maps, showing well defined maxima of the expected heights, fully justified this procedure. The parameters of the chlorine atoms could be determined with a considerable degree of accuracy:

$$\begin{aligned} x_1 &= 0.282, & y_1 &= -0.008, \\ z_1 &= 0.437, & x_2 &= 0.015, \\ y_2 &= -0.215, & z_2 &= -0.105. \end{aligned}$$

The peaks due to the carbon atoms are less marked and partly overlapped by those of the chlorine atoms, but approximate parameters may still be given:

$$\begin{aligned} x_3 &= 0.062, & y_3 &= -0.081, & z_3 &= 0.408, \\ x_4 &= 0.062, & y_4 &= -0.081, & z_4 &= 0.082, \\ x_5 &= 0.028, & y_5 &= -0.194, & z_5 &= 0.245. \end{aligned}$$

Fig. 1 gives a projection of the structure in the (010)-plane (upper half) and in the (001)-plane (lower half). The form of the molecule is shown in Fig. 2. It is easily seen that the configuration is really 1ε , 2ε , 4κ , 5κ , but that the valency angles are not strictly tetrahedral. This deviation from an ideal structure is most strikingly demonstrated by the fact that the