

and valency angles seem, however, to give best agreement with the  $\sigma(r)$ -curve:

$C_1C_2$	1.35 Å
$C_2O$	1.42 Å
$\angle C_2OC_3$	107°
$\angle C_1C_2O$	123°

The C—C and C—O-distances occurring in a planar molecule with these parameters are indicated below the  $\sigma(r)$ -curve by a line diagram. (Full-drawn lines). The maximum IV does not fit with the  $C_1$ — $C_4$ -distance. If we give up the claim of coplanarity, however, and for instance rotate the vinyl groups about the C—O-bonds an angle of 20° in such a way that the  $C_1$ - and  $C_4$ -atoms move in the same direction, a relatively good agreement may be obtained. (Dotted lines in the line diagram.) Because of the uncertainty in the determination of the bond distances and valency angles an accurate determination of the magnitude and form of the deviation from coplanarity is impossible. By varying the distances and angles within reasonable limits we have, however, not been able to find a planar model which can be brought in accordance with the  $\sigma(r)$ -curve. We feel therefore justified in concluding that the deviation from coplanarity is real and that this deviation must be ascribed to the short  $H_2$ — $H_3$ -distance in the undistorted planar model.

In this connection another question related to our problem may be mentioned. Wheland<sup>3</sup> has drawn attention to the surprising fact that the resonance energy of ethyl vinyl ether seems to be slightly *greater* than that of divinyl ether; it would have been expected to be somewhat *smaller* since only a single unstable structure takes part in the resonance in the ethyl vinyl ether molecule, while *two* such structures take part in the resonance of the divinyl ether molecule. This fact might be brought in relation to the unfavourable  $H_2$ — $H_3$ -distance in the planar

## On the Identification of $\alpha$ -Amino adipic Acid by Paper Chromatography

SUNE BERGSTRÖM and KARIN PÅABO

Department of Physiological Chemistry,  
University of Lund, Lund, Sweden

Recent work has shown that  $\alpha$ -amino-adipic acid is an important metabolite. It has been isolated from *Cholera vibrio*<sup>1</sup> and the acid has been found active in transamination<sup>2</sup> in arginine formation<sup>3</sup> and also shown to be an intermediate in the lysine metabolism in mammals<sup>4</sup> and in *Neurospora*<sup>5</sup>.

divinyl ether molecule model. If our assumption of a deviation from coplanarity is correct, the  $\pi$ -orbitals will overlap less completely causing a reduction of the resonance energy. But even if the molecule was assumed to be strictly planar the unfavourable  $H_2$ — $H_3$ -distance would have decreased the stability of the divinyl ether molecule. In the case of the ethyl vinyl ether, on the other hand, the planar configuration is stabilized by the favourable  $H_2$ — $H_3$ -distances. In an ethyl vinyl ether molecule with a planar carbon-oxygen skeleton the  $H_2$ — $H_3$ -distances are approximately 2.3 Å. If any of the carbon atoms are brought out of the plane, one of the  $H_2$ — $H_3$ -distances will decrease and might therefore oppose the deviation from coplanarity.

We wish to express our gratitude to fil.mag. Inga Fischer, University of Stockholm, who has suggested this investigation and placed a sample of divinyl ether at our disposal. We also wish to thank Prof. O. Hassel for having read the manuscript.

1. Ehrenberg, L., and Fischer, I. *Acta Chem. Scand.* **2** (1948) 657.
2. Fischer, I., and Ehrenberg, L. *Acta Chem. Scand.* **2** (1948) 669.
3. Wheland, G. W. *The theory of resonance* (1947) p. 61.

Received March 7, 1949.

Borsook *et al.*<sup>4</sup> reported that they were unable to separate  $\alpha$ -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*<sup>6</sup>, some of which has been found to grow with  $\alpha$ -aminoadipic acid<sup>7</sup>. 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 Edman<sup>8</sup>.

On Munktells filter paper no. 0B<sup>9</sup> with a medium consisting of 45 % *n*-butyric, 45 % isovaleric and 10 % water by volume, a mixture of aspartic, glutamic and  $\alpha$ -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.

1. Blass, J., and Macheboeuf, M. *Helv. Chim. Acta* **29** (1946) 1315.
2. Braunstein, A. E. *Advances in Protein Chemistry* **3** (1947) 40.
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.
6. Fries, N. *Nature* **159** (1947) 199.
7. Bergström, S. Unpublished results.
8. Edman, P. *Arkiv Kemi, Mineral., Geol.* **A 22** (1945) no. 3.
9. Supplied by J. A. Munktells Pappersfabriksaktiebolag, Grycksbo, Sweden. Dimensions: 50 × 50 cm.

Received March 4, 1949.

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

O. HASSEL and E. WANG LUND

*Universitetets Kjemiske Institutt,  
Blindern—Oslo, Norway*

The structure formula of the compound  $C_6H_8Cl_4$  obtained by direct chlorination of cyclohexane<sup>1</sup> 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  $\epsilon$ , 2  $\epsilon$ , 4  $\kappa$ , 5  $\kappa$ . A measurement of the dipole moment<sup>2</sup> supported this view, indicating that the tetrachloride has a configuration corresponding to that of the tetrabromocyclohexane of m.p. 185° earlier investigated<sup>3</sup>.

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:

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