

0.1196 g: 11.98 ml 0.1060*N* NaOH. —
0.4092 g neutralised with NaOH and
made up to 5.00 ml with water: $\alpha_D^{25} = +$
0.425°.

Equiv. wt. Calc. 94.1 Found 94.2
 $[\alpha]_D^{25} = + 5.2^\circ$; $[M]_D^{25} = + 9.8^\circ$.

On comparison with the values given by Von Braun and Werner¹, this preparation seems to contain about 90 % of (+)-acid and 10 % of (—)-acid. As the rotatory power is very low and the data of these authors refer to much more concentrated solutions, this estimation is very approximate. It can however be stated that an optically active carboxylic acid can in this way be converted to a higher homologue without material loss of activity. As the asymmetric carbon atom is not involved in the reactions, (+)-*iso*-propylsuccinic acid and (+)- β -*iso*-propyladipic acid must be sterically related.

The latter acid may be obtained from (+)-limonene¹, and this terpene is thus sterically connected to the alkylsuccinic acids. On the other hand, (+)-limonene is sterically related to (+)-fenchone and (+)-camphor^{7, 8}, which have previously been connected to the alkylsuccinic acids by aid of α -*iso*-propylglutaric acid⁹. These earlier results are corroborated by the present investigation. A further discussion of these matters and a more detailed account of the experiments will be published later.

The author is indebted to Mr. S. Wideqvist for a combustion analysis.

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Electron Diffraction of Cyclooctatetraene Vapour

O. BASTIANSEN AND O. HASSEL

*Universitetets Kjemiske Institutt,
Blindern—Oslo, Norway*

The most direct way for determining the molecular structure of cyclooctatetraene would be the use of interferometric methods based either on X-rays or electrons. Reports on interferometric investigations were published in 1947 by the present authors¹ and by Kaufman, Fankuchen and Mark². The conclusions, however, to which the present authors were led using the electron diffraction sector method were quite different from those reached by Kaufman *et al.* using X-ray crystallographic methods.

In order to check our results two new analyses were carried out, the first with the same material used in our previous investigation (a sample prepared by prof. A. Langseth), the second using new material obtained from The British Oxygen Company. The computation work associated with the second of these new analyses was carried out by a new member of the staff who had no knowledge about the results of the two first analyses. In Fig. 1 the $\frac{\sigma(r)}{r}$ -curve obtained in the very first analysis (A) is compared with those of the two new analyses (B and C). The C-C-C-angle is determined by the ratio of the *r*-values of the two first high maxima of the

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Received February 23, 1949.

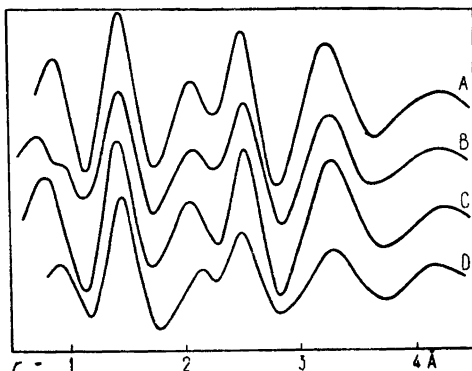


Fig. 1. $\frac{\sigma(r)}{r}$ -curves of cyclooctatetraene A, B, C experimental curves, D theoretical curve based on the «crown» model.

$\frac{\sigma(r)}{r}$ -curve. The angles calculated from the three individual curves are A) 120° , B) 122° , C) 122.5° .

We feel quite safe when drawing the conclusion that the C-C-C-angle is $121.5^\circ \pm 2^\circ$. The r -value of the first high maximum was found to be 1.42_5 \AA , on making exposures of cyclooctatetraene and benzene during the same run and assuming the C-C bond length in benzene to be 1.40 \AA — a value obtained in our earlier work.

We are unable, however, to decide from our investigations whether all the C-C bond distances are equal or if two alternate C-C distances occur, the mean value being 1.42_5 \AA . In the latter case we regard it very improbable, however, that distances corresponding to normal single and double bonds are present as postulated by Kaufman *et al.*

Sets of theoretical $\frac{\sigma(r)}{r}$ -curves have been computed based both on a planar arrangement of the carbon atoms, and on the nonplanar «tub» and «crown» forms of the

molecule. The first alternative is excluded by the fact that the C-C-C-angle is about 122° . The corresponding theoretical curves are also incompatible with such an assumption. A decision between the two nonplanar arrangement just mentioned could in fact be made by comparing the curves, the «crown» model only being able to explain the details of the experimental curve. The theoretical curve D is computed on the basis of the «crown» model.

The $\frac{\sigma(r)}{r}$ -curves obtained using material of different origin are in excellent agreement, a fact which seems to exclude the possibility that the materials are chemically different.

A determination of the positions of the hydrogen atoms will be very difficult. We may mention, however, that there are signs indicating a C-H bond distance smaller than that observed in benzene.

Contrary to the opinion expressed by Kaufman *et al.* we think it proved by thermochemical experiments that the resonance energy of the cyclooctatetraene molecule must be considerable. It seems obvious also, that the structure derived from our electron diffraction work is in good agreement with conclusions drawn from Raman spectroscopical observations³.

We wish to express our gratitude towards The British Oxygen Company for having placed at our disposal quantities of very pure cyclooctatetraene.

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Received April 1, 1949.