

The spectral range within which the boron-nitrogen frequency was found in the present investigation is in good agreement with the frequencies available for other dative bonds to boron in complexes of borane with ligands such as carbon monoxide, phosphorus trifluoride and dimethyl ether.

A more complete presentation of the results will be published elsewhere. This research was supported by the Aeronautical Research Laboratory, Wright Air Development Center, Air Research and Development Command, U.S. Air Force, Wright-Patterson Air Force Base, Ohio.

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<sup>4</sup>Rice, B., Galiano, R. J., and Lehmann, W. J., *J. Phys. Chem.*, **61**, 1222 (1957).

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### Molecular Structure of cyclo-Octatetraene

AN investigation of the nuclear magnetic resonance absorption spectrum of solid *cyclo*-octatetraene has been made with the object of obtaining information about its molecular structure. Previous determinations of the molecular structure of *cyclo*-octatetraene have used the techniques of infra-red and Raman spectroscopy, and of electron diffraction. The results have been explained in terms of three different molecular models: (1) the 'crown' ( $D_4$ ) structure with alternate double and single bonds<sup>1</sup>; (2) the 'crown' ( $D_{4d}$ ) structure with equivalent bonds<sup>2</sup>; (3) the 'tub' ( $D_{2d}$ ) structure with double and single bonds<sup>3</sup>. Subsequent investigations have supported one or other of these models, but with no real agreement as to which is the most likely structure. Theoretical predictions of the vibrational spectra and diffraction patterns of these three molecular configurations show little difference.

In certain cases, information obtained from a study of the nuclear magnetic resonance absorption spectrum can give valuable information distinguishing between various proposed molecular models. Accordingly the second moment of the proton resonance absorption line was measured at a temperature close to the melting point. The mean value obtained was  $7.9 \pm 0.2$  gauss<sup>2</sup>. This has to be compared with the theoretical values calculated for each of the three different configurations. The expected second moment was also calculated for the simple planar model with equivalent bonds, although the above results showed that this was a very unlikely possibility. The results are shown in Table 1.

The C—C and C=C bonds were taken as 1.54 Å. and 1.34 Å. respectively. The equivalent C—C bond

was taken as 1.42 Å., being 2 per cent greater than the corresponding bond-length in benzene, as suggested by Bastiansen *et al.*<sup>2</sup>. The  $D_4$  and  $D_{2d}$  structures were assumed to have angles of 125° between the carbon bonds, the  $D_{4d}$  structure 120° between these bonds and the plane molecule, being in the form of a regular octagon, angles of 135° between the carbon bonds. The C—H bond-length was taken as 1.07 Å. for the  $D_4$  and  $D_{2d}$  structures and as 1.08 Å. for the  $D_{4d}$  and planar structures. The intermolecular contribution to the second moment (due to interactions between protons in different molecules) was calculated on the basis of the orthorhombic crystal structure found by Kaufman, Fankuchen and Mark<sup>4</sup>, with reasonable assumptions regarding the orientation of the molecules in the cell.

Comparison of the experimental and theoretical values for the second moment indicates that the 'tub'  $D_{2d}$  structure is the most probable one for *cyclo*-octatetraene. The  $D_{4d}$  structure gives a second moment which is more than 1 gauss<sup>2</sup> too large, but it must be mentioned that the intermolecular contribution to this second moment amounts to 78 per cent of the total. This is the least accurate part of the calculation because of the assumption made regarding the orientation of the molecule in the unit cell. There is, therefore, some uncertainty in the value 7.05 gauss<sup>2</sup> for the intermolecular part of the second moment, but it is not thought to be so large as to reduce this value by as much as 1 gauss<sup>2</sup>.

The  $D_{2d}$  structure for *cyclo*-octatetraene has recently been confirmed using highly refined electron diffraction techniques by Bastiansen, Hedberg and Hedberg<sup>5</sup>, reported after the completion of the present work.

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### Use of Molecular Sieves in Gas Chromatography for the Determination of the Normal Paraffins in Petroleum Fractions

THE use of Linde molecular sieve 5A in conjunction with gas chromatography<sup>1-3</sup> offers considerable promise as a means of determining *n*-paraffins in hydrocarbon mixtures. Recently, Brenner and Coates<sup>4</sup> reported the application of this technique to the identification of *n*-paraffins in the C<sub>3</sub> to C<sub>11</sub> (gasoline) range. We have successfully used it in a higher molecular weight range, namely, for the determination of the *n*-paraffin content and distribution in kerosines and gas oils. Since this may also be of general interest our method will be outlined here.

The saturated fraction of the kerosine or gas oil was separated by liquid-solid chromatography and a portion run through a 2-metre gas chromatographic

Table 1

Molecular structure	Second moment of absorption lineshape in gauss <sup>2</sup>		
	Intramolecular	Intermolecular	Total
$D_4$	2.38	7.75	10.13
$D_{4d}$	1.99	7.05	9.04
$D_{2d}$	2.99	5.00	7.99
Plane	5.75	5.47	11.22