

Table 1. Carotene content of dried grass and seaweed meal, mg/kg.

Material	Method	
	Paper chromatography	Column chromatography
Dried grass	274, 277, 284, 284	250
»	248, 249, 252	240
Seaweed meal	101, 103	95

the development is interrupted, and the carotene zone is cut out immediately. The strip is then packed tightly in a glass tube (of approximately 8 mm inner diameter) which has a capillary end. The elution is carried out with 5 ml of acetone applied in several small portions and the eluate is made up to precisely 5 ml. The optical density is read in a spectrophotometer at 456 $m\mu$ and the carotene content calculated from the extinction coefficient of β -carotene ($E_{1\text{ cm}}^{1\%} = 2490$ in acetone). No interference with the absorption at 456 $m\mu$ was obtained with the eluates from a blank chromatogram.

The method described above has certain advantages over the conventional method, mainly because it avoids time consuming transference of the carotenes from acetone to petroleum ether. It is simple, requires very little equipment, and may, with a few alterations, be used for microdetermination of carotenes. From Table 1 it is seen that the accuracy of the method was satisfactory. The results obtained with the above procedure were comparable with, although somewhat higher than, those obtained by the conventional analysis³. This is believed to be caused by the higher over all recoveries obtained with the new method.

1. Jensen, A. and Liaaen Jensen, S. *Acta Chem. Scand. In press.*
2. Rutter, L. *Nature* **161** (1948) 435.
3. Haug, A. and Larsen, B. *Norwegian Institute of Seaweed Research, Report No. 16, 1957.*

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The Structure of the Benzene Diazonium Ion

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The structure of benzene diazonium chloride ($C_6H_5N_2Cl$) has been examined by X-ray methods. As the general features of the diazonium group is settled after one Fourier projection owing to the high crystal symmetry, a preliminary report of the first part of the structure determination is given.

Benzene diazonium chloride single crystals were grown from an ethanol solution by slow precipitation with ether at 0°C. The space group is $C222_1$ and the unit cell dimensions are $a = 15.16 \text{ \AA}$, $b = 4.95 \text{ \AA}$, $c = 9.05 \text{ \AA}$ as determined from oscillation and Weissenberg photographs taken at -90°C. With four molecules in the unit cell (calculated density 1.374) this space group demands the molecules to be in special positions, i.e. the molecules have twofold axes of symmetry parallel to the x or y axis.

Integrated Weissenberg photographs were taken at -90°C with rotation about the b axis and the intensities measured with a microphotometer. Approximate x and z parameters were determined from a Patterson projection and refined on the basis of successive Fourier syntheses. Finally, a few cycles of least squares refinements brought the reliability factor R down to 6.5 %. Absorption and secondary extinction corrections were not applied, and the thermal vibrations were regarded as isotropic.

A part of the Fourier projection (plane group pmg ; $c' = c$, $a' = a/2$) along the b axis is shown in Fig. 1. The chloride ion is situated at the origin and the twofold axis of the diazonium ion has the coordinates $(x, 1/4)$. The interatomic distances within the diazonium ion to be found in true lengths in this projection are as follows: $N_1-N_2 = 1.11 \text{ \AA}$, $N_1-C_1 = 1.42 \text{ \AA}$ and $C_1-C_4 = 2.70 \text{ \AA}$. Supposing the line through C_2-C_3 to be parallel to the x axis

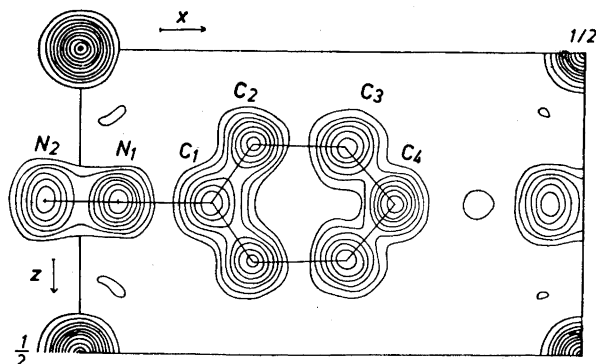


Fig. 1. Electron density projection along the b axis. Contour intervals: $2 \text{ e.}\text{\AA}^{-3}$ for chloride ions and $1 \text{ e.}\text{\AA}^{-3}$ for nitrogen and carbon atoms. The first contour is the 2-electron line.

the distance between these atoms is 1.40 \AA . The angle between the benzene plane and the plane of projection is about 45° .

The conclusion of this investigation is that solid benzene diazonium chloride has a typical ionic structure, the N-N distance corresponds to that of a normal triple bond, and the N_1-C_1 distance is shortened relative to a single C-N bond by an amount to be expected from similar systems where a multiple bond is conjugated to a benzene ring.

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Charge Transfer Compounds with a Halogen Atom linked to Carbon as the Electron Acceptor

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The crystal structures of several addition compounds have recently been determined in which halogen molecules act as electron acceptors. Structures of com-

pounds in which a halogen atom is linked to carbon in the acceptor molecule have not been investigated, however. Compounds like those formed by iodoform with three molecules of quinoline or S_8 therefore appeared to be of considerable interest. The crystal structure of the former has now been determined and the investigation of the sulphur compound has been started.

The unit cell of the quinoline compound is rhombohedral with the hexagonal axes $a = 22.40 \text{ \AA}$ and $c = 4.59 \text{ \AA}$. The rhombohedral unit cell contains one molecule of iodoform which is therefore situated on the trigonal axis (the space group is $R\bar{3}$) whereas the three quinoline molecules occupy general positions. It was not difficult to localize the former, but the lack of symmetry elements other than a threefold axis made the localization of the quinoline molecules more cumbersome.

Fig. 1 shows a difference Fourier map (projection along the trigonal axis) with subtraction of the contribution from iodine atoms. The position of one iodine atom is indicated by an asterisk and the three axes indicated are the projections of the rhombohedral axes into the (111) plane. Already from this projection a linear arrangement N-I-C, might be expected. This expectation is confirmed by the analysis. The N-I distance is found equal to 2.99 \AA , the C-I distance to 2.12 \AA .

These findings also confirm the suggestion that the iodine atoms act as electron acceptors towards the nitrogen atoms and