X-Ray Study of Isoprene, Butadiene and Chloroprene at Low Temperature

IN view of the considerable technical importance of isoprene, butadiene and chloroprene, as the hydrocarbons which on polymerization give natural and synthetic rubber, some interest is attached to the study of their structure. At normal temperatures they are in the liquid or gaseous state, and as it is possible to make exact X-ray structure measurements only in the solid state, the substances have been examined at liquid nitrogen and liquid oxygen temperatures, in a Debye-Scherrer camera, of radius 57 mm., employing a simple technique developed in the Davy Faraday Research Laboratory. Exposures of 30-90 minutes at 100 ma. were given.

Butadiene gives an extremely good X-ray diagram with about 35 measurable lines. Although the photograph provides adequate data as to crystal spacings, it has not been possible to identify the diagram with that of any of the crystal classes of high symmetry. It is probable that this substance is a crystal of a class which makes an analysis by the Debye-Scherrer method extremely difficult, if not impossible. The spacings calculated from the 10 strongest lines, using the Bragg relation, are given in the following table. The intensities are visual estimates.

Line	Butadiene Intensity	Cu K α radiation θ°	d in A.
1	8	9.49	4.67
2	6	10.49	4.23
3	7	10.86	4.08
4	10	12.52	3.55
5	10	12.84	3.47
6	8	13.52	3.30
7	8	13.94	3.19
8	6	19.82	2.27
9	5	20.30	2.22
10	6	21.42	2.08

With long exposures chloroprene never gave diagrams showing more than three lines corresponding to spacings of 3.28, 2.57 and 2.32 A., and two diffuse 'liquid' rings the maxima of which corresponded to spacings of 11.78 and 3.24 A.

Isoprene (Schuckert) never seems to crystallize at all sharply, but gives a glassy mass. This is in accordance with the facts quoted by Beilstein. It is not therefore surprising that only 'liquid' rings appear on the X-ray diagrams. (This substance was also studied using a 35 kw. tube with a rotating iron anticathode. Even a long exposure with an intense X-ray beam failed to bring out any lines.) A photometer trace of one of the films indicated intensity maxima corresponding to spacings of 8.6, 2.4 and 2.0 A.

It is not proposed to continue this work for the present. The purpose of this note is simply to put on record the preliminary results which have been obtained.

I am indebted to Dr. W. J. S. Naunton of Imperial Chemical Industries, Ltd., for supplying me with the chloroprene and butadiene, and to Sir William Bragg and the Managers of the Royal Institution for the facilities they have afforded me.

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A Plant Virus Preparation in a Fully Crystalline State

SPECIFIC proteins, believed to be the viruses themselves, have been isolated from plants infected separately with each of seven viruses of the tobacco mosaic and potato virus 'X' groups¹⁻⁴. Although these proteins have some properties in common with crystalline materials, and can be obtained in both solid and liquid anisotropic states, the solid preparations yet obtained lack the three dimensional regularity characteristic of true crystals. The particles of all these viruses are rod-shaped, and the connexion between this and the formation of fibres or pseudo-crystals has already been discussed^{2,5}.

PREPARATION OF BUSHY STUNT VIRUS CRYSTALLIZED FROM AMMONIUM SULPHATE SOLUTION. $\times 200$.

From tomato plants infected with the virus causing bushy stunt⁶, we have now isolated a specific protein which crystallizes in the form of rhombic dodecahedra. Solutions of this protein do not show anisotropy of flow, and the jelly formed by sedimenting the protein in a centrifugal field of 16,000 times gravity is not birefringent. As the crystals belong to the cubic system, they also are isotropic.

Infections (local lesions) of *Nicotiana glutinosa* can be obtained by rubbing the leaves with 1 c.c. of solutions containing 10⁻⁷ gm. of the protein, and 1 c.c. containing 10⁻⁸ gm. gives a visible precipitate with its antiserum. The activity of a preparation is unaffected by repeated recrystallizations or by sedimentation in a high-speed centrifuge.

After clarifying the infective sap by heating to 60° C. and centrifuging, the protein is isolated by repeated precipitations at room temperature with one quarter saturated ammonium sulphate solution. Impure preparations can also be fractionated by means of trypsin or a high-speed centrifuge. Crystallization is most readily effected by adding ammonium sulphate to a neutral solution at room temperature until the first sign of turbidity appears, immediately cooling to 0° C., and then slowly warming up to room temperature. The protein is much less soluble at room temperature than at 0° C., and as the clear fluid warms it becomes turbid and the protein crystallizes.

Analyses on dried preparations have given the following results: carbon 47, hydrogen 7.3, nitrogen 16, phosphorus 1.3 and carbohydrate 6 per cent.