LETTERS TO THE EDITORS

The Editors do not hold themselves responsible for opinions expressed by their correspondents. No notice is taken of anonymous communications.

A New Method for the Separation of the Amylose and Amylopectin Components of Starch

A NUMBER of methods are available for the separation of the unbranched (amylose) and branched (amylopectin) components of starch. Of these methods, that of T. Schoch[1] has been generally useful but presents certain disadvantages as a routine method inasmuch as it consists in a relatively high-temperature treatment, under pressure, of starch paste with butanol and *iso*amyl alcohol, and this treatment is known to cause some hydrolytic degradation of starch. The amylose component forms a complex with butanol which is insoluble in water, whereas amylopectin either does not form such a complex or, more probably, forms a complex which is soluble in water.

We have had in use for more than a year a method which avoids most of the difficulties of the Schoch method and attains a sharper separation of the starch components. The principle of the new method is the same, namely, the precipitation of amylose as a water-insoluble complex. The precipitant we use is, however, thymol, and the operation is carried out at the temperature of the laboratory.

The starch is dispersed in water by being creamed with cold water and then poured into boiling water with vigorous mechanical stirring. The stirring is continued at the boiling temperature for half an hour; sodium chloride is added to make a 0·1 per cent solution and thereafter the solution is cooled rapidly to room temperature. The optimum concentration of the starch is 1 per cent, but the following procedure has been successfully carried out with 3 per cent solutions. Powdered thymol is now stirred into the liquid in such amount as to ensure complete saturation (0·13 per cent). In a short time the thymol-amylose complex begins to separate and its precipitation is complete in a minimum period of forty-eight hours. The precipitate is removed at the centrifuge, is washed rapidly with (i) water saturated with thymol, (ii) absolute alcohol, and (iii) ether. These operations must be carried out as rapidly as possible and the product dried in a vacuum, otherwise a water-insoluble amylose is obtained.

The amylopectin component is obtained from the mother liquor when the latter is concentrated to a quarter of its bulk at 60° C. and treated with one volume of methylated spirits. The precipitate is washed and dried with alcohol and ether.

As a criterion of purity of the separated components, the blue value, that is, the intensity of the blue colour of the iodine complex prepared under the standard conditions prescribed by McCready and Hassid[2], was measured by means of a Spekker absorptiometer. The yield of amylose precipitated by thymol from potato starch varied between 20 and 22 per cent of the weight of starch, and this material showed blue values between 0·9 and 1·2. Occasionally a higher yield of amylose (up to 25 per cent) was obtained; but such an amylose preparation showed a blue value less than 0·9. The blue value is usually raised if the amylose is dissolved in water and re-precipitated with thymol. We have some reason to believe, however, that more than one amylose type may exist in potato. It is, for example, usually not possible, by reprecipitation, to convert an amylose with a blue value of 0·9 into one with a blue value of 1·2.

The iodine complex of thymol-amylopectin, which is purple in colour, shows a blue value varying between 0·18 and 0·25, most specimens showing a value of 0·22. The corresponding blue values for specimens of potato amylose and amylopectin separated by the butanol method were 1·05 and 0·18 respectively, and an average sample of whole potato starch showed a blue value of 0·41.

With a view to the investigation of the mechanism of amylose-precipitation, we have examined the efficacy as precipitants of a very wide range of organic compounds. Of these, cyclic alcohols (for example, cyclohexanol) and terpene derivatives (such as menthol, borneol or terpin hydrate) are sufficiently active to be worthy of further study. In particular, we have found a combination of thymol and cyclohexanol (or methyl cyclohexanol) to be especially useful in the preparation of pure amylopectin (that is, amylopectins with very low blue values). The amylose so separated contains a higher proportion of amylopectin than does that obtained when thymol is used alone, but amylopectin specimens have been prepared, in good yield (60–70 per cent of the starch), with blue values so low as 0·02. Details of this method of preparing amylopectin will be published later.

W. N. HAWORTH.
S. PEAT.
P. E. SAGROTT.

A. E. Hills Laboratories,
University, Birmingham.
Sept. 11.

[1] Schoch, *J. Amer. Chem. Soc.*, **64**, 2957 (1942).
[2] Hassid and McCready, *J. Amer. Chem. Soc.*, **65**, 1154 (1943).

Lattice Defects in Silver Chloride Crystals

SILVER chloride crystals, with dimensions up to about ½ cm., were obtained by recrystallization of material solidified from the molten state. Fig. 1 shows some recrystallized plates after etching with a dilute solution of sodium thiosulphate.

Laue photographs of these crystals show diffuse bands and spots, the bands being of similar character to those observed recently by Arlman and Kronig[1] (cf. also van Reyen[2]) with tin crystals (Fig. 2).

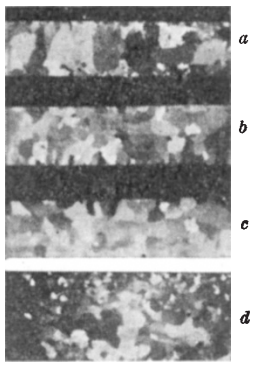

Fig. 1. *a*, *b*, *c*: Rolled and recrystallized plates of silver chloride with large crystals, after etching with sodium thiosulphate (1 per cent solution). Approx. nat. size.

d: Silver chloride plates with large crystals, 'etched' by irradiation with light from a mercury arc lamp. From a determination of the lattice orientation by means of X-rays, it follows that the 'etched' planes are parallel to the cube planes of the crystal lattice.