

The method is in constant use in this Laboratory. Although the procedure is long, it is actually less cumbersome to manipulate than the standard method and much more certain in its results.

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Note on the Esterase Character of Pectase (Pectin-demethoxylase)

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Von Euler¹ proposed the name pecto-lipase for the enzyme pectase, which splits off methoxy groups from pectin and forms a gel from it under suitable conditions. Though this name indicates the esterase character of pectase, it does not specify its action. Other compounds (acetic acid) might also be attached to the molecule by ester linkages, but it is likely that the splitting off of these groups is indifferent from the standpoint of gel formation.

It was of considerable interest to see whether other ester-splitting enzymes are able to produce gels from pectin solutions, a fact of importance in the classification of enzymes. In the course of some work started at this Station several years ago enzymes of different origin and type were tested for their action on pectin. Gels of stiff consistency were obtained by the use of some typical esterases. A preparation from castor beans was used in the first case. The beans were crushed, extracted with ether and dried. This crude preparation forms a gel from apple or lemon pectin in the presence of calcium ion at *PH* 5 and 8, but not at *PH* 3. A commercial lipase preparation (Difco, from pancreas) was also applied, with the same results.

The fact that from pectin gel could be formed by the use of typical ester-hydrolyzing enzymes verifies the earlier assumption about the esterase character of pectase. The more specific name pectin-demethoxylase is proposed for this enzyme.

(1) Von Euler, *Chemie der Enzyme*, Teil 2, Absch. 1, 457 (1928).

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Note on the Preparation of Dibenzoyl-*d*-tartaric Acid

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Considerable quantities of dibenzoyl-*d*-tartaric acid were needed for use in research on the cinchona alkaloids which is in progress in this Laboratory. The only detailed directions for the preparation of this substance which could be found in the literature were in a paper by Zetzsche and Hubacher.¹

(1) Zetzsche and Hubacher, *Helv. Chim. Acta*, 9, 291 (1926).

These authors heated one molecular proportion of tartaric acid with two and seven-tenths moles of benzoyl chloride in order to obtain the desired product. No statement of yield is to be found in any of the papers on the subject. When the directions of Zetzsche and Hubacher¹ were followed exactly, yields of 30 to 37% were obtained.

Since the reaction product of benzoyl chloride and tartaric acid under these conditions is a benzoylated anhydride and not a free acid, it is clear that a dehydration accompanies the benzoylation. It seemed reasonable therefore to suspect that at least three moles of benzoyl chloride (two for benzoylation and one for dehydration) should be used. With this thought in mind, the experiment was repeated according to the following modified directions and a yield close to quantitative was obtained.

One hundred fifty grams of tartaric acid (1 mole) and 450 g. of benzoyl chloride (3.2 moles) were heated in an Erlenmeyer flask slowly up to 150°. Reaction took place mainly between 135 and 140°. The total time of heating was three hours. The contents of the flask were removed and ground in a mortar. Benzoic acid and the excess of benzoyl chloride were removed by extracting with benzene in the following manner. Three hundred cc. of solvent was added, the mixture was warmed, with agitation, just short of the boiling point and then cooled to room temperature. The product was filtered and the extraction was repeated. The material was then washed with several portions of cold benzene. The yield of crude dibenzoyl-*d*-tartaric anhydride, dried at 100°, was 348 g., melting point 155 to 160°. A strictly pure product melting at 173° may be obtained by crystallizing from xylene.

The crude benzene extracted product was placed in a flask with 1500 cc. of water and the mixture was gently boiled for one-half hour, during which time dibenzoyl-*d*-tartaric acid monohydrate collected on the bottom of the flask as a heavy oil. When allowed to stand overnight, the oily product solidified in a crystalline cake. However, if a few crystals of acid from a previous run were added to the aqueous mixture and the mixture was shaken vigorously for a few minutes, the product solidified in small lumps. It was filtered, air-dried and ground. The yield was 359 g., or 93% of the theoretical amount. The substance melted at 84 to 85°, and was sufficiently pure for our purposes. A purer product, melting at 88 to 89°, $[\alpha]_D - 116.0^\circ$, may be obtained if desired by crystallizing from benzene, or more conveniently by washing with a little cold benzene.