Notes

Work is being continued on the reaction of diazo oxides with carbohydrates. The author would be glad to hear of the results of others, especially with the uncommon and not readily available sugars.²

(2) The writer is greatly indebted for a number of samples to: Professor C. S. Hudson of the U. S. Public Health Service; Mr. F. Bates of the U. S. Bureau of Standards; Professor W. C. Austin of Loyola Medical School; Professors H. T. Clarke and M. Heidelberger of College of Physicians and Surgeons, Columbia University.

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The Preparation of Racemic Tartaric Acid

BY ALAN NEWTON CAMPBELL, LOUIS SLOTIN AND STEWART A. JOHNSTON

In preparations of racemic acid by the standard method,¹ we have found that calcium sulfate is an inevitable impurity, despite frequent recrystallizations. This situation is due to the well-known tendency of calcium sulfate to form supersaturated solutions. In previous work² it was found that the racemization of active tartrate could be brought about by much lower concentrations of alkali than are used in the standard preparation. A method designed to yield a product free from calcium sulfate was worked out on this basis as follows: 360 g. of d-tartaric acid is dissolved in two liters of 4 N sodium hydroxide. This solution is gently boiled in a copper flask under reflux for a week. The solution is made distinctly acid with strong hydrochloric acid, and evaporated to a bulk of 500 cc. It is then filtered hot from precipitated sodium chloride and 200 cc. of concentrated hydrochloric acid added to the filtrate. More sodium chloride separates which is again filtered. A liter of 95% alcohol is then added and the precipitated sodium chloride removed. The alcohol is removed by distillation from the steam-bath and the residue allowed to evaporate in an open dish on the steam-bath until the liquid portion has fallen to 150 cc. The thin sirupy liquid obtained is inoculated with a few crystals of racemic acid and allowed to stand overnight. This treatment causes the liquid to set solid. It is macerated with 100 cc. of cold water and filtered. The crude racemic acid thus obtained is dissolved in hot water, boiled with animal charcoal, filtered and allowed to crystallize. The crystals are dried in an oven at 80° to get rid of traces of hydrogen chloride. Further recrystallization from water is necessary to obtain a perfectly pure product. The final product has a melting point, when dehydrated, of 203-204°, is quite inactive and free from chloride. The yield is 150 g. of crystallized product.

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⁽¹⁾ Cohen, "Practical Organic Chemistry," 1928 ed., p. 145: Holleman, Rec. trav. chim., 17, 66 (1898).

⁽²⁾ Campbell and Campbell, THIS JOURNAL, 54, 3834 (1932).

Notes

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.

DEPARTMENT OF CHEMISTRY UNIVERSITY OF MANITOBA WINNIPEG, CANADA Received February 3, 1933 Published June 6, 1933

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

By Z. I. KERTESZ

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 pancrease) was also applied, with the same results.

The fact that from pectin gel could be formed by the use of typical esterhydrolyzing 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). GENEVA, NEW YORK RECEIVED FEBRUARY 16, 1933 PUBLISHED JUNE 6, 1933

Note on the Preparation of Dibenzoyl-d-tartaric Acid

BY C. L. BUTLER AND LEONARD H. CRETCHER

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).