

with ether and the oil left after removing the ether was distilled under reduced pressure. There was obtained a 45.7% yield (based on the diazonium fluorosulfonate) of nearly colorless oil, distilling at 135–137° (5 mm.); d_{25}^{25} 1.2905; n_{25}^{25} 1.5508. When cooled to -78° the oil crystallized, m. p. 33–34.5° (uncor.). The compound had a slightly aromatic odor and was soluble in acetone, methanol, chloroform, benzene and hexane; it was insoluble in

water and ethylene glycol.

Anal. Calcd. for $C_{12}H_9SO_3F$: F, 7.53; hydrolysis value, 7.59 ml. of 0.5221 *N* NaOH. Found: F, 7.40; hydrolysis value, 7.27 ml. of 0.5221 *N* NaOH.³

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MARY W. RENOLL

RECEIVED MARCH 2, 1942

COMMUNICATIONS TO THE EDITOR

THE SYNTHESIS OF LACTOSE AND ITS EPIMER

Sir:

Referring to our articles which describe the syntheses of epi-cellobiose and cellobiose [THIS JOURNAL, **63**, 1724 (1941); **64**, 1289 (1942)], we now report that the combination of acetone-D-mannosan with acetobromo-D-galactose has been accomplished, and that by reactions which are entirely like those used in the indicated syntheses, we have produced, first, the epimer of lactose and, second, lactose itself. The yield of synthetic crystalline α -epi-lactose octaacetate by the indicated combination was 30% and the deacetylation of the octaacetate generated synthetic epi-lactose which is identical with that produced from lactose. Synthetic epi-lactose octaacetate was converted to acetobromo-epi-lactose, which was in turn reduced to the known lactal hexaacetate, from which β -lactose octaacetate was obtained in good yield by oxidation with perbenzoic acid, followed by acetylation. Deacetylation of this octaacetate generated lactose. Publication of the full details will follow. The syntheses of epi-lactose and lactose were announced at the Memphis meeting of the Society in the Division of Organic Chemistry, April 21, 1942.

DIVISION OF CHEMISTRY

NATIONAL INSTITUTE OF HEALTH
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W. T. HASKINS

RAYMOND M. HANN
C. S. HUDSON

RECEIVED MAY 14, 1942

POLAROGRAPHIC DETERMINATION OF CITRININ¹

Sir:

In view of the growing interest in anti-bacterial substances obtained from molds,² it seems desir-

(1) Taken from the thesis of H. W. Hirschy, submitted in partial fulfillment for a Master's Degree in Chemistry, June, 1941.

(2) (a) Abraham, Baker, Chain, Florey, Holiday and Robinsou, *Nature*, **149**, 356 (1942); (b) Dawson, Hobby, Meyer and Chaffee, *Jour. Clin. Invest.*, **20**, 433 (1941); (c) Oxford, Raistrick and Smith, *Chem. and Ind.*, **61**, 22 (1942); (d) Raistrick and Smith, *ibid.*, **60**, 828 (1941); (e) Waksman, *Bact. Rev.*, **5**, 253 (1941); (f) Wiesner, *Nature*, **149**, 356 (1942)

able to publish at this time the results of some preliminary physico-chemical studies on the metabolic product of *Penicillium citrinum*: namely, citrinin, which recently has been shown to inhibit the growth of *Staphylococcus aureus*.^{2d}

Citrinin, $C_{13}H_{14}O_5$, a yellow crystalline solid, m. p. 170–171° (with decomposition), was isolated and purified by the method of Hethering and Raistrick.³ It was obtained from the culture, *P. citrinum* #136-5730.6.⁴ The characteristic properties of our product were in good agreement with those reported previously.³

We have found that citrinin in concentrations of 0.001–0.003 *M* gives well-defined current-voltage curves at the dropping mercury cathode in acid, 75% ethanol solutions, buffered and unbuffered. The limiting current was found to be proportional to the concentration of citrinin. The half-wave potential in the buffered alcoholic solution (*pH* 2.05) is in the range of -0.80 to -0.82 volt applied *versus* the saturated calomel reference electrode. Citrinin is also reduced in 0.1 *N* KCl–75% ethanol solution. It is not reduced in acetate alcoholic buffer (*pH* 6.0) nor in phosphate alcoholic buffer (*pH* 7.4). The manual apparatus used in these studies was similar to that described previously.⁵ Attempts to use standard potentiometric procedures with $TiCl_3$ ⁶ as a reducing agent proved unsuccessful since stable potentials were never reached within a reasonable and workable length of time.

(3) Hethering and Raistrick, *Roy. Soc. Phil. Trans.*, **B220**, 269 (1931).

(4) Furnished through the courtesy of Dr. K. B. Raper, U. S. Department of Agriculture. The A. T. C. collection #6202 failed to give little more than traces of citrinin.

(5) Smith, Kolthoff, Wawzonek and Ruoff, THIS JOURNAL, **63**, 1018 (1941).

(6) (a) Kolthoff and Furman, "Potentiometric Titrations," John Wiley and Sons, Inc., New York, N. Y., 1931, p. 355; (b) Knecht and Hibbert, "Volumetric Analysis," Longmans, Green and Co. London, 1925, p. 53.