[Contribution from the Carbohydrate Laboratory, Bureau of Chemistry, United States Department of Agriculture.]

THE PREPARATION OF XYLOSE FROM CORN COBS.

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In a previous article[1] we published directions for preparing pure crystalline xylose from cottonseed hulls. As the yield was relatively large, namely, 8 to 12%, it seemed that these hulls were an ideal source for xylose. Later experiments have shown, however, that corn (*Zea mays*) cobs are even a better source. The yield is uniformly good, about 12%, the crude xylose is of high purity, and the solutions throughout the course of preparation are less colored than those from cottonseed hulls, even though the preliminary cleaning of the hulls with weak ammonia is omitted when corn cobs are used. It has been shown by Stone and Lotz[2] that crystalline xylose can be prepared from corn cobs, but their yield was less than one per cent. Their method involved the separation of xylan from the cobs and its subsequent hydrolysis to xylose.

The method that has given good results, in the preparation of many kilograms of xylose at different times in this laboratory, is the following:

Seven hundred and fifty g. of broken but not necessarily ground corn cobs are boiled 2 hours with 6 liters of 7% sulfuric acid under a reflux condenser. The insoluble residue is filtered off, and the absorbed liquid is pressed out of the cobs with a hand press. The filtrate is exactly neutralized with pure calcium hydroxide, care being taken that the reaction never becomes alkaline. The calcium sulfate is filtered off, washed with hot water and the washings combined with the filtrate. The filtrate is made just acid to litmus with phosphoric acid, and is decolorized with carbon or bone char, about 30 g. decolorizing carbon being required. The filtrate is boiled under reduced pressure to about 0.5 liter and to it two volumes of 95% ethyl alcohol are added to precipitate the remaining calcium sulfate. After filtration the solution is boiled under reduced pressure to a very thick sirup of 85–90% solids. Should it begin to darken while boiling it may be removed when at about 400 cc. volume and decolorized again. To the sirup sufficient 95% ethyl alcohol is added to cause the sirup to be nearly saturated with alcohol after the homogeneous mixture cools to room temperature. An excess of alcohol causes the separation of two liquid phases. This saturation requires approximately 50 cc. alcohol. During the progress of the crystallization at room temperature or lower more alcohol may be added to prevent the mass from becoming solid, but care should be taken that sirup is not precipitated. Crystallization is usually complete in from 2 to 3 hours, but the solution

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[1] This Journal, 39, 1038 (1917).

[2] *Am. Chem. J.*, 13, 348 (1891).

is ordinarily allowed to stand overnight at 0°. The crystals are filtered off, washed successively with 75%, 95%, and absolute alcohol and dried in a vacuum oven. The sugar is in many cases colorless and pure and the yield is between 10 and 12%, depending upon the cobs that are used. If required, the crude xylose may be recrystallized from water or aqueous alcohol.

In conclusion, we would add that xylose is one of the most easily prepared members of the sugar group when either cottonseed hulls or preferably corn cobs are used as its source. There should no longer be any difficulty in obtaining a supply of this pentose sugar for chemical, bacteriological, medical or other scientific uses.

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[CONTRIBUTION FROM THE SANDERS CHEMICAL LABORATORY OF VASSAR COLLEGE.]

ON THE DINITRO DERIVATIVES OF *p*-DICHLOROBENZENE: 2,5-DICHLORO-1,4-DINITROBENZENE.

BY EDITH H. NASON.

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p-Dichlorodinitrobenzene was first prepared by Jungfleisch[1] by the nitration of mononitro-*p*-dichlorobenzene. He obtained two isomers, α and β, the melting points of which he gave as 87° and 107°, respectively. Englehardt and Latschinoff[2] repeated the process and isolated two compounds which corresponded in appearance to the two isomers obtained by Jungfleisch, but which melted at 104° and 101°, respectively. Later investigators confirmed these melting points and fixed the constitution of the 104° compound as 2,5-dichloro-1,3-dinitrobenzene, that of the 101° being left undetermined. Rütgers[3] studied the reactions of *p*-dichlorodinitrobenzene, prepared by the nitration of *p*-dichlorobenzene. He refers to the β (101°) compound, but states that the amount formed in his preparation was so small as to render its separation unnecessary.

Despite the fact that no reference is made to it by any of these investigators, it seemed probable that the third theoretically possible *p*-dichlorodinitrobenzene was also formed in the nitration of *p*-dichlorobenzene, particularly in view of the work of Jackson, Calhane and Wheeler,[4] who succeeded in isolating the 3 corresponding *p*-dibromodinitro derivatives. Careful recrystallization of the crude product has shown that all 3 isomers are in fact produced in the nitration of *p*-dichlorobenzene, the third, hitherto unnoticed, being formed in considerably larger proportion than either of the other two.

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[1] *Jahresber.*, 1868, 345.

[2] *Z. Chem.*, 6, 234 (1870).

[3] *Inaug. Diss.*, Basle, 1893.

[4] *Am. Chem. J.*, 22, 449 (1899); 28, 451 (1902).