

aluminum and lithium. Typical results are: found: $\text{Al}_2(\text{SO}_4)_3$, 38.27, 38.36; Li_2SO_4 , 12.87, 12.96. Calcd. for $\text{LiAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$: $\text{Al}_2(\text{SO}_4)_3$, 38.68; Li_2SO_4 , 12.43.

The double salt is incongruently soluble at this temperature. This, and the limited region of existence, may explain the failure of many attempts during the last one hundred years to prepare this salt.

The authors are grateful to Professor John E. Ricci of New York University for helpful suggestions and to Dr. M. F. Reynolds of Queens College who assisted in obtaining the photograph shown in Fig. 1.

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RECEIVED SEPTEMBER 27, 1941

Preparation of Maltose Monohydrate by the Deacetylation of Maltose Octaacetate with Barium Methylate

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There has been some difficulty encountered in preparing pure maltose from the octaacetate following the procedure reported by Zemplén.¹ The use of the so-called barium methylate² as a deacetylating agent was found to work very well, however.

Preparation of Barium Methylate.—Slowly add 50 g. of barium monoxide to 150 cc. of absolute methyl alcohol. After the initial reaction has subsided, gently reflux for one hour, dilute to 300 cc., and filter. Standardize with approximately normal sulfuric acid using phenolphthalein indicator and store in refrigerator.

The Deacetylation of the Octaacetate³ of Maltose.—Fifty grams of the octaacetate is dissolved in 500 cc. of absolute methyl alcohol. The solution is cooled to about 10°, and 10 cc. of barium methylate solution added while the flask is well agitated. After an hour or more of intermittent shaking, the flask is cooled in an ice-bath and an exact equivalent of the standard sulfuric acid slowly added. The barium sulfate is allowed to settle overnight and the supernatant liquid decanted. To facilitate the removal of the almost colloidal barium sulfate, the suspension is heated in a water-bath for several minutes with 5 g. of activated carbon plus 5 g. of analytical grade filter-cel. This mixture is filtered until a clear solution is obtained. The water-white solution is concentrated over a steam-bath using a good vacuum. The drying is complete when the

(1) Zemplén, *Ber.*, 59, 1258 (1926); Zemplén, *ibid.*, 60, [2] 1555 (1927); Zemplén and Pacsu, *ibid.*, 62, 1613 (1929).

(2) Weltzien and Singer, *Ann.*, 443, 104 (1925); Isbell, *Bur. Standards J. Research*, 5, 1185 (1930).

(3) The yield of maltose octaacetate can be increased considerably by reclaiming the solid from the alcohol mother liquors and again treating with a portion of acetic anhydride and sodium acetate.

material in the flask is snow white and able to be powdered when prodded with a stirring rod.

Crystallization of Maltose Monohydrate.—The above powdered maltose is weighed quickly and 0.4 cc. of water added for each gram of dry maltose. This mixture is heated in hot water until a thick sirup is formed. Now 65 cc. of 95% ethyl alcohol is added, the solution is heated for several minutes with 2 g. of carbon and 2 g. of filter-cel, and then filtered. The residue may be washed with a small amount of hot 80% alcohol. The clear filtrate is allowed to cool and then seeded with a small crystal of maltose and allowed to crystallize at room temperature with the aid of mechanical stirring. The crystallization is usually complete in about three days, but in order to ensure maximum yield, the maltose should be allowed to stand in the refrigerator for an additional three or four days before filtering and washing with 95% alcohol. The crystals are dried in a vacuum oven at 45° for two days. The yield is 50–60% of the theoretical amount. The specific rotations of five different preparations made by the above procedure varied over the range $[\alpha]^{25}_D +129.5$ to 130.0° .

To compare this maltose with the best commercial preparation and maltose recrystallized from water-alcohol solutions, the reducing power was measured using the method of Blish and Sandstedt.⁴

NUMBER OF CC. OF 0.1 N POTASSIUM FERRICYANIDE REDUCED BY DIFFERENT PREPARATIONS OF MALTOSE

Maltose used, mg.	Sample of maltose			
	Best grade maltose obtained commercially	Good grade maltose recrystallized several times	Sample obtained from another laboratory—considered pure	Maltose prepared through the octaacetate
10.0	3.58	3.60	3.60	3.69
15.4	5.29	5.35	5.34	5.40
20.0	6.76	6.90	6.86	6.87
25.0	8.28	8.40	8.36	8.44

(After the method of Blish and Sandstedt, *Cereal Chem.*, 10, 189 (1933)).

It is apparent that the reducing values of the different preparations are very close to each other.

(4) Blish and Sandstedt, *Cereal Chem.*, 10, 189 (1933).

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RECEIVED OCTOBER 2, 1941

Invert Soaps of Naphthalene

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It was shown in the work by G. Domagk⁴ and R. Kuhn and co-workers⁵ that invert soaps pos-

(1) This communication was part of a paper presented before the Division of Medicinal Chemistry at the Atlantic City Meeting of the American Chemical Society; Abstracts of Papers, 102nd Meeting, American Chemical Society, Atlantic City, New Jersey, September 8–12, 1941, pp. K-8.

(2) Abstracted from the thesis presented by H. Weingarten to the Graduate School of New York University in partial fulfillment of the requirements for the degree of Master of Science, June, 1942.

(3) J. B. Niederl and co-workers, *THIS JOURNAL*, 63, 945, 1475, 1476, 2024 (1941).

(4) G. Domagk, *Dtsch. med. Wschr.*, I, 829 (1935).

(5) R. Kuhn and co-workers, *Ber.*, 73, 1080–1109 (1940).