aluminum and lithium. Typical results are: found: $Al_2(SO_4)_3$, 38.27, 38.36; Li_2SO_4, 12.87, 12.96. Calcd. for $LiAl(SO_4)_2 \cdot 12H_2O$: $Al_2(SO_4)_3$, 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.

DEPARTMENT OF CHEMISTRY
ST. JOHN'S UNIVERSITY
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Preparation of Maltose Monohydrate by the Deacetylation of Maltose Octaacetate with Barium Methylate

BY WILLIAM A. MITCHELL

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 methylate[2] 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 Octaacetate[3] 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 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 $[\alpha]^{26}D$ +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

	Sample of maltose			
Maltose used, mg.	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.

CENTRAL LABORATORIES
GENERAL FOODS CORPORATION
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Invert Soaps of Naphthalene

BY JOSEPH B. NIEDERL AND HERSH WEINGARTEN[1,2,3]

It was shown in the work by G. Domagk[4] and R. Kuhn and co-workers[5] that invert soaps pos-

(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.

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

(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.

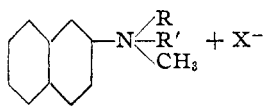
(3) J. B. Niederl and co-workers, THIS JOURNAL, **63**, 945, 1475, 1476, 2024 (1941).

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

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

sessing a chain of sixteen carbon atoms show the highest bactericidal activity. These investigators used benzyl or phenyl radicals in their compounds. It was now thought desirable to prepare similar types of compounds possessing a condensed ring system such as naphthalene, and then to study the bactericidal activity of such compounds.

Thus the following invert soaps were prepared: first the N,N-dimethyl-N-cetyl-β-naphthylammonium iodide (II), and then a few short-chain naphthalene invert soaps such as the N-methyl-N,N-di-*n*-butyl-N-β-naphthyl- (III) and N,N,N-trimethyl-N-β-naphthylammonium salts (IV). The structure of these salts is



	(II)	(III)	(IV)
X^-	= I	I	$CH_3OSO_2O^-$
R	= CH_3	C_4H_9	CH_3
R′	= $C_{16}H_{33}$	C_4H_9	CH_3

The salts are slightly soluble in water.

Phenol coefficient determination following the U. S. Bureau of Standards procedure on *Staphylococcus aureus* showed a phenol coefficient of not more than 0.2. This surprising result of complete inactivation indicates that the cetyl group and *per se* the length of the carbon chain alone are not responsible for high bactericidal action.

Experimental

N,N-Dimethyl-N-cetyl-N-β-naphthylammonium Iodide (II).[6,7]—Seven grams of β-naphthylamine was added to a solution of 15 g. of cetyl bromide in 15 cc. of ethyl alcohol. The mixture was refluxed on a steam-bath for fifteen hours. The precipitate, N-cetyl-N-β-naphthylammonium hydrobromide (Ia), was filtered and recrystallized from ethyl alcohol.

Eleven grams of (Ia) was heated to boiling with 50 cc. of 10% sodium hydroxide solution for two minutes. After cooling, the precipitate was filtered and dissolved in ether. The ether solution was filtered and the filtrate evaporated to dryness. The residue, the N-cetyl-β-naphthylamine (I), was recrystallized from ethyl alcohol.

To a solution of 8 g. of (I) in 240 cc. of ethyl alcohol were added 32 g. of methyl iodide and 8 g. of anhydrous sodium carbonate, and the mixture was refluxed for fifteen hours. After this time, the hot alcohol solution was filtered and the filtrate evaporated until a precipitate appeared. The precipitate (sodium iodide) was again filtered and the filtrate evaporated to dryness. The residue, the invert soap (II), was recrystallized from ethyl acetate.

N-Methyl-N,N-di-*n*-butyl-N-β-naphthylammonium Iodide (III).—Fourteen grams of β-naphthylamine, 20 g. of butyl bromide, and 10 g. of butyl alcohol were refluxed for twenty hours. After this time, the mixture was cooled and neutralized with 10% sodium hydroxide solution. The oily layer was then separated, dried with magnesium sulfate, and, after addition of 15 g. of butyl bromide, refluxed for thirty hours. The mixture was then cooled, neutralized with sodium hydroxide, and the oily layer dried with magnesium sulfate. The N,N-di-*n*-butyl-β-naphthylamine thus obtained was purified by distillation under reduced pressure.

Three grams of the above tertiary amine and 5 g. of methyl iodide, contained in a stoppered Erlenmeyer flask, were kept at room temperature for three days. The methiodide (III) thus obtained was filtered and washed with ether. The crude product was dissolved in ethyl alcohol and reprecipitated by ether.

N,N,N-Trimethyl-N-β-naphthylammonium Methosulfate (IV).—Four grams of β-naphthylamine and 12.5 g. of dimethyl sulfate were heated at 120° in an oil-bath for two hours. After this time, the excess dimethyl sulfate was distilled off under reduced pressure. Upon addition of 100 cc. of methyl alcohol to the oily residue, a precipitate was formed. This precipitate, the methosulfate (IV), was filtered and recrystallized from boiling water.

TABLE I

	Compounds	Formula	M. p. (uncor.), °C.	Analyses, % N Calcd.	Found
I	N-Cetyl-β-naphthylamine	$C_{26}H_{41}N$	64	3.81	3.75
	(a) Hydrobromide[6]	$C_{26}H_{42}NBr$	161	3.12	3.07
II	N,N-Dimethyl-N-cetyl-N-β-naphthylammonium iodide	$C_{28}H_{46}NI$	106	2.67	2.52
III	N-Methyl-N,N-di-*n*-butyl-N-β-naphthylammonium iodide	$C_{19}H_{28}NI$	157	3.52	3.19
IV	N,N,N-Trimethyl-N-β-naphthylammonium methosulfate	$C_{14}H_{19}NSO_4$	288	4.72	4.35

CONTRIBUTION FROM THE CHEMICAL LABORATORIES
OF NEW YORK UNIVERSITY
WASHINGTON SQUARE COLLEGE
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(6) H. Lettre and M. E. Fernholz, *Ber.*, **73**, 436 (1940).

(7) J. R. Stevens and R. H. Beutel, THIS JOURNAL, **63**, 308 (1941).

A New Hydrolysis Product Derived from Bovine Cerebral and Spinal Tissue

BY CARL NIEMANN

Acetylation of the mother liquor obtained by recrystallizing crude sphingosine sulfate (fraction C–S–H_1) from absolute ethanol[1] has led to the isolation of triacetylsphingosine and a hitherto undescribed compound.[2] This latter substance, after repeated recrystallization from methyl ethyl ketone, gives analytical values in good agreement with those demanded by the empirical formula $C_{36}H_{69}NO_4$. Subsequent investigation showed

(1) C. Niemann, THIS JOURNAL, **63**, 1763 (1941).

(2) Parallel experiments were conducted using C–S–H_1 fractions obtained from both brain and spinal cord. As the results of these experiments were essentially identical, only those experiments using C–S–H_1 fractions prepared from brain will be described.