

boiled at 134–137° (25 mm.). Its acetyl derivative melted at 88–89° (89–90°).⁷

N-Nitroso-N-methylmesidine.—N,N-Dimethylmesidine was prepared from nitromesitylené by the method of Emerson, Neumann and Moundres.^{1b} It was converted to N-nitroso-N-methylmesidine by the above procedure, 6 g. (100%) of the latter being obtained from 5.5 g. of the tertiary amine. A sample distilled at 113–117° (3 mm.): n^{20}_D 1.5344, d^{20}_{20} 1.047, M^{20}_D calcd. 53.7, M^{20}_D found 52.8.

N-Methylmesidine.—Six grams of the nitroso derivative was reduced with stannous chloride and hydrochloric acid yielding 2.5 g. (50%) of N-methylmesidine: b. p. 107–118 (30 mm.), n^{20}_D 1.5248, d^{20}_{20} 0.951, M^{20}_D calcd. 49.4, M^{20}_D found 48.0. The *p*-toluenesulfonamide melted at 147.0–147.5° (145–146°)⁸ after one crystallization from aqueous alcohol.

(7) German Patent 176,474, *Chem. Zentr.*, **78**, I, 142 (1907).

(8) Hey, *J. Chem. Soc.*, 1581 (1931).

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Antimony Electrode. A Correction

BY FRANK HOVORKA AND G. H. CHAPMAN

Dr. Duncan A. MacInnes has kindly called our attention to the fact that there is a simple explanation for the difference of the *pH* values for 0.05 *M* potassium acid phthalate that he recommends¹ and the other values quoted in our paper on the "Antimony Electrode."² His value is based on the *pH* scale proposed by MacInnes, Belcher and Shedlovsky,³ and our value is obtained by using Sørensen's value for E_0 , and the two values are not comparable. The difference between the two values is about 0.02 *pH* unit. When our value is recalculated on this proposed scale, the agreement is excellent and is within the experimental error.

(1) MacInnes, "Principles of Electrochemistry," Reinhold Publishing Corporation, New York, N. Y., 1939.

(2) Hovorka and Chapman, *THIS JOURNAL*, **63**, 955 (1941).

(3) MacInnes, Belcher and Shedlovsky, *ibid.*, **60**, 1094 (1938).

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Phenolic Invert Soaps

BY JOSEPH B. NIEDERL AND FRANK A. ABRUSCATO¹

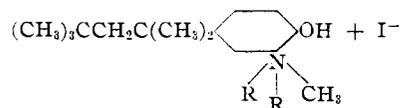
Since none of the previously reported capillary active and water soluble "invert soaps"^{2,3} contain a free phenolic hydroxyl group, it was thought desirable to prepare and study such types of com-

(1) Abstracted from the thesis presented by Frank A. Abruscato to the Graduate School of New York University in partial fulfillment of the requirement for the degree of Master of Science, June, 1941.

(2) R. Kuhn and D. Jerchel, *Ber.*, **73**, 1100 (1940).

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

pounds. For this purpose *p*- $\alpha,\alpha,\gamma,\gamma$ -tetramethoxybutylphenol (diisobutylphenol)⁴ was condensed with formaldehyde and a series of secondary amines. The resulting tertiary amines were then converted into the respective phenolic invert soaps upon treatment with methyl iodide. These quaternary ammonium salts possess the structure



Experimental Procedure

To a solution of 20 g. of diisobutylphenol and 0.1 mole of the secondary amine in 35 cc. of methanol was added with stirring 10 g. of 30% aqueous formaldehyde solution.⁵ The reaction mixture was then allowed to stand at room temperature until two layers had formed. The tertiary amine, the bottom layer, was separated and either crystallized (morpholine, piperidine) or subjected to further purification by precipitation with ammonia of the amine from its filtered solution in dilute acetic acid. The crystalline methiodides were prepared by dissolving the purified amine in an equal amount of methyl iodide and allowing the reaction mixture to stand at room temperature until the quaternary salt crystallized out.

TABLE I

Compounds	Formulas	M. p., °C. (uncor.)	Nitrogen anal.,	
			Calcd.	Found
N-(2-Hydroxy-5- $\alpha,\alpha,\gamma,\gamma$ -tetramethoxybutyl)-benzyl-				
Morpholine	C ₁₉ H ₃₁ NO ₂	44–45	4.59	4.45
Methiodide	C ₂₀ H ₃₄ NIO ₂	176–177.5	3.13	3.03
Piperidine	C ₂₀ H ₃₈ NO	92–93	4.62	4.73
Methiodide	C ₂₁ H ₃₆ NIO	162–163.5	3.15	3.00
Ammonium iodides				
Methyl-diethyl	C ₂₀ H ₃₆ NIO	124–125	3.23	3.41
Methyl-di- <i>n</i> -propyl	C ₂₂ H ₄₀ NIO	135–136.6	3.06	3.04
Methyl-di- <i>n</i> -butyl	C ₂₄ H ₂₄ NIO	132–133	2.86	2.88

(4) J. B. Niederl, *Ind. Eng. Chem.*, **30**, 1269 (1938).

(5) H. A. Bruson, U. S. Patents 2,031,557, 2,033,092 (1936).

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Indole Preparation

BY FLOYD T. TYSON

The author has found that the intramolecular condensation of *o*-formotoluide is a convenient method for the preparation of indole. Verley¹ used sodium amide as the condensing agent. No yields or details with respect to indole preparation were given by Verley although this author describes a procedure for the preparation of 2-

(1) Verley, *Bull. soc. chim.*, **35**, 1039 (1924).