TABLE I

	Compounds	Formulas	M. p., °C. (uncor.)	B. p., °C. (uncor.)	Analyses, % N Calcd.	Analyses, % N Found
I	4-($\alpha,\alpha,\gamma,\gamma$-Tetrametho)-butylphenol-methyl ether[3]	$CH_3OC_6H_4C_8H_{17}$	46	272 (760 mm.)		
II	2-Nitro-	$C_{15}H_{23}NO_3$	58	151 (3 mm.)	5.28	5.12
III	2-Amino-	$C_{15}H_{25}NO$		160 (8 mm.)	5.94	5.86
	(a) Hydrochloride	$C_{15}H_{26}NOCl$	75–77		5.49	5.36
	(b) Benzoyl deriv.	$C_{22}H_{29}NO_2$	111		4.13	4.03
IV	2-Dimethylamino-	$C_{17}H_{29}NO$		163–165 (8 mm.)	5.32	5.21
V	Methiodide	$C_{18}H_{32}NOI$	172		3.46	3.61
VI	Methosulfate	$C_{19}H_{34}NSO_5$	154		3.61	3.57

distilled off under reduced pressure. The residue, the methosulfate (VI), was recrystallized from ethyl acetate. The methosulfate dissolves in water instantaneously.

CONTRIBUTION FROM THE CHEMICAL LABORATORIES
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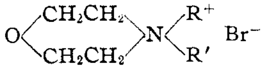
Invert Soaps. Quaternary Morpholonium Salts[1]

BY MARTIN E. MCGREAL AND JOSEPH B. NIEDERL

Since none of the previously reported[2] "invert soaps" contain a mixed heterocyclic radical it was thought desirable to prepare and study invert soaps containing a sulfur–nitrogen (long chain ethers of thiazoline phenols),[3] or an oxygen–nitrogen heterocyclic ring. For the latter purpose N-tertiary morpholines were chosen as starting materials. N-Ethyl- and N-ethylol-morpholine were converted into N-long chain substituted quaternary morpholonium salts upon treatment with lauryl, myristyl and cetyl bromide, respectively. All the resulting quaternary ammonium salts, which possess the structure as given below, proved to be soluble in water.



$$O(CH_2CH_2)_2NRR'^{+}\ Br^{-}$$

(R = —C_2H_5; —CH_2CH_2OH)
(R′ = —$C_{12}H_{25}$; —$C_{14}H_{29}$; —$C_{16}H_{33}$)

Procedure

A 0.1 mole of N-ethyl or of N-ethylol-morpholine was added to 0.1 mole of the respective halide (lauryl, myristyl or cetyl bromide), dissolved in toluene which equaled one-fourth the volume of the reactants. The solution was gently refluxed for four hours and upon cooling a solid precipitated out. The wax-like precipitate was washed with ether and purified by precipitation with anhydrous ether from a concentrated absolute ethyl alcohol solution.

TABLE I

Morpholonium bromides	Formula	M. p. °C. (uncor.)	Analyses, % Br Calcd.	Analyses, % Br Found
N-Ethyl-N-lauryl	$C_{18}H_{38}NOBr$	201	21.98	22.30
N-Ethyl-N-myristyl	$C_{20}H_{42}NOBr$	203	20.40	20.80
N-Ethyl-N-cetyl	$C_{22}H_{46}NOBr$	207	19.00	19.20
N-Ethylol-N-lauryl	$C_{18}H_{38}NO_2Br$	92	21.05	20.85
N-Ethylol-N-myristyl	$C_{20}H_{42}NO_2Br$	95	19.60	19.80
N-Ethylol-N-cetyl	$C_{22}H_{46}NO_2Br$	97	18.34	18.45

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[1] Abstracted from the thesis of M. Buonocore presented to the Graduate School of St. John's University in partial fulfillment of the requirements for the degree of Master of Science, May, 1941.

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

[3] Wm. F. Hart and J. B. Niederl, THIS JOURNAL, **63**, 945 (1941).

The Estimation of Primary Carbinol Groups in Carbohydrates

BY RICHARD E. REEVES

Fleury and Lange[1] observed that glucose is oxidized by periodic acid to yield five mols of formic acid and one mol or formaldehyde, the latter derived from the terminal or primary carbinol group. Other workers[2,3] have applied this reaction to the quantitative estimation of primary carbinol groups by distilling off and determining the formaldehyde. However, they obtained much less than the theoretical amount of formaldehyde from glucose and their method failed seriously with the methyl glucoses and mannitol.

In the present investigation suitable conditions were found for producing the theoretical amount of formaldehyde from a variety of sugars. Glucose, 2,3-dimethyl glucose and mannitol, which gave, respectively, 91, 30 and 89% of the expected amount of formaldehyde by the earlier procedure, will readily give the theoretical yield by the present method. The primary carbinol groups of certain simple sugars may now be determined almost as conveniently and accurately as the reducing group.

The significant changes in the procedure are (a) the oxidation is carried out in the presence of a slight excess of sodium bicarbonate, instead of in acid medium, and (b) the formaldehyde is precipitated directly from the reaction mixture after reduction of iodate and the excess periodate to iodide.

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[1] P. Fleury and J. Lange, *J. pharm. chim.*, **17**, 1, 409 (1933).

[2] P. Karrer and K. Pfaehler, *Helv. Chim. Acta*, **17**, 766 (1934).

[3] N. Ariyama and T. Kitasato, *J. Biochem.* (*Japan*), **25**, 357 (1937).