Anal. for xylitol pentapropionate: Calcd. for $C_{20}H_{32}O_{10}$: C, 55.54; H, 7.46. Found: C, 55.2; H, 7.44.

Anal. for xylitol pentabutyrate: Calcd. for $C_{25}H_{42}O_{10}$: C, 59.74; H, 8.42. Found: C, 59.5; H, 8.38.

Preparation of Xylitol Pentalaurate, Pentamyristate, Pentapalmitate, and Pentastearate.—These esters were prepared by reaction of xylitol with the appropriate acid chloride in the presence of pyridine. The acid chlorides were prepared by the method of Ralston[5] from Eastman acids.

To a solution of 5.0 g. (0.033 mole) of xylitol in 150 g. of dry pyridine, 0.2 mole of acid chloride was added in small portions at a time and the reaction mixture was heated on a steam-bath for four hours under anhydrous conditions. The mixture was extracted with toluene and the toluene extract was washed successively with 3% potassium hydroxide, 3% hydrochloric acid, and distilled water. The extract was decolorized with carbon, and the solvent was removed *in vacuo*, yielding the ester as a solid residue. Yields of 88, 84, 95, and 95% were obtained for the pentalaurate, pentamyristate, pentapalmitate and pentastearate, respectively. Xylitol pentalaurate and pentamyristate were most conveniently purified by recrystallization from 4 parts of acetone at −15°. The palmitic and stearic acid esters were conveniently recrystallized from acetone or toluene at −20°.

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(5) Ralston, This Journal, **61**, 1019 (1939).

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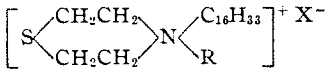
Invert Soaps. Thiomorpholinium Salts[1]

By William F. Hart and Joseph B. Niederl

Studies of the morpholine types of "invert soaps"[2] have been extended to the corresponding sulfur analogs. Recognizing the importance of a "cetyl" group in bactericidal "invert soaps"[3] N-cetylthiomorpholine was studied first. This compound was prepared by condensing cetylamine with mustard gas utilizing the methods of H. T. Clarke[4] and W. E. Lawson and E. E. Reid[5] as follows:

$$C_{16}H_{33}NH_2 + (ClCH_2CH_2)_2S \xrightarrow[C_2H_5OH]{\Delta\ Na_2CO_3} S{<}(CH_2CH_2)_2{>}NC_{16}H_{33}$$

The above tertiary amine upon treatment with appropriate alkyl halides and sulfates was then converted into the respective quaternary thiomorpholinium salts of the "simple invert soaps" types:



R: methyl, ethyl, propyl, benzyl
X: Cl, Br, I, CH_3SO_4-, $C_2H_5SO_4$-

Experimental

N-Cetylthiomorpholine.—Seventy-six grams of cetylamine (b. p. 325° uncor.) was dissolved in 120 ml. of absolute alcohol and 34 g. of anhydrous sodium carbonate added. Fifty grams of mustard gas was then added, and the solution was refluxed for eight hours. The warm solution was filtered to remove inorganic salts, which were washed twice with hot absolute alcohol. The combined alcohol extracts were distilled *in vacuo* to remove the solvent. The residue was then taken up in an excess of dry ether, and saturated with dry hydrogen chloride. The hydrochloride was filtered off and washed repeatedly with dry ether and with acetone. The free base was obtained by taking up the hydrochloride in a concentrated potassium hydroxide solution and extracting with ether. The combined ether extracts were dried over solid potassium hydroxide pellets, and the solvent removed by distillation. The free base was purified by recrystallization from ether.

The picrate was prepared by adding an equal volume of a saturated aqueous solution of picric acid to an aqueous solution of the hydrochloride. This was purified by one recrystallization from alcohol.

Quaternary Thiomorpholinium Salts.—The methiodide and ethiodide were prepared by refluxing N-cetylthiomorpholine the free base with a slight excess of methyl and ethyl iodide, respectively, for three hours, allowing the excess alkyl iodide to evaporate spontaneously at the end of that period. The products were then taken up in warm ethyl acetate in which thiothiomorpholinium iodides are soluble, cooled, filtered, and recrystallized from the same solvent.

The *n*-propyl bromide and benzyl chloride quaternary salts were prepared by refluxing the free base with an equivalent amount of the alkyl halide in toluene solution for six hours, distilling the solvent *in vacuo*, washing with dry acetone, and finally recrystallizing from ethyl acetate.

The alkyl sulfates were prepared by refluxing for four hours equivalent quantities of the free base and the respective di-alkyl sulfates, dimethyl and diethyl sulfate, in half the total volume of dry benzene.

Table I

Compound	Formula	M. p., °C. (uncor.)	Analyses, % N Calcd.	Found
N-Cetylthiomorpholine	$C_{20}H_{41}NS$	78	4.27	4.35
Hydrochloride	$C_{20}H_{42}NSCl$	162	3.82	3.93
Picrate	$C_{26}H_{44}O_7N_4S$	112	10.06	10.15
Methiodide	$C_{21}H_{44}NSI$	244	2.98	3.05
Ethiodide	$C_{22}H_{46}NSI$	205	2.90	3.02
n-Propyl bromide	$C_{23}H_{48}NSBr$	173	3.10	3.13
Benzyl chloride	$C_{27}H_{48}NSCl$	166	3.08	3.12
Methosulfate	$C_{22}H_{47}NS_2O_4$	210 dec.	3.08	3.15
Ethosulfate	$C_{24}H_{51}NS_2O_4$	Oil	2.90	2.97

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(2) J. B. Niederl and co-workers, This Journal, **63**, 1476 (1941); **66**, 840 (1944).

(3) R. Kuhn, *Ber.*, **73**, 1080, 1095, 1100, 1105, 1109 (1940).

(4) H. T. Clarke, *J. Chem. Soc.*, **101**, 1583 (1912).

(5) W. E. Lawson and E. E. Reid, This Journal, **47**, 2821 (1925).

The Preparation of the Trityl Ether of Propylene Glycol

By Nancy Green and Melvin W. Green

During recent years, propylene glycol has been introduced into many types of pharmaceutical preparations and in fact standards governing its purity have been introduced into the National