acid.[3,4] The yield of crude, pale blue dihydrochloride was 6.8 g. (33% based on ethyl bromoacetate). For purposes of analysis a sample was purified by chromatography of an absolute alcoholic solution over hydrochloric acid-washed alumina. Pure samples of the monohydrochloride were readily obtained by diluting the first few fractions with dry acetone, then adding dry ether to incipient turbidity. The white leaflets melted at 189° (microblock). *Anal.* Calcd. for $C_{17}H_{21}N_3O·HCl$: C, 63.84; H, 6.93; N, 13.14; Cl, 11.09. Found: C, 63.68; H, 6.85; N, 13.13; Cl, 11.17.

(N-Phenyl-N-benzylglycyl)-2′-benzamidoethylamide.—This was prepared in excellent yield from the preceding compound by the Schotten–Baumann method and recrystallized from pyridine–water and from alcohol; m. p. 165–166° (microblock). *Anal.* Calcd. for $C_{24}H_{25}N_3O_2$: C, 74.40; H, 6.50; N, 10.85. Found: C, 74.36; H, 6.65; N, 10.64.

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(3) The color test is negative for the ethyl phenylbenzylglycinate.

(4) Bischoff, *Ber.*, **31**, 2675 (1898), reports the benzaldehyde test as characteristic of N-phenyl-N-benzylglycine.

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NEW COMPOUNDS

Symmetrical Morpholinium and Thiamorpholinium Alkyl Sulfates[1]

The previously reported studies in "Symmetrical Morpholinium Alkyl Sulfates"[2] dealt with the reaction of dimethyl, diethyl, di-*n*-butyl, di-*n*-hexyl and di-*n*-hexadecyl sulfates with N-*n*-dodecyl, N-*n*-tetradecyl, N-*n*-hexadecyl and N-*n*-octadecyl morpholines. The purpose of the present communication is to report the extension of these reactions to include di-*n*-octyl, di-*n*-decyl and di-*n*-dodecyl sulfates and the reaction of di-*n*-hexadecyl sulfate with the corresponding previously described thiamorpholines, oxides and dioxides.[3]

TABLE I

SYMMETRICAL N,N-DIALKYL MORPHOLINIUM ALKYL SULFATES $O(CH_2CH_2)_2N(R)(R')^+SO_4R'^-$

R	R′	Formula	M. p., °C. (uncor.)	N analyses, % Calcd.	N analyses, % Found
n-Dodecyl	*n*-Octyl	$C_{32}H_{67}O_5NS$	112	2.43	2.46
n-Tetradecyl	*n*-Octyl	$C_{34}H_{71}O_5NS$	122	2.31	2.37
n-Hexadecyl	*n*-Octyl	$C_{36}H_{75}O_5NS$	132	2.21	2.27
Methyl	*n*-Decyl	$C_{25}H_{53}O_5NS$	99	2.92	2.99
n-Dodecyl	*n*-Decyl	$C_{36}H_{75}O_5NS$	81	2.21	2.26
n-Tetradecyl	*n*-Decyl	$C_{38}H_{79}O_5NS$	79	2.10	2.11
n-Hexadecyl	*n*-Decyl	$C_{40}H_{83}O_5NS$	82	2.03	2.03
Methyl	*n*-Dodecyl	$C_{29}H_{61}O_5NS$	87	2.60	2.57
n-Dodecyl	*n*-Dodecyl	$C_{40}H_{83}O_5NS$	53	2.03	2.06
n-Tetradecyl	*n*-Dodecyl	$C_{42}H_{87}O_5NS$	63	1.95	1.93
n-Hexadecyl	*n*-Dodecyl[a]	$C_{44}H_{91}O_5NS$	93	1.88	1.85
Methyl	*n*-Hexadecyl[b]	$C_{37}H_{77}O_5NS$	99.5	2.16	2.11

[a] Calculated: C, 70.81; H, 12.29. Found: C, 70.88; H, 12.08. [b] Calculated: C, 68.57; H, 11.97; S, 4.93. Found: C, 68.91; H, 11.63; S, 4.84.

Dialkyl sulfates were prepared by the method of Barkenbus and Owen.[4]

N-Alkyl thiamorpholines, oxides and dioxides were prepared by the methods previously described.[3]

TABLE II

SYMMETRICAL N,N-DIALKYL THIAMORPHOLINIUM ALKYL SULFATES

R	R′	Formula	M. p., °C. (uncor.)	N analyses, % Calcd.	N analyses, % Found
Thiamorpholium $S(CH_2CH_2)_2N(R)(R')^+SO_4R'^-$					
n-Dodecyl	*n*-Hexadecyl	$C_{48}H_{99}O_4NS_2$	160	1.71	1.70
n-Tetradecyl	*n*-Hexadecyl	$C_{50}H_{103}O_4NS_2$	127	1.65	1.59
n-Hexadecyl	*n*-Hexadecyl	$C_{52}H_{107}O_4NS_2$	84	1.60	1.63
n-Octadecyl	*n*-Hexadecyl[a]	$C_{54}H_{111}O_4NS_2$	86	1.55	1.57
Thiamorpholinium-1-oxide $OS(CH_2CH_2)_2N(R)(R')^+SO_4R'^-$					
n-Dodecyl	*n*-Hexadecyl	$C_{48}H_{99}O_5NS_2$	124	1.67	1.71
n-Tetradecyl	*n*-Hexadecyl	$C_{50}H_{103}O_5NS_2$	121	1.62	1.64
n-Hexadecyl	*n*-Hexadecyl	$C_{52}H_{107}O_5NS_2$	126	1.57	1.60
n-Octadecyl	*n*-Hexadecyl[b]	$C_{54}H_{111}O_5NS_2$	92	1.52	1.54
Thiamorpholinium-1-dioxide $O_2S(CH_2CH_2)_2N(R)(R')^+SO_4R'^-$					
n-Dodecyl	*n*-Hexadecyl	$C_{48}H_{99}O_6NS_2$	71	1.64	1.68
n-Tetradecyl	*n*-Hexadecyl	$C_{50}H_{103}O_6NS_2$	78	1.60	1.61
n-Hexadecyl	*n*-Hexadecyl	$C_{52}H_{107}O_6NS_2$	117	1.54	1.55
n-Octadecyl	*n*-Hexadecyl[c]	$C_{54}H_{111}O_6NS_2$	116	1.49	1.48

[a] Calculated: C, 71.85; H, 12.39. Found: C, 71.65; H, 12.33. [b] Calculated: C, 70.60; H, 12.18. Found: C, 70.42; H, 12.07. [c] Calculated: C, 69.39; H, 11.97. Found: C, 69.45; H, 11.74.

N,N-Dialkylmorpholinium alkyl sulfates were obtained by the reaction of equimolecular quantities (approximately 0.003 mole) of the N-alkylmorpholine and the appropriate dialkyl sulfate in a tightly stoppered Pyrex test-tube. The reaction mixture was heated to 115° (external temperature) by means of an oil-bath, held at this temperature for six hours and then allowed to remain at room temperature overnight. The reactions employing di-*n*-dodecyl and di-*n*-hexadecyl sulfates were heated at 150° for five hours. The resultant products were washed with 3 cc. of ether at 33°, cooled and centrifuged. The ether layer containing the more soluble unreacted starting materials was decanted. The washed products were crystallized three times from ethyl acetate and dried on a porous tile. Yields by this method varied from 25 to 47%, in most cases from 40 to 45%.

It was found that carrying out the reaction by refluxing the reactants in toluene for eight hours gave somewhat lower yields.

N,N-Dialkylthiamorpholinium Alkyl Sulfates.—Equimolecular quantities (approximately 0.003 mole) of the N-alkylthiamorpholine, oxide or dioxide and di-*n*-hexadecyl sulfate were added to 5 cc. of toluene which had been dried over sodium. The solution was refluxed for four hours, using an oil-bath, with an external temperature of 160–170°. Lower temperatures were found to give incomplete reaction. The toluene was distilled off *in vacuo* a little alcohol was added and distilled *in vacuo* to remove the last traces of toluene. The residue was taken up in ethyl acetate and crystallized from this solvent. In cases where decolorization was necessary, this was done with Darco in alcohol solution. The compounds were recrystallized three times from ethyl acetate or from ethyl acetate containing a little ethyl alcohol.

CHEMICAL LABORATORIES
LAFAYETTE COLLEGE
EASTON, PENNA.
AND ST. JOHN'S UNIVERSITY
BROOKLYN, N. Y.

WILLIAM F. HART
MARTIN E. MCGREAL
CONCETTO F. CAMILLI

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(2) J. B. Niederl and co-workers, THIS JOURNAL, **70**, 618 (1948).

(3) W. F. Hart and J. B. Niederl, *ibid.*, **66**, 1610 (1944); **68**, 714 (1946).

(4) Barkenbus and Owen, *ibid.*, **56**, 1204 (1934).

(5) Original manuscript received January 17, 1949.