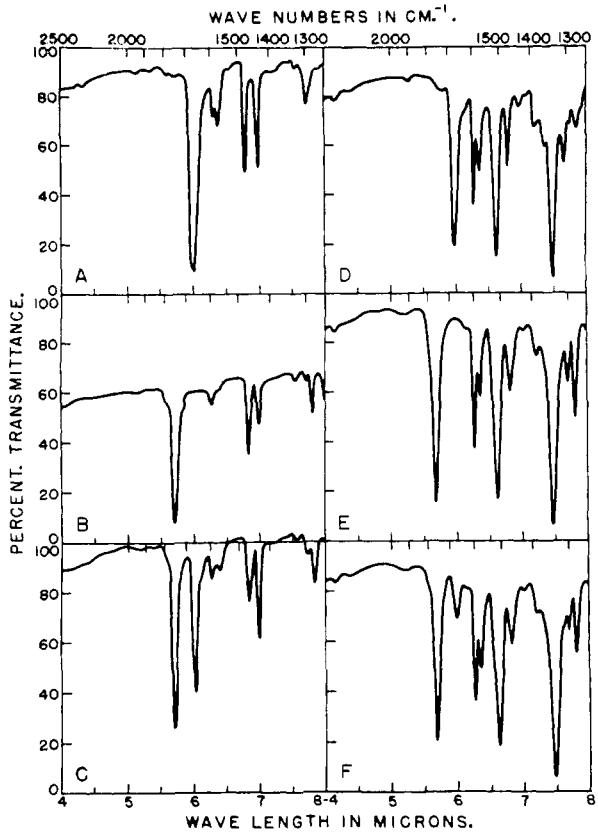


Fig. 1.—Infrared spectra: curve A, symmetrical diphenyl dithiolphthalate; curve B, unsymmetrical diphenyl dithiolphthalate; curve C, mixture of symmetrical and unsymmetrical diphenyl dithiolphthalate; curve D, symmetrical di-(*p*-nitrophenyl) dithiolphthalate; curve E, unsymmetrical di-(*p*-nitrophenyl) dithiolphthalate; curve F, mixture of symmetrical and unsymmetrical di-(*p*-nitrophenyl) dithiolphthalate. All spectra were determined in solid potassium bromide.

ride. The mixture was stirred mechanically at 25° for 18 hours. After the methylene chloride was removed by distillation under reduced pressure, the residue was taken up in 40 ml. of benzene and extracted with successive 40-ml. portions of 5% hydrochloric acid, 5% sodium bicarbonate and water. After drying and removal of the solvent, the residue was crystallized from 10 ml. of absolute ethanol; 0.40 g. (75%), m.p. 109–110°. One recrystallization raised the melting point to 112–113°.

The mixed melting point with authentic ethyl phthalimidoacetate was undepressed.

Treatment of Tryptophan Ethyl Ester with Symmetrical Di-(*p*-nitrophenyl) Dithiolphthalate.—Triethylamine (0.6 ml., 4.6 mmoles) in 20 ml. of methylene chloride was added dropwise with stirring to a solution of 0.66 g. (2.3 mmoles) of tryptophan ethyl ester hydrochloride and 0.50 g. (2.3 mmoles) of symmetrical di-(*p*-nitrophenyl) dithiolphthalate in 45 ml. of methylene chloride. The mixture was stirred for 12 hours at 25°. After the methylene chloride was evaporated under reduced pressure, the residue was taken up in 50 ml. of benzene and extracted successively with 50-ml. portions of water, 5% hydrochloric acid and finally again with water. After drying and removal of the solvent, the residue was crystallized from benzene; 0.60 g. (60%), m.p. 191–192.5°. An analytical sample was prepared by two recrystallizations from benzene.

Anal. Calcd. for $C_{20}H_{12}N_2O_6S_2$: C, 54.55; H, 2.75. Found: C, 54.46; H, 3.04.

The infrared spectrum has only the carbonyl stretching frequency at 1760 cm.$^{-1}$ characteristic of a γ-lactone.

The mixed melting point of I and II, R = *p*-$C_6H_4NO_2$, was 160–165° (depressed).

Treatment of Glycine with Symmetrical Di-(*p*-nitrophenyl) Dithiolphthalate.—A mixture of 0.85 g. (1.15 millimoles) of glycine, 0.50 g. (1.15 mmoles) of the symmetrical isomer I (R = *p*-$C_6H_4NO_2$), and 0.096 g. (1.15 mmoles) of sodium bicarbonate in a solution of 50 ml. of dioxane and 3.0 ml. of water was stirred at 25° for 18 hours. Hydrogen peroxide (0.30 ml. of 30%) was added with continued stirring for 1 hour. After the solvent was evaporated under reduced pressure, the residue was treated with 20 ml. of warm water and the yellow solid was collected by filtration. One recrystallization from benzene gave fine, yellow needles; 0.2 g. (40%), m.p. 191–192.5°. The aqueous filtrate was distilled under reduced pressure, the colorless solid remaining was recrystallized from water; 0.052 g. (24%), m.p. 193–194°.

The mixed melting point with authentic phthaloylglycine (m.p. 194°) was undepressed.

The infrared spectrum of the yellow, crystalline product shows only the band for the γ-lactone and a mixed melting point with the original symmetrical isomer was depressed.

Isomerization of I to II (R = *p*-$C_6H_4NO_2$).—A solution of 0.50 g. (1.14 mmoles) of symmetrical di-(*p*-nitrophenyl) dithiolphthalate and 3.0 ml. (1.14 mmoles) of triethylamine in 30 ml. of methylene chloride was stirred mechanically for 18 hours at 25°. The solution was extracted with two 30-ml. portions of 5% hydrochloric acid and once with 30 ml. of water. After drying, the methylene chloride was evaporated under reduced pressure and the residual yellow solid recrystallized from benzene; 0.15 g. (30%) of fine, yellow needles was obtained, m.p. 188–189°. The mixed melting point with starting material was undepressed. The infrared spectra were identical.

The benzene mother liquor, on storage in a refrigerator, for one week deposited 0.20 g. (40%) of yellow needles, m.p. 158–163°. The infrared spectrum has strong bands in the carbonyl absorption region corresponding to I (1670 cm.$^{-1}$) and II (1760 cm.$^{-1}$).

CAMBRIDGE 39, MASSACHUSETTS

[CONTRIBUTION FROM ABBOTT LABORATORIES]

Local Anesthetics. VI.[1] Alkamine Ethers of Alkyl Hydroxybenzoates

BY M. B. MOORE AND MAYNETTE VERNSTEN

RECEIVED JULY 5, 1956

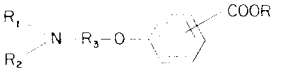
The synthesis of alkamine ethers of the three isomeric series of hydroxybenzoic esters is reported. The lower alkyl esters have local anesthetic properties, and the higher esters are fungistatic.

Only a few carboalkoxyphenyl alkamine ethers have been reported in the literature[2] and none of these include cyclic aminoalkyl ethers. In view of the advantages of the 4-morpholinyl group in aminoalkyl aryl ethers,[1] it appeared desirable to study its effect when combined with a carboalkoxy on the ring. The lower members first prepared were shown to exhibit local anesthetic effect, and

(1) Paper V, THIS JOURNAL, **76**, 4396 (1954).

(2) (a) C. Rohmann and A. Koch, *Arch. Pharm.*, **276**, 154 (1938); (b) R. Fusco, S. Chiavarelli, G. Palazzo and D. Bovet, *Gazz. chim. ital.*, **78**, 951 (1948); *C. A.*, **43**, 6592a (1949).

TABLE I

BENZOIC ESTER ALKAMINE ETHERS, 

A-No.	R	R	$-N<^{R_1}_{R_2}$	B.p. °C.	B.p. Mm.	M.p., °C.	n_D	t	Yield, %	Formula	Analyses, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Nitrogen Calcd.	Nitrogen Found
							ortho series									
6604	CH_3	$(CH_2)_3$	$N(CH_2)_4O$[a]	188–190	2.5		1.5248	25.5	37.5	$C_{15}H_{21}NO_4$					5.014	5.06
						160–161				$C_{15}H_{21}NO_4 \cdot HCl$					4.44	4.48
7276	C_2H_5	$(CH_2)_3$	$N(CH_2)_4O$[a]	163	0.8		1.5213	25.5	63	$C_{16}H_{23}NO_4$	65.55	65.64	7.90	7.81	4.78	4.64
						152–154				$C_{16}H_{23}NO_4 \cdot HCl$	58.26	58.55	7.34	7.27	4.25	4.42
7335	*i*-C_3H_7	$(CH_2)_3$	$N(CH_2)_4O$[a]	168–170	0.8		1.5137	27	20	$C_{17}H_{25}NO_4$	66.42	66.41	8.20	8.18	4.56	4.58
						134–136				$C_{17}H_{25}NO_4 \cdot HCl$	59.38	59.83	7.62	7.43	4.07	4.10
7123	*i*-C_4H_9	$(CH_2)_3$	$N(CH_2)_4O$[a]	196–200	3				25	$C_{18}H_{27}NO_4$	67.26	67.02	8.47	8.49	4.36	4.21
						103–105				$C_{18}H_{27}NO_4 \cdot HCl$	60.41	60.15	7.89	7.81	3.91	3.91
7330	*n*-C_4H_9	$(CH_2)_3$	$N(CH_2)_4O$[a]	164–171	0.25–0.4		1.5138	27.7	57	$C_{18}H_{27}NO_4$	67.26	67.30	8.47	8.41	4.36	4.33
						74–76				$C_{18}H_{27}NO_4 \cdot HCl$	60.41	60.49	7.89	7.72	3.91	3.92
7334	*i*-C_5H_{11}	$(CH_2)_3$	$N(CH_2)_4O$[a]	184	0.3		1.5118	25	64	$C_{19}H_{29}NO_4$	68.00	68.13	8.71	8.73	4.18	4.26
						88–90				$C_{19}H_{29}NO_4 \cdot HCl$	61.36	61.66	8.13	8.11	3.77	3.77
7650	*n*-C_5H_{11}	$(CH_2)_3$	$N(CH_2)_4O$[a]	184–186	1		1.5143	20	56	$C_{19}H_{29}NO_4$	68.00	68.38	8.71	8.33	4.18	4.27
7664	*n*-C_6H_{13}	$(CH_2)_3$	$N(CH_2)_4O$[a]	191	0.5		1.5089	28.5	48	$C_{20}H_{31}NO_4$	68.68	69.23	8.94	8.49	4.31	3.96
				150	.11		1.5092	27		$C_{20}H_{31}NO_4$	68.68	68.94	8.94	8.71	4.01	4.03
7665	*n*-C_7H_{15}	$(CH_2)_3$	$N(CH_2)_4O$[a]	177	.15		1.5020	26	42	$C_{21}H_{33}NO_4$	69.39	69.38	9.15	9.36	3.85	3.68
7920	*n*-C_8H_{17}	$(CH_2)_3$	$N(CH_2)_4O$[a]	217	.9		1.5065	25	27	$C_{22}H_{35}NO_4$	69.99	70.28	9.34	9.12		
8164	*n*-C_9H_{19}	$(CH_2)_3$	$N(CH_2)_4O$[a]	195–196	.15		1.5044	25	20	$C_{23}H_{37}NO_4$	70.55	71.07	9.53	9.43		
8110	*n*-$C_{10}H_{21}$	$(CH_2)_3$	$N(CH_2)_4O$[a]	205	.32		1.5030	25	18	$C_{24}H_{39}NO_4$	71.07	71.13	9.69	9.52		
9937	CH_3	$(CH_2)_2$	$N(CH_3)_2$	101	.18		1.5174	25	11	$C_{12}H_{17}NO_3$	64.55	64.79	7.68	7.84	6.27	6.19
						139–140				$C_{12}H_{17}NO_3 \cdot HCl$	55.49	55.40	6.99	6.95	5.39	5.50
9944	CH_3	$(CH_2)_2$	$N(C_2H_5)_2$	111	.15		1.5103	26	29	$C_{14}H_{21}NO_3$	66.91	66.97	8.42	8.25	5.57	5.60
9951	C_2H_5	$(CH_2)_2$	$N(C_2H_5)_2$	115	.15		1.5049	25	55	$C_{15}H_{23}NO_3$	67.89	67.60	8.71	8.72	5.28	5.15
						106–107				$C_{15}H_{23}NO_3 \cdot HCl$	59.69	59.78	8.01	8.23	4.64	4.48
1-0096	C_2H_5	$(CH_2)_2$	$N(CH_3)_2$	106	.18		1.5100	25	36	$C_{13}H_{19}NO_3$	65.75	65.48	8.07	7.90	5.90	5.75
1-0285	C_2H_5	$(CH_2)_5$	$N(CH_2)_5$[b,f]			108.5–110.5			59	$C_{19}H_{29}NO_3 \cdot HCl$	64.12	64.15	8.51	8.30		
1-0286	C_2H_5	$(CH_2)_5$	$N(CH_2)_4O$[a,f]			113–115			56	$C_{18}H_{27}NO_4 \cdot HCl$	60.41	60.50	7.89	7.74		
1-0287	C_2H_5	$(CH_2)_5$	$N(CH_2)_4$[c,f]			86–88			67.5	$C_{18}H_{27}NO_3 \cdot HCl$	63.24	63.20	8.26	8.26		
1-0288	C_2H_5	$(CH_2)_5$	$N(CH_2)_4NCH_3$[d,f]			213–215			63	$C_{19}H_{30}N_2O_3 \cdot 2HCl$	56.01	56.11	7.91	8.03		
1-0305	CH_3	$(CH_2)_6$	$N(CH_2)_4O_4$[a,f]			99–101			32	$C_{18}H_{27}NO_4 \cdot HCl$	60.41	60.16	7.89	7.92		
1-0371	CH_3	$(CH_2)_6$	$N(CH_2)_4NCH_3$[d,f]			200–202			21	$C_{19}H_{30}N_2O_3 \cdot 2HCl$	56.01	56.19	7.91	7.99		
							meta series									
7686	CH_3	$(CH_2)_3$	$N(CH_2)_4O$[a]	201	0.5		1.5277	26	45	$C_{15}H_{21}NO_4$	64.48	64.44	7.58	7.31	5.01	4.88
7687	C_2H_5	$(CH_2)_3$	$N(CH_2)_4O$[a]	202–205	1.1–1.3		1.5197	27	66.5	$C_{16}H_{23}NO_4$	65.50	65.25	7.90	7.50	4.78	4.87
7792	*n*-C_3H_7	$(CH_2)_3$	$N(CH_2)_4O$[a]	206–208	0.7–0.5		1.5163	26	68.5	$C_{17}H_{25}NO_4$	66.42	66.56	8.20	8.42	4.56	4.78
7797	*n*-C_4H_9	$(CH_2)_3$	$N(CH_2)_4O$[a]	216	1		1.5113	26.5	60	$C_{18}H_{27}NO_4$	67.11	67.08	8.47	8.44	4.36	4.40
7794	*n*-C_5H_{11}	$(CH_2)_3$	$N(CH_2)_4O$[a]	224–226	1				76.5	$C_{19}H_{29}NO_4$	68.03	68.40	8.72	8.62	4.18	4.37

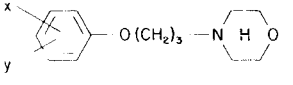
TABLE I (*Continued*)

A-No.	R	R_1	$-N\langle^{R_1}_{R_2}$	B.p. °C.	B.p. Mm.	M.p., °C.	n_D		Yield, %	Formula	Analyses, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Nitrogen Calcd.	Nitrogen Found
							para series									
7791	CH_3	$(CH_2)_3$	$N(CH_2)_4O$[a]	191–194.5	0.5		1.5350	27	61	$C_{15}H_{21}NO_4$	64.48	64.27	7.58	7.33	5.01	5.10
7209	C_2H_5	$(CH_2)_3$	$N(CH_2)_4O$[a]			148–149			39	$C_{16}H_{23}NO_4$·HCl	58.26	58.51	7.33	7.23	4.25	4.33
7216	n-C_3H_7	$(CH_2)_3$	$N(CH_2)_4O$[a]			130–132			57.5	$C_{17}H_{25}NO_4$·HCl	59.38	59.31	7.62	7.84	4.07	4.12
7278	n-C_4H_9	$(CH_2)_3$	$N(CH_2)_4O$[a]			124–126			79	$C_{18}H_{27}NO_4$·HCl	60.41	60.32	7.89	7.70	3.91	4.07
8814	C_6H_{13}	$(CH_2)_3$	$N(CH_2)_4O$[a]			123–126			10	$C_{20}H_{31}NO_4$·HCl	62.24	61.98	8.36	8.39		
8349	n-C_7H_{15}	$(CH_2)_3$	$N(CH_2)_4O$[a]			131–132			18	$C_{21}H_{33}NO_4$·HCl	63.06	63.23	8.57	8.68	3.50	3.47
7593	n-C_8H_{17}	$(CH_2)_3$	$N(CH_2)_4O$[a]			127–129			68.5	$C_{22}H_{35}NO_4$·HCl	63.82	63.31	8.77	8.46		
1-0219	CH_3	$(CH_2)_2$	$N(CH_2)_5$[b,f]			190–191			67	$C_{15}H_{21}NO_3$·HCl	60.09	59.79	7.40	7.26		
1-0220	CH_3	$(CH_2)_2$	$N(CH_2)_4O$[a]	164	0.5		1.5392	25	59	$C_{14}H_{19}NO_4$	63.38	63.10	7.22	7.45	5.34	5.37
						200–202				$C_{14}H_{19}NO_4$·HCl	55.72	55.62	6.68	6.67		
9935[e]	CH_3	$(CH_2)_2$	$N(C_2H_5)_2$			147–148			37	$C_{14}H_{21}NO_3$·HCl	58.36	58.75	7.71	7.86	4.87	5.08
9936[e]	C_2H_5	$(CH_2)_2$	$N(C_2H_5)_2$			151			64	$C_{15}H_{23}NO_3$·HCl	59.69	59.84	8.01	8.19	4.64	4.64
9952	C_2H_5	$(CH_2)_2$	$N(CH_3)_2$			151–153			23	$C_{13}H_{19}NO_3$·HCl	57.03	57.25	7.36	7.62	5.12	5.22

[a] 4-Morpholinyl. [b] 1-Piperidinyl. [c] 1-Pyrrolidinyl. [d] 4-(1-Methylpiperazinyl). [e] Reported in ref. 2a. [f] Method B.

TABLE III

SUBSTITUTED ESTER ETHERS,

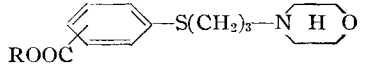


A-No.	x	y	B.p. °C.	B.p. Mm.	M.p., °C.	$n^{25}D$	Yield, %	Formula	Analyses, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found	Nitrogen Calcd.	Nitrogen Found
396	2-$COOCH_3$	4-Br	223–228	1.6			small[a]	$C_{15}H_{20}BrNO_4$	50.29	50.99	5.63	5.59		
6740	4-$COOC_2H_5$	2-OCH_3	188–190	2.5		1.5275	29	$C_{15}H_{21}NO_4$					5.01	5.06
					160–161			$C_{15}H_{21}NO_4$·HCl					4.44	4.48
9443	3-$CH_2CH_2COOCH_3$	2-OCH_3			145–147		10	$C_{18}H_{27}NO_5$·HCl	57.82	57.62	7.55	7.30		
9401	2-$COOCH_3$	5-NH_2			127		5	$C_{15}H_{22}N_2O_4$	61.20	61.45	7.53	7.67		
1-0436	4-$COOCH_3$	2-Br			181–183		72	$C_{15}H_{20}BrNO_4$·HCl	45.61	46.16	5.36	5.45	20.24[b]	19.69[b]

[a] The reaction did not go well apparently due partly to the easy hydrolysis of the ester during manipulation. Attempts to crystallize the hydrochloride were unsuccessful. [b] Bromine analysis.

TABLE IV

BENZOIC ESTER ALKAMINE SULFIDES,



A-No.	R	B.p. °C.	B.p. Mm.	M.p., °C.	$n^{20}D$	Yield, %	Formula	Analyses, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found
6553	2-CH_3			173–175		87	$C_{15}H_{21}NO_3S$·HCl	54.29	54.35	6.68	6.77
6564	4-CH_3			171–172		81	$C_{15}H_{21}NO_3S$·HCl	54.29	54.43	6.68	6.93
7649	2-$C_4H_9(n)$	194–195	0.18		1.5508	71	$C_{18}H_{27}NO_3S$	64.06	64.10	8.01	7.80

TABLE II

HYDROXYBENZOIC ESTERS, HO  COOR

Position	R	B.p. °C.	B.p. Mm.	M.p., °C.	n_D	t	Yield, %	Formula	Analyses, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found
2	C_6H_{13}[a]	109	0.7		1.5019	25	91	$C_{13}H_{18}O_3$	70.24	70.53	8.16	8.21
2	n-C_7H_{15}	113	.25		1.4980	27.5	69	$C_{14}H_{20}O_3$	71.16	71.09	8.53	8.48
2	n-C_9H_{19}[b]	141–144	.7		1.4825	25	55	$C_{16}H_{24}O_3$	72.69	75.04	9.15	10.32
2	n-$C_{10}H_{21}$[b]	144	.55		1.4918	22	46	$C_{17}H_{26}O_3$	73.34	74.20	9.42	9.93
3	n-C_3H_7	118–123	.1	32–33	1.5292	24	85	$C_{10}H_{12}O_3$	66.65	66.46	6.73	6.51
3	n-C_4H_9	152–154	.4		1.5233	24	90	$C_{11}H_{14}O_3$	68.02	68.07	7.27	7.11
3	n-C_5H_{11}	172	.1		1.5180	24.5	81	$C_{12}H_{16}O_3$	69.21	69.09	7.75	7.80
3	n-C_7H_{15}[b]	185–186	1.0		1.5103	25.5	63	$C_{14}H_{20}O_3$	71.16	71.97	8.53	8.68
4	C_6H_{13}[a]	175–178	0.5		1.5178	26.5	87	$C_{13}H_{18}O_3$	70.24	70.75	8.16	8.50
4	n-C_8H_{17}[b]	182–188	0.5	39–41			68	$C_{15}H_{22}O_3$	71.97	72.40	8.86	9.00

[a] The hexyl alcohol used was a sample obtained from a German source; configuration not known. [b] Separation from a by-product, probably the dialkyl ether, was very difficult. The final products from these were satisfactory. Herz, THIS JOURNAL, **67**, 2271 (1941), has reported a number of pure alkyl salicylates, including *n*-octyl.

the work was then extended to include the three isomeric series, higher alkyl esters and other cyclic and non-cyclic amines. In Table I are listed the compounds synthesized, with appropriate physical data. Table II gives similar data for those hydroxybenzoic esters not found in the literature. A limited study was made of the effect of other substituents in the ring, and such compounds are reported in Table III. Similar derivatives of *p*-aminosalicylic acid have recently been reported by Grimme and Schmitz.[3]

A few compounds in which sulfur replaces the ether oxygen are reported in Table IV.

The local anesthetic properties were studied by Dr. J. L. Schmidt and his group, and the fungistatic and bacteriostatic effects by Dr. W. E. Grundy and his group at Abbott Laboratories. The lower members exhibit local anesthetic effects of the procaine type. The higher members are markedly fungistatic, but their activity is vitiated by the presence of serum.

Experimental

Esters of Hydroxybenzoic Acids.—These were prepared in the usual way by refluxing with the appropriate alcohol in the presence of an acid catalyst. Sulfuric acid was used in most cases, but hydrogen chloride would probably be more desirable because of the formation of dialkyl ethers which are hard to separate in the case of the higher alcohols. From such a reaction, di-*n*-octyl ether was isolated, boiling at about 160° at 14 mm., $n^{25}D$ 1.4249.

Anal. Calcd. for $C_{16}H_{34}O$: C, 79.26; H, 14.14. Found: C, 79.56; H, 13.84.

Alkamine Ethers. A.—The sodium or potassium salt of the phenol was formed by reaction with an equivalent of the metal alcoholate in the appropriate alcohol. The alcohol was the same as that used to form the ester, thus avoiding any possibility of ester interchange. The aminoalkyl halide was then added and the product refluxed, usually only a few hours, until a test showed the product no longer to be strongly alkaline. Most of the alcohol was removed under reduced pressure, and the residue partitioned between water and an appropriate organic solvent. The organic layer was separated, dried and distilled to remove the solvent and finally to purify the basic ether ester; in some cases the hydrochloride was formed in the organic solvent and further purified by recrystallization.

B.—Several of the alkamine ethers were prepared *via* the alkyl ω-bromoalkoxybenzoates. These bromides were synthesized by a modification of the method of Marvel and Tannenbaum,[4] using an alcohol as the solvent and replacing the sodium hydroxide by the appropriate sodium alcoholate. By distillation of the alcohol from the reaction mixture, extraction of the residue with ether and evaporation of the ether, a crude product was obtained. Solid methyl 4-(β-bromoethoxy)-benzoate was obtained by extraction of the crude material with hot petroleum ether. The liquid alkyl 2-(ω-bromoalkoxy)-benzoates were recovered by vacuum distillation of the crude oil. The products obtained in these cases were not analytically pure, but were suitable for reaction with an excess of the appropriate amine. Method B was used only for compounds as indicated in Table I.

In the case of esters with larger alkyl groups, difficulties were encountered because of the solubility of these alcohols in organic solvents and their boiling points close to those of the ether esters. In these cases repeated distillation of the bases, or recrystallization of the hydrochlorides was necessary to obtain the pure ether esters.

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NORTH CHICAGO, ILLINOIS

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