logical Coördination Center of the National Research Council. Preparation was accomplished by heating 5 g. of either the corresponding ethyl 2-pyridylaminomethylenemalonate (I)[2] or the 3-carbethoxy-2H-pyrido-1,2-a-pyrimidine-4-one (II)[2] with 250 ml. of 1% aqueous sodium hydroxide solution at 90° for five minutes with II or thirty minutes with I. The solution was filtered hot and made acid to congo red with dilute hydrochloric acid while still hot. The precipitated acid was twice recrystallized from pyridine.

3-(4-Methyl-2-pyridylamino)-acrylic Acid.—M. p. 238° with decarboxylation; yield from I 43%, from II 61%.

Anal. Calcd. for $C_9H_{10}O_2N_2$; N, 15.72; neut. equiv., 178. Found: N, 15.65[3]; neut. equiv., 176.

3-(5-Methyl-2-pyridylamino)-acrylic Acid.—M. p. 258° with decarboxylation; yield from I 36%, from II 67%.

Anal. Calcd. for $C_9H_{10}O_2N_2$: N, 15.72; neut. equiv., 178. Found: N, 15.74[3]; neut. equiv., 180.

(2) G. R. Lappin, THIS JOURNAL, **70**, 3348 (1948).

(3) Microanalysis by the Clark Microanalytical Laboratory, Urbana, Ill.

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Some Quaternary Salts of Carbamates of Amino Alcohols[1]

A series of compounds of the general formula $[R_3'N—A—OCONR_2]^+I^-$ has been prepared (Table I). The unsubstituted carbamates (both R = H) were prepared by method 1, the N-methylcarbamates (R = CH_3, and R = H) by method 2, and the N,N-dimethylcarbamates (both R = CH_3) by method 3.

(1) Reaction of a dialkylamino alcohol with phosgene gave the dialkylaminoalkyl chloroformate, which reacted with ammonia to give the urethan. Condensation with an alkyl iodide gave the quaternary salt.[2]

$$R_2N(CH_2)_nOH \xrightarrow{COCl_2} R_2N(CH_2)_nOCOCl \xrightarrow{NH_3} R_2N(CH_2)_nOCONH_2 \xrightarrow{RI} [R_3N(CH_2)_nOCONH_2]^+I^-$$

(2) Reaction of a dialkylamino alcohol with methyl isocyanate gave a carbamate, which reacted with an alkyl iodide to give the quaternary salt.

$$R_2N(CH_2)_nOH \xrightarrow{CH_3NCO} R_2N(CH_2)_nOCONHCH_3 \xrightarrow{RI} [R_3N(CH_2)_nOCONHCH_3]^+I^-$$

(3) Reaction of a chloro alcohol and phosgene gave the chloroalkyl chloroformate, which was converted to the iodoalkyl carbamate by reaction first with dimethylamine and then with sodium iodide in acetone. Condensation of the iodoalkyl carbamate with a tertiary amine gave the quaternary salt.[3]

$$Cl(CH_2)_nOH \xrightarrow{COCl_2} Cl(CH_2)_nOCOCl \xrightarrow{(CH_3)_2NH} Cl(CH_2)_nOCON(CH_3)_2 \xrightarrow{NaI} I(CH_2)_nOCON(CH_3)_2 \xrightarrow{R_3N} [R_3N(CH_2)_nOCON(CH_3)_2]^+I^-$$

(1) These compounds were prepared for the Office of Scientific Research and Development under Contract OEMsr-136 with Stanford University.

(2) Dalmer and Diehl, U. S. Patent 1,894,162; *C. A.*, **27**, 2533 (1933).

(3) Sprinson, THIS JOURNAL, **63**, 2249 (1941).

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TABLE I

QUATERNARY SALTS OF CARBAMATES OF AMINO ALCOHOLS

Compound	M. p., °C.	Formula	Iodide analyses, % Calcd.	Found
2-Di-*n*-butylaminoethyl carbamate butiodide	99–100	$C_{15}H_{33}IN_2O_2$	31.70	31.47
3-Di-*n*-butylaminopropyl carbamate butiodide	122–123	$C_{16}H_{35}IN_2O_2$	30.62	30.64
3-Di-*n*-amylaminopropyl carbamate amyl iodide	108–110	$C_{19}H_{41}IN_2O_2$	27.80	27.40
2-Diethylaminoethyl N-methylcarbamate ethiodide	90–92	$C_{10}H_{23}IN_2O_2$	38.42	38.52
2-Di-*n*-butylaminoethyl N-methylcarbamate butiodide	100–101.5	$C_{16}H_{35}IN_2O_2$	30.62	30.43
2-Pentamethyleneaminoethyl N-methylcarbamate methiodide	103–105	$C_{10}H_{21}IN_2O_2$	38.66	38.99
3-Di-*n*-butylaminopropyl N-methylcarbamate butiodide	110.5–112	$C_{17}H_{37}IN_2O_2$	29.62	29.36
3-Di-*n*-amylaminopropyl N-methylcarbamate amyliodide	78–83	$C_{20}H_{43}IN_2O_2$	26.97	27.05
1-(3,4-Methylenedioxybenzyl)-2-[(3,4-methylenedioxybenzyl)-methylamino]-ethyl N-methylcarbamate methiodide	155–157	$C_{22}H_{27}IN_2O_6$	23.40	23.30
3-Dimethylamino-*d*-bornyl N-methylcarbamate methiodide	187–189	$C_{15}H_{29}IN_2O_2$	32.02	31.91
2-Diethylaminoethyl N,N-dimethylcarbamate ethiodide	106–107	$C_{11}H_{25}IN_2O_2$	36.86	37.08
Octahydro-N-[2-(dimethylcarbamyloxy)-ethyl]-2-methylpyrrocolinium iodide	150–151.5	$C_{14}H_{27}IN_2O_2$	33.19	33.23

Stilbestrol Esters

Since a new series of testosterone esters[1] was found to have greater androgenic activity than testosterone propionate, similar stilbestrol esters have been prepared to determine whether these esters have any advantage over stilbestrol dipropionate. Two representative esters have been prepared.

Diethylstilbestrol Di-ethoxyacetate.—A solution of 1 g. of diethylstilbestrol (1 mole) in 15 cc. of dry ether and 6 cc. of dry pyridine was prepared. To this was added 2 cc. of ethoxyacetyl chloride (5 mole) in 10 cc. of dry ether. The reaction mixture was refluxed for one hour and 100 cc. more ether was added. This was poured into water and the ether layer separated, washed with dilute sulfuric acid, dilute sodium carbonate solution and water. Evaporation of the ether left 1.40 g. of reddish white powder, m. p. 129–136°. The product was taken up in a large amount of ether and filtered through activated alumina (Aluminum Ore Co. mm. 80 mesh). The red color was adsorbed on the alumina. Evaporation of the ether left a residue which was twice crystallized from 95% ethanol giving a product (1.05 g.) melting at 136.5–137.5°.

Anal. Calcd. for $C_{26}H_{32}O_6$: C, 70.89; H, 7.32. Found: C, 71.16; H, 7.57.

(1) Mooradian and Lawson, in press.