

cator over sulfuric acid the compound melted at 276–278° (uncor.).

*Anal.* Calcd. for  $C_{22}H_{18}S_3O_8N_4$ : N, 9.96; S, 17.09. Found: N, 9.54; S, 16.85.

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### Some Diamino Peptides

Dimethylamine reacted with *p*-nitro- $\beta$ -bromopropionanilide to form *p*-nitro- $\beta$ -dimethylaminopropionanilide, m. p. of the hydrochloride, 200–201°. A portion of this was transformed into the methochloride and both substances were reduced catalytically in alcohol containing hydrogen chloride to give, respectively, *p*-amino- $\beta$ -dimethylaminopropionanilide dihydrochloride (I), colorless felted needles, m. p. 218–219°, and *p*-amino- $\beta$ -dimethylaminopropionanilide methochloride hydrochloride (II), hygroscopic crystals, m. p. 211–212°.

*N,N*-Diethylethylenediamine,<sup>1</sup> prepared by the reduction (sodium and alcohol) of diethylglycine nitrile, reacted with *p*-nitrobenzoyl chloride, forming  $\beta$ -[*p*-nitrobenzoylamidoethyl]-diethylamine hydrochloride, colorless needles melting at 164–5°. This substance and its ethochloride were reduced catalytically in alcoholic hydrogen chloride to  $\beta$ -[*p*-aminobenzoylamidoethyl]-diethylamine dihydrochloride (III), m. p. 176.5–178°, and to  $\beta$ -[*p*-aminobenzoylamidoethyl]-triethylammonium chloride, hydrochloride (IV), which forms stubby prisms melting at 228°.

Reduction of  $\beta$ -dimethylaminopropionitrile gave  $\gamma$ -dimethylaminopropylamine, m. p. of the dihydrochloride, 182–184°. The base reacted with *p*-nitrobenzoyl chloride

Formula	M. p., °C.	Composition
$(n-C_{13}H_{27})C_6H_5NMe_2I$	93–94	$C_{26}H_{46}NI$
$(C_6H_{11}CH_2CH_2)(C_6H_5CH_2)NMe_2Cl$	206 dec.	$C_{17}H_{28}NCl$
$(C_6H_5CH_2)(BrCH_2CH_2)NMe_2Br$	174	$C_{11}H_{17}NBr_2$
$(\alpha-C_{10}H_7CH_2)NEt_3Cl$	197 dec.	$C_{17}H_{24}NCl$

to form  $\gamma$ -[*p*-nitrobenzoylamidopropyl]-dimethylamine hydrochloride, m. p. 190–192°. This was reduced catalytically in alcoholic hydrogen chloride solution to  $\gamma$ -[*p*-aminobenzoylamidopropyl]-dimethylamine dihydrochloride (V), m. p. 184–185°.

Reduction of the *p*-nitrophenylurethan of  $\beta$ -hydroxyethyltriethylammonium chloride yielded  $\beta$ -[*p*-aminophenylcarbamatoethyl]-triethylammonium chloride, hydrochloride (VI), which forms irregular prisms melting at 138–139°.

By the reduction of  $\beta$ -[*p*-nitrobenzoyloxyethyl]-triethylammonium chloride, there was obtained  $\beta$ -[*p*-aminobenzoyloxyethyl]-triethylammonium chloride, hydrochloride (VII), m. p. 214–215°. This has been reported<sup>2</sup> previously, but the synthesis used was not free from ambiguity.

The salts described here were crystallized from absolute alcohol or from alcohol-ethyl acetate mixtures. The analytical data are in the table.

(1) Ristenpart, *Ber.*, **29**, 2526 (1896).

(2) Einhorn and Uhlfelder, *Ann.*, **371**, 138 (1909).

No.	Formula	Analyses, %			
		Calcd. C	Calcd. H	Found C	Found H
I	$C_{11}H_{19}ON_3Cl_2$	47.12	6.84	47.38	6.61
II	$C_{12}H_{21}ON_3Cl_2$	48.96	7.20	48.67	7.27
III	$C_{13}H_{23}ON_3Cl_2$	50.63	7.52	50.67	7.80
IV	$C_{15}H_{27}ON_3Cl_2$	53.56	8.13	53.45	8.04
V	$C_{12}H_{21}ON_3Cl_2$	48.96	7.20	49.05	7.51
VI	$C_{15}H_{27}O_2N_3Cl_2$	51.12	7.73	51.27	7.75
VII	$C_{15}H_{26}O_2N_3Cl_2$	53.39	7.77	53.02	7.62

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### Some New Quaternary Salts

Methylaniline and octadecyl iodide reacted to yield methyloctadecylaniline, a yellow oil, b. p. (3 mm.), 234°. This was treated in benzene solution with methyl iodide forming octadecylphenyldimethylammonium iodide which crystallized in leaflets from ethyl acetate.

Cyclohexylethylamine (prepared from cyclohexylacetic acid by the sequence: acid  $\rightarrow$  acid chloride  $\rightarrow$  amide  $\rightarrow$  nitrile  $\rightarrow$  amine) was methylated by the Clarke-Eschweiler method. The resulting tertiary amine reacted with benzyl chloride in ether to give cyclohexylethylbenzyltrimethylammonium chloride.

Benzyl- $\beta$ -bromoethyltrimethylammonium bromide was prepared from benzyltrimethylamine and ethylene bromide.

Triethylamine and  $\alpha$ -menaphthyl chloride ( $\alpha$ -naphthylmethyl chloride) yielded  $\alpha$ -menaphthyltriethylammonium chloride. Data on these substances are presented in the subjoined table.

Formula	M. p., °C.	Composition	Analyses, %			
			Calcd. C	Calcd. H	Found C	Found H
$(n-C_{13}H_{27})C_6H_5NMe_2I$	93–94	$C_{26}H_{46}NI$	62.25	9.65	62.12	9.69
$(C_6H_{11}CH_2CH_2)(C_6H_5CH_2)NMe_2Cl$	206 dec.	$C_{17}H_{28}NCl$	72.44	10.02	72.15	10.16
$(C_6H_5CH_2)(BrCH_2CH_2)NMe_2Br$	174	$C_{11}H_{17}NBr_2$	40.87	5.31	40.91	5.61
$(\alpha-C_{10}H_7CH_2)NEt_3Cl$	197 dec.	$C_{17}H_{24}NCl$	73.49	8.71	73.41	8.62

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### Some Mono- and Disubstituted Guanidines

The guanidines here described were all prepared by conventional methods. The *S*-methyl-isothiourea sulfate method (generally applicable with amines of moderate strength and water solubility) was used to prepare the five following:  $\beta$ -[*N*-morpholinoethyl]-guanidine sulfate (from aminoethylmorpholine),  $\beta$ , $\beta$ -diethoxyethylguanidine sulfate (from amino acetal), *N,N*-dicyclohexylguanidine sulfate (from dicyclohexylamine), *N*-benzyl-*N*-methylguanidine sulfate (from benzylmethylamine) and  $\delta$ -phenoxybutylguanidine sulfate (from  $\delta$ -phenoxybutylamine prepared by reduction of  $\gamma$ -phenoxybutyronitrile). In all these cases the salt was isolated by evaporation of the reaction mixture