

and crystallized from mixtures of polar and non-polar solvents. Diethoxyethylguanidine sulfate crystallized best from *n*-butanol-ethyl acetate. The others were crystallized from alcohol-ether.

N-Benzyl-N- α -naphthylguanidine Hydrochloride.—Benzyl- α -naphthylamine was refluxed in amyl alcohol with 1 mol of cyanamide and 1.2 mols of hydrogen chloride. On cooling, ether was added and the hydrochlorides separated as a purple sirup. Attempts to crystallize having failed, the mixture was dissolved in water and ammonia added. Some unreacted benzylnaphthylamine separated together with most of the color. The aqueous layer was then basified with sodium hydroxide and the precipitated guanidine taken into ether. After drying over potassium carbonate, alcoholic hydrogen chloride was added. The salt was recrystallized from alcohol-ether mixtures.

N,N'-Dihomoanisylguanidine Hydrochloride.—To a

R	X	Y	M. p., °C.	Formula	Analyses, %			
					Calcd.		Found	
				C	H	C	H	
CH ₃	Cl	I	152	C ₁₇ H ₂₀ O ₂ NCII	46.19	6.62	46.20	6.42
C ₆ H ₅ CH ₂	H	Cl	122-123	C ₂₃ H ₃₄ O ₂ NCl	3.58 ^a	9.05 ^b	3.91 ^a	9.21 ^b
<i>p</i> -ClC ₆ H ₄ CH ₂	H	Cl	166-166.5	C ₂₃ H ₃₀ O ₂ NCl ₂	64.76	7.80	64.85	7.80
<i>p</i> -ClC ₆ H ₄ CH ₂	Cl	Cl	160	C ₂₃ H ₃₀ O ₂ NCl ₃	59.92	7.00	60.12	7.01
<i>p</i> -BrC ₆ H ₄ CH ₂	Cl	Cl	156.5-157	C ₂₃ H ₃₂ O ₂ NCl ₂ Br	54.64	6.38	54.95	6.35

^a N, ^b Cl.

solution of 2 mols of homoanisylamine in absolute ether was added, with shaking and ice-cooling, a solution of 1 mol of cyanogen bromide in ether. After standing one-half hour the ether was evaporated by a stream of dry air, a little absolute alcohol added to homogenize the mixture and the whole was heated three hours in an oil-bath at 150°. The material was then dissolved in water, the base liberated with alkali, and extracted with ether. On drying over potassium carbonate a crystalline solid appeared on the surface of the drying agent. The ether was decanted off, the solid dissolved in chloroform and transformed into the hydrochloride by alcoholic hydrogen chloride. It was recrystallized from alcohol-ether, forming lustrous plates.

N-Methyl-N'- α -naphthylguanidine.—Methylamine and α -naphthylisothiocyanate yielded N,N'-methylnaphthylthiourea. Methylation with methyl sulfate gave the S-methyl derivative which was desulfurized in the usual manner with lead oxide and ammonia.

Data on these compounds are presented in the table.

Substance	M. p., °C.	Empirical formula	Analyses, %			
			Calcd.		Found	
			C	H	C	H
β -N-Morpholinoethylguanidine sulfate	197	C ₇ H ₁₆ ON ₄ (H ₂ SO ₄) _{1/2}	37.99	7.75	38.33	7.52
β,β -Diethoxyethylguanidine sulfate	154	C ₇ H ₁₇ O ₂ N ₃ (H ₂ SO ₄) _{1/2}	37.47	8.09	37.38	7.99
N,N-Dicyclohexylguanidine sulfate	195	C ₁₃ H ₂₅ N ₃ (H ₂ SO ₄) _{1/2}	57.28	9.62	57.05	9.28
N-Benzyl-N-methylguanidine sulfate	252 dec.	C ₉ H ₁₃ N ₃ (H ₂ SO ₄) _{1/2}	50.90	6.65	50.87	6.59
δ -Phenoxybutylguanidine sulfate	199-199.5	C ₁₁ H ₁₇ ON ₃ (H ₂ SO ₄) _{1/2}	51.53	7.08	51.32	7.33
N-Benzyl-N- α -naphthylguanidine hydrochloride	223-224	C ₁₈ H ₁₇ N ₃ ·HCl	69.33	5.82	69.19	5.80
N,N'-Dihomoanisylguanidine hydrochloride	125.5-126.5	C ₁₉ H ₂₆ O ₂ N ₃ ·HCl	62.69	7.21	62.63	7.05
N- α -Naphthyl-N'-methylguanidine hydrochloride	220-220.5 dec.	C ₁₂ H ₁₃ N ₃ ·HCl	61.12	5.99	61.06	5.99

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Some Quaternary Salts from β -Dimethylamino- β' -cymoxydiethyl Ether

A concentrated aqueous solution of sodium thymolate or sodium *p*-chlorothymolate was heated under reflux with β,β' -dichlorodiethyl ether. After separation of the aqueous layer, unreacted dichlorodiethyl ether was removed *in vacuo*, and the residue was heated for seven hours at 145° (*p* = ca. 150 lb.) in a glass-lined bomb with 33% methanolic dimethylamine. After removal of volatile materials on the steam-bath under diminished pressure, the residual tertiary amines were partially purified by solution in acid and extraction with ether; on liberation with alkali, they were obtained as oils which could not be distilled *in vacuo*, but were converted directly into quaternary salts by warming on the steam-bath with the appropriate halides. The salts crystallized from acetone or alcohol on addition of ether.

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N,N-Dimethylethylenediamine and Some Derivatives

The readily available dimethyl glycine nitrile¹ can be reduced by sodium and absolute alcohol to give N,N-dimethylethylenediamine. This is a colorless liquid boiling, when anhydrous, at 107°. As its dehydration is difficult and its dihydrochloride (melting around 160°) is also hygroscopic, it is better characterized through a derivative. The following compounds were prepared as outlined, nitro derivatives being reduced with Adams catalyst in alcoholic solution containing hydrogen chloride. β -[*p*-Nitrobenzoylamidoethyl] dimethylamine hydrochloride (I), formed from *p*-nitrobenzoyl chloride and the

diamine, was hydrogenated catalytically yielding β -[*p*-aminobenzoylamidoethyl]-dimethylamine dihydrochloride (II). The methochloride of I was reduced to β -[*p*-amino-

¹ J. v. Braun, *Ber.*, **40**, 3937 (1907).