α -methyl substitution, and it was concluded that this could be explained most satisfactorily in terms of an induced fold, as bulky substituents near the ester group would tend to hinder efficient fold formation. It can be seen from Table III that a similar pattern is observed in the present hydrolysis studies, a marked drop in the rate of hydrolysis occurring on β -methyl substitution of SCh₂, thus supporting the idea of induced-fold formation. Although low rates of hydrolysis are observed for both the L- and $D-\beta$ -MeSCh₂ isomers, their affinities for the enzyme are different (Table I). The isomer with the higher affinity must also have a high activity, and vice versa, in order that similar rates of hydrolysis be observed. Conversely, when two compounds have the same affinities, differences in their rates of hydrolysis will reflect differences in relative activities. The high affinity of the L- β -methyl isomer, due possibly to an increase in charge on the ester oxygen caused by electron donation from the methyl group, coupled with its low rate of hydrolysis, indicates that fold formation is essential for activity but not for affinity. The electronic influence will be the same in both β enantiomers, and therefore the low affinity of the $D-\beta$ -methyl isomer is probably due to steric interference from the methyl group which in this configuration prevents bond formation between the glutamic acid residue and the ester oxygen.

The most active substrate used in the present study was not SCh_2 but L-(-)- α -MeSCh_2. An analogous increase in the rate of hydrolysis upon L- α -methyl substitution of BuCh was not observed by Mitchard,¹⁵ although in inhibitor studies using the α - and β -methyl analogs of *p*-aminobenzoylcholine, L-*p*-aminobenzoyl- α methylcholine was shown to be a more potent competitive inhibitor of PChE than was *p*-aminobenzoylcholine. The D- α -methyl derivative has a high affinity and correspondingly low rate of hydrolysis, the L- α isomer has a lower affinity and is hydrolyzed rapidly, and SCh₂ has a high affinity and a comparatively high rate of hydrolysis; the activity of the enzyme toward SCh₂ and L- α -MeSCh₂ must therefore be considerably higher than toward D- α -MeSCh₂. It can be seen that interpretation of structure-activity relationships must be undertaken with great care as the maximum velocity values observed are not necessarily indicative of the more fundamental affinity and activity values. The difference in the affinities of the α -methyl isomers will be due to (i) steric interference and (ii) differences in interaction with the hydrophobic site because the α -methyl group is too far from the ester group to exert any significant electronic influence.

It has been found¹⁴ that β -methyl substitution reduces the pharmacological activity much more than does α -methyl substitution. The observation made during the present study that the rates of enzymic hydrolysis follow the same pattern as the pharmacological results indicate that similarities exist between the receptor site for these compounds and the active site of PChE. The K_i values, however, do not follow this pattern, and no definite conclusion can be reached concerning the nature of these similarities. Although the results obtained in the present study do not show the good correlation between inhibitor and hydrolytic studies which was observed by Mitchard,¹⁵ they do appear to follow the same general pattern, indicating that application of the induced-fit theory to PChE is probably valid.

Acknowledgments.—Receipt of a scholarship from the Department for Scientific and Industrial Research is gratefully acknowledged by Caroline L. Vaughan. Appreciation is given to Dr. J. W. Clitherow for his advice during the initial stages of this work.

Sympathetic Nervous System Blocking Agents. V. Derivatives of Isobutyl-, t-Butyl-, and Neopentylguanidine^{1,2}

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Guanidines prepared from simple *n*-alkylamines fail to show adrenergic neurone blocking activity, as determined by the effect on the cat nictitating membrane, while guanidines such as *t*-butylguanidine sulfate and neopentylguanidine sulfate are active. *t*-Octylguanidine hydrochloride (Table I, 5) proved to be the most active member of the alkylguanidine series. It was subjected to extensive pharmacological evaluation and clinical trial. Substitution of one or two methyl groups on the α or β carbons of dialkylaminoalkylguanidines was also investigated. 2-Hexamethyleniminoisobutylguanidine sulfate (20) caused a remarkably long blockade of the sympathetic nervous system.

Mecanylamine³ (I) and pempidine⁴ (II) were the first substances found to possess ganglionic blocking activity which were not quaternary ammonium salts.

(1) Paper IV: J. H. Short and T. D. Darby, J. Med. Chem., 11, 848 (1968).

(2) Presented before the Division of Medicinal Chemistry at the 155th National Meeting of the American Chemical Society, San Francisco, Calif., April 1968.

(3) G. A. Stein, M. Sletzinger, H. Arnold, D. Reinhold, W. Gaines, and K. Pfister, III, J. Am. Chem. Soc., **78**, 1514 (1956).

(4) A. Spinks and E. H. P. Young, *Nature*, **181**, 1397 (1958); G. E. Lee, W. R. Wragg, S. J. Corne, N. D. Edge, and H. W. Reading, *ibid.*, **181**, 1717 (1958).

Both are *t*-carbinamines, that is, the nitrogen atom of each is attached to a carbon atom containing three alkyl substituents. This hydrocarbon bulk which surrounds the nitrogen may be of great significance as far as the activity of I and II are concerned and sug-



gested to us the idea of synthesizing guanidines (VI, VIII) from "bulky" amines such as 2-dialkylaminoisobutylamines (V, $R^1 = R_2N$) and derivatives of neopentylamine (V, $R^1 = CH_3$, R_2NCH_2) and *t*-butylamine (VII, $R^1 = R_2N$).

Very few guanidines have been reported which possess a *t*-carbinamine moiety. Robertson, Biel, and Di-Pierro⁵ investigated a series of compounds related to pempidine and also containing the guanidine group of which III is a typical example.



After our work was completed Rand and Wilson⁶ reported their studies with several derivatives of 2cyclohexylaminoethylguanidine as sympathetic nervous system blocking agents. Included were the α -methyl (IVa), the β -methyl (IVb), and the β , β -dimethyl (IVc) derivatives. They found IVa and IVb to be slightly more active than 2-cyclohexylaminoethylguanidine while IVc was slightly less active.

Chemistry.—We first prepared several alkylguanidines, both unbranched and branched chained. These included *n*-butylguanidine sulfate, *n*-octylguanidine sulfate, *t*-butylguanidine sulfate (VIII, $\mathbb{R}^1 = \mathbb{R}^2 = \mathbb{H}$), *t*-octylguanidine (2,4,4-trimethyl-2-pentylguanidine) hydrochloride (VIII, $\mathbb{R}^1 = (CH_3)_3\mathbb{C}$; $\mathbb{R}^2 = \mathbb{H}$), neopentylguanidine sulfate (VI, $\mathbb{R}^1 = CH_3$; $\mathbb{R}^2 = \mathbb{H}$), 3,3-dimethyl-*n*-butylguanidine sulfate, 3,5,5-trimethyl*n*-hexylguanidine sulfate, and 2,2-diethyl-*n*-butylguanidine sulfate. In addition, 2-phenylisobutylguanidine sulfate (VI, $\mathbb{R}^1 = C_6H_5$; $\mathbb{R}^2 = \mathbb{H}$), *p*-chlorophenyl*t*-butylguanidine hydrochloride (VIII, $\mathbb{R}^1 = p$ -Cl- C_6H_4 ; $\mathbb{R}^2 = \mathbb{H}$), 1-benzyl-3-(*t*-butyl)guanidine hydro-

Method A

 $\begin{array}{c} \mathrm{CH}_{3} & \mathrm{NH} \\ | & | \\ \mathrm{R}^{1}\mathrm{CCH}_{2}\mathrm{NH}_{2} + \mathrm{CH}_{3}\mathrm{SCNHR}^{2} \cdot 0.5\mathrm{H}_{2}\mathrm{SO}_{4} \text{ (or HCl)} \longrightarrow \\ | \\ \mathrm{CH}_{3} \end{array}$



Method B

 $\begin{array}{c} \operatorname{CH}_{3} \\ \downarrow \\ \operatorname{R}^{4}\operatorname{CH}_{2}\operatorname{CNH}_{2} \cdot 0.5\operatorname{H}_{2}\operatorname{SO}_{4} \text{ (or HCl)} + \operatorname{NCNHR}^{2} \longrightarrow \\ \downarrow \\ \operatorname{CH}_{3} \\ \operatorname{VII} \end{array}$

$$\begin{array}{c} \mathrm{CH}_3 \mathrm{NH} \\ \downarrow \\ \mathrm{R}^{1}\mathrm{CH}_2\mathrm{CNH}\mathrm{CNH}\mathrm{R}^{2} \cdot 0.5\mathrm{H}_2\mathrm{SO}_4 \text{ (or HCl)} \\ \downarrow \\ \mathrm{CH}_3 \\ \mathrm{VIII} \end{array}$$

(5) J. E. Robertson, J. H. Biel, and F. DiPierro, J. Med. Chem., 6, 381 (1963).

(6) M. J. Rand and J. Wilson, European J. Pharmacol., 1, 200 (1967).

chloride (VIII, $R^1 = H$; $R^2 = C_6H_5CH_2$), and 1-methyl-3-(*t*-octyl)guanidine hydrochloride (VIII, $R^1 = (CH_3)_3C$; $R^2 = CH_3$) were prepared.

The guanidines possessing a methylene group adjacent to the nitrogen atom could be prepared from the appropriate amine and 2-methyl-2-thiopseudourea sulfate (method A). The unbranched amines and those amines with an ethylene group adjacent to the nitrogen atom (branching at the γ carbon) formed guanidines at room temperature, while neopentylamine (V, R⁴ = CH₃) and the other amines with branching at the β carbon formed the desired guanidines only at higher temperatures.

Method A was unsuccessful with *t*-butylamine (VII, $R^1 = H$) and related compounds in which branching occurs at the α carbon. Tsuji and Ueda⁷ had previously commented on the failure of *t*-butylamine, dimethylamino-*t*-butylamine, and piperidino-*t*-butylamine to react with 2-methyl-2-thiopseudourea sulfate to form the corresponding guanidines.

Variations of method A employing 2-methylpseudourea sulfate (or hydrochloride), 2,5-dimethyl-1guanylpyrazole hydrochloride,⁸ and 1-chloroformamidine hydrochloride⁹ were uniformly unsuccessful with *t*-butylamine and its derivatives (VII). These guanidines (VIII) were prepared by allowing the amine hydrochlorides (monoamines) to react with cyanamide (method B). With the diamines best results were achieved by dissolving the amine in water, adding only 1 equiv of HCl, and allowing the "monohydrochloride" to react with cyanamide.

Neopentylguanidine sulfate (VI, $R^1 = CH_3$; $R^2 = H$), *t*-butylguanidine sulfate (VIII, $R^1 = R^2 = H$), and *t*-octylguanidine hydrochloride (VIII, $R^1 = (CH_3)_3$ -C; $R^2 = H$) showed significant activity, while *n*-butylguanidine sulfate and *n*-octylguanidine sulfate were inactive (see Structure-Activity Relationships section). These results encouraged us to extend this line of investigation to the 2-dialkylaminoethylguanidine series.

Earlier we found 2-diethylaminoethylguanidine sulfate (IX, $R^1 = R^2 = R^3 = R^4 = H$) to be active,⁸ and we decided to investigate the effect on activity of substitution of methyl groups on the ethylene chain. Similar homologs of hexamethyleniminoethylguanidine sulfate (X, $R^1 = R^2 = R^3 = R^4 = H$), also known to be active,¹⁰ were prepared.



2-Diethylamino-*n*-propylguanidine sulfate (IX, $R^1 = CH_3$; $R^2 = R^3 = R^4 = H$) and 2-diethylamino-1-

⁽⁷⁾ T. Tsuji and T. Ueda, Chem. Pharm. Bull. (Tokyo), 12, 946 (1964).
(8) J. H. Short, U. Biermacher, D. A. Dunnigan, and T. D. Leth, J. Med. Chem., 6, 275 (1963).

⁽⁹⁾ The compound was prepared in the manner described in the Experimental Section for the t-butyl homolog. T. B. Johnson and J. M. Sprague, J. Am. Chem. Soc., **61**, 176 (1939), reported mp 182-183°.

⁽¹⁰⁾ R. P. Mull. M. E. Egbert, and M. R. Dapero, J. Org. Chem., 25, 1953 (1960).

methylethylguanidine sulfate (IX, $R^2 = CH_3$; $R^1 = R^3 = R^4 = H$) were prepared by methods A and B, respectively. The amine required for the latter guanidine was prepared by reductive alkylation of ammonia with diethylaminoacetone.

Diethylamino-t-butylguanidine dihydrochloride (IX, $R^1 = R^3 = H$; $R^2 = R^4 = CH_3$) was prepared by method B, while method A was used to prepare the isomeric 2-diethylaminoisobutylguanidine sulfate (IX, $R^1 = R^3 = CH_3$; $R^2 = R^4 = H$).

Diethylamino-*t*-butylamine (VII, $R^1 = (C_2H_5)_2N$) was prepared by reduction of the nitro compound obtained by allowing diethylamine to react with 2-methyl-2-nitro-1-propanol. The diamine was also obtained when diethylamine was allowed to react with 2,2-dimethylaziridine.

Reaction between acetone cyanohydrin and diethylamine gave 2-diethylaminoisobutyronitrile. Attempts to reduce the latter with LiAlH₄ and by catalytic means gave diethylisopropylamine as the only isolable product. The nitrile, however, was hydrolyzed successfully to the corresponding amide and reduction of the latter with diborane gave the desired 2-diethylaminoisobutylamine (V, $\mathbb{R}^1 = (\mathbb{C}_2 \mathbb{H}_5)_2 \mathbb{N}$). LiAlH₄ also effected reduction of the amide, but in much lower yield.

Hexamethylenimino-t-butylamine

VII,
$$R^1 = (CH_2)_6 N$$

and 2-hexamethyleniminoisobutylamine

$$V, \mathbf{R}^1 = (CH_2)_6 N$$

were prepared in the same manner as the corresponding diethyl analogs. The amines, in turn, were converted to hexamethylenimino-*t*-butylguanidine dihydrochloride (X, $R^1 = R^3 = H$; $R^2 = R^4 = CH_3$) and 2-hexamethyleniminoisobutylguanidine sulfate (X, $R^1 =$ $R^3 = CH_3$; $R^2 = R^4 = H$) by methods B and A, respectively.

The reaction between N-methylpiperazine and acetone cyanohydrin gave the expected nitrile, which was hydrolyzed to the corresponding amide, and the latter then was reduced to 2-(4-methylpiperazino)isobutylamine.

$$V, R^1 = CH_3 N$$

The amine was transformed in the usual manner to 2-(4-methylpiperazino)isobutylguanidine sulfate.

$$VI, R^1 = CH_3 N N; R^2 = H$$

Diethylaminoneopentylamine was prepared by reductive alkylation of NH₃ with diethylaminopivaldehyde. Method A was used to convert the amine into diethylaminoneopentylguanidine sulfate (VI, $R^1 = (C_2H_5)_2NCH_2$; $R^2 = H$).

In our earlier work⁸ we had observed that replacement of the guanidine hydrogens of 2-diethylaminoethylguanidine with alkyl groups led to less active or inactive compounds. Nevertheless we prepared several 1,3-disubstituted guanidines in which one substituent was a dialkylaminoalkyl group and the other was a branched alkyl group. 1-(t-Butyl)-3-(2-diethylaminoethyl)guanidine dihydrochloride was obtained by the reaction of diethylaminoethylamine with either tbutylcyanamide (method B) or with 3-(t-butyl)-2methyl-2-thiopseudourea hydrochloride (method A). The same amine was allowed to react with t-octylcyanamide or with 2-methyl-3-(t-octyl)-2-thiopseudourea hydrochloride to give 1-(2-diethylaminoethyl)-3-(t-octyl)guanidine dihydrochloride.

Method B is the more practical method for obtaining the last two guanidines since it is a one-step procedure. Preparation of these guanidines by method A involves a sequence of three reactions, but was of interest from a chemical standpoint. HCl could be added to t-butyleyanamide and t-octyleyanamide to give, respectively, N-(t-butyl)-1-chloroformamidine hydrochloride and 1-chloro-N-(t-octyl)formamidine hydrochloride. Neither of the latter compounds gave the desired guanidines when allowed to react with diethylaminoethylamine. They did, however, react with methanethiol or ethanethiol to give the expected thiopseudoureas, and these underwent reaction in the normal manner, as noted above, to give the desired guanidines, identical in every respect with the substances obtained directly from the amine and the cyanamides.

2-Hexamethyleniminoisobutylamine was allowed to react with t-butylcyanamide and t-octylcyanamide to give, respectively, 1-(t-butyl)-3-(2-hexamethyleniminoisobutyl)guanidine dihydrochloride

VI,
$$R^1 = (CH_2)_6 N; R^2 = (CH_3)_3 C$$

and 1-(2-hexamethyleniminois obutyl)-3-(t-octyl)guanidine dihydrochloride.

VI,
$$\mathbf{R}^1 = (CH_2)_6 \mathbf{N}; \mathbf{R}^2 = (CH_3)_3 CCH_2 C(CH_3)_2$$

The guanidine from 1-(2-aminoethyl)-4-methylpiperazine was found to be active,⁸ and that amine was also allowed to react with *t*-butylcyanamide to give the expected disubstituted guanidine.

The guanidines are tabulated in Table I.

Pharmacology.—The guanidines were examined by the oral route for their effect on the cat nictitating membrane in the manner previously described.^{1,8} The results are compiled in Table I.

2-Hexamethyleniminoisobutylguanidine sulfate (Table I, **20**), when screened at 30 mg/kg, caused a ++ prolapse lasting for 8-9 days. Decreasing the dose to 20 mg/kg did not decrease the duration of the prolapse. It is the only compound we have ever tested that caused a prolapse lasting more than 7 days. It proved to be effective in lowering blood pressure in both renal and neurogenic hypertensive dogs, but it was not effective in doses smaller than 10 mg/kg. Since 2-(2-methylthioethylamino)ethylguanidine sulfate caused a significant decrease in the blood pressure of hypertensive dogs at 2 mg/kg,¹ the latter was chosen for further pharmacological evaluation and clinical trial.

t-Octylguanidine hydrochloride (5) was one of the few compounds we have screened which, like guanethidine, causes a +++ prolapse at 30 mg/kg, and, further, it is the only compound we have observed to have a significant effect at 2 mg/kg. It proved to be

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5 H ₃ SQ | 5 H ₃ S0 ₄
C C C C 20
C C 20
C C 20
5 H ₃ S0 ₄
5 H ₃ S0 ⁴
5 H ₃ | 5 H ₃ SQ ₄
C C C C 20
C C 20
C C 20
5 H ₃ SQ ₄
5 H ₃ | 5 H ₃ S0 ₄
Cl Cl 20
Cl 21
Cl 20
5 H ₃ S0 ₄
5 H ₃ S0 ⁴
5 H ₃ S0 | 5 H ₃ S0 ₄
C C C 20
C C 20
C C 20
5 H ₃ S0 ₄
5 H ₃ S0 ⁴
5 H ₃ S | 5 H ₅ S0 ₄
C C C 20
C C 20
C C 20
5 H ₅ S0 ₄
5 H ₅ S0 ⁴
5 H ₅ S |
| | | $\mathrm{C}_9\mathrm{H}_{21}\mathrm{N}_3\cdot 0.5\mathrm{H}_2\mathrm{SO}_4$ | C ₅ H ₁₃ N ₃ ·0.5H ₂ SO ₄ | | | CaHaNa, HCI
CaHaNa, HCI | $C_{12}H_{19}N_3 \cdot HCl$
$C_9H_{21}N_3 \cdot HCl$ | $C_{12}H_{10}N_3$. HCl
$C_9H_{21}N_3$. HCl | C ₁₂ H ₁₀ N ₃ . HCl
C ₉ H ₂ N ₃ . HCl
C ₉ H ₂ N ₃ . HCl | C ₁₂ H ₁₀ N ₃ · HCl
C ₉ H ₂₁ N ₃ · HCl
C ₁₀ H ₂₃ N ₃ · HCl | C ₁₂ H ₁₀ N ₃ · HCl
C ₉ H ₂₁ N ₃ · HCl
C ₁₀ H ₂₁ N ₃ · HCl
C ₁₀ H ₁₅ N ₃ · HCl
C ₆ H ₁₅ N ₃ · 0. 5H ₂ SO ₄ | C ₁₂ H ₁₀ N ₃ , HCl
C ₉ H ₂₁ N ₃ , HCl
C ₁₀ H ₂₁ N ₃ , HCl
C ₁₀ H ₂₁ N ₃ , 0, 5H ₂ SO ₄
C ₄ H ₁₅ N ₃ , 0, 5H ₂ SO ₄ | C ₁₂ H ₁₀ N ₃ · HCl
C ₉ H ₂₁ N ₃ · HCl
C ₁₀ H ₂₁ N ₃ · HCl
C ₁₀ H ₂₁ N ₃ · 0. 5H ₉ SO ₄
C ₇ H ₁₇ N ₃ · 0. 5H ₉ SO ₄
C ₅ H ₆₁ N ₃ · 0. 5H ₉ SO ₄ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{21}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot 0.5H_3SO_4\\ C_{6}H_{15}N_3\cdot 0.5H_2SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_2SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_2SO_4\\ \end{array}$ | $\begin{array}{c} C_{12}H_{11}N_{3}\cdot HCl\\ C_{3}H_{21}N_{3}\cdot HCl\\ C_{10}H_{23}N_{3}\cdot HCl\\ C_{16}H_{15}N_{3}\cdot 0.5H_{2}SO_{4}\\ C_{7}H_{17}N_{3}\cdot 0.5H_{2}SO_{4}\\ C_{9}H_{21}N_{3}\cdot 0.5H_{2}SO_{4}\\ C_{10}H_{23}N_{3}\cdot 0.5H_{2}SO_{4}\\ \end{array}$ | C ₁₂ H ₁₀ N ₃ . HCl
C ₉ H ₂₁ N ₃ . HCl
C ₁₆ H ₁₂ N ₃ . HCl
C ₆ H ₁₅ N ₃ . 0. 5H ₂ SO ₄
C ₇ H ₁₇ N ₃ . 0. 5H ₂ SO ₄
C ₉ H ₂₁ N ₃ . 0. 5H ₂ SO ₄
C ₁₀ H ₂₃ N ₃ . 0. 5H ₂ SO ₄
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{21}N_3\cdot HCl\\ C_{4}H_{21}N_3\cdot HCl\\ C_{6}H_{15}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{23}N_5\cdot 0.5H_{2}SO_4\\ C_{11}H_{7}N_5\cdot 0.5H_{2}SO_4\\ C_{11}H_{6}CN_3\cdot HCl\\ C_{11}H_{6}CN_3\cdot HCl\\$ | $\begin{array}{c} C_{12}H_{10}N_3\cdot HCl\\ C_{9}H_{21}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{6}H_{15}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{7}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{2}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{2}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{2}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{2}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{2}N_3\cdot 0.5H_{2}N_3\cdot 0.5H_{2}N_3\cdot 0.5H_{2}N_3\\ C_{10}H_{2}N_3\cdot 0.5H_{2}N_3\cdot 0.5H_{2}N_3\\ C_{10}H_{2}N_3\cdot 0.5H_{2}N_3\cdot 0.5H_{2}N_3\\ C_{10}H_{2}N_3\cdot 0.5H_{2}N_3\cdot 0.5H_{2}N_3+0.5H_{2}N$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{11}H_{22}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{6}H_{15}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{17}N_3+0.5H_{17}N_3+0.5H_{17}N_3+0.5H_{17}N_3+0.5H_{17}N_3+0.5H$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{14}H_{21}N_3\cdot HCl\\ C_{16}H_{12}N_3\cdot HCl\\ C_{6}H_{15}N_3\cdot 0.5H_{5}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{5}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{5}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{5}SO_4\\ C_{10}H_{12}N_3\cdot 0.5H_{5}SO_4\\ C_{10}H_{12}N_3\cdot 0.5H_{5}SO_4\\ C_{10}H_{24}N_4\cdot 0.5H_{5}SO_4\\ C_{10}H_{24}N_4\cdot 0.5H_{5}SO_4\\ \end{array}$
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{11}H_{21}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{12}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{3}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{3}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{23}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{10}H_{24}N_4\cdot 0.5H_{2}SO_4\\ \end{array}$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{21}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot 0.5H_3SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{10}H_{12}N_3\cdot 0.5H_3SO_6\\ C_{11}H_{17}N_3\cdot 0.5H_3SO_6\\ C_{11}H_{12}N_3\cdot 0.5H_3SO_6\\ C_{10}H_{24}N_4\cdot 0.5H_3SO_6\\ \end{array}$
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{21}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot 0.5H_3SO_4\\ C_{6}H_{17}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{10}H_{17}N_3\cdot 0.5H_3SO_4\\ C_{10}H_{24}N_1\cdot 0.5H_3SO_4\\ C_{10}H_{24}N_1\cdot 0.5H_3SO_4\\ C_{10}H_{24}N_1\cdot 0.5H_3SO_4\\ \end{array}$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{22}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot 0.5H_3SO_4\\ C_{6}H_{11}N_3\cdot 0.5H_3SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_3SO_4\\ C_{11}H_{12}N_3\cdot 0.5H_3SO_4\\ C_{11}H_{12}N_3\cdot 0.5H_3SO_4\\ C_{11}H_{21}N_4\cdot 0.5H_3SO_4\\ C_{3}H_{20}N_4\cdot H_3SO_4\\ C_{3}H_{20}N_4\cdot H_3SO_4\\ C_{3}H_{20}N_4\cdot H_3SO_4\\ \end{array}$

 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{22}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot 0.5H_3SO_4\\ C_{6}H_{11}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_3SO_4\\ C_{11}H_1CN_3\cdot 0.5H_3SO_4\\ C_{11}H_{12}N_4\cdot 0.5H_2SO_4\\ C_{11}H_{21}N_1\cdot 0.5H_3SO_4\\ C_{11}H_{21}N_1\cdot 0.5H_3SO_4\\ C_{11}H_{21}N_1\cdot 0.5H_3SO_4\\ C_{11}H_{22}N_4\cdot H_2SO_4\\ C_{2}H_{20}N_1\cdot H_2SO_4\\ C_{9}H_{22}N_4\cdot H_{2}SO_4\\ \end{array}$
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{22}N_3\cdot HCl\\ C_{4}H_{25}N_3\cdot 0.5H_{2}SO_4\\ C_{6}H_{15}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{23}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_8\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_8\cdot 0.5H_{2}SO_4\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{6}H_{29}N_4\cdot H_{2}SO_4\\ C_{8}H_{29}N_4\cdot H_{2}SO_4\\ C_{8}H_{29}N_4\cdot H_{2}SO_4\\ C_{9}H_{22}N_4\cdot H_{2}SO_4\\ \end{array}$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{22}N_3\cdot HCl\\ C_{4}H_{25}N_3\cdot 0.5H_{2}SO_4\\ C_{6}H_{15}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{23}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_8\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_8\cdot 0.5H_{2}SO_4\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot H_{2}SO_4\\ C_{3}H_{29}N_4\cdot H_{2}SO_4\\ C_{3}H_{29}N_4\cdot H_{2}SO_4\\ C_{9}H_{22}N_4\cdot H_{2}SO_4\\ \end{array}$
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{22}N_3\cdot HCl\\ C_{4}H_{25}N_3\cdot 0.5H_{2}SO_4\\ C_{6}H_{12}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{23}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_5\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_5\cdot 0.5H_{2}SO_4\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{6}H_{29}N_4\cdot H_{2}SO_4\\ C_{6}H_{29}N_4\cdot H_{2}SO_4\\ C_{9}H_{22}N_4\cdot H_{2}SO_1\\ C_{9}H_{22}N_4\cdot H_{2}SO_1\\ C_{9}H_{22}N_4\cdot 2HCl\\ C_{9}H_{22}N_4\cdot 2HCl\\ \end{array}$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{22}N_3\cdot HCl\\ C_{4}H_{25}N_3\cdot 0.5H_{2}SO_4\\ C_{6}H_{12}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{6}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{9}H_{29}N_4\cdot H_{2}SO_4\\ C_{9}H_{29}N_4\cdot 2HCI\\ C_{9}H_{29}N_4\cdot 2HCI\\ \end{array}$
 | C ₁₂ H ₁₀ N ₃ ·HCl
C ₉ H ₁₂ N ₃ ·HCl
C ₁₀ H ₂₃ N ₃ ·0.5H ₅ SO ₄
C ₆ H ₁₄ N ₃ ·0.5H ₅ SO ₄
C ₇ H ₁₇ N ₃ ·0.5H ₅ SO ₄
C ₉ H ₂₁ N ₄ ·0.5H ₅ SO ₄
C ₁₀ H ₄₇ N ₃ ·0.5H ₅ SO ₄
C ₁₀ H ₂₁ N ₁ ·0.5H ₅ SO ₄
C ₁₀ H ₂₁ N ₁ ·0.5H ₅ SO ₄
C ₁₁ H ₂₂ N ₄ ·H ₂ SO ₄
C ₃ H ₂₂ N ₄ ·H ₂ SO ₄
C ₃ H ₂₂ N ₄ ·H ₂ SO ₄
C ₃ H ₂₂ N ₄ ·H ₂ SO ₄
C ₃ H ₂₂ N ₄ ·H ₂ SO ₄
C ₃ H ₂₂ N ₄ ·H ₂ SO ₄
C ₃ H ₂₂ N ₄ ·2HCl
C ₁₁ H ₃₂ N ₄ ·2HCl | C ₁₂ H ₁₀ N ₃ ·HCl
C ₉ H ₁₂ N ₃ ·HCl
C ₁₀ H ₂₃ N ₃ ·0.5H ₃ SO ₄
C ₆ H ₁₃ N ₃ ·0.5H ₃ SO ₄
C ₇ H ₁₇ N ₃ ·0.5H ₃ SO ₄
C ₉ H ₂₁ N ₄ ·0.5H ₃ SO ₄
C ₁₀ H ₁₇ N ₃ ·0.5H ₃ SO ₄
C ₁₀ H ₂₁ N ₁ ·0.5H ₃ SO ₄
C ₁₁ H ₆ ClN ₃ ·HCl
C ₁₀ H ₂₁ N ₁ ·0.5H ₃ SO ₄
C ₃ H ₂₀ N ₁ ·1.9SO ₄
C ₃ H ₂₀ N ₁ ·1.9SO ₄
C ₃ H ₂₂ N ₄ ·1H ₃ SO ₄
C ₃ H ₂₂ N ₄ ·1H ₃ SO ₄
C ₃ H ₂₂ N ₄ ·2HCl
C ₁₁ H ₃₈ N ₄ ·2HCl
C ₁₁ H ₃₈ N ₄ ·2HCl
C ₁₁ H ₃₈ N ₄ ·2HCl
C ₁₃ H ₃₁ N ₄ ·2HCl
C ₁₃ H ₃₁ N ₄ ·2HCl
 | C ₁₂ H ₁₀ N ₃ ·HCl
C ₉ H ₁₂ N ₃ ·HCl
C ₁₆ H ₂₃ N ₃ ·0.5H ₃ SO ₄
C ₆ H ₁₇ N ₃ ·0.5H ₃ SO ₄
C ₇ H ₁₇ N ₃ ·0.5H ₃ SO ₄
C ₉ H ₂₁ N ₄ ·0.5H ₃ SO ₄
C ₁₀ H ₂₁ N ₄ ·0.5H ₃ SO ₄
C ₁₀ H ₂₁ N ₄ ·0.5H ₃ SO ₄
C ₁₀ H ₂₁ N ₄ ·0.5H ₃ SO ₄
C ₁₀ H ₂₁ N ₄ ·0.5H ₃ SO ₄
C ₈ H ₂₉ N ₄ ·H ₃ SO ₄
C ₈ H ₂₉ N ₄ ·H ₃ SO ₄
C ₉ H ₂₂ N ₄ ·H ₃ SO ₄
C ₉ H ₂₂ N ₄ ·H ₃ SO ₄
C ₁₁ H ₃₆ N ₄ ·2HCl
C ₁₁ H ₃₆ N ₄ ·2HCl | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{2}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{3}H_{31}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{16}ClN_3\cdot HCl\\ C_{11}H_{16}ClN_3\cdot HCl\\ C_{10}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{29}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_4\\ C_{4}H_{24}N_4\cdot 2HCl\\ C_{4}H_{34}N_4\cdot 2HCl\\ C_{4}H_{34}N_4\cdot 2HCl\\ C_{4}H_{34}N_4\cdot 2HCl\\ C_{4}H_{34}N_4\cdot 2HCl\\ C_{4}H_{34}N_4\cdot 0.5H_{2}SO_4\\ C_{4}H_{34}N_4\cdot 0.5H_{2}SO_4\\ \end{array}$
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{12}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{21}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{21}N_4\cdot 0.5H_{2}SO_4\\ C_{10}H_{20}N_4\cdot HCl\\ C_{10}H_{20}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{4}H_{34}N_4\cdot 2HCl\\ C_{11}H_{34}N_4\cdot 2HCl\\ C_{11}H_{34}N_4\cdot 0.5H_{2}SO_4\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{11}H_{24}N_4+ 0.5H_{2}SO_4\\ C_{11}H_{24}N$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{3}H_{12}N_3\cdot HCl\\ C_{4}H_{15}N_3\cdot 05H_{5}SO_4\\ C_{6}H_{15}N_3\cdot 05H_{5}SO_4\\ C_{7}H_{17}N_3\cdot 05H_{5}SO_4\\ C_{9}H_{21}N_3\cdot 05H_{5}SO_4\\ C_{9}H_{21}N_3\cdot 05H_{5}SO_4\\ C_{11}H_{10}N_3\cdot 05H_{5}SO_4\\ C_{11}H_{10}N_3\cdot 05H_{5}SO_4\\ C_{11}H_{10}N_4\cdot 05H_{5}SO_4\\ C_{9}H_{20}N_4\cdot 1H_{5}O_4\\ C_{9}H_{20}N_4\cdot 1H_{5}O_4\\ C_{9}H_{20}N_4\cdot 1H_{5}O_4\\ C_{9}H_{20}N_4\cdot 1H_{5}O_4\\ C_{11}H_{5}N_4\cdot 2HCl\\ C_{11}H_{5}N_4\cdot 2HCl\\ C_{11}H_{5}N_4\cdot 2HCl\\ C_{11}H_{5}N_4\cdot 0.5H_{5}SO_4\\ C_{11}H_{5}N_4+ 0.5H_{5}SO_4\\ C_{11}H_{5}$
 | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{23}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{12}N_3\cdot 0.5H_{2}SO_4\\ C_{7}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{9}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{17}N_8\cdot 0.5H_{2}SO_4\\ C_{10}H_{10}N_4\cdot 0.5H_{2}SO_4\\ C_{10}H_{24}N_4\cdot 0.5H_{2}SO_4\\ C_{10}H_{20}N_4\cdot HCl\\ C_{10}H_{20}N_4\cdot H_{2}SO_4\\ C_{2}H_{20}N_4\cdot H_{2}SO_4\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{10}H_{24}N_4\cdot 2HCl\\ C_{11}H_{24}N_4\cdot 2HCl\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ \end{array}$ | $\begin{array}{c} C_{12}H_{11}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot HCl\\ C_{10}H_{22}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{12}N_3\cdot 0.5H_{2}SO_4\\ C_{11}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{21}H_{17}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{21}N_3\cdot 0.5H_{2}SO_4\\ C_{10}H_{21}N_4\cdot 0.5H_{2}SO_4\\ C_{10}H_{20}N_4\cdot HCl\\ C_{10}H_{20}N_4\cdot H_{2}SO_4\\ C_{20}H_{20}N_4\cdot H_{2}SO_4\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_4\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{3}H_{20}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ C_{11}H_{24}N_4\cdot 0.5H_{2}SO_1\\ \end{array}$
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| | n-C ₄ H ₉ | $n-\mathrm{C_8H_{17}}$ | <i>t</i> -C ₄ H ₉ | нот | 1-C4III9 | /~H~// | l-C ₈ H ₁₇ / | <i>L</i> -C ₈ H ₁₇ / | <i>t</i> -C ₈ H ₁₇ / | <i>t</i> -C _s H ₁₇ /
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 | μ-C₃H₁₇/ μ-C₃H₁₇ (CH₃)₆CCH₂ (CH₃)₆CCH₄ (C₂H₆)₆CCH₄ (C₂H₆)₂CCH₄ (C₂H₆)₂NCH₄ (C₂H₆)₂ | μ-C₃H₁₇/ μ-C₃H₁₇ (CH₃)₆CCH₃ (CH₃)₆CCH₄ (C₃H₆)₅CCH₄ (C₃H₆)₅CCH₄ (C₃H₆)₅NCH₅ (C₃H₆)₂NCH₅ (C₃H₆)₂ (C₃H₆)₃ (C₃H₆)₃ | 4-C ₃ H ₁₇ /
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(C ₂ H ₅) ₂ NCH ₅
(C ₂ H | μ-C₃H₁₇/ μ-C₃H₁₇ μ-C₃H₁₇ (CH₃)₆CCH₄ (CH₃)₈CCH₄ (C₃H₆)₈CCH₄ (C₃H₆)₈CCH₄ (C₃H₆)₈CCH₄ (C₃H₆)₈CCH₄ (C₃H₆)₈DCCH₄ (C₃H₆)₈DNCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₃H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₂NCH₄ (C₄H₆)₃ | $^{h}C_{3}H_{1T}^{r}$
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in in the and the one-fourth of the eye covered by the membrane; ++, one-half, +++, three-quarters. ^a R. B. Fearing and S. W. Fox, J. Am. Chem. Soc.; **76**, 4382 (1954), reported mp 210–211^a. ^a This guani-dine was prepared by method B from t-butyleyanamide and NH,Cl and it was converted to the sulfate with Ag₂SO₄. ^a Prepared by method B from t-butyleyanamide or t-octyleyanamide and the ^a The reaction of t-octyleyanamide with NH₄Cl and with MeNH₃Cl is de-ⁱ The amine was obtained from HCl salt, [G. B. Bachman, H. B. j *p*-Chloro-*a*,*a*-dimethylphenethylamine was converted to the HCl salt, [G. B. Bachman, H. B. cyanamide. ^k The required amine was prepared by reductive alkylation of NH₃ with diethyl-M. Freifelder and Y. H. Ng, *J. Med. Chem.*, **8**, 122 (1965). ¹ The required amine was prepared ^d This guani-The t-octyleyanamide was prepared in these laboratories by Mr. Vincent Johnson The required amine, 1-(2-amino-^b The degree of prolapse is indicated as follows: " The reaction was run in *i*-BuOH. The solvent was removed **76**, 4382 (1954), reported mp 210–211°. by the reduction of the corresponding nitro compound (method E),¹⁸ and by the reaction between diethylamine and 2,2-dimethylaziridine (Experimental Section). manner of method B except that the solution was heated under reflux for 4 hr instead of at 180° . " $C_{4H}O_4$ is fumaric acid." The reaction was run in *i*-BuOH. The residual oil was dissolved in water. The solution was made alkaline with 50% NaOH; the free base was taken up in CHCl₃ and converted to the fumarate salt. Th ethyl)-4-methylpiperazine, has been described.³ ^a All compounds were analyzed for C, H, N. from t-octylamine (Rohm and Haas Co.) and CICN. A The cat receiving the 30-mg/kg dose suffered emesis which accounts for its being more effective at 15 mg/kg. E, EtOII-MesCO; F, EtOII-Skellvsolve B; G, H₂O-MeOII; H, EtOII; J, MeOII-MesCO; K, II₅O-EtOH. f t-C₈H_{IT} = 2,4,4-trimethyl-2-pentyl. scribed in detail by L. S. Luskin and J. H. Short, U. S. Patent 3,140,231 (July 7, 1964); Chem. Abstr., 61, 6927 (1964). O. Platau, J. Am. Chem. Soc., 76, 3972 (1954)] and the salt was allowed to react with cyanamide. aminopivaldehyde (method ${f F}$) and it has been described in a previous report from these laboratories: It has been described by W. M. Bruner, Ind. Eng. Chem., 41, 2860 (1949). Sufficient ethanol was added to form a homogeneous solution. ethyl)-4-methylpiperazine, has been e H₂O-Me₂CO; D, H₂O; Rohm and Haas Co. appropriate amine. Hass, and G. ů,

effective in hypertensive dogs, and was subjected to extensive pharmacological evaluation. 11

Clinical studies showed that 5 was less toxic and caused fewer side effects than guanethidine, but it did not lower blood pressure in hypertensive individuals with the same effectiveness and consistency as did guanethidine.

Structure-Activity Relationships.—The activity of *t*-butylguanidine sulfate (Table I, 3) and neopentylguanidine sulfate (7) contrasted strongly with the lack of activity seen with *n*-butylguanidine sulfate (1) and *n*-octylguanidine sulfate (2). A longer duration of activity was seen with 7 than with 3, indicating that branching at the β carbon might be more desirable than branching at the α carbon.

These first results encouraged us to proceed further with such branched-chain guanidines.

The failure of 3,3-dimethyl-*n*-butylguanidine sulfate (8) to exhibit the desired activity indicated that branching beyond the β carbon was not efficacious for activity. The inactivity of 3,5,5-trimethyl-*n*-hexylguanidine sulfate (10) confirmed this conclusion. This principle was confirmed further by finding that 2,2-diethyl-*n*-butylguanidine sulfate (9) was active. Therefore, further work was limited to preparation of guanidines possessing branching at either the α or β carbons.

t-Octylguanidine hydrochloride (5) proved to be very active (see Pharmacology section). 1-Methyl-3-(toctyl)guanidine hydrochloride (6) was somewhat less active. Reduction of activity in a disubstituted guanidine of this sort when compared with the monosubstituted homolog is in line with previous observations.^{8,12}

Two compounds containing aromatic groups, namely 2-phenylisobutylguanidine sulfate (11) and p-chlorophenyl-t-butylguanidine hydrochloride (12), proved to be inactive. In contrast, 2-(p-chlorophenyl)ethylguanidine hydrochloride¹² had previously been found tohave activity, but of short duration.

In the dialkylaminoalkylguanidine series 2-diethylamino-*n*-propylguanidine sulfate (14) and 2-diethylamino-1-methylethylguanidine sulfate (15) both proved to have substantial activity, but less than 2-diethylaminoethylguanidine sulfate.⁸ Diethylamino-*t*-butylguanidine dihydrochloride (17) proved to be about as active as 2-diethylaminoethylguanidine sulfate, while its isomer, 2-diethylaminoisobutylguanidine sulfate (16), proved to be considerably more active.

A homolog of 16 and 17, diethylaminoneopentylguanidine sulfate (13), was about as active as 17.

Hexamethylenimino-t-butylguanidine dihydrochloride (21) proved to have surprisingly little activity, but its isomer, 2-hexamethyleniminoisobutylguanidine sulfate (20), showed the longest duration of activity of any guanidine we have tested (see Pharmacology section).

The structure-activity relationships outlined here and in the previous papers in this series led us to predict that 2-(4-methylpiperazino)isobutylguanidine sulfate (25) might show exceptional activity. As often happens in medicinal chemistry our prediction proved wrong, since 25 failed to cause a prolapse at 30 mg/kg.

We found in our previous work with dialkylaminoalkylguanidines,⁸ as well as in the benzylguanidine

⁽¹¹⁾ H. G. Schoepke, H. D. Brondyk, L. H. Wiemeler, and J. L. Schmidt,

Arch. Intern. Pharmacodyn., 153, 185 (1965).
 (12) J. H. Short and T. D. Darby, J. Med. Chem., 10, 833 (1967).

series,¹² that one methyl group could be substituted on a guanidine nitrogen (to give 1,3-disubstituted guanidines) without losing activity (cf. 5 and 6), but incorporation of larger *n*-alkyl groups led to inactive compounds. We were interested, therefore, in determining if 1,3-disubstituted guanidines containing a dialkylaminoalkyl group and a branched-chain alkyl group such as a *t*-butyl or *t*-octyl group would possess activity. Five such compounds were prepared (18, 19, 22–24) and were found to be uniformly inactive confirming once again that monosubstituted guanidines are, in all cases, superior to di- and polysubstituted ones.

Testing of the compounds by the oral route raises the question whether a compound might fail to cause a prolapse, not because it is intrinsically inactive, but because it is not absorbed. The ratio of oral toxicity to intraperitoneal toxicity gives an indication of the degree of oral absorption. We have not determined the LD_{50} 's of the compounds described here, but routine toxicity data in mice can give us an approximate oralintraperitoneal ratio. For example, 1 caused no deaths (0/3) in mice at 50 mg/kg, 2/3 deaths at 75 mg/kg, and 3/3 at 100 mg/kg by the intraperitoneal route. By the oral route no deaths (0/3) occurred at 75 mg/kg while 3/3 died at 100 mg/kg. These data give an oralintraperitoneal ratio of about 2:1, indicating that 1 is well absorbed. This ratio is not typical, however. None of the other guanidines are as well absorbed. For **2** the ratio is about 10:1 (intraperitoneal, 0/3, 30 mg/kg; 1/3, 50 mg/kg; 3/3, 75 mg/kg; oral, 0/3, 300 mg/kg; 2/3, 500 mg/kg; 3/3, 750 mg/kg). The remaining inactive compounds all had oral-intraperitoneal toxicity ratios between 5:1 and 10:1 except for 18 which had a ratio of 20:1.

Ratios for the active compounds were comparable. For **3**, **5**, and **7** the ratios were 5:1 or less. Six active compounds (**6**, **14–17**, and **21**) had ratios between 6:1 and 10:1. Compounds **9** and **13** had ratios of about 15:1, while our most active compound, **20**, had a ratio of about 20:1.

The above information indicates that all the guanidines in this series are absorbed to some extent following oral administration, and that we are not likely to have missed a very active compound because of lack of absorption.

This work had led to the following conclusions: (1) alkyl substituents on C-1 or C-2 of simple alkylguanidines lead to active compounds; (2) methyl substituents at C-1 of dialkylaminoalkylguanidines have little effect on activity, while substituents at C-2 usually enhance activity; (3) incorporation of a second substituent (*t*butyl or *t*-octyl) gives inactive compounds.

Experimental Section¹³

N-(*t*-**Butyl**)-**1**-**chloroformamidine** Hydrochloride.—A solution of 49 g (0.5 mole) of *t*-butyleyanamide in 500 ml of dry Et₂O was stirred as 400 ml of 10% ethereal HCl was added during 2 hr. After standing overnight at room temperture a white solid was collected. The yield was 78 g (91%), mp 114-117°. Recrystallization from CHCl₃-THF gave thick colorless prisms melting at 115-117°, lit.¹⁴ mp 110-113°. **3**-(*t*-Butyl)-2-ethyl-2-thiopseudourea Hydrochloride.—A suspension of 85 g (0.5 mole) of N-(*t*-butyl)-1-chloroformamidine hydrochloride and 62 g (1.0 mole) of ethanethiol in 500 ml of 1,2-dimethoxyethane was heated under reflux for 8 hr. The solution was chilled to obtain 66.2 g of white solid, mp 130-132°. Concentrating and chilling the filtrate gave an additional 15 g of product, total yield 81.2 g (83%). A recrystallized portion (Me₂CO) melted at 131-132°. *Anal.* (C₇H₁₆N₂S·HCl) C, H, N.

1-Chloro-N-(*t*-octyl)formamidine Hydrochloride. *-t*-Octylcyanamide (31 g, 0.2 mole) was allowed to react with HCl in the manner described above for the *t*-butyl homolog. The yield of product was 40 g (88%), mp 123-126°. *Anal.* (C₈H₁₉ClN₂-HCl) C, H, N.

2-Methyl-3-(*t*-octyl)-2-thiopseudourea Hydrochloride.—A suspension of 113.6 g (0.5 mole) of 1-chloro-N-(*t*-octyl)formamidine hydrochloride in 750 ml of 1,2-dimethoxyethane and 48 g (1.0 mole) of methanethiol was heated under reflux for 6 hr. The solution was taken to dryness and the residue was dissolved in 200 ml of boiling acetone. The solution was chilled to obtain 64.6 g ($54C_{\rm c}$) of white, glistening leaflets, mp 145–147°. Recrystallization from EtOH–Me₂CO raised the melting point to 148.5–149°. Anal. (C₁₀H₂₂N₂S·HCl) C, H, N.

Preparation of Guanidines. Method A.—A solution of 0.1 mole of the amine and 14 g (0.05 mole) of 2-methyl-2-thiopseudourea sulfate in 25–50 ml of H₂O was heated under reflux for 2–18 hr. The solution was taken to dryness and the residue was crystallized from an appropriate solvent. In a few cases the guanidine precipitated on chilling the reaction solution. Some of the amines were not water soluble and sufficient EtOH was added to form a solution.¹⁶ When 3-(*l*-butyl)-2-ethyl- and 3-(*l*-octyl)-2-methyl-2-thiopseudourea hydrochlorides were used, 0.05 mole of the pseudourea was allowed to react as above with an equivalent amount of the amine in 100 ml of *i*-BuOH. The guanidines are described in Table I.

The use of N-(*t*-butyl)-1-chloroformamidine hydrochloride, and its *t*-octyl homolog, failed to give the guanidines when allowed to react with amines in the above manner.

Method B.—The amine hydrochloride (monoamines) (0.1 mole) or the free amine (diamines) was dissolved in 50 ml of H₂O. To the diamine solutions was added 0.1 mole of HCl or 0.05 mole of H₂SO₄. A slight excess of cyanamide (0.11 mole) was added. When *t*-butyl- and *t*-octyleyanamides were used, enough EtOH was added to form a solution. The reaction solution was placed in a silicone oil bath and the temperature was allowed to rise to 180° during 1 hr as the solvent distilled, and then kept at 180° for 2 hr. The guanidines from the diamines were dissolved in water, a second equivalent of acid (HCl or H₂SO₄) was added, and the solution was taken to dryness. The residue was crystallized from an appropriate solvent. The guanidines are described in Table I.

1-Hexamethylenimino-2-methyl-2-nitropropane.—A solution of 99 g (1.0 mole) of hexamethylenimine and 119 g (1.0 mole) of 1-hydroxy-2-methyl-2-nitropropane in 500 ml of PhII was heated under reflux with a water separator until no further H₂O separated (48–72 hr). The solvent was removed and the residue was distilled to render 180 g (90%) of colorless oil, bp 98–101° (1.5 mm), which solidified, mp 38–40°. Anal. (C₁₀H₂₀N₂O₂) C, H, N.

2-(Substituted amino)isobutyronitriles. Method C.—A solution of 1.0 mole of the amine and 1.0 mole of acetone cyanohydrin in 250 ml of dry PhH was heated under reflux until no more water collected in a water separator (about 18 hr). The solvent was removed and the residue was distilled. The nitriles are described in Table II.

2-(Substituted amino)isobutyramides. Method D.—A solution containing 5 ml of 90% H₂SO₄ for each gram of nitrile was heated on the steam bath for 0.5–2.0 hr. The solution was poured onto ice and made basic with aqueous NH₃. The amide was collected on a filter if it was a solid or taken up in CHCl₈, dried, and distilled. The amides are described in Table II.

Amines by Reductive Alkylation of Ketones. Method E.--The ketone was dissolved in EtOH (4 ml/g) and liquid $\rm NH_3$ was

⁽¹³⁾ Melting points were determined in capillary tubes in a silicone oil bath and are corrected. Where analyses are indicated only by symbols of the elements, analytical results for those elements are within $\pm 0.3\%$ of the theoretical values.

⁽¹⁴⁾ M. Seefelder, German Patent 1,119,258 (Dec 14, 1961); Chem. Abstr., $\mathbf{56},$ 11450 (1962).

⁽¹⁵⁾ The use of ethanol to take the water-insoluble amines into solution does not appear to be essential. Reaction of 2.2-diethyl-n-butylamine (0.05 mole) with 2-methyl-2-thiopseudourea (0.025 mole) sulfate takes place in H_2O (25 ml) to give a 55.5% yield of the expected guandine even though the reaction mixture consists of two layers at the start. A comparable yield (59%) of the guandine was obtained when the reaction was effected in 80%. EtOH (25 ml) because of the insolubility of 2-methyl-2-thiopseudourea sulfate in EtOH. The more soluble hydrochloride salt, on the other hand, gives satisfactory results in EtOH.⁴

			NITRILES AND	AMDES			
No.	R	Mp or bp, °C (mm)	n ²³ D CH	Method	Yield, %	Formula	Analyses
			RCC	° N			
			ĊH	3			
1	$(\mathrm{C}_2\mathrm{H}_5)_2\mathrm{N}$	$76-79 \ (28)^a$	1.4302	\mathbf{C}	57		
2	$(\widetilde{CH_2})_{6}N$	82-84 (1)	1.4662	С	75	${\rm C}_{10}{\rm H}_{18}{\rm N}_2$	C, H, N
3	CH ₃ N	108–110 (1) ^b		С	4 0	$\mathbf{C}_{9}\mathbf{H}_{17}\mathbf{N}_{3}$	С, Н
			$\mathrm{RC}(\mathrm{CH}_{8})_{2}\mathrm{C}$	ONH_2			
4	$(C_2H_{\mathfrak{z}})_2N$	107-108 (7)	1.4600	D	78	$\mathrm{C_8H_{18}N_2O}$	C, H, N
5	$(CH_2)_6N$	$94.5 - 96^{\circ}$		D	55	${\rm C}_{10}{\rm H}_{20}{\rm N}_{2}{\rm O}$	С, Н, N
6	CH ₃ N N	96-97		\mathbf{D}^{d}	60	$C_9H_{19}N_3O$	С, Н, N

TARE II

^a R. A. Jacobson, J. Am. Chem. Soc., **67**, 1996 (1945), reported bp 66–68° (14 mm) and n^{25} D 1.4312. ^b The distillate solidified and melted at 59–60° after being crystallized from Skellysolve B. ^c The amide was crystallized from pentane. ^d The acid solution was poured slowly onto ice to avoid generation of too much heat which causes the amide to decompose. Neutralization was effected with solid K₂CO₃ to keep the volume low because of the water solubility of the amide. It was taken up in CHCl₃ and evaporated, and the residue was crystallized from Skellysolve B.

TADIE III

		± ···									
		А	MINES								
RNH_2											
No.	R	Bp, °C (mm)	$n^{25}D$	Method	Yield, %	Formula	Analyses				
1	$(CH_3)_3CCH_2CH_2$	115 (760) ^a	1.4095	\mathbf{F}	66						
2	$(C_2H_5)_{s}CCH_2$	166 - 168 (760)	1.4391	ь	65	$C_8H_{19}N$	С, Н, N				
3	$C_6H_5C(CH_3)_2CH_2$	106-108 (15)	1.5161	\mathbf{F}^{g}	76	$C_{10}H_{15}N$	Ν				
4	$(C_2H_{\delta})_2NCH_2CH(CH_3)$	$152 - 154 \ (760)^c$	1.4275	\mathbf{E}	69						
5	$(C_2H_5)_2NC(CH_3)_2CH_2$	48-51 (7)	1.4440	F^{d}	50	$\mathrm{C_8H_{20}N_2}$	C, H, N				
		176-178 (760)									
6	$(CH_2)_{a}NC(CH_3)_{a}CH_2$	56-58(0.3)	1.4792	F	85	$\mathrm{C_{10}H_{22}N_2}$	С, Н, N				
7	$(\widetilde{CH_{a}})_{*}NCH_{a}C(CH_{a})_{2}$	78-81 (6)	1.4660	E^{e}	81	${\rm C}_{10}{\rm H}_{22}{\rm N}_2$	C, H, N				
8	CH ₃ NC(CH ₃) ₂ CH ₂	58-59(0.3)	1.4778	F٢	94	$\mathrm{C}_{9}\mathrm{H}_{21}\mathrm{N}_{3}$	С, Н, N				

^a A. P. Howe and H. B. Hass, *Ind. Eng. Chem.*, **38**, 251 (1946), reported bp 112.8–112.9° (745 mm), n^{25} D 1.4122. The starting material, 3,3-dimethyl-*n*-butyramide, was prepared according to the procedure of A. H. Homeyer, F. C. Whitmore, and V. H. Wellingford, *J. Am. Chem. Soc.*, **55**, 4209 (1933). ^b Triethylacetonitrile (Benzol Products Co.) was reduced with LiAlH₄ in the manner previously described for other nitriles.¹² ° L. Haskelberg, *J. Am. Chem. Soc.*, **70**, 2811 (1948), reported bp 154° (760 mm). ^d The yield of amine was 21% when the amide was reduced with LiAlH₄. ^e The amine was prepared by reduction of the corresponding nitro compound (Experimental Section) in the manner of method E except that liquid NH₃ was not added. ^f The trihydrochloride melts at 239-242°. ^e The HCl salt melts at 200-202°. It was recrystallized from MeOH-Et₂O. The starting material, α, α -dimethylphenylacetamide, was obtained from Eli Lilly and Co.

added (same weight as the ketone). Raney Ni was added (20-30% of the weight of amine), and the mixture was shaken in the presence of H₂ (70.3 kg/cm²) at room temperature until the theoretical amount of H₂ was absorbed (2-18 hr). The amines are described in Table III.

Amines by Reduction of Amides with Diborane. Method F.— The reduction was effected according to the procedure of Brown and Heim¹⁶ except that the reaction mixture was heated under reflux for 18 hr. The amines are described in Table III.

Action of LiAlH₄ on 2-Diethylaminoisobutyronitrile.—A solution of 63 g (0.45 mole) of the nitrile in Et₂O (600 ml) was added to a stirred solution of 30 g (0.79 mole) of LiAlH₄ in an ice bath at such a rate that the temperature remained below 10°. The product was a colorless oil, bp 107–109° (760 mm), $n^{25}D$ 1.4044. The yield of diethylisopropylamine was 37 g (72%). Recorded physical constants for diethylisopropylamine are bp 107.5°, $n^{25}D$ 1.4047.³⁷

Reduction of the nitrile with diborane also gave only diethylisopropylamine.

Similar results were obtained when the reduction was carried out in the manner of method E using Rh–C as the catalyst. **Diethylamino**-*t*-**butylamine**.—The reaction of 43.8 g (0.6 mole) of Et₂NH with 21.3 g (0.3 mole) of 2,2-dimethylaziridine in the presence of 60 g (0.45 mole) of anhydrous AlCl₃ in 100 ml of PhH was effected in the manner described by Coleman and Callen¹⁸ for the reaction between Bu₂NH and aziridine. The yield of colorless oil, bp 155–156° (760 mm), n^{25} D 1.4298, was 8.6 g (20%). It proved to be identical in all respects with the material obtained by reduction of 1-diethylamino-2-methyl-2-nitropropane.¹⁹ The absence of the isomer, 2-diethylaminoisobutylamine, was shown by tle.

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⁽¹⁶⁾ H.C. Brown and P. Heim, J. Am. Chem. Soc., 86, 3566 (1964).

⁽¹⁷⁾ R. A. Robinson, J. Org. Chem., 16, 1911 (1951).

⁽¹⁸⁾ G. H. Coleman and J. E. Callen, J. Am. Chem. Soc., 68, 2006 (1946).

⁽¹⁹⁾ M. Freifelder and Y. H. Ng, J. Org. Chem., 30, 4370 (1965).