

**Acyl Radical Replacement in the Reactions of III (R = Alkyl) with Ethyl Orthoformate** (1).— $\alpha$ -Propionyl-*p*-chlorophenylacetonitrile<sup>1</sup> (107 g.) and ethyl orthoformate (214 ml.) were heated together under reflux, reaction being complete after about 3.5 hours. Excess of the orthoester was then distilled *in vacuo*, and the dark residual oil (130 g.) reacted with guanidine (from 56 g. of the hydrochloride) in alcohol on refluxing for 24 hours. After cooling, and standing 48 hours, the crystals of 2,4-diamino-5-*p*-chlorophenyl-6-ethylpyrimidine<sup>1</sup> (38 g.), m.p. 234.5–236°, were collected. The alcoholic liquors were evaporated under reduced pressure, the residual gum stirred in boiling water (2 l.) and sufficient hydrochloric acid added to bring the pH to 5. The hot liquid was filtered from some undissolved viscous oil, basified with sodium hydroxide solution and cooled. The solid thus precipitated was collected, dried and triturated with cold acetone to give 17 g. of crystals. These were extracted several times with 750-ml. portions of hot water, and the extracts cooled to give identical fractions of substantially pure by-product. The residue (2.5 g.) consisted of pure 2,4-diamino-5-*p*-chlorophenyl-6-ethylpyrimidine. When the by-product was extracted with a little hot benzene, and then recrystallized from alcohol, pure 2,4-diamino-5-*p*-chlorophenylpyrimidine<sup>1</sup> (8.8 g.), m.p. 194–195°, was obtained.

(2).— $\alpha$ -Propionyl-*p*-bromophenylacetonitrile<sup>1</sup> (15 g.) was treated with ethyl orthoformate (30 ml.) in the usual manner. The residue after removal of the excess orthoester was heated with guanidine (from 5 g. of the hydrochloride) in alcohol. After cooling, diluting with water and basifying with sodium hydroxide solution, the product was collected and recrystallized from aqueous alcohol to give 2,4-diamino-5-*p*-bromophenyl-6-ethylpyrimidine<sup>1</sup> (8 g.), m.p. 213–214°. On standing, the ethanol-water mother liquors deposited more crystalline material (0.9 g.) which, recrystallized twice from the same solvent, melted at 204–205°. This material was identical in every respect with 2,4-diamino-5-*p*-bromophenylpyrimidine<sup>1</sup> (m.p., mixed m.p., solubility and ultraviolet absorption spectrum).

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## Derivatives of Dimethylaminoethanol and Dimethylaminoethylamine

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Various  $\beta$ -dimethylaminoethyl ethers and a series of disubstituted  $\beta$ -dimethylaminoethylamines have been prepared. These compounds have been assayed for antispasmodic activity against acetylcholine and for antibacterial effectiveness against *Staphylococcus aureus*.

In the course of systematic investigations of  $\beta$ -dimethylaminoethyl substituted compounds, we have prepared various derivatives of dimethylaminoethanol and dimethylaminoethylamine. The former were prepared by means of a Williamson condensation between an appropriate sodium alcoholate and  $\beta$ -dimethylaminoethyl chloride. The compounds prepared are presented in Table I together with data on their antispasmodic and antibacterial activities. The derivatives of dimethylaminoethylamine were prepared, in general, by condensing appropriate secondary amines with  $\beta$ -dimethylaminoethyl chloride. New secondary amines synthesized as intermediates are presented in Table II, and the final products are presented in Table III which includes data on antispasmodic and antibacterial activities.

### Experimental

**Dimethylaminoethyl Ethers.**—One mole of finely divided metallic sodium was added to the required alcohol. Frequently, the alcohol was used in excess to prevent precipitation of the alcoholate. In some instances, non-polar solvents such as toluene or xylene were added for this purpose. After the evolution of hydrogen had ceased, freshly distilled  $\beta$ -dimethylaminoethyl chloride was added, occasionally in xylene, and the temperature was raised to 100° and maintained at that temperature until precipitation of sodium chloride was completed (approximately 24 hours). The reaction mixture was cooled, water added, and the pH was adjusted to approximately 10. The organic layer, frequently with ether added, was separated, dried and the solvent was removed. Products were isolated by fractionation and were purified in some instances by recrystallization of the hydrochlorides.

**Secondary Amines.**—The required secondary amines were prepared using the method of Blicke and Monroe<sup>1</sup> with minor modifications. In general, to two moles of primary amine in ethanol one mole of halide was slowly added. The solution was refluxed for 15 or more hours, cooled, acidified and the alcohol was removed under reduced pressure. Unreacted halide was removed by steam distillation. The residue, suspended in water, was made strongly alkaline and the base was extracted with ether. The secondary amine was then obtained by fractionation. In the preparation of the secondary naphthylamines equimolecular quantities were used at the outset. The pyridyl and quinolyl secondary amines were prepared from the heterocyclic chloro derivatives by treatment with alkyl amines at 160° for 24 hours under pressure; in some instances a copper catalyst was used. Low boiling secondary amines were prepared by allowing solutions to remain at room temperatures for about one week, but, in some instances, reactions were run in xylene under pressure with or without copper catalysts. Amines which apparently have not been reported are presented in Table II.

**Dimethylaminoethylamines.**—Two moles of the appropriate secondary amine and one mole of  $\beta$ -dimethylaminoethyl chloride in xylene were refluxed for 2–4 hours. The reaction mixture was cooled and the hydrochloride of the excess secondary amine was removed by filtration. The product was extracted from the xylene with hydrochloric acid. The acidic aqueous layer was made strongly alkaline and the product was extracted with ether. The ether was removed and the residue was fractionated to yield the desired dimethylaminoethylamine. With lower boiling secondary amines, the reaction was frequently run in sealed tubes at 125–150° overnight. Aromatic secondary amines were treated with 1.2 moles of sodium amide in xylene at temperatures of 125–130° for approximately 72 hours. The reaction mixture was cooled and one mole of  $\beta$ -dimethylaminoethyl chloride was added slowly. The reaction mixture was maintained at 100° for approximately 60 hours and then treated as described above.

(1) F. F. Blicke and E. Monroe, *THIS JOURNAL*, **61**, 91 (1939).

TABLE I  
 $(\text{CH}_3)_2\text{N}-\text{CH}_2-\text{CH}_2-\text{OR}$ 

R <sub>1</sub>	B.p. (Base), °C.	Mn.	Empirical formula	Nitrogen, <sup>a</sup> %		Inhibition acetyl- choline, %	Minimal bacteriostatic concn., μg./100 ml.
				Calcd.	Found		
Methyl <sup>2</sup>	64-68	760	C <sub>6</sub> H <sub>13</sub> NO	13.6	13.8		
Ethyl <sup>3</sup>	116-118	760	C <sub>8</sub> H <sub>15</sub> NO	12.0	11.7		
Propyl	137-140	760	C <sub>7</sub> H <sub>17</sub> NO	10.7	10.8		
Allyl	115-119	760	C <sub>7</sub> H <sub>15</sub> NO	22.0	22.2 <sup>a</sup>		
Butyl <sup>4</sup>	162-166	760	C <sub>8</sub> H <sub>19</sub> NO	9.6	9.7		
Isoamyl	173-177	760	C <sub>9</sub> H <sub>21</sub> NO	8.8	8.7		
Hexyl	105-106	23	C <sub>10</sub> H <sub>23</sub> NO	17.4	17.4 <sup>a</sup>	20	
Heptyl	105-106	13	C <sub>11</sub> H <sub>25</sub> NO	16.3	15.9 <sup>a</sup>		
Octyl	121-125	13	C <sub>12</sub> H <sub>27</sub> NO	7.0	7.0	20	
Nonyl	119-123	5	C <sub>13</sub> H <sub>29</sub> NO	6.5	6.6	30	4
Decyl	131-134	4	C <sub>14</sub> H <sub>31</sub> NO	6.1	6.3		4
Hendecyl	143-145	2	C <sub>15</sub> H <sub>33</sub> NO	5.8	6.1	40	16
Dodecyl	158-162	13	C <sub>16</sub> H <sub>35</sub> NO	5.5	5.8	40	0.5
α-Tetrahydrofurylmethyl	105-107	16	C <sub>9</sub> H <sub>19</sub> NO <sub>2</sub>	8.1	7.9		8
β-Pyridyl	115-116	2	C <sub>9</sub> H <sub>14</sub> N <sub>2</sub> O	16.9	16.9		
Phenyl <sup>6</sup>	93-97	6	C <sub>10</sub> H <sub>15</sub> NO	8.5	8.6		
Cyclohexyl	101-105	24	C <sub>10</sub> H <sub>21</sub> NO	8.2	8.4	20	
Benzyl	118-121	3	C <sub>11</sub> H <sub>17</sub> NO	7.8	8.1		
Thymyl <sup>6</sup>	123-127	3	C <sub>14</sub> H <sub>23</sub> NO	6.3	6.5	65	
Menthyl	103-106	3	C <sub>14</sub> H <sub>29</sub> NO	6.2	6.1	100	

<sup>a</sup> Dumas determinations of nitrogen are given in this and subsequent tables unless otherwise noted; <sup>a</sup> indicates the percentage of HCl in hydrochlorides.

TABLE II

R <sub>1</sub>	R <sub>2</sub>	B.p.		Em- pirical formula	Nitrogen, %	
		(Base), °C.	Mm.		Calcd.	Found
Allyl	<i>i</i> -Propenyl	109	760	C <sub>8</sub> H <sub>11</sub> N	14.4	14.5
Hexyl	Allyl	179-180	760	C <sub>8</sub> H <sub>19</sub> N	9.9	9.8
Hexyl	<i>i</i> -Propyl	172-173	760	C <sub>8</sub> H <sub>11</sub> N	9.8	9.9
Heptyl	<i>i</i> -Propyl	191-193	760	C <sub>10</sub> H <sub>23</sub> N	8.9	8.8
Heptyl	Butyl	222-223	760	C <sub>11</sub> H <sub>25</sub> N	8.2	8.2
Octyl	Propyl	221-223	760	C <sub>11</sub> H <sub>25</sub> N	8.2	8.3
Octyl	<i>i</i> -Propyl	210-212	760	C <sub>11</sub> H <sub>25</sub> N	8.2	8.2
Octyl	Butyl	233-235	760	C <sub>12</sub> H <sub>27</sub> N	7.6	7.4
Octyl	Hexyl	255-257	760	C <sub>14</sub> H <sub>31</sub> N	6.6	6.4
2-Ethylhexyl	Methyl	175-176	760	C <sub>8</sub> H <sub>21</sub> N	9.8	10.0
Cyclohexyl	Allyl	190-193	760	C <sub>8</sub> H <sub>17</sub> N	10.1	10.0
α-Furfuryl	<i>i</i> -Propyl	82-85	19	C <sub>8</sub> H <sub>13</sub> NO	10.1	10.2
α-Furfuryl	Butyl	98-100	9	C <sub>9</sub> H <sub>15</sub> NO	9.2	9.4
α-Naphthyl	Methyl	132-134	2	C <sub>11</sub> H <sub>11</sub> N	8.9	8.8
α-Naphthyl	Hexyl	175-178	2	C <sub>14</sub> H <sub>21</sub> N	6.2	6.0
α-Naphthyl	Octyl	228	9	C <sub>18</sub> H <sub>25</sub> N	5.5	5.4
α-Naphthyl	Nonyl	203-205	1	C <sub>18</sub> H <sub>27</sub> N	5.2	5.0
α-Naphthyl	Decyl	206-208	1	C <sub>20</sub> H <sub>29</sub> N	4.9	4.8
α-Naphthyl	Hendecyl	207-209	1	C <sub>21</sub> H <sub>31</sub> N	4.7	4.6

**Antispasmodic Activity.**—Sections of excised guinea pig ileum were suspended in a 100-ml. bath of oxygenated Tyrode solution at 37°. After equilibration, sufficient acetylcholine bromide was added to evoke a submaximal response (1 mcg./ml. bath fluid). Two such responses determined the mean pretreatment base line contraction height. After washing out the spasmogenic agent the unknown compound under test was added (1 mcg./ml. bath fluid) and, after 5 minutes, without washing, acetylcholine bromide was again added to the bath. The reduction in response to this dose of acetylcholine was taken as an indication of spasmolytic activity. The per cent. inhibition of acetylcholine spasm is given in the appropriate tables. The absence of a value in the tables indicates an inhibition of less than 20% which is taken to mean no significant antispasmodic activity. A dash indicates that the compound was not assayed. Under these conditions of test, atropine completely inhibits the acetylcholine response. Trasentine reduces the response 37%, but Papaverine, aminophyllin and epinephrine produce no effect.

**Antibacterial Assays.**—Individual compounds were dissolved in nutrient broth at a starting concentration of 64 mg. % using a twofold serial dilution method. The stand-

 TABLE III  
 $\text{R}_1\text{R}_2\text{N}-\text{CH}_2-\text{CH}_2-\text{N}(\text{CH}_3)_2$ 

R <sub>1</sub>	R <sub>2</sub>	B.p.		Empirical formula	Nitrogen, %		Inhibition acetyl- choline, %	Minimal bacteriostatic concn. mg./100 ml.
		(Base), °C.	Mm.		Calcd.	Found		
Methyl <sup>7</sup>	Methyl	120	760	C <sub>6</sub> H <sub>16</sub> N <sub>2</sub>	24.1	24.3		
Ethyl <sup>8</sup>	Ethyl	163-165	760	C <sub>8</sub> H <sub>20</sub> N <sub>2</sub>	19.4	19.6	40	
Propyl	Propyl	93-97	27	C <sub>10</sub> H <sub>24</sub> N <sub>2</sub>	16.3	16.3		
Allyl	Allyl	70-73	18	C <sub>10</sub> H <sub>20</sub> N <sub>2</sub>	16.7	16.7		
Allyl	Isopropenyl	90-93	13	C <sub>10</sub> H <sub>19</sub> N <sub>2</sub>	16.7	16.2	20	
Butyl	Methyl	181-183	760	C <sub>9</sub> H <sub>22</sub> N <sub>2</sub>	17.7	18.0		
Butyl	Ethyl	87-89	18	C <sub>10</sub> H <sub>24</sub> N <sub>2</sub>	16.3	16.7		
Butyl	Butyl	201	760	C <sub>12</sub> H <sub>28</sub> N <sub>2</sub>	13.9	14.3		
Butyl	Amyl	136-138	18	C <sub>13</sub> H <sub>30</sub> N <sub>2</sub>	13.1	13.4	30	
Butyl	Hydroxyethyl	116-119	13	C <sub>12</sub> H <sub>24</sub> N <sub>2</sub> O	14.9	14.6		

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ard inoculum was 0.1 ml. of a 1 to 5000 dilution of an 18-hour culture of *Staphylococcus aureus* 209. Final readings were taken after 72 hours incubation. The minimal bacteriostatic concentration was taken as concentration in the first tube showing complete absence of visible growth. Controls were included in every series and each experiment was run in duplicate. Results are recorded in the tables. Absence

TABLE III (Continued)

R <sub>1</sub>	R <sub>2</sub>	B.P.		Empirical formula	Nitrogen, %		Inhibition acetylcholine, %	Minimal bacteriostatic concn. mg./100 ml.
		(Base), °C.	Mm.		Calcd.	Found		
Amyl	Methyl	93-95	20	C <sub>10</sub> H <sub>24</sub> N <sub>2</sub>	16.3	16.6	20	
Amyl	Ethyl	106-108	18	C <sub>11</sub> H <sub>26</sub> N <sub>2</sub>	15.0	15.2		
Amyl	Propyl	116-119	22	C <sub>12</sub> H <sub>28</sub> N <sub>2</sub>	13.9	14.2		32
Amyl	Isopropyl	115-118	20	C <sub>12</sub> H <sub>28</sub> N <sub>2</sub>	13.9	14.1	20	
Amyl	Amyl	136-138	17	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub>	12.3	12.5	20	32
Hexyl	Methyl	109-112	20	C <sub>11</sub> H <sub>26</sub> N <sub>2</sub>	15.0	15.1		
Hexyl	Ethyl	110	17	C <sub>12</sub> H <sub>28</sub> N <sub>2</sub>	13.9	14.2	35	
Hexyl	Propyl	126-129	22	C <sub>13</sub> H <sub>30</sub> N <sub>2</sub>	13.1	13.3		
Hexyl	Isopropyl	145-148	32	C <sub>13</sub> H <sub>30</sub> N <sub>2</sub>	13.1	13.4	25	
Hexyl	Allyl	132-134	20	C <sub>12</sub> H <sub>28</sub> N <sub>2</sub>	13.1	13.4	40	0.5
Hexyl	Butyl	131-134	20	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub>	12.3	12.4		
Hexyl	Methoxyethyl	138-141	7	C <sub>13</sub> H <sub>30</sub> N <sub>2</sub> O	12.2	11.9	35	
Hexyl	Ethoxyethyl	115-116	2	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub> O	11.5	11.9	25	
Heptyl	Methyl	113-116	15	C <sub>12</sub> H <sub>28</sub> N <sub>2</sub>	13.9	14.0		
Heptyl	Ethyl	136-139	20	C <sub>13</sub> H <sub>30</sub> N <sub>2</sub>	13.1	13.3	35	
Heptyl	Propyl	146-149	20	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub>	12.3	12.6	55	
Heptyl	Isopropyl	136-139	16	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub>	12.3	12.6	35	
Heptyl	Butyl	160-163	24	C <sub>15</sub> H <sub>34</sub> N <sub>2</sub>	11.6	12.0	20	
Octyl	Methyl	143-146	23	C <sub>13</sub> H <sub>30</sub> N <sub>2</sub>	13.1	13.4	30	16
Octyl	Ethyl	146-149	22	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub>	12.3	12.6	40	16
Octyl	Propyl	156-159	22	C <sub>15</sub> H <sub>34</sub> N <sub>2</sub>	11.6	11.9	20	16
Octyl	Isopropyl	153-158	20	C <sub>15</sub> H <sub>34</sub> N <sub>2</sub>	11.6	11.9	25	16
Octyl	Butyl	165-167	13	C <sub>16</sub> H <sub>36</sub> N <sub>2</sub>	10.9	11.2	35	16
Octyl	Hexyl	136-139	3	C <sub>18</sub> H <sub>40</sub> N <sub>2</sub>	9.9	9.6	25	1
Octyl	Methoxyethyl	165-167	13	C <sub>15</sub> H <sub>34</sub> N <sub>2</sub> O	10.8	10.4	20	16
Nonyl	Methyl	152-155	18	C <sub>14</sub> H <sub>32</sub> N <sub>2</sub>	12.3	12.5	20	8
Nonyl	Ethyl	164-168	20	C <sub>15</sub> H <sub>34</sub> N <sub>2</sub>	11.6	11.8	30	16
Decyl	Methyl	165-168	20	C <sub>16</sub> H <sub>36</sub> N <sub>2</sub>	11.6	11.7		4
Hendecyl	Methyl	149-152	3	C <sub>16</sub> H <sub>36</sub> N <sub>2</sub>	10.9	11.1	25	1
Dodecyl	Methyl	135-137	2	C <sub>17</sub> H <sub>38</sub> N <sub>2</sub>	10.3	10.4	50	0.12
Myristyl	Methyl	185-187	6	C <sub>19</sub> H <sub>42</sub> N <sub>2</sub>	9.4	9.3		0.25
Cetyl	Methyl	195-199	4	C <sub>21</sub> H <sub>46</sub> N <sub>2</sub>	8.6	8.8	20	0.25
Cyclopentyl	Methyl	95-96	13	C <sub>10</sub> H <sub>22</sub> N <sub>2</sub>	16.4	15.9	40	32
Cyclohexyl	Methyl	116-119	24	C <sub>11</sub> H <sub>24</sub> N <sub>2</sub>	15.2	15.2		
Cyclohexyl	Ethyl	136-138	37	C <sub>12</sub> H <sub>26</sub> N <sub>2</sub>	14.1	14.1		
Phenyl <sup>9</sup>	Methyl	106-107	4	C <sub>11</sub> H <sub>18</sub> N <sub>2</sub>	15.7	15.3		
Phenyl <sup>10</sup>	Butyl	133-134	6	C <sub>14</sub> H <sub>24</sub> N <sub>2</sub>	12.7	12.9	30	16
Phenyl	Octyl	163-166	3	C <sub>18</sub> H <sub>32</sub> N <sub>2</sub>	10.1	10.1	50	2
<i>p</i> -Chlorophenyl	Methyl	143-145	7	C <sub>11</sub> H <sub>17</sub> ClN <sub>2</sub>	13.2	13.3		
<i>p</i> -Chlorophenyl	Butyl	158-160	6	C <sub>14</sub> H <sub>23</sub> ClN <sub>2</sub>	11.1	11.3	50	8
<i>p</i> -Chlorophenyl	Octyl	210-211	9	C <sub>18</sub> H <sub>31</sub> ClN <sub>2</sub>	9.0	9.2		
<i>p</i> -Methoxyphenyl	Methyl	153-156	7	C <sub>12</sub> H <sub>20</sub> N <sub>2</sub> O	13.4	13.7		
<i>p</i> -Methoxyphenyl	Butyl	174-176	10	C <sub>15</sub> H <sub>26</sub> N <sub>2</sub> O	11.2	11.1		
<i>p</i> -Methoxyphenyl	Octyl	211-213	9	C <sub>19</sub> H <sub>34</sub> N <sub>2</sub> O	9.2	9.6	45	
<i>p</i> -Methoxyphenyl	Hydroxyethyl	151-153	13	C <sub>13</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub>	11.8	12.2	30	
Benzyl	Methyl	116-119	10	C <sub>12</sub> H <sub>20</sub> N <sub>2</sub>	14.6	14.8		
$\alpha$ -Naphthyl	Methyl	156-159	3	C <sub>15</sub> H <sub>20</sub> N <sub>2</sub>	12.3	12.4	20	2
$\beta$ -Naphthyl	Methyl	175-178	6	C <sub>15</sub> H <sub>20</sub> N <sub>2</sub>	12.3	12.5	20	0.5
$\alpha$ -Naphthyl	Butyl	175-179	3	C <sub>18</sub> H <sub>26</sub> N <sub>2</sub>	10.4	10.2	20	4
$\alpha$ -Naphthyl	Hexyl	165-168	1	C <sub>20</sub> H <sub>30</sub> N <sub>2</sub>	9.4	9.4		0.12
$\alpha$ -Naphthyl	Octyl	196-199	3	C <sub>22</sub> H <sub>34</sub> N <sub>2</sub>	8.6	8.4	90	0.12
$\alpha$ -Naphthyl	Nonyl	186-188	1.5	C <sub>23</sub> H <sub>36</sub> N <sub>2</sub>	8.2	8.2		0.12
$\alpha$ -Naphthyl	Decyl	199-202	1	C <sub>24</sub> H <sub>38</sub> N <sub>2</sub>	7.9	7.9		0.12
$\beta$ -Pyridyl	Methyl	145-147	10	C <sub>10</sub> H <sub>17</sub> N <sub>3</sub>	23.5	23.4		32
Piperidyl <sup>11</sup>		195	760	C <sub>9</sub> H <sub>20</sub> N <sub>2</sub>	17.9	18.1		20
2-Quinoliny	Methyl	164-167	3	C <sub>14</sub> H <sub>19</sub> N <sub>3</sub>	18.3	18.1	25	32

of value in the table indicates no activity below a concentra-

tion of 64 mg. per 100 ml. A dash indicates that the compound was not assayed.

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