

which the molecule fragments *via* initial losses of CO₂ and of CO, the electron-impact and pyrolytic fragmentations of **6** appear to be qualitatively similar; the same is true of **7**. We are studying the mass spectra and pyrolyses of various aromatic molecules in order to explore the similarities between the two processes.

We hope to develop this technique into a means of predicting a molecule's pyrolysis pathway from its mass spectrum and to elucidate possible electronic relationships between the two processes.

Registry No.—**6**, 711-62-6; **7**, 2435-53-2.

The Pyrolysis of 1,1-Dihexyl-1-methylamine-2-acylimides

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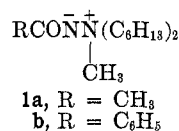
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Pyrolysis of 1,1-dihexyl-1-methylamine-2-acetylimide gave 1-hexene, methyl isocyanate, 1-hexyl-1-methyl-2-acetylhydrazine, and dihexylmethylamine. The corresponding benzoyl derivative gave 1-hexene, dihexylmethylamine, diphenylurea, 2-phenylbenzimidazole, benzanilide, and 1-phenyl-3-methylurazole. The abnormal products isolated result from the reaction of phenyl isocyanate with moisture, the aminimide, and 1-hexyl-1-methyl-2-benzoylhydrazine.

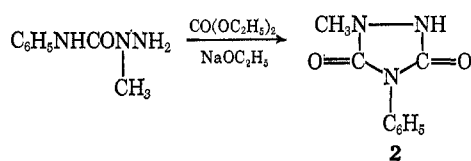
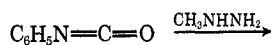
Studies of the pyrolysis of open-chain aminimides have been limited to examples containing three methyl groups,³ two methyls and a 2-acetoxypropyl group,⁴ benzyl and two methyls,⁵ and recently two methyls and a cyclooctyl or 2-phenylpropyl group.⁶ The first two form the tertiary amine and isocyanate and the third type proceeds with rearrangement of the benzyl group. The last two are reported to proceed solely with elimination.

The present work describes the pyrolysis of 1,1-dihexyl-1-methylamine-2-acetylimide (**1a**) and 1,1-dihexyl-1-methylamine-2-benzoylimide (**1b**) and finds that the products formed depend upon the acyl group present.



1,1-Dihexyl-1-methylamine-2-acetylimide (**1a**) upon pyrolysis at 140° gave 1-hexene, methyl isocyanate, 1-hexyl-1-methyl-2-acetylhydrazine, and dihexylmethylamine. Based on the ratio of the last two compounds, the elimination reaction occurred to the extent of 64.5%.

The 2-benzoyl derivative **1b**, when heated at its decomposition point of 175–185°, gave a more complicated mixture consisting of 1-hexene, dihexylmethylamine, benzanilide, diphenylurea, 2-phenylbenzimidazole, and 1-phenyl-3-methylurazole (**2**). The last compound



was identified by comparison with a sample synthesized by the following series of reactions.

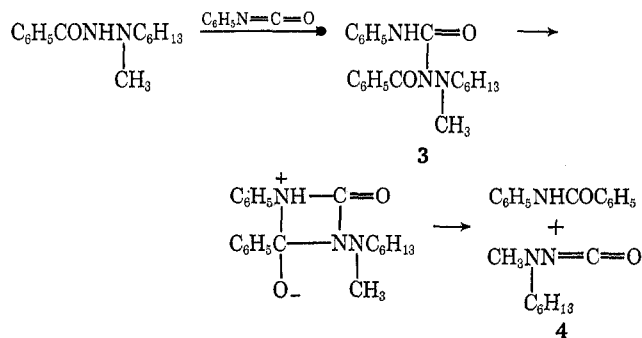
The formation of these products can be rationalized upon the basis of a similar decomposition of the benzoyl derivative **1b** to that found with the acetyl compound **1a**. The absence of phenyl isocyanate or its trimer and 1-hexyl-1-methyl-2-benzoylhydrazine suggests that these compounds are involved in the formation of benzanilide, diphenylurea, 2-phenylbenzimidazole, and 1-phenyl-3-methylurazole (**2**).

2-Phenylbenzimidazole has been isolated previously in the decomposition of 1,1,1-trimethylamine-2-benzoylimide and is considered to be formed from the reaction of phenyl isocyanate with the aminimide.⁷

Diphenylurea is probably formed from phenyl isocyanate and traces of water; no precautions were taken to exclude moisture from the pyrolysis.

Benzanilide and 1-phenyl-3-methylurazole (**2**) result from a reaction of phenyl isocyanate and 1-hexyl-1-methyl-2-benzoylhydrazine. This reaction was verified by heating these two compounds at 170°; 1-hexene, benzanilide, diphenylurea, and 1-phenyl-3-methylurazole were formed.

The initial step in this reaction parallels that observed between phenyl isocyanate and *N*-alkylamides.⁸



The intermediate urea **3** at the temperature used would decompose into benzanilide and methylhexylaminoisocyanate (**4**). The last species (**4**) is not isolated but reacts with phenyl isocyanate and forms a cyclic aminimide.

(1) To whom inquiries should be addressed.

(2) Abstracted in part from the Ph.D. thesis of E. E. Paschke, May 1971.

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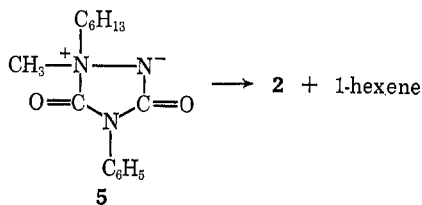
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mide 5. This compound can undergo β elimination and form the urazole 2 and 1-hexene.



The reactions proposed are precedent in the literature. Dimethylaminoisocyanate has been trapped with another isocyanate and forms a cyclic aminimide.⁹ The triethyl analog of 5 is reported to undergo β elimination and to form ethylene.¹⁰

Further verification of this reaction was the trapping of methylhexylaminoisocyanate, prepared from methylhexylhydrazine and phosgene, with phenyl isocyanate; 1-phenyl-3-methylurazole (2) and diphenylurea were obtained as products.

The series of reactions proposed is consistent with the yield of the compounds obtained. The yields of benzanilide (20.5%) and 1-phenyl-3-methylurazole (2) (20.2%) were the same since they are formed from a common intermediate. The yield of 1-hexene (35.6%) was approximately twice that of the benzanilide (30.5%) as expected.

The ratio of rearrangement to elimination was 4:1 for the benzoyl derivative 1b and is higher than that (1:2) observed for the acetyl derivative 1a. Since the decomposition of the former 1b was carried out at 175° and that for the acetyl at 140°, the effect of temperature on the ratio of products from the acetyl derivative was studied. Pyrolysis at 175° gave 67.6% elimination and indicated that the temperature dependence of the reaction was small.

The difference in behavior of the two aminimides (1) can be ascribed to the greater nucleophilic properties of the imide nitrogen in the acetyl derivative 1a. Less delocalization of this charge is involved in this compound (1a) than in the benzoyl derivative 1b. This difference is in agreement with the infrared spectra for the two compounds; the carbonyl absorption for the carbonyl group is at 1560 cm^{-1} for 1b and 1580 cm^{-1} for 1a.

Experimental Section¹¹

1,1-Dihexyl-2-benzoylhydrazine.—A solution of 1,1-dihexylhydrazine¹² (50.0 g, 0.25 mol) and triethylamine (52.2 g, 0.517 mol) in benzene (400 ml) was treated dropwise with a solution of benzoyl chloride (35.3 g, 0.251 mol) in benzene (100 ml) and the resulting solution was refluxed for 1 hr. Addition of water and sodium hydroxide (20 g, 0.5 mol) was followed by separation of the aqueous layer. The aqueous layer was extracted with benzene, the benzene extracts were combined, and the solvent was removed under reduced pressure. The white crystals (62.3 g, 0.205 mol, 82%) obtained were recrystallized from absolute ethanol: mp 75–76°; ir (Nujol) 3280 (NH), 1640 cm^{-1} (CO); nmr (DCCl₃) δ 7.50 (m, 5, C₆H₅), 7.12 (s, 1, NH), 2.81 (t, 4, N(CH₂)₂), 1.10 (m, 22, [CH₂(CH₂)₄]₂).

Anal. Calcd for C₁₉H₃₂N₂O: C, 74.95; H, 10.59; N, 9.20. Found: C, 75.14; H, 10.53; N, 8.99.

1,1-Dihexyl-1-methyl-2-benzoylhydrazonium *p*-Toluenesulfonate.—A solution of 1,1-dihexyl-2-benzoylhydrazine (51.9 g,

0.171 mol) and methyl *p*-toluenesulfonate (36.9 g, 0.198 mol) in acetonitrile (500 ml) was refluxed for 17 hr. Removal of the acetonitrile and excess methyl *p*-toluenesulfonate under reduced pressure gave 65.4 g of an oil which was purified by chromatography on silica gel. Elution with ethyl acetate gave three fractions. The first fraction (8.1 g) consisted of methyl *p*-toluenesulfonate and 1,1-di-*n*-hexyl-2-benzoylhydrazine. Fraction 2 (41.7 g, 0.085 mol, 49.7%) consisted of 1,1-dihexyl-1-methyl-2-benzoylhydrazonium *p*-toluenesulfonate. This compound was first isolated as an oil which crystallized on standing for several days: mp 69–71°; ir (Nujol) 3490 and 3220 (NH), 1680 (CO), 1180 cm^{-1} (SO₃⁻); nmr (DCCl₃) δ 10.50 (broad singlet, 1, NH), 7.45 (m, 9 aromatic hydrogens), 3.90 (t, 4, N(CH₂)₂), 3.57 (s, 3, NCH₃), 2.19 (s, 3, CH₃C₆H₄), 1.11 (m, 22, (C₆H₁₁)₂).

Anal. Calcd for C₂₇H₄₂N₂O₄S: C, 66.08; H, 8.63; N, 5.71. Found: C, 66.09; H, 8.94; N, 5.81.

Fraction 3 was a brown oil (8.07 g, 0.0253 mol, 14.8%) and proved to be 1,1-dihexyl-1-methylamine-2-benzoylimide.

1,1-Dihexyl-1-methylamine-2-benzoylimide.—A solution of 1,1-dihexyl-1-methyl-2-benzoylhydrazonium *p*-toluenesulfonate (23.48 g, 0.0764 mol) in dry methanol (100 ml) was treated with a solution of sodium methoxide (5.05 g, 0.0935 mol) in dry methanol (50 ml), and the resulting solution was heated to boiling. Removal of the methanol gave a viscous oil which was purified by dissolving in water and extracting with chloroform. The oil obtained after removing the chloroform was treated with dry benzene and filtering the traces of sodium *p*-toluenesulfonate which precipitated. Removal of the benzene gave the aminimide (23.66 g, 0.0764 mol, 97.2%) as an oil: ir (neat) 1560 cm^{-1} (CO); nmr (CDCl₃) δ 7.55 (m, 5, C₆H₅), 3.52 (t, 4, N(CH₂)₂), 3.15 (s, 3, NCH₃), 1.13 (m, 22, [CH₂(CH₂)₄]₂).

Anal. Calcd for C₂₀H₃₄N₂O: C, 75.42; H, 10.76; N, 8.80. Found: C, 75.24; H, 10.75; N, 8.62.

Pyrolysis of 1,1-Dihexyl-1-methyl-2-benzoylimide.—The aminimide 1b (12.76 g, 0.040 mol) was heated at 175–185° for 2.3 hr, and volatile products were collected in a receiver cooled with a Dry Ice-acetone mixture. In this manner 1-hexene (1.20 g, 0.0143 mol, 17.8%) was isolated, bp 62–63°. The ir and nmr spectra were identical with those of an authentic sample.

The residue from the pyrolysis upon extraction with hexane gave dihexylmethylamine (6.08 g, 0.0305 mol, 76%): bp 58–59° (0.9 mm); *n*_D²⁰ 1.4337 (lit.¹³ bp 118° (12 mm); *n*_D²⁰ 1.434); nmr (neat) δ 2.27 (t, 4, N(CH₂)₂), 2.11 (s, 3, NCH₃), and 1.13 (m, 22, [CH₂(CH₂)₄]₂).

The hexane-insoluble residue (4.94 g) was chromatographed on silica gel using benzene with increasing amounts of ethyl acetate as solvent. The first fraction contained benzanilide (1.62 g, 8.22 mmol, 20.5%), mp 162–163°. The second compound isolated was 1,3-diphenylurea (0.76 g, 3.58 mmol, 8.9%), mp 241–242°. Fraction 3 contained 2-phenylbenzimidazole (0.75 g, 3.86 mmol, 9.6%), mp 293–294° dec (lit.¹⁴ 299–301°). The ir and nmr spectra were identical with the spectra of an authentic sample.

The fourth compound isolated was 1-phenyl-3-methylurazole (2) (1.55 g, 8.1 mmol, 20.2%), mp 187–188°.

1-Phenyl-3-methylurazole (2).—A solution of 2-methyl-4-phenylsemicarbazide¹⁵ (6.64 g, 0.04 mol), diethyl carbonate (15.77 g, 0.134 mol), and sodium ethoxide (5.68 g, 0.0835 mol) in ethanol (80 ml) was refluxed for 8.5 hr. Removal of the ethanol was followed by treatment with water and neutralization with hydrochloric acid. Extraction with chloroform gave white crystals which were recrystallized from a mixture of acetone and hexane, yield 3.00 g (39.0%). Sublimation at 80° at a pressure of 0.08 mm gave a sample melting at 186–187°: ir (Nujol) 3225 (NH), 1775 and 1685 cm^{-1} (C=O); nmr (DMSO-*d*₆) δ 8.40 (s, 1, NH), 7.50 (m, 5, C₆H₅), 3.15 (s, 3, CH₃).

Anal. Calcd for C₉H₉N₃O₂: C, 56.54; H, 4.74; N, 21.98; mol wt, 191. Found: C, 56.27; H, 4.92; N, 21.75; mol wt (mass spectrum), 191.

***N*-Nitroso-*n*-hexylmethylamine.**—A solution of hexylmethylamine¹⁶ (94.4 g, 0.819 mol) in acetic acid (128 ml) and water (425 ml) was treated simultaneously dropwise with a solution of sodium nitrite (176.0 g, 2.55 mol) in water (425 ml) and a solution of acetic acid (93.5 ml) in water (255 ml). The resulting

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(13) O. Westphal and D. Jerehel, *ibid.*, **73**, 3, 1002 (1940).

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solution was stirred for 2 hr at 55–65° and then extracted with three 200-ml portions of ether. Removal of the ether gave an oil which was purified by distillation: yield 105.4 g (89.2%); bp 108–110° (8 mm); n_D^{25} 1.4524; nmr (neat) δ 4.11, 3.53 (two t, 2, NCH₂), 3.70, 2.96 (two s, 3, NCH₃), 1.11 (m, 11, C₅H₁₁).

Anal. Calcd for C₇H₁₆N₂O: C, 58.80; H, 11.19; N, 19.42. Found: C, 58.58; H, 10.98; N, 19.81.

1-Hexyl-1-methylhydrazine.—A solution of *N*-nitrosohexylmethylamine (104.8 g, 0.727 mol) in anhydrous ether (500 ml) was added dropwise to a solution of lithium aluminum hydride (44.9 g, 1.18 mol) in anhydrous ether (1000 ml). The resulting mixture was refluxed for 1 hr and the excess lithium aluminum hydride was decomposed with water. Filtration of the mixture, followed by removal of the ether, gave an oil which was purified by distillation: yield 71.5 g (75.5%); bp 68–69° (15 mm); n_D^{20} 1.4562; ir (neat) 3380, 3250 cm⁻¹ (NH₂); nmr (neat) δ 3.02 (s, 2, NH₂), 2.45 (t, 2, NCH₂), 2.33 (s, 3, NCH₃), 1.13 (m, 11, C₅H₁₁).

Anal. Calcd for C₇H₁₆N₂: C, 64.56; H, 13.94; N, 21.51. Found: C, 64.91; H, 13.91; N, 21.65.

1-Hexyl-1-methyl-2-benzoylhydrazine.—A well-stirred solution of 1-hexyl-1-methylhydrazine (20.0 g, 0.154 mol) and triethylamine (17.3 g, 0.171 mol) in dry benzene (100 ml) was treated with benzoyl chloride (23.4 g, 0.166 mol) in dry benzene (100 ml), and the resulting mixture was refluxed for 1 hr. The mixture was cooled and treated with sodium hydroxide (14.9 g, 0.373 mol) in water (100 ml). Separation of the layers was followed by a further extraction of the aqueous layer with benzene. Removal of the benzene from the combined extracts gave white crystals (20.4 g, 0.087 mol, 56.5%) which upon recrystallization from ethanol-hexane melted at 81–82°: ir (Nujol) 3290 (NH), 1640 cm⁻¹ (CO); nmr (DCCl₃) δ 8.21 (s, 1, NH), 7.55 (m, 5, C₆H₅), 2.75 (t, 2, NCH₂), 2.63 (s, 3, NCH₃), 1.08 (m, 11, C₅H₁₁).

Anal. Calcd for C₁₄H₂₂N₂O: C, 71.75; H, 9.46; N, 11.96. Found: C, 71.87; H, 9.28; N, 11.99.

Reaction of Phenyl Isocyanate with 1-Hexyl-1-methyl-2-benzoylhydrazine.—A mixture of 1-hexyl-1-methyl-2-benzoylhydrazine (5.80 g, 0.0248 mol) and phenyl isocyanate (3.09 g, 0.0259 mol) was heated to 170° for 3 hr, and the 1-hexene formed was collected in a receiver cooled with Dry Ice in acetone, yield 0.65 g (31.2%).

The pyrolysis residue (7.97 g) was chromatographed on silica gel in the same manner as was described under the pyrolysis of the aminimide. Products isolated in the order listed were benzanilide (1.65 g, 8.37 mmol, 33.8%), 1,3-diphenylurea (0.97 g, 4.57 mmol, 18.5%), 1-hexyl-1-methyl-2-benzoylhydrazine (3.65 g, 15.6 mmol, 62.9%), and 1-phenyl-3-methylurazole (1.56 g, 8.16 mmol, 33%).

Reaction of Hexylmethylaminoisocyanate with Phenyl Isocyanate.—A well-stirred cold solution of phosgene (11.4 g, 0.116 mol) in benzene (90 ml) was treated dropwise with a solution of 1-hexyl-1-methylhydrazine (10.0 g, 0.0773 mol) and quinoline (20.8 g, 0.161 mol) in benzene (50 ml), and the resulting mixture was stirred at room temperature for 1 hr. Removal of quinoline hydrochloride by filtration and benzene and excess phosgene by distillation under reduced pressure gave a viscous oil (17.8 g). The resulting oil was heated at 230–240° and the distillate was added to a solution of phenyl isocyanate (14.9 g, 0.118 mol) in benzene (20 ml). The resulting solution was refluxed for 1 hr, and the excess phenyl isocyanate and benzene were removed under reduced pressure. The white solid (1.86 g) obtained when chromatographed on silica gel using a mixture of benzene and ethyl acetate (1:1) as a solvent gave 1,3-diphenylurea (0.91 g, 4.3 mmol, 7.3%) and 1-phenyl-3-methylurazole (2) (0.61 g, 3.19 mmol, 4.1%).

1,1-Dihexyl-2-acetylhydrazine.—A well-stirred solution of 1,1-dihexylhydrazine (32.65 g, 0.163 mol) in benzene (100 ml) was treated with acetic anhydride (24.8 g, 0.243 mol) in benzene (100 ml). The resulting solution was cooled and treated with a slight excess of aqueous sodium hydroxide. The benzene layer upon removal of the solvent gave an oil which, upon purification by distillation, solidified: yield 35.1 g (0.145 mol, 88.8%); bp 118–120° (0.07 mm); mp 56–58°; ir (Nujol) 3300, 3125 (NH),

1685 cm⁻¹ (C=O); nmr (DCCl₃) δ 7.76 (s, 1, NH), 2.64 (t, 4, N(CH₂)₂), 2.08, 1.91 (2 s, 3, CH₃CO), 1.23 (m, 22, (C₅H₁₁)₂).

Anal. Calcd for C₁₄H₃₀N₂O: C, 69.36; H, 12.48; N, 11.56. Found: C, 69.48; H, 12.53; N, 11.44.

1,1-Dihexyl-1-methyl-2-acetylhydrazonium *p*-Toluenesulfonate.—A solution of 1,1-dihexyl-2-acetylhydrazine (39.9 g, 0.165 mol) and methyl *p*-toluenesulfonate (46.0 g, 0.247 mol) in acetonitrile (100 ml) was refluxed for 29 hr. Removal of the solvent and excess methyl *p*-toluenesulfonate gave a dark brown oil (66.9 g) which was purified by chromatography on silica gel. Elution with ethyl acetate gave an initial fraction of methyl *p*-toluenesulfonate and 1,1-dihexyl-2-acetylhydrazine. The second fraction gave an oil (61.2 g, 0.143 mol, 86.7%) which on the basis of spectra was 1,1-dihexyl-1-methyl-2-acetylhydrazonium *p*-toluenesulfonate: ir (neat) 3480, 3250 (NH), 1710 (CO), 1190 cm⁻¹ (SO₃⁻); nmr (DCCl₃) δ 10.45 (s, 1, NH), 7.45 (q, 4, C₆H₄), 3.85 (t, 4, N-(CH₂)₂), 3.39 (s, 3, NCH₃), 2.21 (s, 3, CH₃C₆H₄), 1.95 (s, 3, CH₃CO), 1.15 (m, 22, (C₅H₁₁)₂).

Anal. Calcd for C₂₂H₄₀N₂O₄S: C, 61.64; H, 9.41; N, 6.54. Found: C, 61.43; H, 9.65; N, 6.59.

1,1-Dihexyl-1-methylamine-2-acetyl-imide (1a).—1,1-Dihexyl-1-methyl-2-acetylhydrazonium *p*-toluenesulfonate (56.6 g, 0.132 mol), in chloroform (100 ml) was stirred with excess aqueous sodium hydroxide until the aqueous layer remained alkaline. The chloroform layer was separated and the solvent was removed under reduced pressure. The oil obtained was dried by adding dry benzene and distilling off the benzene. The product was a brown oil (26.4 g, 0.103 mol, 78%) which was homogeneous according to tlc analysis on silica gel using ethyl acetate as a solvent: ir (neat) 1580 cm⁻¹ (CO); nmr (DCCl₃) δ 3.63 (t, 4, N(CH₂)₂), 3.18 (s, 3, NCH₃), 1.79 (s, 3, CH₃CO), 1.25 (m, 22, (C₅H₁₁)₂).

Anal. Calcd for C₁₅H₃₂N₂O: C, 70.25; H, 12.58; N, 10.93. Found: C, 70.34; H, 12.45; N, 11.11.

Pyrolysis of 1,1-Dihexyl-1-methylamine-2-acetyl-imide.—The aminimide (11.14 g, 0.0434 mol) was heated at 140° for 2 hr and the volatile products were trapped in a solution of aniline in dry toluene. The white solid formed in this solution was filtered and proved to be 1-methyl-3-phenylurea (1.98 g, 0.0131 mol, 32%), mp 149–150°. The filtrate upon distillation gave 1-hexene (2.33 g, 0.0277 mol 68%), bp 62–63°.

The residue (7.67 g) from the pyrolysis was analyzed by gas chromatography on a silicon rubber W98 column at 180° and found to consist of a mixture of dihexylmethylamine and 1-hexyl-1-methyl-2-acetylhydrazine in a ratio of 35.5:64.5. Identification was made by fractional distillation and comparison with authentic samples. Pyrolysis gave similar results at 175°.

1-Hexyl-1-methyl-2-acetylhydrazine.—A solution of 1-hexyl-1-methylhydrazine (20.45 g, 0.157 mol) in benzene (100 ml) was treated dropwise with acetic anhydride (16.0 g, 0.157 mol) in benzene (40 ml). The resulting solution was allowed to cool and then was treated with a slight excess of aqueous sodium hydroxide. The benzene layer upon removal of the solvent gave an oil which was purified by distillation: yield 21.3 g (78.7%); bp 85–87° (0.2 mm); n_D^{20} 1.4568; ir (neat) 3300, 3110 (NH), 1670 cm⁻¹ (C=O); nmr (DCCl₃) δ 8.80, 8.22 (2 s, 1, NH), 2.68 (m, 2, NCH₂), 2.59, 2.52 (2 s, 3, NCH₃), 2.03, 1.89 (2 s, 3, CH₃CO), 1.15 (m, 11, C₅H₁₁).

Anal. Calcd for C₉H₂₀N₂O: C, 62.74; H, 11.70; N, 16.27. Found: C, 62.68; H, 11.90; N, 16.38.

Registry No.—1a, 28538-65-0; 1b, 28538-66-1; 2, 28538-67-2; 1,1-dihexyl-2-benzylhydrazine, 28538-68-3; 1,1-dihexyl-1-methyl-2-benzoylhydrazonium *p*-toluenesulfonate, 28538-69-4; *N*-nitroso-*n*-hexylmethylamine, 28538-70-7; 1-hexyl-1-methylhydrazine, 28538-71-8; 1-hexyl-1-methyl-2-benzoylhydrazine, 28538-72-9; 1,1-dihexyl-2-acetylhydrazine, 28538-73-0; 1,1-dihexyl-1-methyl-2-acetylhydrazonium *p*-toluenesulfonate, 28538-74-1; 1-hexyl-1-methyl-2-acetylhydrazine, 28538-75-2.