A much lower yield (1.7 g.) was obtained when the diacetate of I (7.0 g.) was used in place of the free base.

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Acetophenone Mesitylhydrazone in the Fischer Indole Synthesis. Migration of a Phenacyl Group[1]

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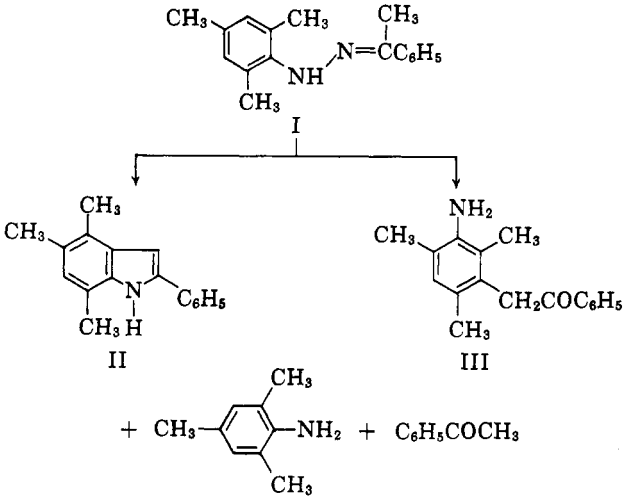
Acetophenone mesitylhydrazone (I) was transformed by zinc chloride in nitrobenzene at 120–135° into 2-phenyl-4,5,7-trimethylindole (II, 10%), product of a 1,2 methyl migration; 3-phenacylmesidine (III, 2%), product of a 1,2 phenacylimino group migration; mesidine (16%); and acetophenone (8%). The indole II and 3-phenacylmesidine (III) were identified with products of independent synthesis. Their formation is rationalized by postulating an intermediate analogous to those proposed to account for similar reactions encountered previously; in this case the intermediate leads in part to II and in part to III. Mesidine and acetophenone are postulated to be derived from the tautomer of I by disproportionation of hydrogen.

The Fischer reaction of acetophenone 2,6-xylylhydrazone, conducted in nitrobenzene with zinc chloride as the promoter, led to 2-phenyl-3a,5-dimethyl-3a,4,7,-7a-tetrahydro[3H]pseudoindolone-4 and 2-phenyl-4,7-dimethylindole.[3] No methyl group migration was involved in the formation of the ketonic product; a 1,2-methyl migration must have accompanied that of the indole. Cyclohexanone mesitylhydrazone, on the other hand, was converted in acetic acid into 6,7,8-trimethyl-1,2,3,4-tetrahydrocarbazole, the result of an apparent 1,4-methyl migration.[4]

The differences in behavior of these two *ortho* dimethylated arylhydrazones under the conditions of the Fischer reaction might be ascribed to the structural differences in the ketone and/or the aryl groups, or differences in the medium might be chiefly responsible. For the purpose of deriving evidence bearing on the structural factors, acetophenone mesitylhydrazone (I) was next selected for investigation in the Fischer reaction; this substance, of course, comprises the ketone fragment of one of the arylhydrazones previously studied and the arylhydrazine moiety of the other. The observations derived from this investigation form the subject of this paper.

Acetophenone mesitylhydrazone (I), prepared in 92% yield from acetophenone and mesitylhydrazine,[4] was not stable in air and accordingly had to be used promptly after its isolation. Like acetophenone 2,6-xylylhydrazone, I underwent reaction in nitrobenzene in the presence of zinc chloride at 120–135°. The reaction mixture afforded four products: 2-phenyl-4,5,7-trimethylindole (II, 10%), a product of 1,2-methyl migration; 3-phenacylmesidine (III, 2%); mesidine (16%); and acetophenone (8%).



Mesidine was characterized through its known N-acetyl derivative, acetophenone by its refractive index and infrared spectrum. The indole II was synthesized independently by the Fischer reaction of acetophenone 2,4,5-trimethylphenylhydrazone.

In addition to II, the Fischer reaction of acetophenone 2,4,5-trimethylphenylhydrazone yielded a compound whose composition and spectroscopic properties were consistent with those to be expected of a higher homolog of the ketonic substance which was the principal product derived from acetophenone 2,6-xylylhydrazone.[3] The ultraviolet, infrared, and n.m.r. spectra left little doubt that the compound was one of the isomers IV or V. The single, broad, vinyl proton signal at τ 4.16 in the n.m.r. spectrum might be consistent with V as well as with IV, since models indicate that the vinyl and the adjoining tertiary hydrogens in V are inclined at a dihedral angle of nearly 90°, so that splitting of the vinyl proton signal might not occur.[5] More

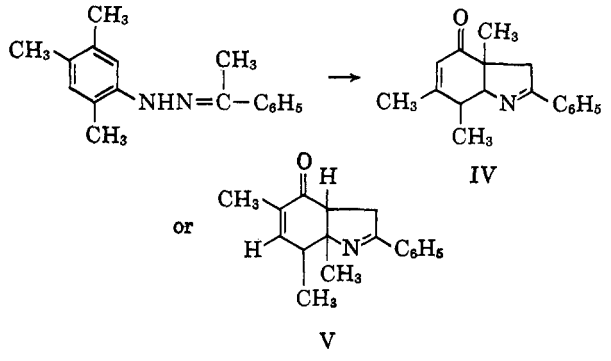
(1) Submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, Carnegie Institute of Technology.

(2) Esso Foundation Fellow, 1961–1962.

(3) R. B. Carlin and D. P. Carlson, *J. Am. Chem. Soc.*, **79**, 3605 (1957); **81**, 4673 (1959).

(4) R. B. Carlin and M. S. Moores, *ibid.*, **81**, 1259 (1959); **84**, 4107 (1962).

(5) *Cf.* M. Karplus, *J. Chem. Phys.*, **30**, 11 (1959); *J. Am. Chem. Soc.*, **85**, 2870 (1963).



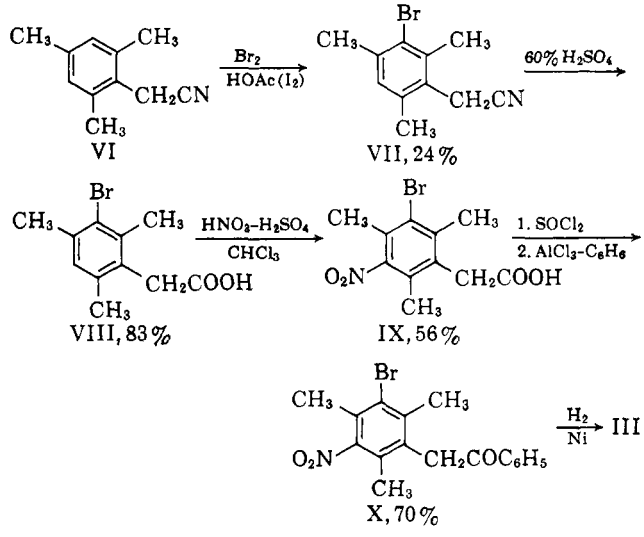
IV

or

V

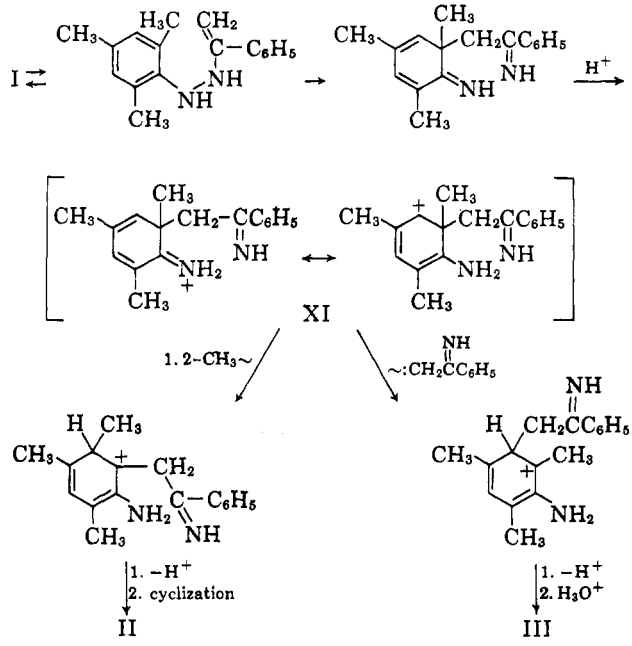
detailed examination will be required before a definitive choice between IV and V can be made.

3-Phenacylmesidine (III) was shown to have the assigned structure by its composition, its spectroscopic and chemical properties, and by independent synthesis. The infrared spectrum showed bands at 2.87 and 2.91 μ (N–H) and an intense band at 5.90 μ (carbonyl conjugated with aromatic unsaturation). The ultraviolet spectrum, showing maxima at 238 mμ (ϵ 22,000) and 287 mμ (ϵ 3000), was represented by a curve that was substantially identical with one constructed by summing the curves for mesidine and acetophenone. Nitrous acid converted III to a clear yellow solution that combined with alkaline β-naphthol to yield a red-brown dye.



The chief obstacle in this synthesis of III was the introduction of but one nitro group into the aromatic ring of mesitylacetonitrile (VI) or the corresponding acid; recorded experience indicated that direct nitration would bring about the introduction of two nitro groups. The most attractive approach to the problem appeared to be one based on a method employed by Smith[6] to prepare mononitro derivatives of the tetramethylbenzenes: a single bromine atom was first introduced into the ring (VII); then, after hydrolysis to VIII, nitration afforded a mononitro monobromo derivative IX, which, following conversion *via* the Friedel–Crafts synthesis to X, underwent simultaneous hydrogenolysis of bromine and hydrogenation of the nitro group to afford III.

The formation of II and III from the Fischer reaction of I can be rationalized by a mechanism that features an intermediate species XI of a type previously proposed to account for 1,2-methyl migrations,[3,7] formation of a hydroindolone derivative,[3,8] and an apparent 1,4-methyl migration.[4] In this instance, the intermediate XI is presumably converted in part to II *via* a 1,2-methyl migration. In this respect the behavior of I parallels that of acetophenone 2,6-xylylhydrazone, but, unlike the latter, I does not also form a ketonic product; instead, the intermediate XI derived from it apparently undergoes 1,2 migration (or even 1,4 migration) of the phenacylimino group ultimately to produce III. Thus, in some circumstances the phenacylimino group must migrate in competition with a geminal methyl group in structural situations analogous to those existing in the dienone–phenol rearrangement (*cf.* ref. 3).



The mechanism represented above and analogous mechanisms postulated heretofore[3,4,7,8] have been subject to uncertainties related to the reaction sequence; for example, a mechanism can be formulated to account for the transformation of XI into II in which cyclization precedes rather than follows methyl migration. However, phenacylimino migration leading to III from XI occurs without any heterocyclic ring closure being possible in the sequence. This observation strongly suggests, but of course does not prove, that methyl migration, as well as phenacylimino migration, proceeds directly from XI, and that cyclization to II follows methyl migration.

Mesidine and acetophenone, both "major" products of the zinc chloride induced transformation of I, could be formed from the ene–hydrazine tautomer of I by a disproportionation reaction entirely similar to that postulated previously to account for an analogous reaction.[8]

Experimental

Acetophenone Trimethylarylhydrazones.—A solution of 150 g. of trimethylarylhydrazine[9] in 500 ml. of 95% ethanol containing

(6) L. I. Smith and C. L. Moyle, *J. Am. Chem. Soc.*, **55**, 1676 (1933); L. I. Smith and D. Tenenbaum, *ibid.*, **57**, 1293 (1935).

(7) R. B. Carlin, W. O. Henley, Jr., and D. P. Carlson, *ibid.*, **79**, 5712 (1957).

(8) R. B. Carlin, A. J. Magistro, and G. J. Mains, *ibid.*, **86**, 5300 (1964).

(9) Preparations of both mesitylhydrazine and of 2,4,5-trimethylphenylhydrazine are reported in ref. 4.

5 ml. of glacial acetic acid was treated with 138 g. of acetophenone. The solution was heated at reflux for 10 min. and then 250 ml. of water was added to the hot solution. Chilling for several hours effected the separation of the arylhydrazone in yellow clumps, which were collected on a filter and pressed dry. Both trimethylarylhydrazones could be kept in a vacuum desiccator for only 1–2 hr. before evidence of decomposition was noticeable; neither could be successfully dried and prepared for analysis. Acetophenone mesitylhydrazone, formed in 92% yield, melted over the range 54–65°; the 2,4,5-trimethylphenylhydrazone, formed in 95% yield, had m.p. 135°.

Transformations of Acetophenone Mesitylhydrazone by Zinc Chloride in Nitrobenzene.—A solution of 233 g. of acetophenone mesitylhydrazone (I), which had been freshly prepared and dried for 1 hr. in a vacuum desiccator immediately prior to use, in 750 ml. of nitrobenzene was treated with 375 g. of freshly fused zinc chloride. The vigorously stirred mixture was warmed gradually to 130°, when the temperature rose abruptly. The mixture was cooled until the temperature declined to 120°; then it was maintained in the range 120–135° for 45 min. The very dark mixture was subjected to steam distillation until the organic material volatile with steam ceased to collect in the distillate.

The organic layer from the distillate, principally nitrobenzene, was extracted with two 100-ml. portions of 10% aqueous hydrochloric acid. Treatment of the acid extract with excess 10% aqueous sodium hydroxide caused the separation of a red oil, which was extracted into 100 ml. of ether. The ether solution was washed with water until the washings were neutral, dried over anhydrous sodium sulfate, filtered, and stripped of solvent under reduced pressure. From the 19.6 g. of the residual red oil, about 0.5 ml. was converted by acetic anhydride and sodium acetate in aqueous medium to a white N-acetyl derivative, m.p. 215–216°, following recrystallization from benzene–cyclohexane. A mixture with authentic 2,4,6-trimethylacetanilide, m.p. 216°, gave no melting point depression.

The residue from the steam distillation was extracted with three 100-ml. portions of ether; emulsions were destroyed by addition of 20% aqueous sodium hydroxide. The ether solutions were washed with water until the washings were neutral and then extracted with 50-ml. portions of 10% aqueous hydrochloric acid until no precipitate was formed when the acid extracts were made basic with 10% aqueous sodium hydroxide. The ether solution, following acid extraction, was washed until neutral and dried over anhydrous sodium sulfate. Following filtration, solvent was evaporated from the solution under reduced pressure, and the 58.6 g. of black residue was subjected to distillation through a short Vigreaux column. Three fractions were collected: (1) 8.6 g., b.p. 55–150° (1 mm.); (2) 21.4 g., b.p. 150–210° (1 mm.); and (3) 28.5 g., tarry residue. Fraction 1, redistilled at atmospheric pressure, afforded a liquid, b.p. 199–200°, whose infrared spectrum was identical with that of an authentic sample of acetophenone. Fraction 2 solidified to a yellow mass, which was subjected to sublimation at 85° (0.01 mm.); the sublimate was recrystallized from *n*-hexane to give 21.4 g. of fine, white needles, m.p. 128–129°. A sample colored Ehrlich's reagent deep red and was identified as **2-phenyl-4,5,7-trimethylindole (II)** which had λ_{max} 252, 313 mμ, ϵ_{max} 22,000, 32,000 (for a 10^{-4} *M* solution in ethanol); and infrared absorption (40 mg./ml. in chloroform) at 2.86 i, 3.34 i, 3.44 i, 3.50 m, 6.21 i, 6.74 i, 6.90 i, 7.15 m, 7.28 m, 7.44 w, 7.54 i, 7.85 w, 8.01 m, 8.32 w, 8.68 w, 9.05 w, 9.33 w, 9.50 w, 9.72 w, 9.96 w, 10.70 w, 11.04 w, and 11.64 m μ.[10]

Anal. Calcd. for $C_{17}H_{17}N$: C, 86.77; H, 7.28; N, 5.95. Found: C, 86.79; H, 7.22; N, 6.10.

The aqueous hydrochloric acid extracts from the ether solution of the residue were made basic with 10% aqueous sodium hydroxide and then extracted with two 100-ml. portions of ether. The ether extract was dried over anhydrous sodium sulfate and filtered; the ether was removed under reduced pressure. The light gray, solid residue was subjected to sublimation at 130° (0.01 mm.). The sublimate was recrystallized from ether to produce 3.8 g. of a fluffy, white solid, m.p. 188–189°, identified as **3-phenacylmesidine (III)** which had λ_{max} 238, 287 mμ, ϵ_{max} 21,000, 3000 (10^{-4} *M* in ethanol); and λ_{max} 242 mμ, ϵ_{max} 16,000 (10^{-4} *M* in 0.1 *N* ethanolic hydrochloric acid); and infrared absorptions (40 mg./ml. in chloroform) at 2.87 w, 2.91 w, 3.32 m, 3.42 m, 3.48 w, 5.90 i, 6.14 i, 6.23 m, 6.30 m, 6.72 i, 6.79 i, 6.90 i, 7.04 w, 7.24 w, 7.53 i, 7.64 m, 7.92 w, 8.12 m, 8.48 m, 9.12 m, 9.72 w, 9.88 w, 9.98 m, 10.12 i, 11.45 m, and 11.88 w μ.[10]

Anal. Calcd. for $C_{17}H_{19}NO$: C, 80.57; H, 7.56; N, 5.53. Found: C, 80.62; H, 7.86; N, 5.52.

Transformation of Acetophenone 2,4,5-Trimethylphenylhydrazone by Zinc Chloride in Nitrobenzene.—A solution of 95 g. of freshly prepared acetophenone 2,4,5-trimethylphenylhydrazone in 500 ml. of nitrobenzene was dried over anhydrous sodium sulfate, filtered, and treated with 150 g. of fused zinc chloride, and the mixture was stirred and warmed to 125°, when the temperature rose rapidly and spontaneously. The mixture was cooled until the temperature again dropped to 125°, then it was maintained for 1 hr. in the temperature range 125–130°. The mixture was subjected to steam distillation, the residue was extracted with two 200-ml. portions of ether, and the ether extracts were washed with water until neutral. The ether solution was then extracted with two 100-ml. portions of 10% aqueous hydrochloric acid and washed with water until neutral; the water washings were added to the hydrochloric acid extract. The extracted ether solution was dried over sodium sulfate and filtered; ether was removed under reduced pressure. The 65.4 g. of residual brown solid was dissolved in hot *n*-hexane. Chilling caused the separation of 25.4 g. (26.3%) of 2-phenyl-4,5,7-trimethylindole (II), white needles, m.p. 128–129°, after further recrystallization from *n*-hexane. A mixture of this sample with one obtained from acetophenone mesitylhydrazone showed no melting point depression. Infrared spectra of samples from the two sources were indistinguishable.

The aqueous acid extracts from the original ether solution were made basic with 10% aqueous sodium hydroxide, and the mixture was extracted with two 50-ml. portions of ether. The ether solution was dried over anhydrous magnesium sulfate and filtered; the solvent was removed under reduced pressure. Sublimation of the residual yellow solid at 120° (0.01 mm.) and recrystallization of the sublimate from ether afforded 6.3 g. (6.6%) of a white crystalline solid, m.p. 131–132°. This substance, on the basis of the following analytical and spectral evidence, is tentatively assigned either the structure **2-phenyl-3a,6,7-trimethyl-3a,4,7,7a-tetrahydro[3H]pseudoindolone-4 (IV)** or the isomeric **2-phenyl-5,7,7a-trimethyl-3a,4,7,7a-tetrahydro[3H]pseudoindolone-4 (V)**. The ultraviolet spectrum had λ_{max} 242 mμ, ϵ_{max} 33,000 (10^{-4} *M* in ethanol); and λ_{max} 240, 273 mμ, ϵ_{max} 19,000, 23,000 (10^{-4} *M* in 0.1 *N* aqueous ethanolic hydrochloric acid). The infrared spectrum (40 mg./ml. in chloroform) showed bands at 3.40 m, 3.52 w, 6.04 i, 6.21 m, 6.30 m, 6.70 w, 6.84 w, 6.92 m, 7.28 m, 7.45 m, 7.55 w, 7.67 m, 7.88 m, 8.55 w, 8.73 w, 8.90 w, 9.05 w, 9.25 w, 9.64 w, 9.78 m, 9.98 w, 10.12 w, 10.37 w, 10.65 w, 10.95 w, 11.20 w, 11.45 m, and 11.60 i μ; and (40 mg./ml. in chloroform saturated with anhydrous hydrogen chloride) 3.36 i, 4.22 m, 6.04 i, 6.12 i, 6.25 m, 6.60 m, 7.04 m, 7.25 m, 7.30 w, 7.50 w, 8.24 i, 9.26 w, 10.79 m, 11.40 m, 11.78 m, 12.70–13.75 i, and 14.95 m μ. The n.m.r. spectrum (165 mg./ml. in carbon tetrachloride) had τ 8.59–8.71 (doublet), J = 12.5 c.p.s.; 8.06 (singlet); 7.51 (doublet), J = 3 c.p.s.; 7.22 (doublet), J = 3 c.p.s.; 7.02 (quartet), J = 12.5 c.p.s.; 6.63 (doublet), J = 2 c.p.s.; 6.35 (doublet), J = 2 c.p.s.; 6.09 (quartet), J = 2 c.p.s.; 7.22–7.51; 6.35–6.63; 4.16 (broad); 2.65 (singlet); and 2.26 (singlet).

Anal. Calcd. for $C_{17}H_{19}NO$: C, 80.59; H, 7.56; N, 5.53. Found: C, 80.41; H, 7.65; N, 5.71.

The ultraviolet and infrared spectra, both in neutral and acid solutions, were strikingly reminiscent of those of an homologous pseudoindolone reported previously.[3] Particularly to be noticed are the division of the single, intense ultraviolet band at 242 mμ, characteristic of the neutral species, into a weaker band at substantially the same wave length and a second band centered at 273 mμ in the salt; and the shift of the 6.21-μ band in the infrared to 6.12 μ in the presence of acid. Both of these phenomena characterized the homolog reported earlier, and their significance was considered in terms of structural features.[3]

3-Bromomesitylacetonitrile (VI).—A solution of 10.5 g. of bromine in 35 ml. of acetic acid was added over a 2.5-hr. period to a solution of 10 g. of mesitylacetonitrile[11] in 150 ml. of glacial acetic acid to which one crystal of iodine had been added; the

(10) w = 50–100% transmission, m = 10–50%, i = 0–10%.

(11) R. C. Fuson and N. Rabjohn, "Organic Syntheses," Coll. Vol. III, John Wiley and Sons, Inc., New York, N. Y., 1955, pp. 557–558.

temperature was maintained at 15°. The stirred mixture was then allowed to warm to room temperature during another 3 hr. During all of this time the reaction vessel was protected from direct light. When the mixture was poured over crushed ice, a yellow solid precipitated, which was collected by filtration, slurried in 200 g. of an ice–water mixture until it became white, again collected by filtration, and washed with water at 0° until the filtrate was neutral. Recrystallization from 95% ethanol afforded 6 g. (24%) of colorless needles: m.p. 114.5–115.5°; infrared spectrum (2 mg. in potassium bromide pellet), 3.40 m, 4.44 m, 6.85 i, 6.95 i, 7.26 m, 7.69 m, 8.08 w, 8.73 i, 9.70 w, 9.84 m, 10.18 i, 10.43 w, 10.85 m, 11.44 i, 12.52 m, and 13.95 m μ; the n.m.r. spectrum (150 mg./ml. in carbon tetrachloride) showed a single aromatic proton at τ 3.36, two methylene protons at 6.6, and nine methyl protons, three each at 7.58, 7.71, and 7.77.

Anal. Calcd. for $C_{11}H_{12}BrN$: C, 55.48; H, 5.08; Br, 33.56. Found: C, 55.53; H, 4.89; Br, 33.78.

3-Bromomesitylacetic Acid (VIII).—A solution of 5 g. of 3-bromomesitylacetonitrile (VII) in 50 ml. of concentrated sulfuric acid and 60 ml. of water was stirred and heated under reflux for 6 hr. The cooled mixture was poured over 200 g. of cracked ice, and the gray solid that precipitated was washed until the washings were neutral. A solution of the solid in 5% aqueous sodium hydroxide was boiled with Norit for 10 min., filtered through a Celite filter pad, cooled, and acidified with concentrated hydrochloric acid. The white solid was first sublimed at 125° (0.1 mm.) and then recrystallized from 125 ml. of 2:3 ethanol–water, when it formed 4.5 g. (83%) of long, silky white needles: m.p. 171.5–173°; infrared spectrum (2 mg. in potassium bromide pellet), 3.35 i, 3.62 m, 3.77 m, 5.86 i, 6.85 m, 7.04 i, 7.23 m, 7.55 m, 8.05 i, 8.20 i, 8.65 m, 9.84 m, 10.15 m, 10.36 w, 10.82 w, 11.18 m, 11.62 m, 12.09 m, 13.70 m, and 14.70 i μ.

Anal. Calcd. for $C_{11}H_{13}BrO_2$: C, 51.38; H, 5.09; Br, 31.08. Found: C, 51.48; H, 5.07; Br, 31.15.

3-Bromo-5-nitromesitylacetic Acid (IX).—A stirred mixture of 5.14 g. of 3-bromomesitylacetic acid (VIII), 5 ml. of concentrated sulfuric acid, and 25 ml. of chloroform was treated with a solution of 1.38 g. of nitric acid (*d* 1.6 g./ml.) in 25 ml. of chloroform; the combined mixture was warmed at 50° for 0.5 hr., cooled to room temperature, washed with two 100-ml. portions of water, and then extracted with two 100-ml. portions of 5% aqueous sodium hydroxide. The aqueous basic solution was acidified with 10% aqueous hydrochloric acid, and the white precipitated solid was collected and washed with water until the washings were neutral. Sublimation of this material at 180–200° (0.1 mm.) and recrystallization of the sublimate from 3:1 chloroform–cyclohexane afforded 3.4 g. (56%) of short, white needles: m.p. 246–247°; infrared spectrum (2 mg. in potassium bromide pellet), 3.30 i, 3.60 w, 3.75 w, 3.90 w, 5.85 i, 6.48 i, 7.04 i, 7.30 i, 7.55 w, 7.78 w, 8.10 i, 8.38 w, 8.45 w, 9.77 m, 10.15 m, 10.86 i, 11.06 m, 11.68 i, 12.05 i, 13.13 m, and 14.30 m μ.

Anal. Calcd. for $C_{11}H_{12}BrNO_4$: C, 43.73; H, 4.00; Br, 26.45; N, 4.63. Found: C, 43.76; H, 4.01; Br, 27.41; N, 4.30.

3-Bromo-5-nitro-1-phenacylmesitylene (X).—A slurry of 3.02 g. of 3-bromo-5-nitromesitylacetic acid (IX) in 100 ml. of dried benzene was stirred and treated with a 10% molar excess of thionyl chloride, and the mixture was heated under reflux until the evolution of hydrogen chloride ceased (total time, about 4 hr.). The clear, yellow solution was cooled to room temperature, treated with 2.7 g. of aluminum chloride, and the mixture was warmed to about 50°, when hydrogen chloride began being evolved copiously. After the evolution of hydrogen chloride had subsided, the mixture was heated under reflux for an additional 2 hr., cooled to room temperature, and poured onto 10 ml. of concentrated hydrochloric acid and 200 g. of ice. The aqueous slurry was extracted with two 50-ml. portions of chloroform, the combined chloroform extracts were washed with water until the washings were neutral, and then extracted with two 50-ml. portions of 5% aqueous sodium hydroxide. Again the chloroform extract was washed with water until neutral, dried, and the solvent was removed under reduced pressure. The yellow residual solid was first purified by sublimation at 150–180° (0.1 mm.) and then by recrystallization from chloroform–cyclohexane to afford 2.4 g. (70%) of a fluffy white solid: m.p. 228–229°; infrared spectrum (2 mg. in potassium bromide pellet), 3.40 m, 5.92 i, 6.25 m, 6.30 w, 6.52 i, 6.84 m, 7.28 w, 7.52 w, 8.03 w, 8.22 i, 8.47 m, 8.35 w, 9.10 w, 9.73 m, 10.00 w, 10.14 i, 10.64 w, 10.98 m, 11.17 w, 11.70 i, 11.96 m, 12.38 w, 13.18 i, 13.45 w, 13.68 w, and 14.63 i μ.

Anal. Calcd. for $C_{17}H_{16}BrNO_3$: C, 56.37; H, 4.45; N, 3.87. Found: C, 56.35; H, 4.60; N, 3.65.

Neutralization of the aqueous sodium hydroxide extract with hydrochloric acid precipitated 0.15 g. of unchanged 3-bromo-5-nitromesitylacetic acid IX.

3-Phenacylmesidine (III).—A solution of 1 g. of 3-bromo-5-nitro-1-phenacylmesitylene (X) in 50 ml. of diglyme was pretreated with about 500 mg. of Raney nickel at 80–90° for 2 hr., filtered, treated with about 500 mg. of fresh Raney nickel, and rocked at 145–155° with hydrogen at 2300 p.s.i. for 2 hr. Solvent was stripped under reduced pressure from the filtered product solution, and the residual solid was dissolved in 50 ml. of chloroform. The solution was extracted with six 10-ml. portions of 10% aqueous hydrochloric acid, the acid extracts were then made basic with 10% aqueous sodium hydroxide, and the precipitated solid was extracted into chloroform. This chloroform solution was washed with water until neutral, dried over anhydrous sodium sulfate, and the solvent was removed under reduced pressure. The residual yellow solid was purified by sublimation at 120–130° (0.1 mm.) and then by recrystallization from ether to a fluffy, white crystalline solid, m.p. 188–189°. A mixture with a specimen formed from acetophenone mesityl hydrazone (I) showed no melting point depression, and the infrared spectra of the two samples showed no differences.