Similar treatment of *s*-diacetylhydrazine yielded no isolable product.

General Procedure for Etherification of I.—To 0.05 mole of I was added 0.5–1.0 mole of methanol and 1–2 drops of 6 *N* hydrochloric acid. The solution was refluxed for a predetermined period of time or until most of the solid had dissolved. Solutions were then neutralized with dilute sodium hydroxide and the products were obtained by crystallization from the excess methanol. Oils were obtained after evaporation of the methanol at room temperature. The conversion to the diether appeared to be quantitative in the examples which were checked for conversion. Yields reported refer to isolable recrystallized product. Pertinent experimental data are summarized below. Comparisons to literature melting points are given in Table II.

N,N′-(1,2-Dimethoxyethylene)bisacetamide, N,N′-(1,2-dimethoxyethylene)bisurethan, and **N,N′-(1,2-dimethoxyethylene)-bisacrylamide** were synthesized in about 40% yield using the general procedure outlined above.

N,N′-(1,2-Dimethoxyethylene)bis(2-pyrrolidone).—The reactants were refluxed for 1 hr.; the infrared spectrum of an aliquot evaporated to dryness indicated 100% conversion to the ether.

1,3-Diacetyl-4,5-dimethoxyimidazolidine.—To 3 g. of Vd was added 100 ml. of methanol and 3 drops of concentrated hydrochloric acid. The solution was refluxed for 16 hr. with an aliquot removed after 8 hr. Evaporation of the solvent in both cases produced oils which, on comparison of their infrared spectra, were found to be identical. Also, the hydroxyl band in the 3-μ region was absent. The n.m.r. spectrum of the oil in chloroform and the infrared spectrum established the oil as the dimethyl ether of Vd. Repeated recrystallization attempts from various solvents failed to produce a solid material.

1,3-Dicarbomethoxy-4,5-dimethoxyimidazolidine.—To 0.016 mole of Ve was added 1.0 mole of methanol and 1 drop of 6 *N* hydrochloric acid. The solution was refluxed for 6 hr. with aliquots removed after 1 and 2 hr. Infrared spectra of the residues indicated that less than 10% Ve remained in the 1-hr. aliquot, a trace in the 2-hr. aliquot, and none after refluxing 6 hr. No other products were formed as determined by these spectra.

A Methyl Ether of N,N′-(1,2-Dihydroxyethylene)bis(methylformamide).—Excess methanol and hydrochloric acid, in addition to those quantities given in the general procedures and an 8-hr. reflux period, were used to produce an oil which on examination of the n.m.r. spectrum (D_2O solvent) appeared to be a mixture of the ether, the dihydroxy compound, N-methylformamide, and glyoxal. The peak from the methoxy protons at 3.32 p.p.m. was significant but only one-fifth of the integrated value of the peaks from the N-methyl protons. Repeated crystallization attempts failed to produce a solid product.

Unsuccessful Etherifications.—N,N′-Dihydroxyethylenebisformamide, 1,4-diformyl-2,3,5,6-tetrahydroxypiperazine, 1,4-diformyl-2,3-dihydroxypiperazine (Vb), and 1,4-diformyl-2,3-dihydroxy-5-methylpiperazine (Vc) were all, except for the tetrahydroxypiperazine, recovered essentially unchanged from refluxing solutions of hydrogen chloride and methanol. The conditions used were stronger than those described in the general procedures, *i.e.*, increased methanol and hydrogen chloride with refluxing from 6 to 16 hr. The tetrahydroxypiperazine was only partially soluble in the methanol and was slightly decomposed by the procedure. No attempt was made to etherify Va since this material has been reported[7] to be unstable in hot alcohol.

Acetylation of 1,3-Diformyl-5,6-dihydroxyimidazolidine (Va).—To 1.8 g. of Va was added 40 ml. of acetic anhydride and 1 drop of concentrated sulfuric acid. After 1 hr. at room temperature the acetic anhydride was evaporated at 40° under vacuum. The residue was crystallized from ethyl acetate producing 1 g. of crystals.

Acetylation of 1,4-Diformyl-2,3-dihydroxypiperazine (Vb).—To 45 ml. of acetic anhydride was added 3.0 g. of Vb (m.p. 192–193° with decomposition starting at about 185°). The mixture was stirred for 1 hr. with little or no reaction occurring. On the addition of 1 drop of concentrated sulfuric acid, Vb rapidly went into solution. After 0.5 hr. the solution was chilled and a precipitate was obtained. The filtrate was evaporated to dryness. The precipitate and the residue had identical infrared spectra and were combined and recrystallized from ethyl acetate and then from water. The purified product and the crude precipitate and residue had essentially the same infrared spectra. The yield was quantitative.

Acetylation of 1:1 Formamide–Glyoxal Adduct.—A mixture of 4 g. of adduct (III, R = H), 90 ml. of acetic anhydride, and 0.5 ml. of concentrated sulfuric acid was heated at 60° for 2 hr. A small quantity of the adduct usually remained undissolved. The mixture was filtered while hot and then it was cooled in ice. There was formed a white crystalline product which was filtered and washed with cold water. The product can be recrystallized from acetic acid or benzene. The infrared spectrum had two carbonyl absorptions, and there was no hydroxyl absorption. The acetylated product is 2,3,5,6-tetraacetoxy-1,4-diformylpiperazine.

Acknowledgment.—The authors wish to express their appreciation to E. R. McCall and G. J. Boudreaux of Southern Regional Research Laboratory for assistance in obtaining the spectral data. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, New York.

Synthesis of Cyclopropanetricarboxamides

A. JOHN SPEZIALE, LOWELL R. SMITH, AND JOAN E. FEDDER

Research Department, Agricultural Division, Monsanto Company, St. Louis 66, Missouri

Received November 17, 1964

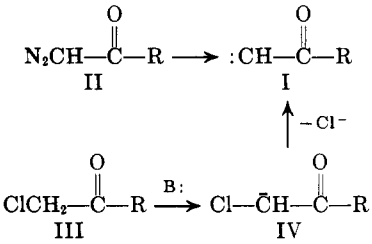
Attempts to prepare carboxamidocarbenes by the action of potassium *t*-butoxide on α-chloroacetamides led to *trans*-1,2,3-cyclopropanetricarboxamides. Evidence for an anionic rather than a carbene mechanism is presented. Hydrolysis of the cyclopropanetricarboxamide yields the triacid. The sequence α-chloroamide to cyclopropanetricarboxamide to *trans*-1,2,3-cyclopropanetricarboxylic acid in a 56% over-all yield appears to be the best synthesis of the latter compound.

Although the existence of carbonylcarbenes I, prepared by the decomposition of diazo compounds II, has been well substantiated,[1] the preparation of these entities by the treatment of α-halocarbonyl compounds with strong base has not been reported. An attempted utilization of an α-halo ester[2] [III, R = $-OC(CH_3)_3$] for the preparation of I produced tar, probably by an acetoacetic ester type condensation. Evidently the loss of chloride ion from IV did not compete favorably with the condensation of IV with III. It appeared that the use of a less reactive carbonyl compound, *i.e.*, an amide,[3] would decrease the condensation and perhaps favor carbene formation.

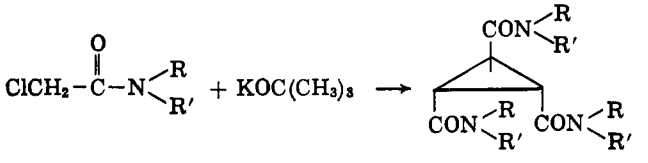
(1) (a) T. Curtius and E. Büchner, *Ber.*, **18**, 2378 (1885); (b) A. Loose, *J. prakt. Chem.*, **79**, 507 (1909); (c) C. Grundmann, *Ann.*, **536**, 29 (1938); (d) W. von E. Doering and L. H. Knox, *J. Am. Chem. Soc.*, **73**, 828 (1951); **78**, 4947 (1956); **83**, 1989 (1961); (e) P. S. Skell and R. M. Etter, *Proc. Chem. Soc.*, 443 (1961).

(2) W. E. Parham and F. C. Lowe, *J. Org. Chem.*, **23**, 1705 (1958).

(3) A. J. Speziale and H. W. Frazier, *ibid.*, **26**, 3176 (1961).

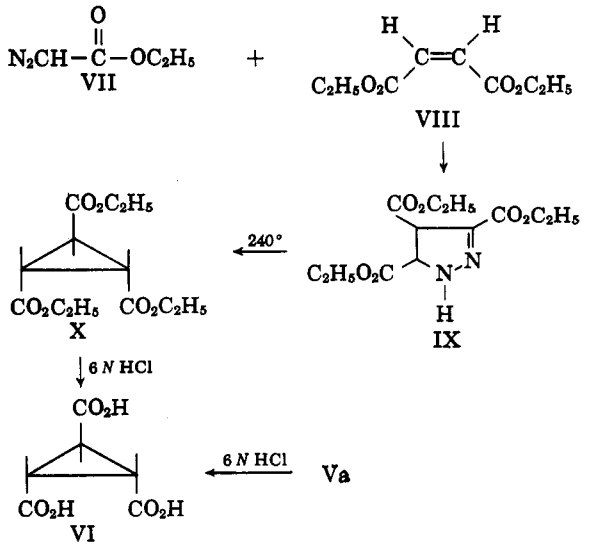


Reaction of N-methyl-α-chloroacetanilide with potassium *t*-butoxide in the presence of cyclohexene led to N,N′,N″-trimethyl-1,2,3-cyclopropanetricarboxanilide (Va), $\lambda_{C=O}$ 1626 cm.$^{-1}$, $\lambda_{max}^{C_2H_5OH}$ 225 mμ (log ϵ 4.35), in 69% yield. No norcarane derivative could be detected. Va was also obtained in ether solution in the absence of cyclohexene in 80% yield. The n.m.r. spectrum of Va exhibited aromatic absorption at τ 2.72, singlets in the ratio 2:1 at 6.78 and 6.93 (two *cis*-CH_3 and one *trans*-CH_3), and complex absorption at 7.28–8.00 (cyclopropane protons). Hydrolysis of Va with refluxing 6 *N* hydrochloric acid gave the water-soluble *trans*-1,2,3-cyclopropanetricarboxylic acid (VI) and N-methylaniline.



Va, R = C_6H_5, R = CH_3
b, R = R′ = CH_3

The structure of Va was established by the independent synthesis of *trans*-1,2,3-cyclopropanetricarboxylic acid (VI). Reaction of ethyl diazoacetate (VII) with diethyl maleate (VIII) formed the pyrazoline IX which on pyrolysis produced triethyl *trans*-1,2,3-cyclopropanetricarboxylate[4] (X). The *trans* configuration of the latter was substantiated by its n.m.r. spectrum which showed two overlapping quadruplets (τ 5.83 and 5.87) and two overlapping triplets (τ 8.75 and 9.79), in addition to cyclopropane proton absorption (τ 7.25–7.75). Hydrolysis of the triester gave the known triacid VI which was identical with the acid derived from the hydrolysis of Va.



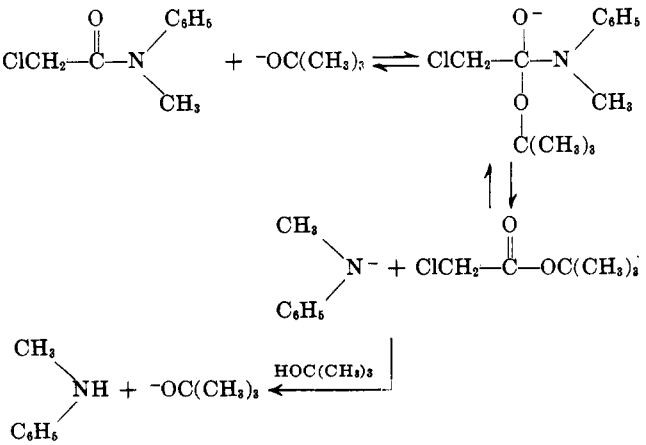
(4) A. Darapsky, *Ber.*, **43**, 1112 (1910).

N,N,N′,N′,N″,N″-Hexamethyl-1,2,3-cyclopropanetricarboxamide (Vb), prepared from N,N-dimethyl-α-chloroacetamide and potassium *t*-butoxide in benzene, showed n.m.r. absorption due to the *cis*-methyl groups at τ 6.91 and 7.17 and the *trans*-methyl groups at 6.77 and 7.12 as well as the cyclopropane protons from 7.26 to 7.70. The nonequivalency of the N-methyl groups of N,N-dimethylamides has been demonstrated.[5]

The reaction of α-chloroacetamides, in particular N-methyl-α-chloroacetanilide, with potassium *t*-butoxide followed by hydrolysis appears to be the best reported synthesis of *trans*-1,2,3-cyclopropanetricarboxylic acid.

The yield of Vb decreased from 50% in benzene to 4% in tetrahydrofuran. N,N-Dimethyl-α-(*t*-butoxy)-acetamide was also isolated in 11 and 39% yields, respectively. The structure of this compound was established by its n.m.r. spectrum which showed absorption of the α-protons at τ 6.03, the nonequivalent N-methyl groups at 6.97 and 7.17, and the *t*-butyl methyl groups at 8.82. Similarly, N-methyl-α-chloroacetanilide with potassium *t*-butoxide in tetrahydrofuran gave Va in 42% yield, N-methylaniline, recovered starting material, and N-methyl-α-(*t*-butoxy)acetanilide. The reduction of the yield of the cyclopropane derivative in the case of the dimethylamide may reflect the lower stability of its anion relative to that of the N-methylanilide.[3] The use of tetrahydrofuran as a solvent appears to enhance displacement of the α-chlorine atom. Since potassium *t*-butoxide is soluble in tetrahydrofuran, it probably exists in this solvent as discrete anions which would be more efficient at displacing the α-chlorine atom of the amide than would be the ion aggregates which would be the probable form of butoxide in benzene or ether.

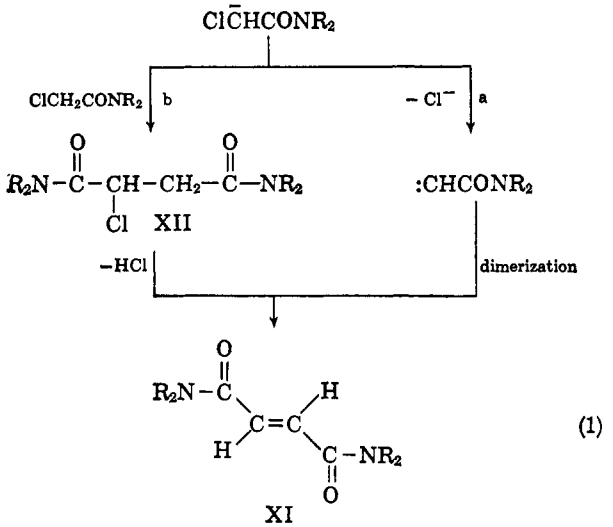
The use of potassium *t*-butoxide in *t*-butyl alcohol with N-methyl-α-chloroacetanilide led only to the isolation of N-methylaniline. Evidently attack at the carbonyl group leading to the tetrahedral intermediate is favored over the displacement reaction. The forward



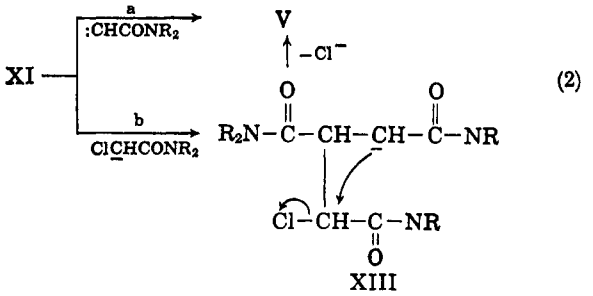
reaction will be favored in that the N-methylanilide ion would immediately abstract a proton from the solvent.

The mechanism of cyclopropane tricarboxamide formation could be formulated in the following ways.

(5) J. A. Pople, W. G. Schneider, and H. J. Bernstein, "High Resolution Nuclear Magnetic Resonance," McGraw-Hill Book Co., Inc., New York, N. Y., 1959, p. 366.



(1)



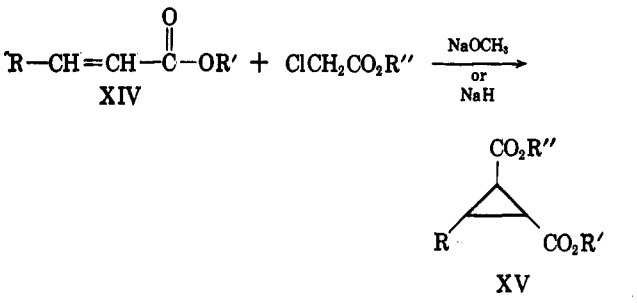
(2)

(3)

Step 1 would involve the formation of N,N'-dimethylfumaranilide either by a carbene (1a) or an anionic displacement reaction (1b). In step 2 the fumaranilide may react with the carbene (2a) directly to form the cyclopropanetricarboxamide or by a Michael addition of the anion (2b) to give XIII which cyclizes to product. Another possible mechanism (eq. 3) would involve three anionic displacements.

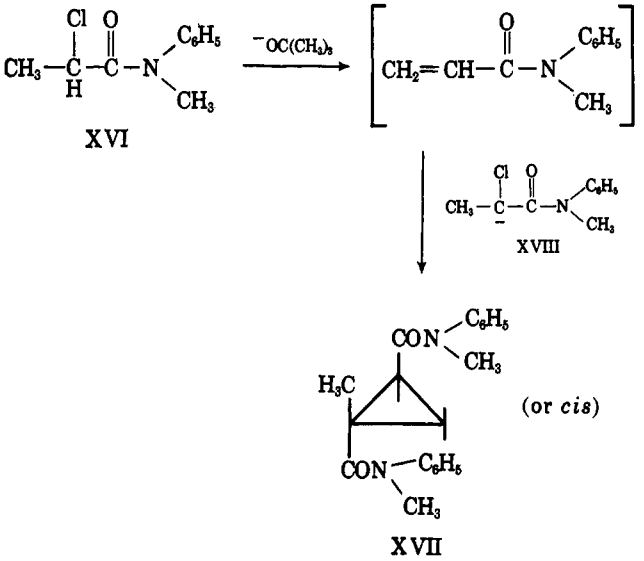
Mechanisms involving carbene intermediates are not likely since we have been unable to trap the carbonylcarbenes with cyclohexene and carbonylcarbenes do not ordinarily dimerize.[1,6] Even if dimerization did take place, it appears unreasonable that the carbene would react with N,N-dimethylfumaranilide in preference to the more nucleophilic cyclohexene. Carbonylcarbenes have been shown to be electrophilic in character.[7] However, the carbene derived from thermal degradation of benzoyldiazomethane reacts with *cis*-dibenzoylethylene (but not with the *trans* compound) to give *cis*-1,2,3-tribenzoylcyclopropane.[6] This experiment, however, was not conducted in the presence of cyclohexene.

Step 2b is analogous to the reaction of α-chloro esters with acrylic esters XIV[8,9] which gives cyclopropanedicarboxylic esters XV. The reaction of ethyl fumarate (XIV, R = $CO_2C_2H_5$; R' = C_2H_5) was unsuccessful in one instance[8] and gave an 18% yield of triethyl *trans*-1,2,3-cyclopropanetricarboxylate (X) in another.[9] However, Va was readily prepared in 80% yield from N-methyl-α-chloroacetanilide and XI with



potassium *t*-butoxide in benzene. Since XI can clearly be an intermediate in the formation of V, the most likely mechanistic path appears to be 1b–2b.

The reaction of N-methyl-α-chloropropionanilide (XVI) with potassium *t*-butoxide in benzene yielded 1,N,N'-trimethyl-1,2-cyclopropanedicarboxanilide (XVII). Its n.m.r. spectrum showed complex aromatic



absorption centered at τ 2.70, a singlet at 6.76 (N-methyl) a singlet at 6.79 (N-methyl), a singlet at 8.83 (C-methyl), and complex absorption at 8.57–8.95 (cyclopropane protons). This reaction is similar to that reported for ethyl α-chloropropionate[10] and probably involves dehydrochlorination to the acrylamide which reacts with the anion XVIII.

Reactions of α-halocarbonyl esters and ketones with strong bases have previously been reported to yield cyclopropane derivatives. Ethyl bromoacetate and sodium led to a 10% yield of triethyl *trans*-1,2,3-cyclopropanetricarboxylate[11] and bromomethyl *t*-butyl ketone[12] with potassium *t*-butoxide gave a 60% yield of *trans*-1,2,3-tripivalylcyclopropane.[13] These reactions do not appear to be of general synthetic usefulness.

The reaction of ethyl chloroacetate and potassium *t*-butoxide in benzene yields a complex mixture of six major and several minor components. Reaction of ethyl chloroacetate with sodium hydride and of phenacyl chloride with potassium *t*-butoxide did not yield any characterizable compounds. Although α-chloroacetamides do not appear to form carbenes on

(6) H. Strzelecka and M. Simalty-Siemiatycki, *Compt. rend.*, **252**, 3821 (1961).
(7) P. S. Skell and R. M. Etter, *Chem. Ind.* (London), 624 (1958).
(8) L. L. McCoy, *J. Am. Chem. Soc.*, **80**, 6568 (1958).
(9) M. Mousseron, R. Fraisse, R. Jacquier, and G. Bonavent, *Compt. rend.*, **248**, 1465 (1959).
(10) D. H. Deutsch and E. R. Buchman, *Experientia*, **6**, 462 (1950).
(11) C. Grundmann, *Ann.*, **555**, 77 (1943).
(12) α-Halo ketones with an α'-proton undergo the Favorski reaction.
(13) M. Charpentier-Morize and P. Colard, *Bull. soc. chim. France*, 1982 (1962).

treatment with potassium *t*-butoxide, success with α,α-dichloroacetamides might be anticipated since a chlorocarbonylcarbene should show increased stability over the unchlorinated derivative. However, reaction of N-methyl-α,α-dichloroacetanilide with potassium *t*-butoxide in ether–cyclohexene led only to N-methylaniline, recovered dichloroamide, and polymeric materials.

Experimental[14]

Reaction of N-Methyl-α-chloroacetanilide with Potassium *t*-Butoxide. A. In the Presence of Cyclohexene.—A mixture of N-methyl-α-chloroacetanilide (18.4 g., 0.1 mole), cyclohexene (41 g., 0.5 mole), and ether (200 ml.) was stirred while potassium *t*-butoxide (16.8 g., 0.15 mole) was added in small portions. The temperature was maintained below 40°. The mixture was stirred for 2 hr. and refluxed for 0.5 hr. Cold water (100 ml.) was added; the mixture was stirred and filtered. The white solid was recrystallized from methylene chloride–hexane. This produced *trans*-N,N′,N″-trimethyl-1,2,3-cyclopropanetricarboxanilide (16.3 g., 0.023 mole, 69%); m.p. 209–210°.

Anal. Calcd. for $C_{27}H_{27}N_3O_3$: C, 73.45; H, 6.15; N, 9.52; mol. wt., 441. Found: C, 72.98; H, 6.14; N, 9.43; mol. wt., 435.

The ether solution was dried over calcium chloride and the solvent was evaporated. Distillation of the small amount of residual oil gave N-methylaniline (0.63 g., 0.006 mole, 6%); b.p. 40° (0.8 mm.).

B. In the Presence of N,N′-Dimethylfumaranilide.—A mixture of N-methyl-α-chloroacetanilide (9.2 g., 0.05 mole), N,N′-dimethylfumaranilide (14.7 g., 0.05 mole), and benzene (200 ml.) was stirred while potassium *t*-butoxide (6.7 g., 0.06 mole) was added in small portions. The temperature was maintained below 30°. The mixture was stirred for 0.5 hr. and then heated at 60° for 2 hr. Cold water (100 ml.) was added and the mixture was filtered giving a white solid. The benzene solution was dried over sodium sulfate and evaporated. The residue and the filtered solid were dissolved in methylene chloride and the addition of hexane caused separation of a white solid. Recrystallization from methylene chloride–hexane gave *trans*-N,N′,N″-trimethyl-1,2,3-cyclopropanetricarboxanilide (17.9 g., 0.04 mole, 80%); m.p. 209–210°. The infrared spectrum was identical with that of the sample prepared as described previously and there was no depression of mixed melting point.

C. In Tetrahydrofuran.—A mixture of potassium *t*-butoxide (5.6 g., 0.05 mole) and tetrahydrofuran (150 ml.) was stirred while N-methyl-α-chloroacetanilide (9.2 g., 0.05 mole) was added in small portions. The mixture was stirred and heated at 50° for 3 hr. The tetrahydrofuran was removed *in vacuo* and the residue was extracted with hot methylene chloride. The methylene chloride solution was washed with water (100 ml.) and dried over anhydrous magnesium sulfate. Evaporation of most of the methylene chloride and the addition of hexane gave *trans*-N,N′,N″-trimethyl-1,2,3-cyclopropanetricarboxanilide (3.1 g., 0.007 mole, 42%); m.p. 209–210°. The infrared spectrum was identical with that of a sample prepared as described previously.

The solvents were evaporated from the mother liquor and the residue was distilled. This produced N-methylaniline (0.25 g., 0.0023 mole, 4.6%); b.p. 40° (1.7 mm.).

Further distillation gave a colorless liquid (1.8 g.), b.p. 122° (1.5 mm.), shown by vapor phase chromatography to be a *ca.* 40:60 mixture. Infrared and n.m.r. spectroscopy indicated that the liquid was a mixture of N-methyl-α-chloroacetanilide and N-methyl-α-(*t*-butoxy)acetanilide. The N-methyl-α-chloroacetanilide separated on standing.

***trans*-1,2,3-Cyclopropanetricarboxylic Acid (VII). A. From *trans*-N,N′,N″-Trimethyl-1,2,3-cyclopropanetricarboxanilide (VIa).**—A mixture of *trans*-N,N′,N″-trimethyl-1,2,3-cyclopropanetricarboxanilide (10.0 g., 0.023 mole) and 6 *N* hydrochloric acid (75 ml.) was refluxed for 72 hr. The solution was cooled, made alkaline with solid sodium hydroxide, and extracted with methylene chloride. The aqueous solution was acidified with hydrochloric acid to pH 2. The water was removed *in vacuo* and the residue was extracted with hot acetone. The acetone solution was treated with charcoal and evaporated to a small volume. The addition of benzene caused separation of a white solid. Recrystallization from acetone–benzene gave *trans*-1,2,3-cyclopropanetricarboxylic acid (2.7 g., 0.016 mole, 70%); m.p. 212–214°, lit. m.p. 213°,[4] 220–221°.[11]

Anal. Calcd.: neut. equiv., 58. Found: neut. equiv., 58.

The methylene chloride extract was dried over sodium sulfate and filtered through charcoal. Evaporation of the methylene chloride gave N-methylaniline (5.7 g., 0.053 mole, 78%).

B. From Triethyl *trans*-1,2,3-Cyclopropanetricarboxylate.—A mixture of triethyl *trans*-1,2,3-cyclopropanetricarboxylate[4] (3.0 g., 0.012 mole) and 6 *N* hydrochloric acid (10 ml.) was refluxed for 1.5 hr. On standing, a white solid precipitated. Recrystallization from acetone–benzene gave *trans*-1,2,3-cyclopropanetricarboxylic acid (1.0 g., 0.0058 mole, 48%); m.p. 212–214°. The infrared spectrum was identical with that of the sample prepared as described previously and there was no depression of a mixture melting point.

Reaction of N,N-Dimethyl-α-chloroacetamide and Potassium *t*-Butoxide. A. In Benzene.—A mixture of potassium *t*-butoxide (33.6 g., 0.3 mole) and benzene (200 ml.) was stirred while N,N-dimethyl-α-chloroacetamide (12.1 g., 0.1 mole) was added rapidly. The temperature was kept below 40°. The mixture was heated and stirred at 50° for 3 hr. Cold water (70 ml.) was added and the mixture was stirred for 0.5 hr. The water layer was extracted with methylene chloride (100 ml.) and the methylene chloride extract was combined with the benzene extract. The combined extracts were dried over sodium sulfate and the solvents were removed *in vacuo*. Distillation gave N,N-dimethyl-α-(*t*-butoxy)acetamide (1.7 g., 0.011 mole, 11%); b.p. 54° (0.3 mm.), $\lambda_{C=O}$ 1634 and λ_{C-O-C} 1079 cm.$^{-1}$.

Anal. Calcd. for $C_8H_{17}NO_2$: C, 60.34; H, 10.76; N, 8.80. Found: C, 60.04; H, 10.75; N, 8.80.

The solid residue was recrystallized from benzene–heptane (with charcoal). This produced *trans*-N,N,N′,N′,N″,N″-hexamethyl-1,2,3-cyclopropanetricarboxamide (4.2 g., 0.0165 mole, 50%); m.p. 116–118°.

Anal. Calcd. for $C_{12}H_{21}N_3O_3$: C, 56.45; H, 8.29; N, 16.46. Found: C, 56.35; H, 8.52; N, 16.50.

B. In Tetrahydrofuran.—A mixture of potassium *t*-butoxide–*t*-butyl alcohol complex[15] (18.6 g., 0.1 mole) and tetrahydrofuran (50 ml.) was stirred at 0° while N,N-dimethyl-α-chloroacetamide (12.1 g., 0.1 mole) was added dropwise. The mixture was stirred for 0.5 hr. and refluxed for 0.5 hr. Potassium chloride was removed by filtration and the tetrahydrofuran was evaporated *in vacuo*. The residue was placed on a column of neutral alumina packed wet with pentane. Elution with pentane gave liquid fractions which were recombined and distilled. This produced N,N-dimethyl-α-(*t*-butoxy)acetamide (6.2 g., 0.039 mole, 39%); b.p. 58–60° (0.6–0.8 mm.). The residue was recrystallized from methylene chloride–heptane (with charcoal). This produced *trans*-N,N,N′,N′,N″,N″-hexamethyl-1,2,3-cyclopropanetricarboxamide (0.35 g., 0.014 mole, 4%); m.p. 116–118°.

1,N,N′-Trimethyl-1,2-cyclopropanedicarboxanilide (XVII).—A mixture of potassium *t*-butoxide (5.6 g., 0.05 mole) and benzene (100 ml.) was stirred at 0° while N-methyl-α-chloropropionanilide (9.65 g., 0.05 mole) was added in small portions. The mixture was stirred for 0.5 hr. and heated at 60° for 2 hr. Cold water (100 ml.) was added and the mixture was stirred. The benzene solution was dried over sodium sulfate, evaporated to a small volume, and placed on a column of neutral alumina packed wet with pentane. Elution with pentane–benzene gave oily fractions with similar infrared spectra. Combination of the fractions and crystallization from hexane gave 1,N,N′-trimethyl-1,2-cyclopropanedicarboxanilide (2.25 g., 0.007 mole, 28%); m.p. 107–109°.

Anal. Calcd. for $C_{20}H_{22}N_2O_2$: C, 74.50; H, 6.88; N, 8.69. Found: C, 74.42; H, 6.82; N, 9.00.

(14) Melting and boiling points are uncorrected. Amides were prepared by the reaction of commercial acid chlorides with the appropriate amines.

(15) A. J. Speziale and K. W. Ratts, *J. Am. Chem. Soc.*, **84**, 854 (1962).