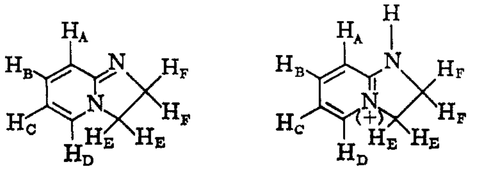
TABLE IV

NMR SPECTRA OF 2,3-DIHYDROIMIDAZO[1,2-*a*]PYRIDINES



I II

| Compd | | Chemical shift, τ | | | | | | |
|---|---|---|---|---|---|---|---|---|
| No. | Substituent | $H_A$ | $H_B$ | $H_C$ | $H_D$ | $H_E$ | $H_F$ | Substituent |
| I[a] | 7-Methyl | 2.92 | ... | 3.59 | 3.12 | 6.10[b] | 6.10[b] | 8.00 |
| II[c] | 7-Methyl | 3.07 | ... | 3.14 | 2.08 | 5.29 | 5.99 | 7.85 |

[a] $CDCl_3$ solution (1.04 *M*). [b] Broad singlet. [c] $D_2O$ solution (1.04 *M*).

6-methyl-, 190–191.2°; 7-methyl-, 215–216°; and 8-methyl-, 288.5–289.59° dec.

*Anal.* Calcd for $C_9H_{11}IN_2$: C, 39.43; H, 4.04; N, 10.22. Found for 6-methyl·$CH_3I$: C, 39.78; H, 4.00; N, 10.41. Found for 7-methyl·$CH_3I$: C, 39.27; H, 3.89; N, 10.02. Found for 8-methyl·$CH_3I$: C, 39.40; H, 4.14; N, 10.40.

**2,3-Dihydro-7-methylimidazo[1,2-*a*]pyridine.**—A solution of 5.4 g (0.05 mole) of 2-amino-5-methylpyridine and 9.4 g (0.05 mole) of 1,2-dibromoethane in 40 ml of 95% aqueous ethanol was heated at reflux for 3 hr. To the resulting brownish red solution was then added an aqueous solution of 10.6 g (0.10 mole) of sodium carbonate in 20 ml of water and refluxing was continued for an additional 12 hr. The cooled reaction mixture was filtered and the filtrate was evaporated to about 20 ml. This solution was then extracted with three 100-ml portions of $CHCl_3$. The combined dried (anhydrous $Na_2CO_3$) $CHCl_3$ extracts were evaporated to dryness to leave a brown oil. Chromatography on neutral grade III alumina yields 2,3-dihydro-7-methylimidazo[1,2-*a*]pyridine (3.4 g) by elution with methanol–ethyl acetate (5:95). Prior to this eluent, ether eluted 0.5 g. of unreacted 2-amino-5-methylpyridine.

The material obtained from the alumina column was further purified by vacuum distillation to yield 3.2 g of the desired compound, bp 170–175° (0.5 mm).

*Anal.* Calcd for $C_8H_{10}N_2$: C, 71.61; H, 7.51; N, 20.88. Found: C, 71.46; H, 7.41; N, 20.59.

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# 1,2 and 1,6 Eliminations in Substituted Xylenes

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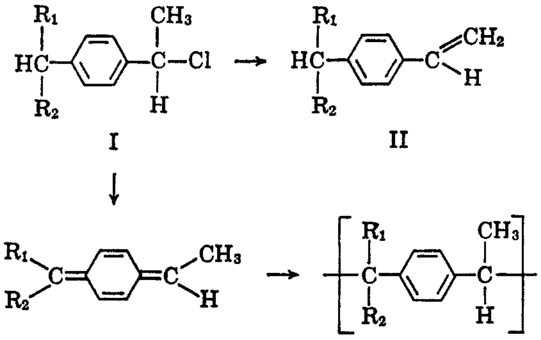
*para*-Substituted benzyl halides such as I are known to undergo E2 elimination[1] to produce the corresponding styrene II, but such halides can also conceivably undergo elimination by a 1,6 process to form the corresponding xylylene III. Examples of 1,6 eliminations in *p*-xylenes are known,[2–7] but in systems such as α-(*p*-tolyl)ethyl halide which can eliminate hydrogen halide by either a 1,2 or 1,6 mechanism, only the 1,2 elimination has been observed.[8] A possible synthetic modification of I which would favor 1,6 elimination is the introduction of conjugated groups, $R_1$ and $R_2$, which would stabilize the xylylene III relative to the corresponding styrene II. 1,6 elimination should also be favored by factors which render the 6 proton more acidic such as adjacent cyano, carbonyl, nitro, etc., groups.



I II III IV

1,6 elimination at the expense of 1,2 elimination has now been observed using the systems I ($R_1$ = CN or COOR; $R_2$ = H, $C_2H_5$, or $C_6H_5$[9]) in which the protons adjacent to the group $R_1$ are quite acidic (p$K_a$ of phenylacetonitrile has been reported to be 15.7 in aqueous ethylenediamine[10]). In these cases dehydrohalogenation with a base as weak as aqueous sodium hydroxide results in the formation of the intermediate xylylenes III which are isolated as high molecular weight polymers. The structures of these polymers were shown to be IV by elemental analysis, their low solubility in common solvents (unlike the corresponding polystyrenes), their high softening temperatures (again in contrast to the corresponding polystyrenes), and their infrared and nmr spectra. Although these polymers could also be formed by a stepwise substitution of the halide I by the corresponding (or polymerically equivalent) carbanion, this was shown not be of importance in the present case. Thus the reaction of I with less than stoichiometric amounts of base still

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[1] H. C. Brown, I. Moritani, and Y. Okamoto, *J. Am. Chem. Soc.*, **78**, 2193 (1956).

[2] L. A. Auspos, L. A. R. Hall, J. K. Hubbard, W. Kirk, J. R. Schaefgen, and S. B. Speck, *J. Polymer Sci.*, **15**, 9 (1955).

[3] F. Johnson and W. A. Nasutavicus, *J. Org. Chem.*, **28**, 1877 (1963); A. Streitweizer and H. F. Koch, *J. Am. Chem. Soc.*, **86**, 404 (1964).

[4] H. F. Winberg, F. S. Fawcett, W. E. Mochel, and C. W. Theobald, *ibid.*, **82**, 1428 (1960).

[5] A. T. Kader and C. J. M. Stirling, *J. Chem. Soc.*, 3425 (1962).

[6] M. Szwarc, *Discussions Faraday Soc.*, **46**, (1947); M. Szwarc, *Nature*, **160**, 403 (1947); J. H. Golden, *J. Chem. Soc.*, 1604 (1961).

[7] Other literature examples such as the dehydrohalogenation of $ClCH_2C_6H_4CH_2COOCH_3$ [J. W. Baker, J. A. Brieus, and D. G. Saunders, *ibid.*, 404 (1956)] also involve the xylylene formed by 1,6 elimination (R. W. Kluiber, unpublished results).

[8] A. Klages, *Ber.*, **35**, 2245 (1902); J. Schramm, *ibid.*, **24**, 1332 (1891).

[9] R. W. Kluiber, *J. Org. Chem.*, **30**, 2037 (1965).

[10] R. Schaal, *J. Chim. Phys.*, **52**, 796 (1955).

results in the formation of high molecular weight polymer. This is characteristic of an addition polymerization (in this case of the xylylene intermediate) not a condensation polymerization. Furthermore, although the *para* isomer of I gives polymer in a rapid and exothermic reaction, the *meta* isomer, which cannot form a stable xylylene, is relatively unreactive. Another route for the formation of the polymer IV, the initial 1,2 elimination to form a styrene and the rearrangement to a xylylene with subsequent polymerization, was shown not be of importance in these reactions.[9]

The mechanism of these eliminations can be E2 or E1cB,[11] and in order to further study the mechanism, these eliminations were also carried out in deuterated solvents. For the system I ($R_1$ = CN, $R_2$ = H), 1,6 elimination using methanolic (OD) potassium hydroxide results in polymer containing somewhat more than half of the equilibrium concentration of deuterium incorporated at the position of the remaining acidic polymer proton. The percentage of incorporated deuterium increases if the initially formed polymer in its swollen form is kept in contact with the deuterated solution, indicating that proton exchange between polymer and solvent does occur, but this process is slow (at least in its latter stages or after the polymer has precipitated) compared to the rate of formation and polymerization of the xylylene intermediate. Essentially no deuterium is found in the polymer formed from the halonitrile I ($R_1$ = CN, $R_2$ = $C_2H_5$), indicating that exchange occurs only with the acidic protons adjacent to the cyano group. The dehydrohalogenation of I ($R_1$ = CN, $R_2$ = H) with potassium hydroxide in deuterium oxide results in the incorporation in the polymer of less deuterium than when methanolic (OD) potassium hydroxide was used. Although these data are subject to several interpretations including merely a question of rate of exchange of the acidic polymer protons in various media, they are in agreement with the idea that in the transition state for 1,6 elimination the carbon–hydrogen bond is completely or almost completely broken. Thus, subject to the above limitations and the question of whether exchange and elimination occur in the same process,[12] the elimination can be described as more E2 (or E2cB) in aqueous potassium hydroxide and more E1cB in the stronger base, methanolic potassium hydroxide.

## Experimental Section[13]

**α-Cyano-α′-hydroxy-α′-methylxylenes.**—From 216 g of phenylacetonitrile, 300 g of acetyl chloride, 750 ml of carbon disulfide, and 720 g of anhydrous aluminum chloride was obtained, by the method of Kunckell,[14] 72 g (24% yield) of *p*-(cyanomethyl)-acetophenone, mp 87–88° (lit.[14] mp 83–84°). The infrared spectrum has characteristic bands at 4.48 (CN stretching) and 5.92 μ (C=O stretching). The nmr spectrum[15] has resonances with chemical shifts of −155 ($CH_3$), −232 ($CH_2$), −447 (aromatic CH groups), and −477 (aromatic CH groups *ortho* to the $-COCH_3$ group) cps. In methanol with added potassium hydroxide the −232-cps resonance disappears because of averaging with the hydroxyl proton indicating rapid exchange. A suspension of this ketone, 45 g in 300 ml of methanol, was reduced with 5 g of sodium borohydride at 5–10°. The methanol solution was concentrated *in vacuo*, diluted with ether, and washed with water to yield upon distillation 37 g of alcohol, bp 135° (0.5 mm), $n^{25.5}_{D}$ 1.5362, mp 35–37°, with characteristic infrared peaks at 2.96 (OH stretching), 4.46 (C≡N), and 12.32 μ (*para*-substituted benzene). The nmr chemical shifts for this alcohol are −85 ($CH_3$), −163 (OH), −220 ($CH_2$), ∼ −290 (CH), and −436 (aromatic CH groups) cps.[15]

*Anal.* Calcd for $C_{10}H_{11}NO$: C, 74.50; H, 6.84. Found: 74.40; H, 6.72.

*m*-(Cyanomethyl)acetophenone[14] was recovered from the mother liquors of the *para* isomer crystallization conducted at −80°. This ketone, bp 125–130° (0.5 mm), $n^{26.5}_{D}$ 1.5440, has characteristic infrared absorptions at 4.46 (C≡N), 5.95 (C=O), 12.65, and 14.50 μ (*meta*-substituted benzene).

*Anal.* Calcd for $C_{10}H_9NO$: N, 8.79. Found: N, 8.80.

By a sequence identical with that for the *para* isomer this *meta* ketone was reduced to α-cyano-α′-hydroxy-α′-methyl-*m*-xylene, bp 127–130° (0.6 mm), $n^{25.5}_{D}$ 1.5357.

*Anal.* Calcd for $C_{10}H_{11}NO$: N, 8.69. Found: N, 8.51.

**Dehydrohalogenation of α-Cyano-α′-chloro-α′-methyl-*p*-xylene.**—α-Cyano-α′-hydroxy-α′-methylxylene (10 g) in 30 ml of ether was converted to the chloride by slowly adding 9 g of thionyl chloride and stirring for 30 min. The solvent and excess reagent were stripped *in vacuo*, and the residue was distilled, bp 100–102° (0.3 mm), to yield 4 g of chloride having characteristic infrared peaks at 4.46 (C≡N) and 12.30 μ (*para*-substituted benzene). The nmr spectrum has peaks having chemical shifts of −109 ($CH_3$), −224 ($CH_2$), −306 (CH), and an $A_2B_2$ system with shifts of −439 and −446 (aromatic CH groups) cps.[15] (A small impurity of *p*-cyanomethylstyrene was also detected.)

*Anal.* Calcd for $C_{10}H_{10}ClN$: C, 66.86; H, 5.61; N, 7.80. Found: C, 67.15; H, 5.80; N, 7.85.

In a typical dehydrohalogenation experiment 10 g of chloride was added with stirring at room temperature to a solution of 3.7 g of potassium hydroxide (85+%) in 50 ml of methanol. A precipitate formed which, after washing in a blender twice with methanol and once each with water and acetone, gave 4.3 g of amorphous (by X-ray) poly-α-cyano-α′-methyl-*p*-xylylene, softening at about 200°, insoluble in chloroform, reduced viscosity 4.55 (0.2 g/100 ml of *p*-chlorophenol, 58.2°). The infrared spectrum, in addition to bands at 4.46 (C≡N) and 11.87 μ (*para*-substituted benzene), shows a band at 7.24 μ (CH symmetrical deformation characteristic of a $C-CH_3$ group). (Analysis was carried out on polymer prepared by the addition of methanolic potassium hydroxide to a methanolic solution of chloride. The polymer, isolated as above and exhaustively washed, was analyzed without further purification owing to its limited solubility in common solvents.)

*Anal.* Calcd for $C_{10}H_9N$: C, 83.88; H, 6.33; N, 9.79. Found: C, 83.46; H, 6.60; N, 9.89.

The *m*-chloro isomer, α-cyano-α′-chloro-α′-methyl-*m*-xylene, was correspondingly prepared, bp 105–107° (0.7 mm), having characteristic infrared absorptions at 4.46 (C≡N), 12.73, 14.32 μ (*meta* substitution).

*Anal.* Calcd for $C_{10}H_{10}ClN$: N, 7.76. Found: N, 7.70.

Attempted dehydrohalogenation with methanolic potassium hydroxide gave, after 5 min of stirring at room temperature, principally recovered chloride, identified by its infrared spectrum.

**α-Cyano-α-ethyl-α′-hydroxy-α′-methylxylenes.**—A mixture of the *para* and *meta* isomers of this alcohol was prepared by acetylation of 50 g of α-ethylphenylacetonitrile[16] using 14 g of aluminum chloride, 60 g of acetyl chloride and 100 ml of carbon disulfide to give, after 48 hr of reflux and work-up, a liquid mixture of *m*- and *p*-acetyl-α-ethylphenylacetonitrile, bp 115–118° (0.5 mm), $n^{25}_{D}$ 1.529, having characteristic infrared absorptions at 4.47 (C≡N), 5.94 (C=O), 12.00 (*para* substitution), 12.54, and 14.43 μ (*meta* substitutions), and nmr chemical shifts of −64 ($CH_3-CH_2$), −117 ($CH_3-CH_2$), −155 ($CH_3CO$), −240 (CH) cps, and complex peaks at approximately −450 and −475 (aromatic protons) cps.

*Anal.* Calcd for $C_{12}H_{13}NO$: C, 76.97; H, 7.00, N, 7.48. Found: C, 76.64; H, 7.30; N, 7.36.

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[11] C. K. Ingold, "Structure and Mechanism in Organic Chemistry," Cornell University Press, Ithaca, N. Y., 1953, p 422.

[12] R. Breslow, *Tetrahedron Letters*, 399 (1964).

[13] All temperatures and pressures are uncorrected.

[14] F. Kunckell, *Ber.*, **39**, 3145 (1906).

[15] All proton nmr spectra were recorded using a Varian A-60 or A-60A 60-Mc spectrometer. Shifts were measured in cps from tetramethylsilane, an internal standard, with deuteriochloroform as solvent. Unless other wise noted the integrated intensities of the peaks and the spin–spin splitting patterns are in agreement with the given structures.

[16] J. V. Murray and J. B. Cloke, *J. Am. Chem. Soc.*, **58**, 2014 (1936).

Reduction with sodium borohydride gave a mixture of the *meta* and *para* alcohols α-cyano-α-ethyl-α′-hydroxy-α′-methyl-*m*- and -*p*-xylene, bp 115–120° (0.6 mm), $n^{25}D$ 1.5220, having characteristic infrared absorptions at 2.95 (OH), 4.49 ($C\equiv N$), 12.00 (*para* substitution), 12.56, and 14.22 μ (*meta* substitution) and nmr chemical shifts at −60 (ethyl $CH_3$), −83 ($CH_3$), −111 (ethyl $CH_2$), −223 (CHCN), −234 (OH), −286 (CH), and −437 (aromatic CH) cps.[15]

*Anal.* Calcd for $C_{12}H_{15}NO$: C, 76.15; H, 7.99; N, 7.40. Found: C, 75.90; H, 8.20; N, 7.38.

An alternate preparation of α-cyano-α-ethyl-α′-hydroxy-α′-methyl-*p*-xylene uncontaminated by the *meta* isomer was made by the interaction of ethyl bromide, 13 g, with the sodium salt of *p*-acetylphenylacetonitrile, prepared from 15 g of the nitrile and sodium amide from 2.4 g of sodium, in 100 ml of refluxing ether. The product, α-ethyl-*p*-acetylphenylacetonitrile, 15 g, isolated after washing of the neutral reaction mixture with water, concentration of the ether, and distillation of the residual oil, bp 120–125° (0.6 mm), $n^{25}D$ 1.5330, has characteristic infrared absorptions at 4.49 ($C\equiv N$), 5.95 (C=O), and 12.07 μ (*para* substitution) and nmr chemical shifts of −63 ($CH_3$), −114 ($CH_2$), −153 ($CH_3CO$–), −232 (CH), −445 (CH aromatic), and −476 (CH aromatic *ortho* to acetyl group) cps. Reduction of this ketone in methanol at 10–20° and with an excess of sodium borohydride gave α-cyano-α-ethyl-α′-hydroxy-α′-methyl-*p*-xylene, bp 110–112° (0.3 mm), $n^{25}D$ 1.5220, having characteristic infrared absorptions at 2.93 (OH), 4.48 ($C\equiv N$), and 12.05 μ (*para* substitution).

*Anal.* Calcd for $C_{12}H_{15}NO$: C, 76.15; H, 7.99; N, 7.40. Found: C, 76.34; H, 8.10; N, 7.47.

Thionyl chloride, 9 g, was used to convert the mixed alcohol, 18 g dissolved in 100 ml of ether, to 12 g of a mixture of α-cyano-α-ethyl-α′-chloro-α′-methyl-*m*- and -*p*-xylene, bp 120–122° (0.4 mm), having characteristic infrared absorptions at 4.48 (C=N), 12.00 (*para* substitution), 12.58, 14.27 (*meta* substitution), and 13.40 μ (C–Cl?) and nmr chemical shifts of −63 ($CH_3$–$CH_2$), −109 ($CH_3$–CHCl–), −115 ($CH_2$), −225 (CH–CN), −305 (CHCl), and *ca.* −440 (aromatic) cps.

*Anal.* Calcd for $C_{12}H_{14}ClN$: C, 69.39; H, 6.79; N, 6.75. Found: C, 69.72; H, 7.15; N, 6.62.

To a solution of the mixed chlorides in methanol was added slowly with stirring methanolic potassium hydroxide until the reaction mixture remained basic. Under these conditions the addition of base initially produces a yellow color which fades as polymer is precipitated. The precipitated polymer was washed with methanol, water, and methane. This polymer has a glass transition temperature at 230° and is soluble in chloroform but can be reprecipitated by addition of petroleum ether (bp 30–50°) or methanol. A sample prepared by the addition of the chloride to the base was found to have a reduced viscosity of 3.0 (N,N-dimethylformamide, 0.2 g/100 ml, 25°).

*Anal.* Calcd for $C_{12}H_{13}N$: C, 84.17; H, 7.65; N, 8.18. Found: C, 84.20; H, 7.71; N, 8.36.

The nmr spectrum of a 3% polymer solution in deuteriochloroform showed broad overlapping peaks centered at −46, −65, and −128 cps of relative intensity 8.0 ($CH_3$, $CH_3$–$CH_2$–), a single broad peak at −187 cps of relative intensity 0.95 (C–H), and broad overlapping peaks at −405, −426, and −445 cps of relative intensity 4.0 (aromatic CH). Characteristic infrared peaks are those found at 4.48 (C=N) and 11.96 μ (*para* substitution).

From the mother liquors of the dehydrohalogenation was obtained by concentration *in vacuo* and distillation α-cyano-α′-chloro-α′-methyl-*m*-xylene, bp 120–122° (0.4 mm), having characteristic infrared absorptions at 4.46 ($C\equiv N$), 12.5, and 14.3 μ (*meta* substitution), and nmr chemical shifts virtually identical with those of the mixed chlorides except for the aromatic proton resonances which consisted of two major peaks at about −437 and −445 cps (neat).[15]

*Anal.* Calcd for $C_{12}H_{14}ClN$: C, 69.39; H, 6.79; N, 6.75. Found: C, 69.50; H, 7.02; N, 6.87.

**Dehydrohalogenation of α-Carbomethoxy-α′-chloro-α′-methylxylene.**—Methyl *p*-acetylphenylacetate was prepared as a mixture of isomers by adding 450 g of aluminum chloride (anhydrous) at 0–10° to a stirred solution of 400 g of methyl phenylacetate, 240 g of acetyl chloride, and 1000 ml of carbon disulfide, refluxing the mixture for 16 hr, pouring into ice and hydrochloric acid, extracting with ether, washing the ether extracts with water, drying, and distilling to yield 86 g. This product was reduced with 10 g of sodium tetrahydridoborate in 400 ml of methanol to yield 43 g of alcohol. The alcohol was dissolved in 90 ml of benzene, and 0.3 ml of pyridine and 35 g of thionyl chloride were added with cooling. After refluxing for 2 hr, the reaction was concentrated *in vacuo*, washed, and distilled, bp 90–107° (0.5 mm). This chloride (4 g) added to a methanolic potassium hydroxide solution gave a low yield of amorphous high molecular weight polymer, insoluble in acetone, containing some potassium as -COOK groups (flame test) and the COOR, C–$CH_3$ *para*-substituted benzene groups (by infrared).

**Dehydrohalogenation in Deuterated Solvents.**—All deuterium analyses were made by Josef Nemeth, Urbana, Ill. (A) *p*-(α-Chloroethyl)phenylacetonitrile, 1.7 g (0.0095 mole), was added to 0.4 g of potassium hydroxide (less than 0.01 mole) in 8 ml of deuterium oxide at room temperature. After 1 hr the polymer was removed by filtration, washed with deuterium oxide and with acetone, and dried. The residual polymer, 0.50 g, was found to contain 1.94 atom % excess deuterium (calcd for complete exchange of one proton, 10.5%). (B) To 5.40 g of a solution of potassium hydroxide (0.0075 mole) in methanol-*d* containing 88% of exchangeable protons as deuterium by nmr (made from 3.56 g of "85%" potassium hydroxide and 37.31 g. of methanol-*d*) was added at room temperature 231 g (0.0013 mole) of chloronitrile I ($R_1$ = CN, $R_2$ = H) as an undiluted liquid. After 5 min the reaction was washed successively with methanol-*d*, deuterium oxide, acetone, and methanol-*d*, and air dried to yield 90 mg (49%) of polymer containing 5.75 atom % excess deuterium (calcd for complete exchange of one proton, 9.7 atom %). (C) Reaction B was repeated except that the product was allowed to stand 24 hr at room temperature. The polymer was found to contain 8.95 atom % excess deuterium. (D) To 0.4 g of "85%" potassium hydroxide in 10 ml of methanol-*d* at −20° was added 2.0 g (0.0095 mole) of chloronitrile I ($R_1$ = CN, $R_2$ = $C_2H_5$). Upon warming, polymer formed which, after the usual washing and drying, produced polymer having 0.11 atom % excess deuterium.

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# Nucleophilic Displacement of Vinylic Halogen in Fluorinated Cycloalkenes. I. Reaction with Pyridines

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Reaction of the fluorinated olefins Ia or Ib (Chart I) and tertiary alkylamines has been reported to give trialkyl (3,3-difluoro-2,4-dioxocyclobutyl)ammonium betaines (II).[2] From the *solvent-free* reaction of Ia and pyridine or substituted pyridines, Pruett and coworkers[3] obtained a mixture of olefin dimers, an olefin trimer, or, in some cases, a quaternary adduct of an olefin dimer. Only from 3-bromopyridine and Ia did they isolate a betaine (IIIf) in low yield after an aqueous work-up.

Herein is reported a more general method for the preparation of fluorinated dioxocycloalkyl pyridinium betaines (III), and the reaction of pyridines with the olefins I has now been extended to the five- and six-membered ring compounds.[4]

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[1] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

[2] (a) K. E. Rapp, *J. Am. Chem. Soc.*, **73**, 5901 (1951); (b) R. L. Pruett, C. T. Bahner, and H. A. Smith, *ibid.*, **74**, 1633 (1952).

[3] R. L. Pruett, C. T. Bahner, and H. A. Smith, *ibid.*, **74**, 1638 (1952).

[4] While this paper was in manuscript, F. H. Megson, M. T. Beachem, and R. F. Stockel [Abstracts, 149th National Meeting of the American Chemical Society, Detroit, Mich., April 1965, p 11P] reported the preparation of IIIb from 1-chloro-2-methoxyhexafluorocyclopentene.