B.—A mixture of 6.36 g. (0.030 mole) of *o*-anilinobenzamide and 6.12 g. (0.060 mole) of acetic anhydride was refluxed for about 20 hr. After the acetic anhydride was removed *in vacuo*, excess ether was added to the residue. The crude solid, m.p. 180–220°, that was obtained was recrystallized from a small amount of hot methanol to give 0.75 g. (9% yield) of **2-acetonylidine-1-phenyl-4(3H)-quinazolinone** (**XV**) as a white solid, m.p. 240–243°. One additional recrystallization from ether gave XV as white crystals; m.p. 241–243°; infrared (CH_2Cl_2) 1699 s (amide carbonyl), 1615 s, and 1568 cm.$^{-1}$ s (either could be the "NH" deformation band or the "ketone" carbonyl absorption of the conjugated system); ultraviolet $\lambda_{max}^{CH_3OH}$ 220 mμ sh (ε 18,600), 268 (5120), 318 (35,090); $\lambda_{min}^{CH_3OH}$ 258 mμ (ε 4620), 282–283 (4420); λ_{max} (0.1 *N* KOH–CH_3OH) 280 mμ sh (ε 5780), 317 (33,150); λ_{min} (0.1 *N* KOH–CH_3OH) 260–264 mμ (ε 5060); λ_{max} (0.1 *N* HCl–CH_3OH) 220 mμ sh area (ε 17,590), 268 sh area (6370), 276 (6810), 318 (29,160); λ_{min} (0.1 *N* HCl–CH_3OH) 257 mμ (ε 5170), 282–285 (6540); $\lambda_{max}^{cyclohexane}$ 216 mμ (ε 20,360), 223 sh (19,640), 278 (7790), 312 (31,270); $\lambda_{min}^{cyclohexane}$ 211 mμ (ε 19,970), 254 (4210), 282 (7440).

Anal. Calcd. for $C_{17}H_{14}N_2O_2$: C, 73.36; H, 5.07; N, 10.07; mol. wt., 278.30. Found: C, 73.09; H, 4.94; N, 10.05; mol. wt., 264.5. (by osmometry[38] in chloroform solution).

Small quantities of compound XIV were obtained from the mother liquors.

2-Acetonylidine-1-phenyl-4(3H)-quinazolinone (XV).—A mixture of 0.495 g. (0.0021 mole) of 2-methyl-1-phenyl-4-quinazolinone (XIV) and 0.224 g. (0.0022 mole) of acetic anhydride was refluxed for about 19 hr. A methanol solution of the residue was partially decolorized with Norit and was then cooled in a Dry Ice–acetone bath. A total of 0.24 g. (41% yield) of XV, m.p. 230–240°, was obtained. One recrystallization from a small volume of methanol with Norit treatment gave 0.14 g. of XV as an off-white solid, m.p. 240–243°.

Acknowledgment.—It is a pleasure to acknowledge the many stimulating discussions with Mr. L. Dorfman and members of his staff throughout the course of this investigation.

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[38] Vapor pressure osmometer, Model 301A, from Mechrolab, Inc., Mountain View, Calif.

A One-Step Synthesis of 1,1-Difluoro Olefins from Aldehydes

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1,1-Difluoro olefins can be prepared in good yield by heating a solution of an aldehyde, triphenylphosphine, and sodium chlorodifluoroacetate. The synthesis appears quite generally applicable to aromatic, aliphatic, and heteroaromatic aldehydes. The yield of the 1,1-difluoro olefin VI from *p*-nitrobenzaldehyde, however, is poor, the major product being *p*-nitro-β,β,β-trifluoroethylbenzene (VII).

1,1-Difluoro olefins can be prepared in good yield by heating a solution of an aldehyde, triphenylphosphine, and sodium chlorodifluoroacetate.[2]

Presumably the reaction proceeds with the *in situ* formation from sodium chlorodifluoroacetate of a carbene (difluoromethylene) which is trapped by triphenylphosphine to form an ylid which undergoes a Wittig reaction with an aldehyde to form the 1,1-difluoro olefin.[3] Formation of difluorocarbene by decomposition of sodium chlorodifluoroacetate in solution has been reported.[4,5] Seyferth demonstrated that chlorocarbene, generated from methylene chloride and butyllithium, reacted with triphenylphosphine to form the ylid.[6] Similar trapping with triphenylphosphine of dichlorocarbene,[7,8] of chlorocarbene,[9] and of methylene,[10,11] and the use, in a Wittig reaction, of the ylids formed have been reported. Speziale[8] was unsuccessful in attempts to trap difluorocarbene, generated from chlorodifluoromethane, with triphenylphosphine, nor could he substantiate Franzen's[11] claims that difluorocarbene generated from dibromodifluoromethane could be trapped with triphenylphosphine.

$$ClCF_2COONa \longrightarrow CF_2 + NaCl + CO_2 \xrightarrow{(C_6H_5)_3P} (C_6H_5)_3P{=}CF_2 \xrightarrow{RCHO} RCH{=}CF_2 + (C_6H_5)_3PO$$

I, R = C_6H_5
II, R = *p*-F–C_6H_4
III, R = *p*-MeO–C_6H_4
IV, R = *n*-C_6H_{13}
V, R = 2-furyl
VI, R = *p*-NO_2–C_6H_4

The synthesis appears quite generally applicable to aromatic, aliphatic, and heteroaromatic aldehydes. Good yields (yields of isolated product in parentheses) were obtained from benzaldehyde (74%), *p*-fluorobenzaldehyde (65%), anisaldehyde (60%), heptaldehyde (52%), and furfural (69%).

No other simple, general route to 1,1-difluoro olefins has been reported. The recorded preparation[12] of β,β-difluorostyrene, for example, consists of seven steps from sodium difluoroacetate, the last step involving pyrolysis at 600°; the over-all yield was 5%.

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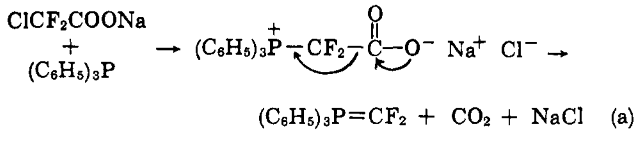
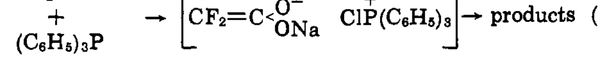
[1] Deceased.

[2] Preliminary communication: S. A. Fuqua, W. G. Duncan, and R. M. Silverstein, *Tetrahedron Letters*, **No. 23**, 1461 (1964). Note that V was erroneously reported as the tetrahydrofuryl compound (p. 1462, last line).

[3] Two other plausible mechanisms were suggested by a referee (a and b).



(a)



$$\begin{matrix}ClCF_2COONa\\+\\(C_6H_5)_3P\end{matrix} \rightarrow \left[CF_2{=}C{<}^{O^-}_{ONa}\quad Cl\overset{+}{P}(C_6H_5)_3\right] \rightarrow \text{products}$$

(b)

[4] J. M. Birchall, G. E. Cross, and R. N. Haszeldine, *Proc. Chem. Soc.*, 81 (1960).

[5] L. H. Knox, Preprints, 2nd International Symposium on Fluorine Chemistry, Estes Park, Colo., July 1962, p. 277; L. H. Knox, E. Velarde, S. Berger, D. Cuadriello, P. W. Landis, and A. D. Cross, *J. Am. Chem. Soc.*, **85**, 1851 (1963).

[6] D. Seyferth, S. O. Grim, and T. O. Read, *ibid.*, **82**, 1510 (1960); **83**, 1617 (1961).

[7] A. J. Speziale, G. J. Marco, and K. W. Ratts, *ibid.*, **82**, 1260 (1960).

[8] A. J. Speziale and K. W. Ratts, *ibid.*, **84**, 854 (1962).

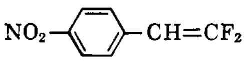
[9] G. Wittig and M. Schlosser, *Angew. Chem.*, **72**, 324 (1960).

[10] V. Franzen and G. Wittig, *ibid.*, **72**, 417 (1960).

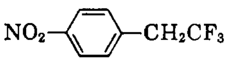
[11] V. Franzen, *ibid.*, **72**, 566 (1960).

[12] M. Prober, *J. Am. Chem. Soc.*, **75**, 968 (1953).

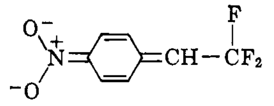
A mixture of two products was obtained from *p*-nitrobenzaldehyde: *p*-nitro-β,β-difluorostyrene (VI, the expected product) in poor yield, and *p*-nitro-β,β,β-trifluoroethylbenzene (VII) as the major product in about 20% yield. Presumably the latter arises from the former by nucleophilic attack by fluoride anion.



VI



VII



The fluorinated olefins liberate hydrogen fluoride on exposure to moist air; fluoride ion may be generated from the hydrogen fluoride by the sodium chloride formed in the reaction.

Initial experiments were carried out by heating all the components in anhydrous dimethoxyethane (monoglyme) at a bath temperature of 90–95° until a quantitative evolution of gas had occurred (wet-test meter). Under these conditions an induction period of variable duration was noted and the total reaction time varied from about 1 to 3 days. Use of anhydrous 2,2′-dimethoxydiethyl ether (diglyme) as a solvent at high temperatures resulted in shorter reaction time, but also introduced the hazard of violent exothermic decomposition of sodium chlorodifluoroacetate. The obvious remedy was dropwise addition of a solution of sodium chlorodifluoroacetate. The problem of its low solubility in diglyme was solved by wrapping the dropping funnel with heating tape to maintain the temperature at 60°.[13]

Other satisfactory solvents were triethylene glycol dimethyl ether (triglyme) and 1-methyl-2-pyrrolidinone. The bath temperatures were 160° for diglyme and triglyme, and 185° for 1-methyl-2-pyrrolidone. At these temperatures, sodium chlorodifluoroacetate eliminates carbon dioxide as rapidly as it is added (over a period of 1–2 hr.). At bath temperatures between about 110 and 140°, buildup of the acetate followed by violent exothermic decomposition was encountered.

Optimum results were obtained with the following ratio of reactants: 1 mole of aldehyde, 1.1 moles of triphenylphosphine, and 1.5 moles of sodium chlorodifluoroacetate.

As indicated above, the 1,1-difluoro olefins liberated hydrogen fluoride on exposure to moist air. However, with the exception of 1,1-difluoro-2-(2-furyl)ethylene (V), they could be distilled through a spinning-band column and appeared stable indefinitely in a sealed tube. Compound V rapidly decomposed at room temperature in an inert atmosphere.

Experimental

Melting points were taken in capillaries in a metal block (Mel-Temp). Infrared spectra were obtained on a Beckman IR5 and a Perkin-Elmer 221, mass spectra on a CEC 21-103C, and n.m.r. spectra (carbon tetrachloride solutions) on a Varian HR60. Gas chromatography was carried out on an Aerograph A90P and A90C.

Sodium Chlorodifluoroacetate.—To a cooled, stirred solution of 60.7 g. (1.516 moles) of sodium hydroxide in 700 ml. of methanol was slowly added a solution of 198.0 g. (1.516 moles) of chlorodifluoroacetic acid (K and K Laboratories) in 300 ml. of methanol; the temperature was held below 40°. The methanol was removed *in vacuo* at 40°. The salt was pulverized and dried overnight at room temperature at 1 mm. The salt was again dried in the same way immediately before use. The solutions were prepared by stirring the finely divided salt in the solvent at 70° for about 5 min.

β,β-Difluorostyrene[12] (I).—The apparatus consisted of a 250-ml. two-necked flask fitted with a reflux condenser, a drying tube, a magnetic stirrer, and a heated equilibrating dropping funnel. The funnel was wrapped with heating tape, and a thermometer was inserted between the funnel and the tape. The solution in the funnel was heated until the thermometer read 60°. All glassware was oven dried and the system was purged with dry nitrogen before the reaction was started.

Triphenylphosphine was used as obtained from M and T Chemicals, Inc. Benzaldehyde was distilled immediately before use. Diglyme and monoglyme were refluxed for 4 hr. over calcium hydride and distilled *in vacuo*. Triglyme was stirred with calcium hydride at 160° for 4 hr. and distilled *in vacuo*. 1-Methyl-2-pyrrolidone was dried over sodium sulfate.

To a stirred, hot (160° bath) solution of 8.5 g. (0.08 mole) of benzaldehyde, 23.1 g. (0.088 moles) of triphenylphosphine, and 10 ml. of anhydrous diglyme was added dropwise over a period of 1–2 hr. a warm (60°) solution of 18.3 g. (0.12 mole) of sodium chlorodifluoroacetate in 50 ml. of anhydrous diglyme. The diglyme and product were flash distilled at 1 mm. and a bath temperature of 100° into a receiver cooled with Dry Ice. This distillate was fractionated through a spinning-band column (18 in. × 6 mm. i.d.). The yield of product collected at a head temperature of 52–54° (40 mm.) was 8.27 g. (74%): n^{20}_{D} 1.4939; $\lambda_{\max}^{\text{film}}$ 3.30, 5.82 ($\text{CH}{=}\text{CF}_2$, strong), 6.89, 7.37, 8.04, 8.52, 10.62, 12.17, 13.32, and 14.43 μ; n.m.r. τ 2.77 (5 protons) and 4.83 (1 proton, pair of doublets, $J_{\text{HF}\ trans}$ = 25 c.p.s., $J_{\text{HF}\ cis}$ = 4.3 c.p.s.)

Anal. Calcd. for $C_8H_6F_2$: C, 68.6; H, 4.32; F, 27.1. Found: C, 68.9; H, 4.45; F, 26.0.

The distilled product gave a single symmetrical peak on gas chromatography under the following conditions: 25% LAC on Chromosorb W, 6 ft. × 0.25 in., 110°, helium flow 41 ml./min., retention time 14.2 min. Gas chromatography of the flash distillate before fractionation showed that the actual yield of product was 10.6 g. (95%).

The product fumes on exposure to moist air, and some etching of glass containers was noted. The product in sealed ampoules appears to keep indefinitely.

Use of triglyme (Ansul Chemical Co.) in place of diglyme under the same conditions resulted in a yield of 71.5% of product.

***p*,β,β-Trifluorostyrene** (II).—A stirred solution of 12.41 g. (0.10 mole) of *p*-fluorobenzaldehyde (freshly distilled), 28.6 g. (0.11 mole) of triphenylphosphine, 16.8 g. (0.11 mole) of sodium chlorodifluoroacetate, and 50 ml. of monoglyme was heated under reflux (drying tube attached) at a bath temperature of 90–95° for 4 days. The apparatus had been oven dried and the system was purged with dry nitrogen before heating. The reaction was stopped when a quantitative amount of carbon dioxide had been evolved. The reaction mixture was cooled and filtered. The filtrate was distilled through a spinning-band column at reduced pressure (60 mm.) to remove monoglyme. The product distilled at a head temperature of 47° (30 mm.): yield 10.2 g. (65%); $\lambda_{\max}^{\text{film}}$ 5.75 (strong $\text{CH}{=}\text{CF}_2$), 6.20, 6.60, 7.05, 7.39, 8.00, 8.48, 10.60, 11.65, and 11.85 μ; n.m.r. τ 2.95 (4 protons, multiplet) and 4.81 (1 proton, two doublets, $J_{\text{HF}\ trans}$ = 25 c.p.s., $J_{\text{HF}\ cis}$ = 5 c.p.s.).

Anal. Calcd. for $C_8H_5F_3$: C, 61.2; H, 2.56. Found: C, 60.4; H, 2.96.

The product gave a peak on g.l.c. (15% silicone DC 550 on Chromosorb W, 5.5 ft. × 0.25. in., 85°, helium flow 34 ml./min.) with retention time of 17.3 min.

***p*-Methoxy-β,β-difluorostyrene** (III).—A stirred solution of 13.6 g. (0.10 mole) of freshly distilled anisaldehyde, 28.6 g.

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[13] Before this solution was devised, several runs were successfully carried out by portionwise addition of the dry acetate powder. This was done, in a closed system, by enclosing the powder in a cloth bag that was tied to the end of the rod of a Trubore stirring apparatus (minus the paddle). Thus the bag could be "dunked" briefly and intermittently into the hot solution and withdrawn out of the vapor zone into an adaptor between the Trubore fitting and the flask joint. This device can give better control than a Soxhlet-type extractor.

(0.11 mole) of triphenylphosphine, 16.8 g. (0.11 mole) of sodium chlorodifluoroacetate, and 50 ml. of monoglyme was heated under reflux (drying tube attached) at a bath temperature of 95° for 3 days. The apparatus had been oven dried and the system was purged with dry nitrogen before heating.

The mixture was cooled to room temperature and sodium chloride (6.2 g., theory 6.4 g.) was removed by filtration. Monoglyme was removed at 60 mm. through a spinning-band column. The product distilled at 49° (1.5 mm.); yield 10.2 g. (60%); $\lambda_{\max}^{\text{film}}$ 3.4, 3.5, 5.75 (strong, $CH{=}CF_2$), 6.19, 6.60, 6.81, 7.40, 7.70, 8.10, 8.43, 8.54, 9.62, 10.63, and 11.90 μ.

Anal. Calcd. for $C_9H_8F_2O$: C, 63.5; H, 4.74; F, 22.23. Found: C, 64.0; H, 4.92; F, 22.25.

The product had a retention time of 12.2 min. on g.l.c. (20% silicone SF96 on firebrick, 5 ft. × 0.25 in., 142°, helium flow 51 ml./min.).

1,1-Difluoro-1-octene[14] **(IV)**.—The apparatus used was described above under β,β-difluorostyrene (I). To a stirred, hot (185° bath) solution of 6.85 g. (0.060 mole) of heptanal, 17.3 g. (0.066 mole) of triphenylphosphine, and 10 ml. of anhydrous 1-methyl-2-pyrrolidone was added dropwise over a period of 1 hr., a warm (60°) solution of 18.3 g. (0.12 mole) of sodium chlorodifluoroacetate in 50 ml. of anhydrous 1-methyl-2-pyrrolidone. The solvent and product were flash distilled at 1 mm. and a bath temperature of 100° into a receiver cooled with Dry Ice. The distillate was fractionated through a spinning-band column (18 in. × 6 mm. i.d.). The following fractions were collected: 4.06 g. (99% pure by gas chromatography) at 118–121°, 0.54 g. (80% pure) at 121–130°, and 0.30 g. (50% pure) at 130–135°; total yield 52%; $\lambda_{\max}^{\text{film}}$ 3.42, 3.50, 5.72 (strong $CH{=}CF_2$), 6.85, 7.70, 8.12, 8.25, 8.48, 10.52, 11.0, and 12.4 μ.

Anal. Calcd. for $C_8H_{14}F_2$: C, 64.8; H, 9.53; F, 25.62. Found: C, 64.2; H, 10.06; F, 24.92.

Gas chromatography on 15% silicone DC 550 on Chromosorb W, 5.5 ft. × 0.25 in., 80°, helium flow 34 ml./min., gave a peak with retention time of 8.0 min.

1,1-Difluoro-2-(2-furyl)ethylene (V).—The apparatus used was described under β,β-difluorostyrene (I). To a stirred hot (160° bath) solution of 4.80 g. (0.05 mole) of freshly distilled furfural (Matheson Coleman and Bell), 14.5 g. (0.055 mole) of triphenylphosphine, and 10 ml. of anhydrous triglyme was added dropwise over a period of 1 hr., a warm (60°) solution of 11.4 g. (0.075 mole) of sodium chlorodifluoroacetate and 25 ml. of anhydrous triglyme. The product (4.46 g., 69% yield) was isolated by flash distillation at a bath temperature of 70° (20 mm.) into a receiver cooled with Dry Ice. A boiling point of 49° (195 mm.) was obtained, but the product resinified to a dark green mass during fractional distillation with the bath temperature at 60°. Analytical samples were collected by g.l.c. The retention time for the product was 10.4 min. and, for monoglyme, 9.4 min. under the following conditions: 15% silicone DC 550 on Chromosorb W, 10 ft. × 0.25 in., 71°, helium flow 40.5 ml./min.

Satisfactory combustion analyses were not obtained because of rapid decomposition of the samples at room temperature. However, the mass spectrum, infrared spectrum, and n.m.r. spectrum provided unequivocal identification: $\lambda_{\max}^{\text{film}}$ 5.74 ($CH{=}CF_2$, strong), 6.68, 7.43, 7.89, 8.01, 8.54, 9.80, 10.25, 10.77, 10.97, 11.30, 12.30, and 13.65 μ; n.m.r. τ 2.60 (1 proton, multiplet), 3.60 (2 protons, multiplet), and 4.60 (1 proton, pair of doublets, $J_{HF\,trans} = 25.7$ c.p.s., $J_{HF\,cis} = 3.3$ c.p.s.) [*Anal.* of mass spectrum. Calcd.[15] for $C_6H_4F_2O$: parent mass 130, parent + 1(6.59% of parent), parent + 2(0.38% of parent). Found: parent mass 130, parent +1(6.54% of parent), parent + 2-(0.38% of parent).].

***p*-Nitro-β,β-difluorostyrene (VI) and *p*-Nitro-β,β,β-trifluoroethylbenzene (VII)**.—The apparatus used was described under β,β-difluorostyrene (I). Because a solution of *p*-nitrobenzaldehyde and triphenylphosphine in diglyme darkened on heating, the aldehyde was added dropwise in this preparation.

To a stirred, hot (160° bath) solution of 23.1 g. (0.88 mole) of triphenylphosphine and 23 ml. of anhydrous diglyme was added dropwise over 1 hr. a warm (60°) solution of 12.1 g. (0.08 mole) of *p*-nitrobenzaldehyde (Eastman), 18.3 g. (0.12 mole) of sodium chlorodifluoroacetate, and 85 ml. of anhydrous diglyme. The diglyme was flash distilled at 100° (1 mm.) (bath temperature). The bath temperature was raised to 130°, whereupon 8 g. of crystalline material was collected in the receiver. Two recrystallizations from petroleum ether (b.p. 60–90°) gave 3.3 g. of a white crystalline product, m.p. 65–66°, which gave a single peak on gas chromatography. The following data established its identity as *p*-nitro-β,β,β-trifluoroethylbenzene (VII): $\lambda_{\max}^{\text{melt}}$ 3.29, 3.40, 3.50, 6.23, 6.55, 7.40, 7.95, 8.29, 8.72, 8.98, 9.22, 9.80, 10.93, 11.10, 11.92, 12.31, 13.35, and 14.20 μ; n.m.r. τ 1.78 (2 protons, doublet, $J_{HH} = 9$ c.p.s.), 2.50 (2 protons, doublet, $J_{HH} = 9$ c.p.s.), and 6.51 (2 protons, quartet, $J_{HF} = 10.5$ c.p.s.) [*Anal.* of mass spectrum. Calcd.[15] for $C_8H_6F_3NO_2$: parent mass 205, parent + 1(9.20% of parent), parent + 2 (0.78% of parent). Found: parent mass 205, parent + 1(9.24% of parent), parent + 2(0.79% of parent).].

Anal. Calcd. for $C_8H_6F_3NO_2$: C, 46.9; H, 2.95; F, 27.8; N, 6.84. Found: C, 47.3; H, 3.35; F, 29.0; N, 6.94.

Gas chromatography on 15% silicone 550 on Chromosorb W, 5.5 ft. × 0.25 in., 140°, helium flow 46 ml./min., gave a peak with retention time 18.9 min.; on 25% LAC on Chromosorb W, 6 ft. × 0.25 in., 190°, helium flow 42 ml./min., retention time was 18.8 min.

The mother liquor from the petroleum ether recrystallization contained two products which were separated by preparative gas chromatography. One of the products was VII, the other (300 mg., 2.1% yield) was *p*-nitro-β,β-difluorostyrene (VI): m.p. 29.5–30.5; $\lambda_{\max}^{\text{melt}}$ 3.27, 3.40, 3.50, 5.80, (strong, $CH{=}CF_2$), 6.25, 6.56, 7.40, 7.92, 8.32, 8.50, 8.71, 8.98, 10.57, 10.92, 11.60, 11.93, 12.30, 13.32, 13.48, 14.18, and 14.40 μ; n.m.r. τ 1.84 (2 protons, doublet, $J_{HH} = 9$ c.p.s.), 2.55 (2 protons, doublet, $J_{HH} = 9$ c.p.s.), and 4.60 (1 proton, 2 doublets, $J_{HF\,trans} = 25$ c.p.s., $J_{HF\,cis} = 3.8$ c.p.s.) [*Anal.* of mass spectrum. Calcd.[15] for $C_8H_5F_2NO_2$: parent mass 185, parent + 1(9.19% of parent), + 2(0.78% of parent). Found: parent mass 185, parent + 1 (9.28% of parent), parent + 2(1.04% of parent).].

Anal. Calcd. for $C_8H_5F_2NO_2$: C, 51.9; H, 2.73; F, 20.53. Found: C, 52.2; H, 3.25; F, 20.60.

Gas chromatography on 15% silicone DC 550 on Chromosorb W, 5.5 ft. × 0.25 in., 140°, helium flow 46 ml./min., gave a retention time of 23.0 min.; on 25% LAC on Chromosorb W, 6 ft. × 0.25 in., 190°, helium flow 42 ml./min., retention time was 18.8 min.

Acknowledgment.—This work was carried out under the internal research program of Stanford Research Institute. The n.m.r. spectra were run by Mr. W. R. Anderson, Jr., and the mass spectra, by Mrs. J. S. Whittick.

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(14) J. L. Anderson, U. S. Patent 2,733,278 (Jan. 31, 1956); *Chem. Abstr.*, **50**, 15575*c* (1956).

(15) R. M. Silverstein and G. C. Bassler, "Spectrometric Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1963, p. 9.