Reaction of Xenon Difluoride with Aromatic Compounds. I. Generation and Electron Spin Resonance Spectra of Radical Cations[1a]

M. J. Shaw,[1b] J. A. Weil,[1b] H. H. Hyman,[1b] and R. Filler[1c]

Contribution from the Chemistry Division, Argonne National Laboratory, Argonne, Illinois 60439, and the Department of Chemistry, Illinois Institute of Technology, Chicago, Illinois 60616.

Received December 5, 1969

Abstract: Esr studies of benzene and substituted benzenes reacting with XeF_2 in CH_2Cl_2 (−80–0°) in the presence of HF have revealed that radical cations of polyphenyls are formed. Esr spectra of the *para*-substituted polyphenyl radicals $R(C_6H_4)_2R^+$, where R = CH_3, F, Cl, and $F–C_6H_4–C_6H_4$, are described. Xenon difluoride is shown to be a useful reagent for generating radical cations, particularly from compounds with relatively high oxidation potential. In some cases, fluorinated 1+ ions are produced.

Radical cations have been shown to be of considerable importance as intermediates in chemical oxidations,[2] electrochemical cyanation,[3,4] methoxylation,[5] and acetoxylation reactions,[6] in radiation chemistry,[7] and in high-pressure mass spectrometry.[8] Their hyperfine splitting constants, obtainable from electron spin resonance measurements, provide information about the unpaired spin distribution in the ground state. Comparison of radical cation and anion hyperfine splitting constants provides a useful test of molecular orbital theories.[9] These models yield charge distributions which are useful in discussing orientation effects acting during attack by nucleophilic reagents. Andreades and Zahnow[3] have correlated orientation effects in electrochemical cyanation with spin densities obtained from esr measurements.

A wide variety of reagents and solvents have been used for the preparation of aromatic radical cations.[10,11] The critical factors for the successful generation and esr measurements of such cations are the use of an oxidizing agent which is selective and has a large electron affinity with respect to the aromatic molecule being oxidized, and the use of a sufficiently polar inert solvent. The temperature selected must be sufficiently low to ensure stability of the radical cation without causing solvent viscosity to become great enough for anisotropy effects in the esr spectra to be deleterious. In addition to these criteria, the work of Aalbersberg, *et al.*,[12] confirms that oxygen causes loss of hyperfine structure in the esr spectra of radical cations and is to be avoided. In view of these requirements, it is not too surprising that the liquid solution esr spectra of the benzene radical cation and its monosubstituted derivatives have not been reported. Radical cations of more easily oxidized substances such as substituted N,N-dimethylanilines,[13] polymethylbenzenes,[14] polymethoxybenzenes,[15] and nitrosobenzene[16] are known.

In a recent communication,[17] we reported that XeF_2, in the presence of small amounts of HF, reacted with benzene to yield fluorobenzene and small amounts of biphenyl, 2- and 4-fluorobiphenyl, plus xenon and hydrofluoric acid. We proposed that fluorination proceeds *via* a nucleophilic attack by HF_2^- on an intermediate benzene 1+ ion, and that the latter radical cation also reacts with benzene to form biphenyl. The purpose of the present investigation was to demonstrate *via* electron spin resonance studies that radical cations are indeed present. We have not observed a spectrum ascribable to the benzene 1+ ion itself, but have obtained confirmatory evidence for the presence of other radical cations. Many of these are the 1+ ions of *para*-disubstituted biphenyls. We have found that the combination of XeF_2 and HF, with CH_2Cl_2 as solvent, is a useful reagent for preparation of a variety of new aromatic radical cations from parent compounds with relatively high oxidation potential.[5,18] Xenon difluoride is easily prepared[19] and is commercially available.[20] The above combination offers several advantages in that temperatures as low as −90° are usable for the solution esr measurements, and

---

[1] (a) Work performed under the auspices of the U. S. Atomic Energy Commission; (b) Argonne National Laboratory; (c) Illinois Institute of Technology.

[2] P. J. Andrulis, M. J. S. Dewar, R. Dietz, and R. L. Hunt, *J. Amer. Chem. Soc.*, **88**, 5473 (1966).

[3] S. Andreades and E. W. Zahnow, *ibid.*, **91**, 4181 (1969).

[4] V. D. Parker and B. E. Burger, *Tetrahedron Lett.*, 4065 (1965).

[5] N. L. Weinberg and H. R. Weinberg, *Chem. Rev.*, **68**, 449 (1968).

[6] L. Eberson, *J. Amer. Chem. Soc.*, **89**, 4669 (1967).

[7] R. B. Cundall and W. Tippett, International Conference on Radiation Chemistry, Argonne National Laboratory, Aug 12, 1968.

[8] (a) S. Wexler and R. P. Clow, *J. Amer. Chem. Soc.*, **90**, 3940 (1968); (b) S. Wexler and L. G. Pobo, *ibid.*, **91**, 7233 (1969); (c) S. Wexler and L. G. Pobo, *J. Phys. Chem.*, 74, 257 (1970).

[9] (a) D. Purins and M. Karplus, *J. Amer. Chem. Soc.*, **90**, 6275 (1968); (b) R. E. Moss and G. K. Fraenkel, *J. Chem. Phys.*, **50**, 252 (1969); (c) E. DeBoer and S. I. Weissman, *J. Amer. Chem. Soc.*, **80**, 4549 (1958).

[10] I. C. Lewis and L. S. Singer, *J. Chem. Phys.*, **43**, 2712 (1965).

[11] G. Vincow, "Radical Ions," E. T. Kaiser and L. Kevan, Ed., Interscience, New York, N. Y., 1968, p 151.

[12] W. Ij. Aalbersberg, J. Gaaf, and E. L. Mackor, *J. Chem. Soc.*, 905 (1961).

[13] B. M. Latta and R. W. Taft, *J. Amer. Chem. Soc.*, **89**, 5172 (1967).

[14] (a) R. Hulme and M. C. R. Symons, *Proc. Chem. Soc.*, 241 (1963); *J. Chem. Soc.*, 1120 (1965); (b) R. Hulme and M. C. R. Symons, *Nature (London)*, 206, 292 (1965).

[15] A. Zweig, W. G. Hodgson, and W. H. Jura, *J. Amer. Chem. Soc.*, **86**, 4124 (1964).

[16] G. Cauquis, M. Genies, H. Lemaire, A. Rassat, and J. P. Ravet, *J. Chem. Phys.*, 47, 4642 (1967).

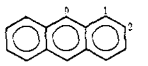
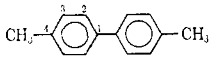
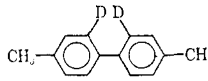
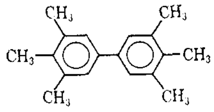
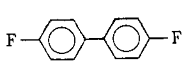
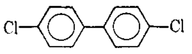
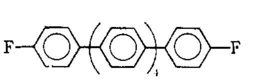
[17] M. J. Shaw, H. H. Hyman, and R. Filler, *J. Amer. Chem. Soc.*, **91**, 1563 (1969).

[18] H. Lund, *Acta Chem. Scand.*, **11**, 491 (1957).

[19] S. M. Williamson, *Inorg. Syn.*, **11**, 147 (1968).

[20] Peninsular Chemresearch Co., Gainesville, Fla.; Ozark-Mahoning Co., Tulsa, Okla.

Table I. Summary of Esr Observations on Radical Cations Produced by XeF_2–HF in CH_2Cl_2 Solvent

Radical 1+ ion	No. of lines obsd	g	Position	Hyperfine splitting,[a] G	Line width, G	Color of solution
 [b]	≥ 75	2.0028_0	1-H	3.06	≤ 0.16	Green
			2-H	1.37		
			9-H	6.47		
			^{13}C	8.48		
 [c]	≥ 62	2.0028_3	2-H	2.65	≤ 0.34	Green, then purple
			3-H	0.08		
			4-CH_3	8.80		
			^{13}C	1.77		
 [d]	≥ 63	2.0028_0	2-H	2.65	≤ 0.40	Green, then purple
			2-D	0.41		
			3-H	Not resolved		
			4-CH_3	8.79		
 [e]	≥ 27	2.0027_3	2-H	2.43	≤ 0.38	Red-brown
			4-CH_3	8.77		
			3-CH_3	Not resolved		
 [f]	≥ 15	2.0034_6	2-H	2.79	≤ 0.22	Green
			4-F	19.56	≤ 0.95	
 [g]	≥ 11	2.0061	H	2.27	≤ 1.27	Blue, then light green
			^{35}Cl	2.27		
			^{37}Cl	1.87		
 [h]	≥ 92	2.0027	Center rings-H	0.60	≤ 0.025	Green
			para-F	3.59		
			meta-H	Not resolved		
			ortho-H	2.24		

[a] No correction for second-order hyperfine terms has been made. [b] Generated from anthracene (0.0020 *M*, measured at −70°). [c] Generated from 4,4′-dimethylbiphenyl (0.00173 *M*, measured at −70°) and toluene (0.10 *M*, measured at −70°). [d] Generated from toluene-*3-d* (0.0925 *M*, measured at −70°). [e] Generated from 1,2,3-trimethylbenzene (0.088 *M*, measured at −70°). [f] Generated from 4,4′-difluorobiphenyl (0.00334 *M*, measured at −75°), 4,4′-dibromobiphenyl (0.0022 *M*, measured at −75°), biphenyl (0.009 *M*, measured at −20°), fluorobenzene (0.046 *M*, measured at −75°), and benzene (0.100 *M*, measured at −25°). [g] Generated from 4,4′-dichlorobiphenyl (0.0019 *M*, measured at −50°) and chlorobenzene (0.100 *M*, measured at −75°). [h] Generated from benzene (0.100 *M*, measured at −30°), biphenyl (0.009 *M*, measured at −65°), and terphenyl (0.0014 *M*, measured at −70°).

the only reduction products are xenon gas and fluoride ions, both diamagnetic.

Results

Hyperfine splitting constants and related experimental details for a number of radical cations are listed in Table I. Experimental conditions were varied when necessary to achieve well-resolved esr spectra. However, a systematic variation of experimental conditions was not attempted. All esr measurements were made in the temperature range of −80–0°. Each radical was generated at least twice to test the reproducibility of the method. Computer simulation, assuming Lorentzian line shapes, gave spectra virtually identical within noise level with those observed. Even brief exposure of solutions of these radical cations to air caused color changes and broadening or loss of the esr signal. Thus, it was necessary to rigorously exclude oxygen. Control experiments in which O_2 was substituted for XeF_2 or in which HF was not added gave no observable esr signals.

(A) **Anthracene 1+ Ion.** Bolton and Fraenkel[21] have characterized the anthracene 1+ ion in sulfuric acid and found $a_9^H = 6.5336$, $a_1^H = 3.0616$, $a_2^H = 1.3796$, $a_9^{C-13} = 8.48$ G. It was found in the present work that this radical could be prepared by oxidation of anthracene with XeF_2, giving virtually the same hyperfine splitting constants (Table I). Thus, the fact that the combination of XeF_2 and HF is capable of producing a radical cation from its parent hydrocarbon is established.

(B) **4,4′-Dimethylbiphenyl 1+ Ion.** Both toluene and 4,4′-dimethylbiphenyl gave an esr spectrum (Figure 1) consisting of a septuplet split twice into quintuplets. The splitting arising from the protons *meta* to the biphenyl linkage was observable only under low-modulation conditions. Comparison of this spectrum with the one obtained from toluene-*3-d* confirmed the assignments presented in Table I. In the case of toluene, a broad resonance was also present at the same *g* value and may be due to the toluene 1+ ion.

(C) **3,3′,4,4′,5,5′-Hexamethylbiphenyl 1+ Ion.** This radical cation was obtained by using 1,2,3-trimethylbenzene as the reactant. The spectrum consisted of a septuplet split into a quintuplet, superimposed on a broad resonance. No *meta* methyl proton splitting was observed. This is consistent with the small coupling constant observed for the *meta* protons in the 4,4′-dimethylbiphenyl 1+ ion.

(D) **4,4′-Difluorobiphenyl 1+ Ion.** Both fluorobenzene and 4,4′-difluorobiphenyl gave the same esr spectrum, which consisted of a triplet split into quintuplets. Line intensities within each quintuplet agreed well with the predicted ratios of 1:4:6:4:1. The hyperfine splitting constants agree well with those ($a^F = 19.28$, $a^H = 2.73$ G) reported by Fisher and Zimmermann[22] for the radical cation of 4,4′-difluorobiphenyl obtained from this biphenyl in $SbCl_5$–CH_2Cl_2 at −90°. They report that the *meta* proton splitting is

---

(21) J. R. Bolton and G. K. Fraenkel, *J. Chem. Phys.*, **40**, 3307 (1964).

(22) P. H. H. Fisher and H. Zimmermann, *Tetrahedron Lett.*, 797 (1969).

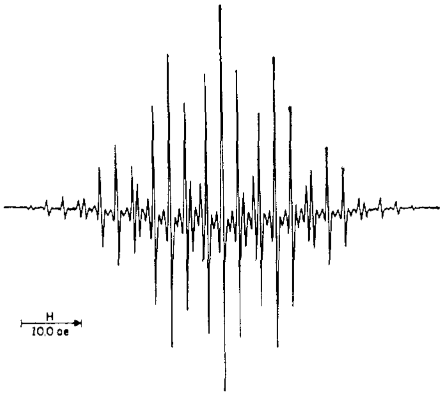


Figure 1. The esr spectrum of the 4,4′-dimethylbiphenyl 1+ ion in CH_2Cl_2 (at *ca.* −70°).

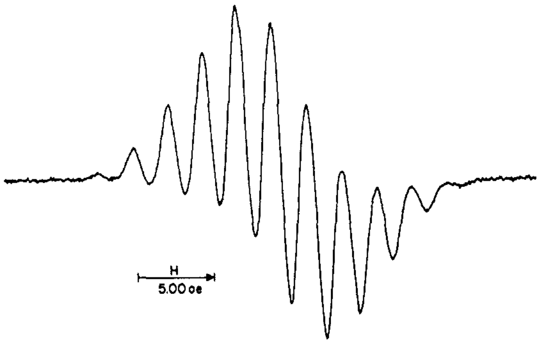


Figure 2. The esr spectrum of the 4,4′-dichlorobiphenyl 1+ ion in CH_2Cl_2 (at *ca.* −50°).

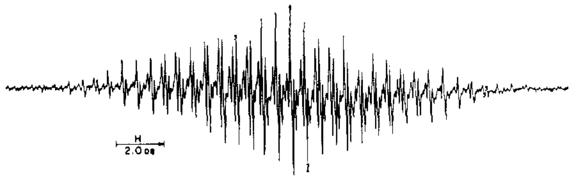


Figure 3. The esr spectrum of the *p,p′*-difluorohexaphenyl 1+ ion in CH_2Cl_2 (at *ca.* −30°).

lost in the line width of 0.8–0.9 G. We find that the spectrum exhibits alternating line widths; *i.e.*, the lines of the center quintuplet have a width of about 0.22 G, while the lines of the two outer quintuplets have a width of about 0.95 G. Thus it seems, at least in our system, that an upper limit of *ca.* 0.05 G for the *meta* proton splitting is appropriate. The line width effect may be due to ion pairing with HF_2^-, analogous to that found with the radical anions produced by alkali metal reduction of various aromatic hydrocarbons.[23] Small additional lines were observable in the center quintuplet; at least some of these may be attributed to ^{13}C splittings.

(E) **4,4′-Dichlorobiphenyl 1+ Ion.** This ion was prepared from both chlorobenzene and 4,4′-dichlorobiphenyl. The esr spectrum (Figure 2) consisted of 11 fairly broad lines. The most reasonable interpretation of this spectrum seems to be that it arises from the dichlorobiphenyl 1+ ion, in which the *ortho* proton and chlorine hyperfine splitting constants fortuitously are nearly identical. The computer-simulated spectrum taking into account the natural isotopic distribution of ^{35}Cl and ^{37}Cl (ratio 0.754/0.246), with the coupling constants given in Table I, agreed well with the observed spectrum.

It is worth noting that two of the samples gave an entirely different spectrum under the "usual" conditions (see Experimental Section). Instead of the observations described above, a four-line spectrum (g = 2.0109, a = 17.46 G), with evidence of further splitting, was observed. Computer simulation of the spectrum, assuming that the hyperfine structure was due only to ^{35}Cl and ^{37}Cl in natural abundance, and using a line width of 4.57 G, yielded an excellent fit to the experimentally observed spectrum. Comparison of these results with the work of Vanderkooi and Poole[24] leaves little doubt that this radical is ClO_2 (g = 2.0103, a = 17.0 ± 0.3 G), present because of oxygen leakage into the Kel-F sample tube. The identification of the ClO_2 radical suggests that, as in the gas phase,[25] one reaction path of the dichlorobiphenyl 1+ ion may involve the elimination of chlorine atoms. The esr spectra of the halogen atoms are known in the gas phase,[26] but it is not expected that these would be observable in solution. It is also possible that chloride ion was displaced by fluoride ion in the radical cation and was then oxidized to atomic chlorine. In a control experiment, we have observed that XeF_2 slowly oxidized gaseous HCl to chlorine, producing Xe and HF.

(F) ***p,p′*-Difluorohexaphenyl 1+ Ion.** Below −70° only a broad resonance was observed, while at temperatures between −70° and −30°, benzene reacted with XeF_2 to give the *p,p′*-difluorohexaphenyl 1+ ion. When the solution was warmed to −25° in the esr cavity, this ion decayed and XeF_2 reacted with benzene to produce the 4,4′-difluorobiphenyl 1+ ion. The *p,p′*-difluorohexaphenyl 1+ ion gave the esr spectrum shown in Figure 3 and was identified in the following manner. Terphenyl reacted (at −70°) with XeF_2 to give the same spectrum, suggesting that the number of benzene rings is a multiple of three. Biphenyl also reacted (at −65°) to give this spectrum, indicating that the number of benzene rings is also a multiple of two (at −25°, biphenyl gave the 4,4′-difluorobiphenyl 1+ ion). Therefore, the radical formed in the low-temperature benzene reaction contains 6*n* rings (*n* = 1, 2, . . .). Computer simulation of the observed spectrum, under the assumption that the protons of the inner rings are all equivalent, gave line positions and intensities consistent with the presence of the *p,p′*-difluorohexaphenyl 1+ ion. Intensities calculated for the *p,p′*-difluorododecaphenyl 1+ ion did not agree with those observed. The *p,p′*-difluorohexaphenyl-d_{24} 1+ ion was prepared from C_6D_6 and exhibited the same fluorine splitting (Table I) as the nondeuterated radical. The 4,4′-difluorobiphenyl-d_8 1+ ion was identified in a similar manner. A third radical in the solution was identified as the partially deuterated 4,4′-difluorobiphenyl 1+ ion containing two *ortho* protons. Since the C_6D_6 used was at least 98%

---

[23] (a) M. C. R. Symons, *J. Phys. Chem.*, **71**, 172 (1967); (b) H. Höfelmann, J. Jagur-Grodzinski, and M. Szwarc, *J. Amer. Chem. Soc.*, 91, 4645 (1969).

[24] N. Vanderkooi and T. R. Poole, *Inorg. Chem.*, **5**, 1351 (1966).

[25] (a) M. K. Carter and G. Vincow, *J. Chem. Phys.*, **47**, 292 (1967); (b) O. Edlund, P. Kinell, A. Lund, and A. Shimizu, *ibid.*, **46**, 3679 (1967).

[26] N. Vanderkooi and J. S. Mackenzie, *Advan. Chem. Ser.*, No. 36, 98 (1962).

isotopically pure (by mass spectral analysis), it is presumed that exchange from HF gave either partially protonated benzene or biphenyl.

(G) **Miscellaneous Data.** Of all the compounds examined, the following gave no esr signal under the usual conditions: *p*-dichlorobenzene, *p*-difluorobenzene, *p*-chlorofluorobenzene, nitrobenzene, and benzotrifluoride. It is not known whether this behavior is due to the fact that the oxidation potential of XeF_2–HF lies below those of these compounds or whether other factors are involved. Iodobenzene and bromobenzene reacted vigorously with XeF_2–HF to yield deep blue solutions which gave no esr signals. The blue color in the former case may be associated with the iodine (I_2^+) cation.[27] The compound 4,4′-dibromobiphenyl also gave a deep blue solution and exhibited the esr spectrum of the 4,4′-difluorobiphenyl 1+ ion. The formation of the latter ion probably is a result of a nucleophilic displacement of Br^- by F^-, similar to that observed by reaction of lutidine with the 9,10-dibromoanthracene radical cation.[28] The compounds *o*-, *m*-, and *p*-xylene, and mesitylene gave esr signals with resolved structure which appeared to arise from the presence of more than one species. These radicals are currently under investigation.

Summary

Several factors appear to determine which radical cation will be observed when a given aromatic compound reacts with XeF_2. The 1+ ion of the parent compound is observed when this cation is sufficiently stable, *e.g.*, anthracene or 4,4′-dimethylbiphenyl radical cations. With benzene, the radical cation observed depends on the reaction temperature; the *p,p′*-difluorohexaphenyl 1+ ion dominates at lower temperatures and the 4,4′-difluorobiphenyl 1+ ion predominates above −25°. It thus appears that polymerization is faster than fluorination at low temperatures, and that fluorination is a termination step. Monosubstituted benzenes generally react to give 4,4′-disubstituted biphenyls and the 1+ ions of the latter. The nature of the biphenyl cation also depends on whether the substituent is replaceable by fluorine.

The use of XeF_2 to produce fluorinated substituted benzenes has been investigated. Preparative experiments, encompassing product analyses and competition reactions directed at the elucidation of the mechanism of fluorination and biphenyl formation, will be presented elsewhere. These have led us to postulate the existence of benzene and substituted benzene 1+ ions as intermediates to the observed biphenyls and their radical cations. Monocylic radical cations have not, in fact, been observed by us, presumably because these are highly reactive species. Under our experimental conditions, in which an excess of substituted benzene is present, it is expected that they would rapidly react to form biphenyls and their 1+ ions.

Experimental Section

Materials. Xenon difluoride was prepared and initially purified according to the procedure of Schreiner, *et al.*,[29] and was free from xenon tetrafluoride. Xenon difluoride was deposited, stored, and dispensed from a Kel-F tube, fitted with a $^1/_4$-in. × $^1/_4$-in. Kel-F valve, containing anhydrous sodium fluoride. The observation that none of the organic compounds reacted until HF was added indicates that the XeF_2 thus treated was free from HF.

Anhydrous hydrogen fluoride with conductivity = 2×10^{-6} mho was used. Benzene, 99.999%; toluene, 99.9% (James Hinton Co., Newport News, Va.); and anthracene, 99.999% (Princeton Organics, Princeton, N. J.) were used. All other materials were of standard commercial grade. Solids were purified by fractional sublimation. All liquids were dried over P_2O_5 and fractionally distilled over anhydrous Na_2SO_4.

Toluene-*3-d* was prepared from *m*-bromotoluene by hydrolysis of the corresponding Grignard reagent with D_2O (99.8%), according to the procedure of Weldon and Wilson.[30] After drying over P_2O_5 and two fractional distillations, a sample was obtained which, by infrared analysis,[31] was shown to be approximately 96.6% toluene-*3-d*. Crude mass spectral analysis indicated that the toluene-*3-d* concentration was at least 88%. No lines due to nondeuterated toluene were detected in the esr spectrum of this compound.

The 4,4′-dichlorobiphenyl was prepared according to the procedure of Shaw and Turner[32] and purified by recrystallization from toluene and light petroleum, and by fractional sublimation, mp 149–150° (lit.[31] 149–150°).

Throughout this investigation, Kel-F and metal vacuum lines and accessories were used.[33]

Esr Sample Preparation. Solutions were prepared by dissolving reactants in CH_2Cl_2 which had been distilled over P_2O_5 under an atmosphere of argon. After a final saturation of these solutions with argon, 0.5 ml was quickly pipetted into a $^1/_4$-in. × 12-in. Kel-F tube which had previously been washed with benzene and evacuated to 2×10^{-6} Torr and then filled with argon. This tube was then connected to a Kel-F "Y" connector *via* a $^1/_4$-in.–$^1/_4$-in. Kel-F valve.[34] The Kel-F esr tube (0.154-in. o.d. × 6 in., wall thickness 0.017 in.), containing about 1×10^{-4} mol of XeF_2, fitted with a $^1/_4$-in.–$^1/_8$-in. Kel-F valve,[34] was connected to the other arm of the Y. The Y assembly was evacuated to 2×10^{-6} Torr. The esr tube was then cooled to *ca.* −75° and evacuated to the same pressure. The organic solution was degassed by alternately freezing at *ca.* −125° and warming to room temperature until a pressure *change* of less than 2×10^{-6} Torr was observed when the organic solution was frozen and opened to the vacuum line. After degassing was complete, 0.5 ml of the organic solution was distilled or poured *in vacuo* into the esr tube at −75°. Then about 3×10^{-4} mol of very pure HF was transferred from a 29-ml Kel-F tube to the esr tube at *ca.* −125°. The tube was warmed with agitation to *ca.* −75° and transferred to the esr cavity at about −75°. The temperature was raised until an esr spectrum was observed. Occasionally, shaking the cold sample for short periods of time at room temperature and subsequent cooling increased the radical concentration. In general the radicals were stable for several hours at the temperature of esr measurement.

Measurements. The esr measurements were carried out at *ca.* 9100 MHz using a standard Varian V4500-10A spectrometer, including a V-4531 multipurpose cavity and a V-4557 variable temperature system. The latter allowed operation down to *ca.* −100° by adjustment of the flow of cold nitrogen gas around the sample tube. Magnetic fields and microwave frequencies were measured by use of a Perkin-Elmer M2 (nmr) gaussmeter plus CMC counter system. Temperatures were measured with a copper–constantan thermocouple inserted in the N_2-flow system just below the sample, and were checked with a calibrated thermocouple inserted into CH_2Cl_2 in an esr tube at the sample site. The simulated spectra were calculated on a CDC 3600 computer using program C168 (Argonne National Laboratory).

Acknowledgment. We are grateful for the assistance given us by Drs. L. Kaplan and K. Wilzbach and also by the Argonne Analytical group, especially Mrs. A. Engelkemeir, with the mass spectral identification of compounds.

---

[27] R. J. Gillespie and J. M. Milne, *Chem. Commun.*, 158 (1966).

[28] V. D. Parker and L. Eberson, *ibid.*, 973 (1969).

[29] F. Schreiner, G. M. McDonald, and C. L. Chernick, *J. Phys. Chem.*, 72, 1162 (1968).

[30] L. H. P. Weldon and C. L. Wilson, *J. Chem. Soc.*, 235 (1946).

[31] M. Takahasi, *Bull. Chem. Soc. Jap.*, 33, 808 (1960).

[32] F. R. Shaw and E. E. Turner, *J. Chem. Soc.*, 285 (1932).

[33] J. H. Canterford and O. T. A. O'Donnell, *Tech. Inorg. Chem.*, 7, 273 (1968).

[34] H. H. Hyman and J. J. Katz, "Non-aqueous Solvent Systems," T. C. Waddington, Ed., Academic Press, New York, N. Y., 1965, p 50.