For nearly pure $CHF_2(CF_3)PCl$, the vapor-phase molecular weight was 186.9 (calcd 186.5). Its volatility (10.7 mm at −45 °C, 45.6 mm at −22.6 °C, and 150 mm at 0 °C) is described by $\log P = 5.538 + 1.75 \log T - 0.005T - 1710/T$ (bp 40 °C, Trouton constant 21.2 eu). Its easily observable infrared peaks (with relative intensities) are at 2950 (0.14, wide), 1333 (0.6), 1294 (14), 1213 (11), 1158 (57), 1088 (35), 1023 (26), 982 sh (3), 956 sh (2), 740 (1.4), 550 (3.0), 480 (0.9), 445 (1.6), and 420 $cm^{-1}$ (1.3).

The volatility of $CH_2F(CF_3)PCl$ (15.7 mm at −22.6 °C or 61 mm at 0 °C) suggests $\log P = 6.729 + 1.75 \log T - 0.0065T - 2030/T$ (bp 60 °C, Trouton constant 21.4 eu). Infrared data: 1430 (0.33), 1380 (0.15), 1309 (0.9), 1260 (0.8), 1169 (19), 1153 (27), 1090 (10), 1034 (10) 922 (0.4), 850 (0.45), 768–720 (0.2), 703 (0.44), 545 (1.6), 518 (1.6), 448 (0.9), 418 $cm^{-1}$ (0.8).

The bis(phosphino)methane $(CF_3)_2PCH_2P(CF_3)Cl$ will be more fully described in another paper. Its unusual geminal-$CF_3$ NMR spectrum left no doubt of its presence. A number of other minor byproducts of the HCl–aminophosphine reaction (with complicated $^{19}F$ NMR spectra near δ 50) were not identified.

**P–H Phosphines.** The reaction series

$$R(CF_3)PCl + HI \rightleftharpoons R(CF_3)PI + HCl \qquad (1)$$

$$RCF_3PI + HI + Hg \rightarrow HgI_2 + R(CF_3)PH \qquad (2)$$

can be completed by three additions of excess HI to the rotatable reaction tube; in the one-run process, the conversion is only 75–80% because the direct removal of HI by Hg intervenes. The yield of HCl may be only half of the calculated value; then $Hg_2Cl_2$ would account for the rest of the chloride. Reaction 2 was shown in separate experiments to be fully as quantitative as the prototype process with $(CF_3)_2PI$.[6]

Tables II and III show the NMR data for both $RCF_3PH$ compounds. Further work with $CHF_2(CF_3)PH$ showed interesting variations which are being studied further by Dr. L. D. Field, a recent member of our Hydrocarbon Institute. For example, $_2J_{FCP}$ for $F_A$ (the downfield member of the AB pattern) varies from 126 Hz in the neat sample at −30 °C to 112 Hz in $DCCl_3$ at 30 °C, and similarly $_2J_{FCPH}$ varies from 17.4 to 13.6 Hz; but for $F_B$ these parameters remain nearly constant—and so do all other parameters not relating to $F_A$ and the PH proton. It might be suggested that $F_A$ and the PH proton have a rubbing contact, with some electrostatic attraction. The effect of this would diminish with dilution and rise of temperature. The concept is justified by the expected bond distances and angles and van der Waals radii; but it is not a final conclusion.

For the purification of $CHF_2(CF_3)PH$ it was considered that the almost equally volatile $CH_3(CF_3)PH$ could be a minor impurity. This could be removed by forming the $BH_3$ complex, which is not appreciably volatile at −78 °C.[7] Then the 98% pure sample had mol wt 151 (calcd 152), and its volatility (6.3 mm at −63.7 °C, 25.8 mm at −45.3 °C, and 102.6 mm at −22.7 °C) suggested the equation $\log P = 6.000 + 1.75 \log T - 0.006T - 1674/T$ (bp 22 °C, Trouton constant 21.3 eu). The significant infrared frequencies are 2967 (0.6), 2335 (0.4), 1404 (0.3), 1308 (4), 1262 (1.6), 1163 (24), 1149 (26), 1092 sh (7), 1070 (1.1), 913 (2.1), 903 (1.8), 855 (3), 824 (2.7) 758 (0.5), 750 (0.5), 720 (0.6), 591 (0.3), 524 (0.8), 445 (1.8), and 360 $cm^{-1}$ (0.2).

For pure $CH_2F(CF_3)PH$, mol wt = 134, as calculated. Its volatility (3.6 mm at −51.8 °C, 15.0 mm at −32.7 °C, 28.7 mm at −22.6 °C, and 103 mm at 0 °C) implies $\log P = 5.756 + 1.75 \log T - 0.005T - 1814/T$ (bp 47 °C, Trouton constant 21.3 eu). Its infrared frequencies included fundamentals scarcely stronger than some overtones or combinations; hence all peaks easily observable at 82 mm pressure with path 113 mm are listed: 2925 (0.29), 2825 (0.05), 2590 (0.22), 2313 (0.22), 2265 (0.18, wide), 2064 (0.05), 1978 (0.04), 1928 (0.02), 1876 (0.07), 1750–1550 (0.07), 1438 (0.29), 1315 (0.9), 1260 (0.8), 1179 (15), 1142 (39), 923 (0.8), 876 (0.45), 830 (3.4), 800 sh (0.7), 742 (0.8), 644 (0.09), 555 (0.09), 520 (0.17), 475 (0.07), 410 (0.37), 360 $cm^{-1}$ (0.35).

The NMR parameters of $CH_2F(CF_3)PH$ did not vary appreciably with dilution.

**Significance.** The new chiral phosphines are probable precursors of numerous ligands suitable for the study of electronic effects in transition-element complexes. The P–H phosphines also could be used for making $(RCF_3PBH_2)_3$ ring compounds offering a wide range of subtly variant properties.

**Acknowledgment.** Thanks are due to Drs. L. D. Field and K. L. Servis for advice about NMR spectra and to Dr. H. Goldwhite for further information about his earlier publication.[2]

**Registry No.** $(CF_3)_2PH$, 460-96-8; $(CF_3)_2PCl$, 650-52-2; $CHF_2(CF_3)PCl$, 4669-82-3; $CH_2F(CF_3)PCl$, 77846-29-8; $(CF_3)_2PCH_2P(CF_3)Cl$, 77846-30-1; $HN[P(CF_3)_2]_2$, 2349-17-9; $CHF_2(CF_3)PNH_2$, 4669-83-4; $CH_2F(CF_3)PNH_2$, 77846-31-2; $CHF_2(CF_3)PH$, 77846-32-3; $CH_2F(CF_3)PH$, 77846-33-4.

---

[6] R. G. Cavell and R. C. Dobbie, *J. Chem. Soc. A*, 1308 (1967).

[7] A. B. Burg, K. K. Joshi, and J. F. Nixon, *J. Am. Chem. Soc.*, **88**, 34 (1966).

---

# Correspondence

## Hydrogen Migration Mechanism for Ligand Substitution Reactions in Metal Carbonyl Hydrides

*Sir:*

It has been known for some time that certain transition-metal carbonyl hydrides are unusually labile in ligand substitution processes.[1] For example, $HCo(CO)_4$ is much more reactive than other $XCo(CO)_4$ molecules.[2] $HMn(CO)_5$ is considerably more reactive than similar $XMn(CO)_5$ molecules.[3] The protonated forms of iron pentacarbonyl and $Fe(CO)_4PPh_3$ are much more labile than the parent molecules.[4] Even the anion $HFe(CO)_4^-$ undergoes ligand exchange far more rapidly than $Fe(CO)_5$ does and also much more rapidly than $CH_3COFe(CO)_4^-$.[5] The anions $Co(CO)_4^-$ and $Fe(CO)_4^{2-}$ are quite inert.

A mechanism explaining this unusual lability (Scheme I) was offered involving a ligand migration of hydrogen to a

---

[1] Basolo, F.; Pearson, R. G. "Mechanisms of Inorganic Reactions"; Wiley: New York, 1967; Chapter 7.

[2] Heck, R. F. *J. Am. Chem. Soc.* **1963**, *85*, 657.

[3] Berry, A.; Brown, T. L. *J. Organomet. Chem.* **1971**, *33*, C67.

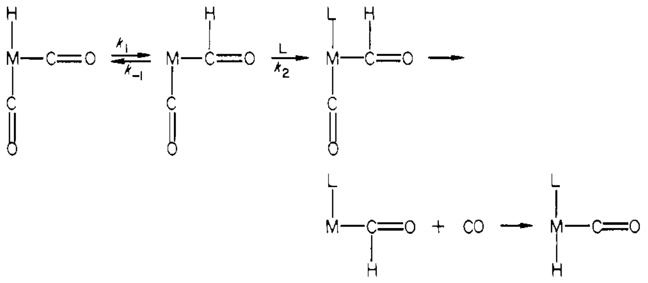
[4] Basolo, F.; Brault, A. T.; Poë, A. J. *J. Chem. Soc.* **1964**, 676.

[5] Collman, J. P.; Finke, R. G.; Matlock, P. L.; Wahren, R.; Komoto, R. G.; Brauman, J. I. *J. Am. Chem. Soc.* **1978**, 100, 1119.

[6] Mawby, R. J.; Basolo, F.; Pearson, R. G. *J. Am. Chem. Soc.* **1964**, *86*, 3994.

0020-1669/81/1320-2741$01.25/0

**Scheme I**



carbonyl group, attack at the vacated coordination site by the entering ligand, loss of a CO ligand, and back-migration of H to the metal.[1] On the assumption that the ligand substitution is essentially irreversible, the rate law for Scheme I is

$$\text{rate} = \frac{k_1k_2[\text{M}][\text{L}]}{k_{-1} + k_2[\text{L}]} \tag{1}$$

where M stands for the metal complex. Either first- or second-order kinetics can be found depending on the relative values of $k_2[\text{L}]$ and $k_{-1}$.

Counter examples exist for the hydride migration hypothesis. The analogous $HRe(CO)_5$ is unusually inert to substitution, reacting by a free radical chain mechanism.[7] The complex $H_2Os(CO)_4$ is not labile, but requires elevated temperatures for substitution[8] as does $HCH_3Os(CO)_4$.[9]

The complexes $HMoCp(CO)_3$ and $HWCp(CO)_3$ substitute phosphines for CO rather slowly.[10] Furthermore a kinetics study showed a second-order rate law and no deuterium isotope effect when $DMoCp(CO)_3$ was used. As Green and coworkers pointed out,[10] this is inconsistent with a hydride migration mechanism. The observed rate law for second-order conditions should be

$$\text{rate} = \frac{k_1k_2[\text{M}][\text{L}]}{k_{-1}} \tag{2}$$

Because of the low frequency of metal–hydrogen stretching vibrations, a large *inverse* isotope effect would be predicted for rate law 2.

Finally, the failure to observe formyl complexes in reactions of metal carbonyl hydrides and the demonstration by Casey and Neumann[11] that formyl complexes are thermodynamically unstable have doubtless made hydrogen migration mechanisms less attractive. To counter this negative evidence, it may be pointed out that only small concentrations of M–CHO are needed in Scheme I. Indeed thermodynamic instability of the intermediate formyl complex is a requirement. Also there are a number of both homogeneous and heterogeneous metal-catalyzed reactions of CO and $H_2$ which seem to demand the formation of formyl complexes at some stage.[12]

Recently we have made several observations which we feel offer strong support for the hydride migration mechanism. They also help to clarify the circumstances where such a mechanism may be expected. Our observations are as follows. (A) In acetone at −70 °C $H_2Fe(CO)_4$ reacts within 3 min with $PPh_3$ to give $H_2Fe(CO)_3PPh_3$ and with $P(OMe)_3$ to give $H_2Fe(CO)_3P(OMe)_3$ and $H_2Fe(CO)_2[P(OMe)_3]_2$.[13] The phosphine and phosphites are cis to both hydrogens, and the hydrogens are probably cis to each other. The same results are found in methanol at room temperature.[14] These substitutions are at least 30 times as fast as the thermal decomposition of $H_2Fe(CO)_4$ in methanol. $H_2Fe(CO)_3PPh_3$ decomposes at a rate similar to that of $H_2Fe(CO)_4$ and yields *trans*-$Fe(CO)_3(PPh_3)_2$ in the presence of triphenylphosphine. (B) $H_2Ru(CO)_4$ in acetone at −70 °C also gives $H_2Ru(CO)_3P(OMe)_3$ in a few minutes with the phosphite cis to hydrogen.[13] Upon warming $H_2Ru(CO)_2[P(OMe)_3]_2$ is formed. (C) In methanol at 25 °C the ion $HRu(CO)_4^-$, as the $PPN^+$ salt, is converted immediately into $HRu_3(CO)_{11}^-$. Adding excess sodium methoxide (about 0.4 M) slows the reaction time down to about 15 min, but the same product is formed.[13]

The last observation may be compared to the behavior of $H_4Ru_4(CO)_{12}$ and $H_2Os(CO)_4$, or their salts, which are quite stable in methanol at 25 °C. Also $H_4Ru_4(CO)_{12}$ undergoes ligand substitution rather sluggishly.[15]

We interpret the behavior of $HRu(CO)_4^-$ as caused by a hydride migration in $H_2Ru(CO)_4$ formed by methanolysis. The entering ligand is the anion $HRu(CO)_4^-$ (see eq 3–5).

$$HRu(CO)_4^- + CH_3OH \rightarrow H_2Ru(CO)_4 + CH_3O^- \tag{3}$$

$$H_2Ru(CO)_4 \rightarrow HRu(CHO)(CO)_3 \tag{4}$$

$$HRu(CHO)(CO)_3 + HRu(CO)_4^- \rightarrow H_2Ru_2(CO)_3CHO^- \text{ etc.} \tag{5}$$

In summary, it may be seen that rapid ligand substitutions are found for $HCo(CO)_4$, $HMn(CO)_5$, $H_2Fe(CO)_4$, and $H_2Ru(CO)_4$, all of which are thermally very unstable with the evolution of hydrogen. Slow ligand substitutions are found for $HRe(CO)_5$, $H_2O(CO)_4$, $H_4Ru_4(CO)_{12}$, $HMoCp(CO)_3$, and $HWCp(CO)_3$, all of which evolve $H_2$ much more slowly than does the first group of compounds.[16] Also substitution of CO by phosphines and phosphites in general increases both the thermal stability and reduces the speed of substituion reactions.[2]

The simplest explanation of the above is that a weak metal–hydrogen bond is a necessary requirement for both a hydride migration mechanism for ligand substitution and easy thermal decomposition. Equations 1 and 2 justify the first point, but the second is more complex. In some cases evolution of hydrogen from a metal hydride is a simple dissociation process (examples, $H_2Fe(CO)_4$,[14] $H_2Co[P(OR)_3]_4^+$,[17] $H_2Ir(CO)_2I_2^+$,[18]). In other cases ($HCo(CO)_4$[19] and $H_2Os(CO)_4$[9]), loss of CO precedes loss of $H_2$. Probably the factors which increase the strength of metal–hydrogen bonds and metal–carbonyl bonds go hand in hand in the usual case.

It is significant that rapid ligand substitutions are found for metal carbonyl hydrides of the first transition series, with one example from the second in Ru. The substitutionally inert compounds contain metals from the second and third transition series. It is fairly well established from surface studies that the strength of metal–hydrogen bonds is greater for the third transition series than for the first.[20] The second transition series seems irregular. The ease with which carbonyl substitution occurs may be a good test for metal–hydrogen bond strengths in metal carbonyl hydrides.

An alternative mechanism for rapid ligand substitution reactions is that a hydride ligand exerts a general activating

---

(7) Byers, A. H.; Brown, T. L. *J. Am. Chem. Soc.* **1975**, *97*, 947.
(8) L'Eplattenier, F.; Calderazzo, F. *Inorg. Chem.* **1968**, *7*, 1290.
(9) Norton, J. R. *Acc. Chem. Res.* **1979**, *12*, 139.
(10) Bainbridge, A.; Craig, P. J.; Green, M. *J. Chem. Soc. A* **1968**, 2715.
(11) Casey, C. P.; Neumann, S. M. *J. Am. Chem. Soc.* **1976**, *98*, 5395.
(12) For example, see: Dombek, B. D. *J. Am. Chem. Soc.* **1980**, *102*, 6855.
(13) Walker, H., unpublished observations.
(14) Mauermann, H., unpublished observations.
(15) Knox, S. A. R.; Kaesz, H. *J. Am. Chem. Soc.* **1971**, *93*, 4594.
(16) $HMoCp(CO)_3$ decomposes at 110 °C and $HWCp(CO)_3$ above 180 °C according to: Fischer, E. O., Hafner, W.; Stahl, H. O. *Z. Anorg. Allg. Chem.* **1955**, *282*, 47.
(17) Muetterties, E. L.; Watson, P. L. *J. Am. Chem. Soc.* **1978**, *100*, 6978.
(18) Mays, M. J.; Simpson, R. N. F.; Stefanini, F. P. *J. Chem. Soc. A* **1970**, 3000.
(19) Markó, L.; Ungvary, F. *J. Organomet. Chem.* **1969**, *20*, 205.
(20) Anderson, J. R. "Chemisorption and Reactions on Metallic Films"; Academic Press: New York, 1971; Vol. 2, Chapter VII. See also: Connor, J. A. *Top. Curr. Chem.* **1977**, 71, 101.

effect for the loss of coordinated CO. Certainly $H^-$ has a strong activating trans effect in many complexes. However its trans effect is hardly greater than that of CO or of $CH_3^-$, as judged both by kinetic and structural data.[21] Furthermore Brown has shown that in octahedral metal carbonyls, the activation occurs specifically in the cis positions to the activating ligand.[22] This is in agreement with the products observed in A and B, where the entering ligands are cis to both hydrides.

However it also has been shown[22] that the donor atoms which give cis activation are N, O, and halogen[23] or carbon in groups such as $CH_3CO$, COOH, and $COOCH_3$.[22] These are relatively hard donor atoms and hydride would be anomalous in this list. Indeed it would be expected that an electron-donating substituent such as $H^-$ would increase the electron density on the metal and make CO loss more difficult. A formyl group would be labilizing, as needed in the final stages of Scheme I. Finally it seems unlikely that CO is being lost as rapidly from $H_2Fe(CO)_4$ as needed for its ligand substitution reactions. This should constitute a path for thermal decomposition as seen for other hydrides.[9,19] However, the kinetics of decomposition of $H_2Fe(CO)_4$ show clearly that this is not the case.[24]

**Acknowledgment.** This work was supported by the National Science Foundation (Grant No. CH77-27147 to R.G.P.) and by the Department of Energy (Contract No. DE-AS03-76SF0034 with P.C.F.).

**Registry No.** $H_2Fe(CO)_4$, 12002-28-7; $H_2Ru(CO)_4$, 42781-58-8; $HRu(CO)_4^-$, 77482-04-3; $H_2Fe(CO)_3PPh_3$, 77482-05-4; $H_2Fe(CO)_3P(OMe)_3$, 77482-06-5; $H_2Fe(CO)_2[P(OMe)_3]_2$, 77482-07-6; *trans*-$Fe(CO)_3(PPh_3)_2$, 21255-52-7; $H_2Ru(CO)_3P(OMe)_3$, 77482-08-7; $H_2Ru(CO)_2[P(OMe)_3]_2$, 77482-09-8; $HRu_3(CO)_{11}^-$, 77482-10-1.

---

(21) Appleton, T. G.; Clark, H. C.; Manzer, L. E. *Coord. Chem. Rev.* **1973**, 10, 335. Frenz, B. A.; Ibers, J. A. Reference 16, p 33.

(22) Atwood, J. D.; Brown T. L. *J. Am. Chem. Soc.* **1976**, *98*, 3155, 3160. Brown, T. L.; Bellus, P. A. *Inorg. Chem.* **1978**, *17*, 3728.

(23) Angelici, R. J. *Organomet. Chem. Rev.* **1968**, *3*, 173.

(24) The rate of decomposition is first order in $H_2Fe(CO)_4$ and independent of $HFe(CO)_4^-$ or added CO, at low CO concentrations.

Department of Chemistry
University of California
Santa Barbara, California 93106

**Ralph G. Pearson***
**Howard W. Walker**
**Heiko Mauermann**
**Peter C. Ford**

*Received January 13, 1981*

---

## Generation of Transition-Metal Polysulfide Ions in the Gas Phase by Sequential Reactions of Metal Ions with Ethylene Sulfide

*Sir:*

The ability of sulfur atoms to form long chains is well-known.[1] Elemental sulfur, itself, is an eight-membered ring with a "crown" structure in its most common allotrope. The structure of the $S_8$ molecule in the gas phase has been determined by electron diffraction as having the same puckered ring structure as in the solid state.[2] An eight-membered ring of seven sulfurs and an imide group, $S_7NH$, has also been characterized.[3]

In addition alkali polysulfides,[4] hydrogen polysulfides,[5] and organic salts of hydrogen polysulfides[6] have been isolated. Characterization of these species by X-ray and Raman spectroscopy indicate that generally these chains all have unbranched and nonplanar structures.[1,7]

Several organometallic complexes with polysulfide ligands are known as well, beginning with $(NH_4)_2Pt[S_5]_3$ first prepared in 1903 and finally characterized by X-ray in 1967.[8] The structure consists of three five-membered sulfur chains coordinated to a central platinum atom forming with Pt three six-membered rings in chair configuration with overall octahedral coordination. Egen and Krause were able to prepare a tetrasulfide-containing complex using a nickel(II) mercaptide as a template.[9] Kopf's research group has prepared $Cp_2MS_5$ (M = V or Ti)[10] and $Cp_2MS_4$ (M = Mo or W)[11] which have crystallographically been confirmed as five-[12] and four-membered[13] nonplanar sulfur rings, respectively. In this correspondence we report the formation of metal polysulfide ions in the gas phase by sequential reactions of metal ions with ethylene sulfide.

Ion cyclotron resonance[14,15] (ICR) has often been successfully applied to inorganic systems.[16] Recently our laboratory developed the technique of laser ionization ICR[17–19] wherein virtually any atomic metal ion may be generated from a pure metal sample and reacted with various background gases for times approaching a second with subsequent mass analysis of charged products. Metal ions may also be generated by electron impact on volatile inorganic complexes, but they may comprise only a few percent of the total ion current in a large background of parent and other fragment ions. Our primary interest has been the development of metal ions as selective chemical ionization reagents,[19] while at the same time developing a mechanistic approach to prediciting reactivity. Sulfur compounds are of particular interest as analogues of oxygenated species, allowing us to investigate "hard–soft" effects on metal reactivity. Also, sulfur is a real problem in the petroleum industry, and several works have indicated that metal complexes may be effective desulfurization agents.[20]

During our studies of metal ion reactions with sulfur compounds, we observed unexpected behavior with ethylene sulfide. Upon reaction with $Fe^+$, $Co^+$, $V^+$, and $Ti^+$ successive attachments of sulfur was the dominant process in each case.

---

(1) O. Foss, *Adv. Inorg. Chem. Radiochem.*, **2**, 237 (1960), and references therein.

(2) C. S. Lu and J. Donohue, *J. Am. Chem. Soc.*, **66**, 818 (1944).

(3) J. Weiss *Z. Anorg. Allg. Chem.*, **305**, 190 (1960).

(4) F. Feher and H. J. Berthold, *Z. Anorg. Allg. Chem.*, **274**, 223 (1953).

(5) H. Kreb and K. H. Muller, *Z. Anorg. Allg. Chem.*, **281**, 187 (1955).

(6) F. Feher and G. Winkhaus, *Z. Anorg. Allg. Chem.*, **288**, 123 (1956).

(7) R. Rahman, S. Safe, and A. Taylor, *Q. Rev., Chem. Soc.*, **24**, 208 (1970).

(8) P. E. Jones and L. Katz, *Acta Crystallogr., Sect. B*, **B25**, 745 (1969).

(9) N. B. Egen and R. A. Krause, *J. Inorg. Nucl. Chem.*, **31**, 127 (1969).

(10) (a) H. Kopf, *Chem. Ber.*, **101**, 272 (1968); (b) H. Kopf, A. Wirl, and W. Kahl, *Angew. Chem., Int. Ed. Engl.*, **10**, 137 (1980).

(11) H. Kopf, W. Kahl, and A. Wirl, *Angew. Chem., Int. Ed. Engl.*, **9**, 801 (1970).

(12) E. F. Epstein, I. Bernal, and H. Kopf, *J. Organomet. Chem.*, **26**, 229 (1971).

(13) B. R. Davis, I. Bernal, and H. Kopf, *Angew. Chem., Int. Ed. Engl.*, **10**, 921 (1971).

(14) T. A. Lehman and M. M. Bursey, "Ion Cyclotron Resonance Spectrometry", Wiley-Interscience, New York, 1976.

(15) J. L. Beauchamp, *Annu. Rev. Phys. Chem.*, **22**, 527 (1971).

(16) See, for example: (a) R. C. Dunbar, *J. Am. Chem. Soc.*, **90**, 5676 (1968); (b) M. S. Foster and J. L. Beauchamp, *ibid.*, **97**, 4808 (1975); (c) J. Allison and D. P. Ridge, *ibid.*, **100**, 163 (1978); (d) J. Allison and D. P. Ridge, *ibid.*, **101**, 4998 (1979); (e) A. E. Stevens and J. L. Beauchamp, *ibid.*, **101**, 6449 (1979).

(17) R. B. Cody, R. C. Burnier, W. D. Reents, Jr., T. J. Carlin, D. A. McCrery, R. K. Lengel, and B. S. Freiser, *Int. J. Mass. Spectrom. Ion. Phys.*, **33**, 37 (1980).

(18) R. C. Burnier, T. J. Carlin, W. D. Reents, Jr., R. B. Cody, R. K. Lengel, and B. S. Freiser, *J. Am. Chem. Soc.*, **101**, 7127 (1979).

(19) R. C. Burnier, G. D. Byrd, and B. S. Freiser, *Anal. Chem.*, **52**, 1641 (1980).

(20) (a) H. Alper and J. Paik, *J. Org. Chem.*, **42**, 3522 (1977); (b) G. Parisod and M. Comisarow, *Adv. Mass Spectrom.*, **8**, 212 (1980).

0020-1669/81/1320-2743$01.25/0