

elimination of  $\text{CpRu}(\text{CO})_2\text{H}$  or via migration of Zr to the  $\text{C}_5\text{H}_5$  ligand bound to Ru followed by  $\beta$ -hydride elimination to give II and ring closure to I. Reversible addition of CO to I explains the formation and  $^{13}\text{C}$ O exchange of 2. Intermediate II has a vacant site at Ru that can be captured by  $\text{PMe}_3$  to eventually produce 3. The formation of ethylene adduct 4 can be explained either by insertion of ethylene into the reactive strained  $\text{Zr}-\text{C}_5\text{H}_4$  bond of I or by reaction of ethylene with II to give metallacyclobutane intermediate III.

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**Supplementary Material Available:** Spectroscopic data for 2-5 and details of the X-ray structure solutions for 2 and 3, with listings of fractional coordinates and thermal parameters, bond distances, and bond angles (9 pages). Ordering information is given on any current masthead page.

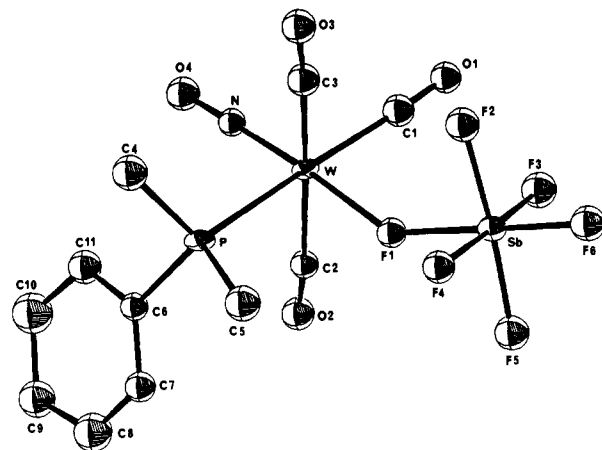
### Coordination of a "Noncoordinating" Anion: X-ray Crystal Structure and $^{31}\text{P}$ NMR Characterization of a Tungsten Nitrosyl Cation Ligated to $\text{SbF}_6^-$

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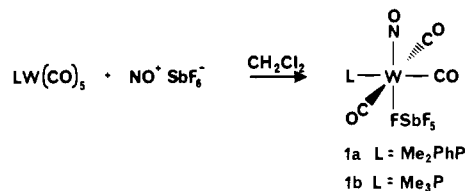
A large body of research on the spectral characterization of carbocations in superacid media has shown that these species are present as fully ionized cations.<sup>1</sup> The simplest of the many noncoordinating anions that are present in these media is the hexafluoroantimonate ion,  $\text{SbF}_6^-$ .<sup>2</sup> The coordinating ability of this anion must be exceedingly poor, and in fact it is known to give covalent adducts only with  $\text{CH}_3^+$  and  $\text{SbF}_5$ , resulting in the monofluorine-bridged species  $\text{CH}_3\text{FSbF}_5$ <sup>1d,3</sup> and  $\text{Sb}_2\text{F}_{11}^-$ .<sup>2</sup> A number of examples of adducts involving other "noncoordinating" anions, including  $\text{ClO}_4^-$ ,  $\text{BF}_4^-$ ,  $\text{PF}_6^-$ , and  $\text{AsF}_6^-$ , are known; in these cases the Lewis acid acceptors are transition metals.<sup>4</sup> We report here what we believe to be the first example of coordination of  $\text{SbF}_6^-$  to a transition metal, including an X-ray diffraction study, NMR evidence that the solid-state coordination persists in solution, and a novel fluxional process in which the  $\text{SbF}_6^-$  ligand apparently "spins" while remaining in the coordination sphere of the metal.

As part of a project whose goal is the high-yield synthesis of tungsten mononitrosyl carbonyl complexes,<sup>5</sup> we combined various



**Figure 1.** ORTEP drawing of  $\text{Me}_2\text{PhP}(\text{CO})_3(\text{NO})\text{W}(\mu\text{-F})\text{SbF}_5$  (**1a**). Selected bond distances (Å) and angles (deg): W-P, 2.518 (5); W-N, 1.795 (17); W-C(1), 2.092 (23); W-C(2), 2.089 (20); W-C(3), 2.052 (21); F(1)-W-N, 176.51 (57); P-W-C(1), 175.36 (61); C(2)-W-C(3), 176.49 (82); F(1)-W-P, 86.43 (32); F(1)-W-C(1), 89.47 (68); F(1)-W-C(2), 89.32 (62); F(1)-W-C(3), 89.08 (67).

tungsten phosphine complexes with  $\text{NO}^+\text{SbF}_6^-$ . While  $\text{Ph}_3\text{PW}(\text{CO})_5$ , as does  $\text{W}(\text{CO})_6$ ,<sup>6b</sup> gave mainly carbonyl-free tungsten dinitrosyl cations,<sup>6</sup> the use of more basic phosphine ligands such as  $\text{Me}_2\text{PhP}$  and  $\text{Me}_3\text{P}$  resulted in rapid and clean consumption of starting material via reaction with a single equivalent of  $\text{NO}^+\text{SbF}_6^-$ . The infrared spectrum of the  $\text{Me}_2\text{PhP}$  adduct **1a**, for



instance, showed bands at 2102 (m), 2012 (s), and 1690 (s)  $\text{cm}^{-1}$  due respectively to two carbonyl ligands trans to each other, another cis carbonyl, and a single nitrosyl ligand,<sup>7</sup> while the  $^1\text{H}$  NMR indicated the presence of a single bound phosphine ligand. Isolation of **1a** and **1b** as yellow crystalline compounds<sup>8</sup> allowed for more definitive characterization.<sup>9</sup> In particular, the mass spectra indicated the coordination of  $\text{SbF}_6^-$ : while no molecular ions were observed, WSb envelopes<sup>10</sup> due to  $\text{M}^+-\text{CO}$  and  $\text{M}^+-2\text{CO}$

(6) (a) Green, M.; Taylor, S. H. *J. Chem. Soc., Dalton Trans.* **1972**, 2629-2631. (b) Sen, A.; Thomas, R. *Organometallics* **1982**, *1*, 1251-1254.

(7) The tungsten dinitrosyl cations referred to in ref 6 typically exhibit nitrosyl bands at 1730-1870  $\text{cm}^{-1}$ , and at most a single carbonyl band around 2160  $\text{cm}^{-1}$ , while the mononitrosyl adducts in ref 5 give corresponding bands at 1670-1725 and 2090-2145  $\text{cm}^{-1}$ .

(8) A typical procedure is as follows: in an inert atmosphere glovebox, powdered  $\text{NO}^+\text{SbF}_6^-$  (0.58 g) was added to 1.01 g of  $\text{Me}_2\text{PhPW}(\text{CO})_5$  in 25 mL of  $\text{CH}_2\text{Cl}_2$ . The stirred solution was maintained under a partial vacuum for 1 h, after which it was concentrated to 10 mL and filtered. After addition of 25 mL of  $\text{CCl}_2\text{FCCl}_2$  and cooling to  $-40^\circ\text{C}$ , the yellow suspension was filtered and the solution concentrated until a precipitate (of **1a**) was observed, ca. 15-20 mL. Cold hexanes (25 mL) was layered on, and the mixture cooled to  $-40^\circ\text{C}$  overnight. After filtering and washing with hexanes, 0.96 g (66% yield) of bright yellow crystals of **1a** were obtained. Both **1a** and **1b** are somewhat air and temperature sensitive, as well as extremely hygroscopic.

(9) **1a**: IR ( $\text{CH}_2\text{Cl}_2$ ) 2102 (m), 2012 (s), 1690 (s)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ )  $\delta$  7.55-7.59 (m, 5H), 2.078 (d,  $J = 8.9$  Hz, 6H); MS (EI),  $m/e$  (for  $^{184}\text{W}$ ,  $^{121}\text{Sb}$ , L =  $\text{Me}_2\text{PhP}$ ) 643 ( $\text{L}(\text{CO})_2(\text{NO})\text{WFSbF}_5^+$ ), 615 ( $\text{L}(\text{CO})(\text{NO})\text{WFSbF}_5^+$ ), 436 ( $\text{L}(\text{CO})_3(\text{NO})\text{W}^+$ ), 427 ( $\text{L}(\text{CO})_2(\text{NO})\text{WF}^+$ ), 399 ( $\text{L}(\text{CO})(\text{NO})\text{WF}^+$ ), 371 ( $\text{L}(\text{NO})\text{WF}^+$ ), 341 ( $\text{LWF}^+$ ); Anal. Calcd for  $\text{C}_{11}\text{H}_{11}\text{NO}_4\text{PSbF}_6\text{W}$ : C, 19.67; H, 1.65; N, 2.09. Found: C, 19.62; H, 1.54; N, 2.04. **1b**: IR ( $\text{CH}_2\text{Cl}_2$ ) 2102 (m), 2010 (s), 1690 (s)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CD}_2\text{Cl}_2$ )  $\delta$  1.804 (d,  $J = 9.5$  Hz); MS (EI),  $m/e$  (for L =  $\text{Me}_3\text{P}$ ) 581 ( $\text{L}(\text{CO})_2(\text{NO})\text{WFSbF}_5^+$ ), 553 ( $\text{L}(\text{CO})(\text{NO})\text{WFSbF}_5^+$ ), 374 ( $\text{L}(\text{CO})_3(\text{NO})\text{W}^+$ ), 365 ( $\text{L}(\text{CO})_2(\text{NO})\text{WF}^+$ ), 337 ( $\text{L}(\text{CO})(\text{NO})\text{WF}^+$ ), 309 ( $\text{L}(\text{NO})\text{WF}^+$ ); Anal. Calcd for  $\text{C}_9\text{H}_9\text{NO}_4\text{PSbF}_6\text{W}$ : C, 11.82; H, 1.49; N, 2.30; P, 5.08; F, 18.70. Found: C, 11.58; H, 1.65; N, 2.38; P, 5.16; F, 18.47.

(10) Tungsten has four abundant isotopes, antimony two; see, e.g.: Gordon, A. J.; Ford, R. A. "The Chemist's Companion"; Wiley: New York, 1972; p 88.

(1) (a) Olah, G. A.; Baker, E. B.; Evans, J. C.; Tolgyesi, W. S.; McIntyre, J. S.; Bastien, I. J. *J. Am. Chem. Soc.* **1964**, *86*, 1360-1373. (b) Olah, G. A.; White, A. M. *Ibid.* **1969**, *91*, 5801-5810. (c) Kirchen, R. P.; Sorensen, T. S. *Ibid.* **1977**, *99*, 6687-6693. (d) Olah, G. A.; Donovan, D. J. *Ibid.* **1978**, *100*, 5163-5169. (e) Arnett, E. M.; Petro, C. *Ibid.* **1978**, *100*, 5408-5416.

(2) While the precise concentrations of the various anions depend on the reaction conditions,  $\text{SbF}_6^-$  can be a major component in  $\text{SbF}_5\text{-HSO}_3\text{F}$  ("magic acid") systems, although  $\text{Sb}_2\text{F}_{11}^-$  is the simplest anion present in media containing  $\text{SbF}_5$  as the only acid: (a) Commeyras, A.; Olah, G. A. *J. Am. Chem. Soc.* **1969**, *91*, 2929-2942. (b) Bacon, J.; Dean, P. A. W.; Gillespie, R. J. *Can. J. Chem.* **1969**, *47*, 1655-1659.

(3) Calves, J.-Y.; Gillespie, R. J. *J. Am. Chem. Soc.* **1977**, *99*, 1788-1792.

(4) (a) Mayfield, H. G.; Bull, W. E. *J. Chem. Soc. A* **1971**, 2279-2281.

(b) Mews, R. *Angew. Chem., Int. Ed. Engl.* **1975**, *14*, 640. (c) Richter, K.; Fischer, E. O.; Kreiter, C. G. *J. Organomet. Chem.* **1976**, *122*, 187-196. (d) Beck, W.; Schloter, K. *Z. Naturforsch., B* **1978**, *33B*, 1214-1222. (e) Wimmer, F. L.; Snow, M. R. *Aust. J. Chem.* **1978**, *31*, 267-278. (f) Regina, F. J.; Wojcicki, A. *Inorg. Chem.* **1980**, *19*, 3803-3807. (g) Mattson, B. M.; Graham, W. A. G. *Ibid.* **1981**, *20*, 3186-3189. (h) Raab, K.; Olgemoller, B.; Schloter, K.; Beck, W. *J. Organomet. Chem.* **1981**, *214*, 81-86. (i) Legzdins, P.; Martin, D. T. *Organometallics* **1983**, *2*, 1785-1791.

(5) Mononitrosyl tungsten carbonyl complexes are available in low overall yield (a-e) and with chelating phosphine ligands (f): (a) Barraclough, C. G.; Bowden, J. A.; Colton, R.; Commons, C. J. *Aust. J. Chem.* **1973**, *26*, 241-245. (b) Colton, R.; Commons, C. J. *Ibid.* **1973**, *26*, 1487-1492. (c) King, R. B.; Saran, M. S.; Anand, S. P. *Inorg. Chem.* **1974**, *13*, 3038-3040. (d) Isaacs, E. E.; Graham, W. A. G. *J. Organomet. Chem.* **1975**, *99*, 119-126. (e) Legzdins, P.; Malito, J. T. *Inorg. Chem.* **1975**, *14*, 1875-1878. (f) Connelly, N. G. *J. Chem. Soc., Dalton Trans.* **1973**, 2183-2188.

ions were observed, as were the corresponding  $R_3P(CO)_3WNO^+$  species.

Slow diffusion of cold hexanes into a cold  $CH_2Cl_2-CCl_2FCClF_2$  solution of **1a** gave yellow parallelepipeds suitable for a single-crystal X-ray diffraction analysis.<sup>11</sup> The structure (Figure 1) is exceptional only in that  $SbF_6^-$  is in fact coordinated; all bond lengths and angles about both tungsten and antimony are relatively normal.<sup>12</sup> The most noticeable angular distortion is in the  $SbF_6^-$  moiety, where the four equatorial fluorine atoms are bent slightly toward the bridging fluorine such that the average  $F_1-Sb-F_{eq}$  angle is  $87.3(12)^\circ$  and the  $F_6-Sb-F_{eq}$  angle  $92.7(8)^\circ$ . The bridging F-Sb bond length of  $1.954(11)$  Å is  $0.09$  Å longer than the average terminal F-Sb bond length of  $1.864(8)$  Å, while the W-F bond length of  $2.169(11)$  Å is  $0.3$  Å longer than the terminal F-Sb bond lengths. Since the tungsten single-bond covalent radius of  $1.62$  Å<sup>12a</sup> is just  $0.26$  Å longer than that of antimony,<sup>13</sup> the tungsten-fluorine interaction must be fairly strong;<sup>14</sup> surprisingly perhaps, it is as strong as (or stronger than) that seen in the other X-ray structures in which coordination of "noncoordinating" anions (specifically  $BF_4^-$  and  $CF_3SO_3^-$ ) has been demonstrated.<sup>15</sup> Only one other moiety appears to possess such a strong interaction with  $SbF_6^-$  in the solid state,<sup>16</sup> namely,  $SbF_5$  in  $Sb_2F_{11}^-$ .<sup>17</sup> The geometry of  $Sb_2F_{11}^-$  is not unlike that seen in **1a**: bridging and terminal Sb-F bond lengths average  $2.01$  and  $1.84$  Å, respectively, while the Sb-F-Sb angle is typically around  $150^\circ$ , compared to the W-F-Sb angle of  $147.15(59)^\circ$ . However, the Sb-F-Sb angle in  $Sb_2F_{11}^-$  ranges from  $146^\circ$  to  $180^\circ$  and is probably related to crystal packing.<sup>17d-f</sup>

The NMR spectrum of **1b** was examined in detail next. In the  $^{19}F$  NMR, only a weak signal, presumably broadened by efficient quadrupolar coupling<sup>18</sup> to the two antimony isotopes ( $I = 5/2, 7/2$ ), was observed. However, in the  $^{31}P$  NMR<sup>19</sup> at  $194$  K, a doublet

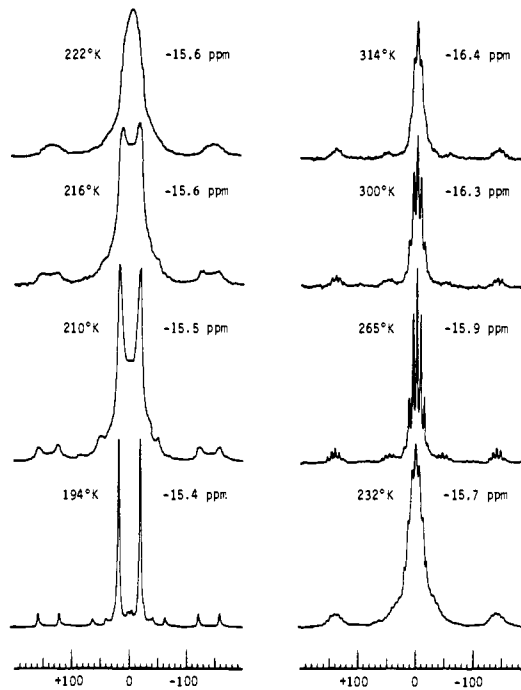


Figure 2. Variable-temperature  $^{31}P$  NMR spectra of **1b**. Scale is in hertz. Small peaks within  $\pm 70$  Hz are spinning side bands, while those centered on  $\pm 140$  Hz are  $^{183}W$  satellites.

was observed ( $-15.4$  ppm,  $J = 37.6$  Hz), due to coupling of the phosphorus to a single fluorine of the coordinated  $SbF_6^-$  (Figure 2). Coupling to  $^{183}W$  (14% natural abundance) was also observed, in the form of two satellite doublets with  $J_{PW} = 280$  Hz. As the temperature was raised, typical coalescence behavior—echoed in the tungsten satellites—was observed, with a broad, nearly featureless, signal appearing at  $222$  K. Above this temperature, the signal sharpened again, until, at  $265$  K, a clean septet was observed at  $-15.9$  ppm. The septet spacing of  $6.64$  Hz implies a phosphorus-fluorine coupling  $J_{PF} = 39.8$  Hz, in which all six fluorines are equivalently coupled to phosphorus. There is only one other report of fluorine coupling of any kind in a complex of a non-coordinating anion, namely, in *mer*-( $CH_3C$ )( $Me_3P$ )( $CO$ ) $_3Cr(\mu-F)BF_3$ , and, remarkably, the  $^{31}P$  NMR at  $-20^\circ C$  consisted of a quintet giving  $J_{PF} = 35.2$  Hz.<sup>4c</sup> Qualitatively, both of these processes must correspond to some type of rapid spinning of the anionic ligand, which nevertheless remains in the coordination sphere of the metal. In the case of **1b** further warming of the sample resulted in sharpening of the septet into a singlet, consistent with the expected rapid intermolecular  $SbF_6^-$  exchange.

Two mechanisms can be imagined for the low-temperature fluxional process in **1b**. The first would involve a concerted "hopping" mechanism in which a single exchange event would result in one of the four equatorial fluorine atoms becoming the new bridging fluorine. The second would involve complete dissociation into  $Me_3PW(CO)_3NO^+$  and  $SbF_6^-$  ions, which remain bound, however, within a solvent cage. The theoretical NMR line shapes<sup>20</sup> predicted by the two mechanisms were found to be virtually identical. The ionic exchange rates were greater than the concerted rates as expected, since an exchange event in the ionic mechanism has a one in six chance of not giving a new fluorine atom in the bridging position. However, both sets of rates gave linear plots of  $\ln(k/T)$  vs.  $1/T$ , which were sufficiently close to give essentially identical activation parameters!<sup>21</sup> Although the two mechanisms cannot be distinguished on the basis of the available data, the ionic mechanism might be favored on the

(11) Crystals of **1a** are monoclinic, space group  $P2_1/n$ ,  $a = 8.838(3)$  Å,  $b = 8.267(2)$  Å,  $c = 25.004(7)$  Å,  $\beta = 94.71(1)^\circ$ ,  $V = 1827.33$  Å<sup>3</sup>,  $Z = 4$ ; data were collected at  $-140^\circ C$ . The structure was solved by means of a Patterson map; an absorption correction ( $\mu = 81.01$  cm<sup>-1</sup>) was applied, and the structure refined steadily to  $R = 0.056$  and  $R_w = 0.070$  for the 1735 independent reflections with  $F_o > 3\sigma(F_o)$ . Only the positions of the three heaviest atoms were refined by using anisotropic thermal parameters, since application of anisotropic thermal parameters to the other atoms did not give meaningful reductions in the residuals.

(12) For some typical tungsten (a-c) and antimony (d-f) bond lengths, see: (a) Churchill, M. R. *Perspect. Struct. Chem.* **1970**, *3*, 91-164. (b) Love, R. A.; Chin, H. B.; Koetzle, T. F.; Kirtley, S. W.; Whittlesey, B. R.; Bau, R. *J. Am. Chem. Soc.* **1976**, *98*, 4491-4498. (c) Hidai, M.; Mizobe, Y.; Sato, M.; Kodama, T.; Uchida, Y. *Ibid.* **1978**, *100*, 5740-5748. (d) Burns, J. H. *Acta Crystallogr.* **1962**, *15*, 1098-1101. (e) Kruger, G. J.; Pistorius, C. W. F. T.; Heyns, A. M. *Acta Crystallogr., Sect. B* **1976**, *B32*, 2916-2918. (f) English, R. B.; Nassimbeni, L. R.; Haines, R. J. *J. Organomet. Chem.* **1977**, *135*, 351-360.

(13) Alcock, N. W. *Adv. Inorg. Chem. Radiochem.* **1972**, *15*, 1-58.

(14) A d<sup>0</sup> tungsten-fluorine nonbridging bond length of  $2.019(14)$  Å has been reported,<sup>12c</sup> as have d<sup>0</sup> nonbridging and bridging bond lengths of  $1.79(2)$  and  $2.18(3)$  Å. A compound related to **1a**,  $F_3Re(\mu-F)Re(CO)_5$ , has a bridging d<sup>0</sup> rhenium-fluorine bond length of  $2.17(4)$  Å, to which must be added about  $0.1$  Å in order to compare to tungsten.<sup>12a</sup> See: (a) Bruce, D. M.; Holloway, J. H.; Russell, D. R. *J. Chem. Soc., Chem. Commun.* **1973**, 321-322. (b) Tucker, P. A.; Taylor, P. A.; Holloway, J. H.; Russell, D. R. *Acta Crystallogr., Sect. B* **1975**, *B31*, 906-908.

(15) (a) Tomlinson, A. A. G.; Bonamico, M.; Dessy, G.; Fares, V.; Scaramuzza, L. *J. Chem. Soc., Dalton Trans.* **1972**, 1671-1674. (b) Gaughan, A. P., Jr.; Dori, Z.; Ibers, J. A. *Inorg. Chem.* **1974**, *13*, 1657-1667. (c) Humphrey, M. B.; Lamanna, W. M.; Brookhart, M.; Husk, G. R. *Ibid.* **1983**, *22*, 3355-3358. (d) Hitchcock, P. B.; Lappert, M. F.; Taylor, R. G. *J. Chem. Soc., Chem. Commun.* **1984**, 1082-1084.

(16) For some weak interactions of cations with  $SbF_6^-$ , see: (a) Boldrini, P.; Gillespie, R. J.; Ireland, P. R.; Schrobilgen, G. *J. Inorg. Chem.* **1974**, *13*, 1690-1694. (b) Passmore, J.; Richardson, E. K.; Whidden, T. K.; White, P. S. *Can. J. Chem.* **1980**, *58*, 851-857. (c) Birchall, T.; Myers, R. D. *Inorg. Chem.* **1981**, *20*, 2207-2211. (d) Passmore, J.; Sutherland, G.; Taylor, P.; Whidden, T. K.; White, P. S. *Ibid.* **1981**, *20*, 3839-3845. (e) Ardon, M.; Bino, A.; Cotton, F. A.; Dori, Z.; Kaftory, M.; Kolthammer, B. W. S.; Kapon, M.; Reisner, G. *Ibid.* **1981**, *20*, 4083-4090.

(17) (a) McRae, V. M.; Peacock, R. D.; Russell, D. R. *J. Chem. Soc., Chem. Commun.* **1969**, 62-63. (b) Lind, M. D.; Christe, K. O. *Inorg. Chem.* **1972**, *11*, 608-612. (c) McKee, D. E.; Zalkin, A.; Bartlett, N. *Ibid.* **1973**, *12*, 1713-1717. (d) Edwards, A. J.; Taylor, P. *J. Chem. Soc., Dalton Trans.* **1973**, 2150-2153. (e) Edwards, A. J.; Sills, R. J. *Ibid.* **1974**, 1726-1729. (f) Gillespie, R. J.; Riddell, F. G.; Slim, D. R. *J. Am. Chem. Soc.* **1976**, *98*, 8069-8072.

(18) Bacon, J.; Gillespie, R. J. *J. Am. Chem. Soc.* **1971**, *93*, 6914-6919.

(19)  $^{31}P$  NMR spectra were measured at  $81$  MHz in  $CD_2Cl_2$  and referenced to external  $PMe_3$  in  $CD_2Cl_2$ , which was fixed at its room temperature value of  $61.52$  ppm upfield of  $H_3PO_4$  (which freezes below  $-50^\circ C$ ).

(20) Line-shape analyses were carried out by using a program written by F. A. L. Anet and modified by S. S. Muira.

(21) Activation parameters (for the ionic mechanism) are  $\Delta H^\ddagger = 8.9 \pm 0.2$  kcal/mol,  $\Delta S^\ddagger = -6.4 \pm 0.8$  eu, and  $\Delta G^\ddagger(230 K) = 10.4 \pm 0.3$  kcal/mol.

grounds that it leads in a natural way to the intermolecular exchange observed at higher temperatures.

The chemistry of these new compounds as well as some related materials<sup>22</sup> is under active investigation. Preliminary results indicate that they possess unusual coordination preferences (SbF<sub>6</sub><sup>-</sup> over ethanol, for instance), that they regioselectively catalyze cyclopentadiene polymerization in high yield,<sup>23</sup> and that they react with metal anions resulting in both dimer formation and nitrosyl transfer. These results will be reported in due course.

(22) For instance, use of the weaker donor ligand acetonitrile (AN) gives analogous chemistry when ANW(CO)<sub>5</sub> is combined with NO<sup>+</sup>BF<sub>4</sub><sup>-</sup>, although some disproportionation to give *mer,cis*-AN<sub>2</sub>(CO)<sub>3</sub>W(NO)<sup>+</sup>BF<sub>4</sub><sup>-</sup> occurs. With NO<sup>+</sup>SbF<sub>6</sub><sup>-</sup>, only disproportionation is observed, so acetonitrile apparently is not a strong enough donor to stabilize the W(μ-F)SbF<sub>5</sub> interaction.

(23) Farahi, J.; Hersh, W. H., unpublished results.

**Acknowledgment.** W.H.H. thanks Andrew Fortney for preparing a large pure sample of **1a**, Professor C. E. Strouse and Dr. C. B. Knobler for assistance in carrying out the X-ray structure determination, and Professor F. A. L. Anet both for suggesting the ionic mechanism and for assistance in carrying out the line-shape analysis. Financial support from the donors of the Petroleum Research Fund, administered by the American Chemical Society, Chevron Research Co., and the UCLA Committee on Research are gratefully acknowledged.

**Supplementary Material Available:** Tables of crystallographic data for **1a** including details of the solution, positional parameters, temperature factors, bond lengths and angles, least-squares planes, and observed and calculated structure factors and theoretical NMR line shapes and rates for **1b** (25 pages). Ordering information is given on any current masthead page.

## Additions and Corrections

**Mechanism of the Reaction of Gas-Phase Iron Ions with Neutral Olefins** [*J. Am. Chem. Soc.* **1984**, *106*, 4307–4316]. D. A. PEAKE, M. L. GROSS,\* and D. P. RIDGE\*

Page 4314: In the paragraph titled Study of Products from Decomposition of FeC<sub>8</sub>H<sub>16</sub><sup>+</sup>, we refer to the product from reaction 17 as FeC<sub>6</sub>H<sub>12</sub><sup>+</sup>, a 1-hexene complex. The Fe(1-octene)<sup>+</sup> gives a FeC<sub>5</sub>H<sub>10</sub><sup>+</sup> fragment which is identical with Fe(1-pentene)<sup>+</sup>. We apologize for any confusion this may have caused.

**Nucleophilic Reactions in Solutions of Nonmicellized Hydrophobic Ammonium Ions** [*J. Am. Chem. Soc.* **1984**, *106*, 7178]. GIRMA BIRESAW, CLIFFORD A. BUNTON,\* CLIFFORD QUAN, and ZHONG-YUN YANG

Page 7182: Table VI footnotes are as follows. <sup>a</sup>Values are 10<sup>3</sup>kΨ, s<sup>-1</sup> at 25.0 °C. <sup>b</sup>In H<sub>2</sub>O:MeCN 70:30 (v/v), 0.01 M NaOH and 0.1 M **1e**. <sup>c</sup>In 2.2 vol % MeCN, 10<sup>-3</sup> M NaOH, 6 × 10<sup>-4</sup> M **1e** and 6 × 10<sup>-3</sup> M **1c**.

**Gas Phase <sup>1</sup>H NMR Spectra and Nitrogen Inversion of Trimethylene Imine** [*J. Am. Chem. Soc.* **1984**, *106*, 7638–7639]. B. RI. FRIEDMAN, J. PAUL CHAUVEL, JR., and NANCY S. TRUE\*

The temperature dependent exchange broadened <sup>1</sup>H NMR spectra reported in this communication result from the *cis* = *trans* nitrogen inversion process for α-methylaziridine. They were incorrectly attributed to nitrogen inversion in the four-membered-ring azetidone. The authors thank F. A. L. Anet of UCLA for bringing this fact to their attention as well as for providing the right assignment of the spectra.