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give even-electron reduction products; behavior similar to that we have observed by $(HP_2Mo_{18}^{6-})$ may be exhibited by three- and five-electron blues. In contrast, the corresponding nonprotonated reduced tungstates do not undergo disproportionation. The difference in the disproportionation reactivity between $(HP_2Mo_{18}^{6-})$ and $(P_2W_{18}^{7-})$ can be attributed to the effect of protonation which diminishes the negative charge on the anions reducing their mutual repulsion.

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Registry No. $P_2Mo_{18}^{6-}$, 57609-49-1; $P_2W_{18}^{6-}$, 12269-70-4; $HP_2Mo_{18}^{6-}$, 80612-23-3; $P_2W_{18}^{7-}$, 12504-08-4.

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An Alternate Preparation of (Trifluoromethanesulfonato)pentaamminecobalt(III) and Some New Applications

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Traditionally, the preparation of pentaamminecobalt(III) complexes has required reaction times of several hours at elevated temperatures (60–90 °C) because of the kinetic stability of the common cobalt(III) reactants. This precludes preparations where the ligand is thermally unstable itself or subject to oxidation by cobalt(III). However, if a good leaving group is in the coordination sphere of the cobalt(III), milder conditions can be used. This is the basis of the methods involving nitrosation of coordinated azide,[1] and alkylation of coordinated acetate,[2] both in poorly coordinating solvents. Most recently this principle has been exploited by Dixon et al.[3] using $CF_3SO_3^-$ as a leaving group after preparing $[(NH_3)_5CoO_3SCF_3](CF_3SO_3)_2$ (I) by dehydrochlorination of $[(NH_3)_5CoCl]Cl_2$ in anhydrous CF_3SO_3H.

The present report gives an alternative preparation of I which is more economical in the essentially quantitative use of CF_3SO_3H. The convenience of the preparations with I have been demonstrated[3] for a number of compounds previously made by other methods. This advantage is shown further here by the preparation of several new compounds, two of which could not be made previously, namely, the fumaronitrile[4] and *p*-nitrophenoxide[5] complexes. In addition, examples are given of preparations done in the presence of 2,6-dimethylpyridine, which is a useful additive when ligand substitution liberates a proton.

Experimental Section

Trifluoromethanesulfonic acid (Aldrich Chemical Co.) was used as supplied and handled by syringe techniques. Reagent grade acetone and sulfolane (tetramethylene sulfone or 1,1-dioxothiolane) were stored over freshly heated (120 °C) Davison 4A molecular sieves and were transferred by syringe or pipet with reasonable care to minimize exposure to atmospheric moisture. Other materials were of reagent grade and were used as supplied unless otherwise indicated. Preparations in acetone or sulfolane were done in Erlenmeyer flasks closed with rubber serum caps.

Electronic spectra were obtained on a Cary 219 spectrophotometer and NMR spectra on a Varian A56/60 spectrometer. Infrared spectra were recorded on a Nicolet FT-7000 spectrophotometer in KBr pellets and as Nujol mulls.

Aquopentaamminecobalt(III) Trifluoromethanesulfonate. To a well-stirred solution of 150 mL of concentrated aqueous ammonia and 20 mL of water were added 33 g of ammonium sulfate and 60 g of cobalt(II) sulfate heptahydrate. Then 50 mL of 30% hydrogen peroxide was added slowly while the solution was warmed on a steam bath. Heating was continued for 2 h, and then the solution was cooled and neutralized with 50% sulfuric acid. The solution was refrigerated overnight, and the red crystalline product was collected by filtration. The crude product was dissolved in 400 mL of warm water and 40 mL of concentrated ammonia. The solution was filtered and neutralized with concentrated sulfuric acid. Neutralization is indicated by the color change from reddish purple $[(NH_3)_5CoOH^{2+}]$ to reddish orange $[(NH_3)_5CoOH_2^{3+}]$. After the solution cooled at ice temperature for several hours, the crystalline product was collected by filtration, washed with ice cold water and methanol, and air-dried to yield 40 g of product. This product has not been characterized fully, but sulfate analysis, as $BaSO_4$, and the molar extinction coefficient at 492 nm[6] are consistent with the formula $[(NH_3)_5CoOH_2](SO_4)(HSO_4)$.

The sulfate salt was converted to the trifluoromethanesulfonate salt by treatment with the stoichiometric amount of barium trifluoromethanesulfonate. The latter was prepared by slowly adding trifluoromethanesulfonic acid to an aqueous slurry of the required amount of barium carbonate until all of the carbonate dissolved. The sulfate salt (40 g) was dissolved in 400 mL of warm water (~60 °C), and the barium trifluoromethanesulfonate solution was added slowly with stirring. The mixture was digested at 60–70 °C for about 1 h, and then cooled to room temperature. The $BaSO_4$ was collected by filtration on a 0.22 μm Millipore filter. The filtrate was evaporated to dryness on a vacuum line while collecting the water in a large volume trap cooled in liquid nitrogen. Any excess CF_3SO_3H will be removed at this stage, and any excess $Ba(CF_3SO_3)_2$ does not affect subsequent preparations.

$[(NH_3)_5CoO_3SCF_3](CF_3SO_3)_2$. Aquopentaamminecobalt(III) trifluoromethanesulfonate loses water when heated at 100–110 °C in vacuo to yield (trifluoromethanesulfonato)pentaamminecobalt(III) trifluoromethanesulfonate (I). The dehydration step has been carried out on 5–10 g samples either in a vacuum drying tube or in a tube furnace. The dehydration seems to require 2–3 h but routinely has been done overnight to ensure completion. The product has a pinkish purple color and is reasonably stable toward hydration in air, although normally it has been stored in a dessicator over calcium sulfate.

$[(NH_3)_5Co(\text{fumaronitrile})](ClO_4)_3$. A solution of I (4 g) in sulfolane (20 mL) with CF_3SO_3H (2 drops) and fumaronitrile (1.1 g) was allowed to react at room temperature for 48 h. Then the solution was added with rapid stirring to a mixture of 100 mL of 2-butanol and 500 mL of ether. The resulting solid was collected by filtration, dissolved in 30 mL of 0.1 M HCl, and precipitated by addition of 70% perchloric acid. The product was recrystallized from 0.1 M perchloric acid and then subjected to chromatography on Dowex 50 W-X2 in the H^+ form. The product was separated from impurities by elution with 2 M NaCl (pH 4.5) and removed from the resin in a batch method with 6 M HCl. Concentrated perchloric acid was added to the HCl solution to precipitate the perchlorate salt. This

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(1) Jordan, R. B.; Sargeson, A. M.; Taube, H. *Inorg. Chem.* **1966**, *5*, 1091.

(2) Jackman, L. M.; Dormish, J. F.; Scott, R. M.; Portman, R. H.; Minard, R. D. *Inorg. Chem.* **1979**, *18*, 1503.

(3) Dixon, N. E.; Jackson, W. G.; Lancaster, M. J.; Lawrance, G. A. Sargeson, A. M. *Inorg. Chem.* **1981**, *20*, 470.

(4) Hua, L., H.-C.; Balahura, R. J.; Fanchiang, Y.-T.; Gould, E. S. *Inorg. Chem.* **1978**, *17*, 3692.

(5) Reid, R.; Fan, F.-R., F.; Fanchiang, Y.-T.; Gould, E. S. *Inorg. Chem.* **1978**, *17*, 2766.

(6) The molar extinction coefficient of $(NH_3)_5CoOH_2^{3+}$ is 47.7 M^{-1} cm^{-1} at 492 nm: Jackson, W. G.; Lawrance, G. A.; Sargeson, A. M. *Inorg. Chem.* **1980**, *19*, 1001.

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Table I. Electronic and Proton NMR Spectra Properties of Pentaamminecobalt(III) Products

ligand	electronic spectrum λ_{max}, nm (ϵ, M^{-1} cm^{-1})	proton NMR, τ[a]		
		trans NH_3	cis NH_3	other
$NC(CH)_2CN$[b]	467 (74.3)[c]	6.56	6.22	3.15–2.48
$NC(CH_2)_2CN$[d]	467 (60.7), 333 (54.4)[c]	6.63	6.23	7.16–6.73
$NCCH_2CN$	467 (62.9), 334 (63.6)[c]	6.50	6.12	5.1–5.2 (br)
$O_2NC_6H_4O^-$	530 (474), 370 (1.50×10^4)	7.30	6.28	3.40–1.94
$H_3CO_2CC_6H_4O^-$	530 (396), 342 (8.00×10^3)[e]	7.33	6.43	6.23 (CH_3), 3.38–2.25 (C_6H_4)
$CH_3C_6H_4SO_2NH^-$[f]	505 (88.0), 290 (1.74×10^3)	6.97	6.67	7.97 (NH), 7.64 (CH_3) 2.72–2.27 (C_6H_4)
C_3H_3NO[g]	469 (60.5), 333 (73.7)[c]	6.45	6.18	2.85, 0.83, 0.55

[a] Shifts relative to residual solvent protons at τ 7.48 in Me_2SO-d_6 with 1–2 drops of H_2SO_4 for the nitrile complexes. [b] Fumaronitrile. [c] Recorded in 0.50 M $HClO_4$. [d] See ref 8 for further details. [e] The ultraviolet spectrum has maxima at 289 nm (8.39×10^3) and 259 nm (1.08×10^4). [f] See ref 7 for further details. [g] Isoxazole.

was recrystallized from dilute perchloric acid, washed with ethanol and ether, and air-dried.

Anal. Calcd for $[(NH_3)_5CoNC(CH)_2CN](ClO_4)_3$: C, 9.23; H, 3.29; N, 18.84. Found: C, 9.21; H, 3.21; N, 18.67. The electronic and ^1H NMR spectra are given in Table I. The C≡N stretches are observed in the infrared spectrum at 2302 (s) and 2240 cm^{-1} (w).

Subsequent work indicates that the ion-exchange step may be replaced by several recrystallizations from 0.1 M perchloric acid and that the preparation may be done in acetone and without the addition of CF_3SO_3H.

$[(NH_3)_5Co$(*p*-nitrophenoxide)$](ClO_4)_2$. Sodium *p*-nitrophenoxide dihydrate (1.0 g) and 3A molecular sieves were stirred in sulfolane at 60–65 °C for 2 h. The solution was cooled for 1 h and then I (3.0 g) was added with occasional stirring and the mixture was allowed to stand overnight. The deep yellow-purple solution was filtered and slowly added to a rapidly stirred solution of 100 mL of 2-butanol in 500 mL of ether. The organic solvent was decanted from the purple oil, and the oil was washed three times with ether and air-dried. The viscous oil was dissolved in water and subjected to cation-exchange chromatography on CGC-271 (Na^+) resin. The red-purple band of the desired complex moved slowly on the column with 0.3 M NaCl as eluent and moved quickly with 0.4 M NaCl. The band of resin containing the product was separated physically and washed with water and then with warm (35–40 °C) 6 M $NaClO_4$ to remove the product. Purple-brown crystals formed after storage of the 6 M $NaClO_4$ solution in a freezer overnight. The product was washed with cold ethanol and ether, air-dried, and stored in a dessicator.

Anal. Calcd for $[(NH_3)_5CoOC_6H_4NO_2](ClO_4)_2$: C, 14.98; H, 3.98; N, 17.47. Found: C, 14.70; H, 3.81; N, 17.19. The electronic and ^1H NMR spectra are given in Table I. The infrared spectrum has absorptions in the aromatic region at 1590 (s) and 1490 (s) cm^{-1}. The corresponding absorptions for the sodium salt of the free ligand appear at 1590 (s) and 1487 (s) cm^{-1}.

$[(NH_3)_5Co$(methyl *p*-oxobenzoate)$](ClO_4)_2 \cdot 3NaClO_4$. A solution of I (3.0 g) in sulfolane (25 mL) with 2,6-dimethylpyridine (0.80 g) and methyl *p*-hydroxybenzoate (2.0 g) was allowed to react for 24 h. The deep purple solution was filtered and added to 100 mL of 2-butanol in 500 mL of ether. The oil was washed with ether, air-dried, dissolved in water, and ion exchanged on CGC-271 (Na^+) weak acid resin. The complex was recovered as described for the *p*-nitrophenoxide complex. The perchlorate salt was washed with ether, air-dried, then dissolved in methanol, and filtered and the solution evaporated to dryness. The solid was dissolved in a minimum of acetone (~4 mL), and the solution was filtered into 60 mL of ether. The brown precipitate was collected, washed with ether, air-dried, and stored in a dessicator. Anal. Calcd for $[(NH_3)_5CoOC_6H_4CO_2CH_3](ClO_4)_2 \cdot 3NaClO_4$: C, 11.15; H, 2.57; N, 8.13. Found: C, 11.08; H, 2.55; N, 7.83. So that the elemental analyses could be verified, a weighed sample of the complex was dissolved in 0.50 M $HClO_4$ and allowed to hydrolyze to $(NH_3)_5CoOH_2^{3+}$. The known molar extinction coefficient of the product at 492 nm[6] was used to calculate the molecular weight of the sample as 887, in good agreement with the theoretical value of 861.5.

The electronic and ^1H NMR spectra are given in Table I. The C═O stretch for the complex appears at 1680 (sh) as compared to 1687 (s) cm^{-1} for the corresponding free ligand.

$[(NH_3)_5Co$(malononitrile)$](ClO_4)_3$. Freshly vacuum distilled malononitrile (2.0 g) was dissolved in sulfolane (10 mL) and allowed to stand over molecular sieves for 4 h. The molecular sieves were removed by filtration and sulfolane (20 mL), I (2.8 g), and CF_3SO_3H (0.4 g) were added to the filtrate with stirring. After 18 h at room temperature, the bright orange solution was added dropwise to 300 mL of rapidly stirred ether. The resulting oil was washed three times with ether by decantation and crystallized from aqueous perchloric acid. The latter step may be unnecessary. The product was dissolved in 0.1 M HBr and filtered, solid LiBr (10 g) was added to the filtrate which was cooled, and a pink impurity was removed by filtration. This filtrate was treated with solid $NaClO_4$ and, after being cooled, yielded a yellow precipitate which was collected by filtration. The treatment with HBr, LiBr, and $NaClO_4$ was repeated. The product was recrystallized from dilute perchloric acid, washed with ethanol and ether, and air-dried. Anal. Calcd for $[(NH_3)_5CoNCCH_2CN](ClO_4)_3$: C, 7.08; H, 3.37; N, 19.28. Found: C, 7.22; H, 3.52; N, 19.25.

The electronic and ^1H NMR spectra are described in Table I. The infrared spectrum has transitions due to the C≡N stretch at 2341 (s) and 2272 (w) cm^{-1}.

$[(NH_3)_5Co$(*p*-toluenesulfonamide)$](ClO_4)_2$. A solution of *p*-toluenesulfonamide (2 g) and 2,6-dimethylpyridine (1 mL) in tetramethylene sulfone was allowed to stand over molecular sieves for 1 h, and then I (3 g) was added. After 4 h at 40 °C, the mixture was filtered into a solution of 60 mL of 2-butanol in 350 mL of ether. The organic solvent was decanted from the resulting oil, and the oil was dried under vacuum (water aspirator). The resulting powder was dissolved in a minimum of methanol, and an equal volume of a concentrated solution of sodium perchlorate in methanol was added. The solution was cooled to −5 °C in the freezing compartment of a refrigerator. The solid was collected by filtration and recrystallized from the minimum volume of methanol.

The product was characterized by comparison of the electronic and ^1H NMR spectra (Table I) to a sample prepared previously, purified by ion-exchange chromatography, and fully characterized.[7]

$[(NH_3)_5Co$(isoxazole)$](ClO_4)_3$. A solution of I (4 g) in sulfolane (20 mL) with isoxazole (1.1 g) was allowed to react at room temperature for 15 h. The orange solution was added to 75 mL of 2-butanol in 400 mL of ether. The resulting solid was dissolved in 35 mL of water and precipitated as the perchlorate by addition of concentrated perchloric acid. This product was collected by filtration and redissolved in a minimum of water (~20 mL). Then 5 mL of concentrated perchloric acid was added and the mixture was cooled quickly to precipitate any $[(NH_3)_5CoOH_2](ClO_4)_3$, which was removed by filtration. The filtrate was treated with 5 mL of concentrated perchloric acid and cooled to 0 °C. The product was collected by filtration and the perchloric acid treatment repeated to yield yellow plates of the final product which were washed with ether and air-dried. Anal. Calcd for $[(NH_3)_5CoC_3H_3NO](ClO_4)_3$: C, 7.04; H, 3.55; N, 16.43. Found: C, 6.77; H, 3.51; N, 16.57.

The electronic and ^1H NMR spectra are given in Table I. The infrared spectrum has an absorption characteristic of ring stretching at 1565 (m) cm^{-1}. The corresponding vibration for the free ligand occurs at 1558 (m) cm^{-1}.

Results and Discussion

The preparation of $[(NH_3)_5CoOH_2](CF_3SO_3)_3$ described here has the advantages that it uses readily available and

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(7) Laird, J. L.; Jordan, R. B. *Inorg. Chem.* **1982**, *21*, 855.

(8) Kupferschmidt, W. C.; Jordan, R. B. *Inorg. Chem.* **1981**, *20*, 3469

inexpensive reagents and makes efficient use of the trifluoromethanesulfonic acid. The product may contain some $Ba(CF_3SO_3)_2$, but this has never been a problem in any of the preparative applications tried. Our experience has been that attempts to prepare $[(NH_3)_5CoOH_2](CF_3SO_3)_3$ by crystallization from a solution of CF_3SO_3H and another salt of $(NH_3)_5CoOH_2^{3+}$ lead to mixed-anion salts which must be recrystallized several times from aqueous CF_3SO_3H. This can consume large amounts of the acid and cobalt(III) before a pure product is obtained.

The benefits of using $[(NH_3)_5CoO_3SCF_3](CF_3SO_3)_2$ (I) as a starting material have been extolled already[3] and are demonstrated further here by the preparation of two previously unattainable products,[4,5] the fumaronitrile and *p*-nitrophenoxide complexes. Only the 2,4- and 2,5-dinitrophenoxide complexes could be prepared previously, but now it seems that a range of phenoxide complexes can be made.

The observation that 2,6-dimethylpyridine can act as a noncoordinating base in the presence of I increases the scope and convenience of the preparation. This feature is illustrated here in the preparations with methyl *p*-hydroxybenzoate and *p*-toluenesulfonamide. In both cases the ligand loses a proton on coordination to $(NH_3)_5Co^{3+}$ but the proton reacts with 2,6-dimethylpyridine to allow the reaction to go to completion.

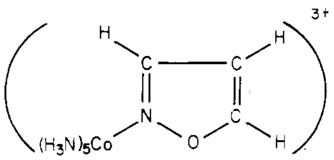
The sensitivity of phenoxide complexes to hydrolysis in aqueous acid has been mentioned by Gould et al.[5] The hydrolyses of the *p*-nitrophenoxide and methyl *p*-oxobenzoate complexes have been studied kinetically, and the reactions were both first order in $[H^+]$ with rate constants of 3.77×10^{-3} and 9.16×10^{-3} M^{-1} s^{-1}, respectively (0.05–0.500 M H^+, in 0.5 M $LiClO_4/HClO_4$, 25 °C). The smaller rate constant with the more electron-withdrawing *p*-nitro substituent is consistent with the reaction scheme

$$H^+ + (NH_3)_5CoOC_6H_4X^{2+} \underset{K_a}{\rightleftharpoons}$$

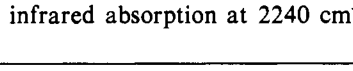
$$(NH_3)_5CoOHC_6H_4X^{3+} \xrightarrow{k_1} (NH_3)CoOH_2^{3+} + HOC_6H_4X$$

Then the measured rate constant is (k_1/K_a) and the larger K_a for *p*-nitrophenoxide would make (k_1/K_a) smaller. There was no indication of saturation in the kinetic data, which indicates that $K_a \gg [H^+]$ at least for $[H^+]$ up to 0.50 M.

The isoxazole complex is an example of a preparation of a new complex with a base sensitive ligand. Isoxazole undergoes ring opening to $NCCH_2CHO$.[9] The NMR and electronic spectra are typical of nitrogen coordination and are consistent with the structure



The isoxazole complex is very susceptible to ring opening in aqueous sodium hydroxide. Stopped-flow studies have shown that the reaction is first order in hydroxide ion with a rate constant of 467 M^{-1} s^{-1} (1.0×10^{-2}–1×10^{-3} M OH^- in 0.50 M $NaClO_4$, 25 °C). The reaction product is strongly colored and has absorption maxima at 500 and 346 nm with molar extinction coefficients of 210 and 1.00×10^3 M^{-1} cm^{-1}, respectively. The structure of the product is tentatively assigned as



$$[(NH_3)_5CoN{\equiv}CCH{=}C(O^-)H]^{2+}$$

based on the infrared absorption at 2240 cm^{-1} and known isoxazole reactions.[9] The chemistry of this system is currently under investigation.

The malononitrile complex originally reported[1] seems to have contained some impurity since the electronic spectrum does not agree with that reported here or by Creaser et al.[10] In fact, our results (Table I) even differ from those of Creaser et al.,[10] who found maximum molar extinction coefficients of 72 and 82 at 474 and 341 nm, respectively. Our experience with a number of these nitrile complexes,[8] of which succinonitrile and fumaronitrile (Table I) are typical, has always given maxima at 467–468 and 333–335 nm. The presence of carboxamido or aquo complex impurities will shift the maxima to longer wavelength.

The malononitrile complex was reported originally[1] to react rapidly with chromium(II). The complex described in the present work does not react unusually rapidly with chromium(II). The reaction rate seems to be rather typical of other nitrile complexes previously reported.[8] However, the reduction is competitive with hydrolysis to the carboxamide and aquo complexes, and no detailed studies have been undertaken.

Finally, to return to the preparative aspect of this work, $[(NH_3)_5CoOH_2](CF_3SO_3)_3$ may be dehydrated by heating it in sulfolane in the presence of molecular sieves. This appears to be a convenient in situ method of generating the reactive sulfolane or trifluoromethanesulfonato complex.

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Registry No. I, 75522-50-8; $[(NH_3)_5CoOH_2](O_3SCF_3)_3$, 69897-22-9; $[(NH_3)_5CoOH_2](SO_4)(HSO_4)$, 80679-80-7; $[(NH_3)_5Co(NC(CH)_2CN)](ClO_4)_3$, 80679-82-9; $[(NH_3)_5Co(OC_6H_4NO_2)](ClO_4)_2$, 80679-84-1; $[(NH_3)_5Co(OC_6H_4CO_2CH_3)](ClO_4)_2{\cdot}3NaClO_4$, 80679-86-3; $[(NH_3)_5Co(NCCH_2CN)](ClO_4)_3$, 15649-18-0; $[(NH_3)_5Co(NHSO_2C_6H_4CH_3)](ClO_4)_2$, 78891-47-1; $[(NH_3)_5Co(ONC_3H_3)](ClO_4)_3$, 80679-88-5.

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(9) Wakefield, B. J.; Wright, D. J. *Adv. Heterocyclic Chem.* **1979**, *25*, 148.

(10) Creaser, I. I.; MacB. Harrowfield, J.; Keene, F. R.; Sargeson, A. M. *J. Am. Chem. Soc.* **1981**, *103*, 3559.

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Perfluoroammonium Salts of Metal Heptafluoride Anions

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Due to its high-energy content and unusual kinetic stability, the NF_4^+ cation is a unique oxidizer. Its salts have found numerous applications such as solid propellant NF_3–F_2 gas generators for chemical HF–DF lasers,[1] ingredients in high detonation pressure explosives,[2] and fluorinating agents for aromatic compounds.[3] Although the NF_4^+ cation has successfully been combined with a large number of different anions in the form of stable salts, all these anions were derived from relatively strong Lewis acids, and their number of ligands did not exceed six. It was therefore of interest to explore whether NF_4^+ salts containing metal heptafluoride anions can exist.

Experimental Section

Apparatus. Volatile materials used in this work were handled in a stainless-steel–Teflon FEP vacuum line. The line and other hardware

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(1) Christe, K. O.; Wilson, R. D.; Schack, C. J. *Inorg. Chem.* **1977**, *16*, 937.

(2) Christe, K. O. U.S. Patent 4 207 124, 1980.

(3) Schack, C. J.; Christe, K. O. *J. Fluorine Chem.* **1981**, *18*, 363.

0020-1669/82/1321-2091$01.25/0