diate compounds, $NaF\cdot YF_3$ and $5NaF\cdot 9YF_3$; one is an unidentified metastable compound of probable formula $5NaF\cdot 3YF_3$; the other three include NaF and two forms of YF_3. Yttrium trifluoride crystallizes from the molten phase in an unknown crystal form which inverts on cooling below 1052° to the orthorhombic form described by Zalkin and Templeton.[7] Lattice parameters and refractive indices of the cubic $NaF\cdot YF_3$–$5NaF\cdot 9YF_3$ solid solution crystals are given in Fig. 2. Inversion of cubic $NaF\cdot YF_3$ crystals results in the formation of uniaxial (+) hexagonal crystals having refractive indices N_ω 1.464, N_ϵ 1.486, with $a = 5.95 \pm 0.03$ Å. and $c = 3.52 \pm 0.03$ Å.[15] The calculated density of these crystals with 1.5 formula weights per unit cell is 4.22 g./cc., as compared with a measured value of 4.2 g./cc. Because of the similarity of stoichiometry and unit-cell dimensions, the hexagonal phase was deduced to be isostructural with β_2-Na_2ThF_6. Although the powder pattern of the ordered form of $5NaF\cdot 9YF_3$ can be indexed assuming a primitive cubic cell with $a = 5.530$ Å., a larger cell is required by stoichiometry; moreover, a noncubic symmetry is indicated by optical data since the crystals are uniaxial (−), with N_α 1.465, N_ω 1.478. An explanation consistent with these observations is that the unit cell is tetragonal with the unique axis being some integral multiple of the pseudo-cubic cell edge, but single-crystal data are needed to test this hypothesis. Evidence that the low-temperature form of $5NaF\cdot 9YF_3$ exists over a small but finite composition range was observed in the powder diffraction patterns at 63.5 and 64 mole % YF_3; through the temperature interval 710 to 537°, the intensities of the superlattice lines were reduced in proportion to the extent that the composition $5NaF\cdot 9YF_3$ deviated from the stoichiometric ratio. The primitive lattice reported by Nowacki[5] is very similar to that of the ordered form of $5NaF\cdot 9YF_3$ and, though slightly larger in apparent cell size, is not deducible as identical. The lattice parameter assigned to the "primitive cubic YF_3" observed by Nowacki was 5.655 ± 0.003 Å. Although Zalkin and Templeton inferred that the primitive cubic phase was $NaF\cdot 3YF_3$,[7] no conclusions can be drawn with respect to the relationship of this phase to the ordered form of $5NaF\cdot 9YF_3$ because crystals observed in our study have never exhibited so large a unit-cell parameter and because quantitative intensity data are not available for comparison.

Acknowledgments.—We are indebted to J. H. Burns and Hugo Steinfink for their helpful advice regarding crystal chemistry. In addition, it is a pleasure to acknowledge the support and encouragement of W. R. Grimes and other members of the ORNL staff.

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[15] J. H. Burns and D. J. Duchamp, "Reactor Chemistry Division Annual Progress Report for Period Ending Jan. 31, 1962," ORNL-3262, p. 15.

Contribution from the Goodyear Atomic Corporation,
Piketon, Ohio

Hexafluorides of Molybdenum, Tungsten, and Uranium. II. Reactions with Nitryl Fluoride, Nitrosyl Fluoride, and Nitrosyl Chloride[1,2]

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Reactions of three metal hexafluorides with nitrosyl and nitryl fluorides resulted in solid compounds of the composition $NO_xF\cdot MF_6$ where x is 1 or 2 and M is W, Mo, or U. Infrared absorption data for these compounds indicate that the ionic form $NO_x{}^+MF_7{}^-$ and the molecular form $NO_xF\cdot MF_6$ contribute to the structure of the solid. Nitrosyl chloride does not act as do the nitrogen oxyfluorides with the hexafluorides but, like nitric oxide,[3] it reduces molybdenum and uranium to complex nitrosyl fluorides of Mo(V) and U(V), with the formation of chlorine. Tungsten hexafluoride does not react with nitrosyl chloride.

Introduction

The study of the action of various nitrogen-containing compounds on the hexafluorides of molybdenum, tungsten, and uranium has been continued in this laboratory. The action of nitrous oxide and nitric oxide toward the hexafluorides has been reported previously.[3] That work suggested that reaction occurred between the hexafluorides and nitrosyl fluoride. In the present investigation the reaction stoichiometry between the hexafluorides and nitryl fluoride, nitrosyl fluoride, or nitrosyl chloride and the physical and chemical properties of the reaction products were determined.

Experimental

Materials.—Purification of the hexafluorides and nitrosyl fluoride has been described.[3] Hydrogen fluoride was purified by the method of Jarry and Davis.[4] Boron trifluoride was used as obtained commercially from the Matheson Company.

Nitryl fluoride was prepared by allowing a slight excess (over

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[1] This work was performed under Contract AT-(33-2)-1 with the U. S. Atomic Energy Commission.

[2] Paper presented in part at the 138th National Meeting of the American Chemical Society, New York, N. Y., September 14, 1960.

[3] J. R. Geichman, E. A. Smith, S. S. Trond, and P. R. Ogle, *Inorg. Chem.*, **1**, 661 (1962).

[4] R. L. Jarry and W. Davis, *J. Phys. Chem.*, **57**, 60 (1953).

TABLE I

PHYSICAL PROPERTIES OF $NO_xF{\cdot}MF_6$ SOLID COMPOUNDS

Compound	Wave numbers, cm.$^{-1}$			Color	Density, g./cc.[a]	Vap. press. at 20°, mm.	Analyses, %		
	NO^+	MF_7^-	NO_2^+				Element	Calcd.	Found
$NOMoF_7$	2328	640		White		3	Mo	37.05	35.89
							F	51.36	51.92
							N	5.41	5.83
$NOWF_7$	2330	620		White	3.7 ± 0.2 at 25°	<1	W	53.00	53.41
	2327						F	38.34	36.70
							N	4.04	3.50
$NOUF_7$	2320	550		Greenish yellow	4.6 ± 0.2 at 25°	<1	U	59.36	58.98
	2318	509					F	33.16	29.66
							N	3.49	3.58
NO_2MoF_7		627	2374[b]	White		53	Mo	34.90	35.06
							F	48.27	45.95
							N	5.10	4.45
NO_2WF_7		620	2377	White		6	W	50.69	49.10
							F	36.65	36.37
							N	3.86	4.47
NO_2UF_7		...[c]	2374	Greenish white	3.8 ± 0.2 at 0°	8	U	57.07	57.23
							F	31.89	28.51
							N	3.36	3.64

[a] At the 95% confidence interval. [b] The ν_2 fundamental for NO_2^+ was not observed. This may result from overlap of the strong broad bands attributed to the MF_7^- ion or the low intensity of the ν_2 band of the NO_2^+ ion. [c] The band expected for the UF_7^- ion is in the region where it would be masked by bands of NO_2F.

stoichiometric) of fluorine to mix by diffusion with dinitrogen tetroxide for 16 hr. at 25° and a total pressure of approximately 1 atmosphere. Excess fluorine was removed by evacuating the product at −196°. Infrared analysis of the product showed a trace impurity of nitrogen dioxide, apparently resulting from the reactivity of nitryl fluoride with the sampling system. This decomposition was minimized by storage in a prefluorinated nickel container.

Nitrosyl chloride, obtained from the Matheson Company, was purified by alternate warming and cooling with evacuation at −78° to remove volatile impurities such as air and chlorine. The nitrosyl chloride was then condensed from a −78° trap into a −196° trap to remove dinitrogen tetroxide. Infrared analysis of the nitrosyl chloride obtained in the −196° trap showed that it contained less than 0.2 mole % nitrogen dioxide.

Solid reaction products were analyzed for molybdenum, tungsten, and uranium as their oxides. Fluorine was determined by a pyrohydrolytic method or the Willard–Winter distillation method.[5] Nitrogen was determined by Devarda's method.[6] Gaseous reaction products were identified by infrared or mass spectrometer analysis.

Equipment.—The general equipment and techniques of the infrared study have been described in an earlier report.[3] A water bath was used for vapor pressure measurements at 20.0 ± 0.05°. Pressure measurements at temperatures above ambient were measured in a large oven controlled within ± 0.5°. Pressure was determined by means of a Taylor Instrument Company absolute pressure transmitter connected to a nickel metal vacuum system in the oven. The pressure was read on a mercury manometer or a Bourdon tube gage which extended from the pressure transmitter outside the oven. A gas displacement method was used for solid density determinations.[7] X-Ray photographs on sealed samples were made with 114.6-mm. diameter North American Philips powder cameras on either a Norelco X-ray or a General Electric XRD-5 unit using nickel-filtered copper radiation.

Reaction with Nitrosyl and Nitryl Fluorides.—Weighed amounts of either nitrosyl or nitryl fluoride and the desired hexafluoride were condensed at −196° into a tared prefluorinated nickel or fluorothene reactor. The reactor was allowed to warm to room temperature for about 1 hr., after which the excess of either component was removed from the solid reaction product by evacuation. Evacuation was continued until a constant vapor pressure was obtained. Vapor pressure measurements and chemical analysis showed that nitrosyl and nitryl fluorides combine with the hexafluorides in a 1:1 molecular ratio to form solids of varying volatility. Infrared examination of the vapor over the solids showed that it consisted of approximately equal molar quantities of the starting components, indicating that the vapor pressure resulted from dissociation of the complex. Also, the infrared spectra of the solids were obtained by carrying out the reactions in a 10-cm. gas absorption cell. The infrared absorption spectra of the solid compounds $NO_xF{\cdot}MF_6$ showed bands which are attributed to the presence of NO_x^+ and MF_7^- ions and bands which are normal for gaseous NO_xF and MF_6 but appear more intense than would be expected for the gases at the dissociation pressure of the solids, indicating that a $NO_xF{\cdot}MF_6$ species was present. The absorption bands characteristic of these ions and other physical properties of the solids are given in Table I. The spectral data are very similar to those described previously for the $NOMoF_6$ and $NOUF_6$ ionic compounds.[3] It should be noted, however, that the NO^+ absorption bands show two maxima of nearly equal intensity for $NOUF_7$ and $NOWF_7$ contrasted to single peaks observed for the NO^+ in $NOUF_6$, $NOMoF_6$, and $NOMoF_7$. The band splitting can only be observed under high resolution such as is provided by a Perkin-Elmer Model 112 instrument described previously.[3] The splitting cannot be explained by isotope effects nor, as has been suggested,[8] by the presence of more than one molecule in the unit cell, in light of the X-ray work on $NOUF_7$.

Properties of NO_xUF_7.—Melting point determinations in a sealed thin-walled glass tube showed that $NOUF_7$ does not melt at temperatures up to 300°, while a melting point on NO_2UF_7 was not obtained because the thermal decomposition pressure is large enough at 140° to destroy the melting point tube. The dissociation pressures were measured in the range 25 to 70°. Fluorine at a known pressure was added to the compounds before the measurements to retard reaction of the NO_xF with the container. Since values of the dissociation pressure corrected for

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(5) W. Wagner, C. J. Hull, and G. E. Markle, "Advanced Analytical Chemistry," Reinhold Publishing Corp., New York, N. Y., 1956, p. 227.

(6) W. F. Hillebrand, G. E. F. Lundell, H. A. Bright, and J. I. Hoffman, "Applied Inorganic Analysis," John Wiley and Sons, Inc., New York, N. Y., 1953, p. 786.

(7) P. A. Faeth and C. B. Willingham, "The Assembly, Calibration and Operation of a Gas Adsorption Apparatus for the Measurement of Surface Area, Pore Volume Distribution and Density of Finely Divided Solids," Mellon Institute of Industrial Research, Pittsburgh, Pa., September, 1955.

(8) R. D. Peacock and D. W. A. Sharp, *J. Chem. Soc.*, 2762 (1959).

the fluorine pressure are nearly the same on heating or cooling of the compound, reversible dissociation is indicated. Corrected results are shown in Table II.

TABLE II
THERMAL DISSOCIATION OF $NO_xF \cdot UF_6$

	0°	20°	25°	35°	36°	41°
	Total pressure (p), mm.					
$NOUF_7$					1	1.5
NO_2UF_7	<1	8	12	25		
	46°	49°	56°	58°	63°	70°
	Total pressure (p), mm.					
$NOUF_7$		3		6		15
NO_2UF_7	60		106		157	246

For $NOUF_7$ and NO_2UF_7, $\log p$ (mm.) $= 11.795 - (3633/T)$ and $11.194 - (3018/T)$, respectively. Calculated heats of dissociation from the equations are 16.6 and 13.8 kcal./mole for $NOUF_7$ and NO_2UF_7. Using the heat of dissociation and the heat of formation of uranium hexafluoride,[9] nitrosyl fluoride,[10] and nitryl fluoride,[11] a $-\Delta H_f$ at 298°K. of 537.4 and 537.8 ± 2 kcal./mole[12] is found for $NOUF_7$ and NO_2UF_7, respectively.

The X-ray powder pattern of $NOUF_7$ indicates a pseudo-cubic symmetry having one formula weight per unit cell with $a_0 = 5.29$ Å. This is analogous to the compound $NOUF_6$.[3] The X-ray density of 4.5 g./cc. compares favorably with the experimentally determined value.

Determinations of the gas density of the vapor over the solid NO_2UF_7 at 20° (see Table III) give an average molecular weight of 237 ± 50[13] compared to the theoretical molecular weight of 208.5 for complete dissociation in the vapor phase. However, this difference may be due to a bias in the method of measurement and not to association.

TABLE III
GAS DENSITY OF $NO_2F \cdot UF_6$

P (measured), mm.	$V \times 10^3$, l. at S.T.P.	Amt. of gas, mg.	D, g./l.	Mol. wt.
8.5	6.7	67	10.0	225
8.3	6.5	72	11.1	246
8.6	6.8	73	10.7	242

Evidence for the presence of the molecular form $NO_2F \cdot UF_6$ in the solid structure was obtained in the following manner. The vapor over the solid NO_2UF_7 was allowed to come to its equilibrium concentration in the gas infrared cell at 25 ± 1° and the absorption spectrum was determined on the Model 21 instrument. The spectrum indicated absorption bands for gaseous NO_2F (1793 cm.$^{-1}$) and UF_6 (1152 cm.$^{-1}$). Then some solid was distilled into the infrared gas cell by warming the reactor containing the NO_2UF_7 with a hair dryer. After the system was permitted to cool to 25 ± 1°, the absorption spectrum was again obtained on the thin film which formed on the cell windows. Absorption bands of the NO_2^+ ion (2374 cm.$^{-1}$) in addition to the NO_2F and UF_6 previously recorded were observed, with the latter bands having a greater absorption than that recorded when only the vapor pressure was present. Repetition of the distillation process caused further increases in the absorption of the NO_2F, UF_6, and NO_2^+ ion bands. Upon evacuation of the cell the original transparency of the silver chloride windows was obtained.

The X-ray powder pattern of NO_2UF_7 is found to best fit a tetragonal symmetry having two formula weights per unit cell with $a_0 = 5.74$ Å. and $c = 2$.

Various chemical properties of NO_xUF_7 were determined. Boron trifluoride and hydrogen fluoride liberate uranium hexafluoride from NO_xUF_7 and form NO_xBF_4[14] and $NO_xF \cdot yHF$,[15] respectively. Nitric oxide liberates NO_xF and forms $NOUF_6$. Hydrolysis of the compounds gives hexavalent uranium and nitric acid, and with $NOUF_7$ also liberates nitric oxide.

$NOWF_7$.—Since the low vapor pressure of the solids $NOWF_7$ and $NOUF_7$ restricts their distillation into the infrared gas cell, another approach was made to determine whether the molecular form existed in these solids.

The reaction in the infrared gas absorption cell of NOF with WF_6 was selected because $NOWF_7$ has a vapor pressure favorable for quantitative measurements and WF_6 is unreactive with such possible contaminants in NOF as nitric oxide[3] and nitrogen dioxide.[16] Nitrosyl fluoride was prepared in the gas absorption cell by treating fluorine with a slight excess of nitric oxide. Tungsten hexafluoride was then forced into the cell in excess to react with all the available NOF. A spectrum obtained immediately exhibited bands in the 2330 and 620 cm.$^{-1}$ regions which are attributed to NO^+ and WF_7^-, at 1845, 767, and 521 cm.$^{-1}$ to NOF, and at 712 cm.$^{-1}$ to WF_6. The cell was then evacuated to remove excess WF_6 and NO. Evacuation continued for approximately 0.5 hr. to an equilibrium pressure of 35 μ. The Perkin-Elmer Model 21 instrument was used to observe changes in the absorption bands for NO^+, NOF, and WF_6 on evacuation. When the cell was isolated from the evacuation system, a pressure increase to approximately 300 μ occurred with a slight increase in absorption of the NOF and WF_6 and a decrease at the NO^+ band. When the cell was re-evacuated a pressure of 35 μ was again obtained and the absorption values of NOF and WF_6 were the same as found previously. Further evacuation and spectral observation showed that the absorption bands of NO^+ ion decreased and those of NOF and WF_6 remained constant. The compound was completely removed from the cell by prolonged evacuation and the cell windows regained their original transparency. It was concluded from this information that the NO^+-WF_7^- dissociates either directly into the original reactants as gases or to the solid molecular complex $NOF \cdot WF_6$ which vaporizes intact or as its gaseous components.

The problem then became one of determining whether the observed absorption of NOF and WF_6 is accounted for by the measured pressure in the cell. If the observed absorption is greater than that for the gaseous components, the existence of the molecular compound $NOF \cdot WF_6$ would be necessary to explain the increased absorption. Since the absorption coefficient of WF_6 gas has been previously determined to be 130.4×10^{-4}/mm. pressure/mm. cell length, quantitative estimates of its concentration were made. The total pressure of the 10-cm. cell containing the sample was 0.3 mm. when isolated and 0.035 mm. when open to the vacuum system. At these pressures, a theoretical absorption for WF_6 gas in a 10-cm. cell length would be 0.196 and 0.023 absorption unit, respectively, assuming half the pressure is derived from WF_6. However, if the pressure resulted from a $NOF \cdot WF_6$ gas identity, the WF_6 absorption would be 0.391 and 0.046 absorption unit, respectively. The absorption actually observed was 0.316 and 0.256 unit, far exceeding the expected theoretical absorption values for the WF_6 as a gas. The data indicate the existence of the $NOF \cdot WF_6$ in the solid phase and the possible existence of the $NOF \cdot WF_6$ gas identity at low pressures.

The X-ray powder diffraction data obtained indicate a low symmetry for $NOWF_7$ in contrast to the high (cubic) symmetry found for $NOUF_7$ and $CsWF_7$.[17] The solid $NOWF_7$ does not melt to a liquid phase but appears to sublime at 185–190°. Anhydrous hydrogen fluoride is found to displace the WF_6 from the $NOWF_7$.

Reaction with Nitrosyl Chloride.—Molybdenum and uranium hexafluorides mixed with an excess of liquid nitrosyl chloride at

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[9] F. D. Rossini, D. D. Wagman, W. H. Evans, S. Levine, and I. Jaffe, "Selected Values of Chemical Thermodynamic Properties," National Bureau of Standards Circular 500, U. S. Govt. Printing Office, Washington, D. C., 1952.

[10] H. S. Johnston and H. J. Bertin, Jr., *J. Am. Chem. Soc.*, **81**, 6402 (1959).

[11] E. Tschuikow-Roux, *J. Phys. Chem.*, **66**, 1636 (1962).

[12] The limits result from the uncertainty in the heat of formation of nitryl fluoride.

[13] At the 95% confidence interval.

[14] Identified by X-ray diffraction analysis.

[15] The value of y is from 3 to 6; see F. Seel, *et al.*, *Angew. Chem.*, **73**, 806 (1961).

[16] J. R. Geichman, E. A. Smith, L. R Swaney, and P. R. Ogle, unpublished work.

[17] G. P. Hargreaves and R. D. Peacock, *J. Chem. Soc.*, 2170 (1958).

25° give $NOMoF_6$ and $NOUF_6$[14] plus chlorine gas, while tungsten hexafluoride does not react. Even at 60° tungsten hexafluoride is unreactive. Liquid tungsten hexafluoride and liquid nitrosyl chloride at 20° in a 1:1 molecular ratio are found to be only partially miscible. The gas-phase reaction between the hexafluorides and nitrosyl chloride was studied in an infrared gas cell to determine if nitrosyl fluoride was an intermediate product of reaction. Nitrosyl fluoride was not observed.

Discussion

The data obtained for the compounds NO_xMF_7 indicate that the following equilibria exist

$$NO_xF(g) + MF_6(g) \rightleftharpoons NO_x F\cdot MF_6(s) \rightleftharpoons NO_x{}^+MF_7{}^-(s) \quad (1)$$

The solid 1:1 molecular addition compound is in equilibrium with the gases NO_xF and MF_6 and is composed of an ionic form $[NO_x{}^+MF_7{}^-]$ and a molecular form $[NO_xF\cdot MF_6]$ where the identities of the NO_xF and MF_6 are retained. Similar structures have been reported for the solids $Cu(NO_3)_2\cdot N_2O_4$,[18] $NOSbCl_6$, and $NOAlCl_4$.[19] The evidence for the presence of the gases NO_xF and MF_6 and the solid ionic structure $[NO_x{}^+MF_7{}^-]$ is readily deduced from the infrared absorption spectra. The presence of the molecular form in the solid is suggested by the low heat of dissociation found for NO_xUF_7 and by the quantitative measurements made for the infrared absorption bands of NO_xF and MF_6 in the solids NO_2UF_7 and $NOWF_7$.

The reactions studied here appear to occur through the initial formation of a 1:1 molecular addition compound. In the case of reaction with nitrosyl and nitryl fluorides, reduction of the metal from the six to the five valence state in the hexafluoro complex ion cannot oxidize the fluoride ion to the fluorine radical, nor does complete dissociation occur by a fluoride ion transfer to form $NO_x{}^+$ and $MF_7{}^-$ ions. Therefore, stability of the complex is accomplished by the partial acceptance of a fluoride ion (polarization) by the metal hexafluoride, accounting for the ionic and molecular structures observed in the solids. In the case of nitrosyl chloride, initial formation of an unstable 1:1 molecular addition compound is indicated since nitrosyl fluoride is not observed as a reaction intermediate, in contrast to its formation when nitric oxide reduces molybdenum and uranium hexafluorides.[3] Thus, reaction would appear to proceed by ionic dissociation of nitrosyl chloride *via* transfer of a chloride ion to the metal hexafluoride, forming an unstable complex ion (MF_6Cl^-) which dissociates to the chlorine radical and the $MF_6{}^-$ ion. Since tungsten hexafluoride is not reduced by nitric oxide,[3] reduction by nitrosyl chloride would not be expected. However, formation of a 1:1 molecular addition complex between nitrosyl chloride and tungsten hexafluoride might be suspected, but it does not occur.

Limits can be established for the values of the heat of formation of the solids $NOMoF_6$ and $NOUF_6$ on the basis of the reactions

$$NOF(g) + MF_6(g) \longrightarrow NOMF_6(s) + F(g) \quad (2)$$

$$NOCl(g) + MF_6(g) \longrightarrow NOMF_6(s) + Cl(g) \quad (3)$$

Since reaction 3 occurs and 2 does not, the $-\Delta H_f$ at 298°K. for $NOMoF_6$ is >398.4 and <416.1 kcal./mole and for $NOUF_6$ is >521.4 and <539.1 kcal./mole. A $-\Delta H_f$ at 298°K. of 382 kcal./mole for molybdenum hexafluoride was used in the calculations.[20]

Peacock and Sharp have reported infrared absorption frequencies of potassium salts of complex fluoro ions.[8] Their data are compared to ours in Table IV. The infrared absorption band in the metal–fluorine region of the $NOMF_7$ and NO_2WF_7 compounds appears to differ enough from the ν_3 absorption of the $MF_6{}^-$ ion to be characteristic of a heptacoordinate $MF_7{}^-$ structure. By analogy, the pseudo-cubic symmetry found for $NOUF_7$ might suggest a pentagonal bipyramid for the structure of the $UF_7{}^-$ ion since that configuration has been found for the $UF_7{}^-$ ion[3] and iodine heptafluoride, both of which have cubic symmetry.[21,22] However, the infrared absorption band in the metal–fluorine region for NO_2MoF_7 does not appear to be appreciably changed from the ν_3 absorption of the $MoF_6{}^-$ ion and suggests the possibility that the structure of $MoF_7{}^-$ is a distorted octahedron. The same metal–fluorine absorption maxima were found to exist for the $UF_6{}^-$ and $UF_7{}^-$ ions but with a reversal in their intensities. Addition of a fluoride ion to an $MF_6{}^-$ anion has been reported to lower the infrared frequency.[8] This is seen to be true for the transformation of $UF_6{}^-$ to $UF_7{}^-$ but not for molybdenum or tungsten.

TABLE IV

INFRARED ABSORPTION FREQUENCIES OF THE HEXAFLUORIDE AND THE FLUOROMETALATE ION

	Absorption frequency, cm.$^{-1}$[a]				
M	MF_6	$MF_6{}^-$		$MF_7{}^-$	
Mo	741[8]	623[8]	615 (b, vs)[3,b]	645[8]	640 (b, vs)[b]
					627[c]
W	712[8]	594[8]	...	620[8]	620 (b, vs)[b,c]
U	623[3]	...	550 (b, vs)[3,b]	...	550 (sh)[b]
			509 (sh)		509 (b, vs)

[a] b, broad; vs, very strong; sh, shoulder. [b] For NO^+ compounds. [c] For $NO_2{}^+$ compounds.

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(19) C. C. Addison and J. Lewis, *Quart. Rev.* (London), **9**, 115 (1955).

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(22) R. P. Burbank and F. N. Bensey, Jr., *J. Chem. Phys.*, **27**, 981 (1957)