termolecular contacts less than 3.5 Å, and those only with carbon atoms. Although dipolar interactions should be important in the packing of the molecules, we cannot recognize a simple or predominant arrangement of dipoles.

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Registry No. TMPD·$2SO_2$, 54657-95-3.

Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 20× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for \$4.50 per photocopy or \$2.50 for microfiche, referring to code number AIC40729V.

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Contribution from the Department of Chemistry and Division of Engineering,
Brown University, Providence, Rhode Island 02912

Preparation, Crystal Structure, and Properties of $NH_4ReO_{1.5}F_3 \cdot H_2O$

F. PINTCHOVSKI, S. SOLED, R. G. LAWLER, and A. WOLD*

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Single crystals of $NH_4ReO_{1.5}F_3 \cdot H_2O$ have been prepared and characterized. The material is synthesized by allowing ammonium perrhenate, rhenium metal, and ammonium hydrogen fluoride to react under a hydrostatic pressure of 20,000 psi at 400°. The product crystallizes in the cubic system, space group $Fm3m$, with $a_0 = 16.563$ (6) Å; $\rho_{\text{exptl}} = 3.57$ (2) g cm^{-3}, and $\rho_{\text{calcd}} = 3.55$ g cm^{-3} for $Z = 32$. The structure consists of isolated $Re_8O_{12}F_{24}$ units bridged by ammonium ions and body-centered rhombic dodecahedra of water molecules. The material has unpaired electrons, as shown by its temperature-dependent paramagnetism, and is an insulator because of the isolation of the $Re_8O_{12}F_{24}$ units.

Introduction

The substitution of fluorine for oxygen in transition metal oxides changes the transport properties of these materials.[1-5] The preparation and properties of members of the series $MO_{3-x}F_x$ (M = W, Mo) have been studied by a number of investigators.[1,3,5] As fluorine is substituted for oxygen into MoO_3 and WO_3, the resulting oxyfluorides $MoO_{3-x}F_x$ and $WO_{3-x}F_x$ become metallic.[3,5]

ReO_3 crystallizes with the perovskite (ABO_3) structure with the A sites vacant. Goodenough[6] has proposed a conduction mechanism for the ReO_3-type compounds involving a strong covalent mixing between the metal t_{2g} and the oxygen p_π orbitals. When the resulting band is partially occupied, as in ReO_3, metallic behavior is observed. Whereas the substitution of oxygen by fluorine increases the electron concentration in this band, it may also decrease the degree of delocalization of the electrons, since fluorine is more electronegative than oxygen. It would, therefore, be of interest to study the effect of the substitution of oxygen by fluorine in metallic ReO_3.

The existence of several unstable, hygroscopic rhenium oxyfluorides such as $ReOF_4$, $ReOF_3$, ReO_3F, and ReO_2F_2 has been described by various investigators,[7-9] but the system $ReO_{3-x}F_x$ has not been reported. An attempt was made to prepare members of this system by hydrothermal synthesis similar to the procedures described for the preparation of $MoO_{3-x}F_x$; however, no appreciable fluorine substitution was observed when aqueous solvents were employed. It was, therefore, necessary to develop a new technique which involves the application of hydrostatic pressure to molten salts. Ammonium hydrogen fluoride was chosen as the molten salt because it is a low-melting solid (125°), a strong fluorinating agent, and a nonaqueous solvent. The reaction of ammonium perrhenate and rhenium metal in the presence of this compound yielded a new phase with the composition $NH_4ReO_{1.5}F_3 \cdot H_2O$. Lustrous black octahedral crystals (0.05–0.50 mm on edge) of this compound were obtained.

This paper describes the preparation of this compound and the characterization of the material by chemical analysis, density measurements, X-ray structure determination, and nuclear magnetic resonance techniques.

Experimental Section

Preparation of Materials. $NH_4ReO_{1.5}F_3 \cdot H_2O$ single crystals were synthesized with the hydrothermal apparatus described previously.[3] A mixture of spectroscopically pure ammonium perrhenate (J. Matthey and Co.), high-purity rhenium metal (Atomergic Chemicals Co.), and recrystallized ammonium hydrogen fluoride in the mole ratio 1:$^1/_{10}$:10 was placed in thin-walled gold tubes, 5-mm i.d. × 10 cm, that were sealed previously at one end. The top of the tube was crimped shut and melt-sealed and the tube was placed in a steel pressure vessel. The temperature and pressure were monitored continuously and maintained within ±5° and ±0.01 kbar, respectively. Crystals were grown by allowing the mixture to react at 400° and 1.36 kbars for 6 days, followed by cooling at a rate of 50°/hr.

Chemical Analysis. The fluoride ion concentration was measured with an electrode (Fluoride Ion Activity Electrode, Model 94-09, Orion Research, Inc.) specifically sensitive to this ion, as described previously.[3]

A gravimetric determination of rhenium by the nitron perrhenate method[10] and of nitrogen by the Kjeldhal method[11] was performed.

Although interference by the fluoride ion prevents a direct quantitative determination of the amount of oxygen present in the compound, the number of protons can be readily determined from nuclear magnetic resonance measurements, since the protons in the compound will exchange rapidly with the water protons in an aqueous solution.[12]

The concentration of exchangeable protons in the sample dissolved in 20% DCl–D_2O (Alfa Inorganics, 99% isotopic purity) was determined from the intensity of the proton NMR signal. Solutions of known molarity of sodium formate (reagent grade, dried for 6 hr at 120°) (solution 1), the unknown and sodium formate (solution 2), and ammonium perrhenate and sodium formate (solution 3) were prepared.

The nonexchangeable proton of the sodium formate provided a standard line whose area was compared to the increase in area of the HOD peak, which results from the exchange between the deuterons of the solvent and the exchangeable protons from either solution 2 or solution 3.

The ammonium perrhenate and sodium formate (solution 3) with four exchangeable protons was used as a control. Solution 1 (sodium formate in DCl–D_2O) was used to determine the background concentration of HOD in the commercially available solvent. The area of this HOD peak was subsequently subtracted from the areas of the HOD peaks in solutions 2 and 3 (column 3 in Table IV).

The number of exchangeable protons was calculated by dividing the ratio of unknown to sodium formate concentration by the corresponding ratio of their NMR peak areas (see formula in Table IV).

Proton nuclear magnetic resonance spectra were recorded on a Varian A-60A spectrometer. The single sharp lines from sodium formate and HOD were integrated repeatedly using the spectrometer's electronic integrator.

Finally, the oxidation number of rhenium was determined by dissolving the sample in acid V^{5+} solution and potentiometrically titrating the reduced vanadium with a standard solution of Ce^{4+}.[13]

Density Measurements. The determination of the density of the crystals was obtained by a hydrostatic technique based on Archimedes' principle[14] with the use of a Mettler H-54 analytical balance. Perfluoro(1-methyldecalin) (Pierce Chemical Co.) was the liquid medium and was first calibrated with a crystal of high-purity silicon (Gallard-Schlesinger, 99.9999%), $\rho = 2.328$ (2) g cm^{-3}.

Magnetic Measurements. Magnetic susceptibility data over the temperature range 77–300°K were obtained with a Faraday balance[15] equipped with a Cahn R.G. Electrobalance. The susceptibility was also measured as a function of field strength (the Honda–Owen technique[16]) to ascertain the presence of ferromagnetic impurities. Susceptibility values were corrected for the core diamagnetism of Re, N, F, and O.

Unit Cell and Intensity Data. From precession photographs, the crystal system, Laue symmetry, and space group were determined. The precise lattice parameter was obtained at ambient room temperature from a least-squares refinement of the angular settings of 12 strong reflections which were centered on a Picker FACS-1 diffractometer using Mo K$\bar{\alpha}$ radiation (λ 0.7107 Å) and equipped with a graphite monochromator.

Powder specimens were also examined by both fast (1° 2θ min^{-1}) and slow ($^1/_4$° 2θ min^{-1}) scan X-ray techniques using a Norelco diffractometer with monochromatic Cu Kα_1 radiation (λ 1.5405 Å) to check for the presence of any additional phase. Three-dimensional intensity data, over one hemisphere of reciprocal space within the annular region $5° < 2\theta < 50°$, were also collected on the FACS-1. A symmetrical scan range in 2θ was used from −0.90 to +0.90° for the Mo Kα peak with allowances made for the Kα_1–Kα_2 separation. Attenuators were inserted automatically if the count rate of the diffracted beam exceeded approximately 9000 counts sec^{-1} during the scan.

Table I. Crystallographic Data for $NH_4ReO_{1.5}F_3{\cdot}H_2O$

System	Cubic
a, Å	16.563 (6)
Absences	*hkl*, $h + k = 2n + 1$ *hkl*, $h + l = 2n + 1$ *hkl*, $k + l = 2n + 1$
Space group	*Fm*3*m*
Z	32
ρ_{calcd}, g cm^{-3}	3.55
ρ_{exptl}, g cm^{-3}	3.57 (2)
μ, cm^{-1} (Mo Kα)	226.2
Crystal habit	Octahedron bounded by {111}, {010}
Crystal size, mm	0.28 × 0.20 × 0.28

Structure amplitudes were calculated in the usual way. The variance in F_o^2 was estimated by the expression

$$\nu(|F_o|^2) = \sigma(|F_o|^2)^2 = (Lp)^{-2}[C + 0.83 + (t_c/2t_b)^2(B_1 + B_2 + 1.65) + (0.05C)^2 + (0.05(B_1 + B_2))^2]$$

where Lp is the Lorentz–polarization factor, C is the total integrated count obtained in time t_c, and B_1 and B_2 are the two background counts each obtained in time t_b. The intensities were corrected for absorption by means of the GONO 9 program;[17] transmission coefficients ranged from 0.058 to 0.14. The average value of $|F_o|^2$ and the standard deviation of the mean, $\sigma(F_o)^2$, were used in the subsequent least-squares refinements.

Solution and Refinement of the Structure. The structure was solved by standard heavy-atom methods and refined by least-squares methods using the program BULS, which is a local variation of the ORFLS program.[18] The function minimized was $\sum w(|F_o| - |F_c|)^2$, with weights taken as the reciprocal variance in $|F_o|$. In all calculations, the atomic scattering factors were taken from Cromer and Waber.[19] The effects of anomalous dispersion for the rhenium atom were included in the calculated structure factors with the appropriate values of $\Delta f'$ and $\Delta f''$ taken from the report of Cromer.[20]

The rhenium and oxygen atoms were located from a three-dimensional Patterson function. The centrosymmetric space group *Fm*3*m* was chosen initially and the successful refinement confirmed this choice. The lower symmetry cubic space groups *F*23, *F*$\bar{4}$3*m*, *F*432, and *Fm*3, which are the only ones consistent with the diffraction data (Table I), were also considered but failed to give a logical ordered model in place of the disordered one required in the centrosymmetric space group *Fm*3*m*. [Also, space groups *F*23 and *F*$\bar{4}$3*m* require two independent rhenium atoms, and the space group *F*432 and *Fm*3 requirements for the F and O atoms of the $Re_8O_{12}F_{24}$ unit are in conflict with the experimental data.] The coordinates and isotropic temperature factor parameters of these atoms were refined by two cycles of least-squares adjustment to give $R = 11.5\%$, $R_w = 14.3\%$. From a succession of difference Fourier maps and least-squares refinements, the positions of all nonhydrogen atoms in the structure were determined. Since oxygen and fluorine have similar scattering powers, it was necessary to determine the fluorine content by direct chemical analysis. The oxygen content was determined by difference. The structure was refined assuming that 50% of the possible water sites were occupied. However, there can be a variation of occupancy by as much as ±10%. Refinement of the complete structure with individual isotropic thermal parameters for all the atoms yielded final values of $R = 3.4\%$, $R_w = 3.6\%$. The structure was confirmed by a final difference synthesis. The highest ripple in the final difference map is of the order of 2.2 electrons Å^{-3} in the neighborhood of the rhenium atoms but no higher than 1.4 electrons Å^{-3} elsewhere. The crystallographic data for the compound are set out in Table I. The positional and thermal parameters of the atoms are shown in Table II. A tabulation of the final values of F_o and $|F_c|$ (in electrons × 10) for the 170 reflections included in the refinement is available.[21]

Results and Discussion

Chemical and Nuclear Magnetic Resonance Analyses. Table III compares the results of the chemical analyses with the calculated values based on the formula $NH_4ReO_{1.5}F_3{\cdot}H_2O$. In Table IV, the analysis for exchangeable protons using nuclear magnetic resonance is shown. The formal oxidation state of rhenium obtained from potentiometric analysis was

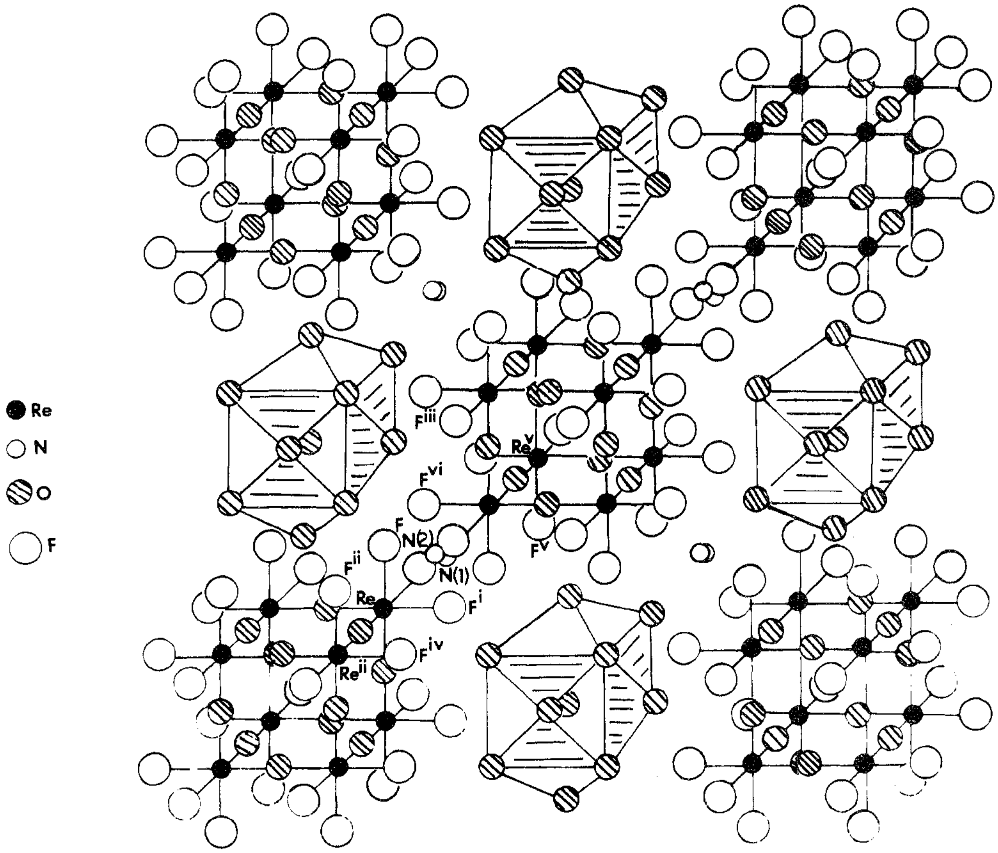

Figure 1. An idealized view of the crystal structure of $NH_4ReO_{1.5}F_3 \cdot H_2O$ viewed down the [010] direction, showing the $Re_8O_{12}F_{24}$ clusters. with bridging ammonium ions and water dodecahedra.

Table II. Final Parameters and Their Standard Deviations

Atom	*x*	*y*	*z*	*B*, Å^2	Wyckoff notation	Point symmetry	No. per unit cell
Re	0.1101 (1)	0.1101 (1)	0.1101 (1)	0.97 (3)	(f)	3*m*	32
O	0.0	0.1126 (8)	0.1126 (8)	1.3 (3)	(h)	*mm*	48
F	0.1176 (5)	0.1176 (5)	0.2316 (6)	2.1 (2)	(k)	*m*	96
N(1)	0.25	0.25	0.25	2.8 (1.3)	(c)	$\bar{4}3m$	8
N(2)	0.25	0.0	0.25	3.7 (9)	(d)	*mmm*	24
$H_2O(1)$	0.0	0.0	0.50	1.1 (2.7)	(b)	*m*3*m*	4 × $^1/_2$ = 2
$H_2O(2)$	0.4009 (25)	0.4009 (25)	0.4009 (25)	6.4 (1.8)	(f)	3*m*	32 × $^1/_2$ = 16
$H_2O(3)$	0.0	0.0	0.2922 (44)	4.0 (2.0)	(e)	4*mm*	24 × $^1/_2$ = 12

Table III. Chemical Analysis of $NH_4ReO_{1.5}F_3 \cdot H_2O$

	% obsd	% calcd
Re	61.6 (2)	61.4
F	18.3 (2)	18.8
N (as NH_3)	5.7 (3)	5.6

5+. Changes in the Re:NH_4ReO_4 molar ratios (0.05–0.50) had no effect on the nature of the product obtained, except for varying the amount of water of hydration present. It is interesting to note that a hydrated product was obtained even though nonaqueous reactants were used. It appears that water is a by-product of the decomposition of ammonium perrhenate.

Description of the Structure. $NH_4ReO_{1.5}F_3 \cdot H_2O$ crystallizes in a new variant of the ReO_3 structure type. In the latter structure, metal atoms occupy the corners of a cubic cell (a_0 = 3.72 Å) with bridging oxygen atoms midway along the edges, so that three-dimensional infinite chains of corner-shared octahedra result. Compounds with this structure include ReO_3,[22] $TiOF_2$,[25] NbO_2F,[26] and TiF_3.[27]

When fluorine is substituted for oxygen in transition metal oxides, a number of possible structure types form. One such possibility is the stabilization of the ReO_3 structure, for example, in $TiOF_2$[25] and NbO_2F.[26] If in ReO_3, two fluorine atoms substitute for an oxygen atom, a phase having the composition $ReO_{3-x}F_{2x}$ would result. If, for $ReO_{3-x}F_{2x}$, $x = ^3/_2$, the composition $ReO_{1.5}F_3$ results. In $NH_4ReO_{1.5}F_3 \cdot H_2O$, there are units of 8 $ReO_{1.5}F_3$ centered on each lattice point of a face-centered cubic lattice of 16.5 Å. The 8 $ReO_{1.5}F_3$ units are comprised of 8 rhenium atoms at the corners of a 3.65-Å cube surrounded by 12 bridging oxygen atoms and 24 terminal fluorine atoms. The geometry of these units is presented in Table V, together with a comparison of bond distances in ReO_3,[22] Re_2O_7,[23] and $ReOF_4$.[24] These 3.65-Å cube units have virtually the same size as an ReO_3 unit cell (3.72 Å).

Every rhenium atom is surrounded by a distorted octahedron (trigonal antiprism) of three oxygen and three fluorine atoms. Each cube unit is well isolated from the next one; the closest approach is 3.53 (2) Å between neighboring fluorine atoms. This maintains the essential geometry of the ReO_3 structure even though the heavy to light atom ratio has become 1:4.5.

A packing diagram of the structure viewed down [010] is presented in Figure 1. The $Re_8O_{12}F_{24}$ units are bridged by NH_4^+ ions parallel to the face and body diagonals of the unit cell and by clusters of body-centered rhombic dodecahedra of water molecules parallel to the cell edges. The ammonium ions occur in two different sites, forming infinite three-dimensional linear chains (N···N separation 4.13 Å) in the

Table IV. Nuclear Magnetic Resonance Analysis[a]

Sample	HCO_2Na	HOD	(HOD)-$(HOD)_1$	No. of exchangeable protons
(1) 0.290 *M* HCO_2Na	103 (8) [16][b]	32 (2) [8]		
(2) 0.290 *M* HCO_2Na 0.0486 *M* unknown	101 (5) [16]	138 (4) [15]	106 (4)	6.3 (5)
(3) 0.290 *M* HCO_2Na 0.0486 *M* NH_4ReO_4	101 (9) [9]	106 (2) [9]	74 (3)	4.4 (6)

[a] The areas of the sodium formate and HOD peaks are shown for the three samples. (The number in parentheses represents the standard deviation.) The number of exchangeable protons in the rhenium compounds in samples 2 and 3 was calculated from

$$\text{no. of protons} = \left(\frac{0.290}{0.0486}\right)\left(\frac{\text{area}[(\text{HOD})\text{-}(\text{HOD})_1]}{\text{area}[\text{HCO}_2\text{Na}]}\right)$$

[b] The number of times a peak was scanned is given in brackets.

Table V. Interatomic Distances and Angles

$NH_4ReO_{1.5}F_3 \cdot H_2O$

Symmetry	Code		
No symbol	x	y	z
i	z	x	y
ii	x	$\bar{y}$	z
iii	$^1/_2 - z$	y	$^1/_2 - x$
iv	z	$\bar{x}$	y
v	$^1/_2 - x$	y	$^1/_2 - z$
vi	$^1/_2 - z$	$\bar{y}$	$^1/_2 - x$
vii	$^1/_2 - x$	$^1/_2 - y$	z
viii	$^1/_2 - x$	$^1/_2 - y$	$1 - z$
ix	$^1/_2 - x$	$-^1/_2 + y$	$1 - z$

Atoms	Distance, Å	Atoms	Distance, Å
Re–O	1.825 (2)	$N(1)\cdots F$	3.12 (1)
Re–F	2.020 (9)	$N(2)\cdots F$	2.95 (1)
$Re\cdots Re^{ii}$	3.647 (2)	$H_2O(1)\cdots H_2O(2)^{vii}$	2.84 (2)
$Re\cdots Re^{v}$	6.554 (2)	$H_2O(1)\cdots H_2O(3)$	3.44 (7)
$F\cdots F^{i}$	2.67 (2)	$H_2O(2)^{vii}\cdots H_2O(3)$	2.94 (5)
$F\cdots F^{ii}$	3.89 (2)	$F\cdots H_2O(3)$	2.93 (3)
$F\cdots F^{iii}$	3.53 (2)	$F\cdots H_2O(2)^{vii}$	2.84 (1)

Atoms	Angle, deg	Atoms	Angle, deg
F–Re–O	92.1 (2)	$H_2O(3)\cdots H_2O(2)^{vii}\cdots H_2O(1)$	73.0 (1.3)
F^{i}–Fe–O	173.2 (3)	$H_2O(2)^{vii}\cdots H_2O(1)\cdots H_2O(2)^{ix}$	109.4 (1.4)
$F\cdots N(2)\cdots F^{iv}$	97.4 (2)	$H_2O(2)^{vii}\cdots H_2O(3)^{iii}\cdots H_2O(2)^{ix}$	104.5 (2.5)
$F\cdots N(2)\cdots F^{v}$	106.4 (2)	$H_2O(3)^{iii}\cdots H_2O(2)^{vii}\cdots H_2O(3)$	111.9 (1.0)
$F\cdots N(2)\cdots F^{vi}$	126.1 (4)	$F\cdots H_2O(3)\cdots F^{ii}$	83.2 (9)

Other Rhenium Compounds

Compd	Atoms	Distance, Å	Atoms	Angle, deg
ReO_3[22]	Re–O	1.87 (1)		
Re_2O_7[23]	Re–O (bridging)	2.08 (2), 1.78 (2), 2.15 (2)		
	Re–O (terminal)	1.66–1.80		
$ReOF_4$[24]	Re–O (terminal)	1.65 (3)	Re–F–Re	139 (4)
	Re–F (terminal)	1.86 (2)		
	Re–F (bridging)	1.99 (3), 2.30 (3)		

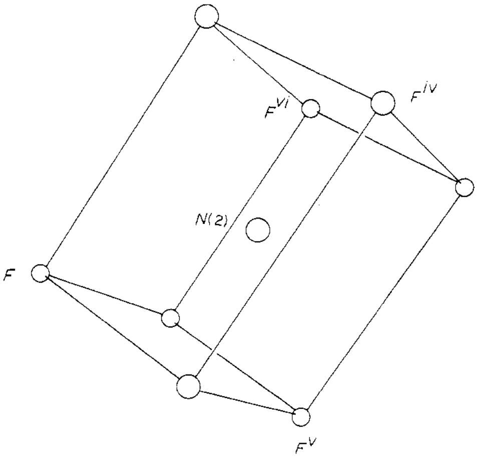

Figure 2. A view of the N(2) environment showing the rectangular prism of fluorine atoms.

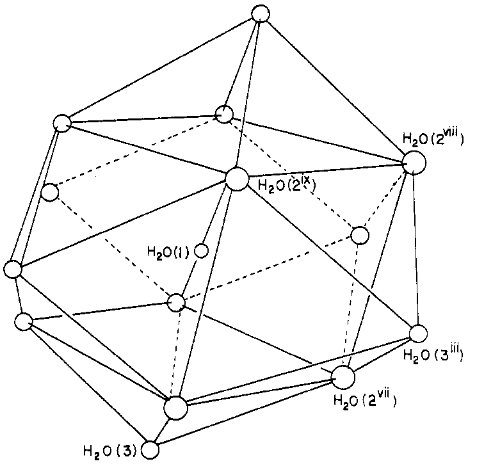

Figure 3. A perspective drawing of the body-centered water dodecahedra.

cavities between the $Re_8O_{12}F_{24}$ units. The first type of NH_4^+ ions (8 per cell) lies along the body diagonals. The NH_4^+ ions are surrounded by 12 fluorine neighbors ($N(1)\cdots F$ = 3.12 (1) Å), three at each leg of a tetrahedron. This same type of coordination around an ammonium ion has been reported for cubic NH_4SiF_6.[28] Neutron diffraction studies of this compound showed a threefold rotational disorder of each hydrogen atom associated with an ammonium ion.

The second group of ammonium ions (24 per unit cell) occupy the center of rectangular prisms (2.7 × 3.5 × 3.9 Å) of fluorine atoms (Figure 2). The prisms lie parallel to the face diagonals of the unit cell and are formed from the fluorine atoms from two neighboring $Re_8O_{12}F_{24}$ units. The eight $N(2)\cdots F$ distances of 2.95 (1) Å are in the range of $N\text{-}H\cdots F$ hydrogen bond formation. If the ammonium tetrahedra are positioned with the hydrogen atoms pointing toward the alternate corners of the rectangular prisms (as the $F\cdots N\cdots F$ angles in Table V suggest), these hydrogen atoms will have a twofold disorder, since the ammonium ions can be positioned into the prism in two ways.

The water molecules are clustered into undistorted body-centered rhombic dodecahedra centered at the middle of each edge (and at the center of the unit cell) and bridging 6 $Re_8O_{12}F_{24}$ units parallel to the cell edges. The internal geometry of the dodecahedra is shown in Figure 3 and listed in Table V. There is hydrogen bonding both within the

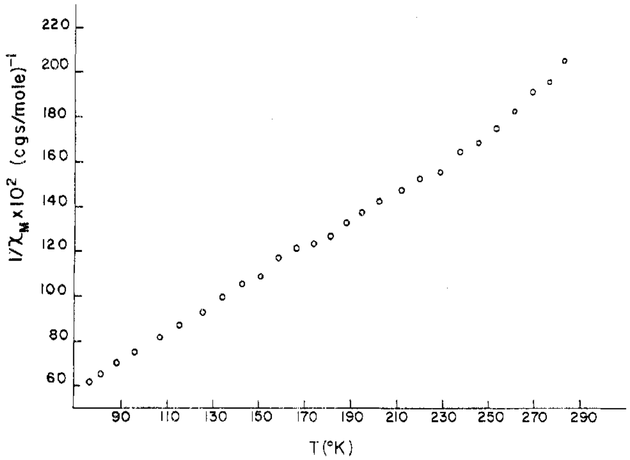

Figure 4. Inverse susceptibility vs. temperature for $NH_4ReO_{1.5}F_3 \cdot H_2O$.

dodecahedra (between $H_2O(1)$ and $H_2O(2^{vii})$ (O⋯O = 2.84 (2) Å) and between $H_2O(2)^{vii}$ and $H_2O(3)$ (O⋯O = 2.94 (5) Å)) and between the dodecahedra and neighboring fluorine atoms. The distance from the water molecules at the tip of a dodecahedron ($H_2O(3)$) to the four nearest fluorine atoms is 2.93 (3) Å, and the distance from each water molecule in the dodecahedron base ($H_2O(2)$) to its nearest fluorine neighbor is 2.84 (1) Å.

Physical Properties. A plot of χ_M^{-1} vs. T (Figure 4) indicates that the material shows temperature-dependent paramagnetic behavior between 77 and 300°K. Although the complexity of the spin–orbit coupling precludes an interpretation of the susceptibility data, it is clear that unpaired electrons are present in the material. Both ReO_3 and $M_{0.6}ReO_3$ (M = Na, K) show metallic behavior since they have delocalized electrons present in a π^* conduction band formed from the overlap of the rhenium t_{2g} and oxygen p_π orbitals.[29,30] The temperature-dependent paramagnetic behavior as well as the high resistivity ($\rho \simeq 10^{10}\ \Omega$ cm) indicates that $NH_4ReO_{1.5}F_3 \cdot H_2O$ is an insulator. Evidently, there are limited regions of electron delocalization (e.g., in the subunits of $Re_8O_{12}F_{24}$), but there are no long-range interactions between these units. This is not surprising considering the isolation of each $Re_8O_{12}F_{24}$ unit, with a rhenium-to-rhenium nearest-neighbor distance of 6.554 (2) Å.

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Registry No. $NH_4ReO_{1.5}F_3 \cdot H_2O$, 54689-15-5; ammonium perrhenate, 13598-65-7; ammonium hydrogen fluoride, 1341-49-7; rhenium, 7440-15-5.

Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for $4.00 for photocopy or $2.50 for microfiche, referring to code number AIC40771Q.

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