

Synthesis and Characterization of TeF₅OF

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Received April 12, 1982

A new method for the synthesis of hypofluorites was discovered utilizing fluorine fluorosulfate as the fluorinating agent. The method was successfully applied to the high-yield synthesis of the new hypofluorite TeF₅OF. The compound was also prepared in lower yield by the fluorination of TeF₅OH with a concentrated NF₄HF₂ solution. The physical properties and infrared, Raman, ¹⁹F NMR and mass spectra of TeF₅OF are reported. The vibrational spectra of TeF₅OCl were redetermined, and complete vibrational assignments are given for TeF₅OF and TeF₅OCl.

Introduction

The number of elements known to form hypofluorites is small and until recently was limited to the following nonmetal main-group elements: H, C, N, O, S, Se, F, and Cl.¹ The synthetic method used for the syntheses of these hypofluorites involved the fluorination of the corresponding hydroxyl compounds of their metal salts with elemental fluorine. An unsuccessful attempt² was made to apply this method to the synthesis of the hitherto unknown TeF₅OF. This failure to prepare TeF₅OF, but the success in the synthesis of TeF₅OCl by an analogous method,² led to the conclusion³ that TeF₅OF is unstable or actually nonexistent.

Our recent success⁴ in preparing a stable iodine hypofluorite and the observation that hypofluorites are generally more stable than the other hypohalites suggested that TeF₅OF should not only exist but should also be stable. In this paper we present data that show that TeF₅OF indeed exists and is stable.

Experimental Section

Materials and Apparatus. Volatile materials were manipulated in a stainless steel vacuum line equipped with Teflon FEP U-Traps, 316 stainless steel bellows-seal valves, and a Heise Bourdon tube-type pressure gauge. Telluric acid was prepared by a literature method⁵ and also purchased from Cerac, Inc., and from Pfaltz and Bauer. Fluorosulfuric acid (Allied) was used both as it was received (light brown color) and after it was distilled to obtain the clear colorless material. Fluorine fluorosulfate was synthesized as described.⁶ The reaction of TeF₅OH with either ClOSO₂F or ClF was used to prepare TeF₄OCl.⁷ Cesium and potassium chloride were oven-dried and then cooled and powdered under the dry N₂ atmosphere of a glovebox.

Infrared spectra were recorded in the range 4000–200 cm⁻¹ on a Perkin-Elmer Model 283 spectrophotometer calibrated by comparison with standard gas calibration points,^{8,9} and the reported frequencies are believed to be accurate to ±2 cm⁻¹. The spectra of gases were obtained with use of either a Teflon cell of 5-cm path length equipped with AgCl windows or a 10-cm stainless steel cell equipped with polyethylene windows that were seasoned with ClF₃. The spectra of matrix-isolated TeF₅OF and TeF₅OCl were obtained at 6 K with an Air Products Model DE202S helium refrigerator equipped with CsI windows. Research grade Ne (Matheson) was used as a matrix material in a mole ratio of 400:1.

The Raman spectra were recorded on a Cary Model 83 spectrophotometer with use of the 488-nm exciting line of an Ar ion laser

and a Claassen filter¹⁰ for the elimination of plasma lines. Quartz tubes (3 mm o.d.), closed by a metal valve, were used as sample containers in the transverse-viewing, transverse-excitation technique. A previously described¹¹ device was used for recording the low-temperature spectra. Polarization measurements were carried out by method VIII as described by Claassen et al.¹⁰

The ¹⁹F NMR spectra were recorded at 84.6 MHz on a Varian Model EM 390 spectrometer. Chemical shifts were determined relative to the CFCl₃ solvent with positive shifts being downfield from CFCl₃.¹² Second-order spectra were analyzed by using the programs NMRIT and NMREN by Swalen.¹³

The mass spectra were recorded with an EAI Quad 300 quadrupole spectrometer at an ionization potential of 40 eV.

Synthesis of TeF₅OH. Telluric acid, H₂TeO₄·2H₂O or Te(OH)₆, was fluorinated to give TeF₅OH by the method of Seppelt and Nothe² with use of HSO₃F as the fluorinating agent. This technique calls for the use of distilled HSO₃F, and initially we encountered difficulty in producing TeF₅OH. Subsequently, it was discovered that adding a few milliliters of H₂O to the reaction mixture and heating the reaction mixture at 160–170 °C for 5–6 h resulted in continuous evolution of TeF₅OH at a slow to moderate rate. Finally, undistilled HSO₃F was employed which furnished TeF₅OH in 70% purified yield; 93.9 mmol of TeF₅OH from 135 mmol of Te(OH)₆ and 1.75 mol of HSO₃F. Fractional condensation was used for the final product purification.

Fluorination of M⁺TeF₅O⁻. The salts CsTeF₅O¹⁴ and KTeF₅O¹⁵ were treated with F₂ in stainless steel cylinders at low temperature. Thus CsTeF₅O (1.43 mmol) and F₂ (4.46 mmol) were allowed to react for 8 days at -45 °C. The only volatile product condensable at -196 °C was TeF₆ (0.38 mmol, 26%). Similarly at -10 °C for 2 weeks a 48% yield of TeF₆ was obtained from the cesium salt. When the potassium salt (2.92 mmol) and F₂ (4.46 mmol) were kept at -45 °C for 6 weeks, again TeF₆ (2.35 mmol, 80%) was the only volatile tellurium compound observed.

Synthesis of TeF₅OF from CsTeF₅O and FOSO₂F. A 30-mL stainless steel Hoke cylinder was loaded with CsTeF₅O (3.42 mmol) in the glovebox. After evacuation and cooling of the cylinder to -196 °C, FOSO₂F (2.79 mmol) was added from the vacuum line. The closed cylinder was slowly warmed to -78 °C in a liquid-nitrogen-CO₂ slush bath and finally kept at -45 °C for 9 days. When the cylinder was recooled to -196 °C, about 4–5 cm³ of noncondensable gas was observed to be present. This was pumped away, and the condensable products were separated by fractional condensation in a series of U-traps cooled at -78, -126, and -196 °C. The -78 °C fraction was TeF₅OH (0.19 mmol) while the -196 °C fraction was TeF₆ (0.49 mmol). A white solid was retained at -126 °C, which changed to a colorless glass and melted, over a range of a few degrees, near -80 °C to a clear, colorless liquid. This material was identified as TeF₅OF (1.91 mmol, 68% yield) on the basis of its vapor density molecular weight: found, 256.2; calculated, 257.6. Further identification was based on its spectroscopic properties (see below) and on the preparation of derivatives.¹⁶ The observed weight loss of the solid (0.375 g) agreed

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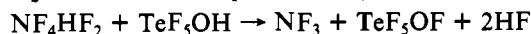
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well with that calculated (0.389 g) for the conversion of 2.79 mmol of CsTeF₅O to CsSO₃F. The following vapor pressure–temperature data of TeF₅OF were measured (*T* in °C, *P* in mm): -79.3, 16; -64.2, 45; -57.6, 63; -46.9, 108; -32.5, 210; -23.0, 312.

Synthesis of TeF₅OF from TeF₅OH and NF₄HF₂. A sample of NF₄HF₂·*n*HF (10.5 mmol) was prepared and concentrated in a Teflon double-U metathesis apparatus, as previously described.¹⁷ To this reactor was added TeF₅OH (10.5 mmol) on the vacuum line at -196 °C. The mixture was allowed to warm slowly to -23 °C and was kept at this temperature for 8 h under a dynamic vacuum. The volatile products were separated by fractional condensation through traps kept at -95, -126, -142, and -210 °C. On the basis of their infrared and ¹⁹F NMR spectra the following products were collected in these traps: -210 °C, NF₃ and a trace of TeF₆; -142 °C, TeF₆ and TeF₅OF in a mole ratio of about 3:1; -126 °C, HF and some TeF₅OF; -95 °C, TeF₅OH and some HF. The white solid residue (0.57 g) decomposed during an attempt to transfer it at ambient temperature to a drybox for further characterization. The overall yield of TeF₅OF was estimated to be about 10–20% with TeF₆ and unreacted TeF₅OH being the principal products.

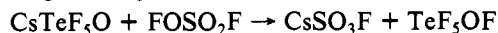
Results and Discussion

Synthesis of TeF₅OF. By analogy to previous attempts^{2,18} to synthesize TeF₅OF from either Hg(TeF₅O)₂ or CsTeF₅O and F₂, the fluorination of either CsTeF₅O or KTeF₅O with F₂ at -45 to -10 °C was unsuccessful and resulted only in TeF₆ formation. Since the decomposition of NF₄XO salts had recently been shown to provide new high-yield syntheses for hypofluorites such as FOClO₃,¹⁹ FOSO₂F,²⁰ and FOIF₄O,⁴ the synthesis of NF₄TeF₅O by metathesis of NF₄SbF₆ and CsTeF₅O in anhydrous HF was attempted. This attempt, however, was preempted by the fact that CsTeF₅O was found to react with anhydrous HF, undergoing a displacement reaction. Recent work¹⁷ in our laboratory had shown that even in cases of Lewis acids that are weaker than HF their NF₄⁺ salts can be prepared by treating NF₄HF₂·*n*HF with this acid. Therefore, this approach was studied for NF₄TeF₅O. Although the NF₄TeF₅O salt itself could not be isolated, it was found that TeF₅OH (which is equivalent to an equimolar mixture of the Lewis acid TeF₄O and HF) reacted with NF₄HF₂·*n*HF at -23 °C to produce TeF₅OF in moderate yield:



Since TeF₆ was the major product, we prefer to interpret this reaction in terms of a fluorination of TeF₅OH by nascent fluorine formed in the decomposition of NF₄HF₂, rather than in terms of a decomposition of an unstable NF₄TeF₅O intermediate. In the latter case, we would expect a near-quantitative yield of TeF₅OF.

A more facile high-yield synthesis of TeF₅OF was discovered by reacting CsTeF₅O with FOSO₂F at -45 °C:



This reaction represents a new synthetic route to hypofluorites. On the basis of the general usefulness of the analogous ClO-SO₂F reagent for the syntheses of hypochlorites,²¹ FOSO₂F may be similarly useful for the synthesis of hypofluorites.

When the synthesis of TeF₅OF from CsTeF₅O and FOSO₂F was carried out above -45 °C, the amount of TeF₆ byproduct sharply increased. For example, at -10 °C and with a reaction time of 7 days, the TeF₆ to TeF₅OF ratio in the product increased to 1:1. The use of an excess of CsTeF₅O in this reaction was found advantageous for the product purification since it eliminates the need for separating TeF₅OF from FOSO₂F.

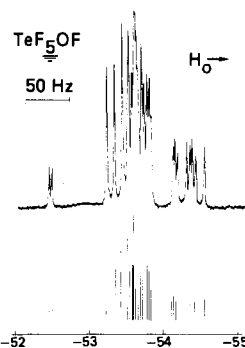


Figure 1. Observed and calculated ¹⁹F NMR spectra of the AB₄ part of TeF₅OF.

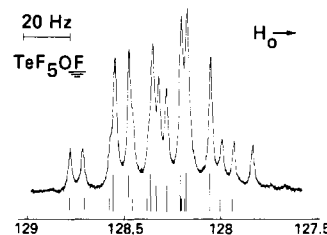


Figure 2. Observed and calculated ¹⁹F NMR spectra of the X part of TeF₅OF.

Table I. Mass Spectrum of TeF₅OF Compared to Those of TeF₅OCl and TeF₅OH

TeF ₅ OF		TeF ₅ OCl		TeF ₅ OH	
assignt	intens	assignt	intens	assignt	intens
TeF ₅ OF ⁺	vw	TeF ₅ OCl ⁺	vww	TeF ₅ OH ⁺	w
TeF ₄ OF ⁺	} vvw	TeF ₄ OCl ⁺	vw	TeF ₄ OH ⁺	vw
TeF ₃ O ⁺					
TeF ₅ ⁺	s	TeF ₄ ⁺	s	TeF ₄ ⁺	vs
TeF ₄ ⁺	w	TeF ₄ ⁺	vw	TeF ₄ ⁺	w
TeF ₃ O ⁺	m	TeF ₃ O ⁺	ms	TeF ₃ O ⁺	s
TeF ₃ ⁺	vs	TeF ₃ ⁺	vs	TeF ₃ ⁺	vs
TeF ₂ ⁺	m	TeF ₂ ⁺	m	TeF ₂ ⁺	m
TeFO ⁺	vw	TeFO ⁺	w	TeFO ⁺	w
TeF ⁺	w	TeF ⁺	w	TeF ⁺	w
Te ⁺	w	Te ⁺	w	Te ⁺	w

Properties of TeF₅OF. This compound is colorless as a gas and liquid. Its vapor pressure–temperature relationship for the range -79 to -23 °C is given by the equation

$$\log[P \text{ (mm)}] = 6.9022 - 1101.2/[T \text{ (K)}]$$

The extrapolated boiling point is 0.6 °C. The derived heat of vaporization is $\Delta H_{\text{vap}} = 5039 \text{ cal mol}^{-1}$ and the Trouton constant is 18.4, indicating little or no association in the liquid phase. Vapor density measurements showed that in the gas phase the compound is also not associated. We were not able to observe a sharp melting point for TeF₅OF because our samples showed a tendency to form a glass near -80 °C. The compound appears to be completely stable at ambient temperature and has been stored in stainless steel cylinders for more than 4 months without any sign of decomposition.

¹⁹F NMR Spectrum. The ¹⁹F NMR spectrum of TeF₅OF in CFCl₃ solution at 28 °C is shown in Figures 1 and 2 and is characteristic for a second-order AB₄X spin system. A computer-aided analysis of the spectrum resulted in the following parameters: $\phi^*(A) = -52.5$, $\phi^*(B_4) = -54.0$, $\phi^*(X) = 128.3$, $J_{AB} = 180 \text{ Hz}$, $J_{AX} = 4.9 \text{ Hz}$, $J_{BX} = 19.0 \text{ Hz}$, $R = 1.20$, $J_{125\text{-Te}^{19}\text{F}} = 3800 \text{ Hz}$. These values are in excellent agreement with those found for numerous other covalent TeF₅O-type compounds.²²

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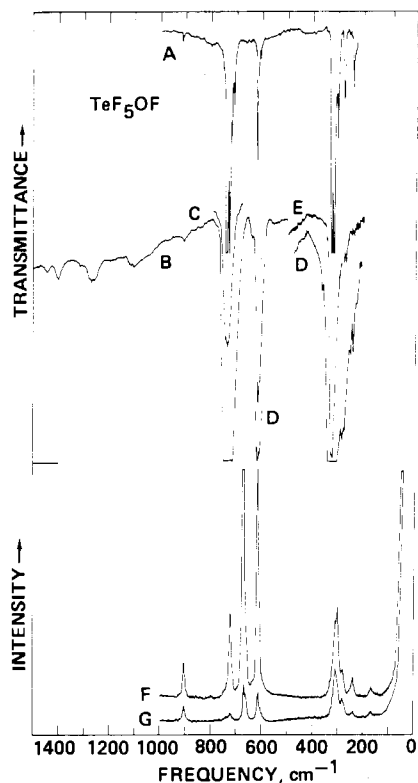


Figure 3. Vibrational spectra of TeF_5OF : trace A, infrared spectrum of TeF_5OF isolated in a neon matrix (mole ratio 400:1) and recorded at 6 K; traces B and C, infrared spectra of the gas, recorded at pressures of 74 and 3 mm, respectively, in a 5-cm path length cell equipped with AgCl windows (the very weak bands at 1272, 1105, 640, and 548 cm^{-1} in spectrum B are due to a trace of FCIO_2 resulting from the ClF_3 used for passivation); traces D and E, infrared spectra of the gas, recorded at pressures of 86 and 8 mm, respectively, in a 10-cm path length cell equipped with polyethylene windows and with polyethylene windows in the reference beam; traces F and G, Raman spectra of the liquid, recorded in 3 mm o.d. quartz tubes at $-55\text{ }^\circ\text{C}$ with the incident polarization parallel and perpendicular, respectively.

Table II. Vibrational Spectra of TeF_5OF

obsd freq, cm^{-1} (rel intens ^a)				
IR		Raman		assign ^b
gas	Ne matrix	liquid, $-55\text{ }^\circ\text{C}$	solid, $-110\text{ }^\circ\text{C}$	
1800 vw				$2\nu_{12}$
1449 vw				$2\nu_1$
1403 w				$\nu_2 + \nu_8$
908 vw		905 (0.4) p	904 (0.8)	ν_{12}
738 vs	738 ^c vs	738 sh, dp	735 sh	ν_8
	727 vs	721 (1.1) p	721 (1.3)	ν_1
718 vw				} impurity?
709 vw				
668 vw		669 (10) p	670 (10)	ν_2
		660 (0.3) dp	662 sh	ν_5
616 m	618 m	613 (3.8) p	613 (4)	ν_3
324 vs	327 vs	325 sh, dp	325 sh	ν_9
	318 vs		319 sh	ν_{10}
	308 vw	309 (1.0) dp	309 (1.6)	ν_7
300 sh	302 m	301 (0.5) p	301 sh	ν_4
280 mw	278 m	279 (0.2) dp	279 (0.2)	ν_{11}
241 mw	239 mw	240 (0.2) p	240 (0.2)	ν_{13}
		166 (0.1) dp	167 (0.1)	ν_{14}

^a Uncorrected Raman intensities (peak heights). ^b For mode description see Table IV. ^c Band shows tellurium isotope fine structure with splittings of about 1.30 cm^{-1} .

Mass Spectrum. The mass spectrum of TeF_5OF is listed in Table I together with the spectra of TeF_5OCl and TeF_5OH , which were measured for comparison. All of the listed frag-

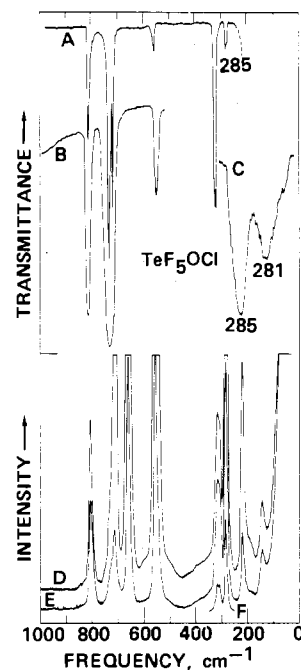


Figure 4. Vibrational spectra of TeF_5OCl : traces A and C, infrared spectra of TeF_5OCl isolated in a neon matrix (mole ratio 400:1) at 6 K; trace B, infrared spectrum of the gas, recorded at a pressure of 27 mm in a 5-cm path length cell equipped with AgCl windows; traces D and E, Raman spectra of the liquid, recorded in 3 mm o.d. quartz tubes at $-80\text{ }^\circ\text{C}$ with the incident polarization parallel and perpendicular, respectively; trace F, spectrum recorded under the same conditions as for trace D, except for a narrower slit width.

Table III. Vibrational Spectra of TeF_5OCl

obsd freq, cm^{-1} (rel intens ^a)			
IR		Raman liquid, $-80\text{ }^\circ\text{C}$	assign ^b
gas	Ne matrix		
1365 vw			$\nu_3 + \nu_{12}$
812 s	814 s 811 s	809 (0.9) p	ν_{12}
732 vs	732 vs ^c	730 sh, dp	ν_8
	718 s	713 (1.6) p	ν_1
		663 (10) p	ν_2
551 m		655 sh, dp	ν_5
	558 m	554 (6.5) p	ν_3
	327 vs	328 sh, dp	ν_9
	322 vs	316 (0.8) dp	ν_{10}
		308 (0.8) dp	ν_7
	285 m		ν_{11}
	281 m	281 (2.8) p	ν_4
		218 (1.1) p	ν_{13}
		141 (0.3) dp	ν_{14}

^a Uncorrected Raman intensities. ^b For mode description see Table IV. ^c Band shows fine structure with splittings of about 1.30 cm^{-1} due to tellurium isotopes.

ments showed the characteristic tellurium isotope pattern, and therefore the individual *m/e* listings were omitted for simplicity. The spectra of all three compounds show weak parent ions and TeF_3^+ as the base peak.

Vibrational Spectra of TeF_5OF and TeF_5OCl . The infrared spectra of gaseous and of neon-matrix-isolated TeF_5OF and the Raman spectra of liquid and solid TeF_5OF were recorded (see Figure 3), and the observed frequencies are summarized in Table II. Since the assignments previously reported²³ for TeF_5OCl could not be reconciled with our results for TeF_5OF , the vibrational spectra of TeF_5OCl were also recorded (see Figure 4 and Table III). The following deviations from the

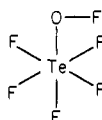
Table IV. Vibrational Spectra of TeF₅OF and TeF₅OCl and Their Assignment Compared to Those of TeF₅Cl

assignt	approx descriptn of mode	obsd freq, cm ⁻¹ (rel intens ^a)							
		TeF ₅ Cl ^b		TeF ₅ OCl		TeF ₅ OF			
		IR (gas)	Raman (liquid)	IR (gas, matrix)	Raman (liquid)	IR (gas, matrix)	Raman (liquid)		
C _{4v}	A ₁ ν ₁	ν(TeF)	711 sh, m	708 (3.1) p	718 s	713 (1.6) p	727 vs	721 (1.1) p	
	ν ₂	ν _s (TeF ₄)	662 vw	659 (10) p		663 (10) p		669 (10) p	
	ν ₃	ν(TeX)	411 ms	413 (7.7) p	551 m	554 (6.5) p	616 m	613 (3.8) p	
	ν ₄	δ _s (TeF ₄)	317 s	312 (0.8) p	281 m	281 (2.8) p	301 m	301 (0.5) p	
	B ₁	ν ₅	ν _s (TeF ₄)		651 (0.8) dp		655 sh, dp		660 (0.3) dp
		ν ₆	δ(TeF ₄)						
	B ₂	ν ₇	δ _{sciss} (TeF ₄)		302 (0.5) dp		308 (0.8) dp		309 (1.0) dp
	E	ν ₈	ν _{as} (TeF ₄)	726 vvs	726 (0.6) dp	732 vs	730 sh, dp	738 vs	738 sh, dp
		ν ₉	δ(FTeF ₄)	325 ms	327 (0.9) dp	327 vs	328 sh, dp	327 vs	325 sh, dp
		ν ₁₀	δ(XTeF ₄)		167 (1.8) dp	322 vs	316 (0.8) dp	318 vs	(309-325)
		ν ₁₁	δ _{as} (TeF ₄)	259 m	259 (1.7) dp	285 m		280 mw	279 (0.2) dp
C _s	A' ν ₁₂	ν(XY)			812 s	809 (0.9) p	908 vw	905 (0.4) p	
	A'' ν ₁₃	δ(TeXY)				218 (1.1) p	240 mw	240 (0.2) p	
	ν ₁₄	τ(TeXY)				141 (0.3) dp		166 (0.1) dp	

^a Uncorrected Raman intensities (peak heights). ^b Data from ref 24.

previous literature data²³ were observed. (i) The infrared spectrum of the gas does not exhibit a very strong band at 708 cm⁻¹. Although our Ne-matrix spectra show the presence of two intense bands at 732 and 718 cm⁻¹, respectively, their frequencies are too close to result in two separate bands in the gas-phase spectrum. (ii) In the Raman spectrum of the liquid the 141-cm⁻¹ band is depolarized and the 809-cm⁻¹ band is polarized. (iii) The infrared spectrum of the neon-matrix sample shows the presence of two fundamental vibrations in the 280-cm⁻¹ region (see trace C of Figure 4).

Using the well-established²⁴ assignments of TeF₅Cl and the revised experimental data of TeF₅OCl for comparison, we can readily assign the vibrational spectra of TeF₅OF (see Table IV), assuming a model with C_{4v} symmetry for the TeF₅O part and C_s symmetry for the TeOF part of the molecule:



Except for the symmetric out of phase, out of plane TeF₄ deformation mode in species B₁, which is usually not observed for pseudooctahedral molecules and is inactive under O_h symmetry, all fundamentals expected for the above C_{4v}-C_s model were observed. The assignments (see Table IV) are straightforward and show for the three molecules almost identical frequencies for the TeF₅ part of the molecules. The modes involving the XY group of this TeF₅XY molecule show the expected mass effects for different X and Y groups. Since

the Te-O stretching mode is expected to couple strongly with the O-Hal stretch and to couple moderately with δ_s(TeF₄) (A₁),²⁵ these modes also exhibit a mass effect.

Comparison of the assignments of Table IV with those previously given²³ for TeF₅OCl shows that with the exception of ν₇ (B₂) and ν₁₃ all the previously given assignments for the deformation modes should be revised. Since a thorough normal-coordinate analysis has previously been carried out²⁴ for TeF₅Cl and since the TeF₅Cl and TeF₅XY spectra are similar, a normal-coordinate analysis of the latter molecules appears unwarranted.

Conclusion. The results of this study show that FOSO₂F is a useful reagent for the synthesis of hypofluorites.²⁶ Furthermore, it is shown that TeF₅OF, as expected from comparison with TeF₅OCl, TeF₅OBr, and FOIF₄O, indeed exists and is a stable molecule. The TeF₅OF molecule was characterized, and the vibrational assignments were made for TeF₅OF and TeF₅OCl.

Acknowledgment. The authors are grateful to R. D. Wilson for his help in some of the experiments, to L. R. Grant for helpful discussions, and to K. Seppelt for a sample of TeF₅OH used in the initial part of this work. This work was financially supported by the Air Force Office of Scientific Research, the Office of Naval Research, and the Army Research Office.

Registry No. TeF₅OH, 57458-27-2; CsTeF₅O, 19610-48-1; KTeF₅O, 19610-51-6; TeF₅OF, 83314-21-0; FOSO₂F, 13536-85-1; NF₄HF₂, 71485-49-9; TeF₅OCl, 41524-13-4.

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