the data for the M(III) states show considerably more scatter than that for M(IV) systems. The slopes of the $\chi_{\rm opt}$ *vs.* q plots are 0.313 (9.4 kK) and 0.165 unit (4.95 kK) which, as for the two 3d oxidation states,[5] are consistent with their interpretation[34] as $(E - A)$ (see Figure 2). We have moreover previously drawn attention[5,6] to the strong correlation which exists between optical electronegativity and the nephelauxetic ratio, and the theoretical basis for this.[14] A similar trend also occurs in the 4d series, as shown in Figure 3, and the data are thus consistent with the earlier results and the interpretation suggested.[5,6,14]

Registry No. $KRuF_6$, 40902-32-7; K_2RuF_6, 40902-49-6; K_3RuF_6, 40902-50-9; Cs_2RhF_6, 16962-25-7.

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Raman Spectra of Fluoro Fluorosulfuryl Peroxide and Trifluoromethyl Fluorosulfuryl Peroxide

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The liquid-phase Raman spectra of $FOOSO_2F$ and CF_3OOSO_2F are reported in the region 2000–0 cm^{-1}. The infrared spectra are also reinvestigated in the region 2000–400 cm^{-1} for the gaseous compounds. Vibrational analyses are made for both molecules based on a description of the normal modes of vibration by the characteristic group vibrations. The O–O stretching vibrations are observed at 890 and 863 cm^{-1} for $FOOSO_2F$ and CF_3OOSO_2F, respectively.

Introduction

The thorough study of the vibrational spectrum of CF_3OOCF_3 by Durig and Wertz[2a] has shown that Raman spectroscopy is a powerful tool in elucidating the O-O stretching frequency and thus the identification of the peroxy linkage in peroxides. While the O-O stretching vibration often gives rise to a weak absorption band in the infrared spectrum, it is usually the most intense band and is highly polarized in the Raman spectrum.

Relatively few inorganic peroxides are known and many of these have not been characterized spectroscopically. While hydrogen peroxide has been examined in detail, extensive vibrational spectral studies are limited to the symmetrical fluorinated peroxides CF_3OOCF_3,[2a] FSO_2OOSO_2F,[2b] and FOOF.[3] The O-O stretching frequency has been identified by Fox, *et al.*,[4] in a number of fluorinated peroxides, by Christe in a vibrational study of dimethyl peroxides,[5] by Aubke and coworkers in a study of the vibrational spectra of bis(fluorosulfuryl) peroxide,[2b] and by us in a vibrational study of bis(pentafluorosulfur) peroxide and fluorosulfuryl pentafluorosulfur peroxide.[6] Virtually no spectroscopic or structural information is available for the mixed, nonsymmetrical derivatives of these peroxides.

This study reports the Raman spectra of the unsymmetrical fluorinated peroxides $FOOSO_2F$ and CF_3OOSO_2F. Only brief reports of their gaseous infrared spectra in the sodium chloride region have appeared.[7,8] A comparison of the two related molecules has provided a basis for detailed vibrational analyses.

Experimental Section

Fluoro fluorosulfuryl peroxide was prepared by the reaction of OF_2 and SO_3 (Allied Chemical Co.)[7] and purified by trap-to-trap distillation. Trifluoromethyl fluorosulfuryl peroxide was prepared by the reaction of $CsOCF_3$ and $S_2O_6F_2$.[9] $S_2O_6F_2$ was synthesized by catalytic fluorination of SO_3,[10] while $CsOCF_3$ was prepared by the reaction of CsF and COF_2 in the presence of acetonitrile.[11] Fractionation of the products yielded large amounts of CF_3OOSO_2F, contaminated with unreacted $S_2O_6F_2$. The purification of CF_3OOSO_2F was accomplished by the addition of elemental iodine, which converted the $S_2O_6F_2$ to the nonvolatile $I(OSO_2F)_3$, followed by trap-to-trap distillation.

Infrared spectra were recorded on a Perkin-Elmer 457 grating spectrometer using a 10-cm gas cell fitted with potassium bromide cell windows (Harshaw Chemicals).

The Raman spectra were measured with a Jarrell Ash 25-300 Raman spectrometer using a Spectra-Physics 250 helium-neon laser for excitation at 6328 Å. The scattered radiation was detected at right angles to the incident beam (transverse viewing axial excitation) by means of a thermoelectrically cooled S-20 photomultiplier tube and photon counting system. Both $FOOSO_2F$ (bp 0°) and CF_3OOSO_2F (bp 12°) were contained as liquids in 5-mm o.d. "optically flat" Pyrex cells at room temperature.

The spectrometer was calibrated from helium-neon plasma emission lines, and the accuracy is estimated to be ±1 cm^{-1}. Depolarization measurements were made by turning the direction of the electric vector of the incident light through 90° by means of a 45° rotation of a half-wave plate. Intensity values were obtained by measuring the area under the peaks.

Results

The Raman spectra of $FOOSO_2F$ and CF_3OOSO_2F as obtained under instrumental conditions of low sensitivity are shown in Figure 1. The observed frequencies, relative intensities, and depolarization ratios are tabulated in Tables I and II for $FOOSO_2F$ and CF_3OOSO_2F, respectively. The gas-phase infrared spectra are given for the region 2000-400 cm^{-1} and were found to be in excellent agreement with the

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Table I. Raman and Infrared Spectra of $FOOSO_2F^a$

Raman (liquid)			Infrared (gas)		
Frequency, cm^{-1}	Rel intensity	ρ	Frequency, cm^{-1}	Intensity	Assignment
1550	1				O_2
1496	6	0.67	1500	s	SO_2 stretch, ν_1
1269	42	0.06			SO_2F_2
1250	100	0.04	1252	s	SO_2 stretch, ν_2
			895	w, sh	510 + 392
890	59	0.08	885	w, sh	O–O stretch, ν_3
880 sh	37	0.12			SO_2F_2
855 sh	31				SO_2F_2
850	51	0.10	855	s	S–F stretch, ν_4
782	44	0.09	780	w–m	S–O stretch, ν_5
738	36	0.05			O–F stretch, ν_6
720	27	0.10	720	s	SO_2 rock, ν_7
595	4		590	w–m	SO_2 bend, ν_8
548	19	0.56	545	w–m	SO_2 rock, ν_9
510	12	0.66	510	w–m	SF wag, ν_{10}
478	13	0.34			SF wag, ν_{11}
470	8	0.32			
392	36	0.42			SO_2F torsion, ν_{12}
324	27	0.33			
308	66	0.30			SOO bend, ν_{13}
253	19	0.30			FOO bend, ν_{14}
242 sh	10	0.32			
125	30	0.62			FO–O torsion, ν_{15}

[a] sh = shoulder, w = weak, m = medium, s = strong, br = broad, ρ = depolarization ratio.

previous reports for the sodium chloride region.[7,8] Assignments of the observed frequencies to the normal vibrational modes are also presented in Tables I and II.

Vibrational Assignments. The molecular structures of $FOOSO_2F$ and CF_3OOSO_2F are unknown, and their reaction chemistry is relatively unexplored. Nevertheless, both peroxides are likely to have nonplanar configurations similar to hydrogen peroxide with the SO_2F and the F or CF_3 groups adjacent to the peroxy linkage in staggered positions. This has been the case for the symmetrical peroxides FOOF,[12] CF_3OOCF_3,[2a] and FSO_2OOSO_2F.[2b] Thus, both $FOOSO_2F$ and CF_3OOSO_2F will possess no symmetry elements and belong to the point group C_1. For this case, all normal modes of vibration are both infrared and Raman active, and all lines are polarized in the Raman effect. This is observed.

The normal modes of vibration are approximated for each molecule by the characteristic group vibrations. The molecules are discussed separately for convenience.

1. $FOOSO_2F$. The 15 vibrational modes of $FOOSO_2F$ can be roughly described as nine SO_2F vibrations; O-F, O-O, and S-O stretching vibrations; SOO and FOO deformations, and a torsional oscillation about the FO-O bond.

Characteristic of covalent sulfuryl compounds[13] are the bands at 1496 and 1250 cm^{-1}, which are assigned to the SO_2 asymmetric and symmetric stretches. Other bands are seen also in this region at 1269 and 1550 cm^{-1}. These are due to SO_2F_2 and O_2, respectively, which arise from the partial decomposition of $FOOSO_2F$ according to

$$FOOSO_2F \rightarrow SO_2F_2 + O_2$$

The presence of molecular oxygen in the sample is seen more clearly in Figure 2. Since $FOOSO_2F$ has been reported to be thermally stable to 50°,[14] the decomposition of $FOOSO_2F$ must be produced by interaction of the sample with the laser beam. Two shoulder bands at 880 and 855 cm^{-1} are also attributed to SO_2F_2.

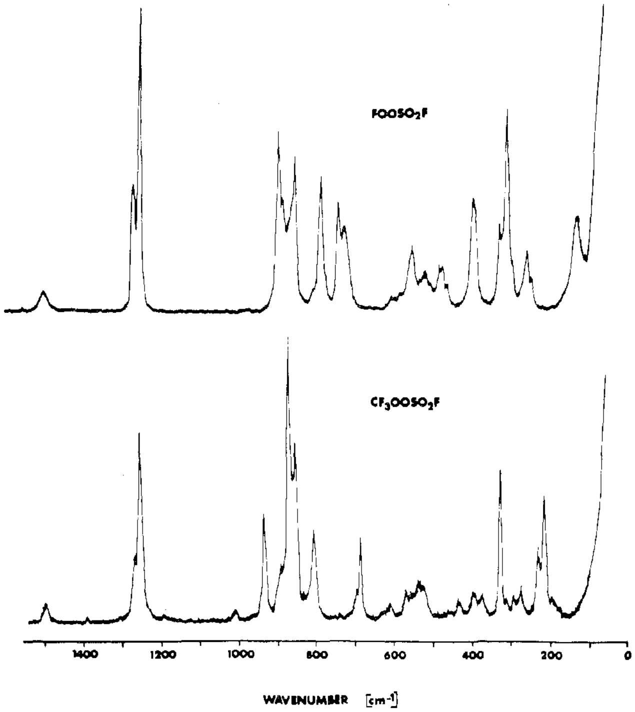

Figure 1. The Raman spectra of $FOOSO_2F$ and CF_3OOSO_2F (1600-0 cm^{-1}): spectral slit width, 5 cm^{-1}; time constant, 0.5 sec; scan speed, 50 cm^{-1}/min; photon counting range, 5 × 10^3 cps ($FOOSO_2F$) and 2 × 10^3 cps (CF_3OOSO_2F).

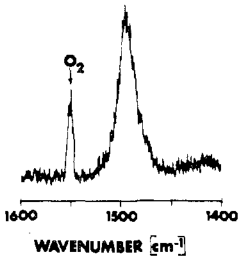

Figure 2. The Raman spectra of $FOOSO_2F$ in the region 1600-1400 cm^{-1}: spectral slit width, 7 cm^{-1}; time constant, 0.5 sec; scan speed, 50 cm^{-1}/min; photon counting range, 1 × 10^3 cps.

Five bands in the region, 900-700 cm^{-1}, are observed at 890, 850, 782, 738, and 720 cm^{-1}. The assignment of the first three bands is relatively easy. The strongest band in this region at 890 cm^{-1}, but which occurs in the infrared as a weak band, is assigned to the O-O stretch. Bands at 850 and 782 cm^{-1} are assigned to the S-F and S-O stretches, respectively, and are similar to those observed for $FOSO_2F$.[2b] The assignment of the two remaining bands at 738 and 720 cm^{-1} to the O-F stretch and one of the SO_2 rocks, respectively, is tentative. While the O-F stretching vibration is normally observed at much higher frequencies, *e.g.*, 880 cm^{-1} in $FOSO_2F$,[2b] it has been assigned to a strong band at 755 cm^{-1} in the infrared spectrum of $FOOCF_3$.[15] The assignment of one of the SO_2 rocks at 720 cm^{-1} also receives some justification from the reported vibrational analysis of $S_2O_5F_2$,[16] where a band seen in both the infrared and Raman at 733 cm^{-1} is assigned to an SO_2 rock. For the case of $S_2O_5F_2$, the observation of an SO_2 rock at relatively high frequencies results from the coupling of the two SO_2F groups. It seems reasonable that the vibrations of $FOOSO_2F$ are also coupled.

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Table II. Raman and Infrared Spectra of $CF_3OOSO_2F^a$

Raman (liquid)			Infrared (gas)		
Frequency, cm^{-1}	Rel intensity	ρ	Frequency, cm^{-1}	Intensity	Assignment
1930	5				COF_2
1494	7	0.65	1490	s	SO_2 stretch, ν_1
1385	1				
1300	1		1300	s	CF_3 stretch, ν_2
1262	22	0.08	1260 sh		SO_2F_2 + CF_3 stretch,^{b} ν_3
1250	67	0.11	1250 br	s	SO_2 stretch, ν_4
1190	1		1190	s	CF_3 stretch, ν_5
1002	4				
929	34	0.48	928	m	CO stretch, ν_6
885	19	0.07			SO_2F_2
863	100	0.09			O–O stretch, ν_7
848	62	0.04	853	s	SF stretch, ν_8
800	30	0.09	803	m–s	SO stretch, ν_9
690 sh	11	0.12			SO_2 rock, ν_{10}
680	16	0.13	680	m	CF_3 deformation, ν_{11}
625 sh	2				CF_3 deformation, ν_{12}
605	4	0.29	602	w–m	SO_2 bend, ν_{13}
562	10	0.71	565	w–m	CF_3 deformation, ν_{14}
532	15	0.65	532	w–m	SO_2 rock, ν_{15}
518	11	0.60	518	w–m	SF wag, ν_{16}
428	6	0.05			CF_3 rock, ν_{17}
390	8	0.56			SO_2F torsion, ν_{18}
365	8	0.33			SF wag, ν_{19}
322	52	0.40			SOO bend, ν_{20}
285	7	0.29			CF_3 rock, ν_{21}
268	11	0.25			
226	25	0.29			O–O torsion, ν_{22}
210	29	0.29			COO bend, ν_{23}
190	7	0.40			Impurity?
180 sh	3	0.33			
Not observed					CF_3 torsion, ν_{24}

[a] sh = shoulder, w = weak, m = medium, s = strong, br = broad, ρ = depolarization ratio. [b] SO_2F_2 impurity in the Raman only; CF_3 stretch in both ir and Raman.

For example, there is likely to be coupling between the O–F and O–O stretches.

Assignments for the remaining SO_2F vibrations are made by analogy to be reported spectra of related molecules containing the fluorosulfate group.[2b,6,16–19] These are as follows: SO_2 bend at 595 cm^{-1}, SO_2 rock at 548 cm^{-1}, SF wags at 510 and 478 cm^{-1}, and SO_2F torsion at 392 cm^{-1}.

The SOO bend is assigned to the relatively strong band at 308 cm^{-1} in analogy with a similar assignment of the SOO bend at 307 cm^{-1} in $S_2O_6F_2$.[2] The FOO bending and the FO–O torsion vibrations are assigned to the bands at 253 and 125 cm^{-1}, respectively. These compare favorably to the assignments of the S–O–F deformation and the S–OF torsional modes in $FOSO_2F$.[2b]

The assignment of the 15 normal modes of vibration for $FOOSO_2F$ is summarized in Table I using the ν_n symbolism. The cause for the splittings of ν_{11}, ν_{13}, and ν_{14} is uncertain.

2. **CF_3OOSO_2F.** The Raman spectrum of CF_3OOSO_2F shows considerable similarity to the spectrum of $FOOSO_2F$. The 24 normal modes of vibration are best approximated in terms of the following group vibrations: nine SO_2F vibrations; nine CF_3 vibrations; C–O, O–O, and S–O stretching vibrations; COO and SOO deformations; and a torsional oscillation about the O–O bond.

Prior to the measurement of the Raman spectrum, the sample of CF_3OOSO_2F showed no bands in the infrared spectrum attributable to impurities. However, bands in the Raman appear at 1930 cm^{-1} (COF_2) and at 1262 and 885 cm^{-1} (SO_2F_2) as a result of the decomposition of CF_3OOSO_2F in the laser beam which proceeds in a manner analogous to $FOOSO_2F$

$$CF_3OOSO_2F \rightarrow COF_2 + SO_2F_2 + {}^1/_2O_2$$

The CF_3 vibrations are most easily assigned since considerable information is available on vibrational spectra of molecules containing the CF_3 moiety.[2a,20–22] The three CF_3 stretching vibrations seen as very weak bands in the Raman are assigned to strong bands in the infrared at 1300, 1260, and 1190 cm^{-1}. The three CF_3 deformations are observed at the frequencies 680, 625, and 562 cm^{-1}. The assignment of the two CF_3 rocking modes is less certain. A comparison with the vibrational spectra of CF_3OOCF_3[2a] and CF_3OOOCF_3[20] suggests the assignment of the CF_3 rocking modes to a weak absorption band at 428 cm^{-1} and a band split at 285 and 268 cm^{-1}. As no bands are seen below 180 cm^{-1}, an assignment for the CF_3 torsional mode, expected to occur at frequencies below 100 cm^{-1},[2a] cannot be made.

The nine SO_2F vibrations in CF_3OOSO_2F, observed at frequencies very close to $FOOSO_2F$, are assigned as follows: SO_2 stretches at 1494 and 1250 cm^{-1}, SF stretch at 848 cm^{-1}, SO_2 rocks at 690 and 532 cm^{-1}, SO_2 bend at 605 cm^{-1}, SF wags at 518 and 365 cm^{-1}, and the SO_2F torsion at 390 cm^{-1}.

It now remains to assign the COOS skeletal vibrations. The CO and SO stretches are seen at 929 and 800 cm^{-1}, respectively. The strongest band in the Raman spectrum

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at 863 cm^{-1} which is highly polarized ($\rho = 0.09$), but is unobserved in the infrared, is clearly the O-O stretching vibration. Thus, the O-O stretching frequency in CF_3OOSO_2F lies between that of CF_3OOCF_3 at 886 cm^{-1} [2a] and FSO_2OOSO_2F at 801 cm^{-1}.[2b] The characteristic absorption band due to the SOO bending vibration is seen at 322 cm^{-1}.

The assignment of the COO bending and O-O torsional modes is difficult due to the unexpected number of bands below 250 cm^{-1}. The two strongest bands at 226 and 210 cm^{-1} are assigned to the torsion and COO bending modes, respectively. The weaker bands at 190 and 180 cm^{-1} may be due to impurities, but no certain explanation can be suggested.

Discussion

Fluoro fluorosulfuryl peroxide, $FOOSO_2F$, is formally derived from the symmetrical peroxides FOOF and FSO_2OOSO_2F. The O-O bond is FSO_2OOSO_2F is rather easily cleaved to form fluorosulfate radicals,[23-25] and the O-O stretching frequency is found at 801 cm^{-1}.[2b] In contrast, the O-O bond in O_2F_2 is much stronger as a result of $[\pi^*\text{-p}]$ σ bonding[26] and the O-O stretching frequency is observed at 1306 cm^{-1}. The reactions of O_2F_2 undoubtedly involve the breaking of O-F bonds,[27-29] as a result of the O-O bond energy being much greater than the OF bond energy ($\Delta H_{\text{diss}} = 103.5$ *vs.* 18 kcal/mol).[30]

The chemistry of $FOOSO_2F$ has not been well investigated. While esr studies show that $FSO_2\cdot$ and $\cdot OOF$ radicals are formed on photolysis of $FOOSO_2F$,[31] the mass spectrum indicates that the O-O bond, not the S-O bond, is more easily ruptured.[2a] The chemistry of CF_3OOSO_2F remains to be elucidated.

The O-O stretching frequencies for $FOOSO_2F$ and CF_3OOSO_2F fall in the region 850-900 cm^{-1} suggesting, coupling of the O-O stretch with other normal modes not withstanding, that the O-O bond energies are similar to that of hydrogen peroxide. While the O-O bonds are expected to be stronger than the O-O bond in FSO_2OOSO_2F, the O-O bond dissociation energies are probably much lower than that for O_2F_2. These conclusions are in agreement with the ^{19}F nmr chemical shifts reported for $FOOSO_2F$ and O_2F_2 at -292 ppm[7,27] and <-800 ppm,[32] respectively (relative to $CFCl_3$). The abnormally low O-O stretching frequencies observed for SF_5OOSF_5 and SF_5OOSO_2F are attributed to extensive vibrational coupling rather than the existence of weak O-O bonds.[6] Some O-O stretching frequencies as determined by Raman studies and given for comparison are: FOOF, 1306 cm^{-1};[33] HOOH, 880 cm^{-1};[34] DOOD, 880 cm^{-1};[34] CF_3OOCF_3, 886 cm^{-1};[2a] CF_3OOSO_2F, 863 cm^{-1}; $FOOSO_2F$, 890 cm^{-1}; FSO_2OOSO_2F, 801 cm^{-1};[2b] SF_5OOSF_5, 751 cm^{-1};[6] SF_5OOSO_2F, 740 cm^{-1}.[6]

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Synthesis and Characterization of Oxotetrafluorotellurate(IV) and Dioxodifluorotellurate(IV) Compounds

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The synthesis and characterization by X-ray powder photography and Raman and infrared spectroscopy of M_2TeOF_4 and $M_2TeO_2F_2$ compounds are described. The M_2TeOF_4 compounds are shown to be isomorphous with M_2SbF_5 compounds. The unit cells of M_2TeOF_4 compounds are shown to be nearly the same as those of the comparable M_2SbF_5 compounds indicating that the anion volumes are very similar. The vibrational spectra of the $M_2TeO_2F_2$ compounds show evidence of oxygen bridging. The infrared and Raman spectra are shown to be consistent with a C_{4v} $TeOF_4^{2-}$ ion and a C_{2v} $TeO_2F_2^{2-}$ ion with oxygens in equatorial positions. Attempted preparations of M_2TeF_6 and $MTeO_2F$ compounds are described.

Introduction

Tellurium normally exhibits a higher coordination number than sulfur and selenium and this is reflected in the compounds of these elements in their highest oxidation state. The six valent oxy acids of S and Se are four-coordinate while that of Te is six-coordinate. The hexafluorides of S and Se show poor acceptor properties while TeF_6 forms adducts which may contain the TeF_7^- and TeF_8^{2-} ions.[1] The stable oxyfluoro anions are SO_3F^-, SeO_3F^-,[2] and

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