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REACTIONS OF $CF_3SF_3{=}NCF_3$ WITH NUCLEOPHILES AND THE PREPARATION OF $CF_3SF_4N(F)R_f$ ($R_f = CF_3$, C_2F_5)

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To Professor George H. Cady on his 70th birthday

SUMMARY

The new compounds, $CF_3SF(O){=}NCF_3$ and $CF_3SF({=}NCH_3){=}NCF_3$, result from nucleophilic displacement reactions of $CF_3SF_3{=}NCF_3$ with water and methylamine, respectively. Higher yields of the di-imine are obtained when $CH_3N(Si(CH_3)_3)_2$ is substituted for CH_3NH_2. Stable fluoroamines, $CF_3SF_4N(F)R_f$ ($R_f = CF_3$, C_2F_5) are prepared by fluorination of the respective amines, $CF_3SF_4N(H)R_f$, with silver difluoride or by the saturation of the double bonds in $CF_3SF_3{=}NCF_3$ and $CF_3SF_4N{=}CFCF_3$ with elemental fluorine in the presence of CsF. The chemical stability of the fluoroamines is significantly greater than that of either the chloroamines or amines.

INTRODUCTION

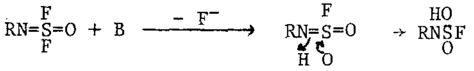
Recently we reported the synthesis of a new compound, $CF_3SF_3NCF_3$ [1], in which the geometry around the sulfur, based on nmr data, was assumed to be distorted trigonal bipyramidal. [2] Reactions with nucleophiles support the chemical inequivalence of the three fluorines bonded to sulfur with two behaving the same chemically while the third is relatively inert. Water, CF_3CH_2OH, and CH_3NH_2 attack the positive sulfur center readily. The reactivity of $>\overset{|}{\underset{|}{S}}{=}N$ is demonstrated markedly by the ease with which saturation occurs. Addition of polar species to $CF_3SF_3NCF_3$ favors a canonical structure of the form $>S^{\delta+}-N^{\delta-}$ which is reasonable and agrees with our observation previously for $R_fN{=}SF_2$, [3] but conflicts with other literature data [4].

The amine and chloramine, $CF_3SF_4N(X)CF_4$ (X = H, Cl) are reluctant precursors to the formation of additional substituted amines due to a strong propensity to either dehydrofluorinate or dechlorofluorinate. However, the amines $CF_3SF_4N(H)R_f$ ($R_f = CF_3$,

C_2F_5) with silver difluoride result in fluoroamines without concomitant hydrazine formation [5]. Higher yields of the stable fluoroamines occur when elemental fluo in the presence of CsF, saturates either the $>\!S\!=\!N$ or $>\!C\!=\!N$ double bond in $CF_3SF_3\!=\!NC$ or $CF_3SF_4N\!=\!CFCF_3$. [6]

RESULTS AND DISCUSSION

The chemical reactivity of sulfonyliminosulfuroxy difluorides was demonstrate by their reactions with water and dilute acids and alkalies to form amines. [7, 8] mechanism which accounts for amine formation was proposed. The geometry of the initial and final compounds is essentially the same.

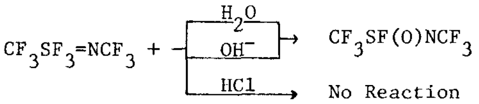


$$RN\!=\!\overset{F}{\underset{F}{S}}\!=\!O + B \xrightarrow{-F^-} RN\!=\!\overset{F}{\underset{H\ O}{S}}\!=\!O \rightarrow RN\overset{HO}{\underset{O}{S}}F$$

R = CH_3SO_2, FSO_2

B = H_2O, OH^-

In this work the hydrolysis of $CF_3SF_3NCF_3$ with water results in the preparation of trifluoromethyliminotrifluoromethylsulfuroxy fluoride and the geometry around S(VI) has changed from trigonal bipyramid to tetrahedral. In the presence of concentrate sodium hydroxide, a similar reaction occurs although no reaction was observed with anhydrous hydrogen chloride.

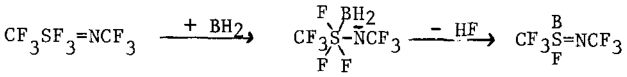


$$CF_3SF_3\!=\!NCF_3 + \begin{cases} \xrightarrow{H_2O} \\ \xrightarrow{OH^-} \end{cases} CF_3SF(O)NCF_3$$

$$\xrightarrow{HCl} \text{No Reaction}$$

Interestingly, the reaction of bis(trifluoromethyl)sulfur difluoride with anhydrous hydrogen chloride in Pyrex glass produces a quantitative yield of bis(trifluomethyl) sulfoxide. [9] However, no reaction occurs with water. This difference in behavior of S(IV) and S(VI) toward nucleophilic substitution reactions can be interpreted in terms of the HSAB principle [10] where the softer nucleophile, Cl^-, attac the softer S(IV) center, but it is less effective with the harder S(VI). An analog argument may be suggested for the harder oxygen-containing nucleophile and its grea effectiveness with S(VI).

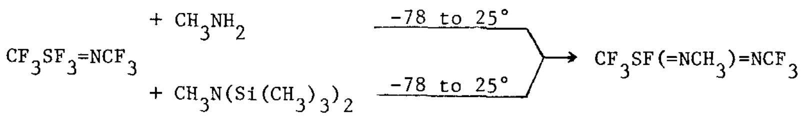
The nucleophilic displacement reaction of $CF_3SF_3NCF_3$ with the alcohol, CF_3CH_2O in the ratio of 1:3 results in the formation of equimolar amounts of $CF_3SF_4N(H)CF_3$ $CF_3SF(O)NCF_3$ accompanied by other unidentified products. The amine is produced by saturation of $>\!S\!=\!N-$ with hydrogen fluoride which is produced in the reaction while $CF_3SF(O)NCF_3$ arises from the decomposition of the unisolated reaction intermediate. In the case of reaction with CH_3NH_2, a second sulfur-nitrogen double bond is formed and the di-imine is stable. Thus, a mechanism which may describe these nucleophilic displacements is



$$CF_3SF_3{=}NCF_3 \xrightarrow{+\ BH_2} CF_3\overset{F\ BH_2}{\underset{F\ F}{S}}{-}NCF_3 \xrightarrow{-\ HF} CF_3\overset{B}{\underset{F}{S}}{=}NCF_3$$

$BH_2 = H_2O, CF_3CH_2OH, CH_3NH_2, OH^-$

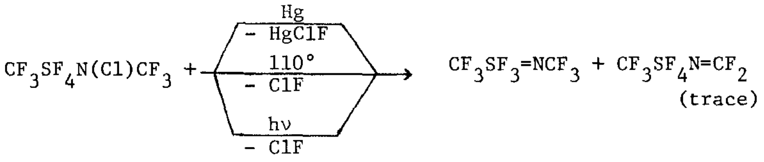
Although it is possible to prepare the di-imine $CF_3SF(=NCH_3)=NCF_3$ from reaction of methylamine with $CF_3SF_3=NCF_3$, the yields of the product are much higher if advantage is taken of the formation of strong Si-F bonds by using an N-silylated derivative. [11, 12]



$$CF_3SF_3{=}NCF_3 \begin{cases} + CH_3NH_2 \xrightarrow{-78 \text{ to } 25°} \\ + CH_3N(Si(CH_3)_3)_2 \xrightarrow{-78 \text{ to } 25°} \end{cases} CF_3SF(=NCH_3)=NCF_3$$

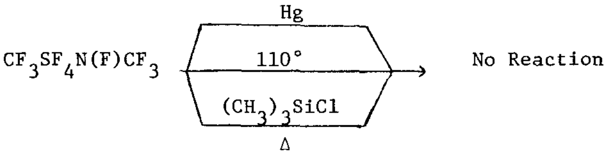
The preparation of a series of fluorinated sulfur(VI) compounds by the saturation of $>\overset{\|}{S}=N-$ of $CF_3SF(O)NCF_3$ was unsuccessful. No reaction occurs with HF or ClF in the presence of CsF. This behavior is analogous to that observed for the iminosulfuroxy difluorides where the addition of >S=N by H_2 or HX failed also. [13] The sharp contrast in the ease of saturation of the sulfur-nitrogen bond in $CF_3SF_3NCF_3$ and in $CF_3SF(O)NCF_3$ must arise in part from the more highly polarized nature of the $\overset{\delta+}{S}-\overset{\delta-}{N}$ bond and a more favorable geometry around S(VI) in the sulfur trifluoride.

N-Chloroamines are often receptive to insertion reactions involving small molecules, such as NO, CO, and Hg, and to photodecomposition reactions to form hydrazines. [14, 15] However, in the case of $CF_3SF_4N(Cl)CF_3$, none of these types of reactions was successful because of the ease of dechlorofluorination reactions, e.g.,



$$CF_3SF_4N(Cl)CF_3 + \begin{cases} \xrightarrow[-\ HgClF]{Hg} \\ \xrightarrow[-\ ClF]{110°} \\ \xrightarrow[-\ ClF]{h\nu} \end{cases} CF_3SF_3{=}NCF_3 + \underset{\text{(trace)}}{CF_3SF_4N{=}CF_2}$$

The amine, $CF_3SF_4N(H)CF_3$ also undergoes dehydrofluorination readily in the presence of CsF. As expected the fluoroamine is much more resistant to reaction than are the amine or chloroamine and in fact is not affected by the following



$$CF_3SF_4N(F)CF_3 \begin{cases} \xrightarrow{Hg} \\ \xrightarrow{110°} \\ \xrightarrow[\Delta]{(CH_3)_3SiCl} \end{cases} \text{No Reaction}$$

This sharp contrast in behavior must arise from a difference in N-X bond strengths and a reverse in polarization, viz $>\overset{\delta+}{N}-\overset{\delta-}{Cl}$ and $>\overset{\delta+}{N}-\overset{\delta-}{F}$.

The fluoroamines were synthesized initially by reactions of amines with silver difluoride,

$$CF_3SF_4N(H)R_f + AgF_2 \overset{\Delta}{\rightarrow} CF_3SF_4N(F)R_f \qquad R_f = CF_3, C_2F_5$$

This is one of the few cases where reactions of CF_3SF_4- compounds differ from those of SF_5- compounds; with $SF_5N(H)R_f$, under thermolytic conditions, hydrazines result

Fluorine has been found to react with sulfur difluoride imides in the presence of CsF [6] by simultaneously saturating the sulfur-nitrogen double bond and increase the oxidation state of sulfur, e.g.,

$$R_fN{=}SF_2 + F_2 \xrightarrow{CsF} R_fN(F)SF_5$$

$$R_f = CF_3, C_2F_5$$

We find that the addition of fluorine to sulfur-nitrogen or carbon-nitrogen double bonds at low temperature in the presence of CsF is a useful alternate route for the high yield preparation of fluoroamines.

$$CF_3SF_3{=}NCF_3 + F_2 \xrightarrow{CsF} CF_3SF_4N(F)CF_3$$

$$CF_3SF_4N{=}CFCF_3 + F_2 \xrightarrow{CsF} CF_3SF_4N(F)C_2F_5$$

EXPERIMENTAL

Apparatus

Infrared spectra were taken by using a Perkin-Elmer Model 457 infrared spectrometer with a 10 cm glass cell fitted with KBr windows and were calibrated against known absorption bands in polystyrene film. Fluorine nuclear magnetic resonance sp tra were determined with a Varian HA-100 NMR spectrometer by using CCl_3F as an internal standard and proton nmr spectra were obtained on a Varian EM-360 with $(CH_3)_4Si$ an internal reference. Mass spectra were obtained using a Hitachi Perkin-Elmer Mod RMU-6E mass spectrometer at 70 eV. Compounds were purified by gas chromatography by utilizing a column constructed of 0.25 in. copper tubing packed with 20% Kel-F oil FS-1265 on Chromosorb P.

Reagents

Chlorine monofluoride and silver difluoride were purchased from Ozark-Mahoning Company. Cesium fluoride was supplied by American Potash. The reagents $CF_3SF_3NCF_3$, $CF_3SF_4N(H)CF_3$, $CF_3SF_4NCFCF_3$ and $CF_3SF_4N(H)C_2F_5$ were prepared by methods reported earlier. [1]

Chlorine monofluoride and fluorine are reactive oxidizing agents and should be handled with caution.

Reactions

1) Reactions of $CF_3SF_3NCF_3$ with nucleophiles.

a) With H_2O

Into a 50 ml Pyrex vessel which contained 2 g of water, 0.4 mmol of $CF_3SF_3NCF_3$ was condensed at -195°. The reaction mixture was warmed to 25° and allowed to remain for 0.5 hr. A nearly quantitative yield of $CF_3SF(O)NCF_3$ was found in a trap at -98° after trap-to-trap separation.

b) With HCl

Anhydrous hydrogen chloride (0.6 mmol) and $CF_3SF_3NCF_3$ (0.3 mmol) were introduced into a 50 ml Pyrex vessel. After 10 hrs at 25 or 80°, no reaction had occurred and $CF_3SF_3NCF_3$ was recovered quantitatively.

c) With 6M NaOH

The compound $CF_3SF_3NCF_3$ was added to 0.5 ml 6 M NaOH in a 50 ml Pyrex vessel. After one hr an essentially quantitative yield of $CF_3SF(O)NCF_3$ was found in a trap at -98° after trap-to-trap distillation.

d) With CF_3CH_2OH

Approximately 0.6 mmol of $CF_3SF_3NCF_3$ was condensed into a 10 ml Hoke metal vessel which contained 0.25 mmol of CF_3CH_2OH.. No CF_3CH_2OH remained after the reaction mixture was kept at 25° for 15 hr. About 0.5 mmol of material was collected in a trap at -78° and about 0.1 mmol of unreacted $CF_3SF_3NCF_3$ was obtained in a trap at -98°. Equal amounts of $CF_3SF(O)NCF_3$ and $CF_3SF_4N(H)CF_3$ were isolated when the mixture was separated by using an 8 ft g.c. column of Kel-F on Chromosorb P.

e) With CH_3NH_2

Monomethylamine (0.8 mmol) and $CF_3SF_3NCF_3$ (0.6 mmol) were condensed into a 75 ml Hoke metal vessel at -195°. The reaction mixture was allowed to warm from -78 to 25° overnight. About 0.15 mmol of $CF_3SF(NCH_3)NCF_3$ was collected in a trap at -78° by using trap-to-trap techniques.

2) Abstraction of chlorine monofluoride from $CF_3SF_4N(Cl)CF_3$

a) With UV light

A quartz vessel which contained 0.5 mmol of $CF_3SF_4N(Cl)CF_3$ was phot lyzed at 3500 Å or with a mixture of wavelengths of uv radiation. Quantitative y of $CF_3SF_3NCF_3$ and ClF were obtained.

b) With mercury

The mixture of $CF_3SF_4N(Cl)CF_3$ (0.5 mmol) and mercury (1 g) in a 10 Pyrex vessel was agitated for one hr at 25°. The mercury salt, HgClF, $CF_3SF_3NCF_3$ (0.4 mmol) and unreacted chloroamine were recovered.

c) With heat

When a quartz vessel containing 0.5 mmol of $CF_3SF_4N(Cl)CF_3$ was kept 110° for 10 hr, equal amounts of $CF_3SF_3NCF_3$ and ClF were found after the reaction ture was fractionally distilled.

3) Reaction of amines with silver difluoride

a) $CF_3SF_4N(H)CF_3$

Into a 75 ml Hoke metal vessel which contained 3 mmol of silver difl ride, 0.75 mmol of $CF_3SF_4N(H)CF_3$ was condensed at -195°. When the reaction mixtur was held at 25° or at 70° for 10 hr, 0.5 mmol of $CF_3SF_4N(F)CF_3$ was found at -78° during trap-to-trap fractionation.

b) $CF_3SF_4N(H)C_2F_5$

In a similar manner, the reaction of $CF_3SF_4N(H)C_2F_5$ (1 mmol) with si difluoride resulted in the synthesis of $CF_3SF_4N(F)C_2F_5$ (0.6 mmol) which was obtain in a trap at -78° following fractional distillation.

4) Saturation of $>\overset{|}{\underset{|}{S}}=N-$ and $>C=N-$ with elemental fluorine

a) $>\overset{|}{\underset{|}{S}}=N-$ double bond

The compound $CF_3SF_3NCF_3$ (1 mmol) was introduced into a 75 ml Hoke me vessel which contained an excess of CsF and the vessel was filled to atmospheric p sure with elemental fluorine at -183°. The reaction mixture was warmed from -78 t 25° over 8 hr. After the fluorine gas was removed by passing through a soda lime trap, the yield of $CF_3SF_4N(F)CF_3$ was 85%.

b) $>C=N-$ double bond

In a similar fashion, a mixture of $CF_3SF_4NCFCF_3$ (1 mmol) and element fluorine in the presence of 3 g of CsF was warmed to 25° from -78°. The yield of $CF_3SF_4N(F)C_2F_5$ was 80%.

5) Reaction of $CF_3SF_3NCF_3$ with $CH_3N(Si(CH_3)_3)_2$

a) Preparation of $CH_3N(Si(CH_3)_3)_2$ [16]

A solution of 60 mmol of $(CH_3)_3SiCl$ in 200 ml of anhydrous ether was placed in a 500 ml round-bottom flask fitted with an efficient reflux condenser and bearing a side arm through which a gas inlet tube was inserted. The methylamine was introduced through the inlet tube continuously for 4 hr. A large amount of white precipitate was formed in the reaction flask. Filtration to remove the precipitate, followed by evaporation of ether resulted in the isolation of a high boiling liquid, $CH_3C(Si(CH_3)_3)_2$ (10 mmol).

b) Reaction of $CH_3N(Si(CH_3)_3)_2$

In a 25 ml Hoke metal vessel, one mmol of $CF_3SF_3NCF_3$ and one mmol of $CH_3N(Si(CH_3)_3)_2$ were mixed. The reaction mixture was warmed from -78 to 25° over 8 hr. After fractional distillation of the mixture, 2 mmol of $(CH_3)_3SiF$ was found at -98° and 0.2 mmol of $CF_3SF(NCH_3)NCF_3$ was collected at -78°.

6) Stability of $CF_3SF_4N(F)CF_3$ and $CF_3SF_4N(H)CF_3$

a) $CF_3SF_4N(F)CF_3$

Into a 10 ml Pyrex vessel containing 1 g of mercury, 0.25 mmol of $CF_3SF_4N(F)CF_3$ was condensed. No reaction was found after the agitation of the mixture for one hr and $CF_3SF_4N(F)CF_3$ was recovered quantitatively.

A 50 ml Pyrex vessel which contained 0.25 mmol of $CF_3SF_4N(F)CF_3$ was heated at 110° for 10 hr. A quantitative recovery of $CF_3SF_4N(F)CF_3$ was made.

Equimolar amounts of $CF_3SF_4N(F)CF_3$ (0.4 mmol) and $(CH_3)_3SiCl$ were distilled into a 15 ml Pyrex vessel. The reaction mixture was kept at 25° or heated at 100° for 10 hr. After fractional distillation, a quantitative recovery of starting materials was obtained.

b) $CF_3SF_4N(H)CF_3$

After agitation of approximately 0.4 mmol of $CF_3SF_4N(H)CF_3$ with 1 g of CsF in a 15 ml Pyrex vessel for 10 hr, $\sim$ 0.4 mmol of $CF_3SF_3NCF_3$ was found.

Mass spectral data, nuclear magnetic resonance spectra, and thermodynamic, elemental analyses and infrared spectral data are given in Tables, 1, 2, and 3, respectively.

ACKNOWLEDGEMENT

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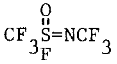
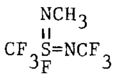
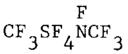
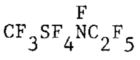
TABLE 1

Mass Spectra Data and Molecular Weight

 $CF_3\overset{O}{\underset{F}{S}}{=}NCF_3$	CF_3^+(very large), CF_3SFN^+(100), CF_3SON^+(82), CF_2SON^+(61), $CFSN^+$(57), CF_3SOFN^+(3), $CF_3SOFNCF_3^+$(3)	216* (219)
 $CF_3\overset{NCH_3}{\underset{F}{S}}{=}NCF_3$	NS^+(12), CH_3NS^+(34), CF_3 (83), CH_3NSN^+(5), CH_3NSF^+(35), CH_2NSF^+(25), $CHNSF^+$(69), CF_3NS^+(16), CF_3NSF^+(29), $CF_3SF(NCH_3)^+$(100), $CF_3SF(NCH_2)^+$(50), $CF_3SF(NCH)^+$(83), $CF_3SF(NCH_3)NCF_3^+$(27)	234* (232)
 $CF_3SF_4\overset{F}{N}CF_3$	CF_3^+(100), SF_2^+(50), CF_2S^+(50), CF_3N^+(23), SF_2N^+(64), SF_3^+(95), CF_3S^+(20), SF_3N^+(8), CF_3SFN^+(67), $CF_3SF_2^+$(16), $CF_3NSF_2^+$(67), $CF_3N(F)SF_2^+$(44)	277* (279)
 $CF_3SF_4\overset{F}{N}C_2F_5$	CF_3^+(95), SF_2^+(51), SF_2N^+(41), SF_3^+(95), CF_3SN^+(71), CF_3S^+(25), CF_3SN^+(55), $C_2F_5^+$(100), $C_2F_5N^+$(35), $CF_3SF_2^+$(23), $C_2F_5N(F)^+$(37), CF_3SFN^+(98), $C_2F_5NS^+$(10), $C_2F_5N(F)S^+$(24), $C_2F_5NSF_2^+$(16), $C_2F_5SF_3^+$(14), $C_2F_5NSF_2^+$(13), $CF_3SNC_2F_5^+$(4)	327* (329)

*Molecular weight found

TABLE 2

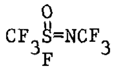
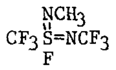
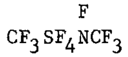
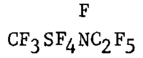
Nuclear magnetic resonance (chemical shift relative to CCl_3F and $(CH_3)_4Si$)

	Chemical Shift								J, Hz
Compound	CF_3S	SF_4	SF	NF	CF_2	CF_3N	CF_3C	CH_3N	
$CF_3\overset{O}{\underset{F}{S}}=NCF_3$	73.2 (d,q)		-51.8 (multi)			45.9 (d,br)			CF_3S-CF_3N = 1.2 $SF-CF_3N$ = 8.0 $SF-CF_3S$ = 16.2
$CF_3\overset{NCH_3}{\underset{F}{S}}=NCF_3$	73.1 (d,q,q)		-61 (multi)			47.4 (d,q,q)		-0.25	CF_3S-CF_3N = 1.2 $SF-CF_3S$ = 26 CH_3-CF_3S = 12 $SF-CF_3N$ = 24 CH_3-CF_3N = 10
$CF_3SF_4\overset{F}{N}CF_3$	65.3 (p)	-37.1 (multi)		45.3 (br)		69.6 (h)			SF_4-CF_3S = 22 $NF-CF_3N$ = 11 SF_4-CF_3N = 11 SF_4-NF = 1.5 CF_3S-NF = 0 CF_3S-CF_3N = 0
$CF_3SF_4\overset{F}{N}C_2F_5$	65.4 (p)	-40.3 (multi)		48.6 (br)	109.5 (d,p)		81.8 (d)		SF_4-CF_3S = 21.5 SF_4-NF = 1.5 CF_3C-CF_2 = 0 CF_3C-SF_4 = 0 CF_3C-NF = 0 CF_3C-NF = 19.5 CF_3S-NF = 0 CF_3S-CF_2 = 0 CF_2-NF = 27.5 CF_2-SF_4 = 13.7

s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; h, hextet; multi, multiplet

TABLE 3

Thermodynamic data, elemental analysis, and infrared spectra

Compound	ΔH_v kcal/mole	ΔS_v (eu)	a^*	b	BP °C	Elemental Analysis F	S	N	C	H	Infrared Spectra
 $CF_3\underset{F}{\overset{O}{\overset{\|}{S}}}=NCF_3$	5.5	18.4	6.89	1195	24						1425(vs), 1340(s), 1250(vs), 1230(s), 1180(vs), 840(m), 810(w), 760(m), 605(m), 570(w)
 $CF_3\underset{F}{\overset{NCH_3}{\overset{\|}{S}}}=NCF_3$	6.9	20.5	7.35	1510	64		11.52 (13.79)**	12.07 (12.07)	15.34 (15.51)	1.42 (1.30)	2930(m), 1450(ms), 1370(s), 1340(s), 1260(s), 1240(vs), 1220(vs), 1180(vs), 920(m), 860(vs)
 $CF_3SF_4\overset{F}{N}CF_3$	7.2	22.9	7.85	1576	44	73.0 (74.9)	11.94 (11.46)	3.89 (5.01)	8.38 (8.60)		1270(vs), 1245(vs), 1220(s), 1170(s), 1035(m), 950(w), 880(s), 800(ms), 740(ms), 680(vs)
 $CF_3SF_4\overset{F}{N}C_2F_5$	7.5	22.4	7.81	1652	62		10.17 (9.73)	4.42 (4.26)	10.70 (10.94)		1350(m), 1250(vs), 1230(s), 1190(s), 1170(s), 1145(m), 1090(s), 1030(w), 890(vs), 860(m), 730(m), 690(vs)

*$\log P_{torr} = a - b/T$; **calculated values