CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF WASHINGTON, SEATTLE 5, WASHINGTON

Reactions of Pentafluorosulfur Hypofluorite[1]

BY STANLEY M. WILLIAMSON AND GEORGE H. CADY

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Pentafluorosulfur hypofluorite has been observed to react quantitatively with perfluorocyclopentene (C_5F_8), tetrafluoroethylene, tetrachloroethylene, ethylene, and vinyl chloride at room temperature to give compounds achieved by heterolysis of the O–F bond of the hypofluorite. The fluorine atom bonds to one carbon and the SF_5O- group bonds to the other carbon of the alkene. Reaction of SF_5OF with vinyl chloride gave only 2-chloro-2-fluoroethyl pentafluorosulfur oxide. Pentafluorosulfur hypofluorite also reacts with sulfur tetrafluoride to give SF_6, F_4SO, F_5SOOSF_5; and F_5SOSF_5; with carbon monoxide to give F_2CO and F_4SO; and with carbon tetrachloride to give F_2CO, Cl_2, and F_5SOSF_5. Pentafluorosulfur hypofluorite begins to irreversibly decompose at 210° into oxygen and sulfur hexafluoride.

Since pentafluorosulfur hypofluorite was first synthesized,[2] the only reported research on it has been the determination of its structure by electron diffraction[3a,b] and the use of it to prepare bis-(pentafluorosulfur) peroxide.[4] It has now been found to add the SF_5O- group and the fluorine atom across some carbon–carbon double bonds in a like manner to CF_3OF, as has been reported by Porter and Cady[5] and by Allison and Cady.[6] It was of interest also to determine whether an addition reaction would occur when the hypofluorite was used to oxidize and increase the coördination number of an atom possessing an unshared pair of electrons. Sulfur tetrafluoride and carbon monoxide were chosen as two examples for the experiment. Reaction with carbon tetrachloride was attempted to see whether the hypofluorite would replace a chlorine atom(s) with a SF_5O- group and/or a fluorine atom. The thermal decomposition of SF_5OF was of general interest for comparison with the reversible decomposition of CF_3OF[7] into F_2CO and F_2.

Equations for the reactions which are described in the experimental part are

$$C_5F_8 + SF_5OF \longrightarrow C_5H_9OSF_5 \qquad (1)$$

$$C_2F_4 + SF_5OF \longrightarrow C_2F_5OSF_5 \qquad (2)$$

$$C_2Cl_4 + SF_5OF \longrightarrow FC_2Cl_4OSF_5 \qquad (3)$$

$$C_2H_4 + SF_5OF \longrightarrow FC_2H_4OSF_5 \qquad (4)$$

$$C_2H_3Cl + SF_5OF \longrightarrow FClC_2H_3OSF_5 \qquad (5)$$

$$SF_4 + SF_5OF \longrightarrow SF_6 + F_4SO + F_5SOOSF_5 + F_5SOSF_5 \qquad (6)$$
(variable proportions)

$$CO + SF_5OF \longrightarrow F_2CO + F_4SO \qquad (7)$$

$$CCl_4 + SF_5OF \longrightarrow F_2CO + 2Cl_2 + F_2SOSF_5 \qquad (8)$$

$$SF_5OF \xrightarrow[\Delta]{} SF_6 + {}^1/{}_2O_2 \qquad (9)$$

Experimental

The general preparative procedures for the first five reactions listed above were essentially the same, *i.e.*, the direct combination of the reactants at room temperature in Pyrex glass reaction vessels by use of standard vacuum line techniques. In this case the total reaction pressure indicated that the reactants combined in a one to one ratio to give the new compounds that are reported. These reactions were carried out at less than atmospheric pressure with nitrogen gas as a diluent so that the vigor of the reactions would be reduced and would not lead to destructive explosions. The nitrogen was removed by pumping the reaction mixture through traps cooled by liquid oxygen. Before any physical measurements were made on the compounds, they were checked for purity, depending on their volatility, either by analytical vapor phase chromatography or fractional codistillation.[8]

Reaction with Perfluorocyclopentene.—Gaseous SF_5OF was added slowly to the liquid C_5F_8 in the presence of nitrogen gas until an equimolar amount was present. Heating of the reaction vessel overnight at 95° was necessary for complete conversion to the addition compound. Rectification of the condensate by means of fractional codistillation gave a single, water-white material, samples of which had vapor densities of 372 and 374 g./GMV. (Theoretical for C_5SOF_{14} is 374.) The equation

$$\log P_{\text{mm}} = 7.9373 - \frac{1.826 \times 10^3}{T}$$

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[1] From the Ph.D. thesis of S. M. Williamson, University of Washington, 1961. Presented at the National Meeting of the American Chemical Society, Chicago, Illinois, September, 1961.

[2] F. B. Dudley, G. H. Cady, and D. F. Eggers, Jr., *J. Am. Chem. Soc.*, **78**, 1553 (1956).

[3] (a) R. A. Crawford, *Dissertation Abstr.*, **19**, 3145 (1959); (b) R. A. Crawford, F. B. Dudley, and K. Hedberg, *J. Am. Chem. Soc.*, **81**, 5287 (1959).

[4] C. I. Merrill and G. H. Cady, *ibid.*, **83**, 298 (1961).

[5] R. S. Porter and G. H. Cady, *ibid.*, **79**, 5625 (1957).

[6] J. A. C. Allison and G. H. Cady, *ibid.*, **81**, 1089 (1959).

[7] R. S. Porter and G. H. Cady, *ibid.*, **79**, 5628 (1957).

[8] G. H. Cady and D. P. Siegwarth, *Anal. Chem.*, **31**, 618 (1959).

represents the vapor pressure as a function of absolute temperature, which gives a normal boiling point of 87.8°, a heat of vaporization of 8,350 cal./mole, and a Trouton constant of 23.1 e.u. The liquid density is given as a function of the centigrade temperature by

$$d_t = 1.939 - 2.68 \times 10^{-3}t \text{ g./ml.}$$

The observed index of refraction at 27.3° of this compound was about 1.29. This value was not read directly since the refractometer had 1.30 as its lowest scale reading. Equation 1 represents the reaction, the product of which gave the following analysis.

Anal. Calcd. for C_5SOF_{14}: C, 16.1; F, 71.2; S, 8.6. Found[9]: C, 15.6; F, 68.9; S, 10.3.

Reaction with Tetrafluoroethylene.—To a 40% mixture by volume of SF_5OF in N_2 was added very slowly a 25% mixture of C_2F_4 in N_2 until a slightly greater than equimolar amount of the latter reactant had been added. (An excessive rate of addition caused explosions.) Fractional codistillation of the condensate gave very little volatile material except for the excess C_2F_4. The remaining material was mostly a single compound, which after the removal of a small after-fraction was a water-white material, the experimental vapor density of which was 260 g./GMV. (Theoretical for C_2SOF_{10} is 262.) The equation

$$\log P_{\text{mm}} = 7.8237 - \frac{1.424 \times 10^3}{T}$$

represents the vapor pressure as a function of absolute temperature, which gives a normal boiling point of 15.0°, a heat of vaporization of 6,510 cal./mole, and a Trouton constant of 22.6 e.u. The liquid density is given as a function of the centigrade temperature by

$$d_t = 1.773 - 3.44 \times 10^{-3}t \text{ g./ml.}$$

The index of refraction of this compound was not determined because of the low boiling point. Equation 2 represents the reaction, the product of which gave the following analysis.

Anal. Calcd. for C_2SOF_{10}: C, 9.2; F, 72.6; S, 12.2. Found[9]: C, 8.4; F, 71.4; S, 11.1.

Reaction with Tetrachloroethylene.—SF_5OF (0.03 mole) with no N_2 was added quickly (2 min.) to 0.03 mole of liquid C_2Cl_4 at 0° in a 0.5-l. Pyrex flask. Some smoking was visible in the flask and the pressure dropped rather quickly, which indicated that the hypofluorite was being consumed. Purification of the product by preparative vapor phase chromatography using a column containing crushed fire-brick coated with tricresyl phosphate operating at 74° gave one main fraction with three other very small fractions, two with retention times less than and one greater than that for the main fraction. This main fraction was a water-white material, consecutive fractions of which had observed vapor densities of 333, 330, and 325 g./GMV at pressures close to 6.4 mm. and at a temperature of 24.5°, for an average value of 329. (Theoretical for $C_2Cl_4SOF_6$ is 328.) Apparently the low vapor pressure of approximately 6 mm. at room temperature caused the somewhat larger than normal experimental error in the observed vapor density values, even though the pressures were read with a cathetometer. The equation

$$\log P_{\text{mm}} = 7.7045 - \frac{2.007 \times 10^3}{T}$$

represents the vapor pressure as a function of absolute temperature, which gives a normal boiling point of 142.9°, a heat of vaporization of 9,180 cal./mole, and a Trouton constant of 22.1 e.u. The liquid density is given as a function of the centigrade temperature by

$$d_t = 1.909 - 1.98 \times 10^{-3}t \text{ g./ml.}$$

The index of refraction was 1.3900 at 27.3°. Equation 3 represents the reaction, the product of which gave the following analysis.

Anal. Calcd. for $C_2Cl_4SOF_6$: C, 7.3; F, 34.8. Found[9]: C, 7.8; F, 37.7. (The last datum is probably not the true value.)

Reaction with Ethylene.—To a 67% mixture by volume of C_2H_4 in N_2 was added very slowly a 20% mixture of SF_2OF in N_2. Slightly less than a stoichiometric amount of SF_5OF was added. Analytical vapor phase chromatography using a column of polypropylene glycol suspended on diatomaceous earth showed that the reaction condensate was a single, pure compound. The water-white liquid had an experimental vapor density of 189 g./GMV at a pressure of 50.0 mm. and a temperature of 26.0°. (Theoretical for $C_2H_4SOF_5$ is 190.) The equation

$$\log P_{\text{mm}} = 8.0489 - \frac{1.873 \times 10^3}{T}$$

represents the vapor pressure as a function of absolute temperature, which gives a normal boiling point of 88.9°, a heat of vaporization of 8,570 cal./mole, and a Trouton constant of 23.7 e.u. The liquid density is given as a function of the centigrade temperature by

$$d_t = 1.679 - 2.13 \times 10^{-3}t \text{ g./ml.}$$

The observed index of refraction at 27.3° of this compound was 1.3026. Equation 4 represents the reaction, the product of which gave the following analysis.

Anal. Calcd. for $C_2H_4SOF_6$: C, 12.7; F, 60.0; S, 16.9; H, 2.1. Found[9]: C, 13.0; F, 58.2; S, 17.0; H, 2.1.

Reaction with Vinyl Chloride.—Pure, undiluted SF_5OF was added very slowly to a 21% mixture by volume of C_2H_3Cl in N_2, until almost a stoichiometric amount had been added. No increase in the pressure of the reaction vessel was observed. The chromatogram for the purification of the reaction condensate, using a column with the same composition and construction as for ethylene, showed two small fractions with retention times less than that for the main fraction. The water-white liquid had an experimental vapor density of 226 g./GMV at a pressure of 48.8 mm. and a temperature of 25.2°. (Theoretical for $C_2H_3ClSOF_6$ is 224.5.) The equation represents the vapor

$$\log P_{\text{mm}} = 8.0526 - \frac{1.874 \times 10^3}{T}$$

pressure as a function of absolute temperature, which gives a normal boiling point of 89.2°, a heat of vaporization of 8,580 cal./mole, and a Trouton constant of 23.7 e.u. The liquid density is given as a function of the centigrade temperature by

$$d_t = 1.730 - 2.14 \times 10^{-3}t \text{ g./ml.}$$

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(9) Schwarzkopf Microanalytical Laboratory, 56-19 37th Ave., Woodside 77, New York.

TABLE I

VAPOR PRESSURE DATA

Obsd. P, mm.	T, °K.	Calcd. P, mm.	Obsd. P, mm.	T, °K.	Calcd. P, mm.
Vapor pressure of $C_5H_9OSF_5$					
70.0	300.7	73.9	431.8	344.3	430.3
118.2	311.7	120.0	502.7	348.3	495.1
156.1	318.2	158.1	562.3	352.0	562.1
189.1	322.7	190.0	633.3	355.5	632.3
221.8	326.4	220.3	697.3	358.2	691.2
260.8	330.4	257.5	747.9	359.8	728.3
295.0	334.3	298.6	761.8	361.0	757.0
381.0	340.5	375.5			
Vapor pressure of $C_2F_5OSF_5$					
104.7	245.0	102.7	449.6	275.4	449.8
145.3	254.3	167.5	540.7	279.7	540.1
207.3	258.5	206.7	592.6	282.0	594.3
254.9	262.4	249.4	661.4	284.5	658.3
310.7	266.8	306.5	728.4	287.2	733.7
369.8	271.1	372.4	788.3	298.0	787.8
Vapor pressure of $FC_2Cl_4OSF_5$					
16.7	314.4	20.9	367.3	390.3	365.0
28.3	324.0	32.4	446.8	396.8	443.1
65.5	342.1	68.8	547.9	404.0	545.4
126.1	359.0	130.1	646.2	410.2	648.3
207.7	372.3	205.9	707.4	413.7	713.2
287.2	383.0	291.3	781.8	417.5	789.4
Vapor pressure of $FC_2H_4OSF_5$					
35.5	290.5	39.9	386.8	343.0	387.5
52.6	298.3	59.0	488.0	349.3	486.1
81.0	306.6	87.2	568.1	354.0	572.7
103.4	311.7	109.6	641.3	357.1	636.7
148.7	317.8	143.0	718.6	360.4	711.1
216.0	327.7	215.4	787.7	363.4	784.9
292.7	335.1	288.1			
Vapor pressure of $FClC_2H_3OSF_5$					
29.4	285.8	31.2	373.4	341.8	370.6
46.7	294.0	47.6	446.7	346.7	443.0
65.3	300.5	65.4	520.1	351.3	521.5
87.7	306.9	88.2	589.1	355.0	592.7
118.8	313.3	117.5	660.1	358.2	660.7
147.6	318.6	147.8	722.3	361.1	727.9
200.7	325.5	197.0	761.0	362.5	762.2
250.2	331.3	248.4			
305.7	336.5	303.8			

TABLE II

t, °C.	d, obsd.	d, calcd.	t, °C.	d, obsd.	d, calcd.
Density of $C_5F_9OSF_5$			Density of $C_2F_5OSF_5$		
0.20	1.938	1.938	−45.5	1.919	1.929
1.10	1.936	1.936	−35.0	1.888	1.893
7.10	1.920	1.920	−29.5	1.870	1.874
10.15	1.912	1.912	−23.6	1.851	1.854
18.25	1.890	1.889	−15.5	1.826	1.826
24.30	1.875	1.874	− 6.7	1.795	1.796
32.70	1.851	1.851	3.7	1.758	1.760
38.13	1.837	1.839	9.5	1.738	1.740
43.70	1.821	1.822	15.1	1.717	1.721
			20.8	1.695	1.701
Density of $FC_2Cl_4OSF_5$			Density of $FC_2H_4OSF_5$		
0.70	1.907	1.908	0.50	1.678	1.678
12.50	1.884	1.884	7.60	1.663	1.663
26.10	1.857	1.857	12.70	1.652	1.652
37.75	1.834	1.834	21.20	1.634	1.634
44.20	1.821	1.821	29.17	1.617	1.617
			37.00	1.600	1.600
			43.75	1.586	1.586
Density of $FClC_2H_3OSF_5$					
0.15	1.730	1.730	24.30	1.679	1.678
4.75	1.718	1.720	30.20	1.665	1.665
12.35	1.704	1.704	37.84	1.649	1.649
19.60	1.689	1.688	42.10	1.639	1.640
			47.60	1.627	1.628

The observed index of refraction at 27.3° of this compound was 1.3271. Equation 5 represents the reaction.

Reaction with Sulfur Tetrafluoride.—Pentafluorosulfur hypofluorite and sulfur tetrafluoride reacted very slowly if at all at room temperature, but when the gases were heated, addition to and fluorination of the SF_4 began to occur appreciably at about 140°. When SF_4 and SF_5OF in a volume ratio of 1.6 to 1 were heated in a nickel reactor, which was equipped with Calrod heating elements, thermocouples, and a Booth-Cromer pressure transmitter,[10] a decrease in the slope of the curve obtained by plotting the reaction pressure *vs.* the reactor temperature indicated only about 30% addition reaction. Fractional codistillation of the reaction mixture gave four compounds, each of which had been reported previously, and they were identified by their respective vapor densities and infrared spectra. The unbalanced equation 6 represents the reaction. All of the SF_5OF had been consumed by the SF_4. The expected addition product, bis-(pentafluorosulfur) oxide,[11] had been reported as the product of the oxidation of sulfur chloride pentafluoride. The product from this reaction consisted of SF_6, F_4SO, F_5SOOSF_5, and F_5SOSF_5 in the relative molar amounts of 7:3:2:1, respectively.

Reaction with Carbon Monoxide.—When the two gases were mixed in a small Pyrex glass vessel at room temperature, no reaction was observed. When a mixture containing 100 mm. pressure of each of the gases was heated in the nickel reactor described above, no change in the slope of the pressure *vs.* temperature curve was observed before the heating was stopped at 165°. The cooling curve was coincident with the heating curve, but the entire contents of the reactor were condensable at −183°; therefore, it was apparent that a reaction had occurred and that the carbon monoxide had been consumed. The reaction mixture was separated by fractional codistillation into essentially equal quantities of carbonyl fluoride (F_2CO) and thionyl tetrafluoride (F_4SO). Equation 7 represents the reaction. The presence of these products was confirmed by their respective vapor densities and infrared spectra. The reaction also was tried at room temperature and high pressure by distilling into a copper tube reactor of 6.3 mm. o.d. and 5 ml. volume 2 g. of SF_5OF and 0.62 g. of CO. After the valve on the tube was closed, the reactor was transferred in a hood directly from the

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[10] S. Katz and J. T. Barr, *Anal. Chem.*, **25**, 619 (1953).

[11] H. L. Roberts, *J. Chem. Soc.*, 2774 (1960).

dewar flask at −196° to within a piece of heavy iron pipe, where it warmed to room temperature. It is not known whether the reaction proceeded slowly or rapidly, but after 2 days the substances F_2CO and F_4SO were found and no other products were detected.

Reaction with Carbon Tetrachloride.—At room temperature, pentafluorosulfur hypofluorite and carbon tetrachloride appeared not to react. Activation of the carbon-chlorine bond by ultraviolet light was considered as a possible means of introducing a SF_5O– group onto the carbon atom. Sixty mm. pressure of CCl_4 and 150 mm. pressure of SF_5OF were added to a 3-l. Pyrex glass flask which was fitted with an internal, water-cooled, 4.5-watt ultraviolet lamp. After 30 hr. of irradiation, the green color of chlorine was seen, and after 40 hr., fractional codistillation showed the products F_2CO, Cl_2, and F_5SOSF_5 to be present. Equation 8 can represent the reaction. Small amounts of SO_2F_2 and SF_6 which also were observed could have resulted from the decomposition of SF_5OF rather than from its reaction with CCl_4. Chlorine was identified by its characteristic color and by its relative retention time during the codistillation. The presence of the other products was confirmed by their respective vapor densities and infrared spectra. No compound was found that would suggest any substitution of chlorine in CCl_4 by the SF_5O– group.

Thermal Decomposition of F_5SOF.—Enough pure SF_5OF was put into the nickel reactor described above to give a pressure of 221 mm. at 23° (2.82 g.). As the vessel was heated slowly to 432°, the pressure was measured frequently and a graph of pressure *vs.* temperature was made. Deviation of the gas from ideal behavior began to occur at about 210° and stopped at about 400°. Upon cooling to room temperature, the pressure behaved almost ideally and gave a final pressure 1.5 times the original 221 mm. The gas was found to be a mixture of oxygen and sulfur hexafluoride formed by the net reaction

$$SF_5OF \xrightarrow[\Delta]{} SF_6 + {}^1/_2O_2 \qquad (9)$$

Discussion

The melting points for the new compounds were not obtained, since on cooling rapidly or slowly to as low as −186°, glasses were formed with or without stirring.

The infrared spectra of the new compounds are shown in Fig. 1 through 5. The C–F stretching frequencies lie in the range 1317 to 1028 cm.$^{-1}$. By analogy with the absorption frequencies for SF_5OF[2] of 935 and 888 cm.$^{-1}$, the S–F absorption range is assigned from 950 to 836 cm.$^{-1}$.

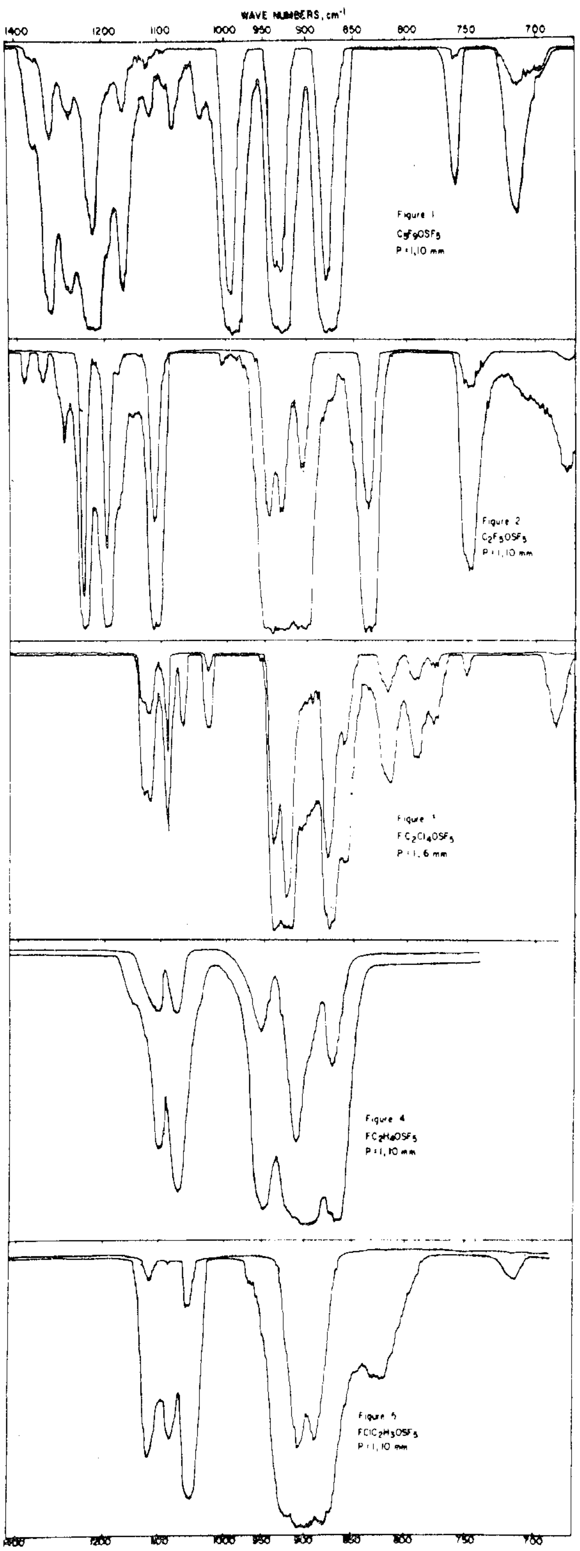

Fig. 1–5.—Infrared spectra in NaCl region, using a 10-cm. cell with AgCl windows in a Perkin-Elmer Model 21 spectrometer.

The mass spectra of the new compounds confirmed their structures as resulting from the addition of a fluorine atom to one carbon and the SF_5O– group to the other carbon of the double bond. For example, from $SF_5OCH_2CH_2F$, the main fragments and their % relative abundance were: $S^{32}F_3^+$, 25%; $OS^{32}F_3^+$, 10%; $S^{32}F_5^+$, 100%; and $C^{12}H_2OS^{32}F_5^+$, 46%. The mass spectrum of the addition compound from C_2H_3Cl and SF_5OF was essentially the same as that from C_2H_4 and SF_5OF, except that the $CFCl^+$ and $C_2H_3FCl^+$

ions also were found in relative abundances of 1% and 32%, respectively.

The nuclear magnetic resonance spectra for the SF_5 groups of the addition compounds from reactions 1 through 5 and of F_5SOSF_5 from reaction 6 have been discussed in detail by Merrill, Williamson, Cady, and Eggers.[12] The spectra due to the fluorine atoms attached to carbon add evidence for the proposed structures. The C–F resonance in SF_5OCH_2CHClF was observed to be a doublet, each part of which was further split into a triplet, the splitting being due to the fluorine–hydrogen nuclear interaction. Likewise for $SF_5OCH_2CH_2F$, the C–F resonance was a triplet, each part of which was further split into a triplet. The previously reported values of the fluorine–hydrogen spin–spin coupling constants[13] in CH_3CH_2F are 60 and 20 c.p.s., respectively, between the fluorine and the methylene and methyl protons. The corresponding coupling constants for SF_5OCH_2CHClF are 50 and 18 c.p.s., and for $SF_5OCH_2CH_2F$ are 51 and 23 c.p.s., ±2 in both cases.

No nuclear interaction was observed between the hydrogen atoms on either carbon or the fluorine atoms on the second carbon from the oxygen and the fluorine atoms of the SF_5 group. The splitting between fluorine atoms attached to the carbon atom bonded directly to oxygen and the equatorial and apical fluorine atoms of the SF_5 group was of the order of 9 and 3 c.p.s., respectively.

The approximate chemical shifts between the four equatorial fluorine atoms of the SF_5 group and the fluorine atom(s) bound to carbon (the latter fluorine resonances always appeared at the higher field strength) for a particular compound at 40 Mc. were 8150, 6400, 5530, 11380, and 8350 c.p.s., respectively, for $C_5F_9OSF_5$, $C_2F_5OSF_5$, $FC_2Cl_4OSF_5$, $FC_2H_4OSF_5$, and $FClC_2H_3OSF_5$.

The n.m.r. spectrum of the last of these substances indicated that SF_5OF had added stereospecifically to vinyl chloride.

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[12] C. I. Merrill, S. M. Williamson, G. H. Cady, and D. F. Eggers, Jr., *Inorg. Chem.*, **1**, 215 (1962).

[13] H. S. Gutowsky, L. H. Meyer, and D. W. McCall, *J. Chem. Phys.*, **23**, 982 (1955).

CONTRIBUTION NO. 751 FROM THE CENTRAL RESEARCH DEPARTMENT, EXPERIMENTAL STATION,
E. I. DU PONT DE NEMOURS AND COMPANY, WILMINGTON, DELAWARE

Iminosulfur Oxydifluoride and Poly-(oxofluorosulfur nitride)

BY G. W. PARSHALL, RICHARD CRAMER, AND R. E. FOSTER

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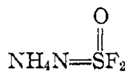
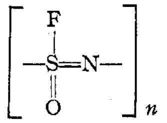
The synthesis and properties of iminosulfur oxydifluoride, $HN{=}SOF_2$, are described. When dehydrofluorinated, this compound yields the rubbery poly-(oxofluorosulfur nitride), which also is directly obtainable from ammonia and sulfur oxytetrafluoride.

A brief account has been given by Seel and Simon[1] of the reaction of ammonia with sulfur oxytetrafluoride to give the ammonium salt of iminosulfur oxydifluoride (I) and poly-(oxofluorosulfur nitride) (II). The reaction of primary amines with sulfur oxytetrafluoride to give a series of N-alkyl- or N-aryliminosulfur oxydifluorides (III) has been described in greater detail.[2] The synthesis and properties of unsubstituted iminosulfur oxydifluoride (IV) are reported in this paper together with details of the



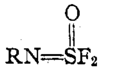
$NH_4N{=}\overset{\overset{O}{\|}}{S}F_2$

(I)



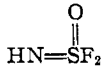
$\left[-\overset{\overset{F}{|}}{\underset{\underset{O}{\|}}{S}}{=}N-\right]_n$

(II)



$RN{=}\overset{\overset{O}{\|}}{S}F_2$

(III)



$HN{=}\overset{\overset{O}{\|}}{S}F_2$

(IV)

[1] F. Seel and G. Simon, *Angew. Chem.*, **72**, 709 (1960).

[2] R. Cramer and D. D. Coffman, *J. Org. Chem.*, **26**, 4010 (1961).