TABLE IV

CALCULATED WATER CONTENT OF BENZENE USED IN SERIES 1

$Ti(OC_2H_5)_4$ concn., $m \times 10^2$	Apparent mol. wt.	App. H_2O content of C_6H_6, $m \times 10^2$
0.702	310	0.37
1.097	372	0.41
1.978	429	0.53
2.48	460	0.53
4.95	562	0.49
6.23	594	0.41
7.76	608	0.37
10.02	630	0.37

argument a sample of "wet" benzene was prepared with a water concentration of 4 × 10^{-3} m by the method of Christian, *et al.*,[7] and a series of molecular weight determinations then was carried out. The results are presented in Table V, and it is noteworthy hat the apparent molecular weight of titanium ethoxide is concentration dependent. The results in Table V show a strikingly similar trend to those in Table I and to the results of Martin and Winter.[4] Moreover, it is clear that the apparent degree of polymerization can be less than unity at very low concentrations. Finally, another series of determinations was carried out with "dry" benzene taking the concentration of titanium ethoxide down to 2 × 10^{-3} m, which was the limit for reasonable accuracy. The molecular weights of titanium ethoxide are given in Table VI and it is clear that there is no significant variation with concentration from 2.0 to 54.9 × 10^{-3} m. For all of the results in Tables II, III, and VI, the mean value of the number average degree of polymerization is 2.82 with a coefficient of variation of ±6%. There is therefore no compelling reason to consider species other than the trimer over the concentration range 2.0–100.2 × 10^{-3} m in benzene at the freezing point. This work also demonstrates the need for exceptional precautions in attempting to make cryoscopic molecular weight measurements on solutions of readily hydrolyzable metal alkoxides.

TABLE V

APPARENT MOLECULAR WEIGHTS OF $Ti(OC_2H_5)_4$ IN "WET" BENZENE

$Ti(OC_2H_5)_4$ concn.			
g./100 g. of C_6H_6	$m \times 10^2$	Apparent mol. wt.	App. deg. of polymerization
0.0560	0.245	204	0.89
0.1602	0.704	300	1.32
0.1780	0.780	310	1.36
0.3750	1.975	441	1.93
0.5450	2.390	485	2.12

TABLE VI

MOLECULAR WEIGHTS OF $Ti(OC_2H_5)_4$ IN BENZENE (SERIES 4)

$Ti(OC_2H_5)_4$ concn.			
g./100 g. of C_6H_6	$m \times 10^2$	Mol. wt.	Deg. of polymerization
0.0456	0.200	736	3.22
0.0655	0.287	605	2.65
0.1009	0.441	726	3.18
0.1620	0.710	590	2.59
0.1805	0.791	607	2.66
1.235	5.486	652	2.86

Infrared spectra and proton magnetic resonance studies are being made in these laboratories on solutions of titanium tetraethoxide over the whole concentration range from about 10^{-2} m to the pure liquid.

Acknowledgment.—We are pleased to acknowledge generous financial support from the Paint Research Institute.

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[7] S. D. Christian, H. E. Affsprung, and J. R. Johnson, *J. Chem Soc.*, 1896 (1963).

CONTRIBUTION FROM THE GORGAS LABORATORY, ROHM & HAAS COMPANY,
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Synthesis and Characterization of Fluorimidodisulfuryl Fluoride, $(FSO_2)_2NF$, and Difluoramidosulfuryl Fluoride, FSO_2NF_2

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The preparation of difluoramidosulfuryl fluoride by the thermal reaction of tetrafluorohydrazine with sulfur dioxide and the preparation of fluorimidodisulfuryl fluoride *via* fluorination of imidodisulfuryl fluoride with elemental fluorine are described. A more complete description of the ultraviolet method of preparation of FSO_2NF_2 is given. Some physical properties of these new materials were determined and their hydrolyses in alkaline solutions were studied.

Introduction

The synthesis of pentafluorosulfur difluoramine, the first member of a new class of NF compounds containing a sulfur–nitrogen single bond, was reported recently by several independent groups.[1-3] Two other examples of this class have now been prepared; difluoramidosulfuryl fluoride[4] and fluorimidodisulfuryl fluoride.

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[1] A. L. Logothetis, G. N. Sausen, and R. J. Shozda, *Inorg. Chem.*, **2**, 173 (1963).

[2] G. H. Cady, D. F. Eggers, and B. Tittle, *Proc. Chem. Soc.*, 65 (1963).

[3] E. C. Stump, Jr., C. D. Padgett, and W. S. Brey, Jr., *Inorg. Chem.*, **2**, 648 (1963).

[4] For a preliminary report on the preparation of FSO_2NF_2 by photolysis of mixtures of tetrafluorohydrazine and sulfur dioxide, see C. L. Bumgardner and M. Lustig, *ibid.*, **2**, 662 (1963).

Results and Discussion

Tetrafluorohydrazine was found to react with sulfur dioxide to produce difluoramidosulfuryl fluoride in good yield. The reaction could be carried out either at 120° under high pressure (*ca.* 100 atm.) or under the influence of ultraviolet irradiation[4] at ambient temperature. The temperature at which the thermal preparation was performed was critical. At 110° and approximately 100 atm. little or no reaction was observed, whereas increasing the temperature to 135° resulted in the fluorination of the sulfur dioxide to sulfuryl fluoride and the formation of nitrogen, nitrogen trifluoride, and small amounts of thionyl fluoride, nitric oxide, and nitrogen dioxide. A similar temperature effect on the reaction between sulfur and tetrafluorohydrazine has been observed. At 135° pentafluorosulfur difluoramine was obtained,[1] while at 220° only sulfur tetrafluoride and sulfur hexafluoride were isolated.[5]

The major sulfur-containing by-product in both the photolytic and thermal reactions was sulfuryl fluoride. No evidence was found in either system for the presence of sulfonyl difluoramine, $SO_2(NF_2)_2$. This suggests that the F_2NSO_2 radical is not an important species in the N_2F_4–SO_2 system and that fluorination of sulfur dioxide to form the FSO_2 radical occurs as the initial step. Coupling of this radical with the difluoramino radical would yield the observed product. The active fluorine in the photolytic reaction is believed to arise from the decomposition of the electronically excited difluoramino radical into fluorine radicals and N-fluoronitrene.[4,6] The source of fluorine in the thermal reaction is more obscure. Since the difluoramino radical is present at low concentration in this system because of the high pressures employed, fluorination by tetrafluorohydrazine rather than the difluoramino radical cannot be ruled out.

The preparation of fluorimidodisulfuryl fluoride was readily accomplished by fluorinating imidodisulfuryl fluoride at ambient temperature. The product was

$$F_2 + (FSO_2)_2NH \longrightarrow (FSO_2)_2NF + HF$$

obtained in a semiquantitative yield and only trace amounts of nitrogen trifluoride and sulfuryl fluoride were found. Fluorimidodisulfuryl fluoride reacts with mercury, so Kel-F oil covered manometers were employed during vacuum line manipulations.

The infrared spectra of both difluoramidosulfuryl fluoride and fluorimidodisulfuryl fluoride contain bands attributable to the S=O asymmetric and symmetric stretching modes. These bands occur at 6.72 and 8.00 μ in the spectrum of the former compound and at 6.69 and 8.02 μ in the latter[7,8] (see Fig. 1 and 2). The NF stretching modes[9–11] are assigned to the band at 9.8 μ in the spectrum of fluorimidodisulfuryl fluoride and to the bands at 10.00 and 10.87 μ in the spectrum of difluoramidosulfuryl fluoride. The S–F stretching vibration in the latter compound is attributed to the very strong band at 11.82 μ. The same mode in NF_2OSO_2F is displaced to slightly longer wave length, *viz.*, 11.93 μ.[10] The asymmetric and symmetric SF stretching modes in fluorimidodisulfuryl fluoride occur at 11.15 and 11.78 μ. These modes are found at 11.46 and 12.14 μ in pyrosulfuryl fluoride[8] which is believed to be related in structure to $(FSO_2)_2NF$.

The F^{19} n.m.r. spectrum of difluoramidosulfuryl fluoride contains a broad band at $-41.7\ \phi$ which is assigned to the fluorine bound to nitrogen and a sharp one at $-24.6\ \phi$ which is due to the S–F group. The area ratio of the two bands (2.00:1) is consistent with the above assignment. Resonance bands for fluorimidodisulfuryl fluoride were found at $-44.9\ \phi$, which is attributed to the fluorine bound to sulfur, and $+28.5\ \phi$, which is due to the N–F fluorine. The ratio of the low-field band to the high-field band is 2.05:1. The large differences in the field positions of the S–F and N–F resonance bands in the two compounds is unexplained. However, the upfield shift in the NF band upon substitution of another group or atom for fluorine is also evident when the F^{19} n.m.r. spectra of chlorodifluoramine ($-141\ \phi$) and dichlorofluoramine ($-129\ \phi$) are compared.[12]

Both difluoramidosulfuryl fluoride and fluorimidodisulfuryl fluoride were found to undergo hydrolysis upon contact with aqueous alkaline solutions. Practically all of the nitrogen-containing species were retained in solution during reaction of the latter compound. Analysis of the solution showed the presence of NO_2^-, $NH_2SO_3^-$, F^-, and SO_4^{2-} ions. Therefore hydrolysis of fluorimidodisulfuryl fluoride may follow the equation

$$3(FSO_2)_2NF + 12OH^- \longrightarrow 2NH_2SO_3^- + NO_2^- + 5F^- + 4SO_3F^- + 4H_2O$$

The hydrolysis of difluoramidosulfuryl fluoride was more complicated. Nitrogen, nitrous oxide, tetrafluorohydrazine, and difluorodiazine were evolved from the solution. The presence of difluorodiazine and tetrafluorohydrazine may arise from the decomposition or oxidation of the NF_2^- ion, which might exist as an active intermediate in solution.[13]

Experimental

Materials.—Imidodisulfuryl fluoride was prepared by the method of Appel.[14] Some difficulty was encountered in the purification of the product by distillation. However, treatment of the crude product with dry sodium chloride in methylene chloride solution followed by distillation was found satisfactory. Fluorine and sulfur dioxide were obtained from the Matheson Company and tetrafluorohydrazine from E. I. du Pont de Nemours and Company.

Preparation of FSO_2NF_2. (A) Thermal Method.—The reaction was performed in a 10-ml., 0.25-in. o.d. copper tube bomb, equipped with a needle valve through which equimolar quantities

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[5] B. Tittle and G. H. Cady, *Proc. Chem. Soc.*, 65 (1963).

[6] M. Lustig, C. L. Bumgardner, and J. K. Ruff, *Inorg. Chem.*, **3**, 917 (1964).

[7] F. B. Dudley and G. H. Cady, *J. Am. Chem. Soc.*, **79**, 573 (1957).

[8] H. C. Clark and H. J. Emeléus, *J. Chem. Soc.*, 190 (1958).

[9] M. K. Wilson and S. R. Polo, *J. Chem. Phys.*, **20**, 1718 (1952).

[10] J. H. Simons, "Fluorine Chemistry," Vol. II, Academic Press, 1954, p. 498.

[11] M. Lustig and G. H. Cady, *Inorg. Chem.*, **2**, 662 (1963).

[12] B. Sukornek, R. F. Stahlane, and J. Gordon, *ibid.*, **2**, 875 (1963).

[13] K. J. Martin, Gorgas Laboratory, unpublished work.

[14] R. Appel and G. Eisenhauer, *Ber.*, **95**, 246 (1962).

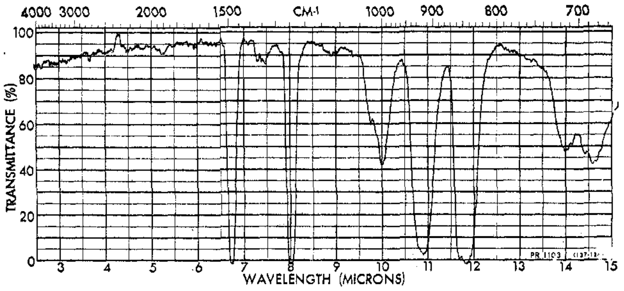

Fig. 1.—Infrared spectrum of FSO_2NF_2 obtained at 24 mm. pressure. The small peak between 7.3 and 7.5 μ is SO_2 impurity.

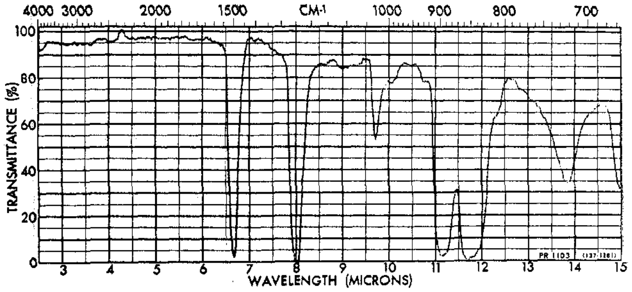

Fig. 2.—Infrared spectrum of $(FSO_2)_2NF$ at 12 mm. pressure. The band at 13.9 μ is due to window absorption.

of SO_2 and N_2F_4 were condensed from a conventional vacuum line. Optimum results were obtained at 100 atm. and 120 ± 1° for 6 days; yield >60% based on SO_2 consumed. Eighty per cent of the SO_2 had been consumed. The observed by-products were N_2, SO_2F_2, SOF_2, and small amounts of NF_3, NO, N_2O, and NO_2. The FSO_2NF_2 was isolated by gas chromatography at 25° using a 20 ft. × 0.25 in. column packed with Dow 710 silicone on Chromosorb W. Further purification by vacuum distillation was necessary to remove the last traces of SO_2F_2 and SOF_2.

(B) **Photolytic Method.**—A mixture of 10 mmoles of N_2F_4 and 10 mmoles of SO_2 was irradiated in a Pyrex vessel with an internally sealed cold cathode mercury resonance (2537 Å.) lamp for 2.5 hr. or until the pressure stopped decreasing. Mass spectral analysis indicated that 8.9 mmoles (89% yield) of FSO_2NF_2 and 3 mmoles of N_2F_2 were formed along with small amounts of N_2O, NO, SiF_4, SOF_2, and SO_2F_2. Products were purified by vacuum line fractionation and gas chromatography as above.

Preparation of $(FSO_2)_2NF$.—Imidodisulfuryl fluoride, 1.9 g., was fluorinated in a glass trap at 25° with a 1:9 mixture of fluorine and helium. The flow rate of F_2 was approximately 2×10^{-2} mole/hr. and the reaction was complete in 2.5 hr. The product, 2.1 g., was retained in a −126° trap placed downstream in the flow system. The only major by-product, silicon tetrafluoride, which presumably arose from the reaction of HF with glass, was found in a −196° trap.

Analysis.—Nitrogen was determined by the Dumas method for both compounds. In order to determine sulfur, the compounds were hydrolyzed as described below. An excess of a 0.1 *N* NaOH solution was admitted to a bulb containing a known weight of each compound. When FSO_2NF_2 was used a gaseous phase formed above the solution. Mass spectral analysis showed that it consisted of N_2F_2, N_2O, and N_2. The liquid phase was heated at 100° for several days to ensure complete hydrolysis of the fluorosulfate to sulfate. Sulfur was then determined gravimetrically as $BaSO_4$. When $(FSO_2)_2NF$ was employed no gas phase formed. The solution was treated as above. Analysis showed the presence of NO_2^-, $SO_3NH_2^-$, F, and SO_4^{2-} ions. Sulfate was determined volumetrically by titration with $BaCl_2$ using a tetrahydroxyquinone indicator. The fluoride content of the solution was determined after distillation by the thorium nitrate method.

Anal. Calcd. for FSO_2NF_2: N, 10.36; S, 22.2. Found: N, 9.98; S, 22.7. Calcd. for $(FSO_2)_2NF$: N, 7.03; S, 32.3; F, 28.2. Found: N, 7.11; S, 32.7; F, 28.8.

Physical Properties.—The physical and spectral properties were determined by known methods. A summary of the physical properties of FSO_2NF_2 and $(FSO_2)_2NF$ is presented in Table 1.

(A) **Melting Point.**—The melting points of FSO_2NF_2 and $(FSO_2)_2NF$ were observed visually by (1) placing the sample in a cold pentane bath which was allowed to warm at a rate of 0.25°/min. The melting point of FSO_2NF_2 was also determined by (2) the method of Stock.[15]

(B) **Vapor Pressures.**—The vapor pressure of FSO_2NF_2 as a function of temperature was measured with a Pyrex spoon gauge immersed in a bath at various temperatures. Some observed pressures are: −79.2°, 33.6 mm.; −55.0°, 117.3 mm.; −40.2°, 283.3 mm.; −28.0°, 477.8 mm.; and −19.6°, 597.7 mm. The Clausius–Clapeyron equation, valid for pressures above 40 mm., is $\log p = 7.518 - 1184/T$. The boiling point, latent heat of vaporization, and Trouton constant are shown in Table I. The vapor pressure measurements for $(FSO_2)_2NF$ were taken by the method of Kellog and Cady.[16] Some of the observed pressures are: 8.5°, 83.5 mm.; 29.0°, 216.7 mm.; 48.0°, 475.9 mm.; and 60.7°, 756.7 mm. The Clausius–Clapeyron equation, valid for pressures greater than 80 mm., is $\log p = 7.980 - 1703/T$. The boiling point and other related quantities for $(FSO_2)_2NF$ are tabulated in Table I. No vapor pressures were measured above the respective boiling points.

(C) **Molecular Weights.**—The molecular weight of FSO_2NF_2 was determined by two methods, mass spectrometrically by observing the effusion rate using mass 52 as the reference peak and by vapor density measurements assuming ideal gas behavior. That of $(FSO_2)_2NF$ was determined only by vapor density measurements.

(D) **Liquid Densities.**—The density of liquid FSO_2NF_2 was determined in a single capillary pycnometer. The temperature dependence is given by the equation $\sigma t = 1.545 - 0.00308t$, where t is °C. The volume coefficient of expansion is 0.00199 at 0°.

(E) **Infrared Spectra.**—The infrared spectra of FSO_2NF_2 and $(FSO_2)_2NF$ were taken in the NaCl region using a Perkin-Elmer Infracord spectrometer and are shown in Fig. 1 and 2. Their spectra in the KBr region were also taken using a Perkin-Elmer Model 21 spectrophotometer. Bands observed for $(FSO_2)_2NF$ in the KBr region were 15.15 (m) and 17.8 μ (vs). Bands observed for FSO_2NF_2 were 14.60 (w), 16.56 (m), 18.87 (w), 20.7 (w), and 21.4 μ (w).

(F) **Mass Spectra.**—A Consolidated Engineering Corporation Model 21-620 spectrometer was used with a heated inlet system and an ionization potential of 60 volts. The mass number, species, and relative abundance for some of the more prominent peaks for FSO_2NF_2 are as follows: 32, S^+ or SO_2^{2+}, 5.1; 33, NF^+, 11.2; 48, SO^+, 7.6; 52, NF_2^+, 12.0; 64, SO_2^+, 9.6; 67, SOF^+, 25.0; 83, SO_2F^+, 100; and 85, $^{34}SO_2F^+$, 4.8; and for $(FSO_2)_2NF$: 48, SO^+, 6.6; 64, SO_2^+, 4.4; 83, SO_2F^+, 100; 85, $^{34}SO_2F^+$, 5.4; and 97, NSO_3F^+, 8.2.

TABLE I

PHYSICAL PROPERTIES OF FSO_2NF_2 AND $(FSO_2)_2NF$

	FSO_2NF_2	$(FSO_2)_2NF$
Melting point range		
Method 1	−110.5 ± 0.5°	−79.9 ± 0.3°
Method 2	−111.3 ± 0.7°	. . .
Boiling point (extrapolated)	−18.2 ± 0.7°	60.8 ± 1.0°
Latent heat of vap., kcal./mole	5.43	7.80
Trouton constant, e.u.	21.29	23.3
Molecular weight		
Vapor density	136.2 (calcd. 135.1)	203 (calcd. 199)
Mass spectrometry	136	

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(15) A. Stock, *Ber.*, **50**, 156 (1917).

(16) K. B. Kellog and G. H. Cady, *J. Am. Chem. Soc.*, **70**, 3986 (1948).

(G) **N.m.r. Spectra.**—The F^{19} n.m.r. spectra of $(FSO_2)_2NF$ and FSO_2NF_2 were obtained on a Varian Model V4310A spectrometer operating at 40 Mc. Samples were measured neat at 25° for $(FSO_2)_2NF$ and $-50°$ for FSO_2NF_2. Trichlorofluoromethane was used as an external reference.

Additional Characterization of $(FSO_2)_2NH$.—In addition to the physical constants of $(FSO_2)_2NH$ given by Appel and Eisenhauer,[14] a single sharp F^{19} resonance was found at $-56.9\ \phi$ and a proton resonance at -9.0 p.p.m. (neat *vs.* TMS). Infrared bands were observed at 3.3, 3.5, 3.7, 6.8, 7.3, 8.5, 8.9, 11.7, 12.6, and 13.2 μ.

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CONTRIBUTION FROM BELL TELEPHONE LABORATORIES,
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The Structure of Ferric Chloride in Neutral and Acid Solutions

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X-Ray diffraction and spectral studies demonstrate that the structure of a concentrated solution of $FeCl_3$ in water is strongly influenced by the presence of H^+ ions. While the coordination about the Fe^{+3} is largely octahedral in concentrated, neutral solutions, the addition of acid results in the formation of a polymer consisting of alternating $FeCl_4$ (tetrahedral) and $FeCl_4(H_2O)_2$ (octahedral) units, with adjacent units sharing a chloride ion. It is also pointed out that the polymerization phenomenon is found only in the concentrated solutions (*ca.* 5 *M* in $FeCl_3$) and not in the dilute solutions normally investigated. About 75% of the Fe^{+3} in neutral solution appears to be $FeCl_4(H_2O)_2$ as deduced from its spectral similarity to the octahedral species in acid solution.

We wish to discuss some further observations on the structure of concentrated (*ca.* 5 *M*) $FeCl_3$ solutions. Earlier X-ray diffraction work[1] was taken to indicate that a significant part of the iron in concentrated aqueous $FeCl_3$ solutions exists as $FeCl_6^{-3}$ in octahedral coordination. On the other hand, Standley and Kruh[2] studied "neutral"[3] and concentrated HCl solutions of $FeCl_3$ using the same technique and claimed that the principal species in *both* solutions is tetrahedral $FeCl_4^-$. In agreement with our work, Standley and Kruh also found that there does not appear to be any iron–water peak in the radial distribution function (RDF) of these solutions. Long before the X-ray work was performed, it already had been determined that in dilute (0.68 *M*) solutions of $FeCl_3$ in concentrated HCl more than 90% of the iron is present as $FeCl_4^-$.[4] Unlike the results found in dilute solution and in contrast to the conclusion reached by Standley and Kruh, who based their analysis mainly on the first peak in the RDF, our recent study of the RDF and optical spectra of concentrated solutions of $FeCl_3$ in H_2O and concentrated HCl leads us to the conclusion that octahedral and tetrahedral forms exist in equilibrium in the solution with octahedral predominating in neutral solution and both octahedral and tetrahedral present in about equal amounts in strongly acidic solutions. In this publication we present the evidence for this and offer an interpretation of our experiments, which is preliminary, but nonetheless of sufficient interest to present at this time.

The RDF curves and the Cl:Fe ratios of the "neutral" and HCl solutions are shown in the upper portion of Fig. 1. The Fe^{+3} mole fractions in the three solutions are equal, the compositions being $Fe_{0.067}Cl_{0.201}(H_2O)_{0.731}$, $Fe_{0.067}Cl_{0.201}(H_2O)_{0.698}(HCl)_{0.034}$, and $Fe_{0.067}Cl_{0.201}(H_2O)_{0.656}(HCl)_{0.078}$ in solutions 1, 2, and 3, respectively. In the water solution (curve 1), the strong Cl–Cl peak at 3.2 Å., consistent with the 3.25 Å. separation expected for *cis* interaction in octahedral coordination, is evident. While the Cl–H_2O peak is also expected in the 3.2 Å. region, the observed intensity is much too high to be explained solely by this interaction.[1,5] The peak at 2.3 Å. is due to Fe–Cl bonding and a consideration of its area shows that there are,

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(1) G. W. Brady, *J. Chem. Phys.*, **26**, 1371 (1958).

(2) G. L. Standley and R. F. Kruh, *ibid.*, **34**, 1450 (1961).

(3) The "neutral" solutions are prepared by the addition of $FeCl_3 \cdot 6H_2O$ to pH 7 water and have a pH of 2 due to a slight hydrolysis.

(4) H. L. Friedman. *J. Am. Chem. Soc.*, **74**, 5 (1952).

(5) Standley and Kruh in their analysis of the "neutral" solution RDF dismissed any contribution of the octahedral Cl–Cl interaction to the peak area at 3.2 Å. Considering the position and the area (490 electrons^2 (el.^{2})) of this peak, we conclude that a strong Cl–Cl contribution is involved. The only other interactions of any possible importance at this distance are the H_2O–H_2O and H_2O–Cl contacts. Standley and Kruh's attempt to account for a large part of the 3.2 Å. area by postulating that 100% of the water participates in tetrahedral H_2O–H_2O interactions (2.9 Å.) is of questionable value because this necessitates an extended solvent structure wherein the presence of the ions is ignored. Since the ratio of ions ($Fe^{+3} + 3Cl^-$) to H_2O at the concentration studied is 1:2.6, there is barely enough solvent for the necessary hydration of the ions, and the tetrahedral water structure cannot possibly exist. This is further evidenced by the lack of a definite peak at 2.9 Å. As pointed out in an earlier paper,[1] we could not detect any Fe–H_2O interaction (expected at 2.0 Å.) and conclude that the H_2O forms a hydration sheath around the Cl atoms, contributing 192 el.^{2} to the area at 3.2 Å. and leaving 298 el.^{2} to be accounted for. Assuming that there are 3.6 H_2O groups about each Cl results in each H_2O having about two H_2O neighbors with a resultant contribution of 72 el.^{2} to the scattering at 3.2 Å. The remaining 226 el.^{2} is then most logically assigned to the *cis* Cl–Cl interaction in an octahedral complex.