deductions of a 1:1 molar composition from the thermodynamic study of the *p*-xylene and *p*-dichlorobenzene Ni(4-mepy)$_4$(SCN)$_2$ by Hart and Smith.[3] Using our single crystal data, we were able to satisfactorily interpret the powder data given in their paper, as shown in Table II. There is no doubt that the clathrate lattice was the same in both investigations.

It was not possible to identify the powder data for the unclathrated complex on the basis of the tetragonal cell of the clathrates and this supports Hart and Smith's conclusion that the clathrated and unclathrated complexes have different crystal structures.

CONTRIBUTION FROM THE GENERAL CHEMICAL DIVISION,
ALLIED CHEMICAL CORPORATION, MORRISTOWN, NEW JERSEY

Dichlorofluoramine[1]

BY BERNARD SUKORNICK, RICHARD F. STAHL,
AND JOSEPH GORDON

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The recent preparation of chlorodifluoramine[2] raised the question of the existence of its more highly chlorinated analog, dichlorofluoramine. We now wish to report the synthesis and characterization of this compound.

Experimental

In a 0.25-in. diameter copper U-tube surrounded by an ice bath was placed 15.0 g. (0.23 mole) of powdered sodium azide. Chlorine monofluoride, diluted with nitrogen, was passed over the solid for 8 hr. at the rate of 2.4 g. (0.044 mole) per hour. During the first 30 min. of reaction, chlorine azide formed almost exclusively. Thereafter, $FNCl_2$ appeared as the major product accompanied by small quantities of F_2NCl. The latter two gases were collected in a −80° trap. Subsequent distillation through an all-metal still equipped with a vapor-phase take-off provided 3.6 g. (20%) of $FNCl_2$, b.p. −2 to −3°. *Anal.* Calcd.: F, 18.28; Cl, 68.24; N, 13.48. Found: F, 18.13; Cl, 68.10; N, 13.47.[3]

The mass spectrum of dichlorofluoramine, obtained on a Consolidated Electrodynamics Model 21–103 mass spectrometer, is given in Table I. The cracking pattern provides strong corroborative evidence for the postulated structure. In particular, observation of the proper chlorine isotope effect for the parent peak confirms its identity.

The absence of any significant parent peak for NF_2Cl would indicate that the sample was relatively free of this compound.

One band was observed in the F^{19} n.m.r. spectrum of dichlorofluoramine. From the data given in Table II, it can be seen that this signal lies very close to the F^{19} chemical shifts for NF_3 and NF_2Cl. Therefore, it is quite likely that the N–F bonds in all three compounds are closely related electronically.

The infrared spectrum of dichlorofluoramine contains two strong bands at 12.0 and 12.2 μ. Both bands lie well within the N–F stretching region, although the one at 12.0 μ is probably best identified with the N–F stretch in $FNCl_2$.

The compound absorbed at 2700 Å. in the ultraviolet. On exposure to a 360-watt high pressure mercury ultraviolet lamp, $FNCl_2$ partially decomposed to *cis*-difluorodiazine and, presumably, chlorine.

TABLE I

FRAGMENTATION PATTERN OF $NFCl_2$

m/e	Ion	Relative intensity
14	N^+	16.8
19	F^+	5.2
24.5	NCl^{+2}	0.80
25.5		0.28
33	NF^+	17.5
49	NCl^+	100
51		32.2
68	$NClF^+$	77.9
70		
84	NCl_2^+	1.6
86		1.1
88		0.19
103	NCl_2F^+	1.68
105		1.05
107		0.20

TABLE II

F^{19} N.M.R. AND INFRARED SPECTRA OF SOME N–F COMPOUNDS

Compound	F^{19} chem. shift, p.p.m.[a]	Principal N–F str. bands, μ
NF_3	-145 ± 1	9.7, 11.0
NF_2Cl[1]	-140.6	10.8, 11.7
$NFCl_2$	-128.7	12.0

[a] Measured relative to $CFCl_3$.

Small quantities of dichlorofluoramine also are formed by the reaction of chlorine azide and chlorine monofluoride. This fact, together with the initial formation of chlorine azide from sodium azide and chlorine monofluoride, suggests that ClN_3 is an intermediate in the formation of $FNCl_2$ *via* the NaN_3–ClF reaction

$$NaN_3 + ClF \longrightarrow ClN_3 + NaF$$

$$ClN_3 \longrightarrow [ClN<] + N_2$$

$$[ClN<] + ClF \longrightarrow FNCl_2$$

At present, however, there is no clear-cut evidence for the existence of the postulated chlorazene intermediate, ClN<.

Milligan[4] photolyzed samples of chlorine azide trapped in an argon matrix at 4.2°K. He attributed the appearance of infrared absorptions at 818 and 824 cm.$^{-1}$ on irradiation and the disappearance of these bands on warming to the formation and decomposition of chlorazene. Dimerization of ClN< also was suggested, but no dichlorodiazene, ClN=NCl, could be isolated.

Although no explosions have been encountered in handling gaseous $FNCl_2$, the liquid is extremely sensitive to friction and shock.

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[2] R. C. Petry, *J. Am. Chem. Soc.*, **82**, 2400 (1960).

[3] Chlorine and fluorine analyses were obtained by reaction with sodium in liquid ammonia at 25° for 148 hr. Nitrogen was determined by a modified Dumas method.

[4] D. E. Milligan, *J. Chem. Phys.*, **35**, 372 (1961).

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF WISCONSIN, MADISON, WISCONSIN

A Silicon Metal Trap for the Safe Disposal of Chlorine Trifluoride

BY RALPH G. CZEREPINSKI AND JOHN L. MARGRAVE

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The standard disposal method for chlorine trifluoride involves the use of a soda lime tower.[1,2] The chlorine