Chemical shifts of methyl protons in trimethylamine adducts decrease in the order $BBr_3 > BCl_3 > BH_3 \geq BF_3 > BMe_3$.[25,26] The simplest interpretation of these results is that they reflect relative electron-withdrawing ability of the acids, but it is well known that with other compounds such a simple explanation often fails. Miller and Onyszchuk show good correlations between the chemical shifts and heats of coordination, dipole moments, and C=O stretching frequencies of corresponding methyl acetate complexes.

From a study of dipole moments Bax, Katritzky, and Sutton have arrived at the following relative order of B–N moments in Me_3NBX_3 adducts: $BF_3 < BH_3 \leq BCl_3 < BBr_3$.[27] In contrast to the greater acceptor strength of BH_3 than BF_3 which was deduced from the dipole and nmr results, Taylor finds the B–N valence force constant much larger for H_3NBF_3 (4.14 mdynes/A) than for H_3NBH_3 (2.90 mdynes/A).[28] The inference of relative bond strengths from force constants involves an assumption about the form of the potential, but this is a much less drastic assumption than those inherent in the above interpretations of nmr spectra and dipole moments. Within the framework of the model which has been presented in this paper, the charge-transfer spectra and polarographic data may be interpreted in terms of the electron-withdrawing power of the acid. It is gratifying that these data agree with Taylor's by indicating BF_3 is a stronger acceptor than BH_3.

Like the nmr and bond moment measurements, the present charge-transfer spectra and oxidation potentials indicate the acceptor order $BBr_3 > BCl_3 > BF_3$. As Miller and Onyszchuk point out, this order was determined for the adducts and cannot be interpreted in terms of reorganization energies, which had been used to explain a similar order for heats of coordination.

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Aminophosphine Chains of CH_3N and CF_3P Units[1]

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Abstract: The new volatile compounds $CF_3P(NHCH_3)_2$ (I, mp −29°; bp estd 135°), $CF_3P(NHCH_3)Cl$ (II, bp estd 102°), and $CH_3N(CF_3PCl)_2$ (III, mp near −50°; bp estd 148°) were made as possible precursors of polymers of the CH_3NPCF_3 unit, and their vapor-phase infrared spectra were accurately recorded. I is stable up to *ca.* 200°, in contrast to the rapid self-aminolysis of a product seeming to be $CF_3P(NH_2)_2$. II is easily aminolyzed to I, and its easy thermal condensation yields CF_3PCl_2 and a slightly volatile product regarded as the P–N–P–N chain compound $CF_3PCl(CH_3NPCF_3)NHCH_3$. I and II are easily cleaved to CF_3PCl_2 by HCl, but III requires a temperature of 140° for such a result, and it is thermally stable above 200°. Aminolysis of III apparently forms the very slightly volatile $CH_3N(CF_3PNHCH_3)_2$, representing the open chain N–P–N–P–N. Numerous combinations of such compounds failed to yield evidence for the formation of $(CH_3NPCF_3)_n$ ring polymers.

The high stability of the compounds $HN[P(CF_3)_2]_2$ and $CH_3N[P(CF_3)_2]_2$, and their resistance to cleavage by dry ammonia or hydrogen chloride,[2] suggested that some stability might be retained in analogous compounds having more labile groups replacing CF_3. In fact, the new volatile compound $CH_3N(CF_3PCl)_2$ proved to be stable and quite resistant to cleavage by HCl, and it could be aminolyzed at the P–Cl bonds without extensive cleavage of the central P–N–P chain.

Also interesting was the quest for the yet unknown $(HNPCF_3)_n$ and $(CH_3NPCF_3)_n$, in view of the reported existence of analogous PN dimer or trimer rings with other side groups.[3] However, at least eight different approaches toward $(CH_3NPCF_3)_n$, including low-pressure vapor-flow methods (to favor small rings), led to no success. The dimer or trimer should be volatile but neither could be obtained by sublimation from the reaction mixtures, nor could anything intelligible be extracted by solvents. However, certain new compounds intended as polymer precursors seem worthy of description.

For example, the diaminophosphine $CF_3P(NHCH_3)_2$ proved to be stable at 180°, resisting the expected aminolytic action (observed at 210°) of the N–H bond to cleave off HCF_3. Such resistance to a reaction analogous to basic hydrolysis would imply a lack of nitrogen-base action, presumably affected by N_{2p} to P_{3d} π bonding.

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(1) Supported by Grant No. GP-3812 from the National Science Foundation, which contributed also through Grants GP-199 and G-14665 toward the purchase of the Beckman IR7 infrared spectrophotometer with NaCl and CsI optics.

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Equally interesting for the same reason is the resistance of the new aminochlorophosphine $CF_3P(NHCH_3)Cl$ to self-aminolysis. Although not a very stable compound, it could be isolated without great difficulty, and its decomposition was not only a condensation with loss of HCl, but also involved a P–N bond cleavage to form CF_3PCl_2.

The experimental work described in the following sections was done by means of a glass high-vacuum manifold, much the same as in the earlier study.[2] These methods made possible the isolation and quantitative study of volatile compounds, so that the equations for some reactions could be written directly from the experimental data. Any deviations from our usual methods are mentioned in the appropriate places.

Bis(methylamino)trifluoromethylphosphine

Quantitative Synthesis. Gaseous CF_3PCl_2 was introduced in small portions into a bulb containing dry methylamine at 25°, with results described by the following equation with quantities in millimoles.

$$\begin{array}{cccc} CF_3PCl_2 + & 4CH_3NH_2 \longrightarrow & CF_3P(NHCH_3)_2 + & 2CH_3NH_3Cl \\ 1.092 & 7.546 & 1.075 & 2.183 \\ & -3.224 & (172\ \text{mg}) & \\ \cline{2-2} & 4.322 & & \end{array}$$

The excess amine was removed through a high-vacuum trap at −50°, and the condensate from that trap was purified further by a high-vacuum reflux column at −33°. This product in a glass bulb at 20 mm pressure was heated up to 180° during 4 days without change, but it formed HCF_3 and unknown products on heating at 210°. In an attempt to form polymers of the CH_3NPCF_3 unit, $CF_3P(NHCH_3)_2$ vapor at a low pressure was passed over P_4O_{10} at temperatures up to 150°, without effect.

Far less stable was a product seeming to be $CF_3P(NH_2)_2$, formed in 75% yield by the reaction of CF_3PCl_2 with ammonia in excess. It melted above 0° but formed HCF_3 well below room temperature, and satisfactory purification did not seem possible. However, the molecular weight of one sample was 140 (calculated, 132), suggesting the formula $CF_3P(NH_2)_2$. Both this compound and $CF_3P(NHCH_3)_2$ would be weaker bases than ammonia (on account of N–P π-dative bonding); thus the great difference in their rates of self-aminolysis requires some explanation, such as steric inhibition by the methyl groups.

Physical Properties. Pure $CF_3P(NHCH_3)_2$ melted in the range −29.5 to −28.5°. Its normal volatility behavior, shown by Table I, is a further evidence of purity.

Table I. Volatility of Liquid $CF_3P(NHCH_3)_2$
($\log P = 7.1770 - 1.75 \log T - 0.0058T - 2650/T$;
$t_{760} = 134.5°$; Trouton constant, 22.4 eu)

Temp, °C	0.0	10.9	18.7	35.6	59.3	68.5	84.3
P_{obsd}, mm	1.42	3.12	5.19	14.6	48.9	74.5	144.3
P_{calcd}, mm	1.43	3.12	5.23	14.5	49.1	74.6	144.0

Confirmation of Formula. The molecular weight of the presumed $CF_3P(NHCH_3)_2$ was determined in the vapor phase as 161.9 (calculated, 160.08). Hydrolysis of 86.0 mg (0.537 mmole) in 20% NaOH (necessarily at 100°) gave 0.521 mmole of HCF_3, showing $0.97CF_3$ per molecule. Further confirmation of the formula came from the rapid reaction with HCl at 25°, according to the following equation with millimole stoichiometry.

$$\begin{array}{cccc} CF_3P(NHCH_3)_2 + & 4HCl \longrightarrow & CF_3PCl_2 + & 2CH_3NH_3Cl \\ 0.481 & 3.020 & 0.477 & 0.968 \\ (76.9\ \text{mg}) & -1.072 & & \\ \cline{2-2} & 1.948 & & \end{array}$$

Methylaminochlorotrifluoromethylphosphine.

Synthesis. The reaction of $CF_3P(NHCH_3)_2$ (mostly in the liquid phase) with gaseous CF_3PCl_2 in slight excess began very rapidly but was allowed 5 hr at 25° for completion. The results are summarized by the millimole stoichiometry accompanying the following equation

$$\begin{array}{lccc} CF_3P(NHCH_3)_2 + & CF_3PCl_2 \longrightarrow & & \\ \quad 2.425 & 2.532 & & \\ & -0.234 & CF_3P(NHCH_3)Cl + X + & CH_3NH_3Cl \\ \cline{2-2} & 2.298 & 3.622 & (0.39Cl^-) \end{array}$$

Here the main product was measured only after purification by the microscale, high-vacuum reflux column, operating first at −78° to remove the excess CF_3PCl_2 and then at −50° for delivery of the desired compound. The less volatile fraction X was employed in an experiment yet to be described.

More of this fraction X was formed when 1.471 mmoles of $CF_3P(NHCH_3)_2$ and 0.463 mmole of CF_3PCl_2 were heated together in a 4-ml sealed tube for 10 hr at 100°, in an attempt to make polymers of the CF_3PNCH_3 unit. Since the nonvolatile product contained only 0.3 mg-atom of Cl^-, most of the chlorine must have been in the slightly volatile products X. These could have included any or all of at least three kinds of condensation products having P–N–P linkages.

Purity and Formula. Since the compound $CF_3P(NHCH_3)Cl$ would have the same composition as an equimolar mixture of the components from which it was made, namely $CF_3P(NHCH_3)_2$ and CF_3PCl_2, it is necessary to demonstrate that our product was a single substance rather than a mixture. Its melting point could not be observed (for it formed a glass at low temperatures, softening near −140°); however, purity was indicated by the narrow temperature range for delivery from the reflux column, and the normal behavior of the vapor tensions shown in Table II confirms this. The infrared spectrum also left no doubt of the absence of the original reactants.

Table II. Volatility of $CF_3P(NHCH_3)Cl$
($\log P = 5.0210 + 1.75 \log T - 0.0033T - 2077/T$;
$t_{760} = 101.6°$; Trouton constant, 22.3 eu)

Temp, °C	0.0	9.4	21.2	47.4	54.8	68.0
P_{obsd}, mm	6.04	10.53	20.5	74.0	102.2	173.5
P_{calcd}, mm	6.02	10.65	20.6	74.0	102.2	173.7

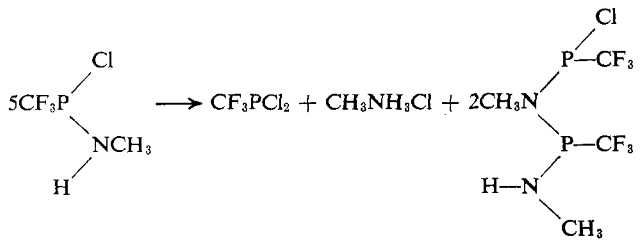
The vapor-phase molecular weight of this product was determined as 167 (calculated, 165.5). Hydrolysis of a 0.265-mmole sample in 10% NaOH (10 hr, 80°) yielded 0.263 mmole of HCF_3. Further confirmation of the formula was accomplished by the quantitative cleavage action of HCl and the similar action of methylamine; the former was extensive at 25° and was completed during 12 hr at 100°, while the latter was immediate in a 1-l. bulb at 25° and was allowed 5 hr for completion. Thus, with millimole stoichiometry

$$\begin{array}{cccc} CF_3P(NHCH_3)Cl \; + & 2HCl & \longrightarrow CF_3PCl_2 \; + & CH_3NH_3Cl \\ 0.245 & 1.504 & 0.247 & 0.244 \\ & \underline{-1.021} & & \\ & 0.483 & & \end{array}$$

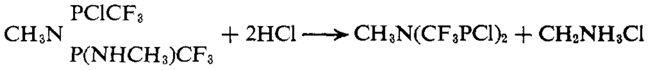
$$\begin{array}{cccc} CF_3P(NHCH_3)Cl \; + & 2CH_3NH_2 & \longrightarrow CF_3P(NHCH_3)_2 \; + & CH_3NH_3Cl \\ 0.388 & 1.386 & 0.344 & 0.347 \\ & \underline{-0.707} & (55\ \text{mg}) & \\ & 0.679 & & \end{array}$$

For both of these reactions, the CH_3NH_3Cl was determined by titration of chloride.

Thermal Condensation. This aminochlorophosphine proved to be appreciably unstable; for example, over a 5-hr period during and after the volatility measurements (Table II), the equilibrium pressure at 0° rose to 6.5 mm, corresponding to a slight formation of CF_3PCl_2 and less volatile material. The more extensive decomposition at 100° is summarized most simply by the somewhat idealized equation



The same main product was obtained in a series of experiments involving mixtures of CF_3PCl_2, $CF_3P(NHCH_3)_2$, and $(CH_3)_3N$, in the liquid or vapor phase or in solution in hexane isomers. It appeared as a colorless liquid having about 0.25 mm vapor tension at 25°. It was analyzed by reaction with HCl at 25°, according to the presumptive equation



$$CH_3N\begin{array}{l} PClCF_3 \\ P(NHCH_3)CF_3 \end{array} + 2HCl \longrightarrow CH_3N(CF_3PCl)_2 + CH_2NH_3Cl$$

However, this conversion was not cleanly simple, for some minor proportions of CF_3PCl_2 appeared in two experiments. For this reason, it was necessary to sum up the component atoms and groups of all products, as quantitatively determined, and subtract the consumed HCl to determine the component parts of the original sample. The results are shown in Table III, with calculated values in parentheses.

Table III. Component Groups in the N–P–N–P–Cl Compound

Sample wt, mg	235	103
Calcd, mmole	0.798	0.350
CF_3P	1.608 (1.596)	0.714 (0.770)
Cl	0.813 (0.798)	0.338 (0.350)
CH_3N	1.545 (1.596)	0.650 (0.700)
N–H bonds	0.687 (0.798)	0.310 (0.350)

These results strongly suggest the assumed formula for the main product of thermal decomposition of $CF_3P(NHCH_3)Cl$. However, its samples never seemed pure enough for any very meticulous characterization. The vapor-phase molecular weight results were 310 and 320 (calculated, 295), and the appreciably low analyses for CH_3N and N–H units might suggest that some CH_3NH groups were replaced by CF_3PCl units on phosphorus. In any case, it appears that this product was an important component of the difficultly volatile fraction previously designated as "X."

This thermal condensation product of $CF_3P(NHCH_3)Cl$ would represent a long step toward formation of polymers of the CH_3NPCF_3 unit, but attempts to complete the process have not yet led to intelligible results. In one experiment, a sample of the condensation product was treated with trimethylamine at elevated temperatures, with some removal of HCl. The desired result would have been a ring closure to form the dimer $(CH_3NPCF_3)_2$, but there was no product which could be volatilized without decomposition. In another experiment, $CF_3P(NHCH_3)Cl$ in a relatively large bulb at 25° absorbed $(CH_3)_3N$ in a 1:1 molar ratio, forming only nonvolatiles. Also, a direct reaction of CF_3PCl_2 with $3CH_3NH_2$ at 100° led to a nonvolatile hexane extract for which an analysis by the HCl method indicated the composition $CF_3P(NHCH_3)_2 \cdot 4CH_3N(CF_3PNHCH_3)_2$. Finally, sodium failed to react with the main condensation product of $CH_3P(NHCH_3)Cl$ in tetrahydrofuran at 25°, and at 60° it reacted to form HCF_3 and nonvolatiles.

Methyliminobis(trifluoromethylchlorophosphine)

Synthesis. The first sample of $CH_3N(CF_3PCl)_2$ was made by the action of HCl in excess (5-ml tube, 25°) upon the slightly volatile product X, which was described in relation to the synthesis of $CF_3P(NHCH_3)Cl$. The stoichiometry was as follows.

$$\begin{array}{cccccc} X \; + & HCl & \longrightarrow & CF_3PCl_2 \; + & CH_3N(CF_3PCl)_2 \; + & \text{nonvol} \\ 102.5 & 1.981 & & 0.060 & 0.326\ \text{mmole} & 0.323Cl^- \\ \text{mg} & \underline{-1.223} & & \text{mmole} & (97.7\ \text{mg}) & (\text{mg-atom}) \\ & 0.759 & & & & \\ & \text{mmole} & & & & \end{array}$$

On the assumption that the nonvolatile product here was all CH_3NH_3Cl, this reaction balance would suggest that X was mostly a mixture of $(CH_3NPCF_3)_n$, $CH_3N(CF_3PCl)_2$, and the condensation product $CF_3PCl(CH_3NPCF_3)NHCH_3$. Any of these could react with HCl to produce major yields of $CH_3N(CF_3PCl)_2$.

A more direct synthesis of $CH_3N(CF_3PCl)_2$, from CF_3PCl_2 by aminolysis, was explored in a series of experiments showing that the optimum ratio of CH_3NH_2 to CF_3PCl_2 was in the range 1.2 to 1.4. The initial 25° reaction had to be continued in a sealed tube for at least 12 days at 100°, presumably because the initially formed $CF_3P(NHCH_3)Cl$ thus reacts further with CF_3PCl_2. Even so, this aspect of the process was difficult to complete, so that the samples still contained $CF_3P(NHCH_3)Cl$ which could not be removed by the usual distillation methods. Hence for purification of the $CH_3N(CF_3PCl)_2$, HCl was employed at 25° to convert $CF_3P(NHCH_3)Cl$ to the easily removable CF_3PCl_2. Then it became fairly easy to purify the desired $CH_3N(CF_3PCl)_2$ by high-vacuum fractional condensation. In this process, a trap at −10° removed a trace of less volatile material; the main product condensed out at −50 to −60°; CF_3PCl_2 was held at −100 to −120°; and HCl went through to the final trap at −196°. The same purification method was used also for syntheses from mixture X.

By this method, better than 85% yields of $CH_3N(CF_3PCl)_2$ could be obtained. The duration of heating at 100° proved to be fairly critical: a 2-hr heating gave no yield at all; and a 5-day heating gave a yield less than 60%, based upon the unrecovered CF_3PCl_2.

Physical Properties. The melting point of $CH_3N(CF_3PCl)_2$ was quite difficult to observe, for only by cooling to −196° and then warming to −78° could crystals be formed; then these melted in a fairly wide range near −50°. However, the vapor tensions showed a normal relation to temperature, as shown in Table IV.

Table IV. Volatility of $CH_3N(CF_3PCl)_2$ ($\log P = 4.915 + 1.75 \log T - 0.003T - 2260/T$; $t_{760} = 148$; Trouton constant, 22.2 eu)

Temp, °C	0.00	10.1	21.6	41.2	61.1	80.1
P_{obsd}, mm	1.24	2.36	4.91	14.25	36.7	82.6
P_{calcd}, mm	1.22	2.39	4.90	14.23	36.8	82.6

Confirmation of the Formula. The vapor-phase molecular weight of this product was 295; calculated for $CH_3N(CF_3PCl)_2$, 299.9. Hydrolysis in 20% NaOH (10 hr at 70°) gave $1.99HCF_3$ per mole, and the remaining aqueous solution was titrated to show $1.99Cl^-$ per mole. Cleavage by HCl failed to occur during 12 hr at 100°, even at 30 atm pressure of HCl; however, a 40-hr heating at 140° gave the following quantitative reaction, with data in millimoles.

$$\begin{array}{llll} CH_3N(CF_3PCl)_2 + & 3HCl \longrightarrow & 2CF_3PCl_2 + & CH_3NH_3Cl \\ 0.403 & 3.278 & 0.796 & 0.399 \\ & -2.074 & & \\ & \overline{1.204} & & \end{array}$$

Thermal Stability. A 4-day exposure of $CH_3N(CF_3PCl)_2$ to 190° had no effect, but during 3 days at 225°, the sample developed a slight yellow coloration, forming a trace of CF_3PCl_2. Thus this compound is appreciably less stable than the previously reported $CH_3N[P(CF_3)_2]_2$[2] and possibly more reactive toward HCl.

A Probable N–P–N–P–N Chain Compound

The reaction of $CH_3N(CF_3PCl)_2$ with CH_3NH_2 in excess gave a nonvolatile product containing $1.94Cl^-$ per mole of the original sample. Thus the main product could be presumed to be the new N–P–N–P–N chain compound $CH_3N(CF_3PNHCH_3)_2$.

The same product was obtained more directly by heating a mixture of 2.761 mmoles of CF_3PCl_2 and 8.302 mmoles of CH_3NH_2 for 40 hr at 100°. A difficult vacuum-line sublimation yielded 41 mg of a product melting in the range 25–30°, so slightly volatile that its vapor tension at 25° could not be measured with any confidence. Its quantitatively monitored reaction with HCl indicated the formula thus

$$\begin{array}{llll} CH_3N(CF_3PNHCH_3)_2 + & 4HCl \longrightarrow & CH_3N(CH_3PCl)_2 + & \\ 0.117\text{ mmole} & 0.967 & 0.118\text{ mmole} & \\ (34.0\text{ mg}) & 0.484 & (35.5\text{ mg}) & \\ & \overline{} & & 2CH_3NH_3Cl \\ & 0.483 & & 0.233\text{ mmole} \\ & \text{mmole} & & \end{array}$$

A sample of this product was employed with CF_3PCl_2 and $2(CH_3)_3N$, in an attempt to close the N–P–N–P–N into the ring compound $(CH_3NPCF_3)_3$. First the mixture of the gaseous reactants was passed over the P_2N_3 compound at 25°, and then the whole mixture was allowed to stand for 10 hr at 0°. The gases were absorbed but no product of interest could be vaporized out of the mixture.

The obverse ring closure also was attempted. A very dilute solution of $CH_3N(CF_3PCl)_2$ and $CF_3P(NHCH_3)_2$ in hexane reacted to some extent, but yielded no product having the volatility expected for $(CH_3NPCF_3)_3$. It seems most probable that $(P–N)_n$ polymer chains (with appropriate end groups) were formed in most of our attempts toward $(CH_3NPCF_3)_n$ rings, but that closure to small rings did not occur effectively under our experimental conditions.

Infrared Spectra

The observed infrared frequencies (cm^{-1}) for three of our new phosphinoamines are listed in Table V, with parenthetical relative intensities defined as before.[2]

Table V. Fundamental Infrared Peaks

Suggested assignment	$CF_3P<\begin{matrix}\overset{H}{NCH_3}\\ \underset{H}{NCH_3}\end{matrix}$	$CF_3P<\begin{matrix}Cl\\ \underset{H}{NCH_3}\end{matrix}$	$CH_3N<\begin{matrix}\overset{Cl}{PCF_3}\\ \underset{Cl}{PCF_3}\end{matrix}$
N–H stretching	3457 (1.5)	3449 (1.5)	. . .
	3423 (0.9)	3406 (1.9)	
C–H stretching	3036 (1.0)	2996 (1.1)	3017 (0.3)
	2965 (4.3)	2972 (1.7)	2968 (0.9)
	2935 (4.1)	2937 (1.8)	
	2915 (3.5)	2917 (1.1)	2909 (0.25)
	2826 (3.0)	2839 (1.0)	2838 (0.18)
N–H bending	1570 (0.2)	. . .	. . .
C–H bending	1485 (0.74)	1487 (0.63)	1466 (1.1)
	1465 (0.72)	1465 (0.56)	1432 (0.5)
	1435 (0.87)	1436 (0.46)	
	1384 (2.9)	1391 (1.7)	
Uncertain	1270 (0.25)	1293 (0.5)	1281 (0.56)
C–F stretching	1191 (22)	1181 (24)	1179sh (27)
	1145 (29)	1164 (21)	1172 (32)
	1119 (26)	1142 (28)	1151 (115)
		1137 (32)	
Fermi overtone	1088 (14)	1095 (6.4)	1091 (10)
CH_3 rocking;	967 (0.20)	995 (0.5)	
P–N stretching;	931 (0.14)	853 (0.2)	857 (24)
CH_3 wagging?	802 (4.0)	823 (3.1)	
		783 (0.8)	
CF_3 sym deform	746 (1.6)	746 (0.6)	739 (2.7)
Uncertain	700 (0.34)	695 (0.14)	644 (1.1)
CF_3 asym deform	551 (1.1)	550–65 (3.1)	557 (4.0)
			545 (3.8)
P–Cl stretching	. . .	493 (4.8)	521 (10)
P–C stretching;	491 (1.5)	483 (5.0)	467 (4.6)
P–N bending?	463 (1.1)	475 (4.8)	
CF_3 rocking	358 (0.9)	386 (1.0)	381 (6.8)
CF_3 wagging or	319 (0.9)	322 (1.1)	(Weak
CH_3 torsion	265 (0.2)	287 (1.0)	bands
		253 (0.8)	not seen)