substitution of D for H is visually evident in the spectra, and the difference between the shifts for NaB_3D_8 and NaB_3H_8 was measured as 0.50 ± 0.01 ppm on the spectrum in Figure 3. The peak separations of the multiplets measured on the spectra in Figures 1d,f and 2, are 1 to 2 Hz smaller than the J_{BH} value of 33.1 ± 0.1 Hz for NaB_3H_8, which was obtained in this study. While this difference may indicate the isotope effect on the coupling constants, the severe overlap of peaks in each of the spectra obscures the true coupling constant values for the partially deuterated triborate species. It was reported[11] that the ^{11}B signal of BD_4^- appears 0.54–0.58 ppm to high field of the BH_4^- resonance and that the J_{BH} value for BD_3H^- is 1 Hz lower than that for BH_4^-.

Experimental Section

Conventional vacuum line techniques were used for the handling of volatile chemicals. Deuterium chloride was prepared by treating PCl_3 with D_2O (99.8% isotopic purity) in the vacuum line. The PCl_3 sample was refluxed for 12 h under 1 atm of pressure of dry nitrogen gas before the D_2O treatment. The generated DCl was fractionated and stored in a stainless steel cylinder. The isotopic purity of the DCl sample as estimated by infrared spectroscopy was higher than 99%. Laboratory stock tetraborane(10) was treated with tetrahydrofuran to prepare the $THF{\cdot}B_3H_7$ sample.[12] Deuteration of $THF{\cdot}B_3H_7$ was performed by the method described elsewhere by using CH_2Cl_2 solvent.[7] The deuterated $THF{\cdot}B_3H_7$ samples were of about 97% isotopic purity. The purity was estimated by assuming complete H–D scrambling at each batch treatment of the $THF{\cdot}B_3H_7$ sample with the DCl. Sodium hydride (K and K Laboratories, Inc., 50% oil suspension) and NaD (Alfa Ventron, 20% in oil, 98% isotopic purity) were washed with diethyl ether in the vacuum line, and the resulting powder was handled in an atmosphere of dry nitrogen. Reagent grade diethyl ether, tetrahydrofuran, and diglyme were stored over $LiAlH_4$ and were distilled from the storage vessels into the vacuum line as needed.

The ^{11}B NMR spectra were recorded on a Varian XL-100-15 instrument equipped with a spin-decoupler unit (Gyrocode), the observed frequency being 32.1 MHz. The ^2H spin was decoupled by using the maxium decoupler power (119db or Hetero High) of the instrument. The samples were not spinned.

Reactions of $THF{\cdot}B_3H_7$ with NaH. A sample of $THF{\cdot}B_3H_7$ (1.20 mmol) was prepared in a 22-mm o.d. Pyrex reaction tube, and a diglyme solution (3 mL) of the adduct was prepared by condensing the solvent in the tube. The solution was transferred, in the vacuum line, into another similar reaction vessel which contained excess NaH (0.1 g). The reaction mixture was allowed to warm to 0 °C and stirred for 15 h. Unchanged NaH was then filtered in the vacuum line. No hydrogen gas evolved and no volatile borane compounds could be detected during this entire process. The ^{11}B NMR spectrum of the clear filtrate indicated the presence of only NaB_3H_8. The resonance signal appears at −29.7 ± 0.2 ppm (J_{BH} = 33.1 ± 0.1 Hz) ($BF_3{\cdot}O(C_2H_5)_2$ standard downfield shift being taken as positive). The experiment with diethyl ether as the solvent was run similarly.

Preparation of the Deuterated NaB_3H_8. Samples of $THF{\cdot}B_3D_7$ were prepared in large reaction vessels (100-mL long-necked flasks for 1.0–1.5 mmol of $THF{\cdot}B_3H_7$) so that a high $DCl/THF{\cdot}B_3H_7$ ratio could be used at each DCl treatment of the batch processes.[7] Generally the reaction mixtures of the triborane adducts and excess NaD (or NaH) were stirred at 0 °C overnight and then filtered into NMR sample tubes. In an experiment a diglyme solution of $THF{\cdot}B_3D_7$ was poured into a 10-mm o.d. Pyrex tube which contained excess NaH, and the tube was sealed. The tube was allowed to warm to room temperature and the reaction was monitored on the NMR instrument while the mixture was agitated occasionally. The triborate signal grew steadily at the expense of the triborane adduct signal and the reaction was virtually complete in 1 h. The ^{11}B NMR spectra of this resulting solution are shown in Figure 1c,d.

The NMR Samples. "$NaB_3H_8 + NaB_3D_8$". The mixture was prepared by breaking an ampule which contained a diglyme solution of NaB_3H_8 (1.3 mmol), in sealed glassware which contained a diglyme solution of NaB_3D_8 (1.4 mmol). The resulting solution was decanted into a 10-mm o.d. tube, which had been attached to the sealed glassware, and the 10-mm tube end was then inserted in the probe of the NMR instrument for the spectral measurements.

"$NaB_3H_4D_4$". A diglyme solution of NaB_3H_8 (1.2 mmol) was placed in a 10-mm o.d. tube, cooled to −196 °C, and 1.3 mmol of $THF{\cdot}B_3D_7$ was sublimed into the tube and the tube sealed. The tube was then allowed to warm to −80 °C and shaken to prepare a uniform solution. The solution was examined for its ^{11}B NMR spectra starting at −80 °C. Below 0 °C the resonance signals (^2H spin decoupled) were too broad to observe the progress of the H–D exchange reaction.

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Registry No. NaB_3D_8, 71341-77-0; NaB_3H_8, 12007-46-4; $THF{\cdot}B_3D_7$, 71341-54-3; $THF{\cdot}B_3H_7$, 12544-89-7; NaD, 15780-28-6; NaH, 7646-69-7; NaB_3D_7H, 71341-74-7; NaB_3H_7D, 71341-75-8; $NaB_3H_4D_4$, 71341-76-9.

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Metal Complexes of Cyclic Triamines. 3. A Kinetic Study of the Acid Hydrolysis of (1,4,7-Triazacyclononane)nickel(II) and (1,5,9-Triazacyclododecane)nickel(II)

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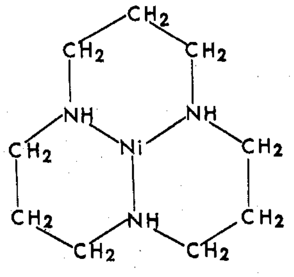
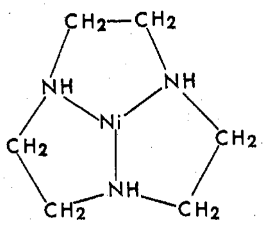
The acid hydrolysis of polyamine nickel(II) chelates proceeds rapidly unless ligand stereochemistry impedes the dissociation process. Thus macrocyclic and polysubstituted amines coordinate in such a fashion that they hinder the normal stepwise unwrapping of the ligand from the metal ion, and nickel(II) complexes of these compounds hydrolyze slowly in aqueous acid solution.[1] Similar inert kinetic behavior is shown by the two triamines tris(anhydro-*o*-aminobenzaldehyde) (TRI)[2] and *cis,cis*-1,3,5-triaminocyclohexane (*c*-tach).[3] Both these amines form tridentate complexes which can only coordinate to the face of the coordination polyhedron of a metal ion. Once a Ni–N bond is broken, the ligand structure restricts the translational freedom of the first nitrogen donor atom so that it cannot be removed very far from the metal.

We have previously reported[4] that the nickel(II) complexes of cyclic triazaalkanes also exhibit similar dissociative characteristics and now we present the results of a kinetic study of the acid hydrolysis of (1,4,7-triazacyclononane)nickel(II), Ni[9]aneN_3^{2+}, and (1,5,9-triazacyclododecane)nickel(II), Ni[12]aneN_3^{2+}. Like TRI and *c*-tach these triazaalkanes coordinate only facially. It might be expected that Ni[12]aneN_3^{2+} would dissociate more rapidly since the trimethylene bridging groups would introduce a greater degree of flexibility and a more facile ligand unwrapping process. This is not the case however. We find the half-life for the dissociation of Ni[12]aneN_3^{2+} in 1 M acid at 30 °C is 54 h while the corresponding time for Ni[9]aneN_3^{2+} dissociation in 1 M acid at 25 °C is only 1.2 h. The difference may lie in strain energy.


Ni[12]aneN_3^{2+}


Ni[9]aneN_3^{2+}

Rate studies were made in the acid range 0.2–5.0 M (HCl) and activation parameters were obtained from the kinetic data by measuring reaction rates at temperatures ranging from 15 to 35 °C for Ni[9]aneN_3^{2+} and 30 to 60 °C for Ni[12]aneN_3^{2+}.

Experimental Section

Materials. The preparation of the ligands has previously been described.[5,6] The complexes were prepared by mixing solutions of nickel(II) nitrate and the appropriate amine hydrochloride in 1:1 molar ratio. Stoichiometric amounts of sodium hydroxide were then added to neutralize the amine hydrochloride and the resulting solutions were heated to 80 °C for 24 h. Solid $Ni(OH)_2$, which initially forms on addition of the sodium hydroxide, dissolves during the course of the heating and the characteristic blue color of the mono complexes develops. Additional heating produced no further change in the absorption spectrum of the solutions. Stock solutions of the complexes were prepared by appropriate dilution of these solutions.

Kinetic Measurements. Kinetic data for the dissociation of the complexes were obtained by following the decay of the visible absorption maximum corresponding to the nickel(II) complex (NiL^{2+}) with time. The initial concentration of the complexes ranged from 2×10^{-3} to 2×10^{-2} M and the concentration of hydrochloric acid ranged from 0.20 to 5.0 M. Measurements on solutions containing acid in the concentration range of 0.20–1.0 M were made at a constant ionic strength of 1.0 by using lithium chloride as the primary salt. For each experiment 5 mL of solution containing the complex and enough lithium chloride to bring the final ionic strength to 1.0 were mixed with 1 mL of standard hydrochloric acid. Volume additivity upon mixing was assumed. Several kinetic runs were made by using nitric and perchloric acids {$\mu = 1.0$ (NO_3^- or ClO_4^-)} in place of hydrochloric acid. No detectable change in rate was observed by using this modified procedure.

Experiments with Ni[9]aneN_3^{2+} were done at 15.0, 25.0, and 35.0 °C and the rate of dissociation of the complex was followed by observing the decay of the absorption maximum (575 nm, ϵ 4.3 cm^{-1} M^{-1}) with time. Kinetic studies with Ni[12]aneN_3^{2+} were done at 30.0, 40.0, 50.0, and 60.0 °C and the dissociation rate was measured by recording the decay of the absorption maximum of the complex (612 nm, ϵ 13.6 cm^{-1} M^{-1}) with time. Except for the absorption due to $Ni(H_2O)_6^{2+}$ (λ_{max} 770 nm, 649 nm (sh)) no other peaks were observed over the course of the reaction.

Table I. Pseudo-First-Order Rate Constants for the Dissociation of Ni[9]aneN_3^{2+a}

$10^2[NiL^{2+}]_0$, M	$[HCl]_0$, M	$10^4 k_{obsd}$, s^{-1}	temp, °C
0.883	1.0	0.379 (0.013)	15
1.06	0.8	0.370 (0.009)	15
1.06	0.6	0.286 (0.005)	15
1.06	0.4	0.262 (0.005)	15
2.10	1.0	1.62 (0.03)	25
0.883	0.8	1.31 (0.09)	25
1.17	0.6	1.17 (0.01)	25
1.17	0.4	0.885 (0.020)	25
0.883	0.2	0.532 (0.009)	25
0.883	1.0	6.56 (0.01)	35
0.883	0.8	4.97 (0.01)	35
0.883	0.6	4.05 (0.06)	35
0.883	0.4	3.03 (0.05)	35
1.06	0.2	1.94 (0.04)	35
0.426	5.0	103 (5)	25

[a] Ionic strength adjusted to 1.00 with LiCl except in cases where $[HCl]_0 = 1.0$ M.

Table II. Pseudo-First-Order Rate Constants for the Dissociation of Ni[12]aneN_3^{2+a}

$10^3[NiL^{2+}]$, M	$[HCl]_0$, M	$10^5 k_{obsd}$, s^{-1}	temp, °C
5.06	1.0	0.355 (0.17)	30
5.06	0.8	0.350 (0.15)	30
5.06	0.6	0.315 (0.03)	30
5.06	0.4	0.300 (0.06)	30
4.22	1.0	1.71 (0.06)	40
4.22	0.8	1.27 (0.14)	40
4.22	0.6	0.929 (0.01)	40
4.22	0.4	0.756 (0.01)	40
5.06	0.2	0.966 (0.01)	40
5.06	1.0	8.64 (0.13)	50
5.06	0.8	6.02 (0.09)	50
5.06	0.6	5.72 (0.02)	50
5.06	0.4	5.95 (0.08)	50
5.06	0.2	5.13 (0.04)	50
5.25	1.0	10.2 (0.01)	60
5.25	0.8	9.79 (0.12)	60
5.25	0.6	8.84 (0.15)	60
5.25	0.4	6.07 (0.05)	60
5.25	0.2	4.87 (0.07)	60
0.426	5.0	4.05 (0.10)	25

[a] Ionic strength adjusted to 1.00 with LiCl except in cases where $[HCl]_0 = 1.0$ M.

Observed rate constants were obtained by a weighted least-squares plot of ln [NiL^{2+}] vs. time. For the determination of complex concentration, the absorbance of $Ni(H_2O)_6^{2+}$ was taken into account and each component of the absorption maximum was assumed to be linearly dependent and obey Beer's law. Thus, the concentration of complex was calculated from eq 1, where $[Ni]_T = [NiL^{2+}] + [Ni^{2+}]$.

$$[NiL^{2+}] = \frac{A_\lambda - \epsilon_\lambda^{Ni^{2+}}[Ni]_T}{\epsilon^{NiL^{2+}} - \epsilon_\lambda^{Ni^{2+}}} \qquad (1)$$

Absorption spectra of solutions were recorded with a Beckman Acta III spectrophotometer. The sample cell holder was thermostated with a constant-temperature water circulator, and all samples were equilibrated at the appropriate temperature prior to measurement.

Results and Discussion

Experimental data for the hydrolysis of Ni[9]aneN_3^{2+} and Ni[12]aneN_3^{2+} in the acid concentration range of 0.20–5.0 M are given in Tables I and II. These data were found to agree with the rate expression given in eq 2, where $k_{obsd} = k$

$$-d[NiL^{2+}]/dt = k_{obsd}[NiL^{2+}] \qquad (2)$$

$+ k_H[H^+]$. There was no acid-independent region observed over the range of acidities studied and our experiments show it is unlikely that further increase in acid strength would bring about independence. Rate constants k and k_H as well as activation parameters were calculated from the data collected at constant ionic strength (0.20–1.0 M) and are presented in Table III.

Table III

Elementary Rate Constants

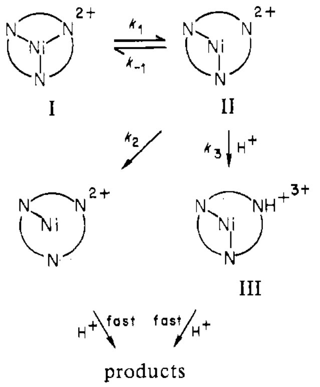
complex	temp, °C	10^5k, s^{-1}	10^5k_H, M^{-1} s^{-1}
Ni[9]aneN$_3^{2+}$	15	1.72 (0.36)	2.18 (0.43)
	25	3.25 (0.67)	13.0 (1.0)
	35	7.6 (2.1)	55.8 (3.2)
Ni[12]aneN$_3^{2+}$	30	0.260 (0.014)	0.100 (0.019)
	40	0.52 (0.24)	1.00 (0.37)
	50	4.17 (0.93)	3.5 (1.4)
	60	3.64 (0.76)	7.2 (1.1)

Activation Parameters[a]

	k		k_H	
complex	$\Delta H^\ddagger$	$\Delta S^\ddagger$	$\Delta H^\ddagger$	$\Delta S^\ddagger$
Ni[9]aneN$_3^{2+}$	12.5	−38	26.8	14
Ni[12]aneN$_3^{2+}$	18.5	−25	28.3	6.6

[a] Units of $\Delta H^\ddagger$ and $\Delta S^\ddagger$ are kcal/mol and entropy units, respectively. Standard deviations in $\Delta H^\ddagger$ and $\Delta S^\ddagger$ for Ni[9]aneN$_3^{2+}$ and Ni[12]aneN$_3^{2+}$ are approximately 10 and 20%, respectively.

Scheme I



There are some marked similarities between the kinetic behavior of these complexes and Ni(c-tach)$^{2+}$.[3] In the case of Ni(c-tach)$^{2+}$, Scheme I was assumed. Although rate expressions derived from this mechanism fit our data, it has recently been criticized by Margerum and co-workers[7] and several changes have been proposed. A mechanism for the dissociation of these complexes based upon those proposals is shown in Scheme II. The principal distinction between the two mechanisms is that there exists in Scheme II a predissociated species (not illustrated) with one donor displaced from the normal chelating position in which solvent has not yet been captured.[7] The solvent reaction of this species is proton promoted. Margerum and co-workers cite the low activation energy (19.3 kcal/mol) for the nonproton rate constant (k) obtained from the Ni(c-tach)$^{2+}$ study as evidence against mechanism 1. We have obtained corresponding activation energies of 13.1 and 19.1 kcal/mol for Ni[9]aneN$_3^{2+}$ and Ni[12]aneN$_3^{2+}$, respectively.

From mechanism 2 we find

$$-d[NiL^{2+}]/dt = \left\{\frac{k_1k_2k_3[H^+] + k_{-1}k_3k_4[H^+] + k_2k_3k_4[H^+]^2}{k_{-1}k_2 + k_{-1}k_3 + k_2k_3[H^+]}\right\}[NiL^{2+}] \quad (3)$$

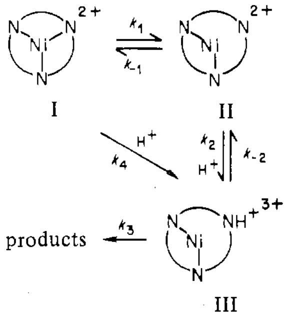
if it is assumed that at low pH

$$k_2k_3[H^+] >> k_{-1}k_2 + k_{-1}k_3$$

the expression for the rate constant becomes

$$k_{obsd} = \frac{k_1k_2 + k_{-1}k_4}{k_2} + k_4[H^+] = k + k_H[H^+] \quad (4)$$

Scheme II



The effect of cyclic ligand structure upon the dissociation rate of the metal complexes is quite dramatic. Although Ni[9]aneN$_3^{2+}$ is 5 orders of magnitude thermodynamically more stable than Ni[12]aneN$_3$[6] (log β_f at 25 °C for Ni[9]aneN$_3^{2+}$ and Ni[12]aneN$_3^{2+}$ are 16.2 and 10.9, respectively), the latter dissociates at a much slower rate. We believe that this rate difference is associated with strain energy effects. It has been shown that when [9]aneN$_3$ coordinates to nickel(II), a sizable trigonal distortion from octahedral geometry results.[8] Spectroscopic studies indicate that Ni[12]aneN$_3^{2+}$ is nearly perfectly octahedral.[6] It is likely therefore that Ni[9]aneN$_3$ is more strained than Ni[12]aneN$_3^{2+}$ and the rates of dissociation can be directly correlated to the strain energy of the complexes. This result is clearly in accord with conclusions drawn from the behavior of macrocyclic complexes.[9] A further demonstration that there exists considerable strain and therefore weaker Ni–N bonds in the Ni[9]aneN$_3^{2+}$ is provided by the smaller $\Delta H^\ddagger$ associated with its hydrolysis. This behavior appears path dependent and amounts to approximately 6 kcal/mol for the non-proton-dependent step.

The recent work of Leugger, Hertli, and Kaden[10] shows that the formation of macrocyclic complexes of the [x]aneN$_4$ (x = 12–16) type is nearly independent of ring size when the ligand is in the monoprotonated (LH$^+$) form. On the assumption that there are no complicating mechanistic steps which occur at higher pH (the pH range of this study is 0 to 1), our data suggest that the rate of formation of Ni[9]aneN$_3^{2+}$ at 25 °C is approximately 6 orders of magnitude greater than Ni[12]aneN$_3^{2+}$. This difference in formation rates may reflect conformational effects similar to those previously proposed to explain the unusual thermodynamic stability of metal–[9]aneN$_3$ complexes (i.e., configurational entropy).[6,11]

The values of $\Delta S^\ddagger$ also appear informative even though there is a great deal of statistical uncertainty in these numbers. If it is assumed that the transition state involves a Ni–N bond weakening (or breaking previous to solvation), then the differences in $\Delta S^\ddagger$ between Ni[9]aneN$_3^{2+}$ might be associated with ligand flexibility. The relatively compact Ni[9]aneN$_3^{2+}$ is less likely to show a significant increase in the degrees of vibrational freedom between its ground and transition states.

In conclusion, the hydrolyses of nickel(II) complexes of these triazaalkanes obey pseudo-first-order acid-dependent kinetics. Although there are several possible mechanisms which might explain this behavior, we feel the mechanism of acid hydrolysis of nickel(II)–amine complexes put forth by Margerum and co-workers serves as a good model for these systems. We further feel that the difference in rates of dissociation of these complexes can be directly correlated to strain energy effects.

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Registry No. Ni([9]aneN$_3$)$^{2+}$, 67163-09-1; Ni([12]aneN$_3$)$^{2+}$, 67163-08-0.

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Preparation and Reactions of Heptafluoroazazcyclopentan-2-one

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The pseudohalogen behavior of $NF_2(OSO_2F)^2$ is well demonstrated in reactions with fluorinated olefins, C_2F_4 and C_3F_6, to form (difluoroamino)(fluorosulfato)-substituted fluoroalkanes.[3] The latter are stable materials which, when reacted with alkali metal fluorides, are converted to α-difluoroamino ketones or α-(difluoroamino)acyl fluorides depending on the position of the fluorosulfate group on the chain. Sulfuryl fluoride is also produced.

We now wish to report that $NF_2(OSO_2F)$ also can be reacted with perfluorocyclobutene to form 1-(difluoroamino)-2-(fluorosulfato)hexafluorocyclobutane which undergoes a defluorosulfurylation reaction and concomitant ring expansion when reacted with potassium fluoride to give heptafluoroazacyclopentan-2-one. The ring is further expanded upon reaction with phosphine.

Results and Discussion

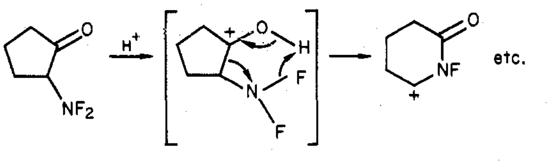
Addition of $NF_2(OSO_2F)$ to the double bond in perfluorocyclobutene requires a higher temperature and longer reaction time than does the analogous reaction with an acyclic perfluoro olefin.[4]

$$NF_2(OSO_2F) + \overline{CF_2CF{=}CFCF_2} \xrightarrow[\text{stainless steel, 22 h}]{160\ °C} NF_2\overline{CFCF_2CF_2C}F(OSO_2F)\ \ 80\%$$

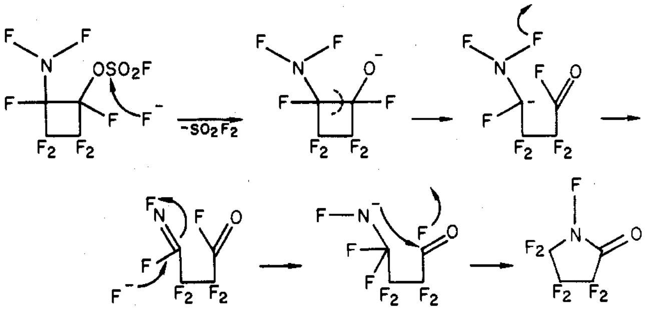
The compound formed is a stable, colorless material which slowly undergoes a defluorosulfurylation reaction at room temperature in the presence of thoroughly dried potassium fluoride to form heptafluoroazacyclopentan-2-one.

$$NF_2\overline{CFCF_2CF_2C}F(OSO_2F) + KF \xrightarrow[20\ h]{25\ °C} \overline{NFCF_2CF_2CF_2C}{=}O + SO_2F_2 \quad (1)$$

Acid-catalyzed ring-expansion reaction routes of a nonfluorinated (difluoroamino)cyclopentane and a (difluoroamino)cyclopentanone have been postulated,[5,6] viz.



In our case, where the reaction is base catalyzed, a likely route can be suggested. Expansion to a five-membered cyclic compound greatly eased the ring strain. This argument is based on the enhanced stability of intermediate A (see discussion concerning KF reaction with (fluorosulfato)heptafluorocyclobutane below). This new cyclic ketone is a volatile, colorless liquid which is stable at 170 °C for at least 30 h. (Trifluoromethyl)hexafluoroazacyclopentan-2-one is synthesized at 350 °C in 70% yield.[7] This great thermal stability is observed also for the unsubstituted hexafluorocyclobutanone, $\overline{CF_2CF_2CF_2C}{=}O$, which is recovered unchanged after being heated for 12 h at 300 °C.[8] The latter is one of the most reactive ketones ever reported. Its reactivity was attributed to a combination of ring strain and the electron-withdrawing influence of the fluorine atoms.[9]



It is interesting to note that when (fluorosulfato)heptafluorocyclobutane, $\overline{CF_2CF_2CF_2C}F(OSO_2F)$, is defluorosulfurylated in the presence of potassium fluoride, the products include hexafluorocyclobutanone and sulfuryl fluoride.[10] Although the reaction conditions are more rigorous (140 °C) than those used in reaction 1, ring opening does not occur. This lends support to the argument that ring opening is enhanced by the greater stability of intermediate A in the difluoroamino case. An analogous intermediate is not possible for the cyclobutanone.

As might be expected, we find that the behavior of heptafluoroazacyclopentan-2-one with PH_3 is quite different from that of hexafluorocyclobutanone where addition of the relatively acidic PH_3 to the polar form of the ketone occurs. PF_3 is found as one of the major products. In addition, a slightly volatile, oily liquid, which spectral and elemental analyses led us to conclude to be a ten-membered heterocycle (I), is formed

$$\overline{NFCF_2CF_2CF_2C}{=}O + PH_3 \xrightarrow[2\ days]{25°C} \underset{\text{I}}{\overline{HNCF_2CF_2CF_2C(O)NHCF_2CF_2CF_2C}(O)} + PF_3 + \text{orange solid}$$

in good yield. The liquid is stable to water. Elemental analyses are in excellent agreement with a substance whose empirical formula is C_4F_6HNO, that is, the five-membered monomer of I. However, mass spectral data strongly support the proposed structure—the highest *m/e* is 347 $(M - 2F - H)^+$ and the base peak is 346 $(M - 2F - 2H)^+$. The fragmentation pattern is given in the Experimental Section. The gas-phase infrared spectrum contains bands at 3480 and 1430 cm^{-1} assigned to ν_{NH} and δ_{NH}. A very strong band at 1830 cm^{-1} with 1805 (sh) cm^{-1} is assigned to $\nu_{C=O}$. This is a slight downward shift from the parent ketone. Strong bands are also found in the C–F stretching region.

In $CDCl_3$, the proton NMR spectrum consists of a single broad peak at δ 8.85 and is assigned to N–H. The ^{19}F NMR spectrum has three resonances at ϕ −92.5 (s), −127.6 (tr), and −134.1 (heptet) in the ratio of 1:1:1. The singlet is assigned

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