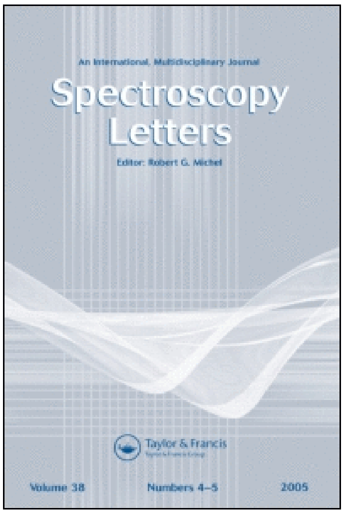

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Fragmentation of Some Hydrazino- and Hydroxylaminohalophosphines Under Electron Impact

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FRAGMENTATION OF SOME HYDRAZINO- AND HYDROXYLAMINOHALOPHOSPHINES UNDER ELECTRON IMPACT

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During the past several years compounds containing P-N bonds have been the object of a number of investigations,[2] many of them motivated by the conjecture that pπ-dπ bonds can form between these two atoms. Recently the behavior of the halophosphonitriles,[3-6] whose bonding is thought to involve extensive pπ-dπ delocalization,[7] and the aminohalophosphines,[8] which contain only a single P-N fragment, toward electron bombardment has been described. Since the framework over which π bonding can occur in the hydrazino- and hydroxylaminohalophosphines[9] is intermediate between these other two classes of compounds we would like to report comparable studies for these latter compounds.

EXPERIMENTAL

All compounds were prepared and purified following procedures appearing in the literature.[9] Their purity was checked by comparison of NMR, and IR spectra, and vapor pressures to reported data.

Mass spectra were run on liquid samples injected into a Hitachi Perkin-Elmer RMU-6D spectrometer at 20 ev and 70 ev ionizing voltages. The source and inlet were not heated to prevent decomposition of the samples. No significant differences were noted between the 20 ev and 70 ev spectra.

RESULTS AND DISCUSSION

The main fragments in the mass spectra of $F_2PN(CH_3)OCH_3$, $F_2PN(CH_3)N(CH_3)_2$, $FP[N(CH_3)OCH_3]_2$, $FP[N(CH_3)N(CH_3)_2]_2$, $Cl_2PN(CH_3)OCH_3$, $Cl_2PN(CH_3)N(CH_3)_2$, $ClP[N(CH_3)OCH_3]_2$, and $ClP[N(CH_3)N(CH_3)_2]_2$ are summarized in Table 1; the complete spectra are listed in Table 2, and the metastable ions observed in these spectra are reported in Table 3. Parent ions were observed for all compounds except $Cl_2PN(CH_3)N(CH_3)_2$ in whose spectrum (parent-Cl) was the highest mass ion detected. As indicated by the fragmentation patterns and supported by metastable ions these molecules fragment, under electron bombardment, through the cleavage of phosphorus-nitrogen, or phosphorus-halogen bonds. As might be expected, since P-Cl bonds are typically weaker than analogous P-F bonds,[10] ions arising from cleavage of the phosphorus-halogen bonds carry a larger percentage of the total ion current in the respective chlorophosphines. Only in the cases of $F_2PN(CH_3)OCH_3$, $F_2PN(CH_3)N(CH_3)_2$, $FP[N(CH_3)OCH_3]_2$and $Cl_2PN(CH_3)N(CH_3)_2$ was fragmentation of the parent ion by C-N, N-N, N-O, or C-H cleavage observed. In all of these cases, however, the amount of such cracking appears to be small with the resultant fragments carrying only about 1% of the ion current.

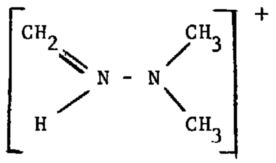
Fragmentation of the trimethylhydrazino derivatives appears to procede largely through P-N bond rupture and the formation of the trimethylhydrazonium ion, $N_2C_3H_9^+$. In fact, with each of these compounds the $N_2C_3H_9^+$ peak is the base peak and between 60%-70% of the total ion current is carried by that ion, $N_2C_3H_8^+$, $N_2C_3H_7^+$, and ions which metastable transitions indicated to have been formed from $N_2C_3H_9^+$. The remaining peaks were uniformly of rather low relative intensity with virtually none of them individually carrying over 3% of the ion current.

TABLE I

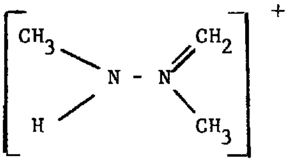
Percent Ion Current ($\%\Sigma_{25}$) of Principal Ions
($R = N_3C_3H_9$ or NOC_2H_6, $X = Cl$ or F)

	Cl_2PR		$ClPR_2$		F_2PR		FPR_2	
Ion	$N_2C_3H_9$	NOC_2H_6	$N_2C_3H_9$	NOC_2H_6	$N_2C_3H_9$	NOC_2H_6	$N_2C_3H_9$	NOC_2H_6
I. Parent	0.0	5.8	1.5	2.4	1.6	14.5	3.7	3.7
II. R	31.6	22.8	37.7	12.3	23.3	4.9	33.8	3.9
III. PX_2	3.1	10.5	---	---	5.8	19.4	---	---
IV. XPR	7.1	5.2	2.0	16.2	1.2	0.2	2.7	11.9
V. (IV-29)	2.9	11.2	1.5	18.6	0.7	6.2	1.1	22.9
VI. (V-X-15)	5.0	3.2	4.5	2.7	0.7	1.4	0.3	3.0

The stability of $N_2C_3H_9^+$ and related fragments is not surprising. In the spectra[11] of $(CH_3)_4N_2$ and other compounds containing the $N_2(CH_3)_3$ moeity the $N_2(CH_3)_3^+$ and associated ions are observed in large abundances probably reflecting the stability of species such as



and



which are formally similar to the relatively stable immonium ions seen in mass spectra of many amines.[12]

With the hydroxylamino derivatives, on the other hand, quite a different picture emerges. Only in the mass spectrum of $Cl_2PN(CH_3)OCH_3$ is the hydroxylimmonium ion, $NOC_2H_6^+$, the base peak, and even with this compound the fraction of the ion current carried by $NOC_2H_6^+$ is only about 23%. Such behavior is not unexpected in view of the greater alkyl substitution in $N_2(CH_3)_3$ and the well known ability of nitrogen to stabilize

TABLE II

Mass Spectra of Hydrazino- and Hydroxylaminohalophosphines
(Each entry consists of respectively, the mass/charge ratio for each ion, $\%\Sigma_{25}$ for that ion, and, for the more important ions, the tentative assignment)

$Cl_2PN(CH_3)N(CH_3)_2$

27, 0.6; 28, 3.8; 30, 2.5; 31, 0.6; 32, 1.2; 33, 0.6; 35, 0.3; 36, 0.6; 40, 0.3; 41, 0.6; 42, 7.5, NC_2H_4; 43, 8.5, NC_2H_5; 44, 10.4, NC_2H_6; 45, 0.6; 46, 2.5, NC_2H_8; 57, 0.6; 58, 0.9; 59, 0.6; 60, 5.0, $PNCH_3$; 66, 1.6; 67, 0.6; 68, 0.6; 71, 0.9; 72, 0.3; $\underline{73}$, $\underline{31.4}$, $N_2C_3H_9$; 74, 1.2; 94, 0.3; 96, 1.6, $^{35}ClPNCH_4$; 98, 0.5, $^{37}ClPNCH_4$; 101, 1.9, $^{35}Cl_2P$; 103, 1.2, $^{35}Cl^{37}ClP$; 110, 2.2, $^{35}ClPN_2CH_5$; 112, 0.7, $^{37}ClPN_2CH_5$; 139, 5.3, $^{35}ClPN_2C_3H_9$; 141, 1.8, $^{37}ClPN_2C_3H_9$.

$ClP[N(CH_3)N(CH_3)_2]_2$

27, 0.8; 28, 3.8; 29, 0.8; 30, 5.3; 31, 0.4; 32, 1.9; 41, 0.8; 42, 7.5, NC_2H_4; 43, 6.8, NC_2H_5; 44, 7.5, NC_2H_6; 45, 0.8; 46, 3.0, NC_2H_8; 57, 1.1; 58, 0.8; 59, 3.0; 60, 4.5, $PNCH_3$; 71, 0.8; 72, 0.4; $\underline{73}$, $\underline{37.7}$, $N_2C_3H_9$; 74, 5.3; 96, 0.8; 110, 1.1, $^{35}ClPN_2CH_5$; 112, 0.4, $^{37}ClPN_2CH_5$; 134, 0.4, $PN_3C_5H_{15}$; 139, 1.5, $^{35}ClPN_2C_3H_9$; 141, 0.5, $^{37}ClPN_2C_3H_9$; 177, 1.1, $PN_4C_6H_{18}$; 212, 1.1, $^{35}ClPN_4C_6H_{18}$; 214, 0.4, $^{37}ClPN_4C_6H_{18}$.

$F_2PN(CH_3)N(CH_3)_2$

27, 0.9; 28, 5.1; 29, 0.5; 30, 4.4; 31, 0.5; 32, 1.9; 36, 0.2; 40, 0.5; 41, 0.7; 42, 11.4, NC_2H_4; 43, 15.6, NC_2H_5; 44, 8.2, NC_2H_6; 45, 0.7; 46, 3.5, NC_2H_8; 50, 0.7, FP; 51, 0.5; 57, 1.4; 58, 2.1; 59, 1.9; 60, 0.5, $PNCH_3$; 69, 5.8, F_2P; 71, 0.9; 72, 0.7; $\underline{73}$, $\underline{23.3}$, $N_2C_3H_9$; 74, 2.1; 78, 0.5, 80, 0.5, $FPNCH_4$; 94, 0.7, FPN_2CH_4; 96, 0.5; 98, 0.2; 112, 0.9, $F_2PN_2CH_3$; 123, 1.2, $FPN_2C_3H_9$; 142, 1.6, $F_2PN_2C_3H_9$.

$FP[N(CH_3)N(CH_3)_2]_2$

27, 0.7; 28, 4.4; 29, 0.7; 30, 4.4; 31, 0.7; 32, 1.7; 40, 0.3; 41, 0.7; 42, 8.1, NC_2H_4; 43, 7.1, NC_2H_5;44, 8.8, NC_2H_6; 45, 0.7; 46, 2.4, NC_2H_8; 50, 0.3, FP; 51, 0.3; 57, 1.0; 58, 0.7; 59, 5.1; 60, 1.4, $PNCH_3$; 71, 1.0; 72, 0.7; $\underline{73}$, $\underline{33.8}$, $N_2C_3H_9$; 74, 4.7; 78, 0.7; 80, 1.0, $FPNCH_4$; 94, 1.7, FPN_2CH_4; 123, 2.7, $FPN_2C_3H_9$; 148, 0.3, $PN_3C_4H_{12}$;196, 3.7, $FPN_4C_6H_{18}$; 197, 0.3.

$Cl_2PN(CH_3)OCH_3$

27, 0.5; 28, 5.0; 29, 1.8; 30, 1.4; 31, 1.6; 32, 0.9; 33, 3.7; 35, 0.5; 36, 0.9; 37, 0.2; 38, 0.3; 42, 0.7; 43, 0.2; 44, 0.7; 45, 1.4, $NOCH_3$; 46, 0.5; 47, 3.2, PO; 49, 0.2; 57, 0.2; 58, 0.7; 59, 1.4; $\underline{60}$, $\underline{22.8}$, NOC_2H_6; 61, 0.7; 62, 0.2; 63, 0.5, 66, 2.3, ^{35}ClP; 67, 1.4; 68, 0.7, ^{37}ClP; 69, 0.5; 94, 0.9; 95, 0.2; 96, 0.7; 97, 8.5; $^{35}ClPOCH_3$; 98,0.2;99,2.7,$^{35}ClPOCH_3$;101, 5.9, $^{35}Cl_2P$, 103, 3.9, $^{35}Cl^{37}ClP$;105;0.7, $^{37}Cl_2P$;110, 0.7; 112, 0.2; 117, 0.4; 119, 0.2; 126, 3.9, $^{35}ClPNOC_2H_6$; 128, 1.3, $^{37}ClPNOC_2H_6$, 130, 0.5; 131, 0.2;

(cont.)

132, 0.5; 146, 0.7, $^{35}Cl_2PNOCH_3$; 148, 0.5, $^{35}Cl^{37}ClPNCCH_3$; 160, 0.7; 161, 5.9, $^{35}Cl_2PNOC_2H_6$; 162, 0.5; 163, 4.1, $^{35}Cl^{37}ClPNOC_2H_6$; 164, 0.2; 165, 0.7, $^{37}Cl_2PNOC_2H_6$.

$ClP[N(CH_3)OCH_3]_2$

27, 0.6; 28, 8.8; 29, 1.8; 30, 1.7; 31, 1.1; 32, 2.2; 33, 2.0; 35, 0.1; 36, 1.3; 38, 0.4; 40, 0.1; 41, 0.1; 42, 0.6; 43, 0.7; 44, 1.8, $NCCH_2$; 45, 1.8, $NOCH_3$; 46, 5.2, $NOCH_4$; 47, 2.7, PO; 49, 0.1; 57, 0.1; 58, 0.4; 59, 1.0; 60, 12.3, NOC_2H_6; 61, 4.1; 62, 0.1; 63, 0.8; 66, 0.4; 67, 0.7; 68, 0.1; 69, 0.3; 73, 0.1; 76, 0.1; 78, 0.1; 79, 0.1; 81, 0.1; 83, 0.1 85, 0.1; 90, 0.1; 92, 0.4; 93, 1.3, $PNOCH_4$; 94, 0.8; 95, 0.4; 96, 0.4; $\underline{97}$, $\underline{14.0}$, $^{35}ClPCCH_3$; 98, 0.3; $\underline{99}$, $\underline{4.6}$, $^{37}ClPCCH_3$; 101, 0.3; 103, 0.1; 105, 0.1; 106, 0.1; 108, 0.3; 110, 0.1; 112, 0.1; 122, 1.1; 126, 11.8, $^{35}ClPNOC_2H_6$; 127, 0.4; 128, 4.4, $^{37}ClPNOC_2H_6$; 129, 0.1; 149, 0.1; 151, 0.4; $PN_2O_2C_4H_{12}$; 161, 0.3; 162, 0.1; 163, 0.1; 186, 1.8, $^{35}ClPN_2O_2C_4H_{12}$; 187, 0.1; 188, 0.6, $^{37}ClPN_2O_2C_4H_{12}$.

$F_2PN(CH_3)OCH_3$

26, 0.2; 27, 1.2; 28, 11.7; 29, 9.7; 30, 1.7; 31, 3.1; 32, 0.8; 33, 1.6 36, 0.6; 38, 0.2; 41, 0.2; 42, 0.6; 43, 0.4; 44, 0.4; 45, 2.1, $NOCH_3$; 46, 1.2, $NCCH_4$; 47, 1.4, PO; 50, 1.4, PF; 51, 0.8; 58, 0.2; 59, 0.8; 60, 4.9, NOC_2H_6; 61, 1.0; 62, 0.2; 67, 1.9; $\underline{69}$, $\underline{19.4}$, PF_2; 71, 0.4; 74, 0.6; 77, 0.2; 78, 0.8; 79, 0.2; 80, 0.2; 81, 6.2, $FPCCH_3$; 84, 0.2; 85, 0.4; 86, 0.6; 87, 0.2; 88, 0.4; 94, 0.4; 96, 1.5, $FPNCCH_4$; 97, 0.8; 98, 0.4; 99, 0.8; 100, 0.4; 101, 0.2; 110, 0.2, $FPNOC_2H_6$; 112, 0.2; 113, 0.6; 114, 2.3, F_2PNOCH_3; 128, 0.6; 129, 14.5, $F_2PNOC_2H_6$; 130, 0.4.

$FP[N(CH_3)OCH_3]_2$

27, 0.7; 28, 8.5; 29, 2.5; 30, 2.3; 31, 1.8; 32, 0.2; 33, 2.5; 42, 0.9; 43, 0.9; 44, 1.4; 45, 2.7, $NOCH_3$; 46, 6.7, $NCCH_4$; 47, 3.0, PO, 50, 0.9, FP; 51, 1.6; 59, 0.5; 60, 3.9, NOC_2H_6; 61, 5.0; 62, 0.2; 63, 0.2; 65, 0.7; 66, 0.2; 67, 0.7; 69, 0.5; 73, 0.5; 74, 0.2; 77, 0.5; 78, 3.4, FPN_2; 79, 0.7; 80, 0.7; $\underline{81}$, $\underline{22.9}$, $FPCCH_3$; 82, 0.5; 83, 0.7; 85, 0.2; 93, 0.5; 94, 0.2; 95, 0.9; 96, 0.2; 97, 0.4; 99, 0.2; 101, 0.9; 110, 11.9, $FPNOC_2H_6$; 126, 0.7; 128, 0.2; 155, 0.5, $FPN_2O_2C_3H_9$; 170, 3.7, $FPN_2O_2C_4H_{12}$; 171, 0.2.

TABLE III

Metastable Ions Observed in the Mass Spectra of the Hydrazino, Hydroxylamino-, and Aminohalophosphines

$Cl_2PN(CH_3)OCH_3$

m/e	Assignment
11.2	97→ 33
13.1	60→ 28
18.2	60→ 33
22.4	161→ 60
22.8	97→ 47
28.6	126→ 60
73.1	126→ 96
74.7	126→ 97
76.5	128→ 99
159.0	161→ 160

$F_2PN(CH_3)OCH_3$

m/e	Assignment
13.9	69→ 31
18.2	60→ 33
27.2	81→ 47
27.9	129→ 60
33.1	61→ 45
34.7	61→ 46
51.0	129→ 81
59.6	110→ 81
65.0	101→ 81
79.1	129→ 101
100.8	129→ 114
127.0	129→ 128

$ClP[N(CH_3)OCH_3]_2$

m/e	Assignment
13.1	60→ 28
18.1	60→ 33
22.8	97→ 47
23.4	186→ 66
33.1	61→ 45
34.7	61→ 46
74.7	126→ 97
76.6	128→ 99
85.2	186→ 126
87.2	188→ 128

$FP[N(CH_3)OCH_3]_2$

m/e	Assignment
18.2	60→ 33
27.2	81→ 47
29.4	122→ 60
33.1	61→ 45
34.8	110→ 61
59.6	110→ 81
71.2	170→ 110

$Cl_2PN(CH_3)N(CH_3)_2$

m/e	Assignment
14.0	73→ 32
24.3	73→ 42
26.5	73→ 44
29.0	73→ 46
32.8	110→ 60
37.5	96→ 60
38.4	139→ 73
66.4	139→ 96
87.0	139→ 110

$F_2PN(CH_3)N(CH_3)_2$

m/e	Assignment
12.4	73→ 30
14.0	73→ 32
15.8	124→ 44
19.6	43→ 29
24.2	73→ 42
26.6	73→ 44
29.0	73→ 46
37.7	142→ 73
52.6	123→ 80
71.9	128→ 94

$ClP[N(CH_3)N(CH_3)_2]_2$

m/e	Assignment
14.0	73→ 32
24.3	73→ 42
25.1	212→ 73
26.5	73→ 44
29.0	73→ 46
30.0	177→ 73

$FP[N(CH_3)N(CH_3)_2]_2$

m/e	Assignment
12.4	73→ 30
14.0	73→ 32
15.8	123→ 44
24.2	73→ 42
26.6	73→ 44
27.2	196→ 73
29.0	73→ 46

$Cl_2PN(CH_3)_2$

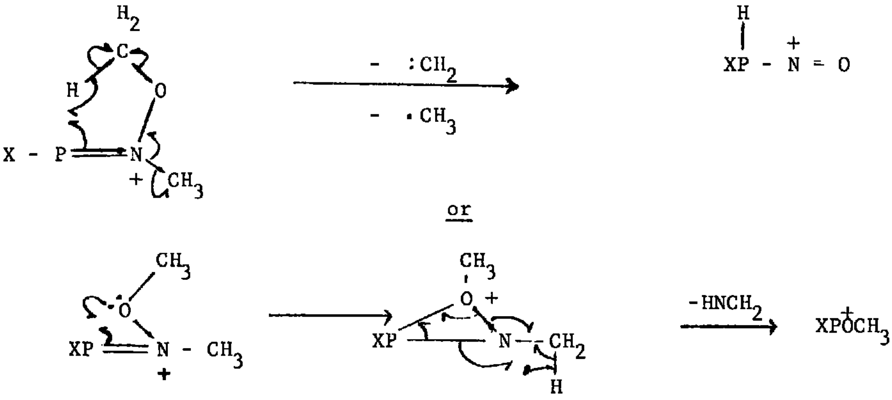
m/e	Assignment
12.2	145→ 42
16.1	110→ 42
17.6	110→ 44
59.7	110→ 81
61.4	112→ 83
82.2	145→ 110
83.5	147→ 112
85.2	149→ 112

$F_2PN(CH_3)_2$

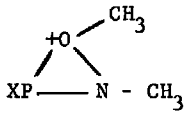
m/e	Assignment
15.7	112→ 42
111	113→ 112

positive ions more effectively than oxygen.[12] In all of the hydroxylamino derivatives, ions corresponding to $X_{n-1}P[N(CH_3)OCH_3]^+_{3-n}$, $X_nP[N(CH_3)OCH_3]^+_{2-n}$, PX^+_n, and fragments arising from them by simple bond cleavage account for a large portion of the total ion current. In addition to these ions, however, large peaks, (in the case of $FP[N(CH_3)OCH_3]_2$ and $ClP[N(CH_3)OCH_3]_2$ the base peaks) are observed for $[XPN(CH_3)OCH_3\text{-}29]^+$ in the spectra of the <u>mono</u> and <u>bis</u> hydroxylaminophosphines, and $[P[N(CH_3)OCH_3]_2\text{-}29]^+$ in the case of the <u>bis</u> derivatives. Metastable ions confirm that these ions result from the cracking of $XPN(CH_3)OCH_3^+$ and $P[N(CH_3)OCH_3]_2^+$ respectively.

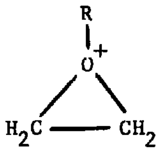
Metastable ions also indicate that the (fragment -29) ions fragment further to ultimately form PO^+. Several possible rearrangements can be written including (using $XPN(CH_3)OCH_3^+$ as an example)



The former process is unlikely since it involves an even electron ion losing a radical to give a radical ion. The abundance of these ions imply their stability and may be more consistent with the second mode described above in which an even electron ion is formed by loss of a stable molecule. Such a mechanism finds some precedent in transannular cleavages previously detected in epoxides.[12] If this mechanism is correct it implies that the structure of the $XPN(CH_3)OCH_3$ ion itself may be

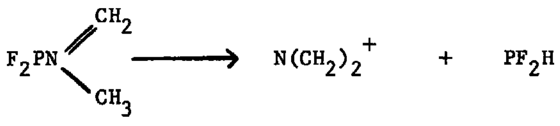


which is formally similar to



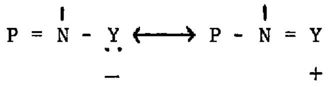
which has been reported to form during the cracking of some ethers.[13]

During the course of this investigation the spectra of $F_2PN(CH_3)_2$ and $Cl_2PN(CH_3)_2$ were also obtained. These agree well with those obtained by Borer and Cohn.[8] We were able to observe several new metastable ions in the spectra of both of these compounds (Table I) which indicate that $Cl_2PN(CH_3)_2$ fragments by a path similar to that of $Cl_2PN(CH_3)N(CH_3)_2$ and $Cl_2PN(CH_3)OCH_3$. That is by initial P-N or P-Cl bond cleavage. $F_2PN(CH_3)_2$, on the other hand, is unique in this series of compounds. As reported by Borer and Cohn[8] the ion $F_2PN(CH_3)_2$ fragments to form an intense ion at M-1. We have observed a metastable ion for the process:



m/e = 112 m/e = 42

implying that a large proportion of the fragmentation of the molecule proceeds through the M-1 ion. (Although M-1 peaks were observed in several of the other compounds studied here they were not intense and no evidence was obtained to indicate that a large amount of the fragmentation proceeded through them.) The formation of a M-1 peak is characteristic in the mass spectra of olefins, representing the formation of a stable allyl ion.[12] The P-N bond in these compounds has often been postulated to contain $d\pi$-$p\pi$ double bond character. Qualitatively if such bonding occurs, it should be maximized when the electronegativity of the substituents bound to the phosphorus is greatest, the electronegativity of the groups bonded to the nitrogen is minimized, and electron donor groups which could participate in resonance forms such as



$$P = \overset{|}{N} - \underset{-}{\underset{\cdot\cdot}{Y}} \longleftrightarrow P - \overset{|}{N} = \underset{+}{Y}$$

are absent. In the compounds studied here $F_2PN(CH_3)_2$ should have the greatest degree of P-N πbond character which might explain its unique fragmentation.

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