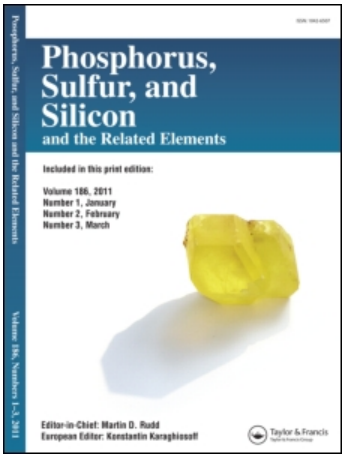

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

SILYL-NITROGEN COMPOUNDS,[1] IV: REACTIONS OF PENTASILA-PHOSPHA-PENTAZENE WITH ALCOHOLS AND CARBOXYLIC ACIDS

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To cite this Article Vasisht, S. K. and Bandhu, Kavita(1997) 'SILYL-NITROGEN COMPOUNDS,[1] IV: REACTIONS OF PENTASILA-PHOSPHA-PENTAZENE WITH ALCOHOLS AND CARBOXYLIC ACIDS', Phosphorus, Sulfur, and Silicon and the Related Elements, 126: 1, 101 — 110
To link to this Article: DOI: 10.1080/10426509708043549
URL: http://dx.doi.org/10.1080/10426509708043549

Phosphorus Sulfur and Silicon, 1997, Vol. 126, pp. 101-110
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SILYL-NITROGEN COMPOUNDS,[1] IV: REACTIONS OF PENTASILA-PHOSPHA-PENTAZENE WITH ALCOHOLS AND CARBOXYLIC ACIDS

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(In final form 24 April, 1997)

Pentakis(trimethylsilyl)–3-phospha–2-pentazene $(Me_3Si)_2N\text{-}N(SiMe_3)\text{-}P{=}N\text{-}N(SiMe_3)_2$ **(1)** reacts with Me_3COH to form oxidative addition products, the partially desilylated [bis(trimethylsilyl) hydrazino][bis(trimethylsilyl)hydrazono](t-butoxy)phosphorane **(2)** and the completely desilylated hydrazino-hydrazono(t-butoxy) phosphorane **(3)**. With CF_3COOH, **(1)** forms [tris(trimethylsilyl) hydrazino] [bis(trimethylsilyl)hydrazono](trifluoroacetato)phosphorane **(4)**. **(1)** and CF_3COOH in the ratio 1:5 produce a partially desilylated oxidative addition product trimethylsilylhydrazino-trimethylsilylhydrazono(trifluoroacetato)phosphorane **(5)** and a desilylated dimer $[H_2N\text{-}\overset{|}{N}\text{-}\overset{|}{P}(OOCCF_3) = N\text{-}NH_2]_2$, **(6)**. With fluorosulfonic acid, **(1)** forms tetrakis(trimethylsilyl) tetrazane $(Me_3Si)_2N\text{-}NH\text{-}NH\text{-}N(SiMe_3)_2$ **(7)** and a desilylated phosphorane $H_2N\text{-}NH\text{-}P(H)(SO_3F){=}N\text{-}NH_2$ **(8)**. Trifluoromethane sulfonic acid forms a desilylated phosphorane $H_2N\text{-}NH\text{-}P(H)(SO_3CF_3) = N\text{-}NH_2$ **(9)**.

INTRODUCTION

Silylated phospha-triazenes,[2,3] -tetrazenes[4] and -pentazenes[1] are well known. Some chemistry of the silylated phosphatriazene has been reported in literature,[5–8] whereas, that of the latter molecules is being investigated in our laboratories. In continuation of our studies[1] of pentasilaphosphapentazene $(Me_3Si)_2N\text{-}N(SiMe_3)\text{-}P{=}N\text{-}N(SiMe_3)_2$ **(1)**, we report here its reactivity towards protonating agents.

RESULTS AND DISCUSSION

Both acyclic as well as cyclic tetrasila-tetrazenes are known to undergo partial or complete desilylation with alcohols and carboxylic acids.[9] Protolysis of the

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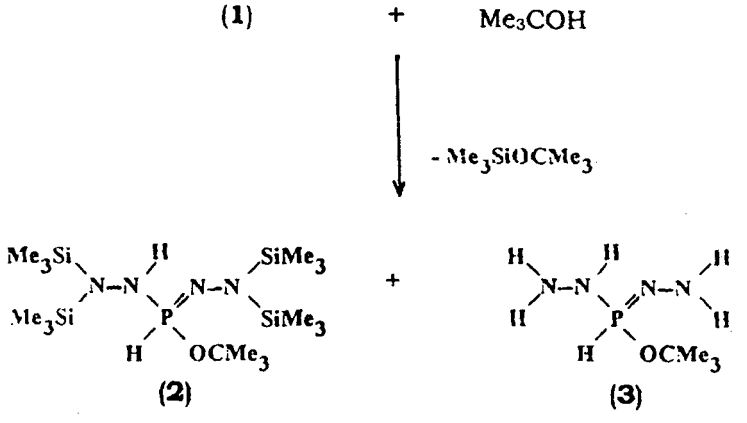
former[10] with CF_3COOH leads to the formation of tetrazene $H_2N\text{-}N{=}N\text{-}NH_2$ (eqn 1) whereas, that of the cyclic cis derivative gives ammonium azide.

$$(Me_3Si)_2N-N=N-N(SiMe_3)_2 \xrightarrow[-4Me_3SiOOCF_3]{+4CF_3COOH} H_2N-N=N-NH_2 \quad \ldots . \quad (1)$$

In both the cases reactivity at –N=N– is not observed. However, electrophillic silylated phosphatriazene[12] and phosphatetrazene[4] have been reported to combine with methanol to form oxidative addition as well as desilylation products. Pentasila-3-phospha-2-pentazene (**1**), on the other hand, reacts with methanol in different proportions with addition at P=N besides forming the partially and completely desilylated products.[1]

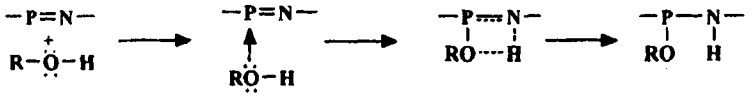
Reaction of (**1**) with t-butanol is very slow at room temperature when both the reactants are taken in equivalent amounts. Reaction of (**1**) and excess Me_3COH yields a partially desilylated oxidative addition product (**2**) and a completely desilylated phosphorane (**3**).



The ^{1}H-NMR spectrum of (**2**) in benzene shows a resonance at δ 0.27 attributed to the four equivalent Me_3Si groups. The butyl group gives a singlet at δ 1.42. The signal at 10.90 is a doublet due to P-H group with an expected large coupling constant $^1J_{(H\text{-}P)} = 612$ Hz.[4,12] The ^{31}P-NMR shows a signal at 25.34 ppm which is characteristic of 4-coordinated P(V).[4,6,12] The infrared spectrum of compound (**2**) shows important bands (cm^{-1}) at 3300 ν(N-H), 2360 ν(P-H) and 1255 ν(P=N) supporting the presence of the three functional groups.

Methanol is already reported to react with phosphatetrazenes to form both phosphorane[4] as well as phosphine[2] derivatives. Reaction of phosphapentazene (**1**) with methanol yields phosphine derivatives,[1] whereas, that of (**1**) with t-butanol leads to the formation of phosphorane. Thus, it is difficult to infer whether an initial 1,1-product or 1,2-product is formed in the reaction of phosphazene and

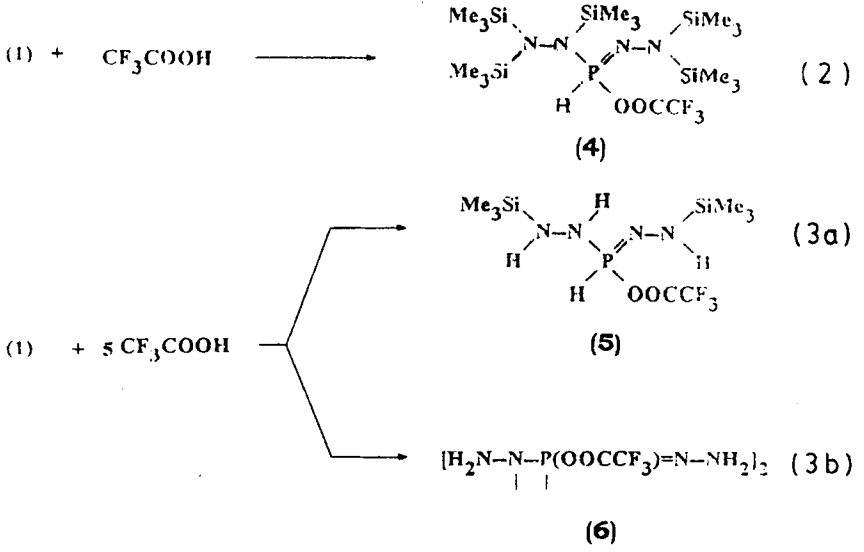
alcohol. However, it is expected that such a reaction may involve an insertion of phosphazene into the polar O-H linkage[15] to form an initial phosphine as follows:



The phosphine, thus formed, may then undergo tautomeric rearrangement to form a phosphorane. The initial addition at –P=N– is also supported by the formation of an intermediate amide in the low temperature reaction of RLi (R = CH_3 or C_4H_9) with tetrasilaphosphatetrazene.[13] The amide reacts with Me_3ECl (E = Si or Sn) to give phosphines some of which then undergo intramolecular rearrangement to form phosphoranes.[13] Such a low temperature insertion of –P=N– into RLi in **(1)** could not be established exclusively because the intermediate amides could not be isolated due to their instability.[14]

The phosphorane **(3)** formed in addition to **(2)** is a white solid insoluble in n-hexane, Et_2O, benzene, CH_2Cl_2 and $CHCl_3$ etc. but soluble in water and D_2O. The ^{31}P-NMR spectrum shows a signal at 25.95 ppm in D_2O. This is the range typical of P(V)[4,6,12] compounds. The infrared spectrum of the pure sample shows important characteristic bands (cm^{-1}) at 3300, 3150 ν(N-H), 2360 ν(P-H) and 1260 ν(P=N) which support structure **(3)** for the compound. Mass spectral analysis of **(3)** shows molecular ion peak at m/Z 166.

Reaction of **(1)** with trifluoroacetic acid proceeds similar to that with t-butanol and leads to an oxidative addition product **(4)** (eqn 2). Besides, desilylation occurs to form a phosphorane **(5)** and a diazadiphosphetidine **(6)**, (eqn 3a, 3b).

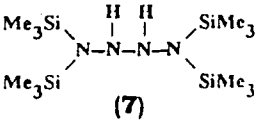


The ^{1}H-NMR spectrum of the oxidative addition product (**4**) in benzene shows resonances at δ 0.36, 0.14, 0.30d $^4J_{H\text{-}P}$ = 0.6 Hz.[4] A doublet at δ 10.93 with $^1J_{H\text{-}p}$ = 669 Hz is attributed to P-H group[4,12] in (**4**). The ^{19}F-NMR spectrum in n-hexane or Et_2O shows a resonance signal at 70.6 ppm due to the CF_3COO group. The ^{31}P-NMR spectrum gives a signal at δ 10.32 characteristic of four coordinated P(V) derivatives.[4,12] The infrared spectrum shows important bands at 2410 ν(P-H), 1700 ν(C=O), 1255 ν(P=N), 1210 ν(C-F), 1050 ν(C-O) and 920 ν(C-F).

The partially desilylated phosphorane (**5**) is a colourless liquid distilling at 70°C/10^{-3} tor. Its spectral data (Experimental) is characteristic of phosphorane structure similar to (**4**).

Phosphazenes –P=N– with less bulky substituents on double bonded nitrogen have greater tendency to dimerise into diazadiphosphetidines.[2] This has been observed in some desilylation reactions of tetrasilaphosphatetrazene.[13] Besides (**4**) and (**5**), reaction of (**1**) with CF_3COOH leads to desilylation to form diazadiphosphetidine (**6**). It is a white solid insoluble in n-hexane, ether, CH_2Cl_2, $CHCl_3$, benzene and THF but is soluble in water, D_2O and methanol. The ^{19}F NMR spectrum of (**6**) shows resonances at δ 74.99 and 75.66 in D_2O corresponding to two nonequivalent CF_3 groups.[13] The ^{31}P-NMR spectrum shows two peaks at δ 0.73 and 4.66 characteristic of P(V) derivatives[4,12]. The spectra showing nonequivalent phosphorus atoms as well as CF_3 groups in (**6**) indicate the possibility of an isomeric mixture.

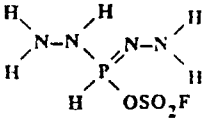
Phosphapentazene (**1**) reacts with fluorosulfonic acid in the ratio 1:5 to yield a tetrazene (**7**)and a phosphorane (**8**). Compound (**7**) distils out as a colourless liquid at 60°C/10^{-3} torr, whereas, compound (**8**) is obtained as a white solid insoluble in almost all organic solvents but soluble in water and D_2O.



(4a)

(**7**)

+

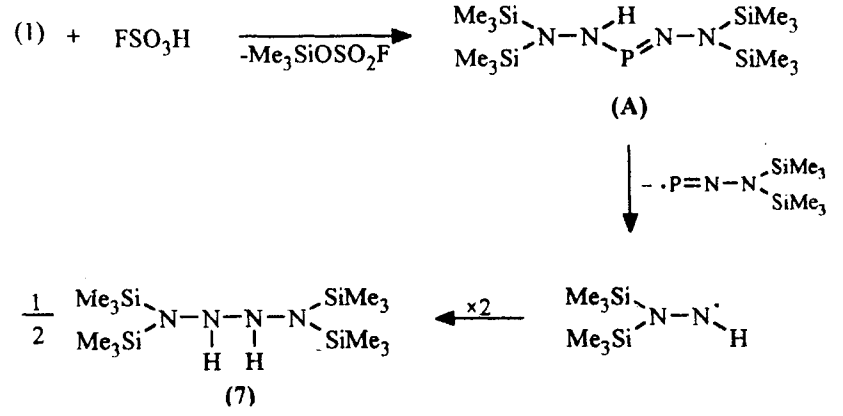


(4b)

(**8**)

The ^{1}H-NMR spectrum of (**7**) in benzene shows a singlet at δ 0.18. ^{29}Si-NMR also shows only one resonance signal at δ 32.66. Mass spectrum shows the molecular ion peak at m/Z 350 and the observed isotopic pattern of the molecular ion agrees well with that calculated for $C_{12}H_{38}N_4Si_4$. Some of the other important fragments are observed at m/Z 349 $(m\text{-}H)^+$, 336 $(m\text{-}Me)^+$, 320 $(m\text{–}2Me)^+$, 277 $(m\text{-}Me_3Si)^+$.

Compound (**7**) is probably formed through an intermediate (A) which decomposes to give bis(trimethylsilyl)hydrazino free radicals. These radicals dimerise on termination to form tetrakis(trimethylsilyl)tetrazane (**7**).

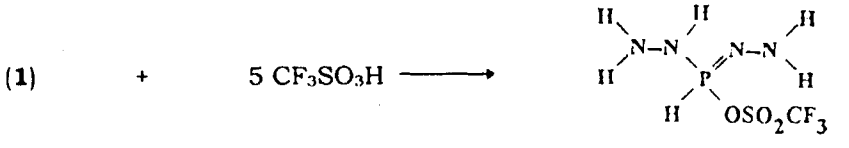


(**A**)

(**7**)

The ^{19}F-NMR spectrum of (**8**) shows a peak at −38.0 ppm in D_2O which corresponds to fluorine of the SO_3F group. The ^{31}P-NMR spectrum shows resonance signal at 0.66 ppm in D_2O which is characteristic of a tetracoordinated P(V)[4,12] compound. Infrared spectrum shows prominent bands (cm^{-1}) at 3300 and 3100 ν(N-H), 2410 ν(P-H), 1255 ν(P=N) and 1050 ν(P-N).

Reaction of CF_3SO_3H with (**1**) yields a completely desilylated oxidative addition product H_2N-NH-P(H)(SO_3CF_3)=N-NH_2 (**9**) which is a white solid insoluble in benzene, ether, n-hexane, $CHCl_3$ but soluble in water, D_2O and THF. Its spectral data (Experimental) is similar to that of (**8**) and characteristic of a phosphorane.



(**9**)

The results indicate that in contrast to tetrasilatetrazene as a source of H_2N-N=N-NH_2, it is difficult to obtain the parent phosphapentazene H_2N-NH-P=N-NH_2 due to higher reactivity at the heteratom or the double bond involving the heteroatom –P=N–.

EXPERIMENTAL

All the investigations were carried out in the absence of air and moisture on a vacuum line connected to a dry nitrogen supply system. Pentasila-3-phospha-2-pentazene[1] was prepared as reported in literature. NMR measurements were recorded on Varian EM 390 and Brucker FT NMR 300 MHz, IR spectra on Perkin Elmer 621 and mass spectra on GC Mass Spectrometer Model 705–250 + (VG). ^{1}H-NMR of Me_3Si protons, δ (ppm) in benzene (Et_2O/CCl_4/n-hexane): $(Me_3Si)_5N_4P$, 0.28 d, 0.21, 0.43 (0.14d, 0.22, 0.34) 2:2:1; $Me_3SiOSiMe_3$ 0.11 (0.06).

General procedure

Pentasila-3-phospha-2-pentazene (**1**) (40 mmol, 18.0 g) dissolved in 50 ml of diethylether/n-hexane maintained at temperatures mentioned in each case and 200 mmol of Me_3COH, CF_3COOH, FSO_3H and CF_3SO_3H diluted with Et_2O/n-hexane were added dropwise. Reaction of (**1**) with an equivalent amount (40 mmol) of CF_3COOH was also carried out. The reaction mixtures were allowed to come to room temperature slowly with stirring. The ^{1}H-NMR spectrum of a part of the filtrate was studied. The main filtrate was evacuated to remove solvent and other volatile compounds up to 60 torr/room temperature and the residue was characterised by ^{1}H-NMR and other methods indicated in each case. These products were separated to isolate various compounds. The products were refractionated/recrystallised to isolate pure fractions which were characterised by the methods described subsequently in each case. The yield of pure major products is based on the amount isolated whereas that of unseparable mixtures or minor products is calculated from ^{1}H-NMR integrated ratios after adding a known amount of cyclohexane or toluene.

Preparation of [bis(trimethylsilyl)hydrazino][bis(trimethylsilyl)hydrazono](t-butoxy)phosphorane (2): Phosphapentazene (**1**) was reacted with t-butyl alcohol in Et_2O at room temperature. The reaction mixture became colourless with white solid at the bottom which was separated by filtration. The colourless filtrate showed ^{1}H-NMR signals at δ 0.20, 0.09 and 0.06. Evacuation of the reaction mixture removed siloxane, $Me_3SiOCMe_3$; the residue left behind was heated in vacuo to obtain a colourless distilled fraction (**2**) at 80°C/10^{-3} torr. It is soluble in common organic solvents.

Analysis

Found: C, 41.20; H, 10.32; N, 12.34; P, 6.76. $C_{16}H_{47}N_4OPSi_4$ Calc.: C, 42.29; H, 10.35; N, 12.34; P, 6.80 %.

^{1}H-NMR in n-hexane δ(ppm) 0.27 [s, 36H, $2(Me_3Si)_2N$], 1.45 [s, 9H, Me_3CO], 10.85 [d, $^1J_{H\text{-}P}$ = 612 Hz, 1H, PH].

Mass spectrum (70 eV) (assignment, relative intensity %): 454 $[(m)^+$, 51], 453 $[(m\text{-}H)^+$, 43], 49 $[(m\text{-}Me)^+$, 25], 437 $[(m\text{-}OH)^+$, 32], 425 $[(m\text{-}C_2H_5)^+$, 22], 381 $[(m\text{-}Me_3CO)^+$, 35], 205 $[((Me_3Si)_2N_2P)^+$, 29], 177 $[(Me_3SiPOCMe_3)^+$, 20], 147 $[Me_3SiOSiMe_3)^+$, 13], 116 $[(MeSiOCMe_3)^+$, 19], 73 $[(Me_3CO)^+$ or $(Me_3Si)^+$, 100].

Preparation of Hydrazino-hydrazono(t-butoxy)phosphorane (3): The white solid obtained after filtration was found to be $H_2N\text{-}NH\text{-}P(H)(OCMe_3)\text{-}N\text{-}NH_2$ **(3)**. It is insoluble in benzene, Et_2O, n-hexane but soluble in water and D_2O.

Analysis

Found: C, 2.31; H, 9.02; N, 32.72. $C_4H_{15}N_4OP$ Calc.: C, 2.40; H, 9.04; N, 33.73 %.

Mass spectrum (70 eV) (assignment, relative intensity %): 166 $[(m)^+$, 27], 165 $[(m\text{-}H)^+$, 13], 164 $[(m\text{–}2H)^+$, 11], 151 $[(m\text{-}CH_3)^+$, 15], 150 $[(m\text{-}NH_2)^+$, 27], 132 $[(m\text{–}2NH_3)^+$, 28], 93 $[(m\text{-}OCMe_3)^+$, 9], 73 $[(Me_3CO)^+$, 75], 57 $[(Me_3C)^+$, 100].

Preparation of [tris(trimethylsilyl)hydrazino][bis(trimethylsilyl)hydra-zono](trifluoroacetato)phosphorane (4): Reaction of **(1)** with CF_3COOH in equivalent amounts was carried out at 50°C. The ^{1}H-NMR spectrum of the reaction mixture showed major resonances at δ 0.29, 0.21. Evacuation of the reaction mixture thoroughly at RT/ 10^{-1} torr, left a light yellow residue. On heating in vacuo this distilled at 100°C/10^{-3} torr to provide **(4)**. It is a colourless liquid miscible with common organic solvents.

Analysis

^{1}H-NMR in Et_2O or n-hexane: δ(ppm) 0.33 [d, $^4J_{H\text{-}P}$ = 0.6 Hz, 18H, $(Me_3Si)_2N\text{-}N$=], 0.28 [s, 18H, $(Me_3Si)_2N$], 0.23 [s, 9H, Me_3SiN), 10.95 [d, $^1J_{H\text{-}P}$ = 669 Hz, 1H, PH].

^{29}Si-NMR in C_6D_6 (TMS external standard): δ(ppm) 11.64 [s, 2Si, $(Me_3Si)_2N\text{-}N$=], 7.31 [s, 2Si, $(Me_3Si)_2N$], 4.10 [s, 1Si, Me_3SiN].

Mass spectrum (70 eV) (assignment, relative intensity %): 566 $[(m)^+$, 5], 547 $[(m\text{-}F)^+$, 5], 532 $[(m\text{-}MeF)^+$, 11], 522 $[(m\text{-}CO_2)^+$, 32], 497 $((m\text{-}CF_3)^+$, 60], 453 $[(m\text{-}CF_3COO)^+$, 62], 420 $[(m\text{–}2Me_3Si)^+$, 55], 380 $((m\text{-}CF_3COOSiMe_3)^+$, 74],

191 ($Me_3SiN{=}PSiMe_3$)$^+$, 186 [($Me_3SiOOCF_3$)$^+$, 52], 113 [(CF_3COO)$^+$, 17], 73 [(Me_3Si)$^+$, 100].

Preparation of (trimethylsilylhydrazino)(trimethylsilylhydrazono)(trifluoroacetato)phosphorane (5): Reaction of (**1**) and CF_3COOH in the stoichiometric ratio 1:5 was carried out at −50°C in n-hexane. The reaction mixture became colourless with white solid at the bottom which was isolated through filtration. The filtrate showed ^{1}H-NMR signals at δ 0.29 and 0.23. The signal at δ 0.29 was due to $CF_3COOSiMe_3$. The mixture was evacuated at RT/10^{-1} torr; the residue obtained was heated in vacuo to get a colourless distilled fraction at 70°C/10^{-3} torr which was found to be (**5**). It is a colourless liquid miscible with common organic solvents.

Analysis

Found: C, 27.40; H, 6.18; N, 15.98; P, 8.71; F, 16.20. $C_8H_{22}N_4O_2PSi_2$Calc.: C, 27.42; H, 6.0; N, 16.00, P, 8.85; F, 16.28 %.

^{1}H-NMR in benzene (n-hexane): δ(ppm) 0.24 (0.30) [s, 18H, $2Me_3SiN$], 6.8 (6.8) [b, s, 1H, HN], 10.4 (10.4) [b, s, 1H, HN], 14.8 (14.8) [b, s, 1H, HN], 10.8 (10.8) [d, $^1J_{H\text{-}P}$ = 594 Hz, s, 1H, PH].

^{19}F-NMR in n-hexane (freon external standard): δ 78.9.

^{31}P-NMR in C_6D_6 (H_3PO_4 external standard): δ 3.00.

^{29}Si-NMR in C_6D_6 (TMS external standard): δ 21.66 [s, 2Si, $2Me_3SiN$].

IR bands (cm^{-1}): 3100 ν(N-H), 2360 ν(P-H), 1700 ν(C=O), 1260 ν(P=N), 1220 ν(C-F), 1050 ν(C-O), 920 ν(C-F).

Mass spectrum (70 eV) (assignment, relative intensity %): 350 [(m)$^+$, 5], 316 [(m-MeF)$^+$, 8], 306 [(m-CO_2)$^+$, 12], 281 [(m-CF_3)$^+$, 10], 195 [(m-Me_2SiCF_3CO)$^+$, 14], 155 [(Me_2SiCF_3CO)$^+$, 61], 147 [($Me_3SiOSiMe_2$)$^+$, 19], 127 [(Me_2SiCF_3)$^+$, 18], 97 [(CF_3CO)$^+$, 15], 73 [(Me_3Si)$^+$, 45].

Preparation of 1,3-diamino-2, 4-dihydrazono-2, 4-bis(trifluoroacetato)-1,3, 2,4-diazadiphosphetidine (6): The white solid obtained after filtration was found to be (**6**). It is insoluble in n-hexane, ether, CH_2Cl_2, $CHCl_3$, benzene and THF but soluble in water, D_2O and methanol.

Analysis

Found: C, 11.66; H, 1.89; N, 27.40; F, 27.92. $C_4H_8N_8P_2F_6O_4$ Calc.: C, 11.76; H, 1.96; N, 27.45; F, 27.94.

IR bands (cm^{-1}): 3250 and 3125 ν(N-H), 1690 ν(C=O), 1260 ν(P=N), 1210 ν(C-F), 1060 ν(C-O), 960 ν(C-F).

Mass spectrum (70 eV) (assignment, relative intensity %): 408 [$(m)^+$, 15], 392 [$(m\text{-}NH_2)^+$, 12], 370 [$(m\text{-}F_2)^+$, 34], 339 [$(m\text{-}CF_3)^+$, 55], 311 [$(m\text{-}CF_3CO)^+$, 45], 294 [$(m\text{-}CF_3COOH)^+$, 31], 214 [$(m\text{–}2CF_3CO)^+$, 65], 205 [$(m\text{-}H_4N_4PCF_3COO)^+ = (m')^+$, 54], 177 [$(m'\text{-}CO)^+$, 18], 136 [$(m'\text{-}CF_3)^+$, 17], 69 [$(CF_3)^+$, 100].

Preparation of tetrakis(trimethylsilyl)tetrazane (7): **(1)** and FSO_3H were allowed to react in n-hexane at –50°C. The reaction mixture became colourless with a white solid at the bottom which was isolated by filtration and ^{1}H-NMR showed resonance peaks at δ 0.32 and 0.15 d. The signal at δ 0.15 was found to be due to Me_3SiF and the concentrated liquid left behind was heated in vacuo to obtain a colourless distilled fraction at 60°C/10^{-3} torr which was found to be **(7)**. Compound **(7)** is a colourless liquid miscible with common organic solvents.

Analysis

Found: C, 41.10; H, 10.72; N, 15.75. $C_{12}H_{38}N_4Si_4$ Calc.: C, 41.14; H, 10.85; N, 16.00 %.

^{1}H-NMR in n-hexane δ(ppm) 0.31 [s, 36H, $2(Me_3Si)_2N$].

IR bands (cm^{-1}): 855 ν(Si_2N), 450 ν(Si-N).

Mass spectrum (70 eV) (assignment, relative intensity %): 350 [$(m)^+$, 15], 349 [$(m\text{-}H)^+$, 20], 336 [$(m\text{-}Me)^+$, 45], 320 [$(m\text{–}2Me)^+$, 70], 277 [$(m\text{-}Me_3Si)^+$, 59], 263 [$(m\text{-}Me_3SiN)^+$, 28], 204 [$(m\text{–}2Me_3Si)^+$, 21], 174 [$(Me_3Si)_2N_2^+$, 27].

Preparation of Hydrazino-hydrazono-(fluorosulphato)phosphorane (8):The white solid obtained after filtration was characterised to be **(8)**. It is insoluble in ether, n-hexane, benzene but soluble in water and D_2O.

Analysis

Found: H, 3.02; N, 28.06; F, 9.80. $H_6FN_4O_3PS$ Calc.: H, 3.12; N, 29.16; F, 9.89 %.

Mass spectrum (70 eV) (assignment, relative intensity %): 192 [$(m)^+$, 15), 176 ($(m\text{-}NH_2)^+$, 11], 160 [$(m\text{-}N_2H_4)^+$, 85),144 [$(m\text{-}SO)^+$, 71], 128 ($(m\text{-}SO_2)^+$, 73], 112 [$(m\text{-}SO_3)^+$, 25], 90 [$(m\text{-}SO_3F)^+$, 55], 73 ($(SO_2F)^+$, 100].

Preparation of Hydrazino-hydrazono(trifluoromethanesulphato)-phosphorane (9): Reaction of Phosphapentazene **(1)** with trifluoromethanesulfonic acid was carried out at –70°C. The reaction mixture became colourless at room temperature when some white precipitates started settling at the bottom. The reaction mixture was filtered to isolate white solid from the colourless filtrate which showed ^{1}H-NMR resonance signal at δ 0.41 due to $Me_3SiSO_3CF_3$which was

removed in vacuo. The white solid obtained was washed with Et_2O and n-hexane, dried in vacuo, and characterised to be **(9)**.

It is a white solid insoluble in benzene, Et_2O, n-hexane, chloroform but soluble in water, D_2O, THF.

Analysis

Found: C, 4.95; H, 2.45; N, 22.10; F, 22.53. $CH_6F_3N_4O_3PS$ Calc.: C, 4.95; H, 2.47; N, 23.14; F, 23.55 %.

^{19}F-NMR in D_2O (freon external standard): δ 79.24.

^{31}P-NMR in D_2O (H_3PO_4 external standard): δ–10.17.

IR bands (cm^{-1}): 3300 and 3150 ν(N-H), 2410 ν(P-H), 1250 ν(P=N), 1050 ν(P-N).

Mass spectrum (70 eV) (assignment, relative intensity %): 242 [$(m)^+$, 9], 223 [$(m\text{-}F)^+$, 18], 210 [$(m\text{-}N_2H_4)^+$, 11], 194 [$(m\text{-}SO)^+$, 25], 185 [$(m\text{-}F_3)^+$, 18], 178 [$(m\text{-}SO_2)^+$, 45], 162 ($(m\text{-}SO_3)^+$, 31], 149 [$(CF_3SO_3)^+$, 31], 125 [$(m\text{-}CF_3SO)^+$, 80], 73 [$(SO_2)^+$, 100].

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