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Synthesis of a *C*-pyridine-substituted 2*H*-1,3,2-diazaphosphole complex and subsequent oxidation to its P^V-sulfide and P^V-selenide derivatives

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Abstract

Thermal ring opening of 2*H*-azaphosphirene tungsten complex **1** in the presence of 2-piperidino carbonitrile and 2-cyanopyridine furnished selectively the 2*H*-1,3,2-diazaphosphole tungsten complex **3**. Liberation of the diazaphosphole ligand by applying various decomplexation reagents failed. However, the corresponding P^V-sulfide **4** and P^V-selenide **5** were obtained by oxidative decomplexation using elemental sulfur and selenium. All products were characterized by NMR spectroscopy and MS spectrometry. The constitution of **3** and **5** could also be established by single crystal X-ray diffraction.

Keywords: *C*-Pyridine; 2*H*-1,3,2-Diazaphosphole; Oxidation; O2H-ataphorphirene; P^v-sulfide; P^v-selenide

1. Introduction

Since their discovery at the end of the 19th century [1], 2,2′-bipyridine [2] (**I**) and their derivatives became important chelating ligand systems in organometallic and coordination chemistry and have found several applications; e.g. in catalysis. Their versatility has spurred the exploration of other bidentate ligands **II**–**IV** having 1,4-diazabutadiene moieties (Scheme 1) and which can be synthesized in good yields from readily available starting materials.

Our interest was to synthesize phosphorus-analogues of 2-pyrazolylpyridines [3] **III** using the thermal ring opening of 2*H*-azaphosphirene complexes [4] in the presence of two different carbonitriles and subsequent decomplexation of the pyridyl-substituted 2*H*-1,3,2-diazaphosphole.

2. Results and discussion

Heating a toluene solution of the 2*H*-azaphosphirene tungsten complex **1** [5] in the presence of 1-piperidino carbonitrile and 2-cyanopyridine yields the 2*H*-1,3,2-diazaphosphole tungsten complex **3** as the sole phosphorus containing product. The formation of **3** most probably [6] occurs via the generation of transient nitrilium-phosphane ylid complex **2** and its trapping with the 2-cyanopyridine, which proceeds regioselectively (Scheme 2).

Complex **3** shows a ^{31}P-NMR resonance at 148.6 ppm and a phosphorus–tungsten coupling constant of 256.7 Hz (Table 1). These values are typical for such complexes, as reported earlier [7], for example complex **8** (Scheme 5), which shows a chemical shift in ^{31}P-NMR of δ 149.9 and a tungsten–phosphorus coupling constant of 259.1 Hz. The ^{13}C-NMR resonances of the ring carbon atoms show values of 160.4 and 163.2 ppm, whereby the assignment is tentative as in previous cases [7]. These values as well as the phosphorus–carbon coupling constants are similar to those of 2*H*-1,3,2-diazaphosphole tungsten complex **8** [8]. Reactions of **3** with elemental sulfur or selenium in excess furnished the

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corresponding phosphorus chalcogenides **4** and **5**, respectively (Scheme 3).

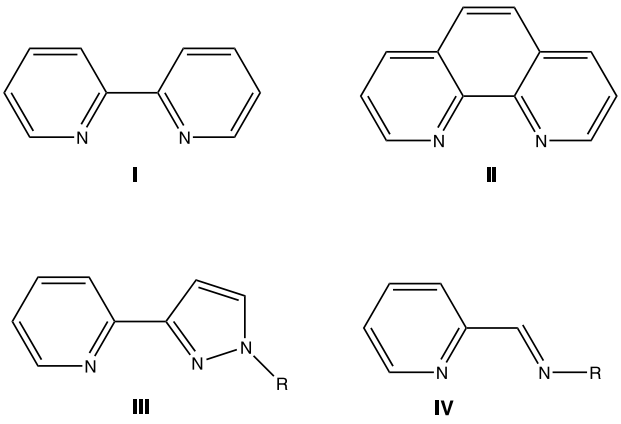
They show ^{31}P resonances at 124.1 (**4**) and 116.9 (**5**), the latter additionally a phosphorus–selenium coupling constant of 775.8 Hz. The ^{13}C-NMR spectra of compounds **4** and **5** show resonances for the ring carbon atoms at 161.4 and 166.0 (**4**) and at 161.5 and 165.0 (**5**), respectively (Table 1). These values of **4** and **5** are similar to those of **3** but the phosphorus–carbon coupling constant of C^4 are significantly increased by about 23 Hz (**4**) and 10.0 Hz for the selenide **5**. A similar effect is observed for the *ipso*-carbon atoms of the pyridine of **4** and **5**, thus showing an increase of about 7 to 9 Hz. These increase of the phosphorus–carbon coupling constants could also be observed in the 2*H*-1,3,2-diazaphospole *P*-sulfide **10**, which was reported earlier [8].

Attempts to obtain the 2*H*-1,3,2-diazaphosphole ligand from **3** via decomplexation by heating with DPPE were unsuccessful. However, 2*H*-1,3,2-diazaphosphole **6** was obtained by deselenization of **5** using $P(NMe_2)_3$ in toluene at ambient temperature affording $SeP(NMe_2)_3$ as the other product (Scheme 4).

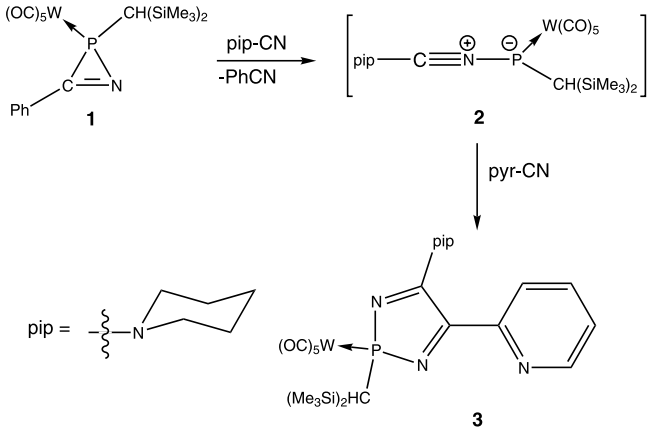
Despite several attempts, it could not be isolated using low-temperature column chromatography.

Compound **6** shows a ^{31}P-NMR resonance at 139.0 ppm, which is very close to the chemical shift of the 2*H*-1,3,2-diazaphosphole **9** (Scheme 5), the only example reported so far, which shows a ^{31}P-NMR resonance at 140.2 ppm [8].

Compound **6** was selectively oxidized at ambient temperature with urea–H_2O_2 giving a product that appeared at 52.0 ppm in the ^{31}P-NMR spectrum of the reaction solution (Scheme 6); we assume the formation of the P^V-oxide **7**, which would be consistent with literature values reported for such P^V-oxides such as 3,4-dimethyl-1-phenyl phosphole-*P*-oxide (42.3 ppm) [9]. Because of decomposition during column chromatography (even at low-temperature), compounds **6** and **7** were not isolated.



Scheme 1. Bidentate ligands **II**–**IV** with 1,4-diazabutadiene moieties.

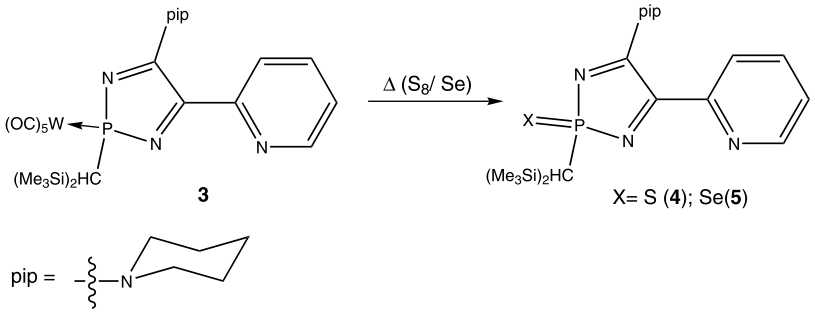


Scheme 2. Formation of 2*H*-1,3,2-diazaphosphole complex **3** via ring opening and trapping of nitrilium phosphane ylid complex **2**.

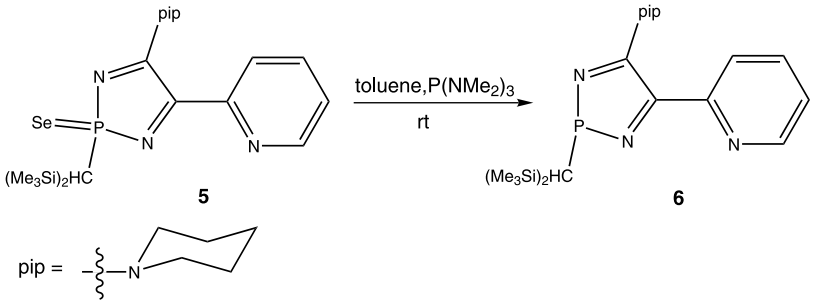
Table 1
Selected NMR-data of **3**–**5** and **8** [7], **10** [8]

Compound	δ ^{31}P (ppm)	δ $^{13}C^{arom.}$	δ $^{13}C^4$	δ $^{13}C^5$
		$^{[3]}J$(P,C) (Hz)	$^{[2+3]}J$(P,C) (Hz)	$^{[2+3]}J$(P,C) (Hz)
3	148.6	154.7	160.4	163.2
	^1J(P,W) = 256.7 Hz	(25.3)	(7.9)	(#)
4	124.1	154.8	161.4	166.0
		34.1	(31.0)	(#)
5	116.9	154.6	161.5	165.0
	^1J(P,Se) = 775.8 Hz	(32.7)	(17.6)	(#)
8 [7]	149.9	135.9	162.3	165.3
	(259.1)	(22.4)	(8.8)	(#)
10 [8]	123.4	135.8	163.6	167.7
		(29.6)	(19.9)	(#)

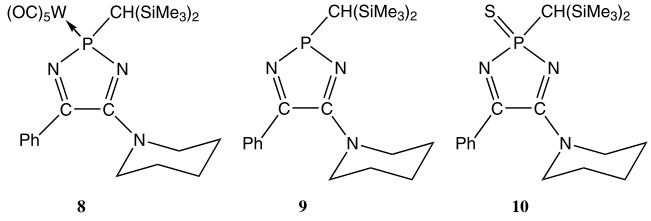
= coupling not resolved.



Scheme 3. Decomplexation of **3** with elemental sulfur and selenium.



Scheme 4. Deselenization of **5** with $P(NMe_2)_3$.



Scheme 5. 2*H*-1,3,2-Diazaphospholes **8**–**10**.

3. Discussion of X-ray data of 3 and 5

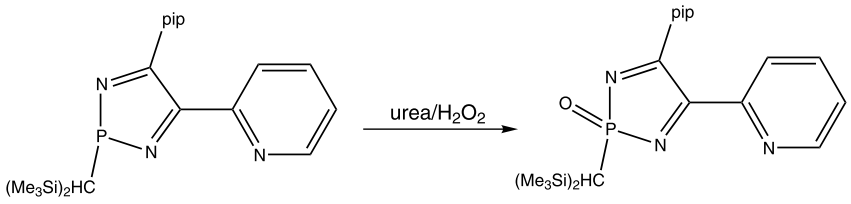
The molecular structures of **3** (Fig. 1) and **5** (Fig. 2) show that the pyridine ring and the P-heterocyclic ring are not coplanar and have torsion angles of 40° (**3**) and 47.8 (**5**), which is a significant difference to 2-pyrazolylpyridines or 2,2′-bipyridines, whereby the latter very often show almost coplanar rings. In the case of 2-pyrazolylpyridines, the torsion angle of the rings vary between 0 and 16°, depending strongly on the bulkyness of the substituents of the 5-membered heterocycle [4]. The lengths of the interconnecting carbon–carbon bond of the 2*H*-1,3,2-diazaphosphole and the pyridine ring in **3** and **5** (C(6)–C(15) 1.468(3) and C(1)–C(10) 1.475(6) Å, respectively) are slightly shorter than in 2,2′-bipyridine [10] (1.493 Å) but quite similar to the parent 2-pyrazolylpyridine [11] (1.472 Å). Complex **3** and selenide **5** have bond lengths and angles as expected for this heterocyclic ring system. It is noteworthy that in the crystal structure of compound **5** there are intermolecular contacts (3.341(10) Å) between one of the hydrogen atoms on C(17) from the piperidine ring and the nitrogen N(4) from the pyridine ring. These contacts were generated by symmetry operators $x, -y+1/2, z-1/2$.

Currently, investigations on the reactivity of these novel ligands towards transition metal ions are underway.

4. Experimental

4.1. General procedures

All reactions and manipulations were carried out under an inert atmosphere of deoxygenated dry nitrogen with the use of standard schlenk-line techniques. All solvents were dried by conventional methods and distilled over nitrogen. All reactions were monitored by ^{31}P-NMR spectroscopy. The ^{1}H-, ^{13}C- and ^{31}P-NMR spectra were recorded on a Bruker AC-200 spectrometer (200 MHz for ^{1}H; 50.3 MHz for ^{13}C and 81.0 MHz for ^{31}P) using (D)chloroform as solvent and internal standard; shifts are given relative to ext. Me_4Si (^{1}H, ^{13}C) or 85% H_3PO_4 (^{31}P). Mass spectra were recorded on a Finnigan Mat 8430 (70 eV, EI); apart from the molecule ions, only the *m*/*z*-values having intensities of more than 20% are given. Infrared spectra were recorded on a Biorad FTIR 165 spectrometer (selected data given). Melting points (m.p.) were obtained on a Büchi 535 capillary apparatus. Elemental analyses were performed using a Carlo Erba analytical gas-chromatograph. The



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Scheme 6. Oxidation of **6** with urea–H_2O_2.

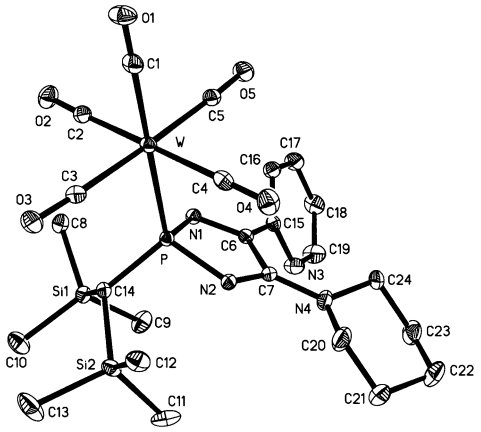

Fig. 1. Molecular structure of **3** in the crystal (hydrogen atoms are omitted for clarity). Selected bond lengths (Å) and angles (°): P–W 2.4916(5), P–N(1) 1.7387(16), P–N(2) 1.6842(16), N(1)–C(6) 1.292(2), N(2)–C(7) 1.300(2), C(6)–C(7) 1.537(3); N(1)–P–N(2) 96.49(8), N(1)–P–W 111.28(13), N(2)–P–W 114.09(12), C(14)–P–Se 118.63(6).

κP-notation in the nomenclature is intended to differentiate between P- and N-coordinations of the appropriate heterocycle to the metal.

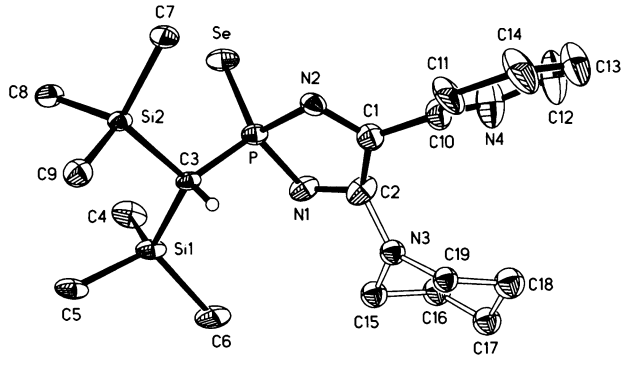

Fig. 2. Molecular structure of **5** in the crystal (hydrogen atoms are omitted for clarity). Selected bond lengths (Å) and angles (°): P–Se 2.097(11), P–N(1) 1.675(3), P–N(2) 1.724(3), N(1)–C(2) 1.303(6), N(2)–C(1) 1.284(5), C(1)–C(2) 1.538(6); N(1)–P–N(2) 97.82(17), N(1)–P–Se 114.36(13), N(2)–P–Se 111.51(12), C(3)–P–Se 116.13(12).

*4.2. {Pentacarbonyl[2-bis(trimethylsilyl)methyl-4-pyridino-5-piperidino-1,3,2-diazaphosphole-κP]-tungsten(0)} (**3**)*

2*H*-Azaphosphirene complex **1** (0.617 g, 1 mmol) was dissolved in 3 ml toluene followed by addition of 2-cyanopyridine (0.312 g, 3 mmol) and piperidinocarbonitrile (0.220 g, 2 mmol) and was heated at 75 °C for 2 h. The excess of toluene was removed in vacuo and the product separated by low-temperature column chromatography [SiO_2, −2 °C, petrol ether(40–60 °C)/Et_2O 87.5:12.5]. Evaporation of the solvents of the third fraction and crystallization from Et_2O at −25 °C yielded **3** as yellow crystals (0.553 g, 76%); m.p. 104 °C; IR (KBr) *ν* 2070 (s), 1987 (s), 1959 (vs), 1911 (vs), 1898 (vs) cm^{-1} (CO); 1578 (w), 1536 (m) cm^{-1} (C=N); ^{1}H-NMR ($CDCl_3$): *δ* 0.12 (s, 9H, $SiMe_3$), 0.27 (s, 9H, $SiMe_3$), 1.37 (d, ^2J(P,H) = 7.2 Hz, 1H, C*H*$SiMe_3$), 1.59 (s br, 6H, NCH_2CH_2), 3.26 (m_c br, 4H, NCH_2), 7.38 (m_c, 1H, 4-Pyr), 7.81 (m_c, 2H, 3,5-Pyr), 8.69 (m_c, 1H, 6-Pyr); ^{13}C{^{1}H}-NMR ($CDCl_3$): *δ* 2.9 (s br, $SiMe_3$), 24.4 (s, $NCH_2CH_2CH_2$), 24.5 (s br, $CH(SiMe_3)_2$), 25.5 (s, NCH_2CH_2), 50.0 (s, NCH_2), 124.2 (s, Pyr), 124.7 (s, Pyr), 137.2 (s, 4-Pyr), 149.8 (s, 6-Pyr), 154.7 (d, ^3J(P,C) = 25.3 Hz, Pyr), 160.4 ($^{[2+3]}J$(P,C) = 7.9 Hz, PN*C*), 163.2 (s br, PN*C*), 197.3 (d, ^2J(P,C) = 7.5 Hz, *cis*-CO), 200.3 (d, ^2J(P,C) = 25.1 Hz, *trans*-CO); ^{31}P{^{1}H}-NMR ($CDCl_3$): *δ* 148.6 (s, ^1J(P,W) = 256.7 Hz); MS (70 eV, EI, ^{184}W) *m/z* 728 ([M^+], 4), 700 ([M^+ −1 CO], 17), 672 ([M^+ −2 CO], 28), 644 ([M^+ −3 CO], 10), 588 ([M^+ −5 CO], 62), 294 ([$WC_6H_{10}N_2^+$], 100), 73 ([$SiMe_3^+$], 23); Anal. Calc. for $C_{24}H_{33}N_4O_5PSi_2W$ (728.50): C, 39.57; H, 4.57; N, 7.69. Found: C, 39.15; H, 4.57; N, 7.54%.

*4.3. 2-Bis(trimethylsilyl)methyl-4-pyridino-5-piperidino-1,3,2-diazaphosphole-P-sulfide (**4**)*

2*H*-Azaphosphirene complex **1** (0.617 g, 1 mmol) and an excess of sulfur (0.10 g) were mixed in 3 ml of toluene and heated at 90 °C for 24 h. The excess of solvent was removed in vacuo and the product separated by low-temperature column chromatography [SiO_2, −10 °C, petrol ether (40–60 °C)/Et_2O 90:10]. Evaporation of the solvents of the second fraction and crystallization from

Et_2O at −25 °C yielded **5** as yellow crystals (0.314 g, 72%); m.p. 102–104 °C (decomp.); IR (KBr) *v* 1584 (s), 1561 (vs) cm^{-1} (C=N); 677 (s) cm^{-1} (P=S); ^{1}H-NMR ($CDCl_3$): δ 0.29 (s, 9H, $SiMe_3$), 0.32 (s, 9H, $SiMe_3$), 0.95 (d, ^2J(P,H) = 21.9 Hz, 1H, C*H*$SiMe_3$), 1.61 (s br, 6H, NCH_2CH_2), 3.26 (m_c br, 4H, NCH_2), 7.32 (m_c, 1H, 4-Pyr), 7.41 (m_c, 2H, 3,5-Pyr), 8.65 (m_c, 1H, 6-Pyr); $^{13}C\{^1H\}$-NMR ($CDCl_3$): δ 2.35 (s, $SiMe_3$), 24.0 (s, $NCH_2CH_2CH_2$), 25.6 (s br, *C*H($SiMe_3$)$_2$), 25.8 (s, NCH_2CH_2), 49.8 (s, NCH_2), 123.9 (s, Pyr), 125.0 (s, Pyr), 137.3 (s, 4-Pyr), 149.0 (s, 6-Pyr), 154.8 (d, ^3J(P,C) = 34.1 Hz, 2-Pyr), 161.4 (d, $^{[2+3]}J$(P,C) = 31.0 Hz, PN*C*), 166.0 (s, PN*C*); $^{31}P\{^1H\}$-NMR ($CDCl_3$): δ 124.1 (s); MS (70 eV, EI, ^{184}W) *m*/*z* 436(20) [M^+], 256(45) [$(C_{11}H_{19}N_2PSi_2)^+$], 245(25) [$(C_{12}H_9N_4P)^+$], 160(28) [$(C_7H_{20}Si_2)^+$], 128(25) [$(C_4H_{11}PSi)^+$], 73(100) [$(SiMe_3)^+$], 64(48) [$(C_5H_4)^+$], 44(22) [$(C_3H_8)^+$]; Anal. Calc. for $C_{19}H_{33}N_4PSSi_2$ (436.17): C, 52.26; H, 7.62; N, 12.83; S, 7.34. Found: C, 52.36; H, 7.60; N, 12.75; S, 7.39%.

4.4. 2-Bis(trimethylsilyl)methyl-4-pyridino-5-piperidino-1,3,2-diazaphosphole-P-selenide (5)

2*H*-Azaphosphirene complex **1** (0.617 g, 1 mmol) and an excess of selenium powder (0.80 g) were mixed in 5 ml of benzonitrile and heated at 95 °C for 48 h. The excess of selenium was filtered and the solvent was removed in vacuo. The product was separated by low-temperature column chromatography [SiO_2, −20 °C, petrol ether (40–60 °C)/Et_2O 50:50]. Evaporation of the solvents of the second fraction and crystallization from Et_2O at −25 °C yielded **5** as yellow crystals (0.242 g, 50%); m.p. 137 °C (decomp.); IR (KBr) *v* 1584 (vs), 1561 (s) cm^{-1} (C=N); 618 (m) cm^{-1} (P=Se); ^{1}H-NMR ($CDCl_3$): δ 0.25 (s, 9H, $SiMe_3$), 0.26 (s, 9H, $SiMe_3$), 1.22 (d, ^2J(P,H) = 23.8 Hz, 1H, C*H*$SiMe_3$), 1.56 (s br, 6H, NCH_2CH_2), 3.32 (m_c br, 4H, NCH_2), 7.35 (m_c, 1H, 4-Pyr), 7.78 (m_c, 2H, 3,5-Pyr), 8.61 (m_c, 1H, 6-Pyr); $^{13}C\{^1H\}$-NMR ($CDCl_3$): δ 2.4 (s, $SiMe_3$), 24.1 (s, $NCH_2CH_2CH_2$), 25.0 (s br, *C*H($SiMe_3$)$_2$), 25.7 (s, NCH_2CH_2), 49.9 (s, NCH_2), 124.2(s, Pyr), 125.0 (s, Pyr), 137.3 (s, 4-Pyr), 149.0 (s, 6-Pyr), 154.6 (d, ^3J(P,C) = 32.7 Hz, 2-Pyr), 161.5 (d, $^{[2+3]}J$(P,C) = 17.6 Hz, PN*C*), 165.0 (s, PN*C*); $^{31}P\{^1H\}$-NMR ($CDCl_3$): δ 116.9 (s, ^1J(P,Se) = 775.8 Hz); MS (70 eV, EI, ^{79}Se) *m*/*z* 484(4) [M^+], 279(42) [$(C_{12}H_{20}N_2PSi_2)^+$], 167(44) [$(C_7H_{10}N_3P)^+$], 149(100) [$(C_6H_4N_3P)^+$]; Anal. Calc. for $C_{19}H_{33}N_4PSeSi_2$ (483.60): C, 47.19; H, 6.88; N, 11.59. Found: C, 47.16; H, 6.94; N, 11.58%.

4.5. 2-Bis(trimethylsilyl)methyl-4-pyridino-5-piperidino-1,3,2-diazaphosphole (6)

Four hundred milligrams (0.826 mmol) of 2*H*-1,3,2-diazaphosphole-*P*-selenide (**5**) dissolved in 2.5 ml of toluene were treated with 135 mg (0.826 mmol) of tris(dimethylamino)phosphine. The reaction mixture was stirred for 2.5 h until **5** was completely consumed (^{31}P-NMR). Besides the tris(dimethylamino)phosphine selenide, the assumed free 2*H*-1,3,2-diazaphosphole **6** was observed by $^{31}P\{^1H\}$-NMR spectroscopy ($CDCl_3$): δ 139.0. The product, decomposed during column chromatography at low-temperatures (−10– −20 °C). For the subsequent oxidation of **6** to **7** with urea–H_2O_2, this crude product was used.

4.6. 2-Bis(trimethylsilyl)methyl-4-pyridino-5-piperidino-1,3,2-diazaphosphole-P-oxide (7)

Compound **6** was dissolved in 2.5 ml of toluene and 188 mg (2 mmol) of urea–H_2O_2 were added at room temperature. The reaction mixture was stirred for 2 h until complete consumption of **6**. The resulting product **7**, the proposed 2*H*-1,3,2-diazaphosphole-*P*-oxide [$^{31}P\{^1H\}$-NMR ($CDCl_3$): δ 52.0], decomposed during column chromatography at low-temperatures (−10– −20 °C).

4.7. Crystal structure determination of 3

Crystal data: $C_{24}H_{28}N_4O_5PSi_2W$, M = 723.50, P(-1), a = 9.0500(8), b = 12.8159(11), c = 14.0679(12) Å, α = 92.351(3), β = 100.320(3), γ = 109.445(3)°, V = 1504.8(2) Å^3, Z = 2, δ_{calc} = 1.597 g cm^{-3}, μ = 4.009 mm^{-1}, T = 133(2) K. A yellow tablet was mounted in inert oil. 33 798 reflections were measured (2θ max 60°) using monochromated Mo–K_α radiation on a Bruker Smart 1000 CCD diffractometer, of which 8787 were unique and used for all calculations (program SHELXL-97 [12]). An absorption correction was based on multiple scans. The structure was refined anisotropically on F^2. All hydrogen atoms were refined with a riding model or using rigid methyl groups. The final wR_1 was 0.0197 with conventional wR_2 = 0.00498, for 340 parameters and 63 restraints; highest peak 1.494, hole −1.494 e Å^{-3}.

4.8. Crystal structure determination of 5

Crystal data: $C_{19}H_{33}N_4PSeSi_2$, M = 483.60, $P2_1/c$, a = 11.9606(14), b = 16.899(2), c = 12.0854(14) Å, β = 96.872(3)°, V = 2425.1(5) Å^3, Z = 4, δ_{calc} = 1.325 g cm^{-3}, μ = 1.725 mm^{-1}, T = 133(2) K. A yellow tablet was mounted in inert oil. 34060 reflections were measured (2θ max 56°) using monochromated Mo–K_α radiation on a Bruker Smart 1000 CCD diffractometer, of which 6009 were unique and used for all calculations (program SHELXL-97 [12]). An absorption correction was based on multiple scans. The structure was refined anisotropically on F^2. All hydrogen atoms were refined with a riding model or using rigid methyl groups. The

final wR_1 was 0.0535 with conventional $wR_2 = 0.1507$, for 279 parameters and 168 restraints; highest peak 0.956, hole -0.930 e Å^3.

5. Supplementary material

Crystallographic data (excluding structure factors) for the structures reported in this paper has been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 218926 and 218927. Copies of this information may be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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