ChemComm

Branched tetrasilane substituted phosphines – synthesis and characterisation of PhSi(SiiPr2)3P and {PhSi(SiMe2)3}2P14

Journal:	*ChemComm*
Manuscript ID:	CC-COM-01-2014-000165.R1
Article Type:	Communication
Date Submitted by the Author:	13-Feb-2014
Complete List of Authors:	von Hänisch, Carsten; University marburg, Fachbereich Chemie Feierabend, Michael; Uni Marburg, Fachbereich Chemie

Cite this: DOI: 10.1039/c0xx00000x

www.rsc.org/xxxxxx

ARTICLE TYPE

Branched tetrasilane substituted phosphines – synthesis and characterisation of $PhSi(Si\mathit{i}Pr_2)_3P$ and $\{PhSi(SiMe_2)_3\}_2P_{14}$†

Michael Feierabend[a] and Carsten von Hänisch*[a]

Received (in XXX, XXX) Xth XXXXXXXXX 200X, Accepted Xth XXXXXXXXX 201X

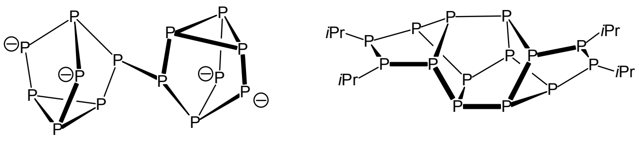
DOI: 10.1039/b000000x

The branched trichlorotetrasilane $PhSi(SiMe_2Cl)_3$ reacts with $P_7(SiMe_3)_3$ under formation of the new oligophosphane $\{PhSi(SiMe_2)_3\}_2P_{14}$ (1), which consists of two $PhSi(SiMe_2)_3$ substituted P_7 norbornane units. The phosphatetrasila[1.1.1]pentane derivative $PhSi(Si\mathit{i}Pr_2)_3P$ (3) was obtained from the reaction of $PhSi(Si\mathit{i}Pr_2Cl)_3$ (2) with Li_3P.

Silicon-phosphorus compounds are still of considerable interest due to their versatile molecular structures and bonding properties. Moreover, they are useful synthons for the formation of several other phosphorus as well as silicon compounds.[1] Silyl groups are also able to stabilise highly reactive phosphorus species such as P_7^{3-}, the silylderivatives of which (e.g. $P_7(SiMe_3)_3$ or $P_7\{Si(SiMe_3)_3\}_3$) are much less reactive concerning oxidation or hydrolysis.[2] As could be shown in recent works, bridging silyl substituents such as bidentate silyl or siloxane groups have major impact on the structures and properties of oligophosphanes.[3] Thus, we decided to investigate to which extend tripodal silyl groups are able to stabilise new P_n-compounds.[4]

Herein, we report on the application of the compound $PhSi(SiMe_2Cl)_3$ as substituent for P_7^{3-}. Moreover, we present the synthesis of the sterically demanding branched trichlorotetrasilane $PhSi(Si\mathit{i}Pr_2Cl)_3$ as well as its reaction with Li_3P.

After work-up of the reaction of $P_7(SiMe_3)_3$ with $PhSi(SiMe_2Cl)_3$ in DME, the compound $\{PhSi(SiMe_2)_3\}_2P_{14}$ (**1**) can be obtained as yellow crystals in 53 % yield. The latter was formed by dimerisation of the P_7 cage through substitution of the silyl groups and represents a silyl derivative of the so far unknown Zintl anion P_{14}^{6-}. The P_{14} framework of compound **1** consists of two P_7 norbornane cages. In both P_7 norbornane subunits, one P_5 ring is substituted by the branched silyl frame in a 1,2,4 positions. These two P_5Si_4 fragments show the same structure as the P_9 cage in Hittorf's phosphorus, and they are connected through a central P_4 ring. Until now, no comparable P_{14} substructure was observed in molecular compounds. The known Zintl ion P_{14}^{4-} consists of two P_7 nortricyclan cages connected by one P-P bond.[5] The compound $P_{14}\mathit{i}Pr_4$, consists – like compound **1** – of two P_7 norbornane units, which are connected by three P-P bonds via P_5 rings in a 1,2,4 position.[6] The P_{14} unit in **1**, however, represents a part within the phosphorus strands in $[(CuI)_8P_{12}]$.[7]



Scheme 1: Structurally characterised ionic/molecular compounds with P_{14} units

The $^{31}P\{^1H\}$ NMR spectrum of **1** shows four signals at 50.9, 9.1, 2.4 and -42.6 ppm corresponding to the four different phosphorus positions within the molecule. Unfortunately, these signals are broad, independent of the temperature and solvent. So, the fine structure can be resolved only for the signal at 50.9 ppm, which shows a triplet structure and represents the symmetrically surrounded phosphorus atoms P4 and P4'.

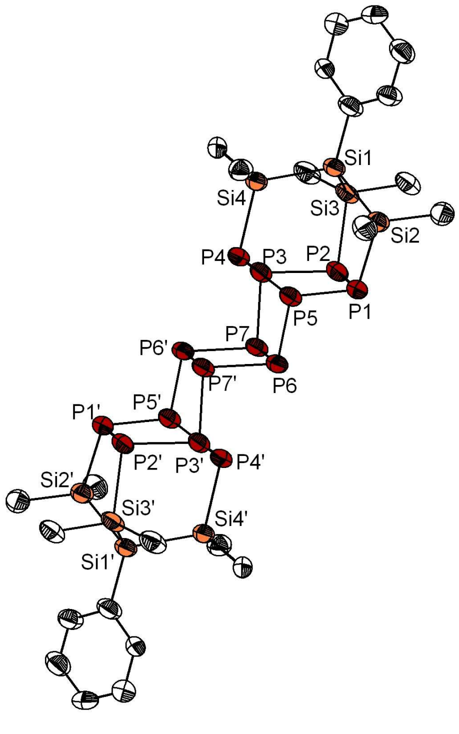
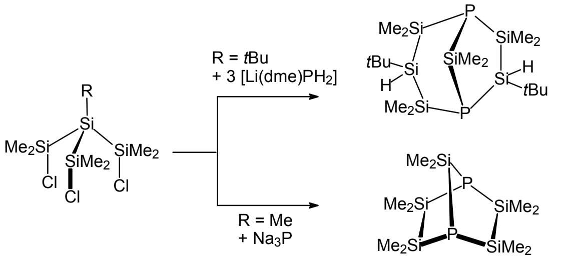


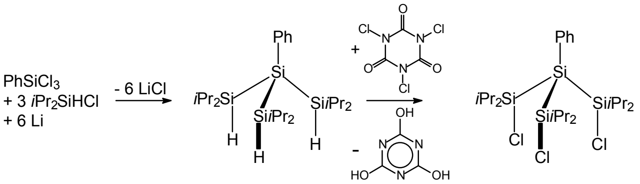
Fig. 1: Molecular structure of **1**; thermal ellipsoids represent a 50% probability level, hydrogen atoms are not shown, selected bond lengths (pm) and angles (°):Si1-Si2 233.5(4), Si1-Si3 232.9(5), Si1-Si4 234.24(4), Si2-P1 229.3(4), Si3-P2 229.9(4), Si4-P4 228.3(4), P1-P2 225.4(4), P1-P5 221.0(4), P2-P3 221.65(4), P3-P4 217.6(4), P4-P5 219.9(4), P5-P6 218.6(4), P6-P7 229.4(4), P6-P7' 224.0(4); Si2-Si1-Si3 98.88(16), Si2-Si1-Si4 107.90(14), Si3-Si1-Si4 106.70(15), P1-Si2-Si1 102.70(15), P2-Si3-Si1 102.57(15), P4-Si4-Si1 110.70(16).

Some years ago, several groups reported on their attempts to synthesise a 1-phospha-2,3,4,5-tetrasila[1.1.1]pentane.[8] These experiments, however, always lead to the formation of other bicyclic silylphosphines as shown in scheme 3.



Scheme 2: Reported attempts to synthesise a 1-phospha-2,3,4,5-tetrasila[1.1.1]pentane and the obtained products[8]

In order to avoid decomposition of the Si_4 substructure, we synthesised the sterically more demanding substituted branched trichlorotetrasilane $PhSi(Si\textit{i}Pr_2Cl)_3$ (**2**) as starting compound. **2** was obtained in a two-step synthesis by a reductive coupling of $PhSiCl_3$ and $\textit{i}Pr_2SiHCl$ with elementary lithium and subsequent chlorination with trichloroisocyanuric acid (TCCA).[9]



Scheme 3: Synthesis pathway of compound **2**

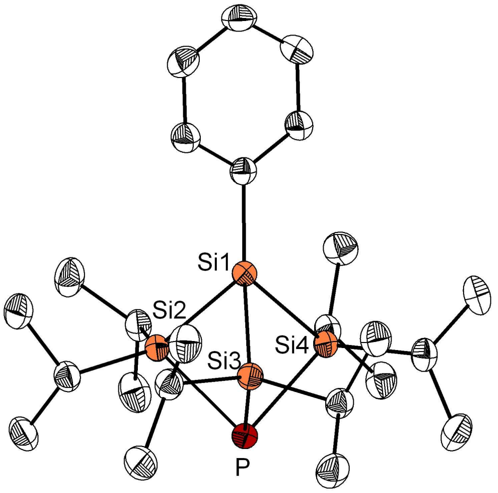


Fig. 2: Molecular structure of **3**; thermal ellipsoids represent a 50% probability level, hydrogen atoms are not shown, selected bond lengths (pm) and angles (°): Si1-Si2 235.46(8), Si1-Si3 235.01(14), Si1-Si4 233.61(12), Si2-P 230.05(14), Si3-P 232.56(9), Si4-P 231.60(11); Si-Si1-Si 81.61(4)-82.52(4), Si1-Si-P 80.19(4)-80.68(4), Si-P-Si 83.30(4)-83.60(4).

After work-up, the reaction of **2** with Li_3P in THF provides the target molecule as a white crystalline solid in small but reproducible yields. Other products could not be characterised to date. The crystal structure analysis shows the strained molecular structure with endocyclic bond angles all below 90°: Si-Si-Si: 81.6 – 82.5°, Si-Si-P: 80.2 – 80.6° and Si-P-Si: 83.3 – 83.6°. The bond length in compound **3,** however, are only slightly longer than the usual single bond between these elements. Worth mentioning are the short distances between the atoms Si2, Si3 and Si4 (307.7(9) – 309.0(12) pm) and between Si1 and P (301.2(10) pm), which are significantly shorter than the sum of the van der Waals radii.

The ^{31}P NMR spectrum of compound **3** shows a typical upfield shift for silylphosphines with -241.7 ppm. In the $^{29}Si\{^1H\}$ NMR, two doublet signals can be observed at 15.4 and -58.2 ppm. The signal at 15.4 ppm corresponds to the $Si\textit{i}Pr_2$ groups and shows a remarkably large $^1J_{Si,P}$ coupling constant of 53.2 Hz. This large coupling constant suggests a high *s*-orbital contribution to the Si-P σ-bonds and matches the results of DFT calculations (see supplementary information), which show a high *p*-orbital character of the phosphorus lone pair and a symmetric bonding orbital with significant contributions of Si1-Si4 and P atomic *s*-orbitals. For comparison, the similar substituted but planar compound $P(Si\textit{i}Pr_3)_3$ shows a $^1J_{Si,P}$ coupling of only 9 Hz.[10]

Acknowledgement

This work was financially supported by the Deutsche Forschungsgemeinschaft (DFG). The authors gratefully acknowledge financial support from Evonik Industries AG, Creavis.

Experimental Section

All working procedures were conducted under rigorous exclusion of oxygen and moisture using a Schlenk line and argon atmosphere. Solvents were dried and freshly distilled before use. NMR spectra were recorded with BRUKER AVANCE 300 or with BRUKER DRX 400. The structural analysis was carried out with appropriate single crystals on an automatic diffractometer (STOE-IPDS-2T, STOE-IPDS-2 or BRUKER D8-Quest). The structures were solved and refined with SHELXS-97[11] and SHELXL-2013[12]. The presentation of crystal structures was effected by DIAMOND3.2. IR vibrational spectra were gathered with the BRUKER ALPHA ATR-FT-IR. The starting materials $PhSi(SiMe_2Cl)_3$[4], $P_7(SiMe_3)_3$[2], and Li_3P[13] were prepared by reported methods.

Crystal data for **1**: $C_{24}H_{46}Si_8P_{14}\cdot 1.5\ C_7H_8$, 100 K, triclinic, P-1, a = 931.0(2), b = 1656.6(4), c = 1880.4(4) pm, α = 88.880(19)°, β = 87.593(19)°, γ = 77.702(18)°, V = 2830.9(11) Å^3, Z = 2, density = 1.327 g cm^3, μ = 0.619 mm^{-1}, F(000) = 1174, GOF = 0.742, Theta range: 1.26 – 24.00°, 12809 reflections collected, 8177 unique (R_{int} = 0.1002). *R*1 (w*R*2) = 0.0716 (0.1586) for 530 parameters and 3183 reflections with $I > 2\sigma(I)$.

Crystal data for **3**: $C_{24}H_{47}Si_4P_1$, 100 K, triclinic, P-1, a = 916.4(3), b = 1020.7(3), c = 1644.1(9) pm, α = 92.88(4)°, β = 94.84(4)°, γ = 110.34(2)°, V = 1431.7(10) Å^3, Z = 2, density = 1.111 g cm^3, μ = 0.273 mm^{-1}, F(000) = 524, GOF = 1.036, Theta range: 1.25 – 25.00°, 9353 reflections collected, 4731 unique (R_{int} = 0.0205). *R*1 (w*R*2) = 0.0247 (0.0657) for 262 parameters and 4266 reflections with $I > 2\sigma(I)$.

1: $PhSi(SiMe_2Cl)_3$ (0.42 g, 1.1 mmol) in dme (20 mL) was added dropwise to a solution of $P_7(SiMe_3)_3$ (0.48 g, 1.1 mmol) in dme (20 mL) at -45 °C. The reaction mixture was allowed to warm up within 4 h to ambient temperature, the solvent was removed under reduced pressure and the residue dissolved in 5 mL toluene. Subsequently, after 2 days yellow crystals of $[\{PhSi(SiMe_2)_3\}_2P_{14}]\cdot 1.5$ tol were obtained at 20 °C as yellow crystals in a yield of 53 % (0.29 g). **Elemental analysis** (%): calc. for $C_{24}H_{46}Si_8P_{14}$: C 29.03, H 4.67. found: C 29.09 , H 5.07
^{1}H-NMR (thd−d$_8$): δ/ ppm = 0.35 (m, C**H**$_3$, 12H), 0.78 (m, C**H**$_3$, 12H), 0.92 (m, C**H**$_3$, 12H), 7.28 (m, C_6**H**$_5$, 6H), 7.39 (m, C_6**H**$_5$, 4H). **^{13}C{^{1}H}-NMR** (thd−d$_8$): δ/ ppm = 2.96 (m, **C**H$_3$), 3.68 (m, **C**H$_3$), 128.24 (s, **C**$_6$H$_5$), 128.66 (s, **C**$_6$H$_5$), 129.11 (s, **C**$_6$H$_5$), 136.56 (s, **C**$_6$H$_5$). **^{29}Si{^{1}H}-NMR** (thd−d$_8$): δ/ ppm = -4.3 (m, **Si**(CH$_3$)$_2$), -5.8 (m, **Si**(CH$_3$)$_2$), -77.5 (s, **Si**C$_6$H$_5$). **^{31}P-NMR** (thd−d$_8$):δ/ppm=51.0(t,$^1J_{PP}$ = 328.1 Hz), 9.1 (m), 2.4(m), -42.6.(m). **MS**(ESI+) m/z (%) calcd.: 992.8153 [M$^+$+H]. found: 992.8265 (45).

$PhSi(Si\textit{i}Pr_2H)_3$: A solution of $PhSiCl_3$ (12.63 g, 0.06 mol) and $\textit{i}Pr_2HSiCl$

(44.96 g, 0.29 mol) in thf (250 mL) was slowly dropped at ambient temperature to a vigorously stirred suspension of Li cuts (3.32 g, 0.48 mol) in thf (250 mL) over 3 h, and stirring was continued for 24 h. The suspension was poured into a mixture of ice and HCl (200 mL, 1 M) and *n*-pentane (100 mL) was added. The organic phase was separated, the aqueous layer was extracted twice with *n*-pentane (100 mL) and the combined organic phases were dried with $MgSO_4$ and filtered. After evaporation of the solvents, the raw product was distilled fractionally under vacuum to afford $PhSi(Si\textit{i}Pr_2H)_3$ (10^{-2} mbar, 120 °C) with a yield of 62.8% (16.9 g).

^{1}H-NMR (C_6D_6): δ/ppm = 1.18 (d, $^3J_{HH}$ = 7.4 Hz, $CH(C\boldsymbol{H}_3)_2$, 18H), 1.20 (d, $^3J_{HH}$ = 7.4 Hz, $CH(C\boldsymbol{H}_3)_2$, 18H), 1.41 (sep, $^3J_{HH}$ = 7.4 Hz, $C\boldsymbol{H}(CH_3)_2$, 3H), 1.42 (sep, $^3J_{HH}$ = 7.4 Hz, $C\boldsymbol{H}(CH_3)_2$, 3H), 4.18 (t, $^1J_{SiH}$ = 2.7 Hz, $Si\boldsymbol{H}$, 3H), 7.11 (m, $C_6\boldsymbol{H}_5$, 3H), 7.80 (m, $C_6\boldsymbol{H}_5$, 2H). **^{13}C{^{1}H}-NMR** (C_6D_6): δ/ ppm = 13.96 (s, $\boldsymbol{C}H(CH_3)_2$), 21.40 (s, $CH(\boldsymbol{C}H_3)_2$), 22.98, (s, $CH(\boldsymbol{C}H_3)_2$), 128.51 (s, $\boldsymbol{C}_6H_5$), 129.29 (s, $\boldsymbol{C}_6H_5$), 135.52 (s, $\boldsymbol{C}_6H_5$), 138.42 (s, $\boldsymbol{C}_6H_5$). **^{29}Si-NMR** (C_6D_6): δ/ppm = -6.4 (d, m, $^1J_{SiH}$ = 172.3 Hz, $\boldsymbol{Si}CH(CH_3)_2$), -81,2 (s, $\boldsymbol{Si}C_6H_5$). **MS**(APCI+) m/z (%) calcd. : 449.2906 [M^+-H], found : 449.2903 (15); **IR** [cm^{-1}]: 463(w), 584(w), 650(m), 698(s), 744(vs), 877(m), 917(m), 1003(m), 1067(m), 1233(vw), 1363(w), 1383(w), 1427(w), 1460(m), 2073(m, Si-H)), 2861(m), 2940(m).

2: A solution of $PhSi(Si\textit{i}Pr_2H)_3$ (16.9 g, 0.038 mol) in thf (250 mL) was cooled down to -20 °C. Afterwards, TCCA (10 g, 0.043 mol) was slowly added and the solution was warmed up to ambient temperature. The solvent was removed under reduced pressure and the residue was dissolved in *n*-pentane (60 mL). The insoluble white precipitate was filtrated and washed two times with *n*-pentane (25 mL). The volume of the combined filtrates was reduced to 50 mL. After 4 days at -8 °C, colourless crystals of $PhSi(Si\textit{i}Pr_2Cl)_3$ were obtained, the yield being 71.0% (14.9 g). **Elemental analysis** (%): calc. for $C_{24}H_{47}Si_4Cl_3$: C 52.00, H 8.55, found C 52.01, H 8.97.

^{1}H-NMR (C_6D_6): δ/ppm = 1.13 (d, $^3J_{HH}$ = 7.4 Hz, $CH(C\boldsymbol{H}_3)_2$, 18H), 1.24 (d, $^3J_{HH}$ = 7.4 Hz, $CH(C\boldsymbol{H}_3)_2$, 18H), 1.70 (sep, $^3J_{HH}$ = 7.4 Hz, $C\boldsymbol{H}(CH_3)_2$, 6H), 7.10 (m, $C_6\boldsymbol{H}_5$, 3H), 8.14 (m, $C_6\boldsymbol{H}_5$, 2H). **^{13}C{^{1}H}-NMR** (C_6D_6): δ/ppm = 19.08 (s, $\boldsymbol{C}H(CH_3)_2$), 19.36 (s, $CH(\boldsymbol{C}H_3)_2$), 20.14 (s, $CH(\boldsymbol{C}H_3)_2$), 129.06 (s, $\boldsymbol{C}_6H_5$), 129.32 (s, $\boldsymbol{C}_6H_5$), 132.31 (s, $\boldsymbol{C}_6H_5$), 138.85 (s, $\boldsymbol{C}_6H_5$). **^{29}Si{^{1}H}-NMR** (C_6D_6): δ/ ppm = 33.4 (s, $\boldsymbol{Si}CH(CH_3)_2$), -70.6 (s, $\boldsymbol{Si}C_6H_5$). **MS**(APCI+) m/z (%) ber.: 517.2126 [M^+-Cl], gef. : 517.2126 [M^+-Cl] (100); **IR** [cm^{-1}]: 424(w), 463(s), 553(vs), 599(s), 621(m), 655(m), 700(m), 735(m), 760(w), 875(s), 991(m), 1233(vw), 1365(w), 1427(w), 1461(m), 2867(m), 2948(m).

3: $PhSi(Si\textit{i}Pr_2Cl)_3$ (1.34 g, 2.42 mmol) in thf (10 mL) was added to a suspension of Li_3P (0.13 g, 2.50 mmol) in thf (100 mL) at -30°C. Subsequently, the reaction mixture was first warmed-up to room temperature and stirred for 16 hours and then heated to reflux for 4 days. Thereafter, the solvent was removed under reduced pressure, and the residue was dissolved in *n*-pentane (25 mL). After filtration, the volume of the solution was reduced to 2 mL and cooled down to -30 °C. Compound **3** was obtained as colourless crystals within 4 days in a yield of 8.8 % (0.10 g). **Elemental analysis** (%): calc. for $C_{24}H_{48}Si_4P$: C 60.19, H 9.89. found: C 60.24, H 10.03.

^{1}H-NMR (C_6D_6): δ/ppm = 1.36 (d, $^3J_{HH}$ = 7.5 Hz, $CH(C\boldsymbol{H}_3)_2$, 18H), 1.38(d, $^3J_{HH}$ =7.5 Hz, $CH(C\boldsymbol{H}_3)_2$, 18H), 1.71 (sep, $^3J_{HH}$ = 7.5 Hz, $C\boldsymbol{H}(CH_3)_2$), 6H), 7.09 (m, $C_6\boldsymbol{H}_5$, 3H), 7.70 (m, $C_6\boldsymbol{H}_5$, 2H). **^{13}C{^{1}H}-NMR** (C_6D_6): δ/ppm = 18.91 (d, $^2J_{CP}$ = 5.2 Hz, $\boldsymbol{C}H(CH_3)_2$), 20.92 (d, $^2J_{CP}$ = 1.1 Hz, $CH(\boldsymbol{C}H_3)_2$), 21.36 (d, $^3J_{CP}$ = 1.5 Hz, $CH(\boldsymbol{C}H_3)_2$), 128.67 (s, $\boldsymbol{C}_6H_5$), 129.22 (s, $\boldsymbol{C}_6H_5$), 133.40 (s, $\boldsymbol{C}_6H_5$), 138.25 (s, $\boldsymbol{C}_6H_5$). **^{29}Si{^{1}H}-NMR** (C_6D_6): δ/ppm = 15.4 (d, $^1J_{SiP}$ = 53.2 Hz, $\boldsymbol{Si}_3P$), -58.2 (d, $^2J_{SiP}$ = 8.5 Hz, $\boldsymbol{Si}C_6H_5$). **^{31}P-NMR**(C_6D_6): δ/ppm = -241.7 (s). **MS**(APCI-) m/z(%) calc.: 479.2565 [M+H]. found: 479.2560 [M+H] (30);

Notes and references

*[a] Fachbereich Chemie,
Hans-Meerwein-Straße, D-35032 Marburg, Germany and Wissenschaftliches Zentrum für Materialwissenschaften (WZMW) der Philipps-Universität Marburg.
E-mail:haenisch@chemie.uni-marburg.de*

*†Electronic Supplementary Information (ESI) available: DFT calculations, Crystallographic data of compound **2**, Proposed mechanism for the formation of compound **1** and ^{31}PNMR spectrum of compound **1**. For crystallographic data see also: CCDC 978105 (**1**), 978107 (**2**) and 978106 (**3**). For ESI and in CIF See DOI: 10.1039/b000000x/*

[1] a) T. Arnold, H. Braunschweig, J. O. C. Jimenez-Halla, K. Radacki, S. S. Sen, *Chem. Eur. J.* **2013**, *19*, 9114-9117; b) R. Rodriguez, T. Troadec, T. Kato, N. Saffon-Merceron, J.-M. Sotiropoulos, A. Baceiredo, *Angew. Chem. Int. Ed.* **2012**, *51*, 7158-7161; c) S. Khan, R. Michel, S. S. Sen, H. W. Roesky, D. Stalke, *Angew. Chem. Int. Ed.* **2011**, *50*, 11786-11789; d) R. Rodriguez, D. Gau, Y. Contie, T. Kato, N. Saffon-Merceron, A. Baceiredo, *Angew. Chem. Int. Ed.* **2011**, *50*, 11492-11495; e) G. Fritz, P. Scheer, *Chem. Rev.* **2000**, *100*, 3341-3401.

[2] a) W. Hönle, G. H. von Schnering, *Z. Anorg. Allg. Chem.* **1978**, *440*, 171-182; b) G. Fritz, W. Hölderich, *Naturwissenschaften* **1975**, *62*, 573-575; c) H. Siegl, W. Krumlacher, K. Hassler, *Silicon Chemistry*, **1999**, 139-145.

[3] a) A. Kracke, C. von Hänisch, *Eur. J. Inorg. Chem.* **2011**, 3374-3380; b) P. Kopecky, C. von Hänisch, F. Weigend, A. Kracke, *Eur. J. Inorg. Chem.* **2010**, 258-265; c) S. Traut, C. von Hänisch, H.-J. Kathagen, *Eur. J. Inorg. Chem.* **2009**, 777-783; d) C. von Hänisch, S. Traut, S. Stahl, *Z. Anorg. Allg. Chem.* **2007**, *633*, 2199-2204; e) C. von Hänisch, E. Matern, *Z. Anorg. Allg. Chem.* **2005**, *631*, 1655-1659.

[4] C. von Hänisch, M. Feierabend, *Z. Anorg. Allg. Chem.* **2013**, *639*, 788-793.

[5] a) V. Miluykov, A. Kataev, O. Sinyashin, P. Lönnecke, E. Hey-Hawkins, *Z. Anorg. Allg. Chem.* **2006**, *632*, 1728-1732; b) N. Korber, *Phosphorus, Sulfer and Silicon*, **1997**, *124*, 339-346.

[6] M. Baudler, H. Jachow, B. Lieser, K.-F. Tebbe, M. Fehér, *Angew. Chem.* **1989**, *101*, 1245-1247.

[7] M. H. Möller, W. Jeitschko, *J. Solid State Chem.* **1986**, *65*, 178-189.

[8] a) M. Driess, M. Reisgys, H. Pritzkow, *Z. Anorg. Allg. Chem.* **1998**, *624*, 1886-1890; b) G. M. Kollegger, U. Katzenbeisser, K. Hassler, C. Krüger, D. Brauer, R. Gielen, *J. Organomet. Chem.* **1997**, *543*, 103-110.

[9] S. Varaprath, D. H. Stutts, *J. Organomet. Chem.* **2007**, *692*, 1892-1897.

[10] a) C. von Hänisch, Z. Anorg. Allg. Chem. **2001**, *627*, 1414-1416; b) M. Driess, C. Monsé, *Z. Anorg. Allg. Chem.* **2000**, *626*, 2264-2268.

[11] G. M. Sheldrick, *Acta Crystallogr., Sect. A* **2008**, *64*, 112–122.

[12] G. M. Sheldrick, *SHELXL*, University of Göttingen, Germany, **2013**.

[13] G. Fritz, R. Blastoch, *Z. Anorg. Allg. Chem.* **1986**, *535*, 63-85.