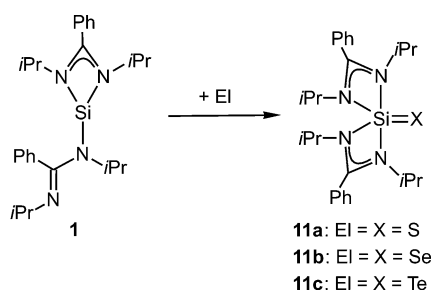


Stable Five-Coordinate Silicon(IV) Complexes with SiN₄X Skeletons (X = S, Se, Te) and Si=X Double Bonds

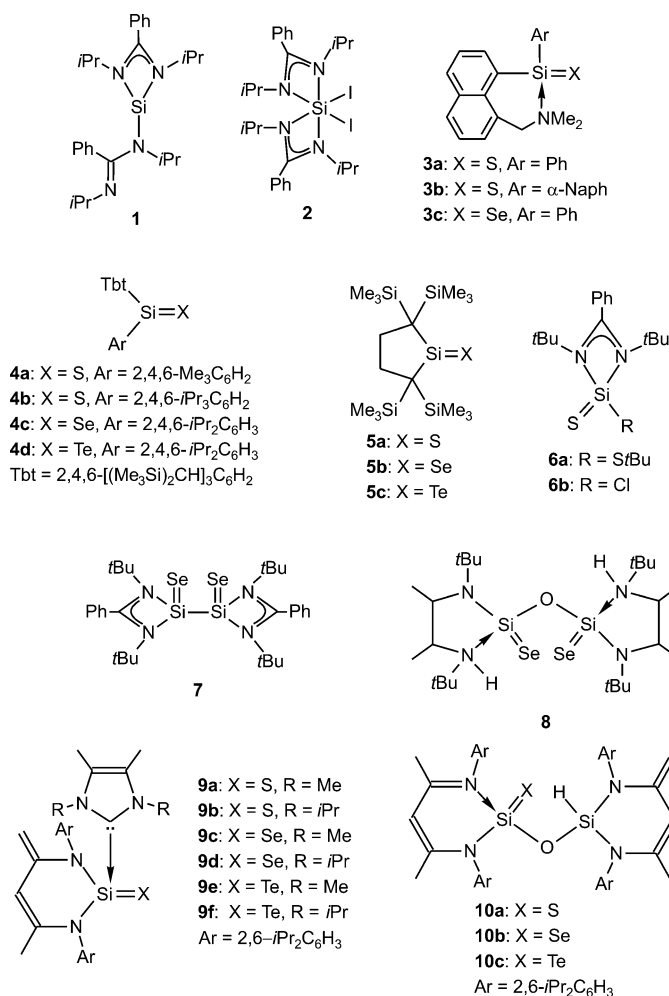
Konstantin Junold, Johannes A. Baus, Christian Burschka, Dominic Auerhammer, and Reinhold Tacke*^[a]

Recently, we have reported the synthesis of the three-coordinate silicon(II) compound **1**, a donor-stabilized thermally stable silylene with one bidentate and one monodentate amidinato ligand.^[1] Treatment of **1** with iodine resulted in an oxidative addition to give the six-coordinate silicon(IV) complex **2** (SiN₄I₂ skeleton); that is, in the formation of **2**, both amidinato ligands of **1** act as bidentate ligands ($\lambda^3\text{Si}^{\text{II}} \rightarrow \lambda^6\text{Si}^{\text{IV}}$).^[1] This result prompted us to study an analogous reaction of **1** with the chalcogens sulfur, selenium, or tellurium, which should potentially afford five-coordinate silicon(IV) complexes with SiN₄X skeletons (X = S, Se, Te) and Si=X double bonds. A series of thermally stable silicon(IV) compounds with Si=X double bonds have already been reported in the literature,^[2–11] such as **3a–c**,^[3] **4a–d**,^[4] **5a–c**,^[5] **6a**,^[6] **6b**,^[7] **7**,^[8] **9a–f**,^[9] and **10a–c**.^[10] However, all these compounds represent three- or four-coordinate silicon(IV) compounds, whereas the reaction of **1** with sulfur, selenium, or tellurium was expected to lead to a new type of five-coordinate silicon(IV) complexes with Si=X bonds.

Indeed, treatment of **1** with one molar equivalent of elemental sulfur, selenium, or tellurium in toluene at 20 °C resulted in the formation of compounds **11a–c**, which were isolated as crystalline solids (yield: **11a** 51%; **11b** 46%; **11c** 72%; Scheme 1).^[12] So far, attempts to prepare the corresponding oxygen analogue of **11a–c** failed. The identities of



Scheme 1. Syntheses of compounds **11a–c**.



11a–c were established by elemental analyses, NMR spectroscopic studies in the solid state and in solution,^[13] as well as crystal structure analyses.^[14] The crystal structure of **11c** was determined with the toluene solvate **11c**·0.7C₆H₅CH₃. The molecular structures of **11a–c** are depicted in Figures 1–3. The studies reported herein were performed in continuation of our systematic investigations on higher-coordinate silicon(IV) and silicon(II) compounds.^[1,15]

The silicon coordination polyhedra of **11a** (two molecules in the asymmetric unit), **11b**, and **11c**·0.7C₆H₅CH₃ (disordered toluene molecules) are best described as strongly dis-

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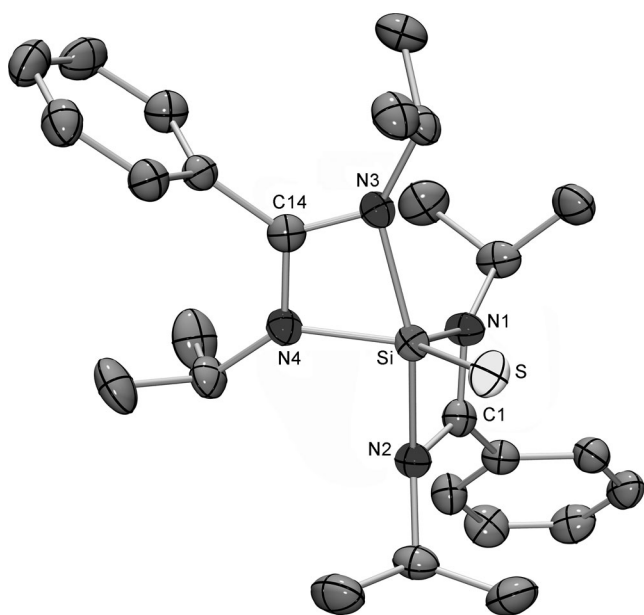


Figure 1. Molecular structure of **11a** (molecule 1) in the crystal (ellipsoids were set at 50% probability; hydrogen atoms were omitted for clarity). Selected bond lengths [Å] and angles [°]: Si–S 2.0193(9), Si–N1 1.8209(17), Si–N2 1.9611(16), Si–N3 1.9562(16), Si–N4 1.8148(16), N1–C1 1.347(2), N2–C1 1.318(2), N3–C14 1.309(2), N4–C14 1.359(2); S–Si–N1 123.43(6), S–Si–N2 103.55(6), S–Si–N3 100.32(6), S–Si–N4 123.90(6), N1–Si–N2 69.07(7), N1–Si–N3 98.99(7), N1–Si–N4 112.67(8), N2–Si–N3 156.07(7), N2–Si–N4 95.91(7), N3–Si–N4 68.88(7), N1–C1–N2 107.38(16), N3–C14–N4 106.33(15); molecule 2: Si–S 2.0229(9), Si–N1 1.8358(16), Si–N2 1.9501(15), Si–N3 1.9682(15), Si–N4 1.8231(16), N1–C1 1.351(2), N2–C1 1.310(2), N3–C14 1.314(2), N4–C14 1.345(2); S–Si–N1 123.66(6), S–Si–N2 99.65(5), S–Si–N3 102.02(6), S–Si–N4 125.01(6), N1–Si–N2 68.83(7), N1–Si–N3 99.46(7), N1–Si–N4 111.30(8), N2–Si–N3 158.31(7), N2–Si–N4 97.71(7), N3–Si–N4 68.79(7), N1–C1–N2 107.16(15), N3–C14–N4 107.57(15).

torted trigonal bipyramids, with one nitrogen atom of each of the two amidinato ligands in the axial positions (N_{ax} –Si– N_{ax} 156.07(7) and 158.31(7) (**11a**), 157.77(8) (**11b**), 157.29(7)° (**11c**); sum of the equatorial bond angles 360.0° (**11a–c**)). The Berry distortions (transition trigonal bipyramid → square pyramid) amount to 18.9/14.8% (**11a**), 17.5% (**11b**), and 15.3% (**11c**).^[16] The equatorial Si–N bond lengths (1.813(2)–1.8358(16) Å) are significantly shorter than the axial Si–N distances (1.9327(17)–1.9682(15) Å), and the Si–X bond lengths (X=S, Se, Te) amount to 2.0193(9) and 2.0229(9) (**11a**), 2.1632(7) (**11b**), and 2.4017(6) Å (**11c**). These Si–X bond lengths are slightly longer than those of the three- and four-coordinate silicon(IV) compounds with Si=X double bonds described in the literature. For comparison, the reported Si–X bond lengths for Si=X double bonds are in the ranges 1.948(4)–2.013(3) (Si=S), 2.0963(5)–2.156(1) (Si=Se), and 2.3210(6)–2.383(2) Å (Si=Te).^[3–10] This slight increase is in line with the change of the silicon coordination number from three or four to five. However, the Si–X bond lengths of **11a–c** are significantly shorter than those reported for the respective equatorial Si–X distances of five-coordinate silicon(IV)

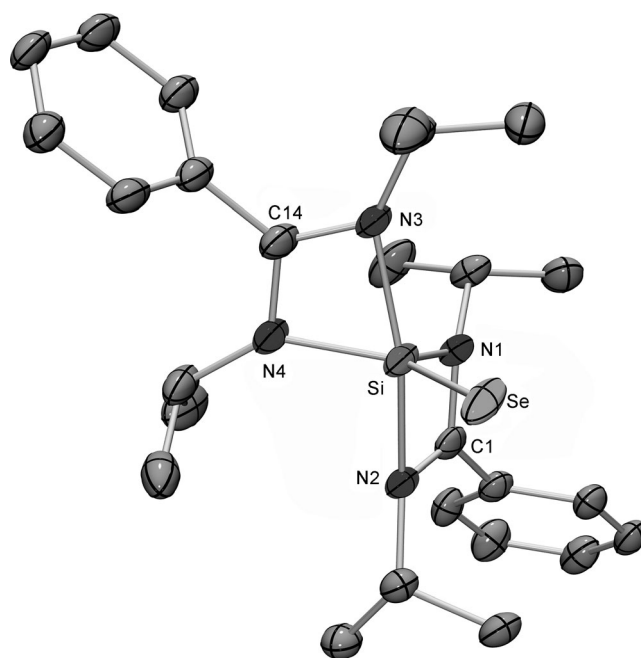


Figure 2. Molecular structure of **11b** in the crystal (ellipsoids were set at 50% probability; hydrogen atoms were omitted for clarity). Selected bond lengths [Å] and angles [°]: Si–Se 2.1632(7), Si–N1 1.8153(18), Si–N2 1.9534(18), Si–N3 1.9550(17), Si–N4 1.813(2), N1–C1 1.338(3), N2–C1 1.314(3), N3–C14 1.306(3), N4–C14 1.356(3); Se–Si–N1 123.73(7), Se–Si–N2 102.72(6), Se–Si–N3 99.50(5), Se–Si–N4 123.19(6), N1–Si–N2 68.96(8), N1–Si–N3 98.92(8), N1–Si–N4 113.07(9), N2–Si–N3 157.77(8), N2–Si–N4 97.99(9), N3–Si–N4 68.90(8), N1–C1–N2 107.32(18), N3–C14–N4 106.64(19).

compounds with Si–X single bonds (Si–S 2.1251(7)–2.2087(8); Si–Se 2.2846(4)–2.3449(7); Si–Te 2.5184(4)–2.5608(12) Å),^[17] indicating a considerable double bond character of the silicon–chalcogen bonds in **11a–c**. The N–C bond lengths in the amidinato ligands of **11a–c** (1.306(3)–1.359(2) Å) are very similar, which can be explained by a high degree of electron delocalization within the NCN skeletons ($N_{ax}=CPh-N_{eq} \leftrightarrow N_{ax}-CPh=N_{eq}$). However, it is interesting to note that the N_{ax} –C distances (1.306(3)–1.318(2) Å) are slightly shorter than the N_{eq} –C bond lengths (1.338(2)–1.359(2) Å), indicating a small preference of the resonance structure $N_{ax}=CPh-N_{eq}$.

The isotropic ²⁹Si chemical shifts of **11a** ($\delta = -74.7$ (solid state)/ -70.7 ppm (solution)), **11b** ($\delta = -85.0/-80.8$ ppm), and **11c** ($\delta = -111.4/-116.5$ ppm) and the isotropic ⁷⁷Se and ¹²⁵Te chemical shifts of **11b** ($\delta = -485.3/-495.7$ ppm) and **11c** ($\delta = -1208.8/-1199.2$ ppm), respectively, in the solid state and in solution (C_6D_6) are very similar, indicating that these three compounds exist also in solution. At 23 °C, the ¹H NMR spectra of **11a–c** showed only one doublet and one septet for the four $CH(CH_3)_2$ groups, and the ¹³C{¹H} NMR spectra revealed only two resonance signals for the four $CH(CH_3)_2$ groups, indicating a rapid exchange of the four nitrogen sites of the two bidentate amidinato ligands. This assumption was further supported by the ¹⁵N{¹H} NMR spectrum of **11b**, which showed only one resonance signal at

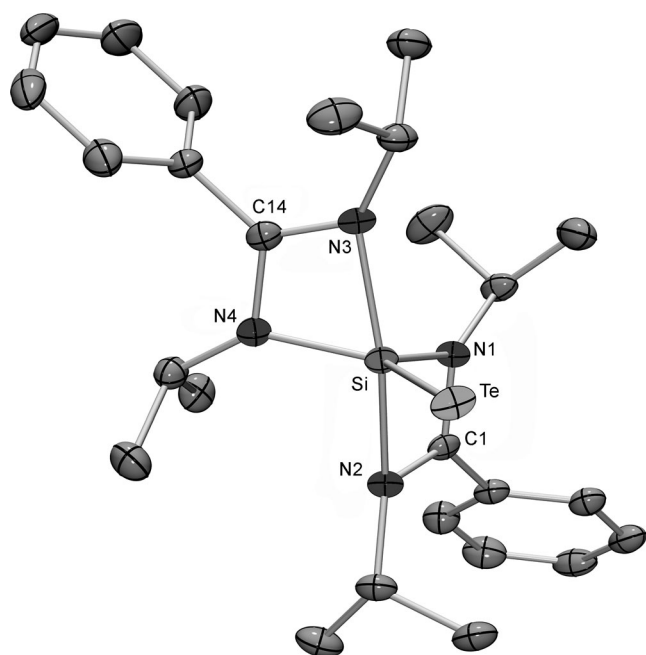


Figure 3. Molecular structure of **11c** in the crystal of **11c**·0.7 C₆H₅CH₃ (ellipsoids were set at 50% probability; hydrogen atoms were omitted for clarity). Selected bond lengths [Å] and angles [°]: Si–Te 2.4017(6), Si–N1 1.8326(18), Si–N2 1.9327(17), Si–N3 1.9474(17), Si–N4 1.8226(17), N1–C1 1.345(3), N2–C1 1.318(3), N3–C14 1.313(3), N4–C14 1.346(2); Te–Si–N1 126.42(6), Te–Si–N2 101.78(5), Te–Si–N3 100.93(5), Te–Si–N4 122.62(6), N1–Si–N2 69.30(7), N1–Si–N3 97.27(8), N1–Si–N4 110.96(8), N2–Si–N3 157.29(7), N2–Si–N4 97.86(8), N3–Si–N4 69.14(7), N1–C1–N2 107.15(17), N3–C14–N4 107.30(17).

$\delta = -208.9$ ppm (solid state: $\delta = -225.1$ (2N), -196.0 , -173.1 ppm).

In conclusion, starting from the easily accessible tri-coordinate silicon(II) compound **1**, the five-coordinate silicon(IV) complexes **11a–c** were synthesized by oxidative addition with sulfur, selenium, or tellurium (transformations of the type $\lambda^3\text{Si}^{\text{II}} \rightarrow \lambda^5\text{Si}^{\text{IV}}$). These compounds represent a new class of five-coordinate silicon(IV) species with SiN₄X skeletons (X = S, Se, Te) and thermally stable Si=X double bonds. Five-coordinate silicon(IV) compounds with silicon–chalcogen double bonds have not yet been reported. The synthesis of **11a–c** emphasizes once again the high synthetic potential of the donor-stabilized silylene **1**.^[1] Future studies have to evaluate the chemical reactivity of the Si=X double bonds of **11a–c** and the synthetic potential of these compounds for the chemistry of higher-coordinate silicon.

Experimental Section

The syntheses were carried out under a dry argon atmosphere in oven-dried glassware by using standard Schlenk techniques. The solvents were dried, purified, and deoxygenated according to standard procedures. The gray selenium (99.7%; ABCR, product no. AB118648, < 70 μ) and tellurium (99.8%; Acros Organics, product no. 31599, 200 mesh) were used as powders. Melting points were measured in sealed glass capillaries by using a Büchi Melting Point B-540 apparatus.

Compound 11a: Toluene (10 mL) was added at 20°C to a mixture of sulfur (37.0 mg, 144 μ mol of S₈) and **1** (500 mg, 1.15 mmol), and the reaction mixture was stirred at this temperature for 15 h. The resulting suspension was concentrated in vacuo to a volume of 2 mL and heated until a clear solution was obtained, which was then cooled slowly to -20°C and kept undisturbed at this temperature for two days. The resulting colorless crystalline solid was isolated by filtration, washed with *n*-pentane (2 $\times$ 5 mL), and dried in vacuo (20°C, 4 h, 0.01 mbar). Yield: 272 mg (583 μ mol, 51%). M.p. 213°C (decomp); ¹H NMR (500.1 MHz, C₆D₆): $\delta = 1.43$ (d, ³J(¹H,¹H) = 6.8 Hz, 24H; CH₃), 3.93 (sept, ³J(¹H,¹H) = 6.8 Hz, 4H; CH₃CHCH₃), 7.08–7.17 and 7.19–7.28 ppm (m, 10H; C₆H₅); ¹³C{¹H} NMR (125.8 MHz, C₆D₆): $\delta = 24.1$ (8C; CH₃), 47.2 (4C; CH₃CHCH₃), 128.1 (4C; *o*-C₆H₅), 128.5 (4C; *m*-C₆H₅), 130.2 (2C; *p*-C₆H₅), 131.9 (2C; *i*-C₆H₅), 172.1 ppm (2C; NCN); ²⁹Si{¹H} NMR (99.4 MHz, C₆D₆): $\delta = -70.7$ ppm; ¹⁵N VACP/MAS NMR (40.6 MHz): $\delta = -224.4$, -222.6 , -196.0 , -174.9 ppm; ²⁹Si VACP/MAS NMR (79.5 MHz): $\delta = -74.7$ ppm (br); elemental analysis (%) calcd for C₂₆H₃₈N₄SSi (466.77): C 66.90, H 8.21, N 12.00, S 6.87; found: C 66.4, H 8.1, N 12.3, S 6.9.

Compound 11b: Toluene (10 mL) was added at 20°C to a mixture of gray selenium (54.5 mg, 690 μ mol) and **1** (300 mg, 690 μ mol), and the reaction mixture was stirred at this temperature for 20 h. The insoluble components were filtered off and discarded, and the filtrate was concentrated in vacuo to a volume of 1 mL. The resulting suspension was heated until a clear solution was obtained, which was then cooled slowly to -20°C and kept undisturbed at this temperature for two days. The resulting yellow crystalline solid was isolated by filtration, washed with *n*-pentane (2 $\times$ 3 mL), and dried in vacuo (20°C, 5 h, 0.01 mbar). Yield: 162 mg (315 μ mol, 46%). M.p. 196°C (decomp); ¹H NMR (500.1 MHz, C₆D₆): $\delta = 1.42$ (d, ³J(¹H,¹H) = 6.8 Hz, 24H; CH₃), 4.00 (sept, ³J(¹H,¹H) = 6.8 Hz, 4H; CH₃CHCH₃), 7.10–7.18 and 7.20–7.26 ppm (m, 10H; C₆H₅); ¹³C{¹H} NMR (125.8 MHz, C₆D₆): $\delta = 24.1$ (8C; CH₃), 47.0 (4C; CH₃CHCH₃), 128.1 (4C; *o*-C₆H₅), 128.5 (4C; *m*-C₆H₅), 130.2 (2C; *p*-C₆H₅), 131.8 (2C; *i*-C₆H₅), 171.7 ppm (2C; NCN); ¹⁵N{¹H} NMR (50.7 MHz, C₆D₆): $\delta = -208.9$ ppm; ²⁹Si{¹H} NMR (99.4 MHz, C₆D₆): $\delta = -80.8$ ppm (⁷⁷Se satellites, ¹J(²⁹Si,⁷⁷Se) = 291.9 Hz); ⁷⁷Se{¹H} NMR (95.4 MHz, C₆D₆): $\delta = -495.7$ ppm; ¹⁵N VACP/MAS NMR (40.6 MHz): $\delta = -225.1$ (2N), -196.0 , -173.1 ppm; ²⁹Si VACP/MAS NMR (79.5 MHz): $\delta = -85.0$ ppm (⁷⁷Se satellites, ¹J(²⁹Si,⁷⁷Se) = 268 Hz); ⁷⁷Se VACP/MAS NMR (76.3 MHz): $\delta = -485.3$; elemental analysis (%) calcd for C₂₆H₃₈N₄SeSi (513.66): C 60.80, H 7.46, N 10.91; found: C 60.5, H 7.5, N 10.9.

Compound 11c: Toluene (15 mL) was added at 20°C to a mixture of tellurium (147 mg, 1.15 mmol) and **1** (500 mg, 1.15 mmol), and the reaction mixture was stirred at this temperature for 16 h. The insoluble components were filtered off and discarded, and the filtrate was concentrated in vacuo to a volume of 3 mL. The resulting suspension was heated until a clear solution was obtained, which was then cooled slowly to -20°C and kept undisturbed at this temperature for one day. The resulting yellow crystalline solid was isolated by filtration, washed with *n*-pentane (2 $\times$ 3 mL), and dried in vacuo (20°C, 5 h, 0.01 mbar). Yield: 468 mg (832 μ mol, 72%). M.p. 242°C (decomp); ¹H NMR (500.1 MHz, C₆D₆): $\delta = 1.39$ (d, ³J(¹H,¹H) = 6.8 Hz, 24H; CH₃), 4.04 (sept, ³J(¹H,¹H) = 6.8 Hz, 4H; CH₃CHCH₃), 7.10–7.18 and 7.19–7.22 ppm (m, 10H; C₆H₅); ¹³C{¹H} NMR (125.8 MHz, C₆D₆): $\delta = 24.4$ (8C; CH₃), 46.8 (4C; CH₃CHCH₃), 128.0 (4C; *o*-C₆H₅; overlapping with the solvent signal), 128.6 (4C; *m*-C₆H₅), 130.3 (2C; *p*-C₆H₅), 131.8 (2C; *i*-C₆H₅), and 170.9 ppm (2C; NCN); ²⁹Si{¹H} NMR (99.4 MHz, C₆D₆): $\delta = -116.5$ ppm (¹²⁵Te satellites, ¹J(²⁹Si,¹²⁵Te) = 832 Hz); ¹²⁵Te{¹H} NMR (157.8 MHz, C₆D₆): $\delta = -1199.2$ ppm; ¹⁵N VACP/MAS NMR (40.6 MHz): $\delta = -224.8$, -222.5 , -186.7 , -184.9 ppm; ²⁹Si VACP/MAS NMR (79.5 MHz): $\delta = -111.4$ ppm (¹²⁵Te satellites, ¹J(²⁹Si,¹²⁵Te) = 809 Hz); ¹²⁵Te{¹H} HPDec/MAS NMR (126.2 MHz): $\delta = -1208.8$ ppm; elemental analysis (%) calcd for C₂₆H₃₈N₄SiTe (562.30): C 55.54, H 6.81, N 9.96; found: C 55.1, H 6.9, N 9.9.

Keywords: selenium • silicon compounds • silylenes • sulfur • tellurium

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- [12] Treatment of **1** with an excess of sulfur, selenium, or tellurium (molar ratio 1:2) resulted also in the formation of **11a–c**.
- [13] Solution ^1H , $^{13}\text{C}\{^1\text{H}\}$, $^{15}\text{N}\{^1\text{H}\}$, $^{29}\text{Si}\{^1\text{H}\}$, $^{77}\text{Se}\{^1\text{H}\}$, and $^{125}\text{Te}\{^1\text{H}\}$ NMR spectra were recorded at 23 °C on a Bruker Avance 500 NMR spectrometer (^1H 500.1 MHz; ^{13}C 125.8 MHz; ^{15}N 50.7 MHz; ^{29}Si 99.4 MHz; ^{77}Se 95.4 MHz; ^{125}Te 157.8 MHz). C_6D_6 was used as the solvent. Chemical shifts were determined relative to internal $[\text{D}_3]\text{C}_6\text{H}_6$ (^1H , $\delta = 7.28$ ppm), internal C_6D_6 (^{13}C , $\delta = 128.0$ ppm), external TMS (^{29}Si , $\delta = 0$ ppm), external formamide (90% w/w in DMSO ; ^{15}N , $\delta = -268.0$ ppm), external Me_2Se (5% w/w in C_6D_6 ; ^{77}Se , $\delta = 0$ ppm), or external Ph_2Te_2 (0.1 M in CDCl_3 ; ^{125}Te , $\delta = 422.0$ ppm). Assignment of the ^{13}C NMR data was supported by DEPT 135 and ^1H , ^{13}C correlation experiments. Solid-state ^{15}N , ^{29}Si , and ^{77}Se VACP/MAS NMR spectra and $^{125}\text{Te}\{^1\text{H}\}$ HPDec/MAS NMR spectra were recorded at 22 °C on a Bruker DSX 400 NMR spectrometer with bottom layer rotors of ZrO_2 (diameter 4 mm (**11a**) or 7 mm (**11b** and **c**) containing about 80 mg (4 mm) or 200 mg (7 mm) of sample (^{15}N 40.6 MHz; ^{29}Si 79.5 MHz; ^{77}Se 76.3 MHz; ^{125}Te 126.2 MHz; external standard TMS (^{13}C , ^{29}Si , $\delta = 0$ ppm), glycine (^{15}N , $\delta = -342.0$ ppm), Me_2Se (^{77}Se , $\delta = 0$ ppm), or $\text{Te}(\text{OH})_6$ (^{125}Te , $\delta = 685.5$ and 692.2 ppm); spinning rate 10 kHz (4 mm) or 7 kHz (7 mm); contact time 3 ms (^{15}N) or 5 ms (^{29}Si , ^{77}Se); 90° ^1H transmitter pulse length 2.6 μs (4 mm) or 3.6 μs (7 mm); repetitions time 4–7 s).
- [14] Crystal structure analyses of **11a**, **11b**, and **11c**-0.7 $\text{C}_6\text{H}_5\text{CH}_3$: Suitable single crystals were mounted in inert oil (perfluoropolyalkyl ether, ABCR) on a glass fiber and then transferred to the cold nitrogen gas stream of the diffractometer (**11a**: Stoe IPDS, graphite-monochromated $\text{MoK}\alpha$ radiation, $\lambda = 0.71073$ Å; **11b** and **11c**-0.7 $\text{C}_6\text{H}_5\text{CH}_3$: Bruker Nonius KAPPA APEX II, Montel mirror, $\text{MoK}\alpha$ radiation, $\lambda = 0.71073$ Å). The structures were solved by direct methods (SHELXS-97) and refined by full-matrix least-squares methods on F^2 for all unique reflections (SHELXL-97).^[18] For the CH hydrogen atoms, a riding model was employed. CCDC-888842 (**11a**), CCDC-888843 (**11b**), and CCDC-888844 (**11c**-0.7 $\text{C}_6\text{H}_5\text{CH}_3$) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Selected data for **11a**: single crystal of dimensions $0.7 \times 0.25 \times 0.25$ mm obtained by crystallization from toluene at -20 °C; $\text{C}_{26}\text{H}_{38}\text{N}_4\text{SSi}$; $M_r = 466.75$; analysis at 173(2) K; monoclinic; space group $P2_1/c$ (no. 14); $a = 17.987(3)$, $b = 16.210(3)$, $c = 19.063(3)$ Å; $\beta = 105.88(2)^\circ$; $V = 5346.2(16)$ Å³; $Z = 8$; $\rho_{\text{calcd}} = 1.160$ g cm⁻³; $\mu = 0.186$ mm⁻¹; $F(000) = 2016$; $2\theta_{\text{max}} = 52.04^\circ$; 57273 collected reflections; 10363 unique reflections ($R_{\text{int}} = 0.0689$); 593 parameters; $S = 0.991$; $R_1 = 0.0392$, [$I > 2\sigma(I)$]; wR_2 (all data) = 0.0863; max/min residual electron density = $+0.429/-0.273$ e Å⁻³. Selected data for **11b**: single crystal of dimensions $0.5 \times 0.4 \times 0.3$ mm obtained by crystallization from toluene at -20 °C; $\text{C}_{26}\text{H}_{38}\text{N}_4\text{SeSi}$; $M_r = 513.65$; analysis at 100(2) K; monoclinic; space group $P2_1/n$ (no. 14); $a = 11.1400(7)$, $b = 16.3972(11)$, $c = 14.7306(9)$ Å; $\beta = 92.894(2)^\circ$; $V = 2687.3(3)$ Å³; $Z = 4$; $\rho_{\text{calcd}} = 1.270$ g cm⁻³; $\mu = 1.462$ mm⁻¹; $F(000) = 1080$; $2\theta_{\text{max}} = 52.84^\circ$; 79882 collected reflections; 5518 unique reflections ($R_{\text{int}} = 0.0529$); 327 parameters; 25 restraints; $S = 1.046$; $R_1 = 0.0334$ [$I > 2\sigma(I)$]; wR_2 (all data) = 0.0734; max/min residual electron density = $+0.536/-0.984$ e Å⁻³. Selected data for **11c**-0.7 $\text{C}_6\text{H}_5\text{CH}_3$: single crystal of dimensions $0.5 \times 0.4 \times 0.3$ mm obtained by crystallization from toluene at -20 °C; $\text{C}_{26}\text{H}_{38}\text{N}_4\text{SiTe}$ -0.7 $\text{C}_6\text{H}_5\text{CH}_3$; $M_r = 626.97$; analysis at 100(2) K; monoclinic; space group $P2_1/n$ (no. 14); $a = 11.6736(8)$, $b = 16.2635(12)$, $c = 17.2246(13)$ Å; $\beta = 104.762(2)^\circ$; $V = 3162.2(4)$ Å³; $Z = 4$; $\rho_{\text{calcd}} = 1.317$ g cm⁻³; $\mu = 1.003$ mm⁻¹; $F(000) = 1292$; $2\theta_{\text{max}} = 55.90^\circ$; 190882 collected reflections; 7594 unique reflections ($R_{\text{int}} = 0.0527$); 305 parameters; 7 restraints; $S = 1.050$; $R_1 = 0.0286$ [$I > 2\sigma(I)$]; wR_2 (all data) = 0.0694; max/min residual electron density = $+1.166/-0.563$ e Å⁻³.
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