[13] X-ray structural analysis. **3**: $C_{22}H_{19}S_2P$, 0.40 × 0.35 × 0.33 mm, hexagonal, space group $R\bar{3}$, $a = 33.059(5)$, $c = 9.001(2)$ Å, $V = 8519(3)$ Å^3, $Z = 18$, $\rho_{calcd} = 1.328$ mg m^{-3}, $2\theta_{max} = 54°$, $\lambda(Mo_{K\alpha}) = 0.71073$ Å, ω scan, 293 K, 2319 measured reflections, 2307 independent, observed $I > 2\sigma(I)$, Lorentz and polarization correction ($\mu = 3.67$ cm^{-1}), 227 refined parameters with $R1 = 0.0582$ and $wR2 = 0.1649$, max./min. residual electron density 0.335/−0.304 e Å^{-3}. **4**: $C_{37}H_{25}N_2O_5PS_2W$, 0.45 × 0.40 × 0.40 mm, monoclinic, space group $C2/c$, $a = 26.378(7)$, $b = 22.272(2)$, $c = 15.479(7)$ Å, $\beta = 123.99(2)°$, $V = 7540.4(4)$ Å^3, $Z = 4$, $\rho_{calcd} = 1.571$ mg m^{-3}, $2\theta_{max} = 54°$, $\lambda(Mo_{K\alpha}) = 0.71073$ Å, ω scan, 293 K, 8218 measured reflections, 8044 independent, observed $I > 2\sigma(I)$, Lorentz and polarization correction ($\mu = 33.31$ cm^{-1}), 437 refined parameters with $R1 = 0.0573$ and $wR2 = 0.1615$. After Lorentz and polarization corrections with HELENA, the structures were solved with SIR-97, and refined with SHELX93 by full-matrix least squares on F^2. Programs: HELENA: A. L. Spek, **1997**, Utrecht University, The Netherlands; SIR-97: A. Altomare, M. C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna, *J. Appl. Crystallogr.* **1998**, *31*, 74; SHELX93: G. M. Sheldrick, **1993**, Universität Göttingen, Germany. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-137515 and -137516. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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The First Structurally Characterized Metal–SeH Compounds: $[LAl(SeH)_2]$ and $[L(HSe)AlSeAl(SeH)L]$[**]

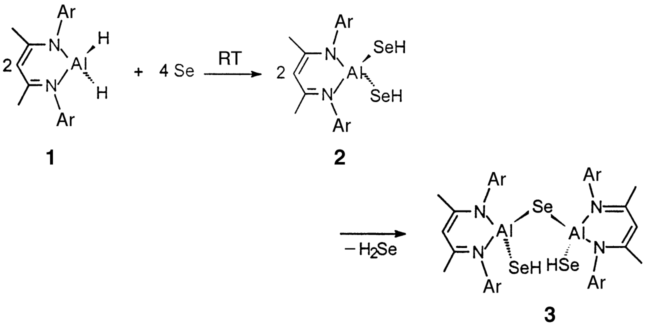
Chunming Cui, Herbert W. Roesky,* Haijun Hao, Hans-Georg Schmidt, and Mathias Noltemeyer

Dedicated to Professor Jürgen Troe
on the occasion of his 60th birthday

Aluminum compounds containing Group 16 elements have been widely studied due to their important applications in chemical vapor deposition (CVD) and catalysis.[1] We have recently shown that organoaluminum dihydrides are very useful precursors for preparing aluminum chalcogenides of the formula $[(RAlE)_n]$ (R = organic ligand; E = S, Se, Te; n = 2,4).[2] A few novel intramolecularly stabilized aluminum chalcogenides, $[\{[N(SiMe_3)C(Ph)C(SiMe_3)_2]AlE\}_2]$[2a] and $[(ArAlE)_2]$ (Ar = 2,6-$(Et_2NCH_2)_2C_6H_3$, 2-Et_2NCH_2-6-MeC_6H_3; E = Se, Te),[2b] have been synthesized. We became interested in employing the same synthetic route using other ligand systems with the aim of studying the mechanism of the reaction and synthesizing monomeric aluminum chalcogenides [RAlE]. Previously we predicted that this reaction proceeds through hydroselenide (SeH) or hydrotelluride (TeH) intermediates.[2] Barron et al. and Oliver et al. discussed the same intermediates for the reaction of Group 13 trialkyl compounds with H_2Se.[3] However, no stable compounds of Group 13 elements with the SeH ligand have been isolated so far. In fact, only a few transition metal complexes (Co, Cr, Fe, Ir, Mn, Pt, Re, Ta, Ti) containing the SeH ligand (both terminal and bridging) are known, but they have not been structurally characterized.[4] Herein we report on the first stable aluminum SeH compounds with the β-diketiminato ligand.

Reaction of the β-diketimine LH (L = N(Ar)C(Me)-CHC(Me)N(Ar), Ar = 2,6-$iPr_2C_6H_3$)[5] with $AlH_3 \cdot NMe_3$ at room temperature in n-hexane leads to the dihydride $[LAlH_2]$ (**1**; see Scheme 1) in high yield. The IR spectrum shows typical asymmetric and symmetric absorptions[6] for Al–H at 1832 and 1795 cm^{-1}, and the mass spectrum gives the highest peak at $[M^+ - H]$, indicating the formation of a monomeric dihydride. Compound **1** is related to $[\{(iPr)_2(ati)\}AlH_2]$ (ati = 2-aminotroponimine), which has a monomeric structure in the solid state.[6b]

Compound **1** reacts smoothly with two equivalents of elemental selenium at room temperature to afford $[LAl(SeH)_2]$ (**2**) in modest yield (Scheme 1). It is noteworthy that **2** is formed even when only one equivalent of Se is used for the reaction. Compound **2** is not stable in solution (n-hexane, THF) over a longer period of time at room temperature, and it slowly eliminates H_2Se to give pale yellow $[L(HSe)AlSeAl(SeH)L]$ (**3**). Compound **3** can also be obtained by the reaction of **1** with two equivalents of Se at 60 °C.



Scheme 1. Synthesis of **2** and **3**. Ar = 2,6-$iPr_2C_6H_3$.

Compounds **2** and **3** have been characterized by multinuclear NMR spectroscopy, mass spectrometry as well as elemental analysis. The ^1H NMR spectra of **2** and **3** show a high-field singlet at $\delta = -2.82$ and -2.83, respectively, which

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[*] Prof. Dr. H. W. Roesky, Dipl.-Chem. C. Cui, H. Hao, H.-G. Schmidt, Dr. M. Noltemeyer
Institut für Anorganische Chemie der Universität
Tammannstrasse 4, 37077 Göttingen (Germany)
Fax: (+49) 551-393373
E-mail: hroesky@gwdg.de

[**] L = N(Ar)C(Me)CHC(Me)N(Ar), Ar = 2,6-$iPr_2C_6H_3$. This work was supported by the Deutsche Forschungsgemeinschaft

can be assigned to SeH.[7] The IR spectra also give weak SeH stretching frequencies at 2318 cm^{-1} for **2** and at 2276 and 2292 cm^{-1} for **3**.[4c] The ^{1}H NMR spectrum of **3** shows two septets for the C*H*Me$_2$ protons (*i*Pr groups), indicating C_s symmetry of the molecule. The ^{77}Se NMR spectrum of **3** gives two broad signals ($\delta = -341$ and -535) in an approximate intensity ratio of 2:1; the former can be assigned to SeH.[8]

The molecular structures of **2** and **3** were determined by X-ray crystallography.[9] Yellow crystals of **2** suitable for X-ray structure analysis were grown from *n*-hexane at −30 °C. Unfortunately, the quality of the crystal structure did not allow location of the hydrogen atoms on the selenium atoms. Nevertheless, the presence of terminal SeH groups was established unequivocally by IR and ^{1}H NMR spectroscopy. The molecular structure of **2** is shown in Figure 1. The Al–Se bond lengths (234.0 and 233.1 pm) are in line with those of the dimeric $[\{[\text{N(SiMe}_3)\text{C(Ph)C(SiMe}_3)_2]\text{AlSe}\}_2]$ (av 235 pm).[2] The Al–N bond distances of **2** are equal, and the Al atom deviates slightly from the NCCCN plane of the ligand.

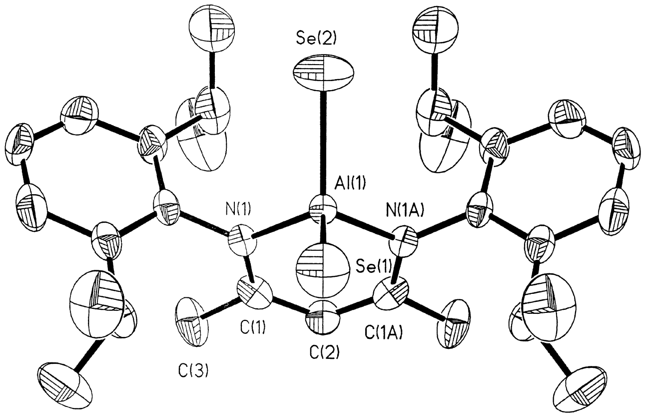

Figure 1. Molecular structure of **2** in the crystal. Hydrogen atoms have been omitted for clarity. Selected bond distances [pm] and angles [°]: Al(1)-N(1) 189.9(6), Al(1)-N(1A) 189.9(6), Al(1)-Se(2) 233.1(3), Al(1)-Se(1) 2.340(3); N(1)-Al(1)-N(1A) 97.1(4), N(1)-Al(1)-Se(2) 113.5(2), N(1A)-Al(1)-Se(2) 113.5(2), N(1)-Al(1)-Se(1) 114.8(2), N(1A)-Al(1)-Se(1) 114.8(2), Se(2)-Al(1)-Se(1) 103.73(12).

The structure of compound **3** is shown in Figure 2.[9] The Al–Se bond lengths (232.6 and 233.3 pm) of the Al-Se-Al unit are among the shortest reported so far. They are slightly shorter than those of the Al–SeH units (237.5 and 237.1 pm) due to the more highly polarized Al-Se-Al bonds. The internal angle at Se(2) (116.88°) is much more open than those of compounds with the dimeric Al$_2$Se$_2$ core (av 78°),[2] and is only slightly larger than that of $[\{[(\text{SiMe}_3)_2\text{CH}]_2\text{Al}\}_2\text{Se}]$ (114.71°),[10] probably due to the bulky nature of the ligand L. The hydrogen atoms could be located, by a ΔF synthesis, at the selenium atoms (Se–H ca. 150 pm); free refinement, however, shortens this distance (as expected for hydrogen atoms on heavy atoms). There is very little information in the literature on the Se–H bond. The Se–H bond lengths computed (with the MINI-1, 3-21-G/LWD and STD-3G basis sets) for organic RSeH compounds range from 142 to 157 pm.[11] Compounds **2** and **3** are well separated monomers in the solid state, which excludes the presence of any intermolecular hydrogen bonds. Furthermore, **2** and **3** are the first structurally characterized organometallic compounds which contain metal–SeH units.

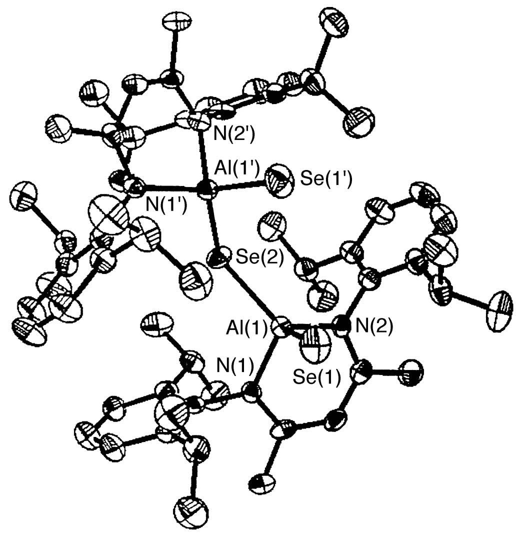

Figure 2. Molecular structure of **3** in the crystal. Hydrogen atoms have been omitted for clarity. Selected bond distances [pm] and angles [°]: Al(1′)-N(2′) 191.2, Al(1′)-N(1′) 191.8(5), Al(1′)-Se(2) 232.6(2), Al(1′)-Se(1′) 237.5(2), Al(1)-N(2) 190.6(5), Al(1)-N(1) 190.6(5), Al(1)-Se(2) 233.3, Al(1)-Se(1) 237.1(2); N(2′)-Al(1′)-N(1′) 96.7(2), N(2′)-Al(1′)-Se(2) 108.2(2), N(1′)-Al(1′)-Se(1′) 109.5(2), N(1′)-Al(1′)-Se(1′) 106.0(2), Se(2)-Al(1′)-Se(1′) 117.29(9), N(2)-Al(1)-N(1) 97.3(2), N(2)-Al(1)-Se(2) 115.4(2), N(1)-Al(1)-Se(2) 108.5(2), N(2)-Al(1)-Se(1) 107.0(2), N(1)-Al(1)-Se(1) 107.5(2), Se(2)-Al(1)-Se(1) 118.69(8), Al(1′)-Se(2)-Al(1) 116.77(7).

The X-ray structural analysis of **2** and **3** disclose the lability of the Se–H bonds,[12] and provide direct evidence that the tendency to form hydrogen bonds is much lower than for the lighter congeners (S, O).[13] The stabilization of **2** and **3** might be attributed to the bulky chelating ligand. Owing to the acidic nature of the SeH moiety, compounds **2** and **3** might be used as synthons for preparing Se-bridged heterometallic compounds. Moreover, the synthesis of **2** and **3** suggests that the selenium atom can insert very easily into the Al–H bond.

An explanation for why the reaction of [LAlH$_2$] with one equivalent of selenium yields **2** instead of [LAl(H)SeH] is presently not available. It is difficult to monitor the reaction by NMR spectroscopy due to its heterogeneous nature. However, it is supposed that the Se$_2$ unit is involved in this reaction. It has been argued[14c] that reactions of diorganodiselenides with $\text{AlH}_3 \cdot \text{NMe}_3$ proceed by initial complexation of a selenium species, creating a hypervalent aluminum center.[14a, 14b] According to this argument the reaction of [LAlH$_2$] with selenium proceeds by coordination of a Se$_2$ unit to the aluminum center, followed by a concerted reduction of the activated Se$_2$ by the two hydride ligands to cleave the Se$_2$ unit. The conversion of **1** to **2** demonstrates the 'umpolung' of the hydridic to the protonic form of the hydrogen atoms. This assumption can well explain our experimental results that **2** is always the product formed at room temperature, regardless of the amount of selenium used. The conversion of **2** into **3** clearly indicates that intermolecular elimination of H$_2$Se is favored over intramolecular elimination.

Experimental Section

1: A solution of LH (2.50 g, 5.98 mmol) in *n*-hexane (40 mL) was added at room temperature to a solution of $AlH_3 \cdot NMe_3$ (0.56 g, 6.29 mmol) in *n*-hexane (10 mL), and the resulting mixture was stirred for 48 h. All volatile components were removed in vacuo, and the crude product was crystallized from *n*-hexane to afford colorless crystals of **1** (2.3 g, yield 86.2 % based on LH); m.p. 194 °C. ^{1}H NMR (200 MHz, C_6D_6): $\delta = 7.18$ (d, 4H, aromatic H of Ar group), 7.10 (m, 2H, aromatic H of Ar group), 4.86 (s, 1H, γ-CH), 3.42 (sept, 4H, C*H*Me$_2$), 1.54 (s, 6H, C*Me*), 1.37, 1.12 (d, 24H, CH*Me*$_2$); ^{13}C NMR (125.75 MHz, C_6D_6): $\delta = 170.2$ (CN), 144.6, 139.4, 124.8 (Ar), 96.2 (γ-CH), 28.48 (*C*HMe$_2$), 25.5, 24.5 (CH*Me*$_2$), 22.9 (C*Me*); IR (KBr, Nujol): $\tilde{\nu} = 1832, 1795\ \mathrm{cm^{-1}}$ (m, Al–H); EI-MS: *m*/*z* (%): 445 (100, $[M^+ - H]$); elemental analysis calcd for $C_{29}H_{43}AlN_2$: C 78.0, H 9.7, N 6.3; found: C 78.2, H 9.7, N 6.7.

2: To a mixture of **1** (0.45 g, 1 mmol) and selenium (0.16 g, 2 mmol) was added toluene (20 mL). The mixture was stirred at room temperature for 15 h, and a light orange solution formed with trace amounts of unchanged selenium. After filtration, the solvent was removed and the residue extracted with *n*-hexane (20 mL). The extract was stored at −30 °C for 2 d to give yellow crystals of **2** (0.35 g, yield 58 %); m.p. 220 °C (decomp.). ^{1}H NMR (500 MHz, C_6D_6): $\delta = 7.15-7.09$ (m, 6H, aromatic H of Ar group), 4.93 (s, 1H, γ-CH), 3.52 (sept, $^3J_{HH} = 6.8$ Hz, 4H, C*H*Me$_2$), 1.51 (s, 6H, C*Me*), 1.35 (d, $^3J_{HH} = 6.8$ Hz, 12H, CH*Me*$_2$), 1.10 (d, $^3J_{HH} = 6.8$ Hz, 6H, CH*Me*$_2$), −2.82 (s, 2H, SeH); ^{27}Al NMR (65.17 MHz, C_6D_6): $\delta = 122$ ($\nu_{1/2} = 260$ Hz); EI-MS: *m*/*z* (%): 604 (8, $[M^+ - H]$), 525 (20, $[M^+ - SeH]$), 403 (100, $[L^+ - Me]$); IR (KBr, Nujol): $\tilde{\nu} = 2318\ \mathrm{cm^{-1}}$ (w, SeH); elemental analysis calcd for $C_{29}H_{43}AlN_2Se_2$: C 57.6, H 7.2, N 4.6; found: C 58.0, H 7.5, N 4.6.

3: This compound was prepared in a similar manner as **2** except that the mixture was stirred at 60 °C for 15 h. After removal of the solvent, the residual was recrystallized from THF at −30 °C to give pale yellow crystals of **3** (0.26 g, yield 46 %); m.p. 250 °C (decomp.). ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.13, 7.03, 6.92$ (m, 12H, aromatic H of Ar group), 5.17 (s, 2H, γ-CH), 3.21 (sept, $^3J_{HH} = 6.7$ Hz, 4H, C*H*Me$_2$), 2.98 (sept, $^3J_{HH} = 6.8$ Hz, 4H, C*H*Me$_2$), 1.61 (s, 12H, C*Me*), 1.10 (d, $^3J_{HH} = 6.75$ Hz, 12H, CH*Me*$_2$), 0.95 (d, $^3J_{HH} = 6.65$ Hz, 12H, CH*Me*$_2$), 0.90 (d, $^3J_{HH} = 6.82$ Hz, 12H, CH*Me*$_2$), 0.84 (d, $^3J_{HH} = 6.82$ Hz, 12H, CH*Me*$_2$), −2.83 (s, 2H, SeH); ^{27}Al NMR (65.17 MHz, $CDCl_3$): $\delta = 116$ ($\nu_{1/2} = 680$ Hz); ^{77}Se NMR (C_6D_6, 500 MHz): $\delta = -341$ (s, SeH, $\nu_{1/2} = 120$ Hz), −535 (s, Al_2Se, $\nu_{1/2} = 90$ Hz); EI-MS: *m*/*z* (%): 1126 (7, $[M^+ - 2H]$), 1048 (90, $[M^+ - H - SeH]$), 443 (100, $[LAl^+ + H]$); IR (KBr, Nujol): $\tilde{\nu} = 2276, 2292\ \mathrm{cm^{-1}}$ (w, SeH); elemental analysis calcd for $C_{58}H_{84}Al_2N_4Se_3$: C 61.8, H 7.5, N 5.0; found: C 62.2, H 7.1, N 5.0.

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[8] Se satellites cannot be observed due to the broadness of the signals. A ^{77}Se NMR spectrum of **2** could not be obtained because of decomposition of the compound in solution.

[9] The data were collected on a Stoe-Siemens four-circle diffractometer using $Mo_{K\alpha}$ radiation. The intensities were recorded with φ and ω scans. The structures were solved by direct methods (SHELXS-96)[15] and refined against F^2 using SHELXL-97.[16] All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were included in the refinement in geometrically ideal positions. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-139091 (**2**) and -139092 (**3**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk). Crystallographic data for **2** ($C_{29}H_{43}AlN_2Se_2$): $M_r = 604.55$, orthorhombic, space group *Pnma*, $a = 1306.5(3)$, $b = 2204.9(4)$, $c = 1055.7(2)$ pm, $\alpha = \beta = \gamma = 90°$, $V = 3.0467(11)\ \mathrm{nm^3}$, $Z = 4$, $\rho_{calcd} = 1.318\ \mathrm{Mg\,m^{-3}}$, $F(000) = 1248$, $\lambda = 71.073$ pm, $T = 203(2)$ K, $\mu = 2.475\ \mathrm{mm^{-1}}$; crystal dimensions $1.00 \times 0.80 \times 0.40$ mm, $7.26 \leq 2\theta \leq 50.02°$; of 2769 reflections collected, 2760 were independent and were used in the structure refinement of 165 parameters; $R1 = 0.1020$ ($I > 2\sigma(I)$), $wR2 = 0.2787$ (all data); min./max. residual electron density $1706/-1408\ \mathrm{e\,nm^{-3}}$. Crystallographic data for **3** ($C_{58}H_{84}Al_2N_4Se_3$): $M_r = 1128.13$, monoclinic, space group $P2_1/n$, $a = 1225.5(3)$, $b = 2734.7(6)$, $c = 1775.3(4)$ pm, $\alpha = 90$, $\beta = 98.29(3)$, $\gamma = 90°$, $V = 5.888(2)\ \mathrm{nm^3}$, $Z = 4$, $\rho_{calcd} = 1.273\ \mathrm{Mg\,m^{-3}}$, $F(000) = 2352$, $\lambda = 71.073$ pm, $T = 203(3)$ K, $\mu = 1.942\ \mathrm{mm^{-1}}$; crystal dimensions $1.00 \times 0.80 \times 0.40$ mm, $7.06 \leq 2\theta \leq 45.06°$; of 8425 reflections collected, 7712 were independent and were used in the structure refinement of 630 parameters; $R1 = 0.0595$ ($I > 2\sigma(I)$), $wR2 = 0.1253$ (all data); min./max. residual electron density $1847/900\ \mathrm{e\,nm^{-3}}$.

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