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anti-16 product can be used as an example to demonstrate the differentiation between the double bonds in the corresponding metathesis product. Careful ozonolysis of *anti*-16 monitored by thin-layer chromatography, followed by reductive workup with lithium aluminum hydride, yielded 79% of the monoprotected aminodiol 17, which offers a variety of possibilities for further elaboration.

Experimental Procedure

Typical procedure for the stereoselective ring-closing metathesis as exemplified by ent-syn-16 (Table 1, entry 11): ent-12 (50 mg, 0.13 mmol) was dissolved in anhydrous benzene (9.2 mL) under argon, [Mo] (4.9 µmol, 5 mol%) was then added, and the resulting mixture was stirred for 3 d at a bath temperature of 80 °C. The solvent was removed and the residue was purified by flash chromatography on silica gel by using tert-butyl methyl ether/petroleum ether (1:40) as eluent to afford 44 mg (97%) of the cyclization product (72% de, GC) as a yellow oil. Selected spectroscopic data: ¹H NMR (400 MHz, CDCl₃): two amide isomers, combined spectrum: $\delta = 0.05$ (s, 6 H), 0.88 und 0.89 (s, 9 H), 1.68 and 1.69 (dd, 3 H, J = 1.5, 6.5 Hz), 3.82 (m, 1H), 3.87 (m, 1H), 4.76-4.97 (m, 2H), 5.11-5.45 and 5.59-5.95 (m, 4H). ¹³C NMR (50 MHz, CDCl₃): two amide isomers, combined spectrum: $\delta = -5.5$ (CH₃), -5.4 (CH₃), 17.5 (CH₃), 17.7 (CH₃), 18.4 (C_a), 25.8 (CH₃), 25.9 (CH₃), 62.4 (CH₂), 67.0 (CH), 67.1 (CH), 68.2 (CH), 68.4 (CH), 126.7 (CH), 127.3 (CH), 128.1 (CH), 128.3 (CH), 129.2 (CH), 129.8 (CH), 130.0 (CH), 153.2 (C_q). HR-MS $(C_{15}H_{23}F_{3}NO_{2}Si, M^{+} - CH_{3})$: calcd 334.1450, found 334.1450. IR (CCl_{4}) : $1/\lambda = 1686 \text{ cm}^{-1}$. Elemental analysis (C₁₆H₂₆F₃NO₂Si): calcd C 54.99 H 7.50 N 4.01, found C 55.09 H 7.50 N 4.24.

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Reductive Carbon-Sulfur Bond Cleavage: A Simple Pathway to Nonstabilized (Lithiomethyl)amines**

Carsten Strohmann* and Bors Cajus Abele

(Lithiomethyl)amines are important building blocks in the synthesis of not only organic but also organoelement compounds.^[11] Nonstabilized^[2] α -heterocarbanions^[3]—containing nitrogen or oxygen as the heteroatom—cannot ordinarily be synthesized by hydrogen—lithium or halogen—lithium exchange using either lithium bases or metallic lithium.^[11] However, (lithiomethyl)amines^[4] and (lithiomethyl)ethers^[5] can usually be obtained by metal—lithium, typically tin—lithium, exchange. Reductive carbon—sulfur bond cleavage permits another pathway to (lithiomethyl)ethers;^[6] in addition it circumvents the chromatographic separation necessary for stannylmethyl compounds.^[5, 7]

During systematic investigations on the synthesis of 1,3dimetalated compounds of general formula M--CR₂-El-CR₂-M (M = e.g. Li, MgBr; El = Group 14-16 element, when necessary containing substituents; R = H, alkyl, aryl),^[8] we were also interested in the preparation of bis(lithiomethyl)amines.^[9] There has only been one report^[10] on the synthesis of nonstabilized, α -alkyl substituted, monometalated α -aminocarbanions using reductive C-S bond cleavage. The synthetic aspects of this reaction have not been further investigated. We report here on a simple pathway to nonstabilized mono(lithiomethyl)amines that are not substituted on the α -carbon atom by means of reductive C-S bond cleavage, as well as on the use of these synthetic building blocks in the construction of organic and organoelement compounds.

The required (phenylthiomethyl)amine starting materials can be synthesized in high yields from thiophenol, formaldehyde, and secondary amines^[11] (Scheme 1). Treatment of the



(phenylthiomethyl)amines with a solution of lithium naphthalide ($\text{LiC}_{10}\text{H}_8$) in THF at -78 °C gives a red solution of the corresponding (lithiomethyl)amine. In order to prove that the

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(lithiomethyl)amine had been formed, the reaction solutions were treated with two equivalents^[12] of chloro(dimethyl)phenylsilane. The corresponding pure (aminomethyl)silanes 7a-f were isolated in 67–80% yield (Table 1).

Table 1. Yields of 7a-7h.

7	NR ₂	Yield [%]	7	NR ₂	Yield [%]
<u>а</u>	N(C ₂ H ₃),	80	e	NC₄H ₈ NCH ₃	68
b	NC ₄ H ₈	72	f	$N(C_2H_4OCH_3)_2$	70
с	NC ₅ H ₁₀	74	g	$N(i-C_3H_7)_2$	27
d	NC ₄ H ₈ O	67	h	$N(C_6H_5)_2$	0 [a]

[a] Isolation of the pure entrapped products of 5h with PhMe₂SiCl has not yet been successful. The trapping reaction with Bu₃SnCl gave diphenyl(tributylstannyl-methyl)amine in 56% yield.

Sterically demanding amines such as diisopropyl(phenylthiomethyl)amine **4g** and diphenyl(phenylthiomethyl)amine **4h** show a somewhat different reaction behavior. Reaction of the (lithiomethyl)amines with the starting material lead to C–C bond formation so that the $\text{LiC}_{10}\text{H}_8$ was not completely consumed. When **4g** was treated with $\text{LiC}_{10}\text{H}_8$ followed by entrapment with PhMe₂SiCl, both **7g** and **8** were isolated (Scheme 2). The structure of further side products has not yet been clarified. Amine **4h** reacts in a similar manner.



In the reaction mixtures of the (lithiomethyl)amines obtained by reductive C-S bond cleavage, a further nucleophile, PhSLi **6**, which is not formed in the tin-lithium exchange reaction is generated. Nevertheless, **6** did not interfere with reactions with ketones, stannanes, and silanes, which furnished products in good yields (Scheme 3). Also stirring the (lithiomethyl)amines



Scheme 3. Reactions of (lithiomethyl)amines: a) $5b + 2 Bu_3SnCl/ - 2 LiCl_1 - Bu_3SnSPh_5 b) 5e + Ph_2CO_5 + 2 H_2O_7 - 2 LiOH_5 - HSPh_5 c) 25a + Ph_2SiCl_2 - 2 LiCl_5 - 2 LiSPh.$

for 15 min at room temperature did not lead to a change in the product composition. Protonation of the metalated carbon atoms or carbenoid reactions^[1b] (for example, the decomposition into lithium amides and methylene) was also not observed. We believe the synthesis of bis(aminomethyl)silanes such as 11 is particularly interesting (Table 2). As far as we know, the only

Table 2. Selected spectroscopic data of the newly synthesized compounds.

7e: yield: 68%; b.p. 121 °C oven temperature/ 10^{-3} mbar; ¹H NMR (200.1 MHz, CDCl₃, 25 °C, TMS): $\delta = 0.28$ (s, ²J(H,Si) = 6.6 Hz, 6H; SiCH₃), 2.10 (s, ²J(H,Si) = 5.3 Hz, 2H; SiCH₂N), 2.18 (s, 3H; NCH₃), 2.30–2.35 (m, 8H; NCH₂CH₂N), 7.25–7.35, 7.45–7.55 (m, 5H; Ph); ¹³C NMR (50.3 MHz, CDCl₃, 25 °C, CDCl₃): $\delta = -2.7$ (2 C, SiCH₃), 46.0 (NCH₃), 49.6 (SiCH₂N), 55.3, 56.9 (4 C, NCH₂CH₂N), 127.7 (2 C, m-Ph), 128.9 (*p*-Ph), 133.6 (*o*-Ph) 139.0 (*ipso*-Ph); ²⁹Si NMR (39.8 MHz, CDCl₃, 25 °C, TMS): $\delta = -7.0$; MS (EI, 70 eV): *m/z* (%) = 248 (5) [*M*⁺], 233 (2) [*M*⁺ - CH₃], 113 (100) [CH₂NC₄H₈NCH₃⁺]; correct elemental analysis.

7f: yield: 70%; b.p. 128 °C oven temperature/ 10^{-3} mbar; ¹H NMR (200.1 MHz, CDCl₃, 25 °C, TMS): $\delta = 0.30$ (s, ²*J*(H,Si) = 6.4 Hz, 6H; SiCH₃), 2.25 (s, 2H; SiCH₂N), 2.58 (t, ³*J*(H,H) = 6.2 Hz, 4H; NCH₂C), 3.23 (s, 6H; OCH₃), 3.34 (t, ³*J*(H,H) = 6.2 Hz, 4H; OCH₂C), 7.25–7.35, 7.45–7.50 (m, 5H; Ph); ¹³C NMR (50.3 MHz, CDCl₃, 25 °C, CDCl₃): $\delta = -2.9$ (2 C, SiCH₃), 46.4 (SiCH₂N), 57.2 (2 C, NCH₂C), 58.7 (2 C, OCH₃), 71.0 (2 C, OCH₂C), 127.7 (2 c, *m*-Ph), 128.9 (*p*-Ph), 133.7 (2 C, *o*-Ph), 138.9 (*ipso*-Ph); ²⁹Si NMR (39.8 MHz, CDCl₃, 25 °C, TMS): $\delta = -6.6$; MS (EI, 70 eV): *m/z* (%) = 281 (4) [*M*⁺], 236 (88) [*M*⁺ - CH₂OCH₃], 204 (6) [*M*⁺ - C₆H₅], 146 (50) [CH₂N(C₂H₄OCH₃)₂]⁺, 102 (100); correct elemental analysis.

7g: yield: 27%; b.p. 95 °C oven temperature/ 10^{-3} mbar; ¹H NMR (200.1 MHz, CDCl₃, 25 °C, TMS): $\delta = 0.28$ (s, ²J(H,Si) = 6.4 Hz, 6H; SiCH₃), 0.86 (d, ³J(H,H) = 6.6 Hz, 12H; NCCH₃), 2.14 (s, 2H; SiCH₂N), 2.86 (sept, ³J(H,H) = 6.6 Hz, 2H; NCHC₂), 7.20-7.30, 7.45-7.50 (m, 5H; Ph); ¹³C NMR (50.3 MHz, CDCl₃, 25 °C, CDCl₃): $\delta = 2.8$ (2 C, SiCH₃), 20.3 (4 C, NC(CH₃)₂) 34.2 (SiCH₂N), 49.0 (2 C, NCHC₂), 127.6 (2 C, m-Ph), 128.7 (p-Ph), 133.7 (2 C, o-Ph), 140.0 (*ipso-Ph*); ²⁹Si NMR (39.8 MHz, CDCl₃, 25 °C, TMS): $\delta = -5.8$; MS (EI, 70 eV): m/2 (%) = 249 (10) [M^{+1}], 234 (10) [M^{+} -CH₃], 114 (100) [CH₂N(C₃H₇)₂]⁺; correct elemental analysis.

11: yield: 45%; b.p. 185 °C oven temperature/ 10^{-3} mbar; ¹H NMR (200.1 MHz, CDCl₃, 25 °C, TMS): $\delta = 0.83$ (t, ³J(H,H) = 7.0 Hz, 12H; NCCH₃), 2.33 (q, ³J(H,H) = 7.0 Hz, 8H; NCH₂C), 2.51 (s, 4H; SiCH₂N), 7.25-7.35, 7.60-7.70 (m, 10H; Ph); ¹³C NMR (50.3 MHz, CDCl₃, 25 °C, CDCl₃): $\delta = 11.4$ (4C, NCCH₃), 41.8 (2 C, SiCH₂N), 50.0 (4C, NCH₂C), 127.6 (2C, *m*-Ph), 129.1 (*p*-Ph), 135.4 (2 C, *o*-Ph), 136.5 (*ipso*-Ph); ²⁹Si NMR (39.8 MHz, CDCl₃, 25 °C, TMS): $\delta = -17.5$; MS (EI, 70 eV): *m/z* (%) = 354 (2) [*M*⁺], 268 (53) [*M*⁺ - CH₂N(C₂H₃)₂], 86 (100) [CH₂N(C₂H₃)₂]⁺; correct elemental analysis.

14: yield: 55%; b.p. 150 °C oven temperature/1013 mbar; ¹H NMR (200.1 MHz, CDCl₃, 25 °C, TMS): $\delta = -0.02$ (s, ²*J*(H,Si) = 6.4 Hz, 18 H; SiCH₃), 1.83 (s, 4 H; SiCH₂N), 2.33 (m, 8 H; NCH₂CH₂N); ¹³C NMR (50.3 MHz, CDCl₃, 25 °C, CD-Cl₃): $\delta = -1.1$ (6 C, SiCH₃), 50.8 (2 C, SiCH₂N), 57.3 (4 C, NCH₂CH₂N); ²³Si NMR (39.8 MHz, CDCl₃, 25 °C, TMS): $\delta = -1.6$; MS (EI, 70 eV): m/z (%) = 258 (20) [M^+], 243 (12) [$M^+ - CH_3$], 229 (2) [$M^+ - CH_3 - CH_2$], 185 (100) [$M^+ - SiCH_3$], 73 (32) [SiCH₃]⁺; correct elemental analysis.

representative of this class that has been reported so far is bis-(dimethylaminomethyl)dimethylsilane.^[13]

The synthesis of multimetalated systems is also possible using this reductive bond cleavage. The product of N,N-bis(lithiomethyl)piperazine 13 entrapped by Me₃SiCl was isolated in 55% yield (Scheme 4). The preparation of further multimetalated, nonstabilized (lithiomethyl)amines is currently under investigation.

Nonstabilized (lithiomethyl)amines can be obtained in high yields by reductive C-S bond cleavage in a two-step synthesis starting from the appropriate amines and starting materials, which are both easily obtainable and inexpensive. These (lithiomethyl)amines can be used as building blocks in further synthesis reactions. The advantages of this method over tinlithium exchange include the shortening of the synthesis by one step (which utilizes relatively expensive alkyl tin compounds) and the circumvention of chromatographic purification of the

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Scheme 4. Synthesis of $N_{,N'}$ -bis(trimethylsilylmethyl)piperazine: a) +4 LiC₁₀H₈/ - 4 C₁₀H₈; b) 4 Me₃SiCl/ - 4 LiCl, -2 Me₃SiSPh.

starting materials. On account of the simple pathway to (phenylthiomethyl)amines and the wide range of applications of the metalation reaction, reductive C–S bond cleavage could develop into an important method for the synthesis of compounds containing the $-CH_2NR_2$ structural unit.

Experimental Procedure

(Phenylthiomethyl)amines: The (phenylthiomethyl)amines were prepared by reported methods [11]. Amines 7a [14], 7b [15], 7c [15], 7d [11], 8 [16], 9 [7a], and 10 [7] have already been reported.

Preparation of the (lithiomethyl)amines and their reactions: To a freshly prepared solution of lithium naphthalide from lithium granules (694 mg, 100 mmol) and naphthalene (12.8 g, 100 mmol) in 150 mL THF at -78 °C was added (phenyl-thiomethyl)amine (50 mmol) in 50 mL THF precooled to the same temperature. After 10 min chloro(dimethyl)phenylsilane (7a-7h; 17.1 g, 100 mmol), dichloro-diphenylsilane (11; 12.7 g, 100 mmol), or chlorotrimethylsilane (14; 10.8 g, 100 mmol) was added. The mixture was warmed to room temperature and then subjected to an aqueous workup. After removal of the volatiles in vacuo, the residue was purified by Kugelrohr distillation.

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The Existence of Stable Tm@C₈₂ Isomers**

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Since the discovery of the fullerene synthesis by Krätschmer and Huffman^[1] in 1990, the study of endohedral fullerenes is one of the main streams in current fullerene research^[2] besides the growing field of chemical derivatization especially of C_{60} . The incorporation of metal atoms into the carbon cage results in an electron transfer from the metal atom to the carbon structure under the formation of a stable ion pair. Because of the steric hindrance by the surrounding fullerene the metal ion cannot leave the carbon cage nor react with other substances. The study of the electron transfer at such "caught" metal cations is a very new field in electrochemical research. Up till now the electron transfer to the carbon cage rather than the intramolecular transfer reactions is the main topic of these studies.

Our recent work concentrated on the preparation of several endohedral fullerenes containing different metals and their characterization by mass spectrometry, cyclic voltammetry, ex situ and in situ ESR, and UV/Vis spectroscopy.^[3] New results were obtained by ex situ and in situ ESR spectroscopy on the composition of the fullerene soot extract, the differences in the redox behavior of empty and endohedral fullerenes, and the existence of different paramagnetic states at endohedral fullerenes. As metals, scandium, yttrium, lanthanum, and the rare earth metals cerium, praseodymium, samarium, holmium, thulium, and lutetium were used. In these studies the first successful mass spectrometric proof of the existence of endohedral thulium fullerene in the soot extract was reported.^[3]

In this paper it will be demonstrated for the first time that three stable $\text{Tm}@C_{82}$ isomers exist, which can be isolated and structurally characterized. These isomers of an endohedral

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