Tetrakis(sulfurdiimido)silane, -germane and -stannane

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Silicon, Germanium, Tin, Sulfur Diimides, Heterocycles, NMR Data

Two tetrakis(sulfurdiimido)silanes [Si(NSN)]₄ (R = 'Bu 1a, SiMe₃ 2b), two germanes [Ge(NSN)]₄ (R = 'Bu 2a, SiMe₃ 2b) and one stannane [Sn(NSN'Bu)]₄ 3a were prepared and characterised by 'H, ¹³C, ¹⁵N, ²⁹Si and ¹¹⁹Sn NMR spectroscopy in solution, and 3a was also studied in the solid state by ¹¹⁹Sn CP/MAS NMR. Whereas 1a, 2a and 2b are monomeric in solution, the ¹¹⁹Sn NMR data suggest that 3a is associated both in solution and in the solid state, and that the tin atoms are hexa-coordinated. The attempted stepwise synthesis of 1a by using one, two, three or four equivalents of K[(NSN)R] led to mixtures of 1a with Cl₃Si(NSN'Bu) 4a, Cl₂Si(NSN'Bu)₂ 5a, and ClSi(NSN'Bu)₃ 6a. Only one sulfurdiimido ligand of the silane 1a reacted with hexachlorodisilane by oxidative addition and cleavage of the Si-Si bond to give the new heterobicyclic derivative 7a which is held together by two different coordinative N-Si bonds.

Introduction

The chemistry of MX₄ compounds (M = Si, Ge, Sn) is well developed for various substituents X such as halides, pseudohalides, chalcogenides, amides, or organyl groups [1, 2]. However, analogous derivatives of the type M(NSN)]₄ have not been reported as yet. Such compounds should be of interest with respect to their structural flexibility and their reactivity, since the M-N bonds [3] are sensitive to moisture and readily soluble in toluene or chlorinated solvents. To improve the solubility of the potassium salts, K[(NSN)R] (R = 'Bu, SiMe₃), and thus accelerates the reaction. The products 1 - 3 are isolated as yellow to orange oils (1b, 2a,b) or orange solids (1a, m.p. 112°C; 3a, m.p. 253°C). They are sensitive to moisture and readily soluble in toluene or chlorinated solvents.

Results and Discussion

Synthesis of the M(NSN)]₄ compounds

The reaction of four equivalents of the potassium salt K[(NSN)R] (R = 'Bu, SiMe₃) with the respective element tetrachloride leads directly to the corresponding tetrakis(sulfurdiimido) compound [eq. (1)]. Addition of 1,2-dimethoxyethane (DME) helps

\[
4 \text{K[(NSN)R]} + \text{MCl}_4 \rightarrow \text{hexane/DME} \rightarrow -78^\circ \text{C}
\]

M(NSN)]₄ + 4 KCl

\[
\begin{array}{c|ccc}
M & \text{Si} & \text{Ge} & \text{Sn} \\
\hline
R = '\text{Bu} & 1a & 2a & 3a \\
R = \text{SiMe}_3 & 1b & 2b & & \\
\hline
\text{Cl}_3\text{Si(NSN'Bu)} & 4a & & & \\
\text{Cl}_2\text{Si(NSN'Bu)}_2 & 5a & & & \\
\text{ClSi(NSN'Bu)}_3 & 6a & & & \\
\hline
\end{array}
\]

product distribution in (%)

\[
x=1 \\
x=2 \\
x=3 \\
x=4
\]

4a 5a 6a 1a

36.3 15.0 3.5 \\
42.0 19.4 5.3 \\
28.0 46.6 25.4 \\
100.0

Reactions of one, two or three equivalents of K[(NSN'Bu)] with SiCl₄ lead to mixtures containing Cl₃Si(NSN'Bu) 4a, Cl₂Si(NSN'Bu)₂ 5a, ClSi(NSN'Bu)₃ 6a and Si(NSN'Bu)₄ (1a) [Eq. (2)]. This indicates that the reactivities of SiCl₄ and of substituted silanes such as Cl₃Si(NSN'Bu) or Cl₂Si(NSN'Bu)₂ are comparable (equation (2)).

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A prominent feature of many sulfur diimides is their fluxionality with respect to E/Z isomerisation. This is evident for alkyl groups as substituents [7-9] and also for numerous sulfur diimides bearing organometallic substituents [10-13]. For sterical reasons, isomers with Z/Z configuration are unfavourable, in particular if a 'Bu or a Me₃Si group is linked to one of the nitrogen atoms. This would leave fifteen configurational isomers for M(NSNR₄)₄ compounds, assuming rigid structures. If the E/E configuration is also excluded as unfavourable because of electron pair repulsion, five principal isomers remain, either with four times Z/E, four times E/Z, or Z/E and E/Z configurations as shown in Scheme 1. This situation corresponds closely to the findings for bis- and tris(sulfurdiimido) compounds [6].

Scheme 1. The five most likely configurations of tetrakis(sulfurdiimido)element compounds M(NSNR₄)₄ (R = 'Bu, SiMe₃; M = Si, Ge, Sn); the first letter (E or Z) refers to the position of the substituent R.

Reaction of tetrakis(N-tert-butylsulfurdiimido)stannane with triethylborane

We have investigated the reactivity of tetrakis(N-tert-butylsulfurdiimido)stannane 3a towards triethylborane in order to compare with the analogous reactions of trimethyltin-substituted sulfur diimides R'(NSN)SnMe₃ (R' = SnMe₃, 'Bu, SiMe₃). The latter have been transformed quantitatively into trimethylstannylnitrogen(diethylborylamino) sulfanes [15]. Although it cannot be excluded that the initial steps of the reaction of 3a with Et₃B (1:4 or 1:8) are similar to that of R'(NSN)SnMe₃, decomposition becomes dominant in the case of 3a. The identified decomposition products [eq. (4)] suggest that S-alkylation, followed by elimination of ethene, is favoured over rearrangement (migration of the stannyl fragment) and N-alkylation [15].

\[ \text{Reaction of tetrakis(N-tert-butylsulfurdiimido)-stannane with triethylborane} \]

\[ \text{R}_1 \text{M(NSN} \text{Bu}_3 \text{)} + \text{Cl}_3 \text{Si-SiCl}_3 \rightarrow \text{hexane} \]

\[ \text{M = Si, } R_1 = \text{NSN} \text{Bu} \]
\[ \text{M = Ge, } R_1 = \text{Me} \]

\[ \text{M = Si, } R_1 = \text{NSN} \text{Bu} \]
\[ \text{M = Ge, } R_1 = \text{Me} \]

\[ \text{Reaction of tetrakis(N-tert-butylsulfurdiimido)-silane with hexachlorodisilane} \]

Treatment of tetrakis(N-tert-butylsulfurdiimido)silane 1a with hexachlorodisilane [eq. (3)], either in an equimolar ratio or in excess, gave an orange oil which was characterised by \(^1\text{H}, \ ^{13}\text{C}, \ ^{14}\text{N}, \ ^{15}\text{N} \text{ and } ^{29}\text{Si NMR in solution. For comparison, the analogous reaction was carried out with methyl-tris(N-tert-butylsulfurdiimido)germane. Apparently, only one}

NMR spectroscopic results

General

The NMR data of all new compounds are given in Tables I (1-3), II (4-6) and III (7, 8). It has been shown that \(^{15}\text{N} \text{ NMR data of sulfur diimides [6,12,13] are particularly useful for the configur-}
Table I. NMR data\(^{[a]}\) of tetrakis(sulfurdiimido) compounds M(NSNR\(^4\))\(_4\) (1a - 3a, 1b, 2b).

<table>
<thead>
<tr>
<th>Compound</th>
<th>(\delta)(^{1}\text{H}) (\text{^13C})</th>
<th>(\delta)(^{29}\text{Si})</th>
<th>(\delta)(^{15}\text{N})</th>
<th>[b]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1a Si(NSN'Bu)(_4)</td>
<td>1.44</td>
<td>29.5/63.0</td>
<td>-74.7</td>
<td>-48.6</td>
</tr>
<tr>
<td>2a Ge(NSN'Bu)(_4) [^{[d]}]</td>
<td>1.31</td>
<td>28.6/63.4</td>
<td>-48.0 (Z)</td>
<td>-71.3 (E)</td>
</tr>
<tr>
<td>3a Sn(NSN'Bu)(_4) [^{[e]}]</td>
<td>1.54</td>
<td>29.9/60.6</td>
<td>-624.0</td>
<td>n.m.</td>
</tr>
<tr>
<td>1b Si(NSNSiMe(_3))(_4)</td>
<td>0.20</td>
<td>0.8</td>
<td>-77.1</td>
<td>-60.7</td>
</tr>
<tr>
<td>2b Ge(NSNSiMe(_3))(_4)</td>
<td>0.09</td>
<td>0.8</td>
<td>+5.2</td>
<td>-49.9</td>
</tr>
</tbody>
</table>

\(^{[a]}\) Measured at +27°C in [D\(_8\)]toluene; coupling constants in Hz: \(^1\text{J}(^{29}\text{Si})\) and \(^2\text{J}(^{29}\text{Si})\) in parentheses; n.m. = not measured; \[^{[b]}\] \(^{15}\text{N}\) NMR methods: A = refocused INEPT pulse sequence with \(^1\text{H}\) decoupling; B = \(^1\text{H}\) inverse-gated decoupling; C = direct measurement, \(^1\text{H}\) coupled; D = direct measurement. \(^{[c]}\) \(^{15}\text{N}\) (\(-40^\circ\text{C}\)) = -49.6 (N'Bu), -83.5 (NSi); \(^{15}\text{N}\) NMR methods: A, D; \[^{[d]}\] \(^{15}\text{N}\) NMR spectrum recorded at -50°C; \[^{[e]}\] Measured at -60°C; all \(^1\text{H}\) and \(^{13}\text{C}\) NMR signals are broad; \(^{[f]}\) \(^{119}\text{Sn}\) CP/MAS = -604.0.

In the case of the germanium derivative 2a, it is evident from all NMR data that several isomers exist in solution. The \(^{15}\text{N}\)(N'Bu) values between -48.0 and -53.6 are in support of the Z positions of this group and the \(^{15}\text{N}\)(NSi) values in the range of -71.3 to -73.6 are indicative of the E positions of these moieties. The \(^{15}\text{N}\) NMR spectra suggest that four isomers with Z/E configuration are present in solution (Fig. 2). The four most likely isomers are (Z/E)\(_4\), (Z/E)\(_3\)(E/Z), (Z/E)\(_2\)(E/Z)\(_2\) and (Z/E)(E/Z)\(_3\) (Scheme 1). The signals of the E/Z parts of the isomers are still broad at -50°C and therefore not all resonances for the four isomers could be observed. It seems that the moieties with E/Z configuration are even less rigid than those with Z/E configuration. The same phenomenon was observed in the case of tris(sulfurdiimido)germanium compounds [6b].

The tetrakis(N-trimethylsilylsulfurdiimido)silicon and -germanium derivatives 1b and 2b possess a more fluxional character in solution than the corresponding N'Bu compounds 1a and 2a. The
Tetrakis(sulfurdiimido)silane, -germane and -stannane

Table II. NMR data\textsuperscript{[a]} of compounds 4a - 6a.

<table>
<thead>
<tr>
<th>Compound</th>
<th>(\delta^1\text{H}^\text{tBu} )</th>
<th>(\delta^{13}\text{C}^\text{tBu} )</th>
<th>(\delta^{29}\text{Si} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cl(_3)Si(NSSi\text{Bu})(_4)</td>
<td>4a</td>
<td>1.24</td>
<td>29.1/65.1</td>
</tr>
<tr>
<td>Cl(_2)Si(NSSi\text{Bu})(_2)</td>
<td>5a</td>
<td>1.32</td>
<td>29.3/64.2</td>
</tr>
<tr>
<td>ClSi(NSSi\text{Bu})(_3)</td>
<td>6a</td>
<td>1.38</td>
<td>29.4/63.5</td>
</tr>
</tbody>
</table>

\textsuperscript{[a]} Measured at +27°C, in C\(_6\)D\(_6\).

usual trend for the influence exerted by electronegative substituents. The same trend is found in the series Cl\(_3\)Si(NSSi\text{Bu}) (4a, \(\delta^{29}\text{Si} -38.7\)), Cl\(_2\)Si(NSSi\text{Bu})\(_2\) (5a, \(\delta^{29}\text{Si} -53.3\)) , ClSi(NSSi\text{Bu})\(_3\) (6a, \(\delta^{29}\text{Si} -65.2\)) and Si(NSSi\text{Bu})\(_4\) (1a, \(\delta^{29}\text{Si} -74.7\)) (Table II, Fig. 4). Furthermore there is a systematic shift of the \(29\text{Si}(\text{SiMe}_3)\) resonances towards lower frequencies when an increasing number of NNSiMe\(_3\) groups is attached to the central element [Me\(_3\)M(NNSiMe\(_3\))\(_4\): \(\delta^{29}\text{Si} 1.6\) (M = Si), 0.1 (M = Ge) \[12a\]; Me\(_2\)M(NNSiMe\(_3\))\(_2\): \(\delta^{29}\text{Si} 2.8\) (M = Si), 1.3 (M = Ge) \[6a\]; MeM(NNSiMe\(_3\))\(_3\): \(\delta^{29}\text{Si} 4.0\) (M = Si), 2.8 (M = Ge) \[6b\]; M(NNSiMe\(_3\))\(_4\): \(\delta^{29}\text{Si} 4.6\) (M = Si, 1b), 5.2 (M = Ge, 2b)].

According to the \(1^\text{H}, 13^\text{C}\) and \(119^\text{Sn}\) NMR spectra the structure of the tin derivative 3a is still fluxional at -80°C. The \(\delta^{119}\text{Sn}\) value (-624.0) of tetrakis(N-\text{tert}-butyl-sulfurdiimido)tin 3a falls in the typical range for hexa-coordinated tin atoms \[18\]. The \(119^\text{Sn}\) resonance is broad both at room temperature and at lower temperatures, possibly as a result of dynamic processes. It seems likely that the increase of the coordination number at the tin atom is caused by intermolecular association \textit{via} the free electron pairs at the nitrogen atoms of the NSN system, analogous to bis- and tris(sulfurdiimido)tin compounds \[6b\]. The structure of 3a in the solid state must be similar as in solution, since the solid-state \(119^\text{Sn}\) CP/MAS NMR spectrum reveals an isotropic \(\delta^{119}\text{Sn}\) value of -604.0, close to the value in solution (-624.0).

Fig. 1. A: 50.6 MHz \(15^\text{N}\{1^\text{H} \text{ inverse-gated}\} \) NMR spectrum of Si(NSSi\text{Bu})\(_4\) (1a), measured in [D\(_6\)]toluene at 300K. Note the fairly broad resonance signals with \(h_{1/2} = 15\) Hz. B: 30.4 MHz \(15^\text{N}\{1^\text{H}\} \) NMR spectrum of Si(NSSi\text{Bu})\(_4\) (1a), measured in [D\(_6\)]toluene at 233 K.

\(\delta^{15}\text{N}(\text{NSiMe}_3)\) values of 1b and 2b are typical averaged values \[12\], indicating fast E/Z-Z/E isomerisation processes (Fig. 3).

\(29^\text{Si}\) and \(119^\text{Sn}\) NMR data

Comparing different sulfurdiimido compounds we have observed that the \(29\text{Si}\) nuclear shielding of the central silicon atom increases with an increasing number of sulfurdiimido groups [Me\(_3\)Si(NNSiMe\(_3\))\(_4\): \(\delta^{29}\text{Si} 1.6 \[12a\]; Me\(_2\)Si(NNSiMe\(_3\))\(_2\): \(\delta^{29}\text{Si} -16.2 \[6a\]; MeSi(NNSiMe\(_3\))\(_3\): \(\delta^{29}\text{Si} -44.5 \[6b\]; Si(NNSiMe\(_3\))\(_4\) (1b): \(\delta^{29}\text{Si} -77.1\)], which is the
Fig. 2. 30.4 MHz $^{15}$N NMR spectrum ($^1$H coupled) of Ge(NSN'Bu)$_4$ (2a), measured in [D$_6$]toluene at 223 K.

Fig. 4. 59.6 MHz $^{29}$Si NMR spectrum ($^1$H coupled; C$_6$D$_6$ solution; 300K) of a mixture of Cl$_{4-n}$Si(NSN'Bu)$_n$ ($n = 1-4$), obtained from the 1:1 reaction of K[(NSN)Bu] with SiCl$_4$.

is therefore suggested that the silicon atoms are penta-coordinated due to an additional coordinative N-Si bond [6a]. The signal for the central Si atom in 7a lies at fairly high frequency, when compared with 1a. This $^{29}$Si deshielding is caused by...

$^{15}$N and $^{29}$Si NMR data of the heterocycles 7a and 8a

Two $^{29}$Si NMR signals are observed for NSiCl$_3$ groups in 7a and 8a at rather low frequencies. It...
Table III. NMR data\(^{[a]}\) of the compounds 7a and 8a.

<table>
<thead>
<tr>
<th>Compound (^{[b]})</th>
<th>(\delta^{13})C</th>
<th>(\delta^{29})Si</th>
<th>(\delta^{15})N</th>
</tr>
</thead>
<tbody>
<tr>
<td>7a</td>
<td>29.6/63.9</td>
<td>-20.0</td>
<td>-83.1</td>
</tr>
<tr>
<td></td>
<td>29.6/64.4</td>
<td>-20.6</td>
<td>-83.9</td>
</tr>
<tr>
<td></td>
<td>31.0/63.4</td>
<td>-20.6</td>
<td>-83.1</td>
</tr>
<tr>
<td>8a</td>
<td>29.5/63.9</td>
<td>-20.6</td>
<td>-83.1</td>
</tr>
<tr>
<td></td>
<td>29.6/64.5</td>
<td>-20.6</td>
<td>-83.1</td>
</tr>
<tr>
<td></td>
<td>31.1/63.1</td>
<td>-20.6</td>
<td>-83.1</td>
</tr>
</tbody>
</table>

\(^{[a]}\) Measured at +27°C, in [D\(_8\)]toluene; \(^{15}\)N NMR spectra were recorded at -50°C, direct measurement, \(^1\)H coupled; \(br\) = broad; \(s\) = singulet; \(^{[b]}\) \(\delta^{1}\)H[corresponding \(\delta^{13}\)C value]: 1.34 (s, 18H, \(^1\)Bu) [29.6/64.4], 1.38 (s, 9H, \(^1\)Bu) [29.6/63.9], 1.51 (s, 9H, \(^1\)Bu) [31.0/63.4]; \(^{14}\)N: -25.0 (br, N\(^{1}\)Bu), -100.0 (br, NSi), region between -270.0 and -320.0 (br, NSiCl\(_3\)).

However, the \(^{14}\)N NMR spectra of 7a and 8a show very broad signals in the region between \(-270\) and \(-320\) for these groups. Apparently both compounds are still involved in dynamic processes; thus it was not possible to detect any \(^{15}\)N resonances at room temperature or to measure the \(^{15}\)N NMR signals by polarisation transfer in order to assign definitely the signals of the =N\(^{1}\)Bu groups.

**Experimental**

All compounds were handled in an atmosphere of dry argon, and carefully dried solvents were used for all syntheses and the preparation of samples for NMR measurements. The potassium salts K[(NSN)R] (R = \(^1\)Bu, SiMe\(_3\)) \(^{[5]}\) were prepared according to literature procedures. Triethylborane and hexachlorodisilane were
commercial products and used without further purification. NMR instruments (all equipped with multinuclear units and variable-temperature control units) for liquid state measurements: JEOL FX 90Q (119Sn NMR), Bruker ARX 250, Bruker AC 300 and Bruker AM 500 (1H, 11B, 13C, 15N, 29Si, 119Sn NMR). Chemical shifts are given with respect to Me2Si [δ1H (C6D6H) = 7.15, (C6D6Si2H) = 2.03; δ13C (C6D6) = 128.0, (C6D6C2H) = 20.4; δ29Si = 0 for δ (29Si) = 19.867184 MHz], EtzO-BF3 [δ11B = 0 for δ (11B) = 32.083971 MHz], neat MeONO2 [δ13N = 0 for δ (13N) = 10.136767 MHz, and δ (14N) = 7.226455 MHz] and neat Me2Sn [δ119Sn = 0 for δ (119Sn) = 37.290665 MHz]. The mutual assignments of 1H, 13C and 15N resonances to the different isomers were achieved by 2D13C/1H and 2D15N/1H heteronuclear shift correlations [based on 1J(13C1H), 2J(13C1H) and 3J(15N1H)]. A Bruker MSL 300 instrument (equipped with a multinuclear double-bearing probe head) served for the solid state 119Sn CP/MAS NMR measurement; the sample was packed in an air-tight insert [20] fitting exactly into the commercial ZrO2 rotor. The spectrum was run at two different spinning speeds for assignment of the isotropic δ value. Electron impact (EI) mass spectra (70 eV): Varian MAT CH 7 (EI-MS, 70 eV) with direct inlet.

Tetrakis(sulfurdiimido) compounds M(NSN)4 (M = Si, Ge, Sn; R = Bu) - 3a; M = Si, Ge; R = SiMe3, 1b,2b).

General procedure:

A suspension of 6 mmol of the potassium sulfurdiimido salt, K[(NSN)R], in DME (30 ml) was added to the element tetrachloride (1.5 mmol) in hexane (20 ml) at -78°C. The mixture was stirred for 30 minutes at -78°C and then allowed to reach room temperature. After filtration the solvent was removed in vacuo, and the compounds were left as yellow to orange oils (1b,2a,b) or orange solids (1a, m.p. 112°C, 3a, m.p. 253°C). In repeated experiments, yields were found to be 70-90% for the N-tert-butyl compounds and 30-70% for the N-trimethylsilyl compounds.

Si(NSN)4Bu (1a); C18H36N8S4Si; M = 496.862 g/mol; EI-MS: m/z (%) = 496 (1) [M1], 439 (2) [M1-57], 425 (78), 379 (81), 369 (60), 313 (60), 257 (100), 211 (70), 182 (20), 150 (15), 136 (23), 57 (1).

Si(NSNSiMe)4 (1b); C24H56N8S4Si3; M = 561.162 g/mol; EI-MS: m/z (%) = 545 (15), 459 (5.5), 339 (6), 294 (5.5), 278 (5), 73 (100).

Synthesis of Cl4-n Si(NSNSiBu)n (n = 1, 4a; n = 2, 5a; n = 3, 6a)

A suspension of the potassium salt K[(NSN)4Bu] (6 mmol) in hexane (50 ml) was added to silicon tetrachloride (6 mmol, 3 mmol or 2 mmol) in hexane (20 ml) at room temperature. The mixture was stirred for 4 h, and after filtering off insoluble material the solvent was removed in vacuo. In each case, a mixture of sulfur diimides was left as a yellow to orange oil.

Reactions of R'M(NSN)4Bu3 with hexachlorodisilane:

Synthesis of 7a and 8a. General procedure:

A suspension of 2 mmol of R'M(NSN)4Bu3 (M = Si, R' = NSNBu, 1a; M = Ge, R' = Me) in 40 ml of hexane was combined with a solution of 2 mmol or 8 mmol, respectively, of hexachlorodisilane in 30 ml of hexane at 0°C. The mixture was allowed to reach room temperature and then stirred for additional 24 h. Finally the solvent was removed in high vacuum. 7a was obtained as an orange, 8a as a yellow oil.

Reactions of tetrakis(N-tert-butylsulfurdiimido)tin 3a with triethylborane (NMR tube)

A solution of 1 mmol of 3a in 2 ml of toluene/[D4]toluene was combined with triethylborane (4 or 8 mmoles, respectively) at -78°C. The mixture was kept for 30 min at this temperature and then allowed to reach r. t. 119Sn NMR showed that 3a was no longer present, and 11B NMR revealed the signals of decomposition products such as the aminoborane Et3BH2Bu (9). By trapping all volatile material, the aminoborane and Et3BH2Bu (10) could be identified by their NMR data.

Et3BNH2Bu (9): δ11B : 46.6 (h1/2 = 110 Hz); δ14N: -250.0; δ13C([D4]toluene, -50°C): 33.2, 52.1 (tBu), 13.1, 11.5 (br, CH2), 10.4, 10.1 (CH3).

Et3B-NH2Bu (10): δ11B : -2.8 (h1/2 = 183 Hz); δ14N: -335.0 (br); δ13C: 13.7 (br, CH2), 10.1 (CH3), 29.0/51.1 (tBu); δ1H: 0.40 (q, 7.7 Hz, CH2), 0.99 (t, 7.7 Hz, CH3), 1.24 (s, tBu), 1.99 (br, NH2); all spectra recorded at +27°C, in C6D6; br = broad; q = quadruplet; t = triplet; s = singulet.

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