# The addition of organometallic reagents to tetramesityldigermene

**Kyle L. Fujdala, David W.K. Gracey, Erica F. Wong, and Kim M. Baines**

**Abstract**: The thermolysis and photolysis of hexamesitylcyclotrigermane in the presence of ethylmagnesium bromide has been investigated. Under photochemical conditions, ethyldimesitylgermane, 1,2-diethyl-1,1,2-trimesityldigermane and ethyl-1,1,2,2-tetramesityldigermane were isolated and, under thermal conditions, 1,2,2-triethyl-1,1-dimesityldigermane and 2,2-diethyl-1,1,1-trimesityldigermane were isolated. The photolysis of hexamesitylcyclotrigermane in the presence of methyllithium has also been investigated. In both cases, the organometallic reagent adds to tetramesityldigermene and dimesitylgermylene formed by photochemical or thermal cleavage of the cyclotrigermane. In the case of the addition of the Grignard reagent, the resulting germyl Grignard reagent undergoes a facile ligand exchange reaction.

*Key words*: digermene, germylene, Grignard reagents, alkyllithium reagents, germylmagnesium compounds, germyllithium compounds.

**Résumé** : On a étudié la thermolyse et la photolyse de l'hexamésitylcyclotrigermane en présence de bromure d'éthylmagnésium. Dans les conditions photochimiques, on a isolé de l'éthyldimésitylgermane, du 1,2-diéthyl-1,1,2-trimésityldigermane et du éthyl-1,1,2,2-tétramésityldigermane alors, que dans des conditions thermiques, on a isolé du 1,2,2-triéthyl-1,1-dimésityldigermane et du 2,2-diéthyl-1,1,1-triméstiyldigermane. On a aussi examiné la photolyse de l'hexamésitylcyclotrigermane en présence de méthyllithium. Dans les deux cas, le réactif organométallique s'additionne au tétraméthyldigermène et au dimésitylgermylène qui se forment par clivage photochimique ou thermique du cyclotrigermane. Dans le cas de l'addition du réactif de Grignard, le réactif germylmagnésien qui en résulte subit une réaction facile d'échange de ligands.

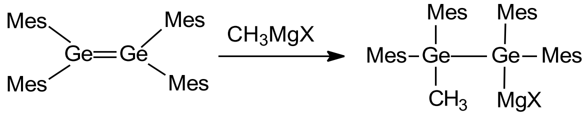
*Mots clés* : digermène, germylène, réactifs de Grignard, réactifs alkyllithium, composés germylmagnésium, composés germyllithium.

[Traduit par la Rédaction]

## Introduction

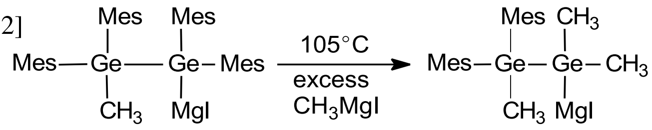
The reactivity of digermenes has been of interest since stable and relatively stable derivatives of these compounds were first synthesized twenty years ago (1). Despite two decades of research in this area, the addition of organometallic reagents remains a relatively unexplored area of digermene chemistry. We have reported that the addition of methylmagnesium halide to $Mes_2Ge{=}GeMes_2$ (Mes = mesityl = 2,4,6-trimethylphenyl) gives (dimesitylmethylgermyl)dimesitylgermylmagnesium bromide in good yield (eq. [1]) (2).

[1]



Furthermore, we discovered that thermolysis of the resulting germyl Grignard reagent in the presence of excess MeMgX leads to a facile ligand exchange reaction in which the mesityl groups on the germanium bonded to the magnesium were exchanged for methyl groups (eq. [2]).

[2]



$$\mathrm{Mes{-}\overset{Mes}{\underset{CH_3}{Ge}}{-}\overset{Mes}{\underset{MgI}{Ge}}{-}Mes} \xrightarrow[\text{excess } \mathrm{CH_3MgI}]{105\,^{\circ}\mathrm{C}} \mathrm{Mes{-}\overset{Mes}{\underset{CH_3}{Ge}}{-}\overset{CH_3}{\underset{MgI}{Ge}}{-}CH_3}$$

A mechanism involving the α-elimination of MesMgX followed by the addition of MeMgX to an intermediate germylene has been proposed to explain the observed ligand exchange (Scheme 1). Evidence in support of the proposed mechanism was provided by trapping the initially formed intermediate germylene with 2,3-dimethylbutadiene. We have also found that methylmagnesium halide adds regioselectively to germasilenes giving germyl Grignard reagents, which undergo an analogous methyl-for-mesityl exchange (2, 3). To investigate not only the generality of the Grignard addition to digermenes and the generality of the ligand exchange reaction, we now describe the addition of ethylmagnesium bromide to tetramesityldigermene generated either photochemically or thermally from hexamesitylcyclotrigermane. Furthermore, we also report on the addition of methyllithium to tetramesityldigermene and the thermal behaviour of the products obtained therefrom.

Received 5 February 2002. Published on the NRC Research Press Web site at http://canjchem.nrc.ca on 11 September 2002.

*Dedicated to Professor Tristram Chivers in recognition of his contributions to the field of inorganic chemistry.*

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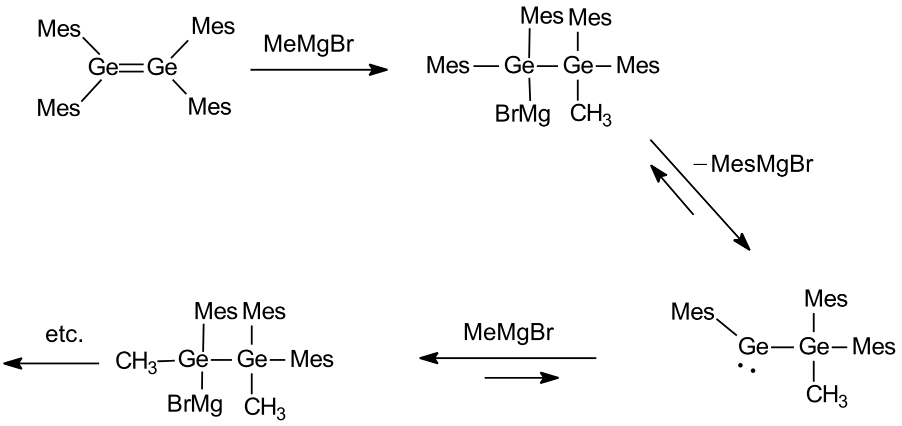
DOI: 10.1139/V02-128

**Scheme 1.**



## Results and discussion

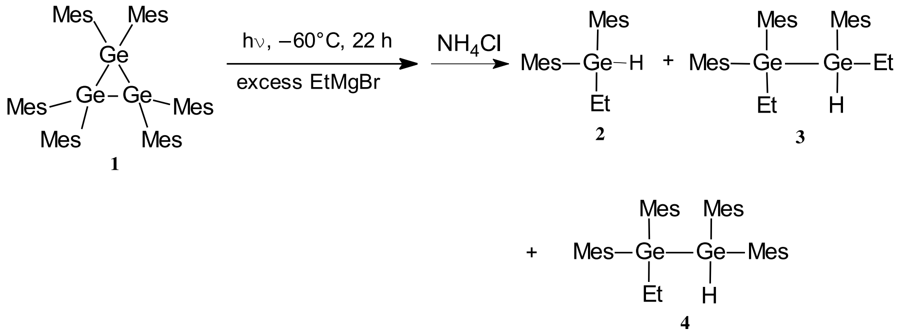
The photolysis of hexamesitylcyclotrigermane (**1**) in toluene at –60°C in the presence of excess ethylmagnesium bromide, followed by hydrolysis using ammonium chloride, led to the isolation of three products: ethyldimesitylgermane (**2**), 1,2-diethyl-1,1,2-trimesityldigermane (**3**), and ethyl-1,1,2,2-tetramesityldigermane (**4**) in a 7.8:2.8:1 ratio (eq. [3]). All new compounds were characterized by $^1$H, $^{13}$C NMR, and IR spectroscopy and mass spectrometry, the details of which are given in the experimental section. In general, the greater shift dispersion of the $^{13}$C NMR spectrum of a given compound was useful in determining the number of different mesityl or ethyl groups and the $^1$H NMR spectrum gave the ratio of mesityl:ethyl:GeH groups. Finally, the high resolution mass spectrum was critical in determining the molecular formula of the compound.

The formation of compounds **2**–**4** can be understood on the basis of our previous mechanistic proposal. The photolysis of hexamesitylcyclotrigermane results in the formation of dimesitylgermylene and tetramesityldigermene (4). The formation of **2** is most easily accounted for by the quenching of ethyldimesitylgermylmagnesium bromide formed by the addition of ethylmagnesium bromide to dimesitylgermylene. Similarly, compound **4** is apparently derived from addition of the ethyl Grignard reagent to tetramesityldigermene to give the germyl Grignard reagent **5** followed by hydrolysis (Scheme 2). Compound **3** is believed to arise from **5** via an α-elimination of mesitylmagnesium bromide in the cold to give the germylgermylene **6**. Ethylmagnesium bromide could then add to **6**, giving **3** after hydrolysis (Scheme 2).

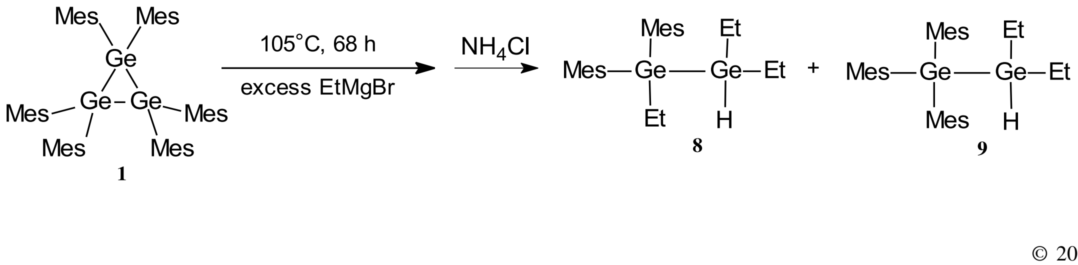
The reactivity observed in the addition of EtMgBr to $Mes_2Ge{=}GeMes_2$ parallels that for the addition of the methyl analog. However, unlike the methyl analog where a ligand exchange was observed only upon thermolysis of the germyl Grignard reagent at 105°C, the ethyl-for-mesityl exchange was observed at low temperature. Presumably, the more facile ligand exchange is the result of greater steric congestion in the germyl Grignard reagent, which is subsequently relieved upon elimination of mesitylmagnesium bromide. Ligand exchange in $t$-$Bu_2MeSi$-$GeMes_2MgX$ was also observed at lower temperatures compared to ligand exchange in $Mes_2MeSi$-$GeMes_2MgX$ (3).

Thermolysis of hexamesitylcyclotrigermane for extended periods of time gave two major products: 1,2,2-triethyl-1,1-dimesityldigermane (**8**) and 2,2-diethyl-1,1,1-trimesityldigermane (**9**) in a 1:1 to 2:1 ratio (eq. [4]). Compounds **8** and **9** were identified using standard spectroscopic techniques.

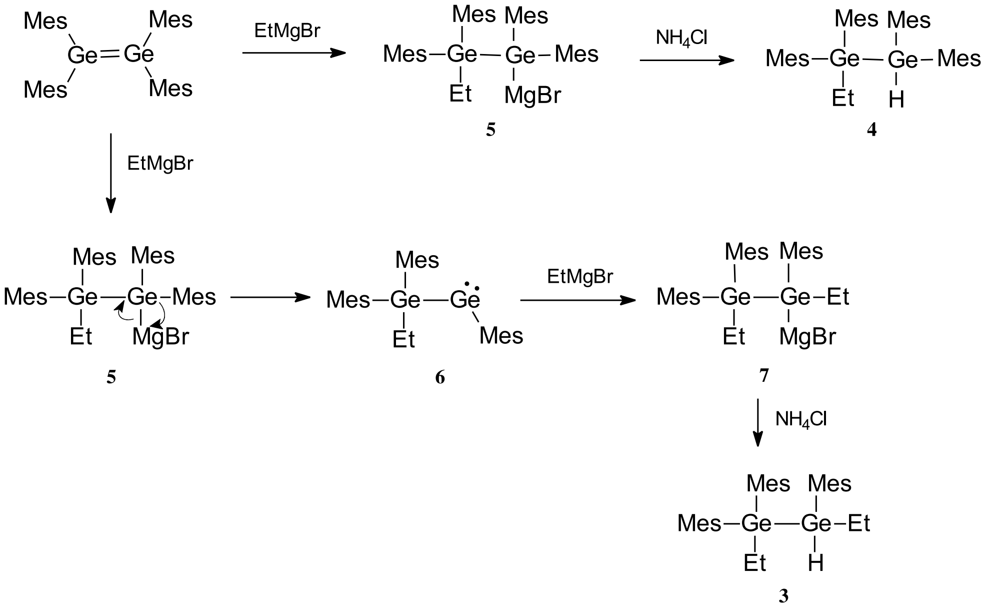
[3]



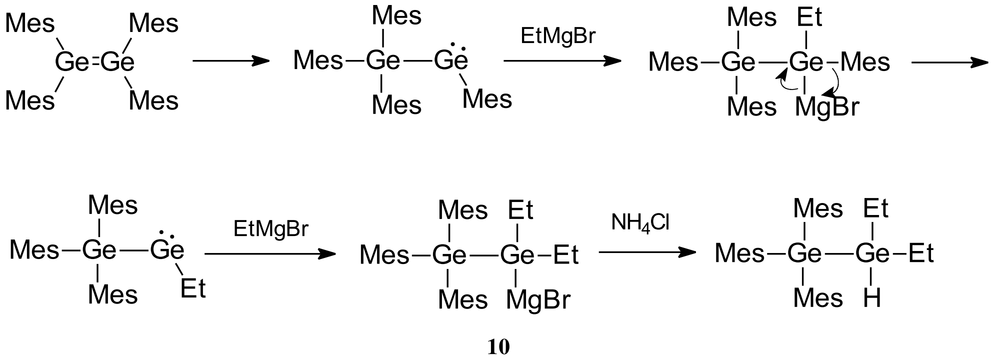
[4]



**Scheme 2.**



**Scheme 3.**



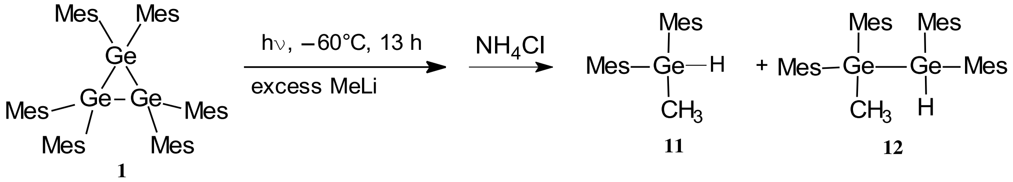
Formation of these two compounds can be easily explained based on our current mechanism. Compound **8** appears to be the result of an α-elimination of mesitylmagnesium bromide from germyl Grignard **7** to give ethyldimesitylgermylethylgermylene, followed by the addition of ethylmagnesium bromide to give **8** after hydrolysis. The formation of compound **9** is interesting since an analogous product was not observed in the addition of MeMgBr to the digermene. Tetramesityldigermene is known to undergo a facile mesityl migration to give trimesitylgermylmesitylgermylene (5). Addition of ethylmagnesium bromide to this germylene, followed by α-elimination of mesitylmagnesium bromide and the addition of a second equivalent of ethylmagnesium bromide, would result in the formation of germyl Grignard **10**, which upon quenching would give the observed product (Scheme 3). As in the analogous reaction with methylmagnesium halide, no apparent product derived from dimesitylgermylene was isolated. Presumably, ethylmagnesium bromide adds to Mes$_2$Ge: to give ethyldimesitylgermylmagnesium bromide; however, under the reaction conditions, complete ligand exchange occurs to give triethylgermane (after hydrolysis), which was not isolated. Thermolysis for shorter periods of time (2 h) gave a more complex reaction mixture. In addition to compounds **8** and **9**, compounds **2** and **3** were formed, providing evidence for the intermediate formation of ethyldimesitylgermylmagnesium bromide and the germyl Grignard **7**.

Again, there are both similarities and differences observed in comparison to the analogous thermolysis of hexamesitylcyclotrigermane with the methyl Grignard reagent. A facile ligand exchange was also observed in this system; however, the rate of addition of the bulkier ethyl group appears to compete with the 1,2-mesityl shift at 105°C.

Given the interesting reactivity of germyl Grignard reagents and the absence of any reported additions of alkyllithium reagents to Group 14 dimetallenes, we also investigated the addition of methyllithium to Mes$_2$Ge=GeMes$_2$ under similar conditions.

The photolysis of hexamesitylcyclotrigermane in the presence of methyllithium at –60°C followed by quenching the reaction mixture with a solution of ammonium chloride gave two products: methyl-1,1,2,2-tetramesityldigermane (2) (**11**)

[5]



and dimesitylmethylgermane (6) (**12**) in modest isolated yields (eq. [5]). The compounds were identified by comparison of their spectroscopic data to that of authentic samples. Given that methyllithium does not react with the cyclotrigermane in the dark, it is reasonable to assume that methyllithium adds to both tetramesityldigermene and dimesitylgermylene (generated by photolysis of the cyclotrigermane) in the same manner as Grignard reagents. Thermolysis of hexamesitylcyclotrigermane in the presence of methyllithium gave only a complex reaction mixture, in contrast to the analogous reaction with methyl Grignard reagent.

In summary, the results described here for the addition of ethylmagnesium bromide to tetramesityldigermene and dimesitylgermylene parallel those previously observed for the addition of methylmagnesium bromide to digermenes and germasilenes. Hence, it has become increasingly apparent that the addition of Grignard reagents to both digermenes and germylenes and the ligand exchange of germyl Grignard reagents are all general reactions. Furthermore, alkyllithium reagents also appear to readily add to digermenes and germylenes, although thermolysis of the resulting mixture of germyllithium reagents does not lead to isolable species. We continue to explore the addition of a variety of organometallic reagents to digermenes and the chemistry of the resulting germyl metallic reagents.

## Experimental

All experiments were carried out in flame-dried glassware under an inert argon atmosphere. Toluene, diethyl ether, and THF were freshly distilled from sodium/benzophenone. Ethylmagnesium bromide was prepared from commercially available ethyl bromide and magnesium (Lancaster Chemical Co.). The concentration of the Grignard reagent was determined by titration, prior to use. Chromatographic separations and purifications were carried out using a Chromatotron (Harrison Research) or on conventional silica gel preparative plates. Photolyses were carried out at 350 nm in a Rayonet photochemical reactor. The solution to be photolyzed was placed in a Schlenk tube and suspended in an immersion well filled with methanol at –60°C. The temperature of the methanol was maintained using a NESLAB ULT-80 low temperature bath circulator.

NMR spectra were recorded on a Varian Gemini 200, an XL-300, or a Varian Gemini 300 NMR spectrometer using benzene-$d_6$ as a solvent. IR spectra were recorded ($cm^{-1}$) as thin films on a PerkinElmer 2000 FT-IR spectrometer. A Finnegan MAT model 8200 mass spectrometer, with an ionizing voltage of 70 eV, was used to obtain electron impact mass spectra (recorded in mass-to-charge units (*m/z*) with ion identity and peak intensities relative to the base peak in parentheses).

### Photolysis of hexamesitylcyclotrigermane in the presence of excess ethylmagnesium bromide

A solution of $Ge_3Mes_6$ (48 mg, 0.052 mmol) and freshly prepared ethylmagnesium bromide (1 mL, 1.5 mmol in $Et_2O$) in toluene (3 mL) was photolyzed (350 nm) for 22 h at –60°C. At the end of the photolysis, the solution was bright yellow-orange. The cold reaction mixture was quenched with aqueous ammonium chloride. The aqueous and organic phases were then separated. The aqueous phase was extracted with three portions of hexanes and the extracts were combined with the organic phase. The organic phase was dried over anhydrous magnesium sulfate and then filtered to remove the solid. Removal of the solvent in vacuo yielded a clear, colourless oil. $^1H$ NMR spectroscopic analysis of the reaction mixture showed three major products in a 7.8:1:2.8 ratio (**2**:**4**:**3**). The reaction mixture was separated by TLC using 8% $CH_2Cl_2$ in hexanes as the eluent to give ethyldimesitylgermane (**2**, 15.3 mg, 87%) and ethyl-1,1,2,2-tetramesityldigermane (**4**, 8.5 mg, 25%). Compound **3** was not isolated in pure form.

*Ethyldimesitylgermane (2)*:

mp 76.5–78°C. IR (thin film) ($cm^{-1}$): 2045 (Ge-H). $^1H$ NMR (ppm) δ: 6.73 (s, 4H, Ar-H), 5.44 (t, *J* = 4.2 Hz, 1H, Ge-H), 2.35 (s, 12H, Mes *o*-$CH_3$), 2.11 (s, 6H, Mes *p*-$CH_3$), 1.34–1.45 (m, 2H, Et-$CH_2$), 1.12 (t, *J* = 7.7 Hz, 3H, Et-$CH_3$). $^{13}C$ NMR (ppm) δ: 143.46 (Mes-C), 138.12 (Mes-C), 134.25 (Mes-C), 129.08 (Mes-CH), 23.89 (Mes *o*-$CH_3$), 21.02 (Mes *p*-$CH_3$), 11.58 ($CH_2$), 11.26 ($CH_3$). MS *m/z*: 342 ($[M]^+$, 10), 313 ([$HGeMes_2$], 100), 222 ([GeEtMes], 34), 191 ([GeMes], 42), 119 ([Mes], 18), 105 (25). HR-MS calcd. for $C_{20}H_{28}{}^{74}Ge$: 342.1403; found: 342.1393.

*Ethyl-1,1,2,2-tetramesityldigermane (4)*:

IR (thin film) ($cm^{-1}$): 2032 (Ge-H). $^1H$ NMR (ppm) δ: 6.72 (s, 4H, Ar-H), 6.70 (s, 4H, Ar-H), 5.92 (s, 1H, Ge-H), 2.36 (s, 12H, Mes *o*-$CH_3$), 2.21 (s, 12H, Mes *o*-$CH_3$), 2.10 (s, 6H, Mes *p*-$CH_3$), 2.09 (s, 6H, Mes *p*-$CH_3$), 1.92 (q, *J* = 7.8 Hz, 2H, Et-$CH_2$), 1.1 (t, *J* = 7.8 Hz, 3H, Et-$CH_3$). $^{13}C$ NMR (ppm) δ: 143.80, 143.73, 137.83, 137.48, 137.45, 136.58 (all C), 129.44 (Mes-CH), 128.96 (Mes-CH), 24.90, 24.85, 21.00, 20.91 (all $CH_3$), 15.20 ($CH_2$), 11.06 ($CH_3$). CI-MS *m/z* (isobutane): 651 ($[M - H]^+$, 5), 533 ($[M - Mes]^+$, 13), 431 ([$GeMes_3$], 17), 341 ([$GeMes_2Et$], 100), 311 ([$GeMes_2$ – H], 22), 223 ([GeEtMesH], 18), 191 ([GeMes], 10), 121 ([MesH + H], 88). HR-MS calcd. for $C_{38}H_{50}{}^{72}Ge^{74}Ge$-H: 651.2267; found: 651.2276.

### Thermolysis of hexamesitylcyclotrigermane in the presence of excess ethylmagnesium bromide

A solution of $Ge_3Mes_6$ (50.3 mg, 0.055 mmol) and freshly prepared ethylmagnesium bromide (1 mL, 1.65 mmol in $Et_2O$) in toluene (4 mL) was heated for 68 h at 105°C. After a few minutes of heating, the clear and colourless solution became light yellow and some solid precipitated from solution. At the end of the thermolysis, the solution was cloudy grey in colour with a white solid precipitate. Following the thermolysis, the reaction mixture was quenched with aqueous ammonium chloride. The aqueous and organic phases were then separated. The aqueous phase was extracted with three portions of hexanes and the extracts were combined with the organic phase. The organic phase was dried over anhydrous magnesium sulfate and then filtered to remove the solid. Removal of the solvent in vacuo yielded a clear, colourless oil. $^1$H NMR spectroscopic analysis of the reaction mixture showed two major products (**8** and **9**) in a 1:1 or 2:1 ratio. The product mixture was separated by TLC using 10% $CH_2Cl_2$ in hexanes as the eluent to give 2,2-diethyl-1,1,1-trimesityldigermane (**9**, 1.1 mg, 3.8%) and 1,2,2-triethyl-1,1-dimesityldigermane (**8**, 4.8 mg, 18.5%).[2]

*1,2,2-Triethyl-1,1-dimesityldigermane (8)*:

Oily solid. IR (thin film) (cm$^{-1}$): 1996 (Ge-H). $^1$H NMR (ppm) δ: 6.72 (s, 4H, Mes-H), 4.28 (m, 1H, Ge-H), 2.35 (s, 12H, Mes *o*-$CH_3$), 2.11 (s, 6H, Mes *p*-$CH_3$), 1.65 (q, *J* = 7.8 Hz, 2H, Et-$CH_2$), 0.95–1.15 (m, 13H, 2 Et + Et-$CH_3$). $^{13}$C NMR (ppm) δ: 142.80, 137.64, 137.57, 129.26 (CH), 24.89 ($CH_3$), 20.94 ($CH_3$), 14.86 ($CH_2$), 11.98 ($CH_3$), 11.03 ($CH_3$), 5.76 ($CH_2$). MS *m/z*: 472 ([M]$^+$, 2), 443 ([M – Et]$^+$, 4), 415 ([M – 2Et + H]$^+$, 2), 385 ([M – 3Et]$^+$, 1), 341 ([GeMes$_2$Et], 100), 313 ([GeMes$_2$ + H], 20), 221 ([GeEtMes – H], 6), 193 ([GeMes], 36), 119 ([Mes], 14). HR-MS calcd. for $C_{24}H_{43}{}^{72}Ge^{74}Ge$: 472.1406; found: 472.1390.

*2,2-Diethyl-1,1,1-trimesityldigermane (9)*:

mp 176–180°C. IR (thin film) (cm$^{-1}$): 1982 (Ge-H). $^1$H NMR (ppm) δ: 6.75 (s, 6H, Mes-H), 4.18 (tt, *J* = 1.0 Hz, *J* = 5.0 Hz, 1H, Ge-H), 2.27 (s, 18H, Mes *o*-$CH_3$), 2.11 (s, 9H, Mes *p*-$CH_3$), 1.21 (pseudo t, 6H, Et-$CH_3$), 0.8–1.1 (m, 4H, Et-$CH_2$). $^{13}$C NMR (ppm) δ: 143.82, 138.86, 137.85, 129.57, 24.92, 20.95, 13.42, 8.14 ($CH_2$). MS *m/z*: 561 ([M – H]$^+$, 17), 533 ([M – Et]$^+$, 4), 443 ([M – Mes]$^+$, 47), 431 ([GeMes$_3$], 100), 311 ([GeMes$_2$ – H], 7), 191 ([GeMes], 12), 119 ([Mes], 10). HR-MS calcd. for $C_{31}H_{43}{}^{72}Ge^{74}Ge$ ([M – H]): 561.1797; found: 561.1807.

The experiment was repeated (49 mg $Ge_3Mes_6$, 1 mL of EtMgBr in $Et_2O$, toluene (1.5 mmol, 3 mL), 105°C), except the reaction mixture was heated for 2 h only. Under these conditions the following products were obtained: Mes$_3$Ge-GeHEt$_2$ (**9**), Mes$_2$GeEtH (**2**), Mes$_2$EtGe-GeEt$_2$H (**8**), and a fourth product tentatively identified as Mes$_2$EtGe-GeHEtMes (**3**) in a 3.3:2.9:1:1.2 ratio. The products were separated by TLC using 6% $CH_2Cl_2$ in hexanes as the eluent. Compound **3** was obtained as a mixture with Mes$_3$Ge-GeHEt$_2$.

*1,2-Diethyl-1,1,2-trimesityldigermane (3)*:

$^1$H NMR (ppm) δ: 6.74 (s), 6.70 (s, 2H, Mes-H), 5.21 (dd, *J* = 3 Hz, *J* = 6 Hz, 1H, Ge-H), 2.31 (s, 6H, Mes $CH_3$), 2.20 (s, 6H, Mes $CH_3$), 2.18 (s, 6H, Mes $CH_3$), 2.12 (s, 6H, Mes $CH_3$), 2.11 (s, 3H, Mes $CH_3$), 1–1.8 (m, Et). $^{13}$C NMR (ppm) δ: 144.20, 142.99, 138.50, 137.65, 137.60, 133.33, 129.25 (CH), 129.17 (CH), 128.81 (CH), 25.05 ($CH_3$), 24.91 ($CH_3$), 24.88 ($CH_3$), 20.94 ($CH_3$), 14.69 ($CH_2$), 12.62 ($CH_3$), 11.00 ($CH_3$), 9.87 ($CH_2$). MS *m/z*: 562 ([M]$^+$).

### Photolysis of hexamesitylcyclotrigermane in the presence of methyllithium

Methyllithium (1.0 mL of a 1.4 M solution in diethyl ether, 1.4 mmol) was added to a Schlenk tube containing a clear, pale yellow solution of $Ge_3Mes_6$ (53 mg, 0.057 mmol) dissolved in toluene (4 mL). The Schlenk tube was then sealed and placed in an immersion well, cooled to –60°C, and then irradiated for 13 h. After photolysis, the clear, deep yellow solution was warmed to room temperature and was quenched with aqueous ammonium chloride. The aqueous and organic phases were then separated. The aqueous phase was extracted with four portions of hexanes and the extracts were combined with the organic phase. The organic phase was washed three times with water and then dried over anhydrous magnesium sulfate. After filtration to remove the solid, removal of the solvent in vacuo yielded a pale yellow solid. The product mixture was separated by preparative TLC using hexanes–dichloromethane (99:1) as the eluent to give 1,1,2,2-tetramesitylmethyldigermane (2) (**12**, 16 mg, 44%) and dimesitylmethylgermane (6) (**11**, 7 mg, 38%), which were identified by comparison of their $^1$H NMR spectra with authentic samples.

### Attempted reaction of hexamesitylcyclotrigermane with methyllithium in the dark

Methyllithium (1.0 mL of a 1.4 M solution in diethyl ether, 1.4 mmol) was added to a Schlenk tube containing a clear, pale yellow solution of $Ge_3Mes_6$ (50 mg, 0.054 mmol) dissolved in toluene (4 mL). The Schlenk tube was covered in aluminum foil and left undisturbed for 14 h at room temperature (rt). No change in the colour of the solution was observed. Saturated aqueous ammonium chloride was then added to the solution. The aqueous and organic phases were then separated. The aqueous phase was extracted with four portions of hexanes and the extracts were combined with the organic phase. The organic phase was washed three times with water and then dried over anhydrous magnesium sulfate. After filtration to remove the solid, removal of the solvent in vacuo yielded a pale yellow solid. The solid was identified as hexamesitylcyclotrigermane (41.6 mg, 83%) by $^1$H NMR spectroscopy.

### Thermolysis of hexamesitylcyclotrigermane in the presence of methyllithium

Methyllithium (1.0 mL of a 1.4 M solution in diethyl ether, 1.4 mmol) was added to a Schlenk tube containing a clear, pale yellow solution of $Ge_3Mes_6$ (50 mg, 0.054 mmol) dissolved in toluene (4 mL). The Schlenk tube was then placed in an oil bath at 100°C for 14 h. The orange-brown solution was allowed to cool to rt. The colour of the solution changed to pale yellow upon quenching with saturated ammonium chloride; a gas was evolved. The aqueous and organic phases were then separated. The aqueous phase was extracted with four portions of hexanes and the extracts were combined with the organic phase. The organic phase was washed three times with water and then dried over anhydrous magnesium sulfate. After filtration to remove the solid, removal of the solvent in vacuo yielded a yellow oil. Analysis

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[2] Due to the small quantities of samples, it was impossible to obtain enough material for elemental analysis.

of the oil by $^{1}H$ NMR spectroscopy revealed a complex mixture of products, which could not be separated or identified.

## Acknowledgements

The financial support of the Natural Sciences and Engineering Research Council of Canada (NSERC) is gratefully acknowledged.

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