Supplementary Information for the Communication

Entitled

A Blue Digermene (t-Bu₂MeSi)₂Ge=Ge(SiMe-t-Bu₂)₂

Vladimir Ya. Lee, Kiera McNeice, Yuki Ito, and Akira Sekiguchi

Department of Chemistry, Graduate School of Pure and Applied Sciences,

University of Tsukuba, Tsukuba, Ibaraki 305-8571, Japan
Experimental Section

General Procedure. All experiments involving air-sensitive compounds were performed using high-vacuum line techniques or in an argon atmosphere using MBRAUN MB 150B-G glove box. All solvents were dried and degassed over potassium mirror in vacuum prior to use. NMR spectra were recorded on Bruker AC-300FT (\$H NMR at 300.1 MHz; $^{13}$C NMR at 75.5 MHz; $^{29}$Si NMR at 59.6 MHz) and Bruker AV-400FT (\$H NMR at 400.2 MHz; $^{13}$C NMR at 100.6 MHz; $^{29}$Si NMR at 79.5 MHz) NMR spectrometers.

Experimental Procedure and Spectral Data for Compound 3.

An excess of tert-butyl isonitrile (83 mg, 1.00 mmol) was added to tetrakis(di-tert-butylmethylsilyl)digermene 1 (150 mg, 0.19 mmol) by vacuum transfer. Dry oxygen-free benzene (1 ml) was then introduced into the reaction tube by vacuum transfer, and the resulting mixture was stirred for 10 min at room temperature. The color of the reaction mixture changed from dark blue to pale yellow. The solvent, excess tert-butyl isonitrile and side product (isobutene) were removed under vacuum, leaving 3 as a colorless solid in 97% yield. $^1$H NMR (C$_6$D$_6$, ppm): $\delta$0.15 (s, 6 H, CH$_3$), 0.98 (s, 18 H, C(CH$_3$)$_3$), 1.11 (s, 18 H, C(CH$_3$)$_3$), 3.77 (s, 1 H, Ge–H). $^{13}$C NMR (C$_6$D$_6$, ppm): $\delta$ –5.81 (CH$_3$), 21.61 (C(CH$_3$)$_3$), 21.98 (C(CH$_3$)$_3$), 28.88 (C(CH$_3$)$_3$), 29.34 (C(CH$_3$)$_3$), 124.34 (CαN). $^{29}$Si NMR (C$_6$D$_6$, ppm): $\delta$19.94.


A freshly prepared solution of lithium naphthalenide (2.63 mmol) in THF (5 ml) was added under an Ar atmosphere to the solution of tetrakis(di-tert-butylmethylsilyl)digermene 1 (500 mg,
0.65 mmol) in THF (5 ml) at –78 °C. After the reaction mixture was gradually warmed to room temperature, the solvent was evaporated and the residue was washed with dry hexane several times to give dilithiogerane 4 as a pale-yellow powder (519 mg, 58%). 4 was identified by the comparison of its NMR spectral data with those previously reported for the authentic 1,1-dilithiogerane (t-Bu₂MeSi)₂GeLi₂.¹

Figure 1. UV–Vis spectrum of digermene 1 measured in hexane.