Supplemental material for

{Ge$_{10}$Si[Si(SiMe$_3$)$_3$]$_4$(SiMe$_3$)$_2$Me}$^-$: A Ge$_{10}$Si framework reveals a structural transition onto elemental germanium

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Experimental procedure:

Liquid germanium was treated with HCl under high vacuum at 1600°C, and the resulting gaseous products were condensed together with 150 ml of a mixture of toluene/N$_n$Pr$_3$ (5:1) (150 ml) at a surface at −196°C. During the reaction 20mmol HCl and 16.3 mmol Ge (1.208g) were used. After the cocondensate was allowed to warm to −78°C a dark red emulsion of a dark red oil and a pale yellow solution was obtained. The emulsion was treated with a toluene solution of LiSi(SiMe$_3$)$_3$·3THF (9.33 g, 19.8 mmol), cooled to −78°C. The reaction mixture was then heated slowly to room temperature under stirring leading to a dark red solution. Evaporating the solvent in vacuum leads to a dark red almost black residue, which when extracted with pentane gave a dark red pentane extract, from which orange crystals of {Ge$_9$[Si(SiMe$_3$)$_3$]$_3$}Li(THF)$_4$·3THF (250 mg, 0.15 mmol) are obtained on concentrating. Afterwards the solvent is changed to hexane and the hexane solution is heated to 40°C for 4 hours. On cooling to room temperature dark red plate like crystals of {Ge$_{10}$Si[Si(SiMe$_3$)$_3$]$_4$(SiMe$_3$)$_2$Me}Li(THF)$_4$ (100 mg, 0.045 mmol) precipitate.
Masspectra of the anion \( \text{Ge}_{10}\text{Si}[\text{Si(SiMe}_{3}]_{3}\text{]}_{4}\text{(SiMe}_{3}\text{)}_{2}\text{Me} \)^{-}

Experiments were performed on an IonSpec ULTIMA FT/ICR-MS, equipped with a 7 T super conducting magnet and an external electrospray (Analytica) ion source.