

Supplemental material for

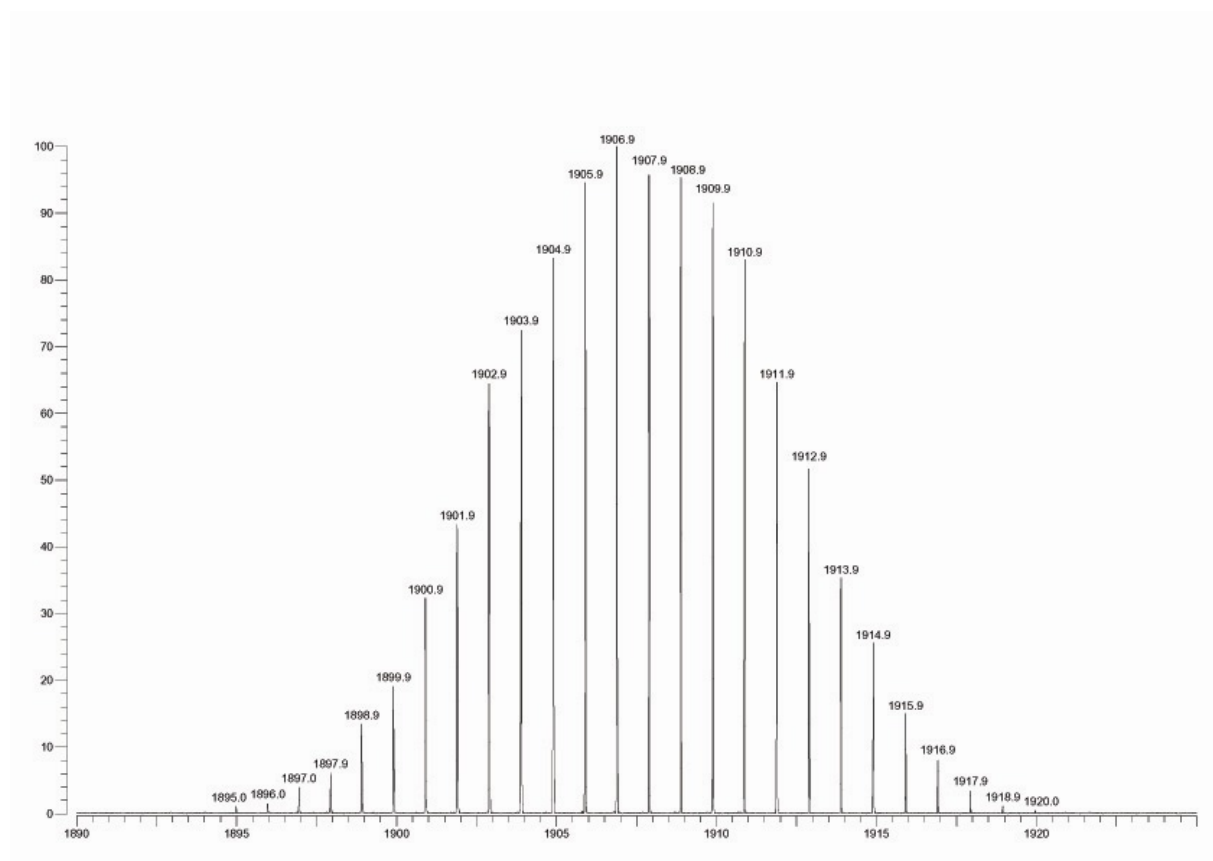
$\{\text{Ge}_{10}\text{Si}[\text{Si}(\text{SiMe}_3)_3]_4(\text{SiMe}_3)_2\text{Me}\}^-$: A Ge_{10}Si framework reveals a structural transition onto elemental germanium

Andreas Schnepf

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/*Nn*Pr₃ (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(SiMe₃)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 {Ge₉[Si(SiMe₃)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 {Ge₁₀Si[Si(SiMe₃)₃]₄(SiMe₃)₂Me}Li(THF)₄ (100 mg, 0.045 mmol) precipitate.

Massspectra of the anion $\{\text{Ge}_{10}\text{Si}[\text{Si}(\text{SiMe}_3)_3]_4(\text{SiMe}_3)_2\text{Me}\}^-$



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.