

**Electronic Supporting Information for manuscript**

**“On the coordination chemistry of organochalcogenolates  $R^{NMe_2}E^-$  and  $R^{NMe_2}E^+O^-$  ( $E = S, Se$ ) onto lead(II) and lighter divalent tetrel elements<sup>‡</sup>”**

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### S1. Synthesis of $\text{Ge}(\text{TePh})_2[\text{N}(\text{SiMe}_3)_2]_2$

$\text{Ph}_2\text{Te}_2$  (0.16 g, 0.40 mmol) and  $\text{Ge}[\text{N}(\text{SiMe}_3)_2]_2$  (0.16 g, 0.40 mmol) were dissolved in toluene (30 mL) and the solution was stirred at r.t. overnight. Removal of the solvent under vacuum then afforded a dark orange solid. Recrystallisation in a 1:2 mixture of toluene and pentane afforded large red-orange crystals of the title compound. Isolated yield 0.22 g (69%).  $^1\text{H}$  NMR (benzene- $d_6$ , 400.13 MHz, 298 K):  $\delta$  = 7.99–7.97 (m, 4H, arom- $H$ ), 7.06–7.02 (m, 2H, arom- $H$ ), 6.96–6.92 (m, 4H, arom- $H$ ), 0.43 (s, 36H,  $\text{N}(\text{Si}(\text{CH}_3)_3)_2$ ) ppm.  $^{13}\text{C}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 100.61 MHz, 298 K):  $\delta$  = 142.48, 129.88, 129.34, 114.23 (all arom- $C$ ), 7.54 ( $\text{N}(\text{Si}(\text{CH}_3)_3)_2$ ,  $^1J_{\text{C-Si}}$  = 56.5 Hz) ppm.  $^{29}\text{Si}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 79.49 MHz, 298 K):  $\delta$  = 5.66 ( $^1J_{\text{Si-C}}$  = 56.5 Hz) ppm.  $^{125}\text{Te}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 126.24 MHz, 298 K):  $\delta$  = +447 ppm. Anal. Calcd for  $\text{C}_{24}\text{H}_{46}\text{N}_2\text{Te}_2\text{Si}_4\text{Ge}$  (802.82  $\text{g}\cdot\text{mol}^{-1}$ ): C 35.9, H 5.8, N 3.5. Found: C 35.8, H 5.8, N 3.4.

### S2. Synthesis of $\text{Ge}(\text{SePh})_2[\text{N}(\text{SiMe}_3)_2]_2$

$\text{Ph}_2\text{Se}_2$  (0.14 g, 0.44 mmol) and  $\text{Ge}[\text{N}(\text{SiMe}_3)_2]_2$  (0.17 g, 0.44 mmol) was dissolved in toluene (30 mL) and the mixture was stirred overnight at room temperature. Removal of the solvent under vacuum then afforded a pale yellow solid. Recrystallisation in a 1:2 mixture of toluene and pentane afforded large colourless crystals of the title compound. Isolated yield 0.19 g (61%).  $^1\text{H}$  NMR (benzene- $d_6$ , 400.13 MHz, 298 K):  $\delta$  = 7.83–7.81 (m, 4H, arom- $H$ ), 7.02–6.99 (m, 6H, arom- $H$ ), 0.41 ppm (s, 36H,  $\text{N}(\text{Si}(\text{CH}_3)_3)_2$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 74.57 MHz, 298 K):  $\delta$  = 138.51, 129.62, 128.92, 128.25 (all arom- $C$ ), 7.54 ppm ( $\text{N}(\text{Si}(\text{CH}_3)_3)_2$ ,  $^1J_{\text{C-Si}}$  = 56.6 Hz).  $^{29}\text{Si}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 79.49 MHz, 298 K):  $\delta$  = 5.39 ( $^1J_{\text{Si-C}}$  = 56.6 Hz) ppm.  $^{77}\text{Se}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 76.31 MHz, 298 K):  $\delta$  = +282 ppm. Anal. Calcd for  $\text{C}_{24}\text{H}_{46}\text{N}_2\text{Se}_2\text{Si}_4\text{Ge}$  (705.54  $\text{g}\cdot\text{mol}^{-1}$ ): C 40.9, H 6.6, N 4.0. Found: C 41.0, H 6.6, N 4.1.

### S3. Synthesis of $\text{Sn}(\text{TePh})_2[\text{N}(\text{SiMe}_3)_2]_2$

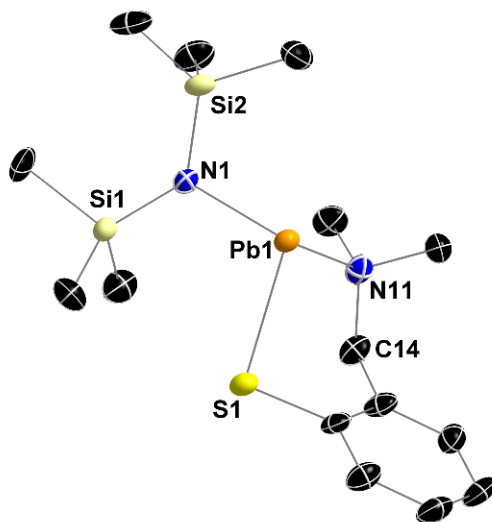
$\text{Ph}_2\text{Te}_2$  (0.22 g, 0.40 mmol) and  $\text{Sn}[\text{N}(\text{SiMe}_3)_2]_2$  (0.24 g, 0.54 mmol) were dissolved in toluene (30 mL) and the solution was stirred overnight at room temperature. Removal of the solvent under vacuum then afforded a red-orange solid. Recrystallisation in a 1:2 mixture of toluene and pentane afforded large red-orange crystals of the title compound. Isolated yield 0.31 g (67%).  $^1\text{H}$  NMR (benzene- $d_6$ , 300.13 MHz, 298 K):  $\delta$  = 7.88 (dd,  $^3J_{\text{H-H}} = 7.6$  Hz,  $^4J_{\text{H-H}} = 0.8$  Hz, 4H, arom- $H$ ), 7.02 (t,  $^3J_{\text{H-H}} = 7.6$  Hz, 2H, arom- $H$ ), 6.91 (t,  $^3J_{\text{H-H}} = 7.6$  Hz, 4H, arom- $H$ ), 0.37 (s, 36H,  $\text{N}(\text{Si}(\text{CH}_3)_3)_2$ ) ppm.  $^{13}\text{C}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 75.48 MHz, 298 K):  $\delta$  = 143.12, 130.03, 129.37, 109.43 (all arom- $C$ ), 7.07 ( $\text{N}(\text{Si}(\text{CH}_3)_3)_2$ ,  $^3J_{\text{C-}^{119}\text{Sn}} = 12.3$  Hz,  $^1J_{\text{C-Si}} = 55.5$  Hz) ppm.  $^{29}\text{Si}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 79.49 MHz, 298 K):  $\delta$  = 5.66 ( $^2J_{\text{Si-}^{119}\text{Sn}} = 10.3$  Hz) ppm.  $^{119}\text{Sn}\{^1\text{H}\}$  NMR (benzene- $d_6$ , 149.21 MHz, 25 °C):  $\delta$  = -442 ( $^1J_{^{119}\text{Sn-Te}} = 4424$  Hz) ppm.  $^{125}\text{Te}\{^1\text{H}\}$  NMR ( $\text{C}_6\text{D}_6$ , 126.24 MHz, 298 K):  $\delta$  = +288 ppm. Anal. Calcd for  $\text{C}_{24}\text{H}_{46}\text{N}_2\text{Te}_2\text{Si}_4\text{Sn}$  (848.89 g·mol $^{-1}$ ): C 34.0, H 5.5, N 3.3. Found: C 34.0, H 5.5, N 3.4.

### S4. Synthesis of $[\{2-(\text{Me}_2\text{NCH}_2)\text{C}_6\text{H}_4\text{SeCH}_2\text{C}(\text{Me})_2\text{OH}\}\text{H}]^+[\text{H}_2\text{N}\{\text{B}(\text{C}_6\text{F}_5)_3\}_2]^-$

$[\text{H}(\text{OEt}_2)_2]^+[\text{H}_2\text{N}\{\text{B}(\text{C}_6\text{F}_5)_3\}_2]^-$  (0.95 g, 0.80 mmol) was added in fractions with a bent finger to a colourless solution of  $2-(\text{Me}_2\text{NCH}_2)\text{C}_6\text{H}_4\text{SeCH}_2\text{C}(\text{Me})_2\text{OH}$  (0.23 g, 0.80 mmol) in  $\text{Et}_2\text{O}$  (30 mL). The resulting solution was stirred at 25 °C for 4 h. An oily material formed upon addition of pentane (120 mL). The supernatant was removed by filtration, and the oil was dried *in vacuo* to yield a pale yellow solid that was further washed with pentane (2 × 20 mL). After drying under vacuum, the title compound was isolated as a fine pale yellow powder. Yield 0.81 g (76 %). X-ray quality crystals were grown from a dichloromethane/pentane mixture at room temperature.  $^1\text{H}$  NMR ( $\text{CD}_2\text{Cl}_2$ , 500.13 MHz, 298 K):  $\delta$  = 9.36 (br, 1H, OH), 7.82 (d, 1H,  $^3J = 7.5$  Hz, arom- $H$ ),

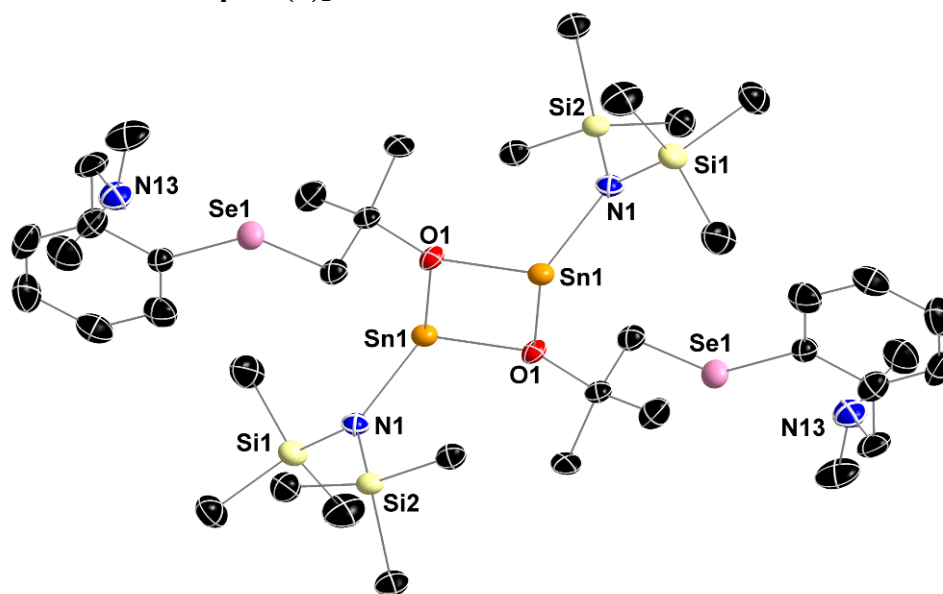
7.51 (dt, 1H,  $^3J = 7.5$  Hz,  $^4J = 1.0$  Hz, arom-*H*), 7.45 (dt, 1H,  $^3J = 7.5$  Hz,  $^4J = 1.0$  Hz, arom-*H*), 7.32 (dd, 1H,  $^3J = 8.0$  Hz,  $^4J = 1.0$  Hz, arom-*H*), 5.68 (br, 2H,  $H_2N$ ), 4.36 (d, 2H,  $^2J = 5.0$  Hz, ArCH<sub>2</sub>), 3.19 (s, 2H, SeCH<sub>2</sub>), 2.92 (d, 6H,  $J = 5.0$  Hz, ArCH<sub>2</sub>NH(CH<sub>3</sub>)<sub>2</sub>), 1.86 (s, 1H, ArCH<sub>2</sub>NHMe<sub>2</sub>), 1.40 (s, 6H, SeCH<sub>2</sub>C(CH<sub>3</sub>)<sub>2</sub>OH).  $^{13}C\{^1H\}$  NMR (CD<sub>2</sub>Cl<sub>2</sub>, 100.62 MHz, 298 K):  $\delta = 149.50, 147.14, 140.78, 138.31, 135.82, 138.79, 133.90, 133.19, 132.48, 131.42, 130.42, 120.95, 74.20$  (SeCH<sub>2</sub>CMe<sub>2</sub>OH), 64.75 (ArCH<sub>2</sub>), 47.03 (SeCH<sub>2</sub>), 43.75 (ArCH<sub>2</sub>NH(CH<sub>3</sub>)<sub>2</sub>), 29.03 (SeCH<sub>2</sub>C(CH<sub>3</sub>)<sub>2</sub>OH) ppm.  $^{19}F\{^1H\}$  NMR (CD<sub>2</sub>Cl<sub>2</sub>, 376.53 MHz, 298 K):  $\delta = -132.9$  (d, 12F,  $^3J_{F-F} = 18.4$  Hz, *o*-F),  $-160.2$  (t, 6F,  $^3J_{F-F} = 20.3$  Hz, *p*-F),  $-165.67$  (t, 12F,  $^3J_{F-F} = 19.2$  Hz, *m*-F) ppm.  $^{11}B$  NMR (CD<sub>2</sub>Cl<sub>2</sub>, 128.39 MHz, 298 K):  $\delta = -8.31$  ppm.  $^{77}Se\{^1H\}$  NMR, (CD<sub>2</sub>Cl<sub>2</sub>, 76.31 MHz, 298 K):  $\delta = +187$  ppm. Anal. Calcd. for C<sub>49</sub>H<sub>24</sub>B<sub>2</sub>F<sub>30</sub>N<sub>2</sub>OSe (1327.26 g·mol<sup>-1</sup>): C 44.3, H 1.8, N 2.1; Found: C 44.5, H 1.8, N 2.1. Single-crystals suitable for XRD studies were grown by recrystallisation from a mixture of dichloromethane and pentane.

#### S5. X-ray structure of complex 6



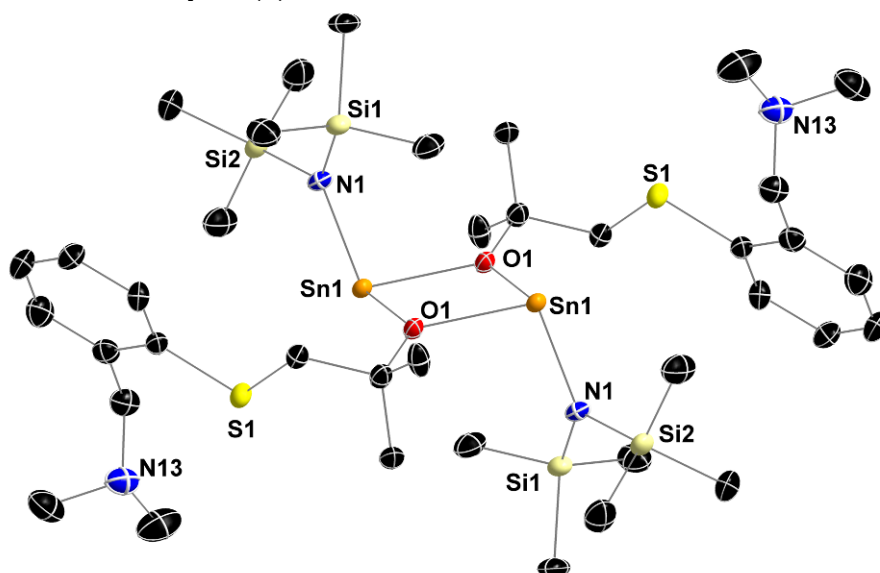
ORTEP representation of the molecular solid-state structure of Pb[SC<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>NMe<sub>2</sub>)<sub>2</sub>][N(SiMe<sub>3</sub>)<sub>2</sub>] (**6**), with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°): Pb(1)–N(1) = 2.248(5), Pb(1)–N(11) = 2.634(6), Pb(1)–S(1) = 2.6476(17), N(1)–Si(1) = 1.724(6), N(1)–Si(2) = 1.727(6); N(1)–Pb(1)–N(11) = 97.08(19), N(1)–Pb(1)–S(1) = 100.79(15), N(11)–Pb(1)–S(1) = 83.09(13).

### S6. X-ray structure of complex (8)<sub>2</sub>



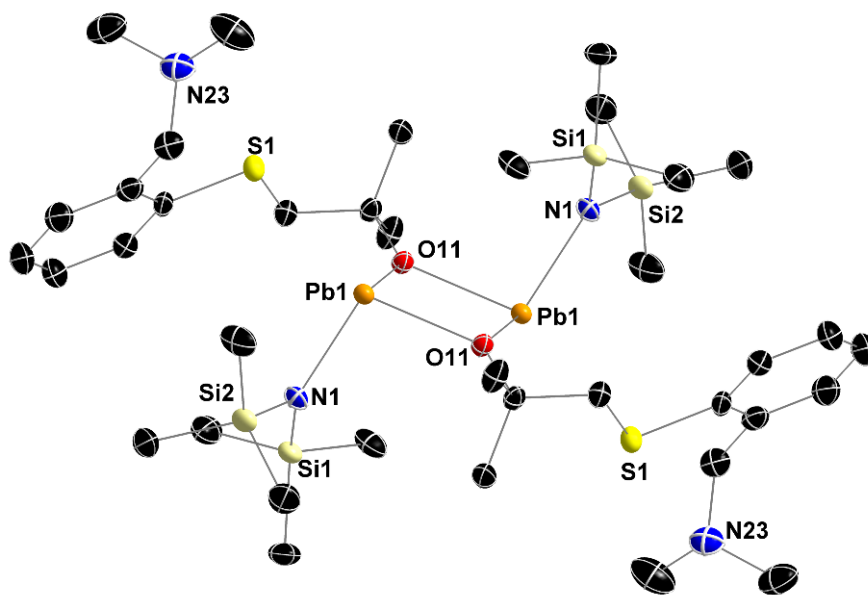
ORTEP diagram of the molecular structure of the dimer of  $\text{Sn}[\text{OC}(\text{Me})_2\text{CH}_2\text{SeC}_6\text{H}_4(\text{CH}_2\text{NMe}_2)_2][\text{N}(\text{SiMe}_3)_2]$  [(8)<sub>2</sub>], with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Sn}(1)\text{--N}(1) = 2.136(5)$ ,  $\text{Sn}(1)\text{--O}(1)^{\#1} = 2.168(4)$ ,  $\text{Sn}(1)\text{--O}(1) = 2.177(4)$ ;  $\text{N}(1)\text{--Sn}(1)\text{--O}(1)^{\#1} = 103.68(17)$ ,  $\text{N}(1)\text{--Sn}(1)\text{--O}(1) = 101.17(17)$ ,  $\text{O}(1)^{\#1}\text{--Sn}(1)\text{--O}(1) = 71.65(17)$ .

### S7. X-ray structure of complex (9)<sub>2</sub>



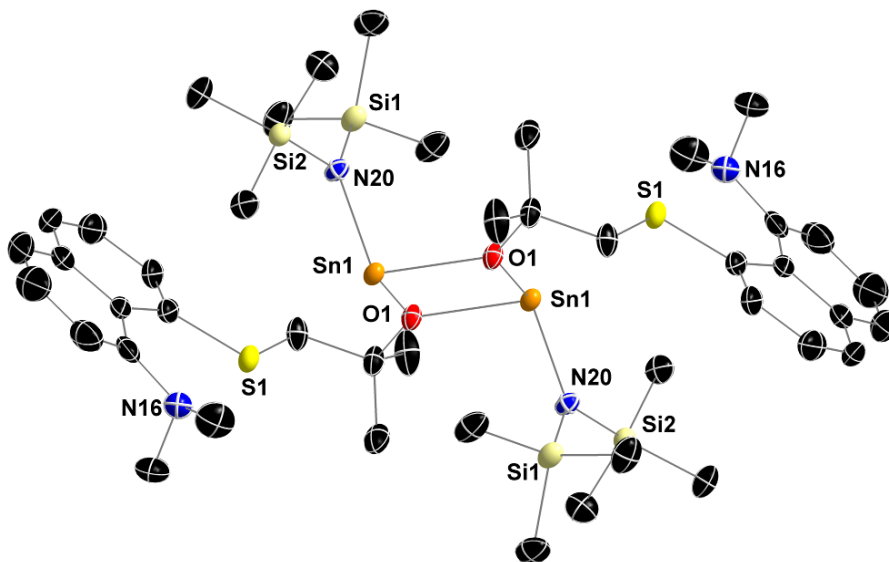
ORTEP diagram of the molecular structure of the dimer of  $\text{Sn}[\text{OC}(\text{Me})_2\text{CH}_2\text{SC}_6\text{H}_4(\text{CH}_2\text{NMe}_2)_2][\text{N}(\text{SiMe}_3)_2]$  [(9)<sub>2</sub>], with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Sn}(1)\text{--N}(1) = 2.150(5)$ ,  $\text{Sn}(1)\text{--O}(1) = 2.168(4)$ ,  $\text{Sn}(1)\text{--O}(1)^{\#1} = 2.250(4)$ ;  $\text{N}(1)\text{--Sn}(1)\text{--O}(1) = 103.09(18)$ ,  $\text{N}(1)\text{--Sn}(1)\text{--O}(1)^{\#1} = 99.46(17)$ ,  $\text{O}(1)\text{--Sn}(1)\text{--O}(1)^{\#1} = 71.78(17)$ .

### S8. X-ray structure of complex (11)<sub>2</sub>



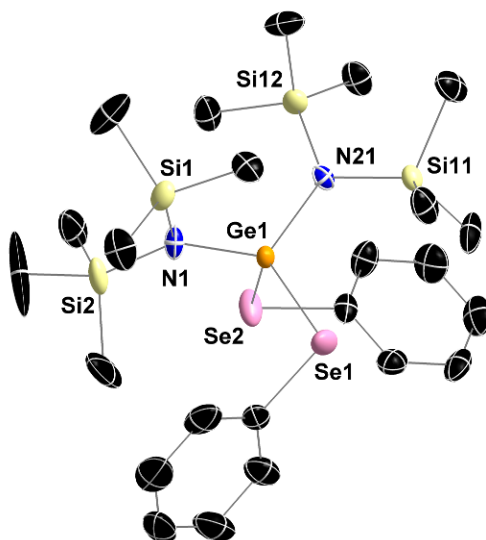
ORTEP diagram of the molecular structure of the dimer of  $\text{Pb}[\text{OC}(\text{Me})_2\text{CH}_2\text{SC}_6\text{H}_4(\text{CH}_2\text{NMe}_2)_2][\text{N}(\text{SiMe}_3)_2]$  [(11)<sub>2</sub>], with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Pb}(1)\text{--N}(1) = 2.2571(19)$ ,  $\text{Pb}(1)\text{--O}(11) = 2.2729(16)$ ,  $\text{Pb}(1)\text{--O}(11)^{\#1} = 2.3572(16)$ ;  $\text{N}(1)\text{--Pb}(1)\text{--O}(11) = 103.35(6)$ ,  $\text{N}(1)\text{--Pb}(1)\text{--O}(11)^{\#1} = 98.24(6)$ ,  $\text{O}(11)\text{--Pb}(1)\text{--O}(11)^{\#1} = 71.62(7)$ .

### S9. X-ray structure of complex (12)<sub>2</sub>



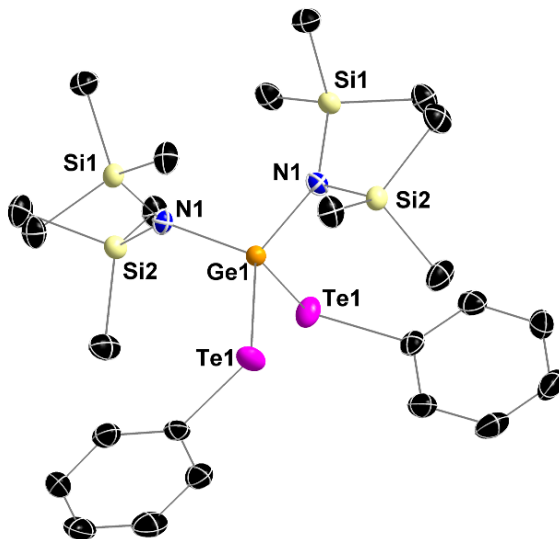
ORTEP diagram of the molecular structure of the dimer of  $\text{Sn}[\text{OC}(\text{Me})_2\text{CH}_2\text{S}\{1\text{-(8-(NMe}_2\text{)naphthalen-1-yl)}\}][\text{N}(\text{SiMe}_3)_2]$  [(12)<sub>2</sub>], with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Sn}(1)\text{--N}(20) = 2.147(2)$ ,  $\text{Sn}(1)\text{--O}(1) = 2.165(2)$ ,  $\text{Sn}(1)\text{--O}(1)^{\#1} = 2.282(2)$ ;  $\text{N}(20)\text{--Sn}(1)\text{--O}(1) = 102.77(8)$ ,  $\text{N}(20)\text{--Sn}(1)\text{--O}(1)^{\#1} = 100.25(9)$ ,  $\text{O}(1)\text{--Sn}(1)\text{--O}(1)^{\#1} = 71.86(9)$ .

### S10. X-ray structure of $\text{Ge}(\text{SePh})_2[\text{N}(\text{SiMe}_3)_2]_2$



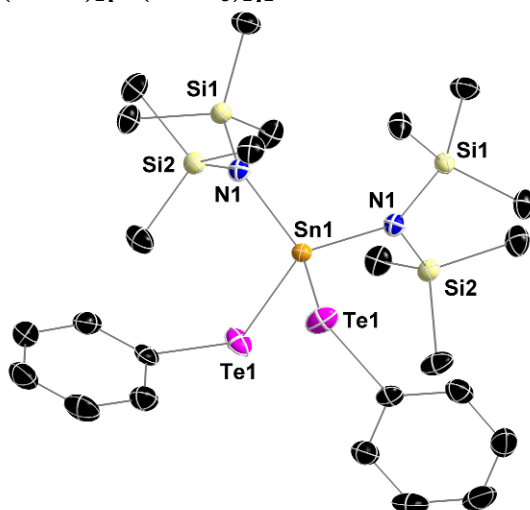
ORTEP diagram of the molecular structure of the dimer of  $\text{Ge}(\text{SePh})_2[\text{N}(\text{SiMe}_3)_2]_2$ , with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Ge}(1)\text{--}\text{N}(1) = 1.860(3)$ ,  $\text{Ge}(1)\text{--}\text{N}(21) = 1.863(3)$ ,  $\text{Ge}(1)\text{--}\text{Se}(2) = 2.3706(5)$ ,  $\text{Ge}(1)\text{--}\text{Se}(1) = 2.3868(5)$ ;  $\text{N}(1)\text{--}\text{Ge}(1)\text{--}\text{N}(21) = 115.74(13)$ ,  $\text{N}(1)\text{--}\text{Ge}(1)\text{--}\text{Se}(2) = 105.37(9)$ ,  $\text{N}(21)\text{--}\text{Ge}(1)\text{--}\text{Se}(2) = 111.88(9)$ ,  $\text{N}(1)\text{--}\text{Ge}(1)\text{--}\text{Se}(1) = 109.49(9)$ ,  $\text{N}(21)\text{--}\text{Ge}(1)\text{--}\text{Se}(1) = 106.73(8)$ ,  $\text{Se}(2)\text{--}\text{Ge}(1)\text{--}\text{Se}(1) = 107.36(2)$ .

### S11. X-ray structure of $\text{Ge}(\text{TePh})_2[\text{N}(\text{SiMe}_3)_2]_2$



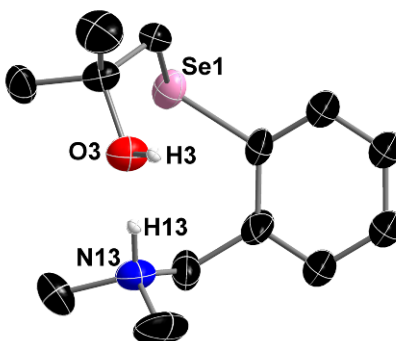
ORTEP diagram of the molecular structure of the dimer of  $\text{Ge}(\text{TePh})_2[\text{N}(\text{SiMe}_3)_2]_2$ , with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Ge}(1)\text{--}\text{N}(1)^{\#1} = 1.8656(17)$ ,  $\text{Ge}(1)\text{--}\text{N}(1) = 1.8657(17)$ ,  $\text{Ge}(1)\text{--}\text{Te}(1)^{\#1} = 2.6197(2)$ ,  $\text{Ge}(1)\text{--}\text{Te}(1) = 2.6198(2)$ ;  $\text{N}(1)^{\#1}\text{--}\text{Ge}(1)\text{--}\text{N}(1) = 112.86(10)$ ,  $\text{N}(1)\text{--}\text{Ge}(1)\text{--}\text{Te}(1)^{\#1} = 107.32(5)$ ,  $\text{N}(1)^{\#1}\text{--}\text{Ge}(1)\text{--}\text{Te}(1) = 107.32(5)$ ,  $\text{N}(1)\text{--}\text{Ge}(1)\text{--}\text{Te}(1) = 110.63(5)$ ,  $\text{Te}(1)^{\#1}\text{--}\text{Ge}(1)\text{--}\text{Te}(1) = 107.995(12)$ .

**S12. X-ray structure of  $\text{Sn}(\text{TePh})_2[\text{N}(\text{SiMe}_3)_2]_2$**



ORTEP diagram of the molecular structure of the dimer of  $\text{Sn}(\text{TePh})_2[\text{N}(\text{SiMe}_3)_2]_2$ , with thermal ellipsoids set at 50% probability. Hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°):  $\text{Sn}(1)\text{--}\text{N}(1) = 2.0623(19)$ ,  $\text{Sn}(1)\text{--}\text{N}(1)^{\#1} = 2.0624(19)$ ,  $\text{Sn}(1)\text{--}\text{Te}(1)^{\#1} = 2.7690(2)$ ,  $\text{Sn}(1)\text{--}\text{Te}(1) = 2.7690(2)$ ;  $\text{N}(1)\text{--}\text{Sn}(1)\text{--}\text{N}(1)^{\#1} = 112.17(11)$ ,  $\text{N}(1)\text{--}\text{Sn}(1)\text{--}\text{Te}(1)^{\#1} = 110.07(5)$ ,  $\text{N}(1)^{\#1}\text{--}\text{Sn}(1)\text{--}\text{Te}(1)^{\#1} = 107.85(5)$ ,  $\text{N}(1)\text{--}\text{Sn}(1)\text{--}\text{Te}(1) = 107.85(5)$ ,  $\text{N}(1)^{\#1}\text{--}\text{Sn}(1)\text{--}\text{Te}(1) = 110.07(5)$ ,  $\text{Te}(1)^{\#1}\text{--}\text{Sn}(1)\text{--}\text{Te}(1) = 108.788(11)$ .

**S13. X-ray structure of  $[\{2\text{-(Me}_2\text{NCH}_2\text{)C}_6\text{H}_4\text{SeCH}_2\text{C(Me)}_2\text{OH}\}\text{H}]^+ \cdot [\text{H}_2\text{N}\{\text{B(C}_6\text{F}_5)_3\}_2]^-$**



ORTEP representation of the main component of the cation in the molecular structure of  $[\{2\text{-(Me}_2\text{NCH}_2\text{)C}_6\text{H}_4\text{SeCH}_2\text{C(Me)}_2\text{OH}\}\text{H}]^+ \cdot [\text{H}_2\text{N}\{\text{B(C}_6\text{F}_5)_3\}_2]^-$ , with thermal ellipsoids set at 50% probability.

**S14. Tables of crystallographic data**

|                                                            | <b>5</b>                                                                                       | <b>6</b>                                                                                      | <b>7</b>                                                                                       |
|------------------------------------------------------------|------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------|
| Formula                                                    | C <sub>15</sub> H <sub>30</sub> N <sub>2</sub> Pb <sub>1</sub> Se <sub>1</sub> Si <sub>2</sub> | C <sub>15</sub> H <sub>30</sub> N <sub>2</sub> Pb <sub>1</sub> S <sub>1</sub> Si <sub>2</sub> | C <sub>24</sub> H <sub>46</sub> N <sub>2</sub> Se <sub>2</sub> Si <sub>4</sub> Sn <sub>1</sub> |
| CCDC                                                       | 1006879                                                                                        | 1006880                                                                                       | 1006881                                                                                        |
| Mol. wt.                                                   | 580.74                                                                                         | 533.84                                                                                        | 751.60                                                                                         |
| Crystal system                                             | Triclinic                                                                                      | Triclinic                                                                                     | Monoclinic                                                                                     |
| Space group                                                | P -1                                                                                           | P -1                                                                                          | C 2/c                                                                                          |
| <i>a</i> (Å)                                               | 8.8755(2)                                                                                      | 8.8858(4)                                                                                     | 8.7208(4)                                                                                      |
| <i>b</i> (Å)                                               | 12.2474(2)                                                                                     | 12.1481(6)                                                                                    | 17.4865(9)                                                                                     |
| <i>c</i> (Å)                                               | 13.0612(2)                                                                                     | 12.9552(6)                                                                                    | 21.7958(9)                                                                                     |
| $\alpha$ (°)                                               | 63.6070(10)                                                                                    | 63.6440(10)                                                                                   | 90                                                                                             |
| $\beta$ (°)                                                | 70.8920(10)                                                                                    | 71.551(2)                                                                                     | 98.565(2)                                                                                      |
| $\gamma$ (°)                                               | 75.5130(10)                                                                                    | 75.792(2)                                                                                     | 90                                                                                             |
| <i>V</i> (Å <sup>3</sup> )                                 | 1192.50(4)                                                                                     | 1179.59(10)                                                                                   | 3286.7(3)                                                                                      |
| <i>Z</i>                                                   | 2                                                                                              | 2                                                                                             | 4                                                                                              |
| Density (g/cm <sup>3</sup> )                               | 1.617                                                                                          | 1.503                                                                                         | 1.519                                                                                          |
| Abs. coeff., (mm <sup>-1</sup> )                           | 8.698                                                                                          | 7.339                                                                                         | 3.154                                                                                          |
| <i>F</i> (000)                                             | 556                                                                                            | 520                                                                                           | 1512                                                                                           |
| Crystal size, mm                                           | 0.28 × 0.17 × 0.07                                                                             | 0.29 × 0.18 × 0.1                                                                             | 0.48 × 0.23 × 0.03                                                                             |
| $\theta$ range, deg                                        | 3.07 to 27.48                                                                                  | 1.81 to 27.62                                                                                 | 2.91 to 27.49                                                                                  |
|                                                            | −11 < <i>h</i> < 10                                                                            | −10 < <i>h</i> < 11                                                                           | −9 < <i>h</i> < 11                                                                             |
| Limiting indices                                           | −15 < <i>k</i> < 12                                                                            | −13 < <i>k</i> < 15                                                                           | −22 < <i>k</i> < 22                                                                            |
|                                                            | −16 < <i>l</i> < 13                                                                            | 0 < <i>l</i> < 16                                                                             | −28 < <i>l</i> < 23                                                                            |
| <i>R</i> (int)                                             | 0.0258                                                                                         | 0.0000                                                                                        | 0.0434                                                                                         |
| Reflections collected                                      | 8413                                                                                           | 5435                                                                                          | 11413                                                                                          |
| Reflec. Unique [ <i>I</i> >2 $\sigma$ ]                    | 5371                                                                                           | 5435                                                                                          | 3763                                                                                           |
| Completeness to $\theta$ (%)                               | 98.3                                                                                           | 99.3                                                                                          | 99.5                                                                                           |
| Data/restraints/param.                                     | 5371 / 0 / 198                                                                                 | 5435 / 0 / 198                                                                                | 3763 / 0 / 156                                                                                 |
| Goodness-of-fit                                            | 1.139                                                                                          | 1.033                                                                                         | 1.057                                                                                          |
| <i>R</i> <sub>1</sub> [ <i>I</i> >2 $\sigma$ ] (all data)  | 0.0270 (0.0304)                                                                                | 0.0360 (0.0403)                                                                               | 0.0331 (0.0464)                                                                                |
| <i>wR</i> <sub>2</sub> [ <i>I</i> >2 $\sigma$ ] (all data) | 0.0803 (0.0813)                                                                                | 0.1217 (0.1244)                                                                               | 0.0668 (0.0708)                                                                                |
| Largest diff. e <sup>−</sup> ·Å <sup>−3</sup>              | 1.193 and −1.010                                                                               | 1.487 and −1.610                                                                              | 0.443 and −0.504                                                                               |

|                                                            | (8) <sub>2</sub>                                                                                              | (9) <sub>2</sub>                                                                                             | (10) <sub>2</sub>                                                                                             |
|------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------|
| Formula                                                    | C <sub>38</sub> H <sub>76</sub> N <sub>4</sub> O <sub>2</sub> Se <sub>2</sub> Si <sub>4</sub> Sn <sub>2</sub> | C <sub>38</sub> H <sub>76</sub> N <sub>4</sub> O <sub>2</sub> S <sub>2</sub> Si <sub>4</sub> Sn <sub>2</sub> | C <sub>38</sub> H <sub>76</sub> N <sub>4</sub> O <sub>2</sub> Pb <sub>2</sub> Se <sub>2</sub> Si <sub>4</sub> |
| CCDC                                                       | 1006882                                                                                                       | 1006883                                                                                                      | 1006884                                                                                                       |
| Mol. wt.                                                   | 1128.73                                                                                                       | 1034.95                                                                                                      | 1305.68                                                                                                       |
| Crystal system                                             | triclinic                                                                                                     | triclinic                                                                                                    | triclinic                                                                                                     |
| Space group                                                | P -1                                                                                                          | P -1                                                                                                         | P -1                                                                                                          |
| <i>a</i> (Å)                                               | 10.431(5)                                                                                                     | 8.9518(9)                                                                                                    | 8.9533(3)                                                                                                     |
| <i>b</i> (Å)                                               | 10.948(2)                                                                                                     | 10.6790(11)                                                                                                  | 10.7807(4)                                                                                                    |
| <i>c</i> (Å)                                               | 11.369(4)                                                                                                     | 13.8023(15)                                                                                                  | 13.9822(5)                                                                                                    |
| $\alpha$ (°)                                               | 100.052(13)                                                                                                   | 101.636(5)                                                                                                   | 101.8840(10)                                                                                                  |
| $\beta$ (°)                                                | 100.25(2)                                                                                                     | 104.069(5)                                                                                                   | 103.3150(10)                                                                                                  |
| $\gamma$ (°)                                               | 94.638(17)                                                                                                    | 93.483(5)                                                                                                    | 93.6150(10)                                                                                                   |
| <i>V</i> (Å <sup>3</sup> )                                 | 1249.6(8)                                                                                                     | 1245.2(2)                                                                                                    | 1276.49(8)                                                                                                    |
| <i>Z</i>                                                   | 1                                                                                                             | 2                                                                                                            | 2                                                                                                             |
| Density (g/cm <sup>3</sup> )                               | 1.5                                                                                                           | 1.38                                                                                                         | 1.699                                                                                                         |
| Abs. coeff., (mm <sup>-1</sup> )                           | 2.586                                                                                                         | 1.217                                                                                                        | 8.139                                                                                                         |
| <i>F</i> (000)                                             | 572                                                                                                           | 536                                                                                                          | 636                                                                                                           |
| Crystal size, mm                                           | 0.3 × 0.2 × 0.1                                                                                               | 0.38 × 0.29 × 0.19                                                                                           | 0.1 × 0.05 × 0.02                                                                                             |
| $\theta$ range, deg                                        | 2.91 to 27.52                                                                                                 | 3.1 to 27.46                                                                                                 | 3.07 to 27.48                                                                                                 |
| Limiting indices                                           | −13 < <i>h</i> < 13                                                                                           | −11 < <i>h</i> < 11                                                                                          | −11 < <i>h</i> < 11                                                                                           |
|                                                            | −14 < <i>k</i> < 12                                                                                           | −12 < <i>k</i> < 13                                                                                          | −12 < <i>k</i> < 13                                                                                           |
|                                                            | −14 < <i>l</i> < 14                                                                                           | −17 < <i>l</i> < 17                                                                                          | −18 < <i>l</i> < 18                                                                                           |
| <i>R</i> (int)                                             | 0.0597                                                                                                        | 0.0355                                                                                                       | 0.0355                                                                                                        |
| Reflections collected                                      | 12220                                                                                                         | 15449                                                                                                        | 17730                                                                                                         |
| Reflec. Unique [ <i>I</i> >2 $\sigma$ ]                    | 4291                                                                                                          | 5289                                                                                                         | 4938                                                                                                          |
| Completeness to $\theta$ (%)                               | 0.984                                                                                                         | 0.985                                                                                                        | 0.952                                                                                                         |
| Data/restraints/param.                                     | 5674 / 0 / 245                                                                                                | 5620 / 0 / 245                                                                                               | 5554 / 0 / 245                                                                                                |
| Goodness-of-fit                                            | 1.023                                                                                                         | 1.193                                                                                                        | 1.027                                                                                                         |
| <i>R</i> <sub>1</sub> [ <i>I</i> >2 $\sigma$ ] (all data)  | 0.0631 (0.0839)                                                                                               | 0.0507 (0.0531)                                                                                              | 0.0237 (0.0312)                                                                                               |
| <i>wR</i> <sub>2</sub> [ <i>I</i> >2 $\sigma$ ] (all data) | 0.1598 (0.1766)                                                                                               | 0.1493 (0.1481)                                                                                              | 0.0433 (0.0454)                                                                                               |
| Largest diff. e <sup>−</sup> Å <sup>−3</sup>               | 2.92 and −2.284                                                                                               | 2.92 and −1.557                                                                                              | 0.862 and −0.772                                                                                              |

|                                                            | (11) <sub>2</sub>                                                                                            | (12) <sub>2</sub>                                                                                            | (13) <sub>2</sub>                                                                                            |
|------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|
| Formula                                                    | C <sub>38</sub> H <sub>76</sub> N <sub>4</sub> O <sub>2</sub> Pb <sub>2</sub> S <sub>2</sub> Si <sub>4</sub> | C <sub>44</sub> H <sub>76</sub> N <sub>4</sub> O <sub>2</sub> S <sub>2</sub> Si <sub>4</sub> Sn <sub>2</sub> | C <sub>44</sub> H <sub>76</sub> N <sub>4</sub> O <sub>2</sub> Pb <sub>2</sub> S <sub>2</sub> Si <sub>4</sub> |
| CCDC                                                       | 1006885                                                                                                      | 1006886                                                                                                      | 1006887                                                                                                      |
| Mol. wt.                                                   | 1211.89                                                                                                      | 1107.01                                                                                                      | 1283.94                                                                                                      |
| Crystal system                                             | triclinic                                                                                                    | monoclinic                                                                                                   | monoclinic                                                                                                   |
| Space group                                                | P -1                                                                                                         | P21/c                                                                                                        | P21/n                                                                                                        |
| <i>a</i> (Å)                                               | 8.9509(3)                                                                                                    | 15.6263(11)                                                                                                  | 13.0728(5)                                                                                                   |
| <i>b</i> (Å)                                               | 10.6875(4)                                                                                                   | 8.4904(4)                                                                                                    | 11.7745(4)                                                                                                   |
| <i>c</i> (Å)                                               | 13.9731(5)                                                                                                   | 21.1602(11)                                                                                                  | 17.6806(5)                                                                                                   |
| $\alpha$ (°)                                               | 102.1710(10)                                                                                                 | 90                                                                                                           | 90                                                                                                           |
| $\beta$ (°)                                                | 103.790(2)                                                                                                   | 107.158(3)                                                                                                   | 99.5030(10)                                                                                                  |
| $\gamma$ (°)                                               | 93.8130(10)                                                                                                  | 90                                                                                                           | 90                                                                                                           |
| <i>V</i> (Å <sup>3</sup> )                                 | 1259.36(8)                                                                                                   | 2682.5(3)                                                                                                    | 2684.15(16)                                                                                                  |
| <i>Z</i>                                                   | 1                                                                                                            | 2                                                                                                            | 4                                                                                                            |
| Density (g/cm <sup>3</sup> )                               | 1.598                                                                                                        | 1.37                                                                                                         | 1.589                                                                                                        |
| Abs. coeff., (mm <sup>-1</sup> )                           | 6.887                                                                                                        | 1.135                                                                                                        | 6.468                                                                                                        |
| <i>F</i> (000)                                             | 600                                                                                                          | 1144                                                                                                         | 1272                                                                                                         |
| Crystal size, mm                                           | 0.11 × 0.08 × 0.05                                                                                           | 0.2 × 0.08 × 0.06                                                                                            | 0.55 × 0.48 × 0.43                                                                                           |
| $\theta$ range, deg                                        | 3.09 to 27.46                                                                                                | 3.07 to 27.48                                                                                                | 3.12 to 27.48                                                                                                |
| Limiting indices                                           | −11 < <i>h</i> < 11                                                                                          | −20 < <i>h</i> < 20                                                                                          | −15 < <i>h</i> < 16                                                                                          |
|                                                            | −12 < <i>k</i> < 13                                                                                          | −10 < <i>k</i> < 11                                                                                          | −13 < <i>k</i> < 15                                                                                          |
|                                                            | −18 < <i>l</i> < 18                                                                                          | −26 < <i>l</i> < 27                                                                                          | −22 < <i>l</i> < 20                                                                                          |
| <i>R</i> (int)                                             | 0.0328                                                                                                       | 0.0713                                                                                                       | 0.0402                                                                                                       |
| Reflections collected                                      | 20184                                                                                                        | 23307                                                                                                        | 22836                                                                                                        |
| Reflec. Unique [ <i>I</i> >2 $\sigma$ ]                    | 5408                                                                                                         | 4714                                                                                                         | 5153                                                                                                         |
| Completeness to $\theta$ (%)                               | 0.994                                                                                                        | 0.998                                                                                                        | 0.987                                                                                                        |
| Data/restraints/param.                                     | 5741 / 0 / 245                                                                                               | 6117 / 0 / 272                                                                                               | 6080 / 0 / 272                                                                                               |
| Goodness-of-fit                                            | 1.042                                                                                                        | 1.028                                                                                                        | 1.044                                                                                                        |
| <i>R</i> <sub>1</sub> [ <i>I</i> >2 $\sigma$ ] (all data)  | 0.0185 (0.0206)                                                                                              | 0.0394 (0.0577)                                                                                              | 0.0233 (0.0321)                                                                                              |
| <i>wR</i> <sub>2</sub> [ <i>I</i> >2 $\sigma$ ] (all data) | 0.0432 (0.0424)                                                                                              | 0.0693 (0.0754)                                                                                              | 0.0493 (0.0515)                                                                                              |
| Largest diff. e <sup>−</sup> Å <sup>−3</sup>               | 0.966 and −0.481                                                                                             | 0.557 and −0.529                                                                                             | 1.002 and −1.14                                                                                              |

|                                                            | <b>14</b>                                                                                                     | <b>15</b>                                                                                                     | <b>(16)<sub>2</sub></b>                                                                                                      |
|------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------|
| Formula                                                    | C <sub>26</sub> H <sub>28</sub> F <sub>12</sub> N <sub>2</sub> O <sub>2</sub> Se <sub>2</sub> Sn <sub>1</sub> | C <sub>26</sub> H <sub>28</sub> F <sub>12</sub> N <sub>2</sub> O <sub>2</sub> Pb <sub>1</sub> Se <sub>2</sub> | C <sub>98</sub> H <sub>44</sub> B <sub>4</sub> F <sub>60</sub> N <sub>4</sub> O <sub>2</sub> Pb <sub>2</sub> Se <sub>2</sub> |
| CCDC                                                       | 1006888                                                                                                       | 1006889                                                                                                       | 1006890                                                                                                                      |
| Mol. wt.                                                   | 905.11                                                                                                        | 993.61                                                                                                        | 3064.91                                                                                                                      |
| Crystal system                                             | triclinic                                                                                                     | triclinic                                                                                                     | monoclinic                                                                                                                   |
| Space group                                                | P -1                                                                                                          | P -1                                                                                                          | P 2 <sub>1</sub>                                                                                                             |
| <i>a</i> (Å)                                               | 9.9934(2)                                                                                                     | 10.0061(3)                                                                                                    | 11.9038(6)                                                                                                                   |
| <i>b</i> (Å)                                               | 11.6181(2)                                                                                                    | 11.5976(3)                                                                                                    | 14.3044(7)                                                                                                                   |
| <i>c</i> (Å)                                               | 15.2377(3)                                                                                                    | 15.2067(4)                                                                                                    | 32.5897(18)                                                                                                                  |
| $\alpha$ (°)                                               | 72.2840(6)                                                                                                    | 72.1480(10)                                                                                                   | 90                                                                                                                           |
| $\beta$ (°)                                                | 82.4962(7)                                                                                                    | 83.0470(10)                                                                                                   | 90.138(2)                                                                                                                    |
| $\gamma$ (°)                                               | 67.4339(7)                                                                                                    | 67.7890(10)                                                                                                   | 90                                                                                                                           |
| <i>V</i> (Å <sup>3</sup> )                                 | 1556.04(5)                                                                                                    | 1555.06(7)                                                                                                    | 5549.3(5)                                                                                                                    |
| <i>Z</i>                                                   | 2                                                                                                             | 2                                                                                                             | 2                                                                                                                            |
| Density (g/cm <sup>3</sup> )                               | 1.932                                                                                                         | 2.122                                                                                                         | 1.834                                                                                                                        |
| Abs. coeff., (mm <sup>-1</sup> )                           | 3.261                                                                                                         | 7.866                                                                                                         | 3.836                                                                                                                        |
| <i>F</i> (000)                                             | 880                                                                                                           | 944                                                                                                           | 2936                                                                                                                         |
| Crystal size, mm                                           | 0.28 × 0.14 × 0.08                                                                                            | 0.44 × 0.27 × 0.17                                                                                            | 0.53 × 0.31 × 0.18                                                                                                           |
| $\theta$ range, deg                                        | 2.95 to 27.48                                                                                                 | 2.95 to 27.48                                                                                                 | 2.91 to 27.48                                                                                                                |
|                                                            | −12 < <i>h</i> < 12                                                                                           | −11 < <i>h</i> < 12                                                                                           | −15 < <i>h</i> < 15                                                                                                          |
| Limiting indices                                           | −15 < <i>k</i> < 15                                                                                           | −15 < <i>k</i> < 15                                                                                           | −18 < <i>k</i> < 18                                                                                                          |
|                                                            | −19 < <i>l</i> < 17                                                                                           | −18 < <i>l</i> < 19                                                                                           | −42 < <i>l</i> < 42                                                                                                          |
| <i>R</i> (int)                                             | 0.0296                                                                                                        | 0.0477                                                                                                        | 0.0512                                                                                                                       |
| Reflections collected                                      | 23659                                                                                                         | 25256                                                                                                         | 49272                                                                                                                        |
| Reflec. Unique [ <i>I</i> >2 $\sigma$ ]                    | 7026                                                                                                          | 7109                                                                                                          | 25119                                                                                                                        |
| Completeness to $\theta$ (%)                               | 98.4                                                                                                          | 99.6                                                                                                          | 99.6                                                                                                                         |
| Data/restraints/param.                                     | 7026 / 0 / 410                                                                                                | 7109 / 0 / 410                                                                                                | 25119 / 4 / 722                                                                                                              |
| Goodness-of-fit                                            | 1.020                                                                                                         | 1.024                                                                                                         | 1.021                                                                                                                        |
| <i>R</i> <sub>1</sub> [ <i>I</i> >2 $\sigma$ ] (all data)  | 0.0212 (0.0269)                                                                                               | 0.0265 (0.0325)                                                                                               | 0.0604 (0.0673)                                                                                                              |
| <i>wR</i> <sub>2</sub> [ <i>I</i> >2 $\sigma$ ] (all data) | 0.0455 (0.0477)                                                                                               | 0.0530 (0.0546)                                                                                               | 0.1561 (0.1612)                                                                                                              |
| Largest diff. e <sup>−</sup> Å <sup>−3</sup>               | 0.460 and −0.342                                                                                              | 1.021 and −1.010                                                                                              | 1.541 and −2.065                                                                                                             |

|                                                            | Ge(SePh) <sub>2</sub> [N(SiMe <sub>3</sub> ) <sub>2</sub> ] <sub>2</sub>                       | Ge(TePh) <sub>2</sub> [N(SiMe <sub>3</sub> ) <sub>2</sub> ] <sub>2</sub>                       | Sn(TePh) <sub>2</sub> [N(SiMe <sub>3</sub> ) <sub>2</sub> ] <sub>2</sub>                       |
|------------------------------------------------------------|------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------|
| Formula                                                    | C <sub>24</sub> H <sub>46</sub> Ge <sub>1</sub> N <sub>2</sub> Se <sub>2</sub> Si <sub>4</sub> | C <sub>24</sub> H <sub>46</sub> Ge <sub>1</sub> N <sub>2</sub> Si <sub>4</sub> Te <sub>2</sub> | C <sub>24</sub> H <sub>46</sub> N <sub>2</sub> Si <sub>4</sub> Sn <sub>1</sub> Te <sub>2</sub> |
| CCDC                                                       | 1006891                                                                                        | 1006892                                                                                        | 1006894                                                                                        |
| Mol. wt.                                                   | 705.50                                                                                         | 802.78                                                                                         | 848.88                                                                                         |
| Crystal system                                             | triclinic                                                                                      | monoclinic                                                                                     | monoclinic                                                                                     |
| Space group                                                | P -1                                                                                           | C 1 2/c 1                                                                                      | C 2/c                                                                                          |
| <i>a</i> (Å)                                               | 8.9113(4)                                                                                      | 8.6866(2)                                                                                      | 8.7218(3)                                                                                      |
| <i>b</i> (Å)                                               | 11.4671(4)                                                                                     | 17.9348(4)                                                                                     | 17.9841(5)                                                                                     |
| <i>c</i> (Å)                                               | 17.4235(6)                                                                                     | 21.2488(5)                                                                                     | 21.6618(6)                                                                                     |
| $\alpha$ (°)                                               | 97.6430(10)                                                                                    | 90                                                                                             | 90                                                                                             |
| $\beta$ (°)                                                | 94.825(2)                                                                                      | 98.1587(9)                                                                                     | 98.7190(10)                                                                                    |
| $\gamma$ (°)                                               | 112.1520(10)                                                                                   | 90                                                                                             | 90                                                                                             |
| <i>V</i> (Å <sup>3</sup> )                                 | 1616.80(11)                                                                                    | 3276.90(13)                                                                                    | 3358.47(18)                                                                                    |
| <i>Z</i>                                                   | 2                                                                                              | 4                                                                                              | 4                                                                                              |
| Density (g/cm <sup>3</sup> )                               | 1.449                                                                                          | 1.627                                                                                          | 1.679                                                                                          |
| Abs. coeff., (mm <sup>-1</sup> )                           | 3.363                                                                                          | 2.842                                                                                          | 2.622                                                                                          |
| <i>F</i> (000)                                             | 720                                                                                            | 1584                                                                                           | 1656                                                                                           |
| Crystal size, mm                                           | 0.48 × 0.25 × 0.18                                                                             | 0.41 × 0.26 × 0.06                                                                             | 0.57 × 0.36 × 0.31                                                                             |
| $\theta$ range, deg                                        | 2.92 to 27.48                                                                                  | 2.91 to 27.48                                                                                  | 2.96 to 27.47                                                                                  |
|                                                            | −11 < <i>h</i> < 11                                                                            | −11 < <i>h</i> < 10                                                                            | −10 < <i>h</i> < 11                                                                            |
| Limiting indices                                           | −14 < <i>k</i> < 14                                                                            | −22 < <i>k</i> < 23                                                                            | −23 < <i>k</i> < 18                                                                            |
|                                                            | −21 < <i>l</i> < 22                                                                            | −24 < <i>l</i> < 27                                                                            | −27 < <i>l</i> < 28                                                                            |
| <i>R</i> (int)                                             | 0.0406                                                                                         | 0.0393                                                                                         | 0.0273                                                                                         |
| Reflections collected                                      | 18080                                                                                          | 14204                                                                                          | 14291                                                                                          |
| Reflec. Unique [ <i>I</i> >2 $\sigma$ ]                    | 7359                                                                                           | 3758                                                                                           | 3815                                                                                           |
| Completeness to $\theta$ (%)                               | 99.2                                                                                           | 99.9                                                                                           | 98.9                                                                                           |
| Data/restraints/param.                                     | 7359 / 0 / 310                                                                                 | 3758 / 0 / 156                                                                                 | 3815 / 0 / 156                                                                                 |
| Goodness-of-fit                                            | 1.033                                                                                          | 1.071                                                                                          | 1.111                                                                                          |
| <i>R</i> <sub>1</sub> [ <i>I</i> >2 $\sigma$ ] (all data)  | 0.0404 (0.0553)                                                                                | 0.0226 (0.0269)                                                                                | 0.0231 (0.0273)                                                                                |
| <i>wR</i> <sub>2</sub> [ <i>I</i> >2 $\sigma$ ] (all data) | 0.0913 (0.0961)                                                                                | 0.0580 (0.0598)                                                                                | 0.0516 (0.0529)                                                                                |
| Largest diff. e <sup>−</sup> Å <sup>−3</sup>               | 0.726 and −0.659                                                                               | 0.466 and −0.402                                                                               | 0.673 and −0.601                                                                               |

| <hr/> [ {2-(Me <sub>2</sub> NCH <sub>2</sub> )C <sub>6</sub> H <sub>4</sub> SeCH <sub>2</sub> C(Me) <sub>2</sub> OH}H] <sup>+</sup> ·[H <sub>2</sub> N{B(C <sub>6</sub> F <sub>5</sub> ) <sub>3</sub> } <sub>2</sub> ] <sup>-</sup> <hr/> |                                                                                                              |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|
| Formula                                                                                                                                                                                                                                   | C <sub>49</sub> H <sub>24</sub> B <sub>2</sub> F <sub>30</sub> N <sub>2</sub> O <sub>1</sub> Se <sub>1</sub> |
| CCDC                                                                                                                                                                                                                                      | 1006893                                                                                                      |
| Mol. wt.                                                                                                                                                                                                                                  | 1327.28                                                                                                      |
| Crystal system                                                                                                                                                                                                                            | monoclinic                                                                                                   |
| Space group                                                                                                                                                                                                                               | P 21/c                                                                                                       |
| <i>a</i> (Å)                                                                                                                                                                                                                              | 16.5296(6)                                                                                                   |
| <i>b</i> (Å)                                                                                                                                                                                                                              | 15.9662(6)                                                                                                   |
| <i>c</i> (Å)                                                                                                                                                                                                                              | 20.9740(7)                                                                                                   |
| <i>α</i> (°)                                                                                                                                                                                                                              | 90                                                                                                           |
| <i>β</i> (°)                                                                                                                                                                                                                              | 105.7030(10)                                                                                                 |
| <i>γ</i> (°)                                                                                                                                                                                                                              | 90                                                                                                           |
| <i>V</i> (Å <sup>3</sup> )                                                                                                                                                                                                                | 5328.8(3)                                                                                                    |
| <i>Z</i>                                                                                                                                                                                                                                  | 4                                                                                                            |
| Density (g/cm <sup>3</sup> )                                                                                                                                                                                                              | 1.654                                                                                                        |
| Abs. coeff., (mm <sup>-1</sup> )                                                                                                                                                                                                          | 0.850                                                                                                        |
| <i>F</i> (000)                                                                                                                                                                                                                            | 2616                                                                                                         |
| Crystal size, mm                                                                                                                                                                                                                          | 0.1 × 0.05 × 0.04                                                                                            |
| <i>θ</i> range, deg                                                                                                                                                                                                                       | 2.96 to 27.49                                                                                                |
| Limiting indices                                                                                                                                                                                                                          | −18 < <i>h</i> < 21                                                                                          |
|                                                                                                                                                                                                                                           | −20 < <i>k</i> < 20                                                                                          |
|                                                                                                                                                                                                                                           | −25 < <i>l</i> < 23                                                                                          |
| <i>R</i> (int)                                                                                                                                                                                                                            | 0.0785                                                                                                       |
| Reflections collected                                                                                                                                                                                                                     | 28791                                                                                                        |
| Reflec. Unique [ <i>I</i> >2σ]                                                                                                                                                                                                            | 11484                                                                                                        |
| Completeness to <i>θ</i> (%)                                                                                                                                                                                                              | 96.4                                                                                                         |
| Data/restraints/param.                                                                                                                                                                                                                    | 11484 / 12 / 766                                                                                             |
| Goodness-of-fit                                                                                                                                                                                                                           | 0.886                                                                                                        |
| <i>R</i> <sub>1</sub> [ <i>I</i> >2σ] (all data)                                                                                                                                                                                          | 0.0646 (0.1592)                                                                                              |
| <i>wR</i> <sub>2</sub> [ <i>I</i> >2σ] (all data)                                                                                                                                                                                         | 0.1380 (0.1670)                                                                                              |
| Largest diff. e·Å <sup>-3</sup>                                                                                                                                                                                                           | 0.678 and −0.449                                                                                             |

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