

Supporting information for

**Synchrotron XRD and NMR evidence of germanium redistribution during silylation of
BEC-type germanosilicate**

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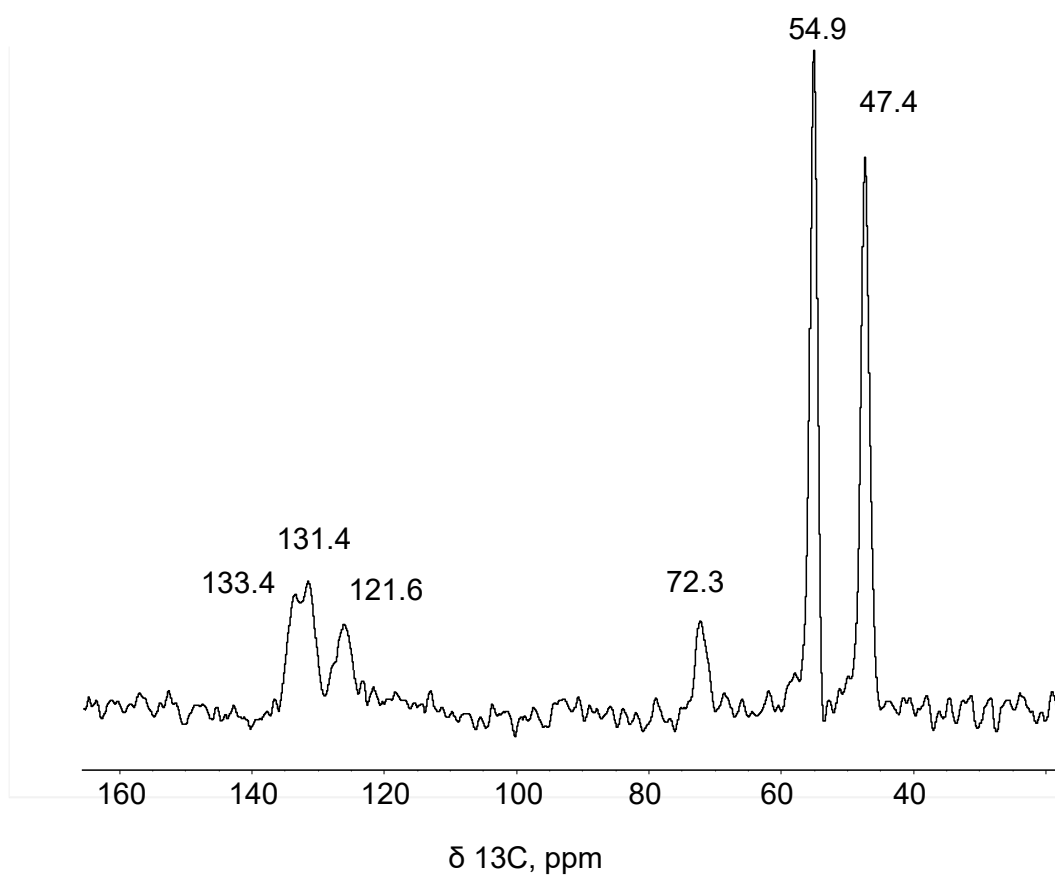


Fig. S1. ^{13}C CP MAS NMR spectra of as-made Ge-BEC.

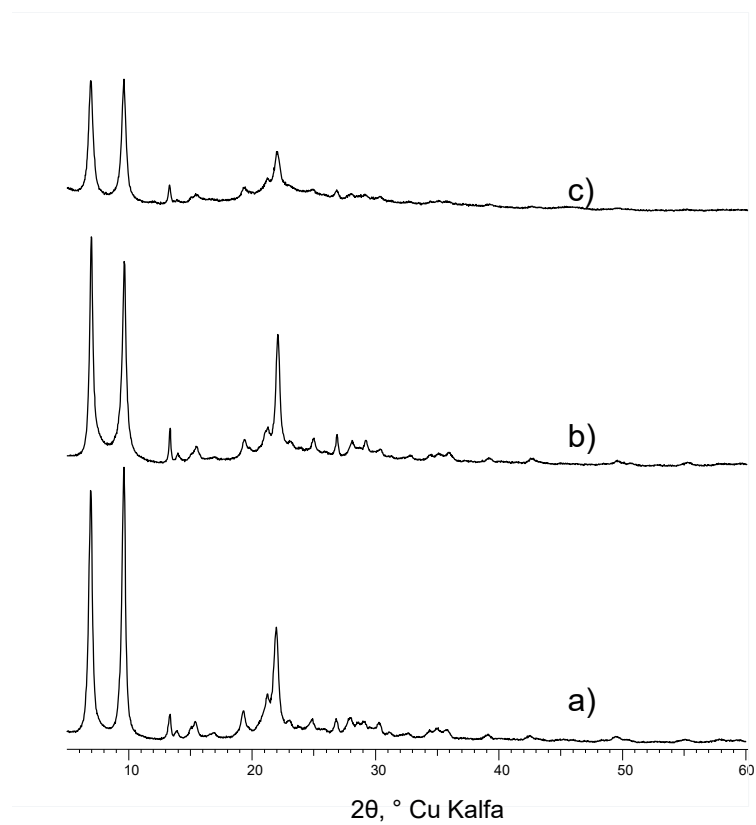


Fig. S2. XRD patterns for calcined Ge-BEC stored for: a) 1 day, b) 5 months, c) 12 months after calcination.

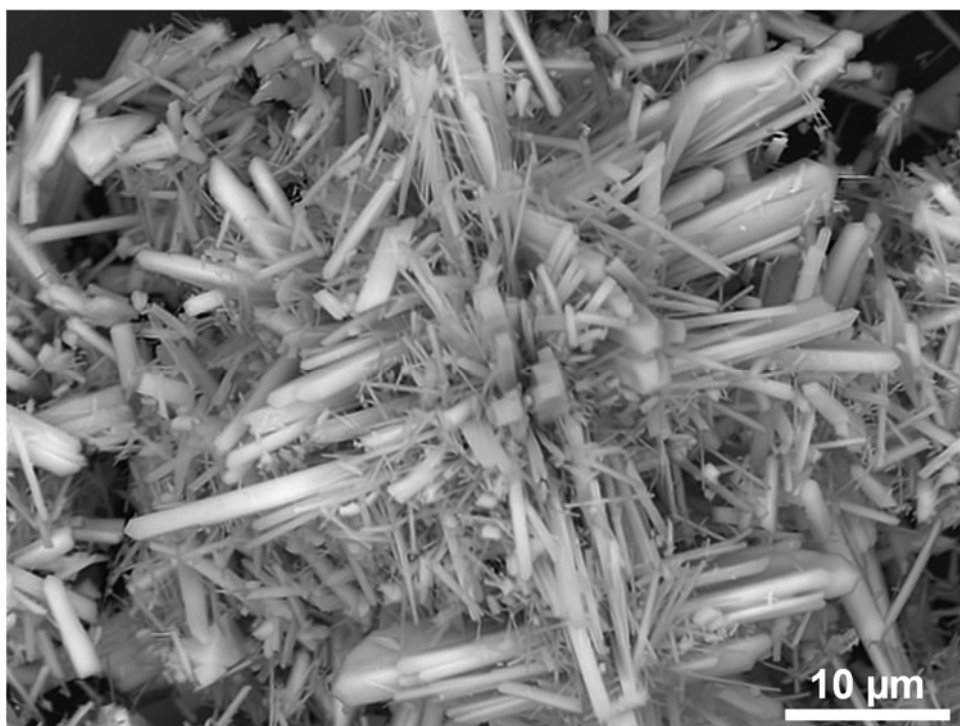


Fig. S3. SEM image of calcined Ge-BEC sample.

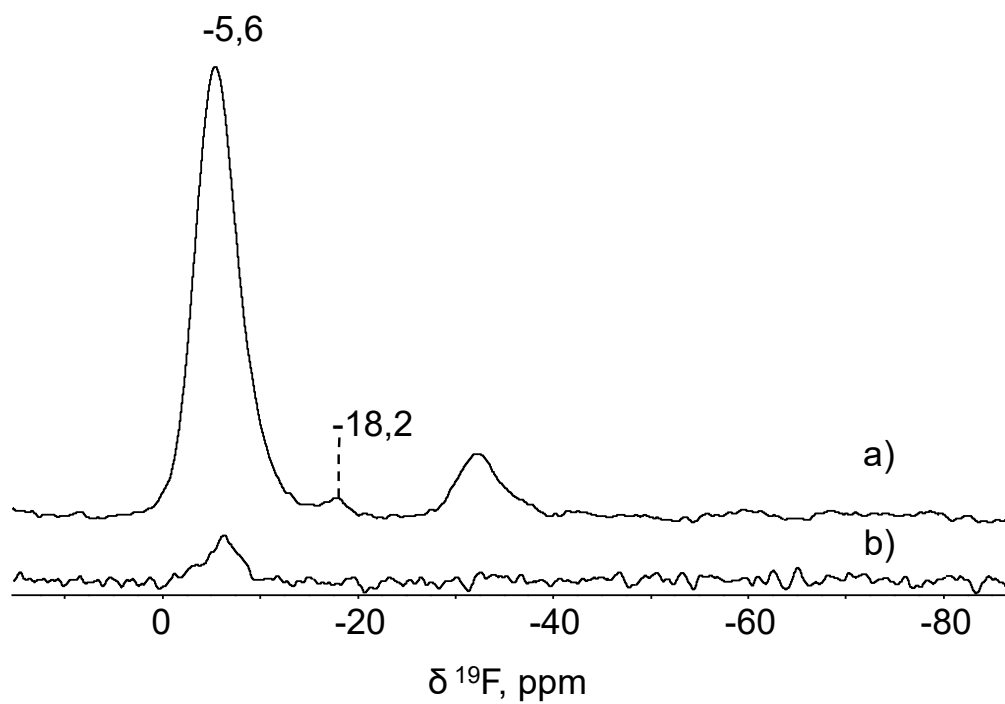


Fig. S4. ^{19}F MAS NMR spectra of a) as-made Ge-BEC and b) calcined Ge-BEC samples.

Table S1. Atomic coordinates and s.o.f. for atomic positions in the crystal structure of as-made Ge-BEC sample (see also Table 1 for the unit cell dimensions).

In the Rietveld refinement, for the atoms T1-T3 and O1-O7 one common U_{iso} was refined to $0.018(3) \text{ \AA}^{-2}$, and for the rest atoms another common U_{iso} was refined to $0.10(1) \text{ \AA}^{-2}$.

	<i>x</i>	<i>y</i>	<i>z</i>	s.o.f.
T1	0.3797(5)	0.1209(5)	0.1158(4)	1
T2	0.1929(5)	0.1929(5)	0.25	1
T3	0.0	0.1232(6)	0.1173(5)	1
O1	0.0	0.0	0.1381(5)	1
O2	0.1012(4)	0.1797(5)	0.1680(5)	1
O3	0.3455(6)	0.0	0.1348(4)	1
O4	0.5	0.1461(5)	0.1517(5)	1
O5	0.3039(5)	0.1854(6)	0.1952(5)	1
O6	0.3569(4)	0.1598(5)	0.0	1
O7	0.0	0.1532(4)	0.0	1
N14	0.458(3)	0.223(4)	0.485(3)	0.17(1)
N15	0.570(3)	0.061(4)	0.525(4)	0.17(1)
C16	0.506(3)	0.180(3)	0.389(4)	0.17(1)
C17	0.552(5)	0.071(3)	0.415(4)	0.17(1)
C18	0.622(4)	0.156(4)	0.559(3)	0.17(1)
C19	0.547(4)	0.250(4)	0.558(3)	0.17(1)
C20	0.392(4)	0.139(4)	0.533(3)	0.17(1)
C21	0.468(4)	0.055(3)	0.574(5)	0.17(1)
C22	0.391(4)	0.317(3)	0.466(3)	0.17(1)
C23	0.442(5)	0.403(4)	0.407(3)	0.17(1)
C24	0.403(4)	0.439(3)	0.316(4)	0.17(1)
C25	0.444(4)	0.524(3)	0.266(4)	0.17(1)
C26	0.527(3)	0.578(4)	0.309(4)	0.17(1)
C27	0.563(3)	0.549(3)	0.403(3)	0.17(1)
C28	0.526(4)	0.459(4)	0.447(3)	0.17(1)
F1	0.0	0.5	0.5	0.90(2)
O8w	0.5	0.709(7)	0.400(8)	0.22(3)

Table S2. Si/Ge ratios in Ge-BEC, Ge-BEC-Si-343 and Ge-BEC-Si-373.

	BEC-7	Ge-BEC-Si-343	Ge-BEC-Si-373
T1	52(1)/48(1)	55(2)/45(2)	52(1)/48(1)
T2	81(1)/19(1)	82(2)/18(2)	85(1)/15(1)
T3	94(2)/6(2)	95(2)/5(2)	100(2)/0