Electronic Supplementary Information

Immobilization of Isolated Dimethyltin Species on Crystalline Silicates through Surface Modification of Layered Octosilicate

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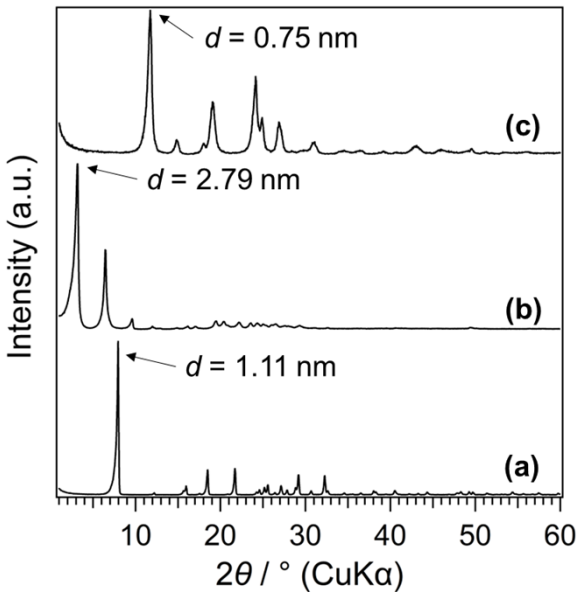


Fig. S1 Powder XRD patterns of (a) Na-Oct, (b) C_{16}TMA-Oct, and (c) H-Oct.

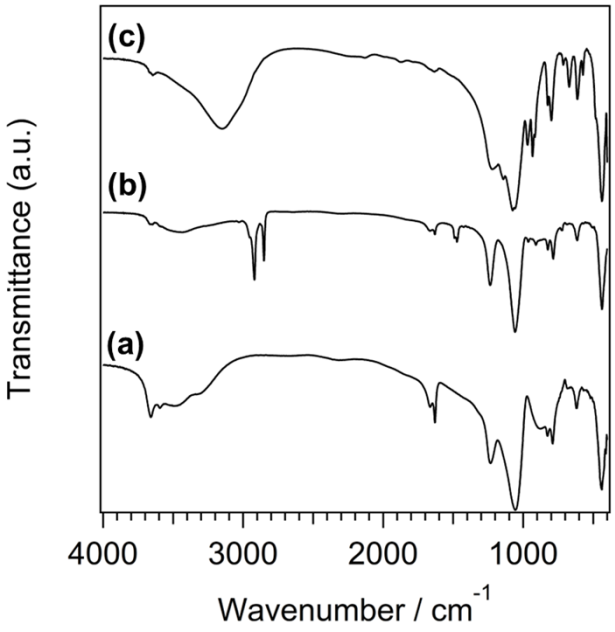


Fig. S2 FT-IR spectra of (a) Na-Oct, (b) C_{16}TMA-Oct, and (c) H-Oct.

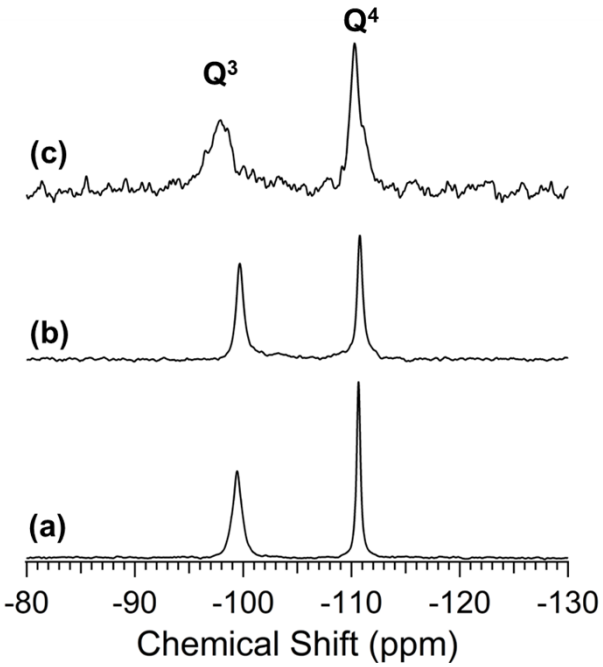


Fig. S3 ^{29}Si MAS NMR spectra of (a) Na-Oct, (b) C_{16}TMA-Oct, and (c) H-Oct.

Table S1 Chemical shifts and integral intensity ratios of the ^{29}Si MAS NMR signals (Fig. S3) for Na-Oct, C_{16}TMA-Oct, and H-Oct.

Samples	Chemical Shift (ppm)		Integral intensity ratio
	Q^3	Q^4	Q^3/Q^4
Na-Oct	−100	−111	1.00
C_{16}TMA-Oct	−100	−111	1.00
H-Oct	−98	−110	1.00

Table S2 Carbon, nitrogen, silicon, and sodium contents in Na-Oct, C_{16}TMA-Oct, H-Oct, and Me_2Si-Oct.

Samples	C / wt%	N / wt%	Si / wt%	Na / wt%
Na-Oct	-	-	30.6	5.4
C_{16}TMA-Oct	36.8	2.4	18.7	0.3
H-Oct	-	-	41.9	0.5
Me_2Si-Oct	5.2	0.3	40.6	-

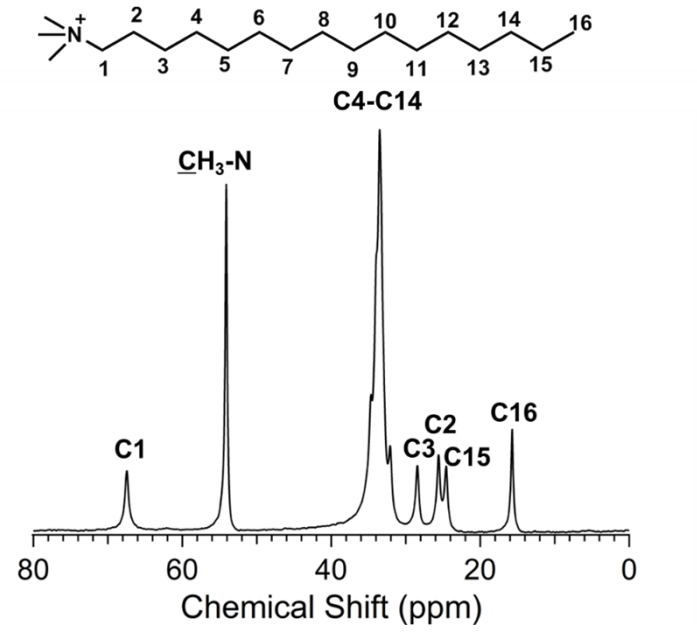


Fig. S4 ^{13}C CP/MAS NMR spectrum of C_{16}TMA-Oct.
The signals were assigned according to the previous report.[1]

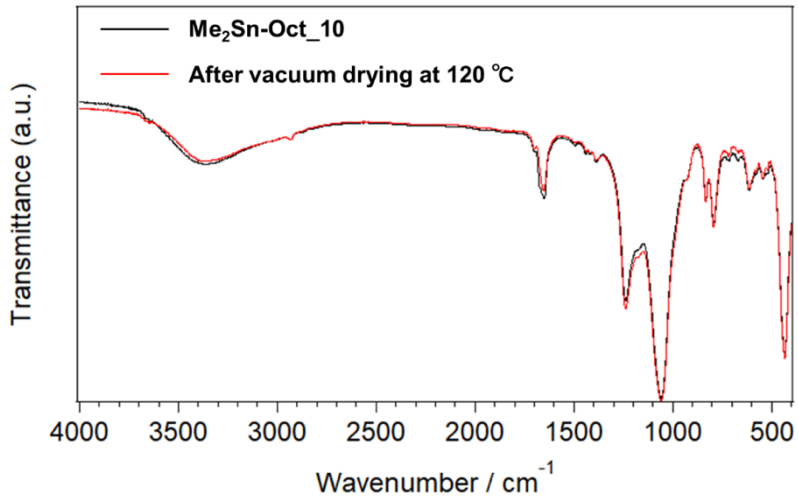
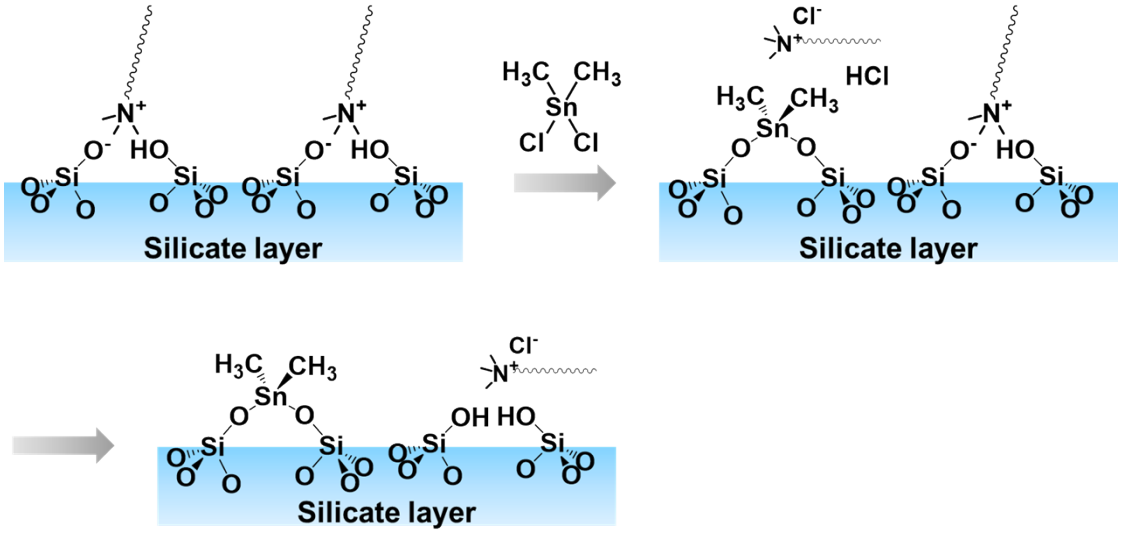


Fig. S5 FT-IR spectra of (black line) Me_2Sn-Oct_10 and (red line) Me_2Sn-Oct_10 after drying.



Scheme S1 Possible reaction scheme for the elimination of two $C_{16}TMA^+$ cations by the reaction of one Me_2SnCl_2 molecule.

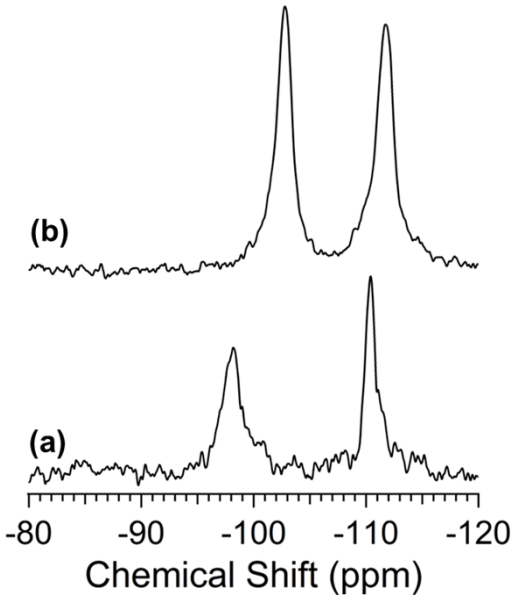


Fig. S6 ^{29}Si MAS NMR spectra of (a) H-Oct and (b) H-Oct_heat.

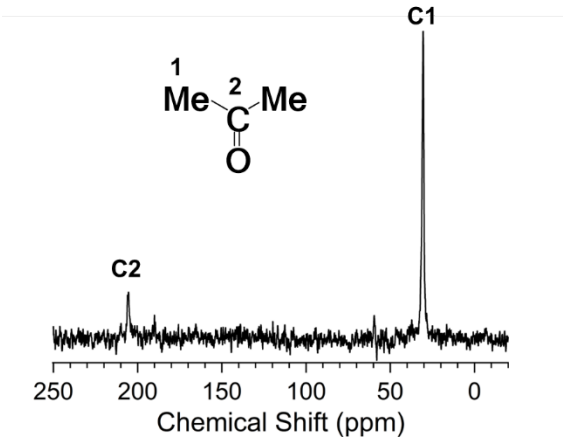


Fig. S7 ^{13}C CP/MAS NMR spectrum of H-Oct_heat.

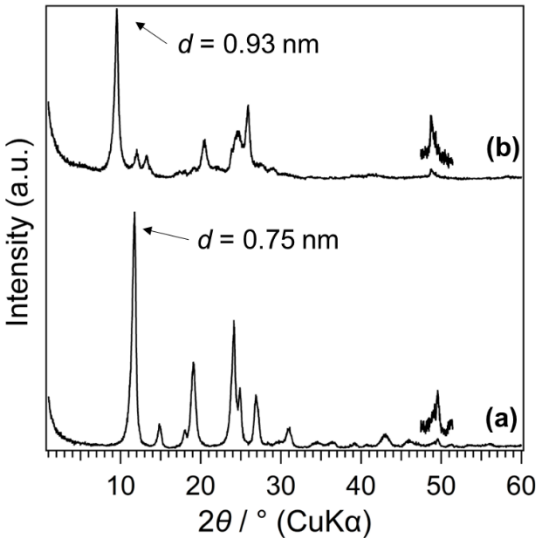


Fig. S8 Powder XRD patterns of (a) H-Oct and (b) H-Oct_heat.

H-Oct and H-Oct_heat were analyzed by ^{13}C CP/MAS NMR (Fig. S7) and powder XRD (Fig. S8). The presence of acetone in H-Oct_heat was confirmed by ^{13}C NMR spectrum. The basal spacing of H-Oct_heat increased from that of H-Oct, which was generally consistent with the profile of the XRD pattern for acetone-adsorbed H-octosilicate reported previously.[2] These results suggested that acetone as a washing solvent was intercalated between the layers.

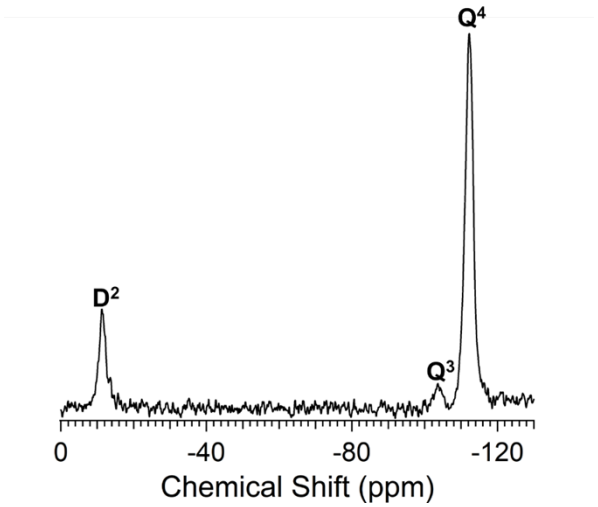


Fig. S9 ^{29}Si MAS NMR spectrum of Me_2Si-Oct.

Table S3 Integral intensity ratios of the ^{29}Si MAS NMR signals (Fig. S9) and the degree of silylation for Me_2Si-Oct.

Samples	Integral intensity ratio			Degree of silylation
	D^2	Q^3	Q^4	/ %
Me_2Si-Oct	0.43	0.12	1.88	88

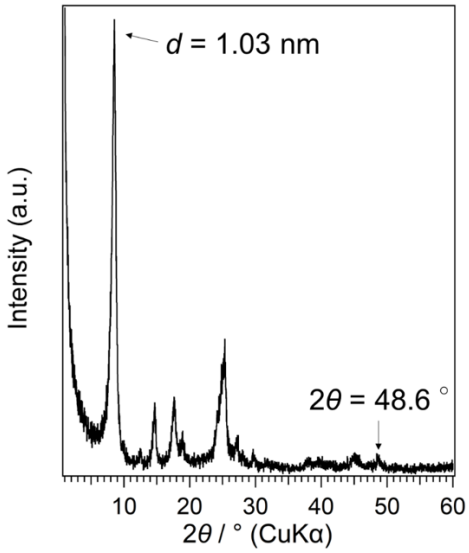
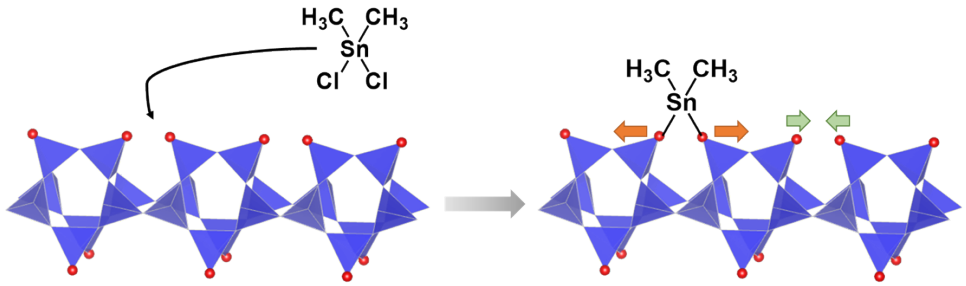
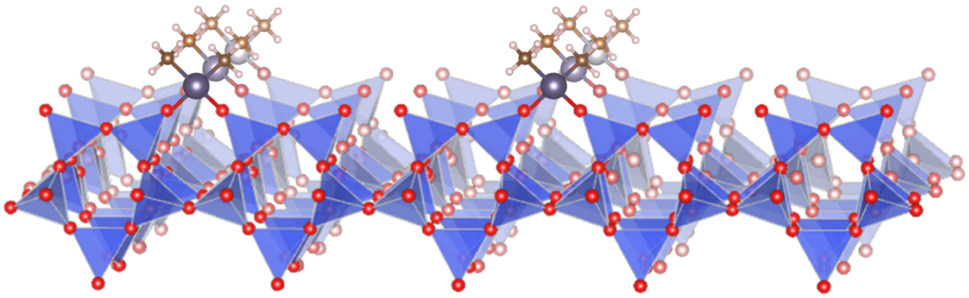


Fig. S10 Powder XRD pattern of Me_2Si-Oct.

(a)



(b)



Scheme S2 (a) A possible mechanism that explains the limited degree of dimethyltin-modification (approximately 50%). (b) A possible structure of Me_2Sn-Oct_10.

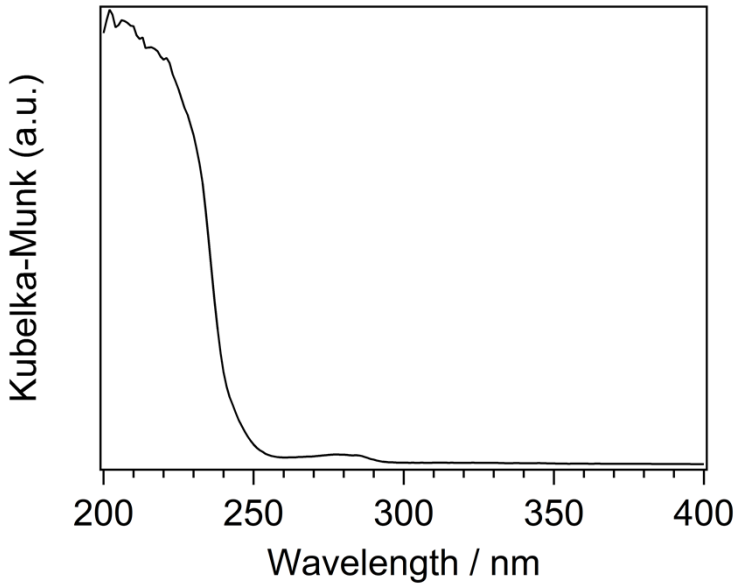


Fig. S11 UV–Vis spectrum of dimethyltin oxide.

Table S4 The values of the absorption edge for each spectrum.

Samples	Absorption edges / eV
Sn foil	29195.0
SnO	29196.8
SnO_2	29200.6
DMTO	29198.6
Me_2Sn-Oct_0.1	29197.2
Me_2Sn-Oct_0.25	29197.1
Me_2Sn-Oct_0.5	29197.2
Me_2Sn-Oct_10	29197.2

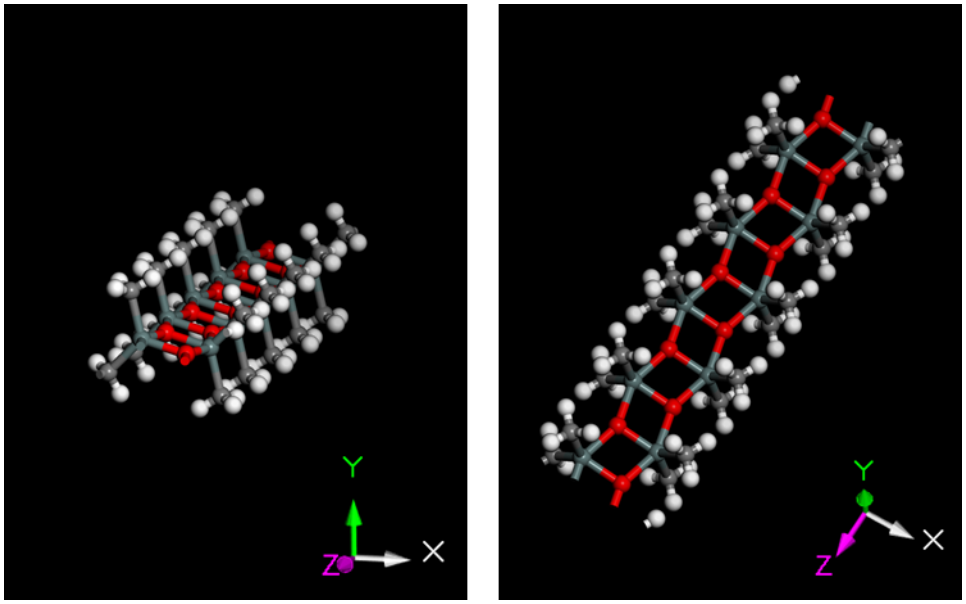


Fig. S12 A structural model of dimethyltin oxide.

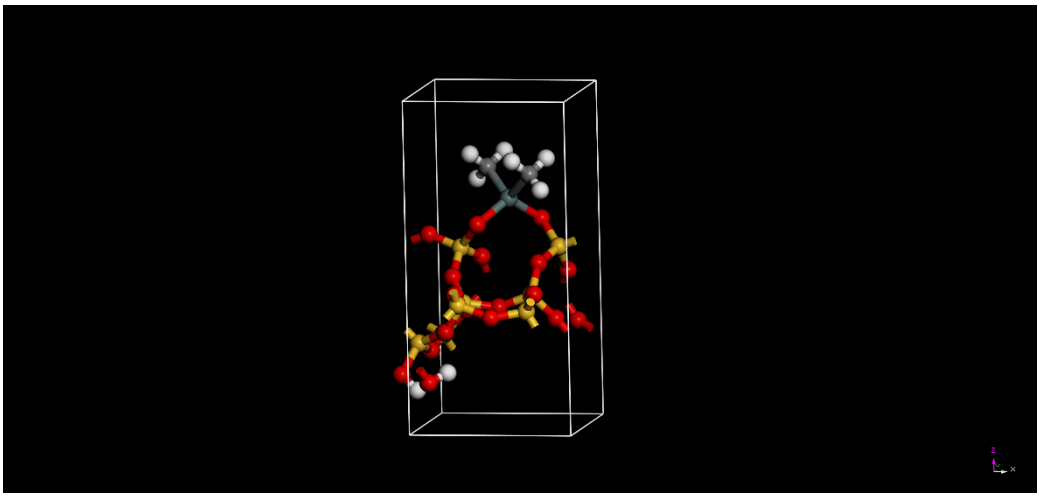


Fig. S13 A structural model of Me_2Sn-Oct_*X*.

References

1 B. B. Kharkov and S. V. Dvinskikh, *J. Phys. Chem. C*, 2013, **117**, 24511–24517.

2 M. Borowski, O. Kovalev and H. Gies, *Microporous Mesoporous Mater.*, 2008, **107**, 71–80.