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Synthesis of Janus cube containing Si-H moieties

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Synthesis of Si-H/Janus cube 8

To a mixture of diethyl ether (150 ml), triethylamine (14 ml, 100 mmol) and trichlorosilane (8.8 ml, 90 mmol) was added a diethyl ether solution (150 ml) of *all-cis*-[$Bu^{i}SiO(OH)]_4$ **6** (9.5 g, 20 mmol) at 0 ºC. After stirring at room temperature for 24 h, the solvents were removed under reduced pressure. Hexane was added to the residue, and the resulting suspension was filtered through Celite® to remove inorganic salts to give the crude material containing *all-cis*-[$Bu^{i}SiO(OSiHCl_2)]_4$ **7** which was used for the next step without purification. To a mixture of water (100 ml) and acetone (800 ml) was added a ethereal solution (200 ml) of the material from the previous step at 0 ºC, and the mixture was then stirred at room temperature for 2 days. The organic solvents were removed under reduced pressure. Extraction of the residue with toluene/water gave clear solution. The organic layer was concentrated and purified by recycle-type preparative HPLC (GPC, JAIGEL 1H+2H, eluent: toluene) to afford product **8** (102 mg, 0.16 mmol, 0.8%).

White solid, mp 80 °C. ^{1}H NMR (C_6D_6) δ: 0.73 (d, $^{3}J_{H\text{-}H}$ = 6.9 Hz, 8H), 1.01 (d, $^{3}J_{H\text{-}H}$ = 6.5 Hz, 24H), 1.93-2.02 (m, $^{3}J_{H\text{-}H}$ = 6.9 Hz, 4H), 4.45 (s, $^{1}J_{Si\text{-}H}$ = 166 Hz, 4H). ^{13}C NMR (C_6D_6) δ: 22.6 (CH_2), 24.2 (CH), 25.8 (CH_3). ^{29}Si NMR (C_6D_6) δ:–67.3, –84.5; IR (KBr): 478.31, 613.32, 688.54, 742.54, 777.26, 837.05, 1031.85, 1110.92(Si-O), 1230.50, 1332.72, 1465.80, 1627.81, 2268.13(Si-H), 2871.81, 2925.81, 2954.74, 3419.56 cm^{-1}. HRMS *m*/*z*: $[M+Na]^+$ 671.05660. Elemental Analysis: Found (%): C, 29.99; H, 6.50. Calc. for $C_{16}H_{40}O_{12}Si_8$ (%): C, 29.60; H, 6.21. Elemental analyses and high-resolution MS were carried out at the Microanalytical Laboratory of the Institute for Chemical Research, Kyoto University.

NMR and HRMS charts

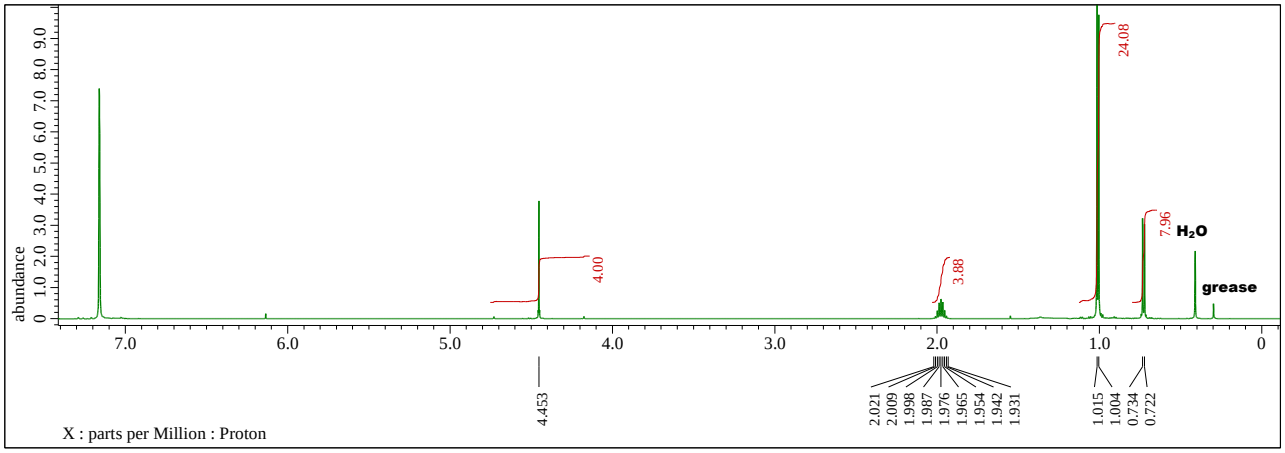

Figure S1. ^{1}H NMR (600 MHz, C_6D_6) spectrum of **8**

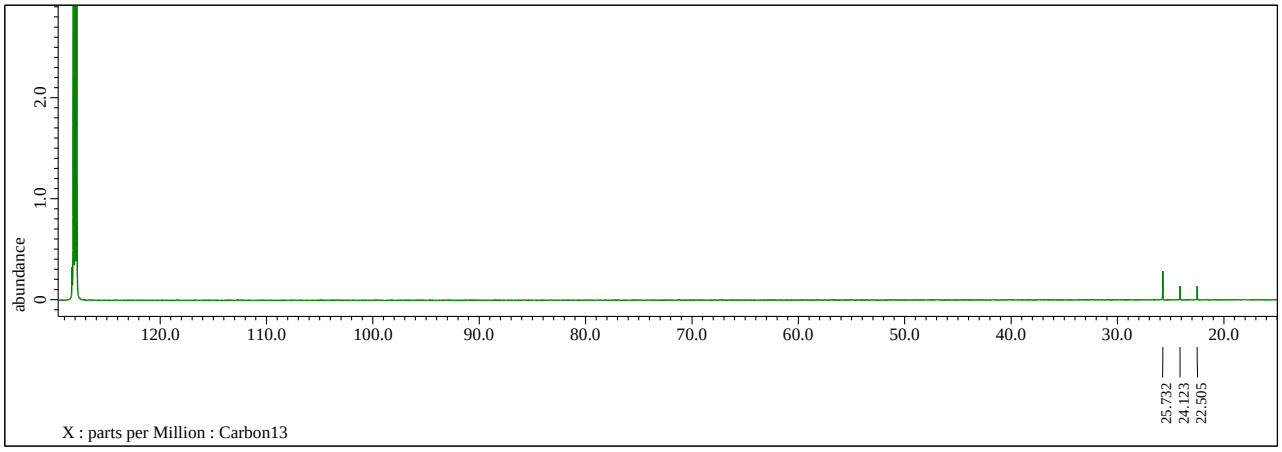

Figure S2. ^{13}C NMR (151 MHz, C_6D_6) spectrum of **8**

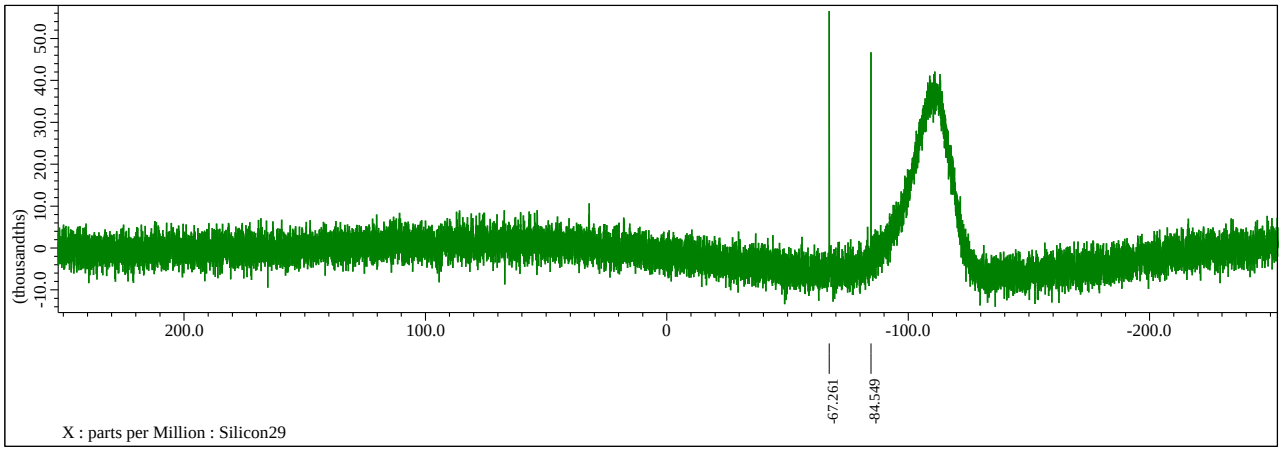

Figure S3. ^{29}Si NMR (119 MHz, C6D6) spectrum of **8**

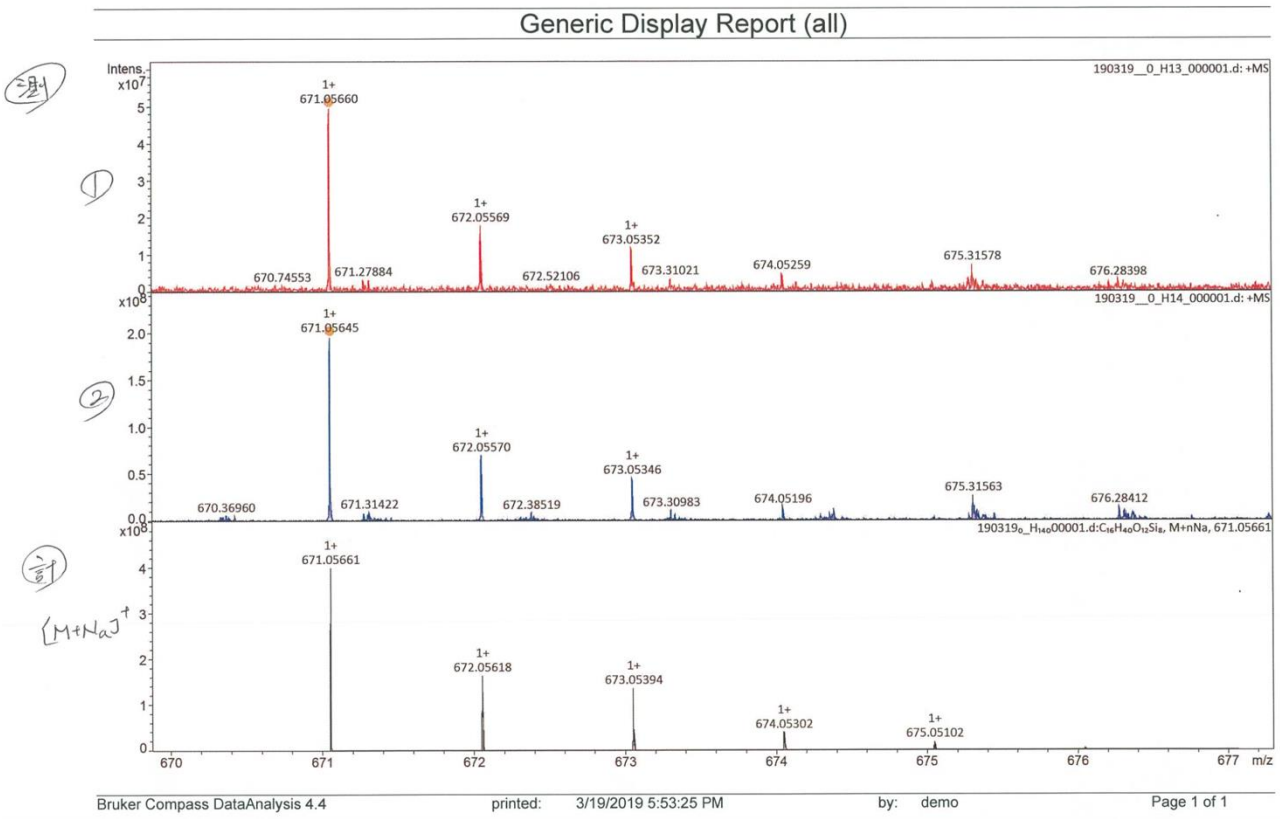

Figure S4. HRMS of **8**