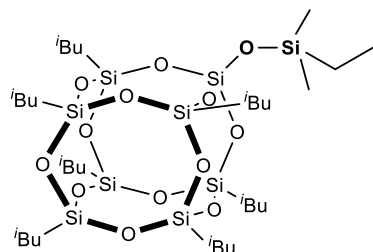


Ru-Catalyzed Dehydrogenative Silylation of POSS-Silanols with Hydrosilanes: Its Introduction to One-Pot Synthesis

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The spectroscopic data of obtained silsesquioxane derivatives



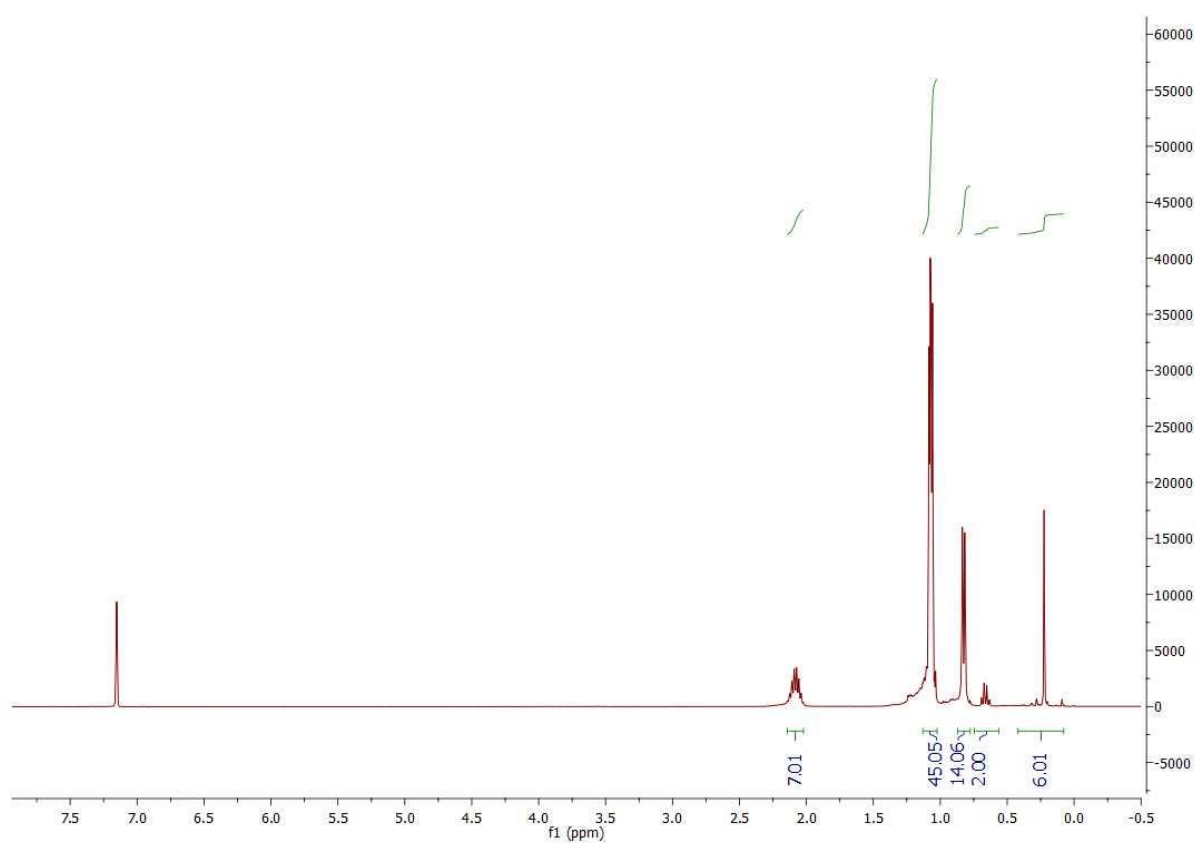
Compound (**1**) was obtained as solid in 98% yield.

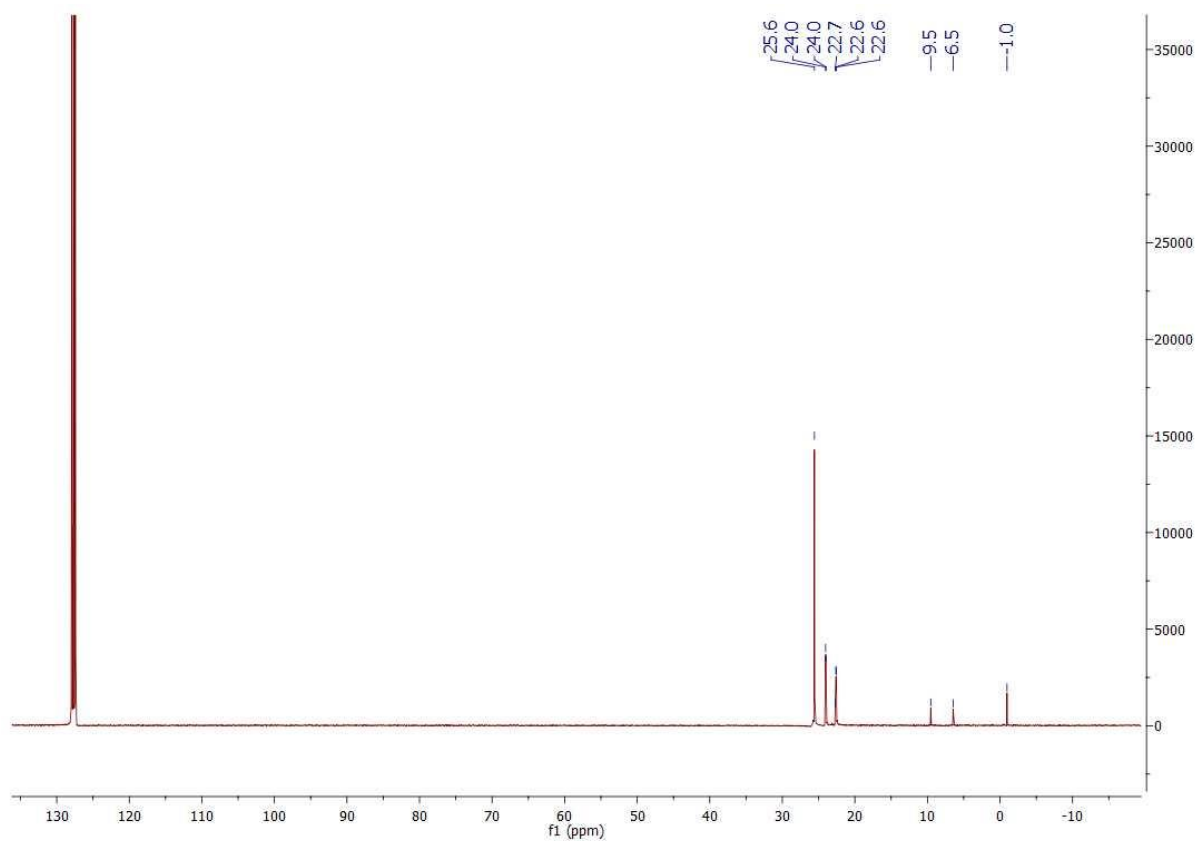
^1H NMR (400 MHz, C_6D_6) δ (ppm) = 0.23 (s, 6H), 0.66 (q, $J = 7.9$ Hz, 2H), 0.83 (d, $J = 7.0$ Hz, 14H), 1.03- 1.11 (m, 45H), 2.01-2.14 (m, 7H).

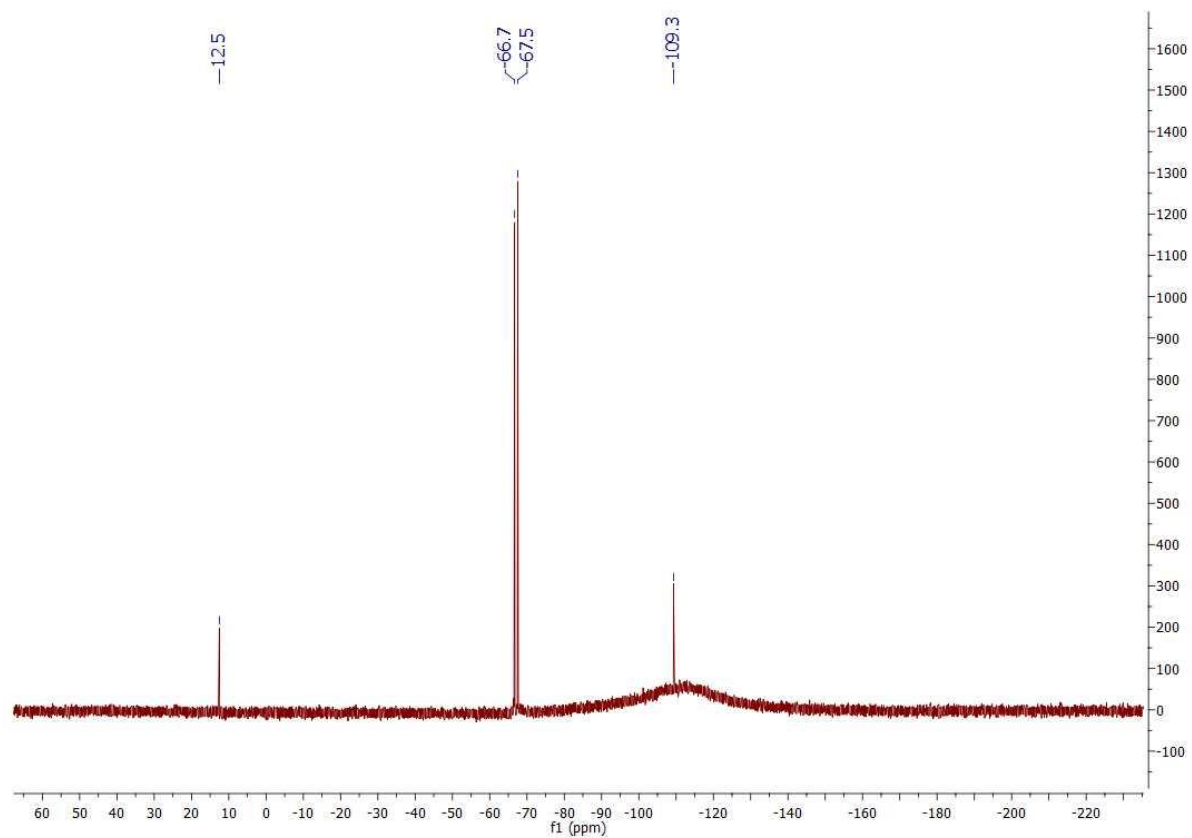
^{13}C NMR (101 MHz, C_6D_6) δ (ppm) = 1.0, 6.5, 9.5, 22.6, 22.6, 22.7, 24.0, 24.0, 25.6.

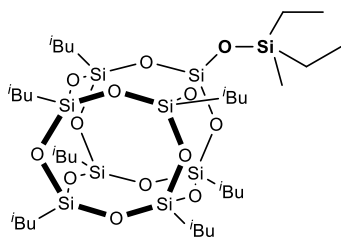
^{29}Si NMR (79 MHz, C_6D_6) δ (ppm) = 12.5, -66.7, -67.5, -109.3.

EA: $\text{C}_{32}\text{H}_{74}\text{O}_{13}\text{Si}_9$ (918.305): calcd. C 41.79, H 8.11; found C 41.54, H 8.21.









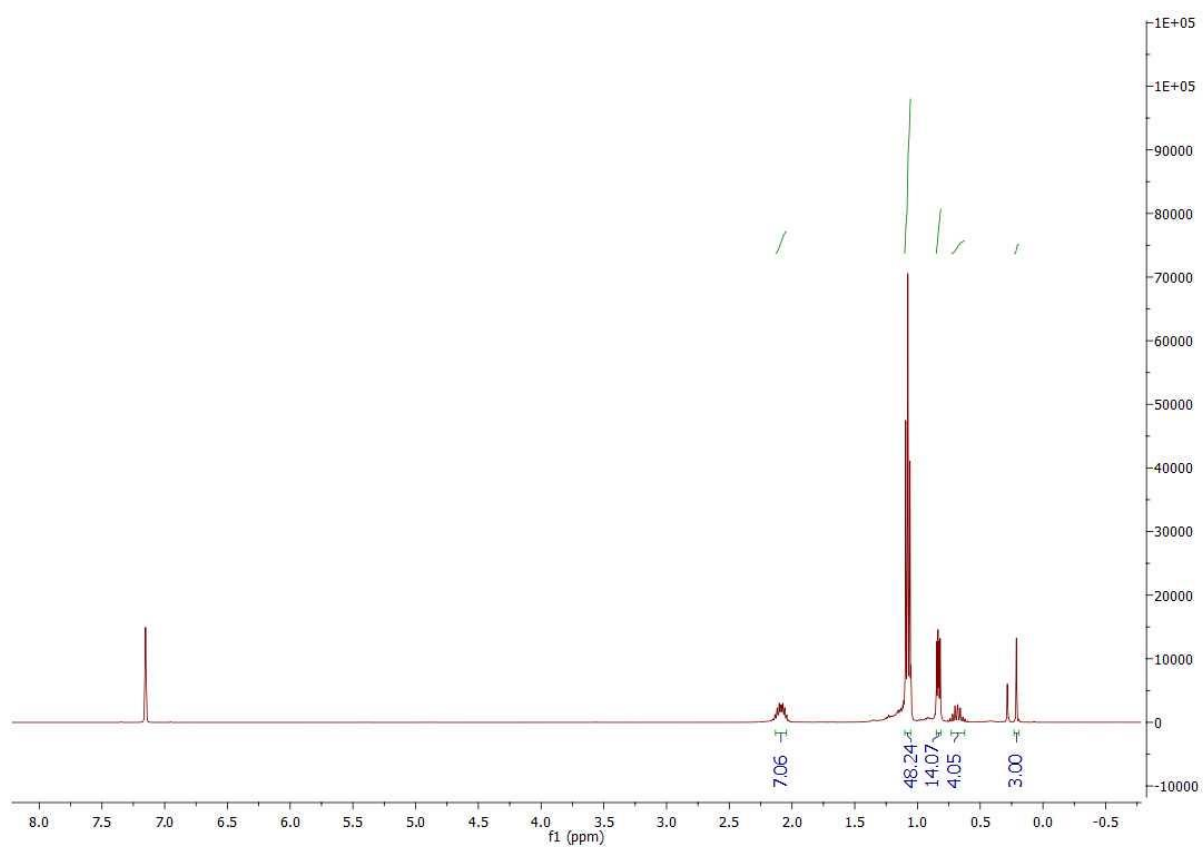
Compound (**2**) was obtained as solid in 92% yield.

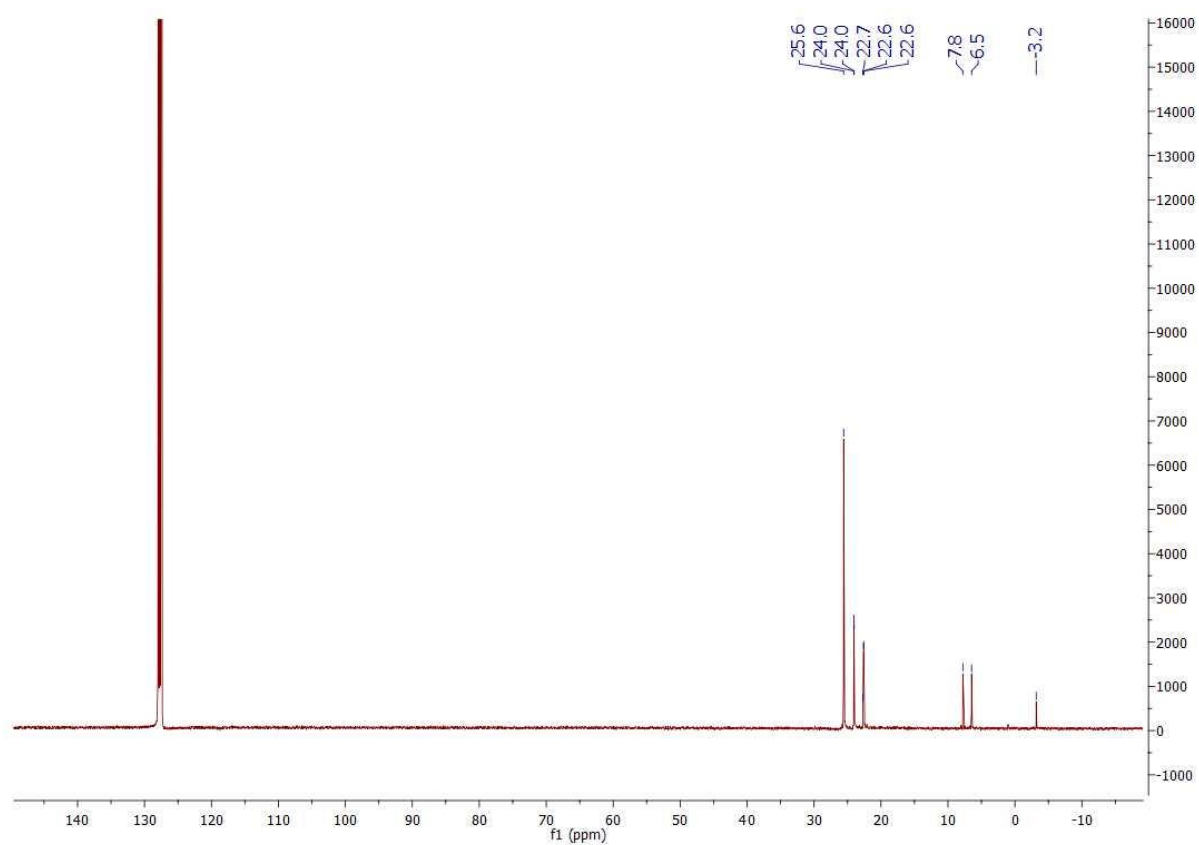
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.21 (s, 3H), 0.62-0.74 (m, 4H), 0.81-0.86 (m, 14H), 1.05-1.10 (m, 48H), 2.04-2.14 (m, 7H).

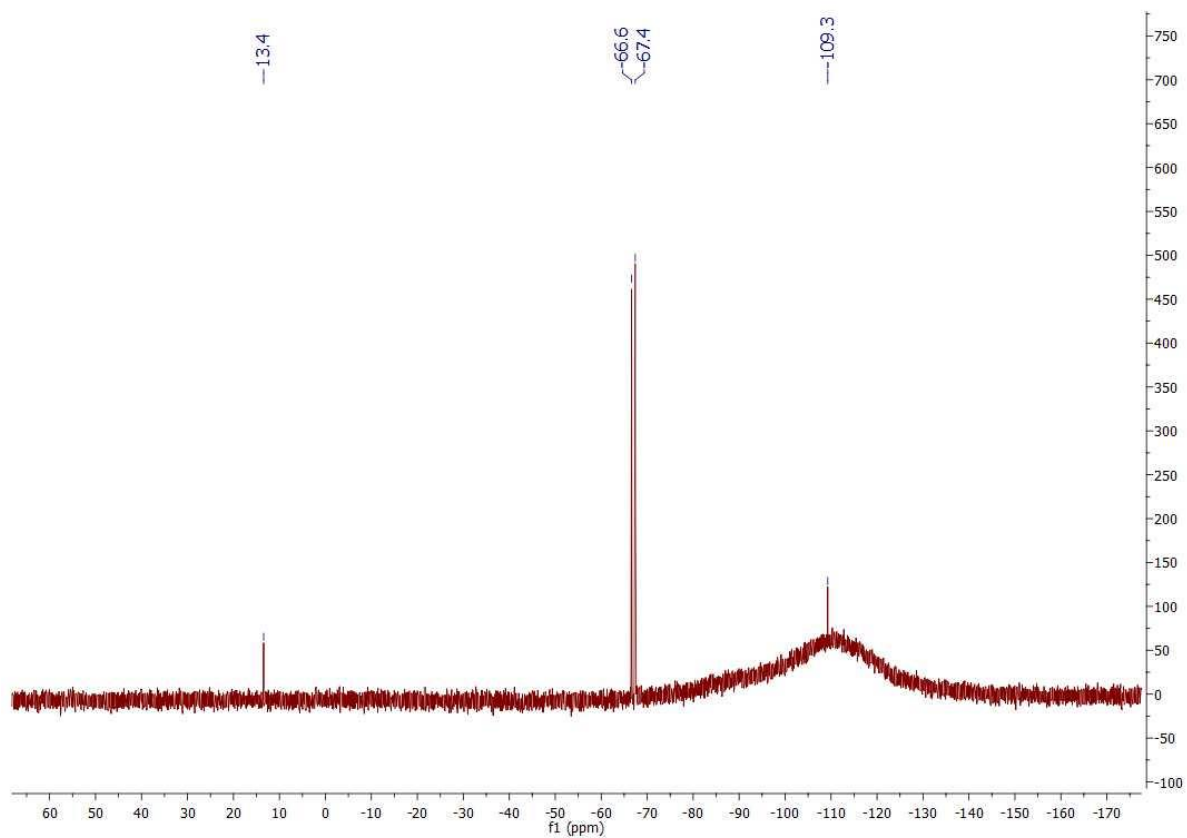
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -3.2, 6.5, 7.8, 22.6, 22.6, 22.7, 24.0, 24.0, 25.6.

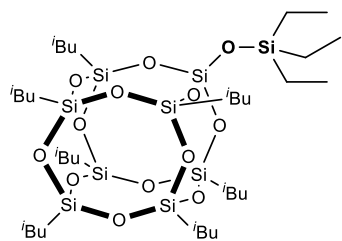
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 13.4, -66.6, -67.4, -109.3.

EA: C₃₃H₇₆O₁₃Si₉ (932.321): calcd. C 42.45, H 8.20; found C 42.52, H 8.28.









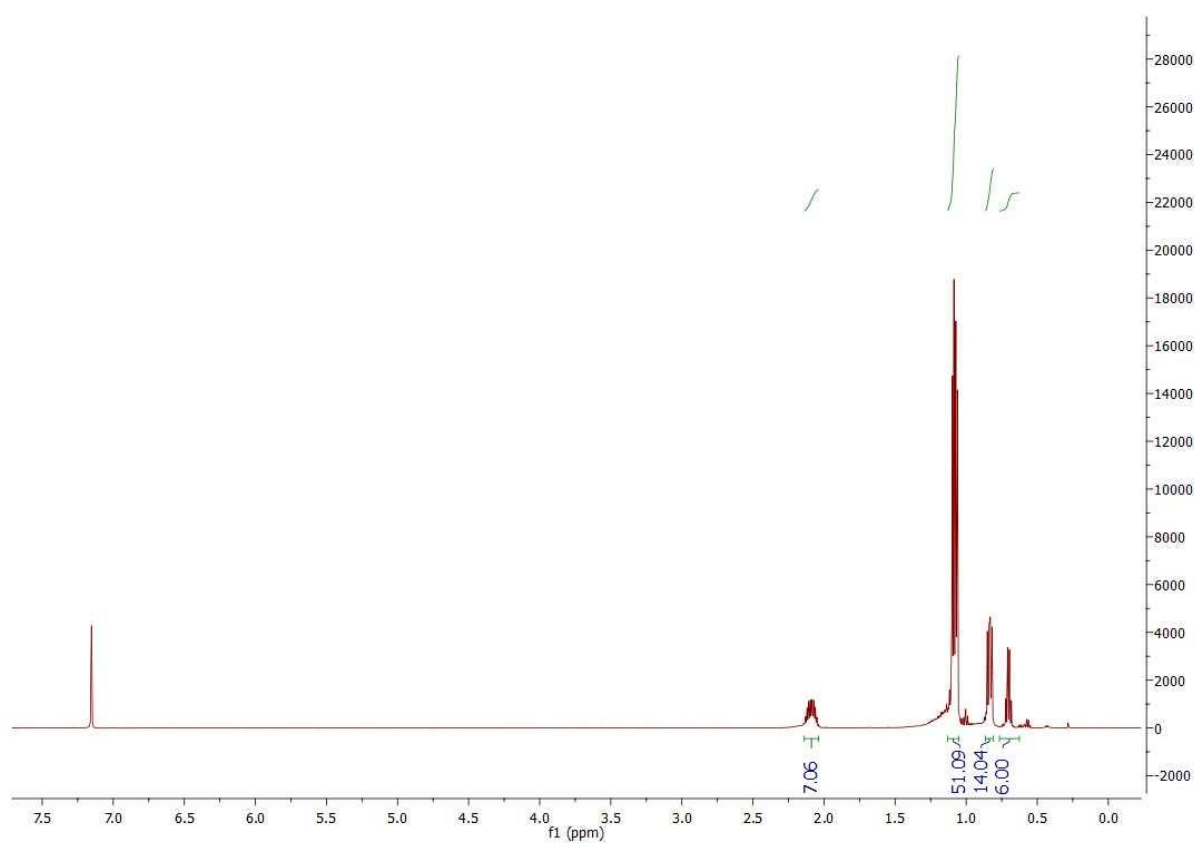
Compound (**3**) was obtained as solid in 91% yield.

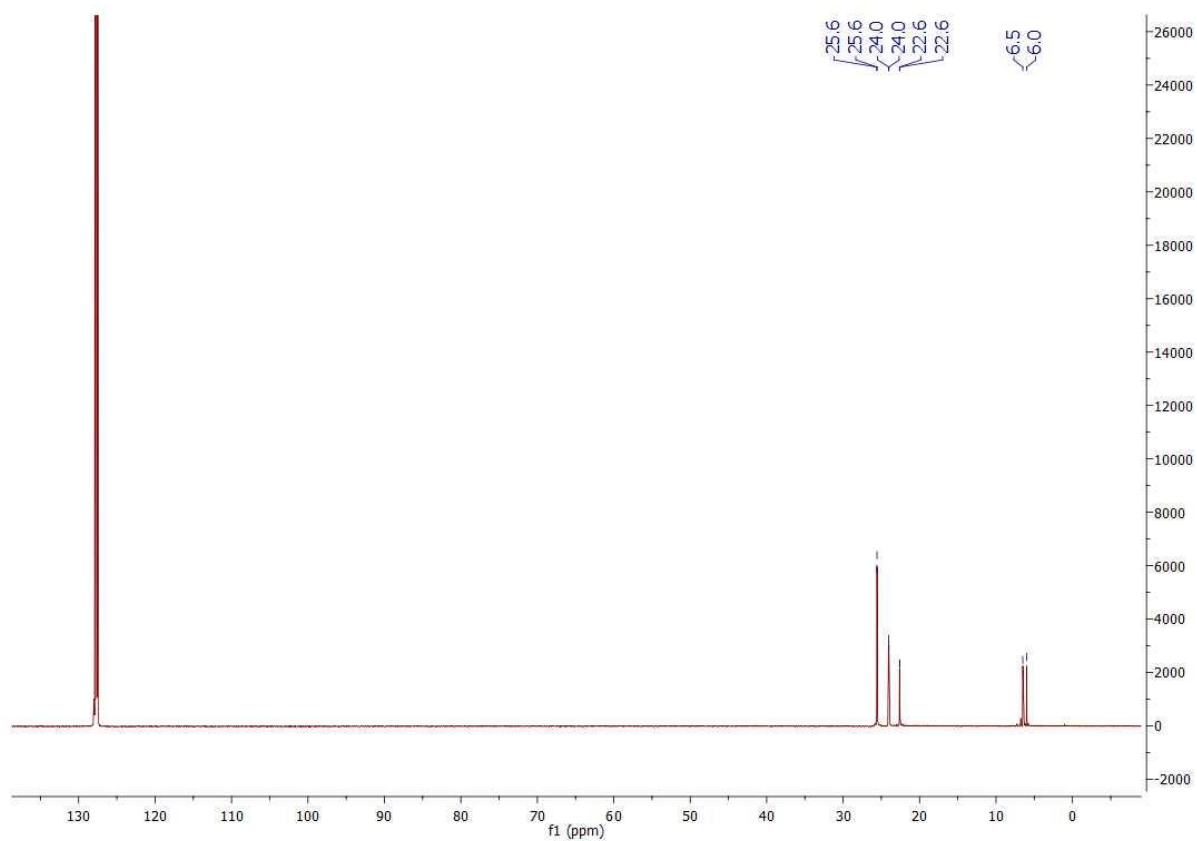
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.7 (q, J = 8.0 Hz, 6H), 0.81-0.86 (m, 14H), 1.05-1.13 (m, 51H), 2.04-2.14 (m, 7H).

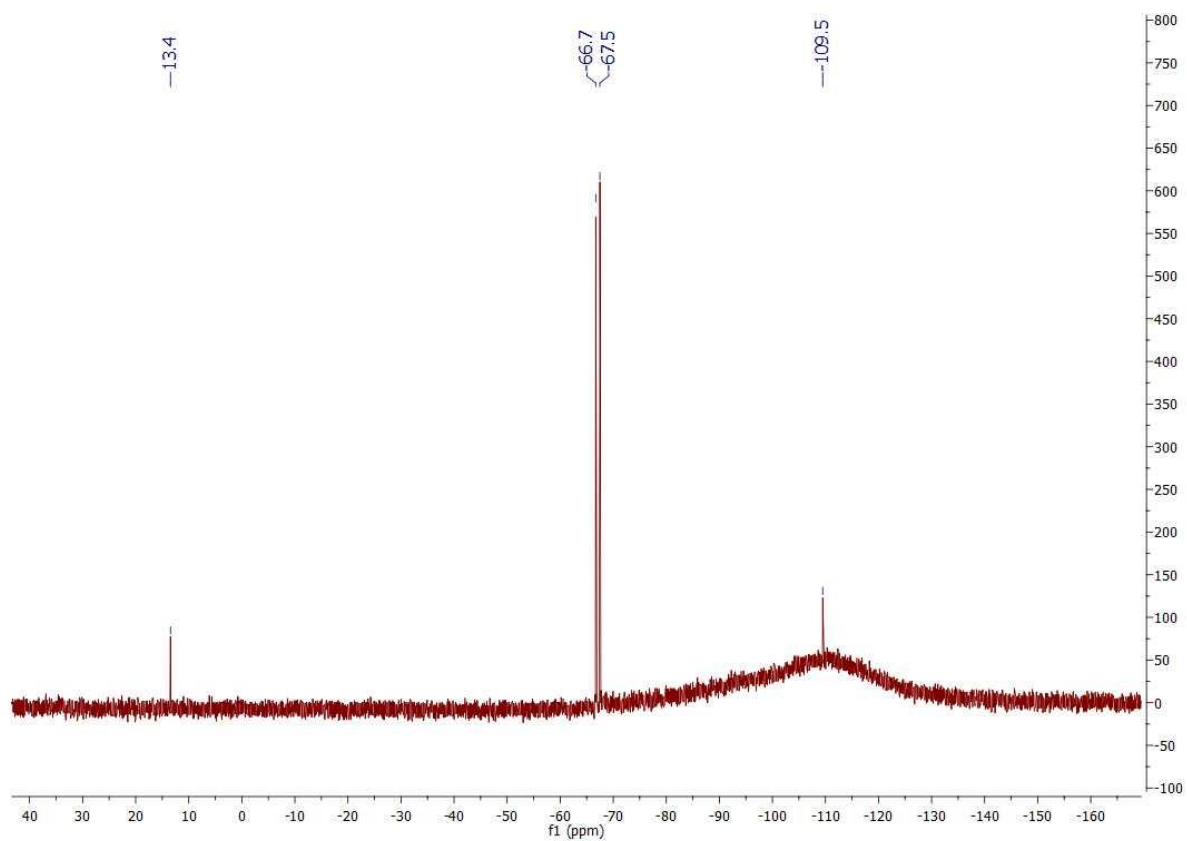
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 6.0, 6.5, 22.6, 22.6, 24.0, 24.0, 25.6, 25.6.

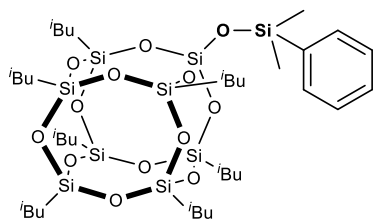
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 13.4, -66.7, -67.5, -109.5.

EA: C₃₄H₇₈O₁₃Si₉ (946.337): calcd. C 43.09, H 8.30; found C 42.92, H 8.18.









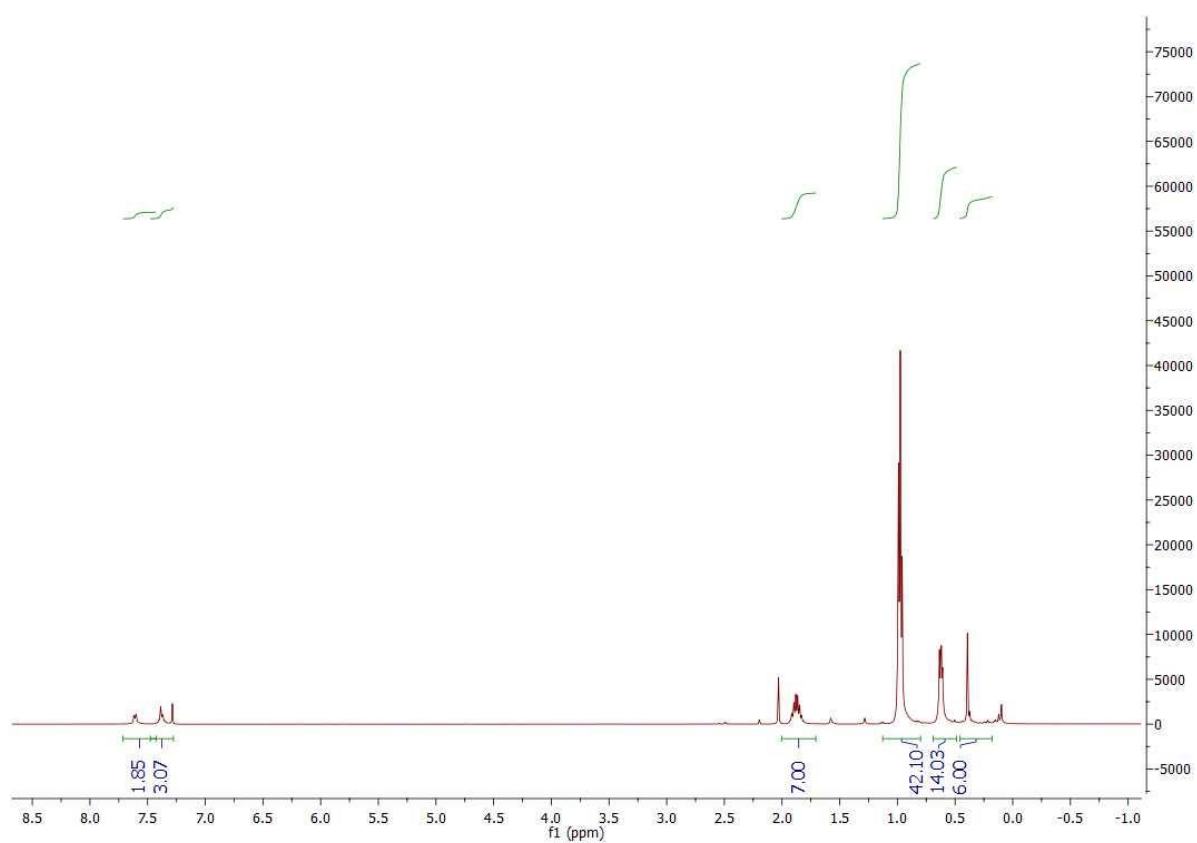
Compound (**4**) was obtained as solid in 96% yield.

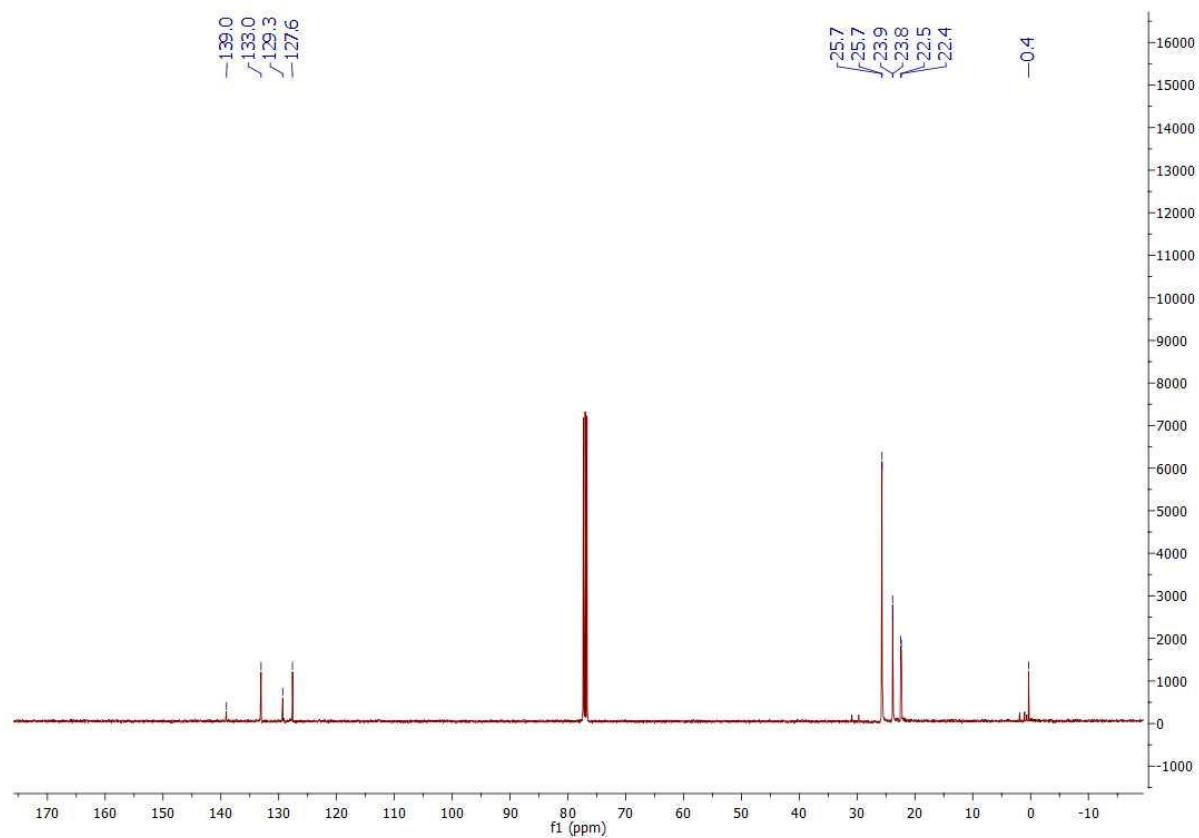
^1H NMR (400 MHz, C_6D_6) δ (ppm) = 0.39 (s, 6H), 0.59-0.65 (m, 14H), 0.94-1.01 (m, 42H), 1.82-1.92 (m, 7H), 7.30-7.45 (m, 3H), 7.55-7.65 (m, 2H).

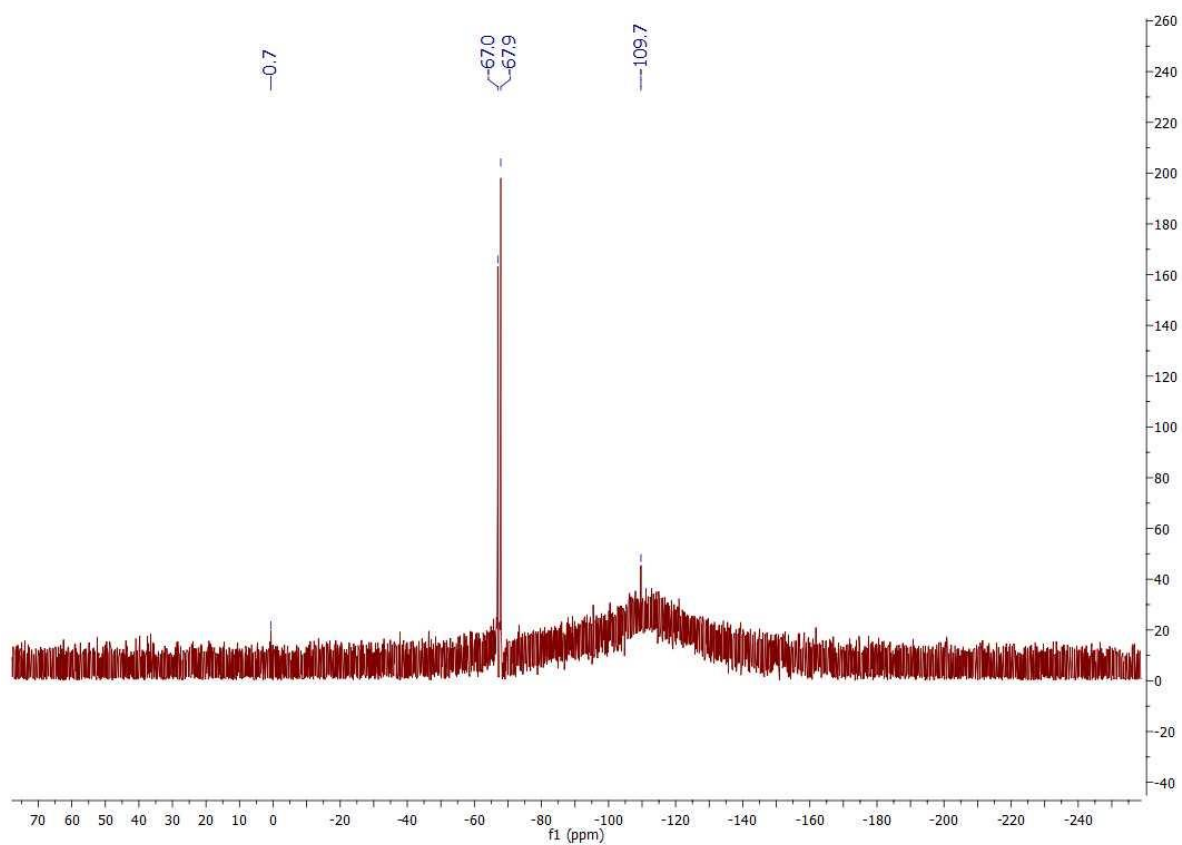
^{13}C NMR (101 MHz, C_6D_6) δ (ppm) = 0.4, 22.4, 22.5, 23.8, 23.9, 25.7, 25.7, 127.6, 129.3, 133.0, 139.0.

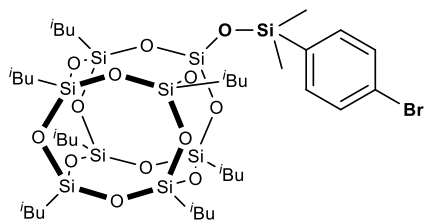
^{29}Si NMR (79 MHz, C_6D_6) δ (ppm) = 0.7, -67.0, -67.9, -109.7.

EA: $\text{C}_{36}\text{H}_{74}\text{O}_{13}\text{Si}_9$ (966.305): calcd. C 44.68, H 7.71; found C 44.83, H 7.83.









Compound (**5**) was obtained as solid in 89% yield.

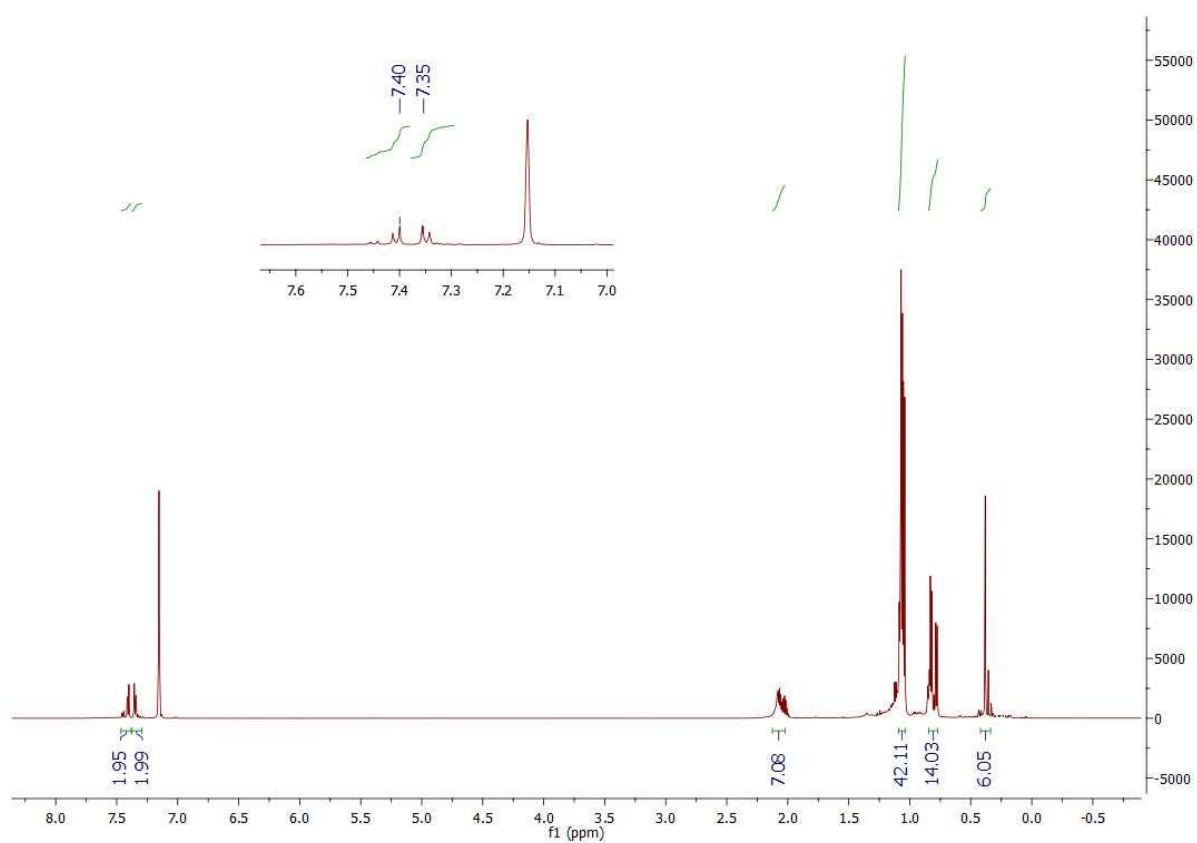
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.35-0.42 (m, 6H), 0.77-0.86 (m, 14H), 1.03-1.11 (m, 42H), 1.99-2.12 (m, 7H), 7.33-7.37 (m, 2H), 7.40-7.44 (m, 2H).

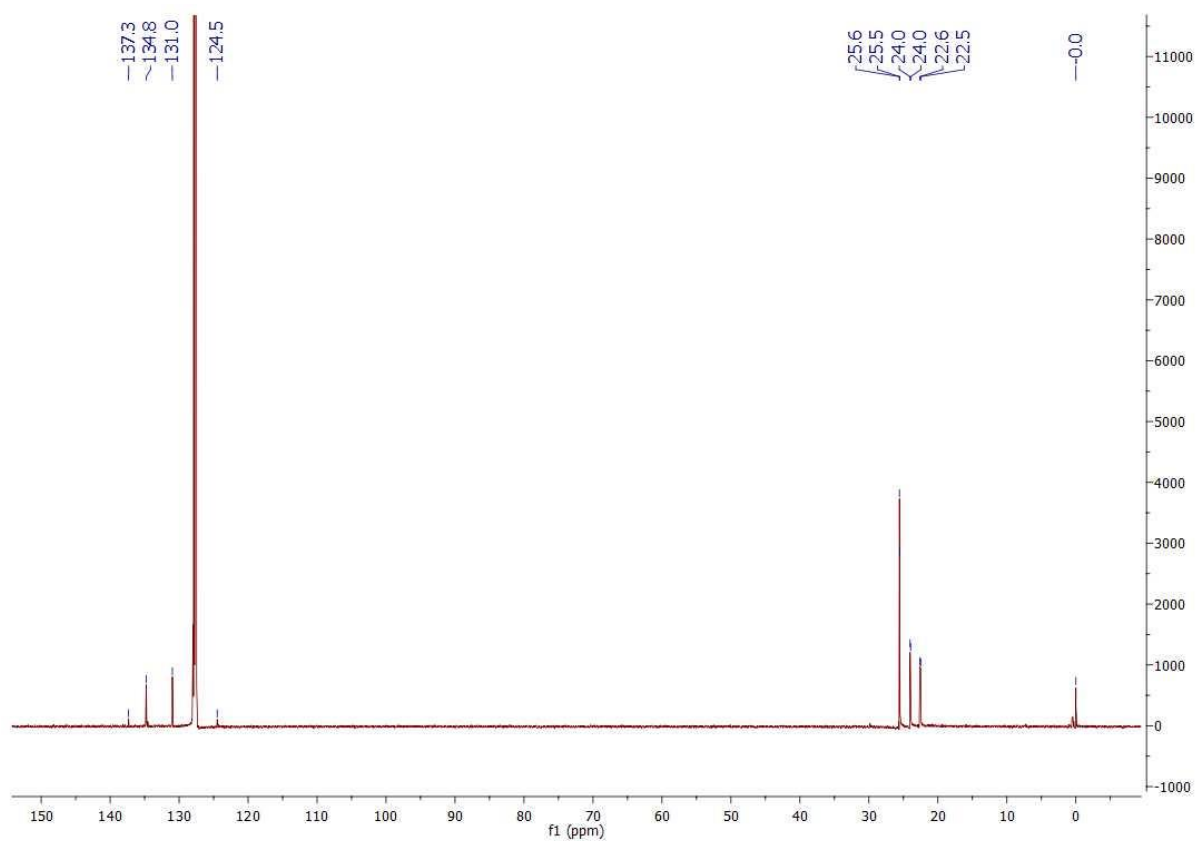
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 0.0, 22.5, 22.6, 24.0, 24.0, 25.5, 25.6, 124.5, 131.0, 134.8, 137.3.

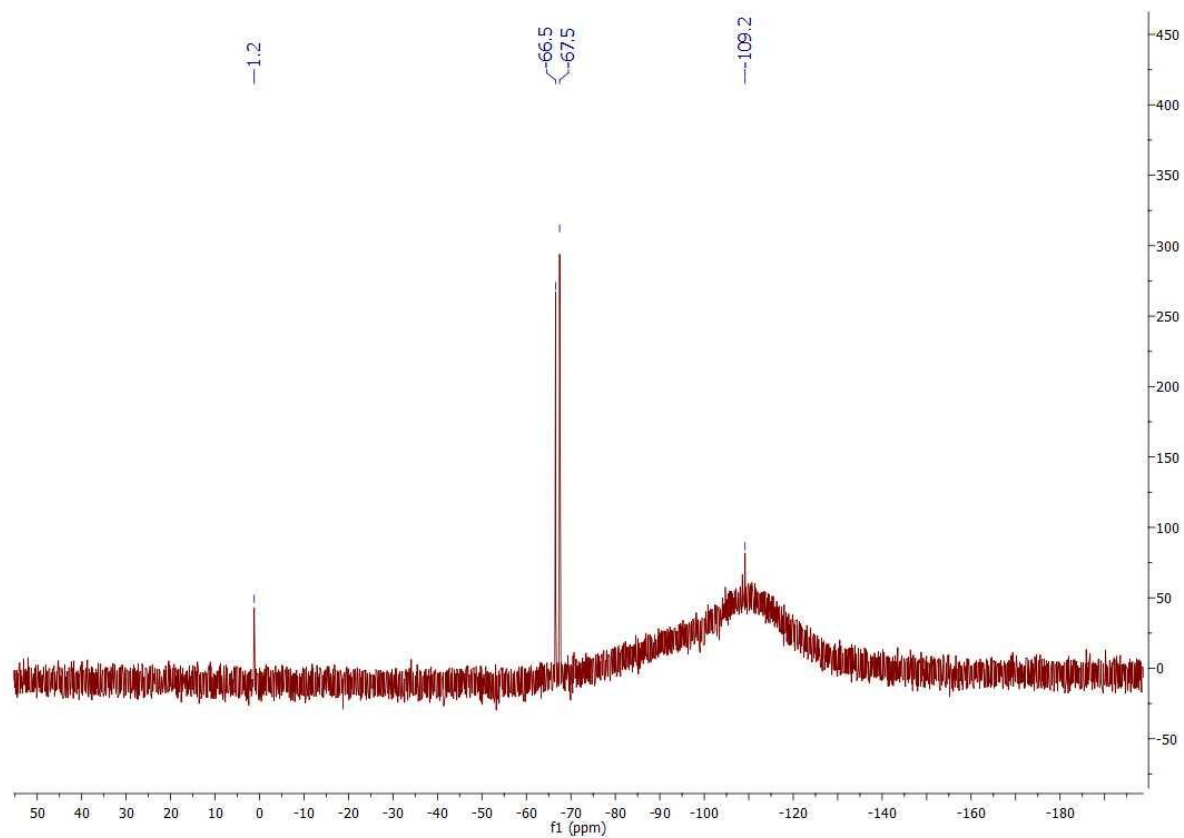
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 1.2, -66.5, -67.5, -109.2.

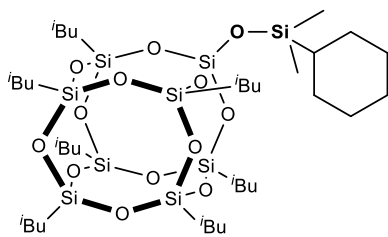
EA: C₃₆H₇₃BrO₁₃Si₉ (1044.216): calcd. C 41.31, H 7.03; found C 41.45, H 7.16.

HRMS: calcd. for C₃₆H₇₃BrO₁₃Si₉Na 1067.21; found 1067.21.









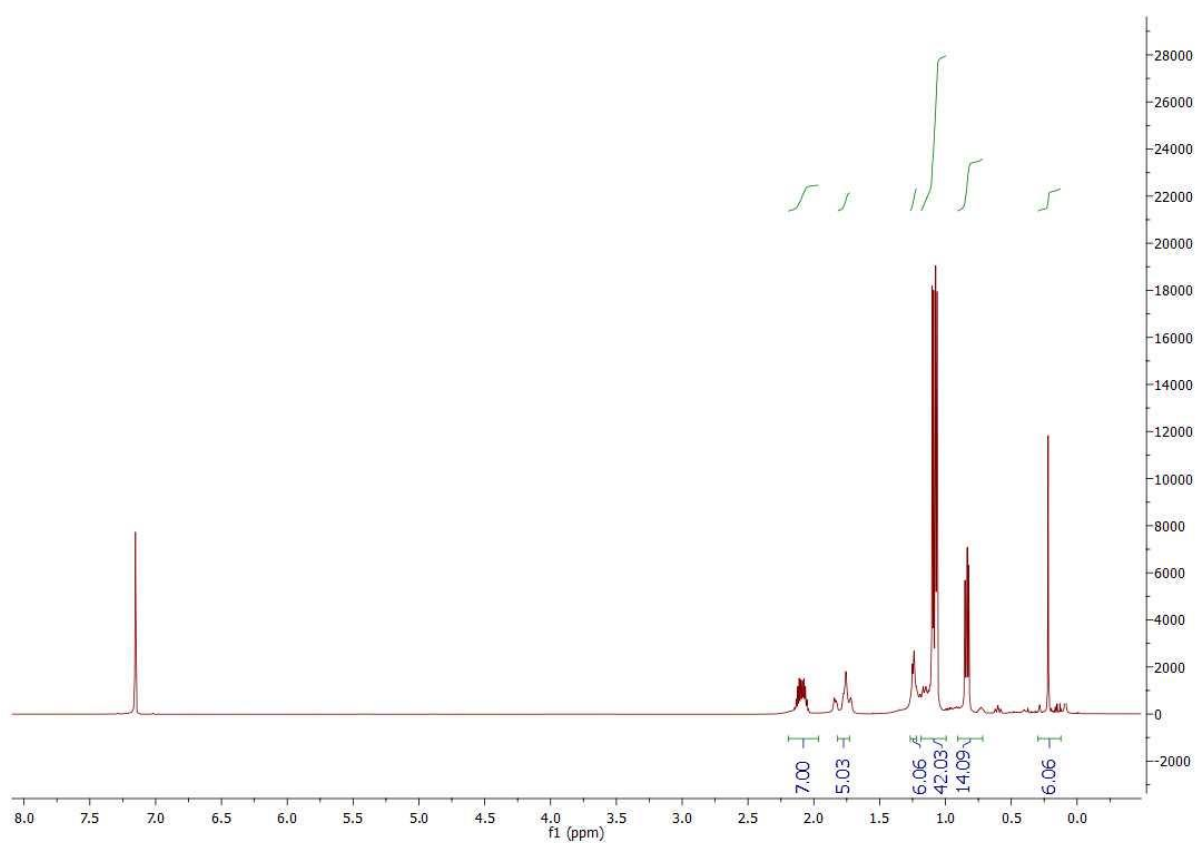
Compound (**6**) was obtained as oil in 85% yield.

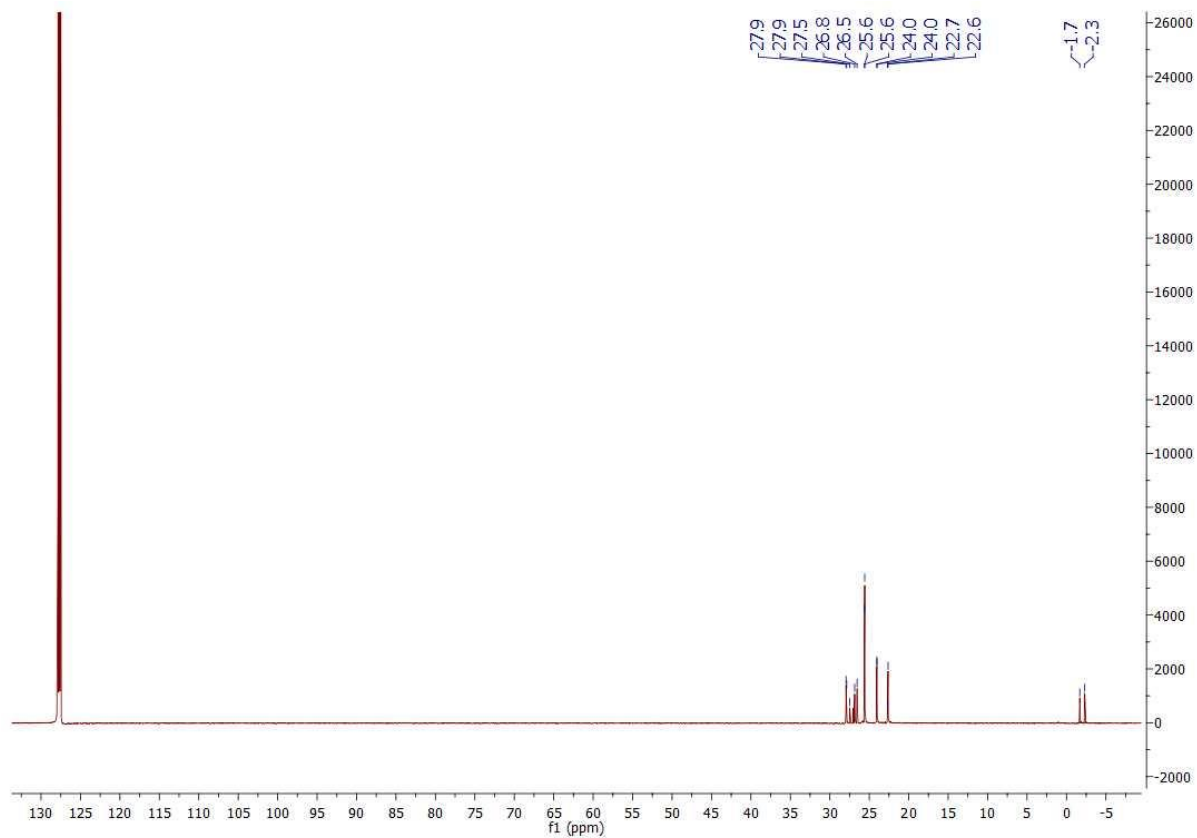
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.22 (s, 6H), 0.81-0.87 (m, 14H), 0.99-1.18 (m, 42H), 1.23-1.27 (m, 6H), 1.72-1.78 (m, 5H), 2.04-2.14 (m, 7H).

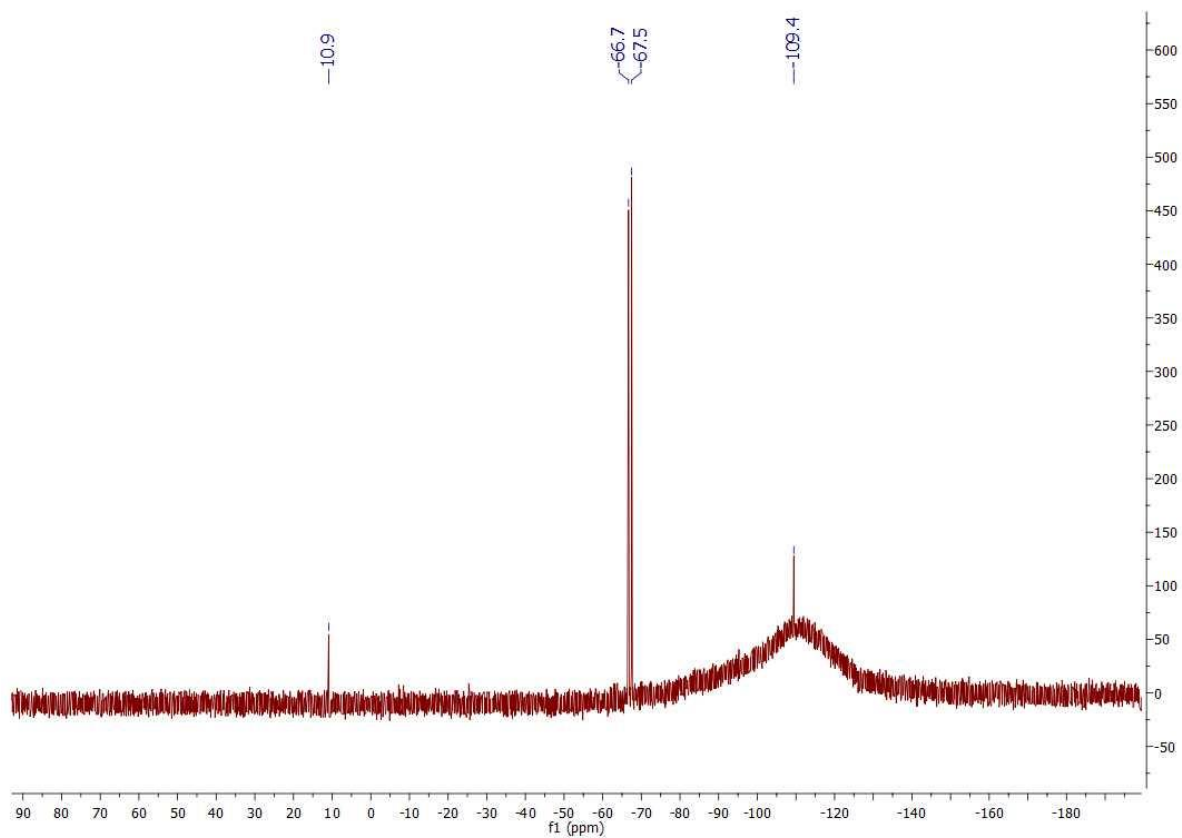
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -2.3, -1.7, 22.6, 22.7, 24.0, 24.0, 25.6, 25.6, 26.5, 26.8, 27.5, 27.9, 27.9.

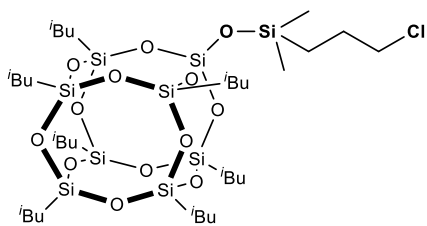
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 10.9, -66.7, -67.5, -109.4.

EA: C₃₆H₈₀O₁₃Si₉ (972.352): calcd. C 44.40, H 8.28; found C 44.53, H 8.15.









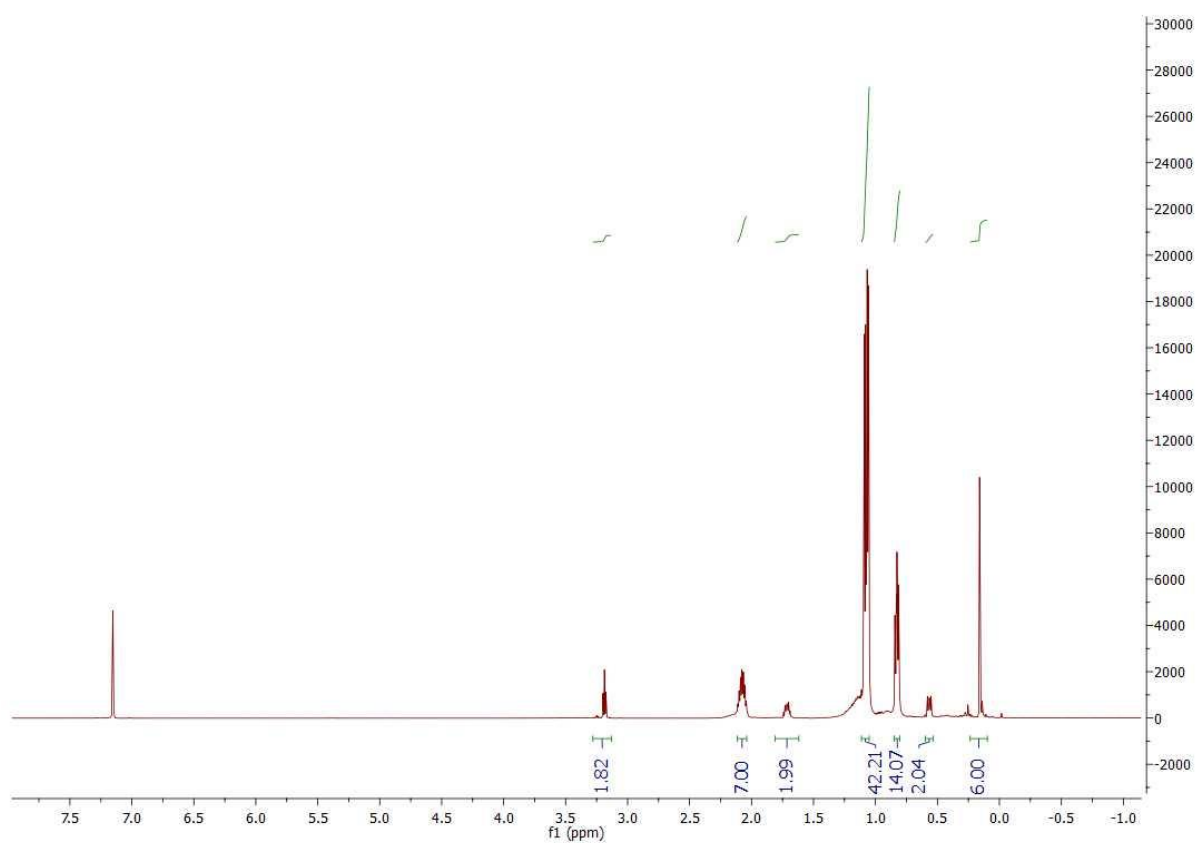
Compound (**7**) was obtained as oil in 87% yield.

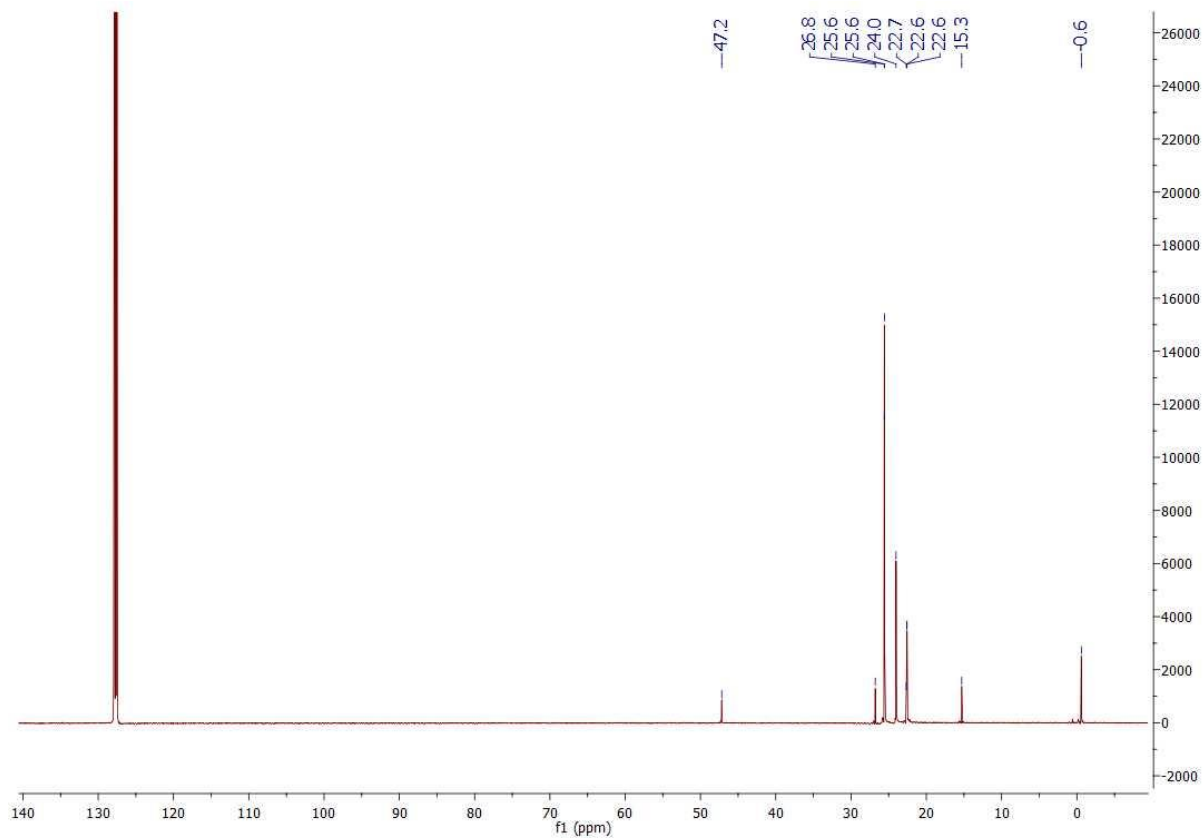
^1H NMR (400 MHz, C_6D_6) δ (ppm) = 0.16 (s, 6H), 0.53-0.60 (m, 2H), 0.79-0.85 (m, 14H), 1.03-1.11 (m, 42H), 1.67-1.74 (m, 2H), 2.02-2.12 (m, 7H), 3.19 (t, $J = 7\text{Hz}$, 2H).

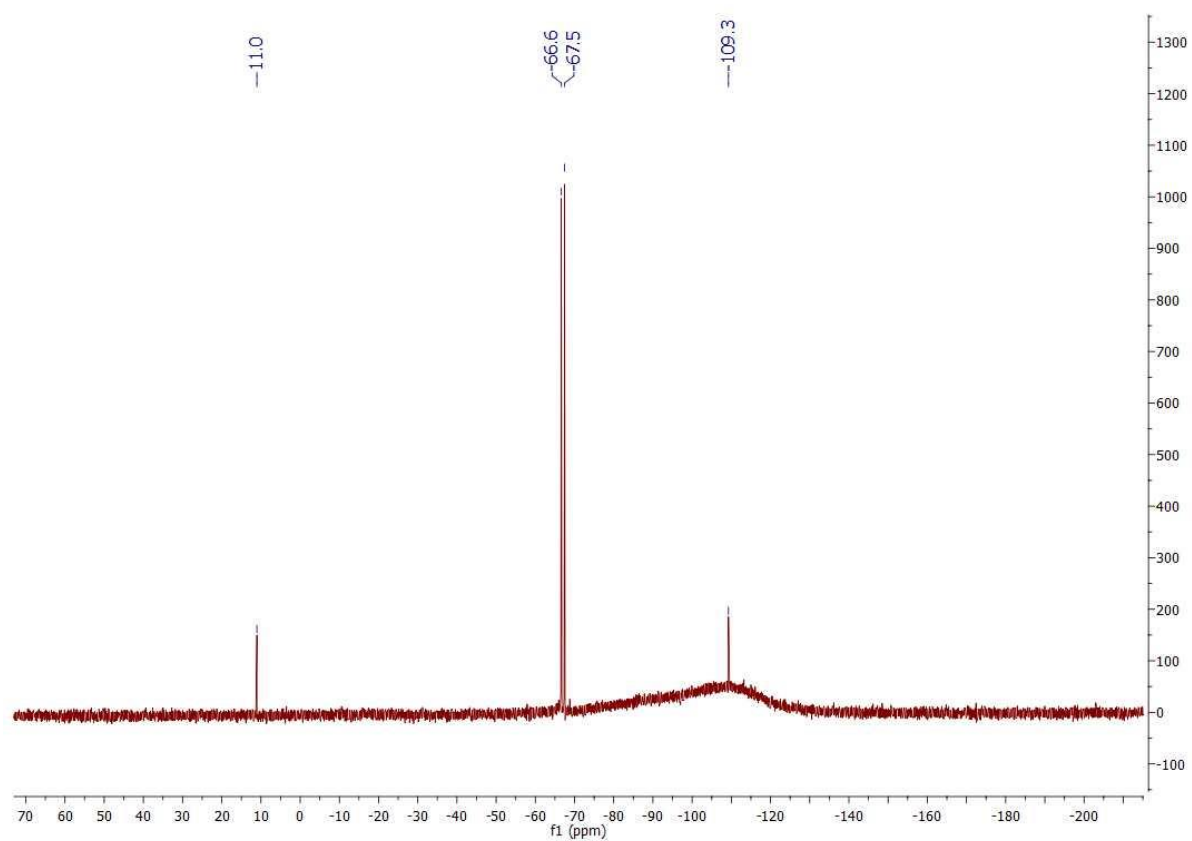
^{13}C NMR (101 MHz, C_6D_6) δ (ppm) = -0.6, 15.3, 22.6, 22.6, 22.7, 24.0, 25.6, 25.6, 26.8, 47.2.

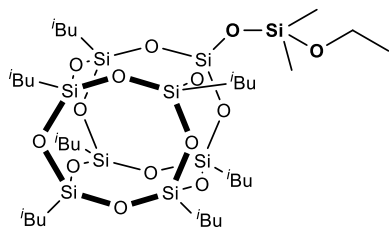
^{29}Si NMR (79 MHz, C_6D_6) δ (ppm) = 11.0, -66.6, -67.5, -109.3.

EA: $\text{C}_{36}\text{H}_{80}\text{ClO}_{13}\text{Si}_9$ (966.282): calcd. C 40.94, H 7.81; found C 40.83, H 7.89.









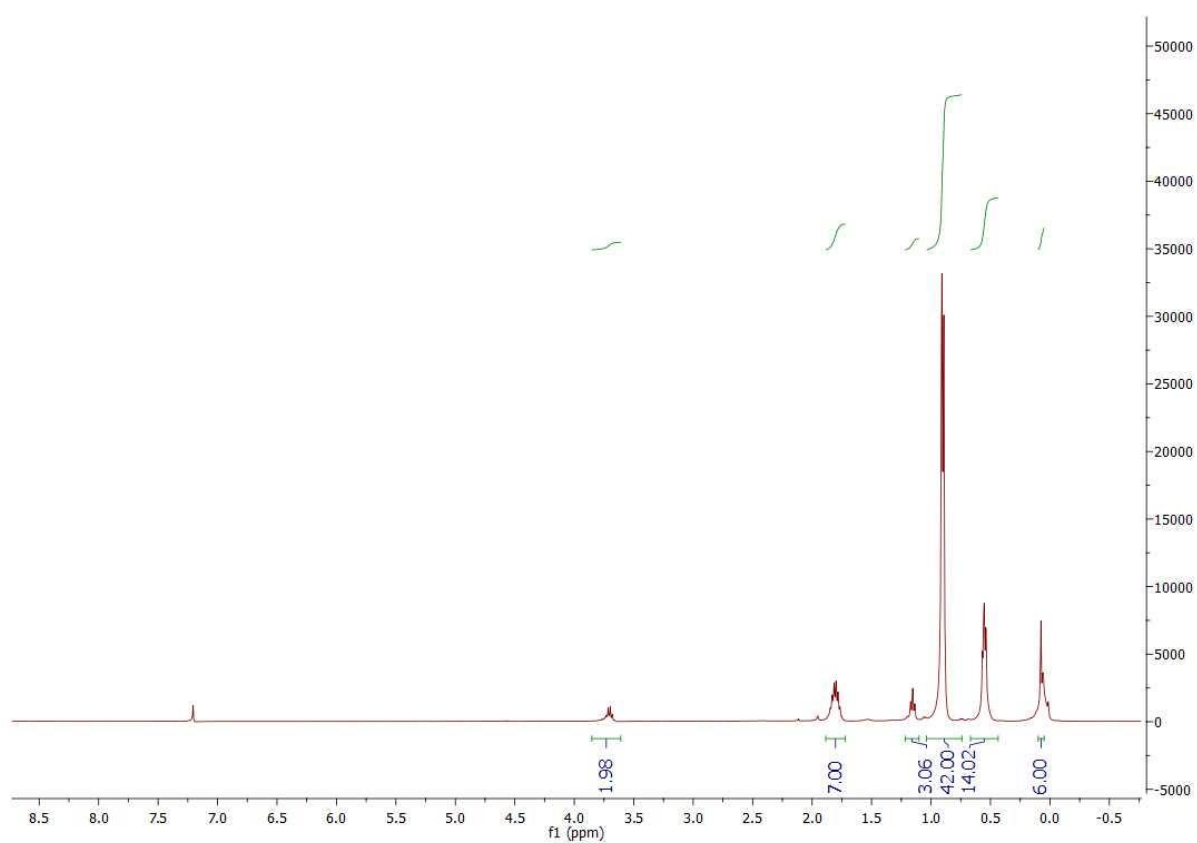
Compound (**8**) was obtained as solid in 89% yield.

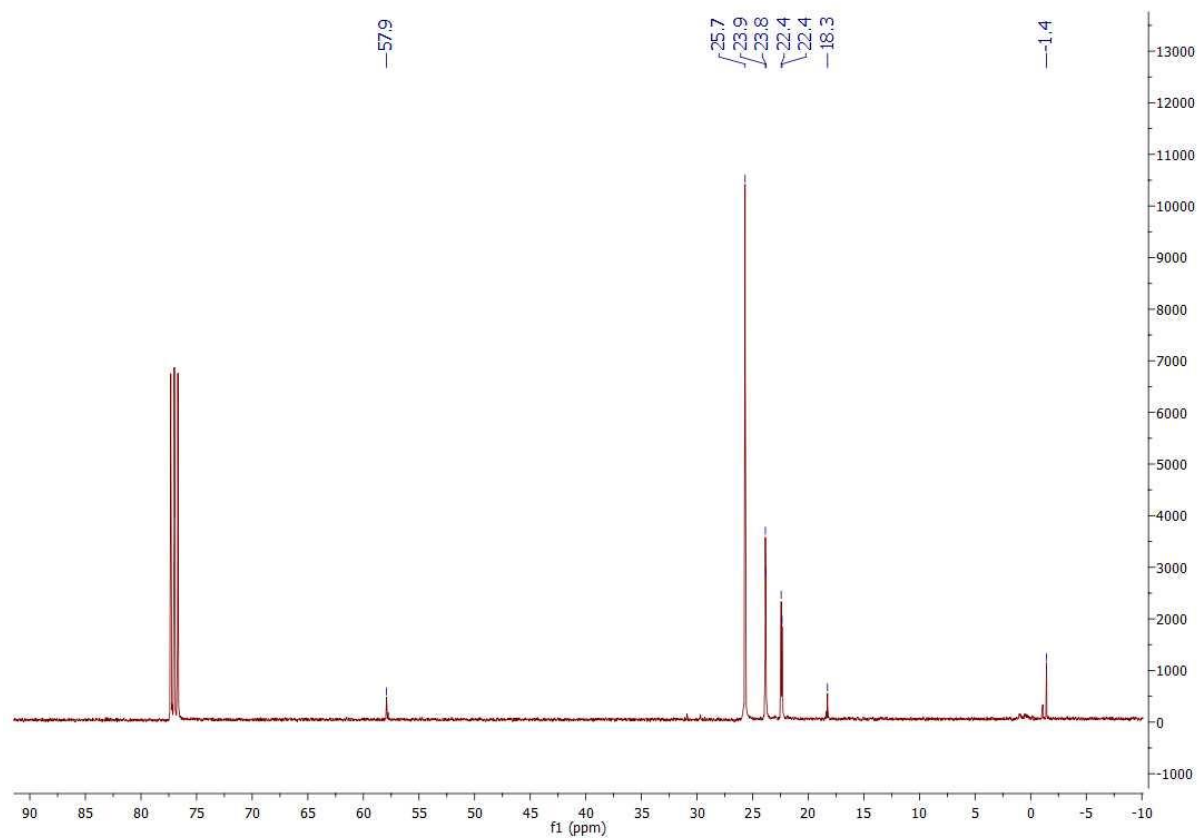
¹H NMR (400 MHz, CDCl₃) δ (ppm) = 0.04-0.10 (m, 6H), 0.48-0.58 (m, 14H), 0.84-0.98 (m, 42H), 1.15 (t, J = 7.0 Hz, 3H), 1.76-1.85 (m, 7H), 3.65-3.74 (m, 2H).

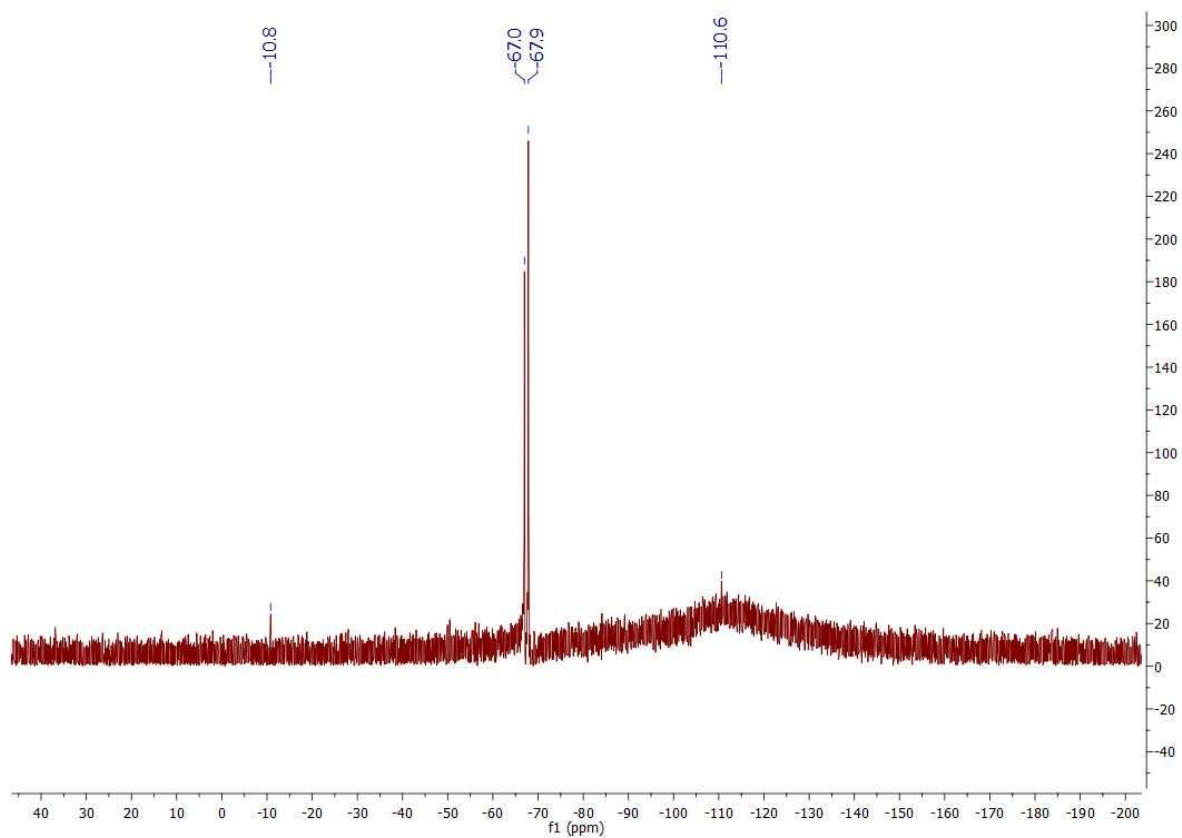
¹³C NMR (101 MHz, CDCl₃) δ (ppm) = -1.4, 18.3, 22.4, 22.4, 23.8, 23.9, 25.7, 57.9.

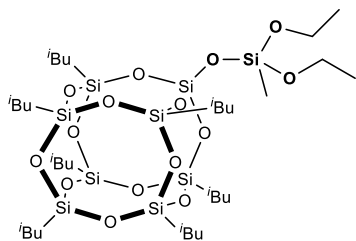
²⁹Si NMR (79 MHz, CDCl₃) δ (ppm) = -10.8, -67.0, -67.9, -110.6.

EA: C₃₂H₇₄O₁₄Si₉ (934.300): calcd. C 41.08, H 7.97; found C 41.23, H 8.11.









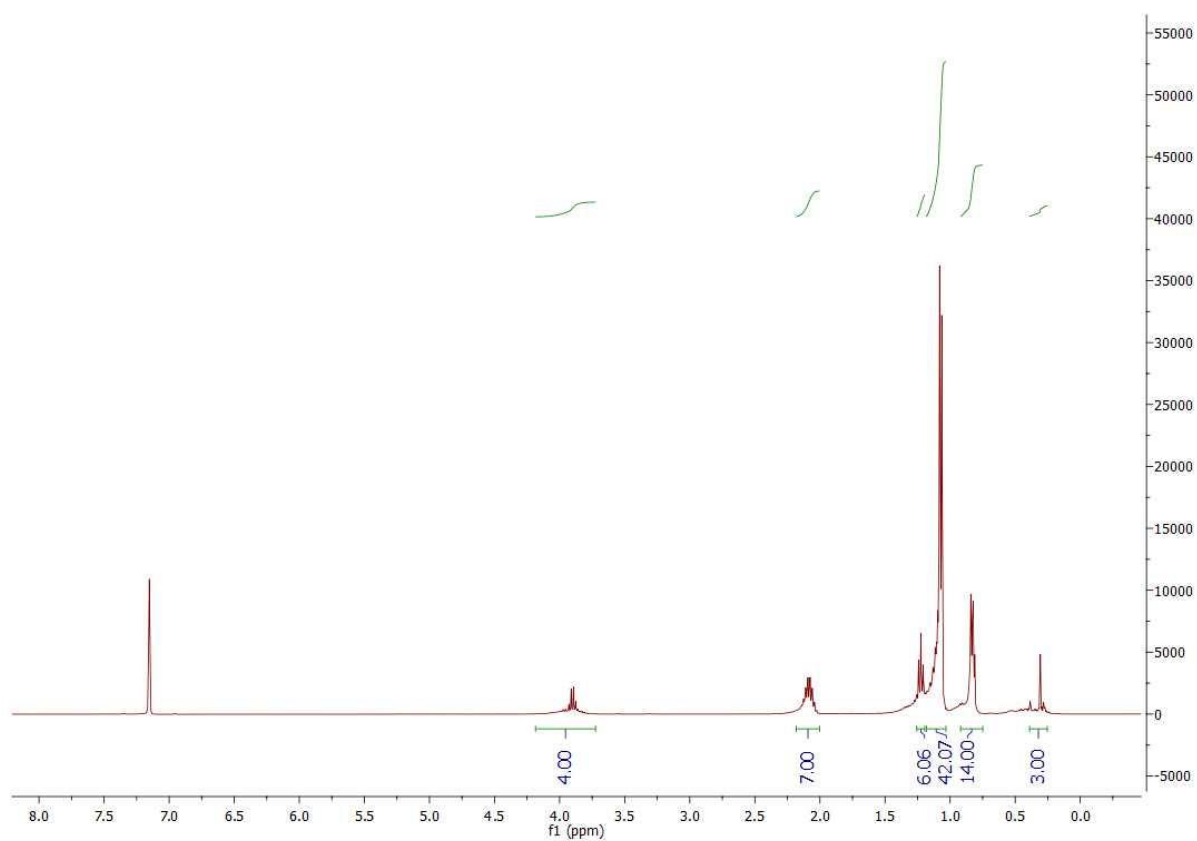
Compound (**9**) was obtained as oil in 93% yield.

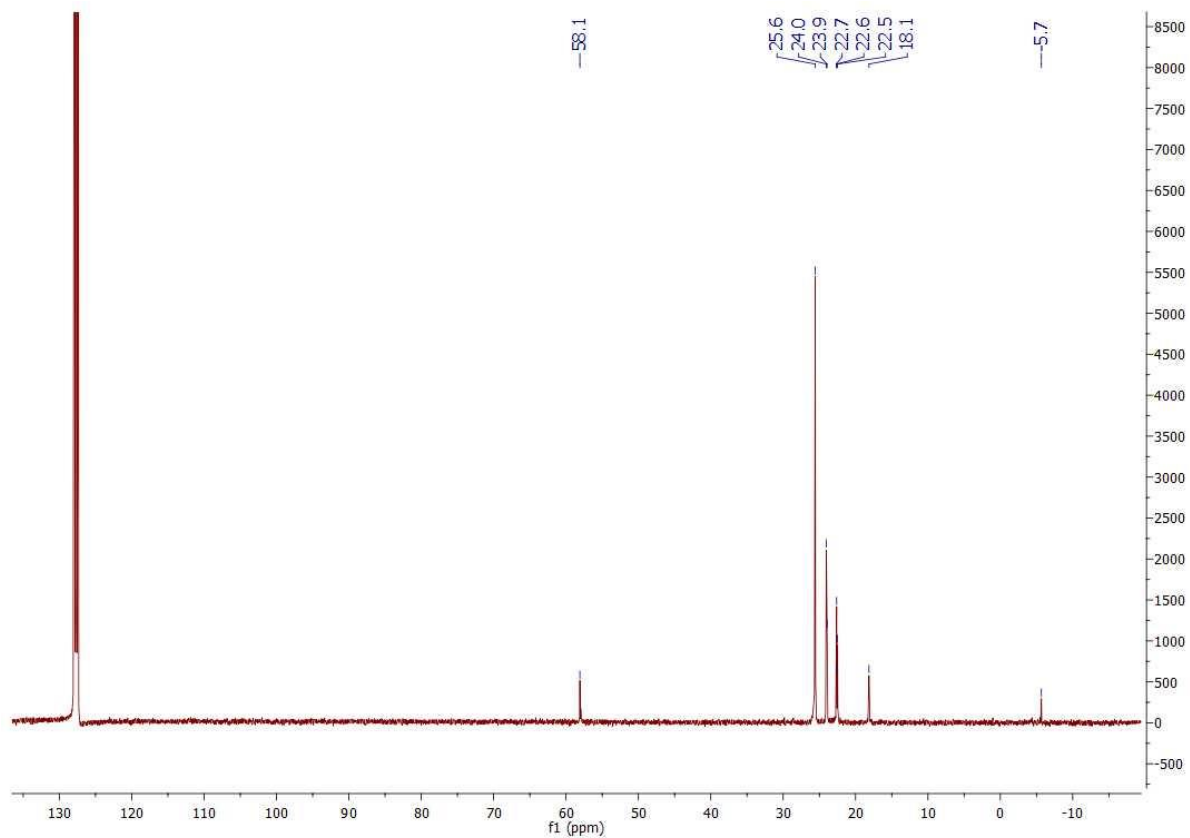
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.31 (s, 3H), 0.80-0.86 (m, 14H), 1.05-1.15 (m, 42H), 1.22 (t, *J* = 7.0 Hz, 6H), 2.02-2.14 (m, 7H), 3.86-3.94 (m, 4H).

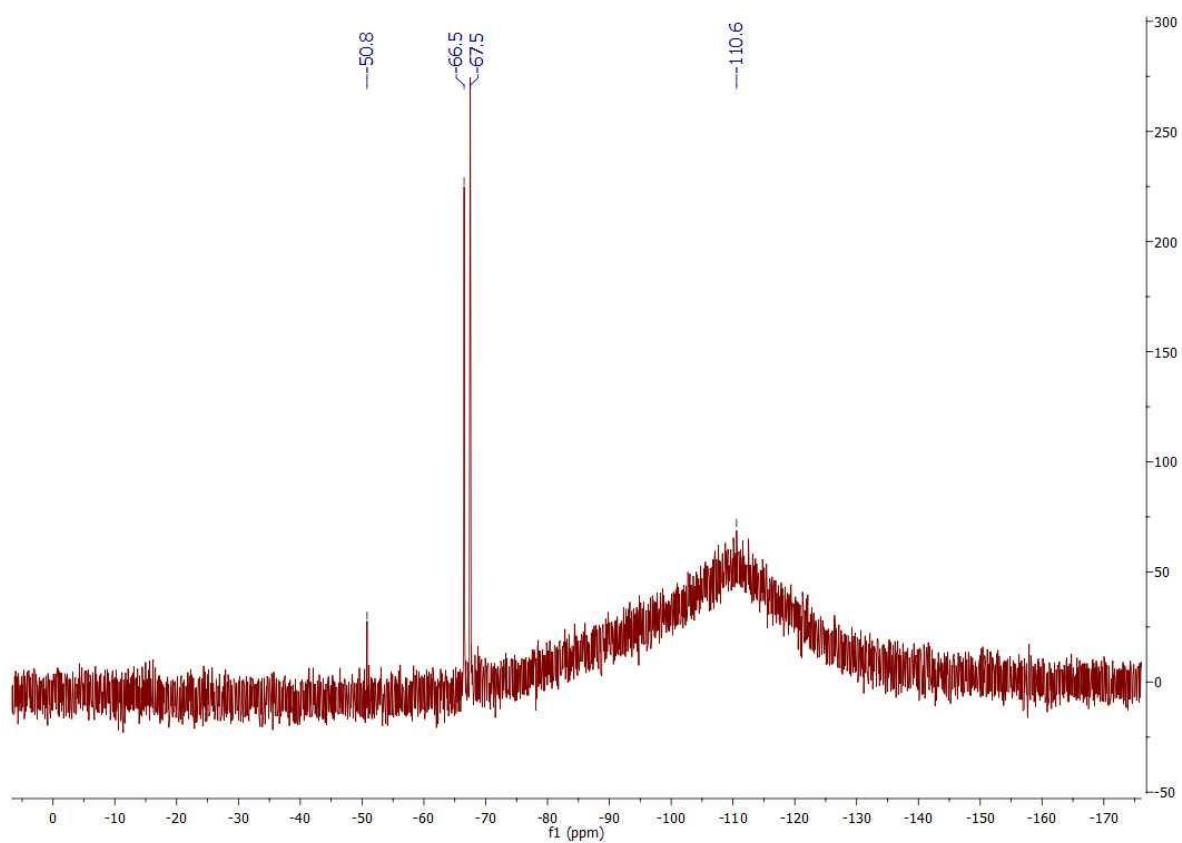
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -5.7, 18.1, 22.5, 22.6, 22.7, 23.9, 24.0, 25.6, 58.1.

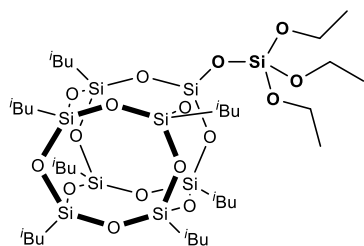
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -50.8, -66.5, -67.5, -110.6.

EA: C₃₂H₇₆O₁₅Si₉ (964.311): calcd. C 41.04, H 7.93; found C 41.18, H 8.12.









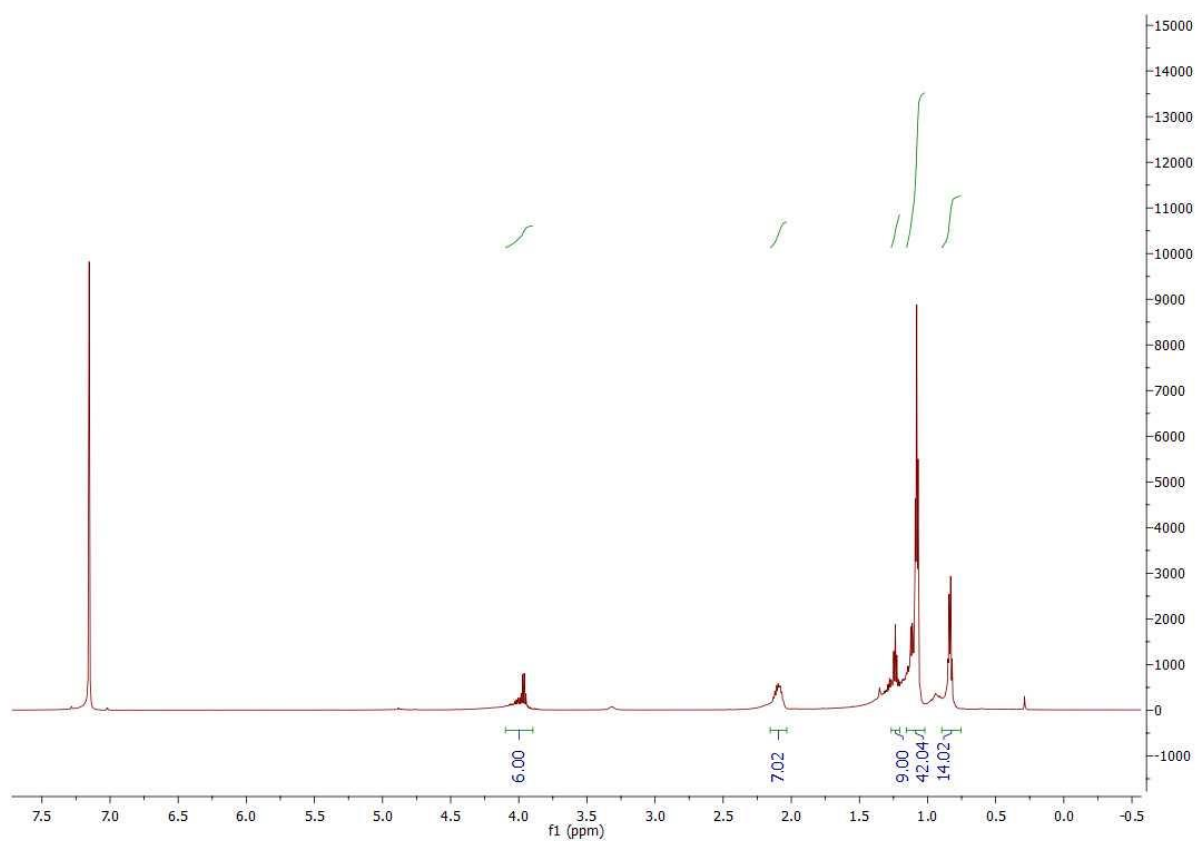
Compound (**10**) was obtained as oil in 91% yield.

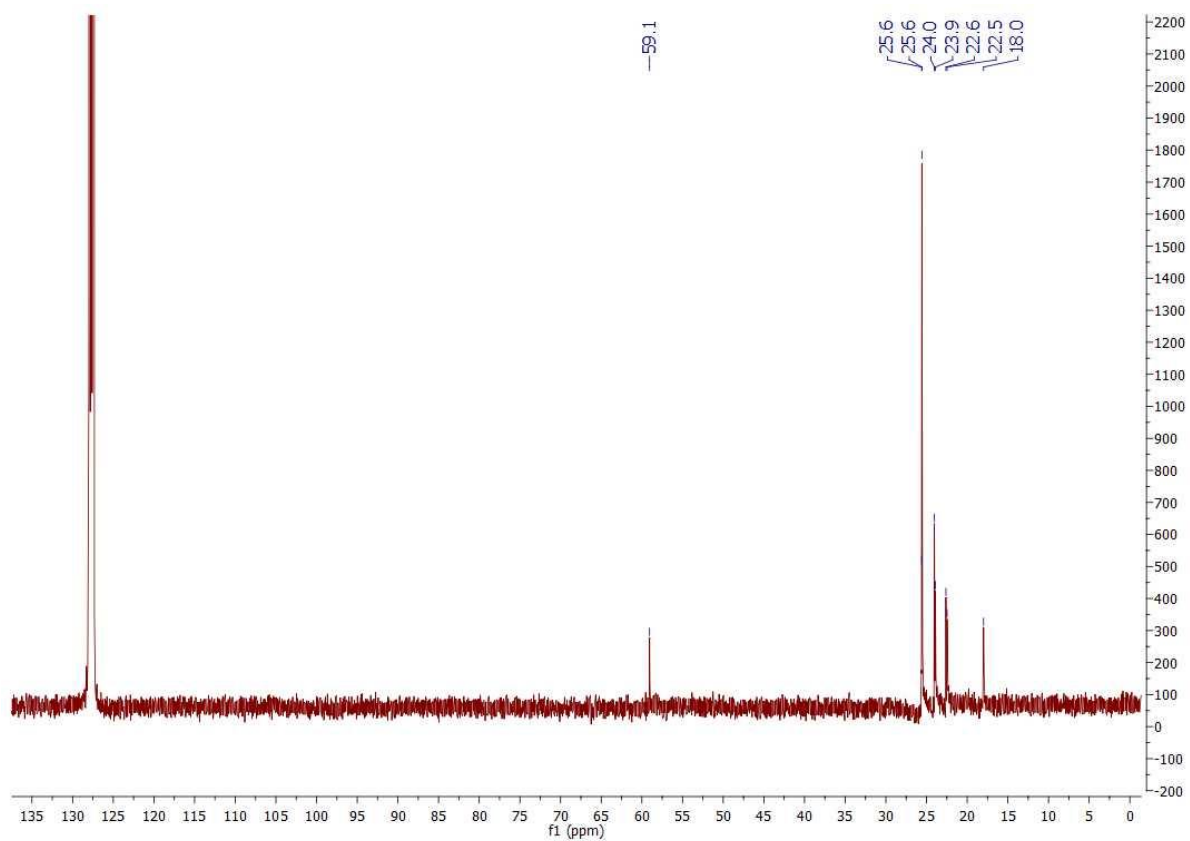
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.80-0.86 (m, 14H), 1.05-1.14 (m, 42H), 1.24 (t, J = 7.0 Hz, 9H), 2.03-2.13 (m, 7H), 3.90-4.00 (m, 6H).

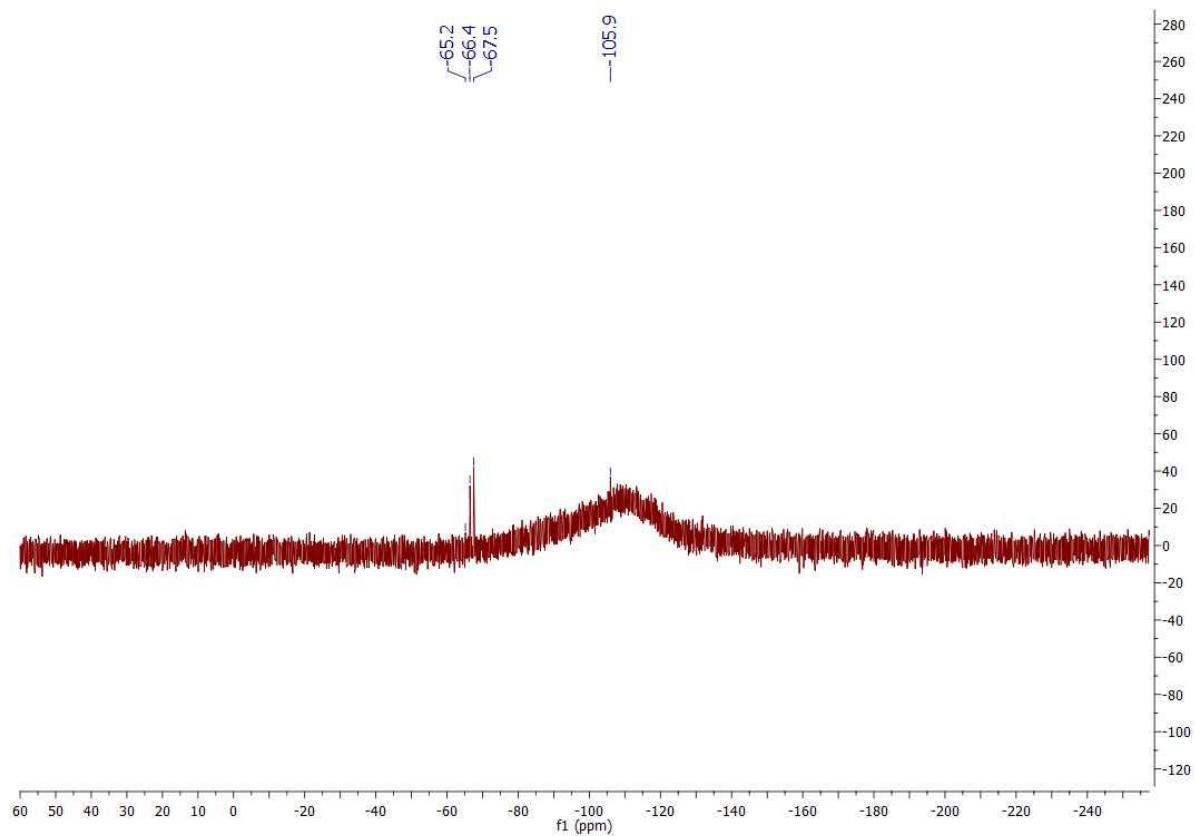
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 18.0, 22.5, 22.6, 23.9, 24.0, 25.6, 25.6, 59.1.

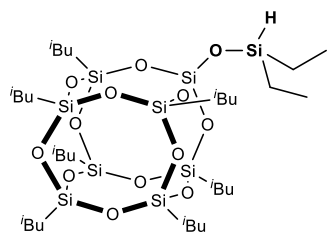
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -65.2, -66.4, -67.5, -105.9.

EA: C₃₄H₇₈O₁₆Si₉ (994.321): calcd. C 41.01, H 7.90; found C 41.23, H 8.05.









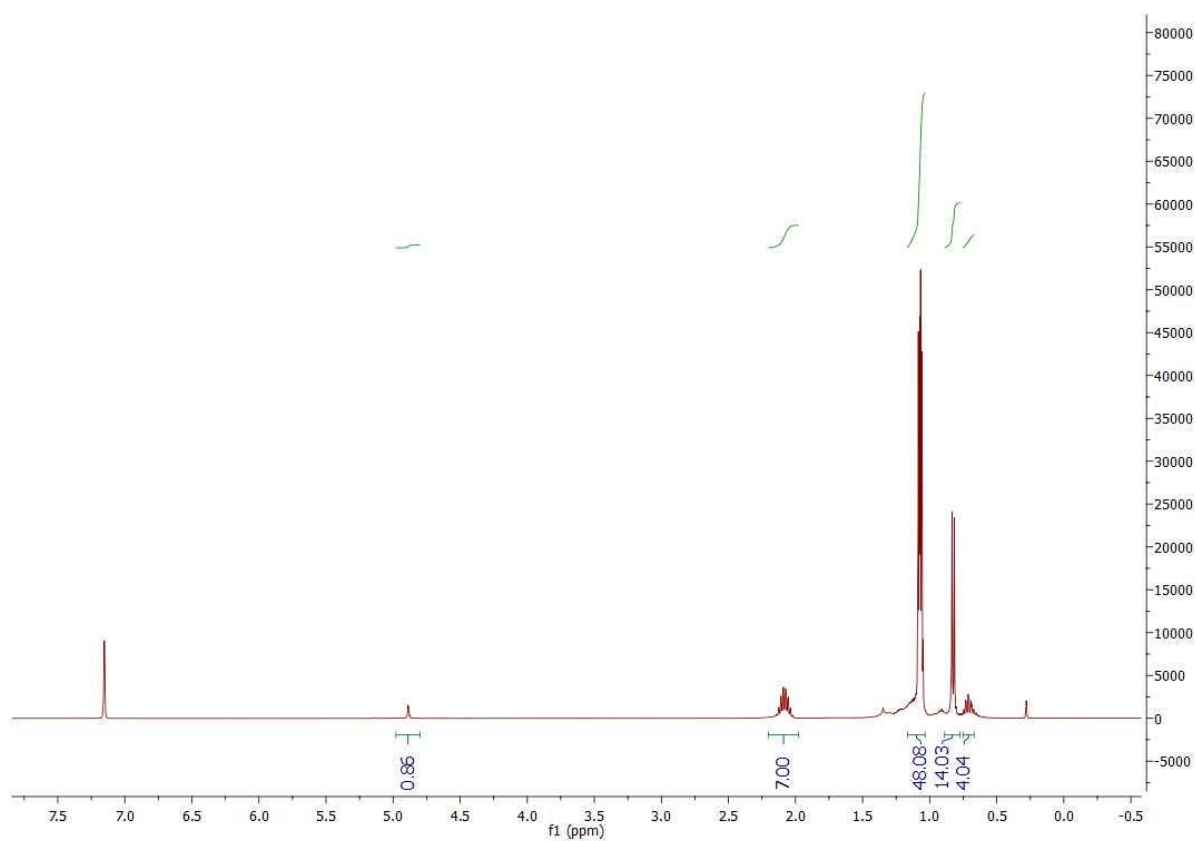
Compound (**11**) was obtained as solid in 97% yield.

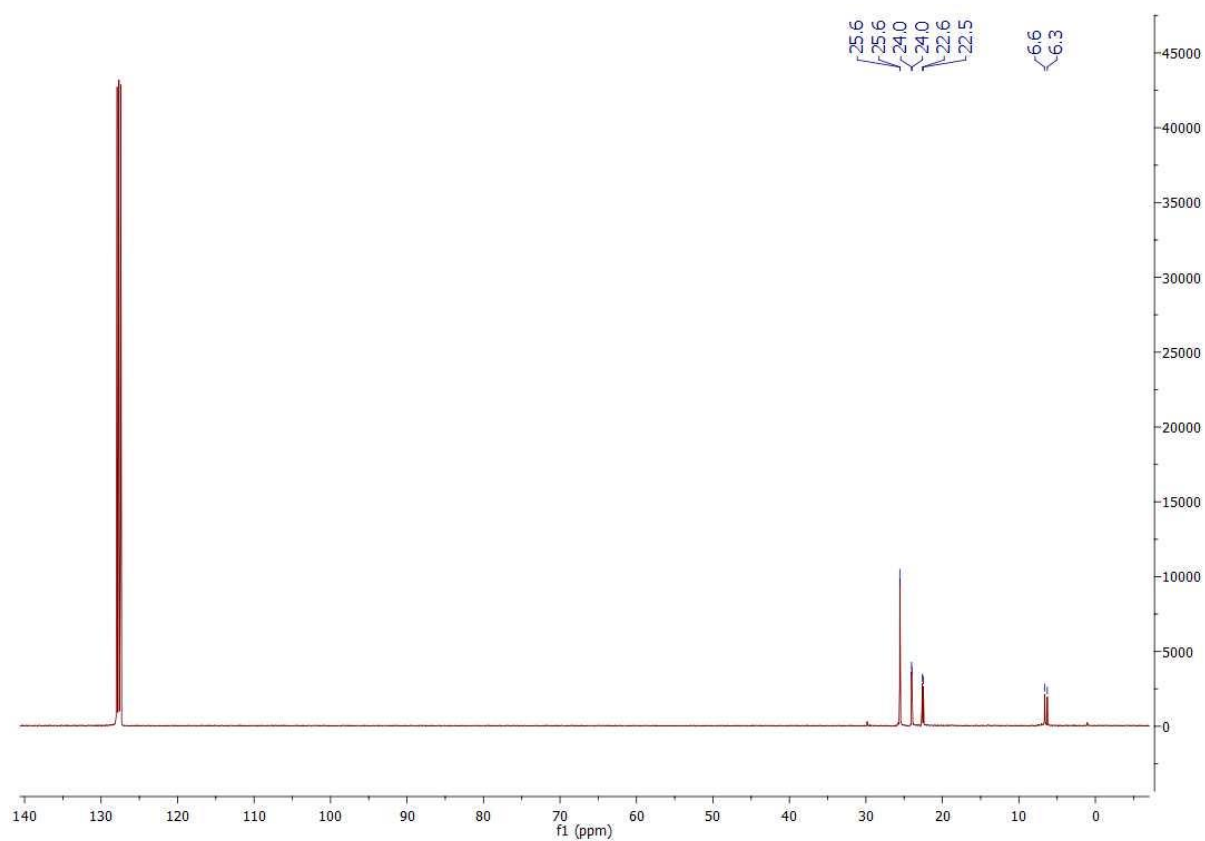
^1H NMR (400 MHz, C_6D_6) δ (ppm) = 0.66-0.74 (m, 4H), 0.77-0.88 (m, 14H), 1.03-1.14 (m, 48H), 2.01-2.20 (m, 7H), 4.88-4.91 (m, 1H).

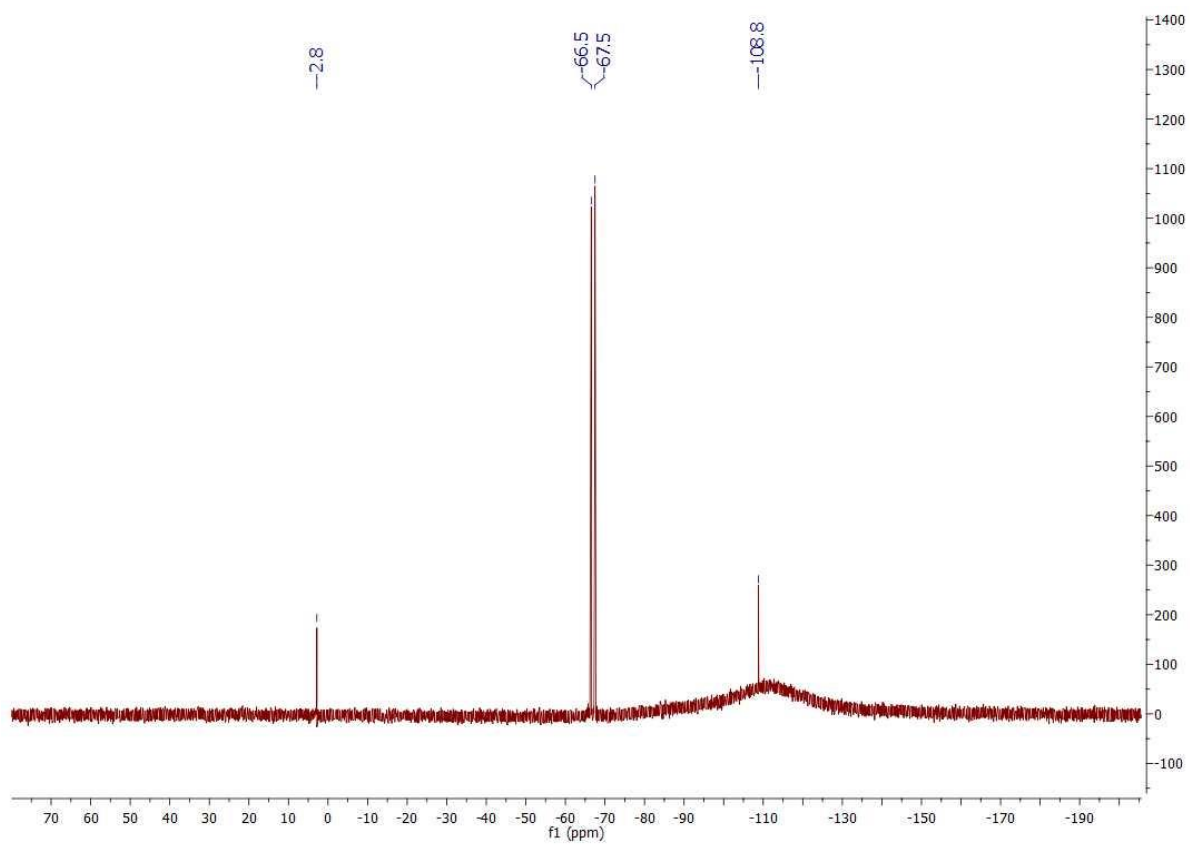
^{13}C NMR (101 MHz, C_6D_6) δ (ppm) = 6.3, 6.6, 22.5, 22.6, 24.0, 24.0, 25.6, 25.6.

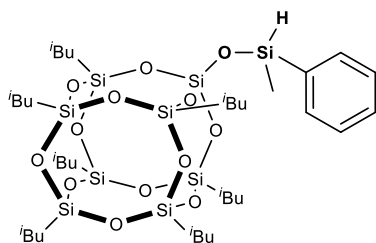
^{29}Si NMR (79 MHz, C_6D_6) δ (ppm) = 2.8, -66.5, -67.5, -108.8.

EA: $\text{C}_{32}\text{H}_{74}\text{O}_{13}\text{Si}_9$ (918.305): calcd. C 41.79, H 8.11; found C 41.63, H 8.01.









Compound (**12**) was obtained as solid in 95% yield.

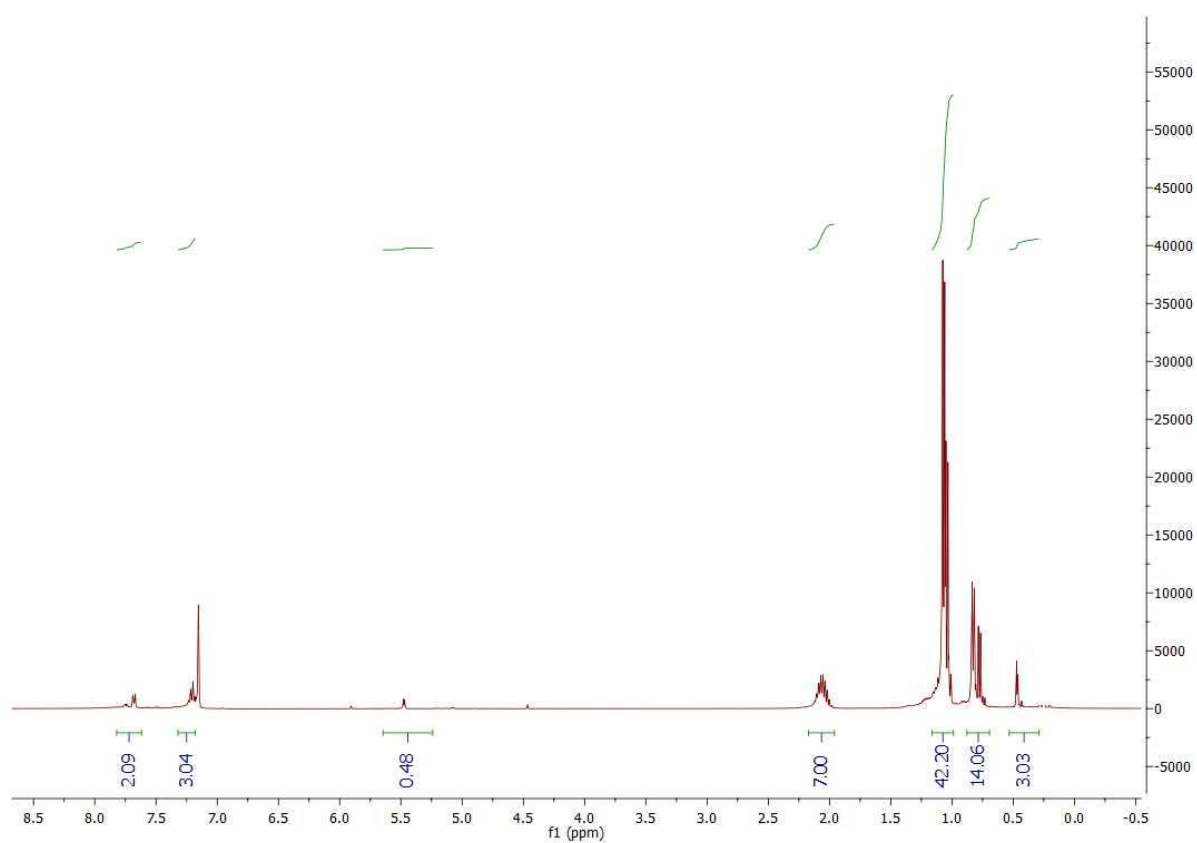
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.35-0.55 (m, 3H), 0.73-0.87 (m, 14H), 1.00-1.11 (m, 42H), 1.98-2.12 (m, 7H), 5.46-5.49 (m, 1H), 7.18-7.26 (m, 3H), 7.66-7.77 (m, 2H).

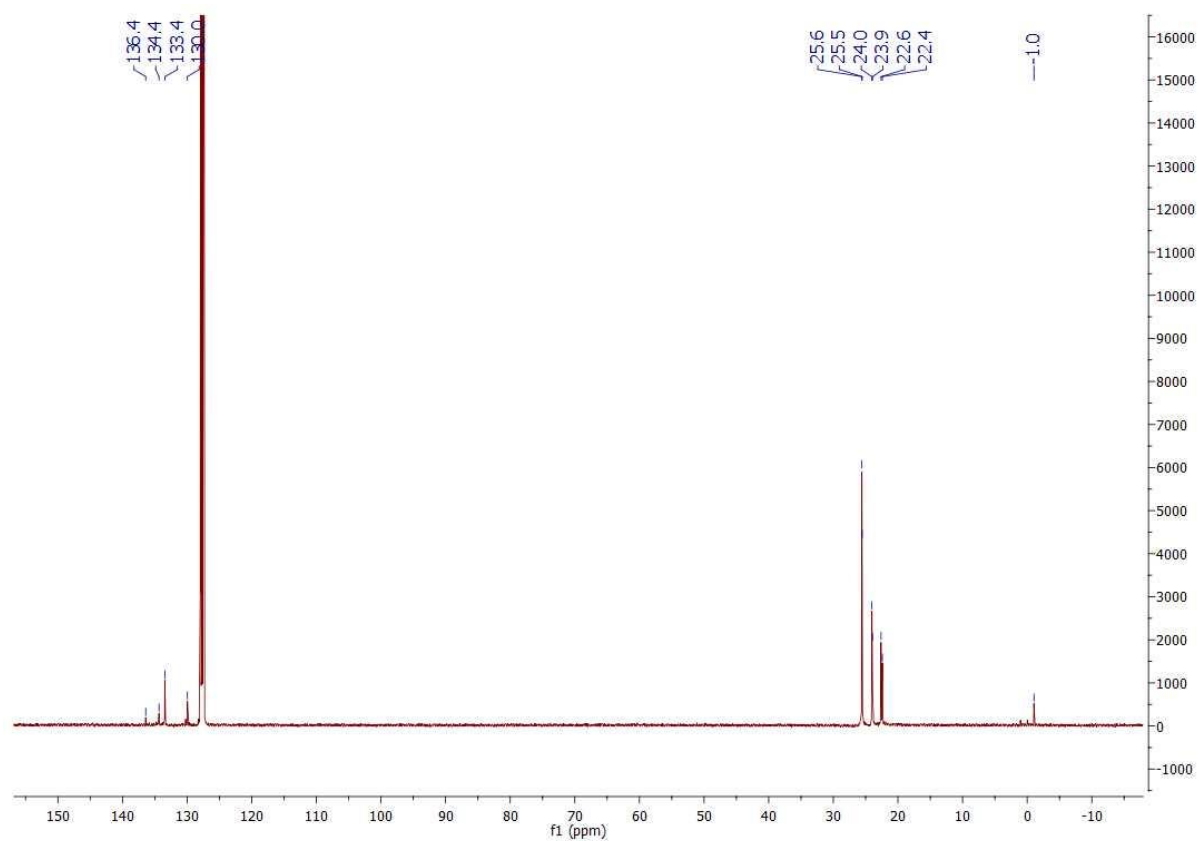
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -1.0, 22.4, 22.6, 23.9, 24.0, 25.5, 25.6, 130.0, 133.4, 134.4, 136.4.

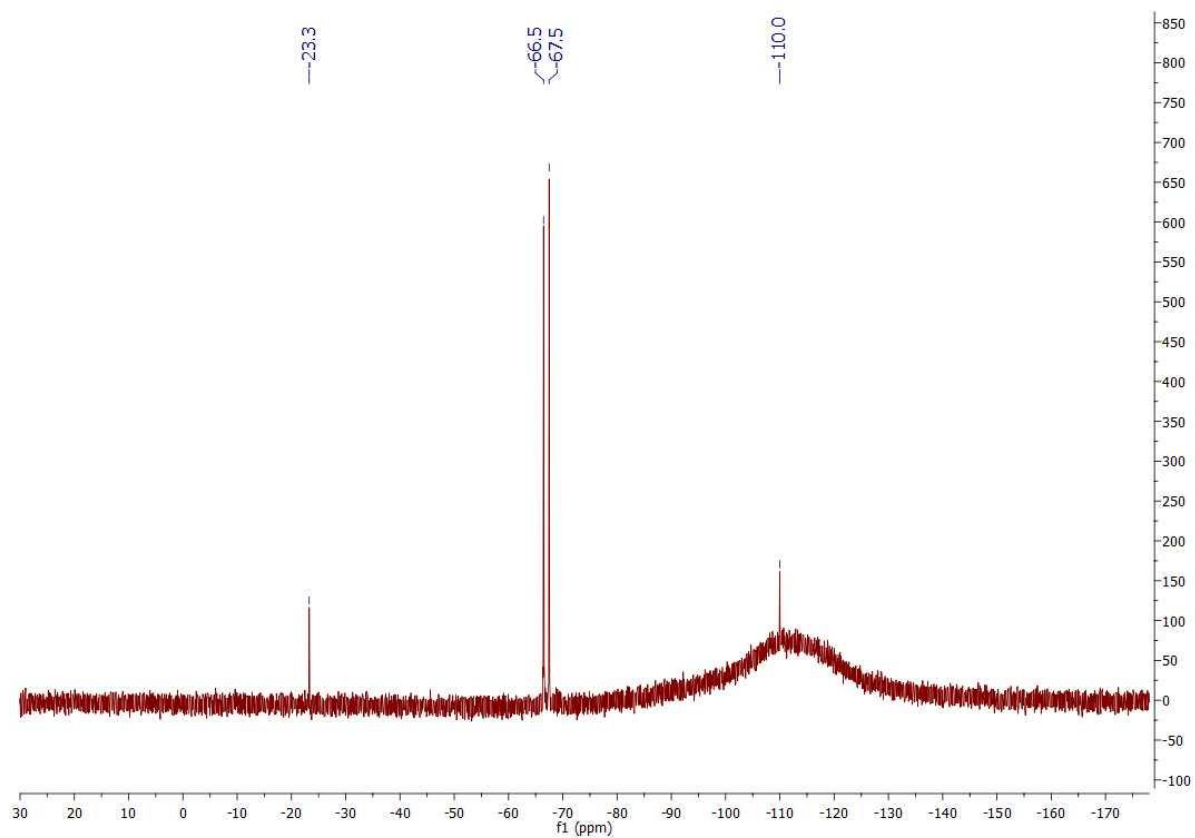
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -23.3, -66.5, -67.5, -110.0.

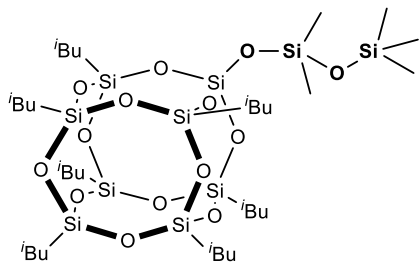
EA: C₃₅H₇₂O₁₃Si₉ (952.290): calcd. C 44.08, H 7.61; found C 43.94, H 7.85.

HRMS: calcd. for C₃₅H₇₂O₁₃Si₉Na 975.28; found 975.28.









Compound (**13**) was obtained as oil in 93% yield.

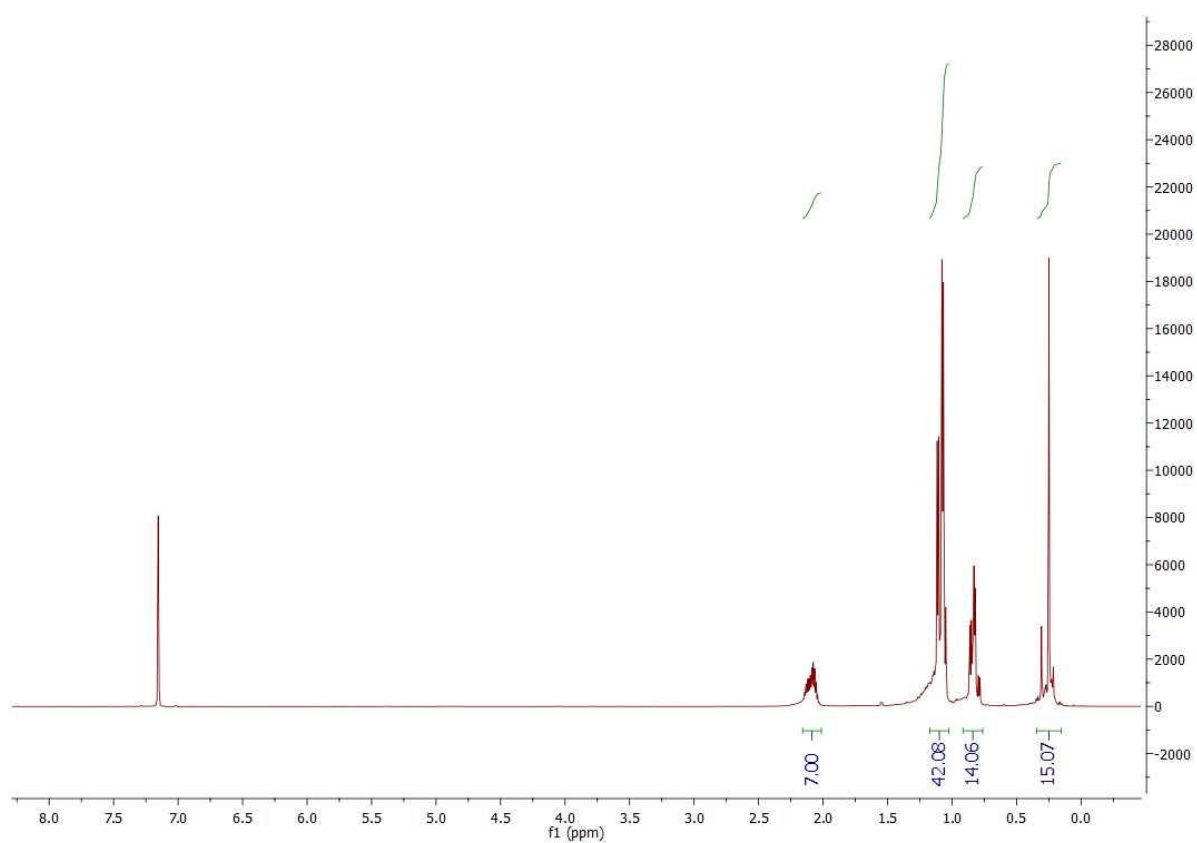
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.20-0.32 (m, 15H), 0.77-0.87 (m, 14H), 1.04-1.14 (m, 42H), 2.03-2.15 (m, 7H).

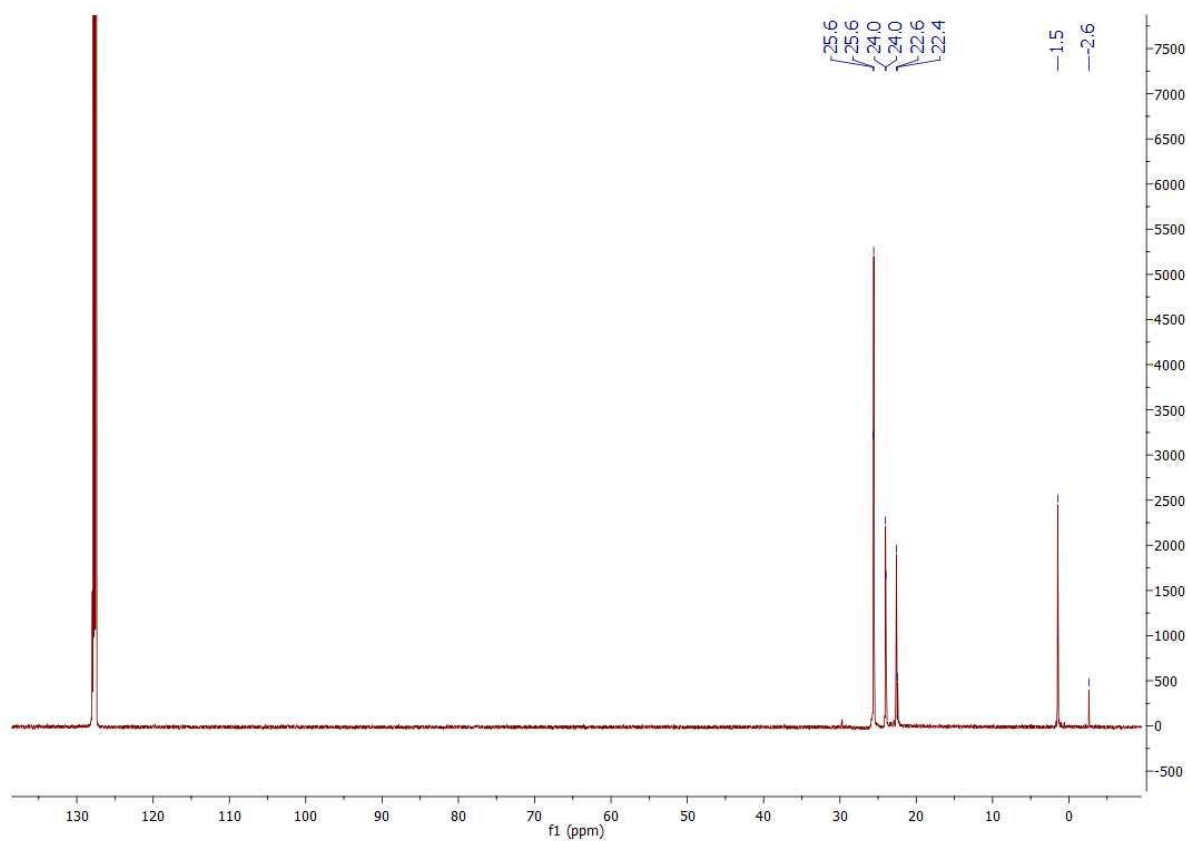
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -2.6, 1.5, 22.4, 22.6, 24.0, 24.0, 25.6, 25.6.

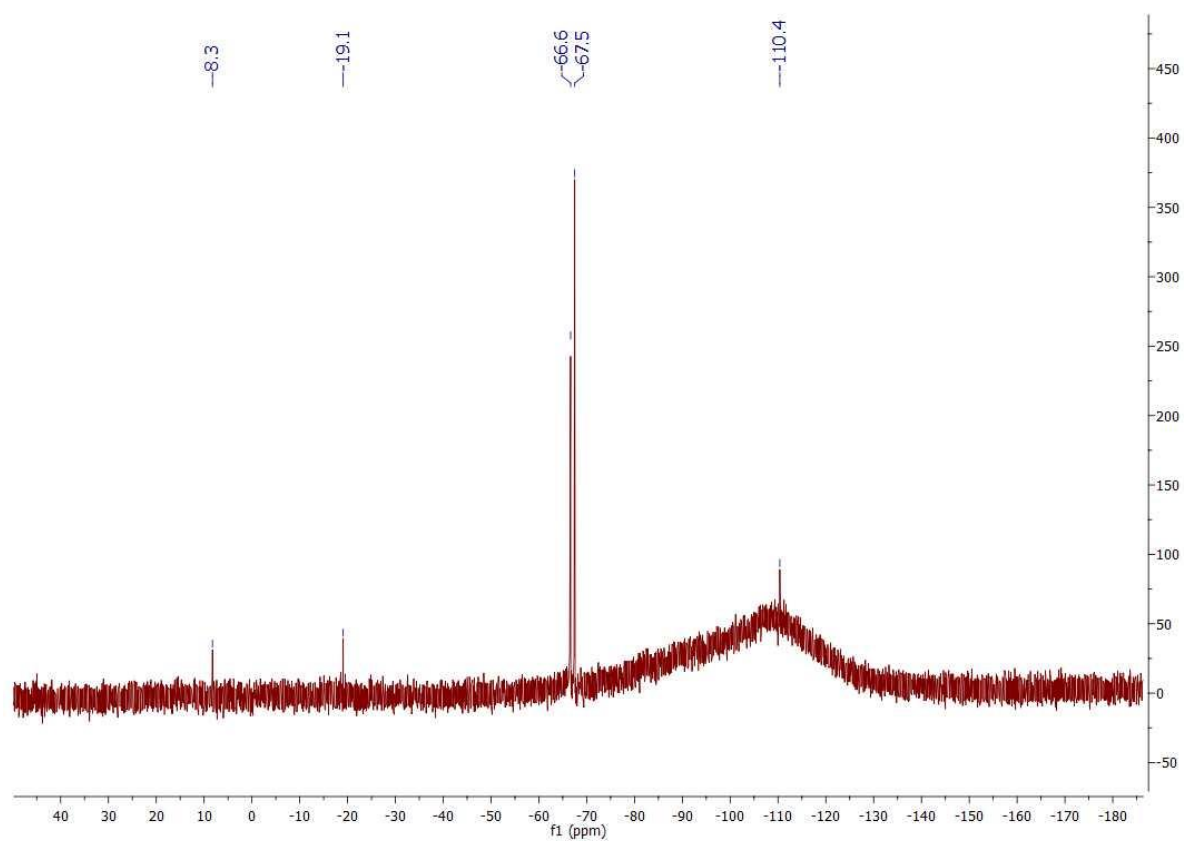
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 8.3, -19.1, -66.6, -67.5, -110.4.

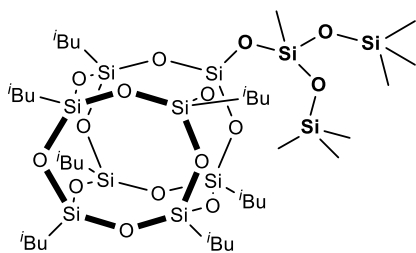
EA: C₃₃H₇₈O₁₄Si₁₀ (978.308): calcd. C 40.45, H 8.02; found C 40.27, H 7.95.

HRMS: calcd. for C₃₃H₇₈O₁₄Si₁₀Na 1001.30; found 1001.30.









Compound (**14**) was obtained as oil in 82% yield.

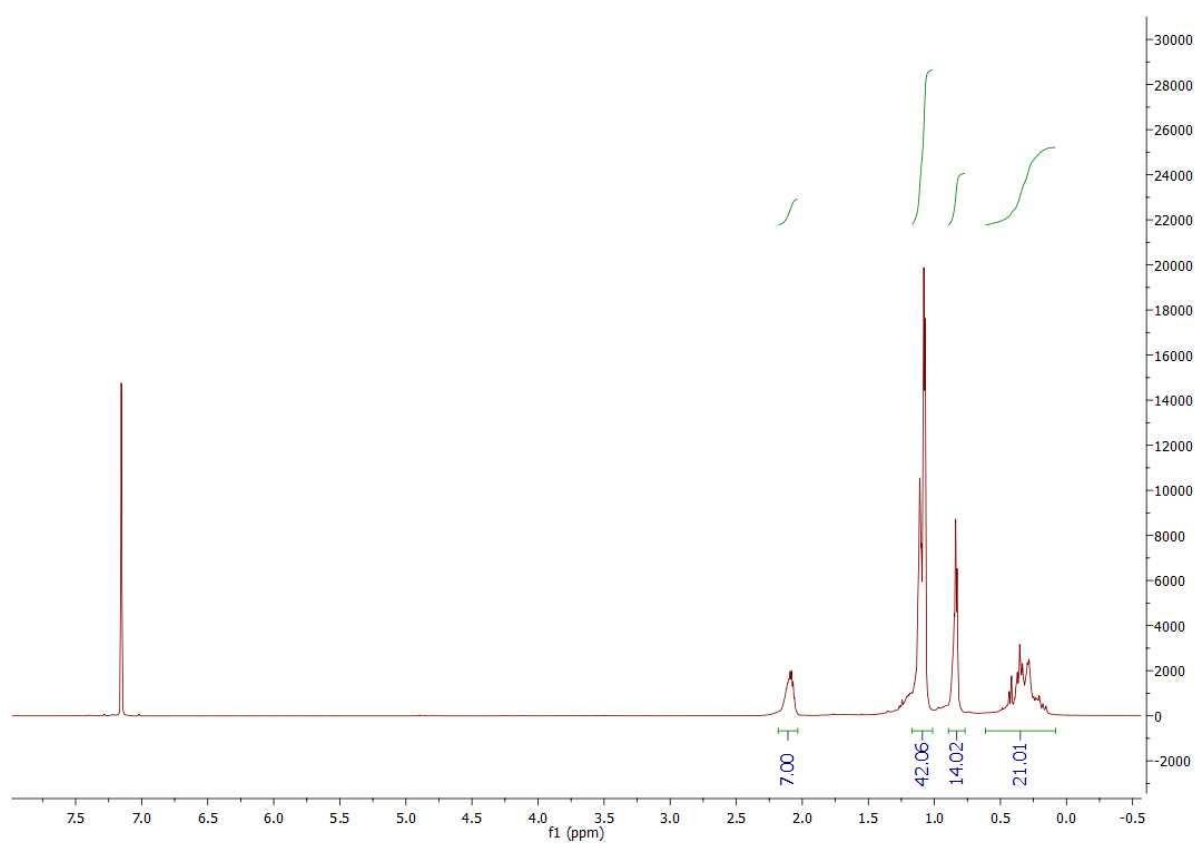
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.10-0.45 (m, 21H), 0.81-0.89 (m, 14H), 1.05-1.16 (m, 42H), 2.04-2.16 (m, 7H).

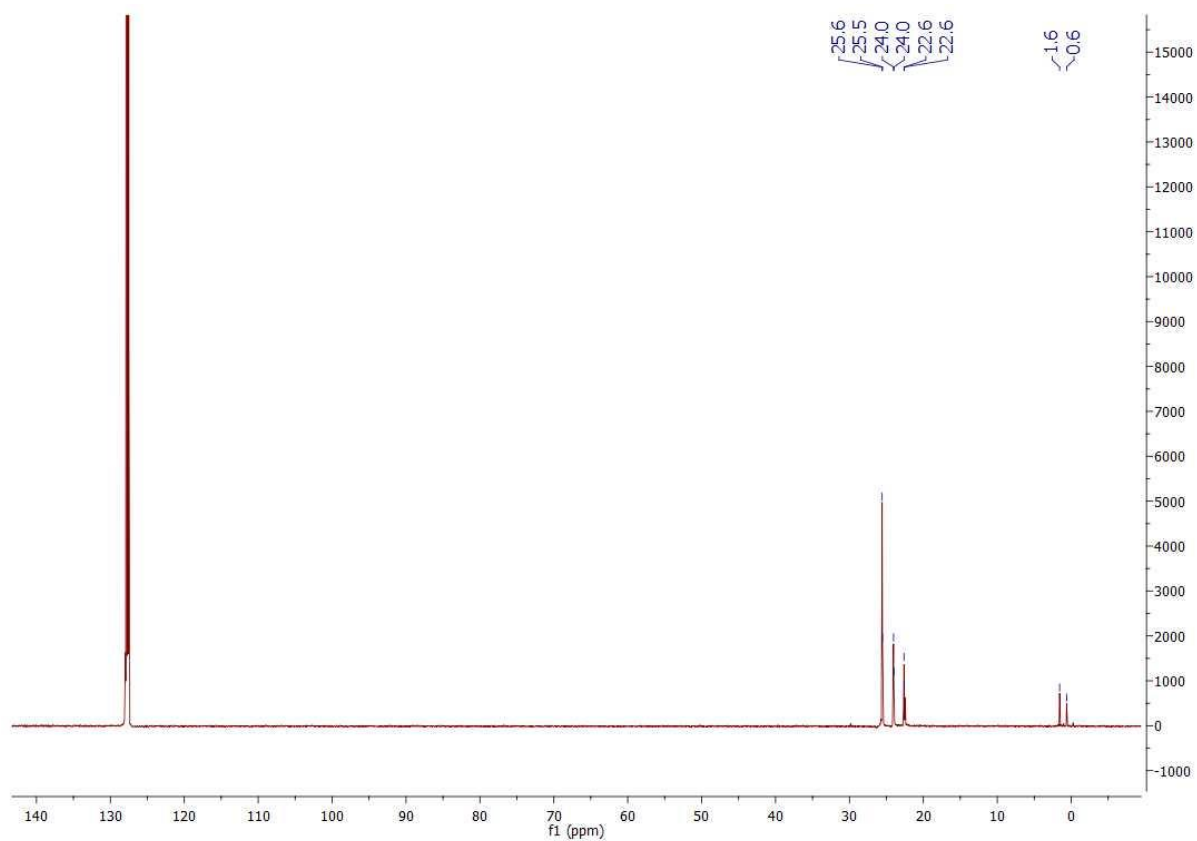
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 0.6, 1.6, 22.6, 22.6, 24.0, 24.0, 25.5, 25.6.

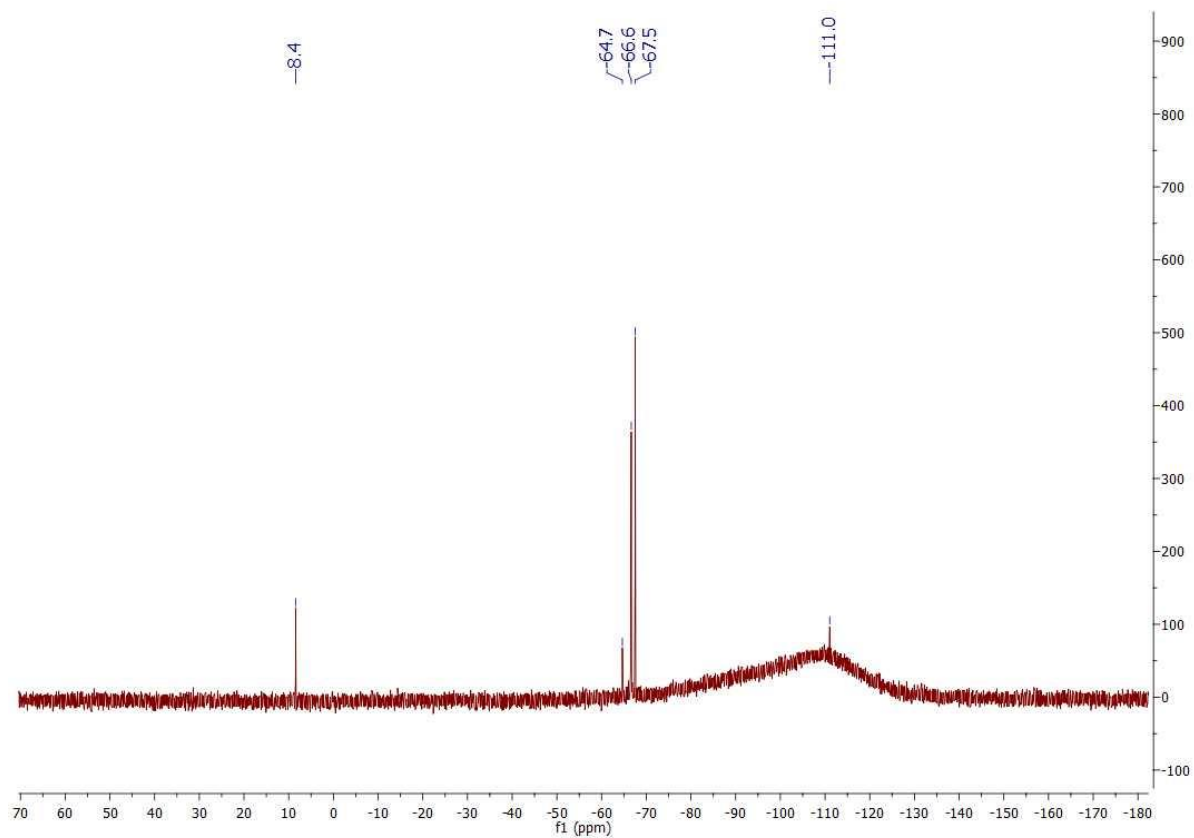
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 8.4, -64.7, -66.6, -67.5, -110.0.

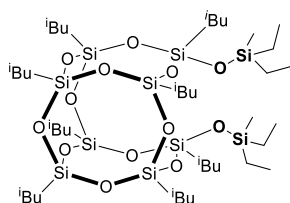
EA: C₃₅H₈₄O₁₅Si₁₁ (1052.327): calcd. C 39.89, H 8.03; found C 40.11, H 8.16.

HRMS: calcd. for C₃₅H₈₄O₁₅Si₁₁Na 1075.32; found 1075.32.









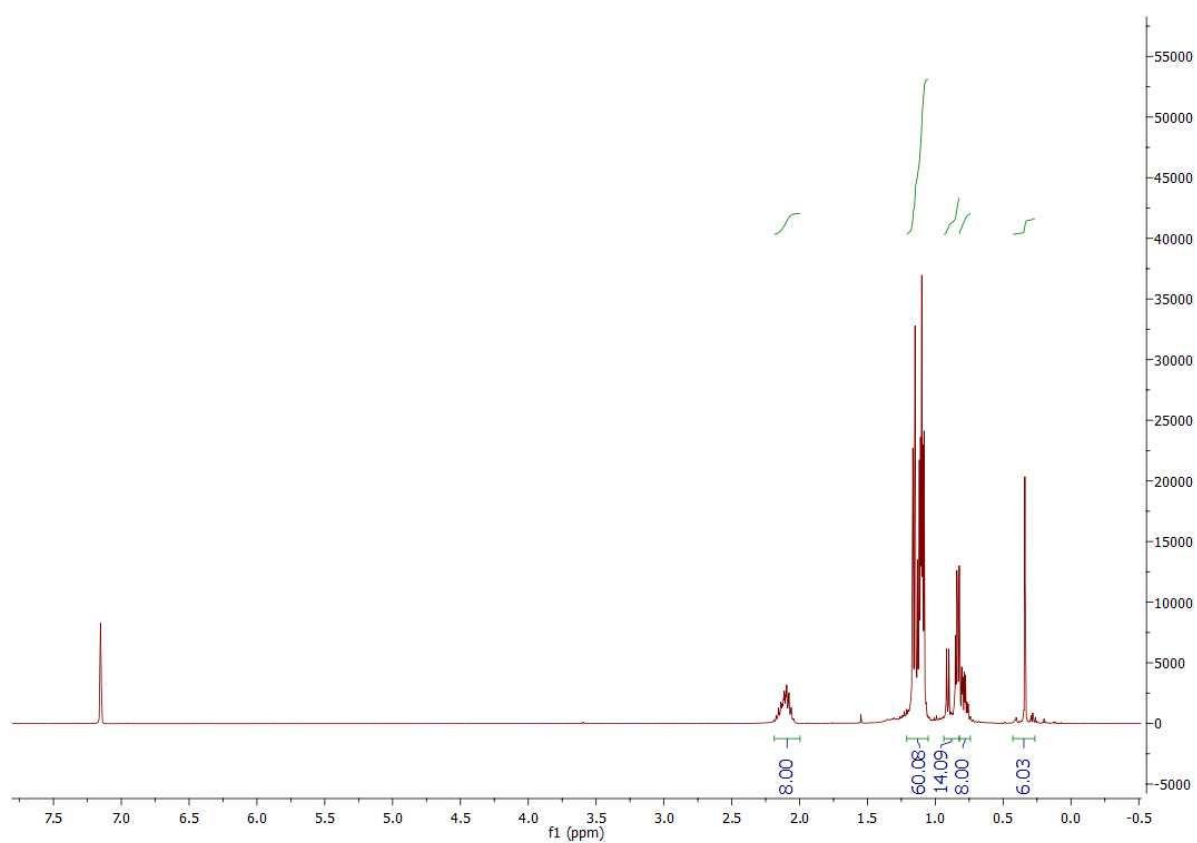
Compound (**15**) was obtained as oil in 95% yield.

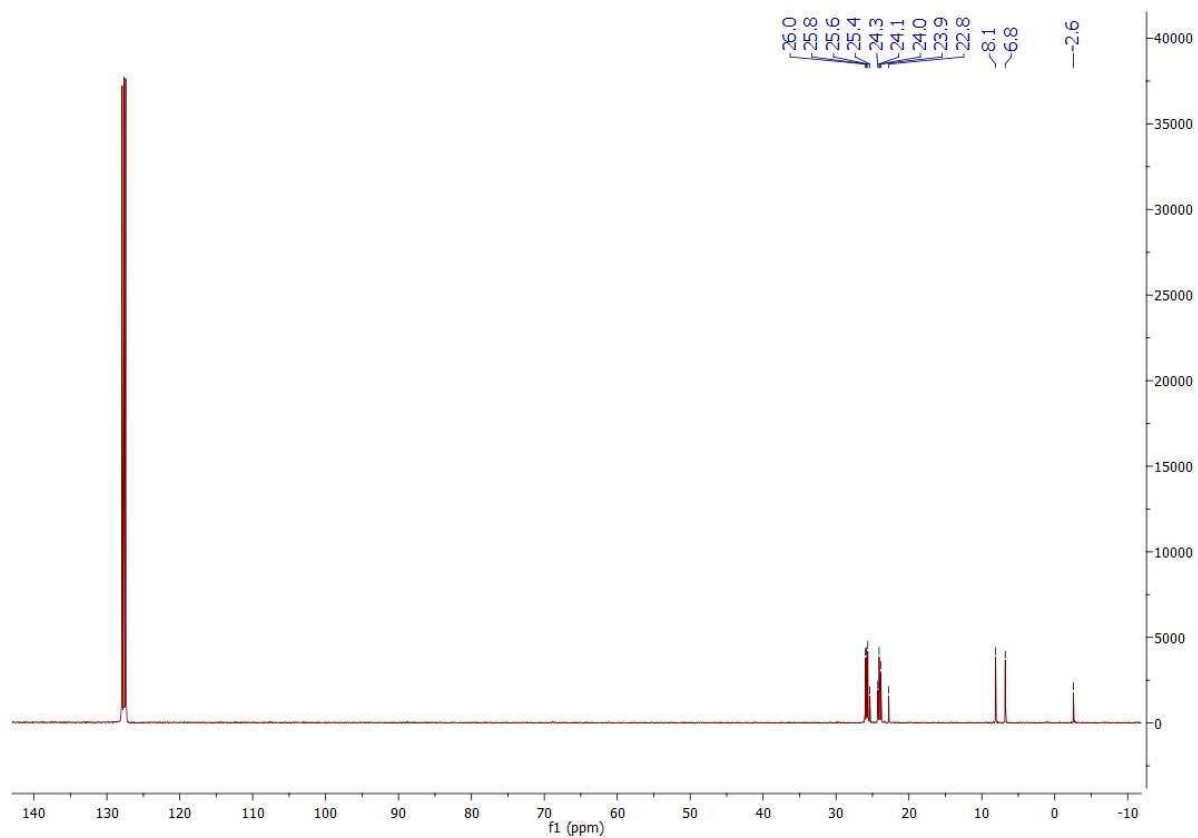
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.34 (s, 6H), 0.74-0.82 (m, 8H), 0.83-0.94 (m, 14H), 1.06-1.20 (m, 60H), 2.03-2.19 (m, 8H).

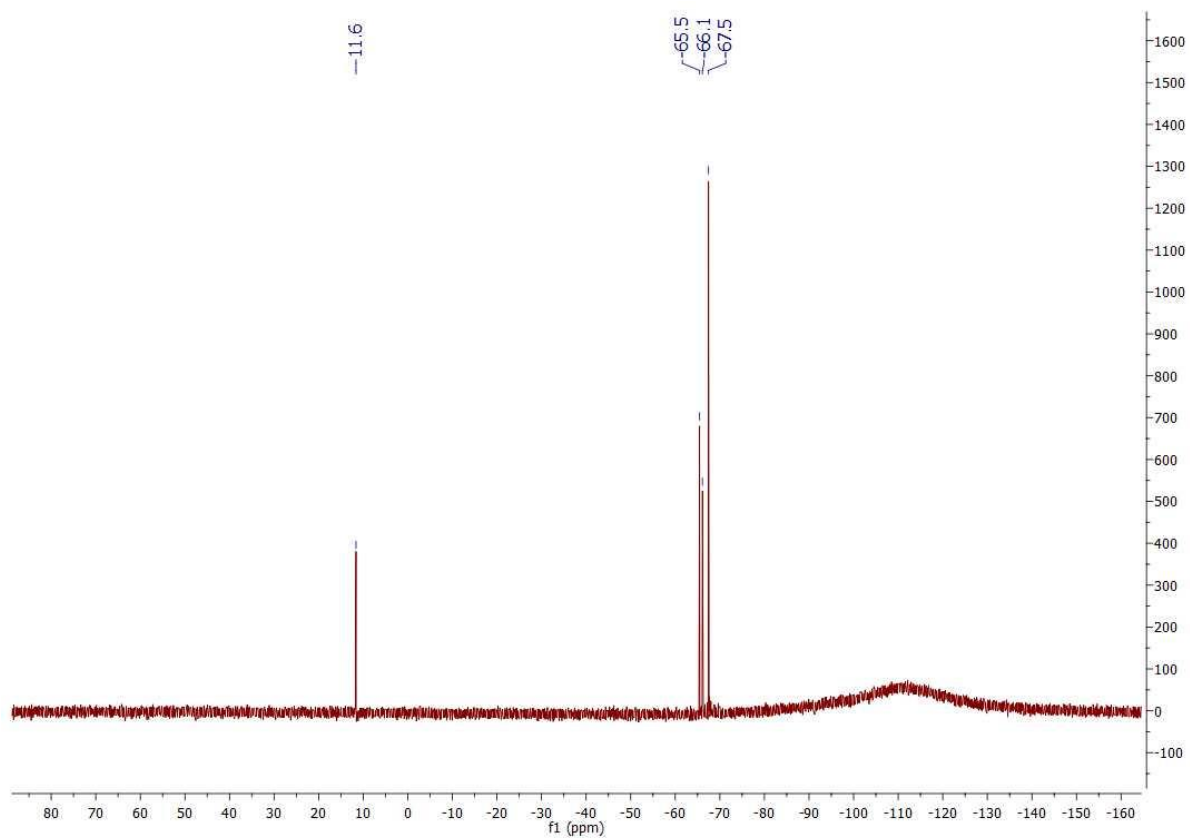
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -2.6, 6.8, 8.1, 22.8, 23.9, 24.0, 24.1, 24.3, 25.4, 25.6, 25.8, 26.8.

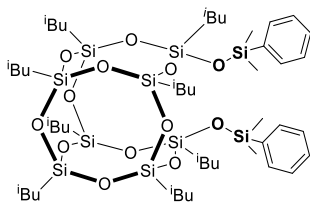
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 11.6, -65.5, -66.1, -67.5.

EA: C₄₂H₉₈O₁₃Si₁₀ (1090.470): calcd. C 46.19, H 9.05; found C 45.93, H 8.92.









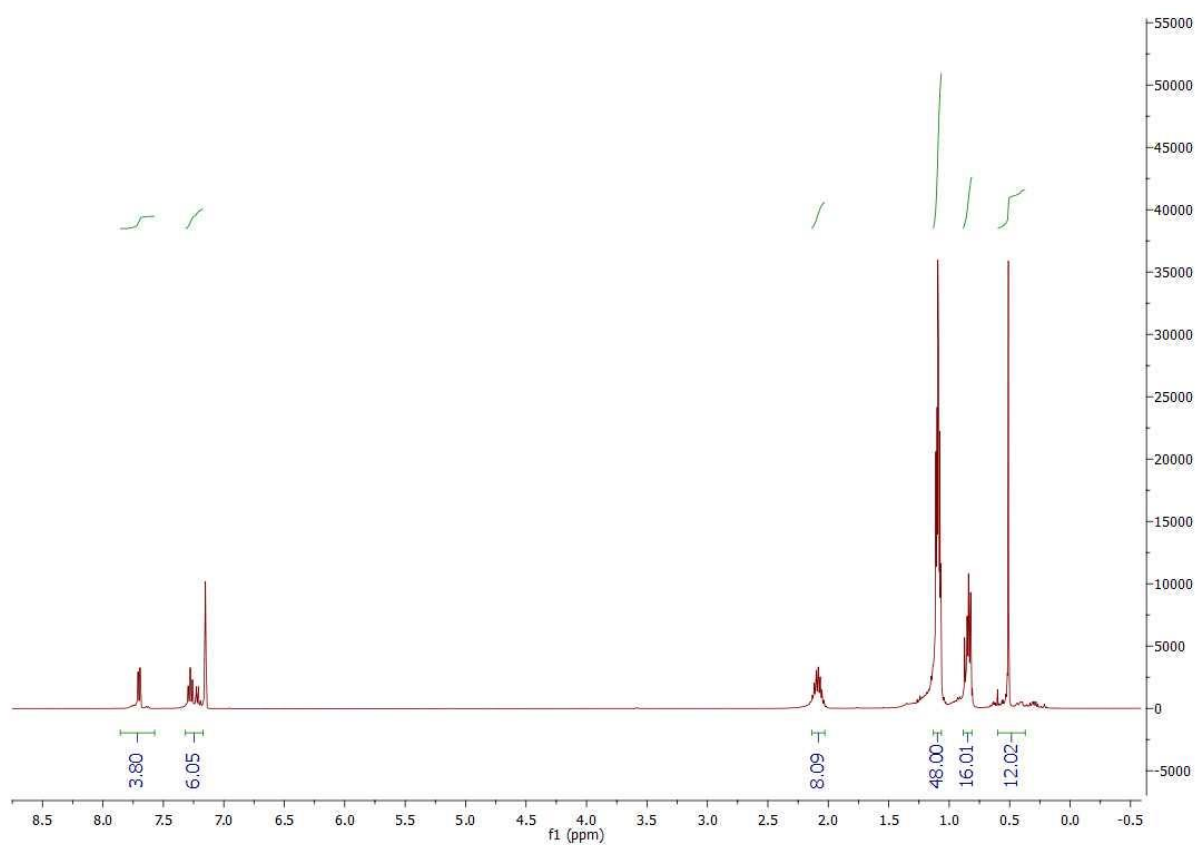
Compound (**16**) was obtained as oil in 88% yield.

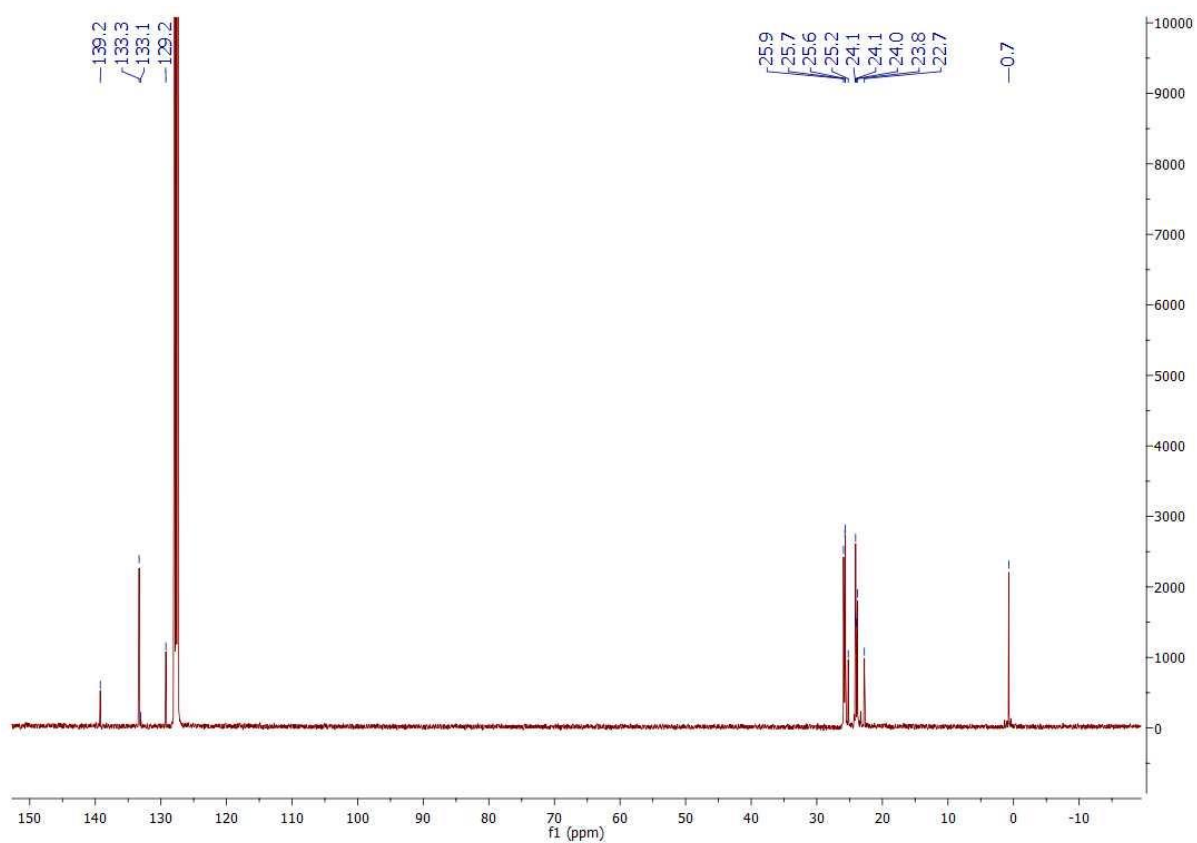
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.51 (s, 12H), 0.81-0.88 (m, 16H), 1.06-1.14 (m, 48H), 2.01-2.15 (m, 8H), 7.20-7.32 (m, 6H), 7.68-7.75 (m, 4H).

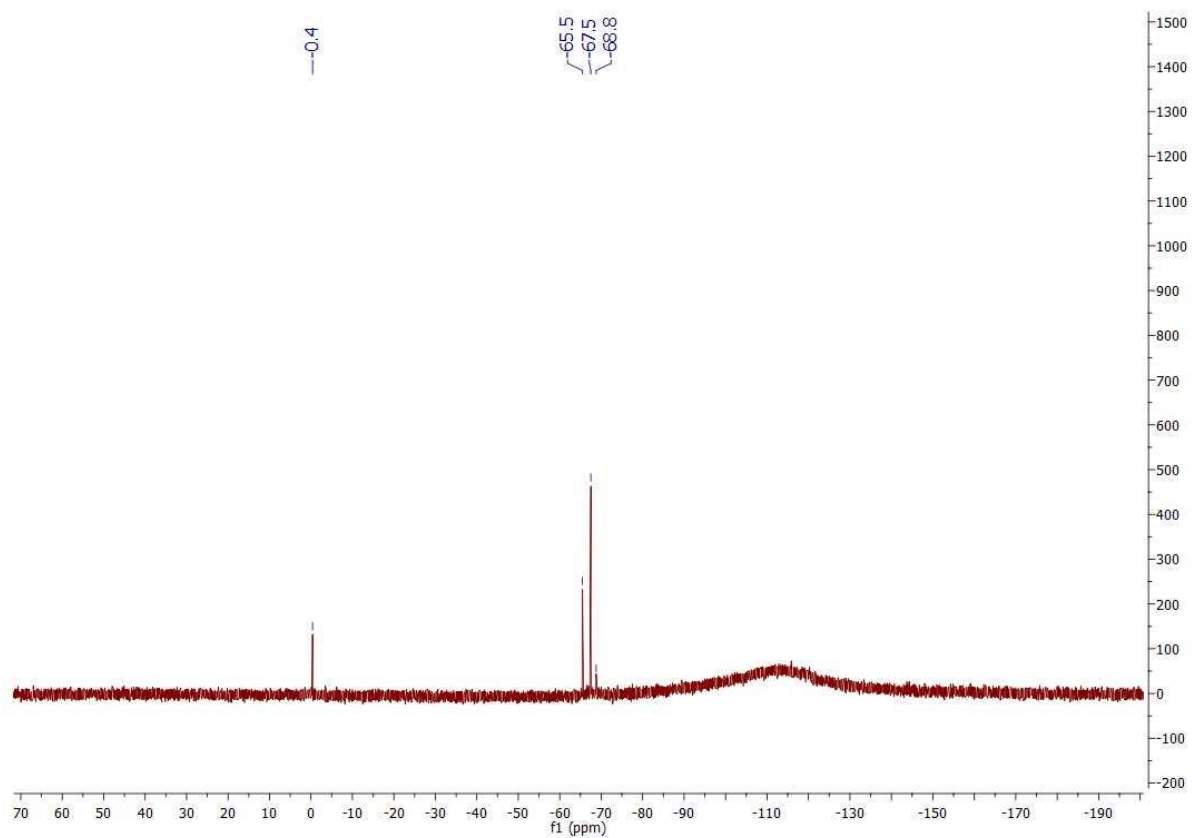
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 0.7, 22.7, 23.8, 24.0, 24.1, 24.1, 25.2, 25.6, 25.7, 25.9, 129.2, 133.1, 133.3, 139.2.

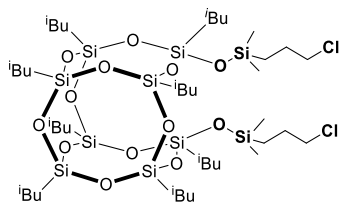
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -0.4, 65.5, 67.5, 68.8.

EA: C₄₈H₉₄O₁₃Si₁₀ (1158.439): calcd. C 49.70, H 8.17; found C 49.83, H 8.26.









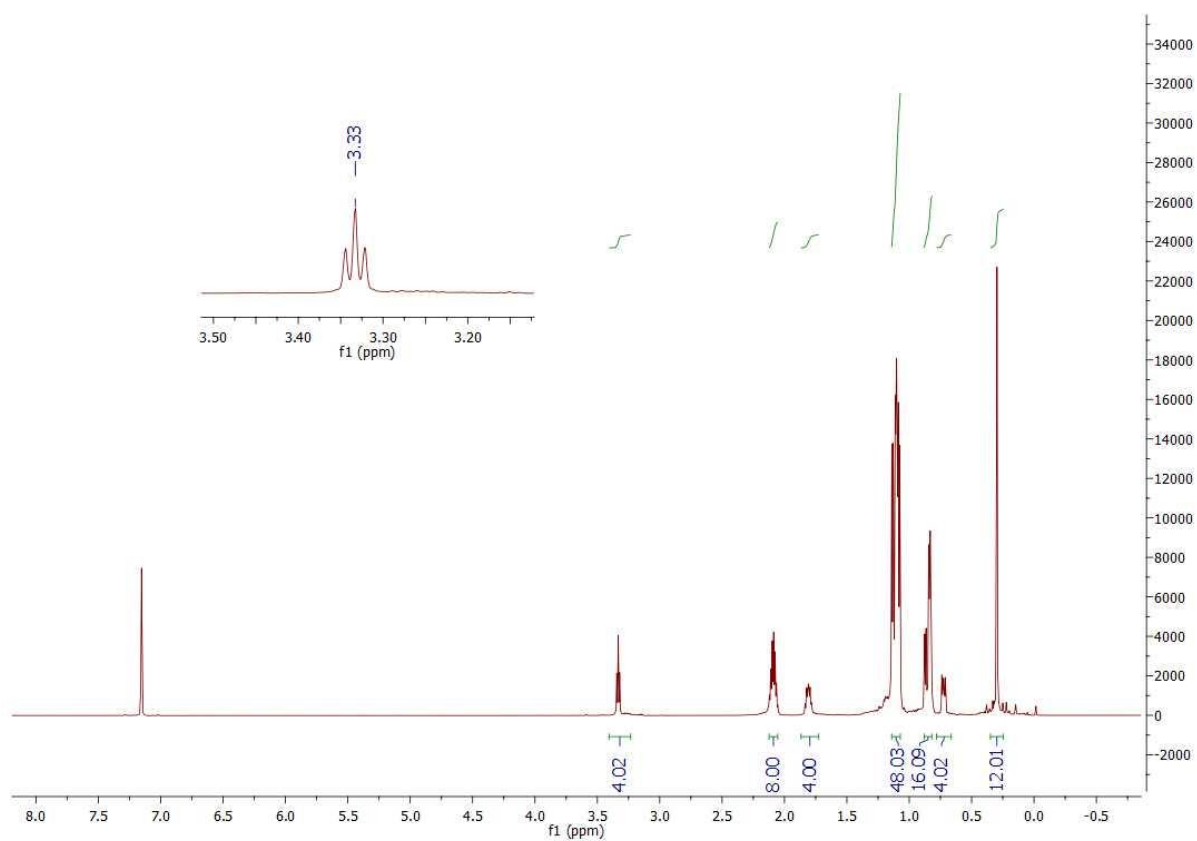
Compound (**17**) was obtained as oil in 81% yield.

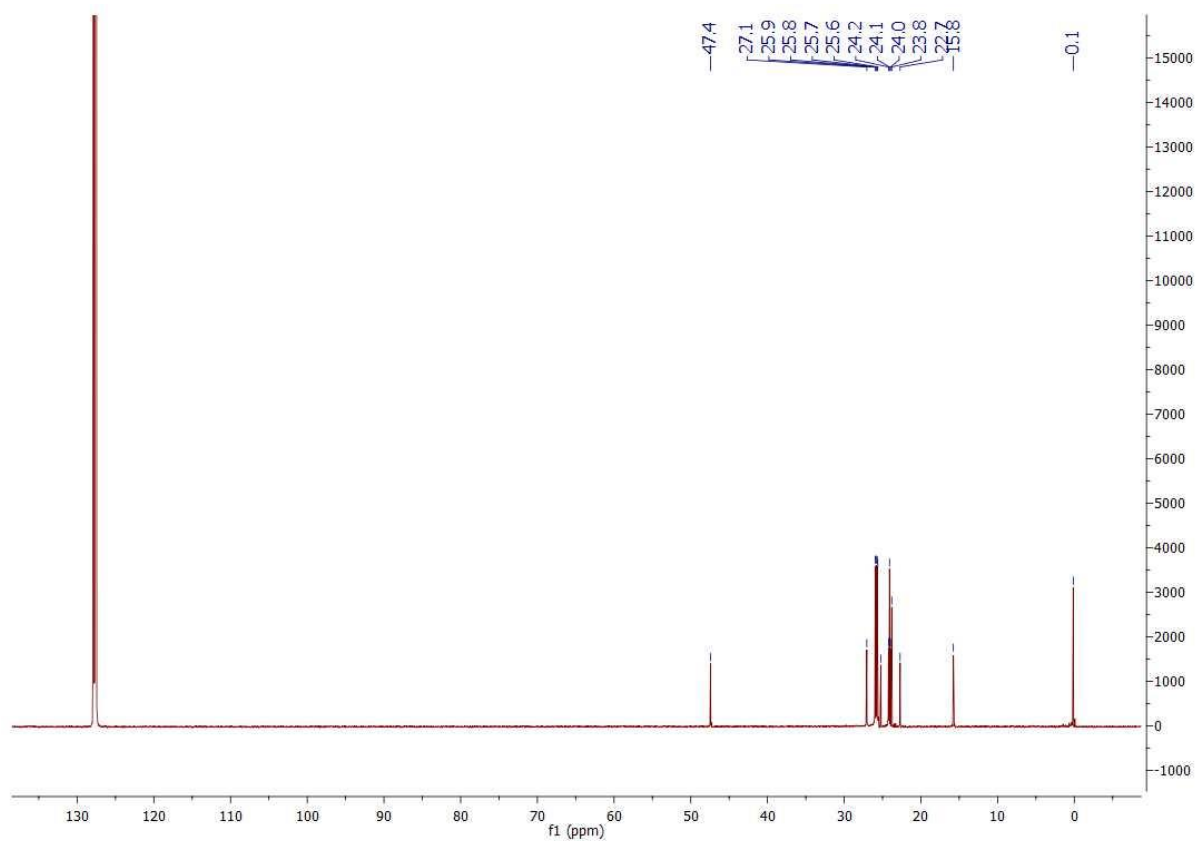
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.30 (s, 12H), 0.69-0.75 (m, 4H), 0.82-0.89 (m, 16H), 1.06-1.15 (m, 48H), 1.77-1.84 (m, 4H), 3.33 (t, J = 6.9 Hz, 4H).

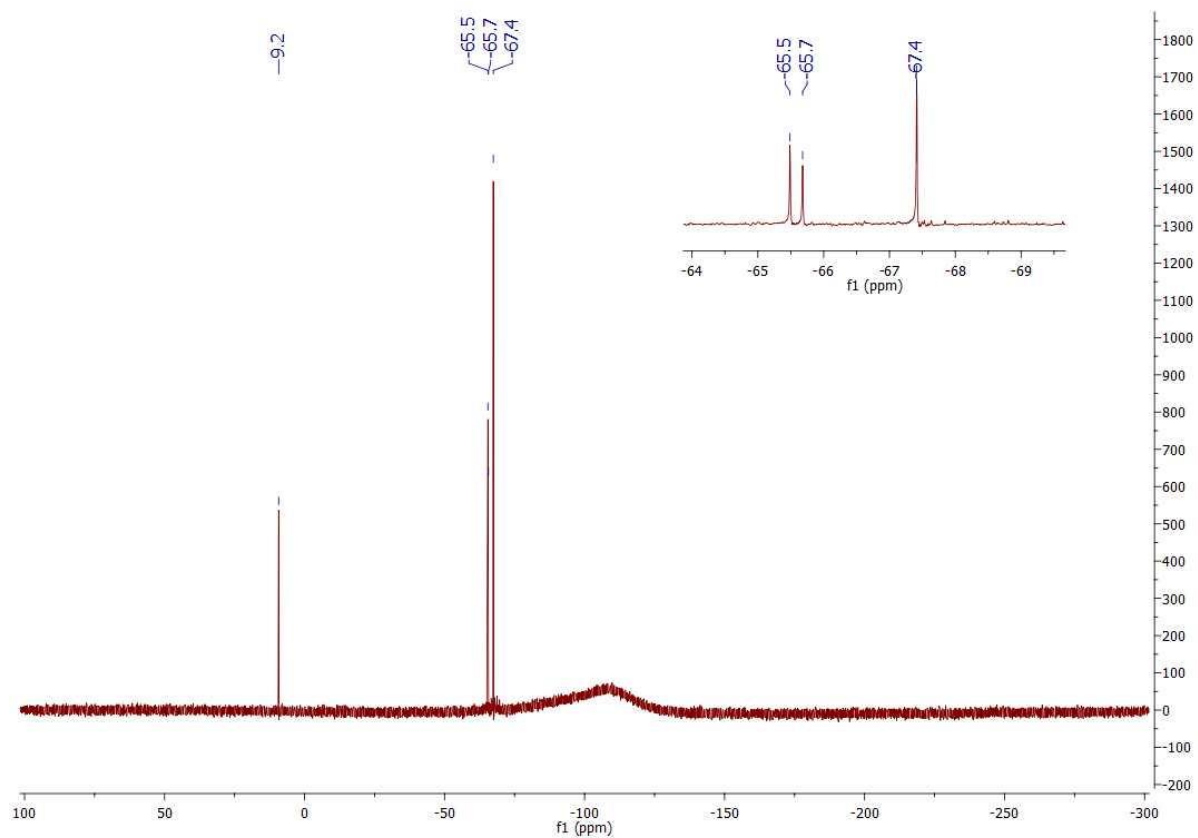
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 0.1, 15.8, 22.7, 23.8, 24.0, 24.1, 24.2, 25.2, 25.6, 25.7, 25.8, 25.9, 27.1, 47.4.

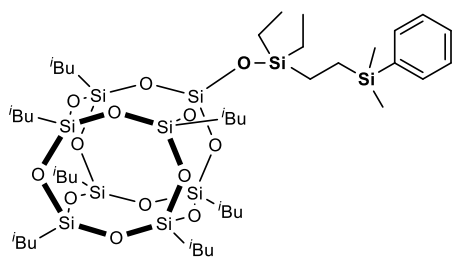
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 9.2, -65.5, -65.7, -67.4.

EA: C₄₂H₉₆Cl₂O₁₃Si₁₀ (1158.392): calcd. C 43.45, H 8.34; found C 43.63, H 8.44.









Compound (**18**) was obtained as oil in 89% yield.

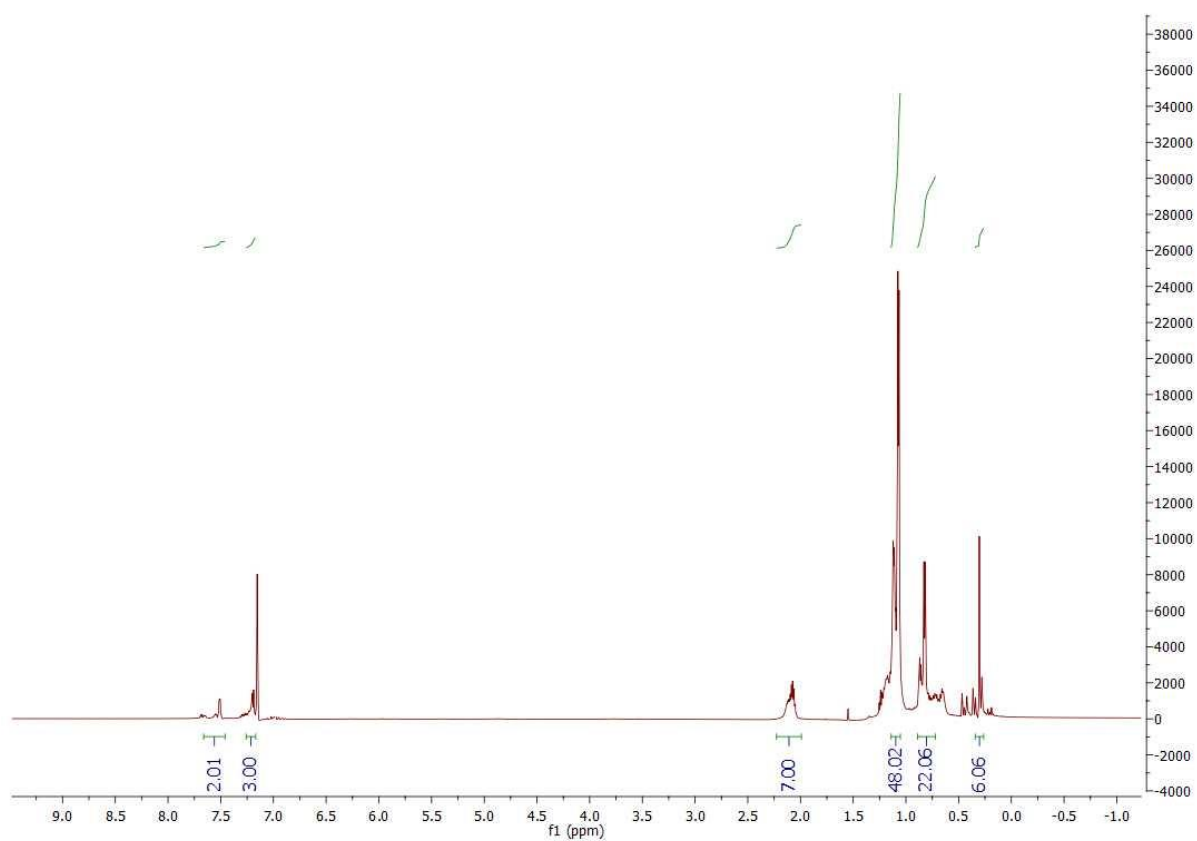
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.22-0.35 (m, 6H), 0.72-0.90 (m, 22H), 1.04-1.16 (m, 48H), 2.01-2.16 (m, 7H), 7.18-7.24 (m, 3H), 7.46-7.64 (m, 2H).

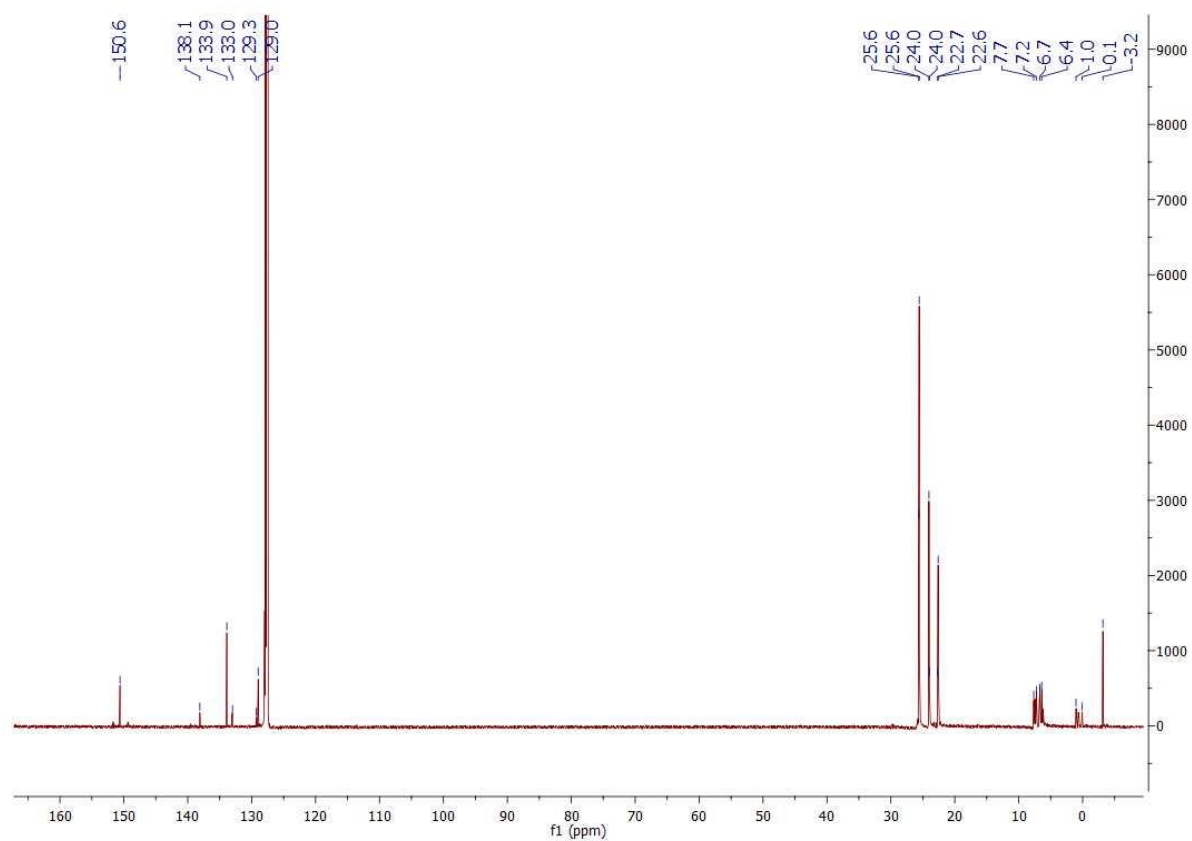
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = -3.2, 0.1, 1.0, 6.4, 6.7, 7.2, 7.7, 22.6, 22.7, 24.0, 24.0, 25.6, 25.6, 129.0, 129.3, 133.0, 133.9, 138.1, 150.6.

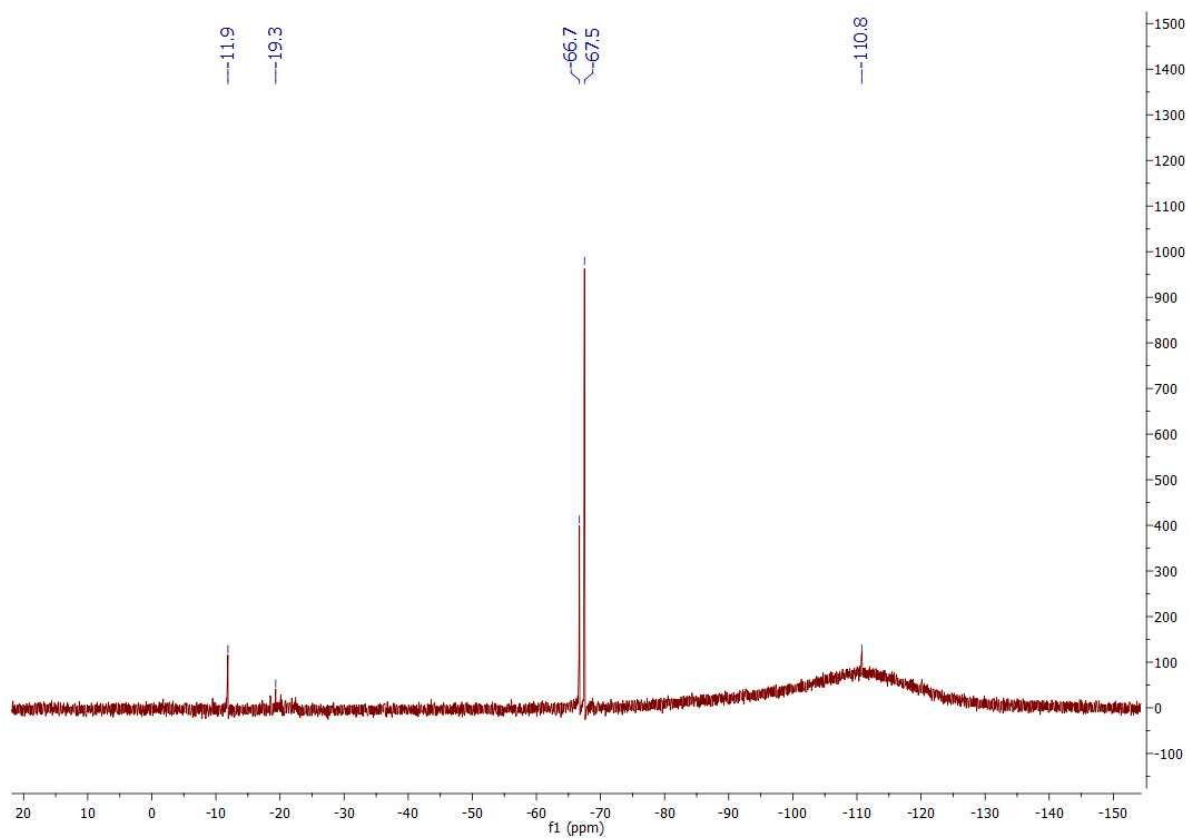
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -11.9, -19.3, -66.7, -67.5, -110.8.

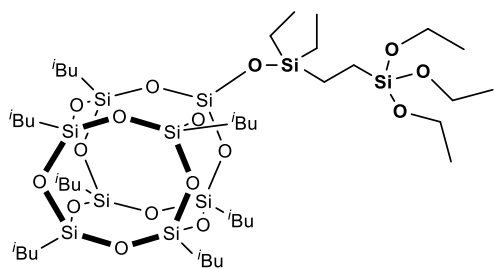
EA: C₄₂H₈₈O₁₃Si₁₀ (1080.392): calcd. C 46.62, H 8.20; found C 46.81, H 8.32.

HRMS: calcd. for C₄₂H₈₈O₁₃Si₁₀Na 1103.38; found 1103.38.









Compound (**19**) was obtained as oil in 84% yield.

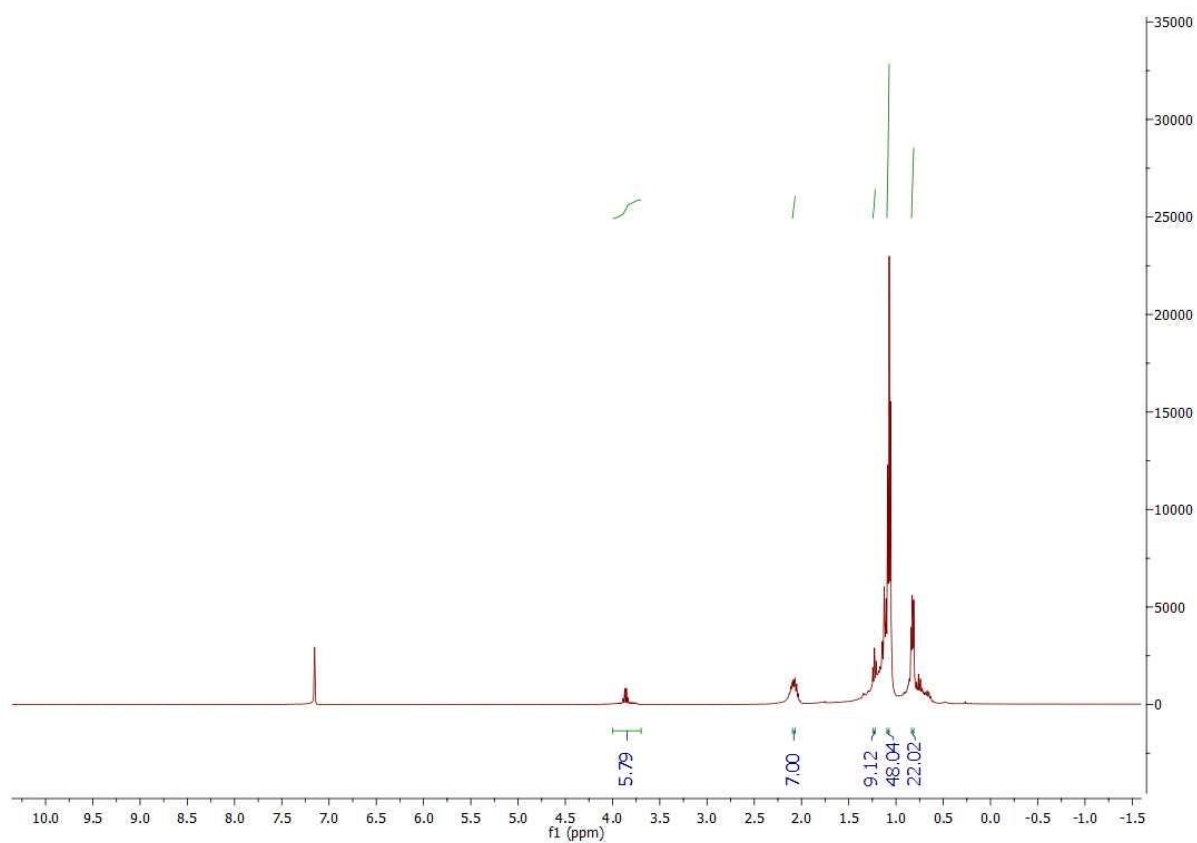
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.80-0.86 (m, 22H), 1.04-1.13 (m, 48H), 1.21-1.25 (m, 9H), 2.05-2.12 (m, 7H), 3.86 (q, *J* = 7.0 Hz, 6H).

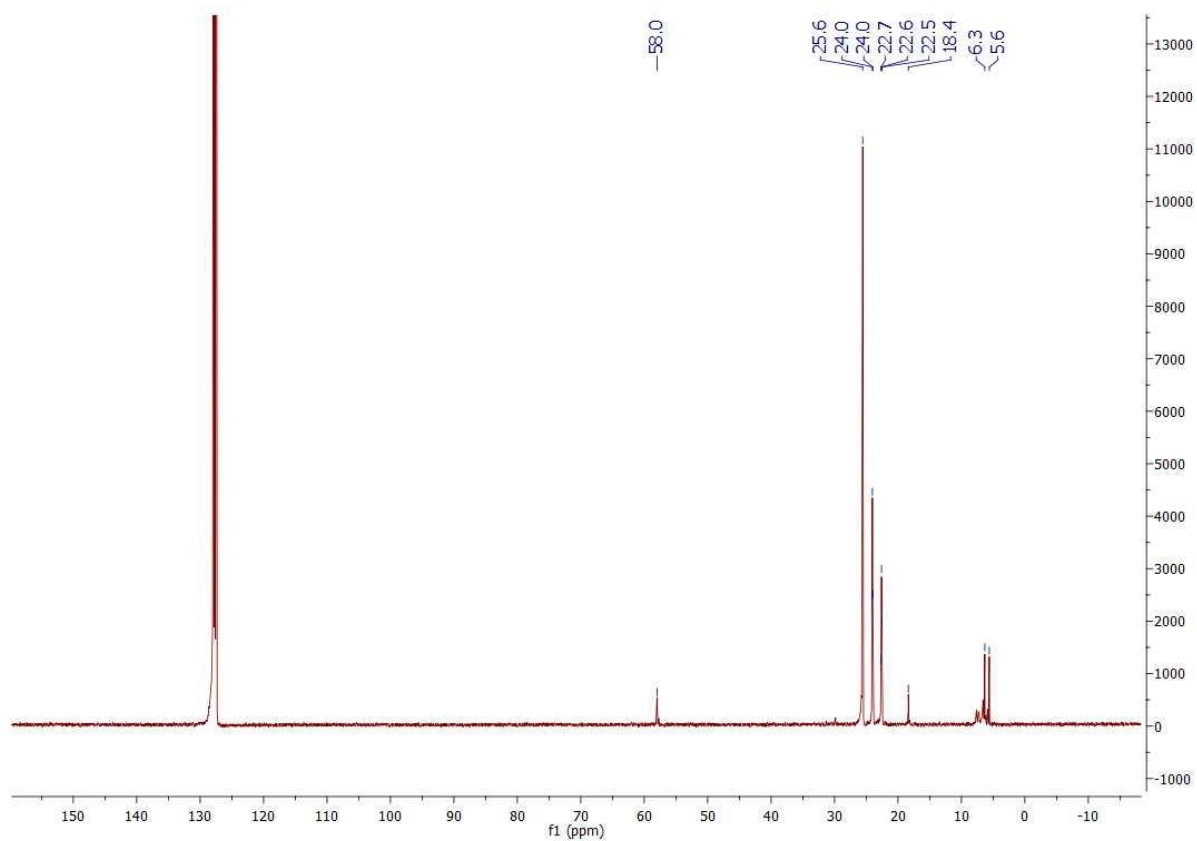
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 5.6, 6.3, 18.4, 22.5, 22.6, 22.7, 24.0, 24.0, 25.6, 58.0.

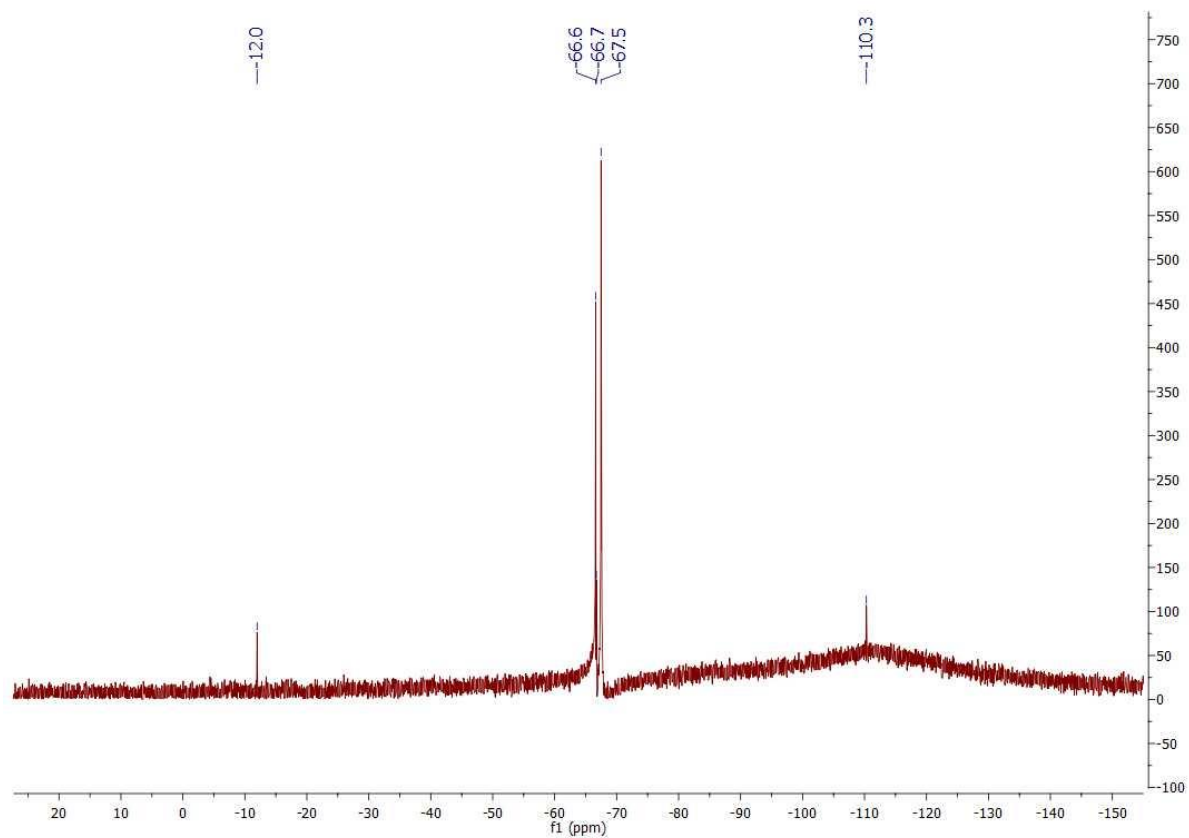
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -12.0, -66.6, -66.7, -67.5, -110.3.

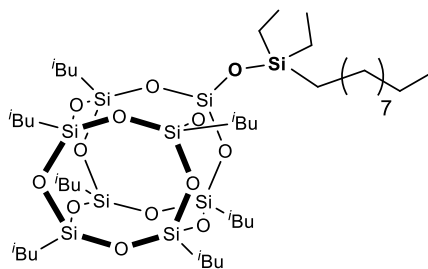
EA: C₄₀H₉₂O₁₆Si₁₀ (1108.408): calcd. C 43.28, H 8.35; found C 43.46, H 8.47.

HRMS: calcd. for C₄₀H₉₂O₁₆Si₁₀Na 1131.40; found 1131.40.









Compound (**20**) was obtained as oil in 88% yield.

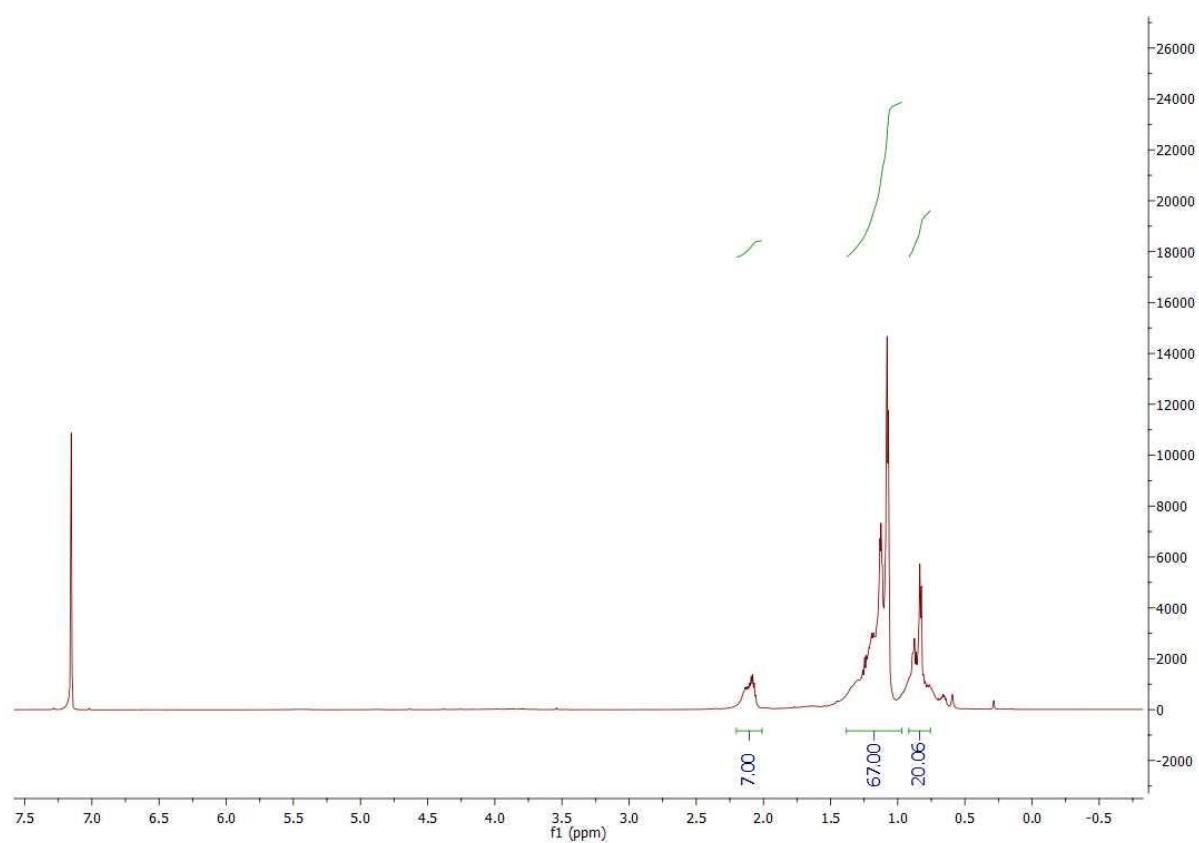
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.75-0.90 (m, 20H), 1.05-1.30 (m, 67H), 2.03-2.17 (m, 7H).

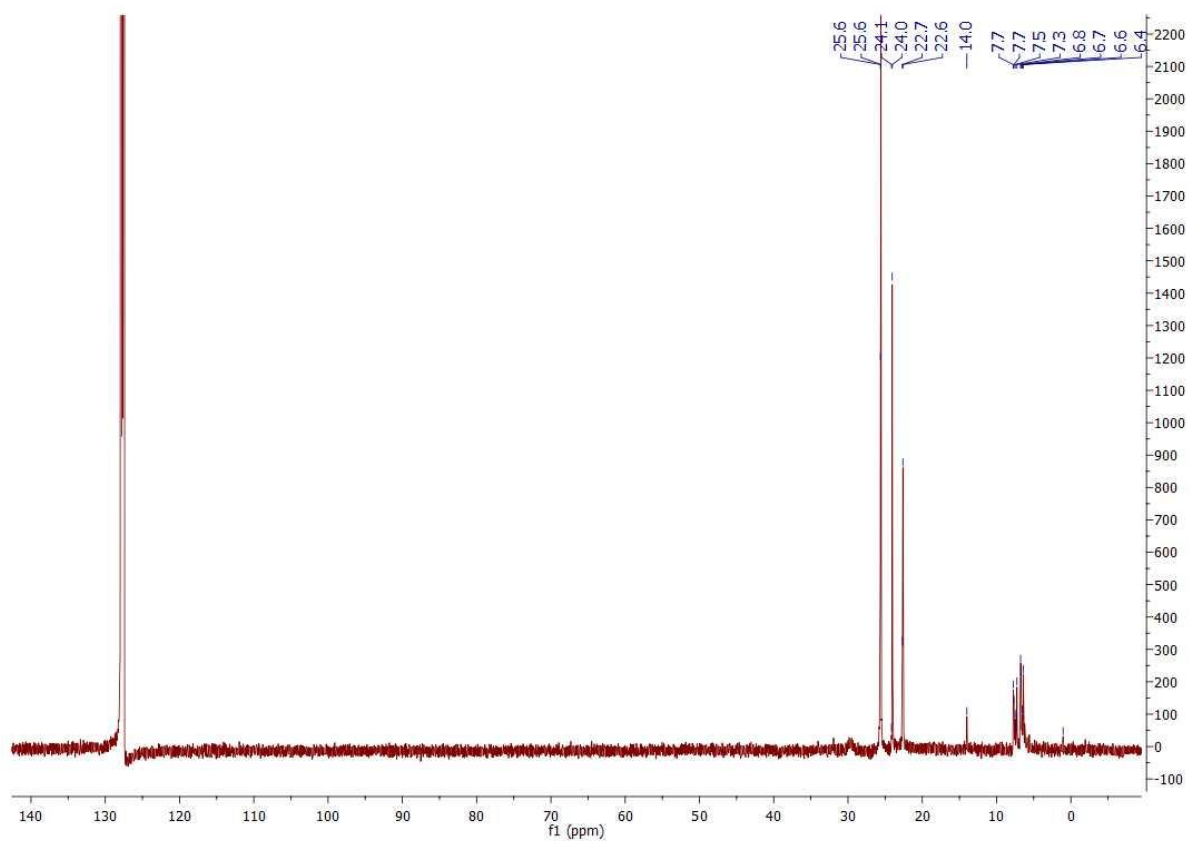
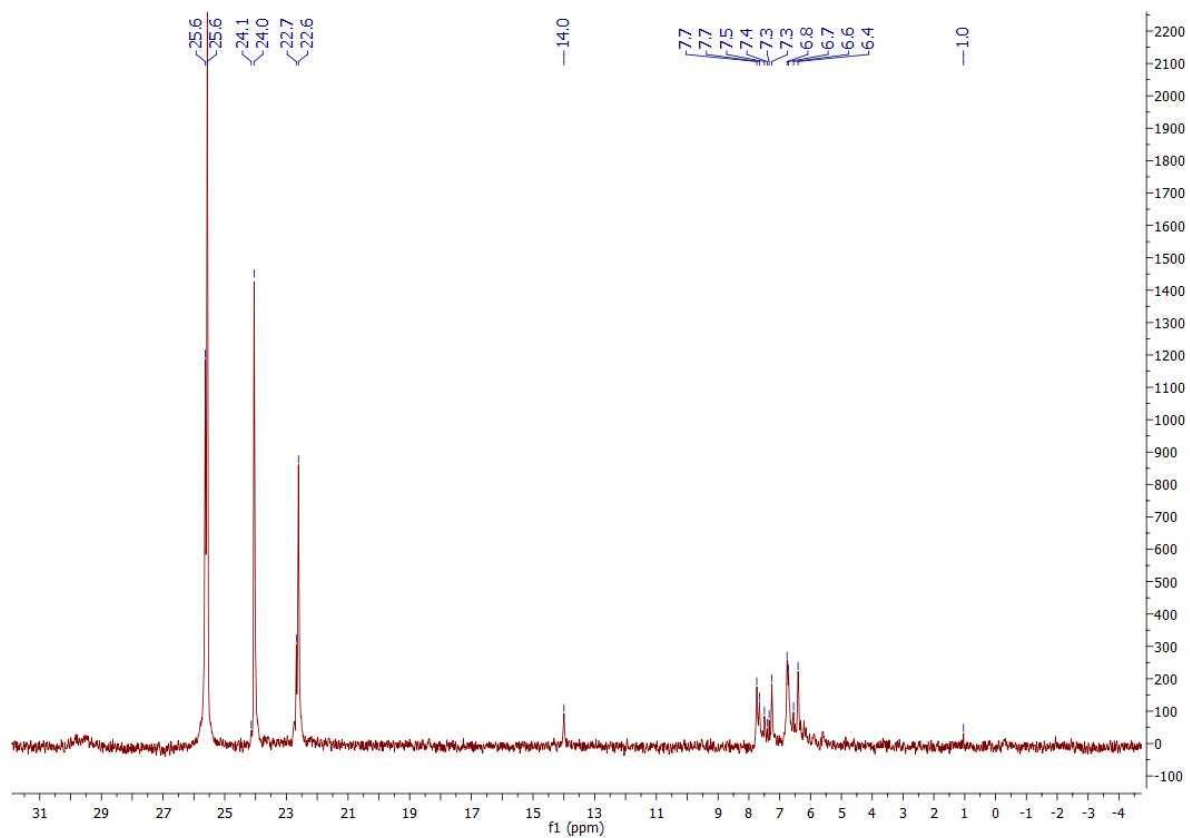
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 1.0, 6.4, 6.6, 6.7, 6.8, 7.3, 7.3, 7.4, 7.5, 7.7, 7.7, 14.0, 22.6, 22.7, 24.0, 24.1, 25.6, 25.6.

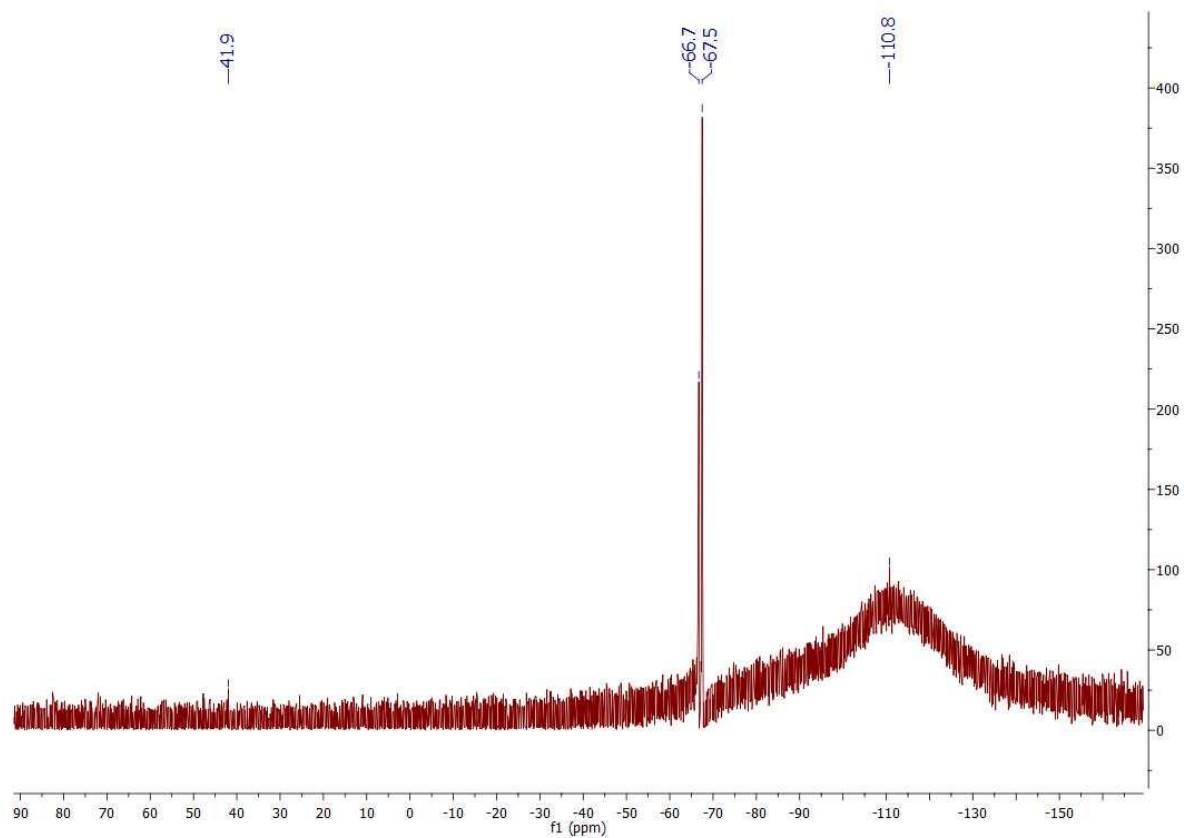
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 41.9, -66.7, -67.5, -100.8.

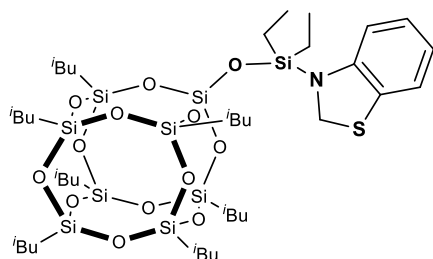
EA: C₃₆H₈₂O₁₃Si₉ (974.368): calcd. C 44.31, H 8.47; found C 44.20, H 8.38.

HRMS: calcd. for C₃₆H₈₂O₁₃Si₉Na 997.36; found 997.36.









Compound (**21**) was obtained as oil in 93% yield.

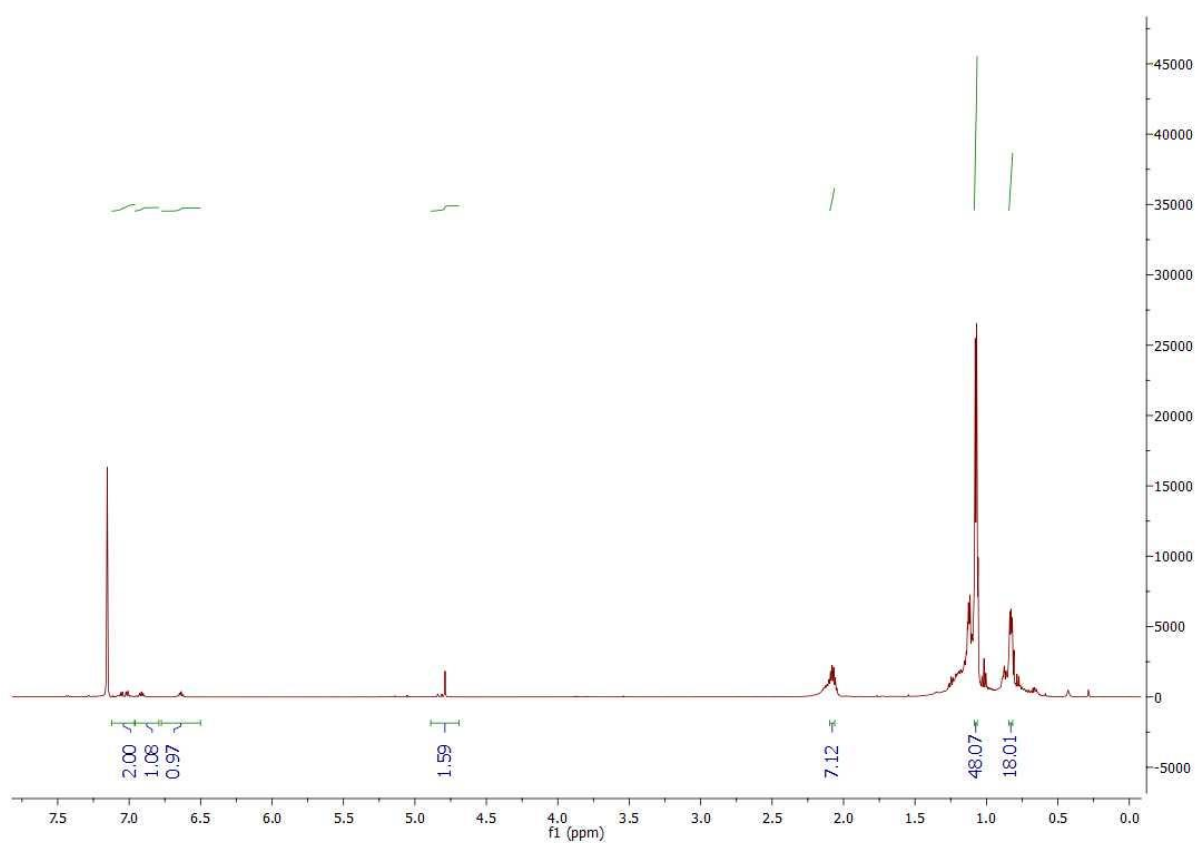
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.81-0.86 (m, 18H), 1.05-1.10 (m, 48H), 2.03-2.10 (m, 7H), 4.79 (s, 2H), 6.62-6.65 (m, 1H), 6.90-6.94 (m, 1H), 6.99-7.09 (m, 2H).

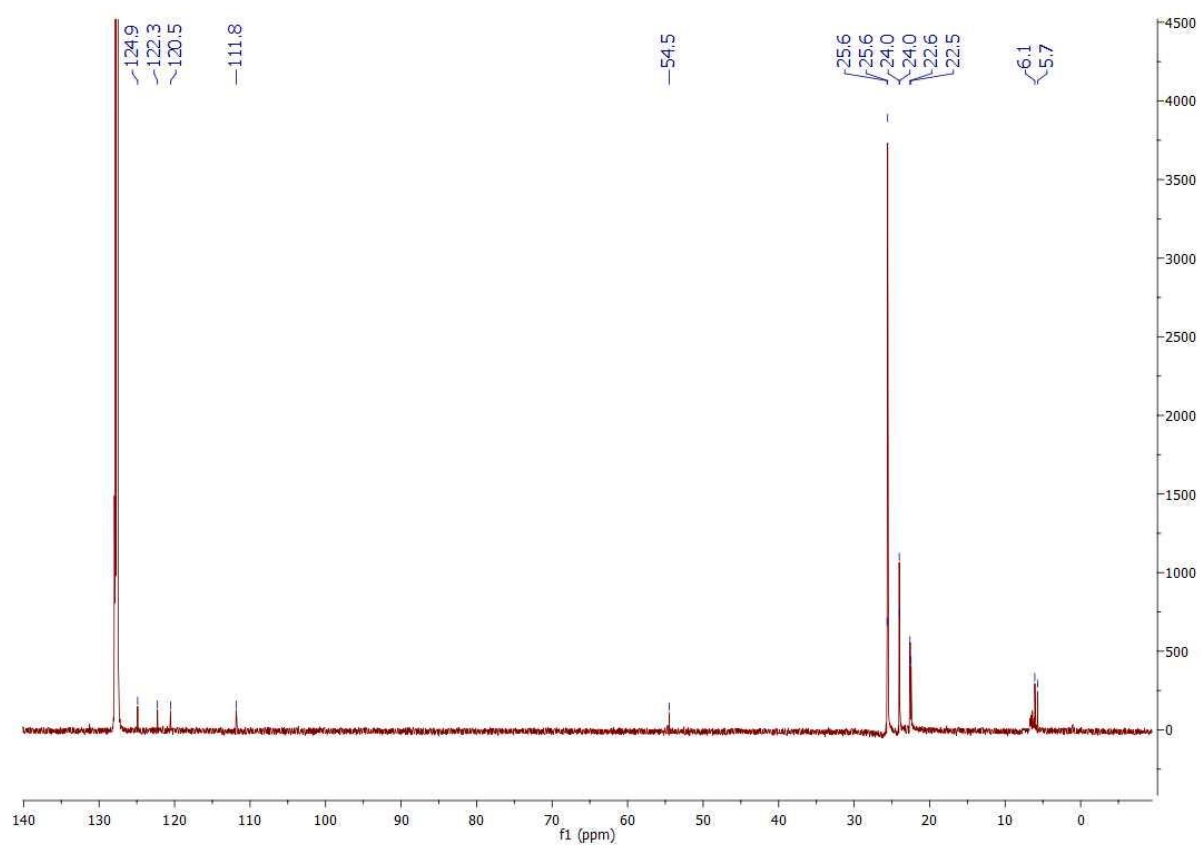
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 5.7, 6.1, 22.5, 22.6, 24.0, 24.0, 25.6, 25.6, 54.5, 111.8, 120.5, 122.3, 124.9.

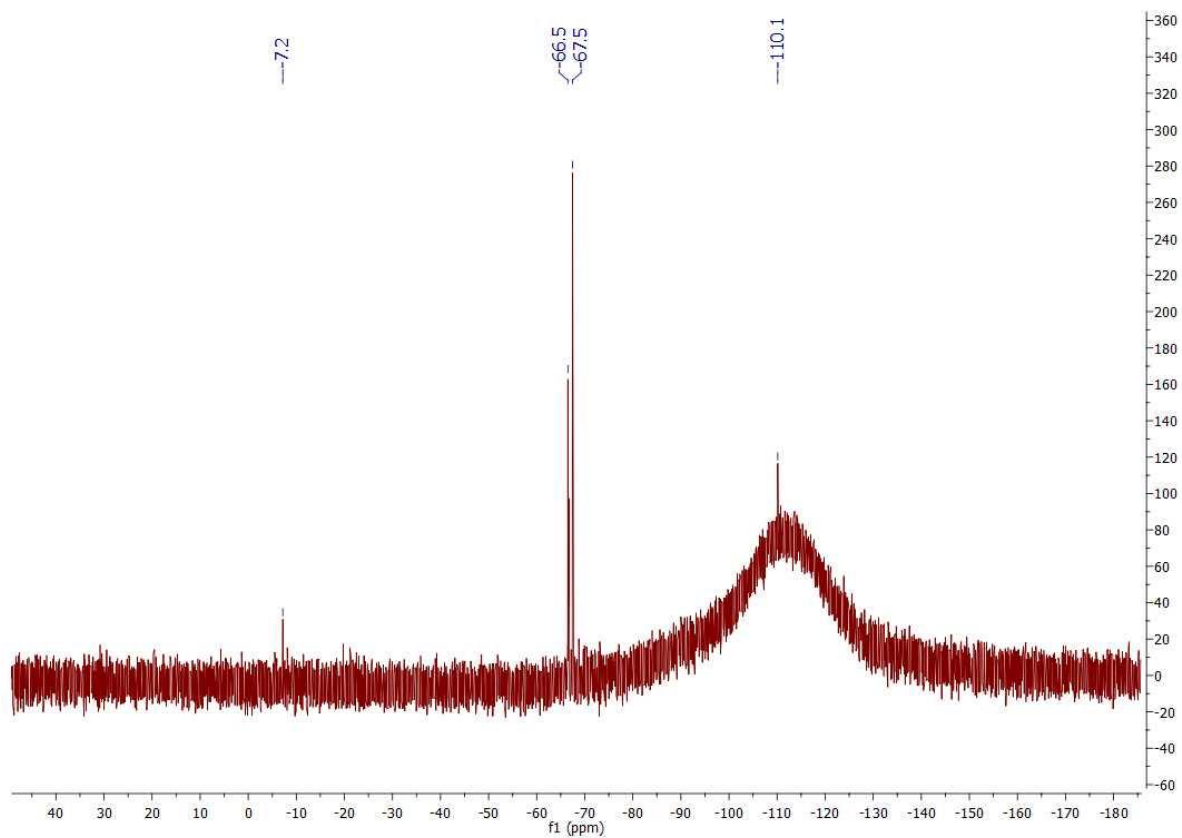
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -7.2, -66.5, -67.5, -110.1.

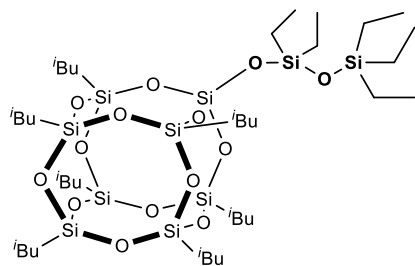
EA: C₃₉H₇₉NO₁₃SSi₉ (1053.320): calcd. C 44.41, H 7.55; found C 44.29, H 7.67.

HRMS: calcd. for C₃₉H₇₉NO₁₃SSi₉Na 1076.31; found 1076.31.









Compound (**22**) was obtained as oil in 86% yield.

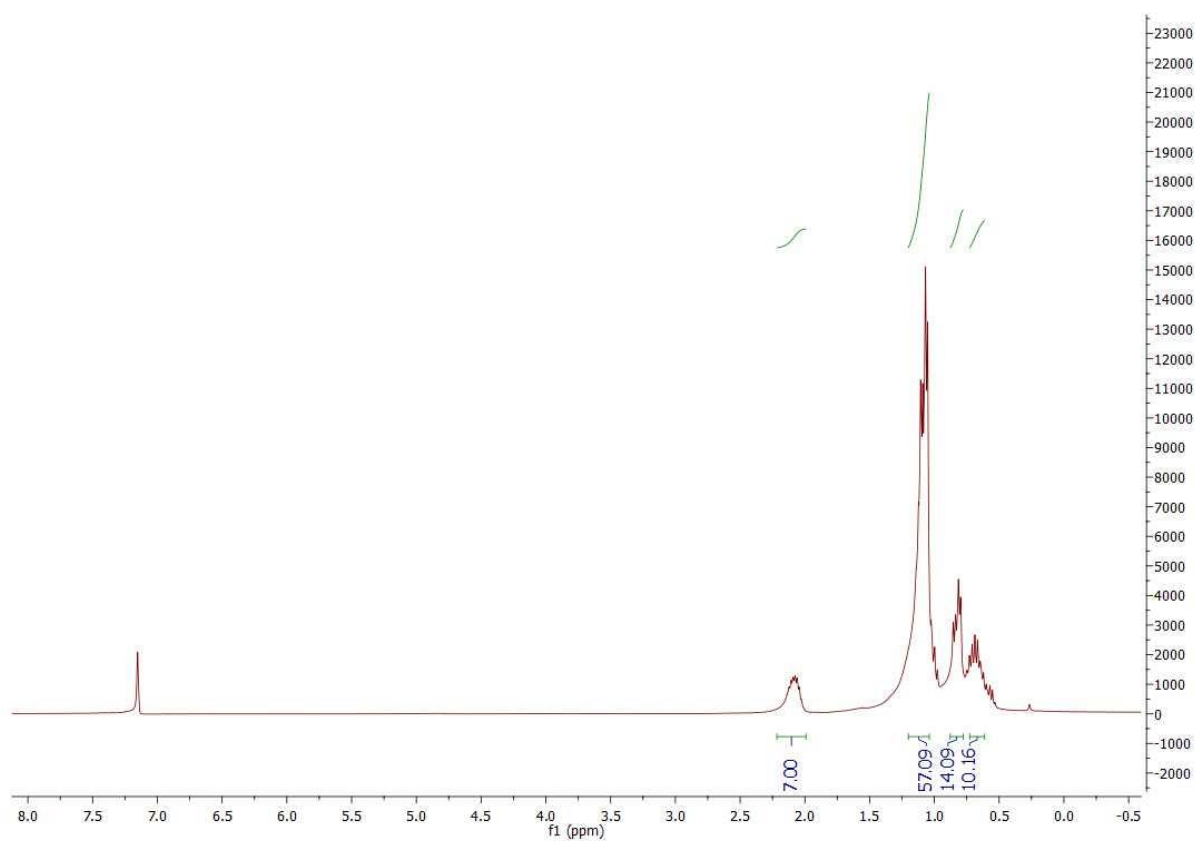
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.62-0.72 (m, 10H), 0.77-0.87 (m, 14H), 1.04-1.20 (m, 57H), 1.99-2.20 (m, 7H).

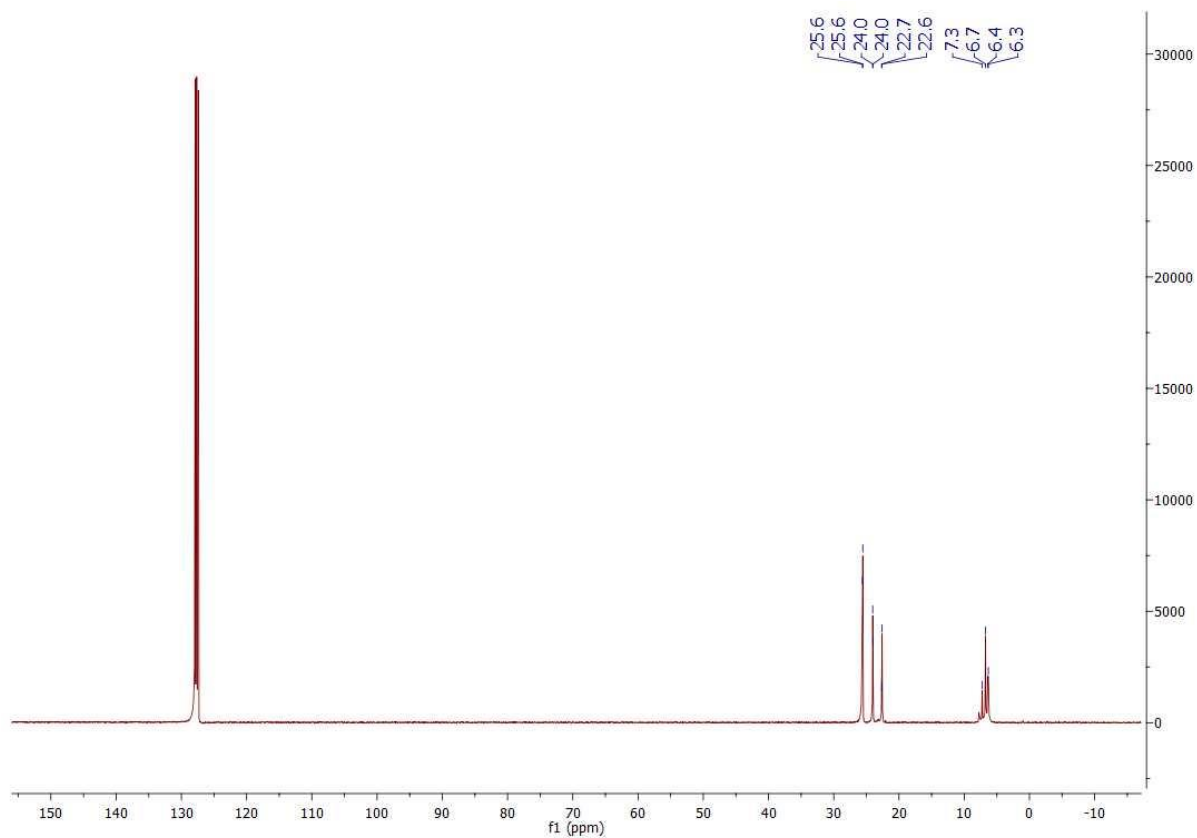
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 6.3, 6.4, 6.7, 7.3, 22.6, 22.7, 24.0, 24.0, 25.6, 25.6.

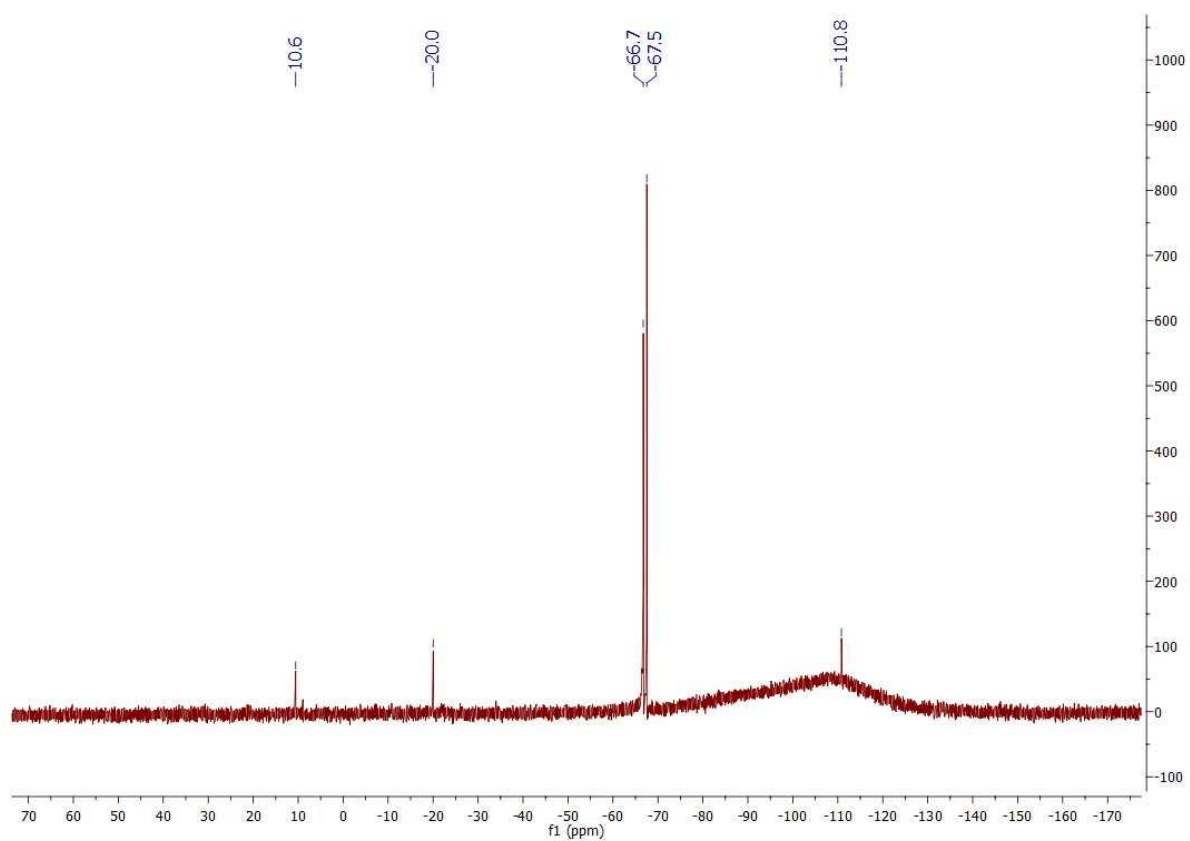
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 10.6, -20.0, -66.7, -67.5, -110.8.

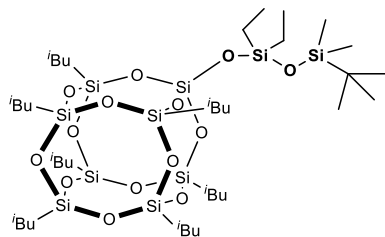
EA: C₃₈H₈₈O₁₄Si₁₀ (1048.387): calcd. C 43.47, H 8.45; found C 43.61, H 8.58.

HRMS: calcd. for C₃₈H₈₈O₁₄Si₁₀Na 1071.38; found 1071.38.









Compound (**23**) was obtained as oil in 90% yield.

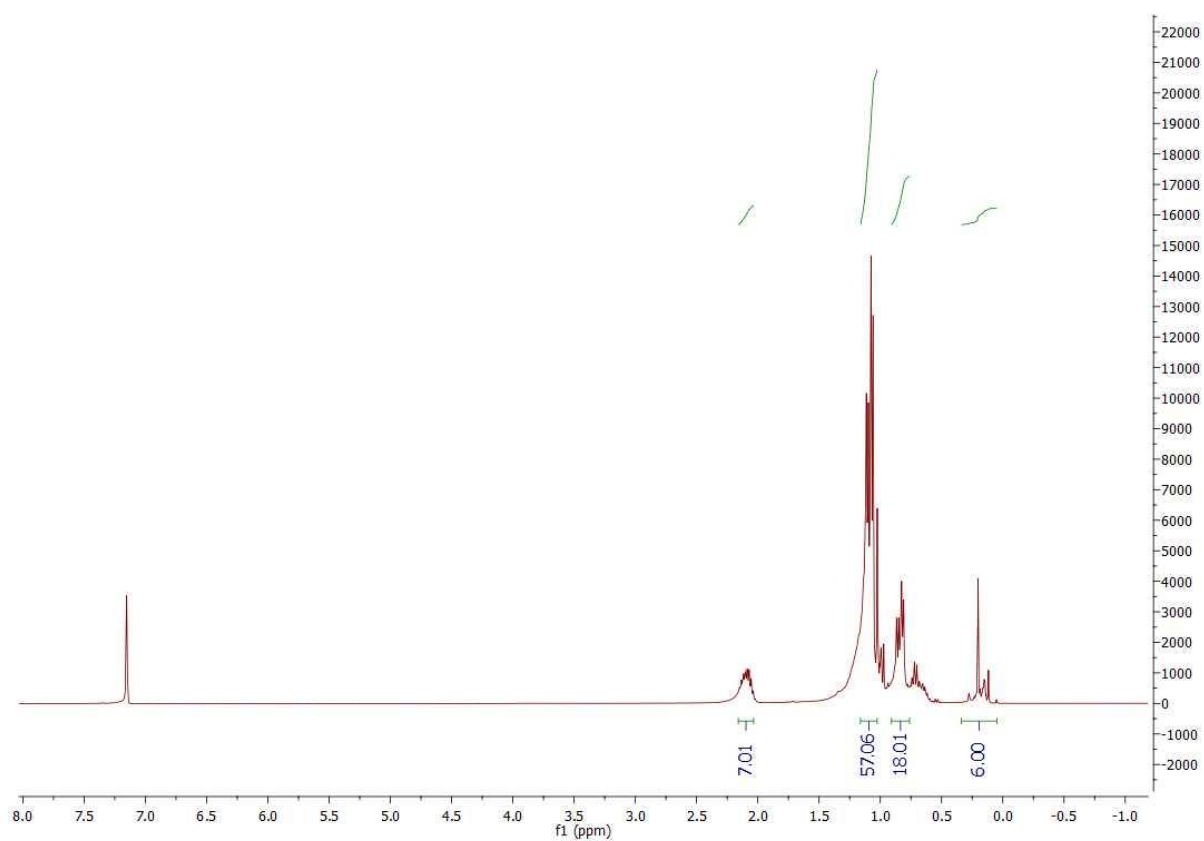
¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.05-0.25 (m, 6H), 0.76-0.90 (m, 18H), 1.02-1.16 (m, 57H), 2.04-2.16 (m, 7H).

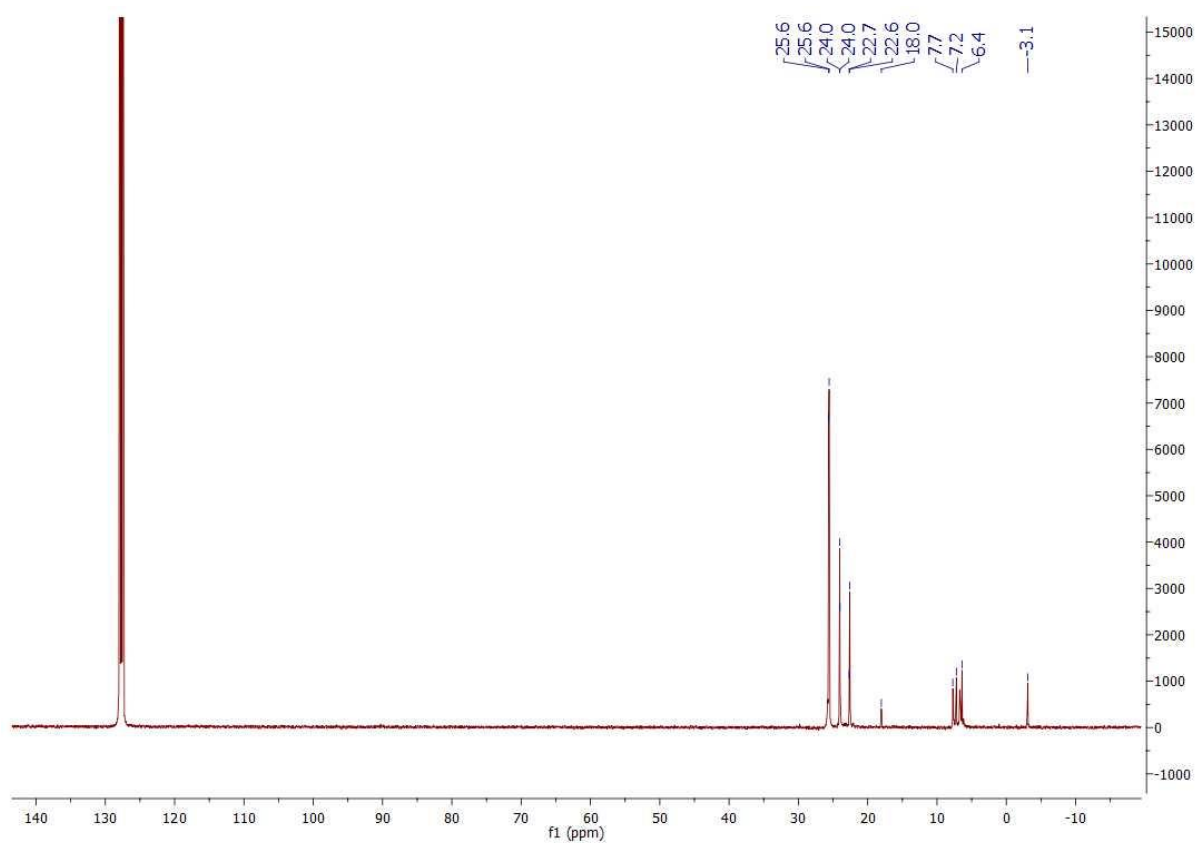
¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 3.1, 6.4, 7.2, 7.7, 18.0, 22.6, 22.7, 24.0, 24.0, 25.6, 25.6.

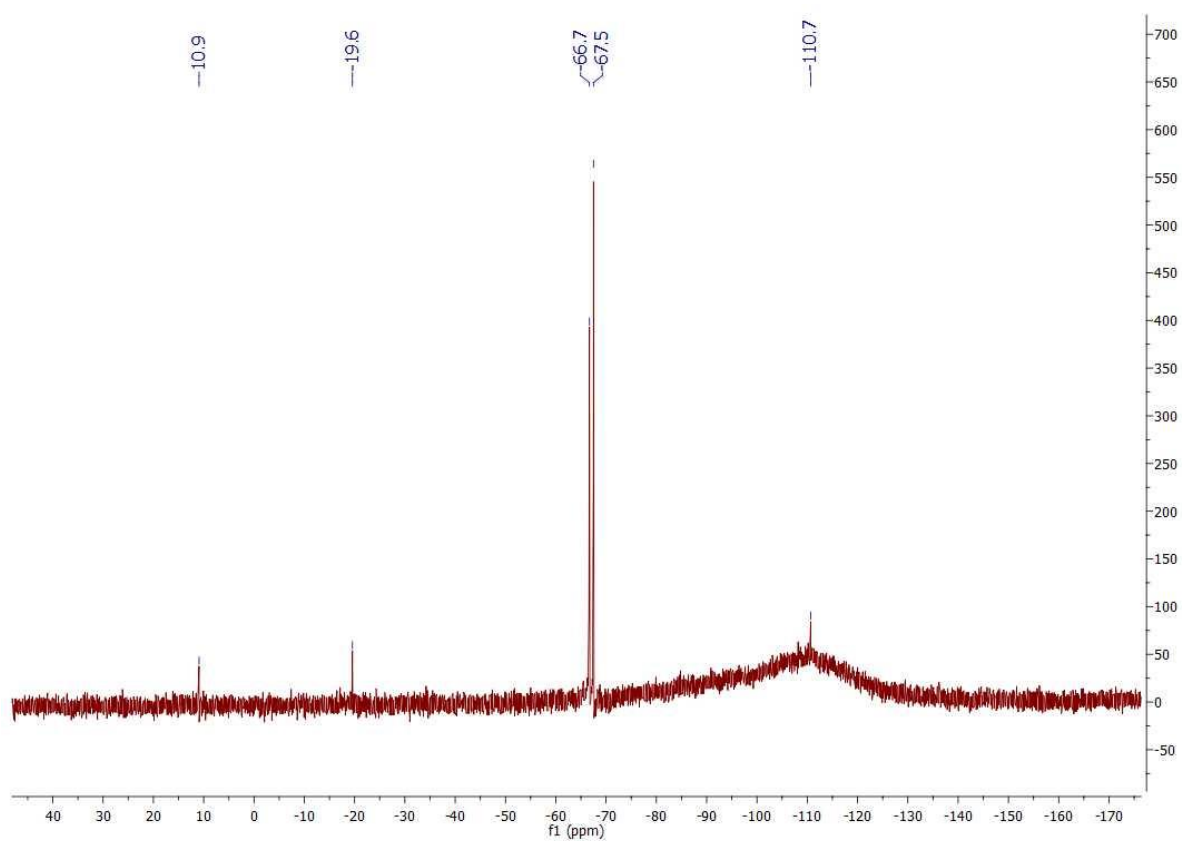
²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = 10.9, -19.6, -66.7, -67.5, -100.7.

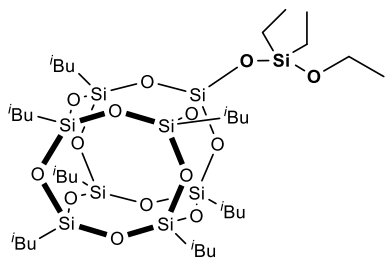
EA: C₃₈H₈₈O₁₄Si₁₀ (1048.387): calcd. C 43.47, H 8.45; found C 43.69, H 8.64.

HRMS: calcd. for C₃₈H₈₈O₁₄Si₁₀Na 1071.38; found 1071.38.









Compound (**24**) was obtained as oil in 91% yield.

¹H NMR (400 MHz, C₆D₆) δ (ppm) = 0.78-0.87 (m, 18H), 1.05-1.13 (m, 51H), 2.02-2.14 (m, 7H).

¹³C NMR (101 MHz, C₆D₆) δ (ppm) = 6.8, 7.2, 7.7, 22.6, 22.7, 24.0, 24.0, 25.6, 25.6, 58.4.

²⁹Si NMR (79 MHz, C₆D₆) δ (ppm) = -66.7, -67.5, -110.8.

EA: C₃₄H₇₈O₁₄Si₉ (962.331): calcd. C 42.37, H 8.16; found C 42.52, H 8.04.

HRMS: calcd. for C₃₄H₇₈O₁₄Si₉Na 985.32; found 985.32.

