Electronic Supplementary Material (ESI) for Inorganic Chemistry Frontiers.

Supporting Information File for:

Silylation of Silanols with Hydrosilanes via Main-Group Catalysis: The Synthesis of Unsymmetrical Siloxanes and Hydrosiloxanes

Krzysztof Kuciński,*[a] Hanna Stachowiak, [a] Grzegorz Hreczycho*[a]

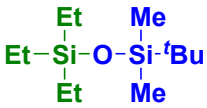
[a]*Faculty of Chemistry, Adam Mickiewicz University in Poznań, Ul. Uniwersytetu Poznańskiego 8, 61-614 Poznań (Poland)*

***kucinski.k@amu.edu.pl**

Table Of Contents

Characterization data

1-*Tert*-butyl-3,3,3-triethyl-1,1-dimethyldisiloxane (**3a**) was obtained as colorless oil in 91% yield.



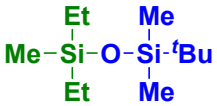
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 0.97 (t, *J* = 7.9 Hz, 9H, *SiCH$_2$***CH$_3$**), 0.90 (s, 9H, *Si(CH$_3$)$_2$C*(**CH$_3$**)$_3$), 0.55 (q, *J* = 8.0 Hz, 6H, *Si***CH$_2$***CH$_3$*), 0.05 (s, 6H, *Si*(**CH$_3$**)$_2$*C(CH$_3$)$_3$*).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 25.7, 18.2, 6.8, 6.3, -2.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 9.5, 9.3.

MS (EI) m/z (rel. int.): 247 (16%, M^+), 232 (5, M^+-CH_3), 217 (9, M^+-C_2H_5), 205 (4), 189 (10, M^+-C_4H_9), 177 (100), 161 (33), 149 (11), 133 (9), 119 (5), 105 (9).

1-*Tert*-butyl-3,3-diethyl-1,1,3-trimethyldisiloxane (**3b**) was obtained as colorless oil in 90% yield.



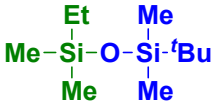
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 0.95 (t, *J* = 7.9 Hz, 6H, *SiCH$_2$***CH$_3$**), 0.89 (s, 9H, *Si(CH$_3$)$_2$C*(**CH$_3$**)$_3$), 0.53 (q, *J* = 7.7 Hz, 4H, *Si***CH$_2$***CH$_3$*), 0.04 (s, 9H, *Si*(**CH$_3$**)$_2$*C(CH$_3$)$_3$ and Si(C$_2$H$_5$)$_2$***CH$_3$**).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 25.7, 18.1, 8.2, 6.8, -2.5, -2.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 9.6, 9.1.

MS (EI) m/z (rel. int.): 232 (10%, M^+), 217 (10, M^+-CH_3), 203 (10, M^+-C_2H_5), 175 (50, M^+-C_4H_9), 159 (100, M^+-C_4H_9 and -CH_3).

1-*Tert*-butyl-3-ethyl-1,1,3,3-tetramethyldisiloxane (**3c**) was obtained as colorless oil in 90% yield.



^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 0.95 (t, *J* = 7.9 Hz, 3H, *SiCH$_2$***CH$_3$**), 0.88 (s, 9H, *Si(CH$_3$)$_2$C*(**CH$_3$**)$_3$), 0.52 (q, *J* = 7.9 Hz, 2H, *Si***CH$_2$***CH$_3$*), 0.06 (s, 6H, *SiC$_2$H$_5$*(**CH$_3$**)$_2$), 0.03 (s, 6H, *Si*(**CH$_3$**)$_2$*C(CH$_3$)$_3$*).

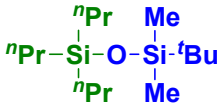
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 25.7, 18.0, 10.1, 6.8, -0.3, -2.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 9.7, 8.1.

MS (EI) m/z (rel. int.): 219 (5%, M^+), 203 (10, M^+-CH_3), 189 (10, M^+-C_2H_5), 161 (100, M^+-C_4H_9), 146 (25), 131 (10).

EA: $C_{10}H_{26}OSi_2$ (218.15)**:** calcd. C 54.97, H 12.00; found C 55.05, H 12.10

1-*Tert*-butyl-1,1-dimethyl-3,3,3-tripropyldisiloxane (**3d**) was obtained as colorless oil in 84% yield.



^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 1.41 – 1.32 (m, 6H, *SiCH$_2$***CH$_2$***CH$_3$*), 0.98 (t, *J* = 7.2 Hz, 9H, *Si(CH$_2$)$_2$***CH$_3$**), 0.88 (s, 9H, *Si(CH$_3$)$_2$C***(CH$_3$)$_3$**), 0.59 – 0.51 (m, 6H, *Si***CH$_2$***CH$_2$CH$_3$*), 0.03 (s, 6H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*).

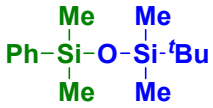
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 25.8, 18.5, 18.5, 18.2, 16.8, -2.8.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 9.2, 6.1.

MS (EI) m/z (rel. int.): 205 (100%), 161 (50), 147 (70, M^+-Si(^{n}Pr)$_3$), 105 (60).

EA: $C_{15}H_{36}OSi_2$ (288.23)**:** calcd. C 62.42, H 12.57; found C 62.38, H 12.50

1-*Tert*-butyl-1,1,3,3-tetramethyl-3-phenyldisiloxane (**3e**) was obtained as colorless oil in 89% yield.



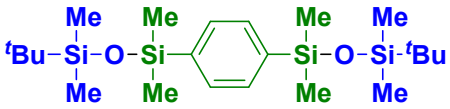
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.70 – 7.59 (m, 2H, *Si(CH$_2$)$_2$***Ph**), 7.51 – 7.36 (m, 3H, *Si(CH$_2$)$_2$***Ph**), 0.94 (s, 9H, *Si(CH$_3$)$_2$C***(CH$_3$)$_3$**), 0.38 (s, 6H, *Si***(CH$_2$)$_2$***Ph*), 0.09 (s, 6H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 140.2, 133.0, 129.2, 127.7, 25.7, 18.1, 1.0, -2.8.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 11.5, -2.8.

MS (EI) m/z (rel. int.): 267 (5%, M^+), 251 (90, M^+-CH_3), 209 (100, M^+-C_4H_9), 193 (27), 147 (20), 135 (8, M^+-Si$(CH_3)_2$Ph).

1,4-Bis(3-(*tert*-butyl)-1,1,3,3-tetramethyldisiloxanyl)benzene (**3f**) was obtained as colorless oil in 93% yield.



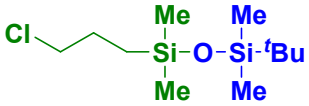
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.59 (s, 4H, *Si(CH$_2$)$_2$***Ph**), 0.93 (s, 18H, *Si(CH$_3$)$_2$C***(CH$_3$)$_3$**), 0.37 (s, 12H, *Si***(CH$_2$)$_2$***Ph*), 0.08 (s, 12H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 141.1, 132.2, 25.7, 18.1, 1.0, -2.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 11.5, -2.9.

MS (EI) m/z (rel. int.): 439 (32%, M^+-CH_3), 397 (100, M^+-C_4H_9), 384 (18), 355 (10), 342 (14), 310 (21), 267 (20), 251 (14), 236 (14), 221 (10), 147 (13).

1-*Tert*-butyl-3-(3-chloropropyl)-1,1,3,3-tetramethyldisiloxane (**3g**) was obtained as colorless oil in 91% yield.



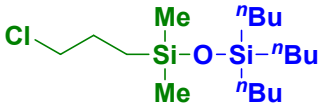
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 3.53 (t, *J* = 7.0 Hz, 2H, *Si(CH$_3$)$_2$CH$_2$CH$_2$***CH$_2$***Cl*), 1.98 – 1.67 (m, 2H, *Si(CH$_3$)$_2$CH$_2$***CH$_2$***CH$_2$Cl*), 0.88 (s, 9H, *Si(CH$_3$)$_2$C***(CH$_3$)$_3$**), 0.72 – 0.46 (m, 2H, *Si(CH$_3$)$_2$***CH$_2$***CH$_2$CH$_2$Cl*), 0.10 (s, 6H, *Si***(CH$_3$)$_2$***CH$_2$CH$_2$CH$_2$Cl*), 0.04 (s, 6H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 47.9, 27.1, 25.7, 18.0, 16.0, 0.3, -2.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 10.6, 6.6.

MS (EI) m/z (rel. int.): 189 (50%), 167 (100), 147 (60, M^+-C_4H_9 and CH_3 (4x)).

1,1,1-Tributyl-3-(3-chloropropyl)-3,3-dimethyldisiloxane (**3h**) was obtained as colorless oil in 89% yield.



^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 3.53 (t, *J* = 7.0 Hz, 2H, *Si(CH$_3$)$_2$CH$_2$CH$_2$***CH$_2$***Cl*), 2.10 – 1.72 (m, 2H, *Si(CH$_3$)$_2$CH$_2$***CH$_2$***CH$_2$Cl*), 1.41 – 1.22 (m, 12H, *SiCH$_2$***CH$_2$CH$_2$***CH$_3$*), 0.91 (t, *J* = 6.9 Hz, 9H, *SiCH$_2$CH$_2$CH$_2$***CH$_3$**), 0.68 – 0.61 (m, 2H, *Si(CH$_3$)$_2$***CH$_2$***CH$_2$CH$_2$Cl*), 0.60 – 0.43 (m, 6H, *Si***CH$_2$***CH$_2$CH$_2$CH$_3$*), 0.09 (s, 6H *Si***(CH$_3$)$_2$***CH$_2$CH$_2$CH$_2$Cl*).

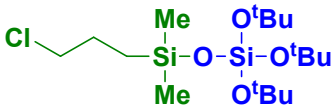
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 47.9, 27.2, 26.6, 25.5, 16.1, 15.3, 13.8, -0.4.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 7.8, 5.9.

MS (EI) m/z (rel. int.): 251 (72, M^+-C_7H_{15}), 233 (100), 217 (38), 195 (16), 179 (12, M^+-(^{n}Bu)$_3$), 161 (12), 155 (14), 139 (27), 121 (6), 105 (7).

EA: $C_{17}H_{39}ClOSi_2$ (288.23)**:** calcd. C 58.15, H 11.20; found C 58.12, H 11.24

1,1,1-*Tert*-butoxy-3-(3-chloropropyl)-3,3-dimethyldisiloxane (**3i**) was obtained as colorless oil in 98% yield.



^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 3.54 (t, *J* = 7.1 Hz, 2H, *Si(CH$_3$)$_2$CH$_2$CH$_2$***CH$_2$***Cl*), 2.00 – 1.82 (m, 2H, *Si(CH$_3$)$_2$CH$_2$***CH$_2$***CH$_2$Cl*), 1.31 (s, 27H, *SiOC***(CH$_3$)$_3$***), 0.91 – 0.51 (m, 2H, *Si(CH$_3$)$_2$***CH$_2$***CH$_2$CH$_2$Cl*), 0.15 (s, 6H, *Si***(CH$_3$)$_2$***CH$_2$CH$_2$CH$_2$Cl*).

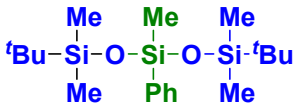
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 72.3, 48.0, 31.3, 27.0, 15.9, -0.1.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 8.0, -99.5.

MS (EI) m/z (rel. int.): 383 (9%, M^+-CH_3), 327 (39, M^+-$OC(CH_3)_3$), 271 (32), 229 (24), 213 (13), 193 (22), 171 (65), 153 (100), 135 (33), 57 (40).

EA: $C_{17}H_{39}ClO_4Si_2$ (398.21)**:** calcd. C 51.16, H 9.85; found C 51.18, H 9.76

1,5-Di-tert-butyl-1,1,3,5,5-pentamethyl-3-phenyltrisiloxane (**5a**) was obtained as colorless oil in 90% yield.



^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.71 – 7.55 (m, 2H, *SiCH$_3$***Ph***), 7.47 – 7.28 (m, 3H, *SiCH$_3$***Ph***), 0.92 (s, 18H, *Si(CH$_3$)$_2$C***(CH$_3$)$_3$***), 0.36 (s, 3H, *Si***CH$_3$***Ph*), 0.10 (s, 6H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*), 0.07 (s, 6H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*).

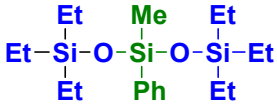
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 138.5, 133.2, 129.4, 127.5, 25.7, 18.1, -0.2, -2.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 11.1, -35.2.

MS (EI) m/z (rel. int.): 367 (20%, M^+-CH_3), 326 (100, M^+-C_4H_9), 306 (4), 284 (72), 268 (8), 252 (8), 222 (10), 135 (6).

EA: $C_{19}H_{38}O_2Si_3$ (382.22)**:** calcd. C 59.62, H 10.01; found C 59.68, H 9.92

1,1,1,5,5,5-Hexaethyl-3-methyl-3-phenyltrisiloxane (**5b**) was obtained as colorless oil in 92% yield.



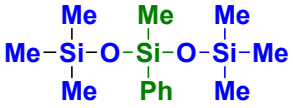
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.72 – 7.53 (m, 2H, *SiCH$_3$***Ph**), 7.43 – 7.34 (m, 3H, *SiCH$_3$***Ph**), 0.96 (t, *J* = 7.9 Hz, 18H, *SiCH$_2$***CH$_3$**), 0.59 (q, *J* = 7.9 Hz, 12H, *Si***CH$_2$***CH$_3$*), 0.35 (s, 3H, *Si***CH$_3$***Ph*).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 138.8, 133.2, 129.3, 127.5, 6.7, 6.2, -0.1.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 10.8, -35.8.

MS (EI) m/z (rel. int.): 367 (5%, M^+-CH_3), 354 (100, M^+-C_2H_5), 324 (4), 306 (2), 295 (2).

1,1,1,3,5,5,5-Heptamethyl-3-phenyltrisiloxane (**5c**) was obtained as colorless oil in 86% yield.



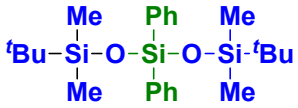
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.69 – 7.56 (m, 2H, *SiCH$_3$***Ph**), 7.46 – 7.36 (m, 3H, *SiCH$_3$***Ph**), 0.32 (s, 3H, *Si***CH$_3$***Ph*), 0.16 (s, 18H, *Si***CH$_3$**).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 138.5, 133.2, 129.4, 127.6, 1.9, 0.0.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 8.5, -34.2.

MS (EI) m/z (rel. int.): 284 (100%, M^+-CH_3), 268 (3, M^+-CH_3 (2x)), 252 (2), 221 (3), 135 (2).

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3,3-diphenyltrisiloxane (**5d**) was obtained as colorless oil in 95% yield.



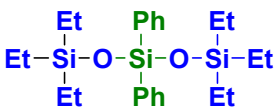
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.68 – 7.55 (m, 4H, *Si***Ph**), 7.51 – 7.33 (m, 6H, *Si***Ph**), 0.90 (s, 18H, *Si(CH$_3$)$_2$C***(CH$_3$)$_3$**), 0.04 (s, 12H, *Si***(CH$_3$)$_2$***C(CH$_3$)$_3$*).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 136.4, 134.4, 129.7, 127.5, 25.8, 18.2, -2.8.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 12.5, -47.4.

MS (EI) m/z (rel. int.): 430 (6%, M^+-CH_3), 388 (100, M^+-C_4H_9), 368 (2), 346 (34), 310 (19), 284 (10), 268 (13), 252 (5), 135 (5).

1,1,1,5,5,5-Hexaethyl-3,3-diphenyltrisiloxane (**5e**) was obtained as colorless oil in 94% yield.



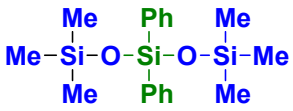
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.73 – 7.58 (m, 4H, *Si***Ph**), 7.51 – 7.30 (m, 6H, *Si***Ph**), 0.93 (t, *J* = 7.9 Hz, 18H, *SiCH*$_2$**CH**$_3$), 0.59 (q, *J* = 7.8 Hz, 12H, *Si***CH**$_2$*CH*$_3$).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 136.7, 134.3, 129.6, 127.5, 6.7, 6.3.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 12.2, -48.4.

MS (EI) m/z (rel. int.): 416 (100%, M^+-C_2H_5), 386 (5), 367 (5), 357 (5), 299 (5), 107 (5).

1,1,1,5,5,5-Hexamethyl-3,3-diphenyltrisiloxane (**5f**) was obtained as colorless oil in 87% yield.



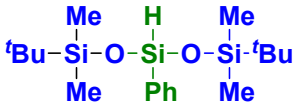
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.67 – 7.56 (m, 4H, *Si***Ph**), 7.48 – 7.28 (m, 6H, *Si***Ph**), 0.16 (s, 18H, *Si***CH**$_3$).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 136.7, 134.3, 129.7, 127.6, 1.9.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 9.7, -47.5.

MS (EI) m/z (rel. int.): 346 (100%, M^+-CH_3), 283 (8), 268 (13).

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxane (**6a**) was obtained as colorless oil in 86% yield.



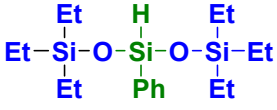
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.63 – 7.55 (m, 2H, *Si***Ph**), 7.46 – 7.37 (m, 3H, *Si***Ph**), 5.06 (s, 1H, *Si***H**), 0.92 (s, 18H, *Si(CH*$_3$*)*$_2$*C***(CH**$_3$**)**$_3$*)*), 0.10 (d, *J* = 6.1 Hz, 12H, *Si***(CH**$_3$**)**$_2$*C(CH*$_3$*)*$_3$).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 137.2, 133.1, 130.0, 127.7, 25.7, 18.2, -3.1.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 13.2, -49.4.

MS (EI) m/z (rel. int.): 368 (5%, M^+), 354 (9, M^+-CH_3), 312 (100), 296 (6), 284 (5), 267 (10), 252 (5), 204 (5), 135 (5).

1,1,1,5,5,5-Hexaethyl-3-phenyltrisiloxane (**6b**) was obtained as colorless oil in 96% yield.



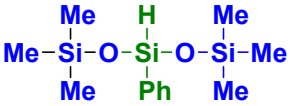
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.65 – 7.58 (m, 2H, *Si***Ph**), 7.46 – 7.36 (m, 3H, *Si***Ph**), 5.08 (s, 1H, *Si***H**), 0.97 (t, *J* = 7.9 Hz, 18H, *SiCH*$_2$**CH$_3$**), 0.61 (q, *J* = 7.8 Hz, 12H, *Si***CH$_2$***CH*$_3$).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 137.4, 133.0, 129.9, 127.7, 6.7, 6.1.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 13.0, -49.7.

MS (EI) m/z (rel. int.): 368 (7%, M^+), 340 (100, M^+-C_2H_5), 309 (5), 281 (5), 252 (5).

1,1,1,5,5,5-Hexamethyl-3-phenyltrisiloxane (**6c**) was obtained as colorless oil in 85% yield.



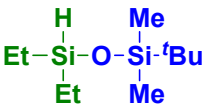
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.74 – 7.56 (m, 2H, *Si***Ph**), 7.48 – 7.32 (m, 3H, *Si***Ph**), 5.01 (s, 1H, *Si***H**), 0.18 (s, 18H, *Si***CH$_3$**).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 137.0, 133.1, 130.0, 127.8, 1.7.

^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 10.7, -49.4.

MS (EI) m/z (rel. int.): 283 (17%, M^+-CH_3), 269 (100), 253 (5), 207 (16), 191 (8), 135 (17), 75 (7), 59 (4), 45 (7).

1-*Tert*-butyl-3,3-diethyl-1,1-dimethyldisiloxane (**6d**) was obtained as colorless oil in 85% yield.



^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 4.51 (s, 1H, *Si***H**), 0.99 (t, *J* = 7.9 Hz, 6H, *SiCH*$_2$**CH$_3$**), 0.90 (s, 9H, *Si(CH*$_3$*)*$_2$*C***(CH$_3$)$_3$**), 0.62 (qd, *J* = 7.9, 2.2 Hz, 4H, *Si***CH$_2$***CH*$_3$), 0.06 (s, 6H, *Si***(CH$_3$)$_2$***C(CH*$_3$*)*$_3$).

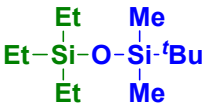
^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm) = 25.7, 18.2, 7.1, 6.6, -3.3.

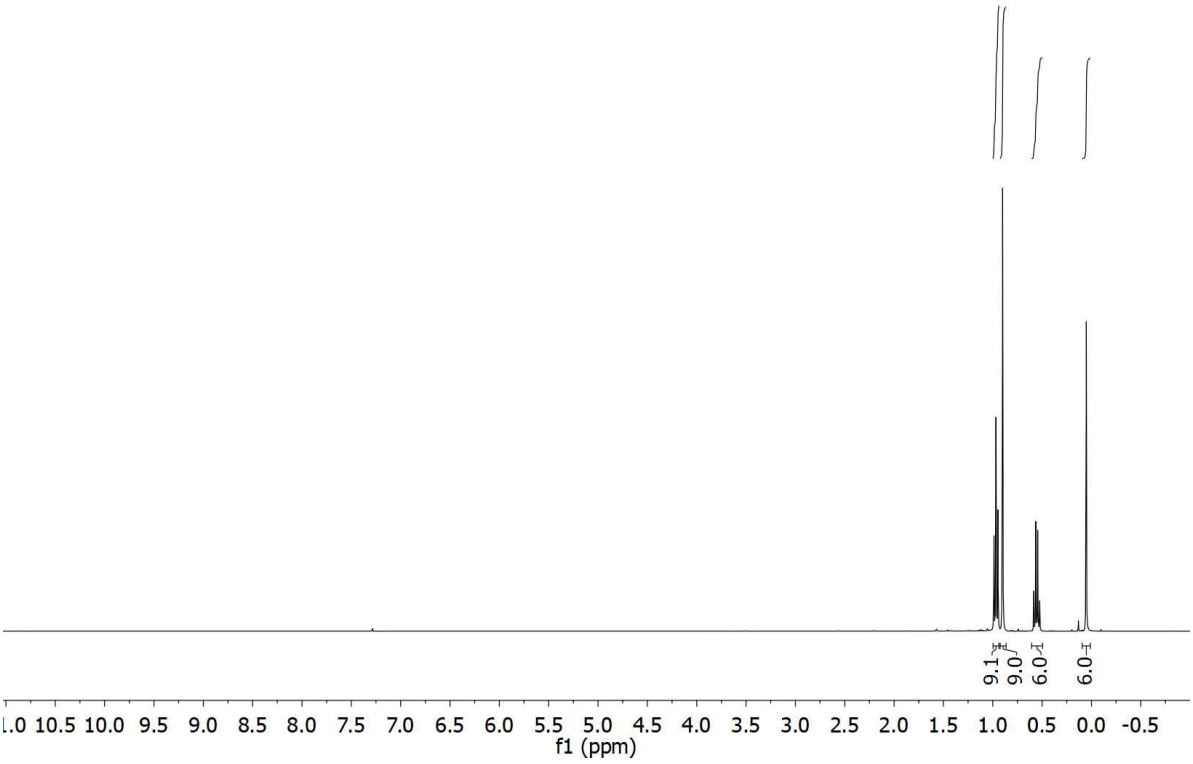
^{29}Si NMR (79 MHz, $CDCl_3$) δ (ppm) = 11.8, -0.7.

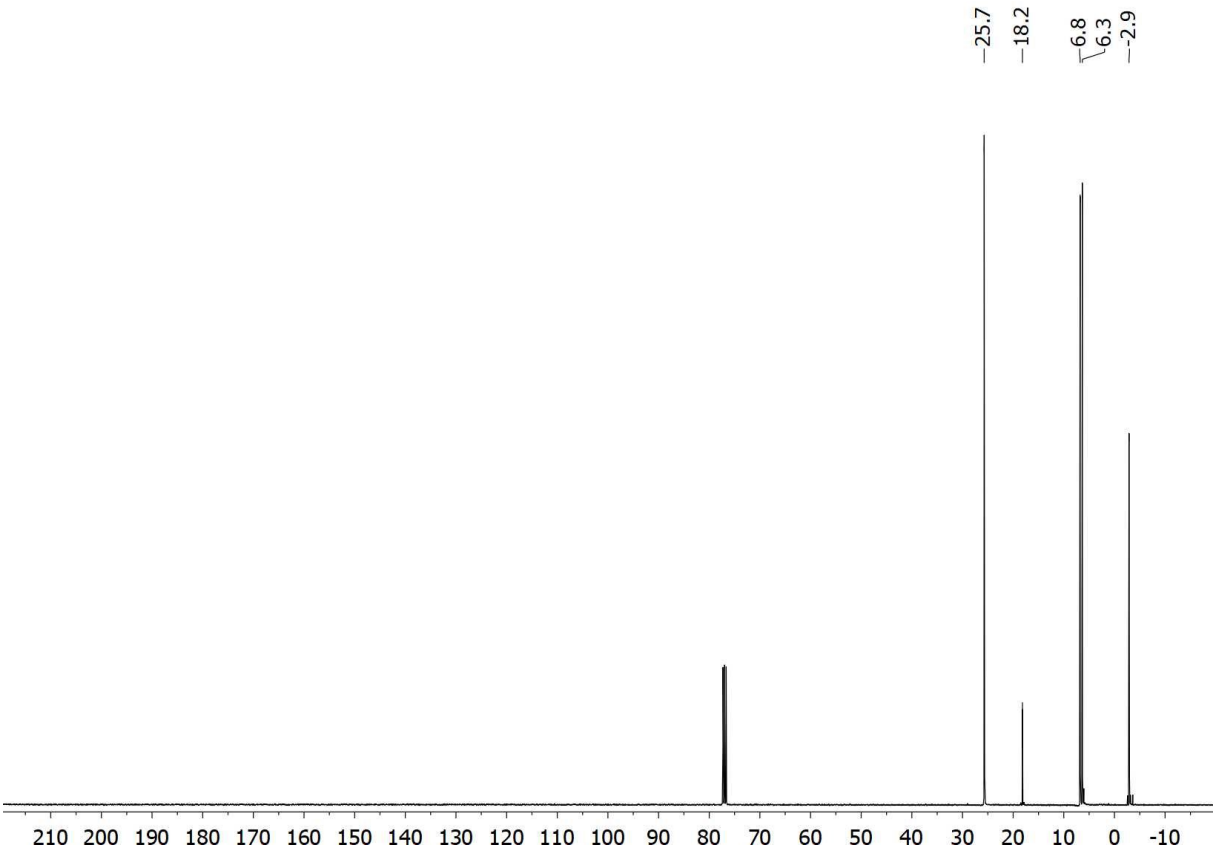
MS (EI) m/z (rel. int.): 219 (27%, M^+), 204 (11, M^+-CH_3), 191 (18), 175 (15), 163 (100), 147 (25), 133 (12), 118 (5), 105 (31).

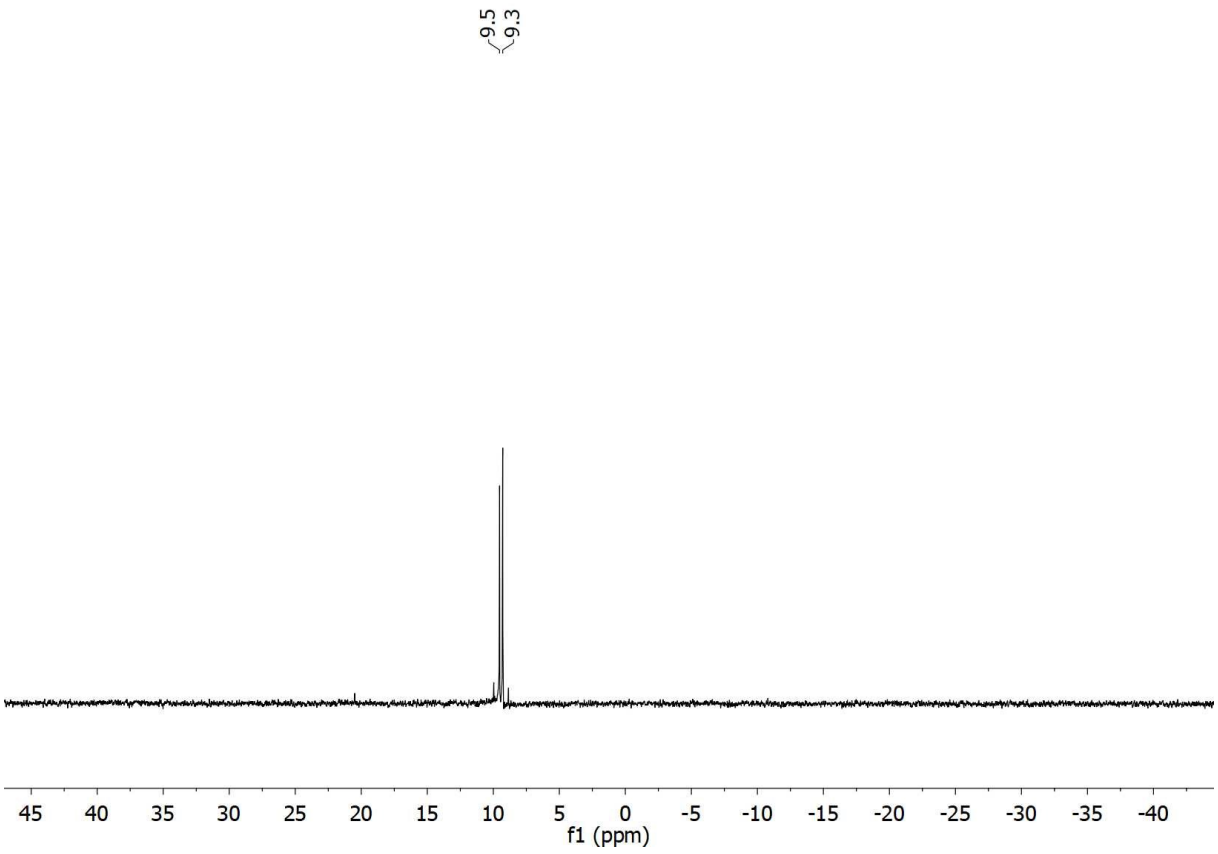
^{1}H, ^{13}C and ^{29}Si NMR spectra

1-*Tert*-butyl-3,3,3-triethyl-1,1-dimethyldisiloxane (**3a**)

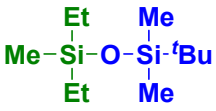


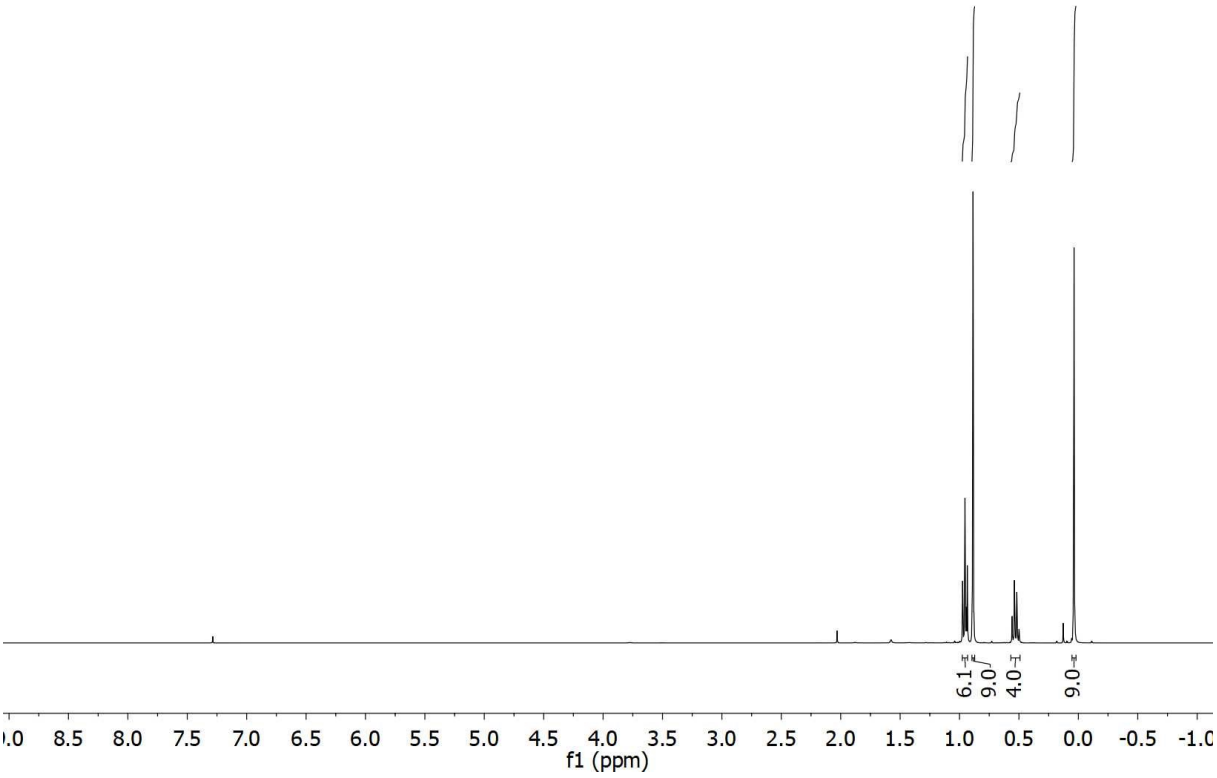


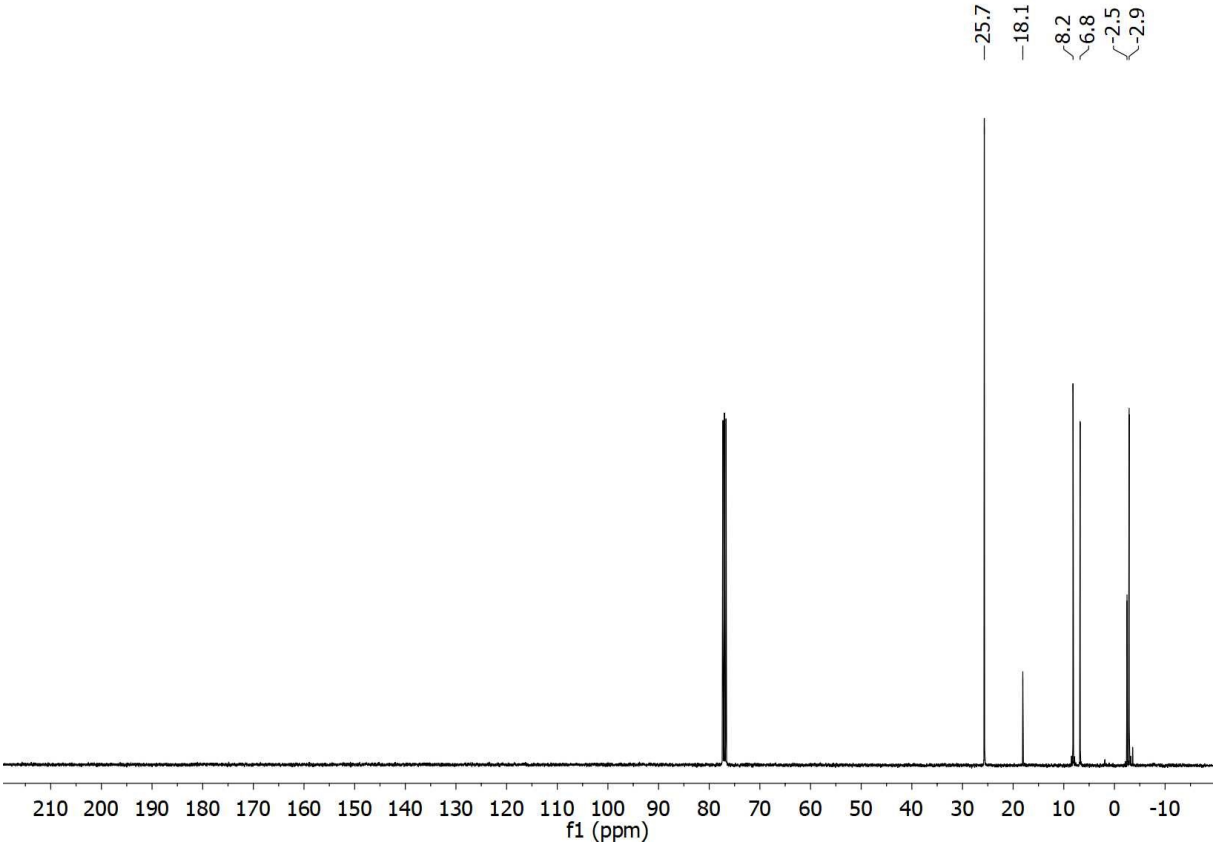


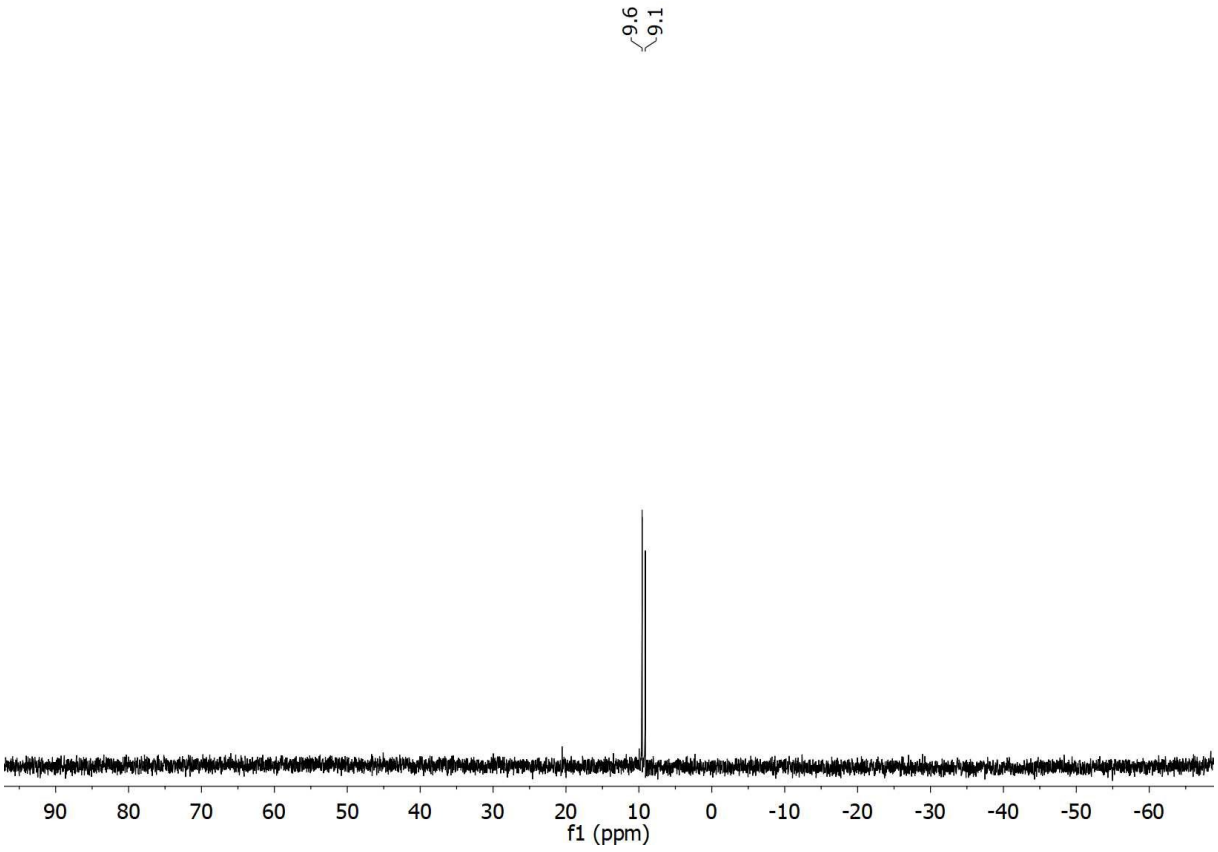

9.5
9.3
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

1-*Tert*-butyl-3,3-diethyl-1,1,3-trimethyldisiloxane (**3b**)

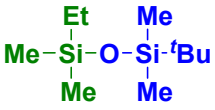


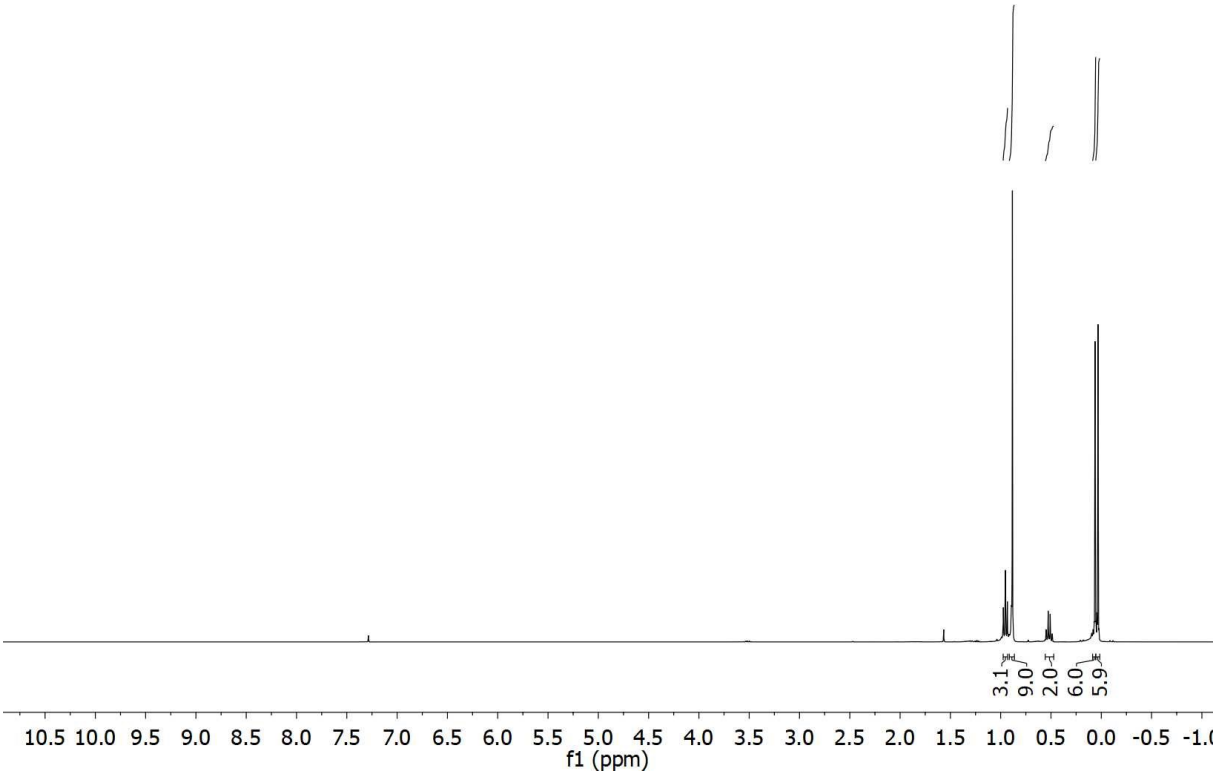


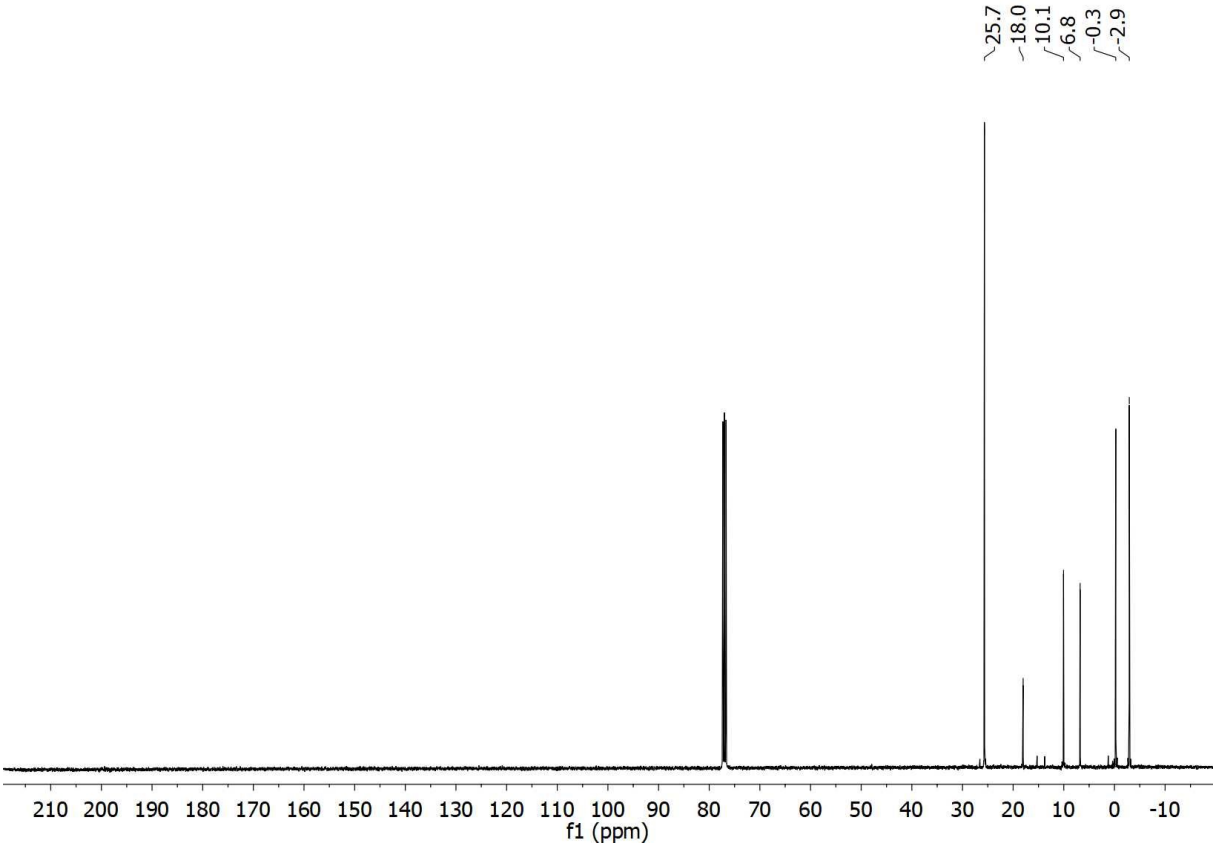


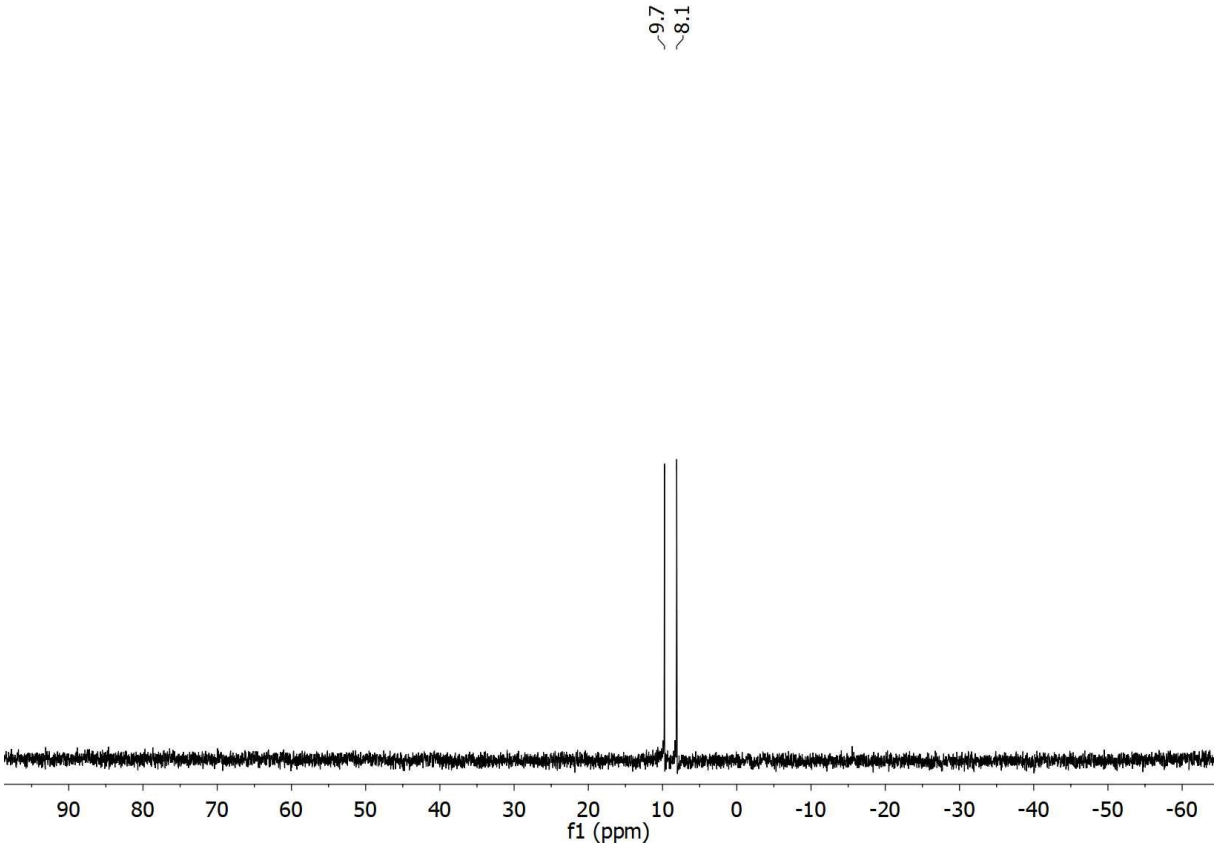

9.6
9.1
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
f1 (ppm)

1-*Tert*-butyl-3-ethyl-1,1,3,3-tetramethyldisiloxane (**3c**).

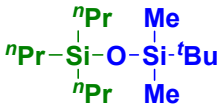


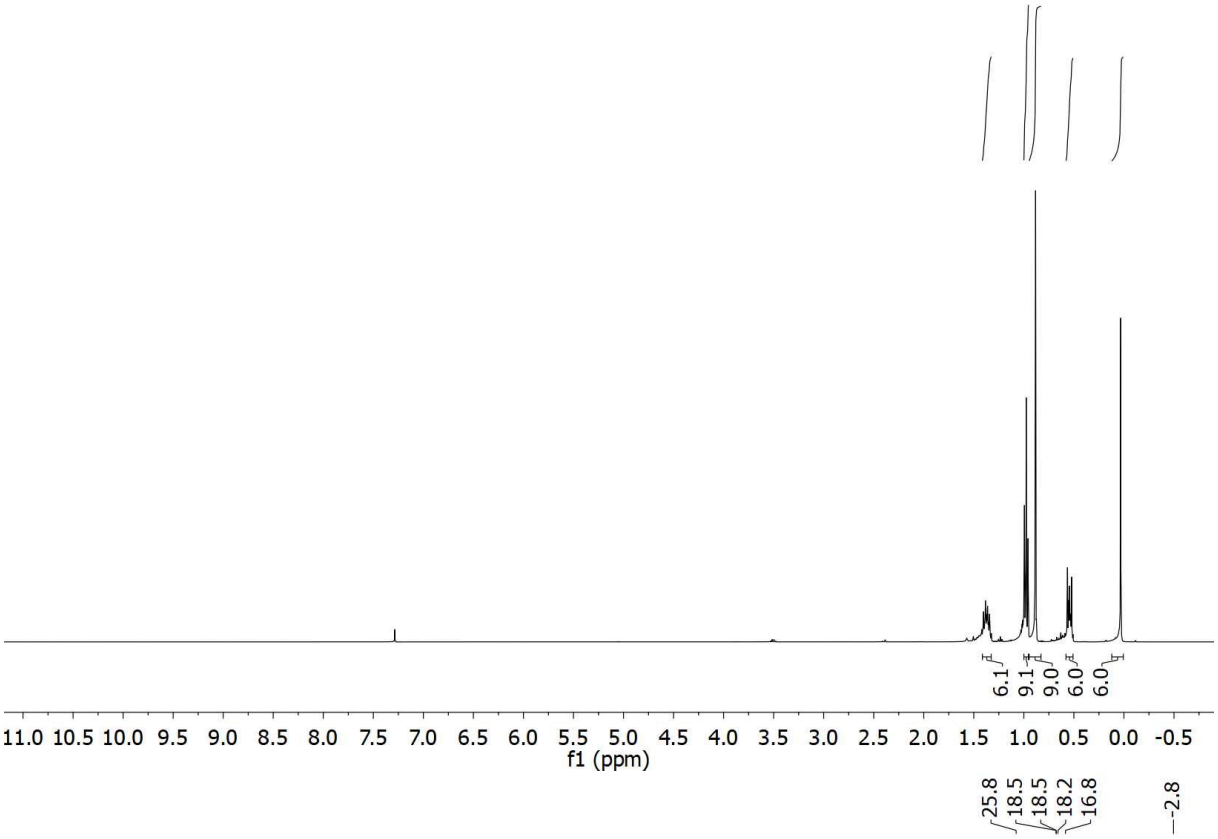


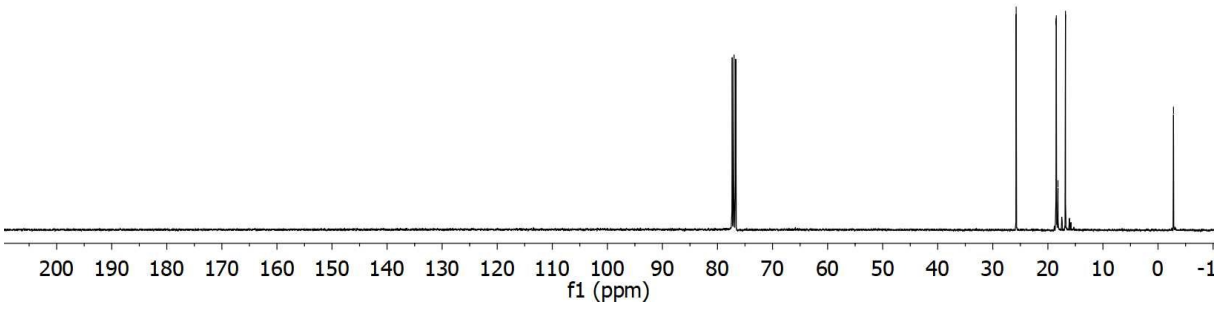


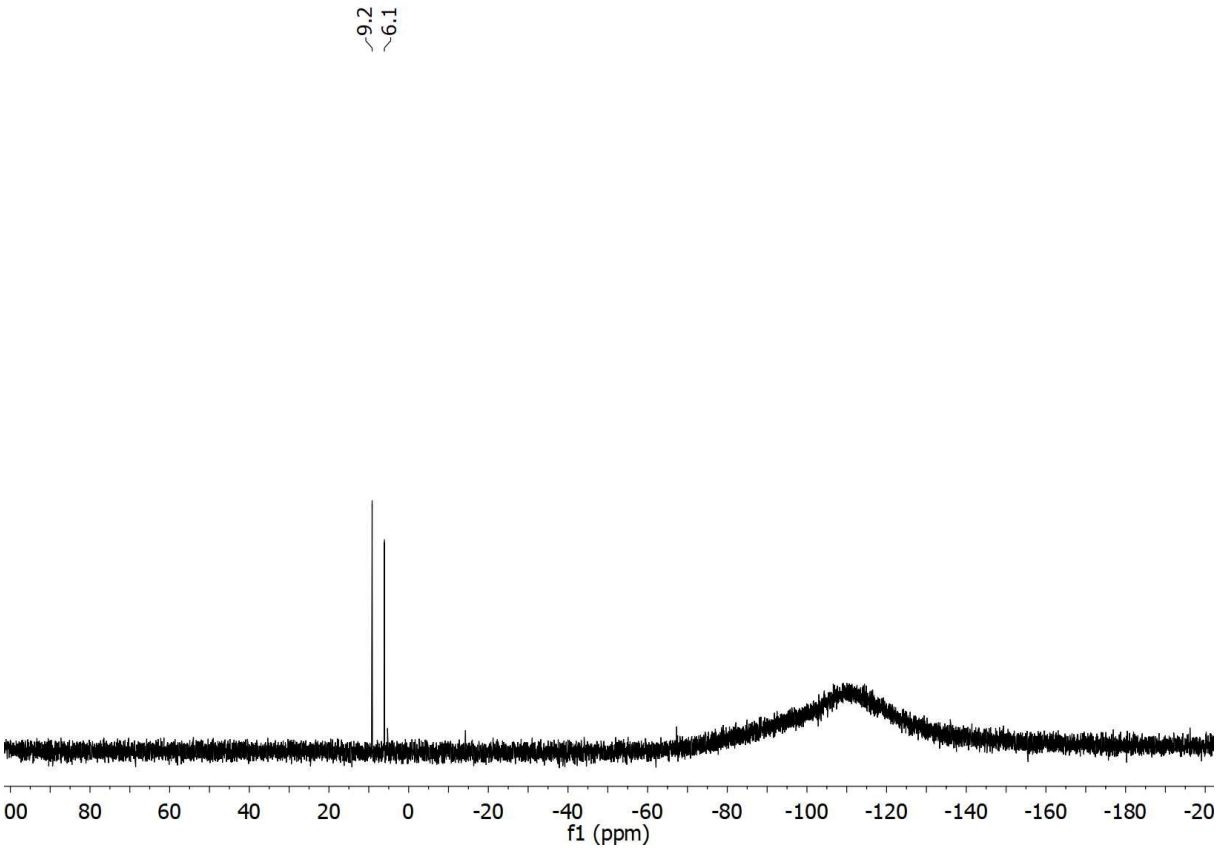

9.7
8.1
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
f1 (ppm)

1-*Tert*-butyl-1,1-dimethyl-3,3,3-tripropyldisiloxane (**3d**)

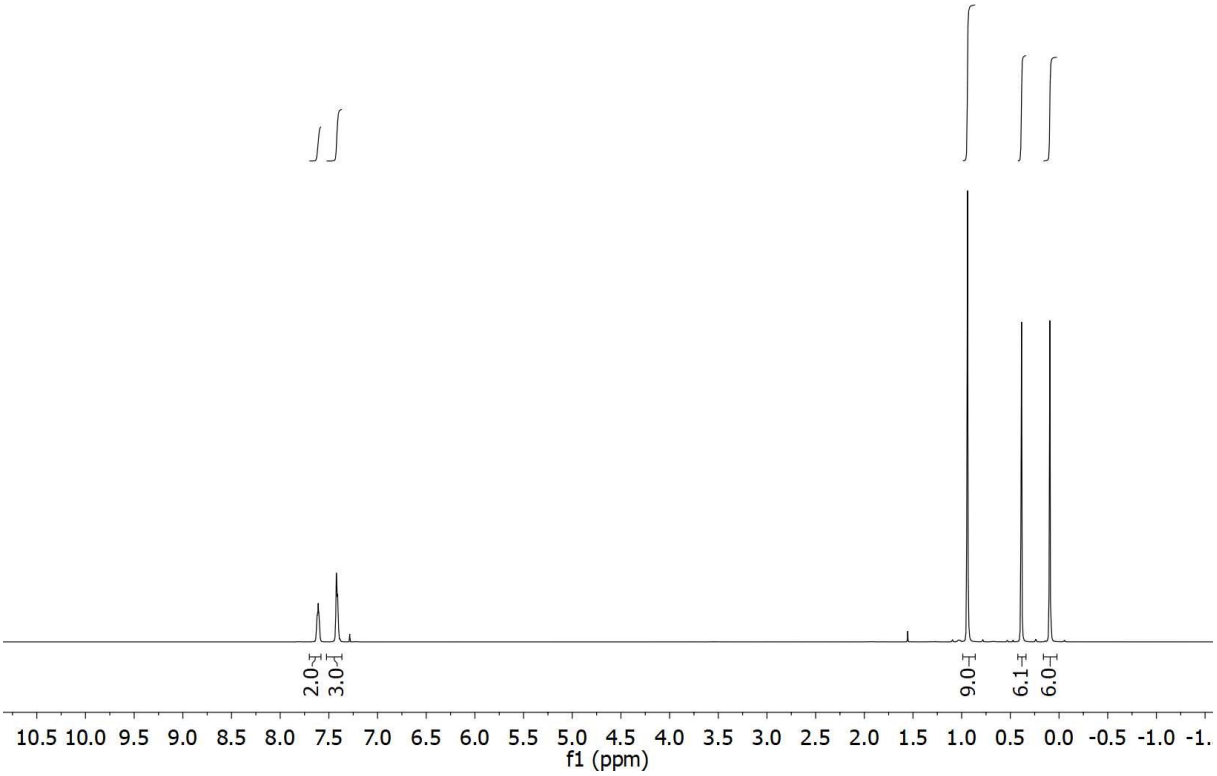


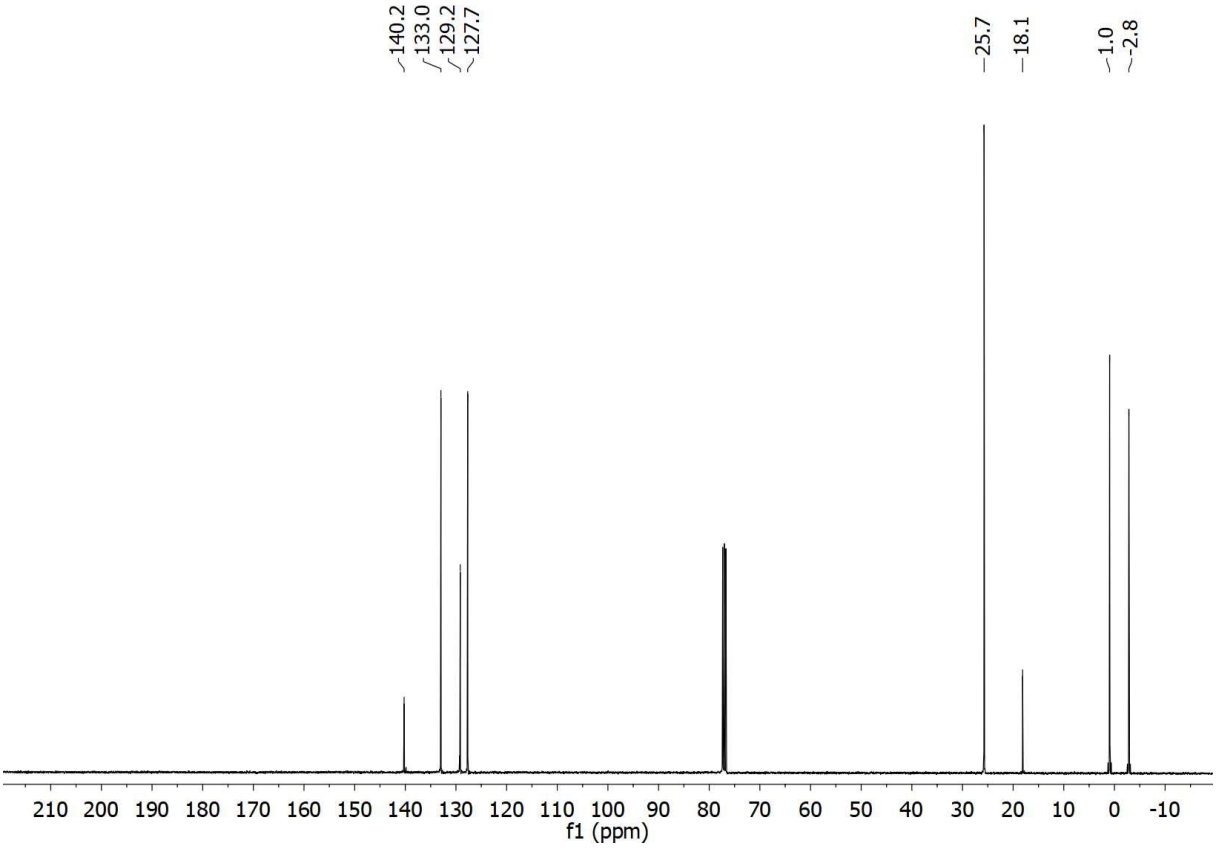


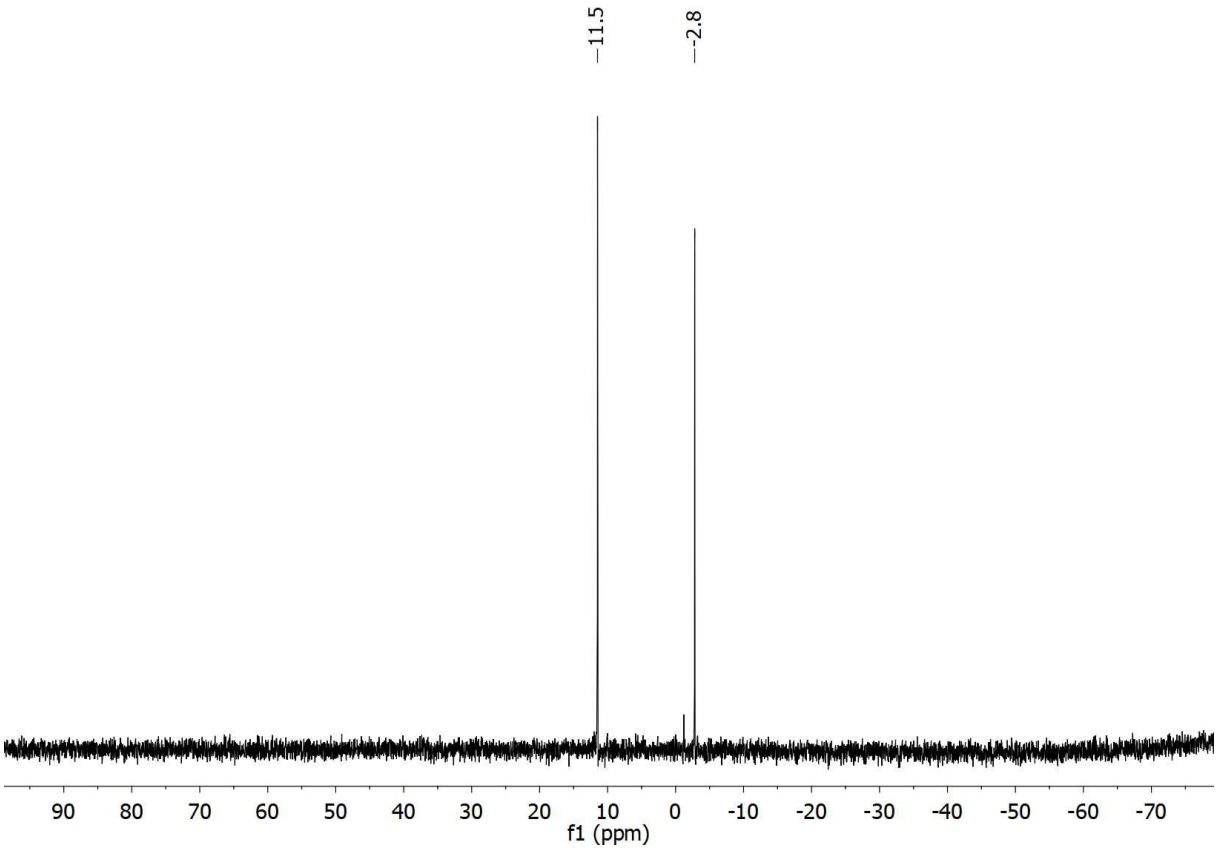



9.2
6.1
00
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-20
f1 (ppm)

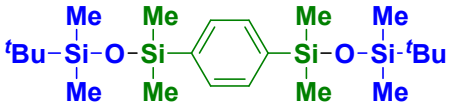
1-*Tert*-butyl-1,1,3,3-tetramethyl-3-phenyldisiloxane (**3e**).

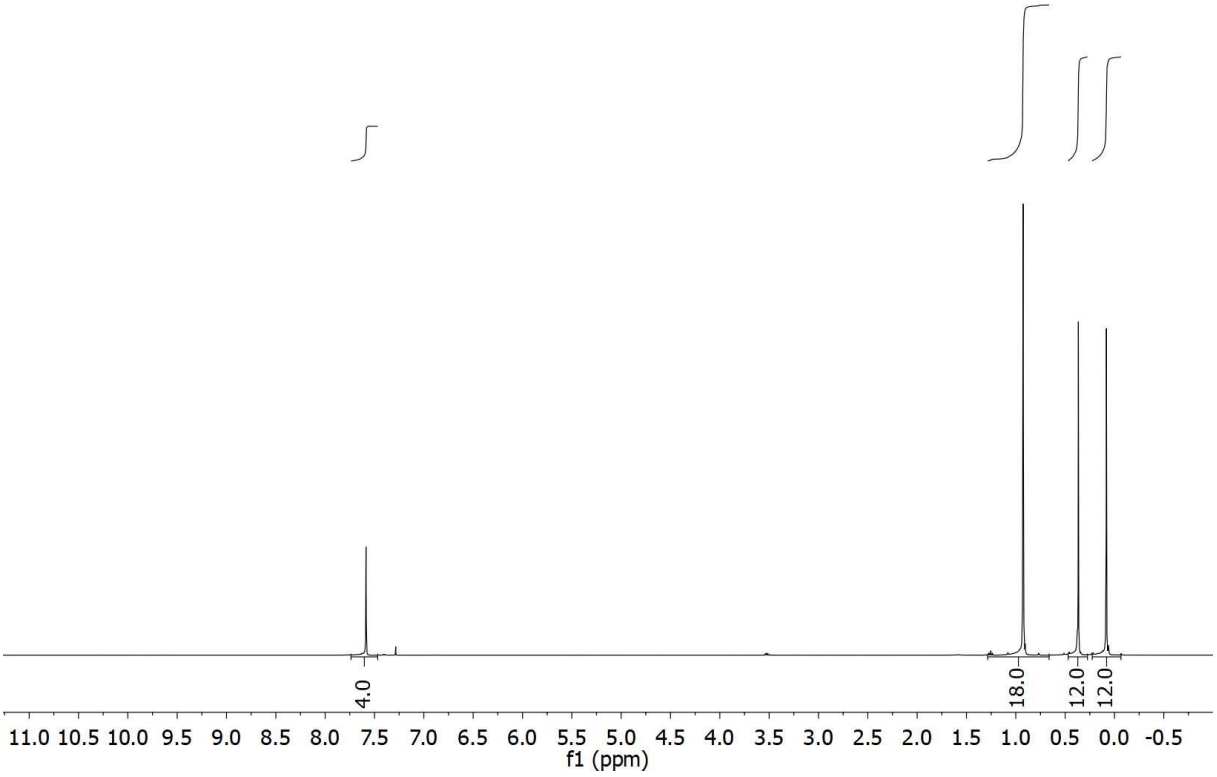


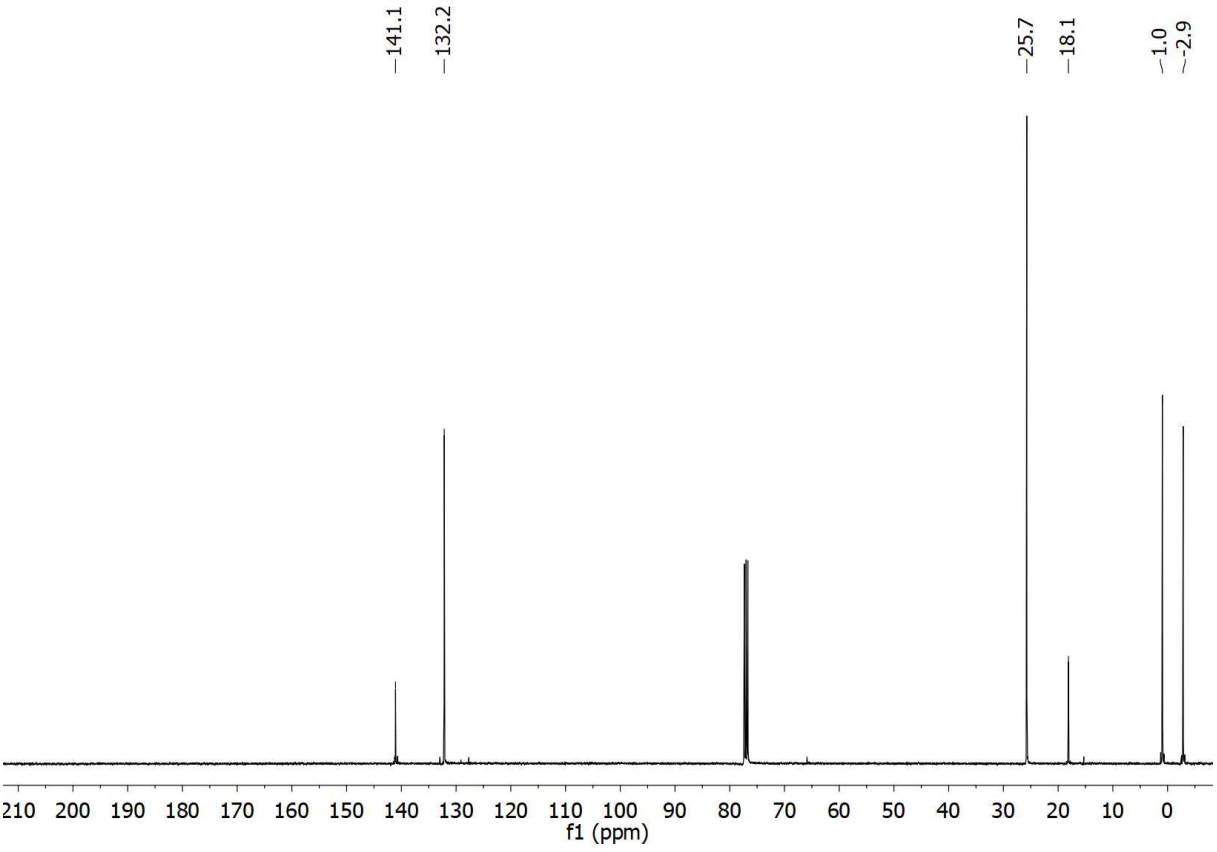


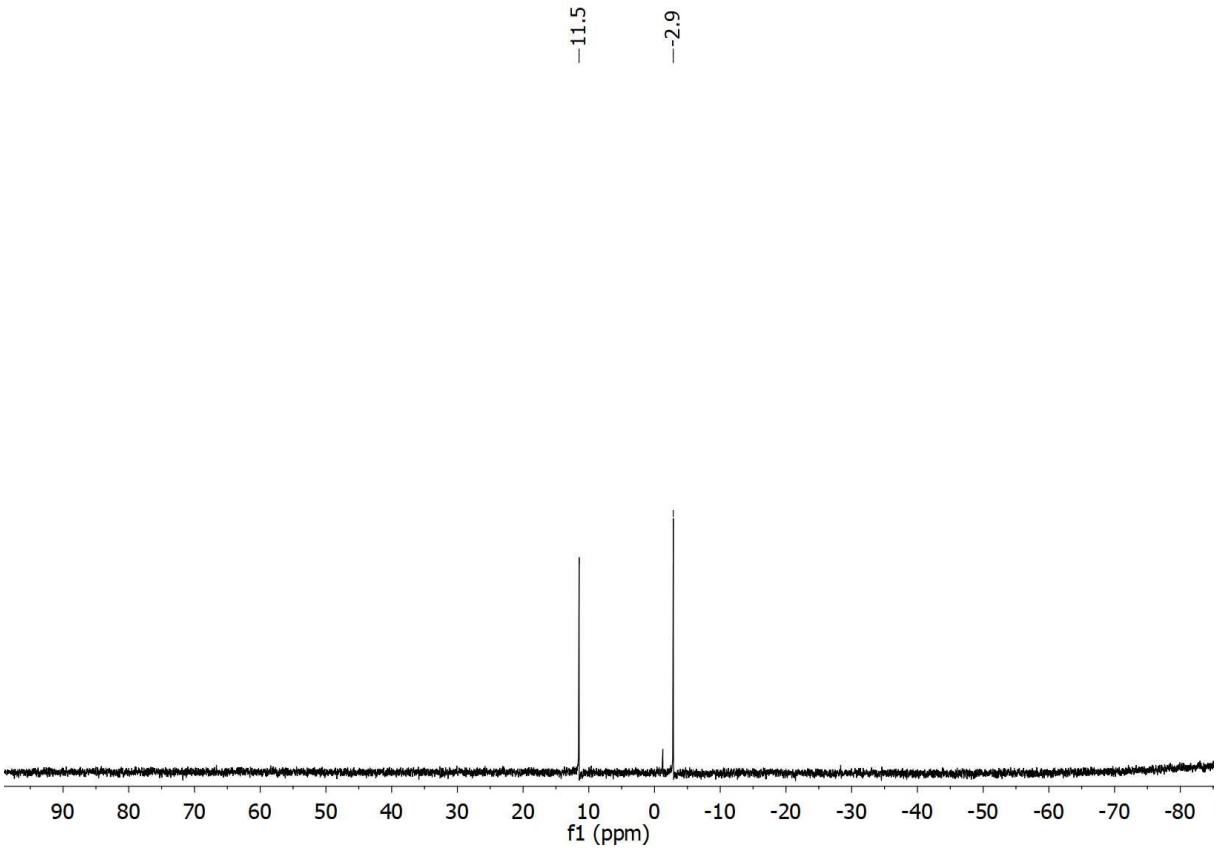

11.5
2.8
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
f1 (ppm)

1,4-bis(3-(*tert*-butyl)-1,1,3,3-tetramethyldisiloxanyl)benzene (**3f**) was obtained as colorless oil in 93% yield.

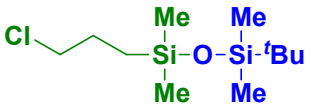


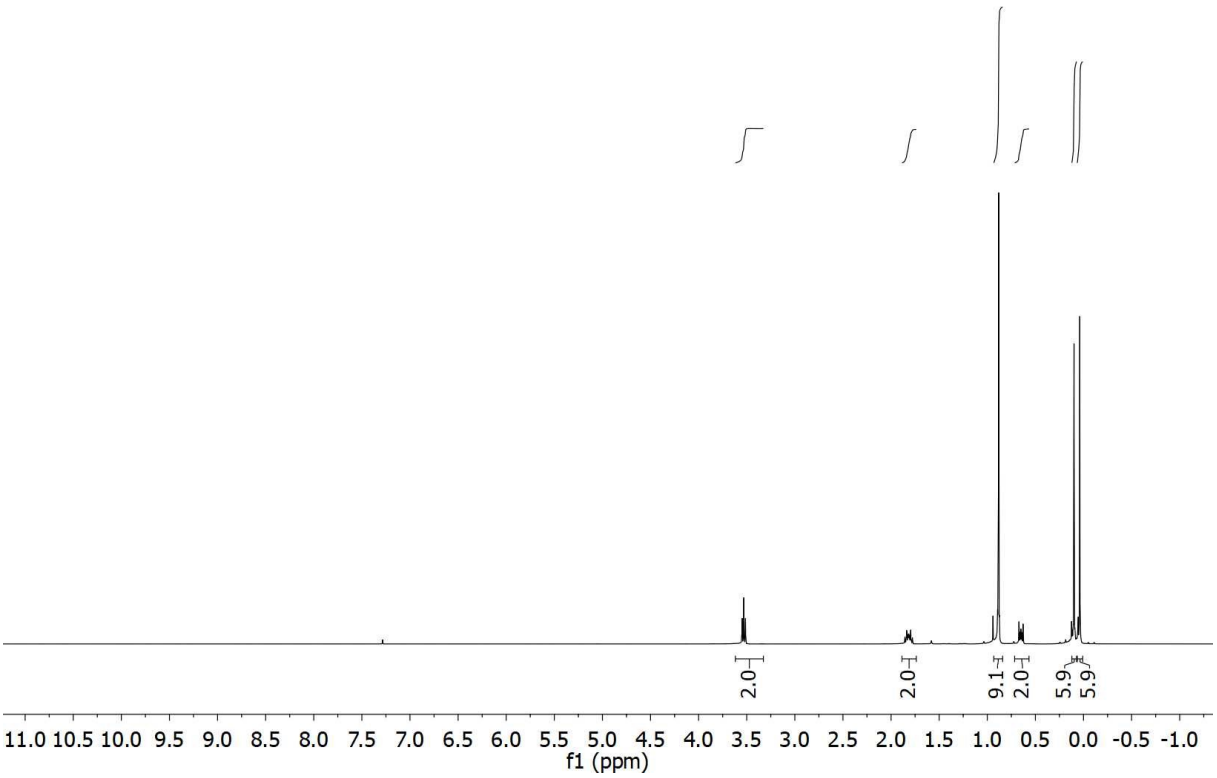


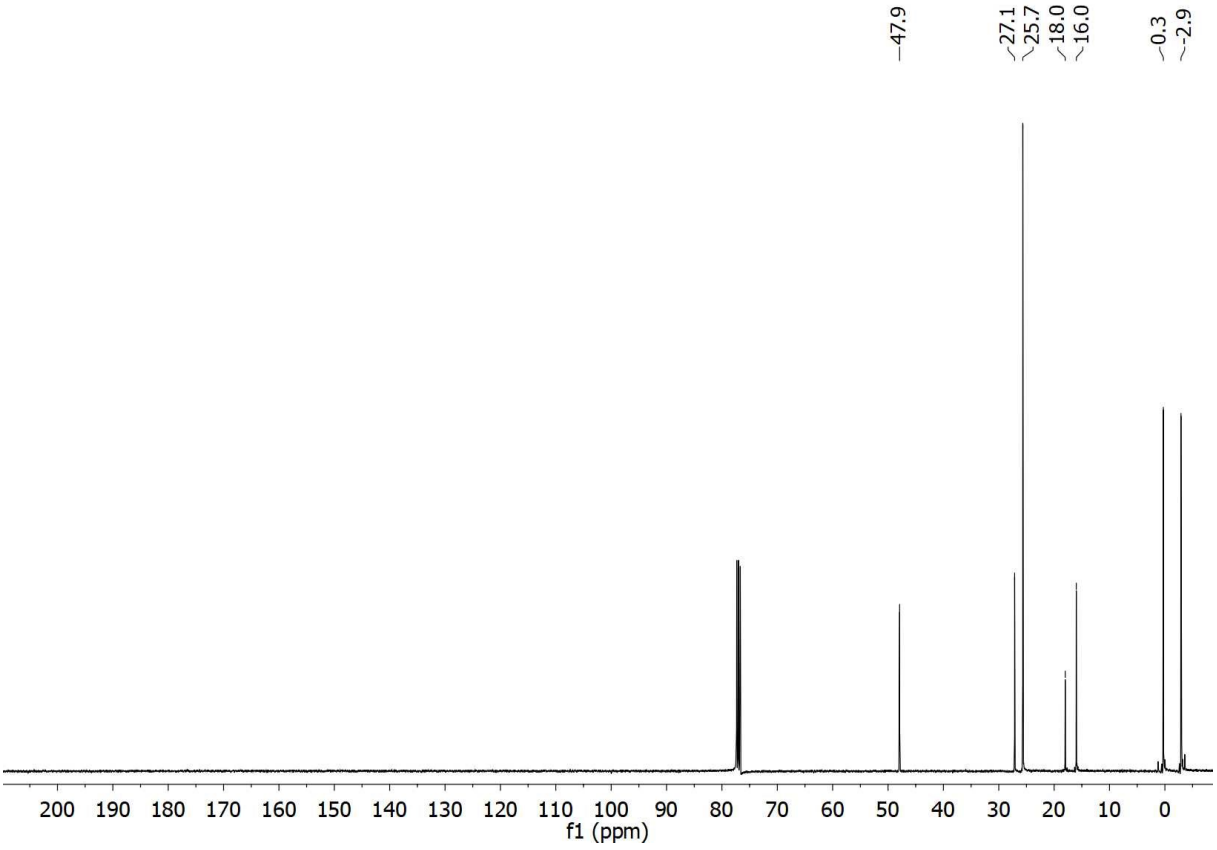


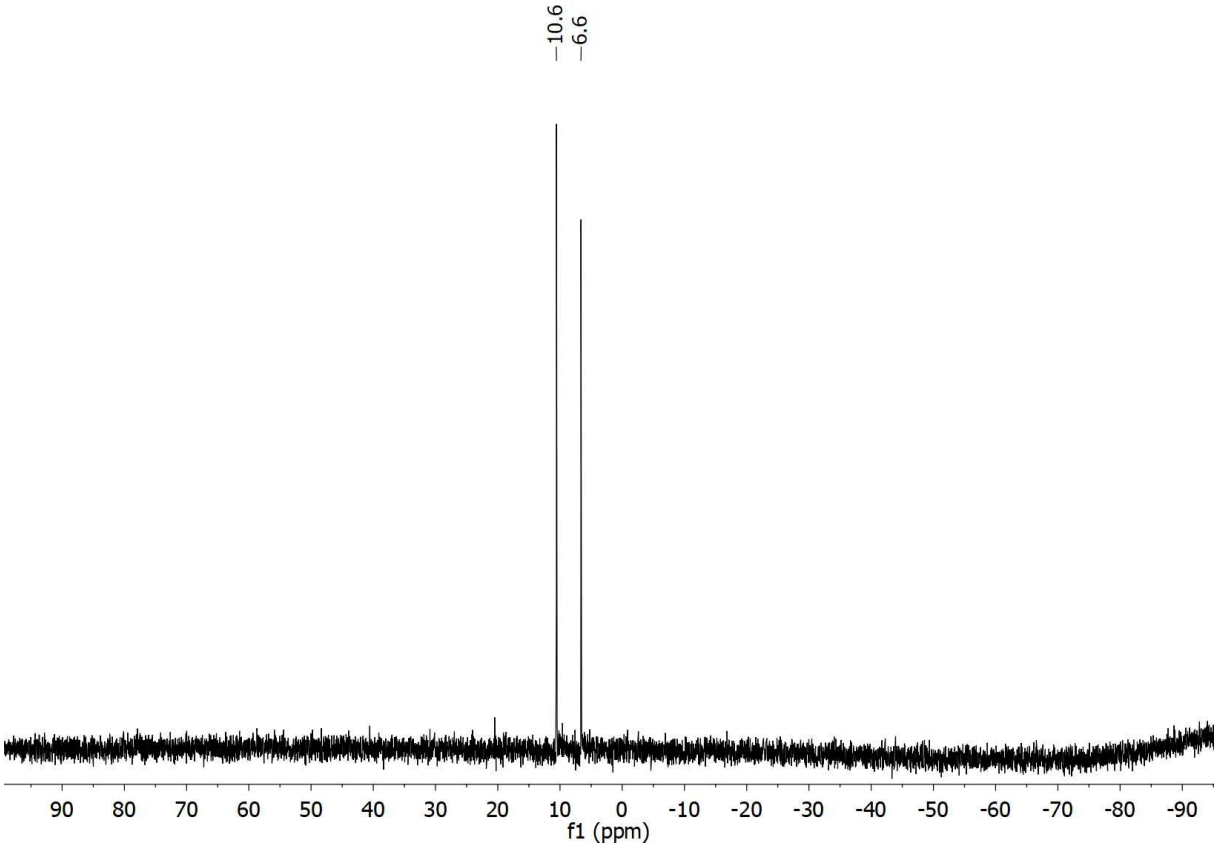

11.5
2.9
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

1-*Tert*-butyl-3-(3-chloropropyl)-1,1,3,3-tetramethyldisiloxane (**3g**).

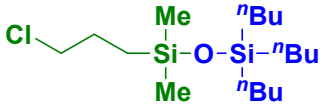


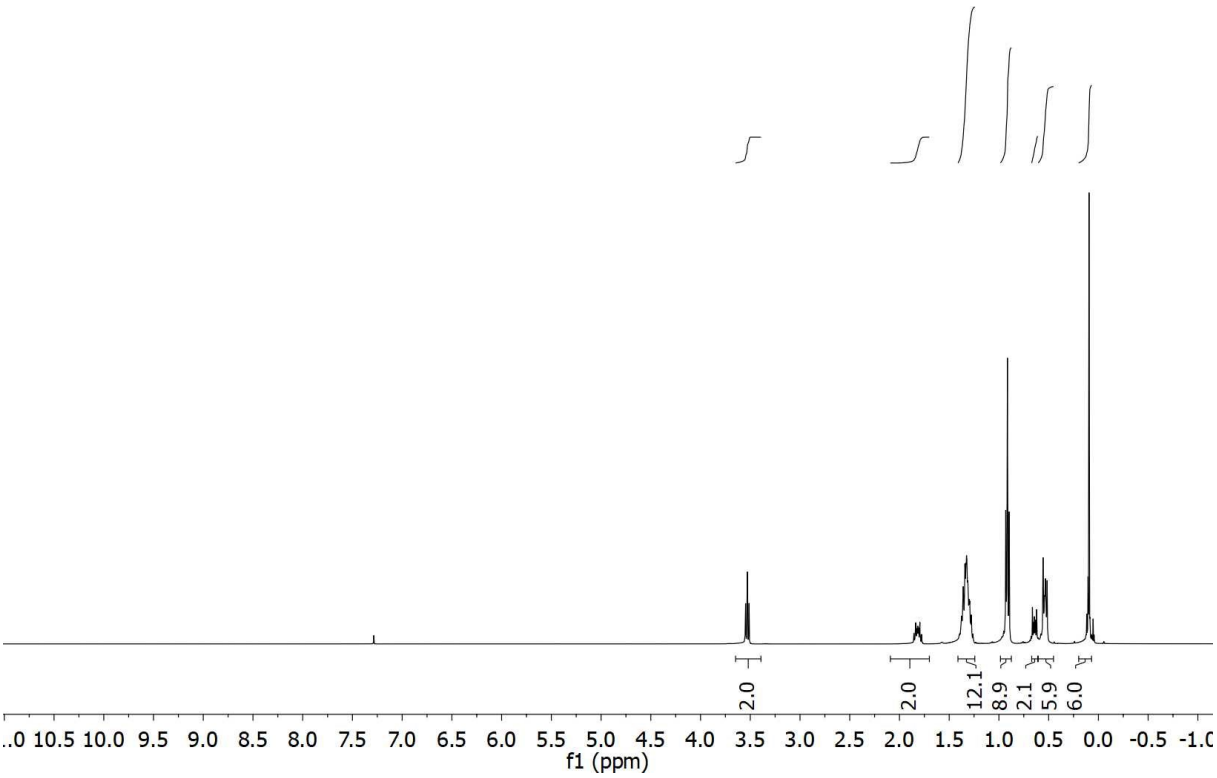


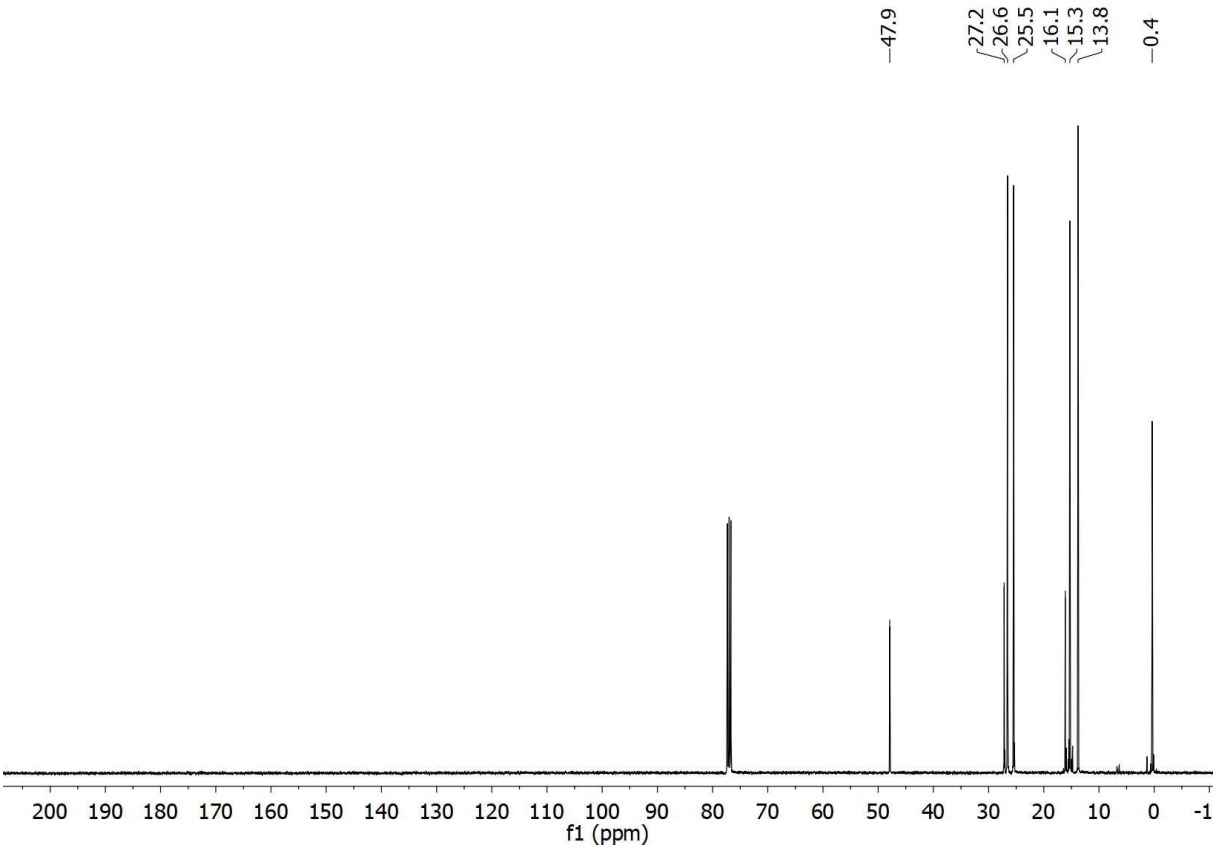


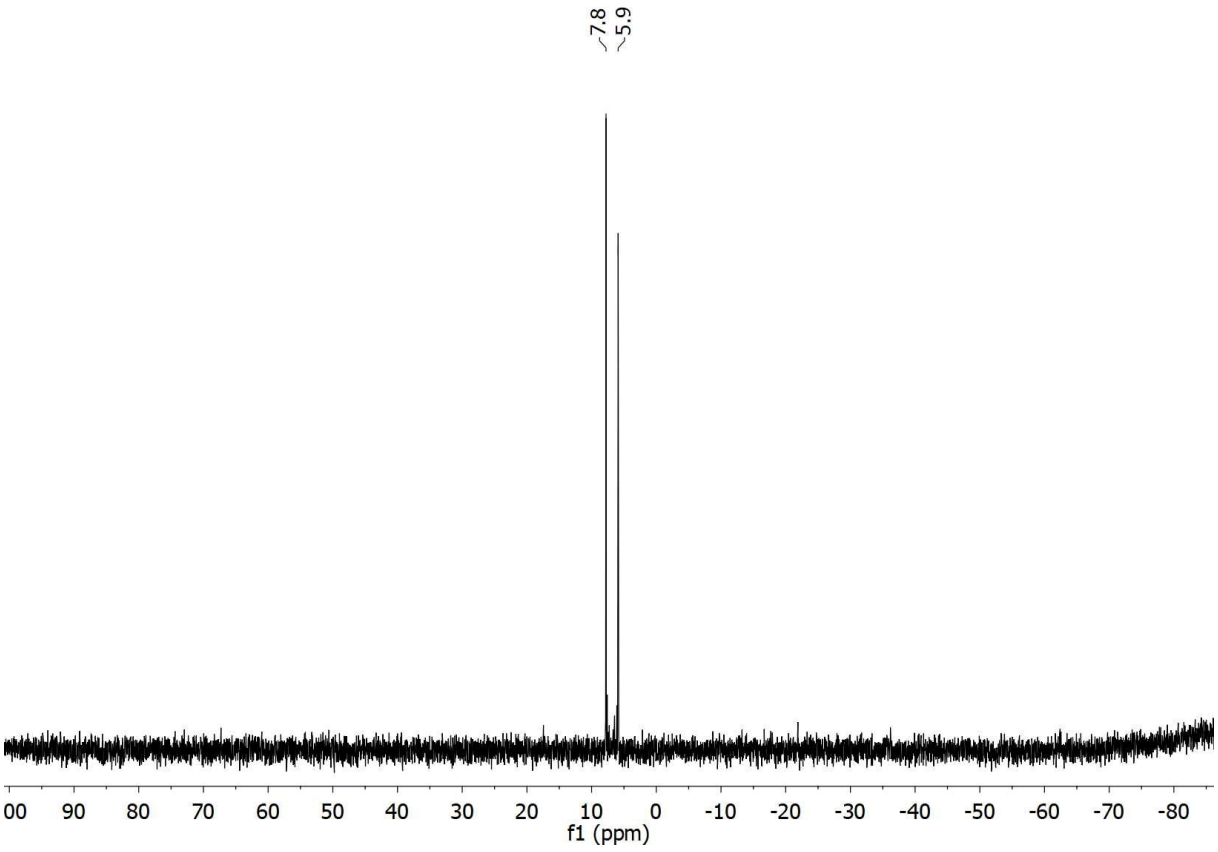

10.6
6.6
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

1,1,1-tributyl-3-(3-chloropropyl)-3,3-dimethyldisiloxane (**3h**).

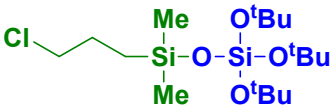


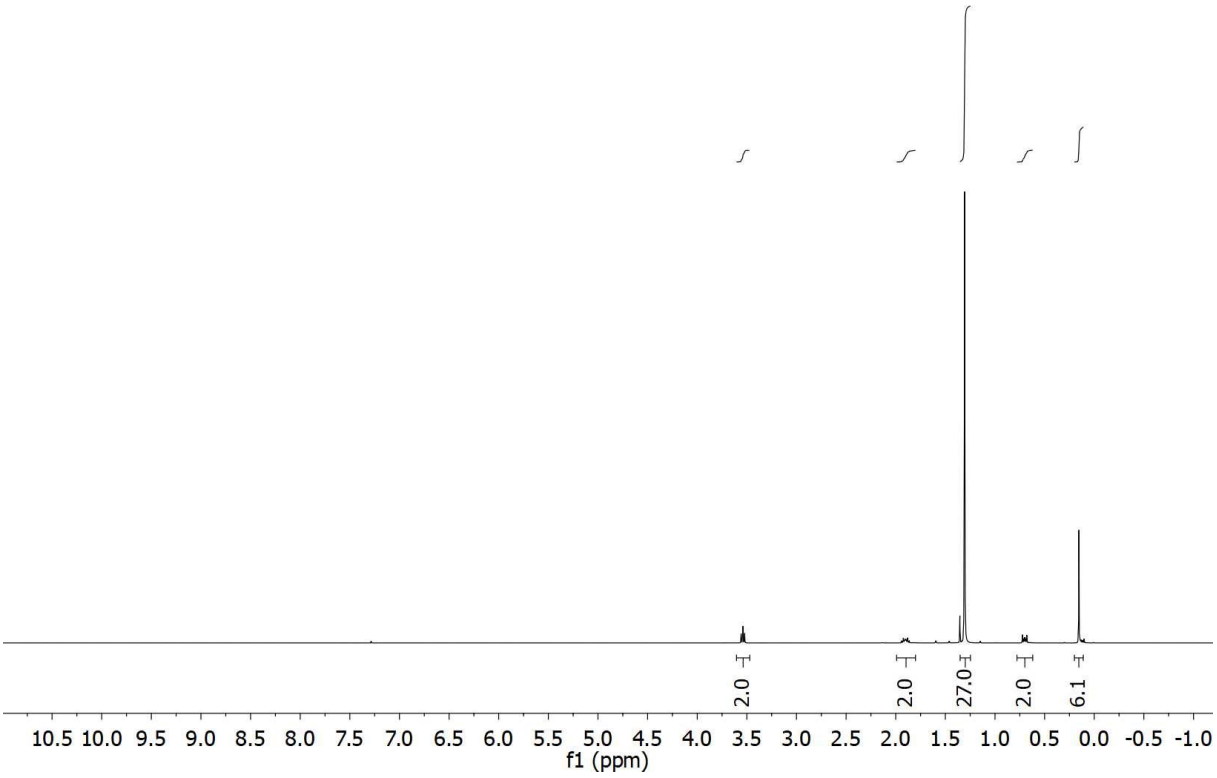


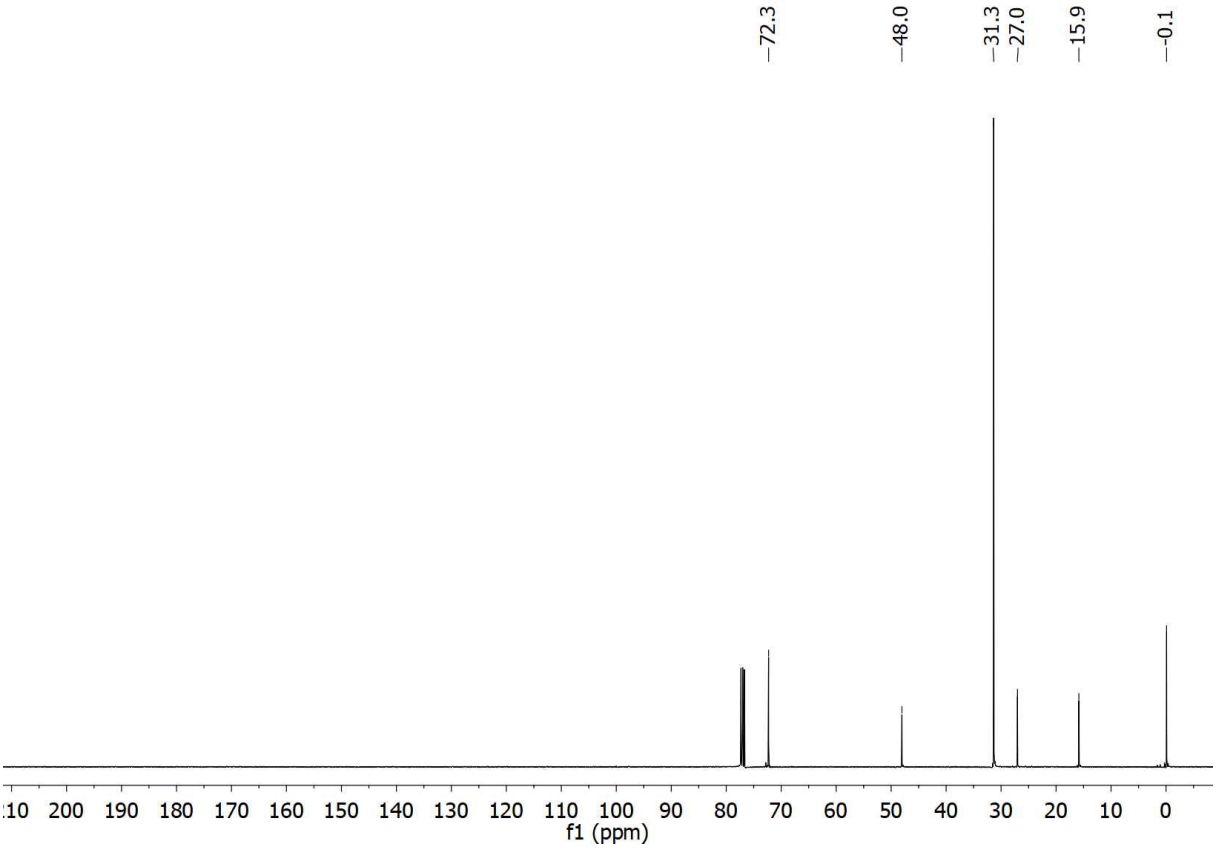


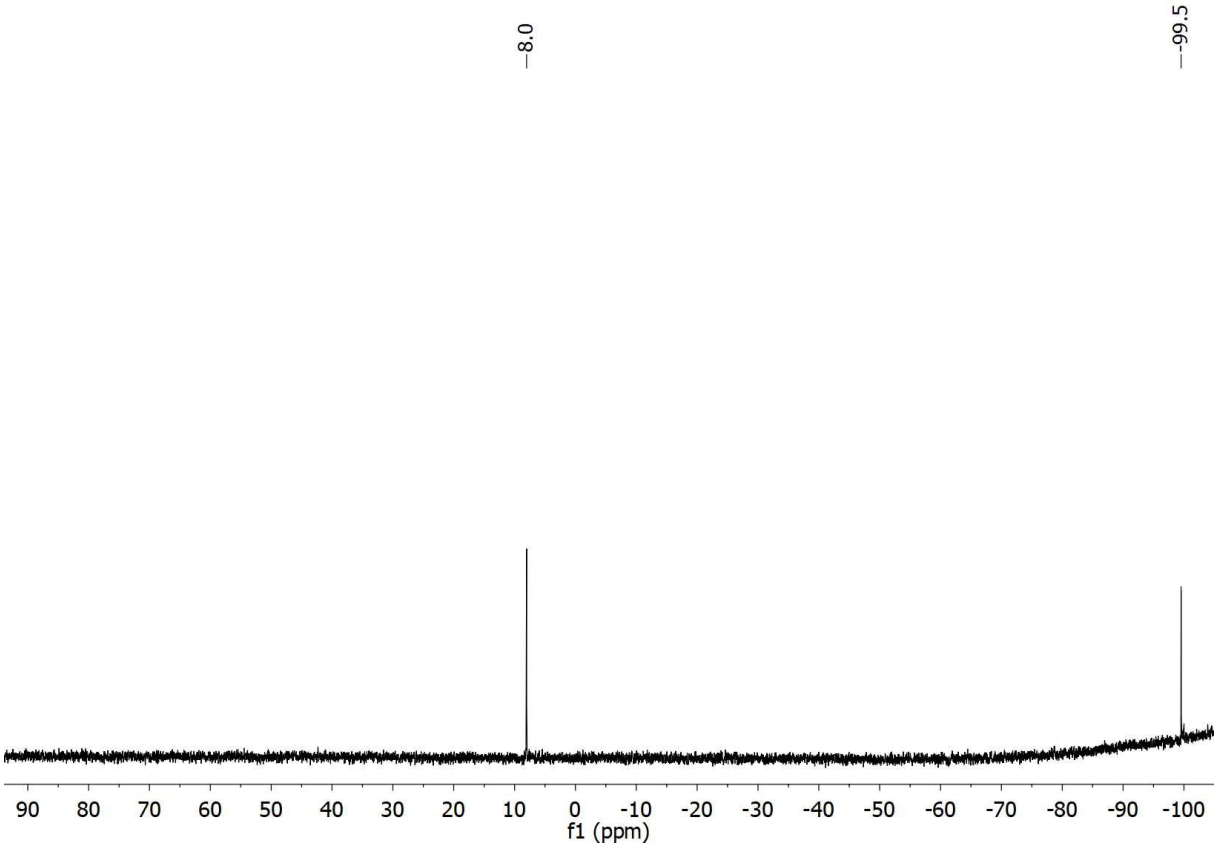

7.8
5.9
00 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

1,1,1-*Tert*-butoxy-3-(3-chloropropyl)-3,3-dimethyldisiloxane (**3i**).

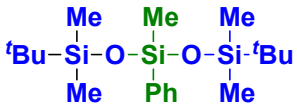


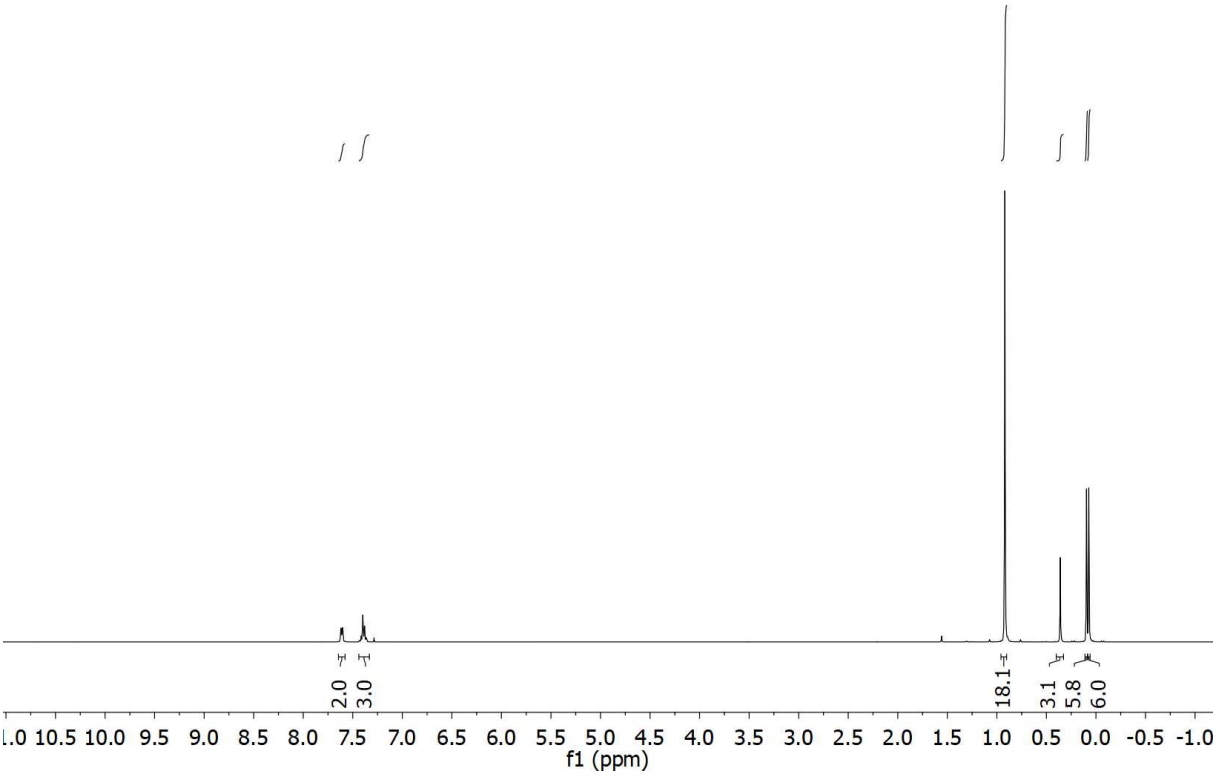


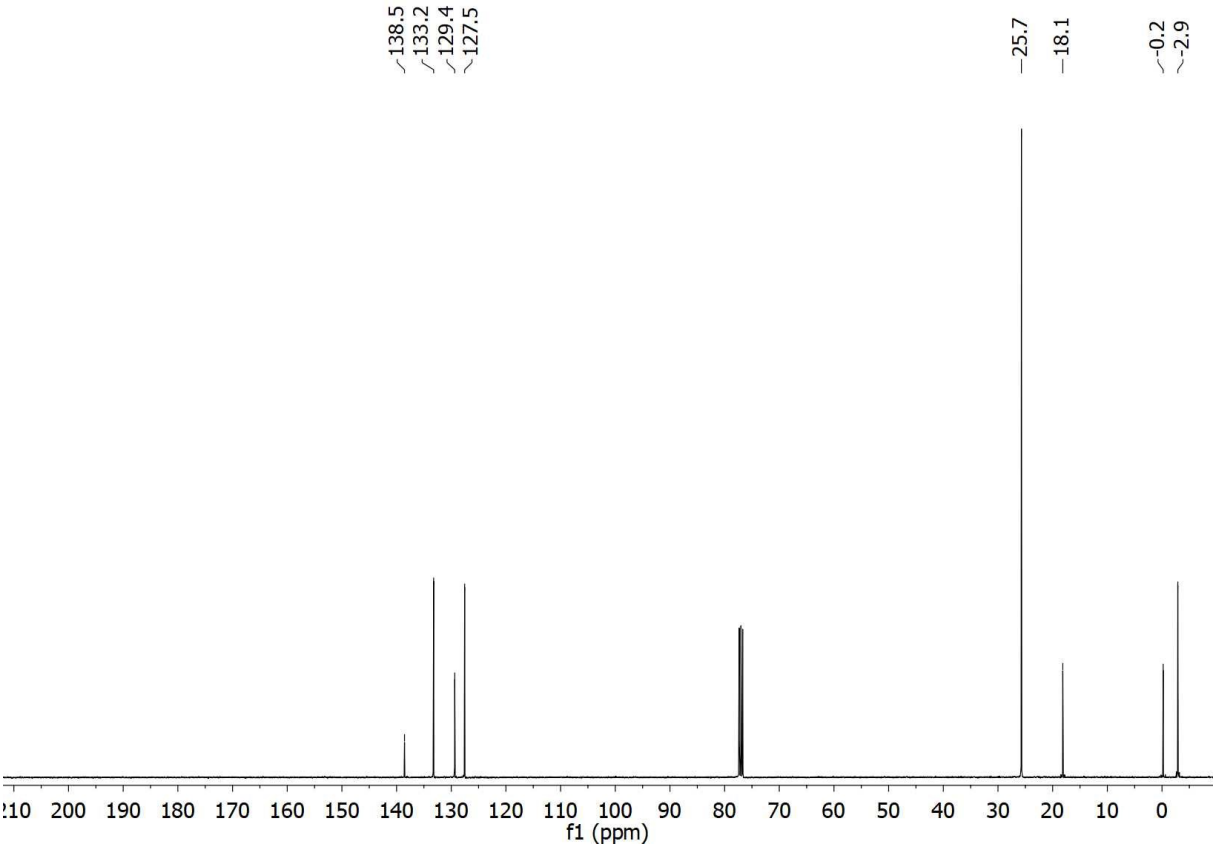


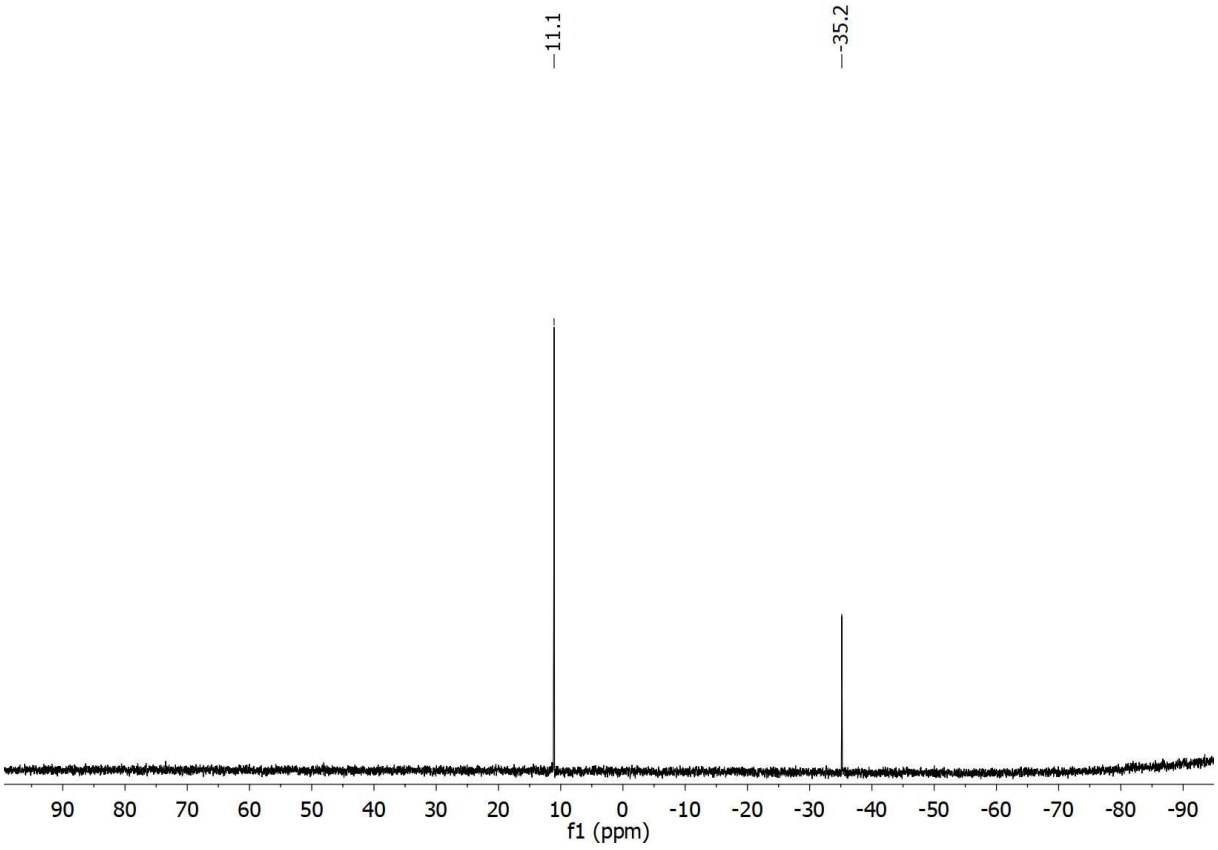

8.0
-99.5
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

1,5-Di-tert-butyl-1,1,3,5,5-pentamethyl-3-phenyltrisiloxane (**5a**).

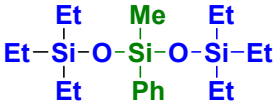


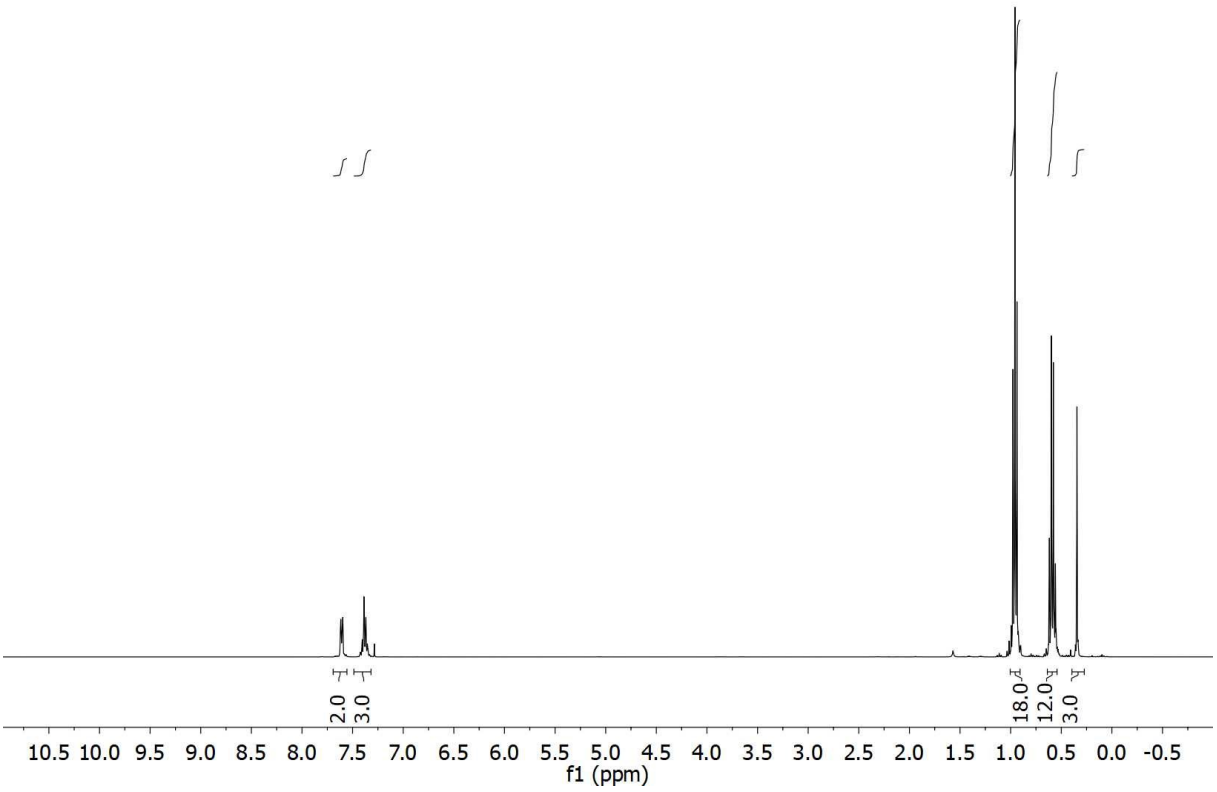


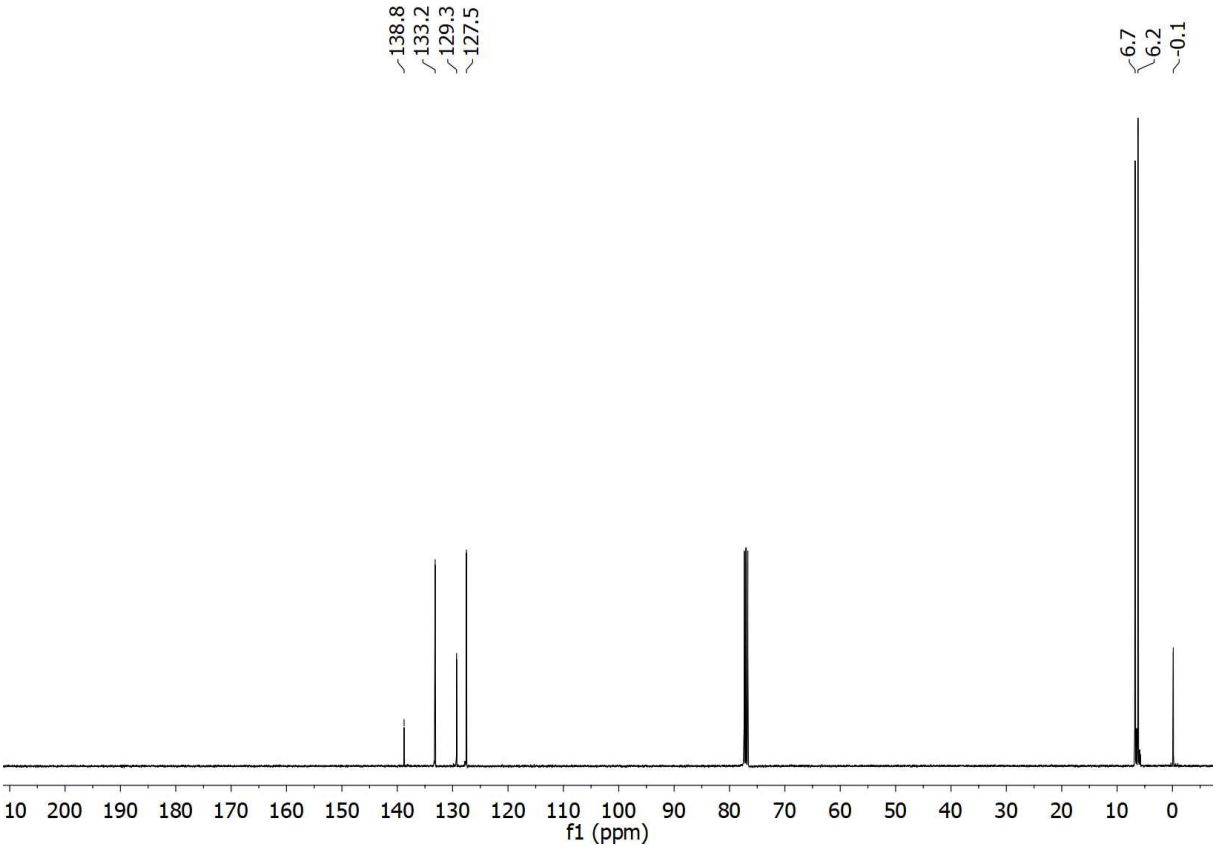


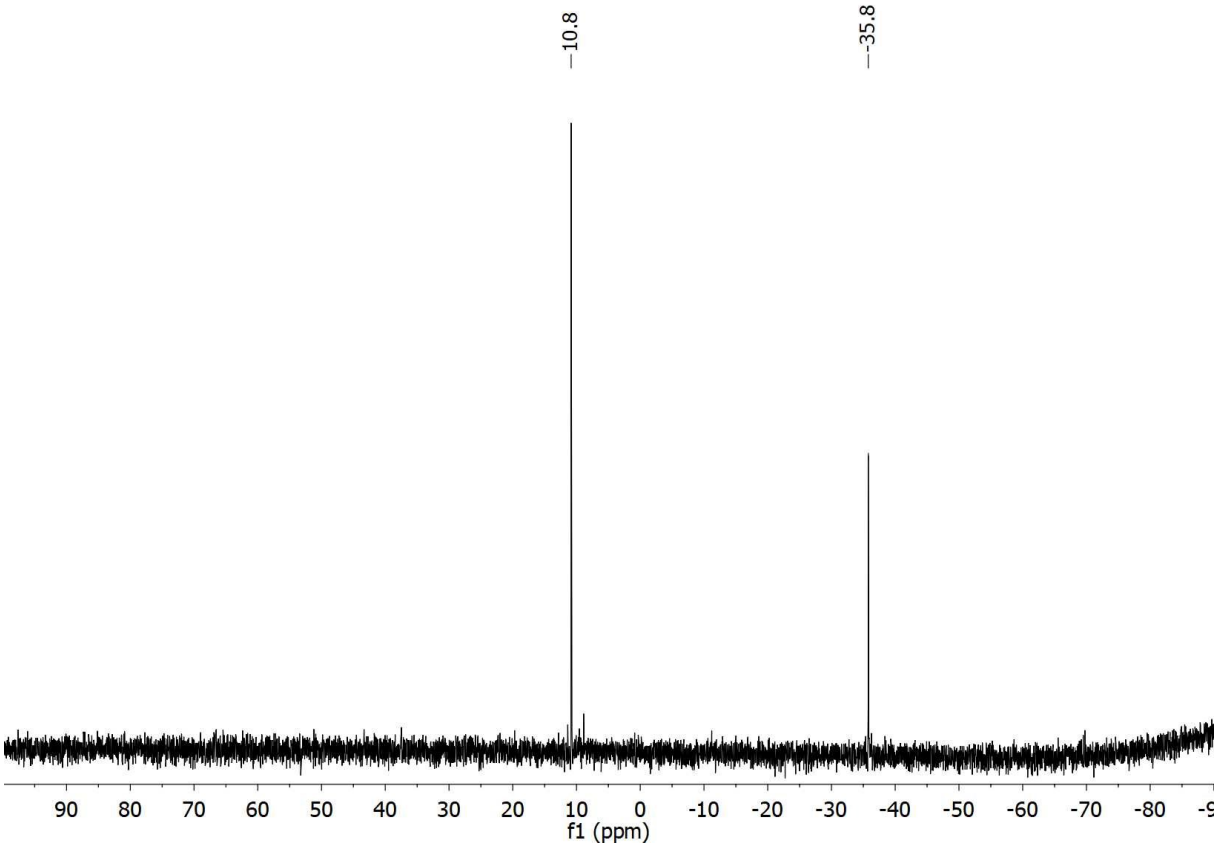

11.1
-35.2
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

1,1,1,5,5,5-Hexaethyl-3-methyl-3-phenyltrisiloxane (**5b**).

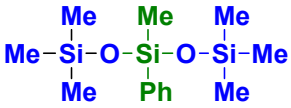


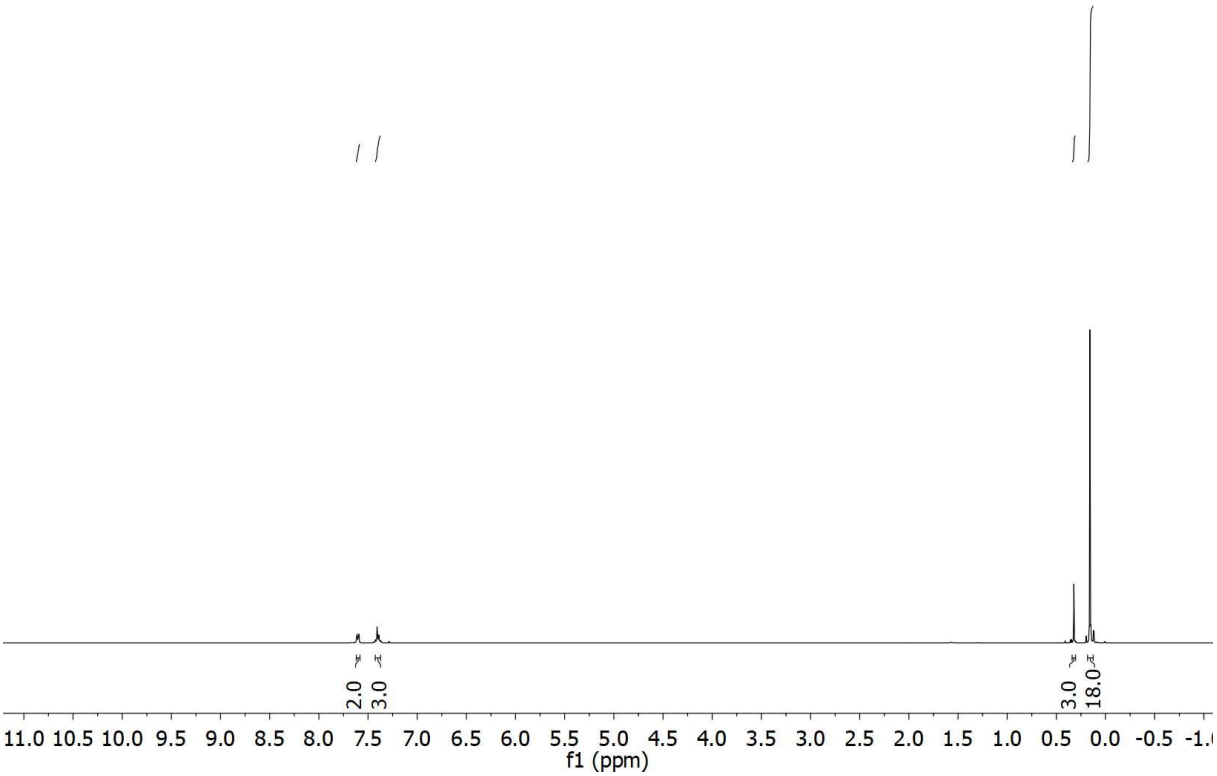


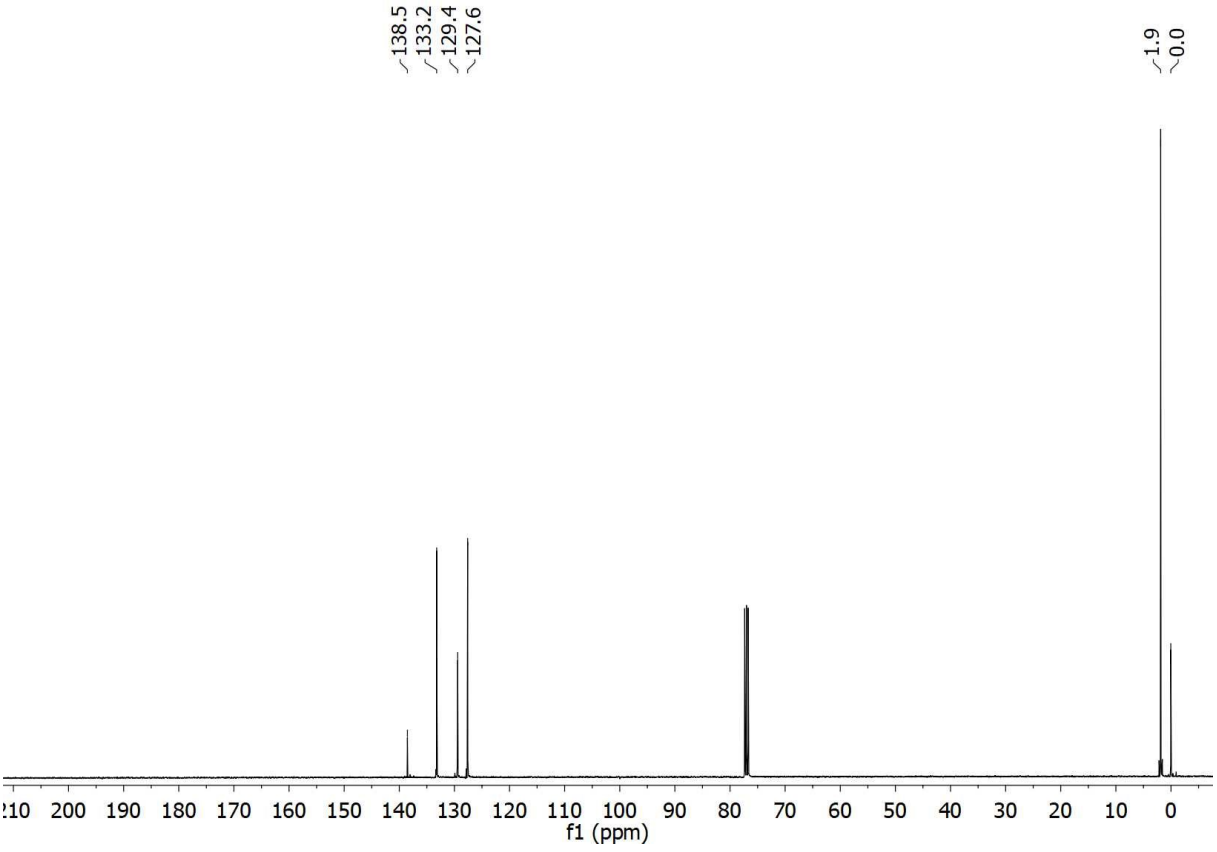


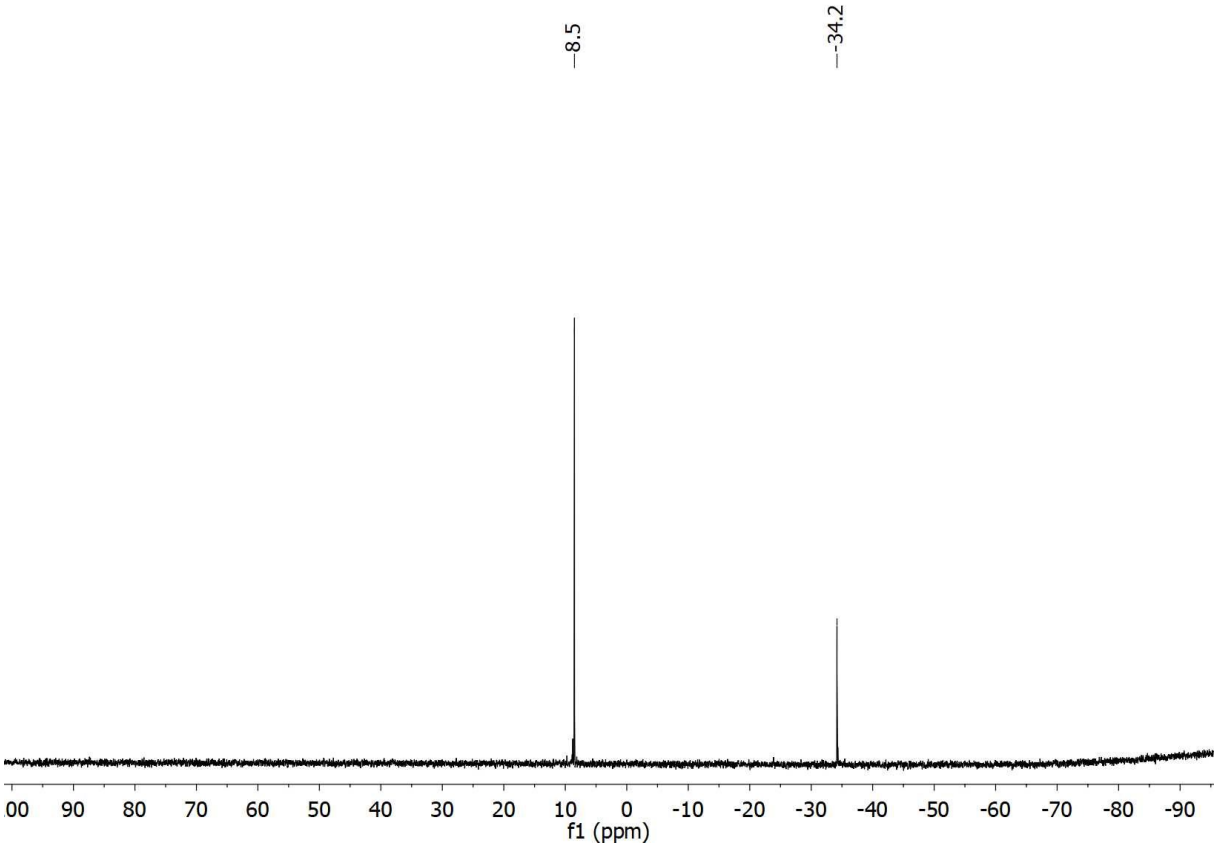

10.8
-35.8
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

1,1,1,3,5,5,5-Heptamethyl-3-phenyltrisiloxane (**5c**).

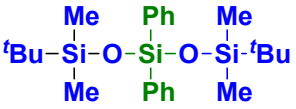


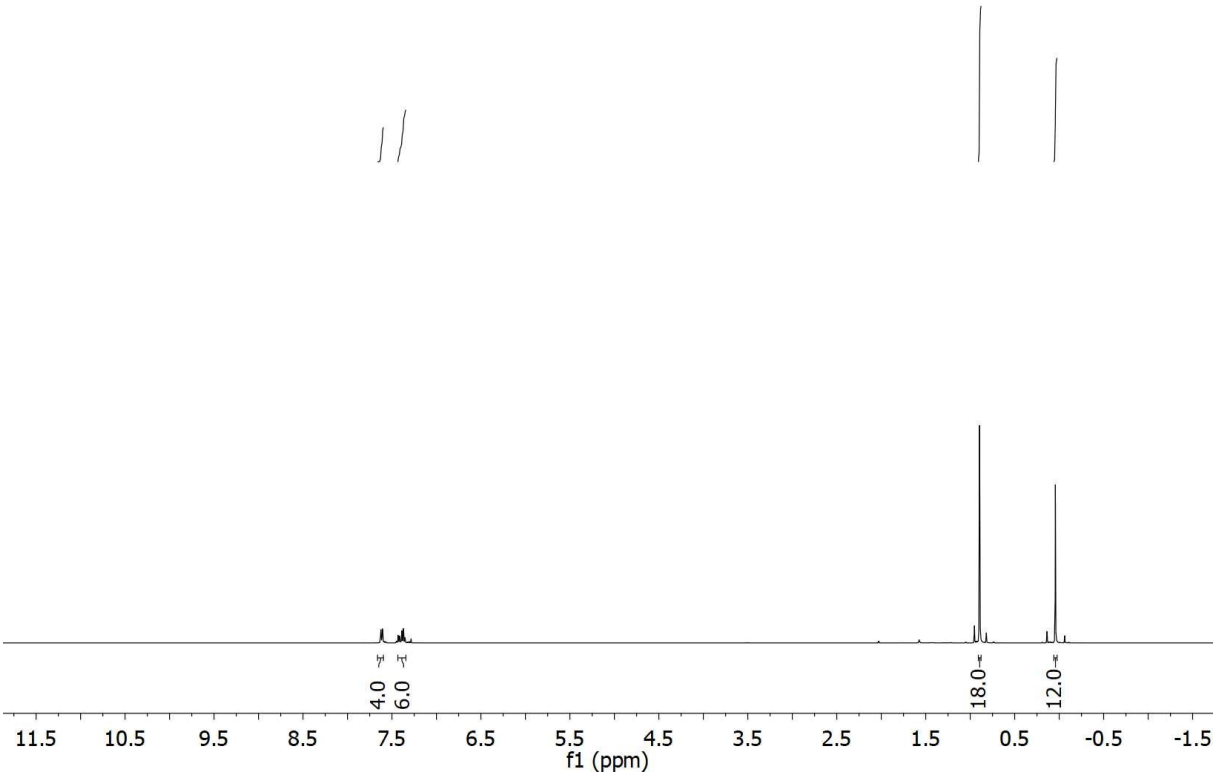


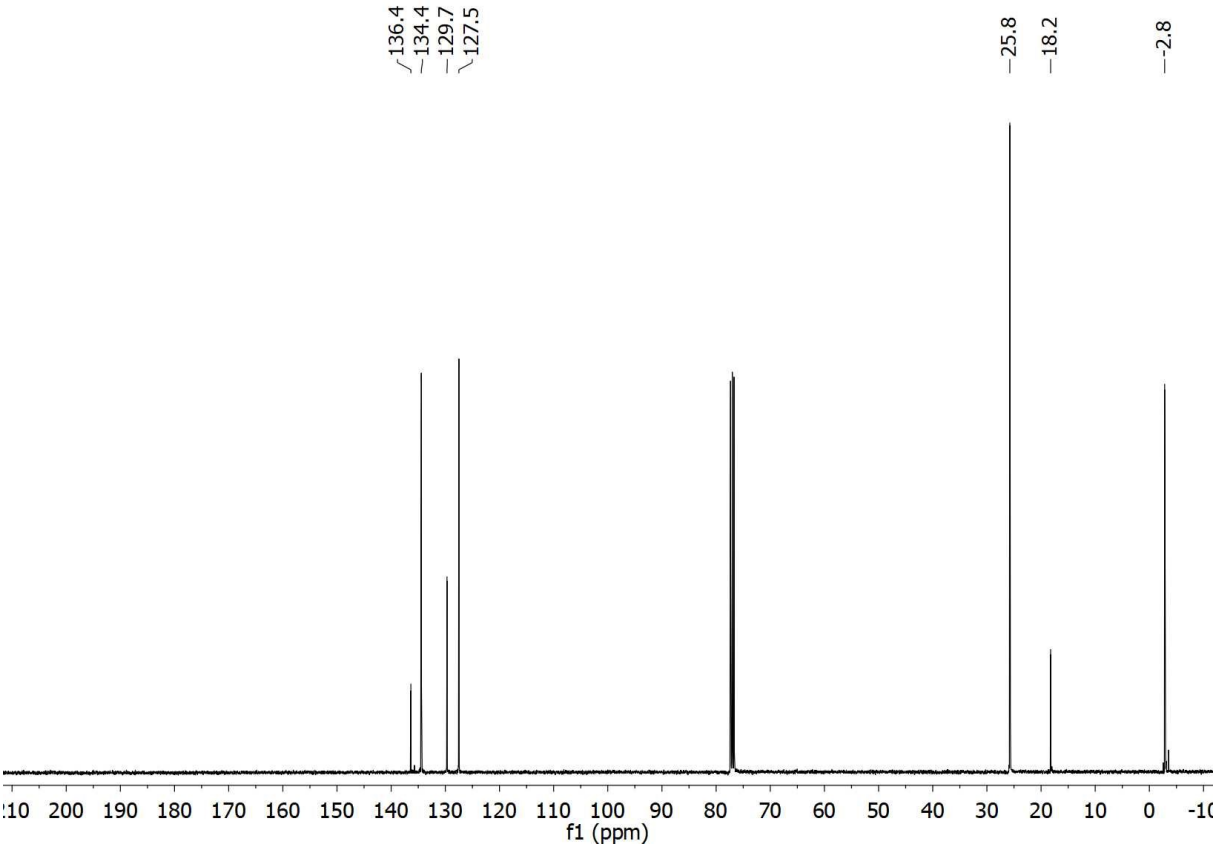


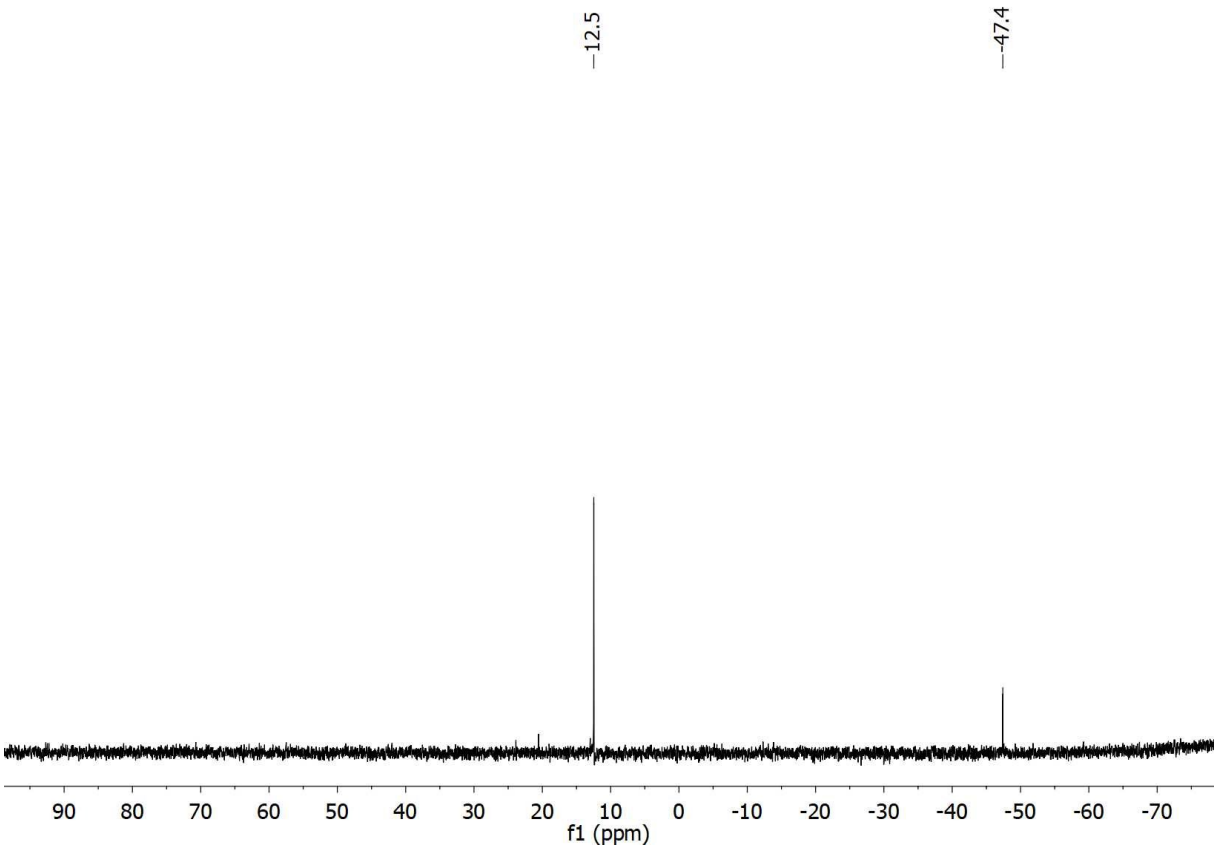

8.5
-34.2
00 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3,3-diphenyltrisiloxane (**5d**).

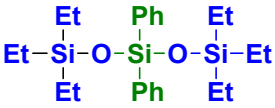


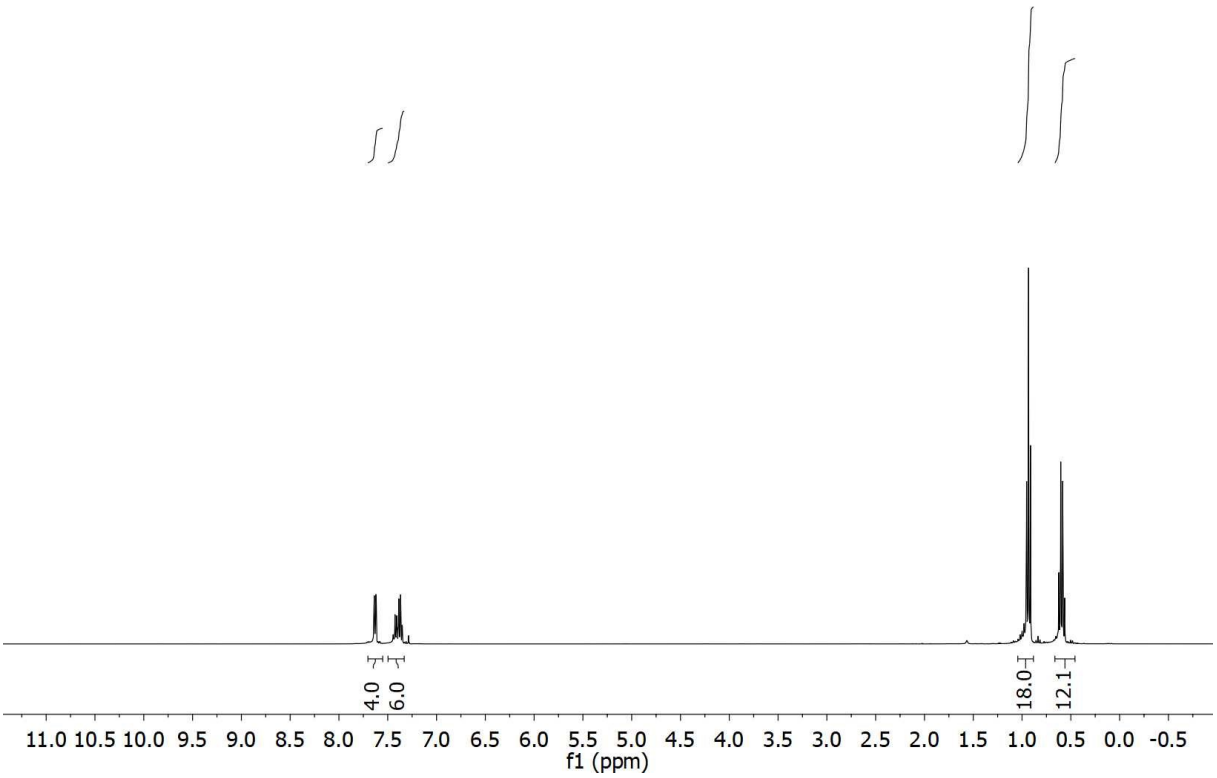


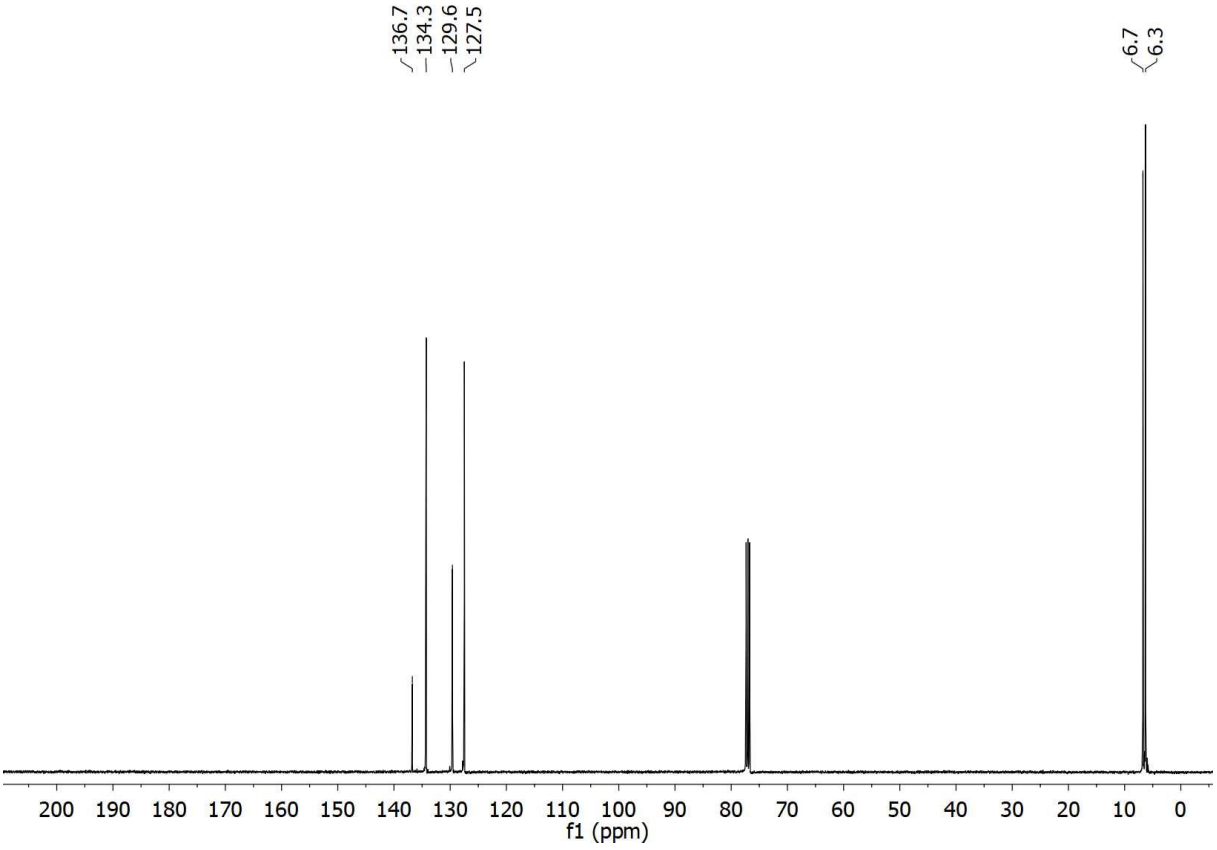


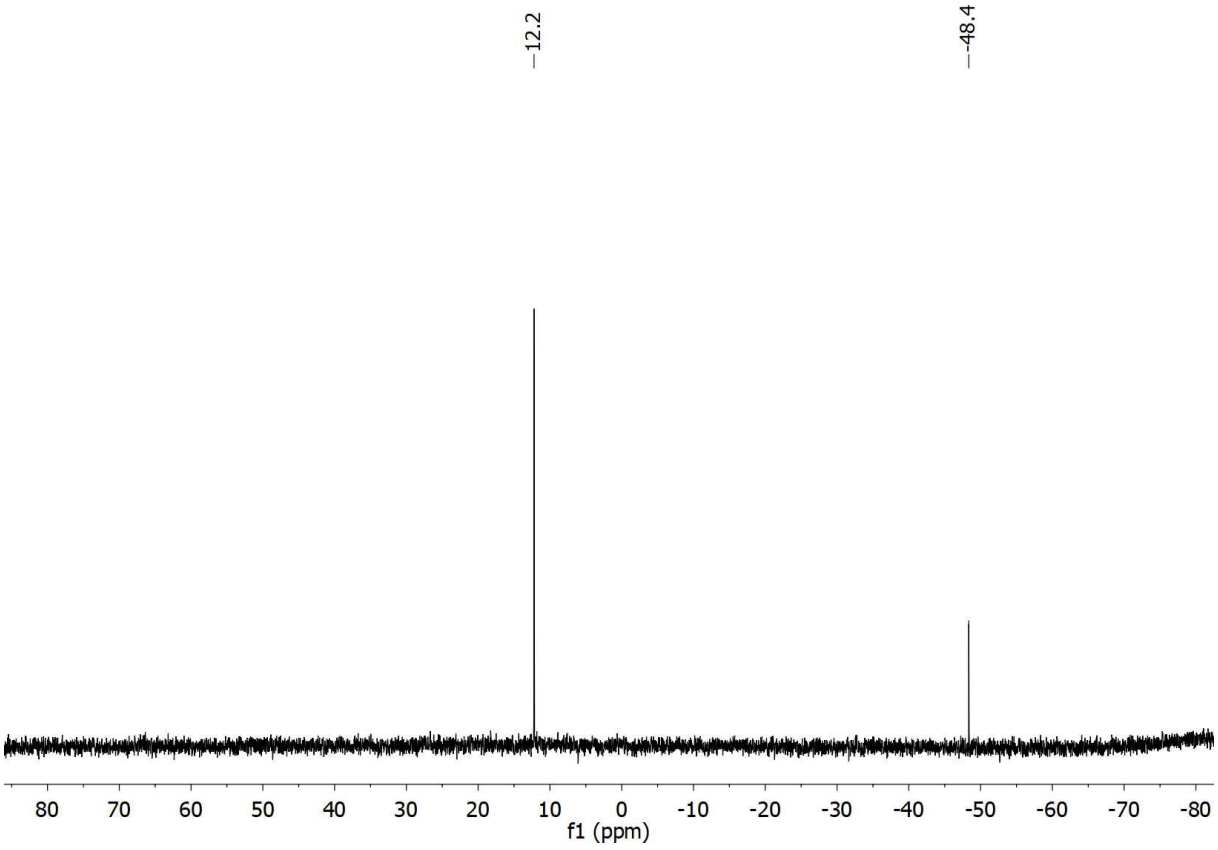

-12.5
-47.4
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
f1 (ppm)

1,1,1,5,5,5-Hexaethyl-3,3-diphenyltrisiloxane (**5e**).

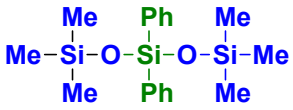


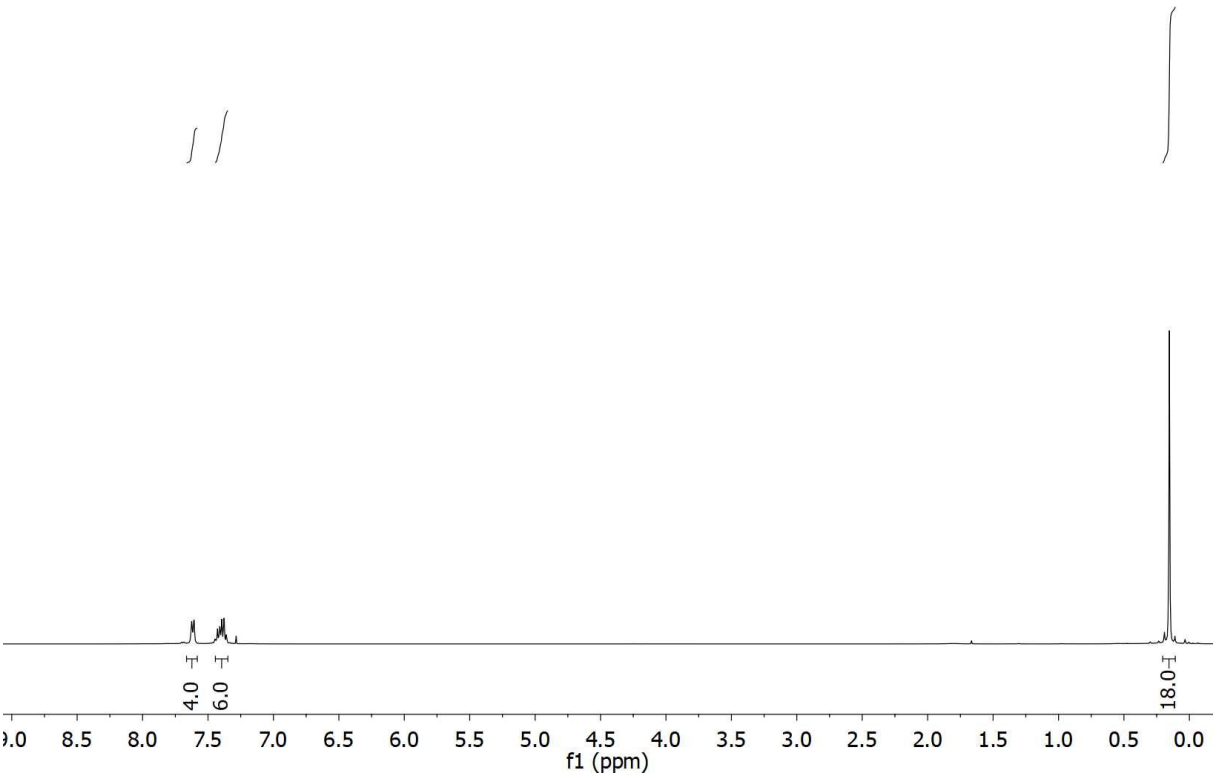


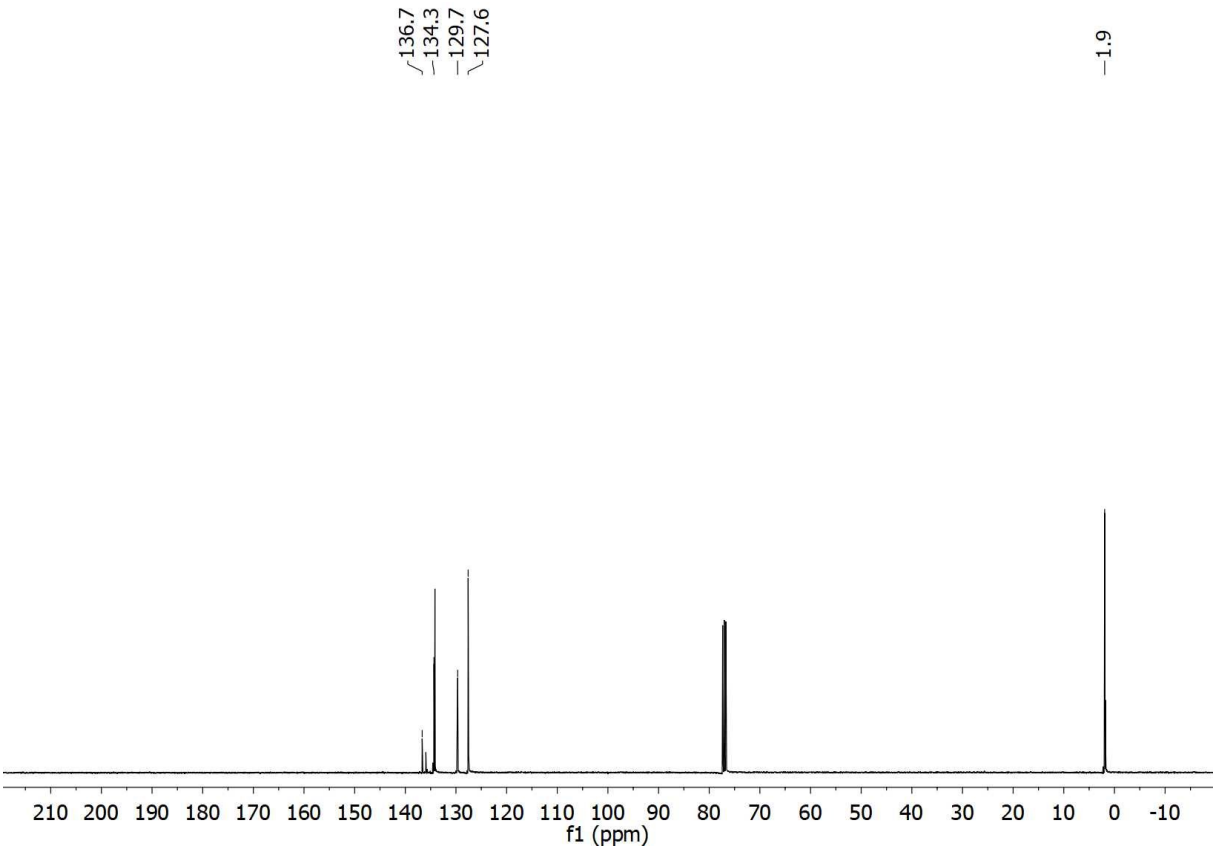


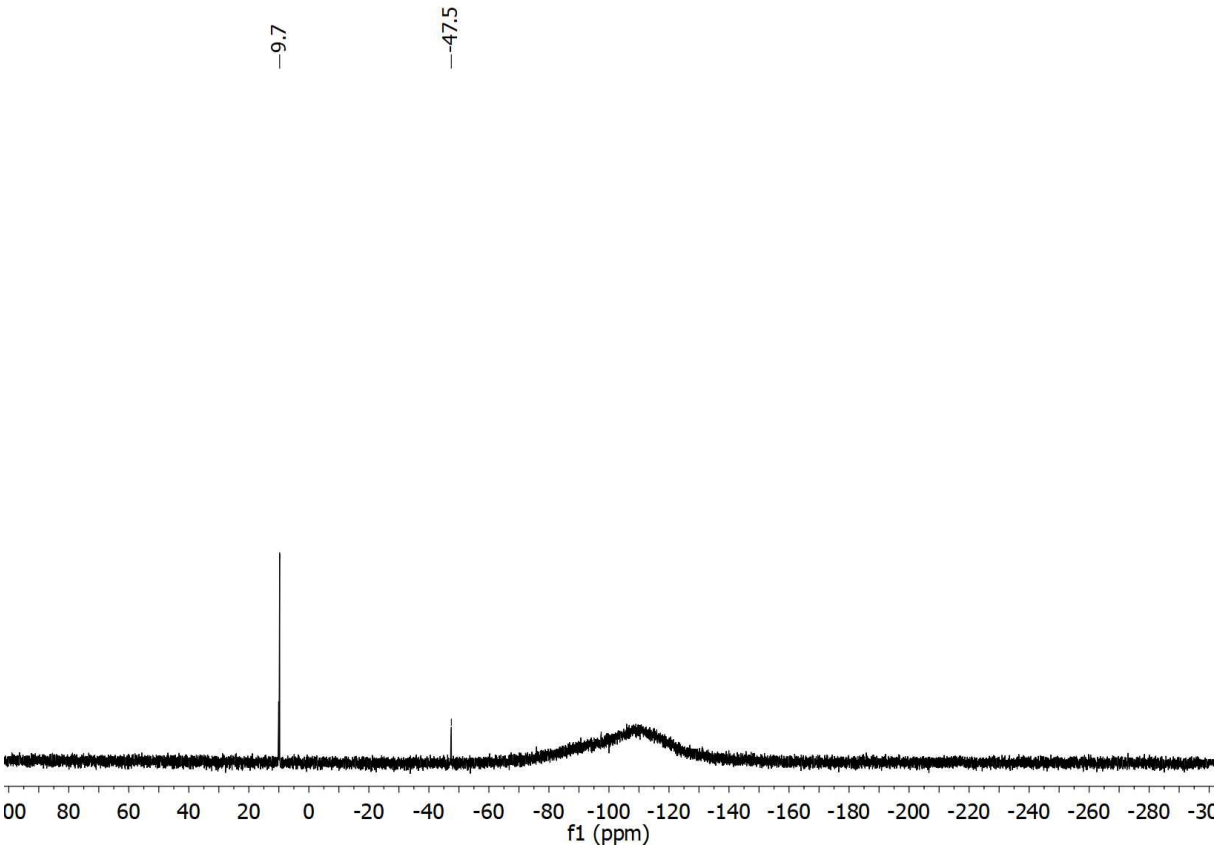

12.2
-48.4
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)

1,1,1,5,5,5-Hexamethyl-3,3-diphenyltrisiloxane (**5f**).

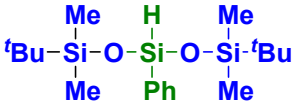


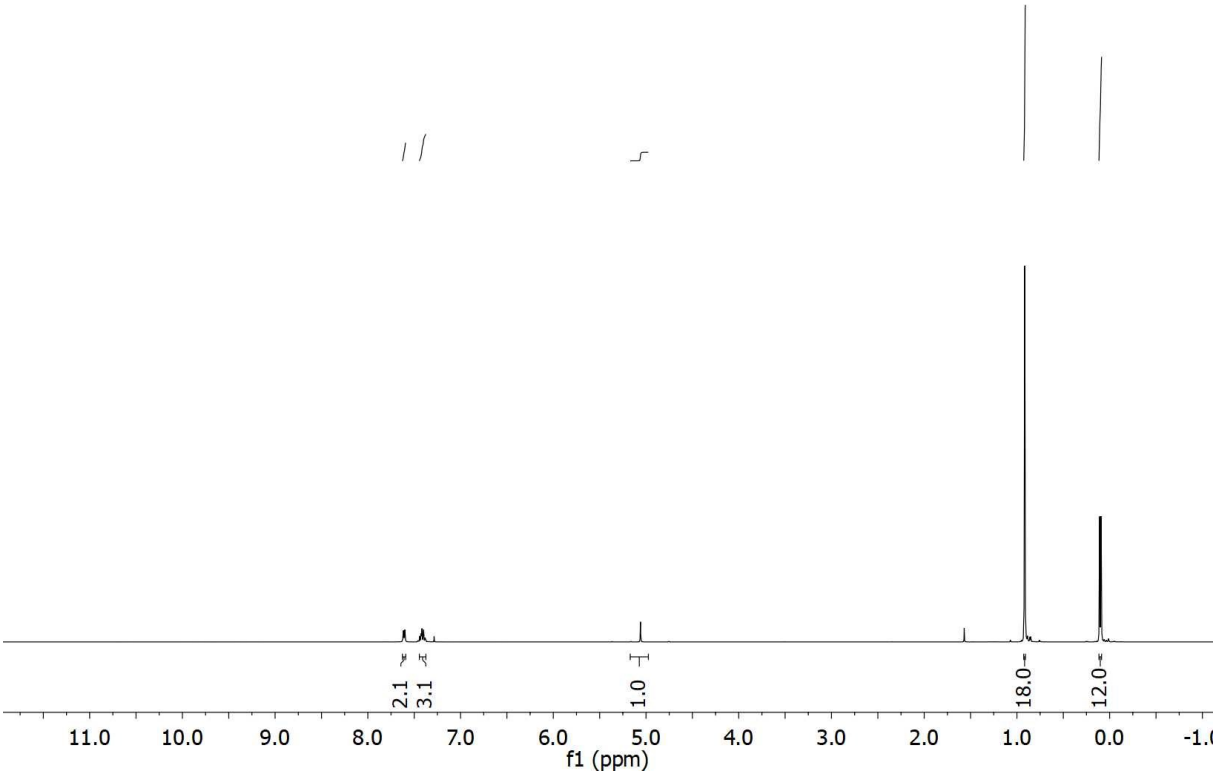


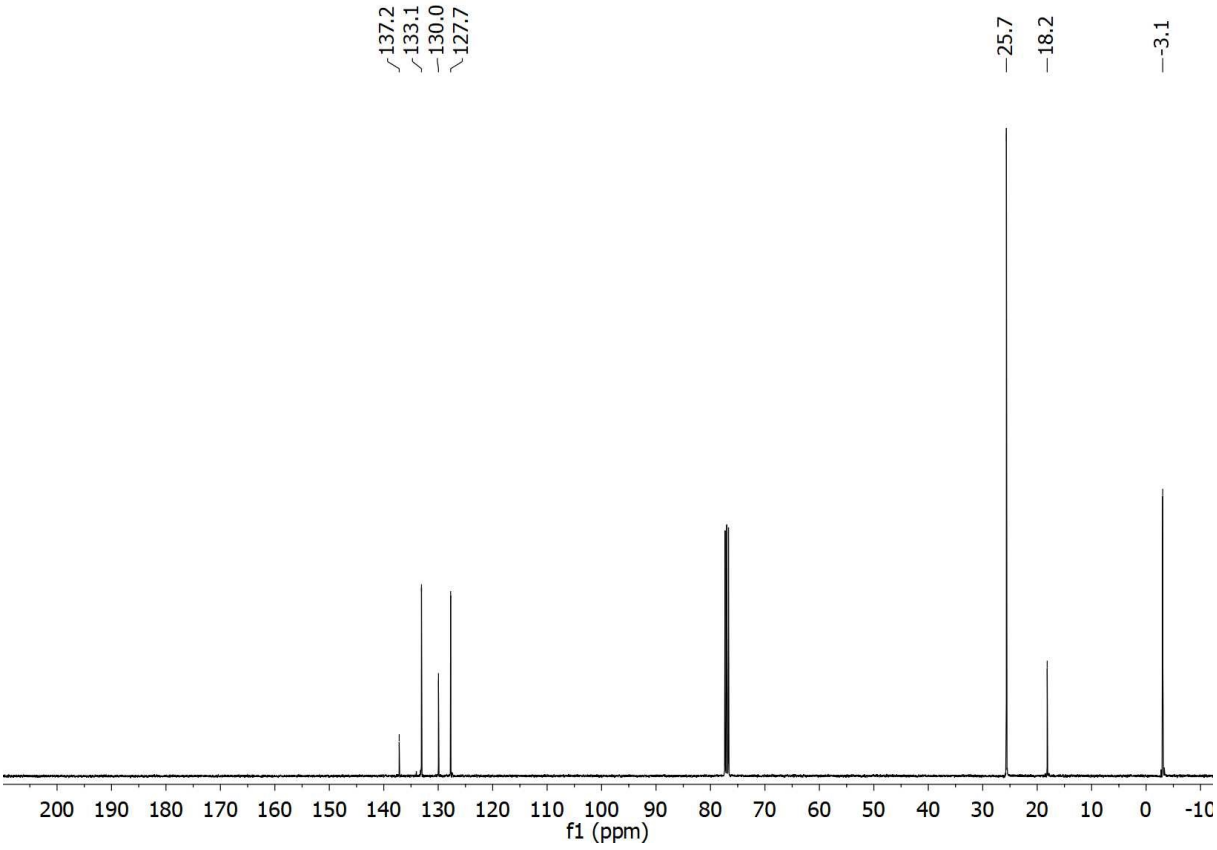


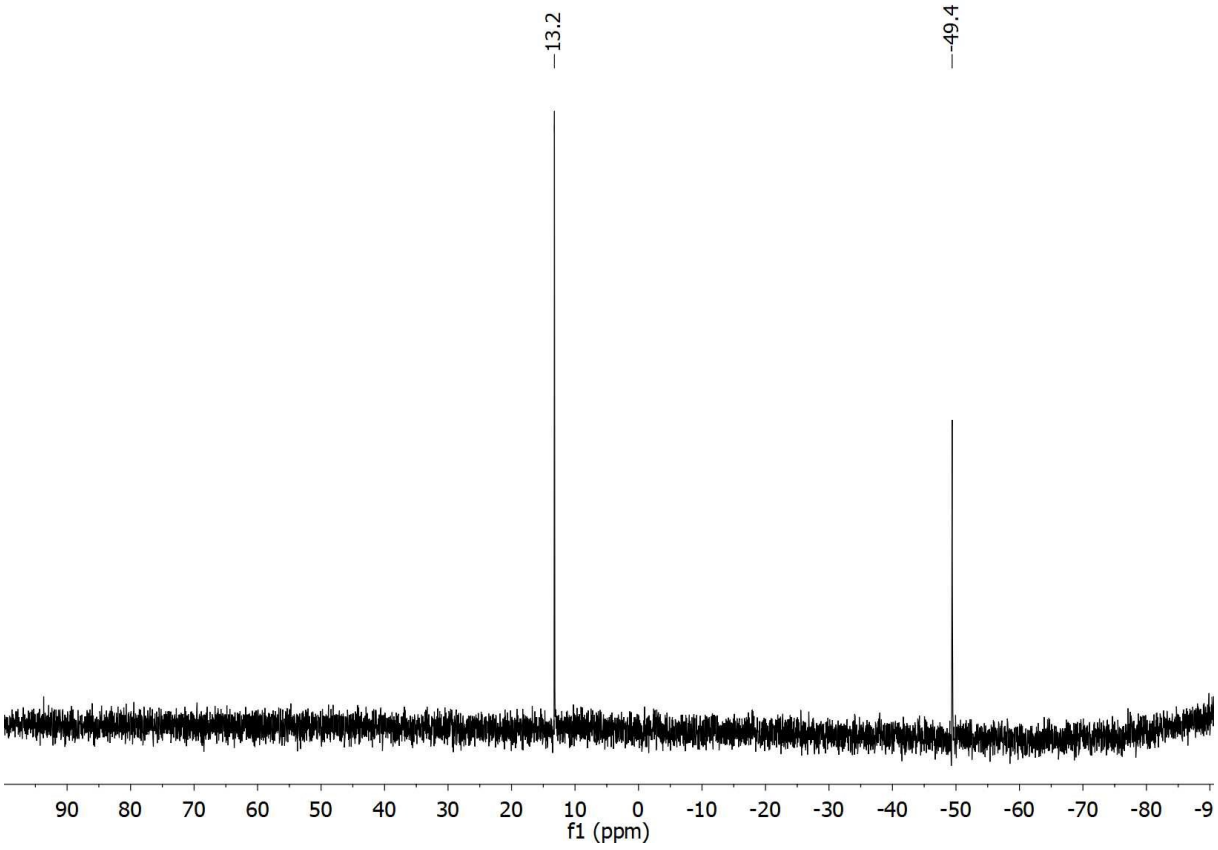

-9.7
-47.5
00 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -30
f1 (ppm)

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxane (**6a**).

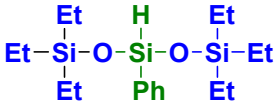


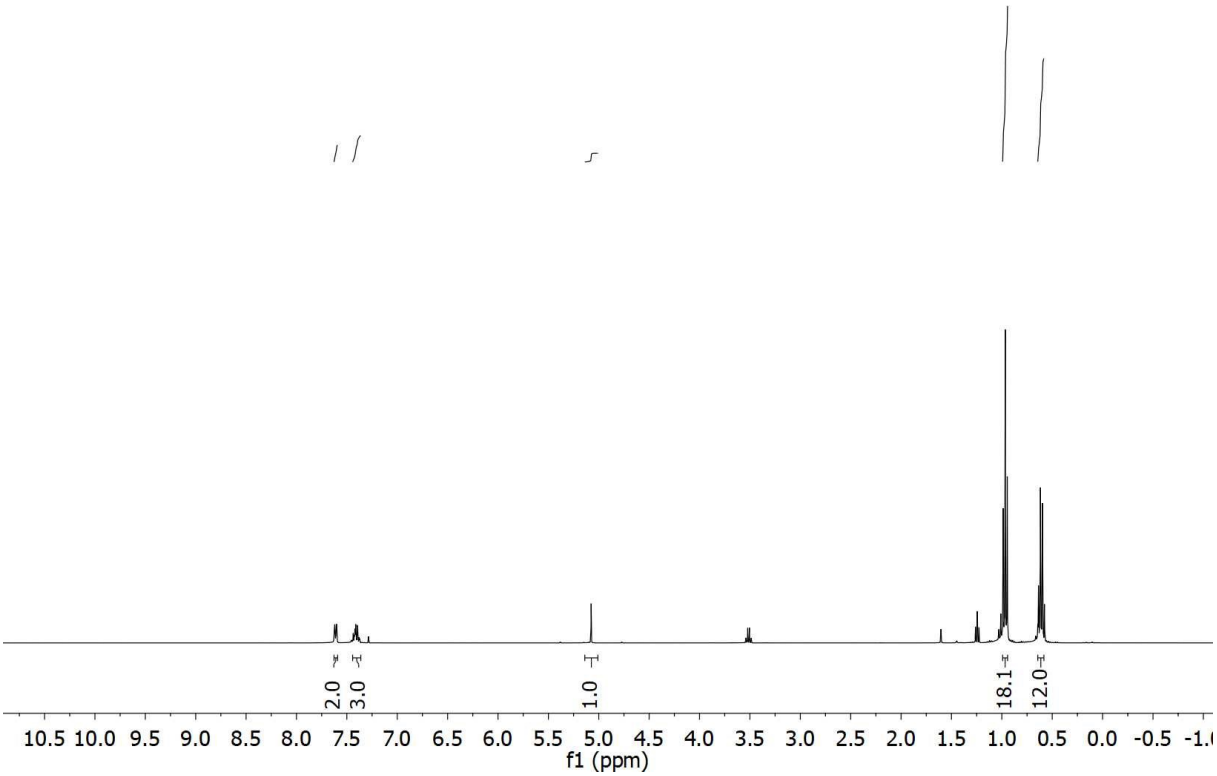


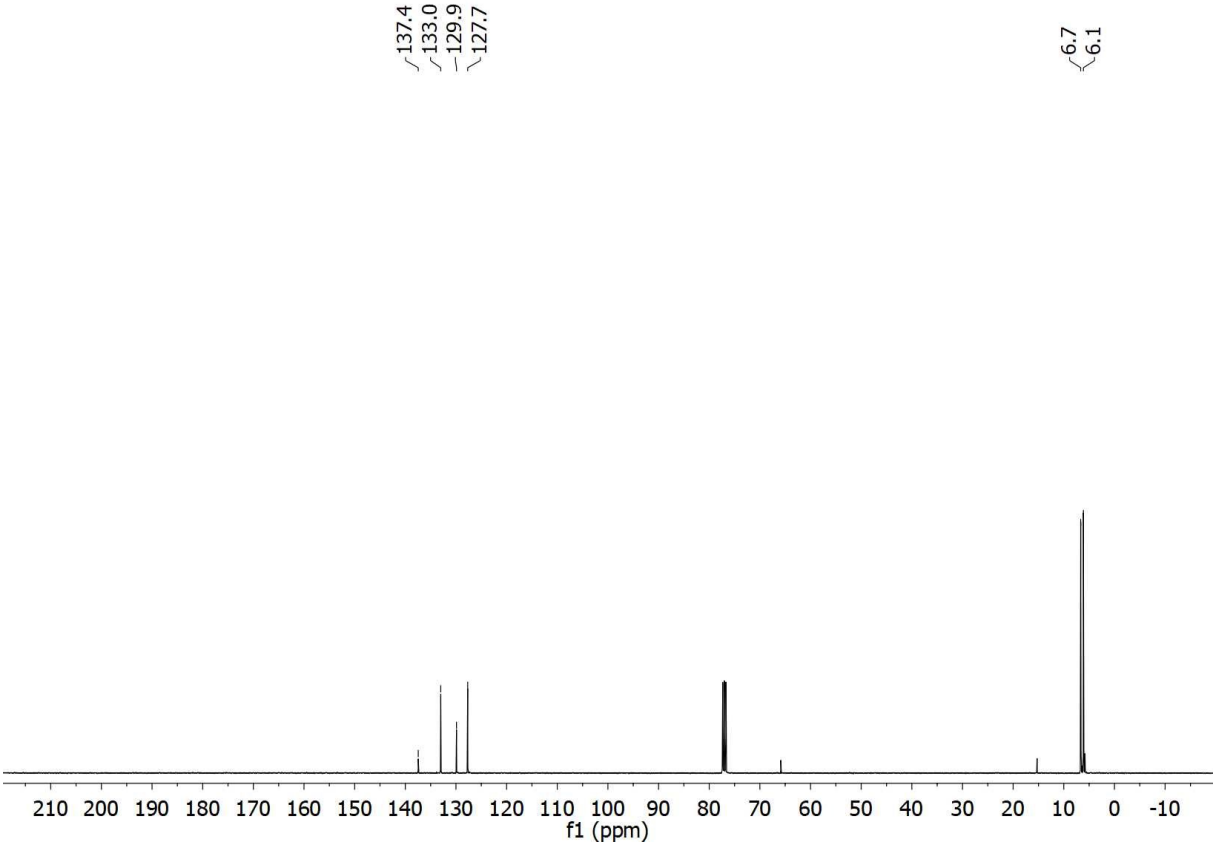


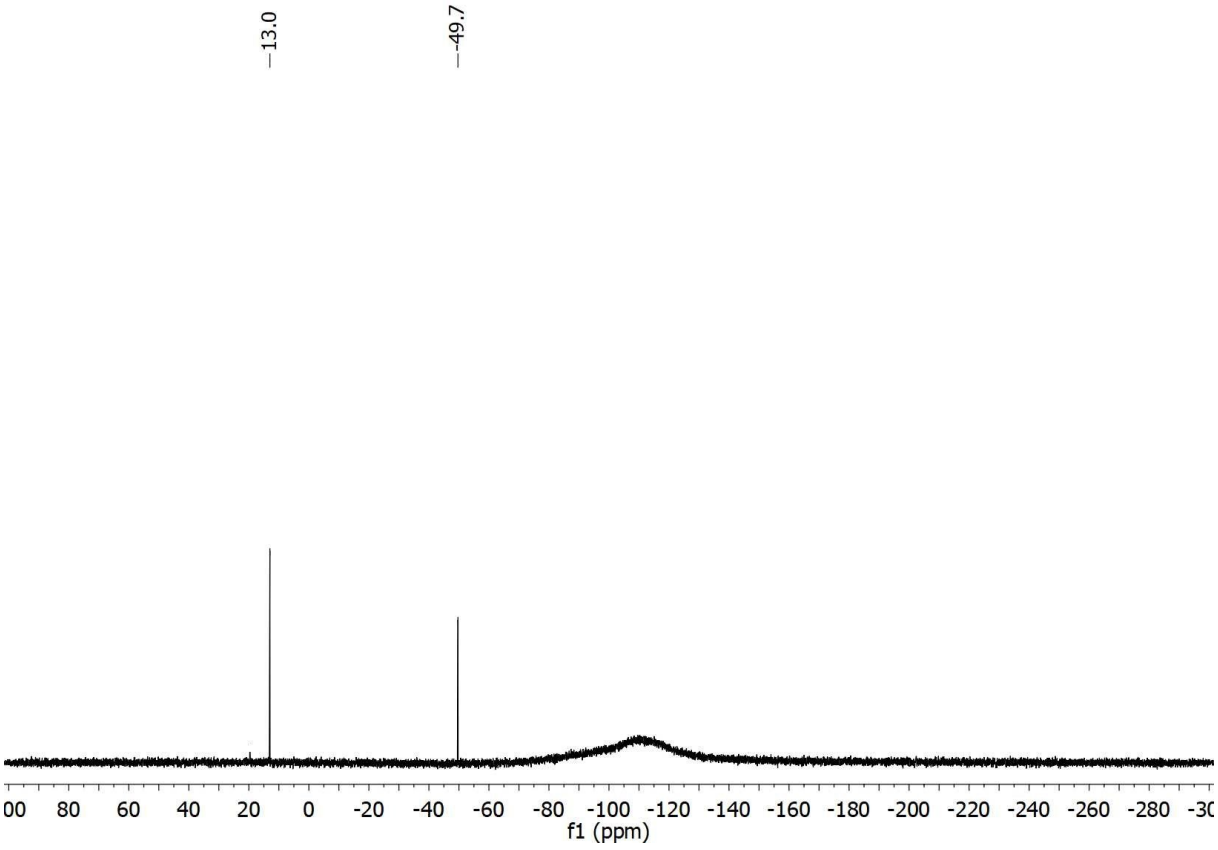

13.2
-49.4
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -9
f1 (ppm)

1,1,1,5,5,5-Hexaethyl-3-phenyltrisiloxane (**6b**).

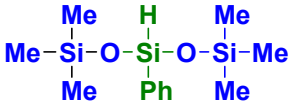


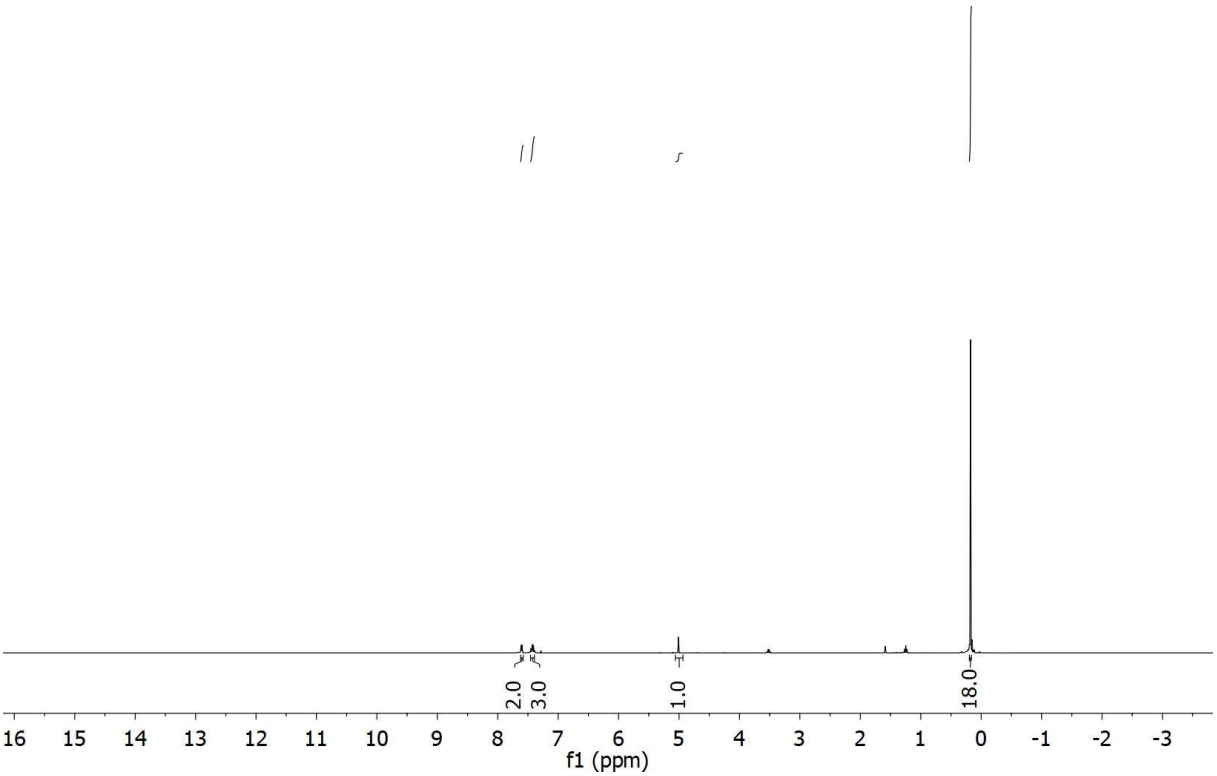


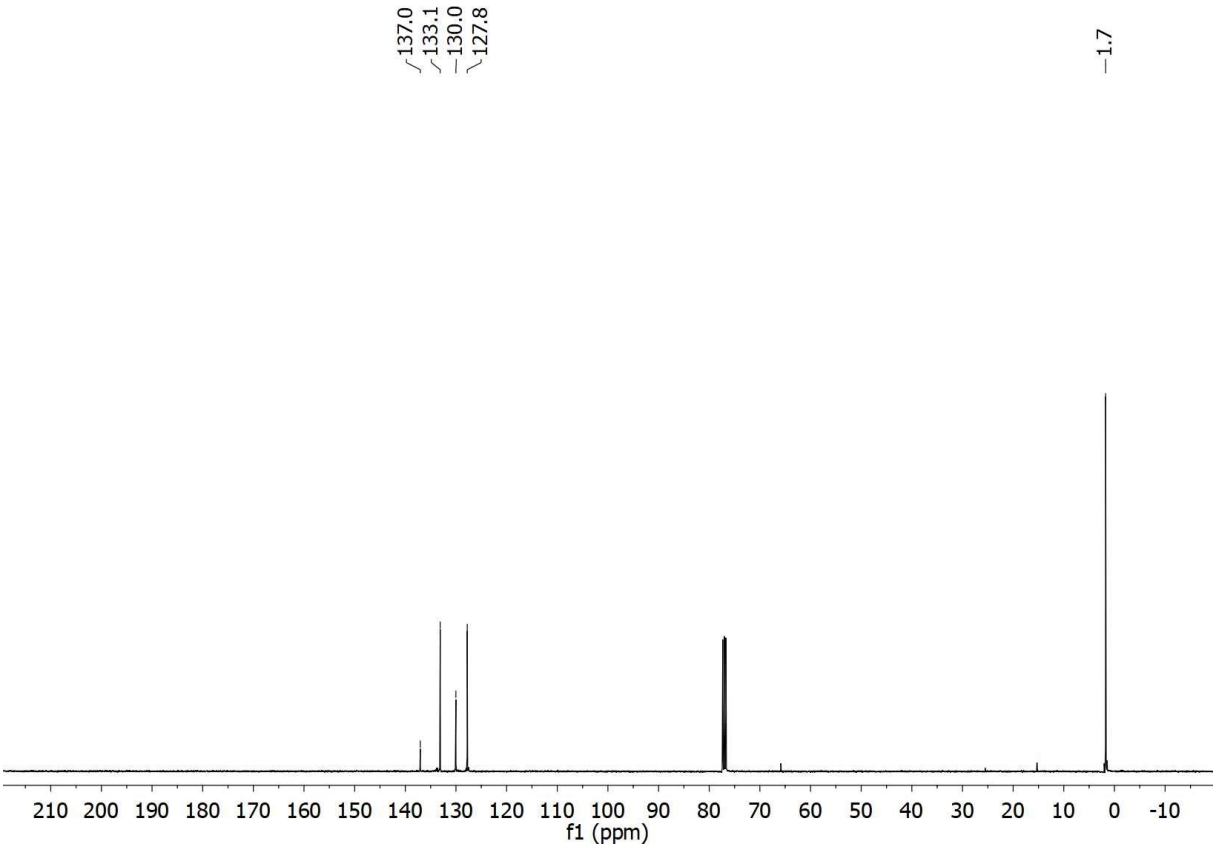


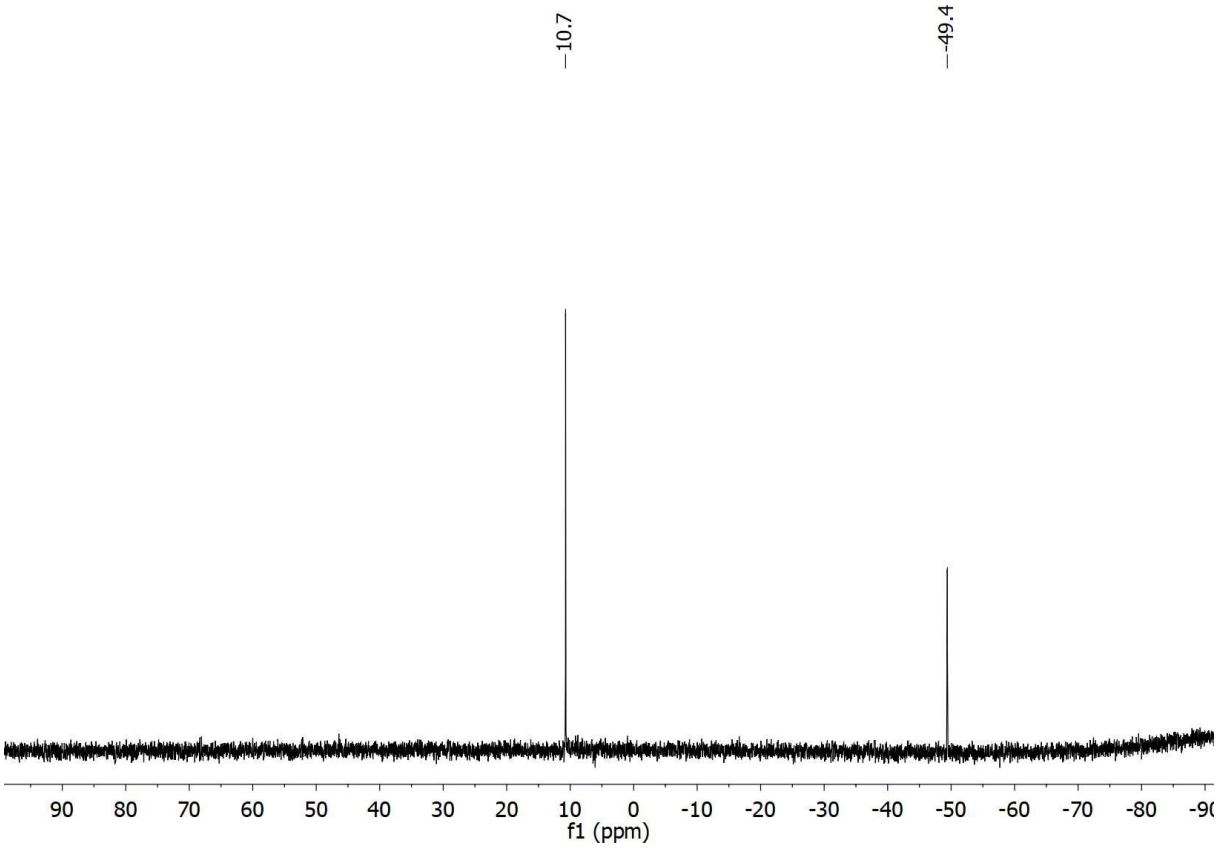

-13.0
-49.7
00 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -30
f1 (ppm)

1,1,1,5,5,5-Hexamethyl-3-phenyltrisiloxane (**6c**).

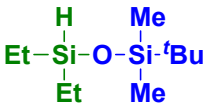


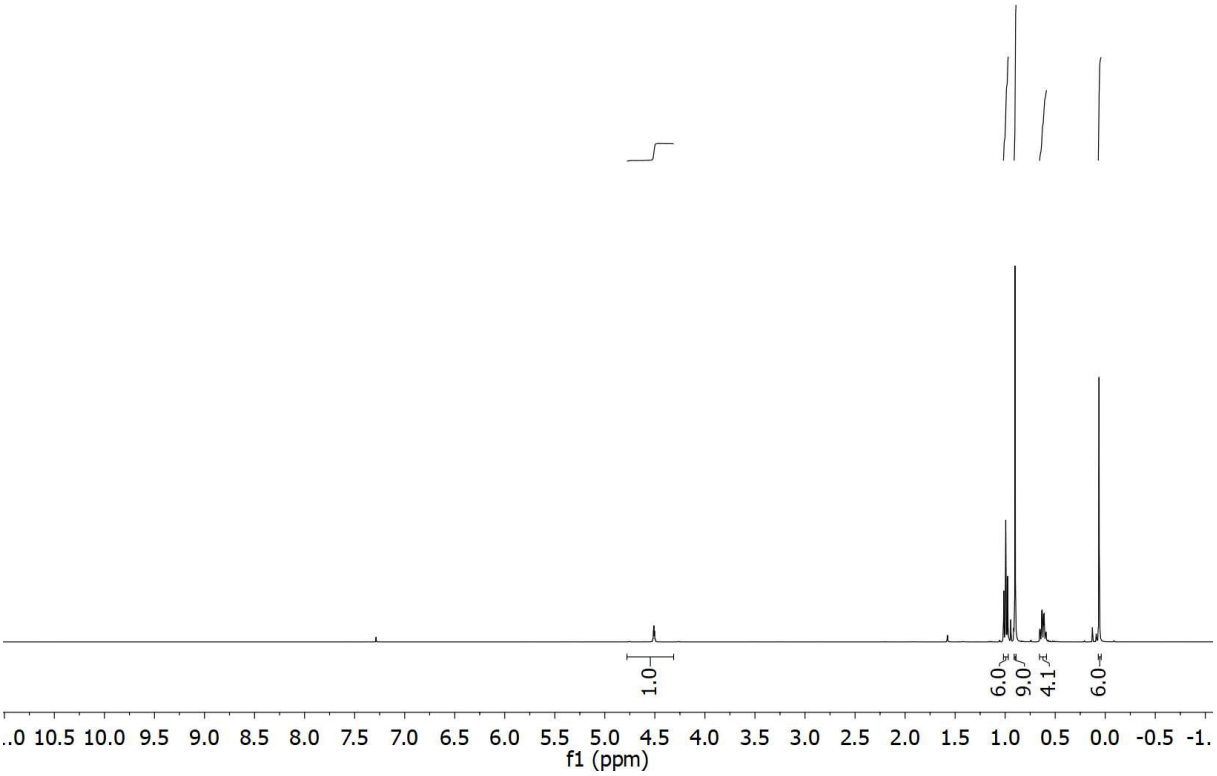


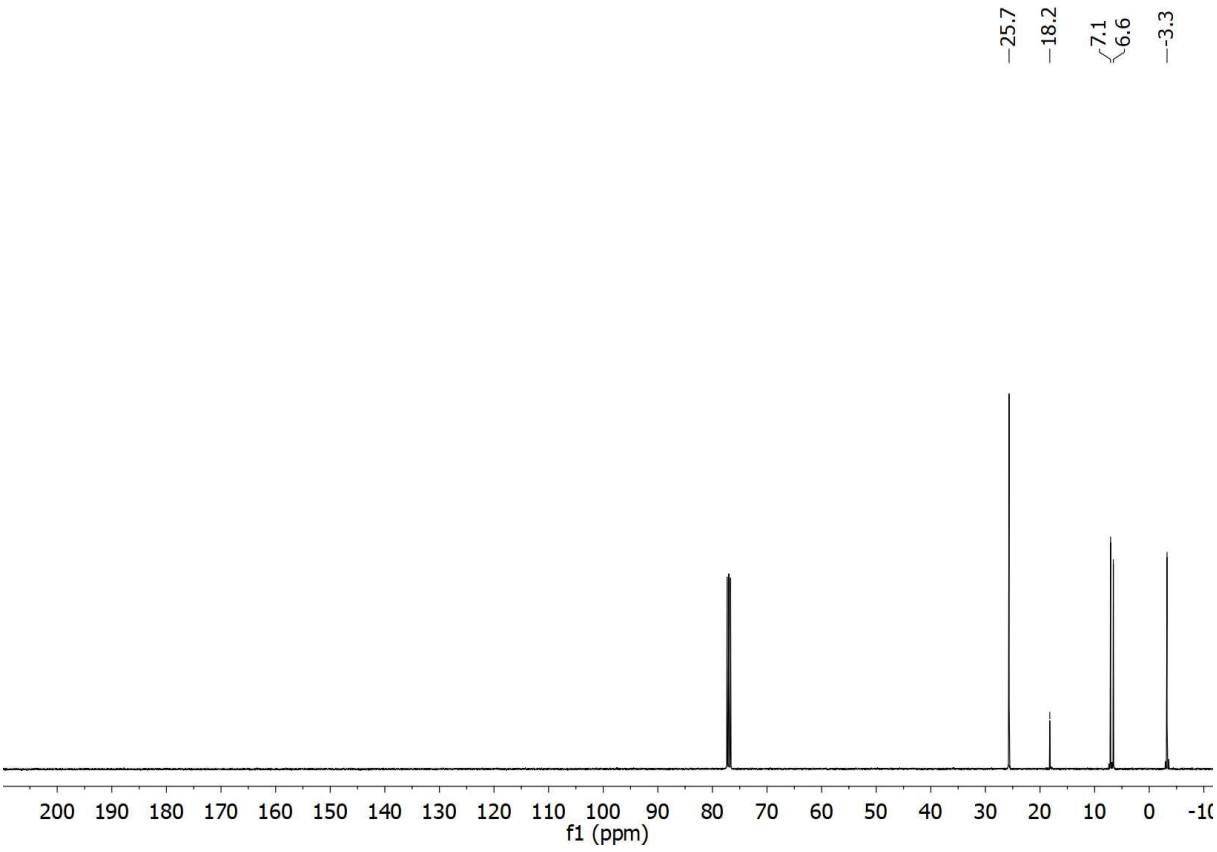


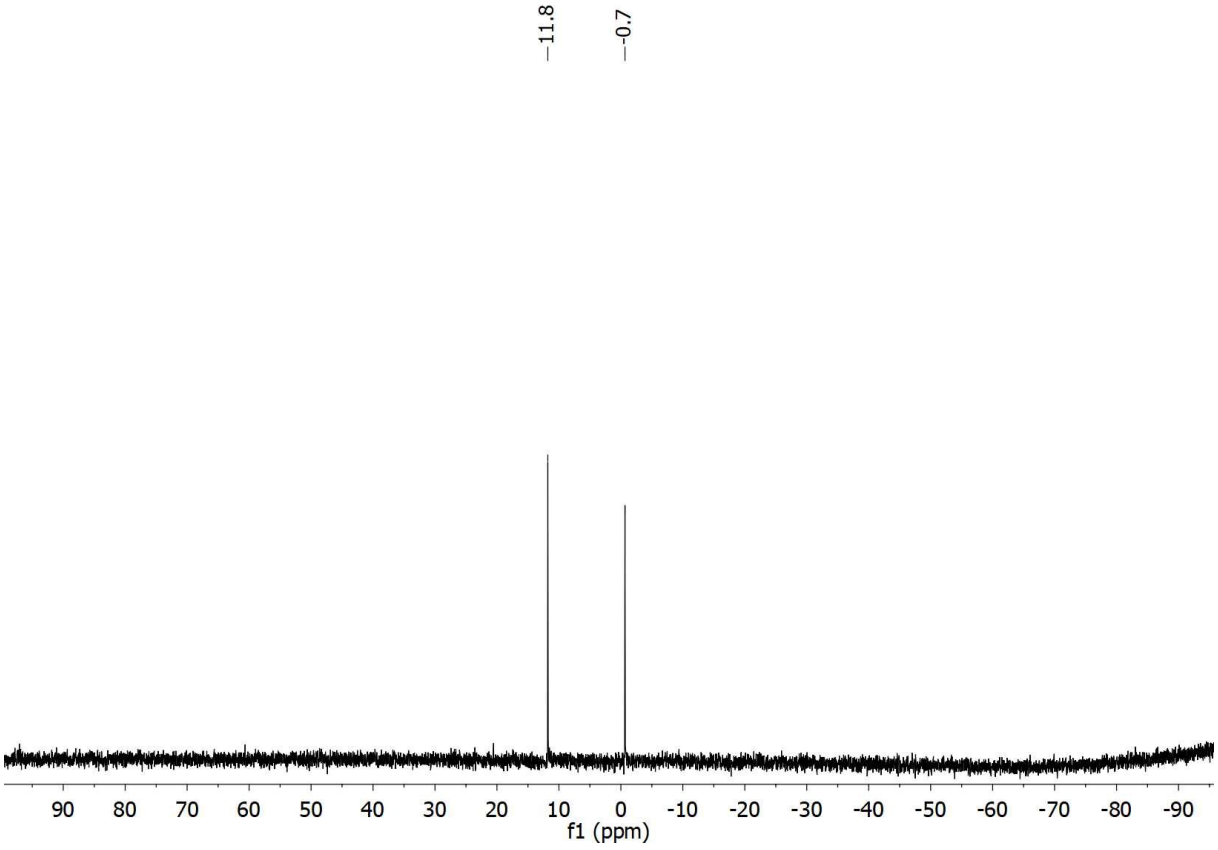

10.7
-49.4
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -9(
f1 (ppm)

1-*Tert*-butyl-3,3-diethyl-1,1-dimethyldisiloxane (**6d**).








11.8
-0.7
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)