Supporting Information

Streamlining Si-O Bond Formation Through Cobalt-Catalyzed Dehydrocoupling

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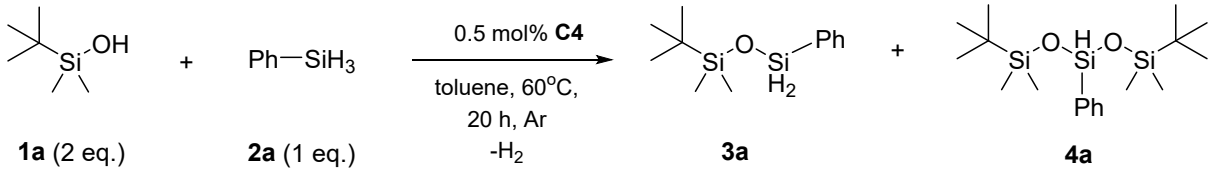
CONTENT:

GENERAL INFORMATION

Air- and moisture-sensitive reactions were carried out under an argon atmosphere using standard Schlenk techniques or a glove box. Solvents used for all experiments were purchased from Honeyweel or Sigma Aldrich (Merck), dried over calcium hydride (CaH_2) and purified by distillation. THF was additionally dried over sodium with a benzophenone system. Co-complexes were prepared following previously reported methods, using reagents purchased from Sigma Aldrich (Merck).[1] Commercially available hydrosilanes (e.g., phenylsilane, n-hexylsilane, methylphenylsilane, and diphenylsilane) as well as silanols (e.g., trimethylsilanol, triethylsilanol, dimethyl(phenyl)silanol, etc.) were purchased from Sigma Aldrich (Merck), ABCR GmBH, Ambeed, Apollo Scientific or Acros Organics, dried over calcium hydride and purified by distillation. Other hydrosilanes (e.g., methyl-p-tolylsilane) were synthesized from the corresponding chlorosilanes *through* reduction using a well-known procedure involving $LiAlH_4$ as reducing agent. The progress of reactions (conversion of silanols and hydrosilanes) was monitored by GC chromatography using Bruker Scion 460-GC and Agilent 5977B GC/MSD with Agilent 8860 GC System. The structures of products were determined by NMR spectroscopy and MS spectrometry. The ^{1}H NMR (400 or 600 MHz), ^{13}C NMR (101 or 151 MHz) and ^{29}Si NMR (79 or 119 MHz) spectra were recorded on Bruker Avance III HD NanoBay spectrometer, using benzene-d_6 (C_6D_6) or chloroform-d ($CDCl_3$) as the solvents. Deuterated solvents were purchased from Sigma Aldrich (Merck).

OPTIMIZATION OF REACTION CONDITIONS

Table 1. Optimization of SiO–H silylation.[a]



1a (2 eq.) **2a** (1 eq.) **3a** **4a**

Entry	Variation of standard condition	Conversion of **2a** [%] [b]	Selectivity [**3a**] : [**4a**] [%] [d]
1	no change	100 (98) [c]	0 : 100
2	no catalyst	0	-
3	1 equiv. of **1a** with **C6**	99	100 : 0
4	$CoCl_2$ instead of **C4**	5	-
5	**C1** instead of **C4**	82	12 : 88
6	**C2** instead of **C4**	80	9 : 91
7	**C3** instead of **C4**	84	10 : 90
8	**C5** instead of **C4**	96	0 : 100
9	**C6** instead of **C4**	98	0 : 100
10	0.25 mol% of **C4**	90	0 : 100
11	in 40°C	43	0 : 100
12	under air	34	0 :100
13	in tetrahydrofuran	98	0 : 100
14	in chlorobenzene	87	14 : 86
15	neat	89	0 : 100

[a] General reaction conditions: **1a** (4 mmol, 2 equiv.), **2a** 2 mmol, 1 equiv.), **C4** (0.5 mol%), under an argon atmosphere, 60°C, 20 h. [b] Conversion of **2a** determined by GC. [c] Isolated yield. [d] Selectivity of [mono]:[double] dehydrogenative coupling products determined by GC.

GENERAL SYNTHETIC PROCEDURES

The synthesis of compounds **3a-d**

To a 12mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C6** in toluene (0.5 mol%), phenylsilane (2 mmol, 1 equiv.), silanol (tert-butyldimethylsilanol, (2,3-dimethylbutan-2-yl)dimethylsilanol, triisopropylsilanol, and triisobutylsilanol; 3 mmol, 1.5 equiv.) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60˚C for 20 hours. After this time, the reaction mixture was dosed with 2 mL of pentane and left for 15 minutes to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product. The pure products were identified by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopies and mass spectrometry (MS).

The synthesis of compounds **4a-j**

To a 12 mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C4** in toluene (0.5 mol%), hydrosilane (phenylsilane, n-hexylsilane, p-tolylsilane, (2 mmol, 1 equiv.), silanol (*e.g.*, tert-butyldimethylsilanol, trimethylsilanol, tributylsilanol, *etc.*; 4 mmol, 2 equiv.) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60˚C for 20 hours. After this time, the reaction mixture was dosed with 2 mL of pentane and left for 15 minutes to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product. The pure products were identified by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopies and mass spectrometry (MS).

The synthesis of compounds **5a-k**

To a 12 mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C4** in toluene (0.5 mol%), hydrosilane (diphenylsilane and methylphenylsilane (2 mmol, 1 equiv.)), silanol (*e.g.*, tert-butyldimethylsilanol, trimethylsilanol, tributylsilanol, *etc.*; 2 mmol, 1 equiv.) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60˚C for 20 hours. After this time, the reaction mixture was dosed with 2 mL of pentane and left for 15 minutes to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product. The pure products were identified by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopies and mass spectrometry (MS).

The synthesis of compounds **6a,b,d-g**

To a 12 mL vial equipped with a magnetic stirring bar, 0.03 M solution of catalyst **C4** in toluene (0.5 mol%), hydrosilane (phenylsilane (2 mmol, 1 equiv.)), alcohol ((3-methyl-3-pentanol, cyclohexanol (4 mmol, 2 equiv.)) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60˚C for 20 hours. After this time, the reaction mixture was dosed with approximately 2 mL of

pentane and left for 15 min. to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product. The pure products were identified by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopies and mass spectrometry (MS).

The synthesis of compound **6c**

To a 12mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C4** in toluene (0.5 mol%), phenylsilane (2 mmol, 1 equiv.) and ethanol (6 mmol, 3 equiv.)) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60°C for 20 hours. After this time, the reaction mixture was dosed with approximately 2 mL of pentane and left for 15 min to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product. The pure products were identified by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopies and mass spectrometry (MS).

The synthesis of compounds **6h-i**

To a 12 mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C4** in C_6D_6 (0.05 mol%), phenylsilane (2 mmol, 1 equiv.) and amine (piperidine and morpholine (4 mmol, 2 equiv.)) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60°C for 20 hours. After this time, we added 1 equiv. of trimetoxybenzene as a chemical standard in a J-Young type NMR tube. Aminosilane conversions were measured by ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopy by integrating residual silane vs. product.

The synthesis of compound **7a**

To a 50 mL Schlenk flask equipped with a magnetic stirring bar, 1 mol% palladium on carbon catalyst, THF, H_2O (5 equiv.) and **4a** (1 equiv.) were added under an inert gas atmosphere. Subsequently, the reaction mixture was stirred at 65°C for 24 hours. After this time, the reaction mixture was filtered and the volatile residues were evaporated to yield a pure product. The pure product was identified by ^{1}H, ^{13}C and ^{29}Si NMR spectroscopies and mass spectrometry (MS).

The synthesis of compound **7b**

To a 50 mL round-bottom flask, TCCA (0.4 equiv.) in 1,2-DCE was added, followed by the dropwise addition of silane (**4a**) (1 equiv.) in 1,2-DCE. The reaction mixture was stirred at room temperature for 4 hours. Afterwards, the solution was filtered from the resulting precipitate and rinsed with DCE (3×2 mL). The combined DCE fractions were concentrated under vacuum. Subsequently, vinylmagnesium bromide in THF (3 equiv.) was added and the mixture was stirred at room temperature for 7 days. Afterwards, water and hexane were added, and the residue organic phase was purified via silica

gel column chromatography using hexane as the eluent. The pure product was identified by ^{1}H and ^{13}C NMR spectroscopies and mass spectrometry (MS).

The synthesis of compound **7c**

To a 50 mL Schlenk flask equipped with a magnetic stirring bar, 0.03 mol% Karstedt catalyst, toluene, **4b** (1 mmol, 1 equiv.), diethoxy(methyl)(vinyl)silane (12 mmol, 1.2 equiv.) were added under an inert gas atmosphere. Subsequently, the reaction mixture was stirred at 60^{o}C for 24 hours. After this time, the reaction mixture was rinsed with 3 x 2 mL of pentane. The solution was evaporated to yield a pure product. The pure product was identified by ^{1}H and ^{13}C NMR spectroscopies and mass spectrometry (MS).

The synthesis of compounds **7d-f**

To a 50 mL Schlenk flask equipped with a magnetic stirring bar, 0.06 mol% Karstedt catalyst, toluene, **4a** (1 mmol, 1 equiv.), diethoxy(methyl)(vinyl)silane, dec-1-ene or 2-((allyloxy)methyl)oxirane (15 mmol, 1.5 equiv.) were added under an inert gas atmosphere. Subsequently, the reaction mixture was stirred at 60˚C for 24 hours. After this time, the reaction mixture was rinsed with 3x2 mL of pentane. The solution was evaporated to yield a pure product. The pure products were identified by ^{1}H and ^{13}C NMR spectroscopies and mass spectrometry (MS).

Scaled-up synthesis of compound **4b**

To a 50 mL vial equipped with a magnetic stirring bar, 0.03 M solution of catalyst **C4** in toluene (0.5 mol%), hydrosilane (phenylsilane 0.0055 mol, 1 equiv.), silanol (trimethylosilanol; 0.011 mol, 2 equiv.) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60˚C for 20 hours. After this time, the reaction mixture was dosed with 10 mL of pentane and left for 15 minutes to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product (1.5339 g, 95%).

The synthesis of compound **4a** with TEMPO

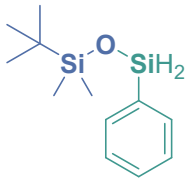
To a 12 mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C4** in toluene (0.5 mol%), phenylsilane (2 mmol, 1 equiv.), tert-butyldimethylsilanol (4 mmol, 2 equiv.), and TEMPO (2 mmol, 1 equiv.) were added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60˚C for 20 hours. After this time, the reaction mixture was dosed with 2 mL of pentane and left for 15 minutes to precipitate the catalyst. The resulting mixture was distilled using the trap-to-trap method yielding the pure product. The pure products were identified by mass spectrometry(MS).

The synthesis of compound **4a** with Quadra-Pure TU

To a 12 mL vial equipped with a magnetic stirring bar, 0.03M solution of catalyst **C4** in toluene (0.5 mol%), phenylsilane (2 mmol, 1 equiv.), tert-butyldimethylsilanol (4 mmol, 2 equiv.), and Quadra-Pure TU was added under an inert gas atmosphere (glove box). Subsequently, the reaction mixture was stirred at 60^{o}C for 20 hours. After this time, the reaction mixture was dosed with 2 mL of pentane and left for 15 minutes to precipitate the catalyst. The resulting mixture was checked by GC-MS. No product was observed in this case.

CHARACTERISATION DATA FOR ALL PRODUCTS

1-(Tert-butyl)-1,1-dimethyl-3-phenyldisiloxane 3a



1-(Tert-butyl)-1,1-dimethyl-3-phenyldisiloxane was obtained as a liquid in 77% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

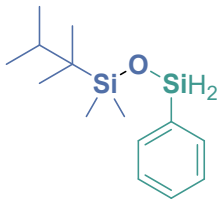
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.55 (dq, J = 5.0, 2.7 Hz, 2H), 7.20 – 7.07 (m, 3H), 5.30 (s, 2H), 0.86 (s, 9H), 0.00 (s, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 134.8, 133.9, 130.2, 128.0, 25.4, 18.1, -3.7.

^{29}Si NMR: (79) Hz, Benzene-d_6) δ 15.3, -29.7.

EI-MS m/z (rel. int.): 195 (3%), 181 (100), 165 (10), 151 (7), 135 (8), 121 (6).

1-(2,3-Dimethylbutan-2-yl)-1,1-dimethyl-3-phenyldisiloxane 3b



1-(2,3-Dimethylbutan-2-yl)-1,1-dimethyl-3-phenyldisiloxane was obtained as a liquid in 94% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

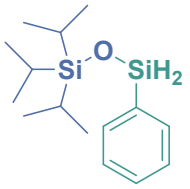
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.68 – 7.62 (m, 2H), 7.22 – 7.17 (m, 3H), 5.38 (s, 2H), 1.61 (p, J = 6.9 Hz, 1H), 0.92 – 0.83 (m, 12H), 0.13 (s, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 134.8, 133.9, 130.1, 127.9, 34.0, 25.0, 19.9, 18.3, -1.6.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 16.0, -29.8.

EI-MS m/z (rel. int.): 195 (5%), 181 (100%), 151 (4), 121 (10), 107 (5), 84 (30).

1,1,1-Triisopropyl-3-phenyldisiloxane 3c



1,1,1-Triisopropyl-3-phenyldisiloxane was obtained as a liquid in 73% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

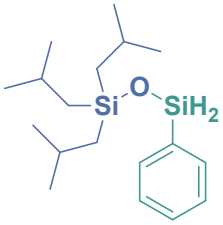
^{1}H NMR: (400 MHz, Benzene-d_6) δ 77.67 (dq, J = 5.7, 3.5, 2.5 Hz, 2H), 7.17 (m, 3H), 5.46 (s, 2H), 1.32 – 0.79 (m, 21H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 135.4, 134.3, 130.5, 128.3, 17.9, 12.9.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 11.4, -29.0.

EI-MS m/z (rel. int.): 265 (41%), 244 (2), 209 (53), 167 (12), 153 (100).

1,1,1-Triisobutyl-3-phenyldisiloxane 3d



1,1,1-Triisobutyl-3-phenyldisiloxane was obtained as a liquid in 70% yield. The title compound was previously unknown.

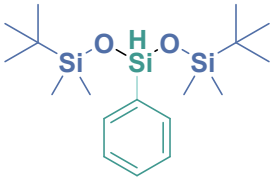
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.49 – 7.43 (m, 2H), 6.99 – 6.95 (m, 3H), 5.19 (s, 2H), 1.71 – 1.63 (m, 3H), 0.79 – 0.77 (m, 18H), 0.45 (d, J = 7.0 Hz, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 134.9, 134.0, 133.2, 130.1, 27.0, 26.2, 24.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 10.2, -30.8.

EI-MS m/z (rel. int.): 237 (100%), 209 (30), 195 (16), 181 (47), 167 (51), 151 (20).

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxane 4a



1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxane was obtained as a liquid in 98% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

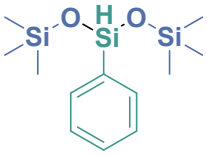
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.76 – 7.72 (m, 2H), 7.26 – 7.19 (m, 3H), 5.41 (s, 1H), 1.03 – 0.91 (m, 18H), 0.14 (d, J = 6.7 Hz, 12H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.4, 133.4, 130.5, 128.2, 25.8, 18.4, -2.8.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 13.8, -48.0.

EI-MS m/z (rel. int.): 311 (75%), 269 (54), 239 (21), 179 (20), 135 (100), 73 (46).

1,1,1,5,5,5-Hexamethyl-3-phenyltrisiloxane 4b



1,1,1,5,5,5-Hexamethyl-3-phenyltrisiloxane was obtained as a liquid in 90% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

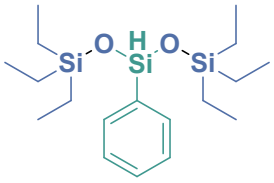
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.77 – 7.72 (m, 2H), 7.24 – 7.20 (m, 3H), 5.40 (s, 1H), 0.17 (s, 18H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.3, 133.4, 130.5, 128.2, 1.8.

^{29}Si NMR: (79 MHz, MHz, Benzene-d_6)) δ 10.7, -49.3.

EI-MS m/z (rel. int.): 283 (8%), 269 (74), 251 (5), 191 (32), 135 (100), 127 (30).

1,1,1,5,5,5-Hexaethyl-3-phenyltrisiloxane 4c



1,1,1,5,5,5-Hexaethyl-3-phenyltrisiloxane was obtained as a liquid in 93% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

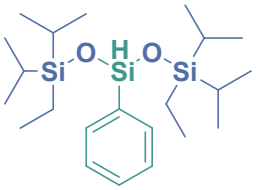
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.87 – 7.66 (m, 2H), 7.28 – 7.17 (m, 3H), 5.42 (s, 1H), 1.00 (t, J = 7.9 Hz, 18H), 0.62 (q, J = 7.8 Hz, 12H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.4, 132.9, 130.1, 127.8, 6.6, 6.1.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 12.9, -48.8.

EI-MS m/z (rel. int.): 339 (100%), 311(10), 283 (5), 253 (2), 197 (4), 135 (15), 107 (18).

1,5-Diethyl-1,1,5,5-tetraisopropyl-3-phenyltrisiloxane 4d



1,5-Diethyl-1,1,5,5-tetraisopropyl-3-phenyltrisiloxane was obtained as a liquid in 86% yield. The title compound was previously unknown.

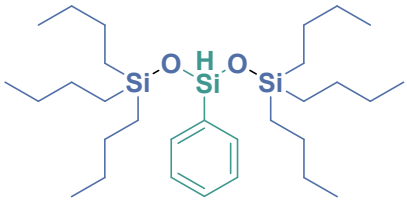
^{1}H NMR: (401 MHz, Benzene-d_6) δ 7.81 – 7.77 (m, 2H), 7.26 – 7.19 (m, 3H), 5.49 (d, J = 1.4 Hz, 1H), 1.13 – 0.98 (m, 34H), 0.70 – 0.63 (m, 4H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.8, 133.2, 130.4, 128.1, 17.6, 13.4, 7.4, 4.1.

^{29}Si NMR: (79 MHz, MHz, Benzene-d_6)) δ δ 11.9, -48.5.

EI-MS m/z (rel. int.): 395 (8%), 381 (100), 353 (7), 339 (9), 237 (6), 141 (30), 121 (31).

1,1,1,5,5,5-Hexabutyl-3-phenyltrisiloxane 4e



1,1,1,5,5,5-Hexabutyl-3-phenyltrisiloxane was obtained as a liquid in 80% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

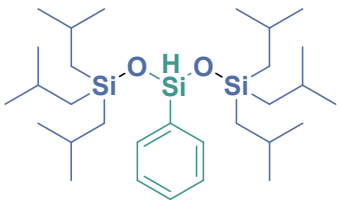
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.86 – 7.70 (m, 2H), 7.28 – 7.16 (m, 3H), 5.47 (s, 1H), 1.50 – 1.34 (m, 24H), 0.93 (t, J = 7.1 Hz, 18H), 0.76 – 0.69 (m, 12H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.5, 133.0, 130.1, 127.8, 26.6, 25.5, 15.3, 13.7.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 10.3, -48.9.

EI-MS m/z (rel. int.): 409 (100%), 367 (30), 325 (27), 283 (16), 241 (15), 199 (46), 106 (27).

1,1,1,5,5,5-Hexaisobutyl-3-phenyltrisiloxane 4f



1,1,1,5,5,5-Hexaisobutyl-3-phenyltrisiloxane was obtained as a liquid in 82% yield. The title compound was previously unknown.

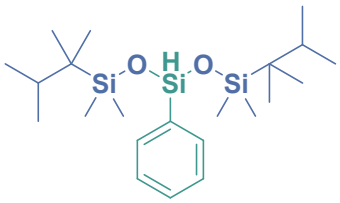
^{1}H NMR: (401 MHz, Benzene-d_6) δ 7.83 – 7.74 (m, 2H), 7.26 – 7.19 (m, 3H), 5.46 (d, *J* = 0.4 Hz, 1H), 1.95 (dq, *J* = 13.3, 6.7 Hz, 6H), 1.05 (dd, *J* = 6.6, 0.5 Hz, 36H), 0.79 – 0.71 (m, 12H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.8, 133.4, 130.4, 128.1, 27.8, 26.7, 24.6.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 8.1, -49.6.

EI-MS m/z (rel. int.): 479 (59%), 423 (44), 367 (40), 311 (40), 255 (37), 211 (39), 199 (100), 113 (45).

1,5-Bis(2,3-dimethylbutan-2-yl)-1,1,5,5-tetramethyl-3-phenyltrisiloxane 4g



1,5-Bis(2,3-Dimethylbutan-2-yl)-1,1,5,5-tetramethyl-3-phenyltrisiloxane was obtained as a liquid in 94% yield. The title compound was previously unknown.

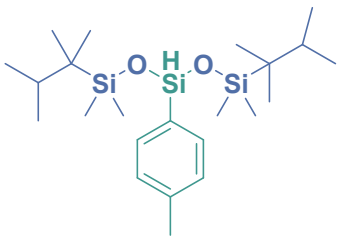
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.80 – 7.73 (m, 2H), 7.29 – 7.19 (m, 3H), 5.40 (d, *J* = 0.9 Hz, 1H), 1.66 (ddd, *J* = 13.7, 7.3, 6.5 Hz, 2H), 1.01 – 0.93 (m, 8H), 0.92 (dd, *J* = 3.6, 0.9 Hz, 16H), 0.27 – 0.14 (m, 12H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.5, 133.3, 130.4, 128.2, 34.3, 25.2, 20.3, 20.3, 18.8, -0.5.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 13.7, -48.3.

EI-MS m/z (rel. int.): 339 (29%), 297 (5), 269 (100), 255 (39), 239 (15), 193 (10), 135 (72).

1,5-Bis(2,3-dimethylbutan-2-yl)-1,1,5,5-tetramethyl-3-(p-tolyl)trisiloxane 4h



1,5-Bis(2,3-dimethylbutan-2-yl)-1,1,5,5-tetramethyl-3-(p-tolyl)trisiloxane was obtained as a liquid in 88% yield. The title compound was previously unknown.

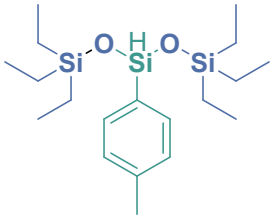
^{1}H NMR: (600 MHz, Benzene-d_6) δ 7.68 – 7.64 (m, 2H), 7.09 – 7.05 (m, 2H), 5.39 (s, 1H), 2.11 – 2.09 (m, 3H), 1.71 – 1.63 (m, 2H), 0.98 – 0.90 (m, 24H), 0.26 – 0.18 (m, 12H).

^{13}C NMR: (151 MHz, Benzene-d_6) δ 140.1, 134.1, 133.5, 129.0, 34.4, 25.3, 21.5, 20.4, 18.8, -0.5.

^{29}Si NMR: (119 MHz, Benzene-d_6) δ 13.0, -48.3.

EI-MS m/z (rel. int.): 353 (24%), 311 (2), 283 (100), 269 (32), 253 (14), 194 (52), 121 (7).

1,1,1,5,5,5-Hexaethyl-3-(p-tolyl)trisiloxane 4i



1,1,1,5,5,5-Hexaethyl-3-(p-tolyl)trisiloxane was obtained as a liquid in 84% yield. The title compound was previously unknown.

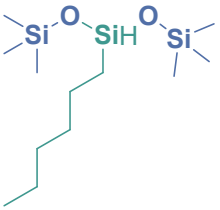
^{1}H NMR: (600 MHz, Benzene-d_6) δ 7.72 – 7.69 (m, 2H), 7.10 – 7.07 (m, 2H), 5.46 (s, 1H), 2.10 – 2.09 (m, 3H), 1.07 – 0.99 (m, 18H), 0.69 – 0.63 (m, 12H).

^{13}C NMR: (151 MHz, Benzene-d_6) δ 140.2, 134.3, 133.4, 129.0, 21.5, 7.0, 6.6.

^{29}Si NMR: (119 MHz, Benzene-d_6) δ 12.7, -48.3.

EI-MS m/z (rel. int.): 353 (100%), 323 (7), 267 (5), 162 (10), 149 (24), 134 (20), 121 (42).

3-Hexyl-1,1,1,5,5,5-hexamethyltrisiloxane 4j



3-Hexyl-1,1,1,5,5,5-hexamethyltrisiloxane was obtained as a liquid in 71% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

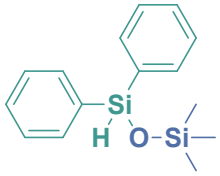
^{1}H NMR: (400 MHz, Benzene-d_6) δ 4.98 (t, J = 1.2 Hz, 1H), 1.55 – 1.44 (m, 2H), 1.38 – 1.22 (m, 6H), 0.90 – 0.85 (m, 3H), 0.69 – 0.62 (m, 2H), 0.18 (s, 18H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 32.6, 31.6, 22.6, 22.2, 17.5, 13.9, 1.4.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 8.9, -36.0.

EI-MS m/z (rel. int.): 291 (6%), 277 (45), 207 (100), 193 (80), 133 (10), 73 (71).

1,1,1-Trimethyl-3,3-diphenyldisiloxane 5a



1,1,1-Trimethyl-3,3-diphenyldisiloxane was obtained as a liquid in 91% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

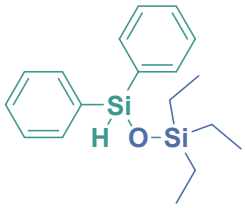
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.76 – 7.59 (m, 4H), 7.23 – 7.15 (m, 6H), 5.79 (s, 1H), 0.11 (s, 9H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 135.9, 134.2, 130.0, 127.9, 1.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 11.9, -22.4.

EI-MS m/z (rel. int.): 271 (3%), 257 (21), 241 (3), 194 (47), 179 (100), 135 (42).

1,1,1-Triethyl-3,3-diphenyldisiloxane 5b



1,1,1-Triethyl-3,3-diphenyldisiloxane was obtained as a liquid in 95% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

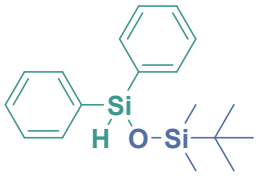
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.75 – 7.65 (m, 4H), 7.23 – 7.16 (m, 6H), 5.84 (s, 1H), 0.95 (t, J = 7.9 Hz, 9H), 0.58 (q, J = 7.8 Hz, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.2, 134.2, 130.0, 127.9, 6.6, 6.1.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 14.3, -22.1.

EI-MS m/z (rel. int.): 285 (100%), 257 (22), 229 (10), 207 (8), 183 (18), 151 (68).

1-(Tert-butyl)-1,1-dimethyl-3,3-diphenyldisiloxane 5c



1-(Tert-butyl)-1,1-dimethyl-3,3-diphenyldisiloxane was obtained as a liquid in 92% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

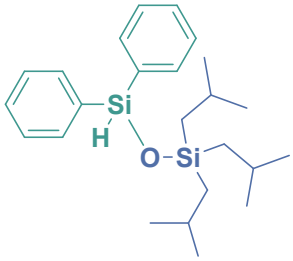
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.71 – 7.63 (m, 4H), 7.18 (ddt, J = 4.1, 3.3, 1.4 Hz, 6H), 5.81 (s, 1H), 0.93 (s, 9H), 0.08 (s, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.0, 134.2, 130.0, 127.9, 25.5, 18.2, -3.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 14.7, -21.8.

EI-MS m/z (rel. int.): 257 (100%), 241 (5), 195 (10), 179 (72), 165 (12) 135 (18).

1,1,1-Triisobutyl-3,3-diphenyldisiloxane 5d



1,1,1-Triisobutyl-3,3-diphenyldisiloxane was obtained as a liquid in 73% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

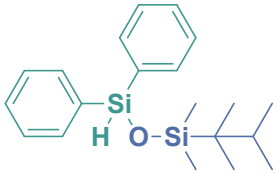
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.77 – 7.68 (m, 4H), 7.22 – 7.16 (m, 6H), 5.85 (s, 1H), 1.89 (dh, J = 13.3, 6.6 Hz, 3H), 0.97 (d, J = 6.6 Hz, 18H), 0.72 (d, J = 6.9 Hz, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.1, 134.3, 130.0, 127.9, 27.4, 26.3, 24.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 9.7, -22.4.

EI-MS m/z (rel. int.): 341 (24%), 320 (6), 285 (82), 229 (100), 183 (10), 151 (50).

1-(2,3-Dimethylbutan-2-yl)-1,1-dimethyl-3,3-diphenyldisiloxane 5e



1-(2,3-Dimethylbutan-2-yl)-1,1-dimethyl-3,3-diphenyldisiloxane was obtained as a liquid in 83% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

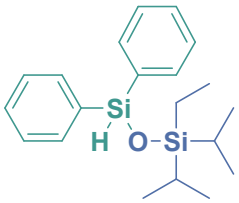
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.71 – 7.65 (m, 4H), 7.21 – 7.16 (m, 6H), 5.81 (s, 1H), 1.62 (hept, J = 6.9 Hz, 1H), 0.94 – 0.83 (m, 12H), 0.14 (s, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.0, 134.3, 130.0, 127.9, 34.0, 25.1, 20.0, 18.4, -1.1.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 14.9, -22.0.

EI-MS m/z (rel. int.): 257 (100%), 241 (7), 195 (70), 155 (5), 135 (20), 121 (16).

1-Ethyl-1,1-diisopropyl-3,3-diphenyldisiloxane 5f



1-Ethyl-1,1-diisopropyl-3,3-diphenyldisiloxane was obtained as a liquid in 87% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

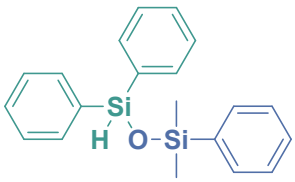
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.76 – 7.67 (m, 4H), 7.21 – 7.16 (m, 6H), 5.87 (s, 1H), 1.08 – 0.90 (m, 17H), 0.66 – 0.57 (m, 2H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.3, 134.2, 130.0, 127.9, 17.3, 13.1, 7.09, 3.7.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 12.6, -21.8.

EI-MS m/z (rel. int.): 299 (100%), 271 (15), 227 (8), 183 (12), 165 (41), 151 (37).

1,1-Dimethyl-1,3,3-triphenyldisiloxane 5g



1,1-Dimethyl-1,3,3-triphenyldisiloxane was obtained as a liquid in 94% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

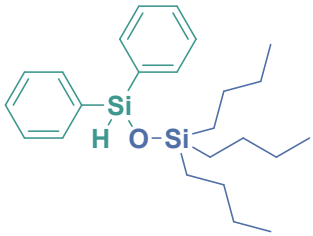
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.67 – 7.51 (m, 6H), 7.20 – 7.14 (m, 9H), 5.85 (s, 1H), 0.33 (s, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 138.8, 135.7, 134.3, 133.1, 130.0, 129.4, 127.9, 127.8, -0.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 2.0, -21.4.

EI-MS m/z (rel. int.): 334 (2%), 319 (35), 256 (12), 241 (100), 197 (41), 178 (50).

1,1,1-Tributyl-3,3-diphenyldisiloxane 5h



1,1,1-tributyl-3,3-diphenyldisiloxane was obtained as a liquid in 76% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

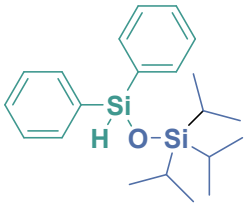
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.73 – 7.67 (m, 4H), 7.25 – 7.16 (m, 6H), 5.86 (s, 1H), 1.44 – 1.27 (m, 12H), 0.87 (t, J = 7.1 Hz, 9H), 0.71 – 0.63 (m, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.2, 134.3, 130.0, 127.9, 26.5, 25.4, 15.1, 13.6.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 11.8, -22.2.

EI-MS m/z (rel. int.): 313 (87%), 278 (10), 271 (67), 229 (100), 193 (5), 165 (10), 151 (73).

1,1,1-Triisopropyl-3,3-diphenyldisiloxane 5i



1,1,1-Triisopropyl-3,3-diphenyldisiloxane was obtained as a liquid in 85% yield. The title compound was known in the literature,[3] and all spectroscopic data are in agreement.

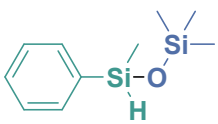
^{1}H NMR: (600 MHz, Benzene-d_6) δ 7.77 – 7.68 (m, 4H), 7.23 – 7.18 (m, 6H), 5.91 (s, 1H), 1.09 – 1.06 (m, 18H).

^{13}C NMR: (151 MHz, Benzene-d_6) δ 136.7, 134.6, 130.4, 128.3, 18.0, 13.1.

^{29}Si NMR: (119 MHz, Benzene-d_6) δ 10.6, -21.8.

EI-MS m/z (rel. int.): 313 (100%), 285 (10), 257 (11), 243 (17), 179 (52), 165 (49).

1,1,1,3-Tetramethyl-3-phenyldisiloxane 5j



1,1,1,3-Tetramethyl-3-phenyldisiloxane was obtained as a liquid in 92% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

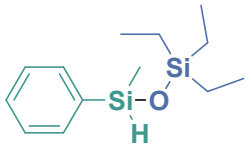
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.62 – 7.58 (m, 2H), 7.24 – 7.18 (m, 3H), 5.37 (q, *J* = 2.9 Hz, 1H), 0.36 (d, *J* = 2.9 Hz, 3H), 0.10 (s, 9H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.7, 133.3, 129.7, 127.9, 1.37, 0.5.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 10.6, -14.3.

EI-MS m/z (rel. int.): 210 (10%), 195 (100), 179 (15), 165 (7), 132 (47), 117 (6).

1,1,1-Triethyl-3-methyl-3-phenyldisiloxane 5k



1,1,1-Triethyl-3-methyl-3-phenyldisiloxane was obtained as a liquid in 81% yield. The title compound was known in the literature,[2] and all spectroscopic data are in agreement.

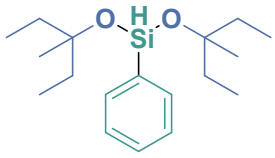
^{1}H NMR: (400 MHz, Benzene-d_6δ 7.70 – 7.54 (m, 2H), 7.27 – 7.17 (m, 3H), 5.41 (q, *J* = 2.8 Hz, 1H), 0.96 (t, *J* = 8.0 Hz, 9H), 0.56 (q, *J* = 8.0 Hz, 6H), 0.39 (d, *J* = 2.8 Hz, 3H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.9, 133.3, 129.7, 127.8, 6.6, 6.1, -0.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ 13.0, -14.2.

EI-MS m/z (rel. int.): 223 (100%), 195 (39), 179 (7), 167 (46), 151 (7), 121 (30).

Bis((3-methylpentan-3-yl)oxy)(phenyl)silane 6a



Bis((3-methylpentan-3-yl)oxy)(phenyl)silane was obtained as a liquid in 90% yield The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

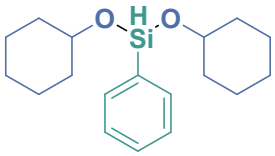
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.87 – 7.77 (m, 2H), 7.26 – 7.17 (m, 3H), 5.47 (s, 1H), 1.56 (p, *J* = 7.6 Hz, 8H), 1.23 (s, 6H), 0.88 (dt, *J* = 11.5, 7.5 Hz, 12H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 138.1, 134.0, 133.6, 129.7, 78.0, 34.0, 26.1, 8.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -47.3.

EI-MS m/z (rel. int.): 293 (3%), 279 (41), 195 (40), 139 (90), 123 (100).

Bis(cyclohexyloxy)(phenyl)silane 6b



Bis(cyclohexyloxy)(phenyl)silane was obtained as a liquid in 90% yield. The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

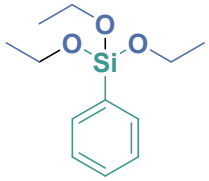
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.95 – 7.78 (m, 2H), 7.32 – 7.17 (m, 3H), 5.39 (s, 1H), 3.99 (td, *J* = 9.0, 4.3 Hz, 2H), 2.03 – 1.83 (m, 4H), 1.76 – 1.45 (m, 8H), 1.33 (dtd, *J* = 10.7, 6.9, 5.9, 3.6 Hz, 2H), 1.13 (ddd, *J* = 10.3, 7.4, 3.0 Hz, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 135.0, 134.6, 130.6, 128.2, 72.0, 35.9, 25.8, 24.1.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -34.5.

EI-MS m/z (rel. int.): 303 (7%), 261 (11), 226 (80), 179 (15), 139 (67), 123 (100).

Triethoxy(phenyl)silane 6c



Triethoxy(phenyl)silane was obtained as a liquid in 81% yield. The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

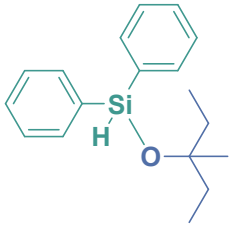
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.91 – 7.82 (m, 2H), 7.28 – 7.20 (m, 3H), 3.93 – 3.80 (m, 6H), 1.18 (tt, *J* = 7.0, 1.0 Hz, 9H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 135.2, 132.1, 130.5, 128.1, 58.9, 18.5.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -57.8.

EI-MS m/z (rel. int.): 240 (31%), 225 (4), 195 (76), 181 (27), 162 (45), 147 (100), 119 (30).

((3-Methylpentan-3-yl)oxy)diphenylsilane 6d



((3-Methylpentan-3-yl)oxy)diphenylsilane was obtained as a liquid in 76% yield. The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

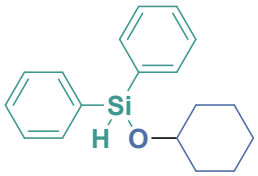
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.77 – 7.70 (m, 4H), 7.24 – 7.17 (m, 6H), 5.84 (s, 1H), 1.60 – 1.50 (m, 4H), 1.18 (s, 3H), 0.85 (t, *J* = 7.5 Hz, 6H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.7, 130.2, 128.2, 78.3, 34.1, 26.2, 8.7.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -23.4.

EI-MS m/z (rel. int.): 269 (3%), 255 (81), 199 (78), 183 (100), 155 (8), 122 (37).

(Cyclohexyloxy)diphenylsilane 6e



(Cyclohexyloxy)diphenylsilane was obtained as a liquid in 82% yield. The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

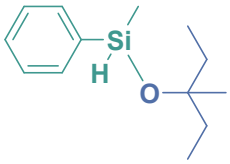
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.84 – 7.66 (m, 4H), 7.17 (s, 6H), 5.78 (s, 1H), 3.86 (tt, *J* = 8.6, 3.9 Hz, 1H), 1.85 (dq, *J* = 12.6, 3.6 Hz, 2H), 1.66 – 1.42 (m, 4H), 1.34 – 1.25 (m, 1H), 1.07 (qt, *J* = 10.8, 5.0 Hz, 3H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 135.3, 135.0, 130.4, 128.3, 72.9, 35.5, 25.8, 24.0.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -15.0.

EI-MS m/z (rel. int.): 281 (5%), 239 (13), 204 (95), 199 (38), 183 (100), 161 (29), 123 (50).

Methyl((3-methylpentan-3-yl)oxy)(phenyl)silane 6f



Methyl((3-methylpentan-3-yl)oxy)(phenyl)silane was obtained as a liquid in 90% yield. The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

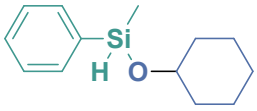
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.74 – 7.59 (m, 2H), 7.26 – 7.18 (m, 3H), 5.40 (q, *J* = 2.8 Hz, 1H), 1.48 (dddd, *J* = 13.1, 7.3, 5.5, 2.4 Hz, 4H), 1.12 (s, 3H), 0.83 (td, *J* = 7.5, 4.8 Hz, 6H), 0.39 (d, *J* = 2.9 Hz, 3H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 138.1, 133.6, 129.5, 127.8, 77.2, 33.8, 25.6, 8.2, -0.3.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -15.9.

EI-MS m/z (rel. int.): 207 (3%), 193 (92), 137 (100), 129 (6), 121 (57), 105 (15).

(Cyclohexyloxy)(methyl)(phenyl)silane 6g



(Cyclohexyloxy)(methyl)(phenyl)silane was obtained as a liquid in 85% yield. The title compound was known in the literature,[4] and all spectroscopic data are in agreement.

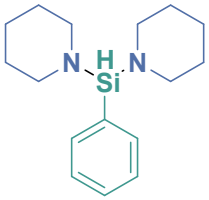
^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.74 – 7.62 (m, 2H), 7.32 – 7.18 (m, 3H), 5.34 (qd, *J* = 2.9, 0.9 Hz, 1H), 3.71 (tt, *J* = 8.8, 3.9 Hz, 1H), 1.90 – 1.73 (m, 2H), 1.68 – 1.38 (m, 4H), 1.31 (tdd, *J* = 9.7, 5.0, 1.9 Hz, 1H), 1.15 – 0.95 (m, 3H), 0.40 (dd, *J* = 2.8, 0.9 Hz, 3H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 137.2, 134.1, 130.2, 128.2, 72.6, 35.6, 25.8, 24.1, -1.6.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -6.4.

EI-MS m/z (rel. int.): 219 (3%), 205 (8), 177 (30), 142 (100), 137 (48), 121 (99).

1,1'-(Phenylsilanediyl)dipiperidine 6h



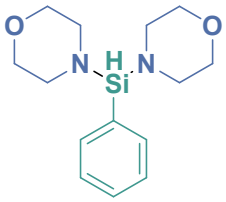
1,1'-(Phenylsilanediyl)dipiperidine was obtained in 99% yield. The title compound was known in the literature,[5] and all spectroscopic data are in agreement.

^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.72 – 7.67 (m, 2H), 7.30 – 7.19 (m, 3H), 5.03 (s, 1H), 2.94 (tt, *J* = 7.9, 4.0 Hz, 8H), 1.51 – 1.45 (m, 4H), 1.38 – 1.32 (m, 8H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 136.5, 134.6, 129.4, 127.8, 46.4, 27.7, 25.5.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -20.0.

4,4'-(Phenylsilanediyl)dimorpholine 6i



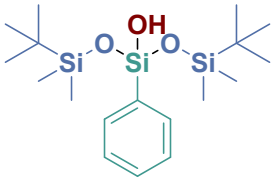
4,4'-(Phenylsilanediyl)dimorpholine was obtained in 99% yield. The title compound was known in the literature,[5] and all spectroscopic data are in agreement.

^{1}H NMR: (400 MHz, Benzene-d_6) δ 7.59 – 7.50 (m, 2H), 7.25 – 7.19 (m, 3H), 4.87 (s, 1H), 3.41 (t, *J* = 4.6 Hz, 9H), 2.79 (q, *J* = 3.5 Hz, 8H).

^{13}C NMR: (101 MHz, Benzene-d_6) δ 134.7, 134.6, 129.9, 128.0, 68.0, 45.6.

^{29}Si NMR: (79 MHz, Benzene-d_6) δ -19.6.

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxan-3-ol 7a



1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxan-3-ol was obtained as a liquid in 93% yield. The title compound was previously unknown.

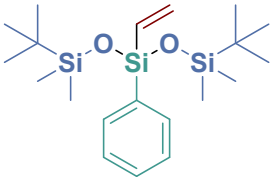
^{1}H NMR: (600 MHz, Benzene-*d*6) δ 7.79 – 7.76 (m, 2H), 7.25 – 7.18 (m, 3H), 1.01 (s, 1H), 1.00 – 0.96 (m, 18H), 0.16 (t, *J* = 7.8 Hz, 12H).

^{13}C NMR: (151 MHz, Benzene-d_6) δ 135.0, 134.4, 130.2, 128.0, 25.9, 18.3, -2.8.

^{29}Si NMR: (119 MHz, Benzene-d_6) δ 12.1, -69.2.

EI-MS m/z (rel. int.): 327 (27%), 269 (15), 252 (30), 238 (4), 207 (100), 193 (25), 132 (12).

1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyl-3-(vinyloxy)trisiloxane 7b



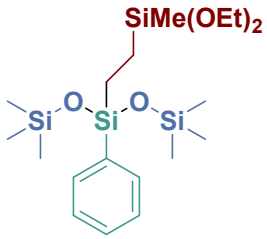
1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyl-3-(vinyloxy)trisiloxane was obtained as a liquid in 62% yield. The title compound was previously unknown.

^{1}H NMR: (600 MHz, Chloroform-*d*) δ 7.62 – 7.54 (m, 2H), 7.42 – 7.32 (m, 3H), 6.22 – 6.04 (m, 2H), 5.86 (dd, *J* = 19.6, 4.8 Hz, 1H), 0.88 (d, *J* = 4.3 Hz, 18H), 0.11 – 0.00 (m, 12H).

^{13}C NMR: (151 MHz, Chloroform-*d*) δ 136.6, 135.6, 134.5, 133.9, 129.5, 127.5, 25.7, 18.1, -2.8.

EI-MS m/z (rel. int.): 337 (95%), 295 (100), 281 (80), 267 (64), 193 (43), 147 (52), 135 (97).

1,5-Di-tert-butyl-3-(2-(diethoxy(methyl)silyl)ethyl)-1,1,5,5-tetramethyl-3-phenyltrisiloxane 7c



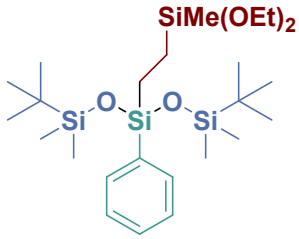
1,5-Di-tert-butyl-3-(2-(diethoxy(methyl)silyl)ethyl)-1,1,5,5-tetramethyl-3-phenyltrisiloxane was obtained as a liquid in 92% yield. The title compound was previously unknown.

^{1}H NMR: (600 MHz, Chloroform-*d*) δ 7.61 – 7.53 (m, 2H), 7.39 – 7.31 (m, 3H), 3.74 (qd, *J* = 7.0, 0.4 Hz, 3H), 1.29 – 1.15 (m, 6H), 0.93 – 0.86 (m, 19H), 0.77 – 0.70 (m, 2H), 0.63 – 0.54 (m, 2H), 0.11 (s, 2H), 0.09 – 0.04 (m, 13H).

^{13}C NMR: (151 MHz, Chloroform-*d*) δ 137.1, 133.6, 129.3, 127.5, 58.0, 25.7, 18.3, 18.1, 7.2, 5.1, -2.8, -5.6.

EI-MS m/z (rel. int.): 471 (67%), 397 (38), 279 (100), 205 (21), 135 (80), 73 (75) .

3-(2-(Diethoxy(methyl)silyl)ethyl)-1,1,1,5,5,5-hexamethyl-3-phenyltrisiloxane 7d



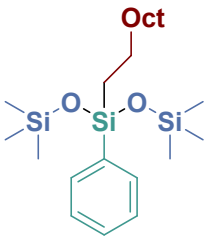
3-(2-(Diethoxy(methyl)silyl)ethyl)-1,1,1,5,5,5-hexamethyl-3-phenyltrisiloxane was obtained as a liquid in 88% yield. The title compound was previously unknown.

^{1}H NMR: (600 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 7.0 Hz, 2H), 7.36 (dt, *J* = 14.6, 7.1 Hz, 3H), 3.76 (dq, *J* = 21.0, 7.1 Hz, 4H), 1.21 (t, *J* = 7.1 Hz, 6H), 0.79 – 0.47 (m, 4H), 0.12 (d, *J* = 13.0 Hz, 21H).

^{13}C NMR: (151 MHz, Chloroform-*d*) δ 137.16 133.5, 129.3, 127.5, 58.0, 18.3, 7.3, 4.8, 1.8, -5.7.

EI-MS m/z (rel. int.): 429 (5%), 283 (100), 267 (10), 253 (10), 193 (7), 135 (72) .

3-Decyl-1,1,1,5,5,5-hexamethyl-3-phenyltrisiloxane 7e



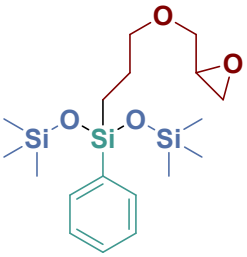
3-Decyl-1,1,1,5,5,5-hexamethyl-3-phenyltrisiloxane was obtained as a liquid in 89% yield. The title compound was previously unknown.

^{1}H NMR: (600 MHz, Chloroform-*d*) δ 7.58 – 7.55 (m, 2H), 7.37 (q, *J* = 6.7, 5.6 Hz, 3H), 1.28 (d, *J* = 5.7 Hz, 12H), 0.91 (t, *J* = 6.9 Hz, 3H), 0.76 – 0.70 (m, 2H), 0.14 (s, 22H).

^{13}C NMR: (151 MHz, Chloroform-*d*) δ 137.8, 133.4, 129.2, 127.4, 33.2, 31.8, 29.6, 29.5, 29.3, 29.2, 22.9, 22.6, 16.3, 14.0, 1.8.

EI-MS m/z (rel. int.): 409 (5%), 346 (3), 283 (100), 269 (10), 207 (4), 135 (42).

1,1,1,5,5,5-Hexamethyl-3-(3-(oxiran-2-ylmethoxy)propyl)-3-phenyltrisiloxane 7f



1,1,1,5,5,5-Hexamethyl-3-(3-(oxiran-2-ylmethoxy)propyl)-3-phenyltrisiloxane was obtained as a liquid in 97% yield. The title compound was previously unknown.

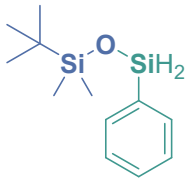
^{1}H NMR: (600 MHz, Chloroform-*d*) δ 7.57 – 7.52 (m, 2H), 7.39 – 7.32 (m, 3H), 3.65 (dd, *J* = 11.5, 3.2 Hz, 1H), 3.48 – 3.33 (m, 3H), 3.12 (ddt, *J* = 5.8, 4.2, 2.9 Hz, 1H), 2.78 (dd, *J* = 5.1, 4.1 Hz, 1H), 2.59 (dd, *J* = 5.1, 2.7 Hz, 1H), 1.69 – 1.58 (m, 2H), 0.75 – 0.67 (m, 2H), 0.15 – 0.11 (m, 18H).

^{13}C NMR: (151 MHz, Chloroform-*d*) δ 137.2, 133.4 129.4, 127.5, 74.0, 71.3, 50.8, 44.3, 23.1, 12.3, 1.8.

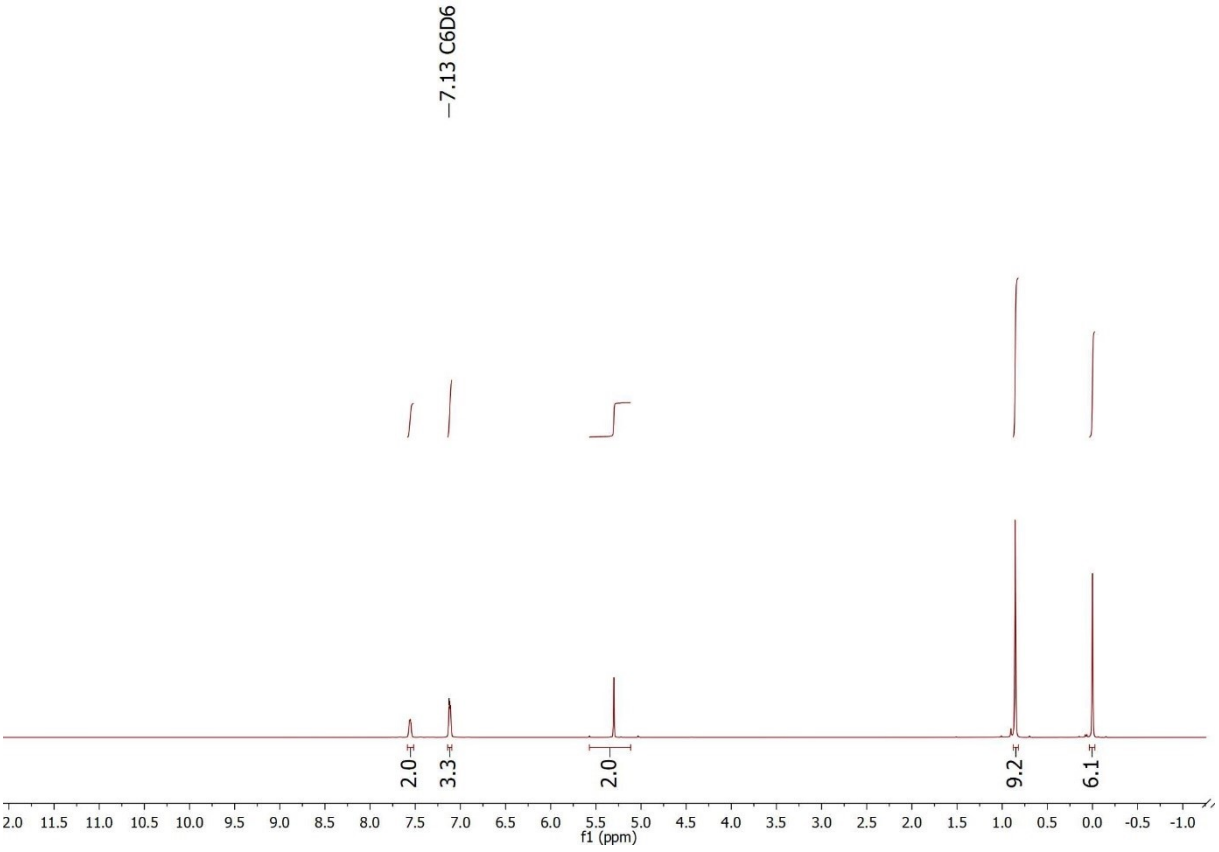
EI-MS m/z (rel. int.): 311 (5%), 283 (79), 253 (29), 207 (95), 193 (32), 135 (100).

SPECTRA FOR ALL PRODUCTS

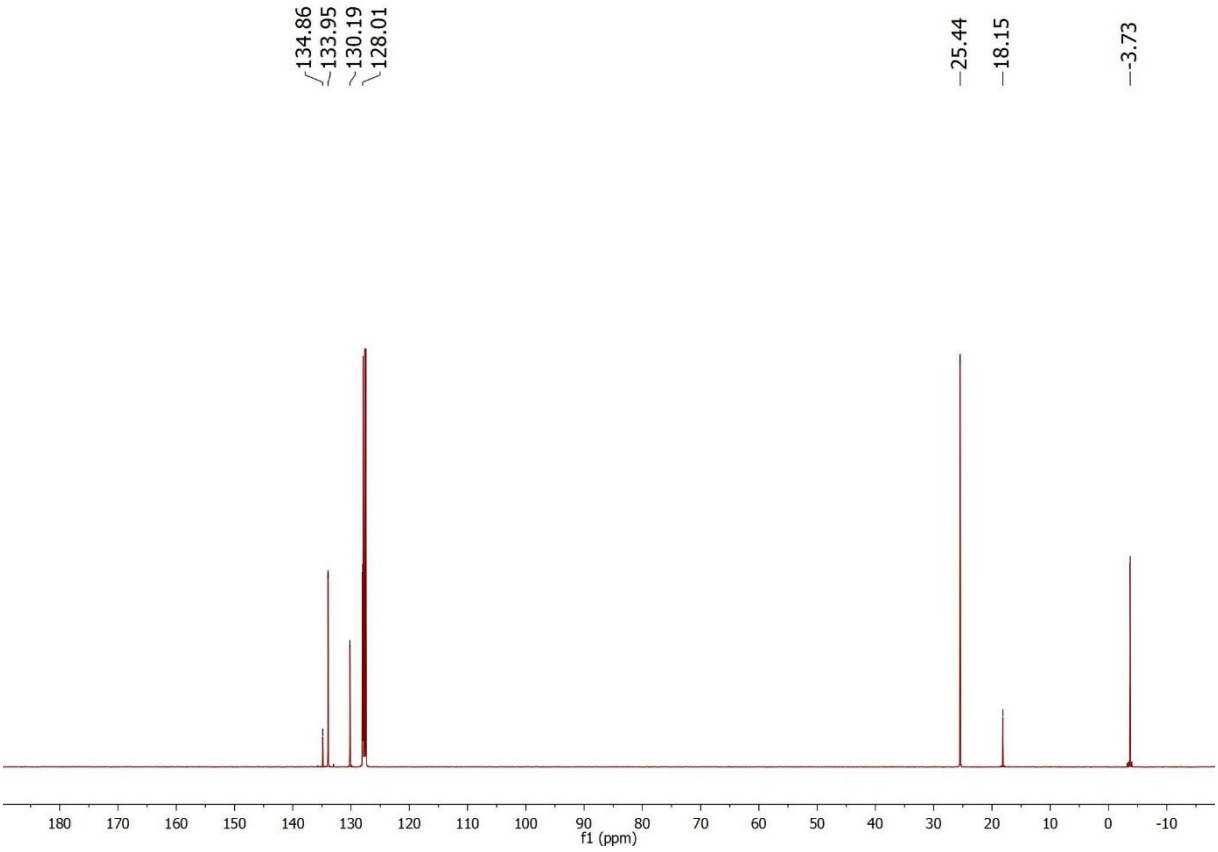
1-(Tert-butyl)-1,1-dimethyl-3-phenyldisiloxane 3a



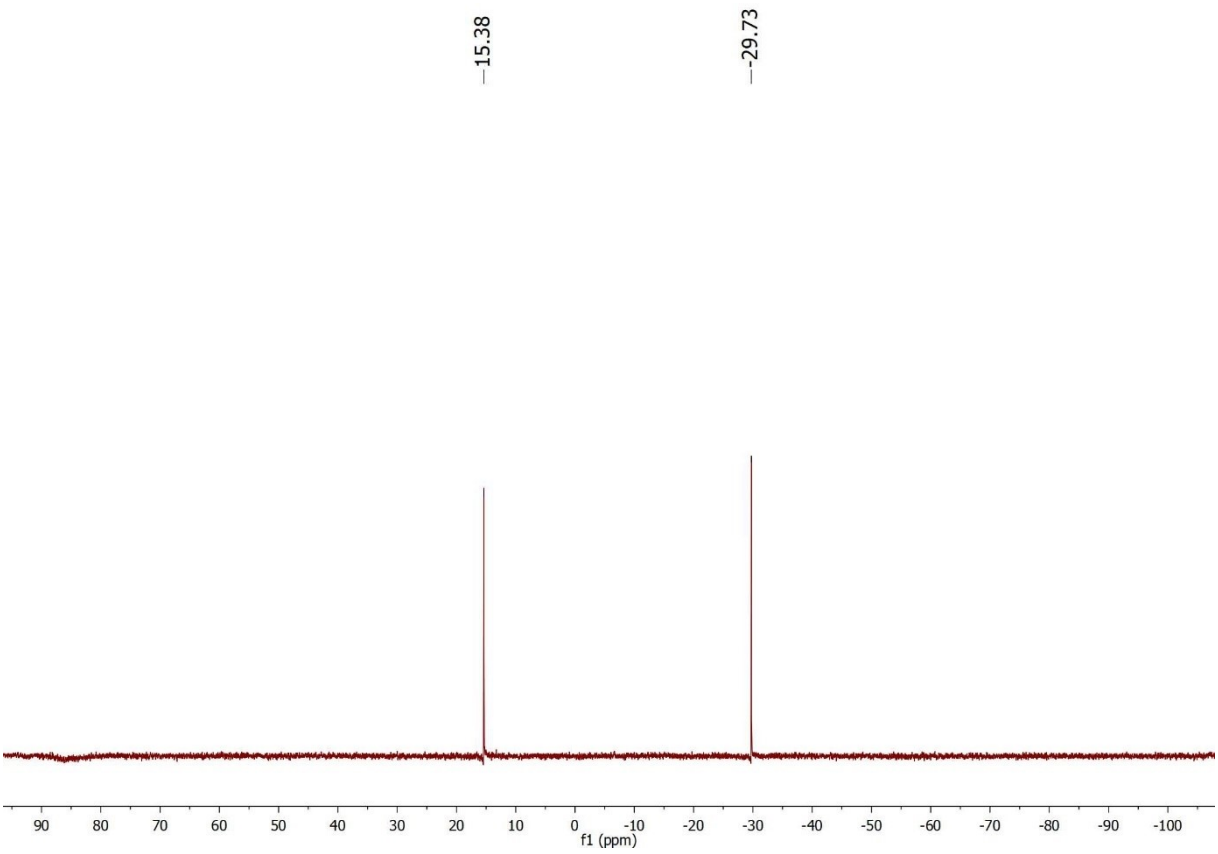
^{1}H NMR



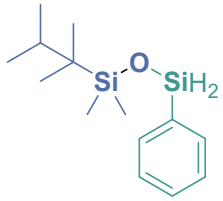
^{13}C NMR



^{29}Si NMR



1-(2,3-Dimethylbutan-2-yl)-1,1-dimethyl-3-phenyldisiloxane 3b



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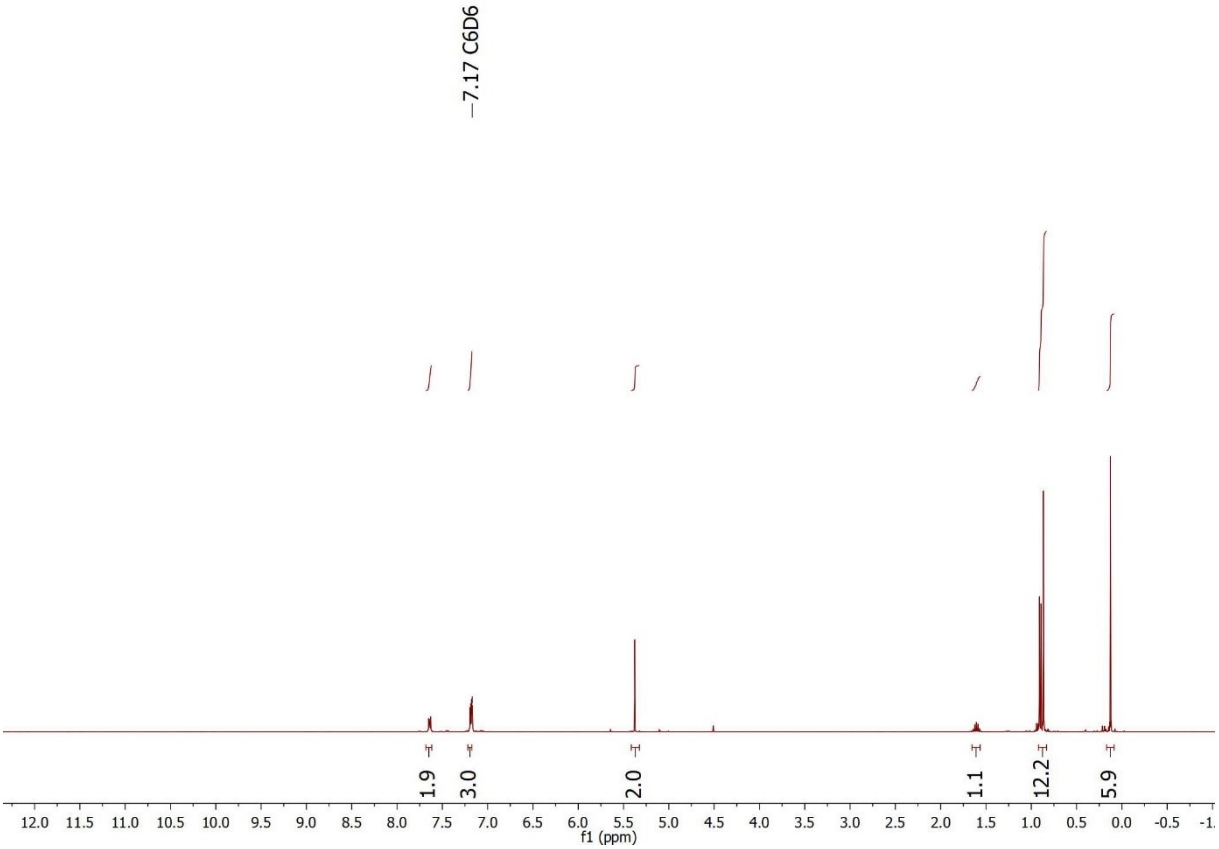

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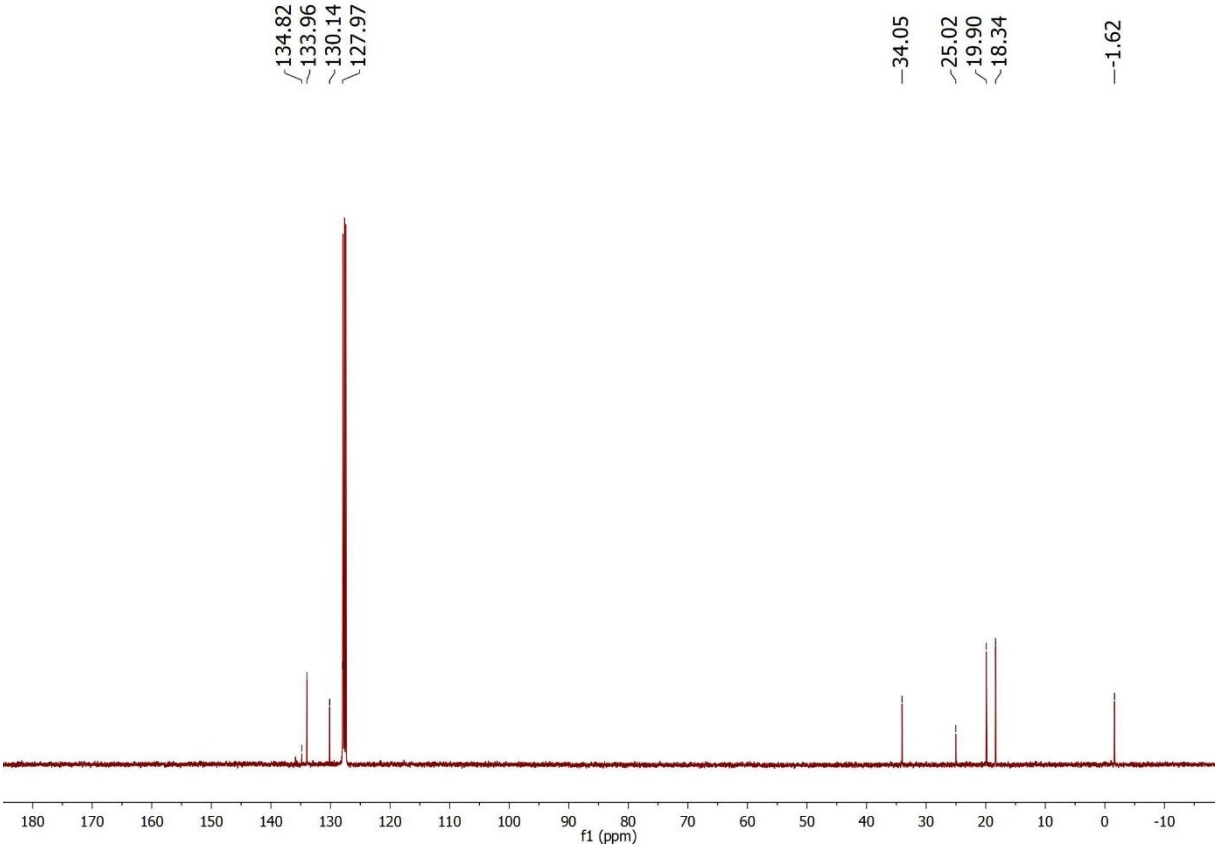
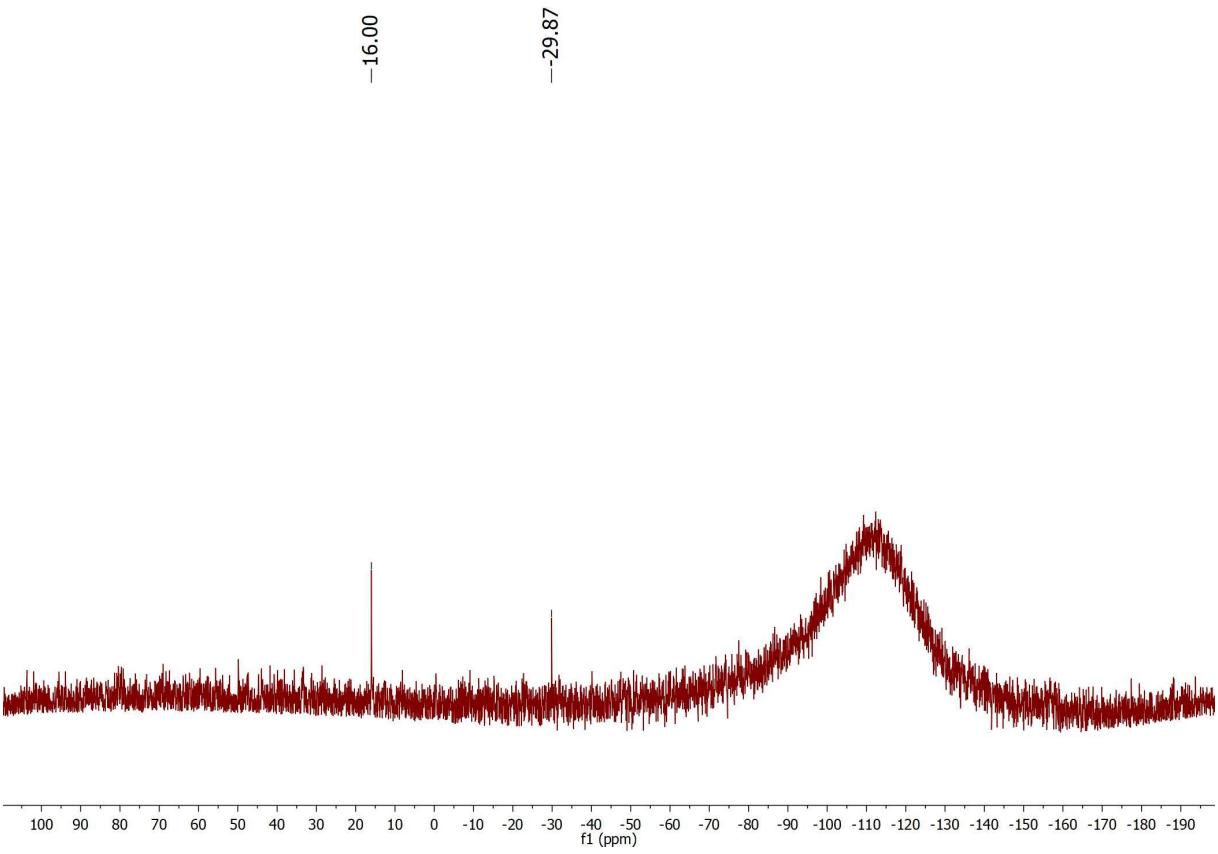
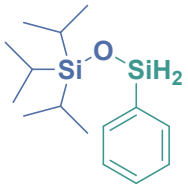


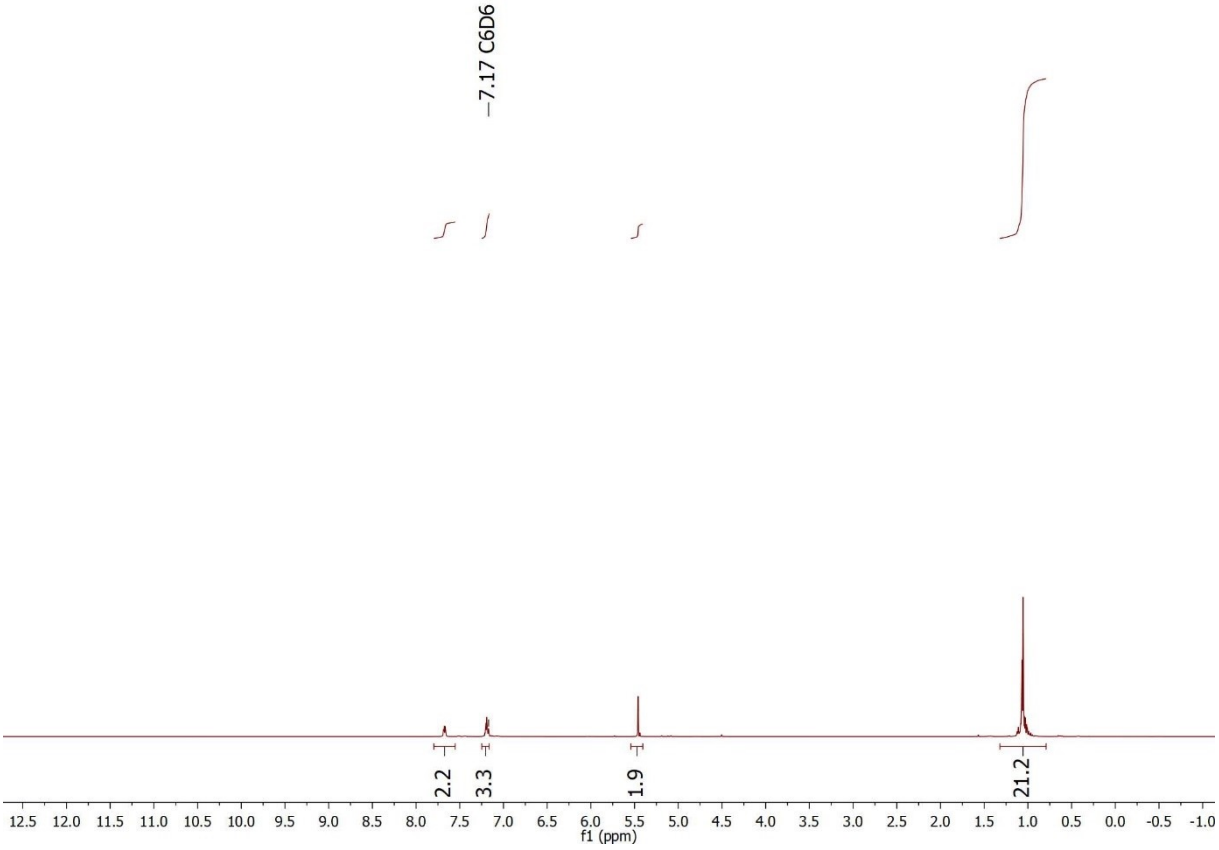
^{29}Si NMR



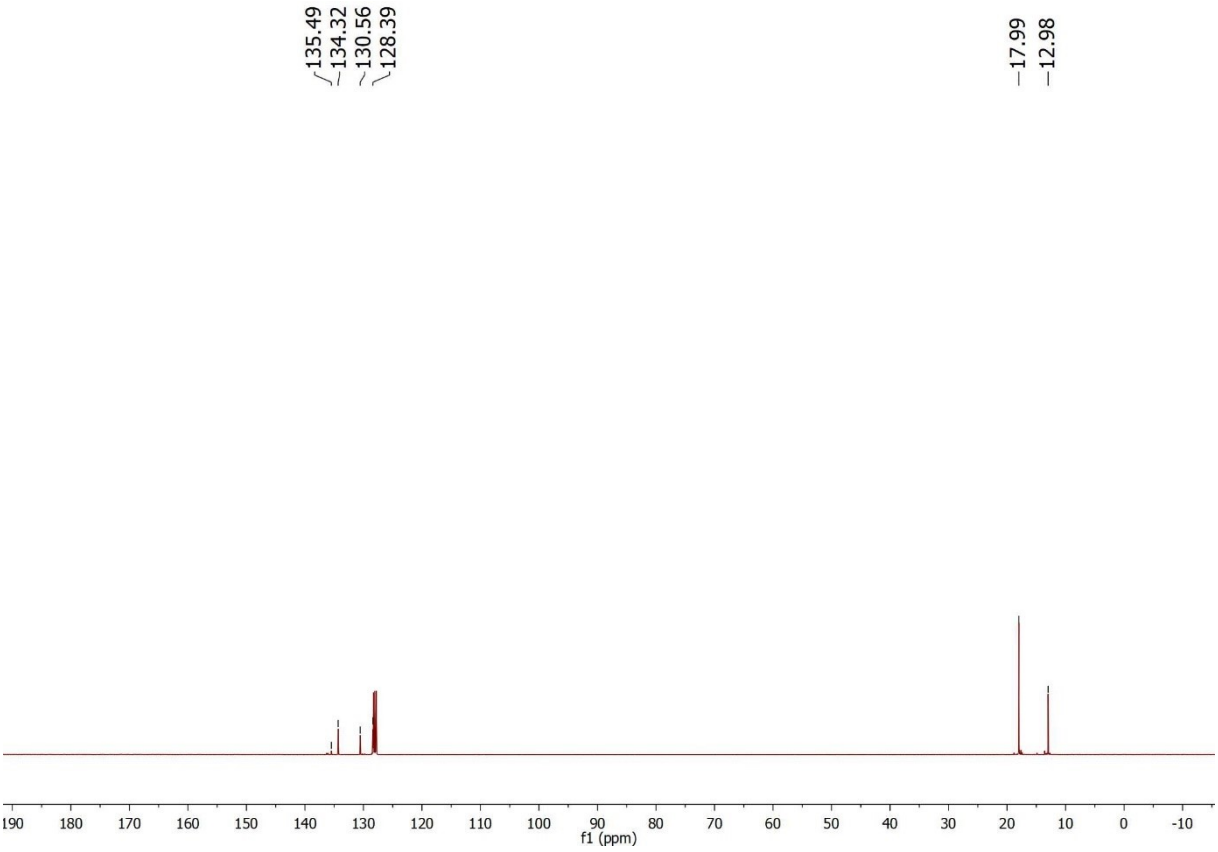
1,1,1-Triisopropyl-3-phenyldisiloxane 3c



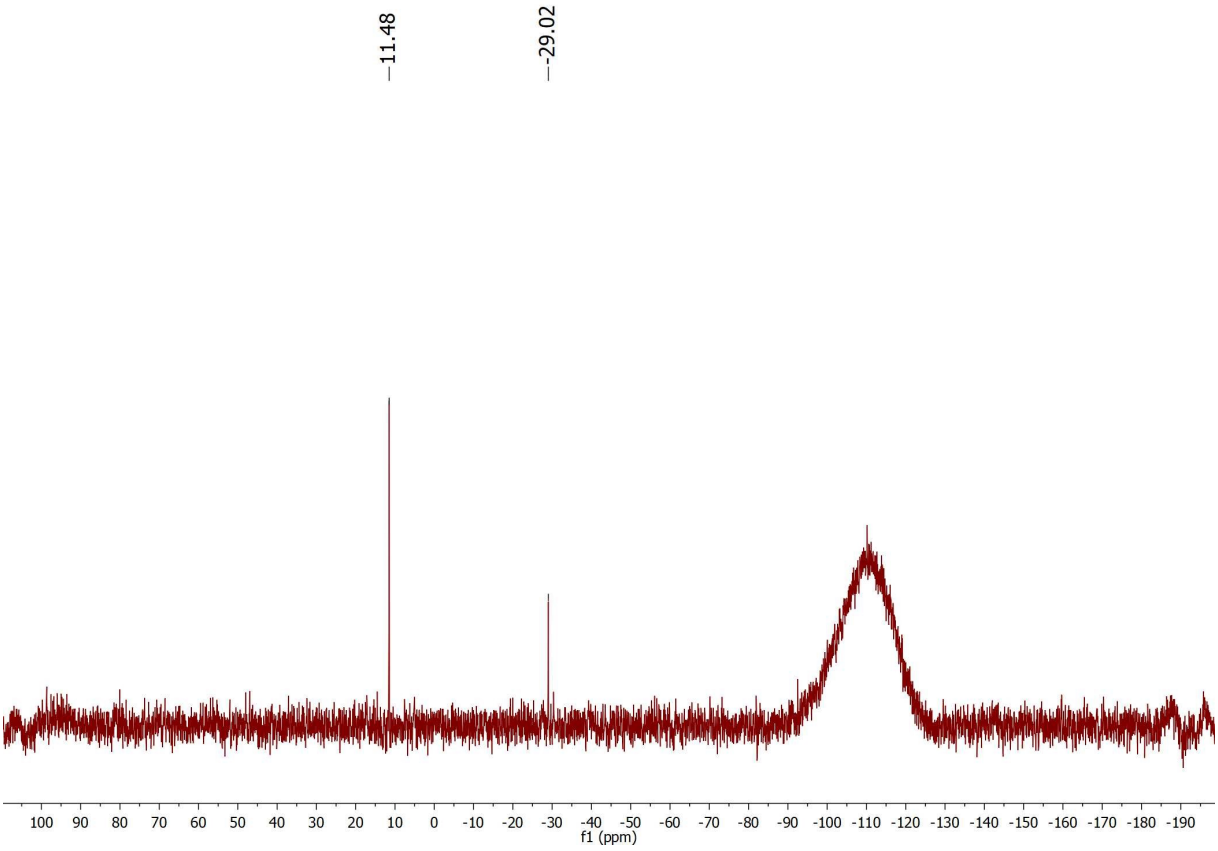
^{1}H NMR



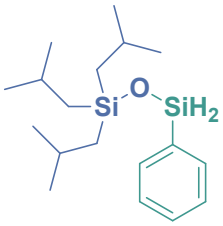
^{13}C NMR



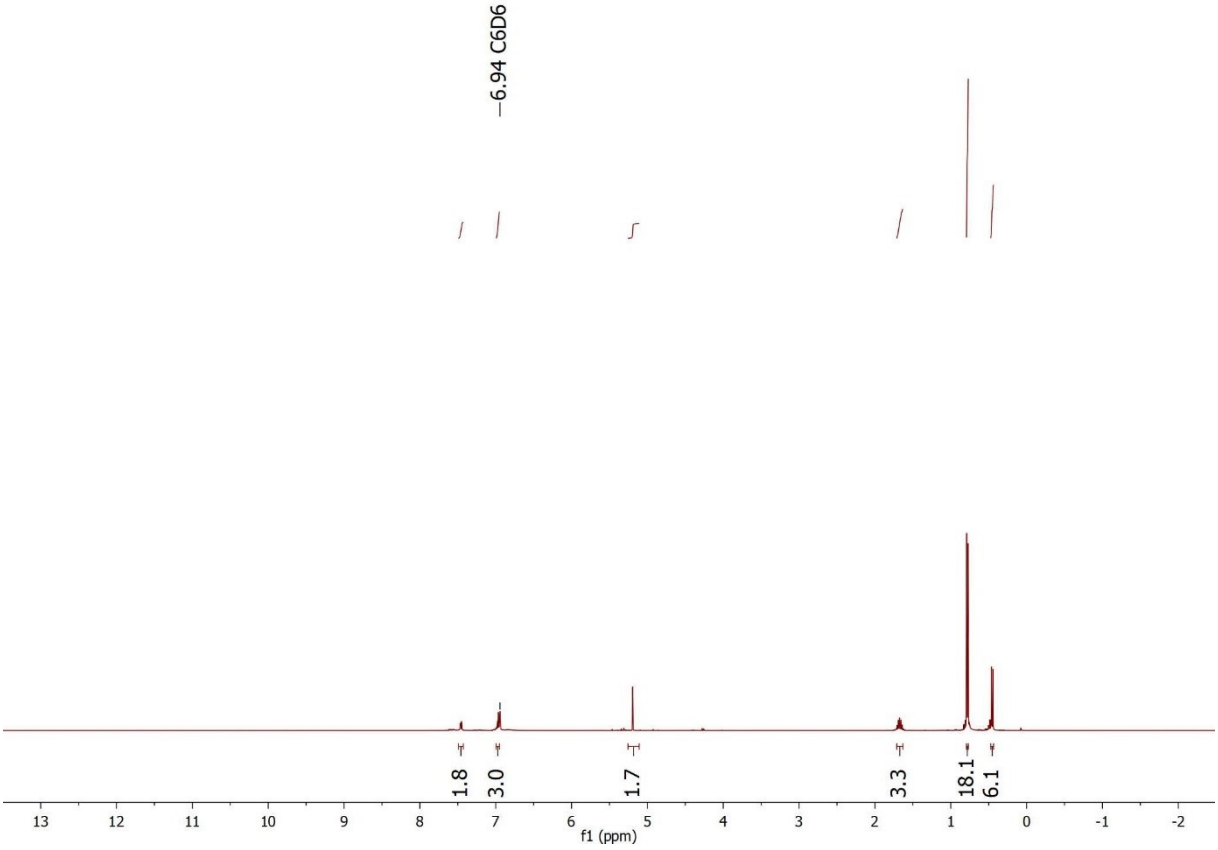
^{29}Si NMR



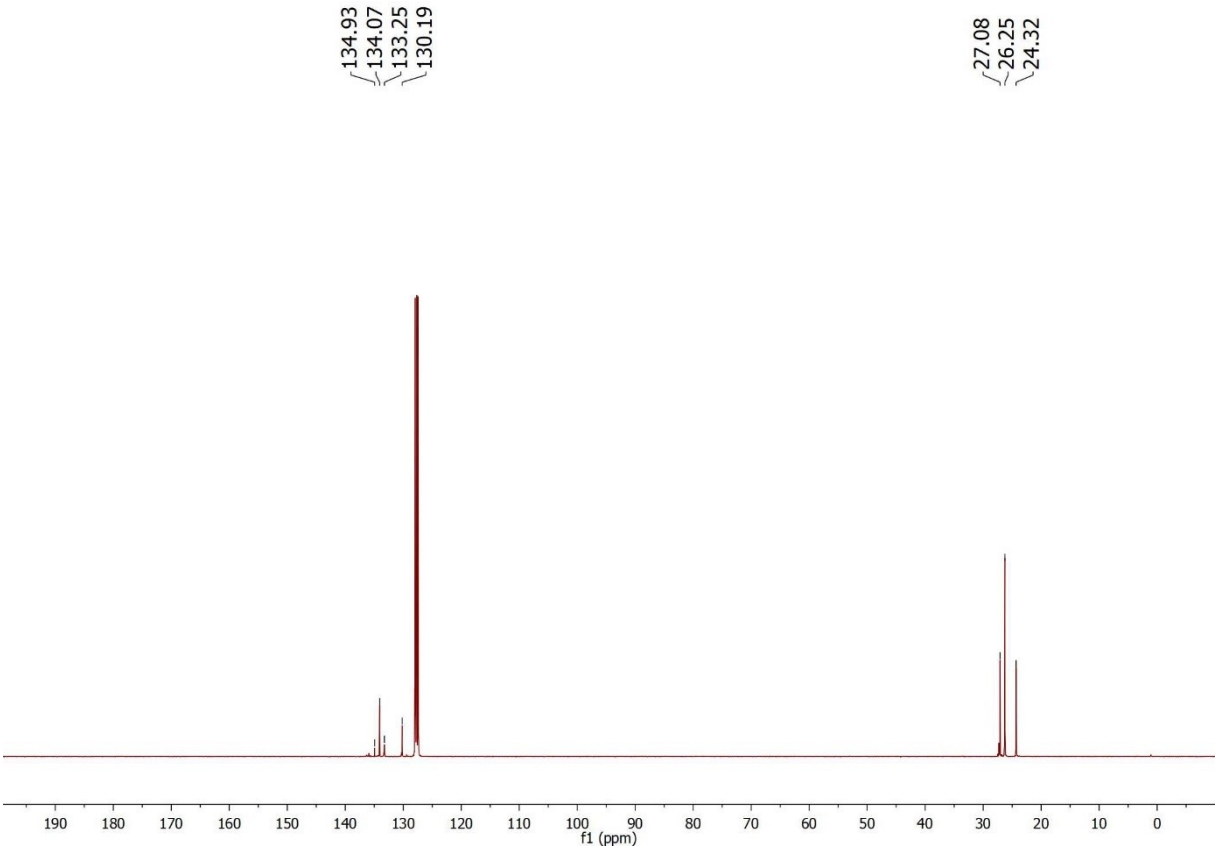
1,1,1-Triisobutyl-3-phenyldisiloxane 3d



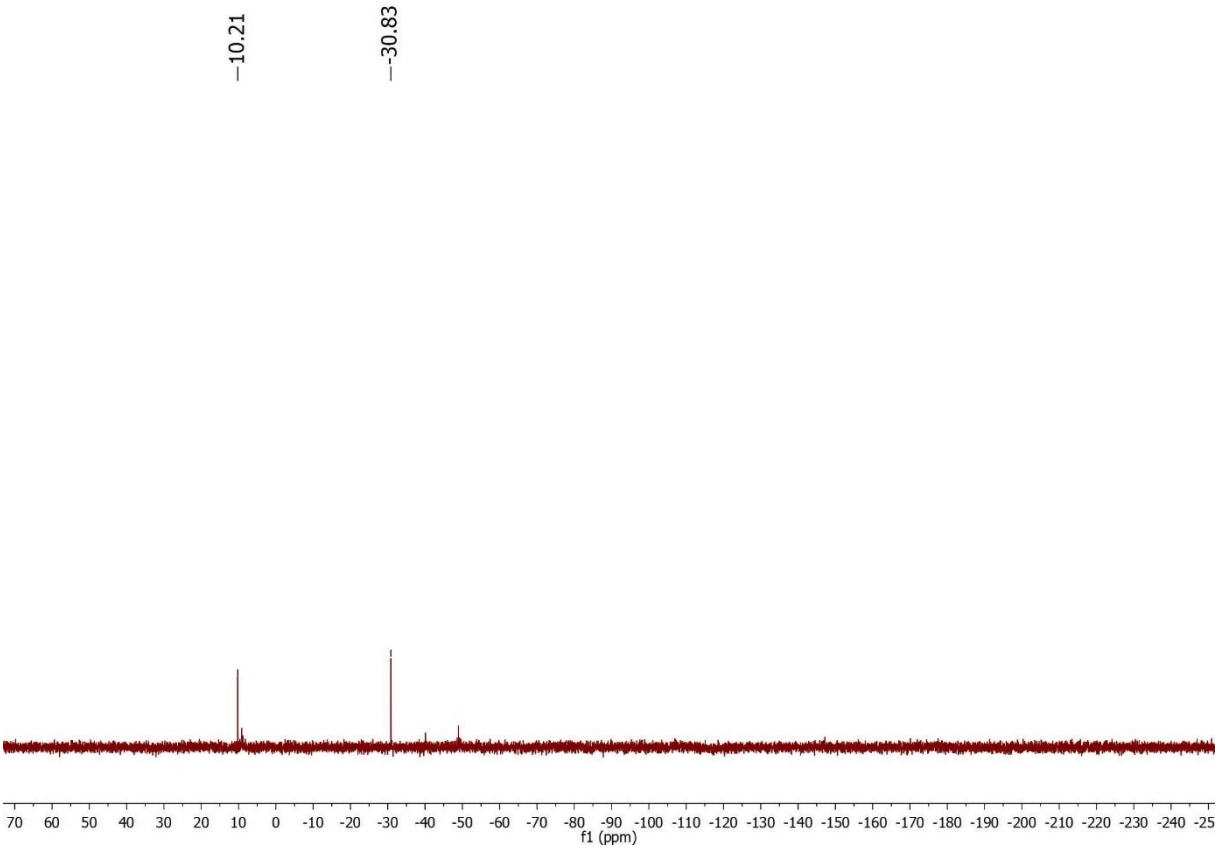
^{1}H NMR



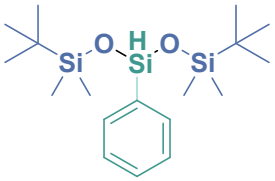
^{13}C NMR



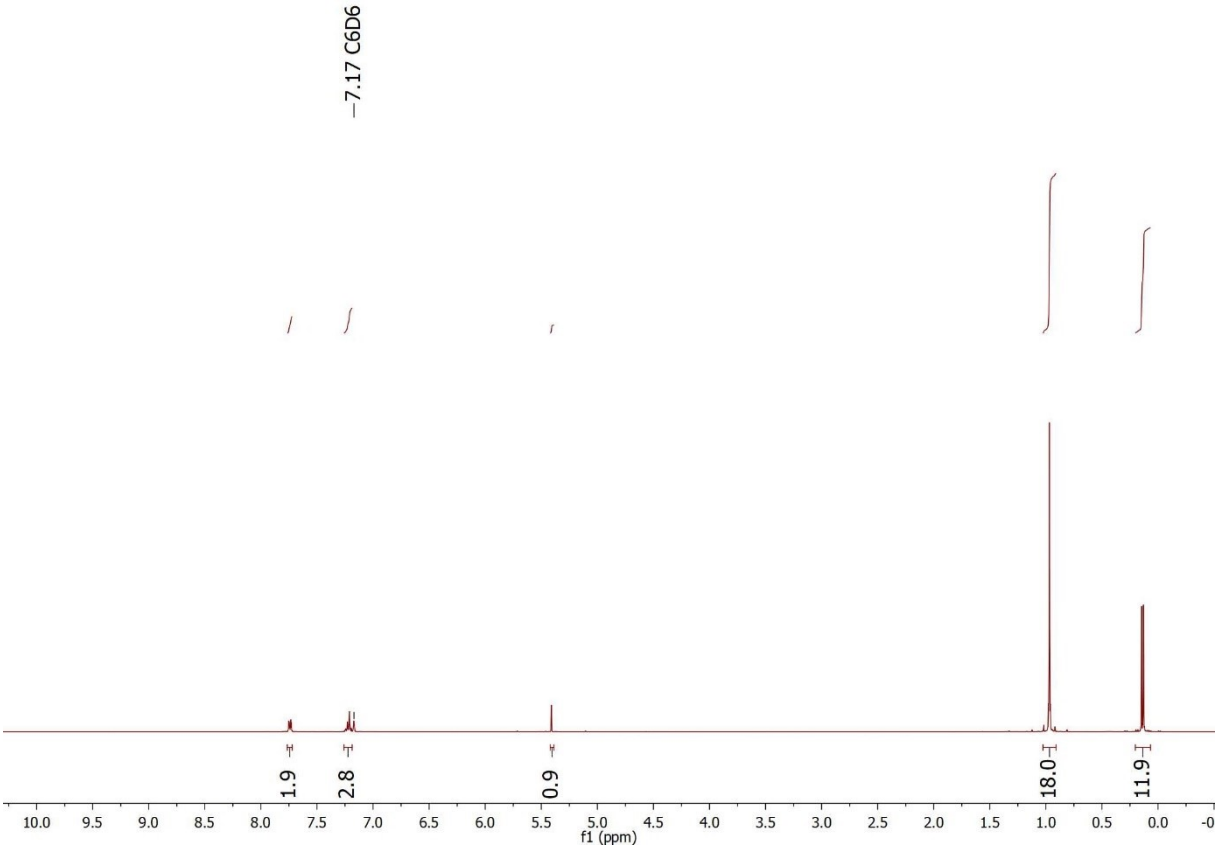
^{29}Si NMR



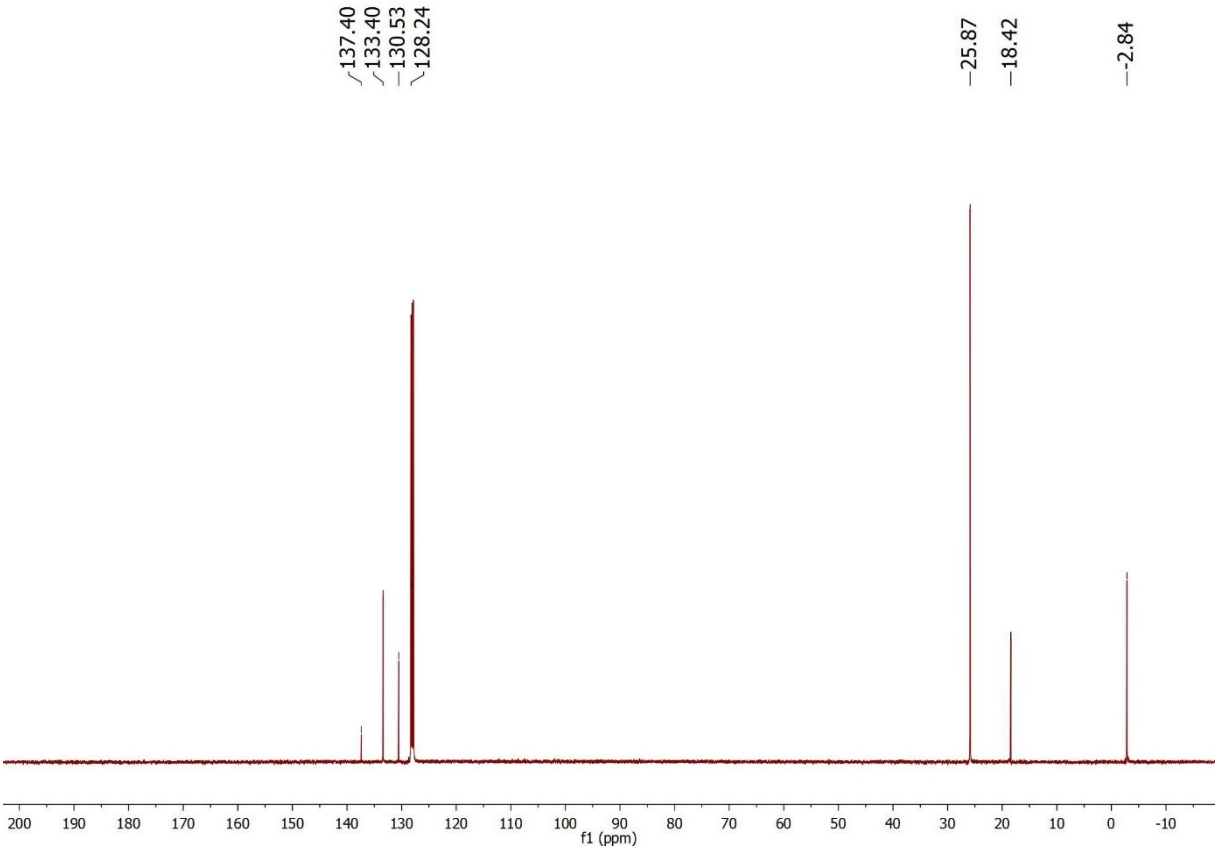
1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxane 4a



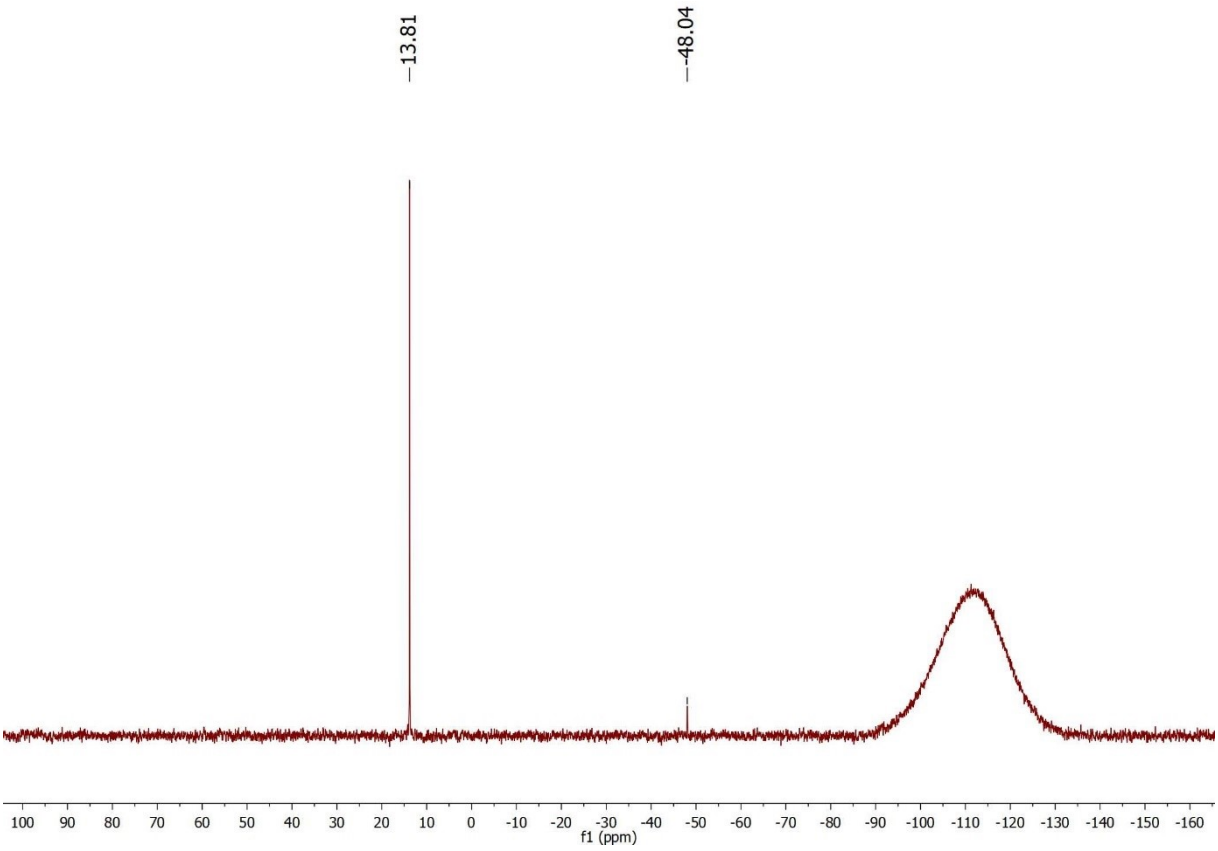
^{1}H NMR



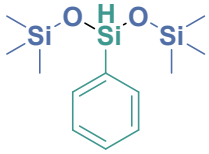
^{13}C NMR



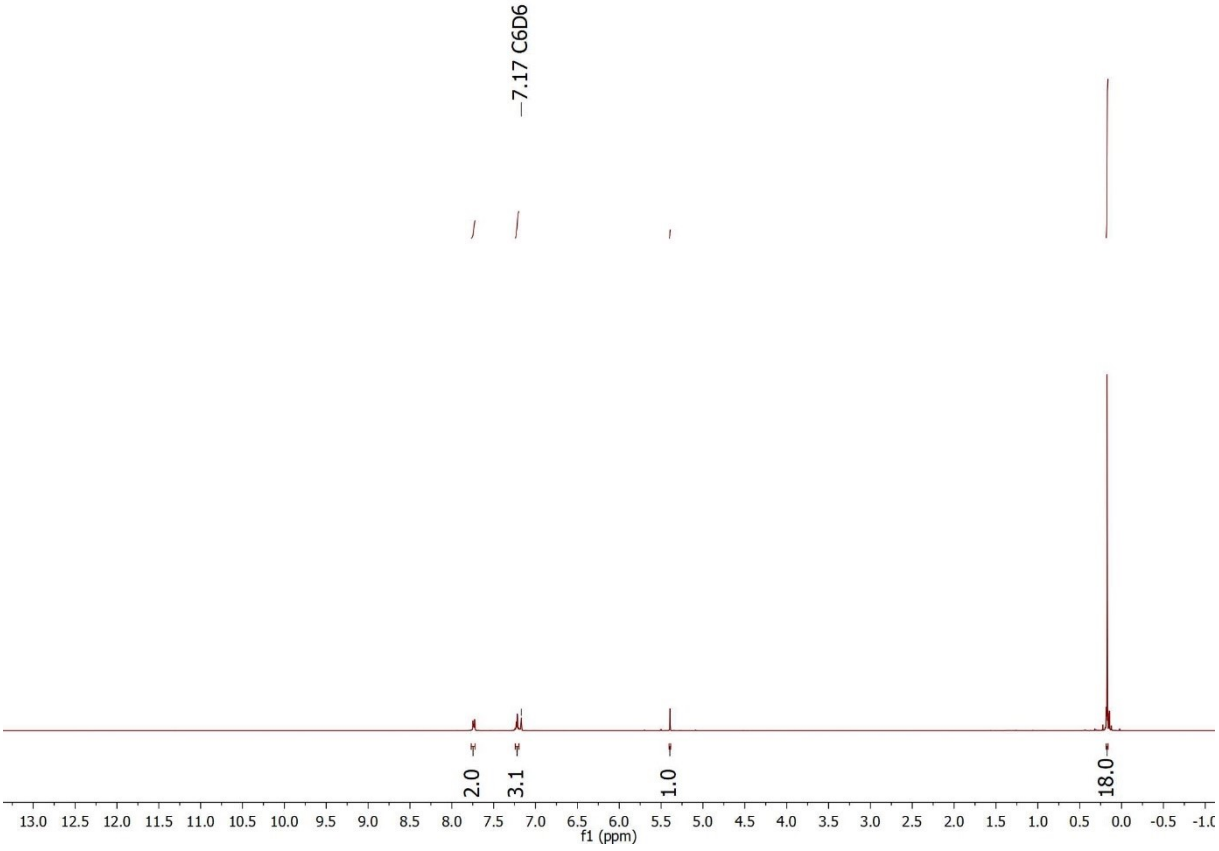
^{29}Si NMR



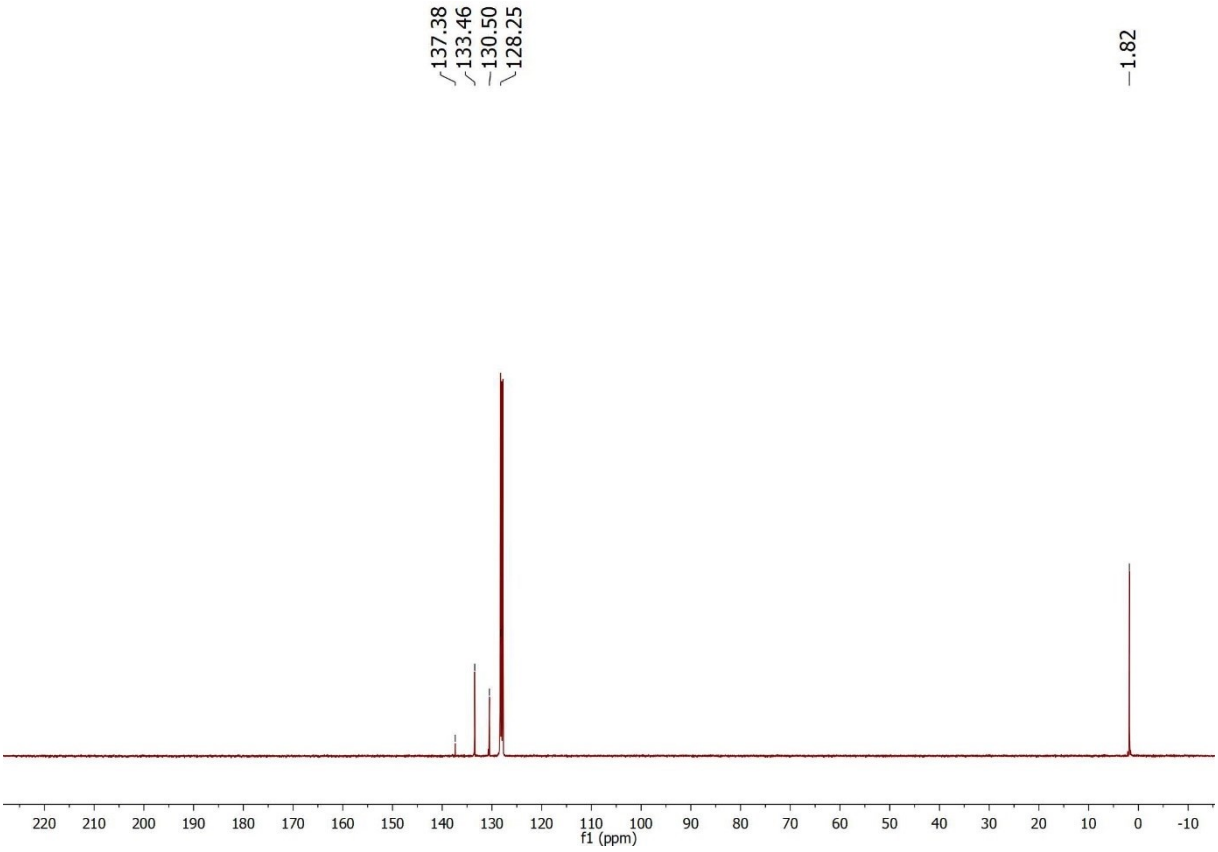
1,1,1,5,5,5-Hexamethyl-3-phenyltrisiloxane 4b



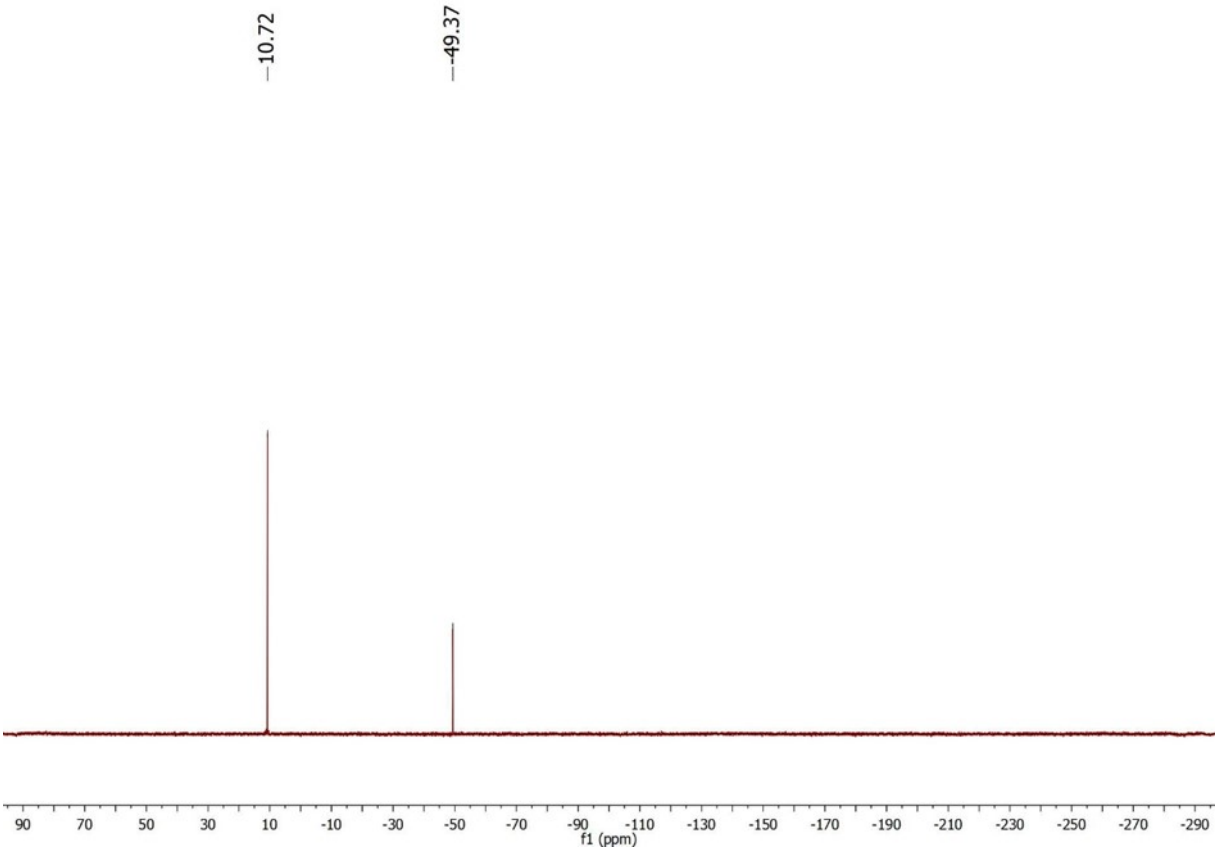
^{1}H NMR



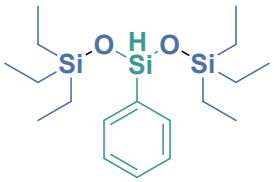
^{13}C NMR



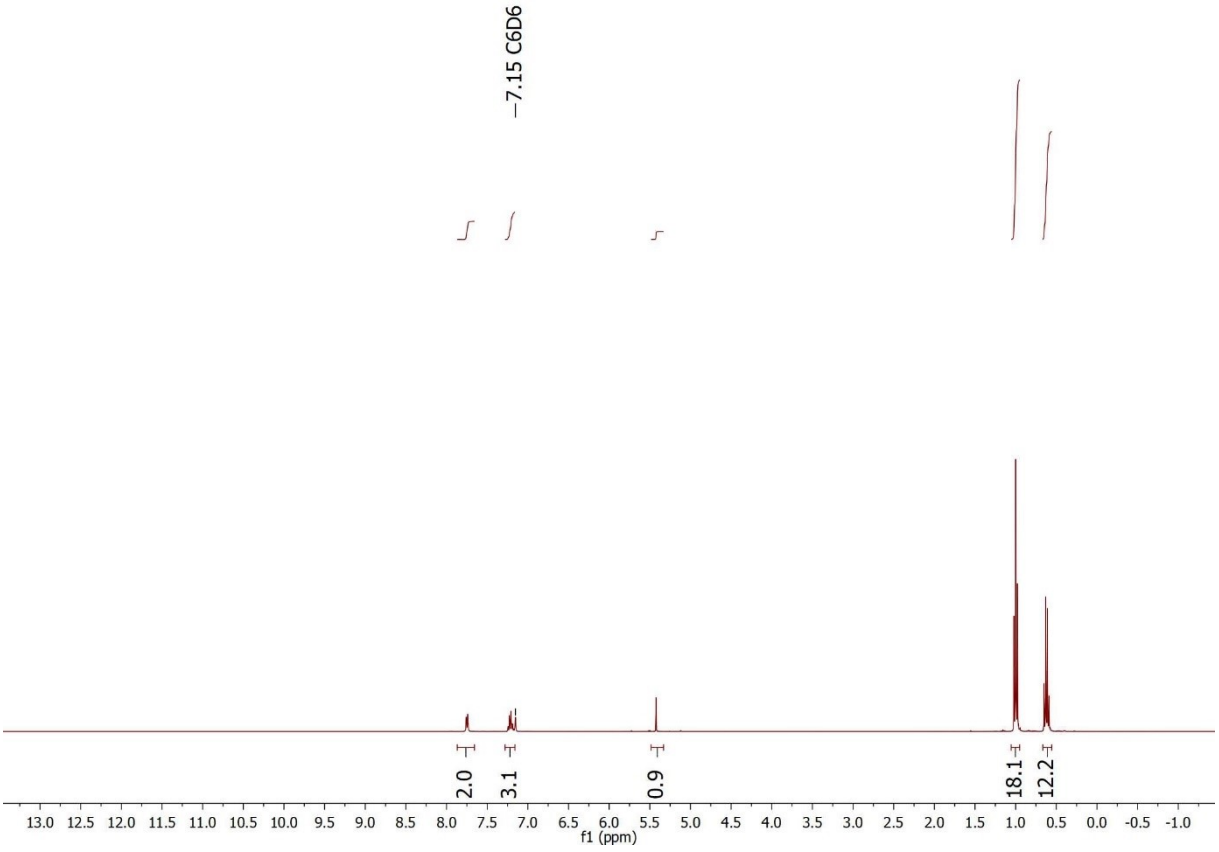
^{29}Si NMR



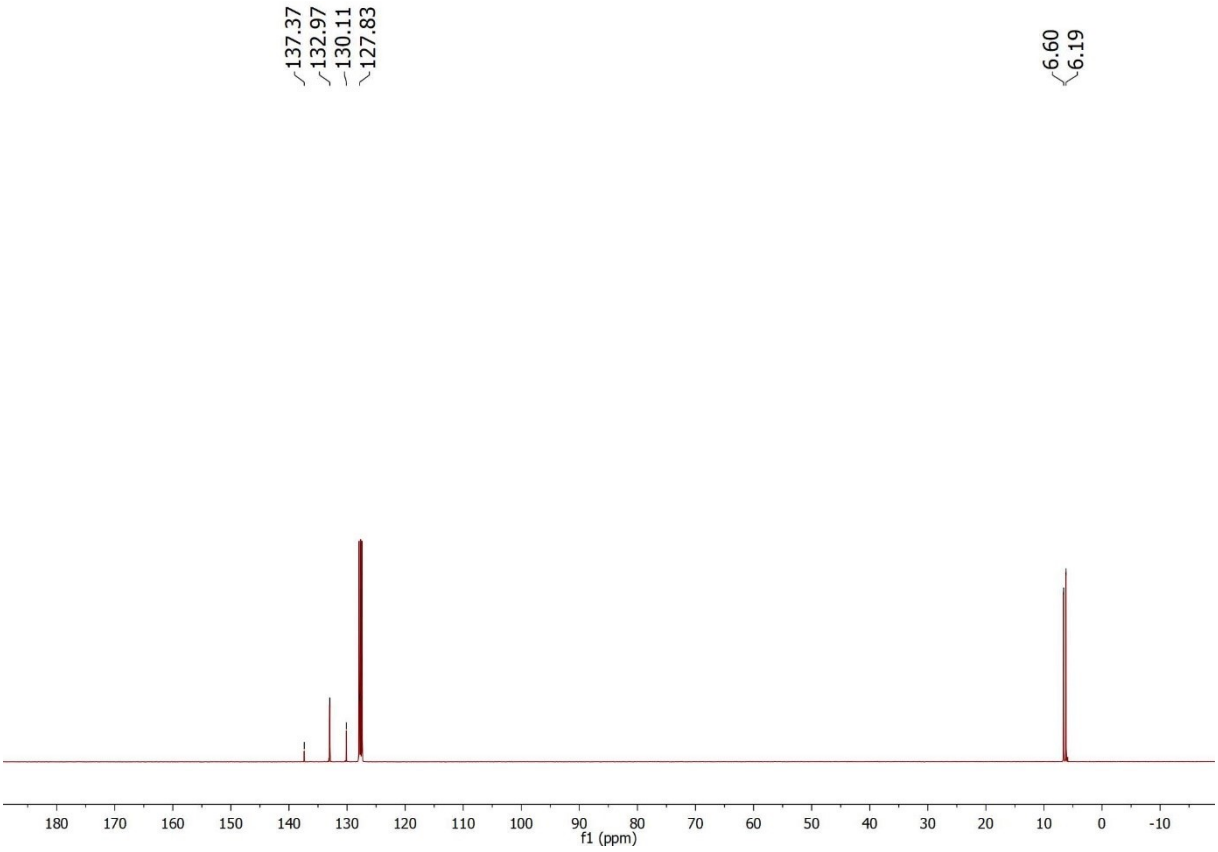
1,1,1,5,5,5-Hexaethyl-3-phenyltrisiloxane 4c



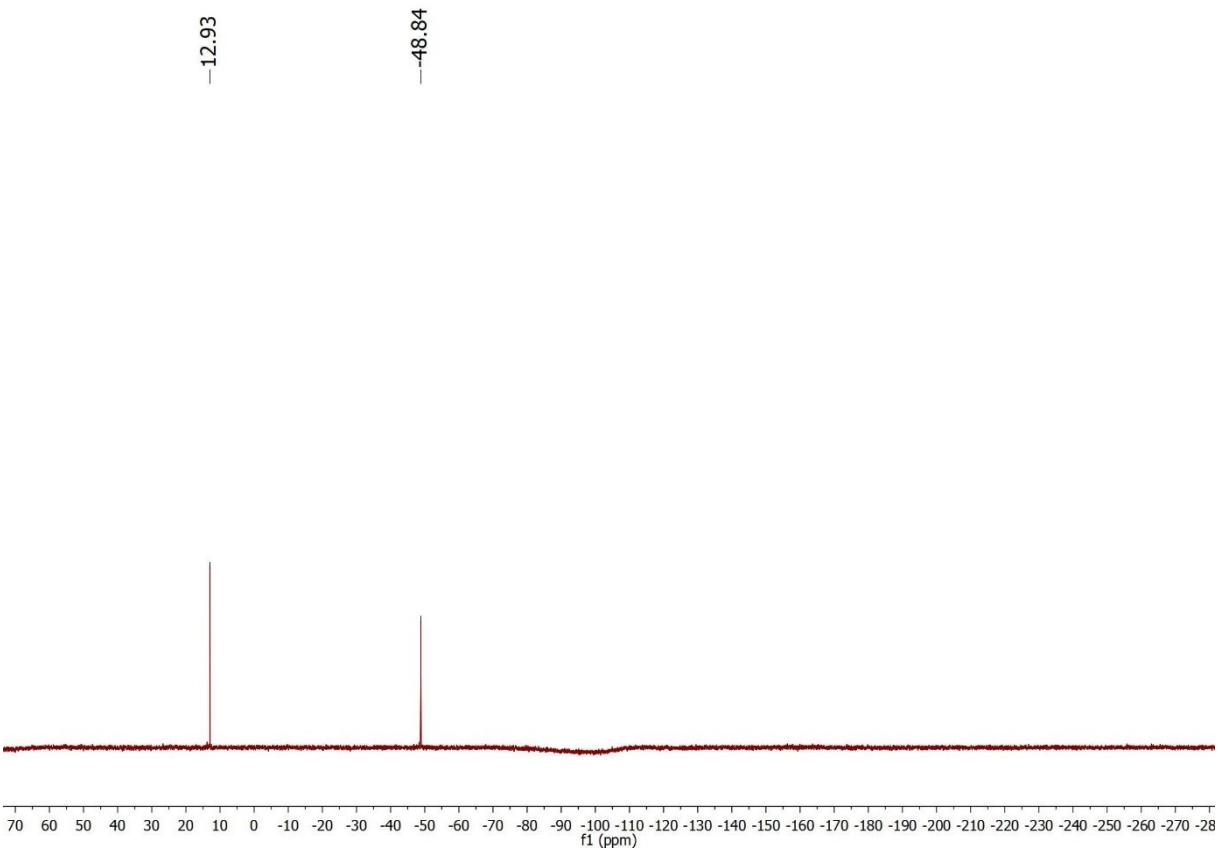
^{1}H NMR



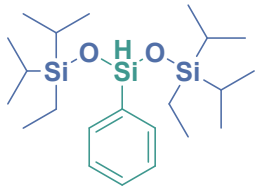
^{13}C NMR



^{29}Si NMR



1,5-Diethyl-1,1,5,5-tetraisopropyl-3-phenyltrisiloxane 4d



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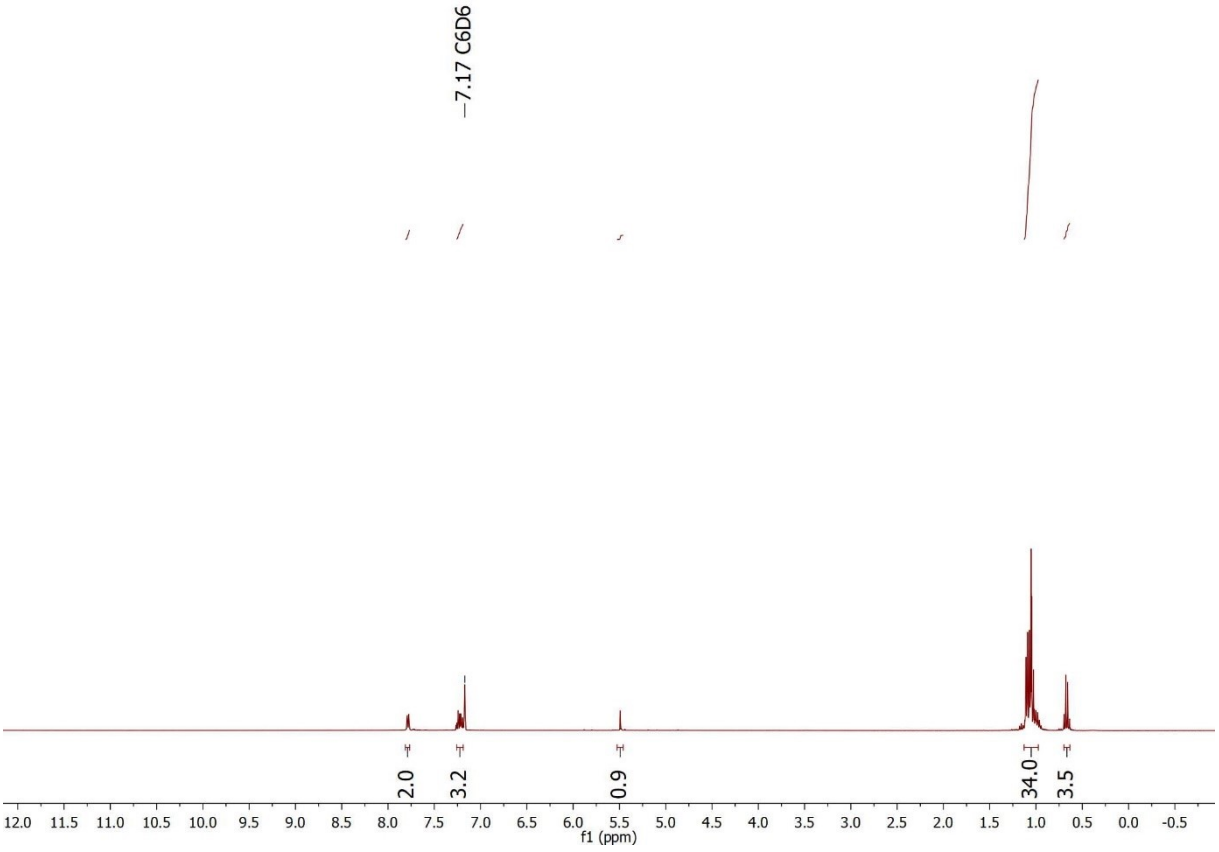

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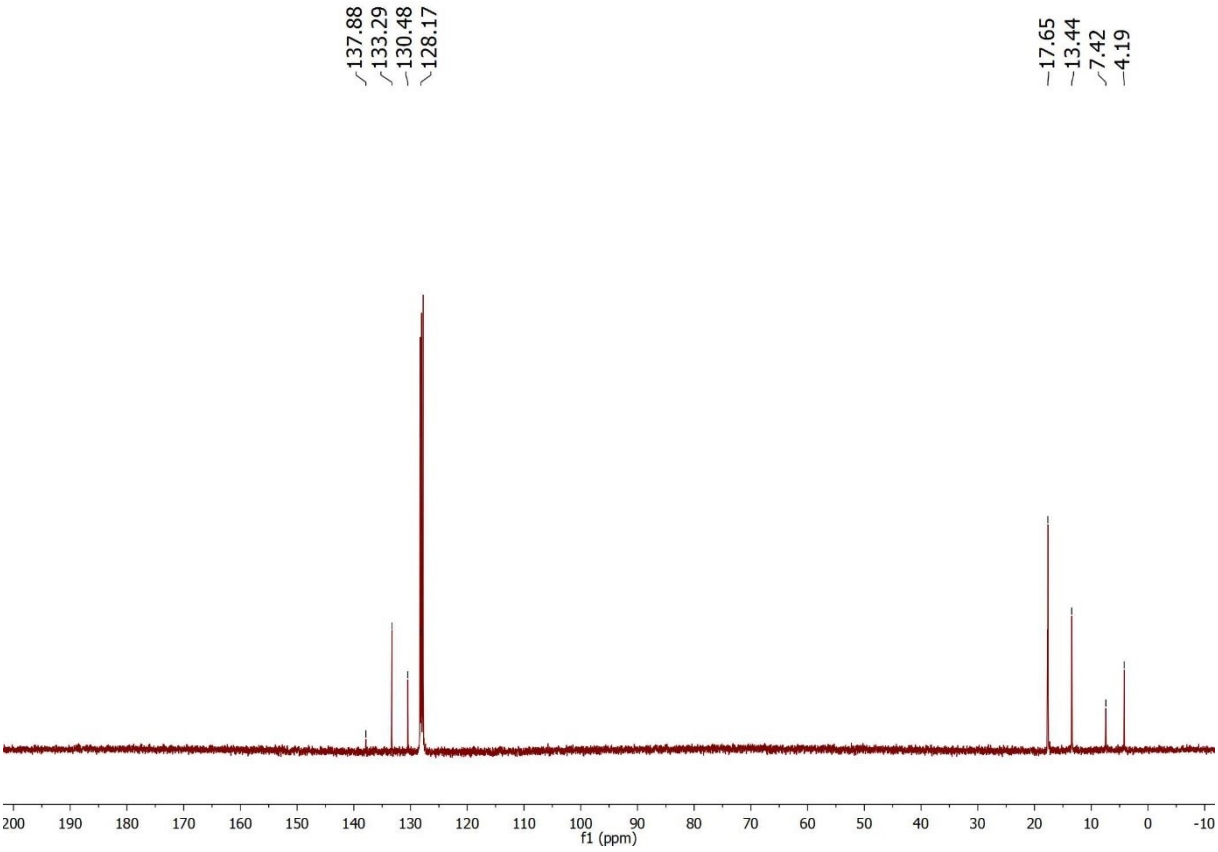

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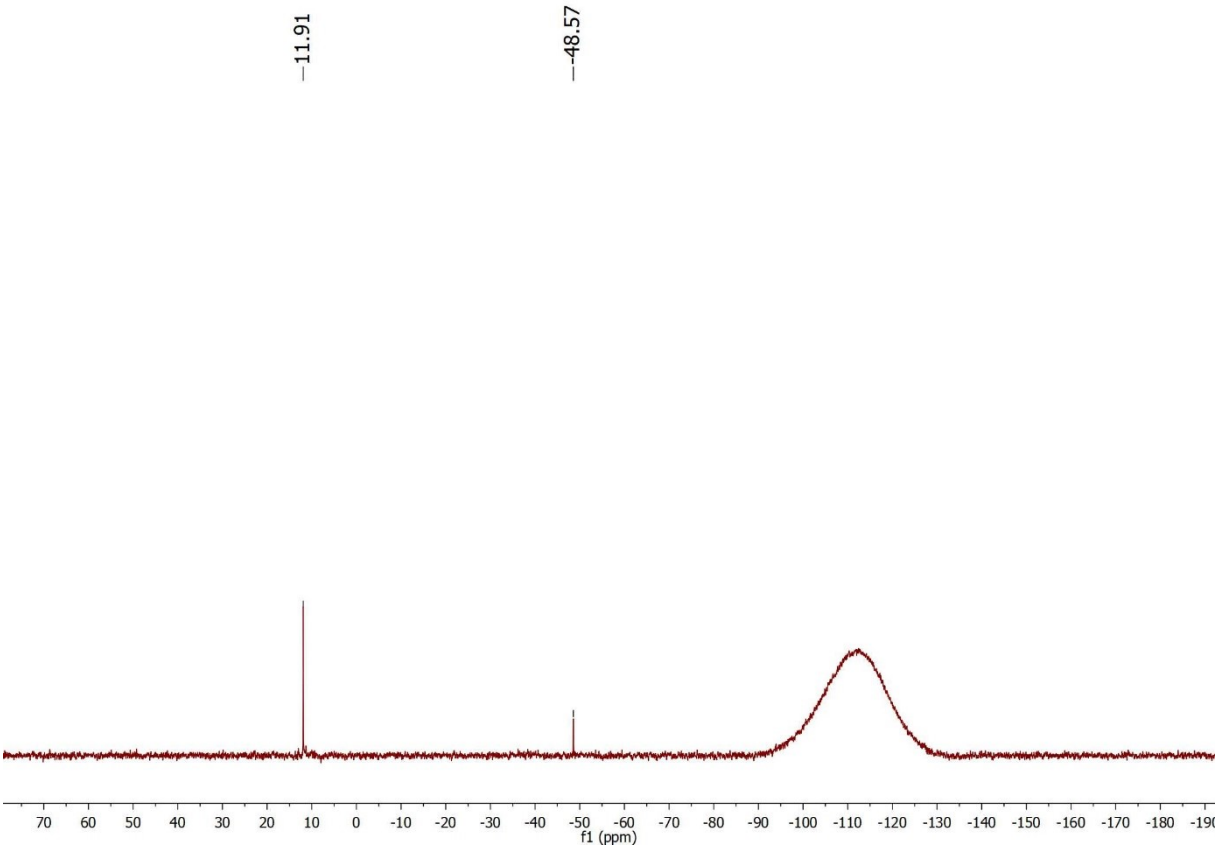
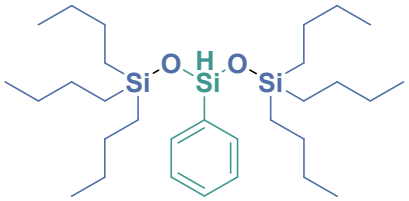


1,1,1,5,5,5-Hexabutyl-3-phenyltrisiloxane 4e



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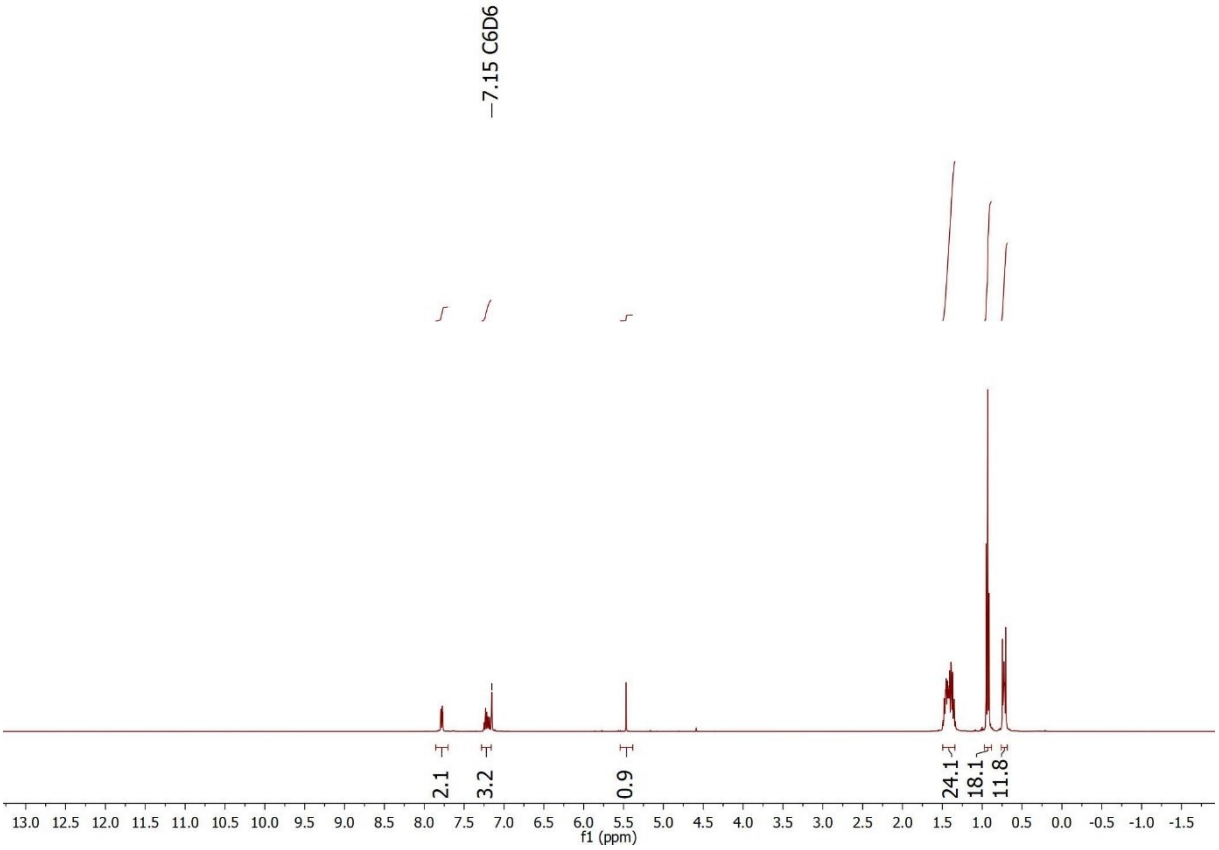

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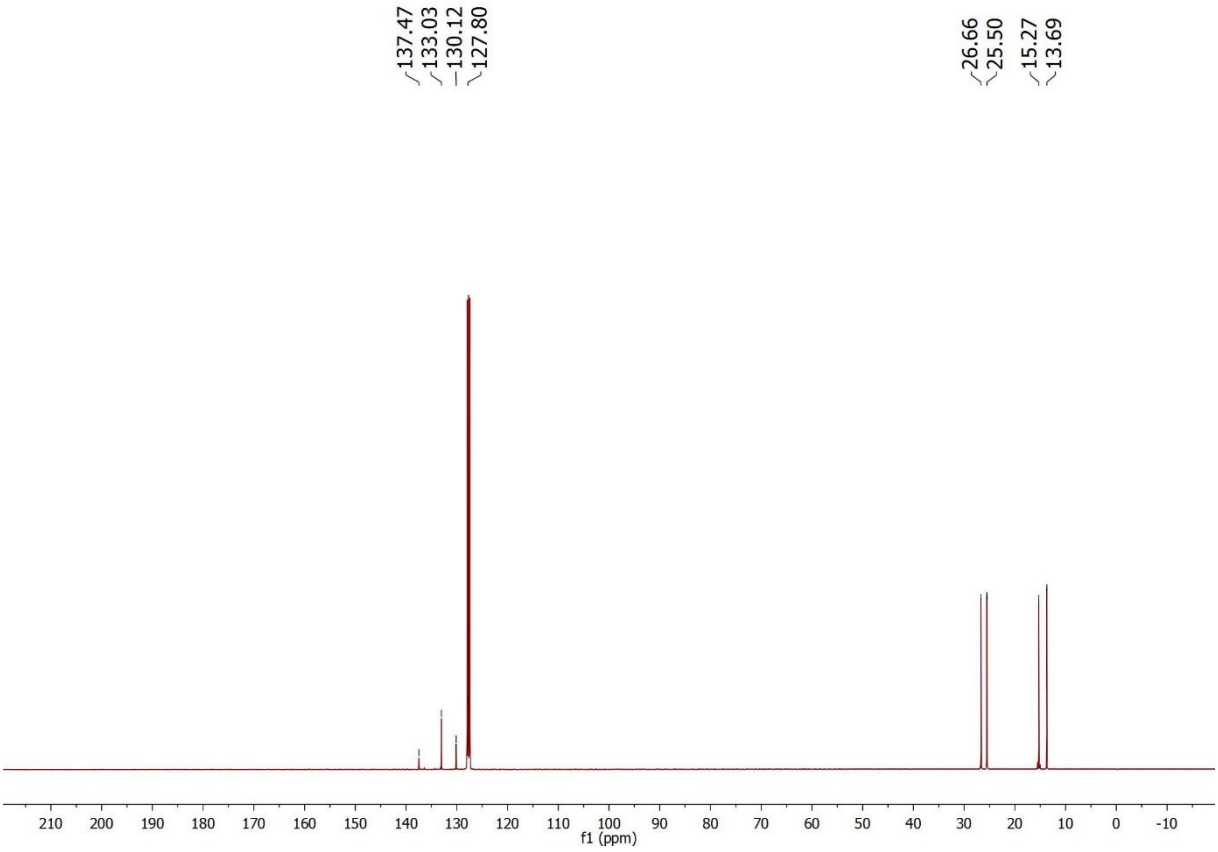

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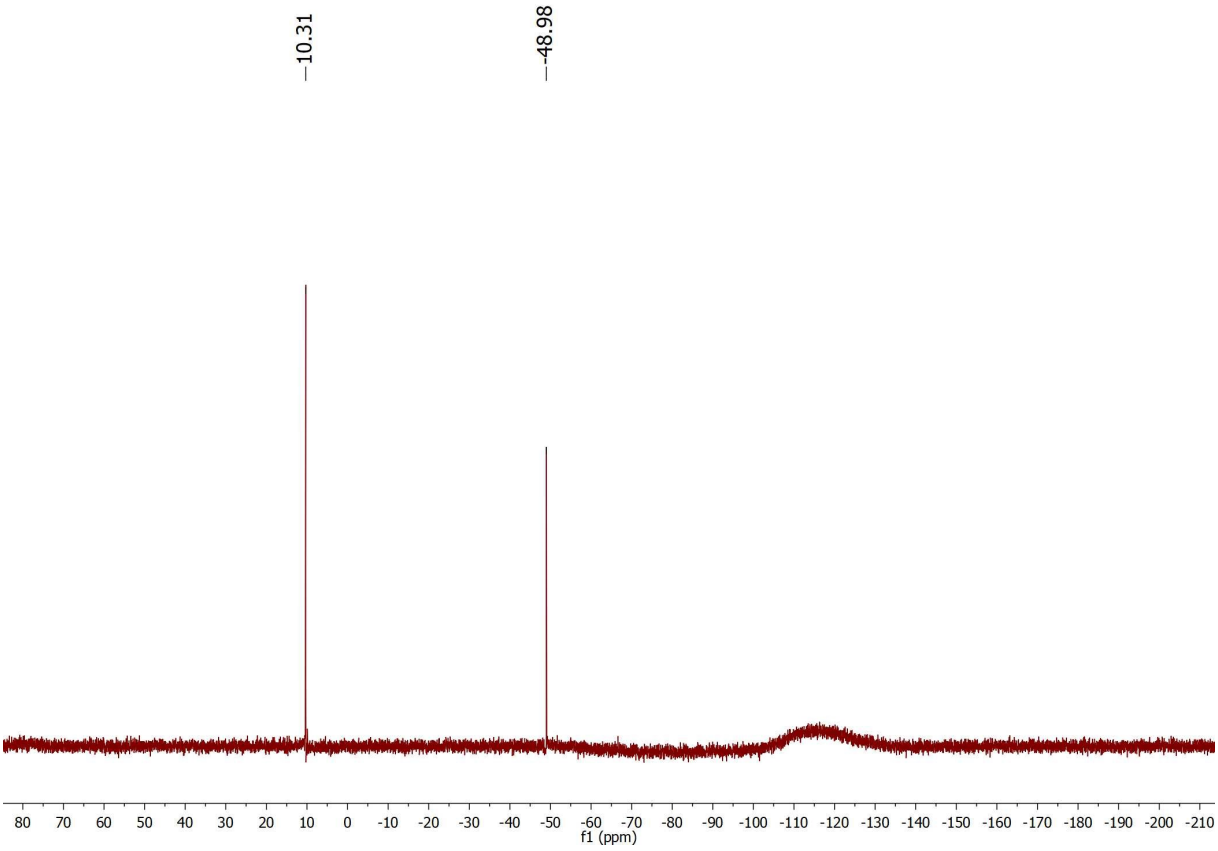
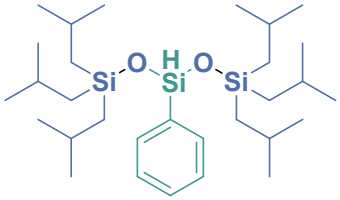


1,1,1,5,5,5-Hexaisobutyl-3-phenyltrisiloxane 4f



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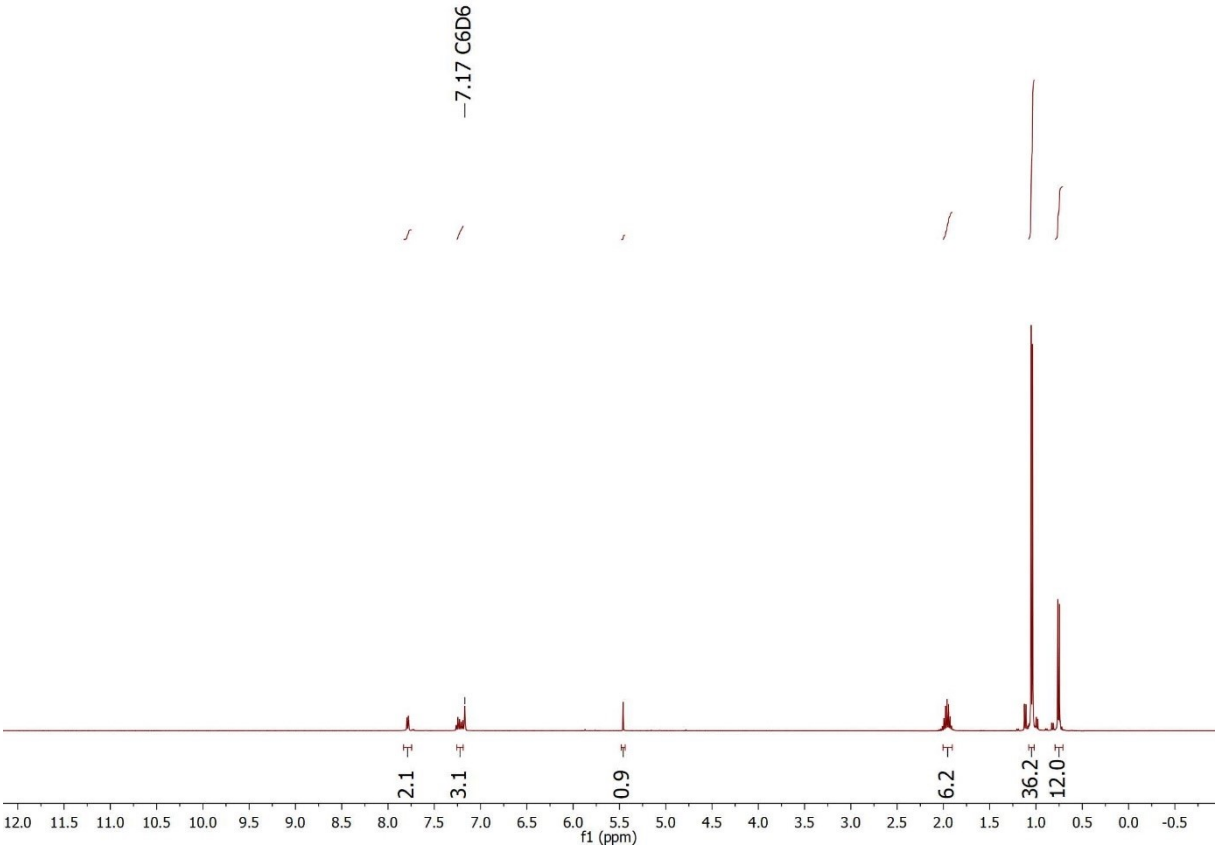

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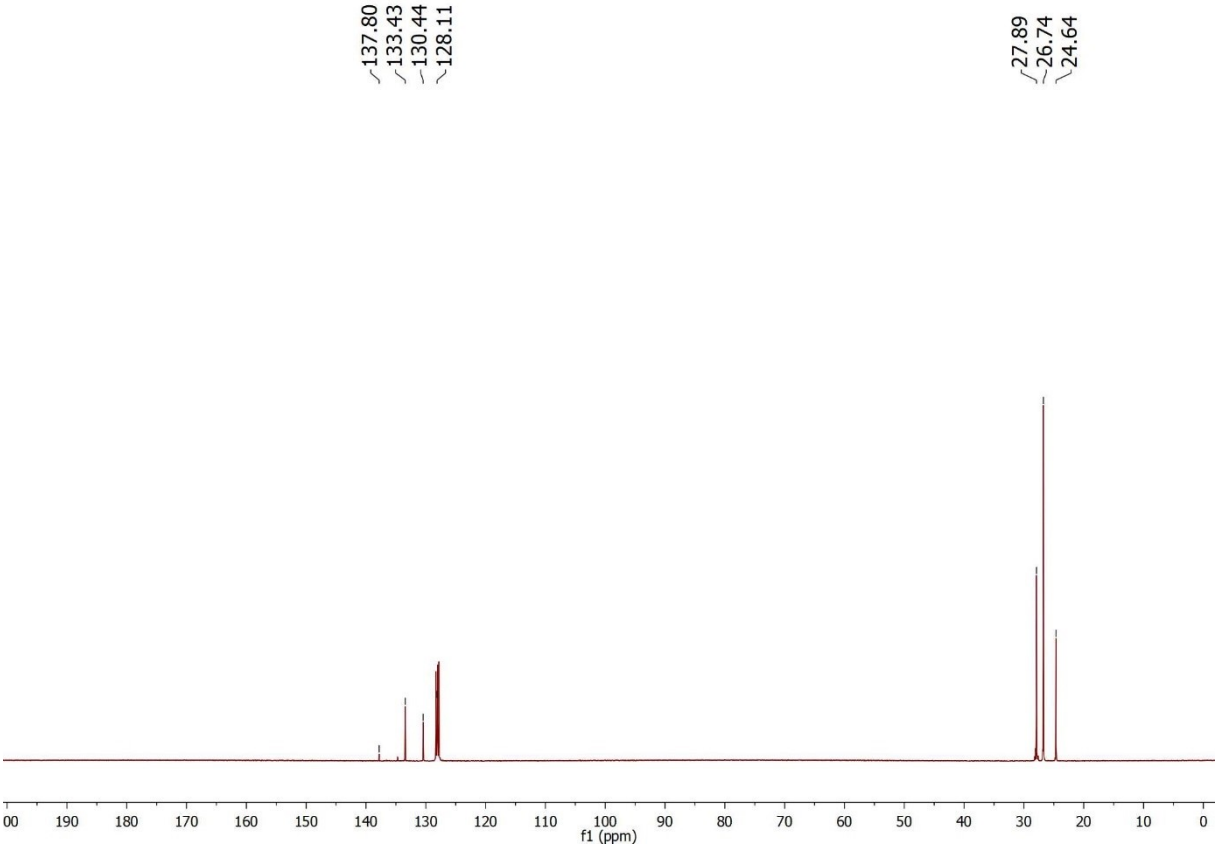

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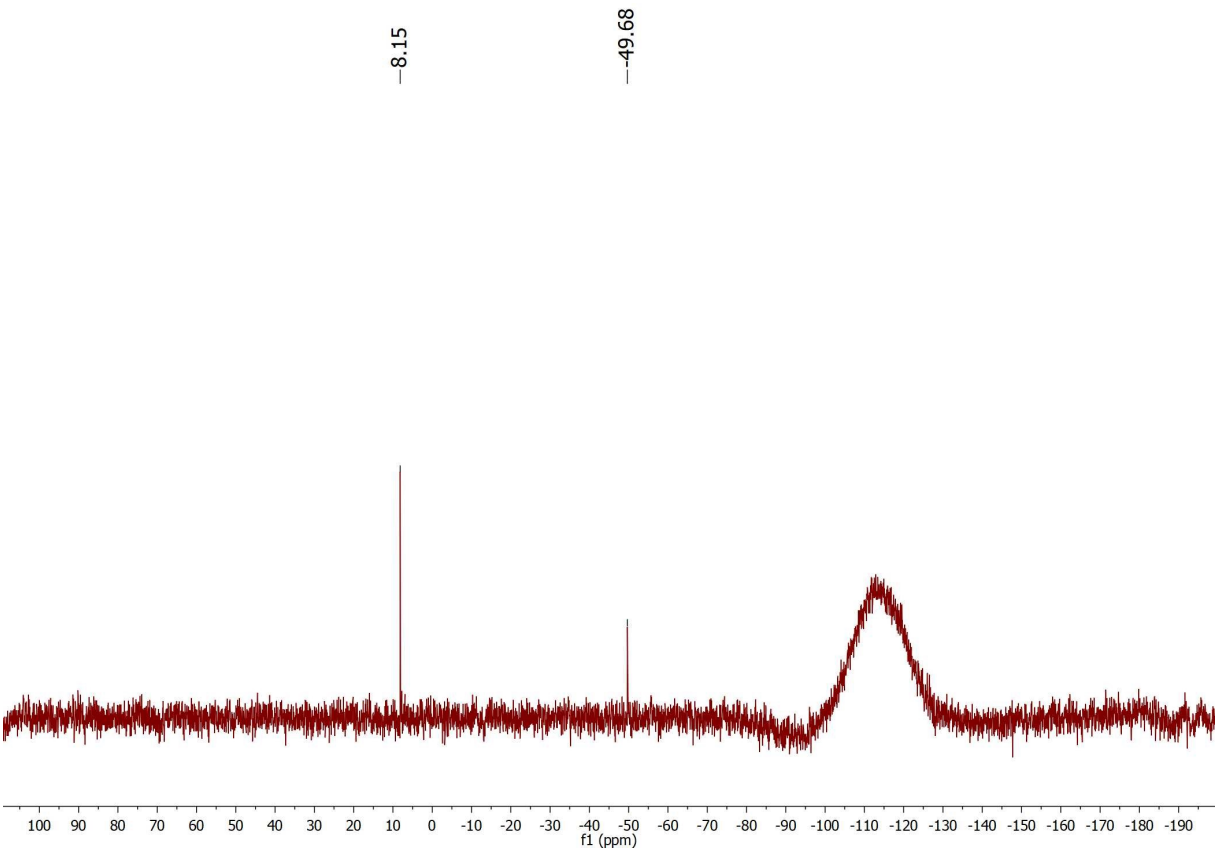
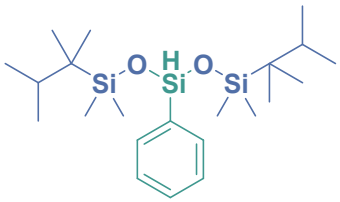


1,5-Bis(2,3-dimethylbutan-2-yl)-1,1,5,5-tetramethyl-3-phenyltrisiloxane 4g



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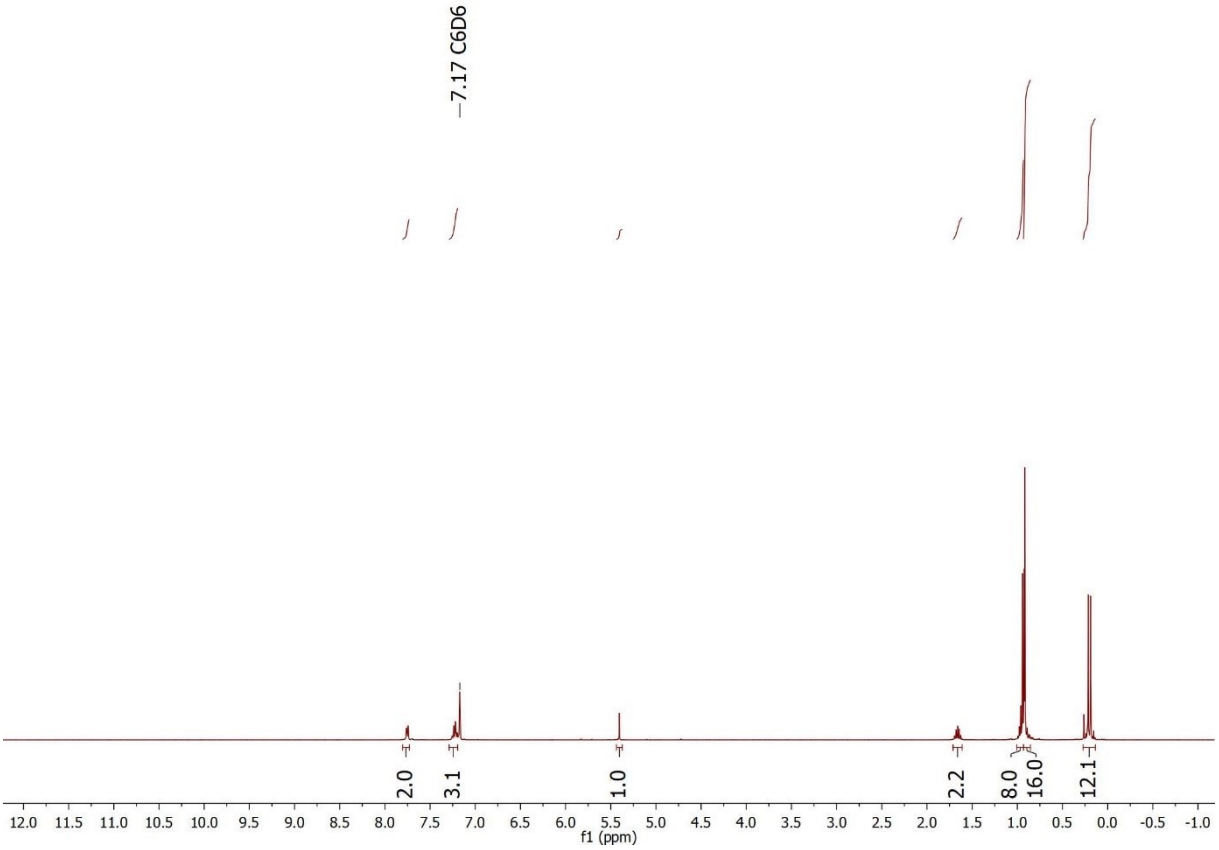

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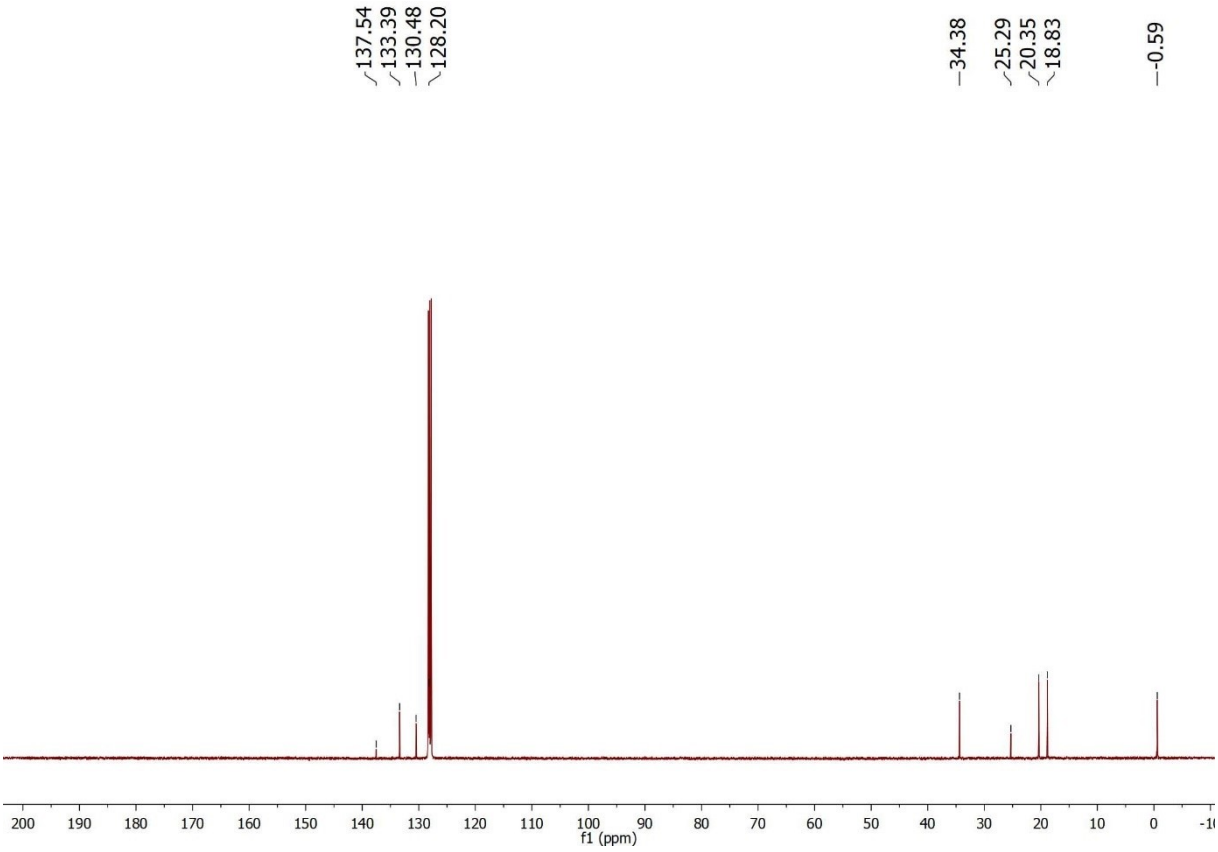

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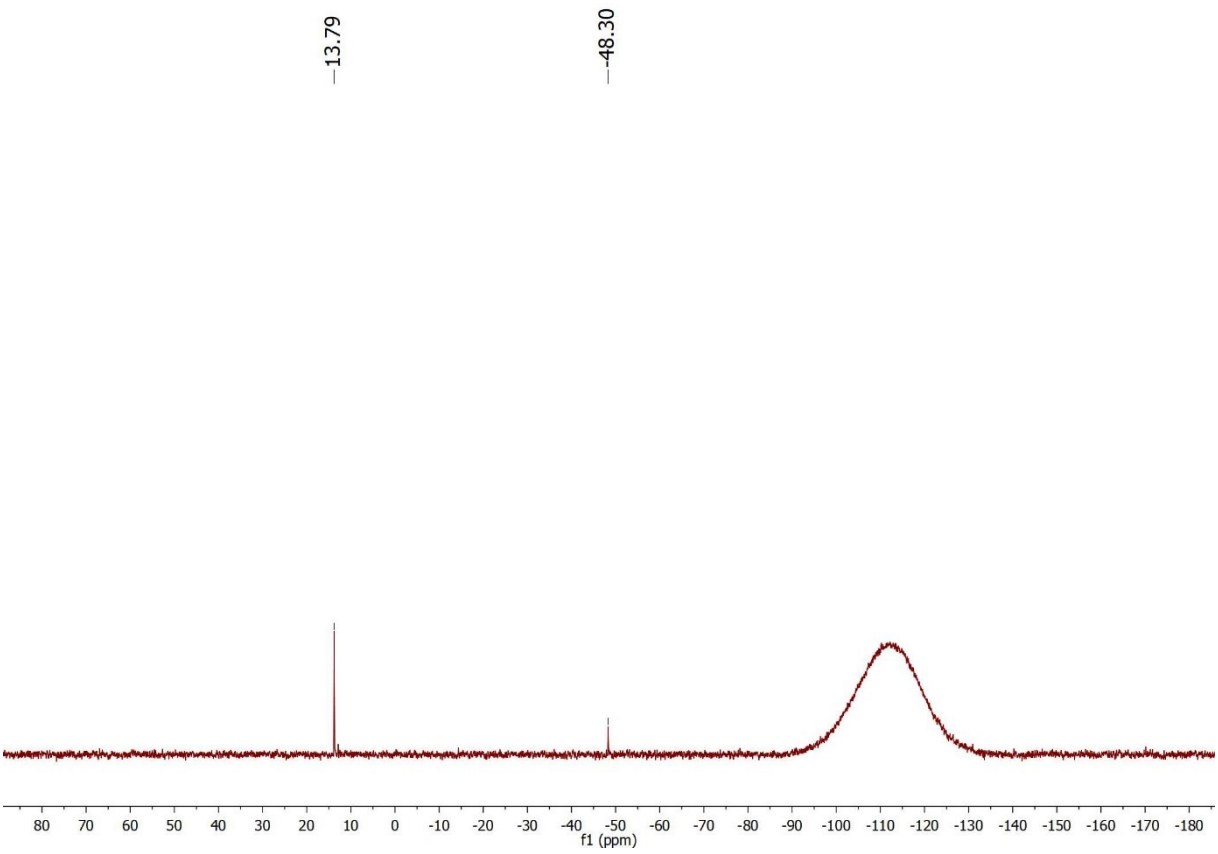
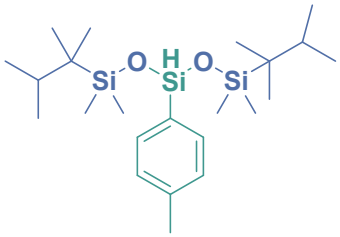


1,5-Bis(2,3-dimethylbutan-2-yl)-1,1,5,5-tetramethyl-3-(p-tolyl)trisiloxane 4h



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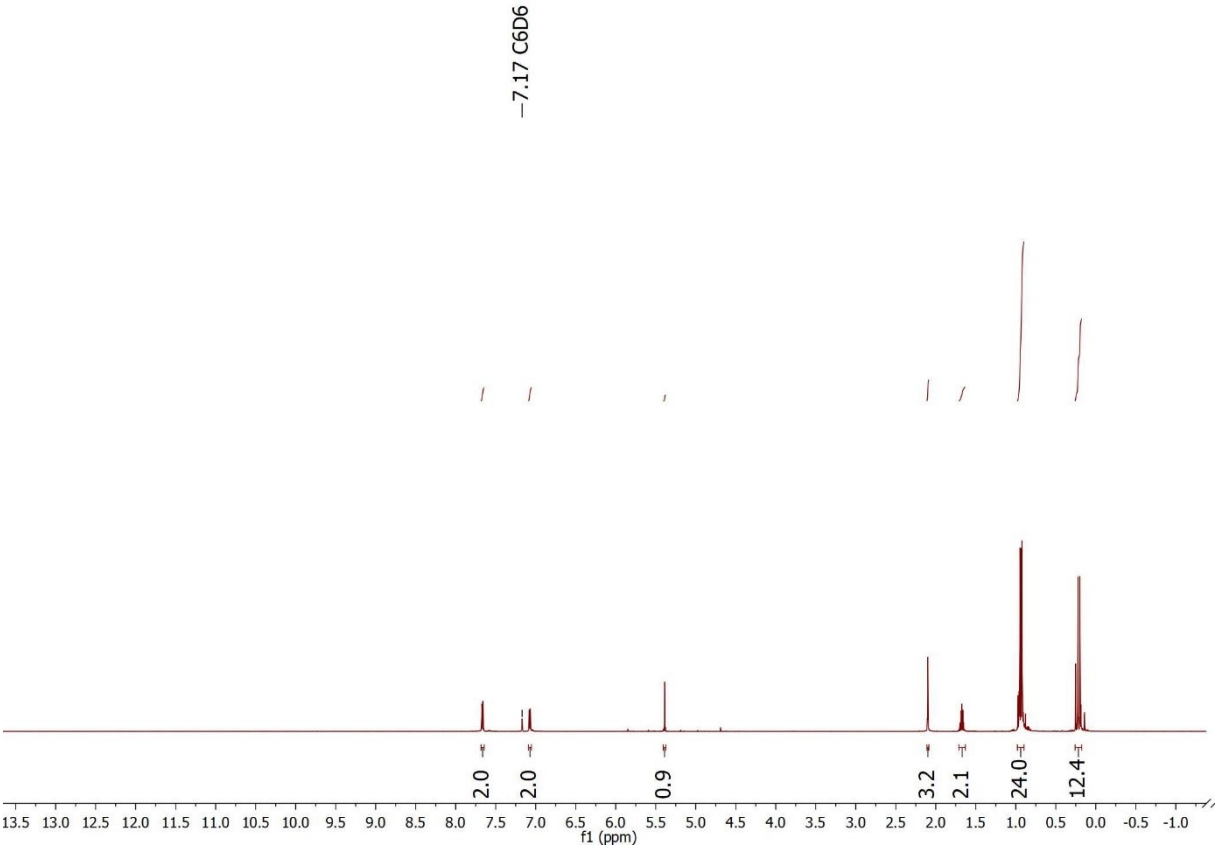

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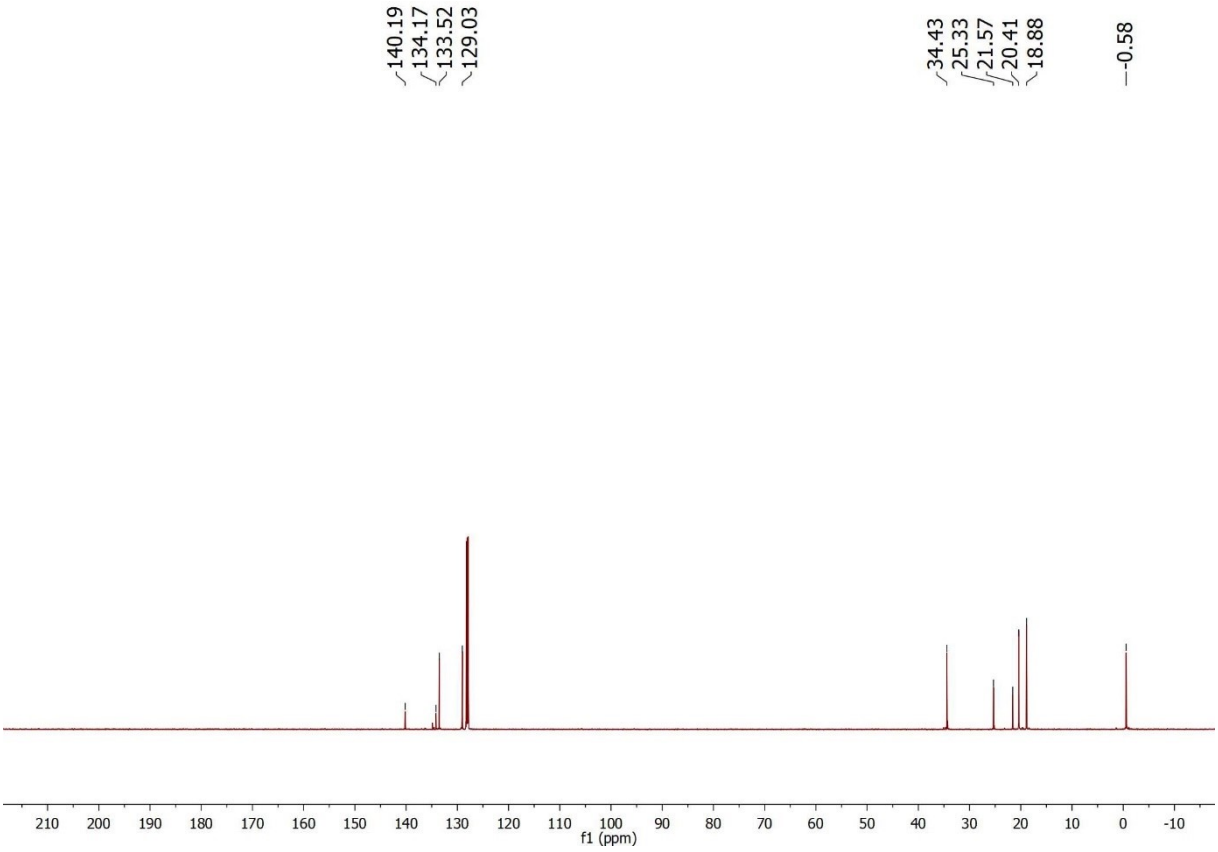
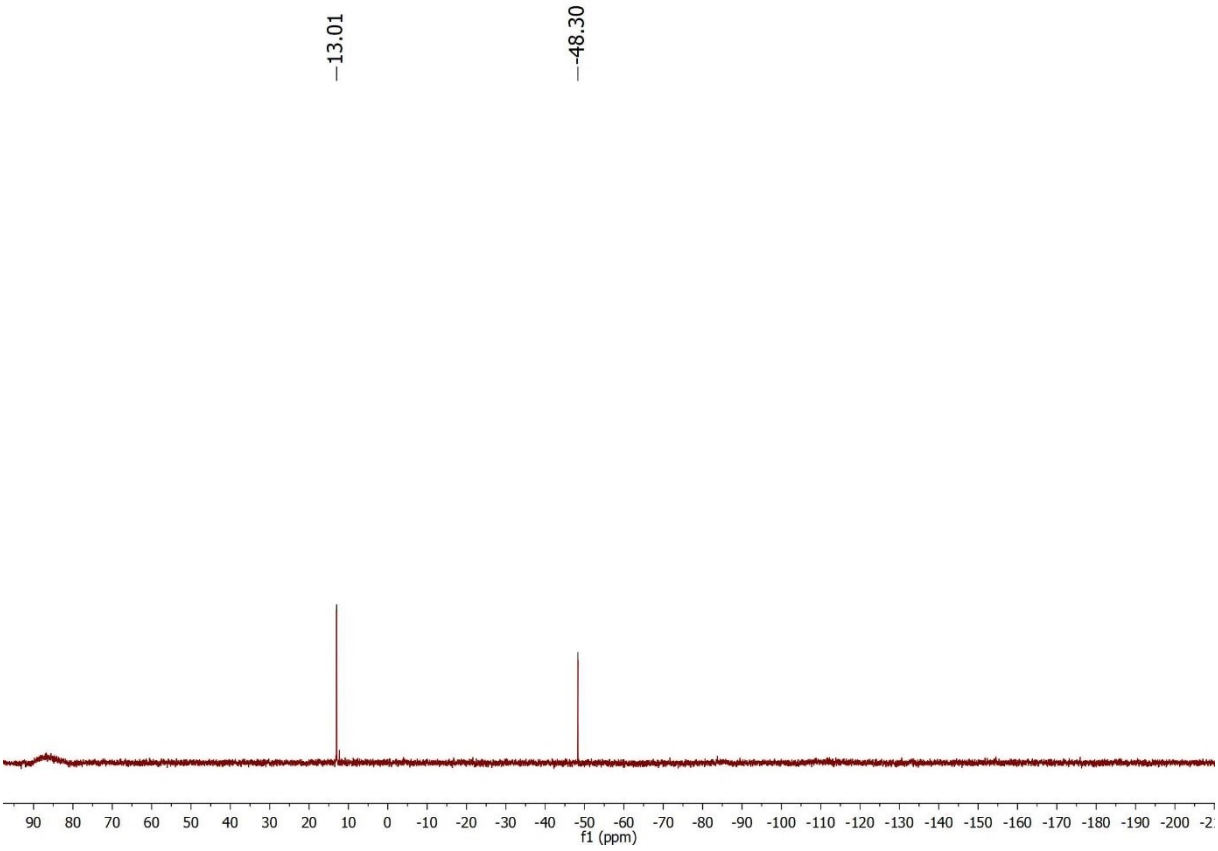
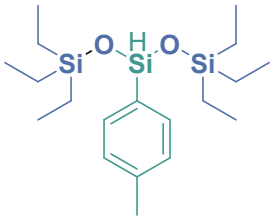


^{29}Si NMR



1,1,1,5,5,5-Hexaethyl-3-(p-tolyl)trisiloxane 4i



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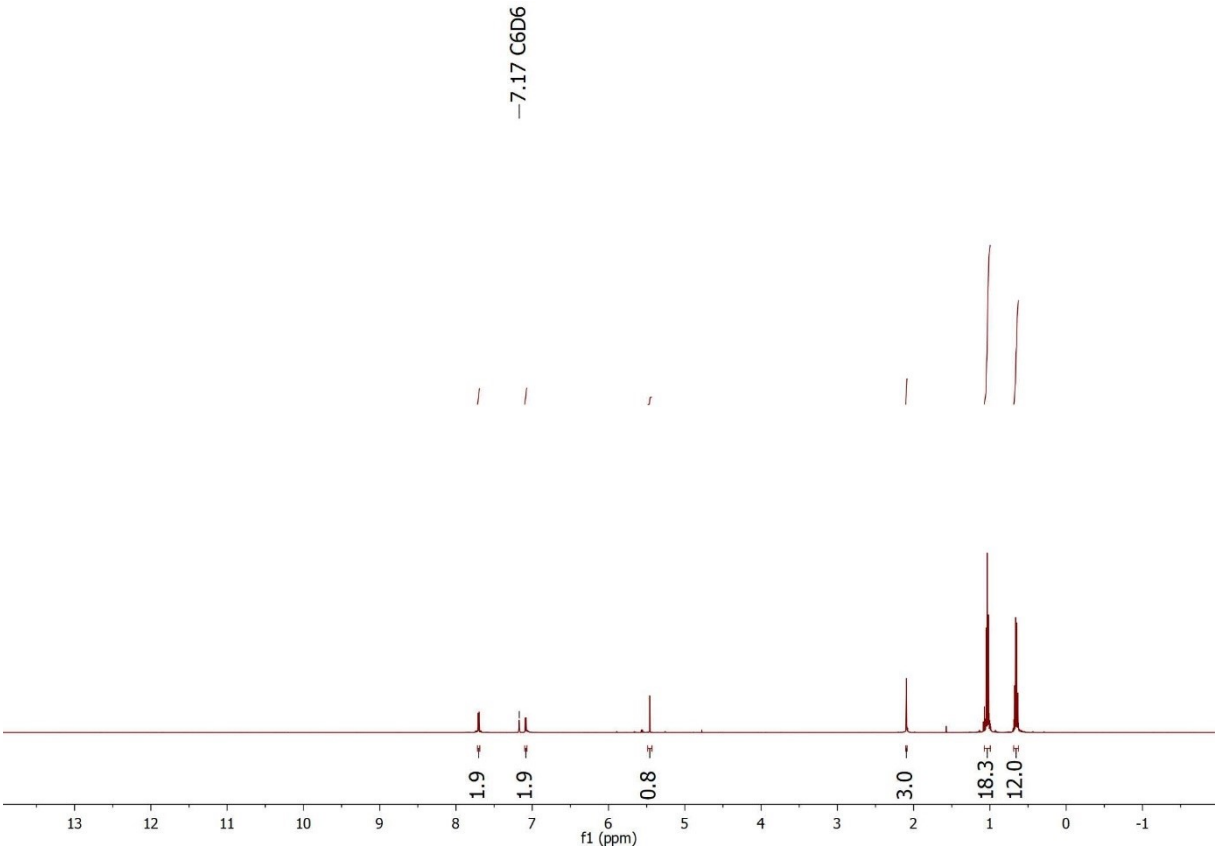

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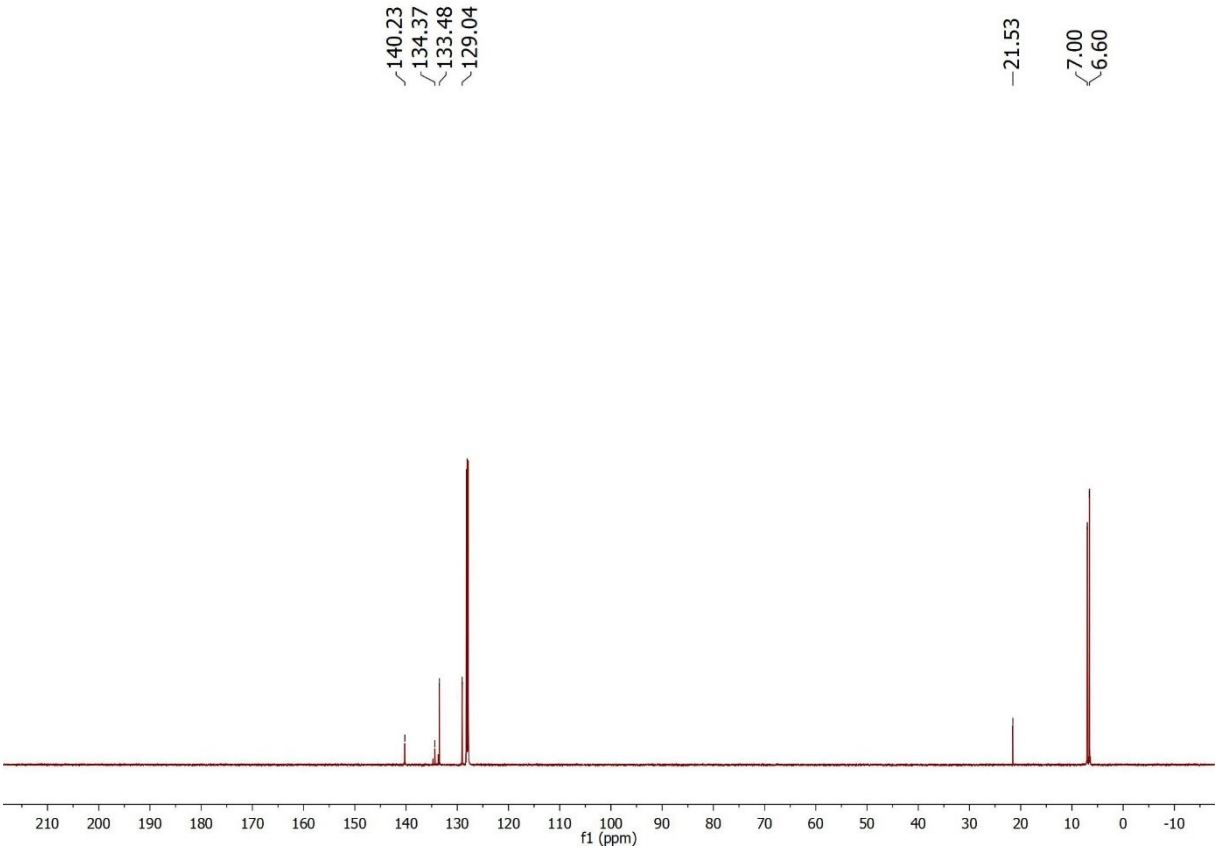

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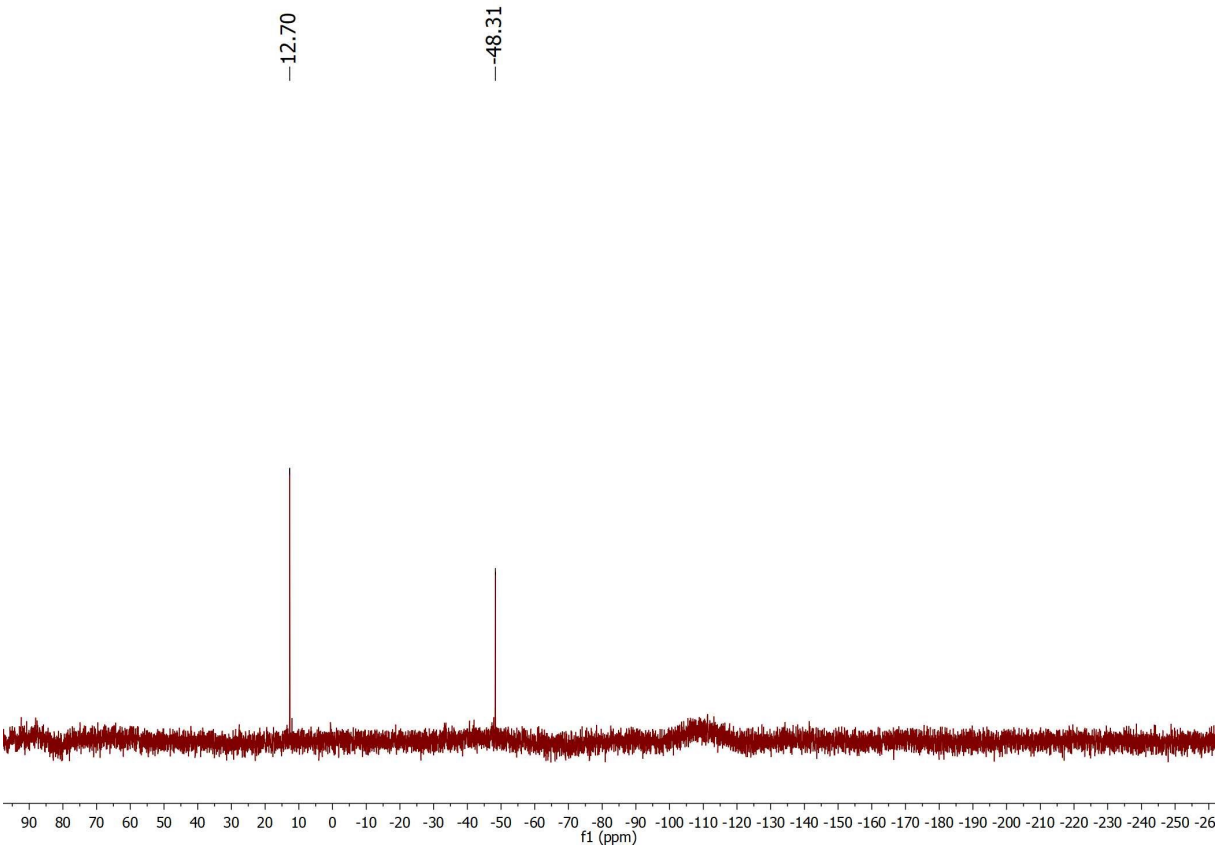
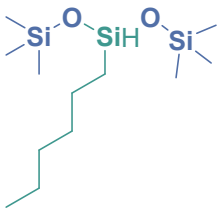


3-Hexyl-1,1,1,5,5,5-hexamethyltrisiloxane 4j



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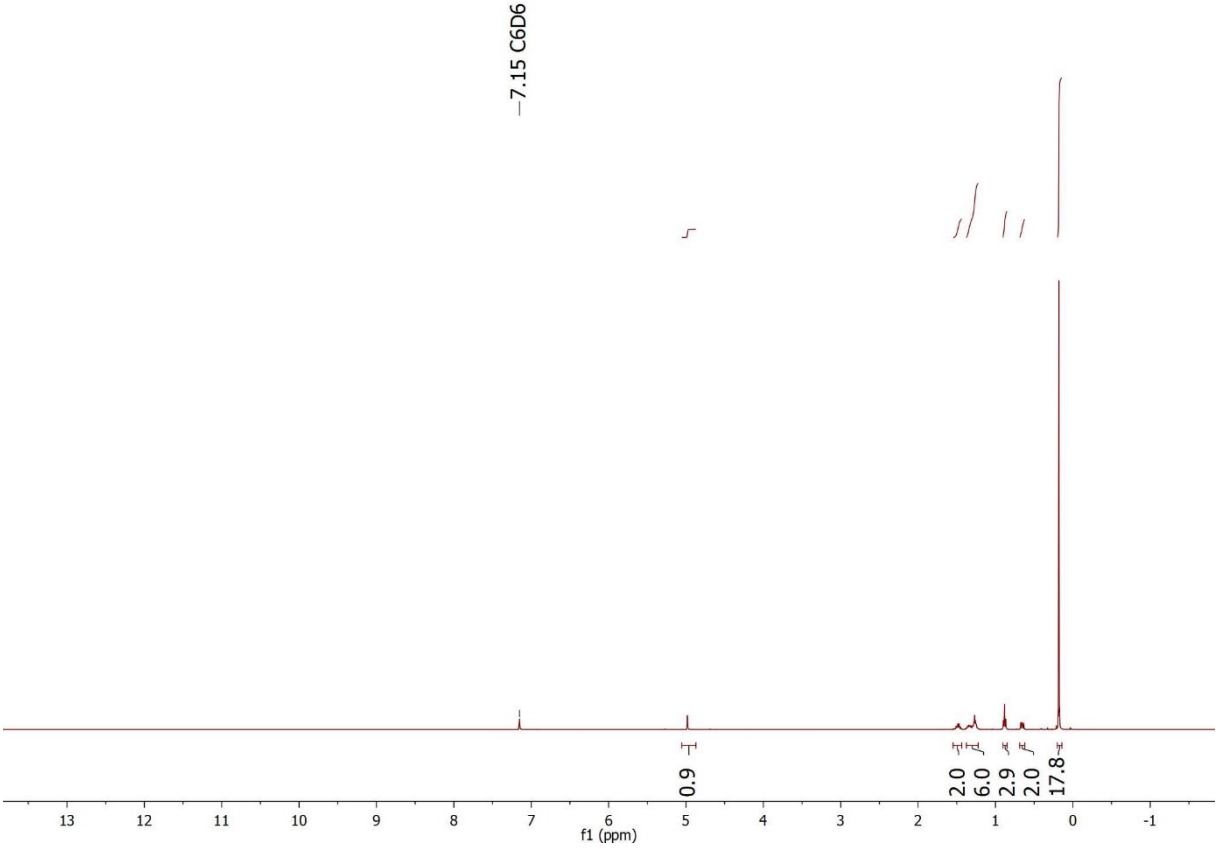

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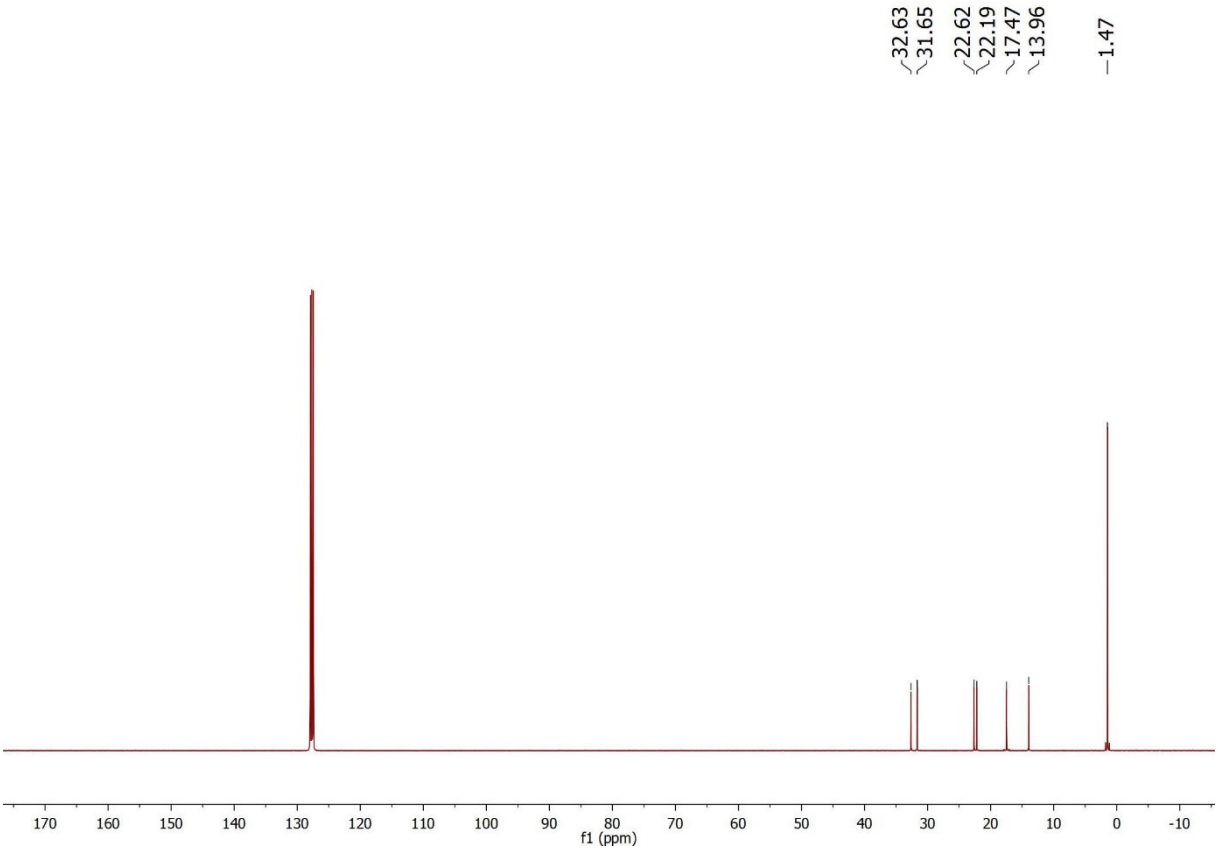

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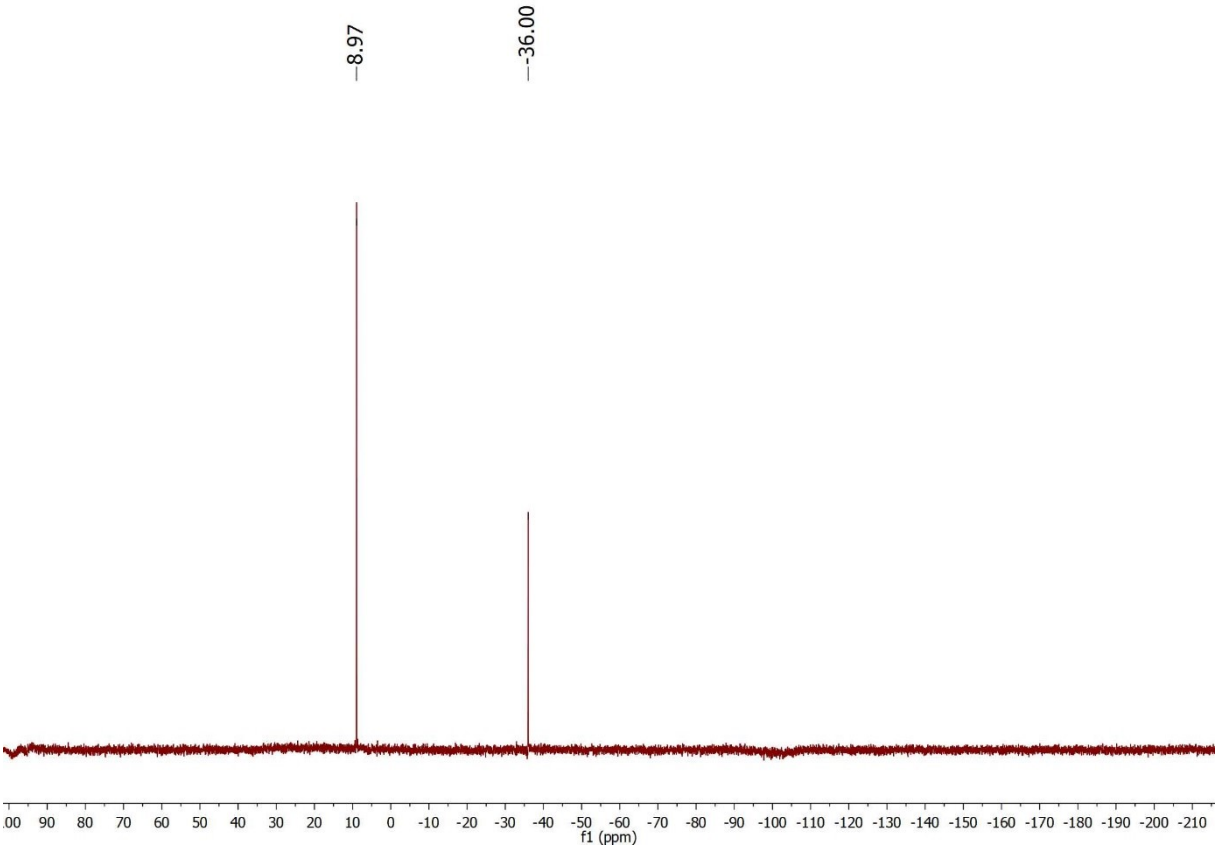
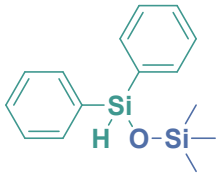
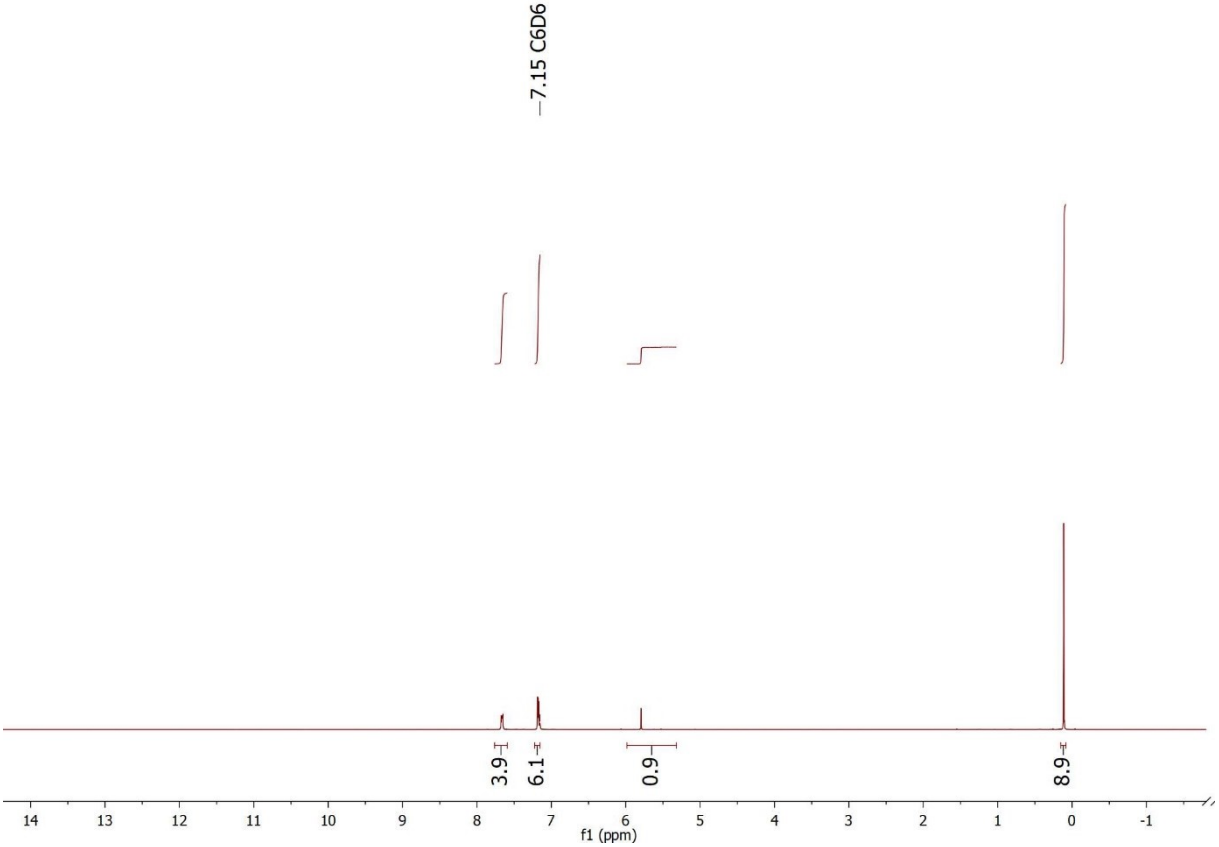


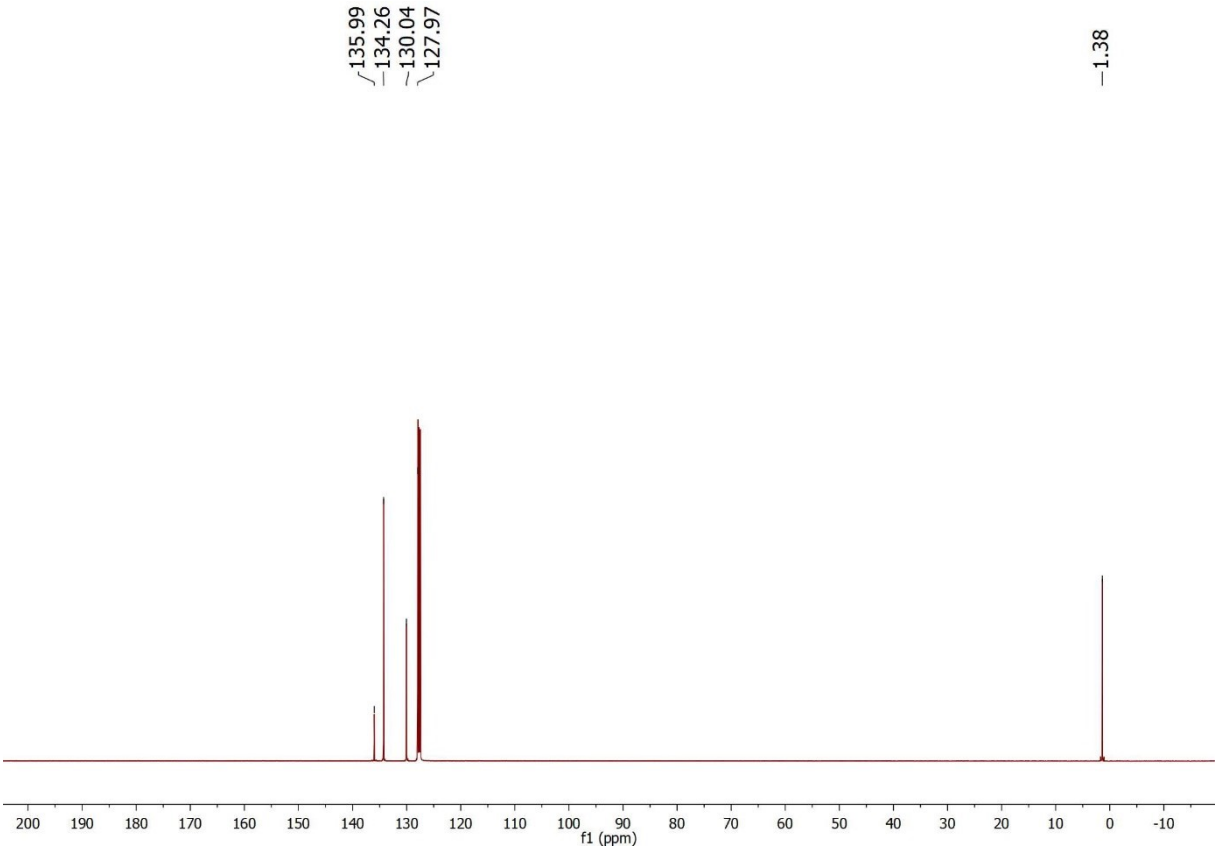
1,1,1-Trimethyl-3,3-diphenyldisiloxane 5a



^{1}H NMR



^{13}C NMR



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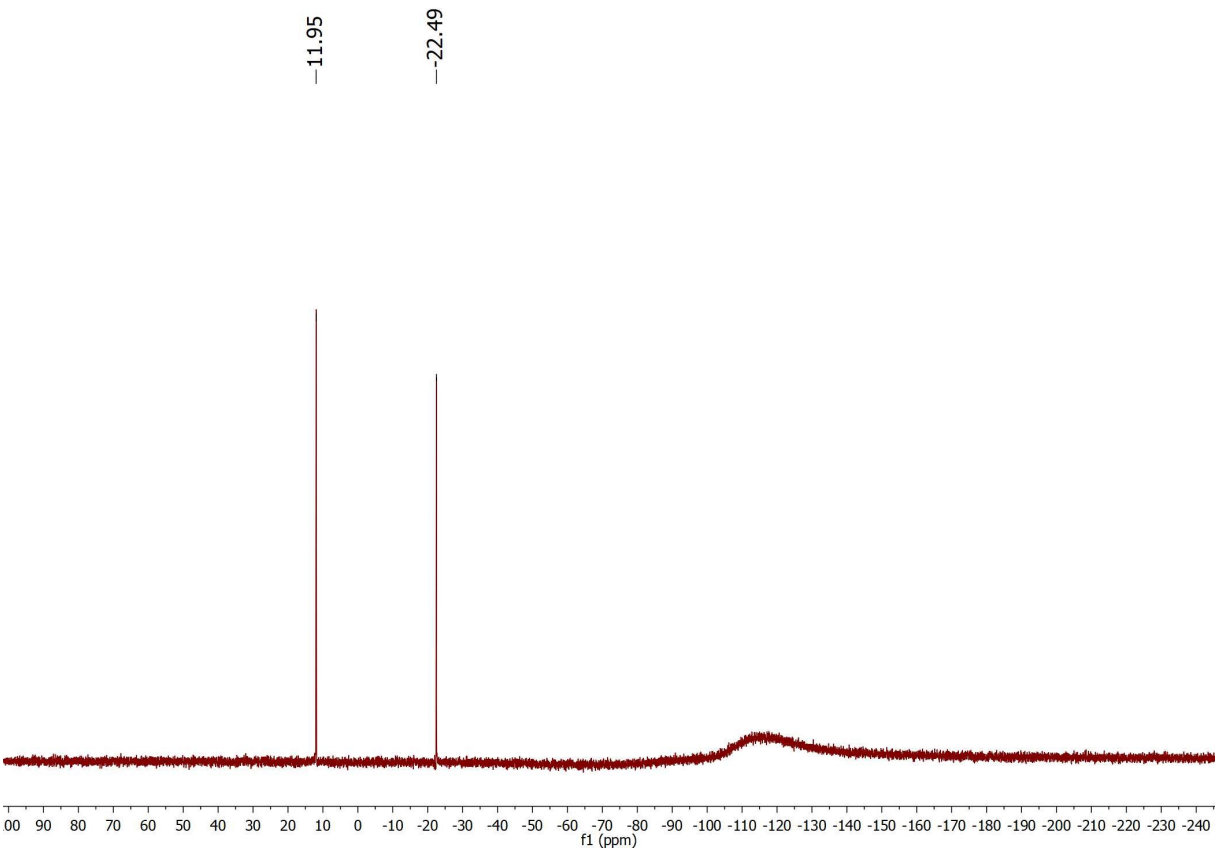
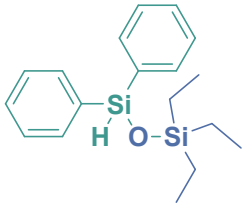


1,1,1-Triethyl-3,3-diphenyldisiloxane 5b



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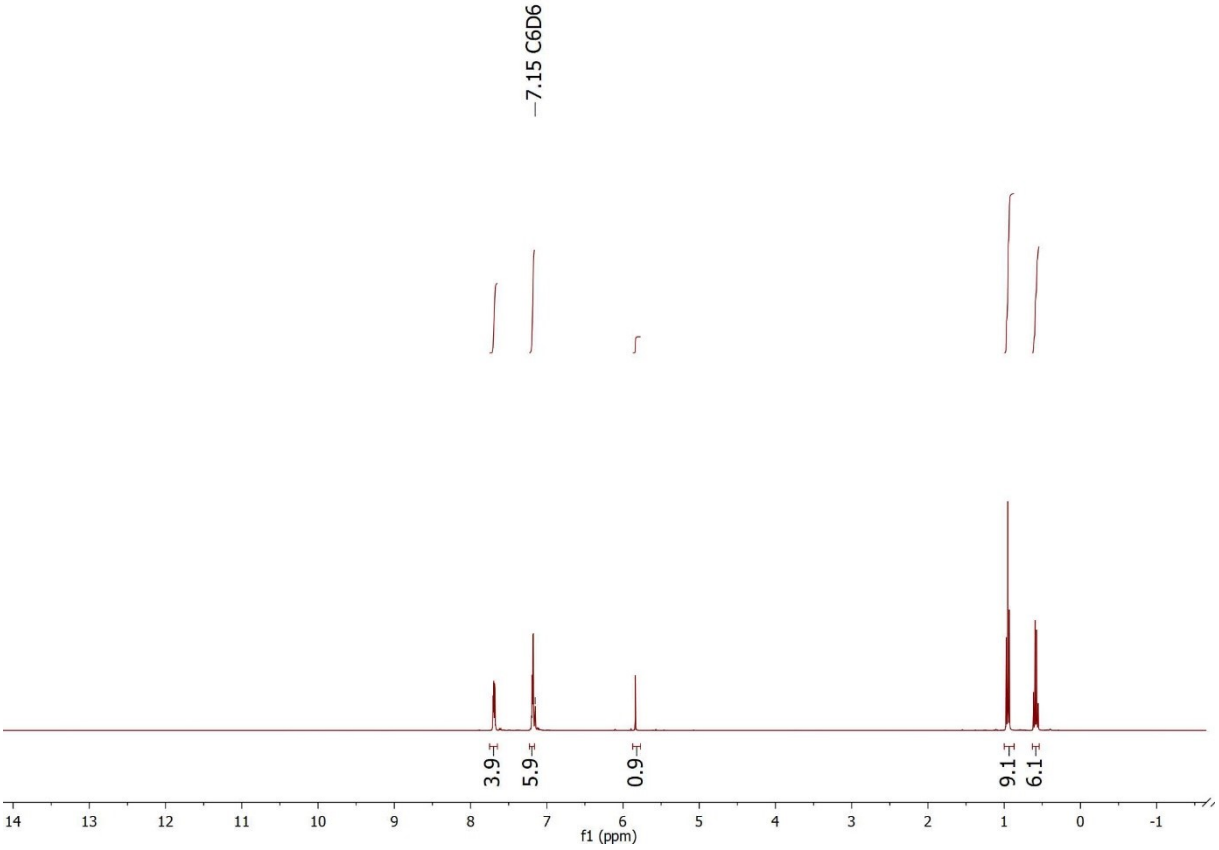
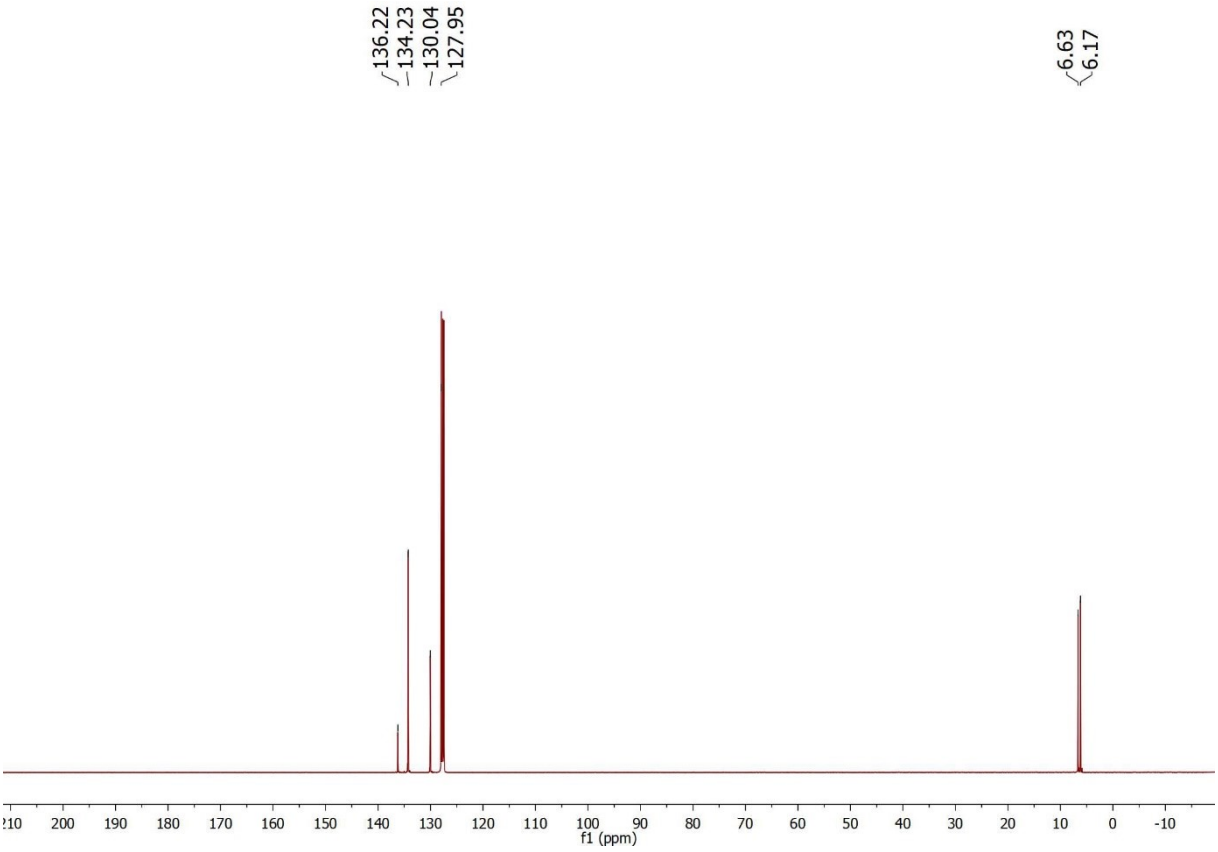
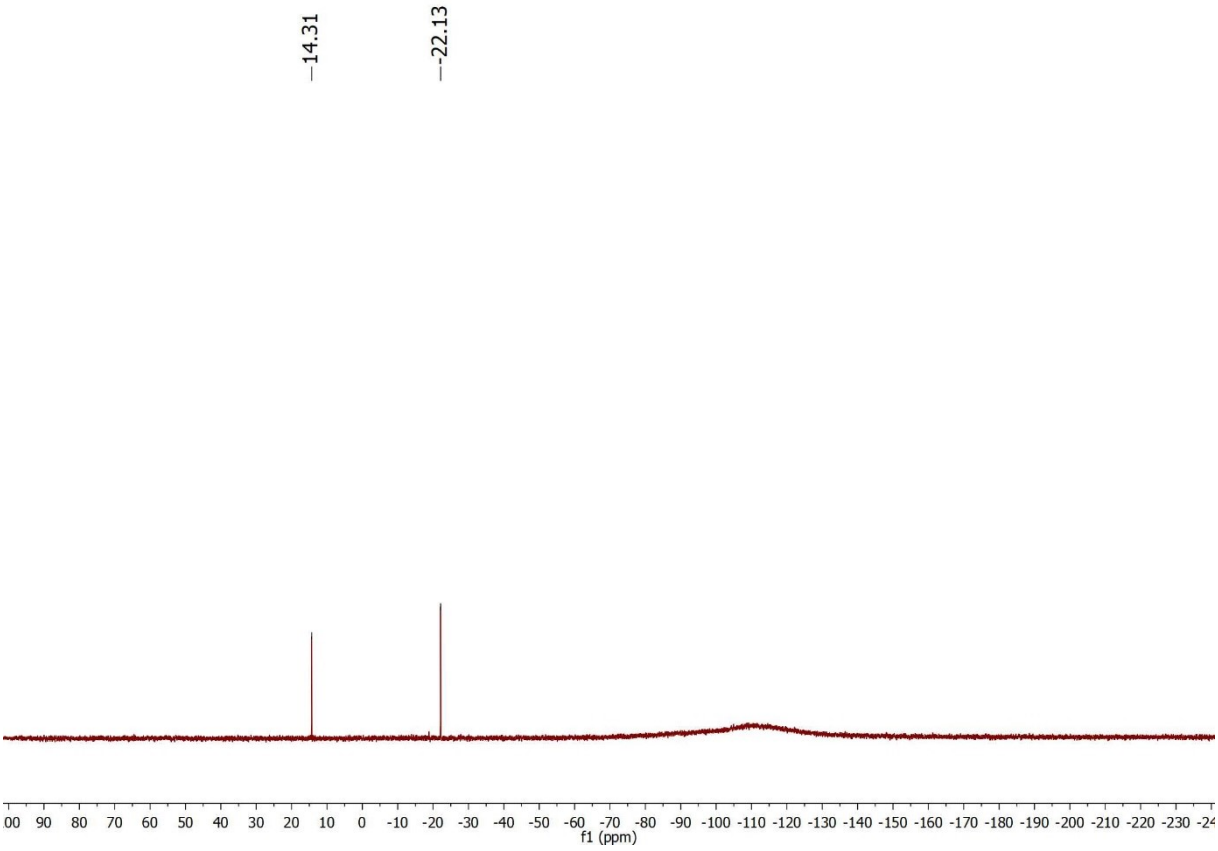


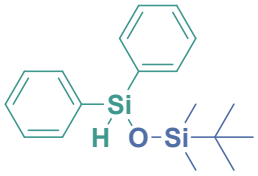
^{13}C NMR



^{29}Si NMR



1-(Tert-butyl)-1,1-dimethyl-3,3-diphenyldisiloxane 5c



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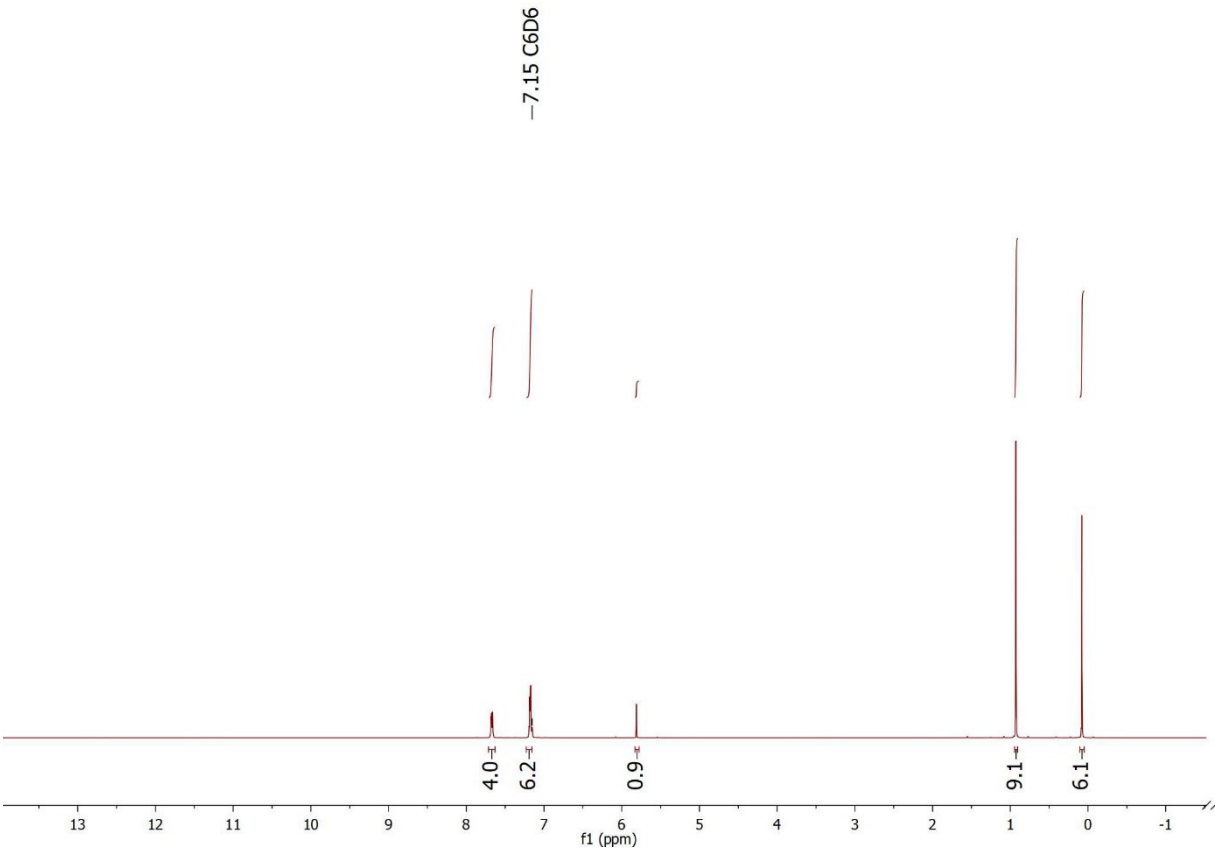

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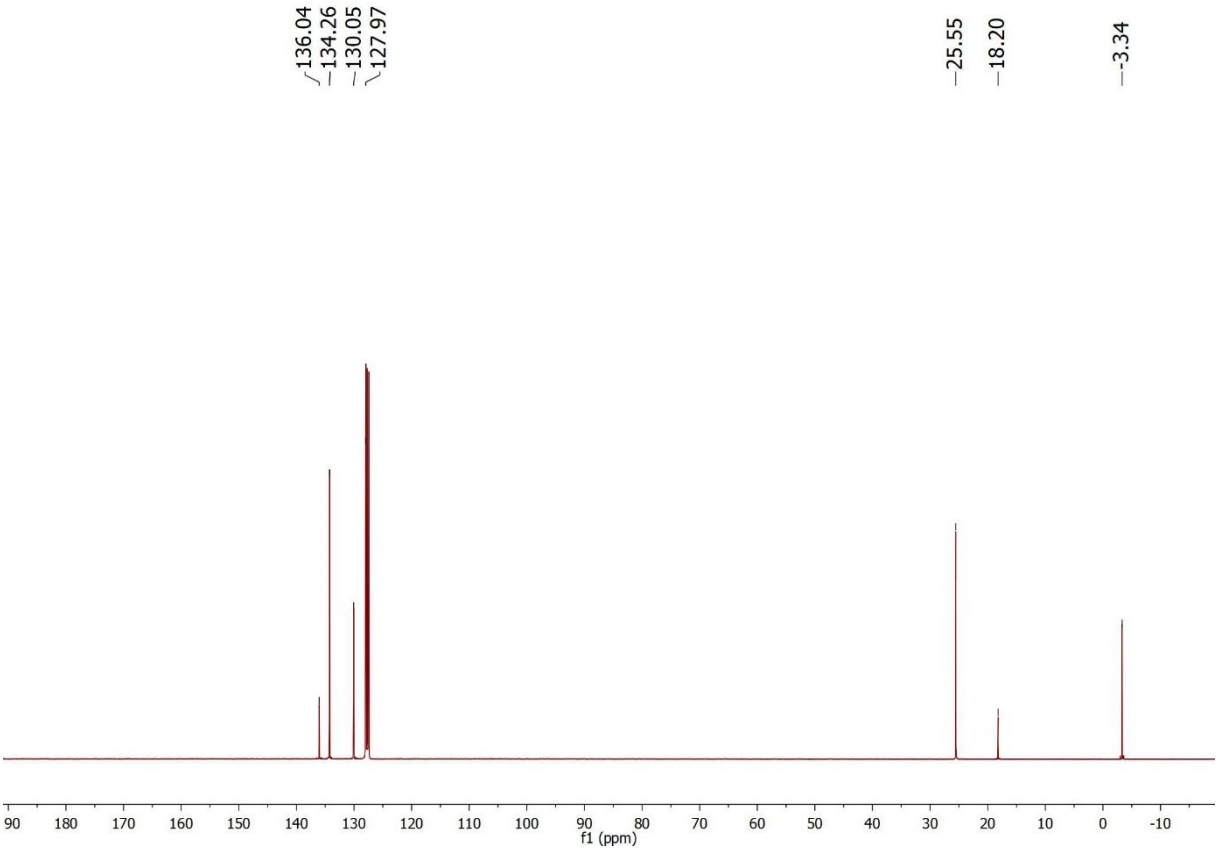
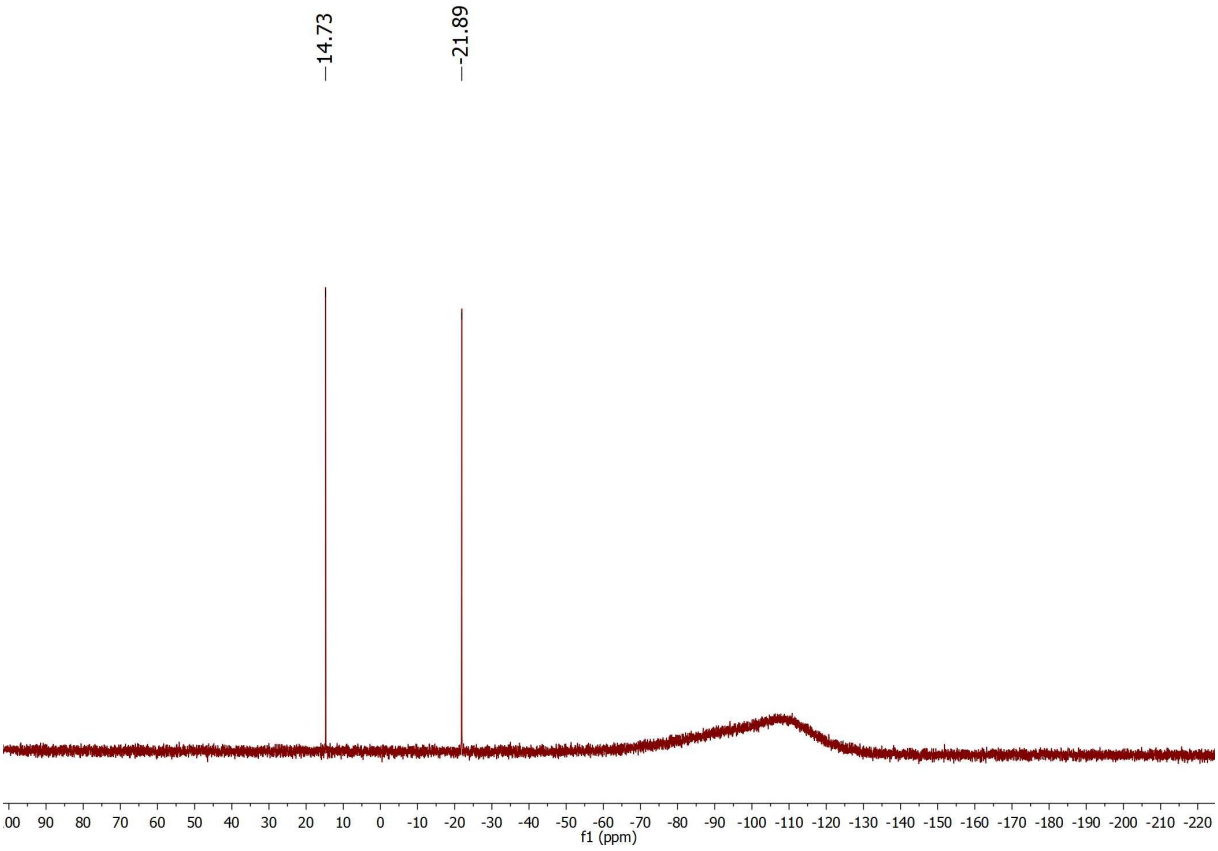
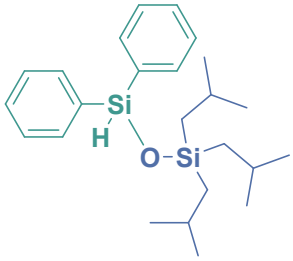


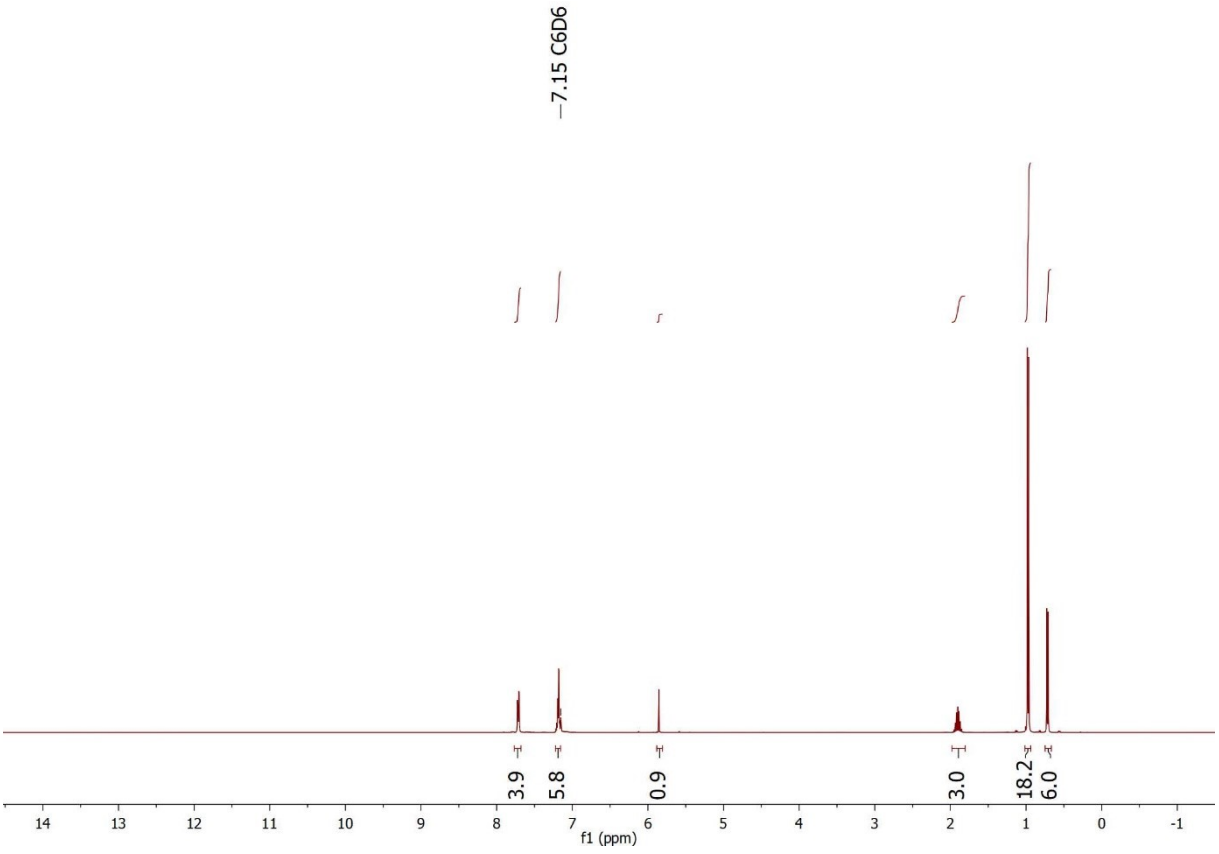
^{29}Si NMR



1,1,1-Triisobutyl-3,3-diphenyldisiloxane 5d



^{1}H NMR



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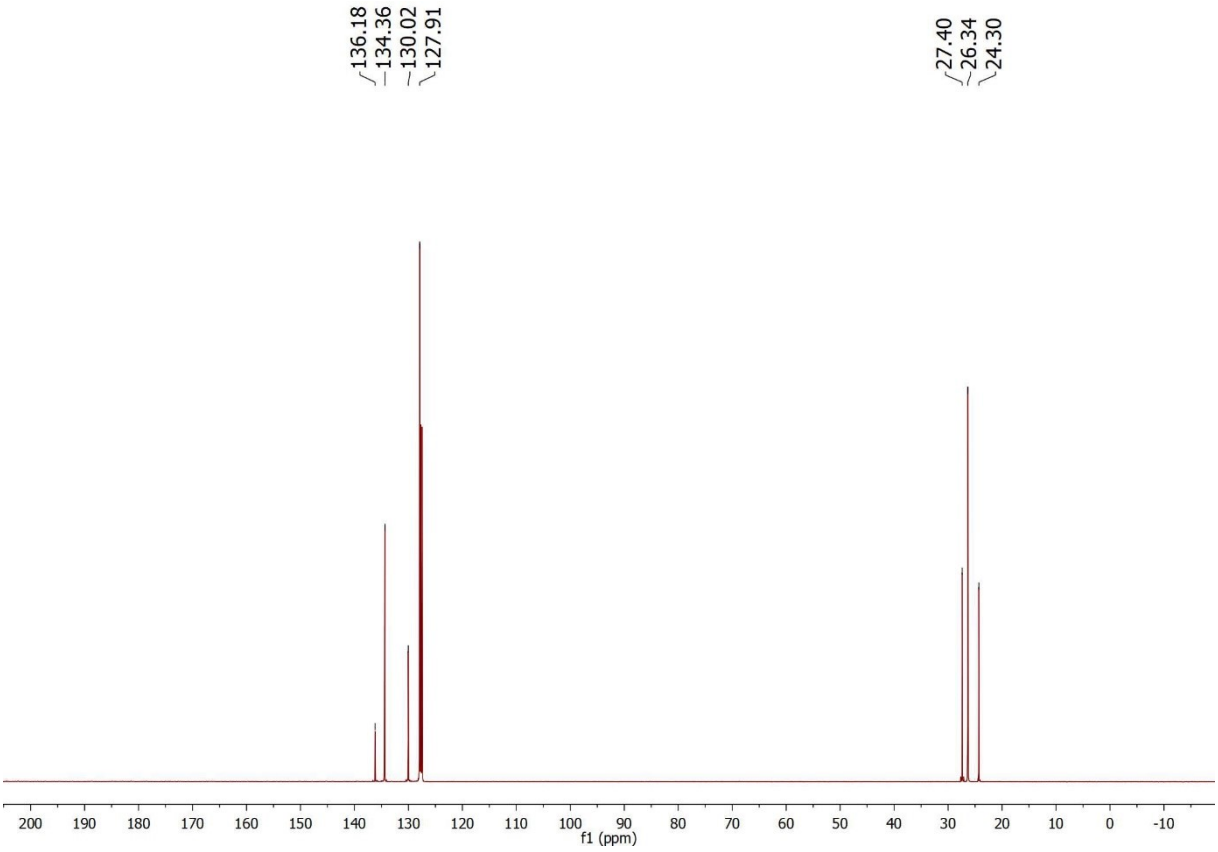

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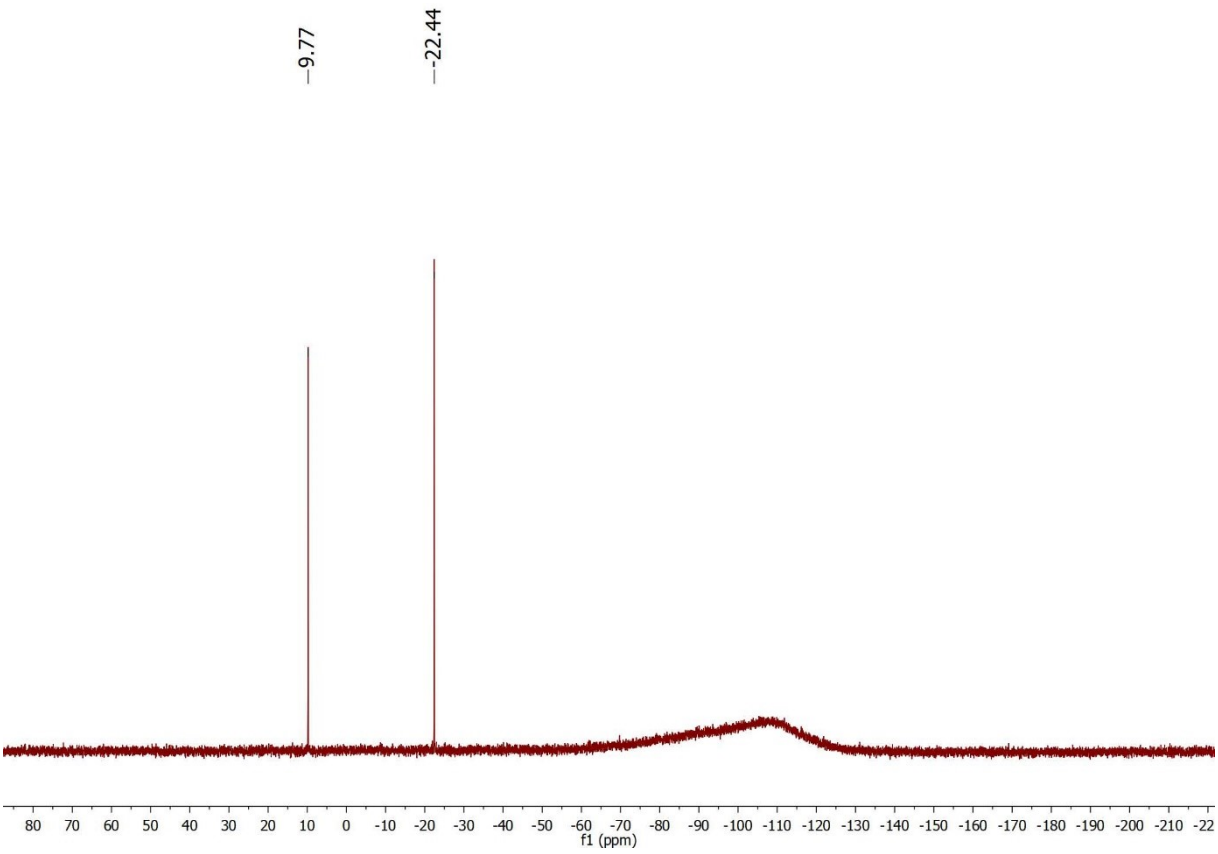
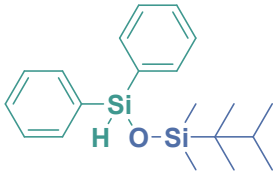


1-(2,3-Dimethylbutan-2-yl)-1,1-dimethyl-3,3-diphenyldisiloxane 5e



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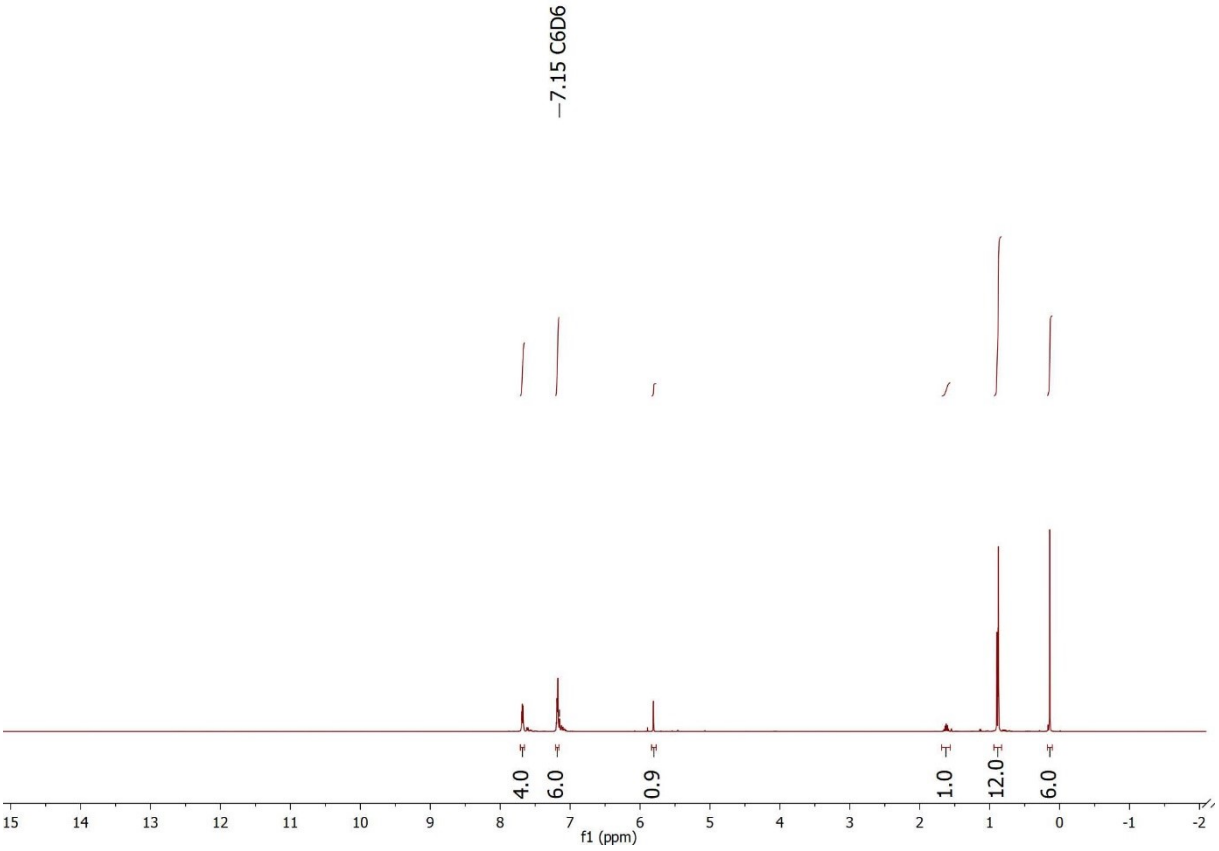
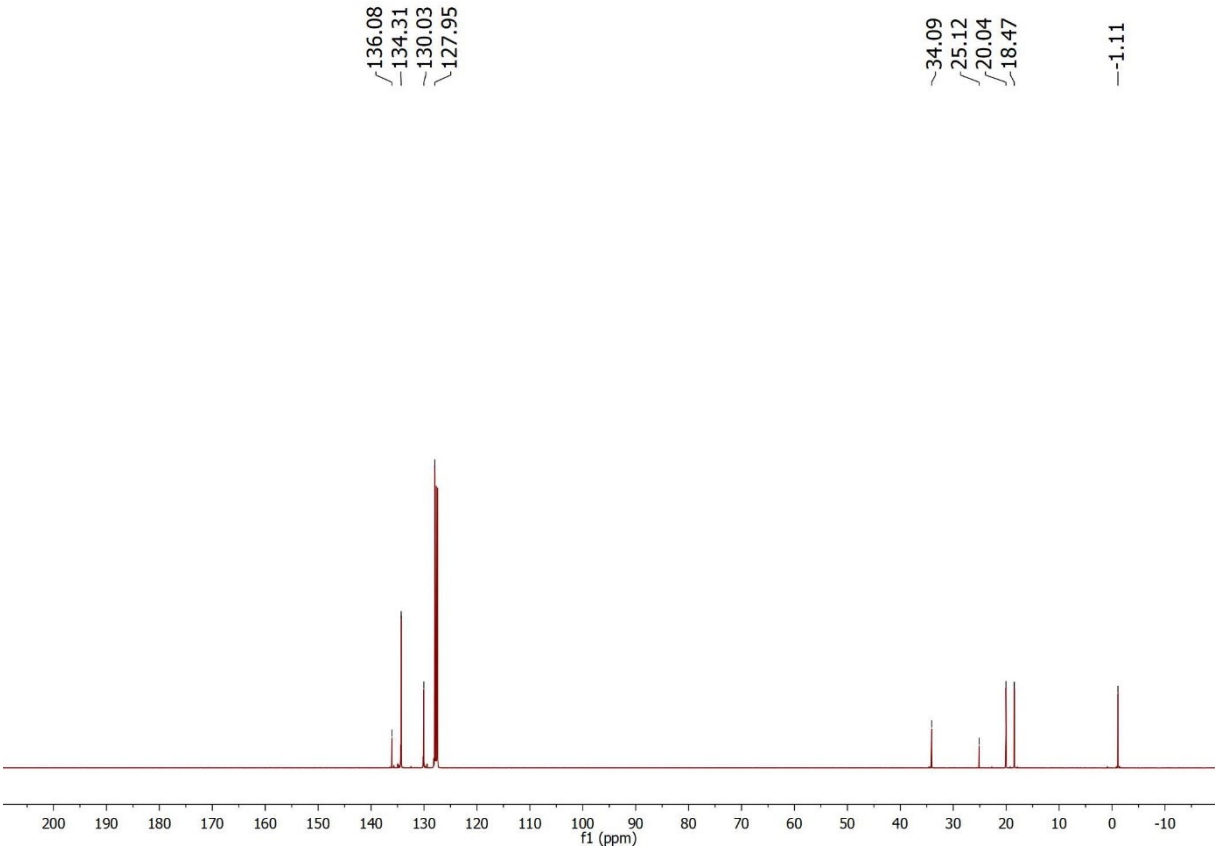


^{13}C NMR



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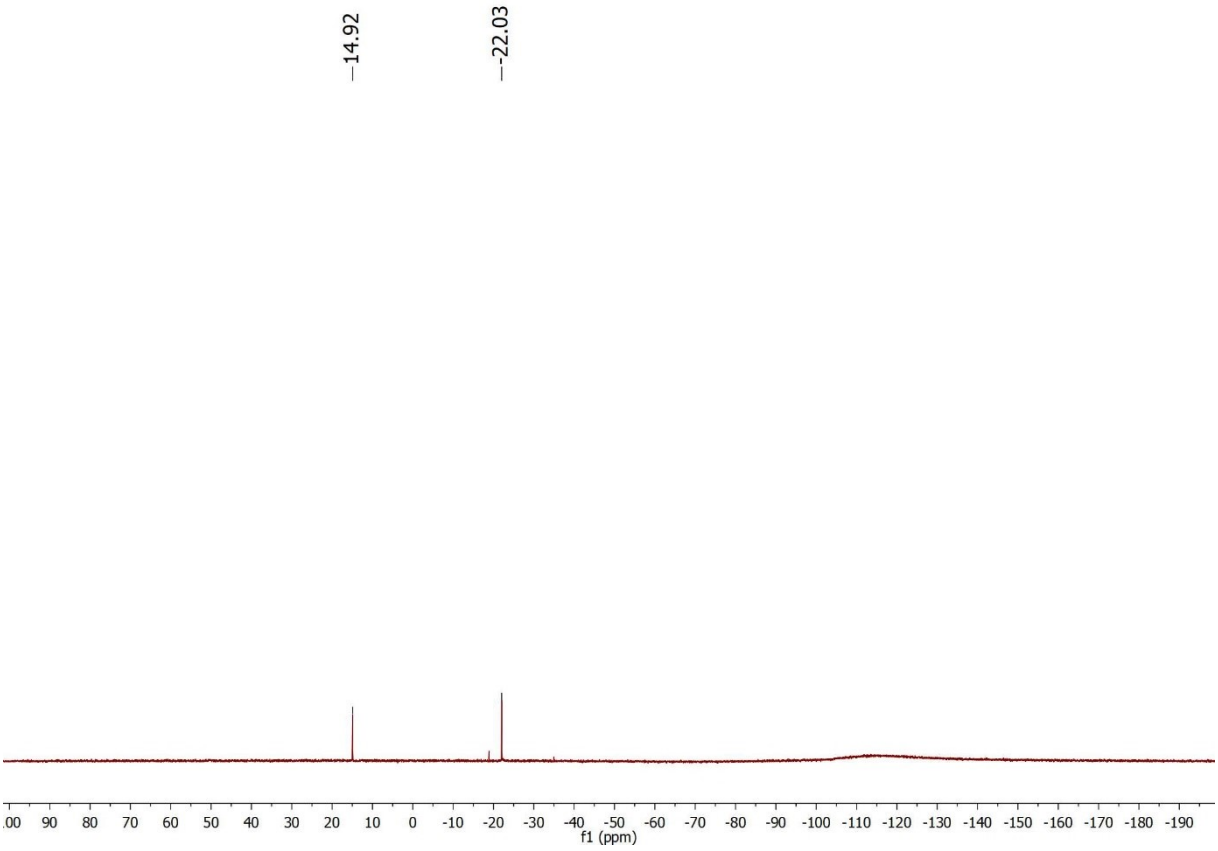
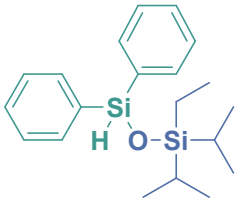
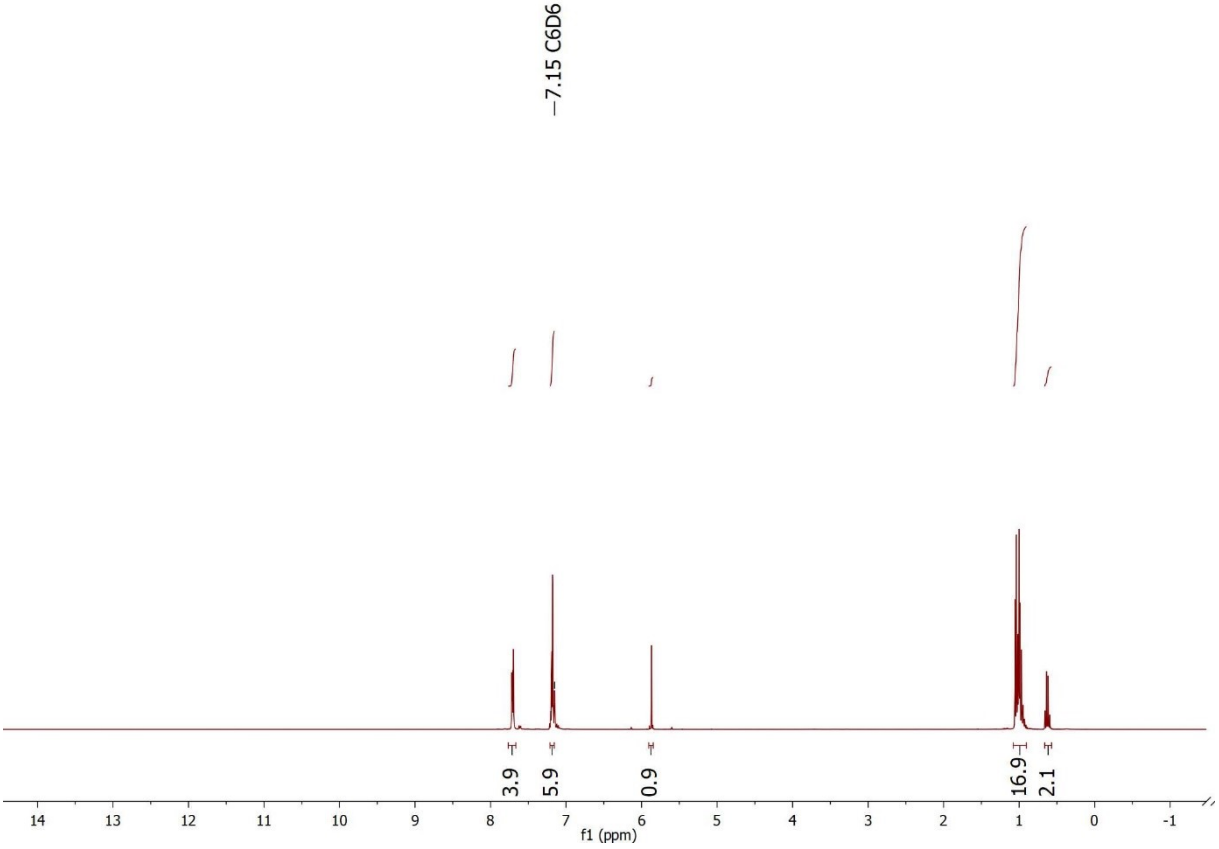


1-Ethyl-1,1-diisopropyl-3,3-diphenyldisiloxane 5f



^{1}H NMR



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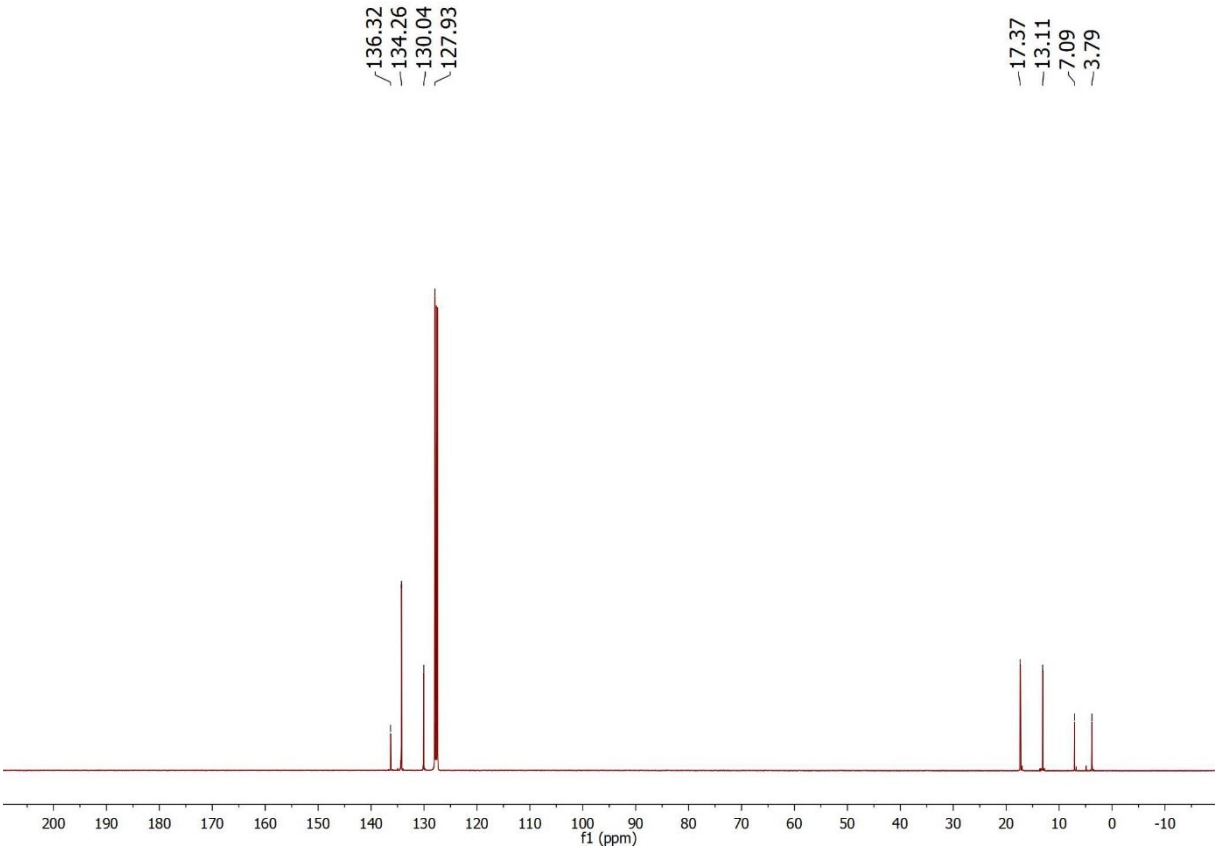
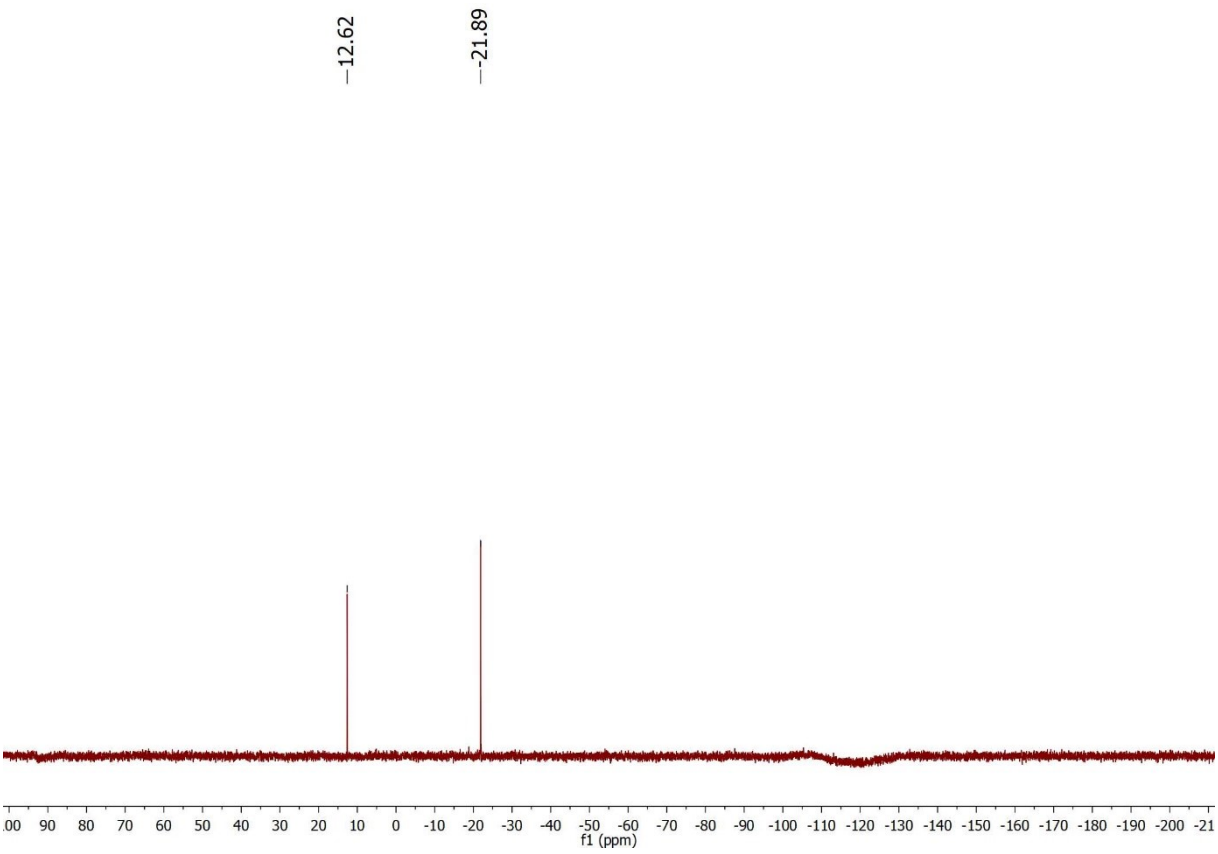
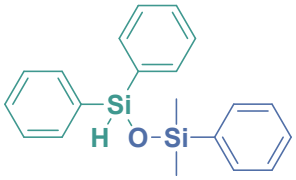


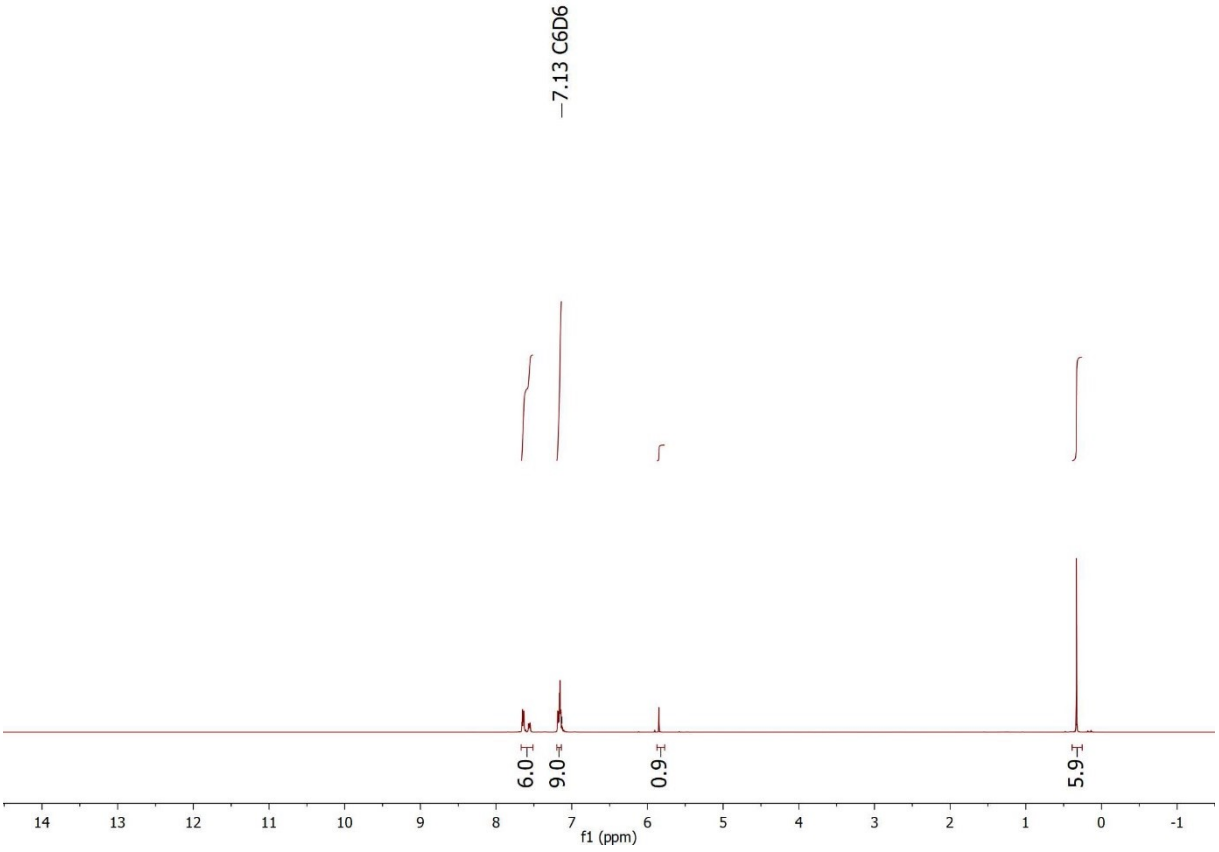
^{29}Si NMR



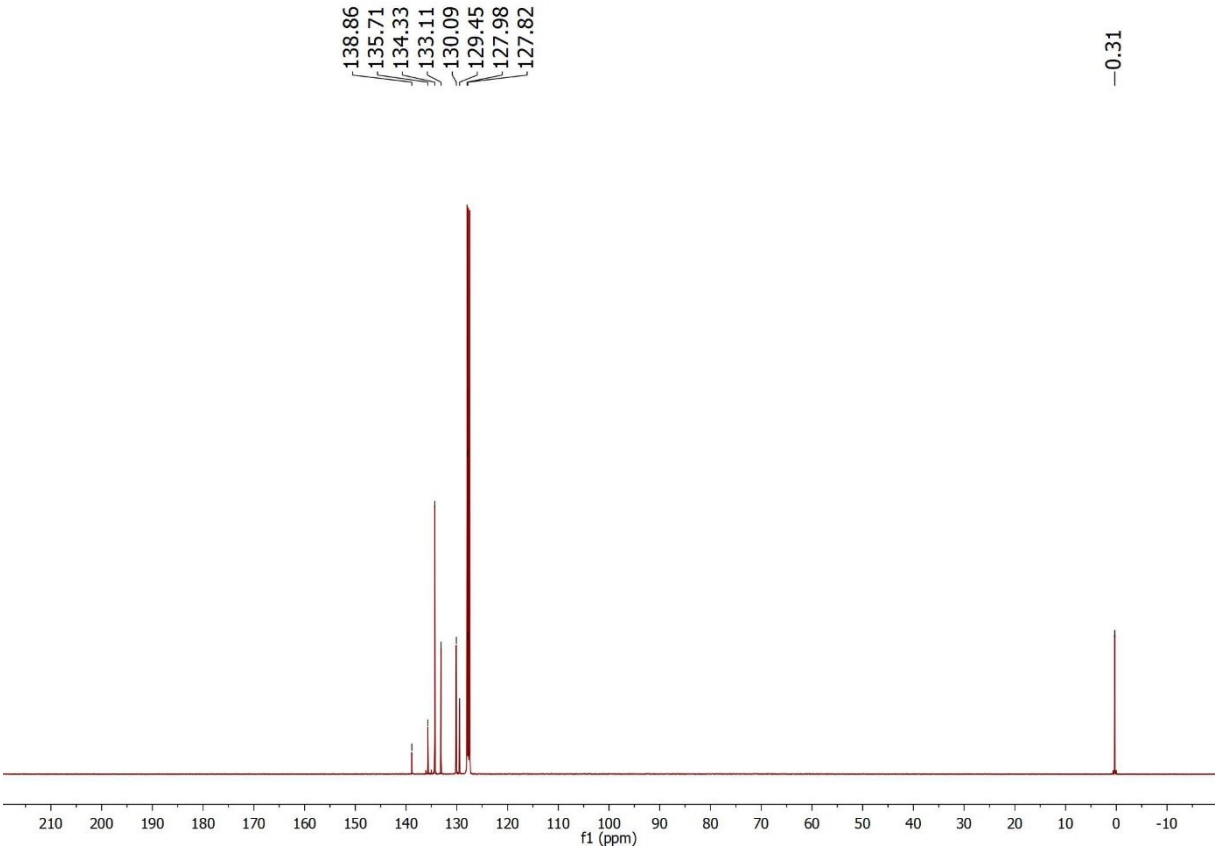
1,1-Dimethyl-1,3,3-triphenyldisiloxane 5g



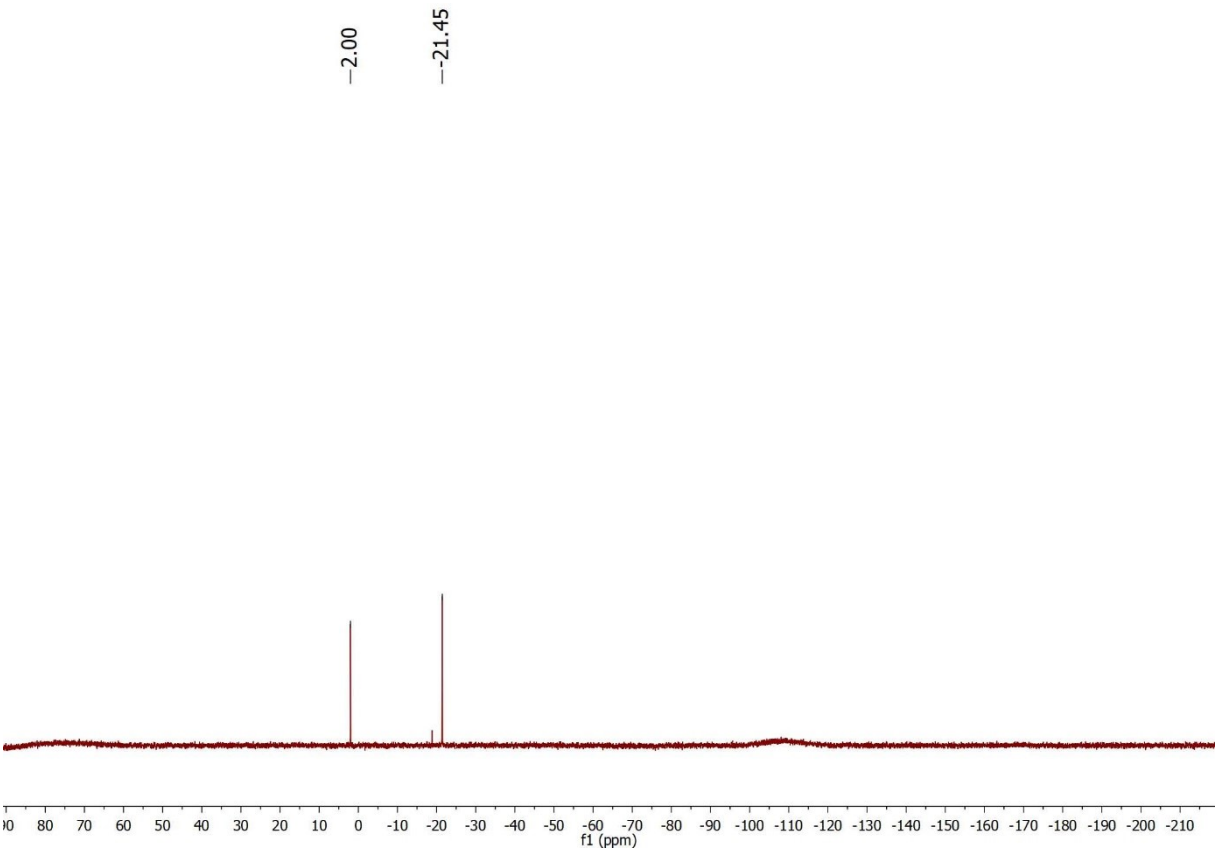
^{1}H NMR



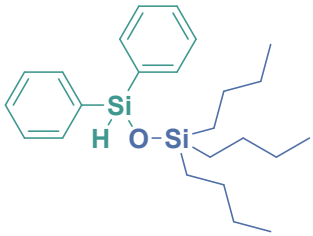
^{13}C NMR



^{29}Si NMR



1,1,1-Tributyl-3,3-diphenyldisiloxane 5h



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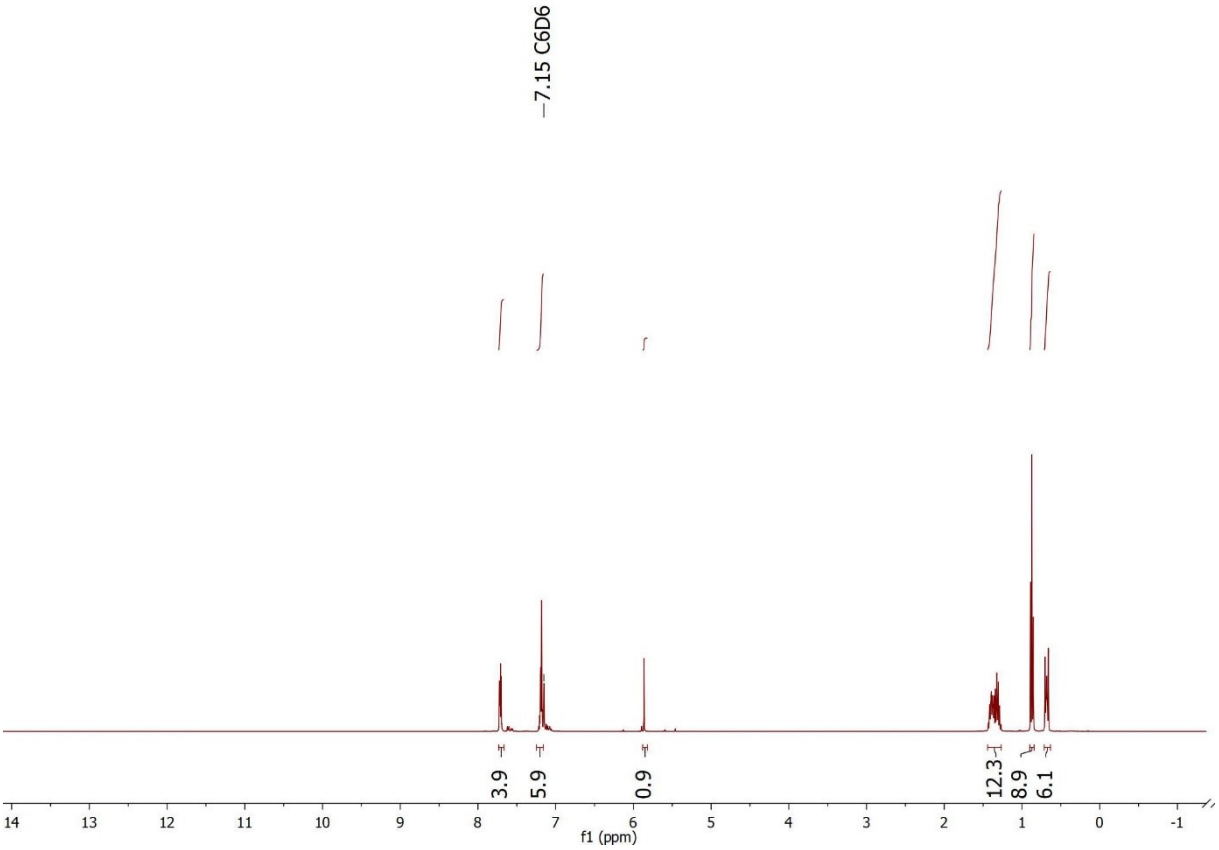

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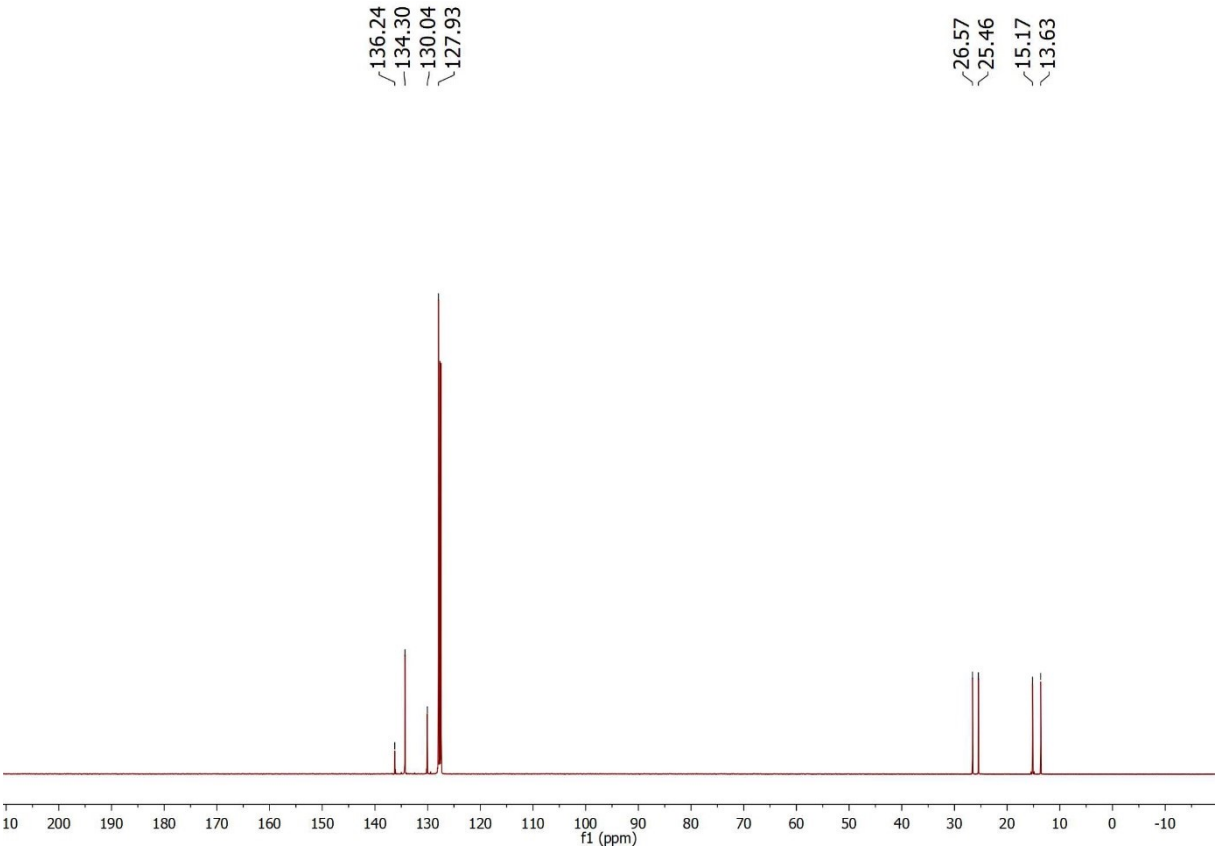

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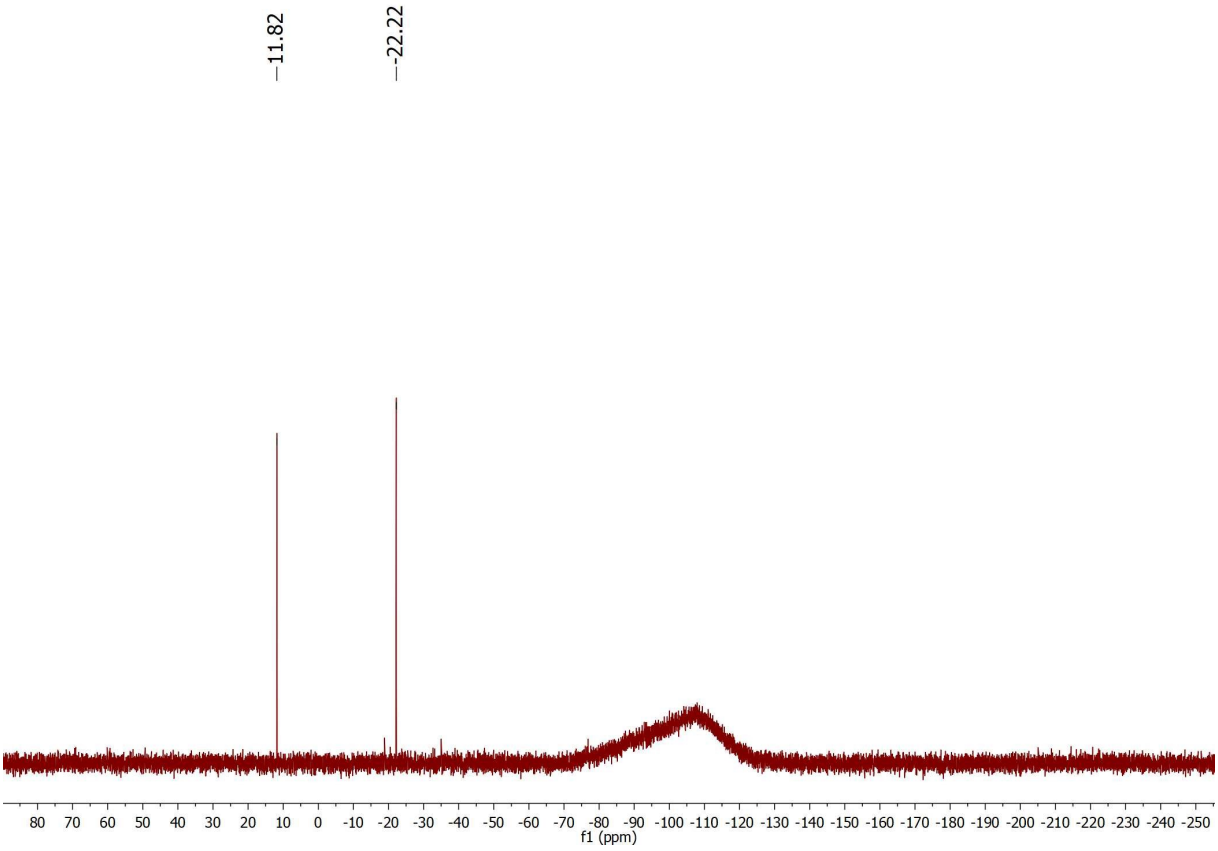
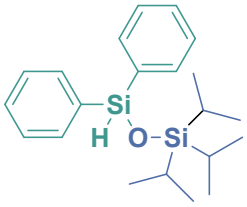


1,1,1-Triisopropyl-3,3-diphenyldisiloxane 5i



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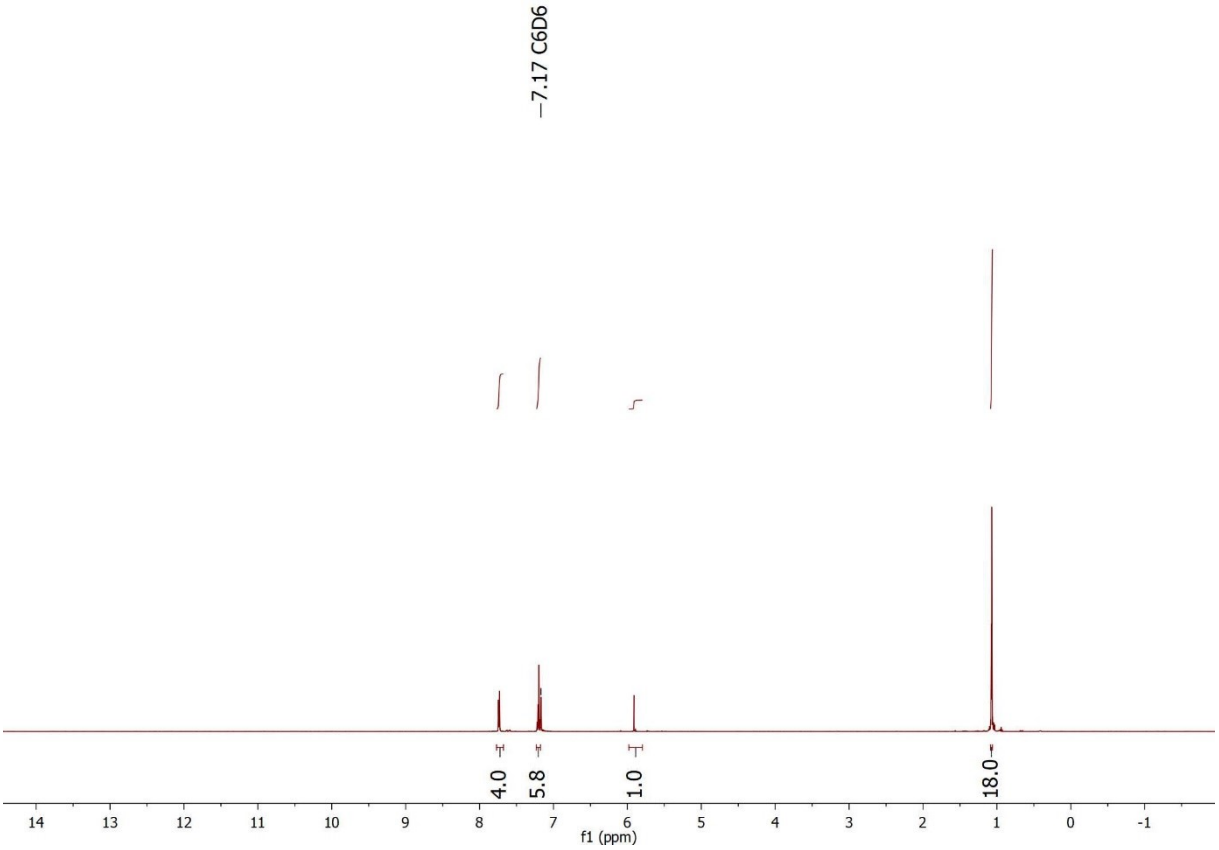

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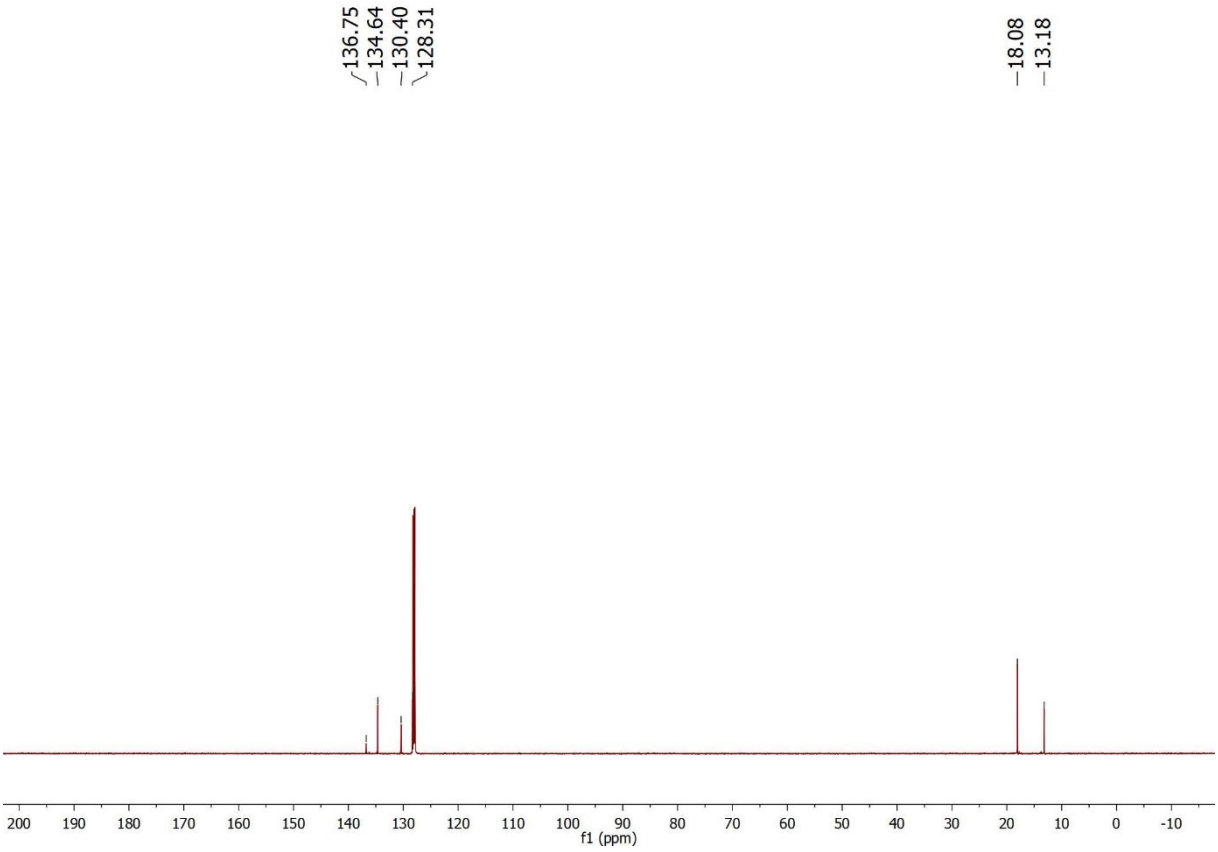

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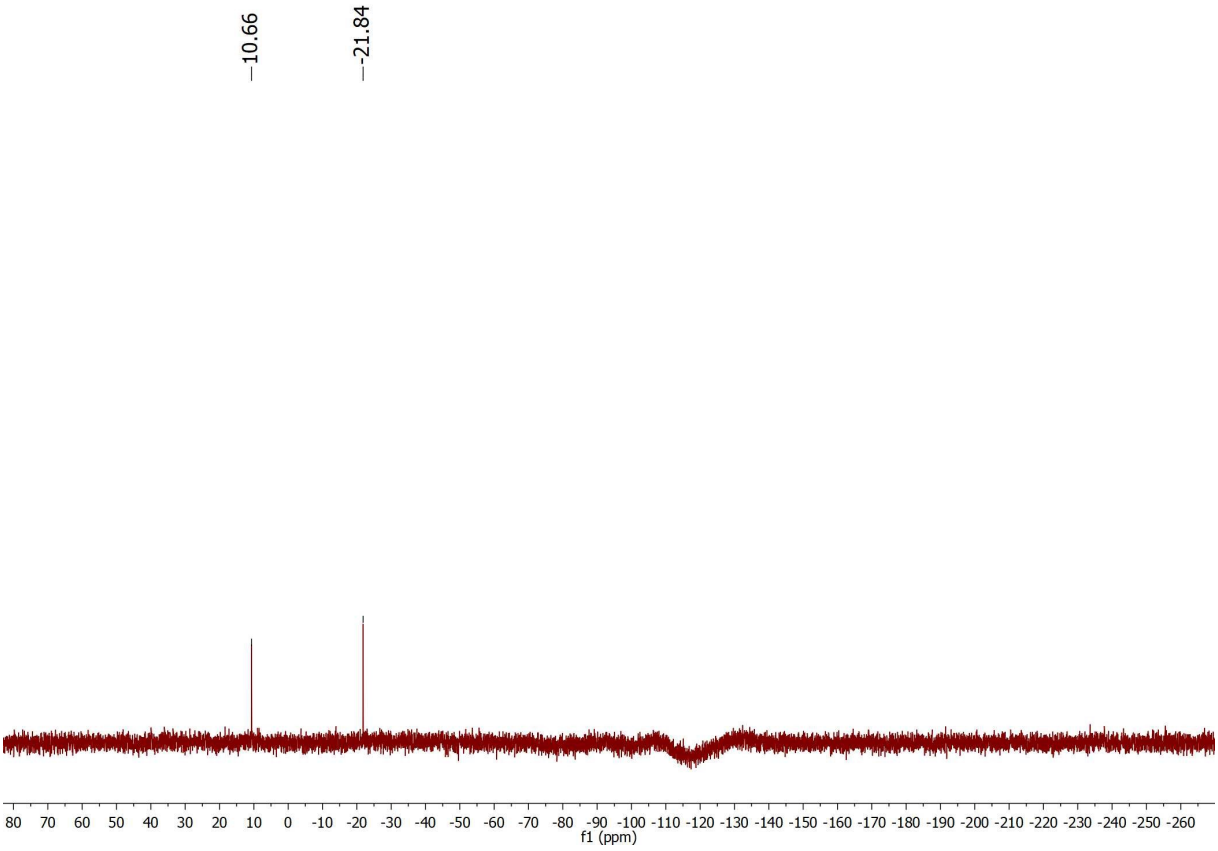
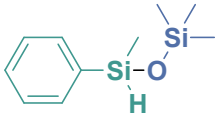


1,1,1,3-Tetramethyl-3-phenyldisiloxane 5j



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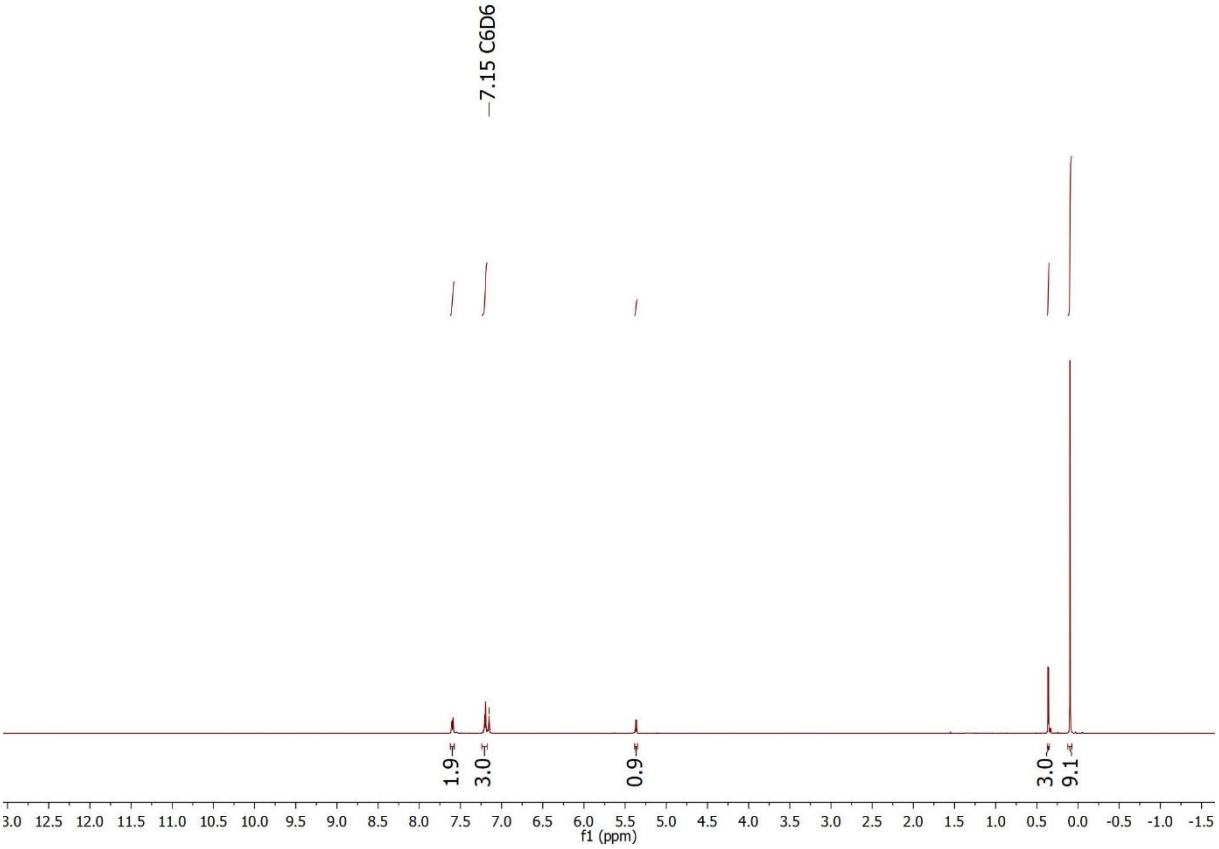

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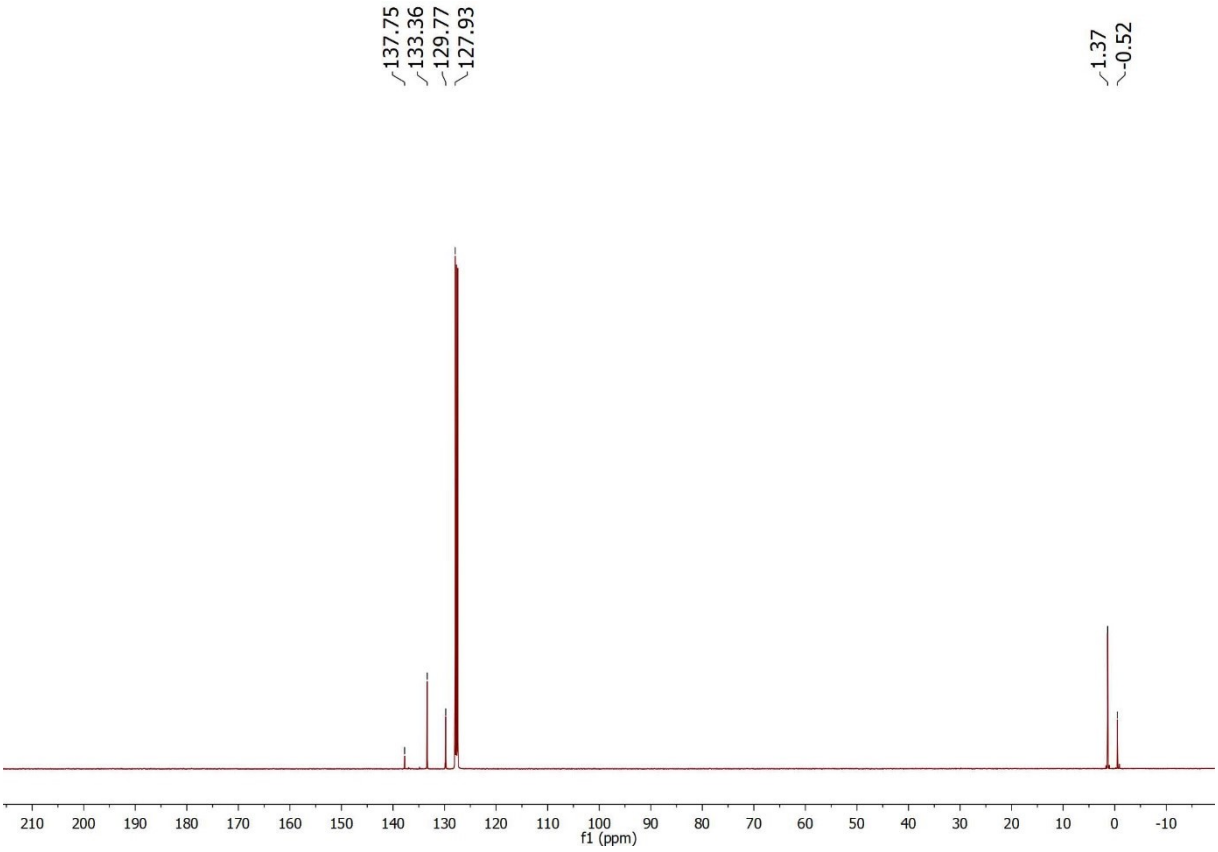

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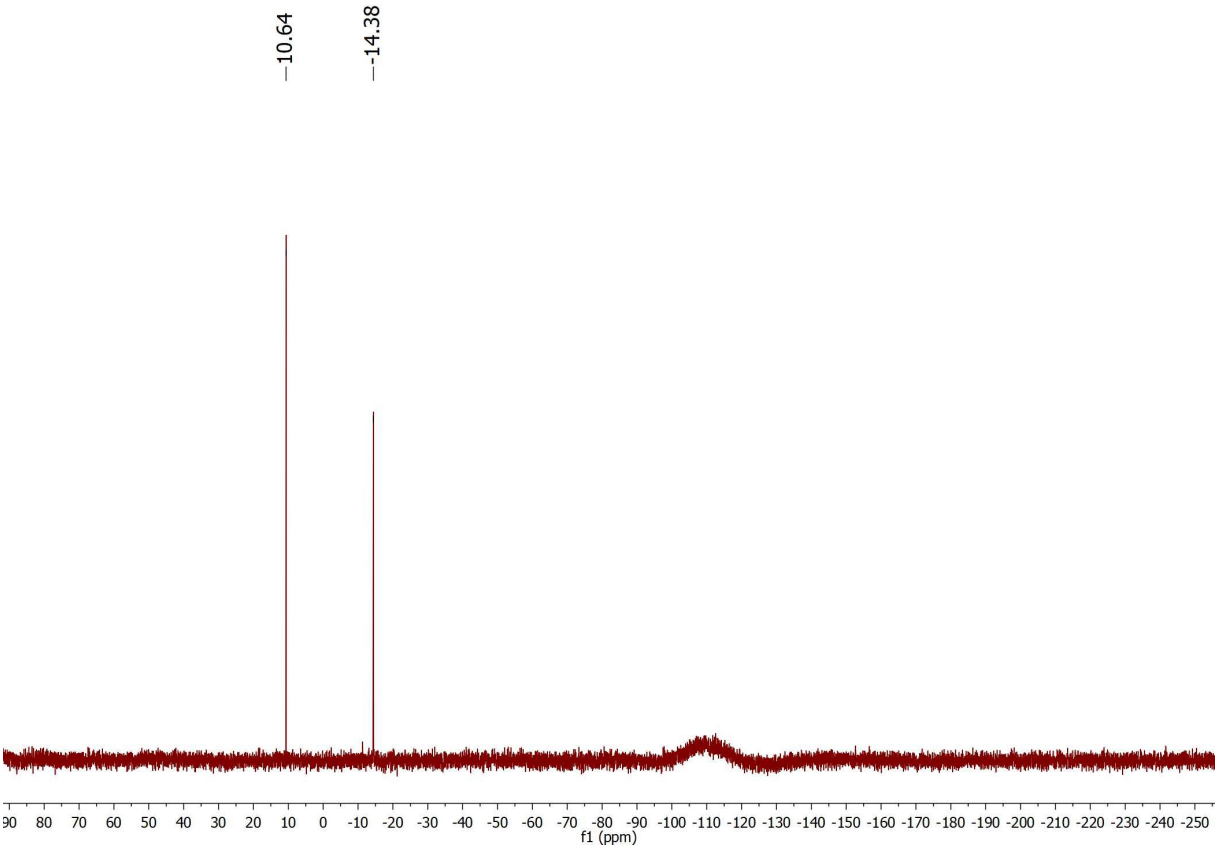
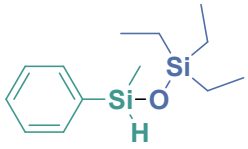


1,1,1-Triethyl-3-methyl-3-phenyldisiloxane 5k



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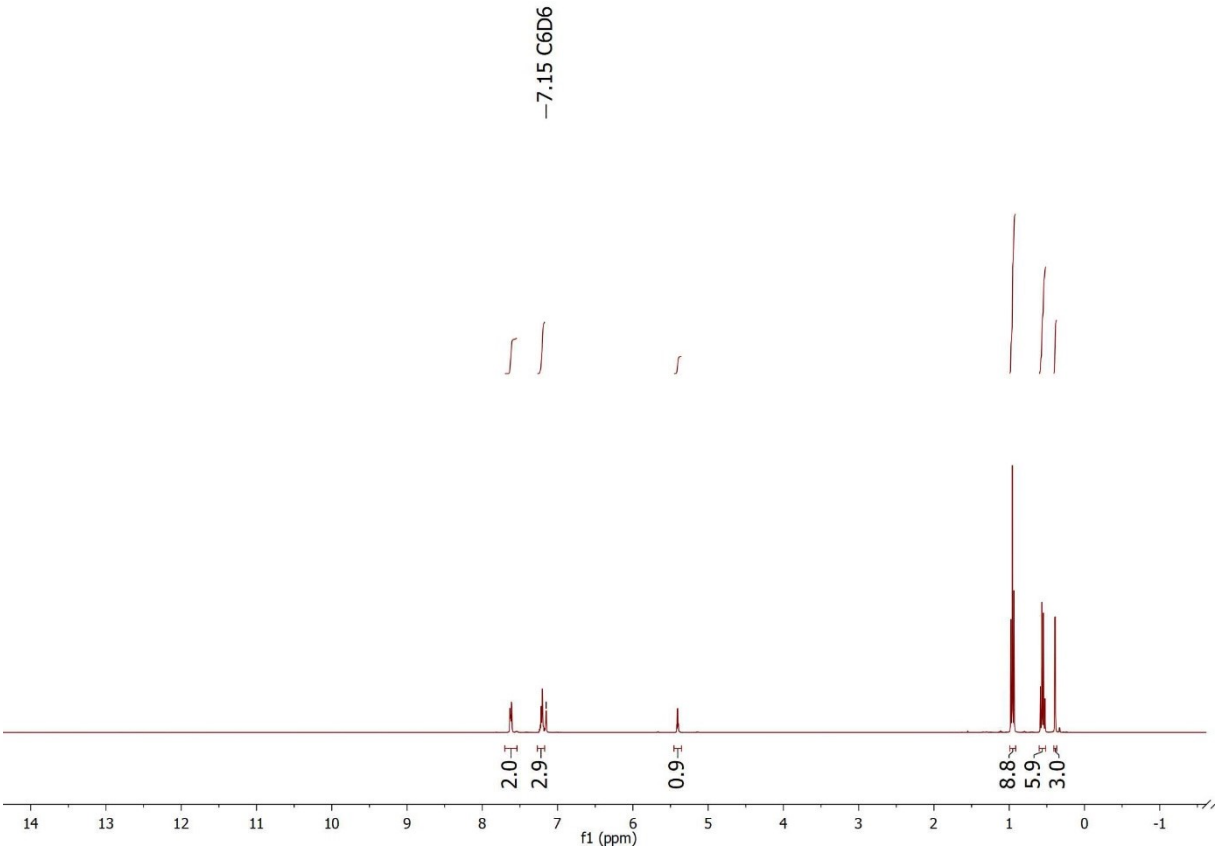

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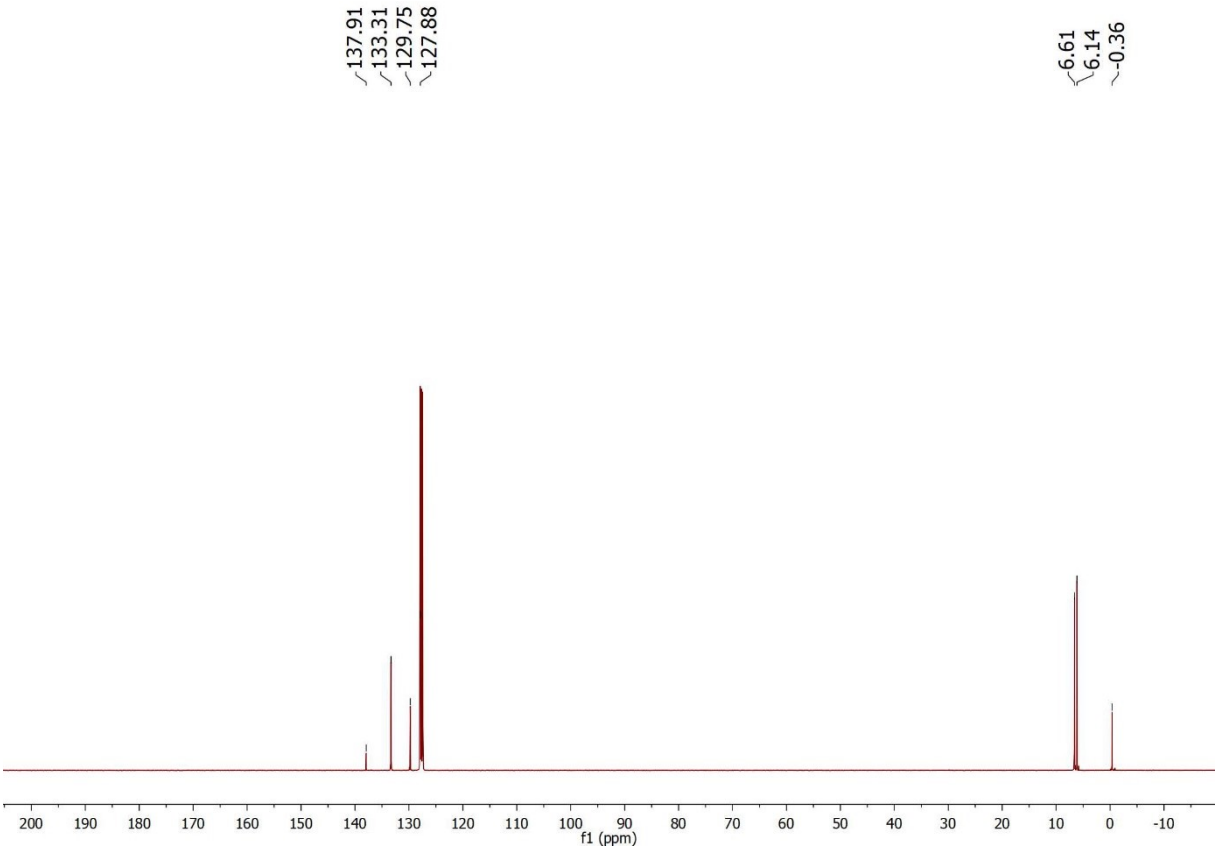

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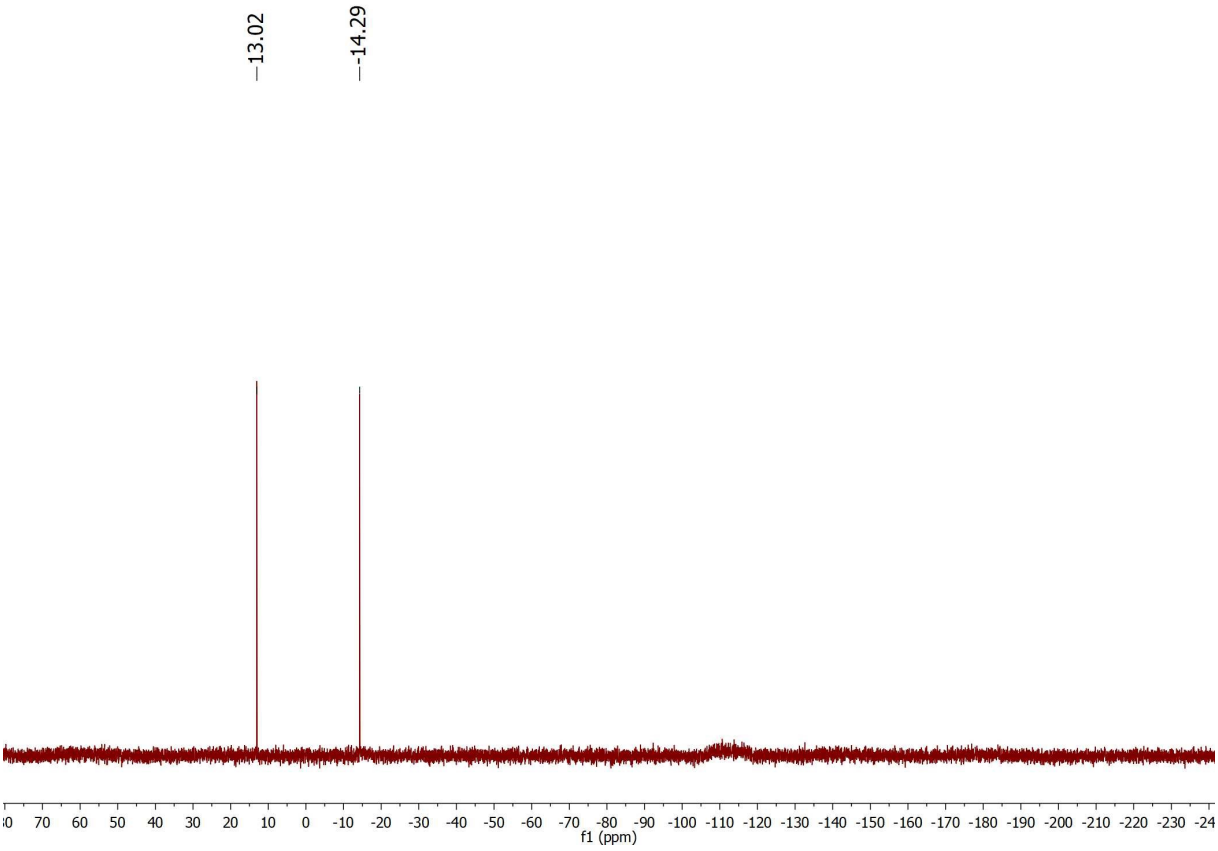
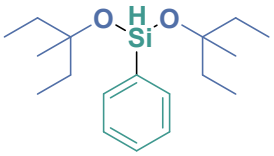


Bis((3-methylpentan-3-yl)oxy)(phenyl)silane 6a



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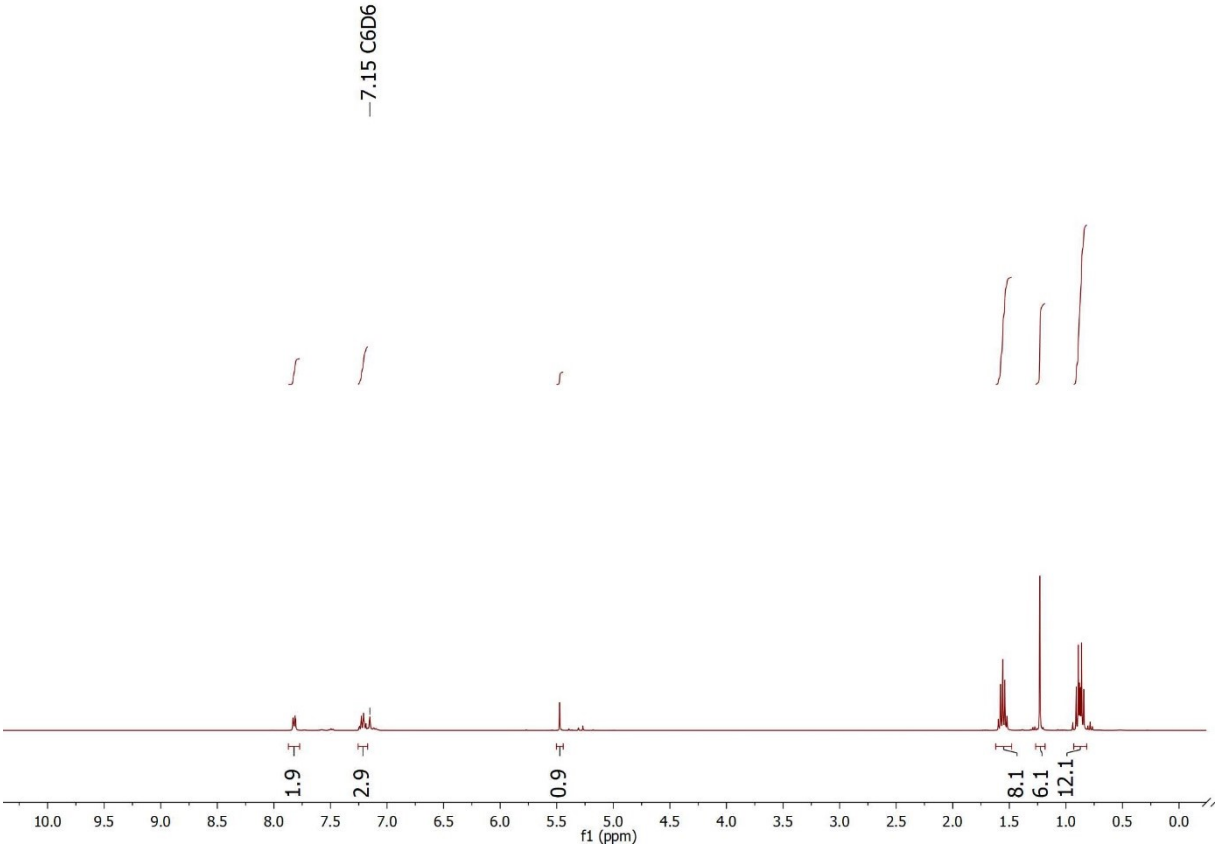

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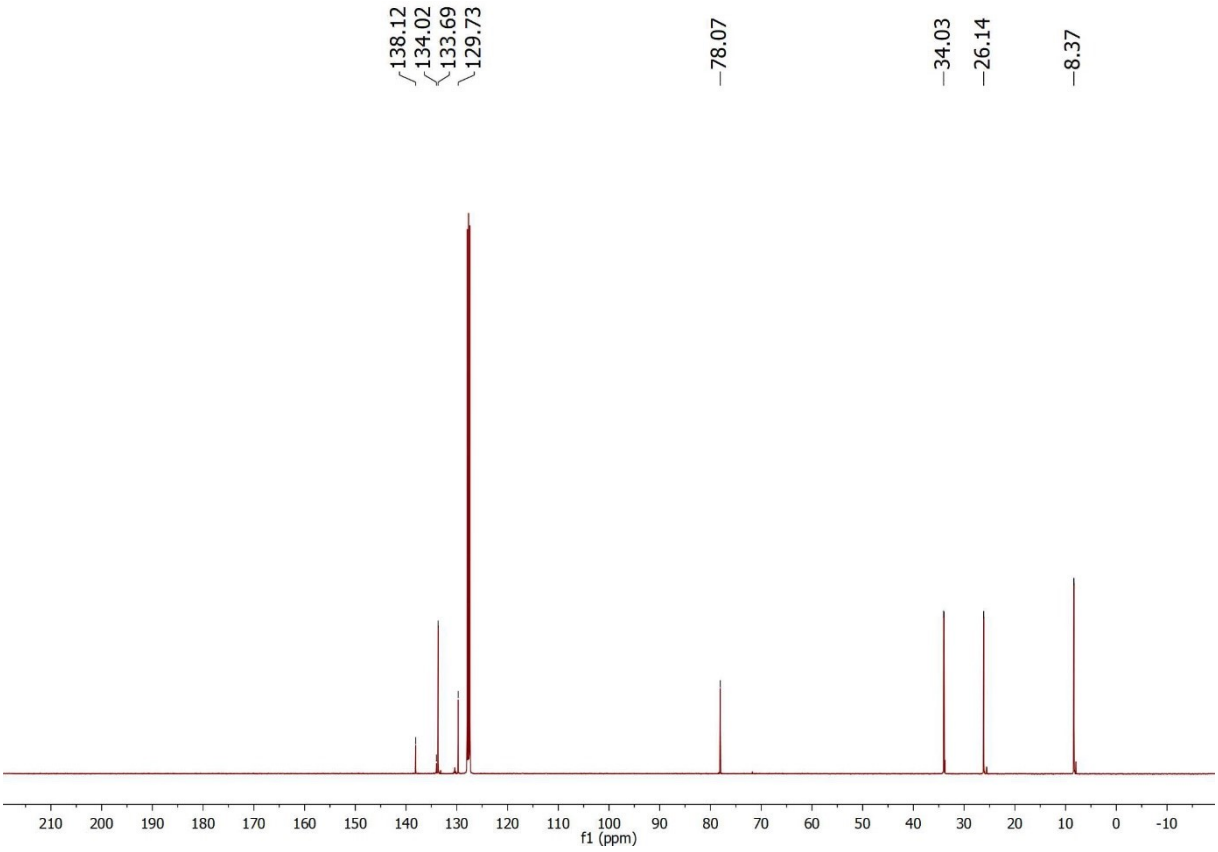

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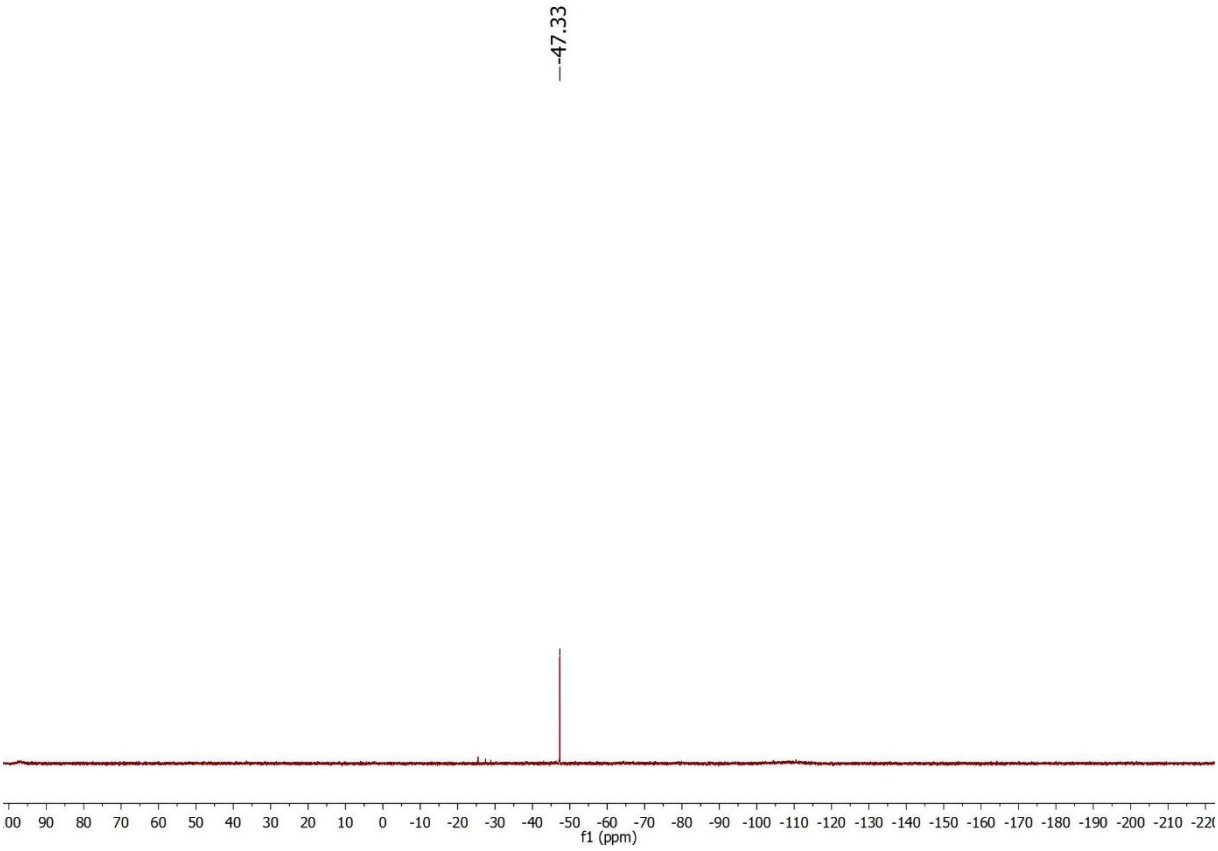
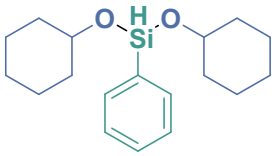


Bis(cyclohexyloxy)(phenyl)silane 6b



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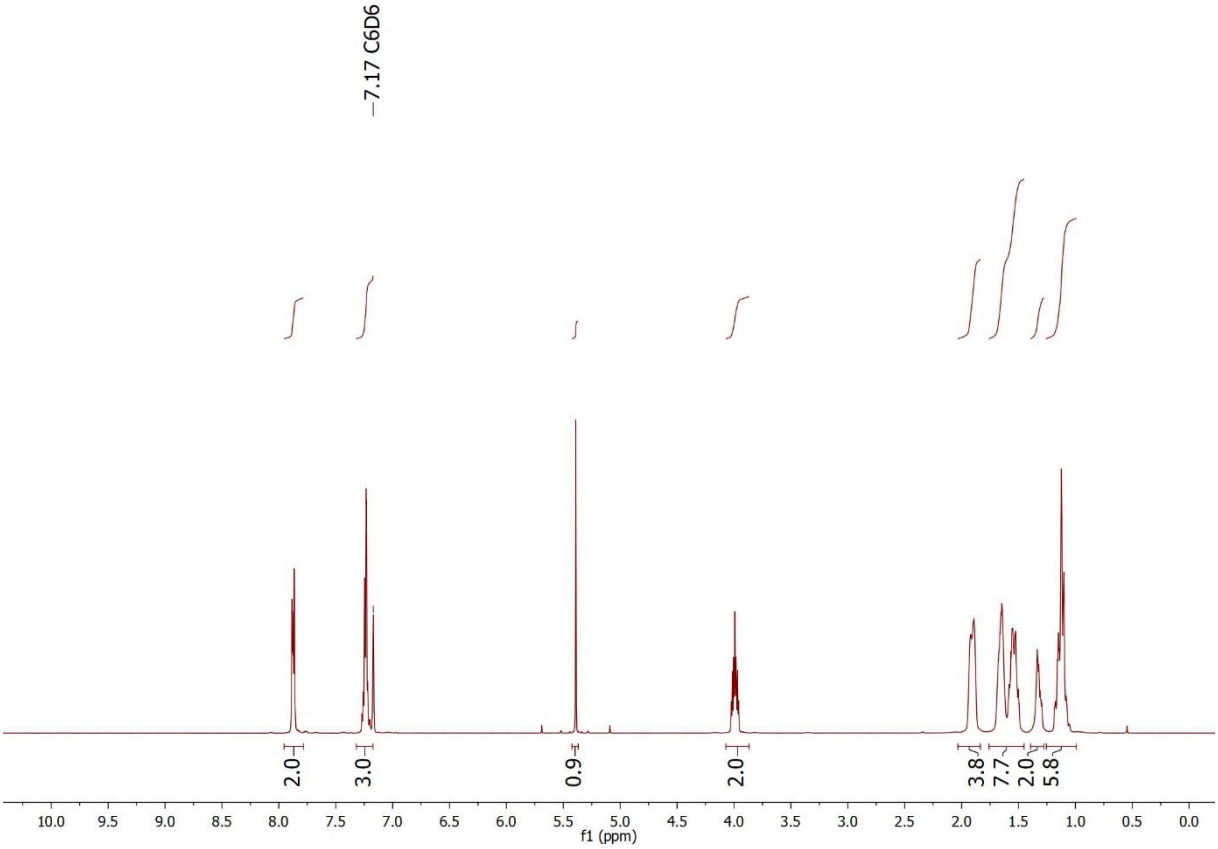

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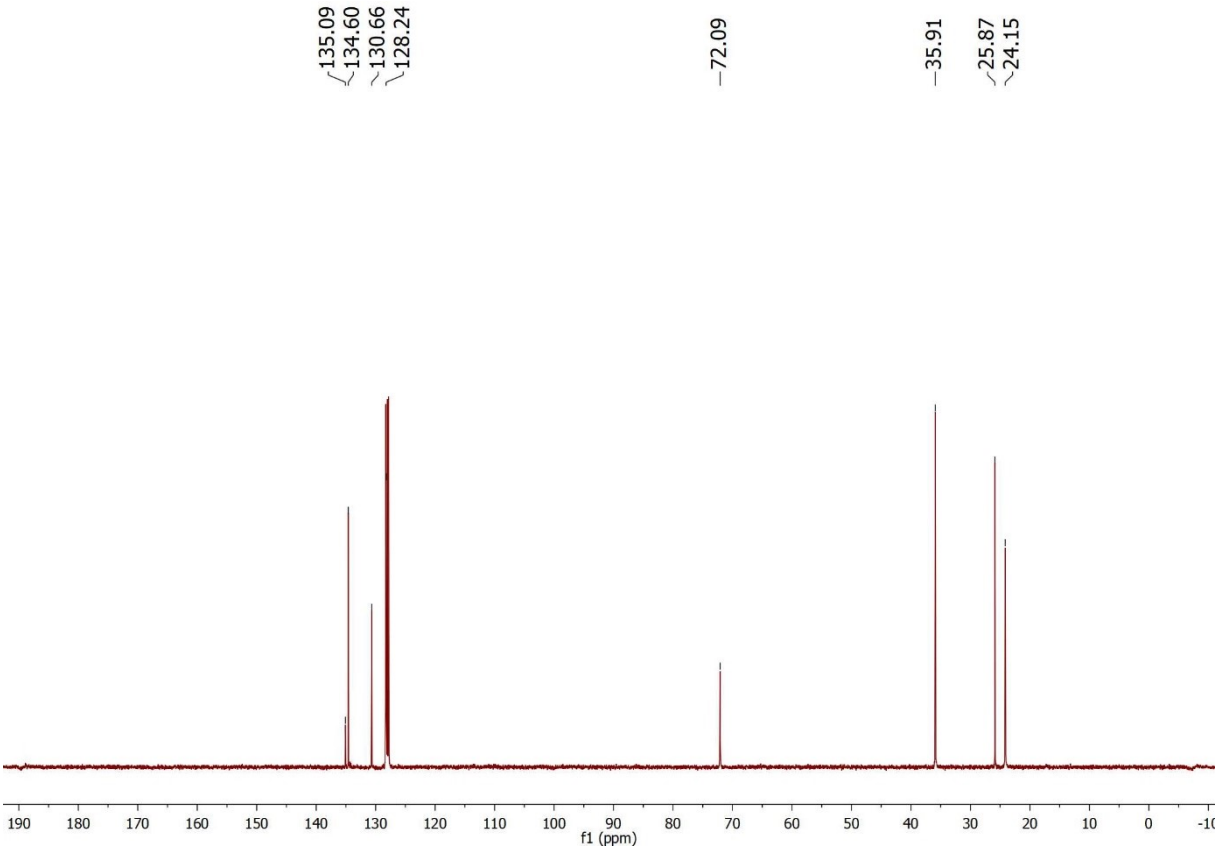

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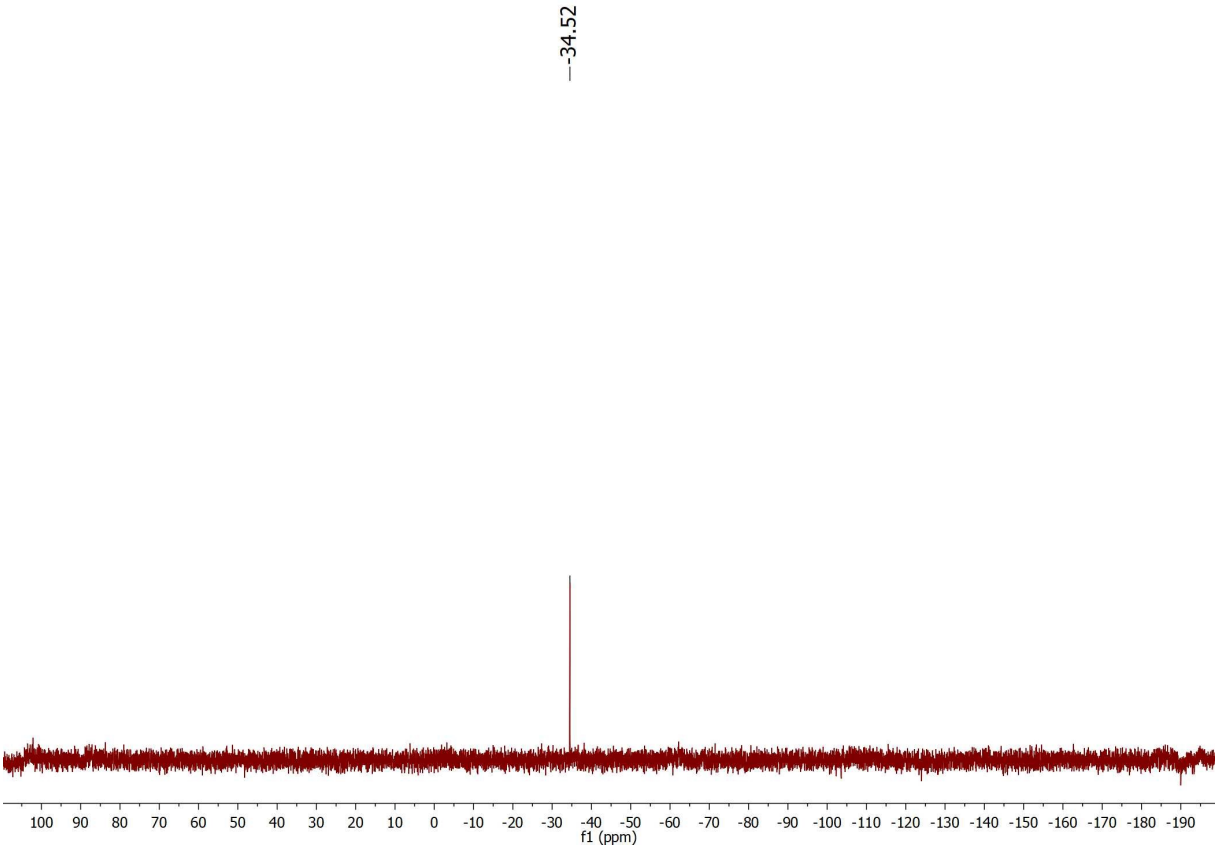
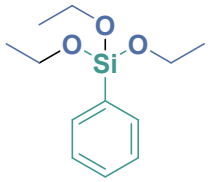


Triethoxy(phenyl)silane 6c



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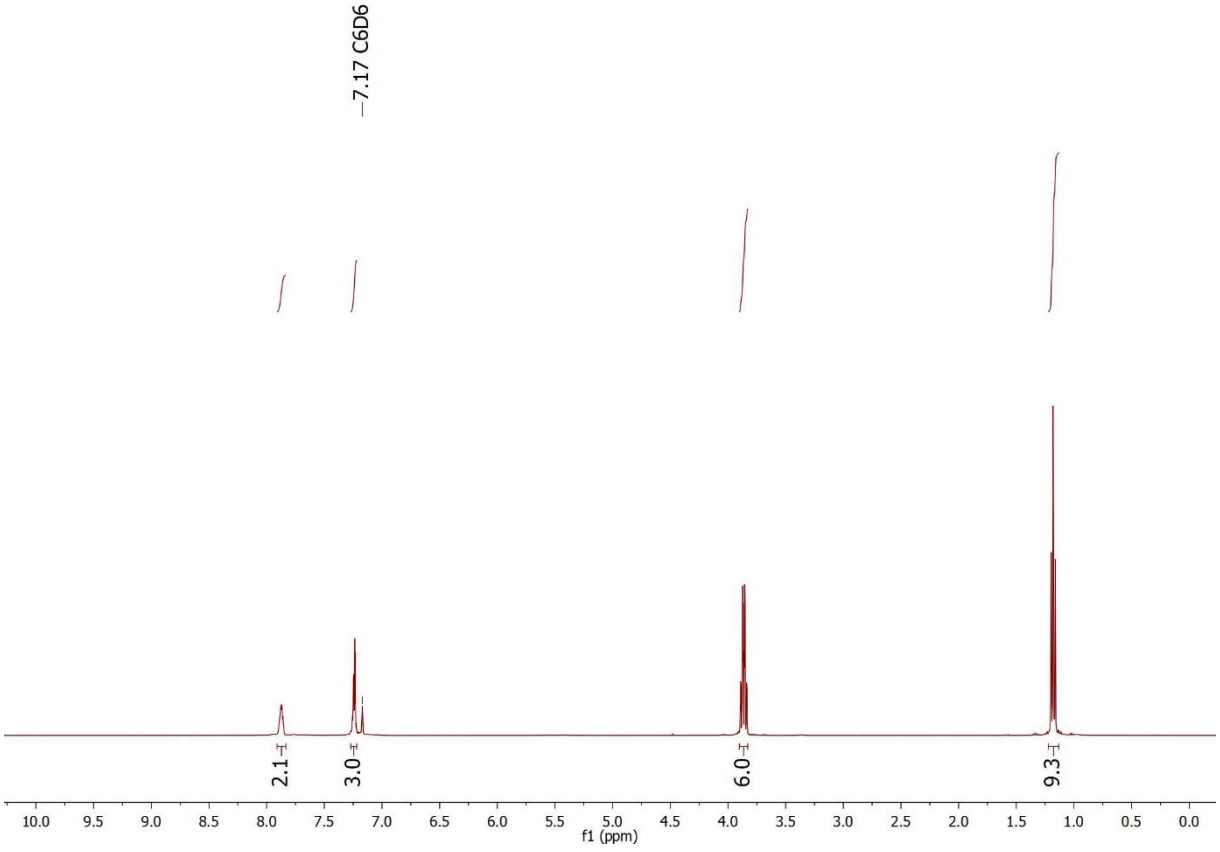

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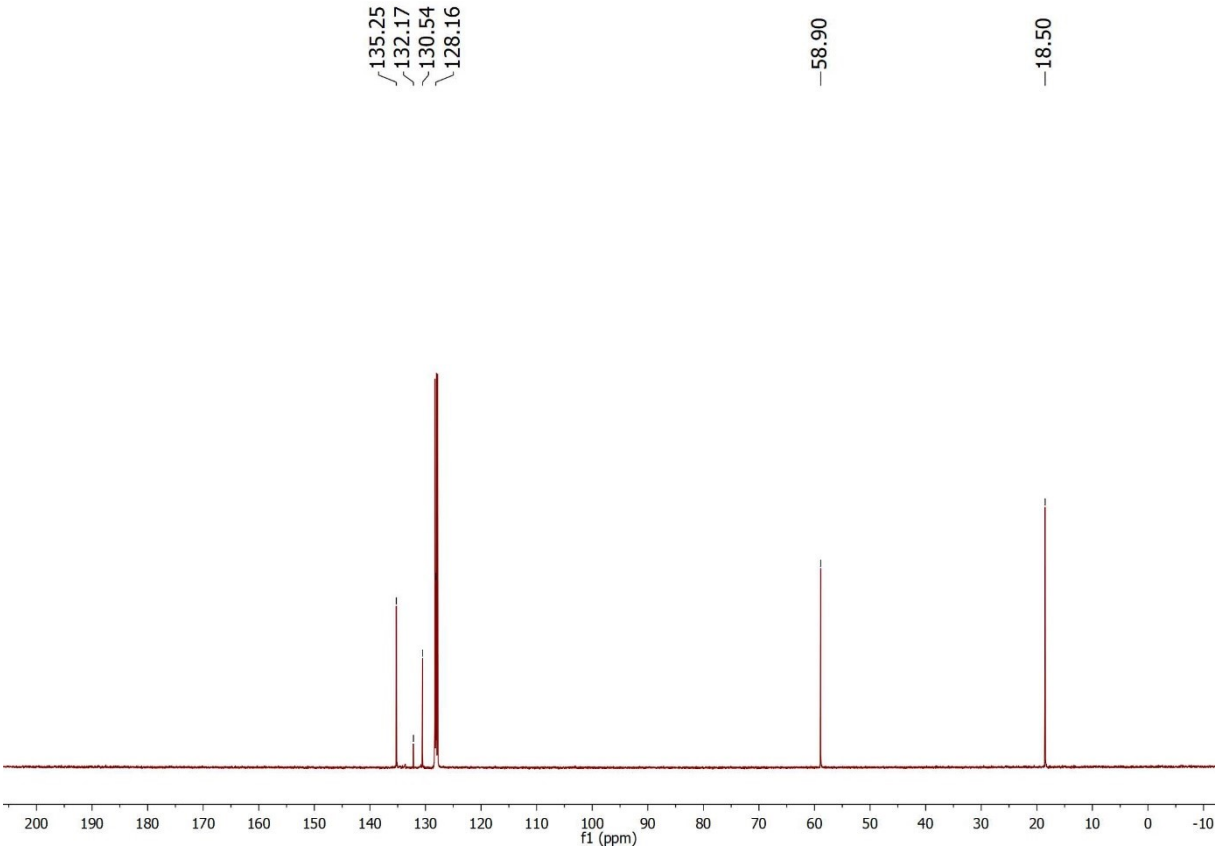

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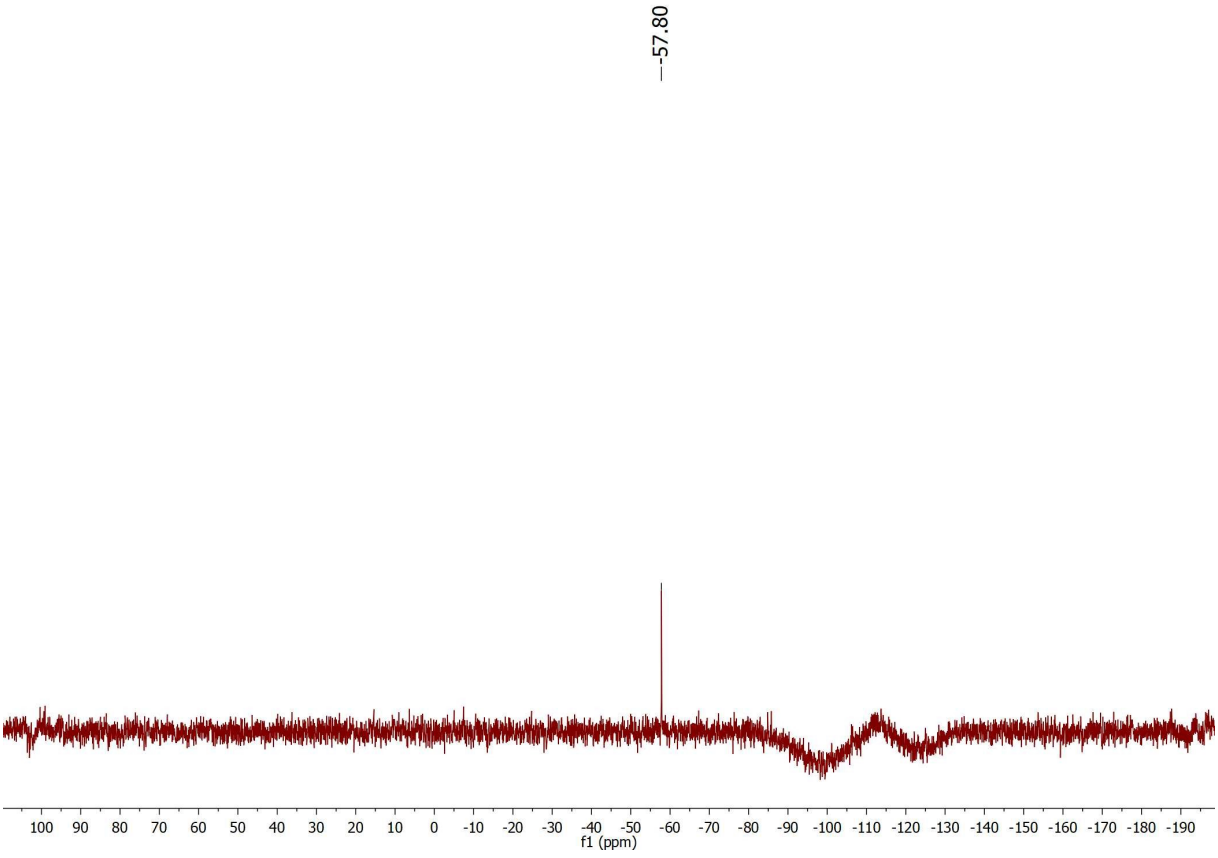
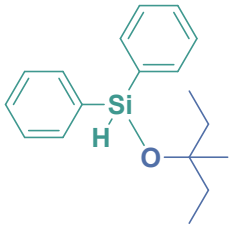
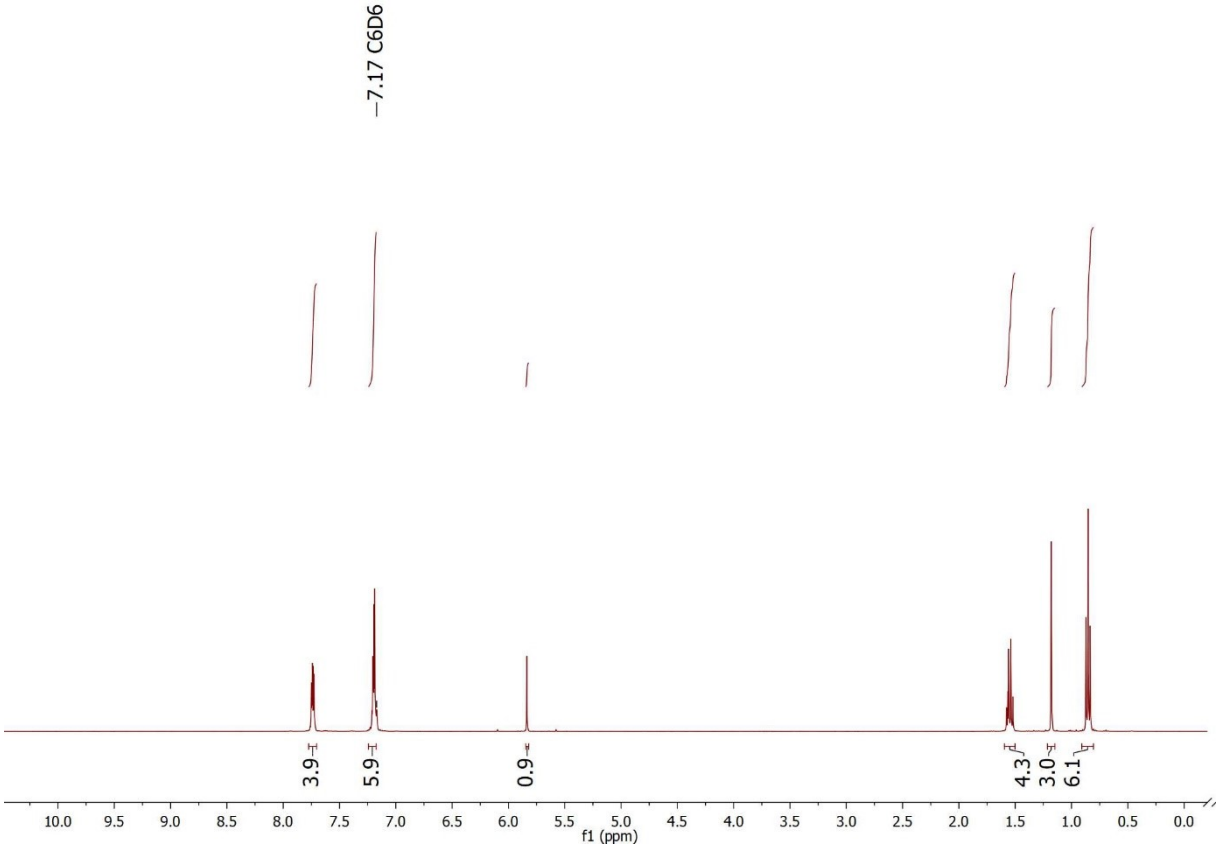


((3-Methylpentan-3-yl)oxy)diphenylsilane 6d



^{1}H NMR



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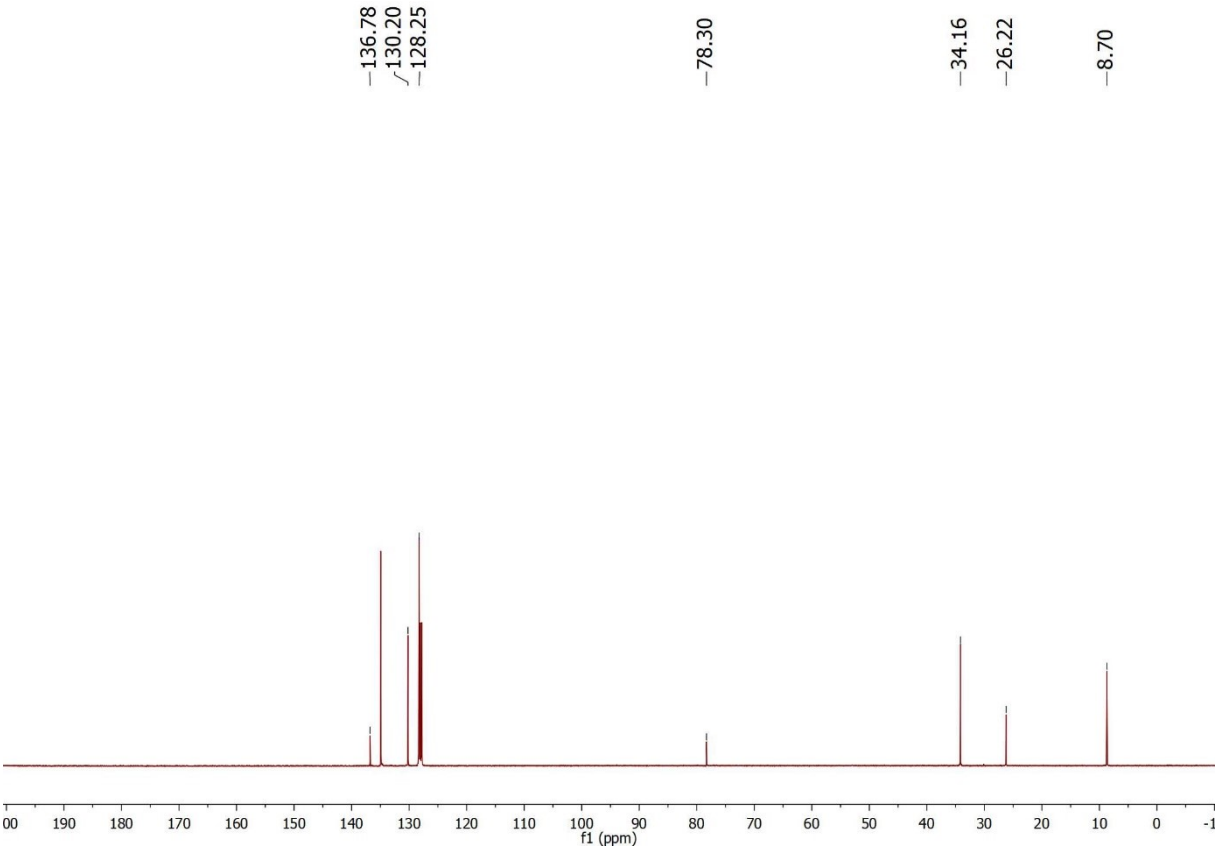
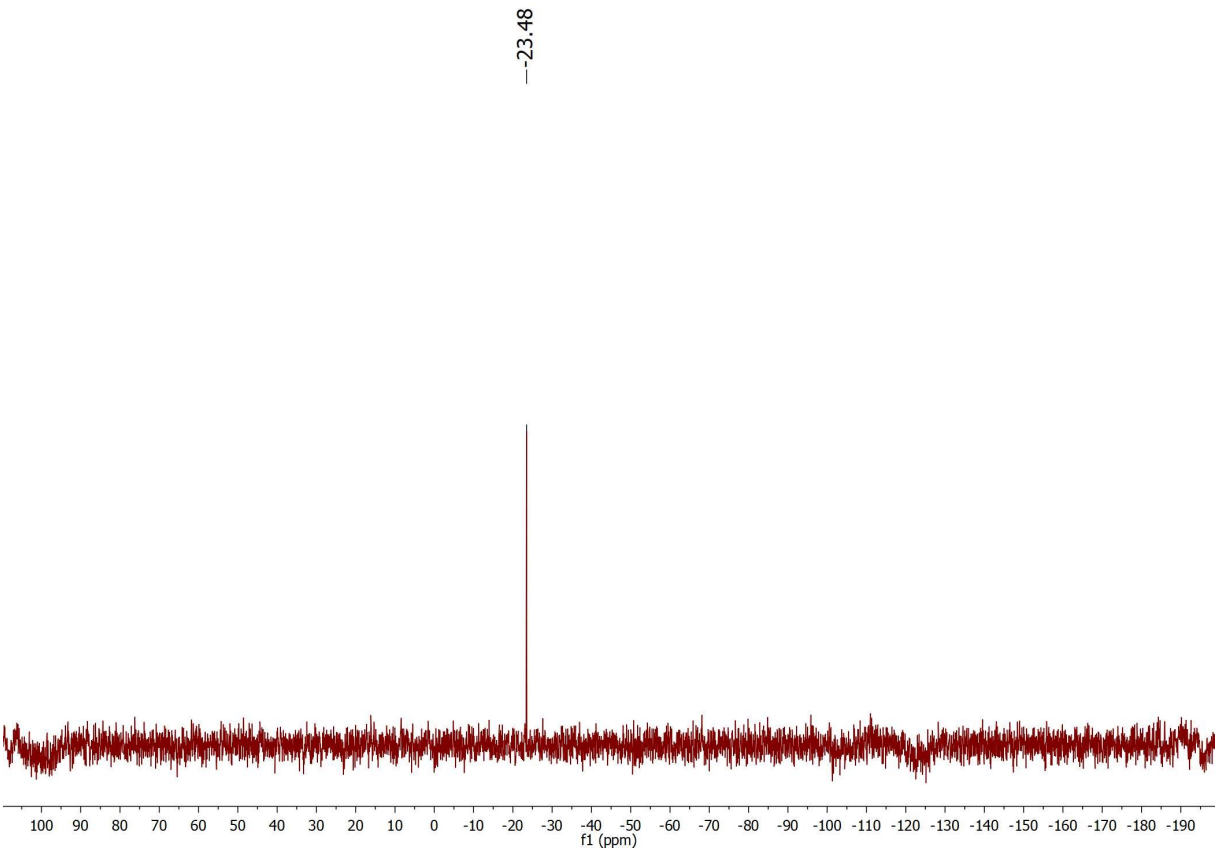
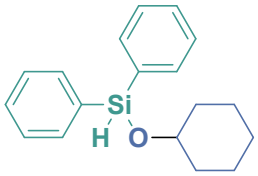


^{29}Si NMR



(Cyclohexyloxy)diphenylsilane 6e



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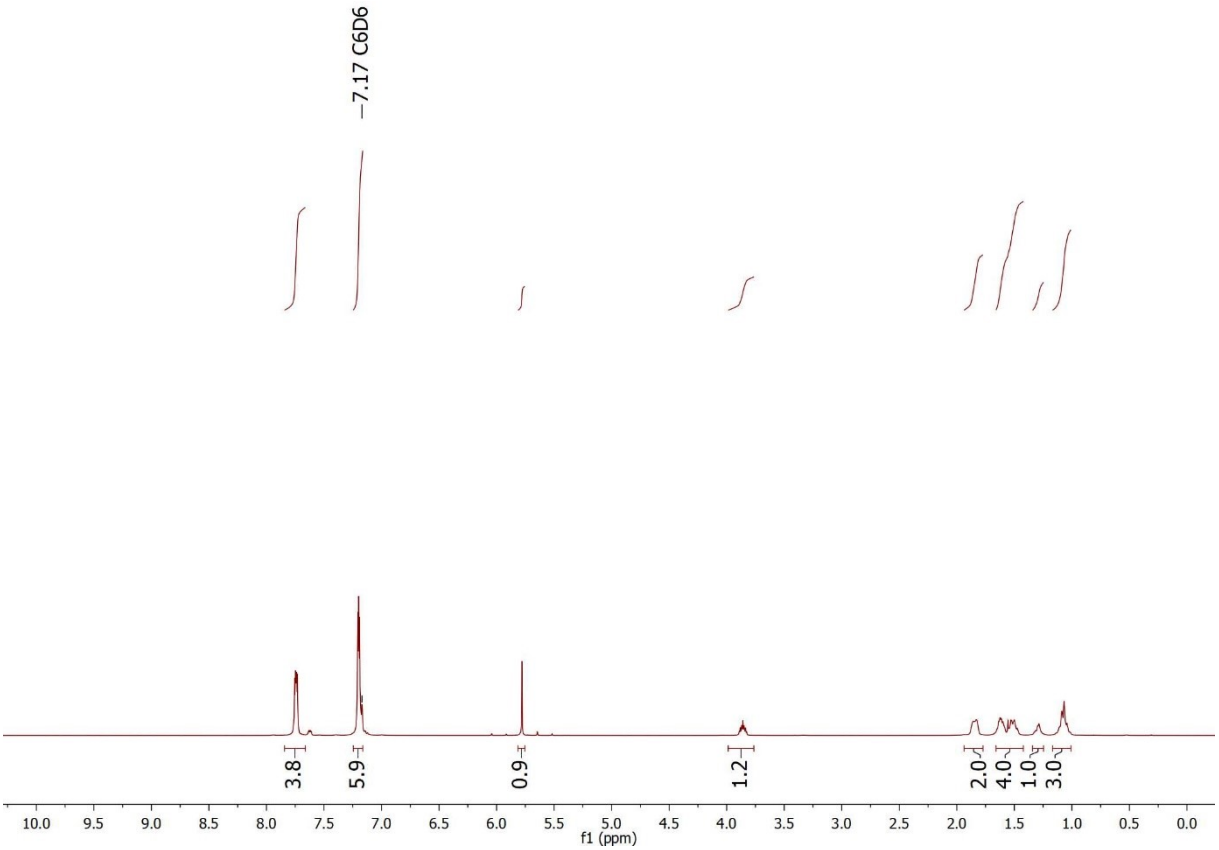

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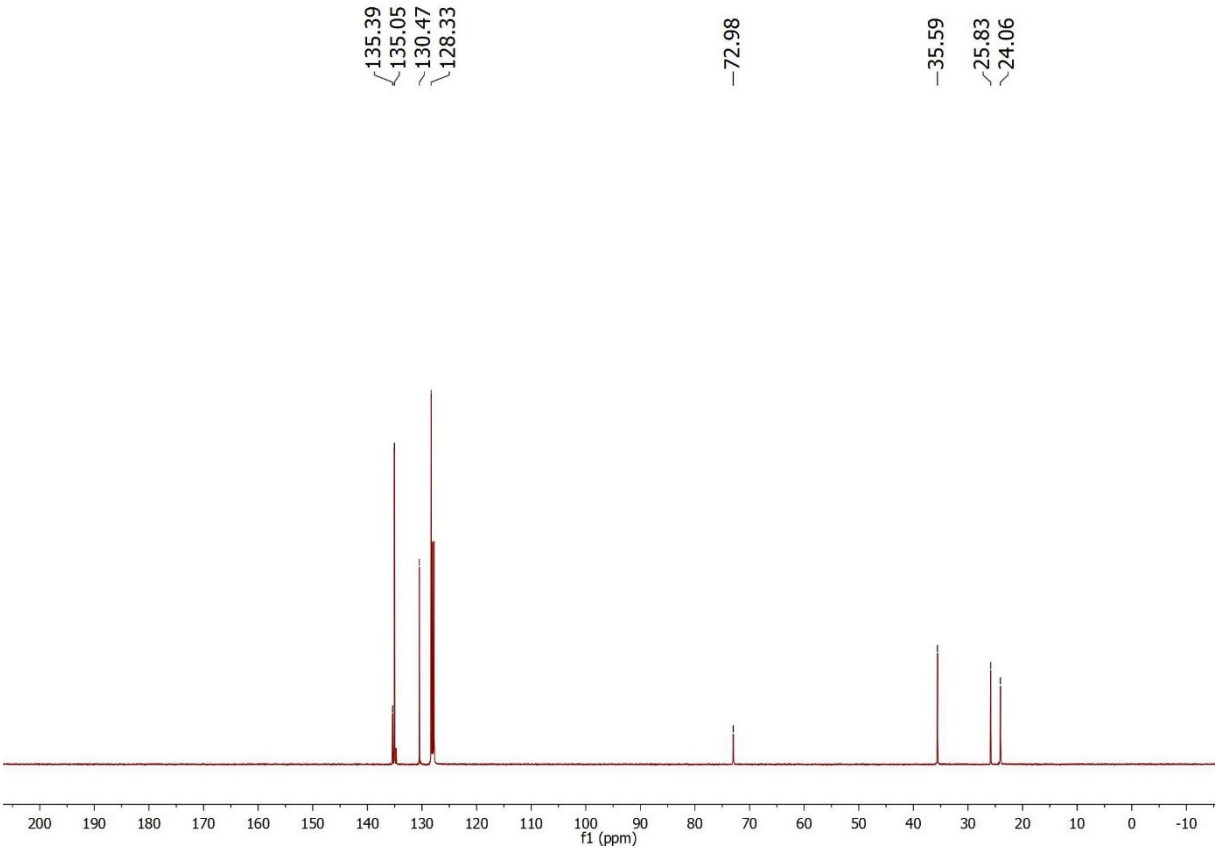
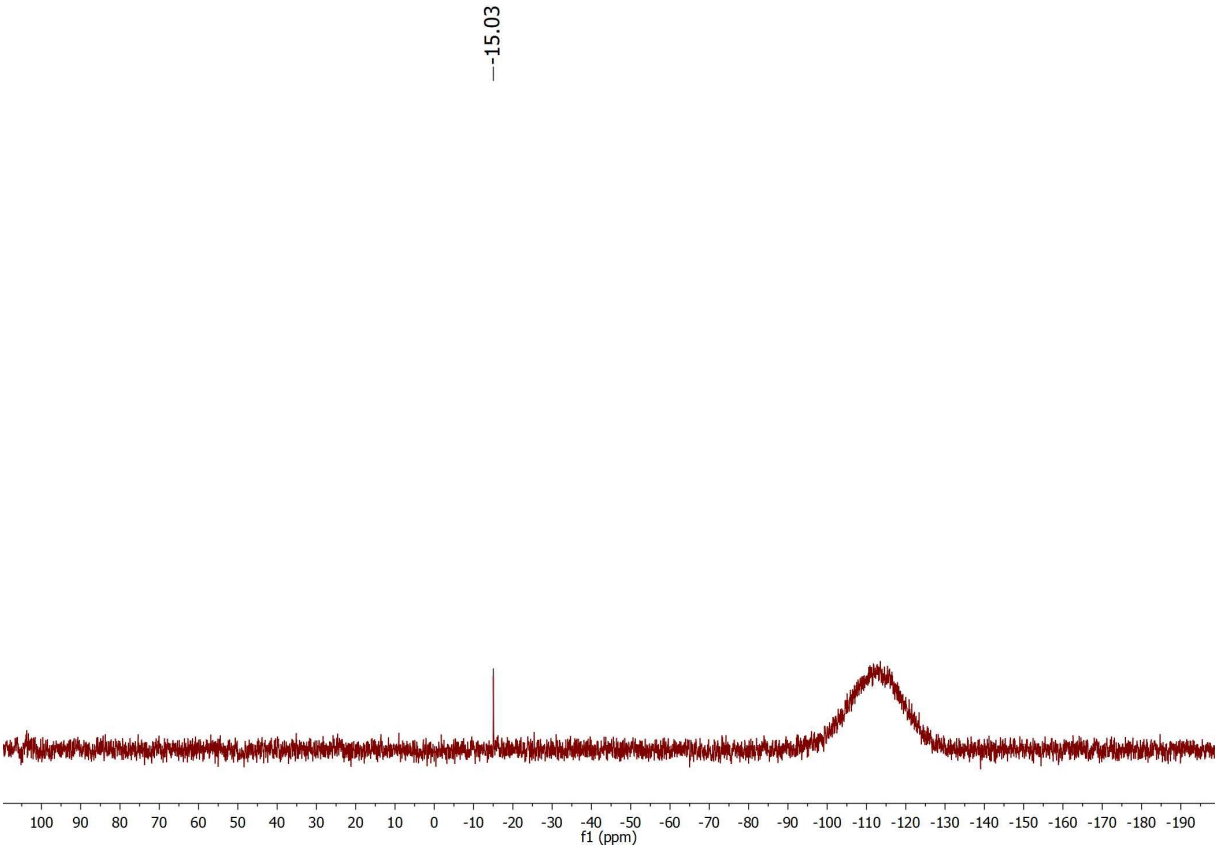
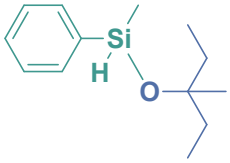


^{29}Si NMR



Methyl((3-methylpentan-3-yl)oxy)(phenyl)silane 6f



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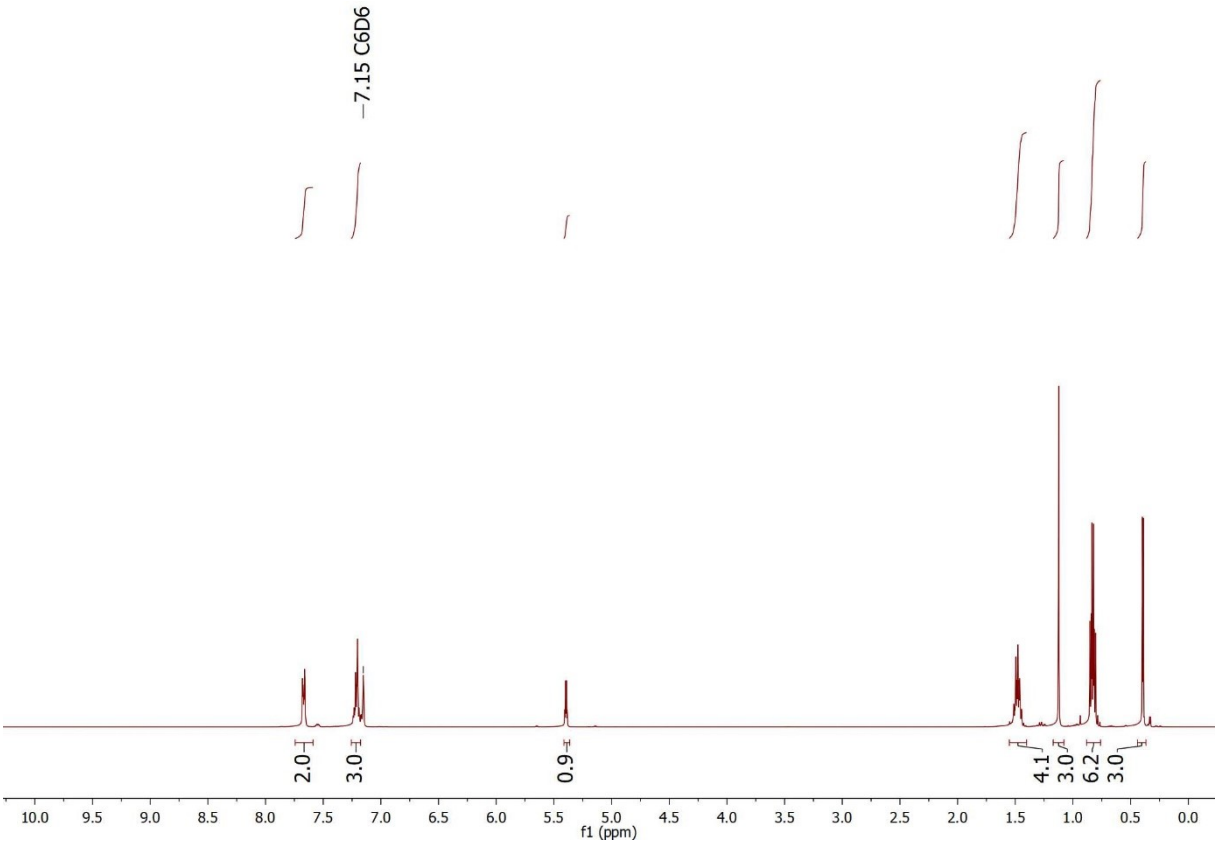

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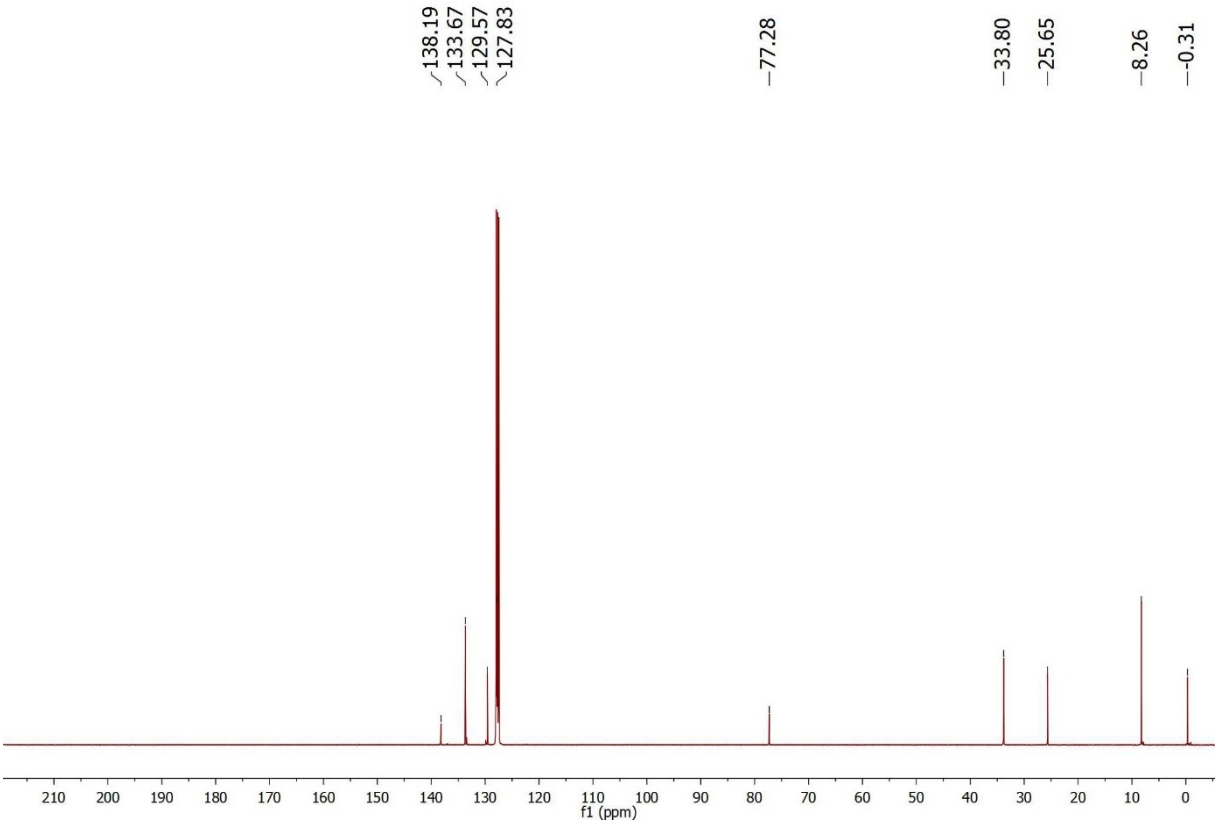
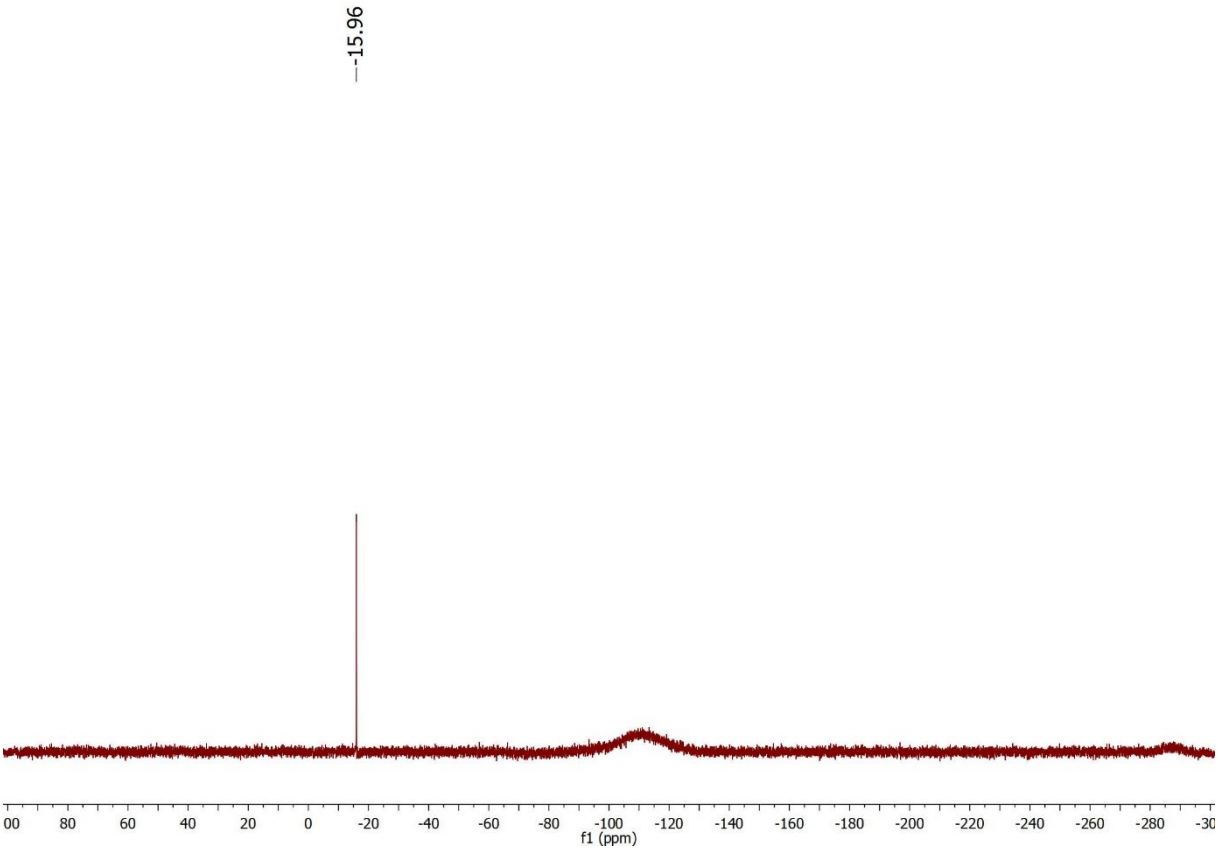
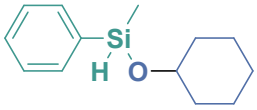


^{29}Si NMR



(Cyclohexyloxy)(methyl)(phenyl)silane 6g



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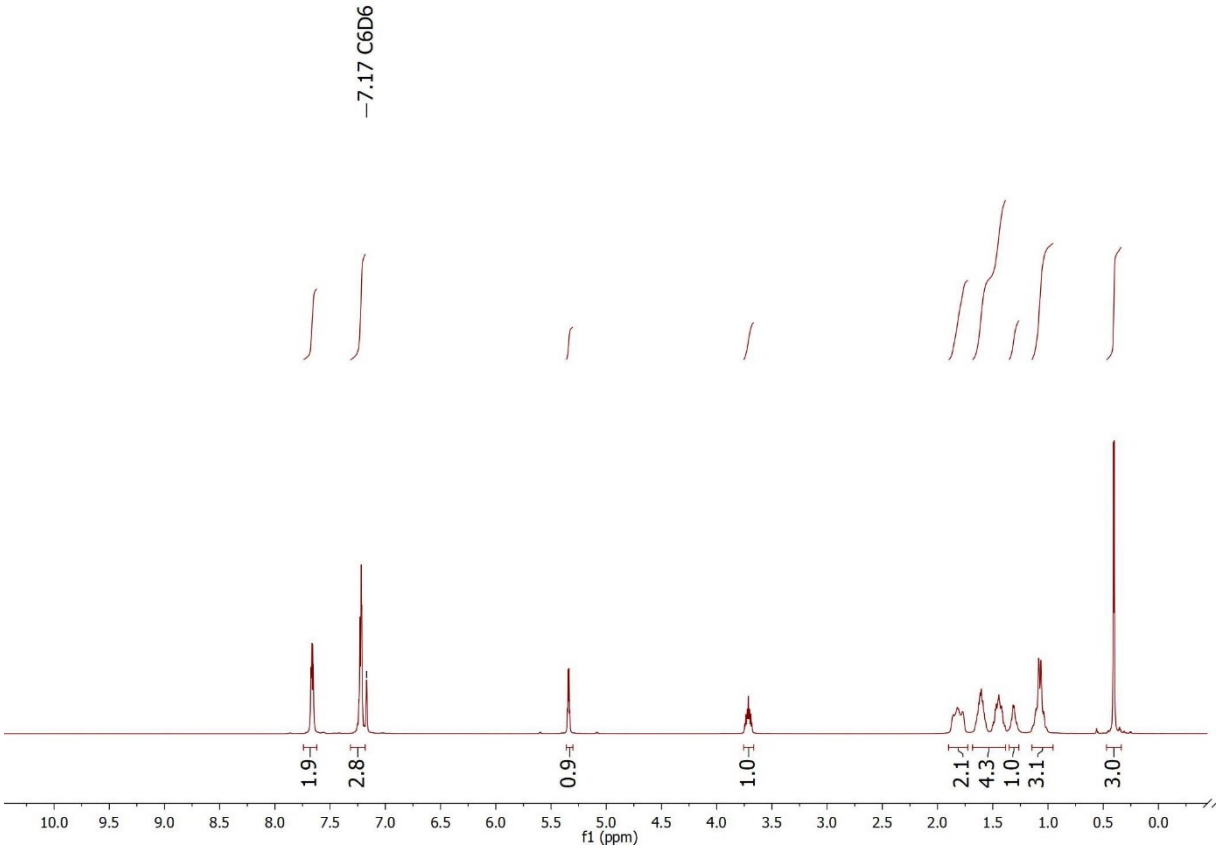

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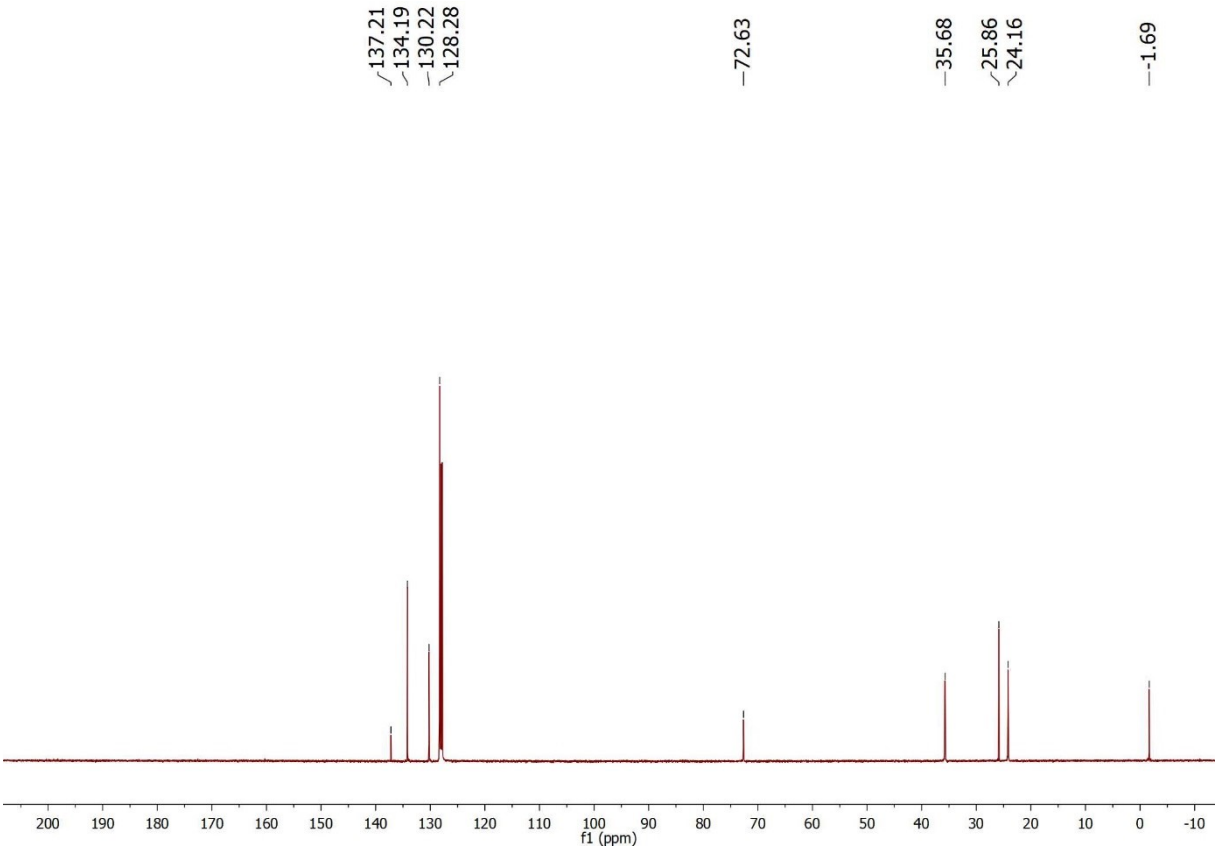

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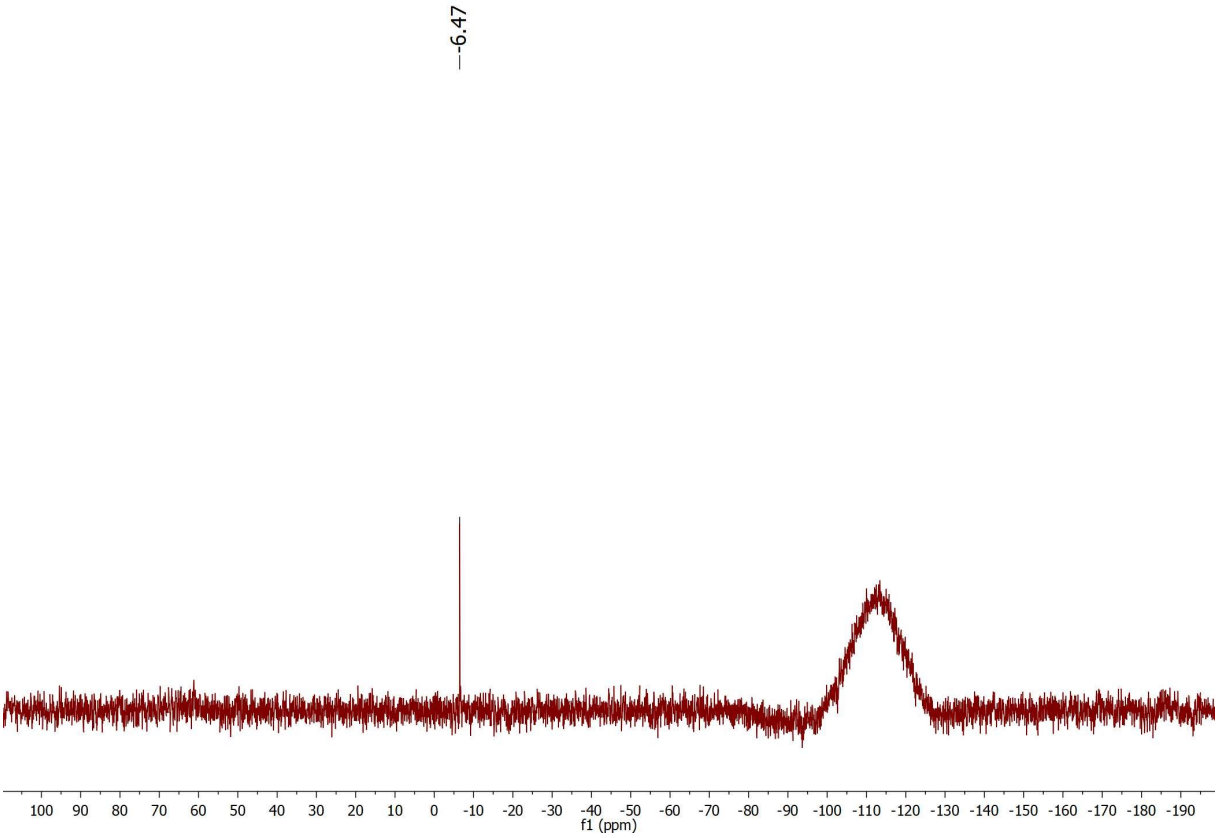
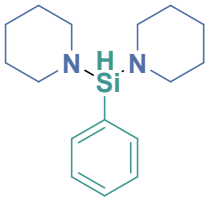
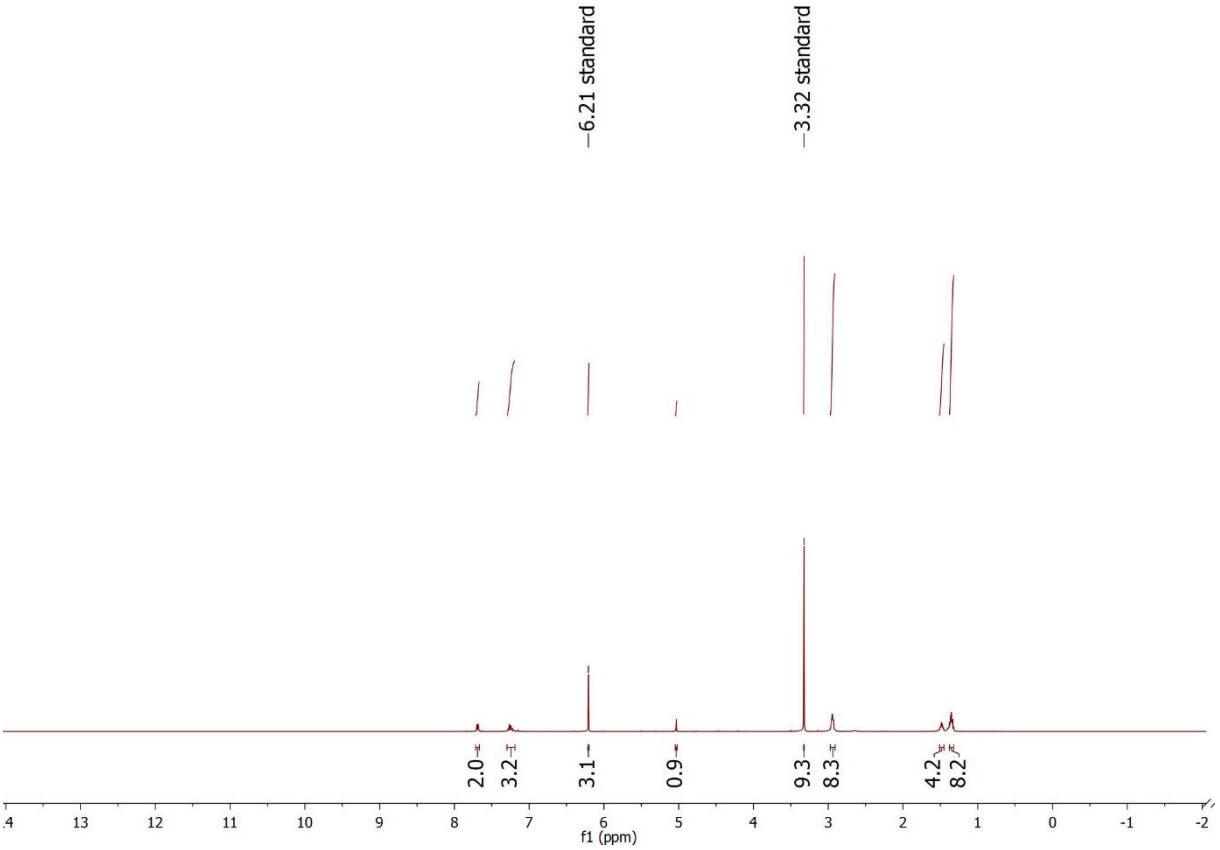


1,1'-(Phenylsilanediyl)dipiperidine 6h



^{1}H NMR



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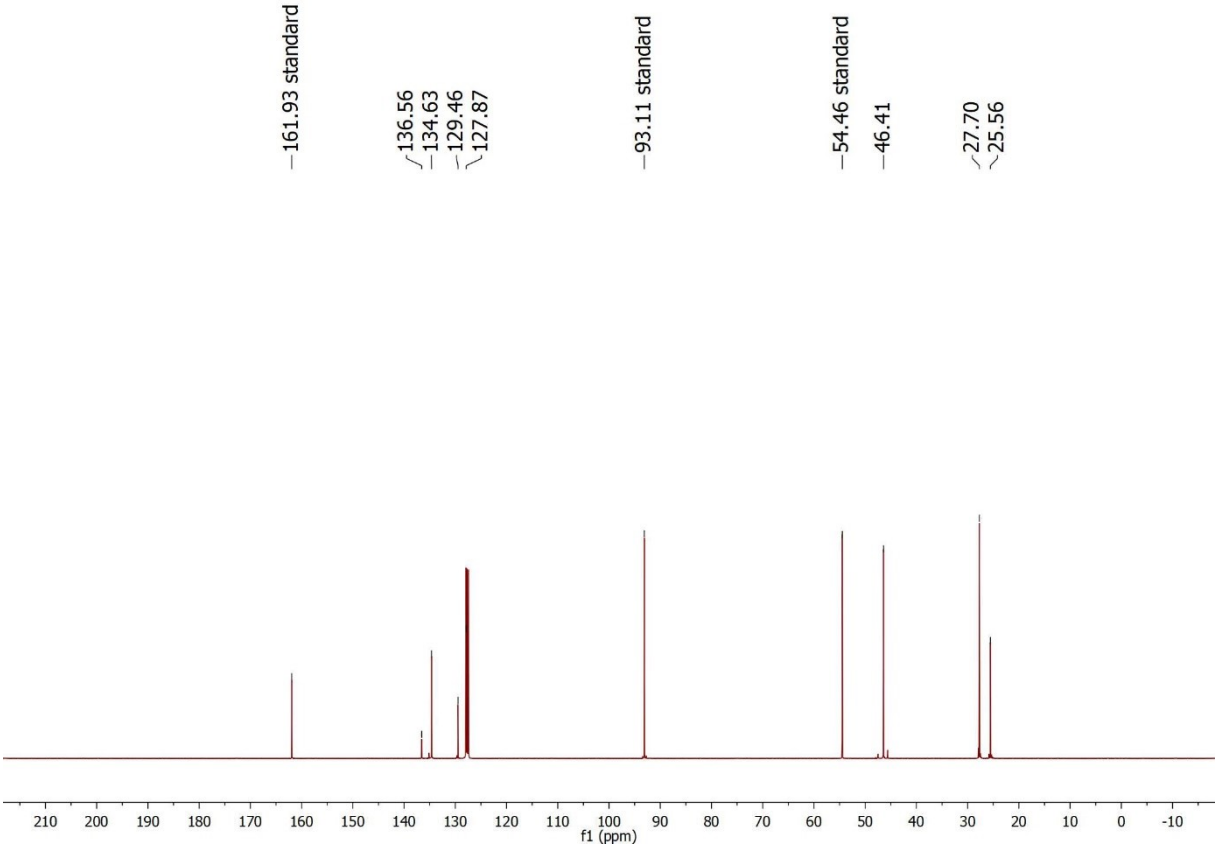
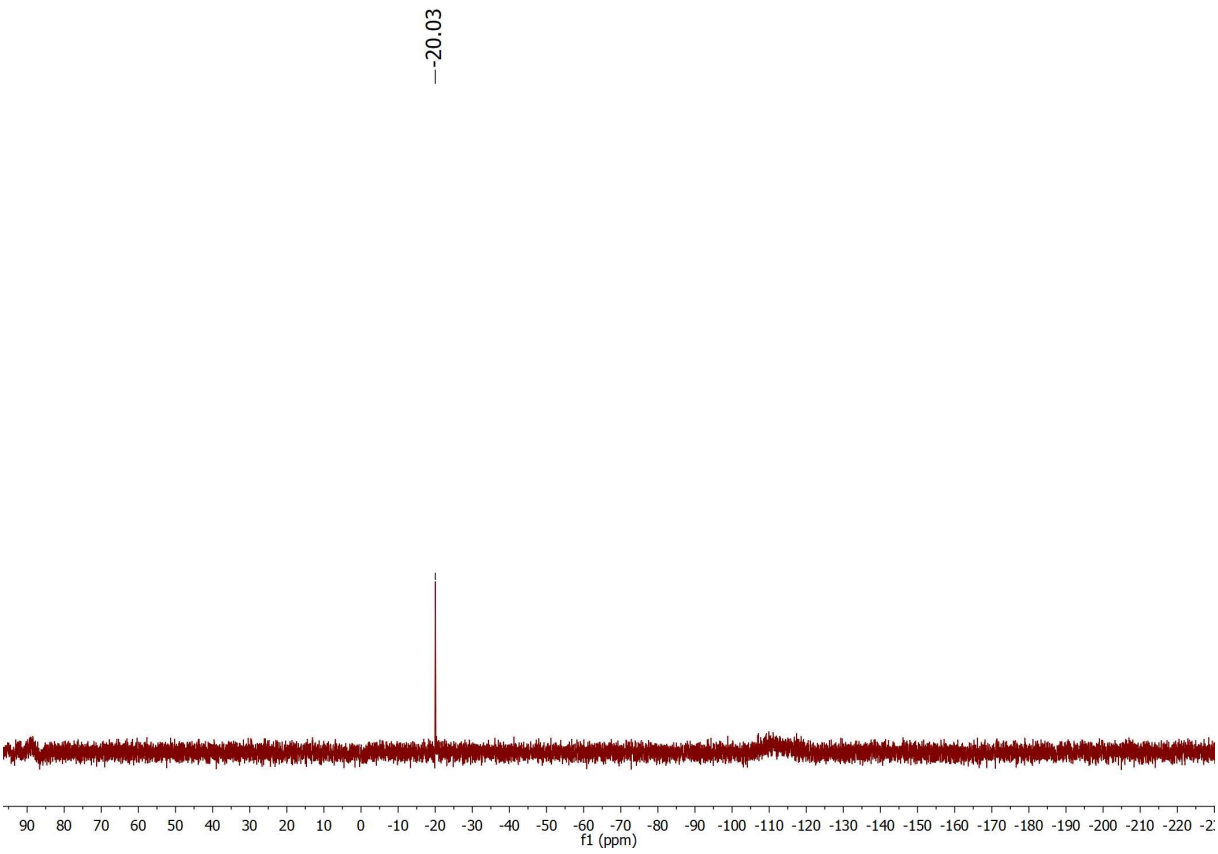
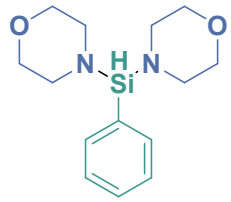


^{29}Si NMR



4,4'-(Phenylsilanediyl)dimorpholine 6i



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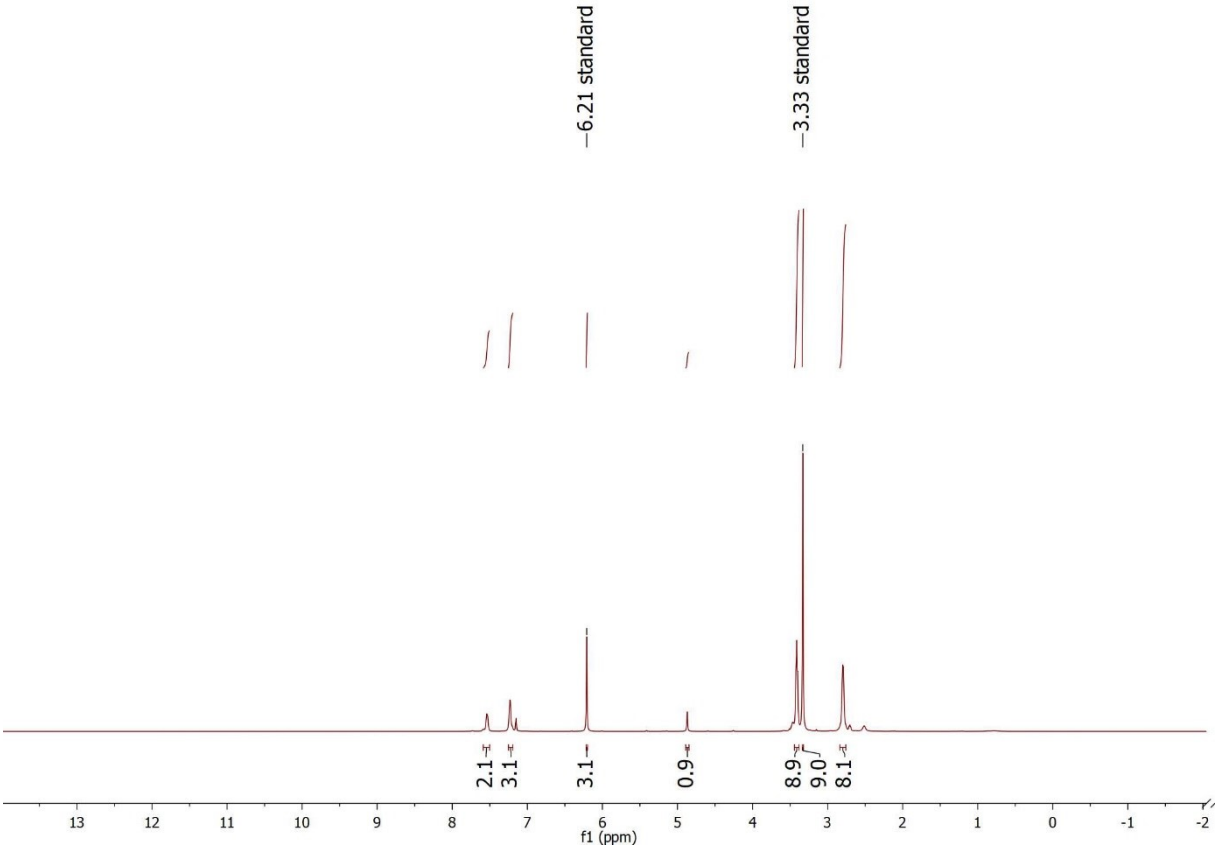

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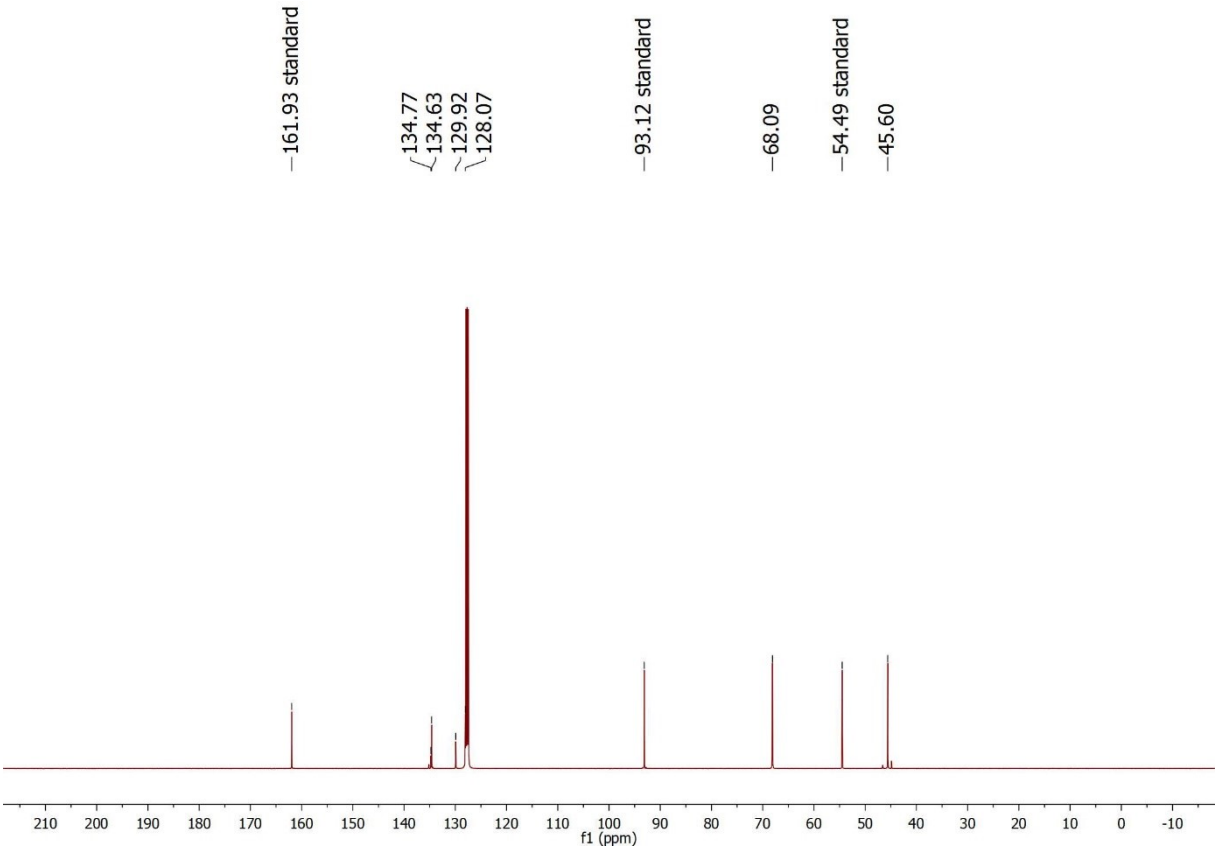
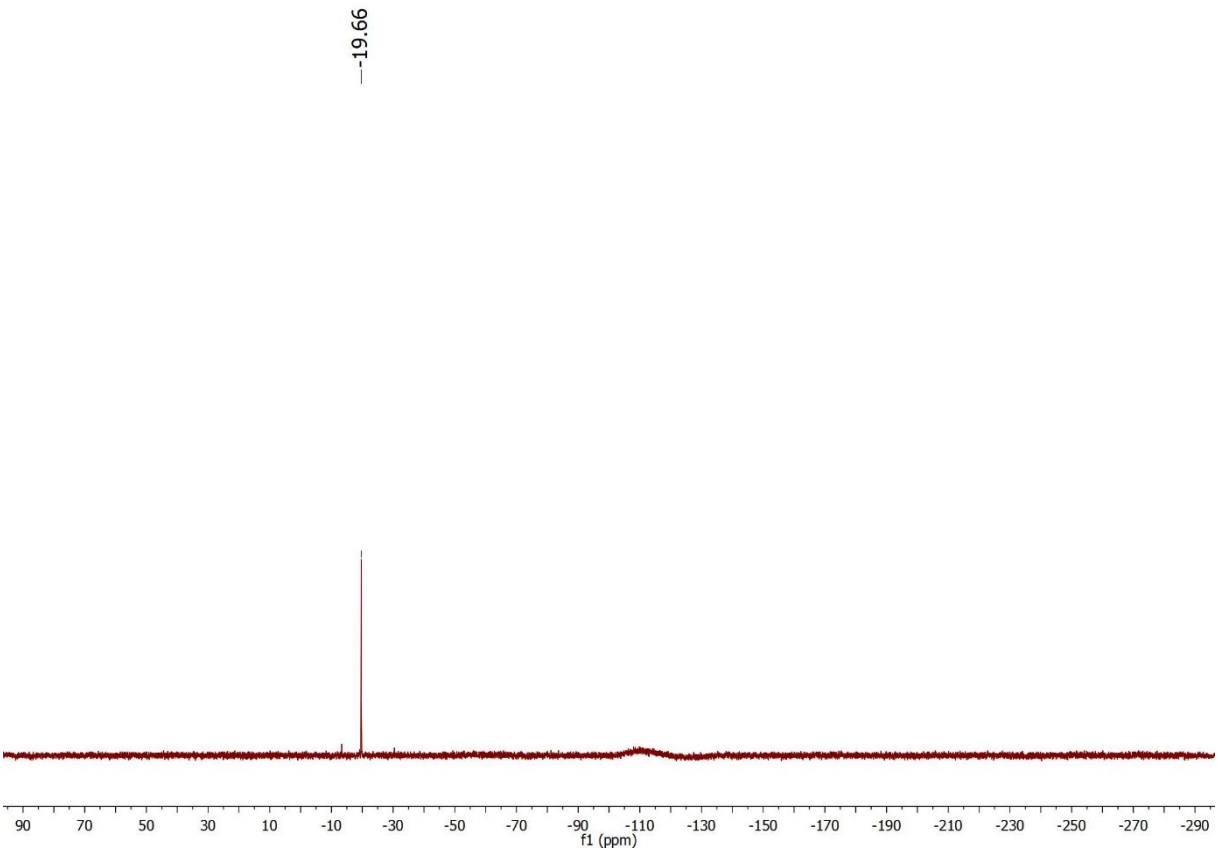
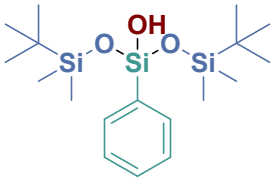


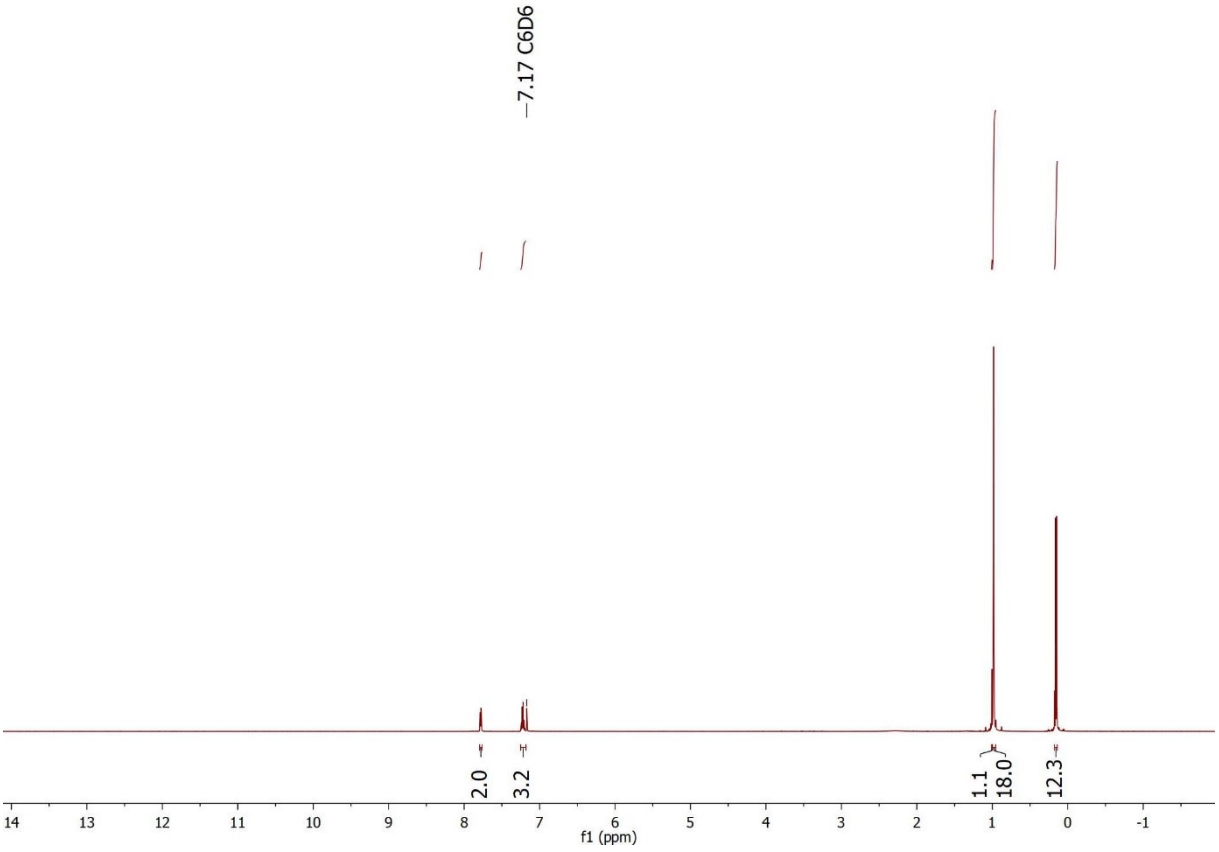
^{29}Si NMR



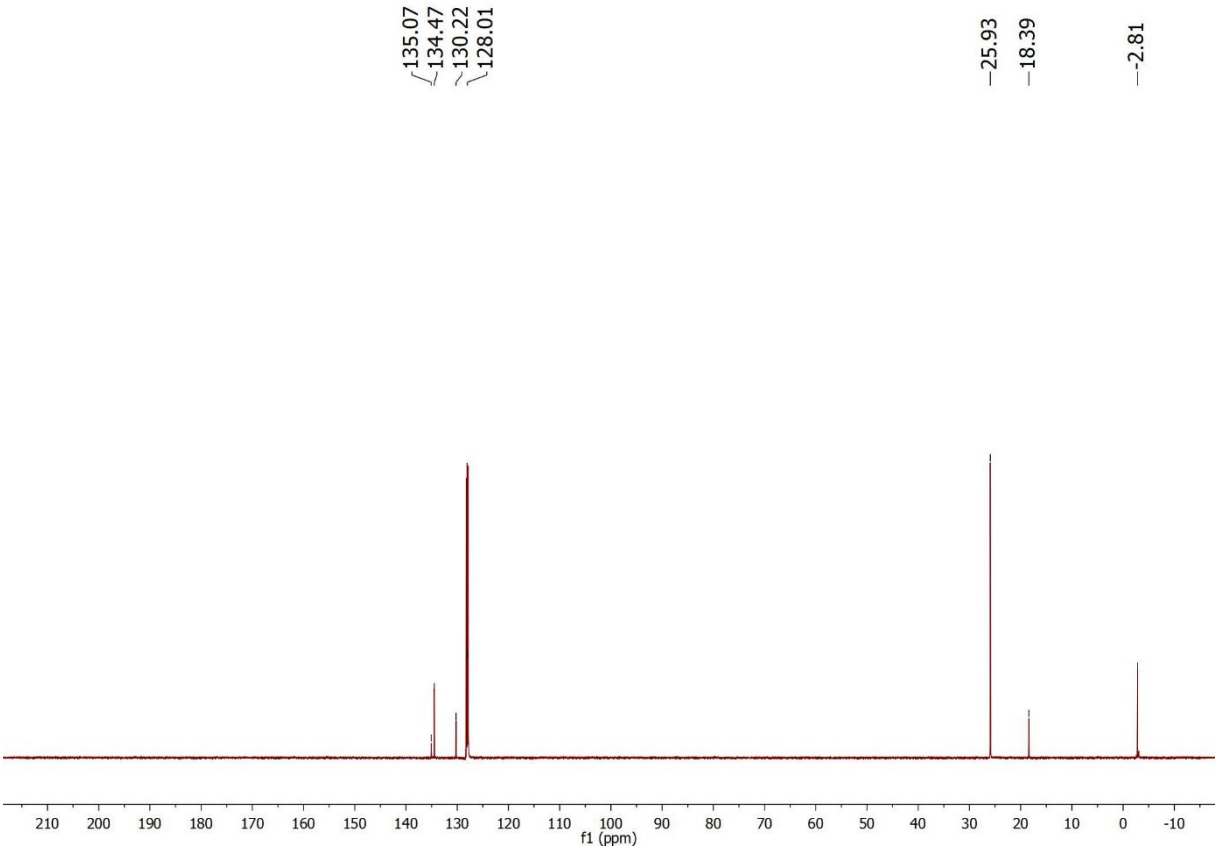
1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyltrisiloxan-3-ol 7a



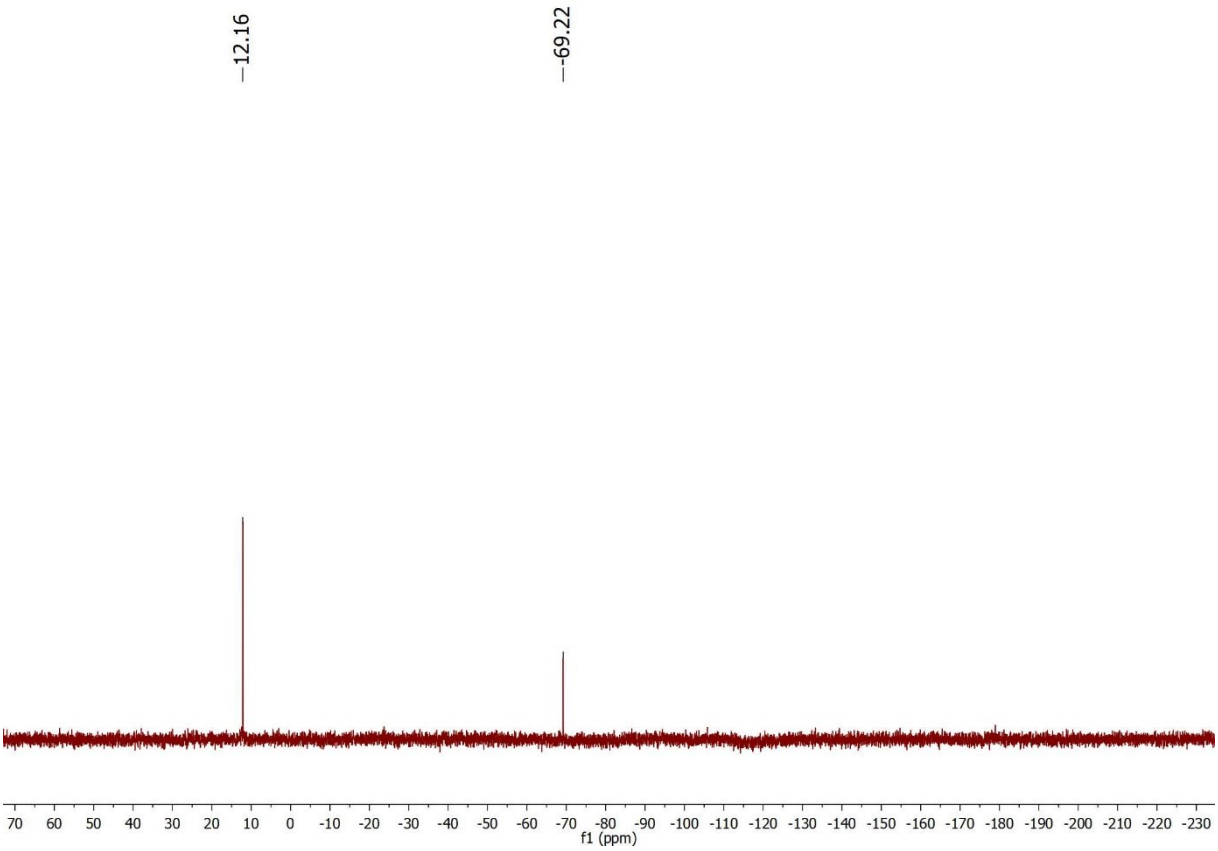
^{1}H NMR



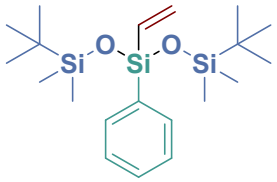
^{13}C NMR



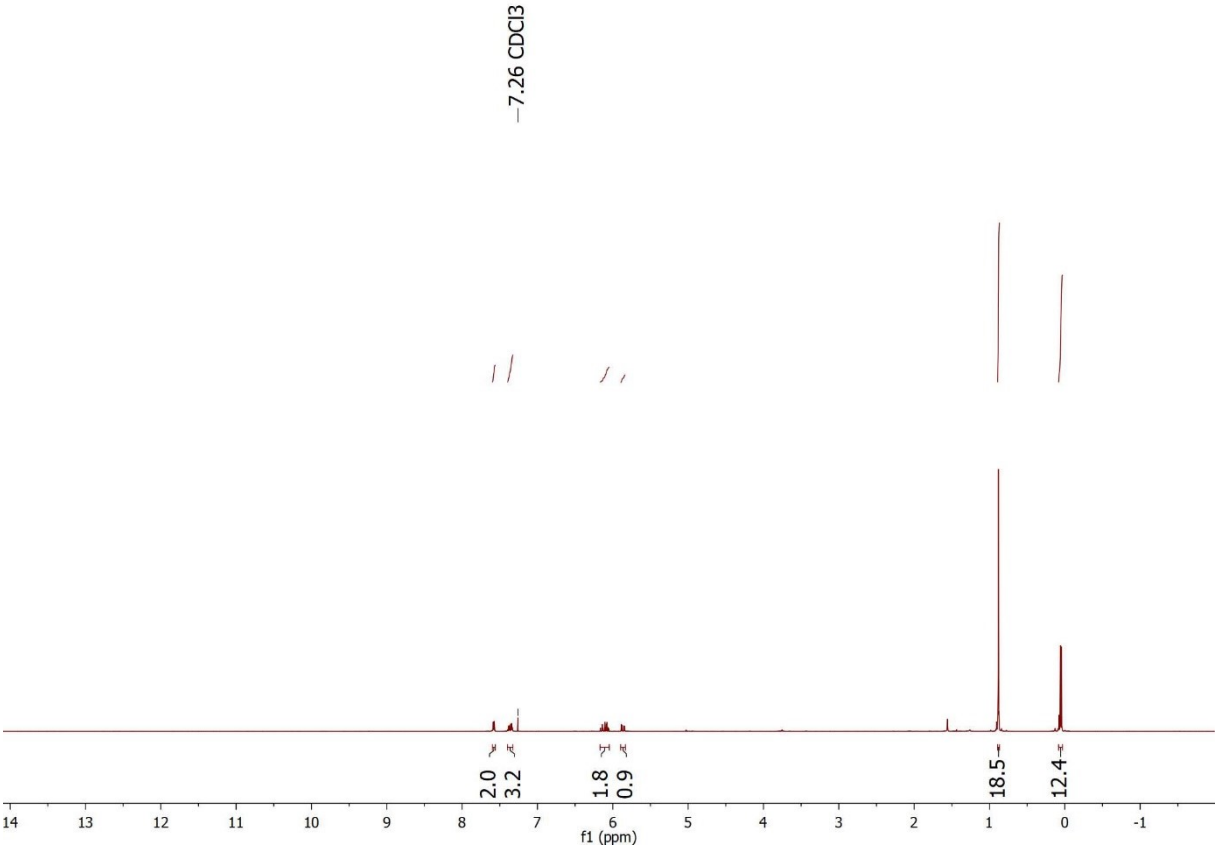
^{29}Si NMR



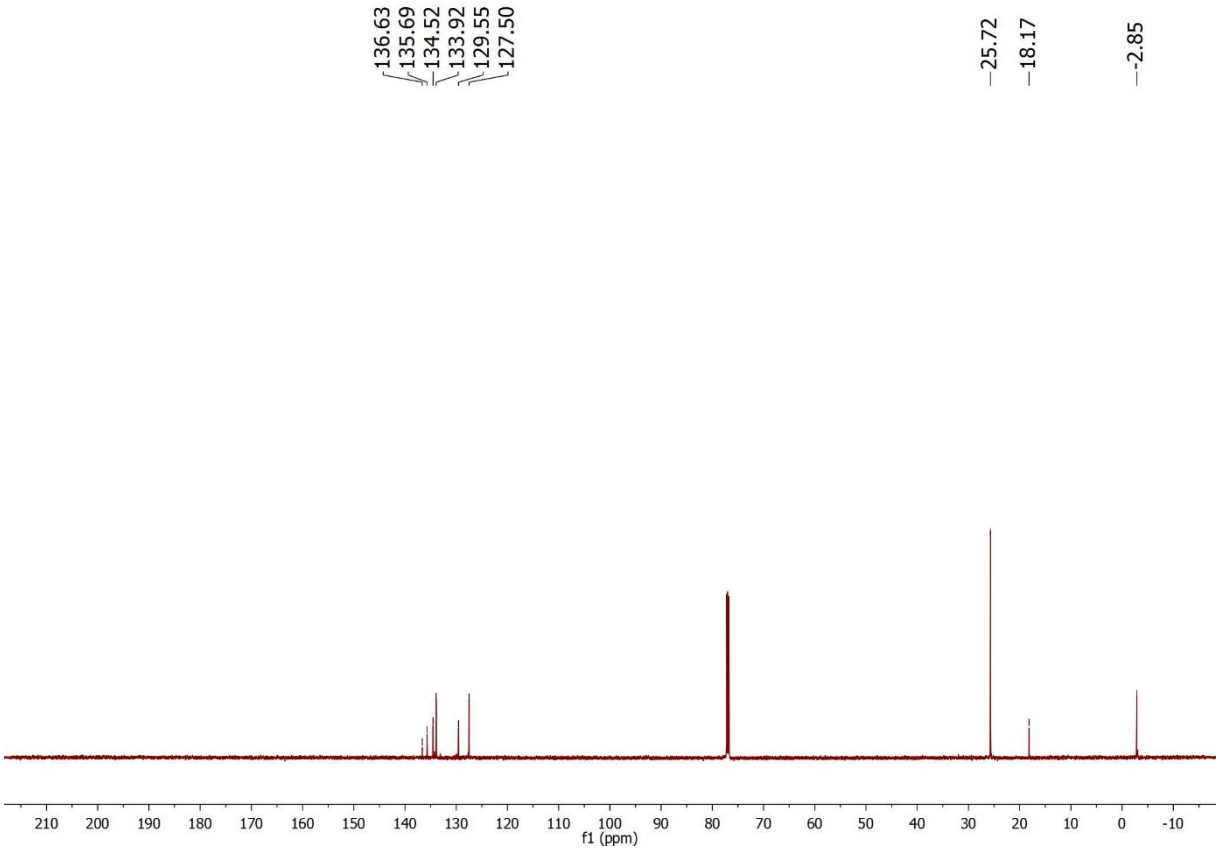
1,5-Di-tert-butyl-1,1,5,5-tetramethyl-3-phenyl-3-(vinyloxy)trisiloxane 7b



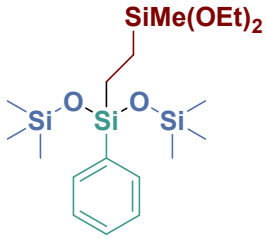
^{1}H NMR



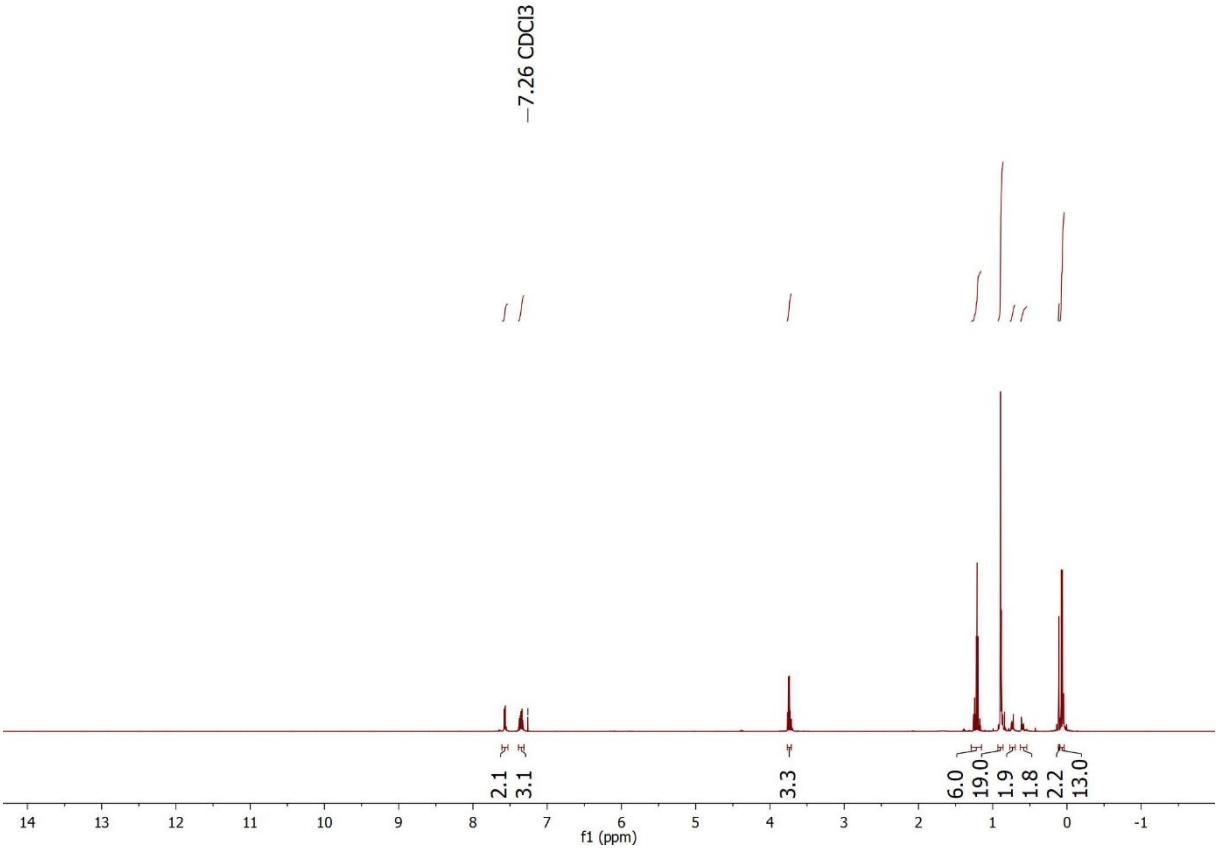
^{13}C NMR


136.63
135.69
134.52
133.92
129.55
127.50
25.72
18.17
-2.85
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

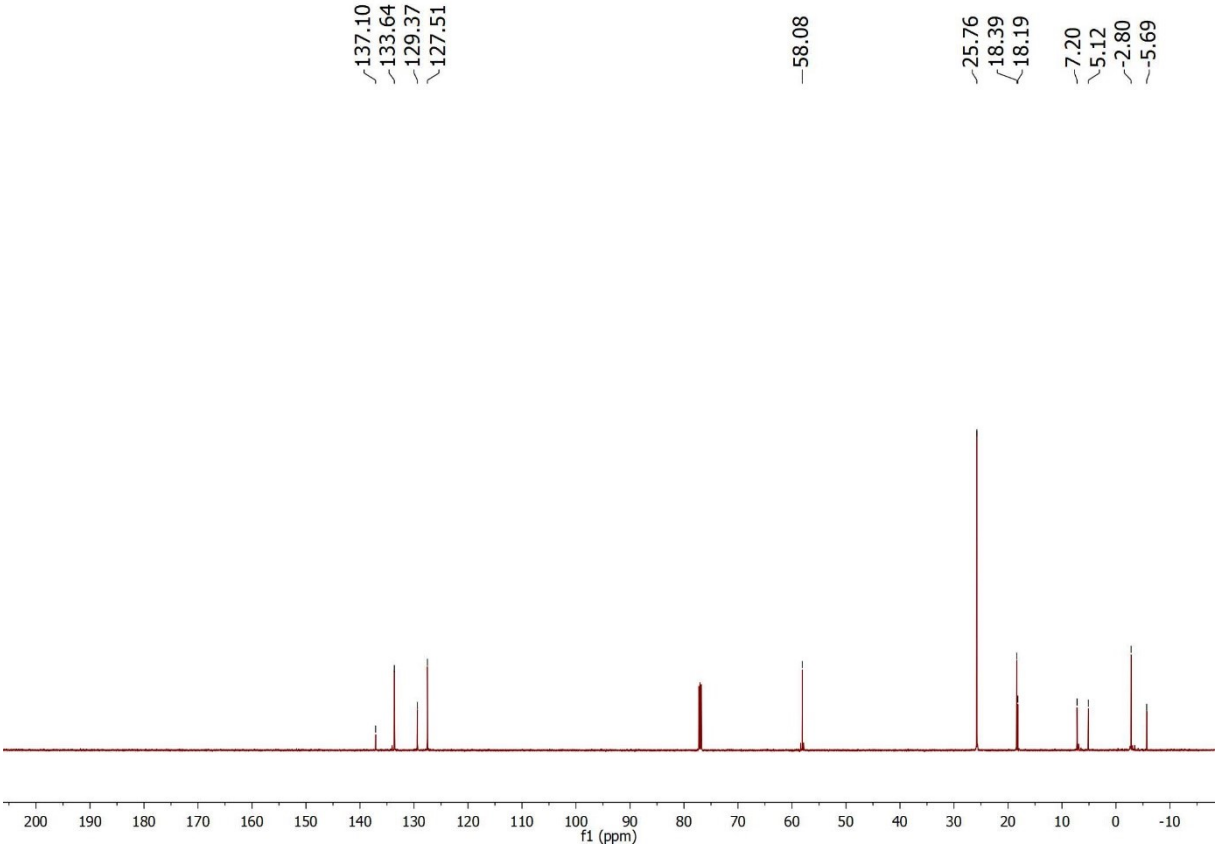
1,5-Di-tert-butyl-3-(2-(diethoxy(methyl)silyl)ethyl)-1,1,5,5-tetramethyl-3-phenyltrisiloxanec 7c



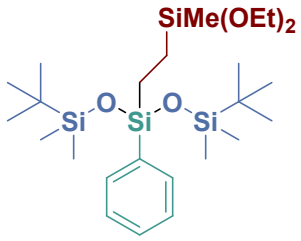
^{1}H NMR



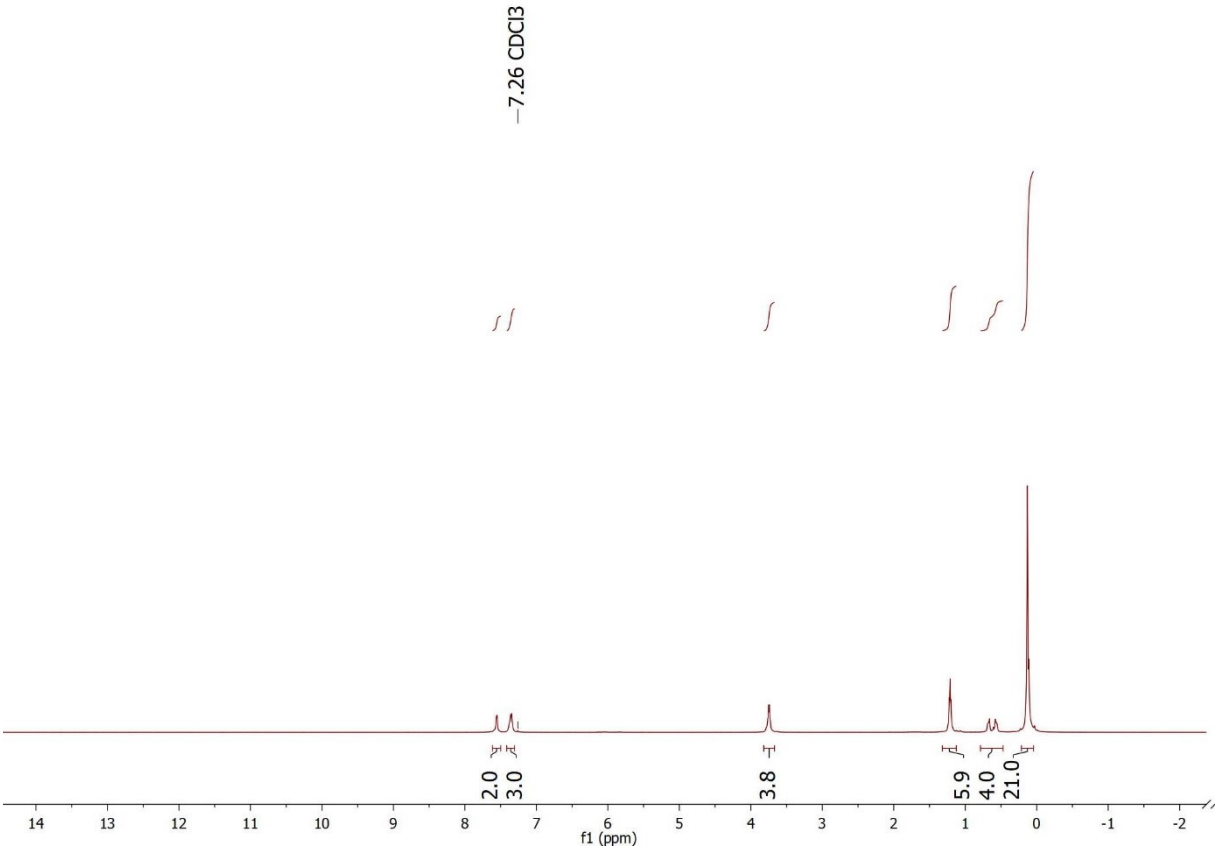
^{13}C NMR


137.10
133.64
129.37
127.51
58.08
25.76
18.39
18.19
7.20
5.12
-2.80
-5.69
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

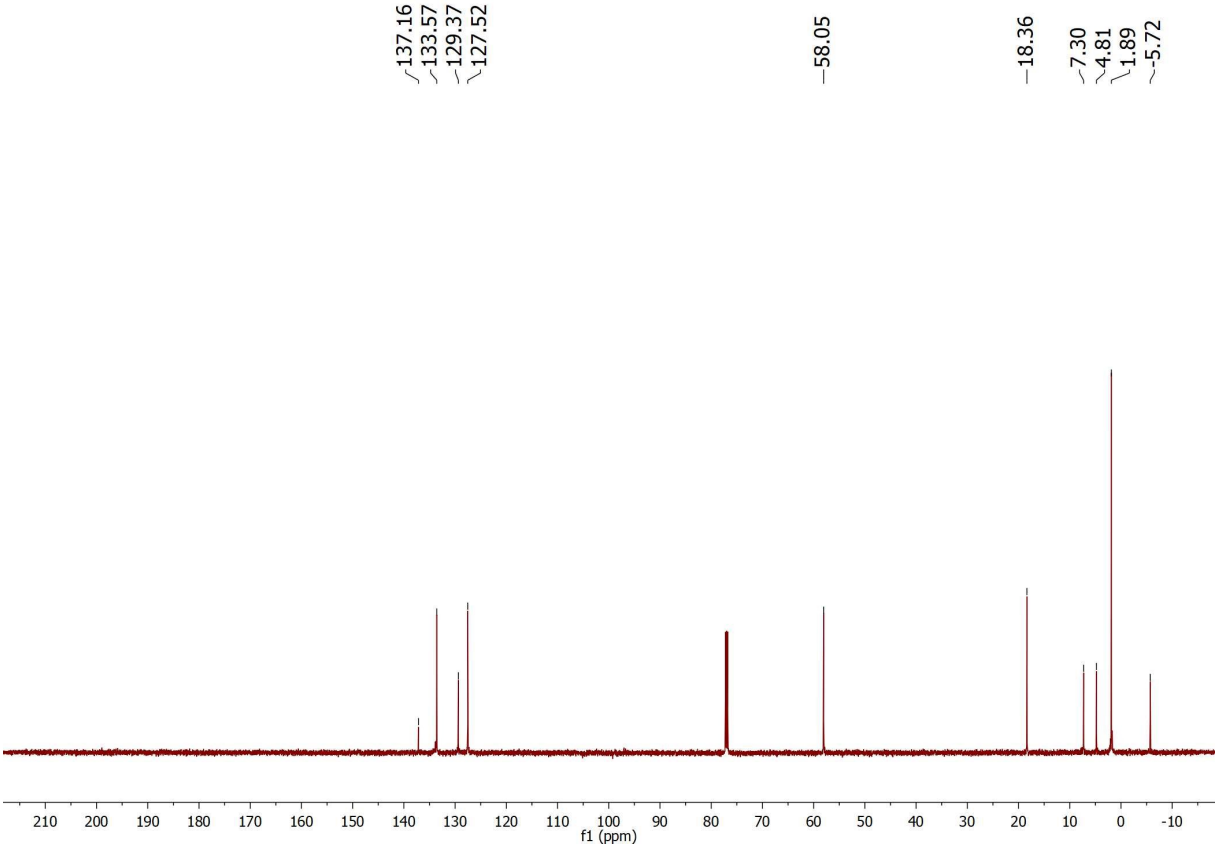
3-(2-(Diethoxy(methyl)silyl)ethyl)-1,1,1,5,5,5-hexamethyl-3-phenyltrisiloxane 7d



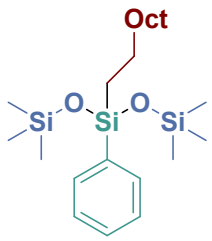
^{1}H NMR



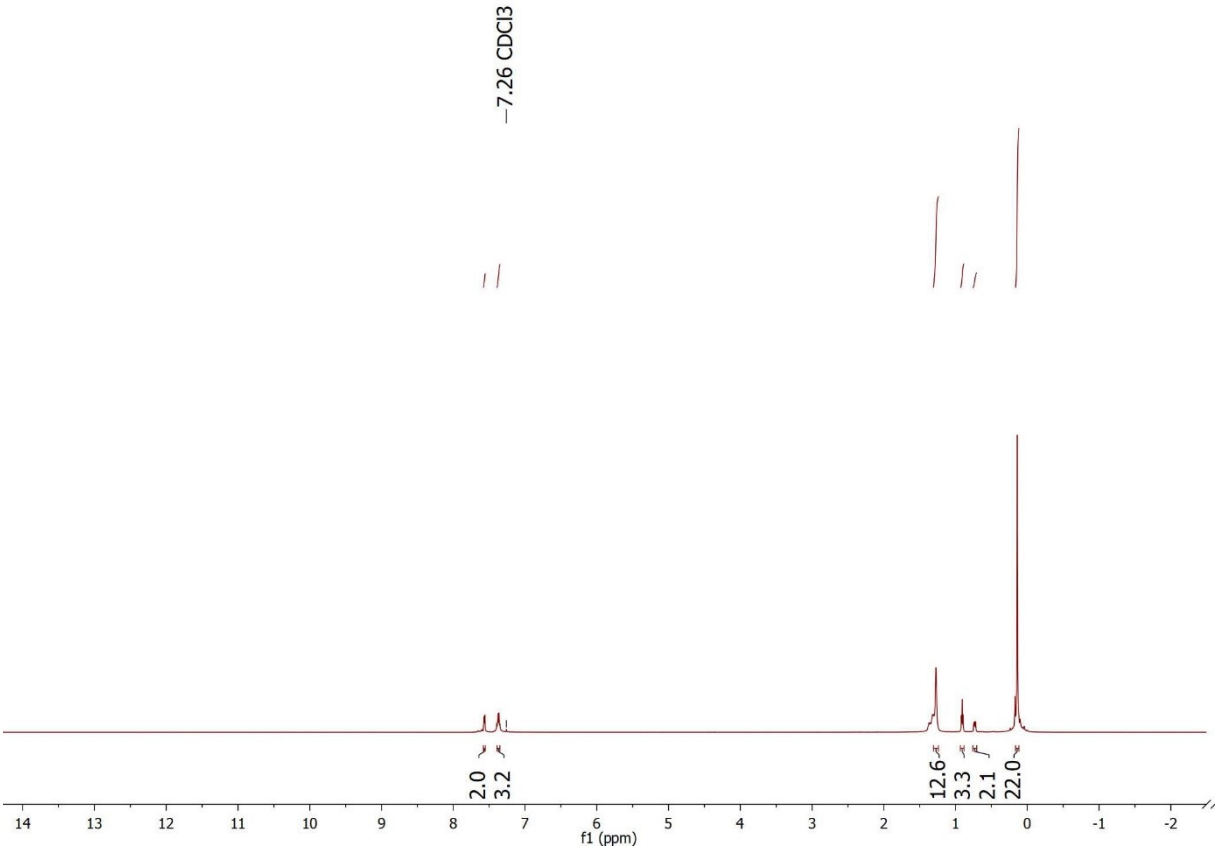
^{13}C NMR


137.16
133.57
129.37
127.52
58.05
18.36
7.30
4.81
1.89
-5.72
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

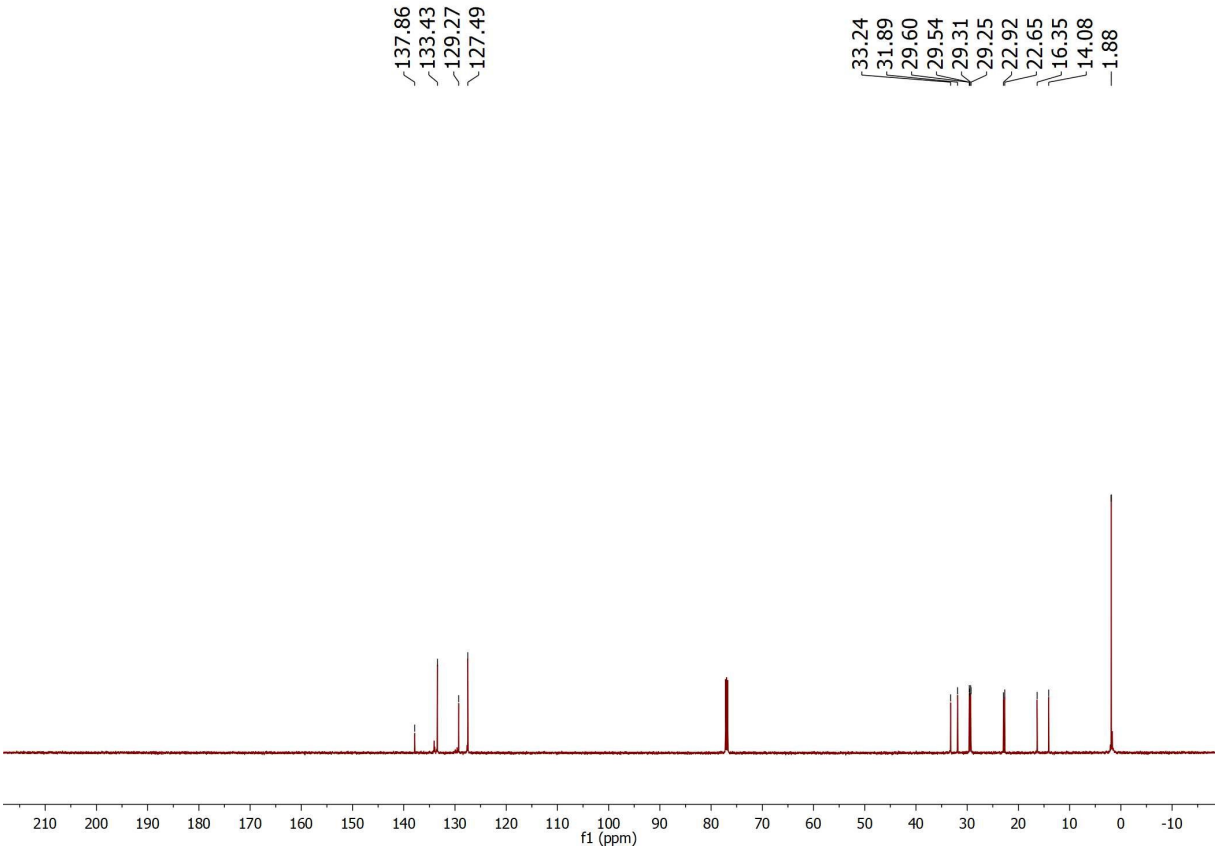
3-Decyl-1,1,1,5,5,5-hexamethyl-3-phenyltrisiloxane 7e



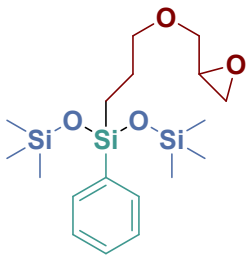
^{1}H NMR



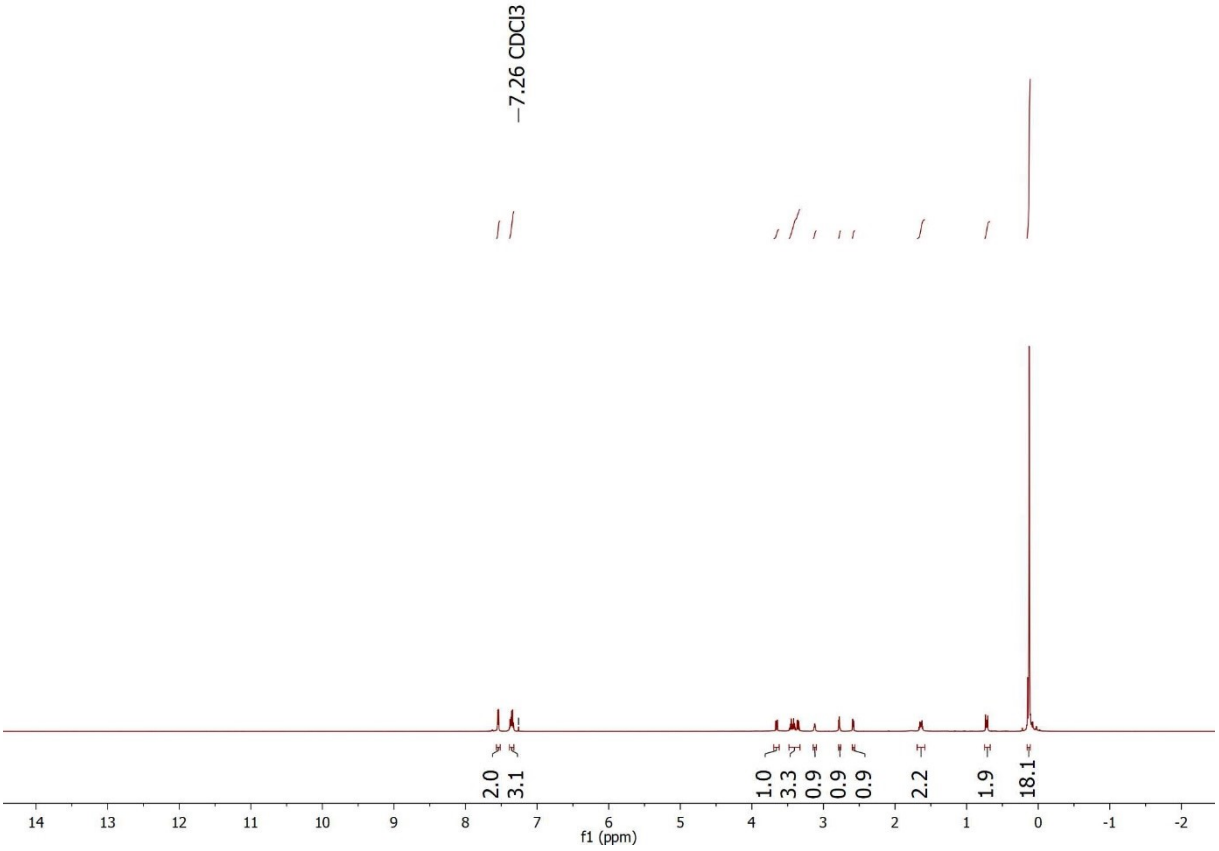
^{13}C NMR


137.86
133.43
129.27
127.49
33.24
31.89
29.60
29.54
29.31
29.25
22.92
22.65
16.35
14.08
1.88
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

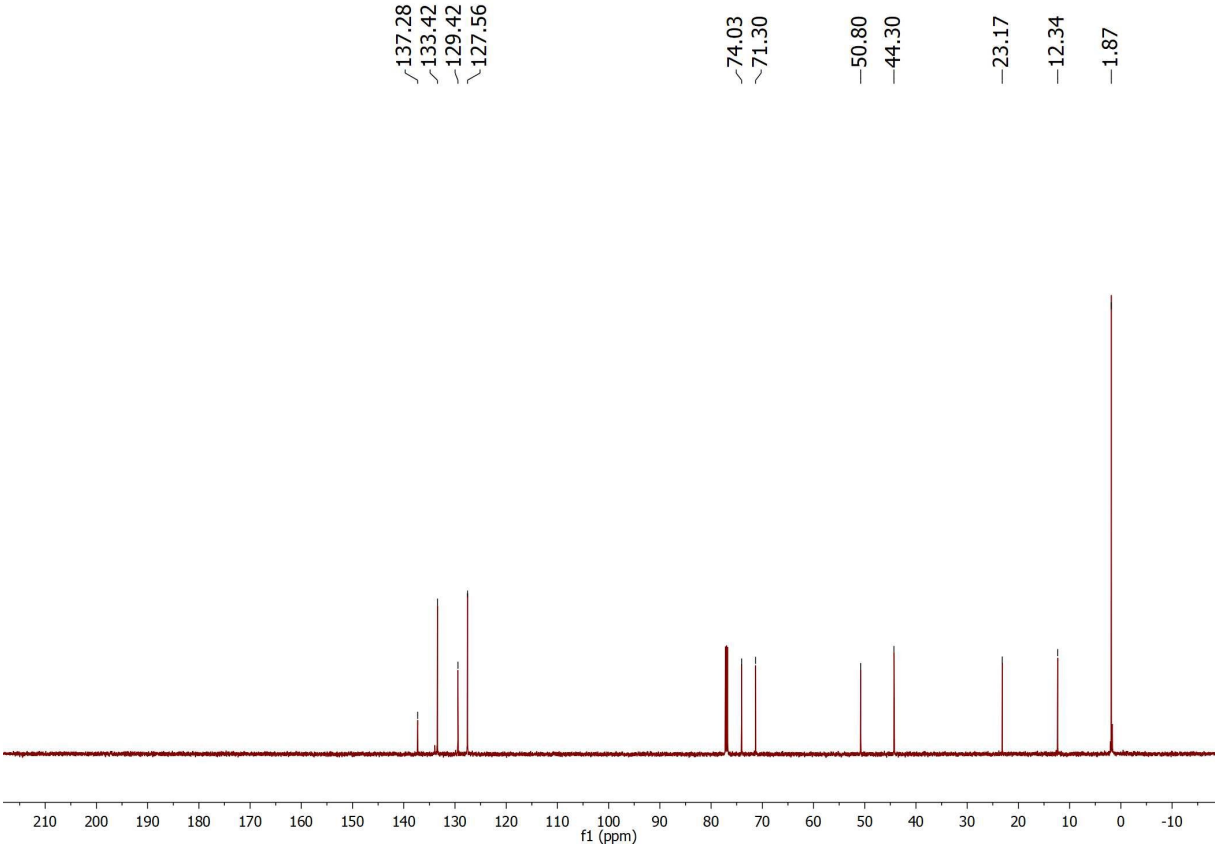
1,1,1,5,5,5-Hexamethyl-3-(3-(oxiran-2-ylmethoxy)propyl)-3-phenyltrisiloxane 7f



^{1}H NMR



^{13}C NMR


137.28
133.42
129.42
127.56
74.03
71.30
50.80
44.30
23.17
12.34
1.87
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

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