Electronic Supplementary Material (ESI) for Polymer Chemistry.

Supplementary Information

Building-Block Approach to Discrete and Sequence-Specific Oligosiloxanes

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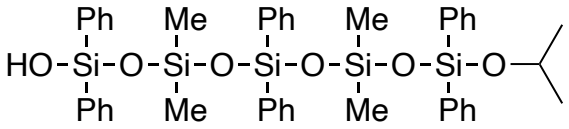
General Information

All experimental manipulations were performed under a nitrogen atmosphere using Schlenk techniques or a MBRAUN LABmaster Pro SP glove box. NMR spectra were recorded on a Bruker AVANCE III HD (^{1}H NMR at 600 MHz; ^{13}C{^{1}H} NMR at 150 MHz; ^{29}Si{^{1}H} NMR at 119MHz) NMR spectrometer with a CryoProbe. The ^{1}H NMR (600 MHz), ^{13}C{^{1}H} NMR (150 MHz) and ^{29}Si{^{1}H} NMR (119 MHz) chemical shifts are reported in δ (ppm) and are referenced to tetramethysilane (TMS: δ = 0 ppm) or the residual solvent resonance as an internal standard. The matrix-assisted laser desorption ionization time-of-flight (MALDI-TOF) mass spectra were collected on a Bruker Autoflex Speed. Differential scanning calorimetry (DSC) was carried out at a heating rate of 5 K/min on a Hitachi High-Tec DSC7020 calorimeter. The samples were sealed in aluminum pans. X-ray diffraction measurements were carried out on a Rigaku R-AXIS IV X-ray diffractometer with a CuKα line (λ = 0.1542 nm, 40 kV, 30 mA). The samples were sealed in Lindman glass capillaries (1.5 mm o. d., 10 μm wall thickness). The sear viscosity was measured on an Anton-Paar MCR302 rheometer equipped with Peltier temperature control devices using a parallel plate (25 mmϕ). Column chromatography was performed with a Yamazen EPCLC AI-580S using a Universal^{TM} Column Premium (silica gel, 30 μm). Gel Permeation Chromatography (GPC) was performed with a JAI LaboACE LC-780 using a JAIGEL-2HR-40 column.

All chemicals were reagent grade and used as received without further purification. Tris(pentafluorophenyl)borane ($B(C_6F_5)_3$), diphenylsilane (Ph_2SiH_2), 1,1,5,5-tetramethyl-3,3-diphenyltrisiloxane, diphenylsilanediol were purchased from Tokyo Chemical Industry Co., Ltd. Toluene, *n*-hexane, acetone, CH_2Cl_2, trichloroisocyanuric acid, chlorotrimethylsilane (Me_3SiCl), chlorodimethylsilane (Me_2HSiCl), triethylamine (Et_3N), 2-propanol and $CDCl_3$ were purchased from Fujifilm Wako Pure Chemical Corporation. Alumina (activated, neutral, Brockmann I) was purchased from Aldrich. Palladium-activated carbon (Pd/C) was purchased from N.E.CHEMCAT. 1,1,3,3,5,5,7,7-octamethylsiloxane ($^{H}MD_2M^{H}$) and 1,1,3,3,5,5,7,7,7-nonamethyltetrasiloxane-1-ol were prepared by previously reported procedures[S1,2].

Preparation of Building Blocks and Starting Materials

9-Isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (2)



$B(C_6F_5)_3$ (51.2 mg, 0.1 mmol) was dissolved in toluene (80 mL). To the mixture was added acetone (2.95 mL, 40 mmol) and then 1,1,5,5-tetramethyl-3,3-diphenyltrisiloxane (6.65 mL, 20 mmol) with stirring at room temperature. After 30 min, Ph_2SiH_2 (7.37 mL, 40 mmol) was added. After 30 min, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (60 mL) and cooled down to –80 °C and kept under constant stirring. To the solution was added trichloroisocyanuric acid (1.53 g, 6.67 mmol) in portions. After the addition was completed, the cooling bath was removed, and the solution was allowed to reach room temperature for 12 h. To the reaction mixture was added triethylamine (5.6 mL, 40 mmol) and 2-propanol (3.1 mL, 40 mmol). After 4 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (60 mL) again. To the solution was added 5% palladium carbon (40 mg, 0.02 mmol) and H_2O (0.72 g, 40 mmol), and the reaction mixture was refluxed. After 12 h, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2). The crude product was purified by silica-gel column chromatography (*n*-hexane: CH_2Cl_2 = 1:1) to give 9-isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (**2**) (7.21 g, 45%) as a colorless oil.

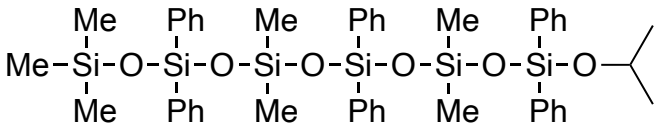
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.59-7.50 (m, 12H), 7.41-7.33 (m, 6H), 7.32-7.20 (m, 12H), 4.11 (septet, J = 6.1 Hz, 1H), 2.31 (s, 1H), 1.12 (d, J = 6.1 Hz, 6H), 0.10, (s, 6H), 0.07 ppm (s, 6H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ 135.5, 135.0, 134.9, 134.6, 134.3, 134.2, 130.1, 129.9, 129.8, 127.72, 127.66, 127.6, 65.7, 25.6, 1.3, 1.2 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ –18.3, –19.1, –38.3, –41.7, –48.0 ppm.

HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{43}H_{50}O_6Si_5Na]^+$ (M+Na): 825.24; found 825.26.

1-Isopropoxy-3,3,7,7,11,11,11-heptamethyl-1,1,5,5,9,9-hexaphenylhexasiloxane (3)



9-Isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (**2**) (1.21 g, 1.5 mmol) was dissolved in CH_2Cl_2 (6.0 mL). To the solution was added Et_3N (553 μL, 3.0 mmol) and Me_3SiCl (381 μL, 3.0 mmol) at room temperature and then the mixture was stirred overnight. The reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was passed through a short silica-gel pad (eluent: *n*-hexane/CH_2Cl_2 = 5:1) to give 1-isopropoxy-3,3,7,7,11,11,11-heptamethyl-1,1,5,5,9,9-hexaphenylhexasiloxane (**3**) (1.31 g, >95%) as a colorless oil.

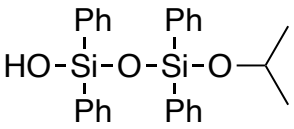
^{1}H NMR (600 MHz, $CDCl_3$): 7.61-7.55 (m, 4H), 7.55-7.48 (m, 8H), 7.39-7.31 (m, 6H), 7.30-7.24 (m, 8H), 7.21 (dd, *J* = 7.5, 7.3 Hz, 4H), 4.12 (sept, *J* = 6.1 Hz, 1 H), 1.12 (d, *J* = 6.1 Hz, 6 H), 0.08-0.05 (m, 15 H), 0.04 ppm (s, 6 H)

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 136.1, 135.6, 135.0, 134.6, 134.3, 134.2, 129.8, 129.72, 129.68, 127.61, 127.56, 65.6, 25.6, 1.9, 1.33, 1.30 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ 9.9, –19.4, –19.5, –41.9, –47.8, –48.1 ppm.

HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{46}H_{58}O_6Si_6Na]^+$ (M+Na): 897.27; found 897.29.

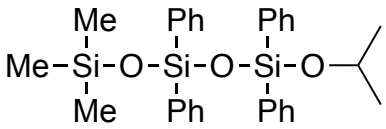
3-Isopropoxy-1,1,3,3-tetraphenyldisiloxan-1-ol



$B(C_6F_5)_3$ (102.4 mg, 0.20 mmol) was dissolved in toluene (80 mL). To the solution was added Ph_2SiH_2 (7.37 mL, 40 mmol) and then acetone (1.47 mL, 20 mmol) over 5 min at room temperature. After 10 min, acetone (1.47 mmol, 20 mmol) was added. After 1 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (60 mL). To the solution was added 5% palladium carbon (40 mg, 0.04 mmol) and H_2O (0.72 g, 40 mmol), and the reaction mixture was refluxed. After 16 h, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2). The crude product was purified by silica-gel column chromatography (eluent: *n*-hexane/ CH_2Cl_2 = 1:2) to give 3-isopropoxy-1,1,3,3-tetraphenyldisiloxan-1-ol (4.28 g, 47%) as a colorless oil.

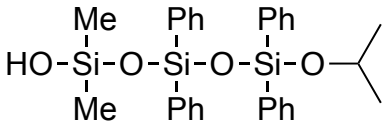
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.70-7.58 (m, 8H), 7.44-7.35 (m, 4H), 7.35-7.27 (m, 8H), 4.19 (septet, *J* = 6.1 Hz, 1H), 2.78 (br s, 1H), 1.11 ppm (d, *J* = 6.1 Hz, 6H).
^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 134.65, 134.62, 134.44, 134.38, 130.2, 130.1, 127.79, 127.75, 66.0, 25.5 ppm.
^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ –36.9, –40.1 ppm.
HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{27}H_{28}O_3Si_3Na]^+$ (M+Na): 479.15; found 479.14.

1-Isopropoxy-5,5,5-trimethyl-1,1,3,3-tetraphenyltrisiloxane (8)



$B(C_6F_5)_3$ (128 mg, 0.25 mmol) and isopropoxytrimethylsilane (870 μL, 5.0 mmol) were dissolved in toluene (20 mL). To the solution was added Ph_2SiH_2 (922 μL, 5.0 mmol). After 20 min, acetone (369 μL, 5.0 mmol) was added. After 20 min, Ph_2SiH_2 (922 μL, 5.0 mmol) was added. After 20 min, acetone (553 μL, 7.5 mmol) was added. After 30 min, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was purified by GPC (eluent: *n*-hexane) to give 1-isopropoxy-5,5,5-trimethyl-1,1,3,3-tetraphenyltrisiloxane (**8**) (1.30 g, 49%) as a colorless oil.
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.64-7.55 (m, 8H), 7.40-7.34 (m, 4H), 7.32-7.27 (m, 8H), 4.16 (septet, *J* = 6.1 Hz, 1H), 1.09 (d, *J* = 6.1 Hz, 6H), 0.01 ppm (s, 9H).
^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 135.8, 134.8, 134.7, 134.3, 129.9, 129.8, 127.6, 65.8, 25.5, 1.8 ppm.
^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ 10.1, –41.1, –47.3 ppm.
HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{30}H_{36}O_3Si_3Na]^+$ (M+Na): 551.19; found 551.17.

5-Isopropoxy-1,1-dimethyl-3,3,5,5-tetraphenyltrisiloxan-1-ol (9)



3-Isopropoxy-1,1,3,3-tetraphenyldisiloxan-1-ol (5.86 g, 12.8 mmol) was dissolved in CH_2Cl_2 (40 mL). To the solution was added Et_3N (2.7 mL, 19.3 mmol) and then $HSiMe_2Cl$ (2.1 mmol, 19.3 mmol) with stirring at room temperature. After 1 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude

product was dissolved in THF (60 mL) again. The solution was added to a mixture of 5% palladium carbon (40 mg, 0.02 mmol) and H_2O (0.72 g, 40 mmol) in THF (30 mL) with stirring at room temperature. After 30 min, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2) to give 5-isopropoxy-1,1-dimethyl-3,3,5,5-tetraphenyltrisiloxan-1-ol (**9**) (6.72 g, 99%) as a colorless solid.

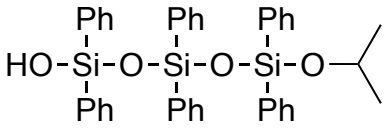
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.64-7.59 (m, 8H), 7.43-7.35 (m, 4H), 7.35-7.28 (m, 8H), 4.15 (septet, J = 6.1 Hz, 1H), 2.54 (s, 1H), 1.10 (d, J = 6.1 Hz, 6H), 0.03 ppm (s, 6H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ 135.3, 134.8, 134.24, 134.22, 130.2, 130.0, 127.8, 127.7, 66.3, 25.4, 0.2 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ –9.0, –39.8, –46.6 ppm.

HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{29}H_{34}O_4Si_3Na]^+$ (M+Na): 553.17; found 553.16.

5-Isopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol



$B(C_6F_5)_3$ (51.2 mg, 0.10 mmol) and $Ph_2Si(OH)_2$ (4.33 g, 20 mmol) was dissolved in toluene (80 mL). To the solution was added Ph_2SiH_2 (7.37 mL, 40 mmol) with stirring at 0 ˚C. After the addition, the cooling bath was removed, and the solution was allowed to reach room temperature. After 30 min, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (60 mL) and cooled down to –80 °C and kept under constant stirring. To the solution was added trichloroisocyanuric acid (1.53 g, 6.67 mmol) in portions. After the addition was completed, the cooling bath was removed, and the solution was allowed to reach room temperature for 12 h. To the reaction mixture was added triethylamine (5.6 mL, 40 mmol) and 2-propanol (3.1 mL, 40 mmol). After 3 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (60 mL) again. To the solution was added 5% palladium carbon (40 mg, 0.02 mmol) and H_2O (0.72 g, 40 mmol), and the reaction mixture was refluxed. After 12 h, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2). The crude product was purified by silica-gel column chromatography (CH_2Cl_2) to give 5-isopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol (3.77 g, 29%) as a colorless solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.59-7.55 (m, 4H), 7.55-7.49 (m, 8H), 7.38-7.32 (m, 6H),

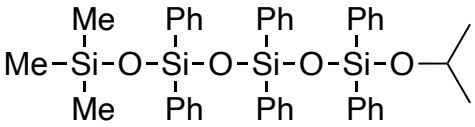
7.28-7.19 (m, 12H), 4.10 (septet, J = 6.1 Hz, 1H), 3.33 (s, 1H), 1.04 ppm (d, J = 6.1 Hz, 6H).

$^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ 134.8, 134.73, 134.72, 134.39, 134.37, 133.91, 130.1, 130.05, 130.0, 127.71, 127.66, 127.6, 66.4, 25.3 ppm.

$^{29}Si\{^{1}H\}$ NMR (119 MHz, $CDCl_3$): δ –36.8, –39.4, –45.0 ppm.

HRMS (MALDI-TOF): m/z calcd for $[C_{39}H_{36}O_4Si_3Na]^+$ (M+Na): 677.20; found 677.18.

1-Isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (11)



5-Isopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol (982 mg, 1.5 mmol) was dissolved in CH_2Cl_2 (6 mL). To the solution was added Et_3N (553 μL, 3.0 mmol) and Me_3SiCl (381 μL, 3.0 mmol) at room temperature and then the mixture was stirred overnight. The reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was passed through a short silica-gel pad (eluent: *n*-hexane/CH_2Cl_2 = 5:1) to give 1-isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (**11**) (1.09 g, >95%) as a colorless oil.

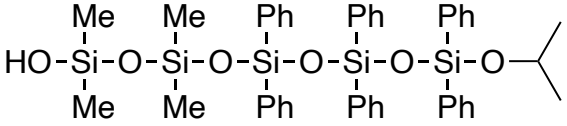
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.56-7.49 (m, 8H), 7.49-7.44 (m, 4H), 7.37-7.29 (m, 6H), 7.26-7.16 (m, 12H), 4.05 (septet, J = 6.1 Hz, 1H), 0.99 (d, J = 6.1 Hz, 6H), –0.1 ppm (s, 9H)

$^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ 135.6, 134.9, 134.7, 134.55, 134.53, 134.34, 129.9, 129.8, 129.7, 127.54, 127.51, 127.50, 65.8, 25.5, 1.7 ppm.

$^{29}Si\{^{1}H\}$ NMR (119 MHz, $CDCl_3$): δ 10.0, –41.2, –46.6, –47.2 ppm.

HRMS (MALDI-TOF): m/z calcd for $[C_{42}H_{46}O_4Si_4Na]^+$ (M+Na): 749.24; found 749.22.

9-Isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenylpentasiloxan-1-ol (12)



5-Isopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol (3.93 g, 6.0 mmol) was dissolved in CH_2Cl_2 (12 mL). To the solution was added Et_3N (1.7 mL, 12 mmol) and then Me_2HSiCl (1.3 mL 12 mmol) with stirring at room temperature. After 1 h, the reaction mixture was

passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (25 mL). The solution was added to a solution of 5% palladium carbon (10.8 mg, 0.0054 mmol) and H_2O (0.20 g, 10.8 mmol) in THF (5 mL) with stirring at room temperature. After 30 min, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2). The crude product was dissolved in CH_2Cl_2 (20 mL). To the solution was added Et_3N (1.5 mL, 10.8 mmol) and then Me_2HSiCl (1.2 mL, 10.8 mmol) with stirring at room temperature. After 1 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (25 mL) again. The solution was added to a solution of 5% palladium carbon (10.8 mg, 0.0054 mmol) and H_2O (0.19 g, 10.4 mmol) in THF (5 mL) with stirring at room temperature. After 30 min, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2) to give 9-isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenylpentasiloxan-1-ol (**12**) (4.04 g, 84%) as a colorless oil.

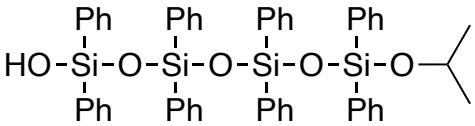
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.56-7.52 (m, 4H), 7.52-7.46 (m, 8H), 7.37-7.30 (m, 6H), 7.25-7.17 (m, 12H), 4.04 (septet, J = 6.1 Hz, 1H), 1.53 (s, 1H), 0.99 (d, J = 6.1 Hz, 6H), –0.05 (s, 6H), –0.06 ppm (s, 6H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ 135.2, 134.9, 134.7, 134.6, 134.5, 134.4, 129.94, 129.89, 129.8, 127.59, 127.56, 127.55, 65.8, 25.5, 0.9, 0.0 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ –10.9, –20.0, –41.0, –46.5, –47.5 ppm.

HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{43}H_{59}O_6Si_5Na]^+$ (M+Na): 825.24; found 825.24.

7-Isopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol



$B(C_6F_5)_3$ (102.4 mg, 0.10 mmol) was dissolved in toluene (40 mL). To the solution was added Ph_2SiH_2 (1.84 mL, 10 mmol) and then acetone (370 μL, 5.0 mmol) over 5 min at room temperature. After 10 min, acetone (737 μL, 10 mmol) was added. After 1 h, Ph_2SiH_2 (1.84 mL, 10 mmol) was added. After 1h, acetone was added (370 μL, 5.0 mmol). After 1 h, the reaction mixture was passed through a short alumina pad (eluent: CH_2Cl_2). The crude product was dissolved in THF (30 mL). To the solution was added 5% palladium carbon (20 mg, 0.04 mmol) and H_2O (0.36 g, 20 mmol), and the reaction mixture was refluxed. After 6 h, the reaction mixture was passed through a short celite

pad (eluent: CH_2Cl_2). The crude product was purified by silica-gel column chromatography (eluent: *n*-hexane/ CH_2Cl_2 = 1:2) to give 7-isopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol (1.24 g, 29%) as a colorless solid.

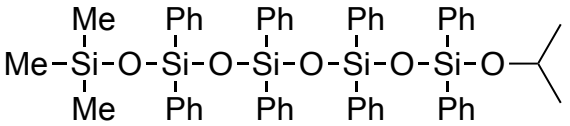
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.50-7.41 (m, 16H), 7.36-7.28 (m, 8H), 7.21-7.12 (m, 16H), 4.02 (septet, *J* = 6.1 Hz, 1H), 2.65 (br s, 1H), 0.96 ppm (d, *J* = 6.1 Hz, 6H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 134.7, 134.62, 134.61, 134.58, 134.5, 134.4, 134.2, 129.99, 129.97, 129.93, 129.90, 127.62, 127.60, 66.1, 25.3 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ –37.6, –40.4, –45.7, –46.2 ppm.

HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{51}H_{48}O_5Si_4Na]^+$ (M+Na): 875.25; found 875.24.

1-Isopropoxy-9,9,9-trimethyl-1,1,3,3,5,5,7,7-octaphenylpentasiloxane (14)



7-Isopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol (1.24 g, 1.45 mmol) was dissolved in CH_2Cl_2 (6.0 mL). To the solution was added Et_3N (553 μL, 3.0 mmol) and Me_3SiCl (381 μL, 3.0 mmol) at room temperature and then the mixture was stirred overnight. The reaction mixture was passed through a short alumina pad (eluent: CH_2Cl_2). The crude product was passed through a short silica-gel pad (eluent: *n*-hexane/CH_2Cl_2 = 5:1) to give 1-isopropoxy-9,9,9-trimethyl-1,1,3,3,5,5,7,7-octaphenylpentasiloxane (**14**) (1.34 g, >95%) as a colorless oil.

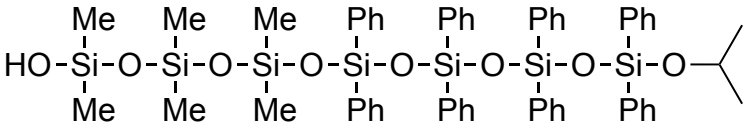
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.48-7.44 (m, 4H), 7.44-7.37 (m, 12H), 7.34-7.26 (m, 8H), 7.19-7.06 (m, 16H), 3.99 (septet, *J* = 6.1 Hz, 1H), 0.93 (d, *J* = 6.1 Hz, 6H), –0.11 ppm (s, 9H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 135.6, 134.73, 134.72, 134.69, 134.59, 134.57, 134.5, 134.3, 129.78, 129.75, 129.6, 127.53, 127.49, 127.47, 127.4, 65.8, 25.4, 1.7 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ 9.9, –41.2, –46.45, –46.53, –47.3 ppm.

HRMS (MALDI-TOF): *m*/*z* calcd for $[C_{54}H_{56}O_5Si_5Na]^+$ (M+Na): 947.30; found 947.29.

13-Isopropoxy-1,1,3,3,5,5-hexamethyl-7,7,9,9,11,11,13,13-octaphenylheptasiloxan-1-ol (15)



7-Isopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol (6.49 g, 7.61 mmol) was dissolved in CH_2Cl_2 (30 mL). To the solution was added Et_3N (1.7 mL, 7.61 mmol) and Me_2HSiCl (1.3 mL, 12 mmol) with stirring at room temperature. After 1 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (25 mL) again. The solution was added to a solution of 5% palladium carbon (15 mg, 0.0076 mmol), H_2O (0.27 g, 15.2 mmol) in THF (5 mL) with stirring at room temperature. After 30 min, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2). The crude product was dissolved in CH_2Cl_2 (20 mL). To the solution was added Et_3N (1.7 mL, 7.61 mmol) and then Me_2HSiCl (1.3 mL, 12 mmol) with stirring at room temperature. After 1 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (25 mL) again. The solution was added to a solution of 5% palladium carbon (15 mg, 0.0076 mmol mmol), H_2O (0.27 g, 15.2 mmol) in THF (5 mL) with stirring at room temperature. After 30 min, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2). The crude product was dissolved in CH_2Cl_2 (30 mL). To the solution was added Et_3N (1.7 mL, 7.61 mmol) and then Me_2HSiCl (1.3 mL, 12 mmol) with stirring at room temperature. After 1 h, the reaction mixture was passed through a short alumina pad (eluent: *n*-hexane). The crude product was dissolved in THF (25 mL) again. The solution was added to a solution of 5% palladium carbon (15 mg, 0.0076 mmol), H_2O (0.27 g, 15.2 mmol) in THF (5 mL) with stirring at room temperature. After 30 min, the reaction mixture was passed through a short celite pad (eluent: CH_2Cl_2) to give 13-isopropoxy-1,1,3,3,5,5-hexamethyl-7,7,9,9,11,11,13,13-octaphenylheptasiloxan-1-ol (**15**) (7.59 g, 93%) as a colorless oil.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.50-7.36 (m, 16H), 7.33-7.25 (m, 8H), 7.17-7.06 (m, 16H), 3.99 (sept, J = 6.1 Hz, 1H), 1.82 (s, 1H), 0.93 (d, J = 6.1 Hz, 6H), 0.04 (s, 6H), –0.07 (s, 6H), –0.13 ppm (s, 6H).

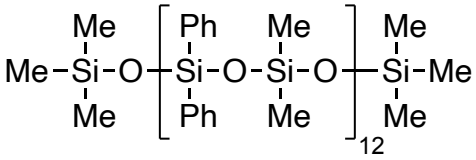
$^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 135.2, 134.70, 134.65, 134.56, 134.55, 134.46, 134.4, 129.79, 129.77, 129.76, 129.7, 127.5, 127.49, 127.47, 127.4, 65.8, 25.4, 0.91, 0.85, 0.23 ppm.

$^{29}Si\{^{1}H\}$ NMR (119 MHz, $CDCl_3$): δ -10.7, -20.5, -21.0, -41.2, -46.4, -46.49, -47.5 ppm.

HRMS (MALDI-TOF): *m/z* calcd for $[C_{57}H_{66}O_8Si_7Na]^+$ (M+Na): 1097.30; found 1097.34.

Preparation of 26-mer 1, 7, 10, and 13

1,1,1,3,3,7,7,11,11,15,15,19,19,23,23,27,27,31,31,35,35,39,39,43,43,47,47,51,51,51-Triacontamethyl-5,5,9,9,13,13,17,17,21,21,25,25,29,29,33,33,37,37,41,41,45,45,49,49-tetracosaphenylhexacosasiloxane (1)



$B(C_6F_5)_3$ (25.6 mg, 0.05 mmol) and 1-isopropoxy-3,3,7,7,11,11-heptamethyl-1,1,5,5,9,9-hexaphenylhexasiloxane (**3**) (875 mg, 1.0 mmol) was dissolved in toluene (4.0 mL). To the solution was added $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) with stirring at room temperature. After 20 min, 9-isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (**2**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, **2** (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, **2** (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, trimethylsilanol (104 μL, 1.0 mmol) was added. After 20 min, the reaction mixture was purified by silica-gel column chromatography (eluent: *n*-hexane:CH_2Cl_2 = 2:1), and then further purified by GPC (eluent: *n*-hexane) to give 1,1,1,3,3,7,7,11,11,15,15,19,19,23,23,27,27,31,31,35,35,39,39,43,43,47,47,51,51,51-triacontamethyl-5,5,9,9,13,13,17,17,21,21,25,25,29,29,33,33,37,37,41,41,45,45,49,49-tetracosaphenylhexacosasiloxane (**1**) (1.83 g, 53%) as a colorless solid.

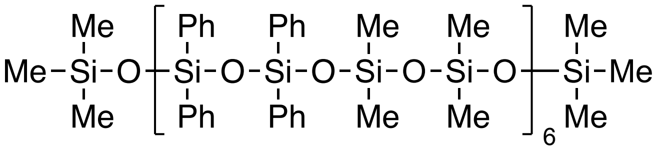
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58-7.43 (m, 48H), 7.36-7.27 (m, 24H), 7.27-7.23 (m, 8H), 7.20-7.09 (m, 40H), 0.05 (s, 9H), 0.03 (s, 6H), 0.023, (s, 6H), 0.015 (s, 6H), 0.003 (s, 9H), –0.01 (s, 6H), –0.02 (s, 6H), –0.019-–0.049 ppm (m, 42H).

$^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ 136.1, 135.9, 135.6, 135.53, 135.52, 134.3, 134.24, 134.18, 129.7, 127.60, 127.55, 127.5, 1.9, 1.7, 1.31, 1.27, 1.25 ppm.

$^{29}Si\{^{1}H\}$ NMR (119 MHz, $CDCl_3$): δ 10.0, 7.5, –19.26, –19.27, –19.28, –19.29, –19.5, –20.4, –20.8, –21.7, –47.8, –48.0, –48.06, –48.08, –48.3 ppm.

HRMS (MALDI-TOF): *m/z* calcd for $[C_{174}H_{210}O_{25}Si_{26}Na]^{+}$ (M+Na): 3449.91; found 3449.88.

1,1,1,3,3,5,5,11,11,13,13,19,19,21,21,27,27,29,29,35,35,37,37,43,43,45,45,51,51,51-Triacontamethyl-7,7,9,9,15,15,17,17,23,23,25,25,31,31,33,33,39,39,41,41,47,47,49,49-tetracosaphenylhexacosasiloxane (7)



$B(C_6F_5)_3$ (25.6 mg, 0.05 mmol) and 1-isopropoxy-5,5,5-trimethyl-1,1,3,3-tetaraphenyltrisiloxane (**8**) (529 mg, 1.0 mmol) was dissolved in toluene (4.0 mL). To the solution was added $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) with stirring at room temperature. After 20 min, 5-isopropoxy-1,1-dimethyl-3,3,5,5-tetraphenyltrisiloxane-1-ol (**9**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**9**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**9**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**9**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**9**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, acetone (73.7 μL, 1.0 mmol) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, trimethylsilanol (104 μL, 1.0 mmol) was added. After 20 min, the reaction mixture was purified by silica-gel column chromatography (eluent: *n*-hexane:CH_2Cl_2 = 2:1), and then further purified by GPC (eluent: *n*-hexane) to give 1,1,1,3,3,5,5,11,11,13,13,19,19,21,21,27,27,29,29,35,35,37,37,43,43,45,45,51,51,51-triacontamethyl-7,7,9,9,15,15,17,17,23,23,25,25,31,31,33,33,39,39,41,41,47,47,49,49-tetracosaphenylhexacosasiloxane (**7**) (1.58 g, 46%) as a colorless oil.

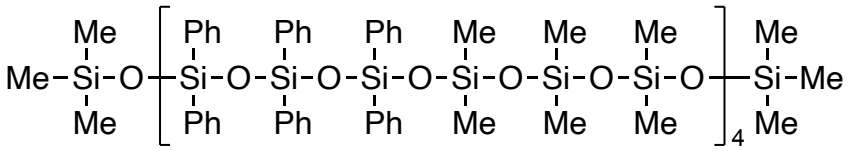
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.59-7.46 (m, 48H), 7.35-7.17 (m, 72H), 0.03 (s, 9H), –0.03 (s, 9H), –0.06 (s, 6H), –0.08 (s, 6H), –0.187 (s, 6H), –0.191 (s, 6H), –0.195 (s, 6H), –0.199 (s, 6H), –0.21 ppm (m, 36H).

$^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ 135.8, 135.6, 135.52, 135.49, 135.4, 134.4, 134.34, 134.31, 134.28, 129.8, 127.54, 127.51, 1.8, 1.7, 1.03, 1.02, 0.9 ppm.

$^{29}Si\{^{1}H\}$ NMR (119 MHz, $CDCl_3$): δ 10.1, 7.3, –20.40, –20.41, –20.43, –20.8, –21.2, –47.3, –47.68, –47.71, –47.73 ppm.

HRMS (MALDI-TOF): *m/z* calcd for $[C_{174}H_{210}O_{25}Si_{26}Na]^+$ (M+Na): 3449.91; found 3449.92.

1,1,1,3,3,5,5,7,7,15,15,17,17,19,19,27,27,29,29,31,31,39,39,41,41,43,43,51,51,51-Triacontamethyl-9,9,11,11,13,13,21,21,23,23,25,25,33,33,35,35,37,37,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (10)



$B(C_6F_5)_3$ (25.6 mg, 0.05 mmol) and 1-isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (**11**) (727 mg, 1.0 mmol) was dissolved in toluene (4.0 mL). To the solution was added $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) with stirring at room temperature. After 20 min, 9-isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenyltetrasiloxane-1-ol (**12**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**12**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**12**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, 1,1,3,3,5,5,5-heptamethyltrisiloxane-1-ol (262 mg, 1.1 mmol) was added. After 20 min, the reaction mixture was purified by silica-gel column chromatography (eluent: *n*-hexane:CH_2Cl_2 = 2:1), and then further purified by GPC (eluent: *n*-hexane) to give 1,1,1,3,3,5,5,7,7,15,15,17,17,19,19,27,27,29,29,31,31,39,39,41,41,43,43,51,51,51-triacontamethyl-9,9,11,11,13,13,21,21,23,23,25,25,33,33,35,35,37,37,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (**10**) (1.66 g, 48%) as a colorless oil.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.54-7.38 (m, 48H), 7.35-7.25 (m, 24H), 7.21-7.11 (m, 48H), 0.06, (s, 9H), –0.01 (s, 6H), –0.076 (s, 6H), –0.078 (9H), –0.11 (6H), –0.17 (s, 6H), –0.176 (s, 6H), –0.177-–0.19 (m, 24H), –0.226 (s, 6H), –0.227 (s, 6H), –0.23 ppm (s, 6H).

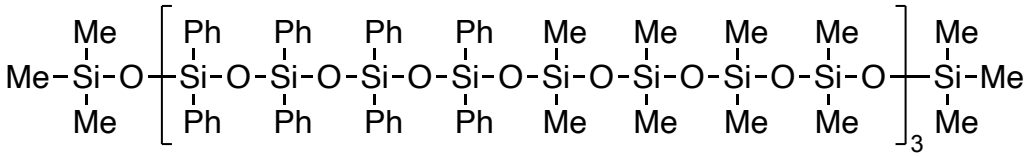
^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$): δ 135.6, 135.33, 135.29, 135.28, 135.00, 134.97, 134.95, 134.52, 134.50, 134.48, 134.38, 134.35, 134.3, 129.8, 129.7, 127.50, 127.46, 1.8, 1.7, 1.1, 1.0, 0.94, 0.93 ppm.

^{29}Si{^{1}H} NMR (119 MHz, $CDCl_3$): δ 10.0, 7.3, -20.67, –20.71, –21.4, –21.6, –21.9, –

46.5, –46.6, –47.2, –47.59, –47.60, –47.61 ppm.

HRMS (MALDI-TOF): *m/z* calcd for $[C_{174}H_{210}O_{25}Si_{26}Na]^+$ (M+Na): 3449.91; found 3449.89.

1,1,1,3,3,5,5,7,7,9,9,19,19,21,21,23,23,25,25,35,35,37,37,39,39,41,41,51,51,51-Triacontamethyl-11,11,13,13,15,15,17,17,27,27,29,29,31,31,33,33,43,43,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (13)



$B(C_6F_5)_3$ (25.6 mg, 0.05 mmol) and 1-isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (**14**) (727 mg, 1.0 mmol) was dissolved in toluene (4.0 mL). To the solution was added $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) with stirring at room temperature. After 20 min, 9-isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenyltetrasiloxane-1-ol (**15**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, (**15**) (803 mg, 1.0 mmol) in toluene (2 mL) was added. After 20 min, $^{H}MD_2M^{H}$ (328 μL, 1.0 mmol) was added. After 20 min, 1,1,3,3,5,5,7,7,7-nonamethyltetrasiloxane-1-ol (344 mg, 1.1 mmol) was added. After 20 min, the reaction mixture was purified by silica-gel column chromatography (eluent: *n*-hexane:CH_2Cl_2 = 2:1), and then further purified by GPC (eluent: *n*-hexane) to give 1,1,1,3,3,5,5,7,7,9,9,19,19,21,21,23,23,25,25,35,35,37,37,39,39,41,41,51,51,51-triacontamethyl-11,11,13,13,15,15,17,17,27,27,29,29,31,31,33,33,43,43,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (**13**) (1.99 g, 58%) as a colorless oil.

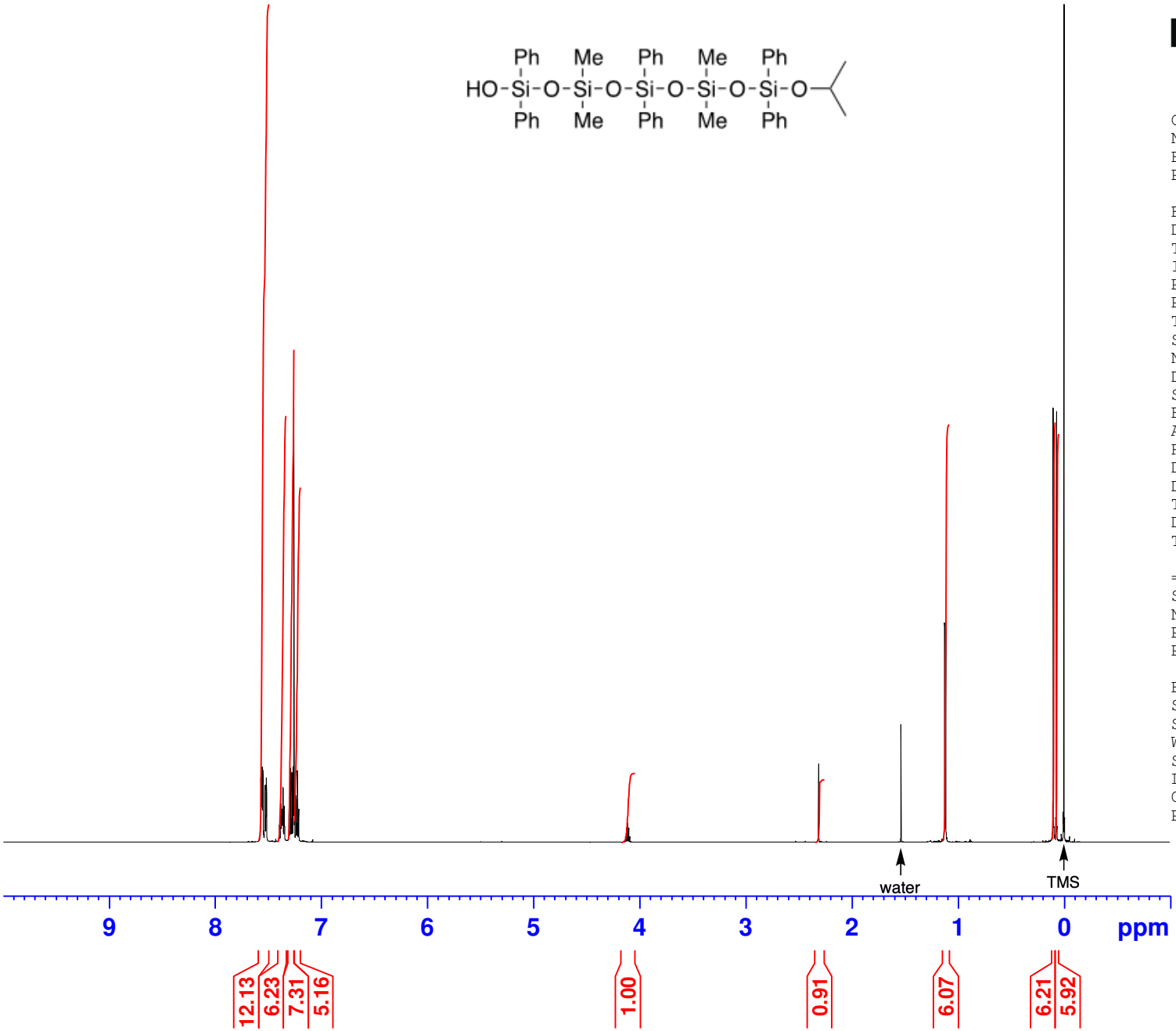
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.45-7.35 (m, 48H), 7.31-7.23 (m, 24H), 7.16-7.04 (m, 48H), 0.07 (s, 9H), 0.02 (s, 6H), 0.01 (s, 6H), –0.08 (s, 6H), –0.11 (s, 9H), –0.14 (s, 6H), –0.16 (m, 24H), –0.18 ppm (m, 24H).

$^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ 135.6, 135.28, 135.25, 134.8, 134.74, 134.72, 134.6, 134.54, 134.52, 134.52, 134.46, 134.37, 134.35, 134.3, 129.7, 129.6, 127.5, 127.43, 127.41, 1.8, 1.7, 1.1, 1.0, 0.93, 0.91 ppm.

$^{29}Si\{^{1}H\}$ NMR (119 MHz, $CDCl_3$): δ 9.9, 7.3, –20.67, –20.70, –21.4, –21.79, –21.82, –21.83, –21.84, –22.1, –46.39, –46.41, –46.44, –47.3, –47.6 ppm.

HRMS (MALDI-TOF): *m/z* calcd for $[C_{174}H_{210}O_{25}Si_{26}Na]^+$ (M+Na): 3449.91; found 3449.89.

Figure S1. 9-Isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (2)
1H NMR

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PROCNO                  1

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DE                15.65 usec
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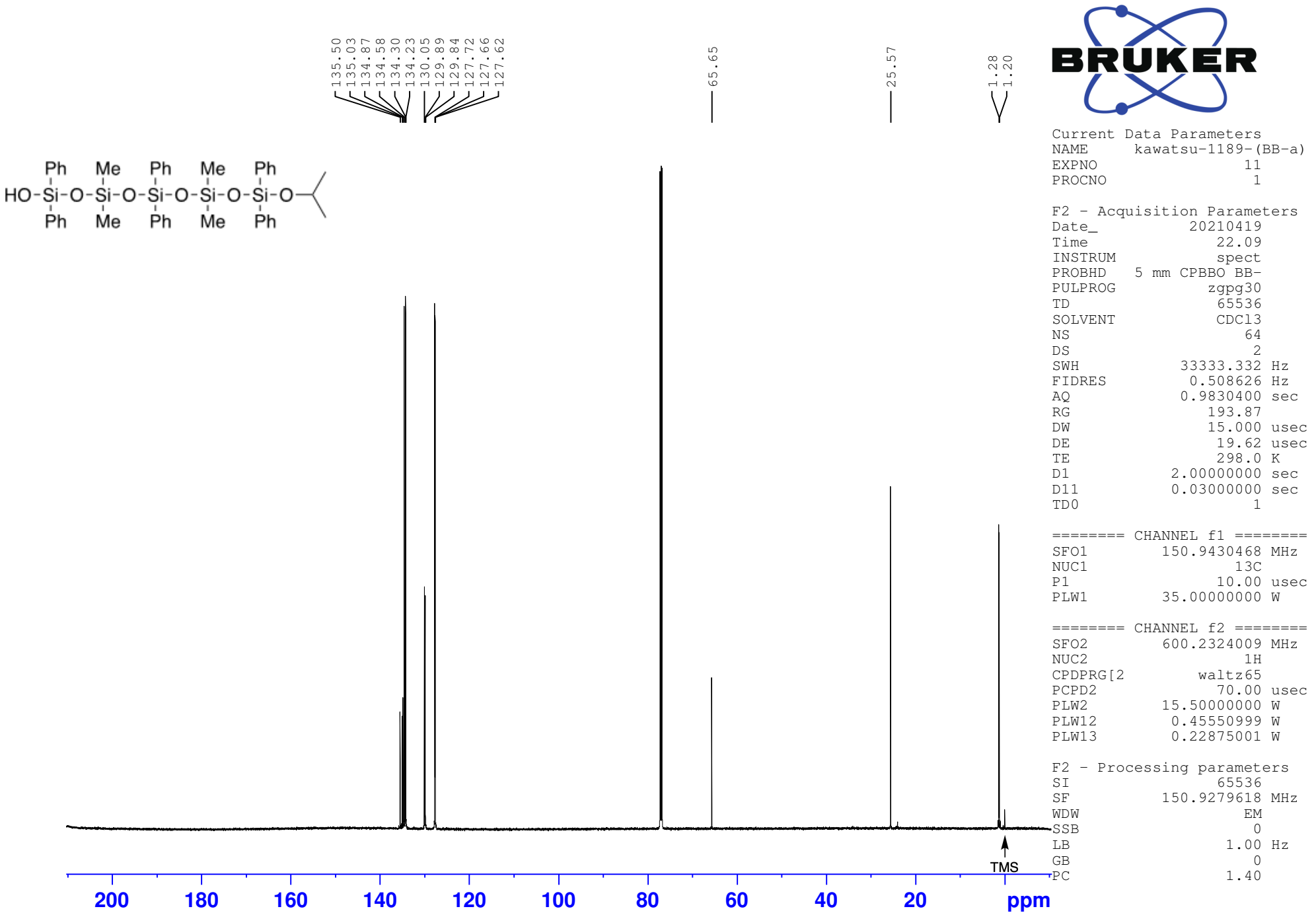
Figure S2. 9-Isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (2)
13C NMR

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RG               193.87
DW               15.000 usec
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PC                 1.40
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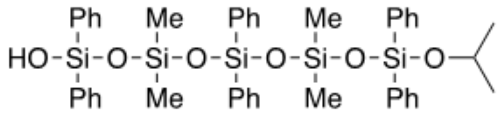
Figure S3. 9-Isopropoxy-3,3,7,7-tetramethyl-1,1,5,5,9,9-hexaphenylpentasiloxane-1-ol (2)
29Si NMR

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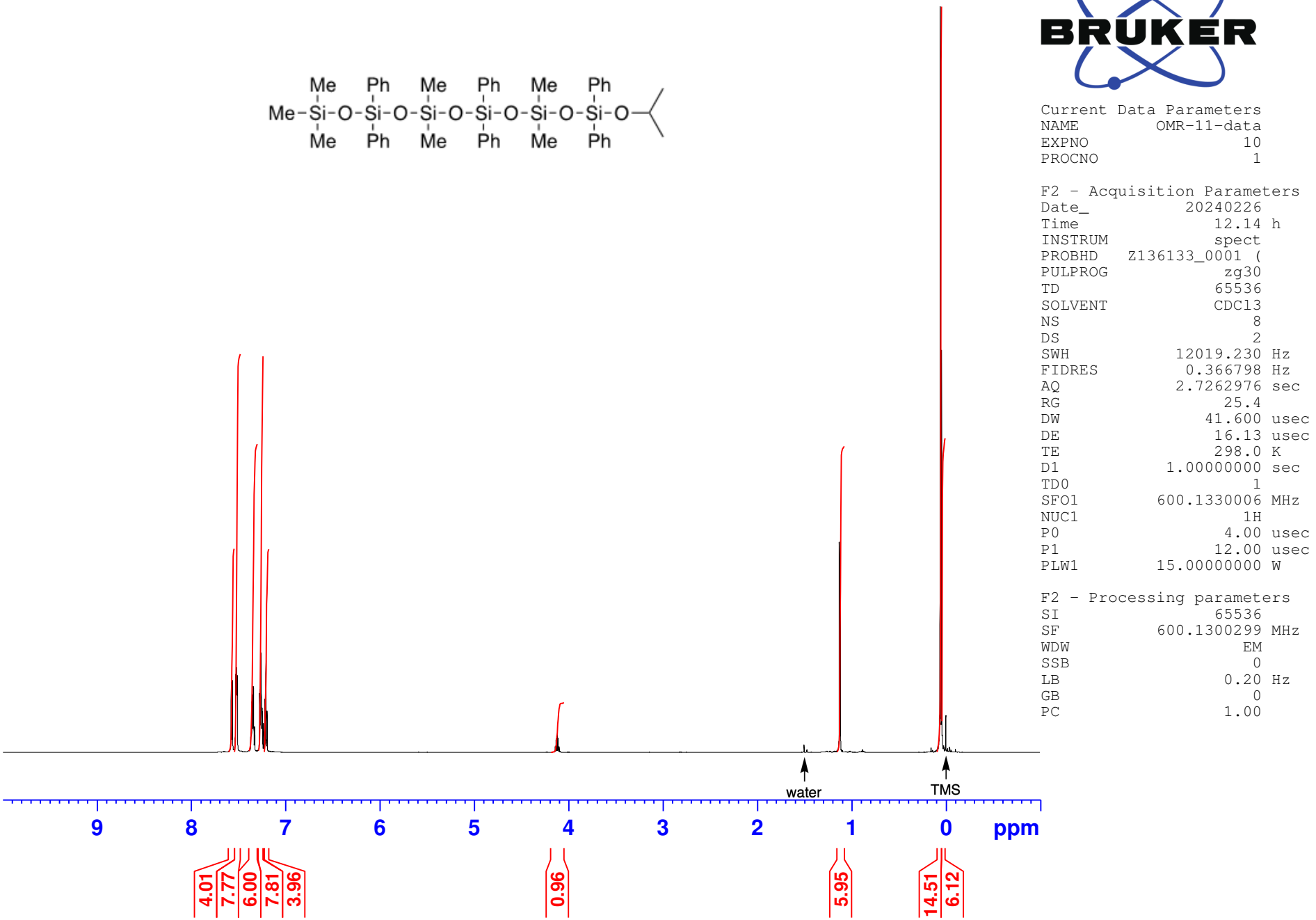
PULPROG          zgig30
TD                131072
SOLVENT            CDCl3
NS                   256
DS                     2
SWH            48076.922 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                193.87
DW                10.400 usec
DE                18.90 usec
TE                298.0 K
D1           10.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         119.2488170 MHz
NUC1                29Si
P1                 12.00 usec
PLW1         44.50000000 W

======== CHANNEL f2 ========
SFO2         600.2324009 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2              70.00 usec
PLW2         15.50000000 W
PLW12         0.45550999 W

F2 - Processing parameters
SI                131072
SF           119.2488188 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40
```

Figure S4. 5-Isopropoxy-3,3,7,7,11,11,11-heptamethyl-1,1,5,5,9,9-hexaphenylhexasiloxane (3)
1H NMR

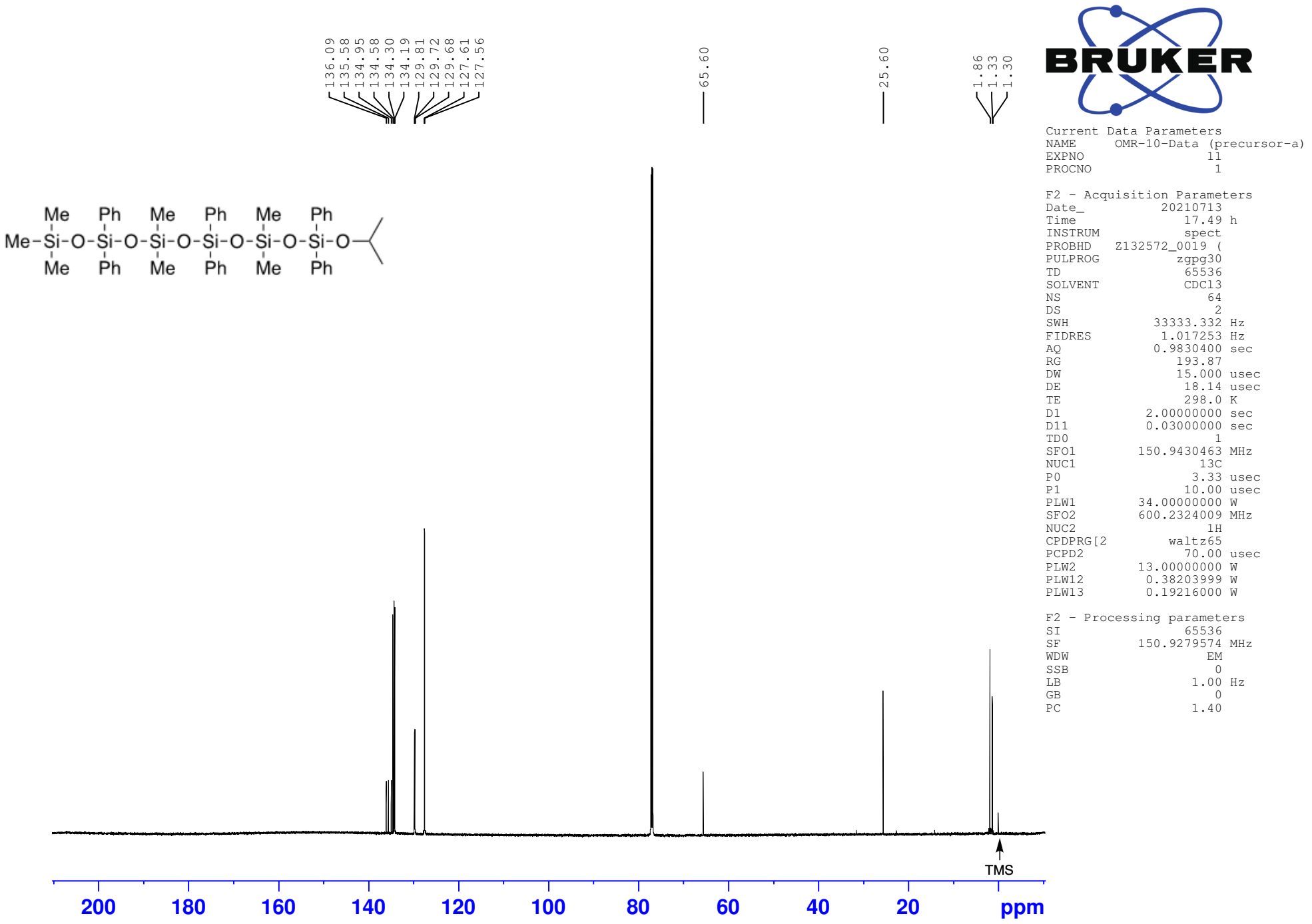



```
Current Data Parameters
NAME         OMR-11-data
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240226
Time               12.14 h
INSTRUM            spect
PROBHD   Z136133_0001 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ             2.7262976 sec
RG                  25.4
DW                41.600 usec
DE                16.13 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         600.1330006 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300299 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S5. 5-Isopropoxy-3,3,7,7,11,11,11-heptamethyl-1,1,5,5,9,9-hexaphenylhexasiloxane (3)
13C NMR

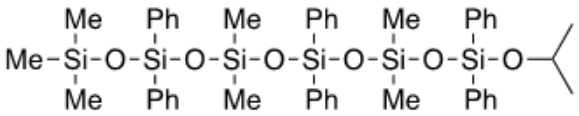



```
Current Data Parameters
NAME      OMR-10-Data (precursor-a)
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210713
Time              17.49 h
INSTRUM             spect
PROBHD    Z132572_0019 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           33333.332 Hz
FIDRES         1.017253 Hz
AQ             0.9830400 sec
RG                193.87
DW                15.000 usec
DE                18.14 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         150.9430463 MHz
NUC1                 13C
P0                3.33 usec
P1               10.00 usec
PLW1         34.00000000 W
SFO2         600.2324009 MHz
NUC2                 1H
CPDPRG[2          waltz65
PCPD2            70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W
PLW13         0.19216000 W

F2 - Processing parameters
SI                65536
SF           150.9279574 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

Figure S6. 5-Isopropoxy-3,3,7,7,11,11,11-heptamethyl-1,1,5,5,9,9-hexaphenylhexasiloxane (3)
29Si NMR

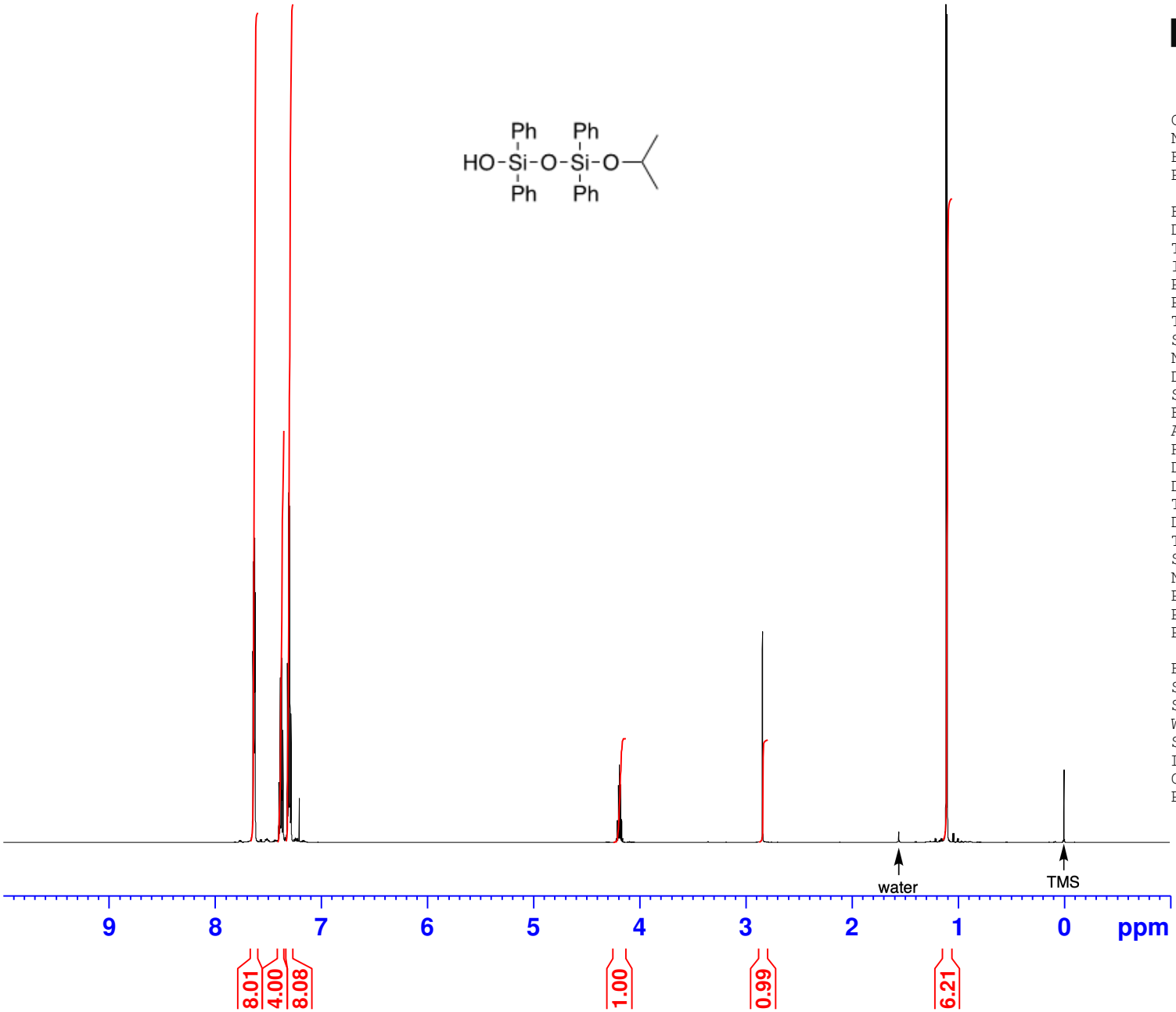



```
Current Data Parameters
NAME      OMR-10-Data (precursor-a)
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210713
Time              18.40 h
INSTRUM            spect
PROBHD    Z132572_0019 (
PULPROG           zgig30
TD                131072
SOLVENT             CDCl3
NS                    256
DS                      2
SWH           48076.922 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                193.87
DW                10.400 usec
DE                19.38 usec
TE                298.0 K
D1           10.00000000 sec
D11           0.03000000 sec
TD0                     1
SFO1         119.2488165 MHz
NUC1                29Si
P0                 4.00 usec
P1                12.00 usec
PLW1         45.00000000 W
SFO2         600.2324009 MHz
NUC2                  1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W

F2 - Processing parameters
SI                131072
SF           119.2488171 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40
```

Figure S7. 3-isopropoxyIsopropoxy-1,1,3,3-tetraphenyldisiloxan-1-ol
1H NMR

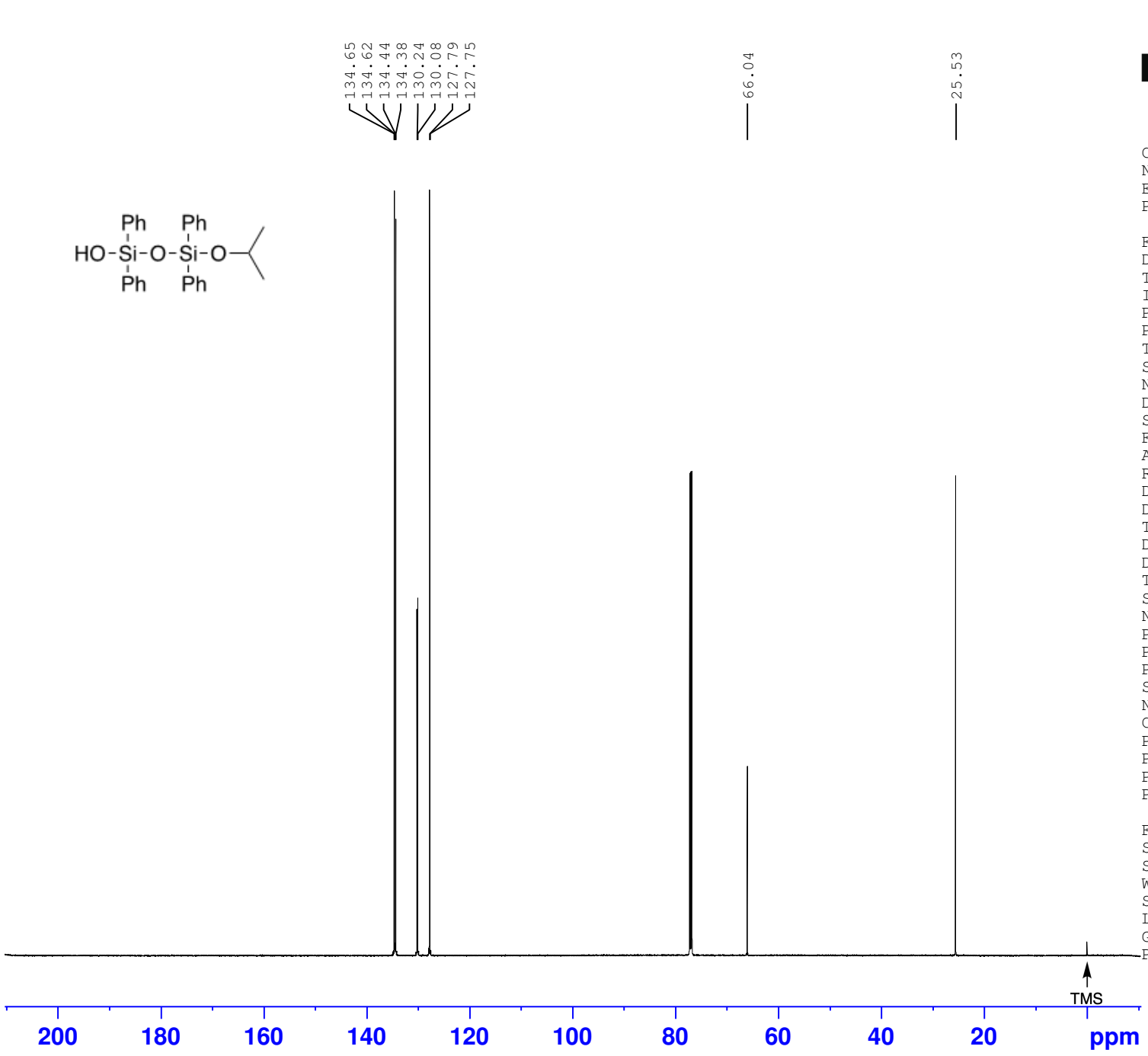



```
Current Data Parameters
NAME         OMR-21-data
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210727
Time                0.02 h
INSTRUM            spect
PROBHD   Z136133_0001 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ             2.7262976 sec
RG                  25.4
DW                41.600 usec
DE                16.13 usec
TE                297.8 K
D1             1.00000000 sec
TD0                    1
SFO1         600.1330006 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300446 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S8. 3-IsopropoxyIsopropoxy-1,1,3,3-tetraphenyldisiloxan-1-ol
13C NMR

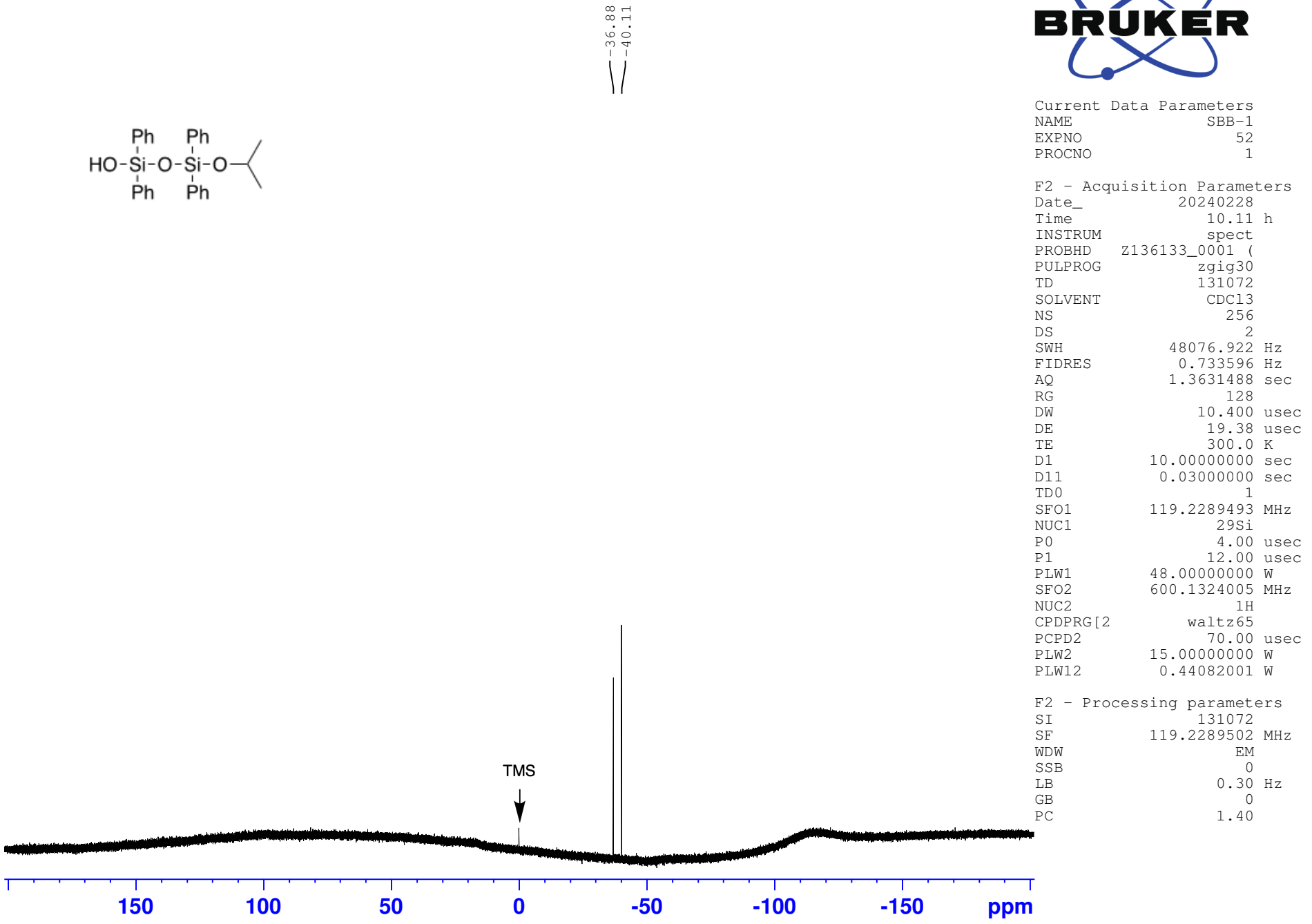



```
Current Data Parameters
NAME                  SBB-1
EXPNO                    71
PROCNO                    1

F2 - Acquisition Parameters
Date_              20240228
Time                  11.59 h
INSTRUM               spect
PROBHD    Z136133_0001 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        2
SWH              33333.332 Hz
FIDRES            1.017253 Hz
AQ               0.9830400 sec
RG                      203
DW                  15.000 usec
DE                  18.14 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           150.9178988 MHz
NUC1                    13C
P0                   3.33 usec
P1                  10.00 usec
PLW1           48.00000000 W
SFO2           600.1324005 MHz
NUC2                    1H
CPDPRG[2          waltz65
PCPD2               70.00 usec
PLW2           15.00000000 W
PLW12           0.44082001 W
PLW13           0.22172999 W

F2 - Processing parameters
SI                    65536
SF             150.9028150 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Figure S9. 3-IsopropoxyIsopropoxy-1,1,3,3-tetraphenyldisiloxan-1-ol
29Si NMR

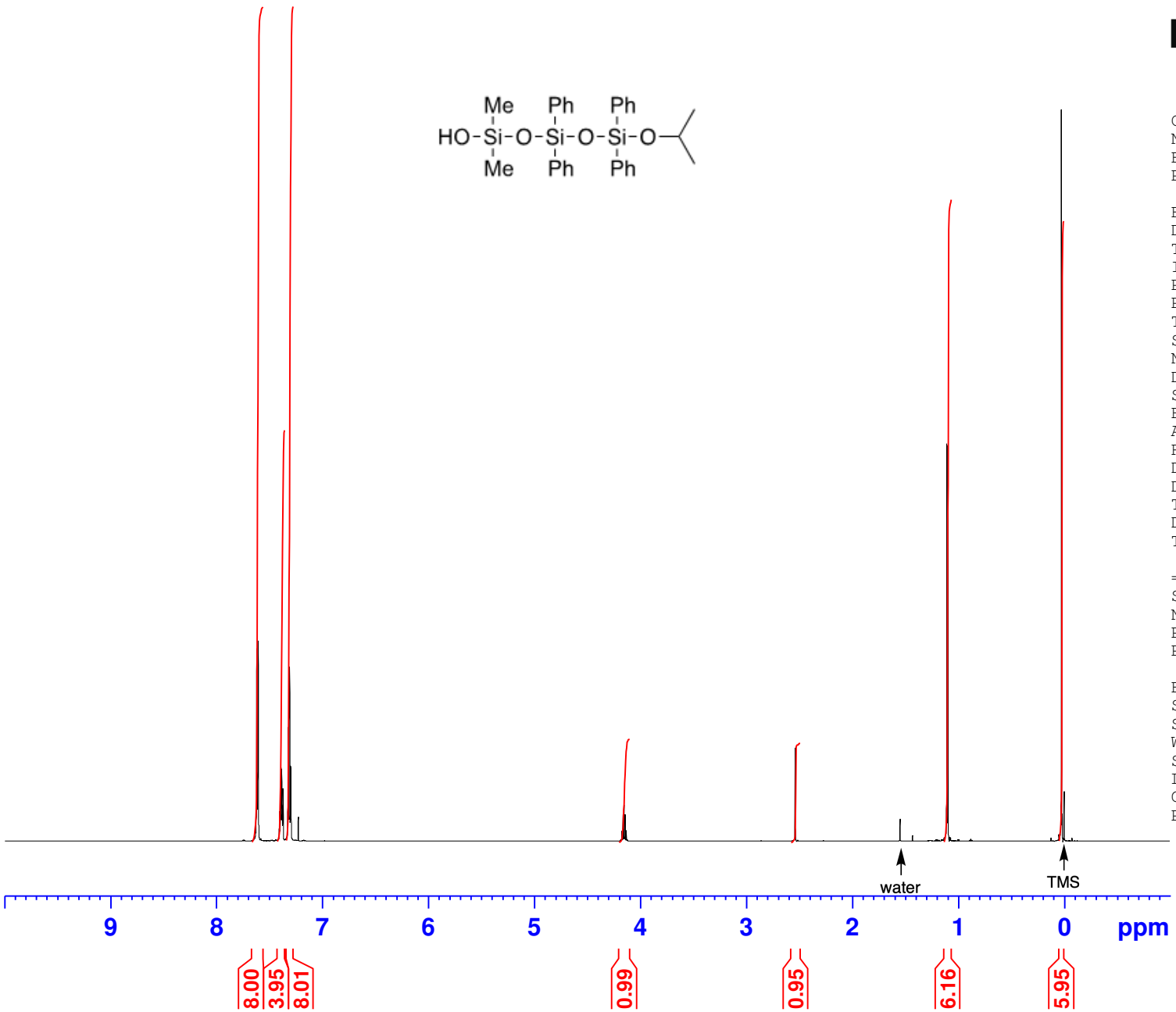



```
Current Data Parameters
NAME                 SBB-1
EXPNO                   52
PROCNO                   1

F2 - Acquisition Parameters
Date_             20240228
Time                 10.11 h
INSTRUM              spect
PROBHD   Z136133_0001 (
PULPROG             zgig30
TD                  131072
SOLVENT              CDCl3
NS                     256
DS                       2
SWH              48076.922 Hz
FIDRES            0.733596 Hz
AQ               1.3631488 sec
RG                     128
DW                  10.400 usec
DE                  19.38 usec
TE                  300.0 K
D1            10.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          119.2289493 MHz
NUC1                 29Si
P0                  4.00 usec
P1                 12.00 usec
PLW1          48.00000000 W
SFO2          600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               70.00 usec
PLW2          15.00000000 W
PLW12          0.44082001 W

F2 - Processing parameters
SI                  131072
SF            119.2289502 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.40
```

Figure S10. 5-Isopropoxy-1,1-dimethyl-3,3,5,5-tetraphenyltrisiloxan-1-ol (9)
1H NMR




```
Current Data Parameters
NAME      kawatsu-1217-(BB-b)
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20210316
Time               18.04
INSTRUM            spect
PROBHD   5 mm CPBBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                12.86
DW               41.600 usec
DE               15.65 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.2330011 MHz
NUC1                1H
P1                12.00 usec
PLW1         15.50000000 W

F2 - Processing parameters
SI                 65536
SF           600.2300339 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S11. 5-Isopropoxy-1,1-dimethyl-3,3,5,5-tetraphenyltrisiloxan-1-ol (9)
13C NMR

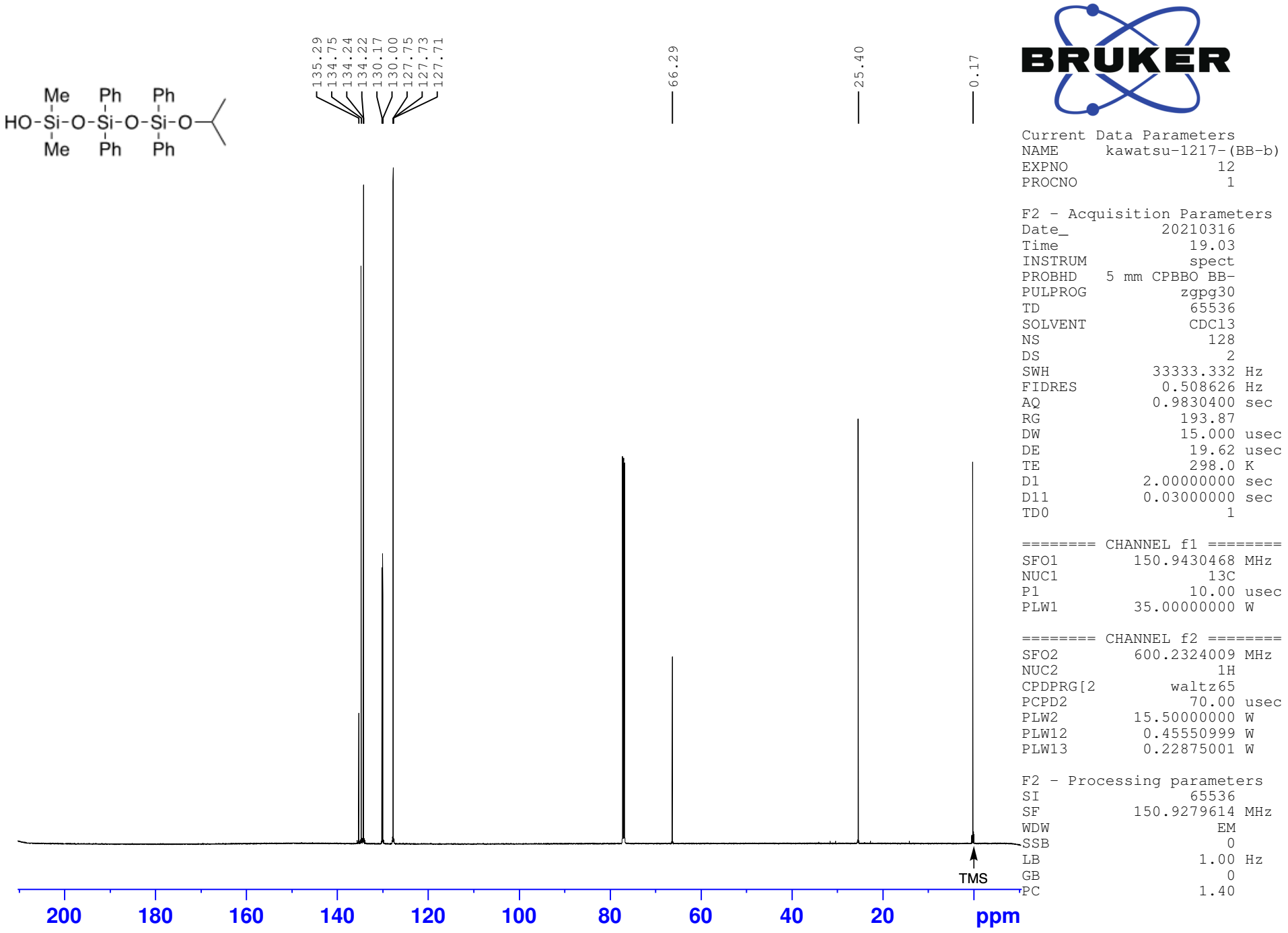
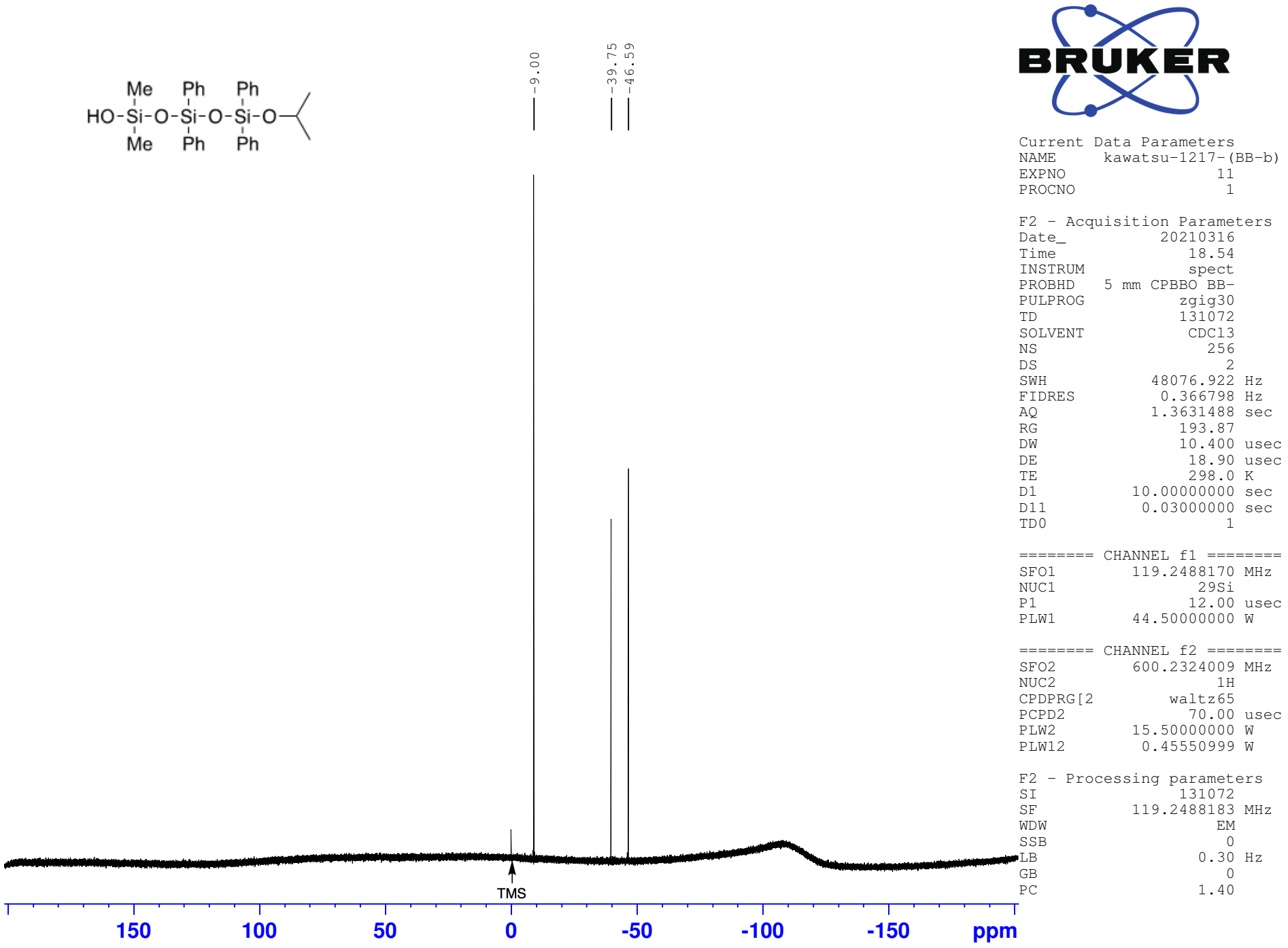


Figure S12. 5-Isopropoxy-1,1-dimethyl-3,3,5,5-tetraphenyltrisiloxan-1-ol (9)
29Si NMR




```
Current Data Parameters
NAME      kawatsu-1217-(BB-b)
EXPNO                      11
PROCNO                      1

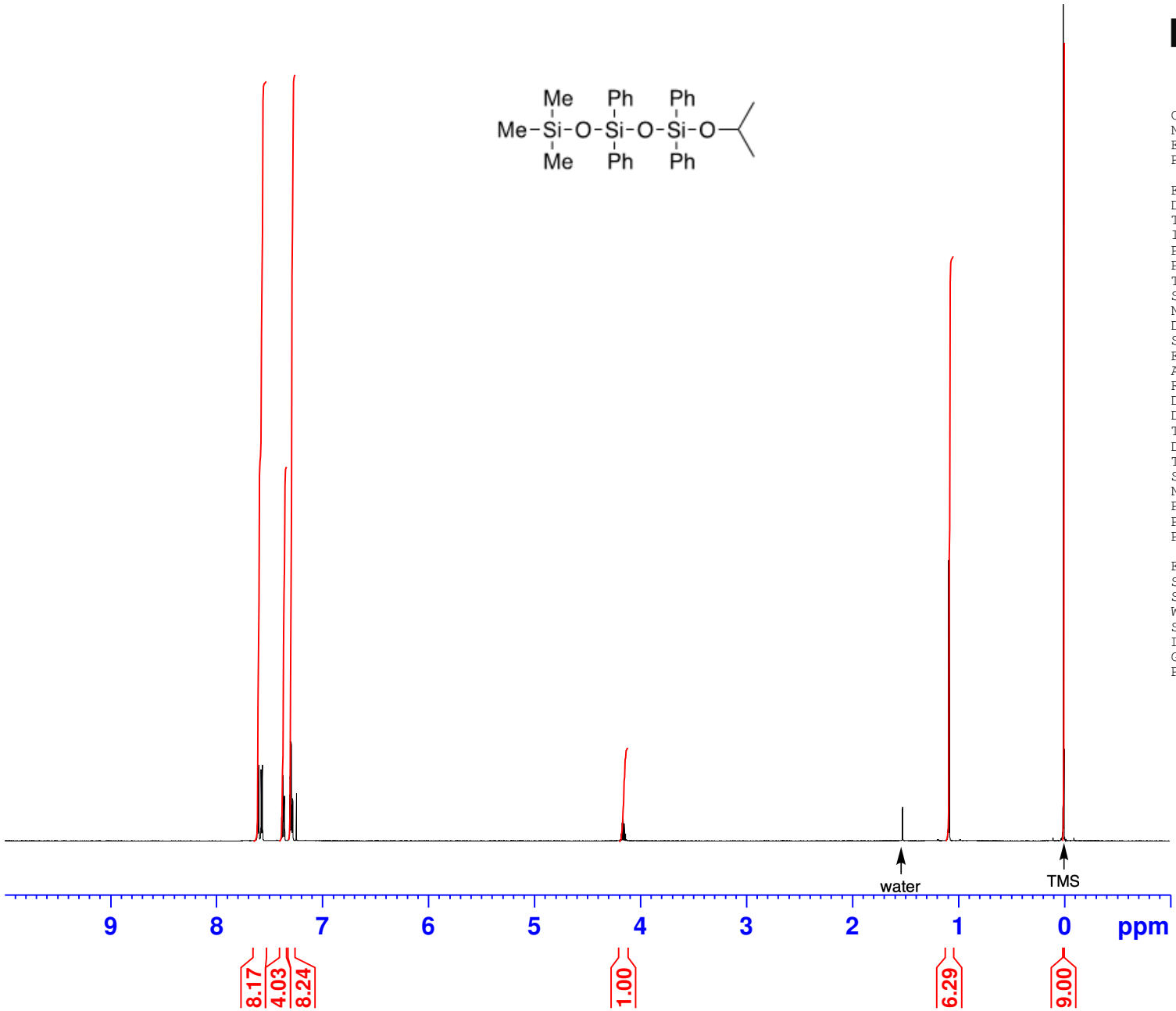
F2 - Acquisition Parameters
Date_            20210316
Time                18.54
INSTRUM             spect
PROBHD    5 mm CPBBO BB-
PULPROG           zgig30
TD                131072
SOLVENT            CDCl3
NS                   256
DS                     2
SWH            48076.922 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                193.87
DW                10.400 usec
DE                18.90 usec
TE                298.0 K
D1           10.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         119.2488170 MHz
NUC1                29Si
P1                 12.00 usec
PLW1         44.50000000 W

======== CHANNEL f2 ========
SFO2         600.2324009 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2              70.00 usec
PLW2         15.50000000 W
PLW12         0.45550999 W

F2 - Processing parameters
SI                 131072
SF           119.2488183 MHz
WDW                    EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40
```

Figure S13. 5-Isopropoxy-5,5,5-trimethyl-1,1,3,3-tetraphenyltrisiloxane (8)
1H NMR

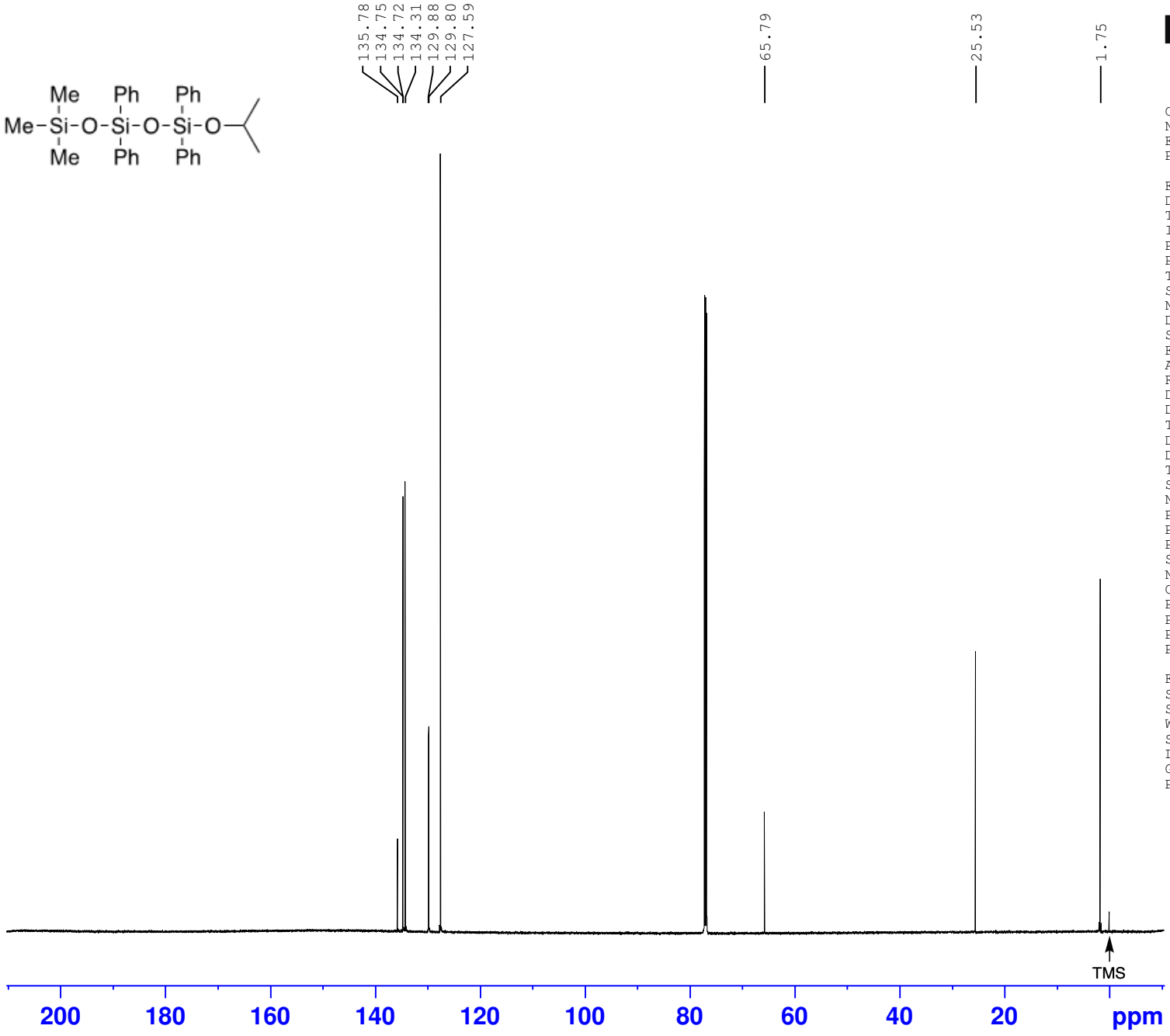



```
Current Data Parameters
NAME      OMR-20-Data (precursor-b)
EXPNO                 63
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210714
Time                9.32 h
INSTRUM            spect
PROBHD    Z132572_0019 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ              2.7262976 sec
RG                 19.72
DW                41.600 usec
DE                16.13 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         600.2330011 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.2300231 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S14.5-Isopropoxy-5,5,5-trimethyl-1,1,3,3-tetraphenyltrisiloxane (8)
13 NMR



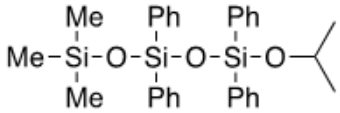
BRUKER

```
Current Data Parameters
NAME      OMR-20-Data (precursor-b)
EXPNO                 64
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210714
Time                9.36 h
INSTRUM            spect
PROBHD    Z132572_0019 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           33333.332 Hz
FIDRES         1.017253 Hz
AQ             0.9830400 sec
RG                193.87
DW                15.000 usec
DE                18.14 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         150.9430463 MHz
NUC1                 13C
P0                3.33 usec
P1               10.00 usec
PLW1         34.00000000 W
SFO2         600.2324009 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W
PLW13         0.19216000 W

F2 - Processing parameters
SI                65536
SF           150.9279578 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

Figure S15. 5-Isopropoxy-5,5,5-trimethyl-1,1,3,3-tetraphenyltrisiloxane (8)
29Si NMR

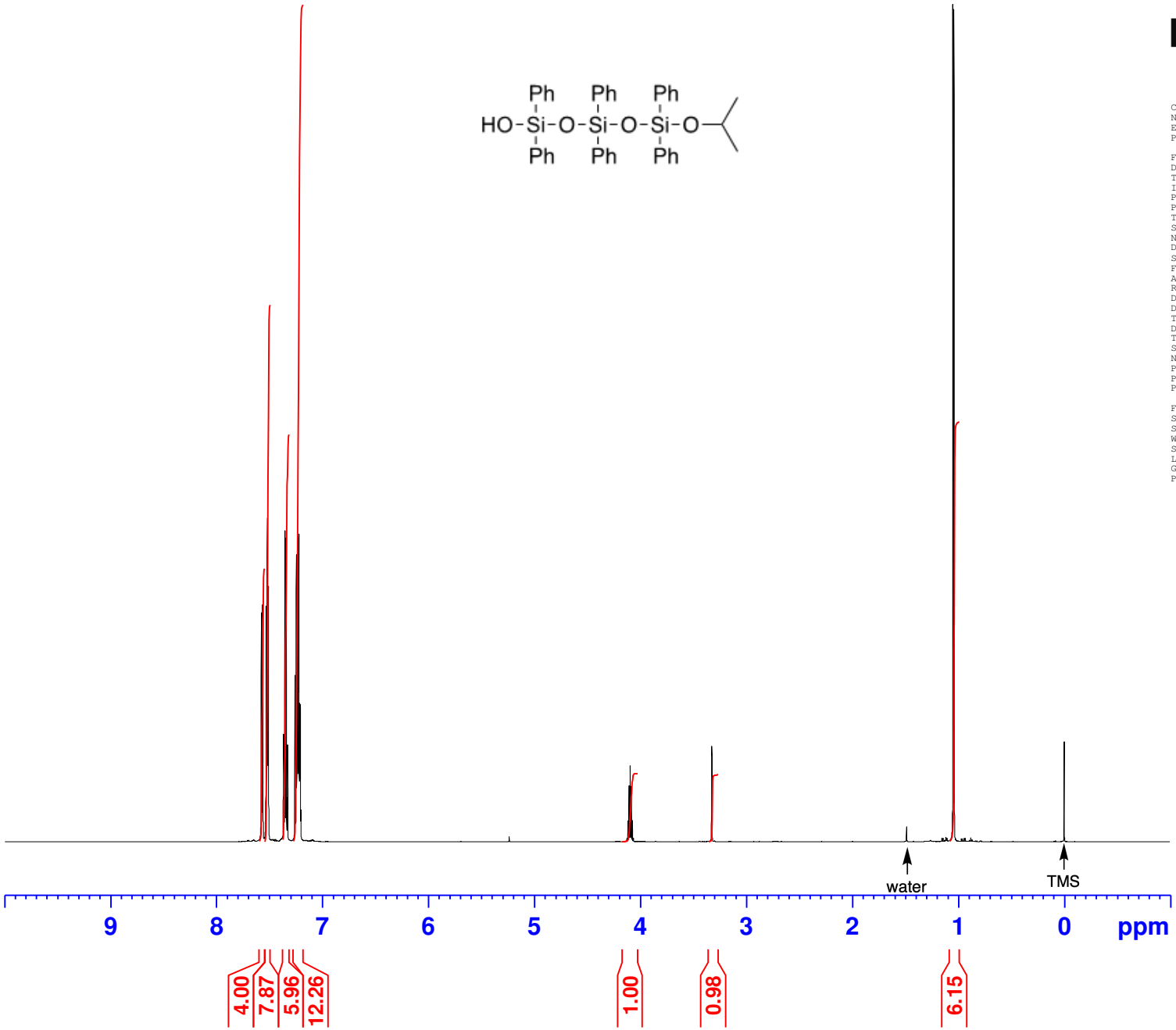



```
Current Data Parameters
NAME      OMR-20-Data (precursor-b)
EXPNO                 65
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210714
Time               10.27 h
INSTRUM             spect
PROBHD    Z132572_0019 (
PULPROG            zgig30
TD                 131072
SOLVENT             CDCl3
NS                    256
DS                      2
SWH            48076.922 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                 193.87
DW                 10.400 usec
DE                 19.38 usec
TE                 298.0 K
D1           10.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1         119.2488165 MHz
NUC1                29Si
P0                 4.00 usec
P1                12.00 usec
PLW1         45.00000000 W
SFO2         600.2324009 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2             70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W

F2 - Processing parameters
SI                 131072
SF           119.2488173 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40
```

Figure S16. 5-isopropoxyIsopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol
1H NMR

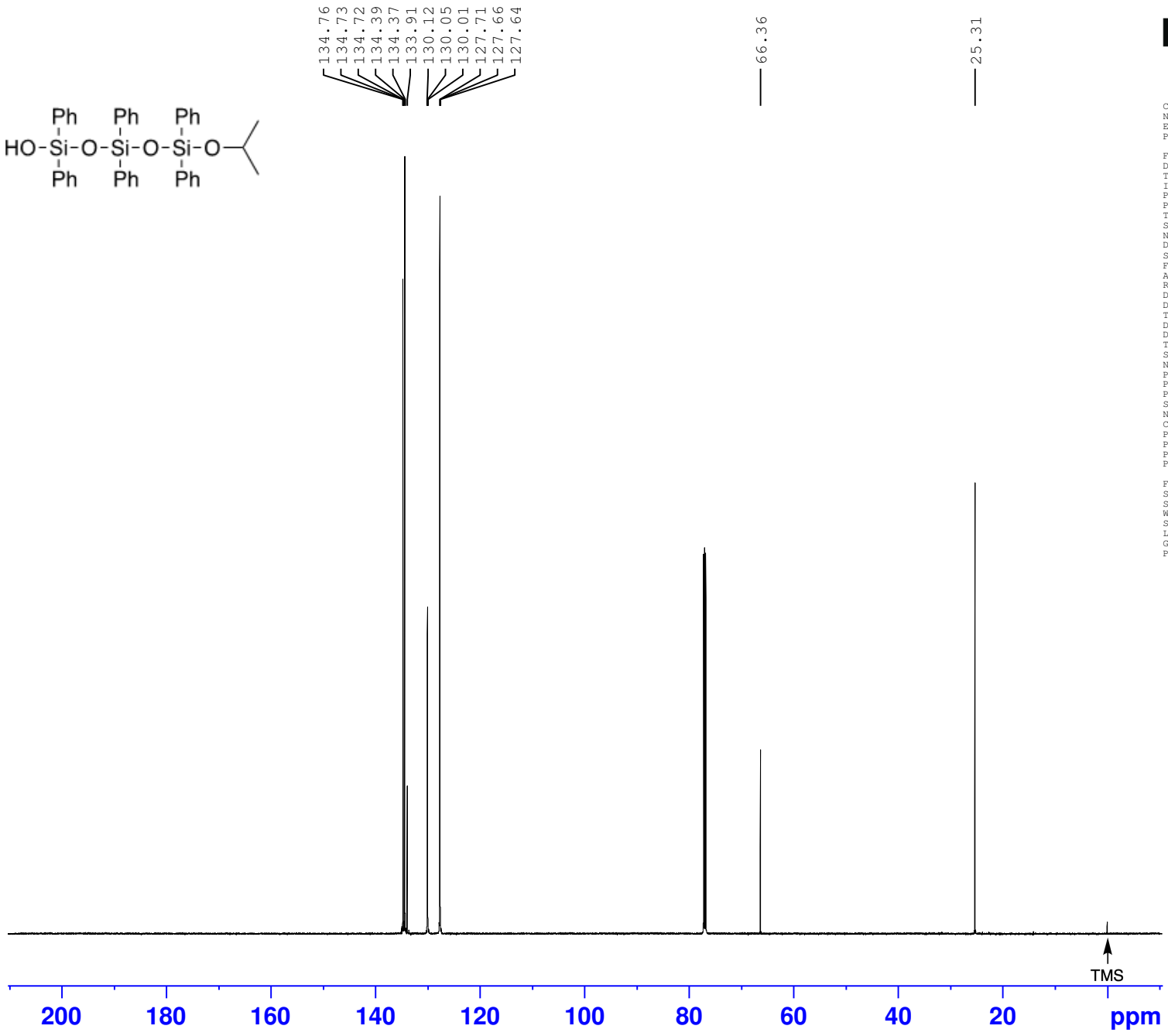



```
Current Data Parameters
NAME      kawatsu-1112-(HOPh2Si-O-Ph2Si-O-Ph2SiOiPr)
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210726
Time              22.03 h
INSTRUM             spect
PROBHD   Z136133_0001 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12019.230 Hz
FIDRES         0.366798 Hz
AQ             2.7262976 sec
RG                    16
DW               41.600 usec
DE               16.13 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         600.1330006 MHz
NUC1                  1H
P0                4.00 usec
P1               12.00 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300462 MHz
WDW                   EM
SSB                    0
LB                0.20 Hz
GB                     0
PC                1.00
```

Figure S17. 5-IsopropoxyIsopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol
13C NMR

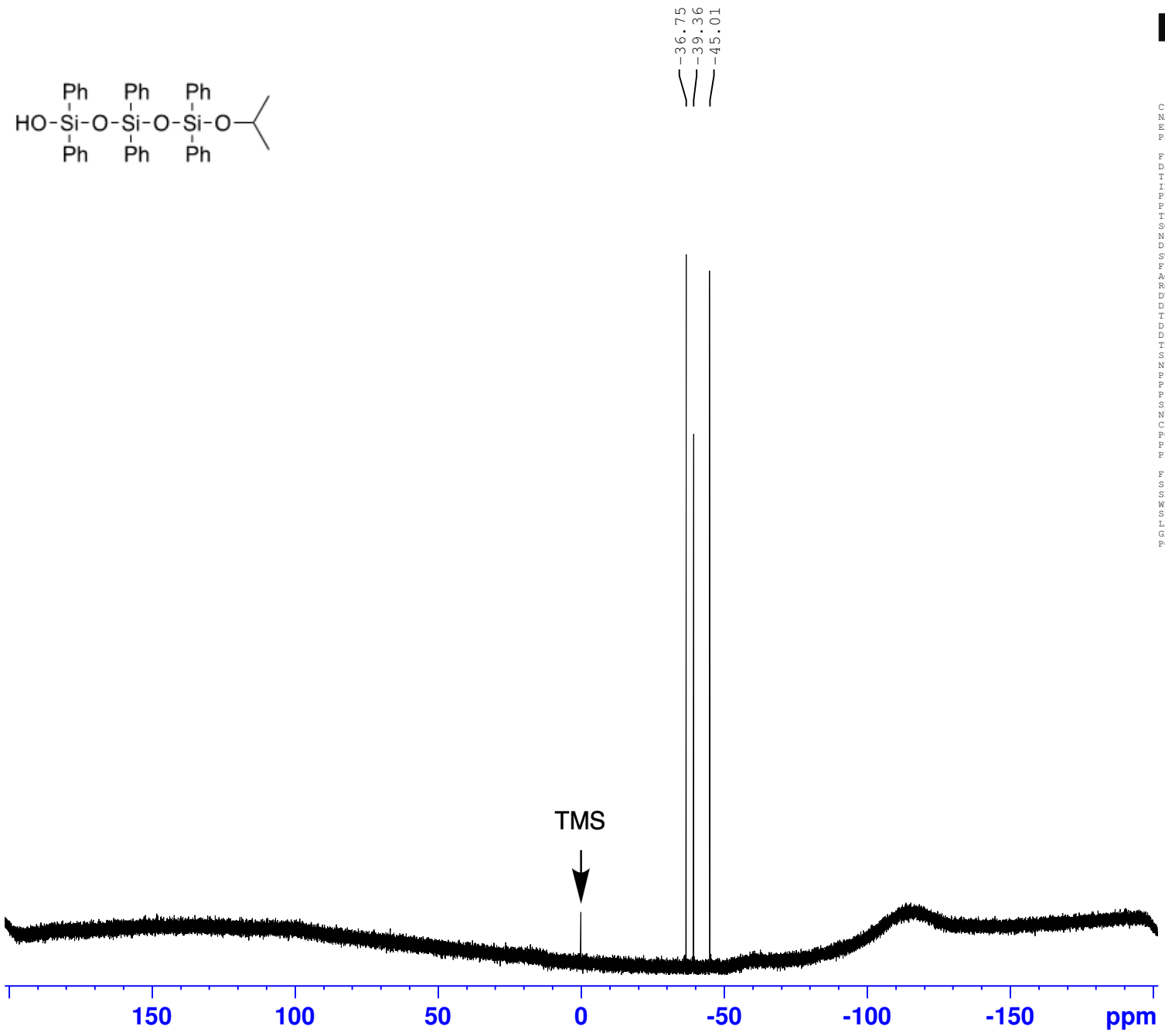


Current Data Parameters
NAME kawatsu-1112-(HOPh2Si-O-Ph2Si-O-Ph2SiOiPr)
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210726
Time 22.10 h
INSTRUM spect
PROBHD Z136133_0001 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 33333.332 Hz
FIDRES 1.017253 Hz
AQ 0.9830400 sec
RG 203
DW 15.000 usec
DE 18.14 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9178988 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 48.00000000 W
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 15.00000000 W
PLW12 0.44082001 W
PLW13 0.22172999 W

F2 - Processing parameters
SI 65536
SF 150.9028199 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S18. 5-IsopropoxyIsopropoxy-1,1,3,3,5,5-hexaphenyltrisiloxan-1-ol
29Si NMR

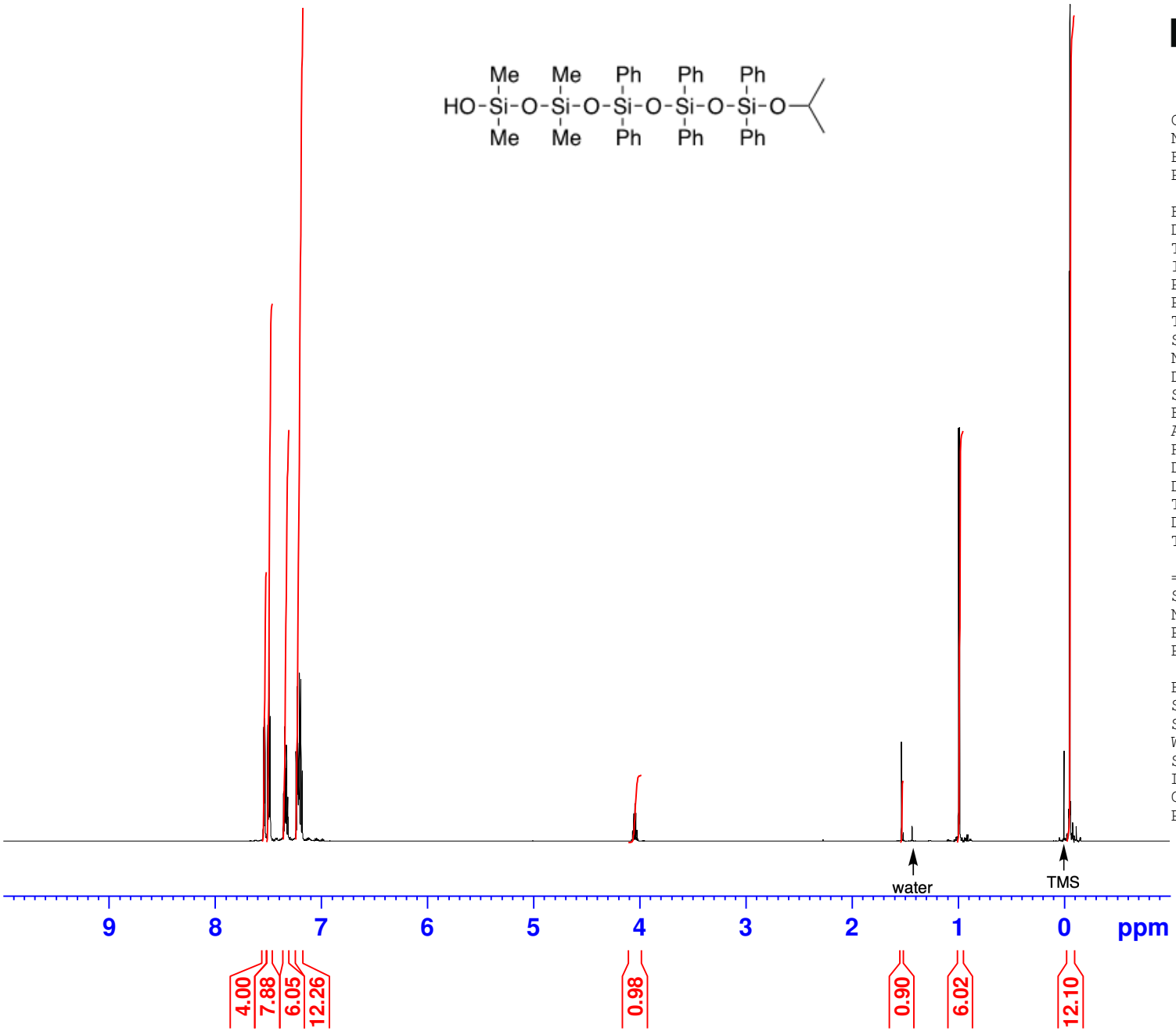



```
Current Data Parameters
NAME      kawatsu-1112-(HOPh2Si-O-Ph2Si-O-Ph2SiOiPr)
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210726
Time              23.01 h
INSTRUM             spect
PROBHD   Z136133_0001 (
PULPROG           zgig30
TD                131072
SOLVENT             CDCl3
NS                    256
DS                      2
SWH           48076.922 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                    144
DW               10.400 usec
DE               19.38 usec
TE                300.0 K
D1          10.00000000 sec
D11          0.03000000 sec
TD0                     1
SFO1        119.2289493 MHz
NUC1               29Si
P0                 4.00 usec
P1                12.00 usec
PLW1        48.00000000 W
SFO2        600.1324005 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2        15.00000000 W
PLW12        0.44082001 W

F2 - Processing parameters
SI                131072
SF          119.2289528 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.40
```

Figure S19.9-Isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenylpentasiloxan-1-ol (12)
1H NMR



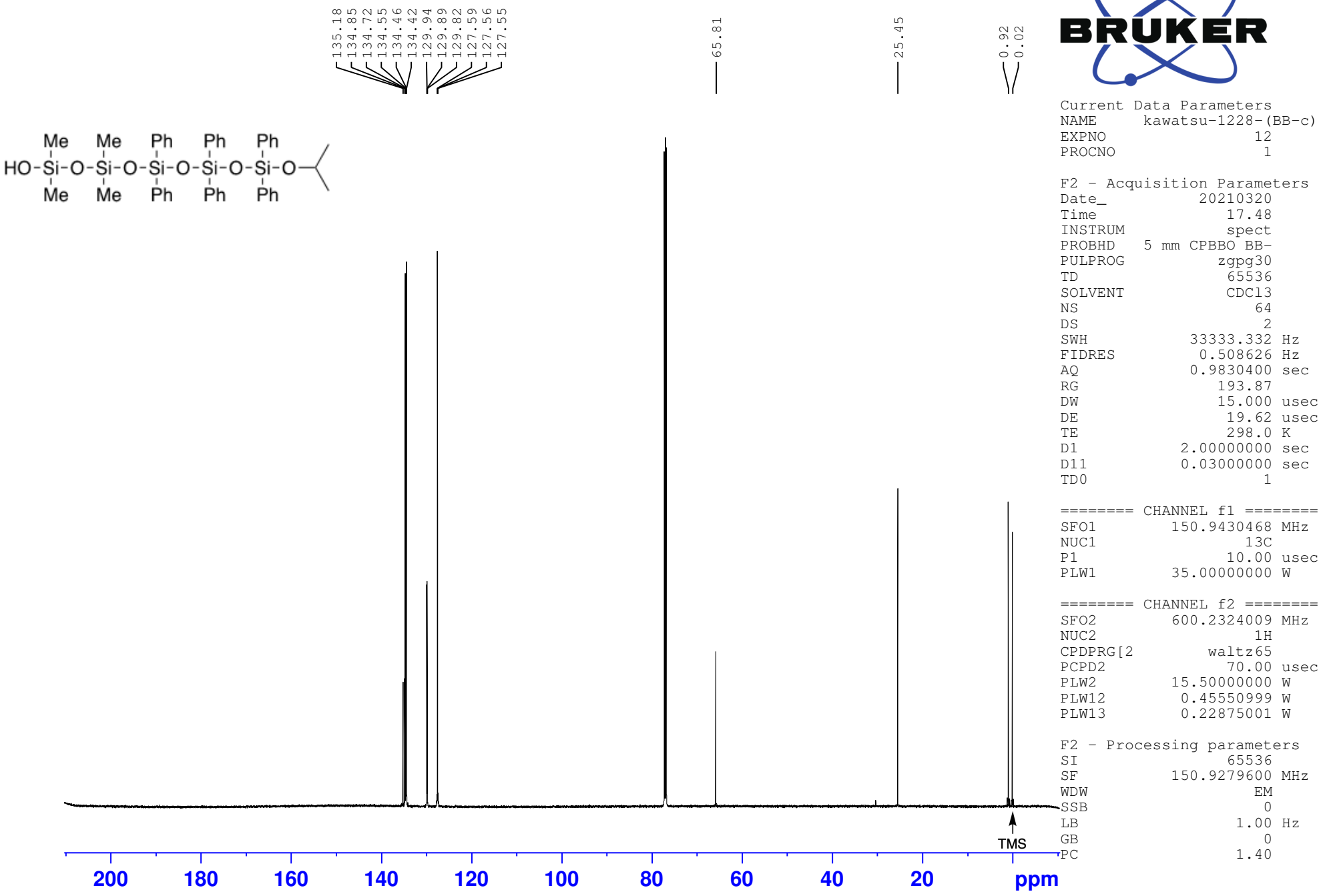
Current Data Parameters
NAME kawatsu-1228-(BB-c)
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210320
Time 16.52
INSTRUM spect
PROBHD 5 mm CPBBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 13.78
DW 41.600 usec
DE 15.65 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.2330011 MHz
NUC1 1H
P1 12.00 usec
PLW1 15.50000000 W

F2 - Processing parameters
SI 65536
SF 600.2300298 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

Figure S20. 9-Isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenylpentasiloxan-1-ol (12)
13 NMR




```
Current Data Parameters
NAME      kawatsu-1228-(BB-c)
EXPNO                  12
PROCNO                  1

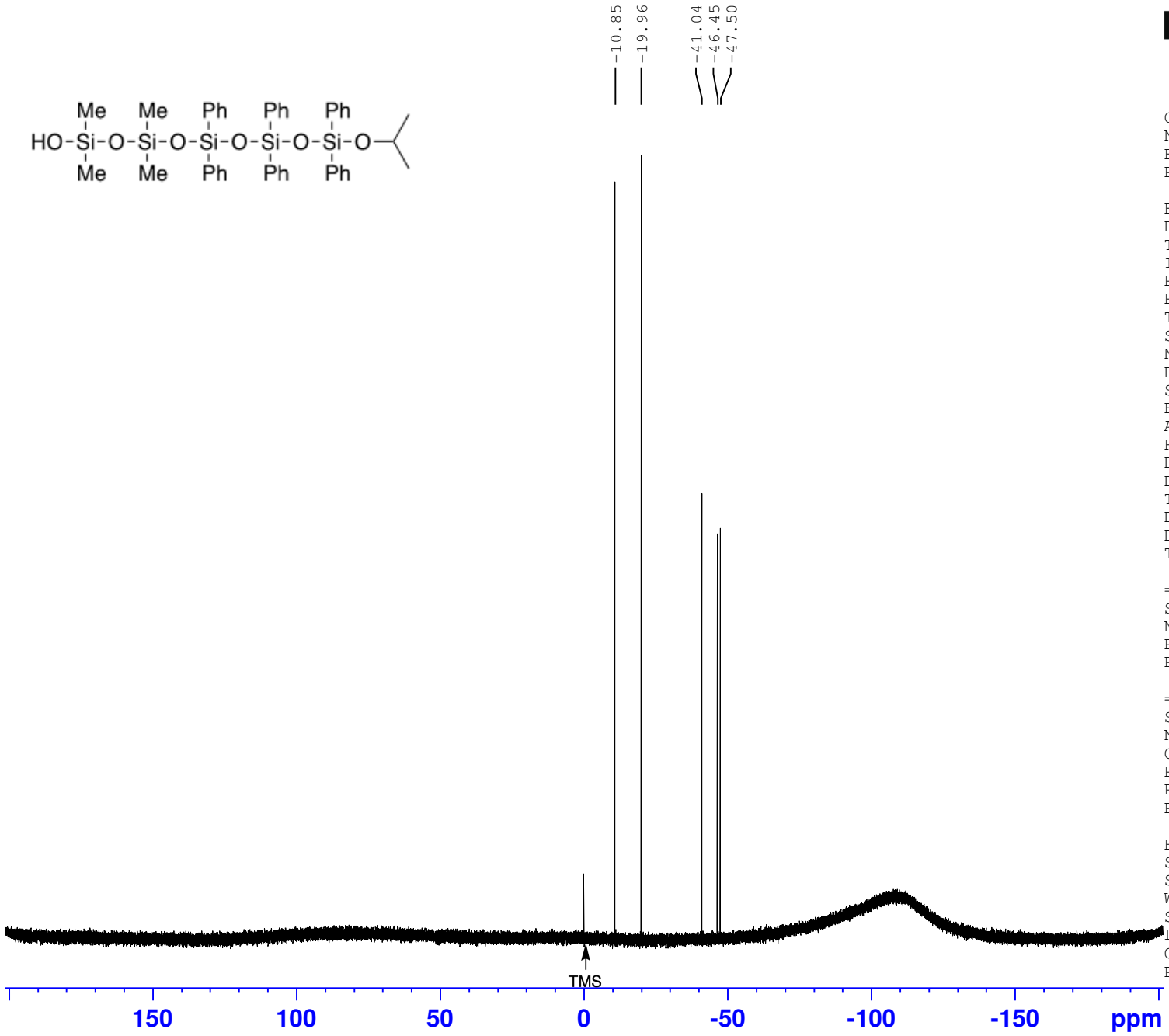
F2 - Acquisition Parameters
Date_           20210320
Time              17.48
INSTRUM           spect
PROBHD   5 mm CPBBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           33333.332 Hz
FIDRES         0.508626 Hz
AQ            0.9830400 sec
RG               193.87
DW               15.000 usec
DE               19.62 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9430468 MHz
NUC1               13C
P1               10.00 usec
PLW1        35.00000000 W

======== CHANNEL f2 ========
SFO2        600.2324009 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2            70.00 usec
PLW2        15.50000000 W
PLW12        0.45550999 W
PLW13        0.22875001 W

F2 - Processing parameters
SI                65536
SF          150.9279600 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```

Figure S21. 9-Isopropoxy-1,1,3,3-tetramethyl-5,5,7,7,9,9-hexaphenylpentasiloxan-1-ol (12)
29Si NMR



BRUKER

```
Current Data Parameters
NAME      kawatsu-1228-(BB-c)
EXPNO                  11
PROCNO                  1

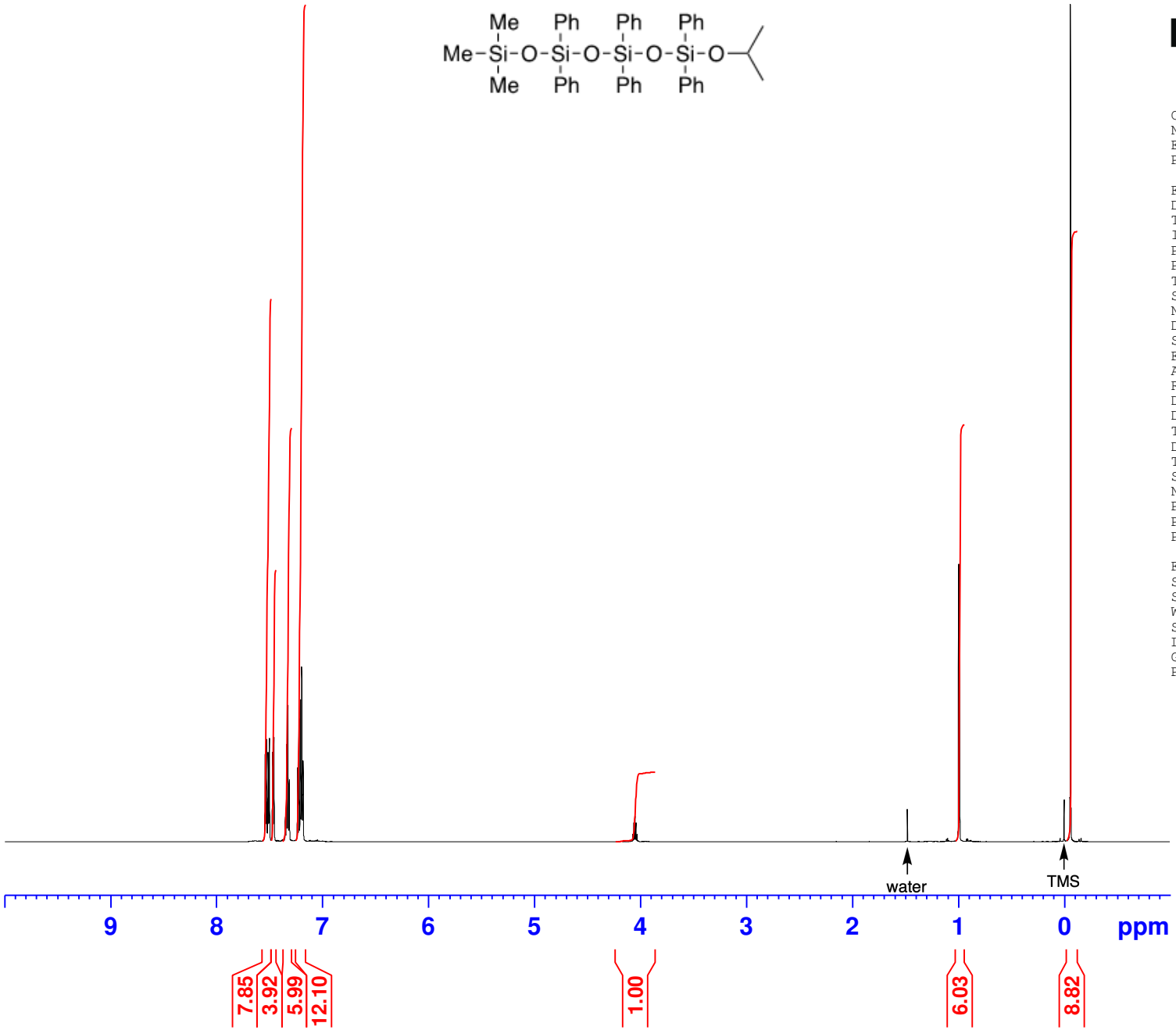
F2 - Acquisition Parameters
Date_           20210320
Time               17.43
INSTRUM            spect
PROBHD   5 mm CPBBO BB-
PULPROG          zgig30
TD               131072
SOLVENT            CDCl3
NS                   256
DS                     2
SWH           48076.922 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.87
DW               10.400 usec
DE               18.90 usec
TE               298.0 K
D1          10.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        119.2488170 MHz
NUC1               29Si
P1                12.00 usec
PLW1        44.50000000 W

======== CHANNEL f2 ========
SFO2        600.2324009 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2        15.50000000 W
PLW12        0.45550999 W

F2 - Processing parameters
SI               131072
SF          119.2488180 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.40
```

Figure S22. 1-Isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (11)
1H NMR

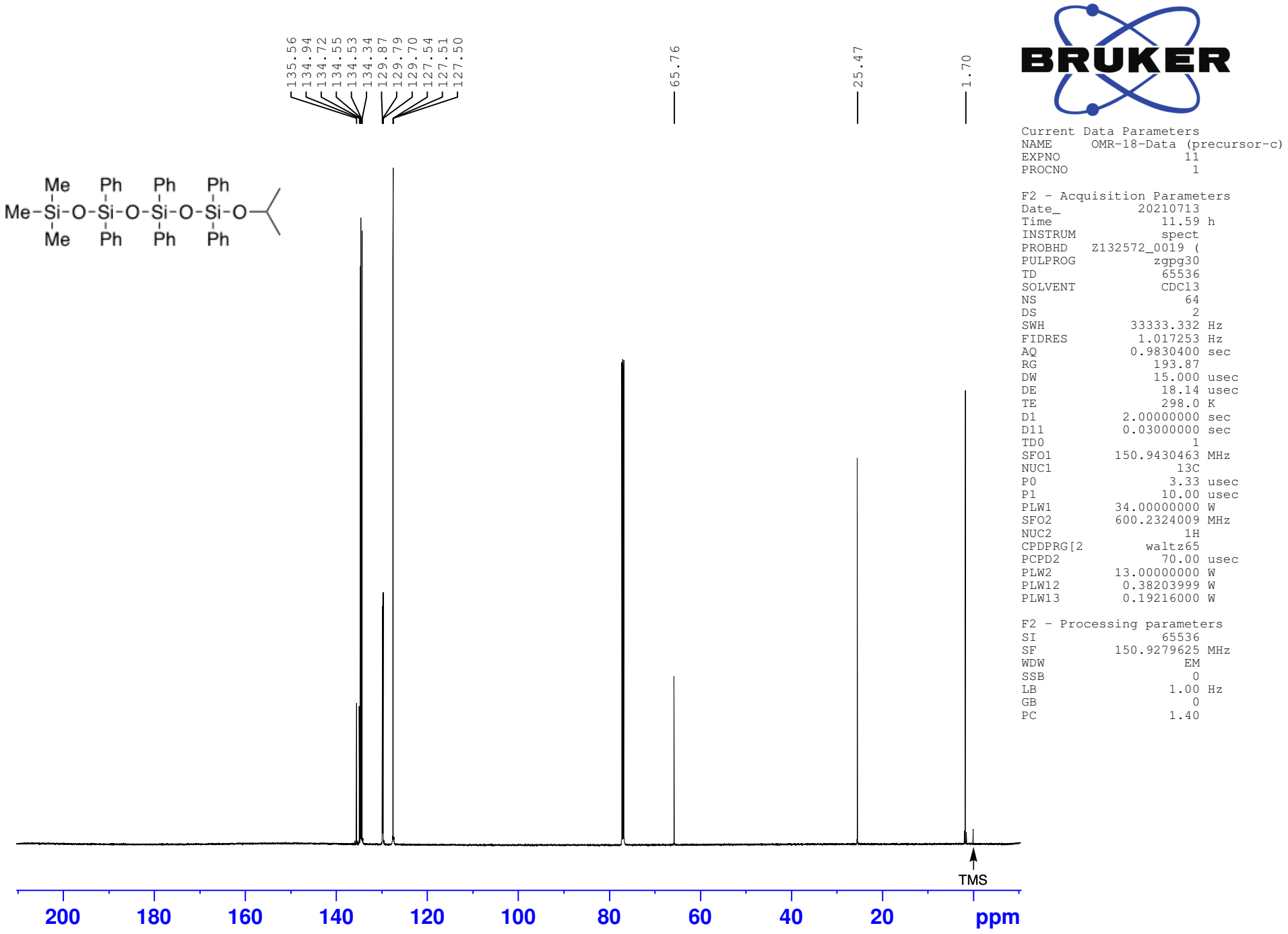



```
Current Data Parameters
NAME      OMR-18-Data (precursor-c)
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210713
Time              14.37 h
INSTRUM            spect
PROBHD    Z136133_0001 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12019.230 Hz
FIDRES         0.366798 Hz
AQ             2.7262976 sec
RG                    18
DW                41.600 usec
DE                16.13 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         600.1330006 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300370 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S23. 1-Isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (11)
13C NMR

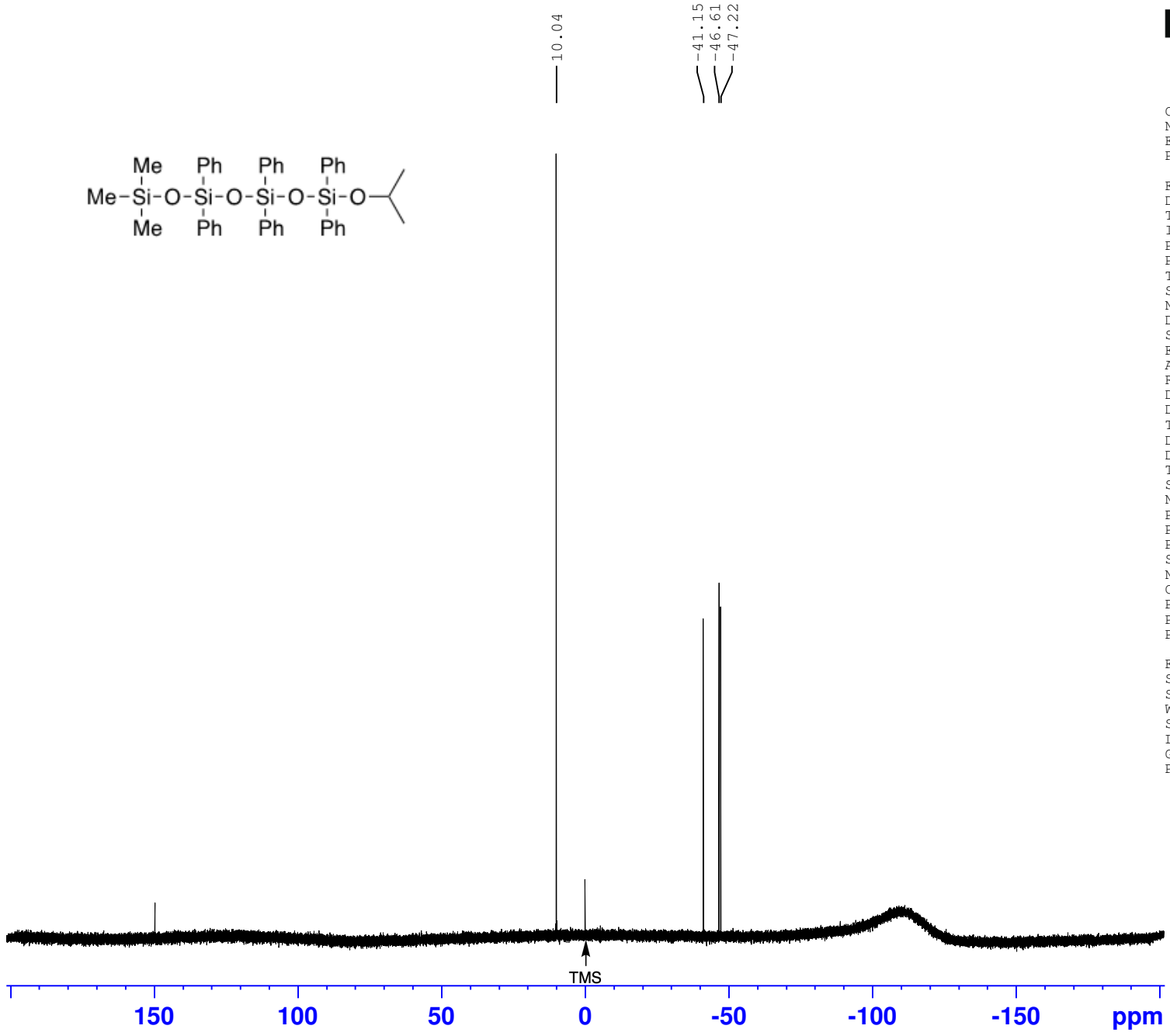



```
Current Data Parameters
NAME      OMR-18-Data (precursor-c)
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210713
Time              11.59 h
INSTRUM            spect
PROBHD    Z132572_0019 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           33333.332 Hz
FIDRES         1.017253 Hz
AQ             0.9830400 sec
RG                193.87
DW                15.000 usec
DE                 18.14 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        150.9430463 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1        34.00000000 W
SFO2        600.2324009 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2        13.00000000 W
PLW12        0.38203999 W
PLW13        0.19216000 W

F2 - Processing parameters
SI                65536
SF          150.9279625 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Figure S24. 1-Isopropoxy-7,7,7-trimethyl-1,1,3,3,5,5-hexaphenyltetrasiloxane (11)
29Si NMR

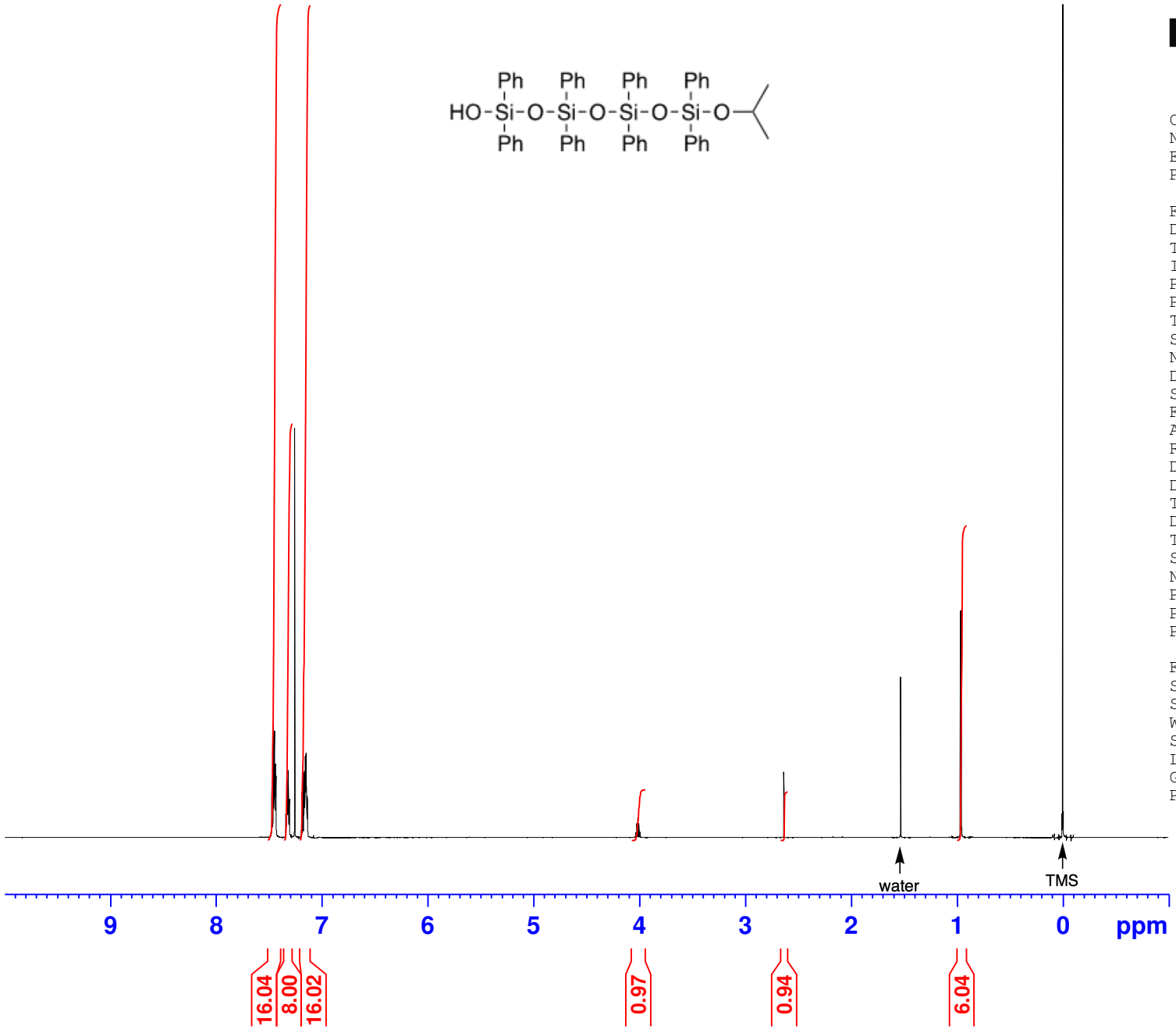



```
Current Data Parameters
NAME      OMR-18-Data (precursor-c)
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210713
Time              11.53 h
INSTRUM            spect
PROBHD    Z132572_0019 (
PULPROG           zgig30
TD                131072
SOLVENT             CDCl3
NS                    256
DS                      2
SWH           48076.922 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                193.87
DW                10.400 usec
DE                19.38 usec
TE                298.0 K
D1           10.00000000 sec
D11           0.03000000 sec
TD0                     1
SFO1         119.2488165 MHz
NUC1                29Si
P0                 4.00 usec
P1                12.00 usec
PLW1         45.00000000 W
SFO2         600.2324009 MHz
NUC2                  1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W

F2 - Processing parameters
SI                131072
SF           119.2488192 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40
```

Figure S25. 7-IsopropoxyIsopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol 1H
1H NMR

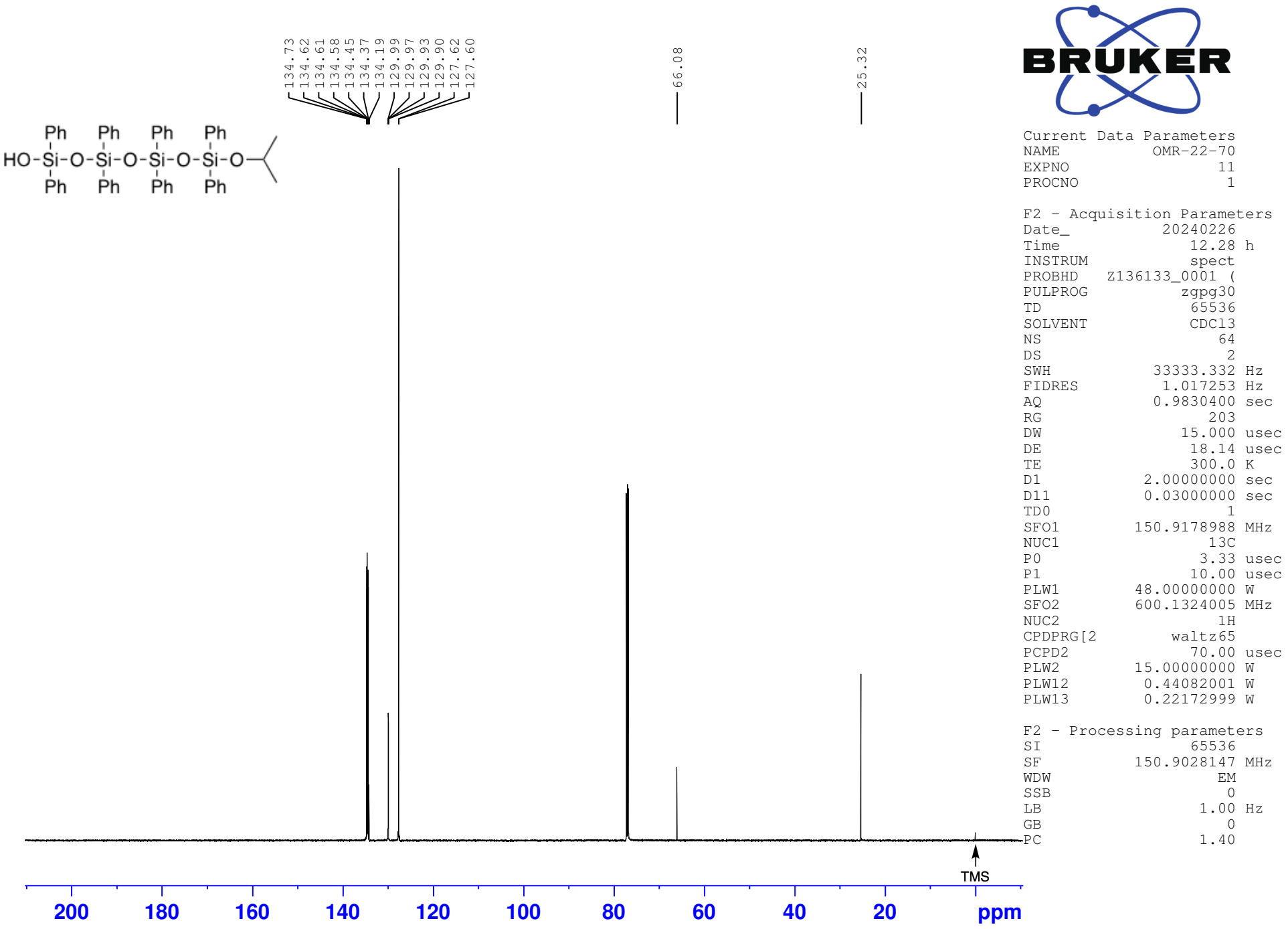



```
Current Data Parameters
NAME                  OMR-22
EXPNO                    700
PROCNO                     1

F2 - Acquisition Parameters
Date_               20211004
Time                   15.45 h
INSTRUM                spect
PROBHD    Z132572_0019 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH               12019.230 Hz
FIDRES             0.366798 Hz
AQ                2.7262976 sec
RG                    27.99
DW                   41.600 usec
DE                   16.13 usec
TE                   298.0 K
D1               1.00000000 sec
TD0                        1
SFO1            600.2330011 MHz
NUC1                      1H
P0                    4.00 usec
P1                   12.00 usec
PLW1            13.00000000 W

F2 - Processing parameters
SI                    65536
SF              600.2300161 MHz
WDW                      EM
SSB                       0
LB                    0.20 Hz
GB                        0
PC                    1.00
```

Figure S26. 7-IsopropoxyIsopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol
13C NMR

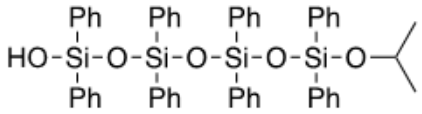



```
Current Data Parameters
NAME          OMR-22-70
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20240226
Time              12.28 h
INSTRUM           spect
PROBHD   Z136133_0001 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           33333.332 Hz
FIDRES         1.017253 Hz
AQ            0.9830400 sec
RG                  203
DW               15.000 usec
DE               18.14 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        150.9178988 MHz
NUC1                13C
P0               3.33 usec
P1              10.00 usec
PLW1        48.00000000 W
SFO2        600.1324005 MHz
NUC2                1H
CPDPRG[2        waltz65
PCPD2            70.00 usec
PLW2        15.00000000 W
PLW12        0.44082001 W
PLW13        0.22172999 W

F2 - Processing parameters
SI                65536
SF          150.9028147 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Figure S27. 7-IsopropoxyIsopropoxy-1,1,3,3,5,5,7,7-octaphenyltetrasiloxan-1-ol
29Si NMR

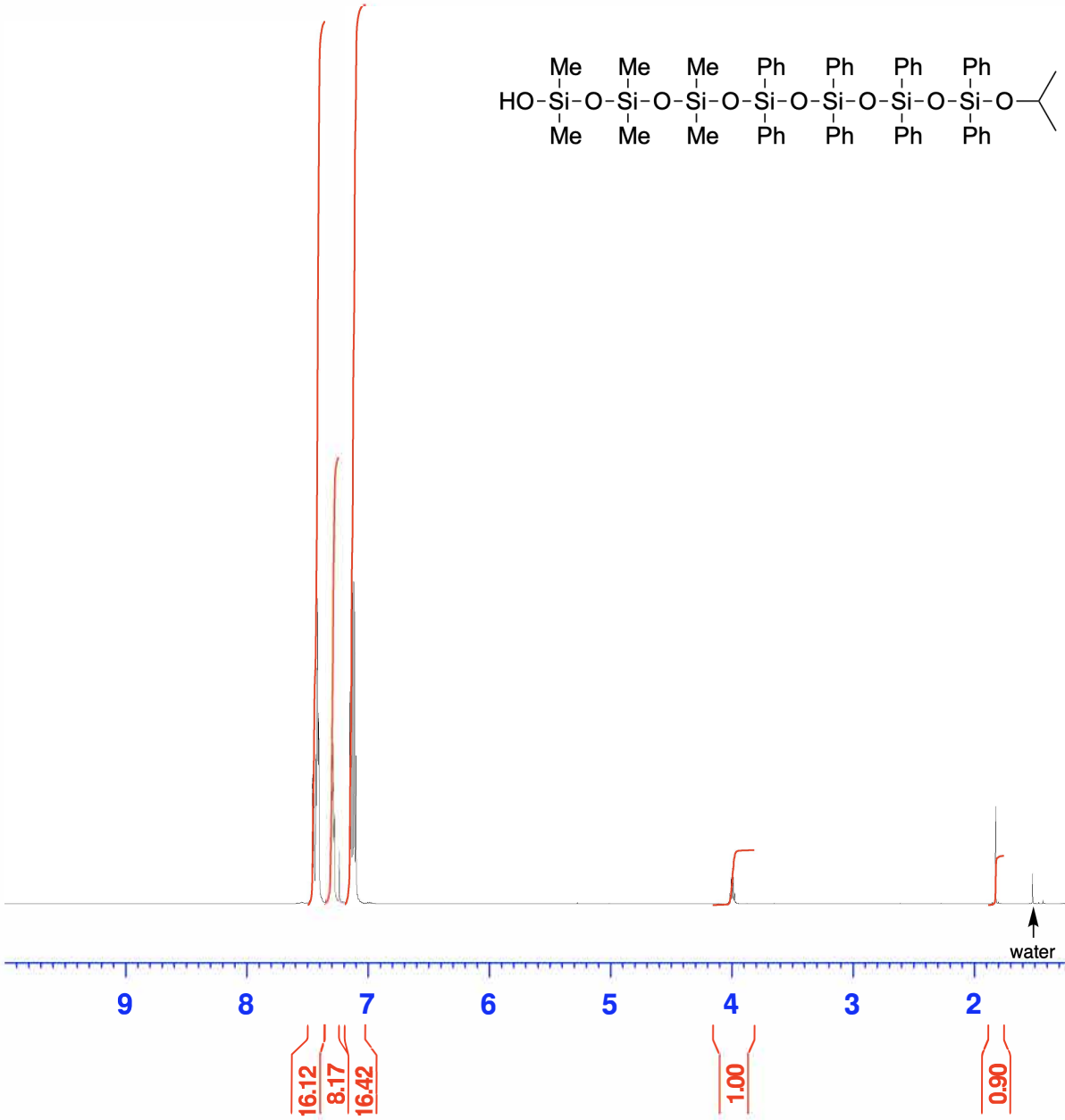



```
Current Data Parameters
NAME                  OMR-22
EXPNO                    120
PROCNO                     1

F2 - Acquisition Parameters
Date_               20210802
Time                   22.54 h
INSTRUM                spect
PROBHD    Z136133_0001 (
PULPROG               zgig30
TD                    131072
SOLVENT                CDCl3
NS                       256
DS                         2
SWH               48076.922 Hz
FIDRES             0.733596 Hz
AQ                1.3631488 sec
RG                       144
DW                    10.400 usec
DE                    19.38 usec
TE                    300.0 K
D1              10.00000000 sec
D11              0.03000000 sec
TD0                        1
SFO1            119.2289493 MHz
NUC1                   29Si
P0                     4.00 usec
P1                    12.00 usec
PLW1            48.00000000 W
SFO2            600.1324005 MHz
NUC2                   1H
CPDPRG[2            waltz65
PCPD2                 70.00 usec
PLW2            15.00000000 W
PLW12            0.44082001 W

F2 - Processing parameters
SI                    131072
SF              119.2289497 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.40
```

Figure S28. 13-Isopropoxy-1,1,3,3,5,5-hexamethyl-7,7,9,9,11,11,13,13-octaphenylheptasiloxan-1-ol (15)
1H NMR



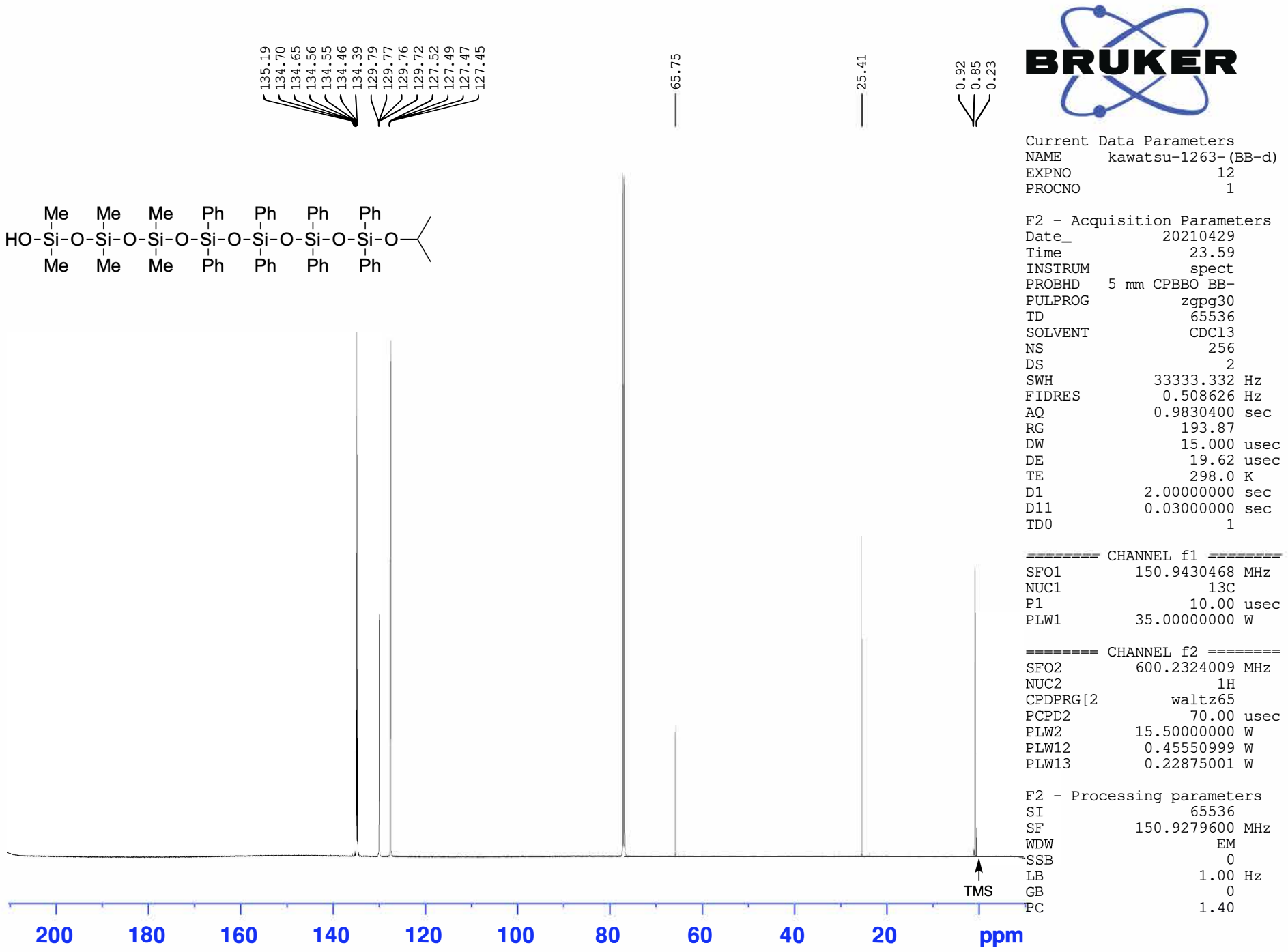

```
Current Data Parameters
NAME      kawatsu-1263-(BB-d)
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20210429
Time              22.05
INSTRUM           spect
PROBHD   5 mm CPBBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                12.86
DW               41.600 usec
DE               15.65 usec
TE               298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         600.2330011 MHz
NUC1                 1H
P1                12.00 usec
PLW1         15.50000000 W

F2 - Processing parameters
SI                65536
SF           600.2300294 MHz
WDW                  EM
SSB                   0
LB                0.20 Hz
GB                    0
PC                1.00
```

Figure S29. 13-Isopropoxy-1,1,3,3,5,5-hexamethyl-7,7,9,9,11,11,13,13-octaphenylheptasiloxan-1-ol (15)
13C NMR




```
Current Data Parameters
NAME      kawatsu-1263-(BB-d)
EXPNO                  12
PROCNO                  1

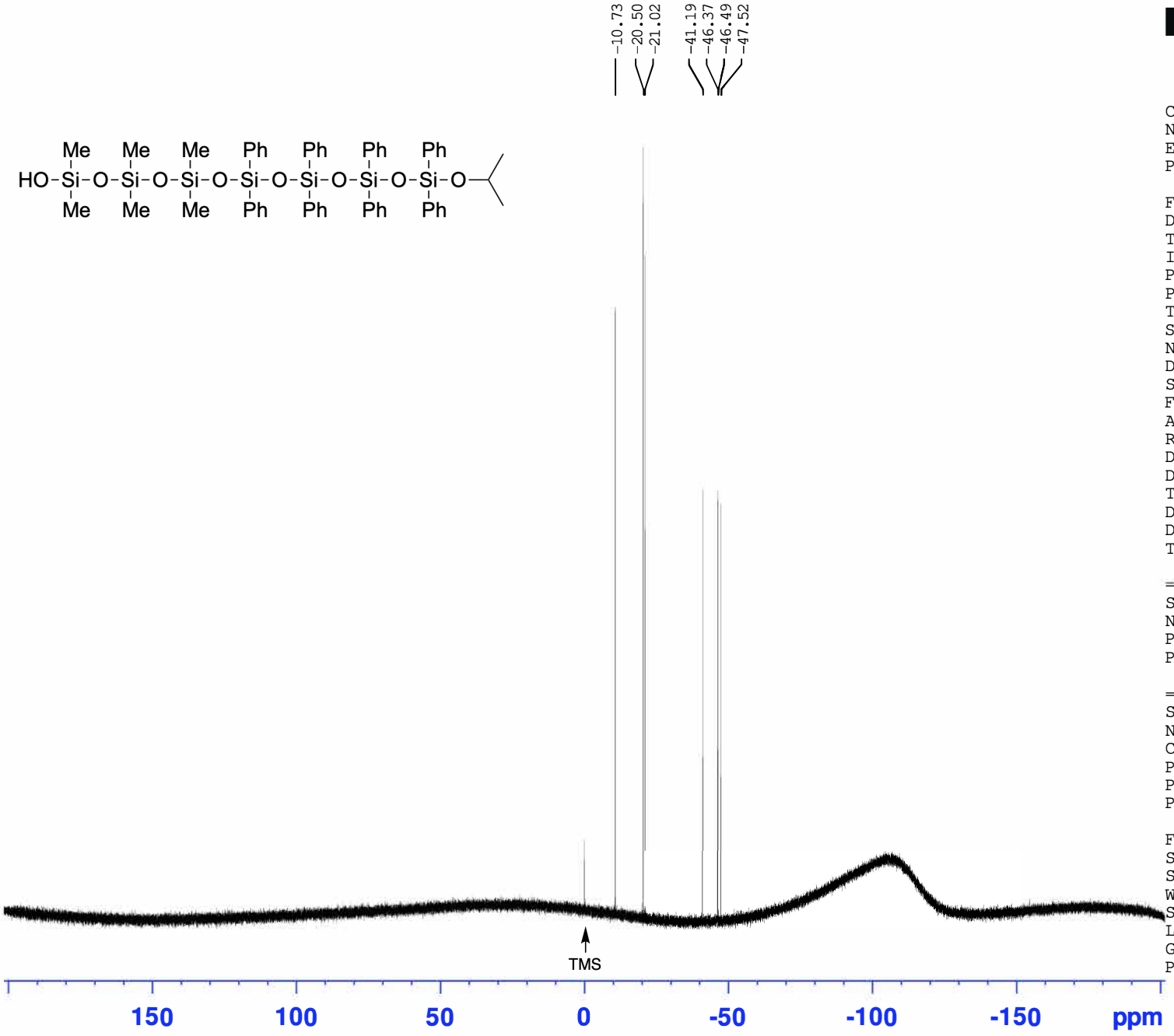
F2 - Acquisition Parameters
Date_           20210429
Time              23.59
INSTRUM           spect
PROBHD   5 mm CPBBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    2
SWH           33333.332 Hz
FIDRES         0.508626 Hz
AQ            0.9830400 sec
RG               193.87
DW               15.000 usec
DE               19.62 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         150.9430468 MHz
NUC1                13C
P1                10.00 usec
PLW1         35.00000000 W

======== CHANNEL f2 ========
SFO2         600.2324009 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         15.50000000 W
PLW12         0.45550999 W
PLW13         0.22875001 W

F2 - Processing parameters
SI                65536
SF           150.9279600 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Figure S30. 13-Isopropoxy-1,1,3,3,5,5-hexamethyl-7,7,9,9,11,11,13,13-octaphenylheptasiloxan-1-ol (15)
29Si NMR




```
Current Data Parameters
NAME      kawatsu-1263-(BB-d)
EXPNO                  11
PROCNO                  1

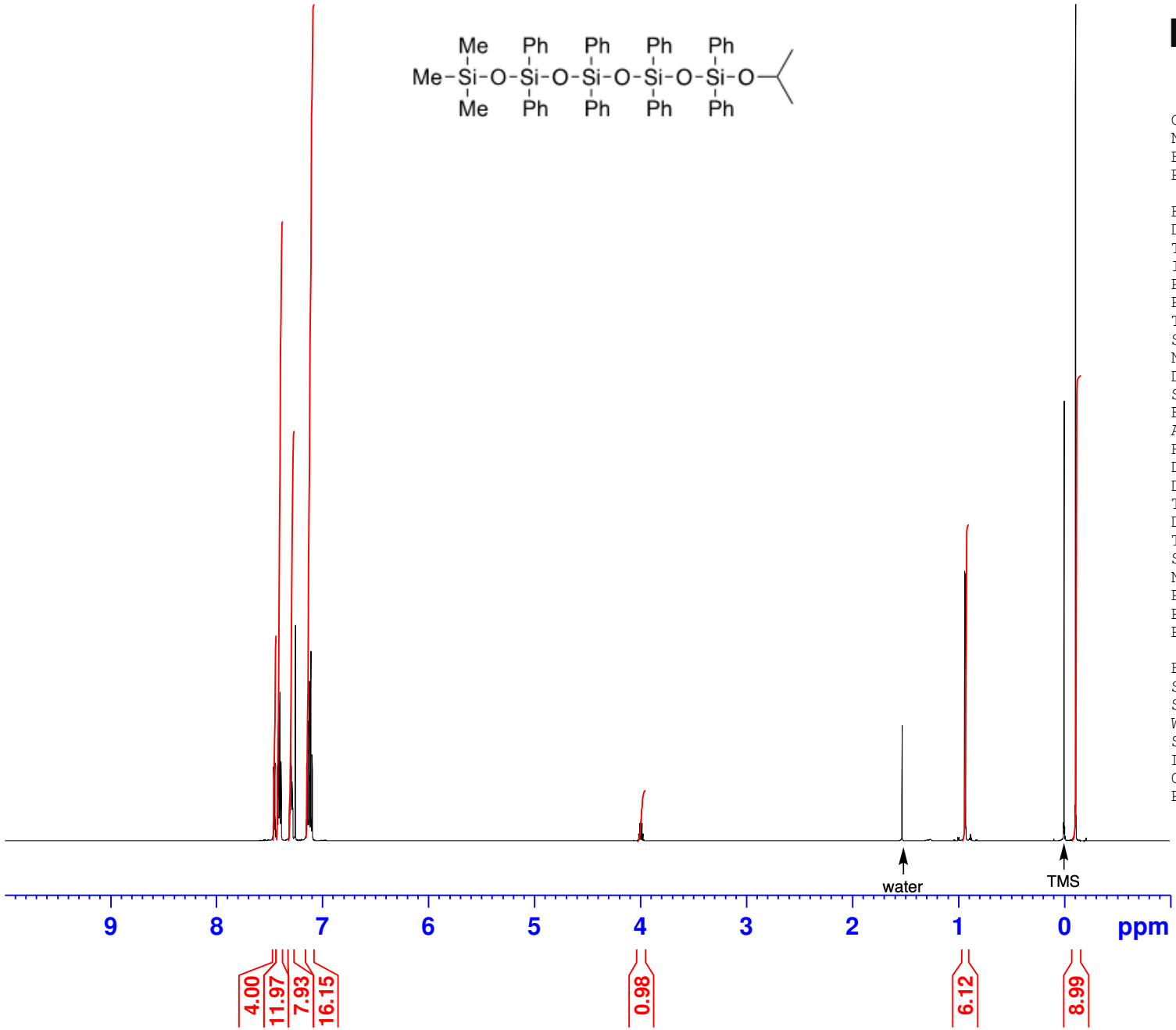
F2 - Acquisition Parameters
Date_           20210429
Time              23.44
INSTRUM           spect
PROBHD   5 mm CPBBO BB-
PULPROG          zgig30
TD              131072
SOLVENT           CDCl3
NS                  512
DS                    2
SWH           48076.922 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.87
DW               10.400 usec
DE               18.90 usec
TE               298.0 K
D1          10.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        119.2488170 MHz
NUC1               29Si
P1                12.00 usec
PLW1        44.50000000 W

======== CHANNEL f2 ========
SFO2        600.2324009 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2             70.00 usec
PLW2        15.50000000 W
PLW12        0.45550999 W

F2 - Processing parameters
SI              131072
SF          119.2488179 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC                1.40
```

Figure S31.1-Isopropoxy-9,9,9-trimethyl-1,1,3,3,5,5,7,7-octaphenylpentasiloxane (14)
1H NMR




```
Current Data Parameters
NAME                 OMR-19
EXPNO                   170
PROCNO                    1

F2 - Acquisition Parameters
Date_               20210805
Time                   12.44 h
INSTRUM                spect
PROBHD    Z132572_0019 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH               12019.230 Hz
FIDRES             0.366798 Hz
AQ                2.7262976 sec
RG                     24.68
DW                    41.600 usec
DE                    16.13 usec
TE                    298.0 K
D1               1.00000000 sec
TD0                       1
SFO1            600.2330011 MHz
NUC1                     1H
P0                     4.00 usec
P1                    12.00 usec
PLW1            13.00000000 W

F2 - Processing parameters
SI                    65536
SF              600.2300173 MHz
WDW                      EM
SSB                       0
LB                     0.20 Hz
GB                        0
PC                     1.00
```

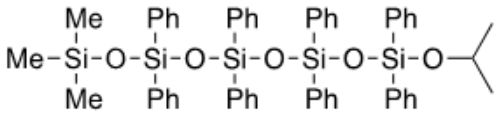
Figure S32. 1-Isopropoxy-9,9,9-trimethyl-1,1,3,3,5,5,7,7-octaphenylpentasiloxane (14)
13C NMR

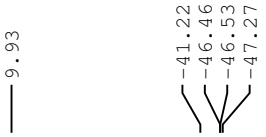

```
Current Data Parameters
NAME       kawatsu-1284
EXPNO                11
PROCNO                1

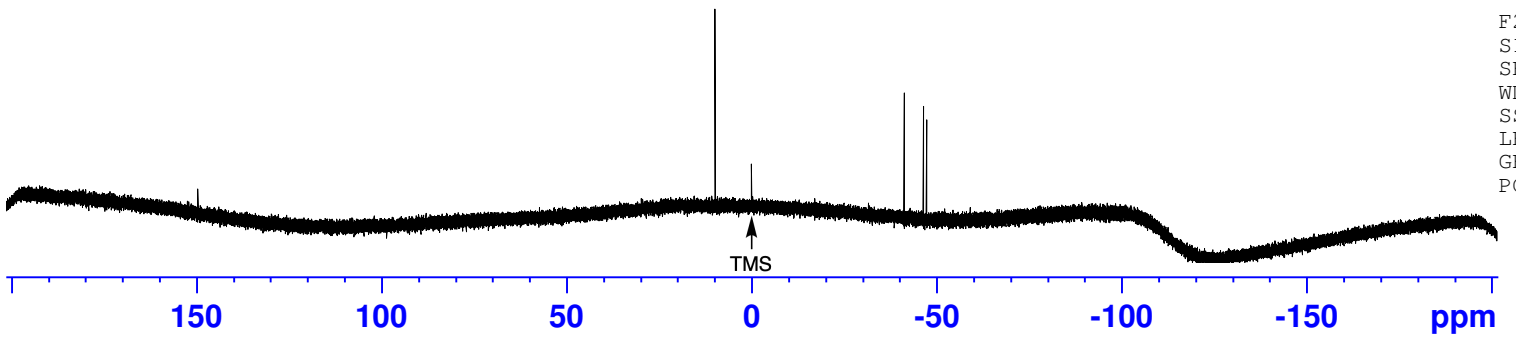
F2 - Acquisition Parameters
Date_          20240226
Time              11.51 h
INSTRUM            spect
PROBHD   Z136133_0001 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           33333.332 Hz
FIDRES         1.017253 Hz
AQ            0.9830400 sec
RG                  203
DW               15.000 usec
DE               18.14 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        150.9178988 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1         48.00000000 W
SFO2        600.1324005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         15.00000000 W
PLW12         0.44082001 W
PLW13         0.22172999 W

F2 - Processing parameters
SI                65536
SF          150.9028106 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Figure S33. 1-Isopropoxy-9,9,9-trimethyl-1,1,3,3,5,5,7,7-octaphenylpentasiloxane (14)
29Si NMR





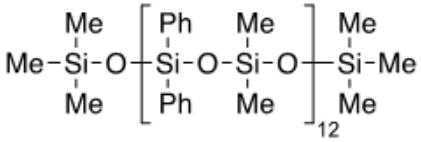



```
Current Data Parameters
NAME       kawatsu-1284
EXPNO                12
PROCNO                1

F2 - Acquisition Parameters
Date_          20240227
Time               0.53 h
INSTRUM           spect
PROBHD   Z132572_0019 (
PULPROG          zgig30
TD               131072
SOLVENT            CDCl3
NS                   256
DS                     2
SWH            48076.922 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                193.87
DW                10.400 usec
DE                19.38 usec
TE                298.0 K
D1            10.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          119.2488165 MHz
NUC1                29Si
P0                 4.00 usec
P1                12.00 usec
PLW1          45.00000000 W
SFO2          600.2324009 MHz
NUC2                  1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2          14.00000000 W
PLW12          0.41143000 W

F2 - Processing parameters
SI                131072
SF          119.2488165 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.40
```

Figure S34. 1,1,1,3,3,7,7,11,11,15,15,19,19,23,23,27,27,31,31,35,35,39,39,43,43,47,47,51,51,51-Triacontamethyl-5,5,9,9,13,13,17,17,21,21,25,25,29,29,33,33,37,37,41,41,45,45,49,49-tetracosaphenylhexacosasiloxane (1)
1H NMR

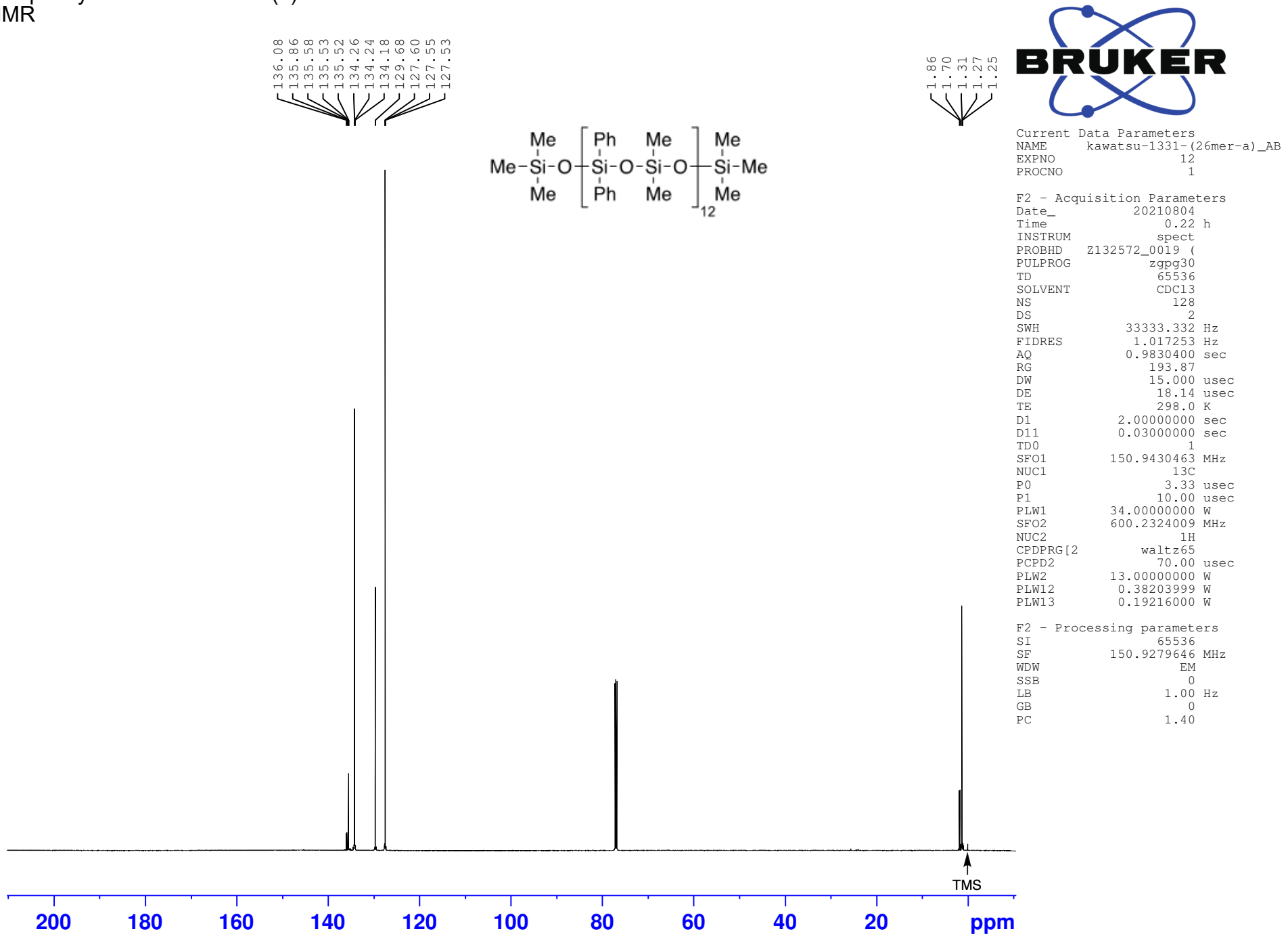



```
Current Data Parameters
NAME      kawatsu-1331-(26mer-a)_AB
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210803
Time              22.33 h
INSTRUM           spect
PROBHD    Z132572_0019 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           12019.230 Hz
FIDRES         0.366798 Hz
AQ             2.7262976 sec
RG                 12.86
DW                41.600 usec
DE                16.13 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         600.2330011 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.2300459 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S35. 1,1,1,3,3,7,7,11,11,15,15,19,19,23,23,27,27,31,31,35,35,39,39,43,43,47,47,51,51,51-Triacontamethyl-5,5,9,9,13,13,17,17,21,21,25,25,29,29,33,33,37,37,41,41,45,45,49,49-tetracosaphenylhexacosasiloxane (1)
13C NMR

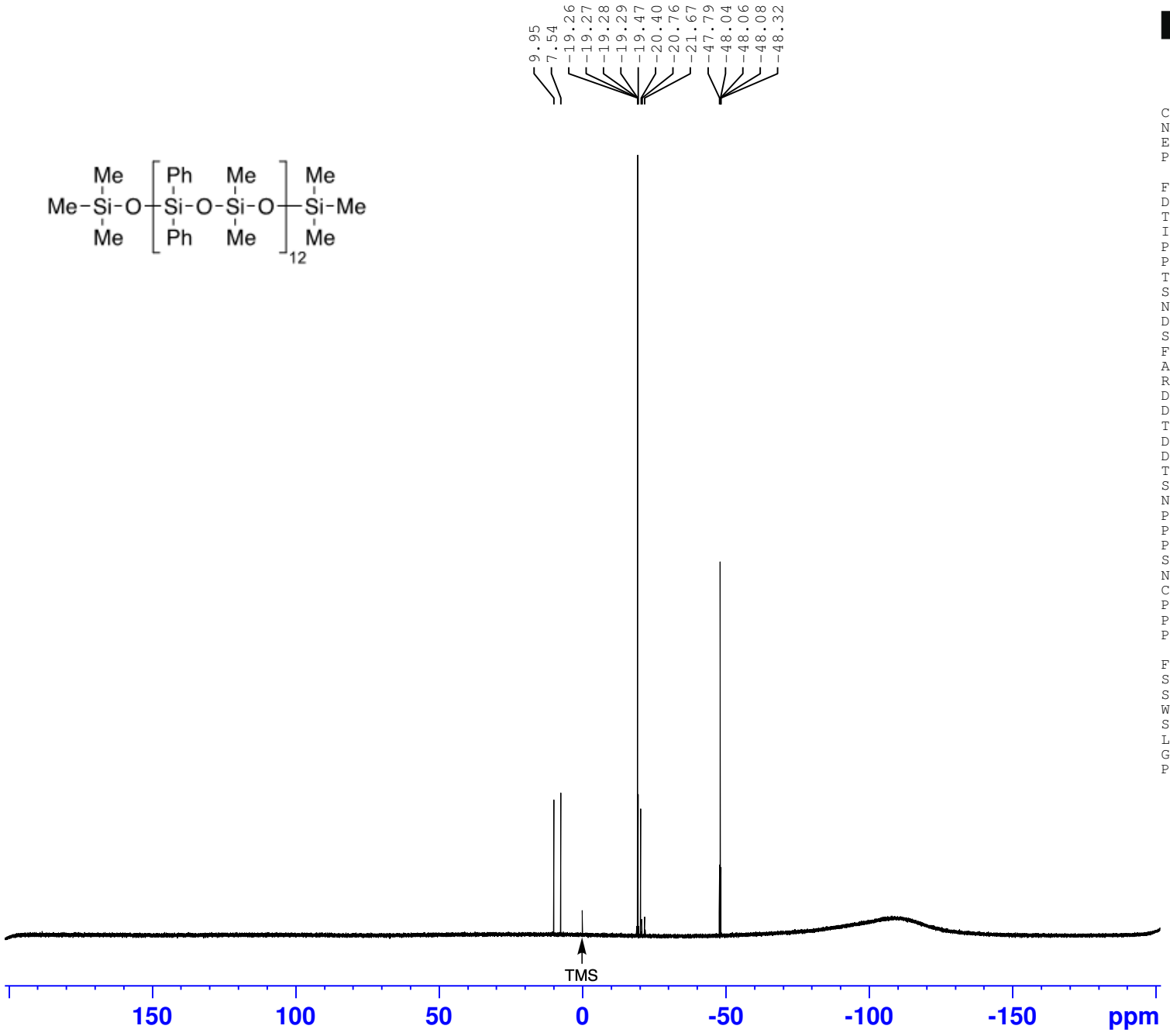


Current Data Parameters
NAME kawatsu-1331-(26mer-a)_AB
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210804
Time 0.22 h
INSTRUM spect
PROBHD Z132572_0019 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 33333.332 Hz
FIDRES 1.017253 Hz
AQ 0.9830400 sec
RG 193.87
DW 15.000 usec
DE 18.14 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9430463 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 34.00000000 W
SFO2 600.2324009 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 13.00000000 W
PLW12 0.38203999 W
PLW13 0.19216000 W

F2 - Processing parameters
SI 65536
SF 150.9279646 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S36. 1,1,1,3,3,7,7,11,11,15,15,19,19,23,23,27,27,31,31,35,35,39,39,43,43,47,47,51,51,51-Triacontamethyl-5,5,9,9,13,13,17,17,21,21,25,25,29,29,33,33,37,37,41,41,45,45,49,49-tetracosaphenylhexacosasiloxane (1)
29Si NMR

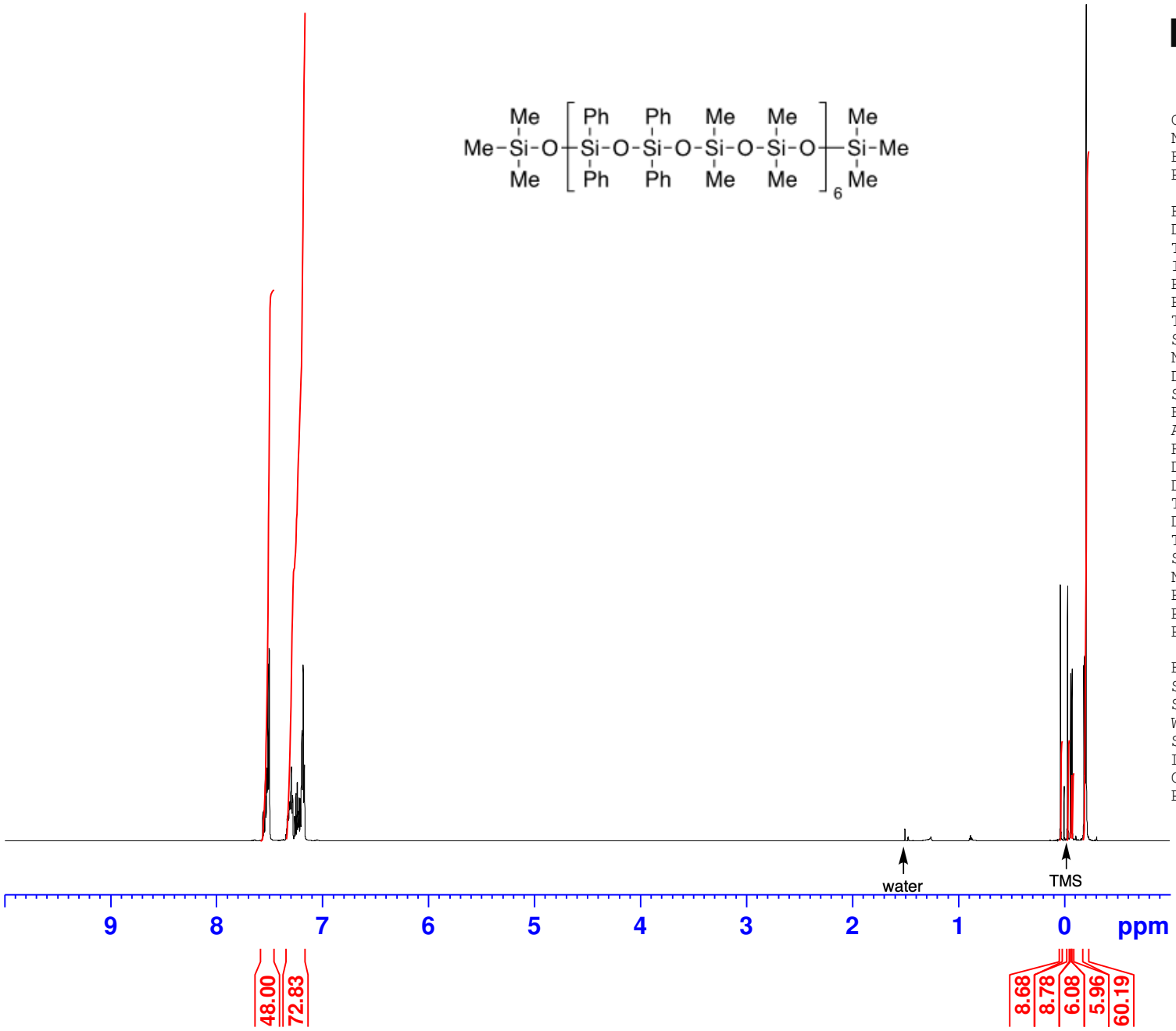


Current Data Parameters
NAME kawatsu-1331-(26mer-a)_AB
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210804
Time 0.13 h
INSTRUM spect
PROBHD Z132572_0019 (
PULPROG zgig30
TD 131072
SOLVENT CDCl3
NS 512
DS 2
SWH 48076.922 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 193.87
DW 10.400 usec
DE 19.38 usec
TE 298.0 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 119.2488165 MHz
NUC1 29Si
P0 4.00 usec
P1 12.00 usec
PLW1 45.00000000 W
SFO2 600.2324009 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 13.00000000 W
PLW12 0.38203999 W

F2 - Processing parameters
SI 131072
SF 119.2488199 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

Figure S37.1,1,1,3,3,5,5,11,11,13,13,19,19,21,21,27,27,29,29,35,35,37,37,43,43,45,45,51,51,51-Triacontamethyl-7,7,9,9,15,15,17,17,23,23,25,25,31,31,33,33,39,39,41,41,47,47,49,49-tetracosaphenylhexacosasiloxane (7)
1H NMR

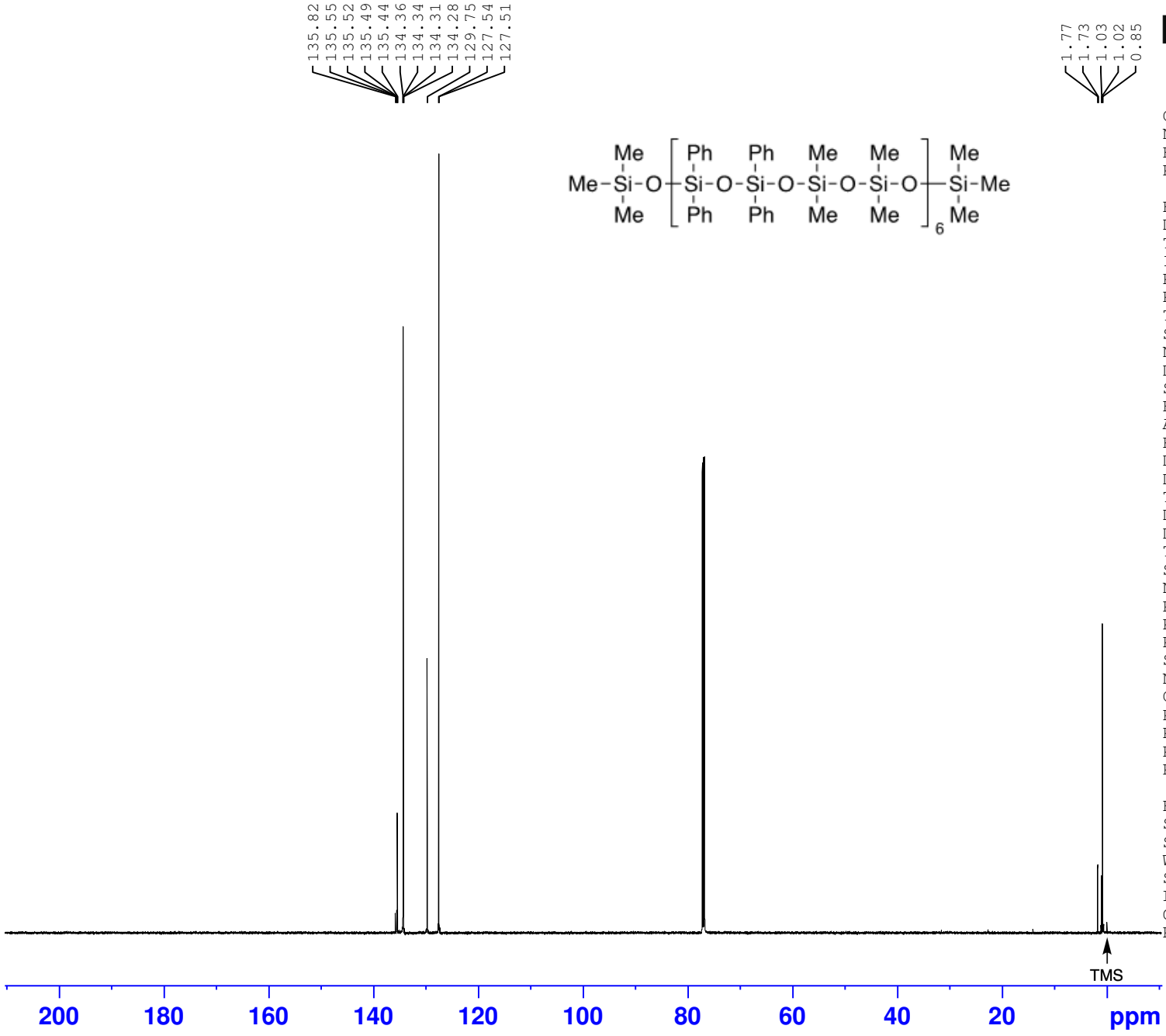



```
Current Data Parameters
NAME      kawatsu-1342-data
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20240226
Time                12.04 h
INSTRUM             spect
PROBHD    Z136133_0001 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ             2.7262976 sec
RG                  25.4
DW                41.600 usec
DE                16.13 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         600.1330006 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300279 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S38. 1,1,1,3,3,5,5,11,11,13,13,19,19,21,21,27,27,29,29,35,35,37,37,43,43,45,45,51,51,51-Triacontamethyl-7,7,9,9,15,15,17,17,23,23,25,25,31,31,33,33,39,39,41,41,47,47,49,49-tetracosaphenylhexacosasiloxane (7)
13C NMR

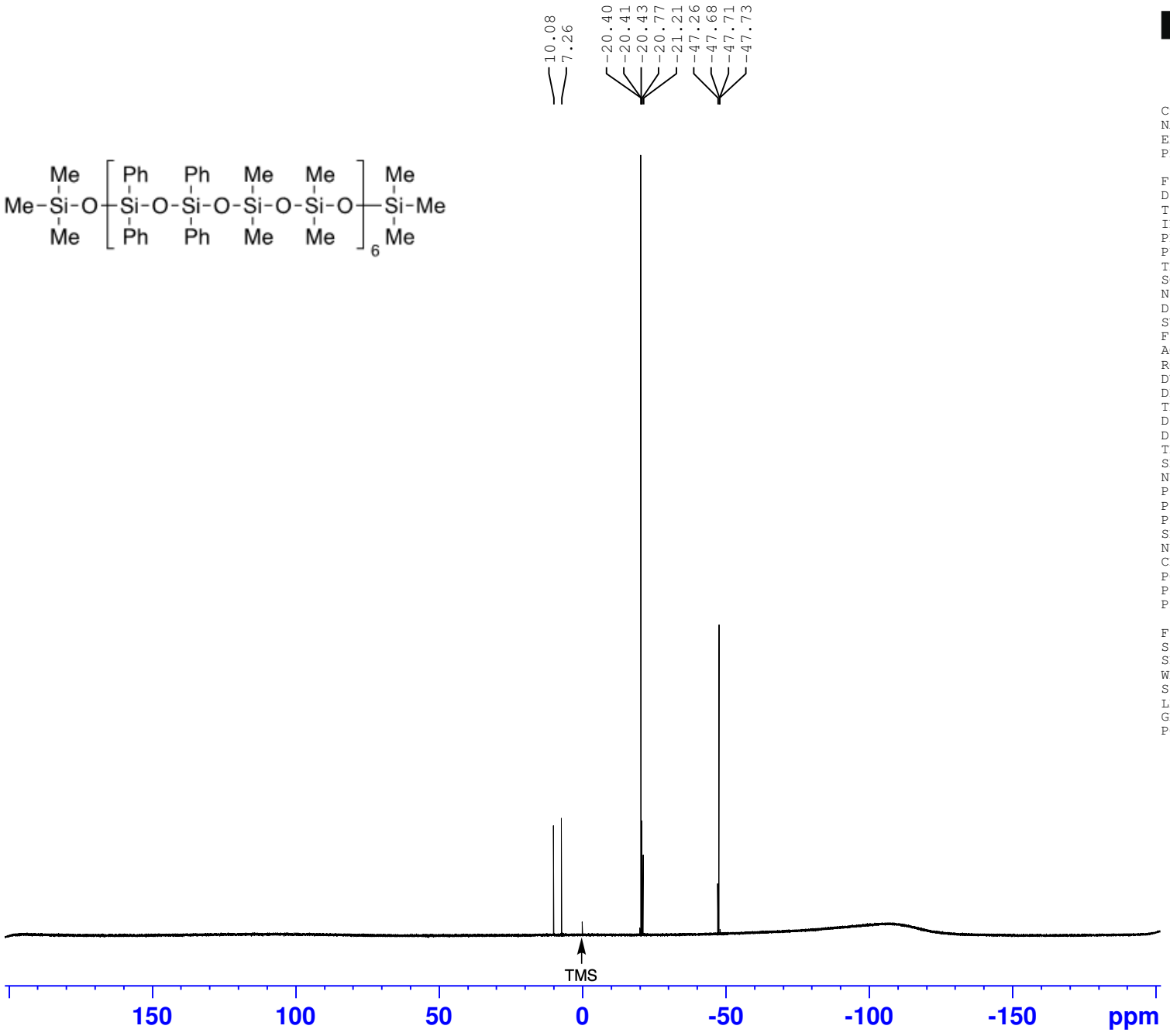


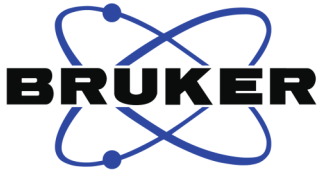

```
Current Data Parameters
NAME      kawatsu-1342-data
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_            20240226
Time                12.09 h
INSTRUM             spect
PROBHD    Z136133_0001 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    64
DS                     2
SWH            33333.332 Hz
FIDRES          1.017253 Hz
AQ             0.9830400 sec
RG                   203
DW                15.000 usec
DE                18.14 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         150.9178988 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1         48.00000000 W
SFO2         600.1324005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         15.00000000 W
PLW12         0.44082001 W
PLW13         0.22172999 W

F2 - Processing parameters
SI                 65536
SF           150.9028133 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Figure S39. 1,1,1,3,3,5,5,11,11,13,13,19,19,21,21,27,27,29,29,35,35,37,37,43,43,45,45,51,51,51-Triacontamethyl-7,7,9,9,15,15,17,17,23,23,25,25,31,31,33,33,39,39,41,41,47,47,49,49-tetracosaphenylhexacosasiloxane (7)
29Si NMR



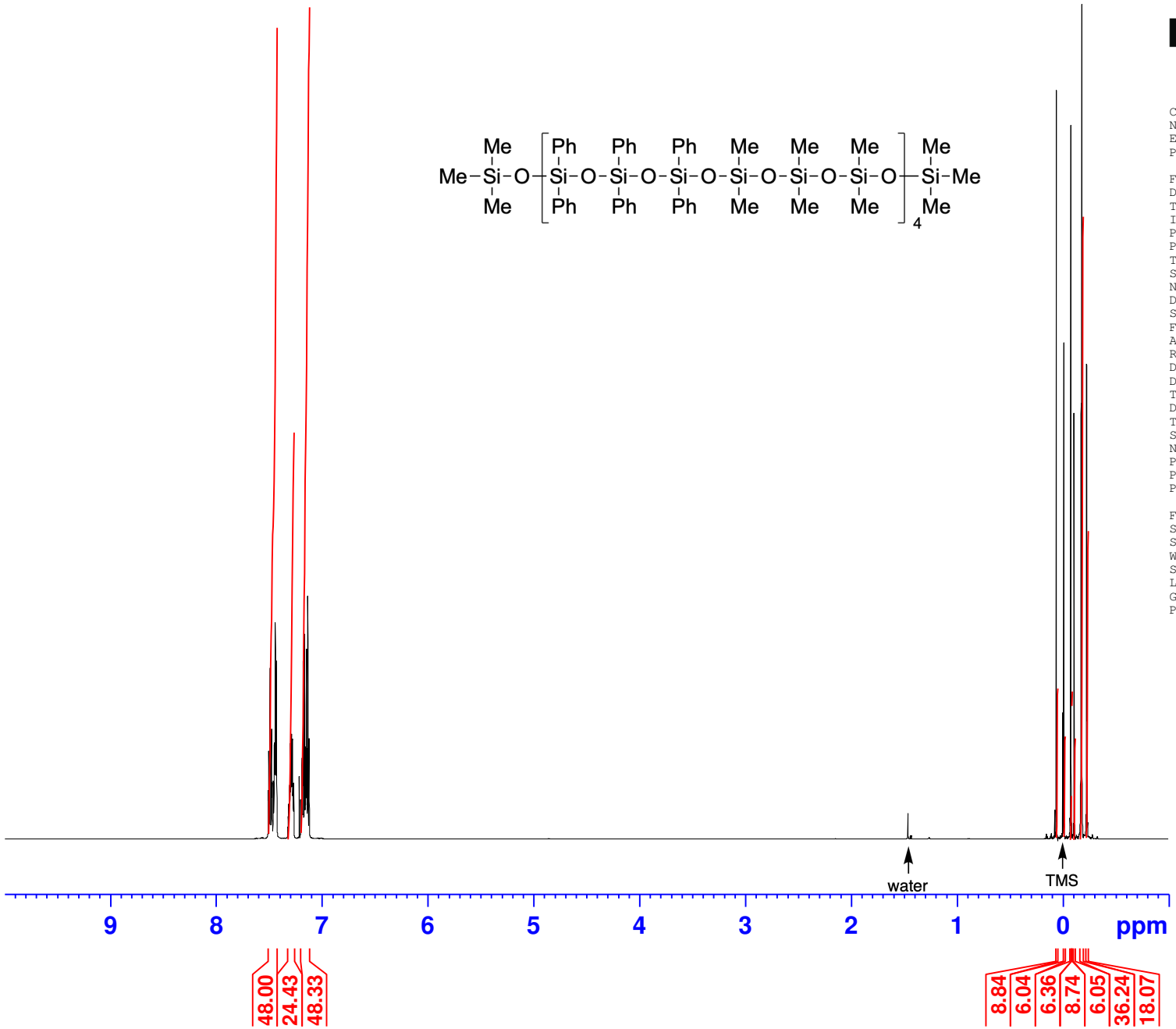



```
Current Data Parameters
NAME      kawatsu-1342-(26mer-b)_AABB
EXPNO                  25
PROCNO                  1

F2 - Acquisition Parameters
Date_            20210804
Time                2.13 h
INSTRUM             spect
PROBHD    Z132572_0019 (
PULPROG            zgig30
TD                 131072
SOLVENT             CDCl3
NS                    512
DS                      2
SWH             48076.922 Hz
FIDRES           0.733596 Hz
AQ              1.3631488 sec
RG                 193.87
DW                 10.400 usec
DE                 19.38 usec
TE                 298.0 K
D1            10.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          119.2488165 MHz
NUC1                29Si
P0                  4.00 usec
P1                 12.00 usec
PLW1          45.00000000 W
SFO2          600.2324009 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2              70.00 usec
PLW2          13.00000000 W
PLW12          0.38203999 W

F2 - Processing parameters
SI                 131072
SF            119.2488204 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40
```

Figure S40. 1,1,1,3,3,5,5,7,7,15,15,17,17,19,19,27,27,29,29,31,31,39,39,41,41,43,43,51,51,51-Triacontamethyl-9,9,11,11,13,13,21,21,23,23,25,25,33,33,35,35,37,37,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (10)
1H NMR

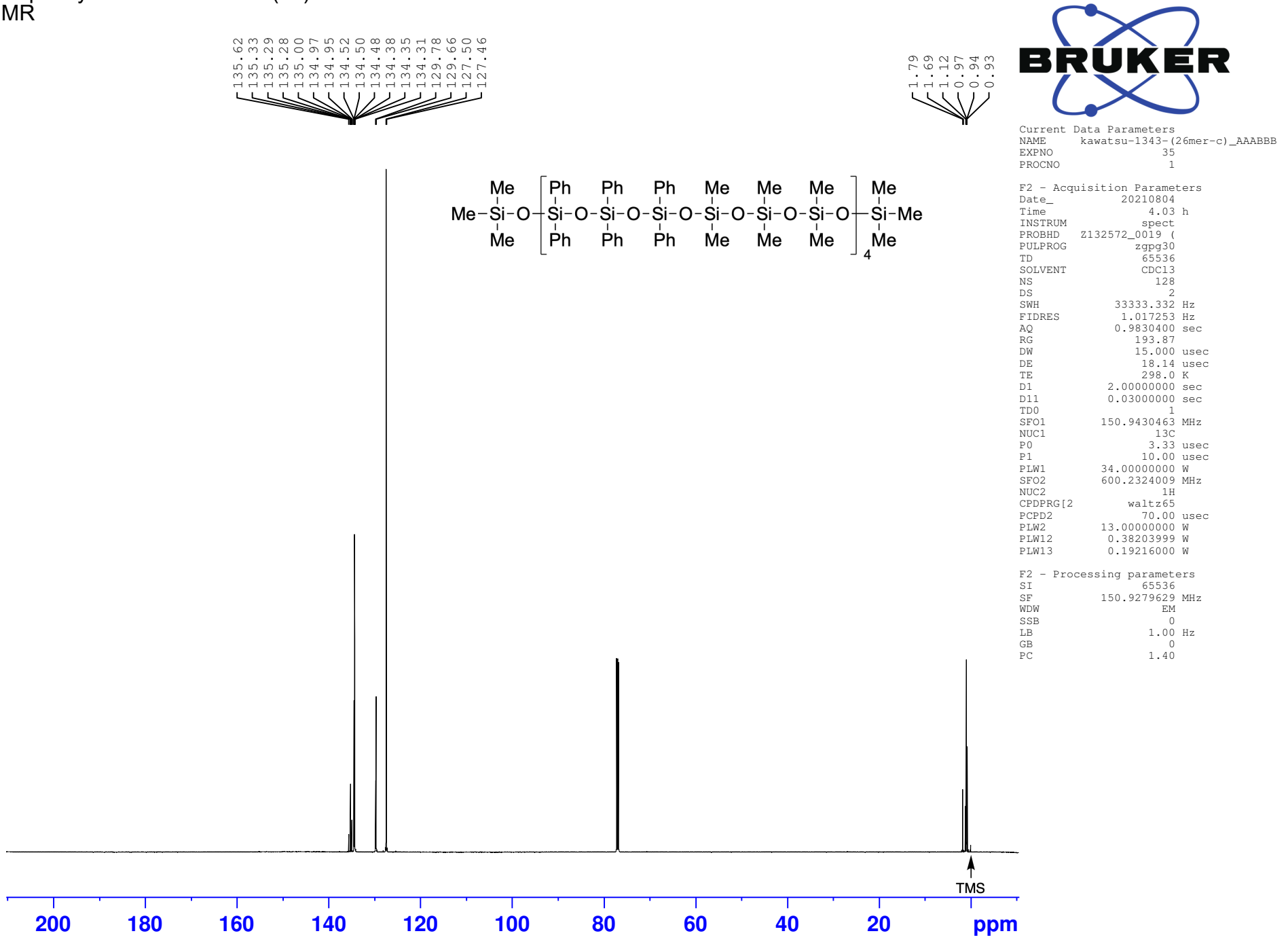



```
Current Data Parameters
NAME      kawatsu-1343-(26mer-c)_AAABBB
EXPNO                 33
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210804
Time                2.16 h
INSTRUM           spect
PROBHD   Z132572_0019 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12019.230 Hz
FIDRES          0.366798 Hz
AQ             2.7262976 sec
RG                  17.4
DW                41.600 usec
DE                16.13 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         600.2330011 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.2300409 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00
```

Figure S41. 1,1,1,3,3,5,5,7,7,15,15,17,17,19,19,27,27,29,29,31,31,39,39,41,41,43,43,51,51,51-Triacontamethyl-9,9,11,11,13,13,21,21,23,23,25,25,33,33,35,35,37,37,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (10)
13C NMR

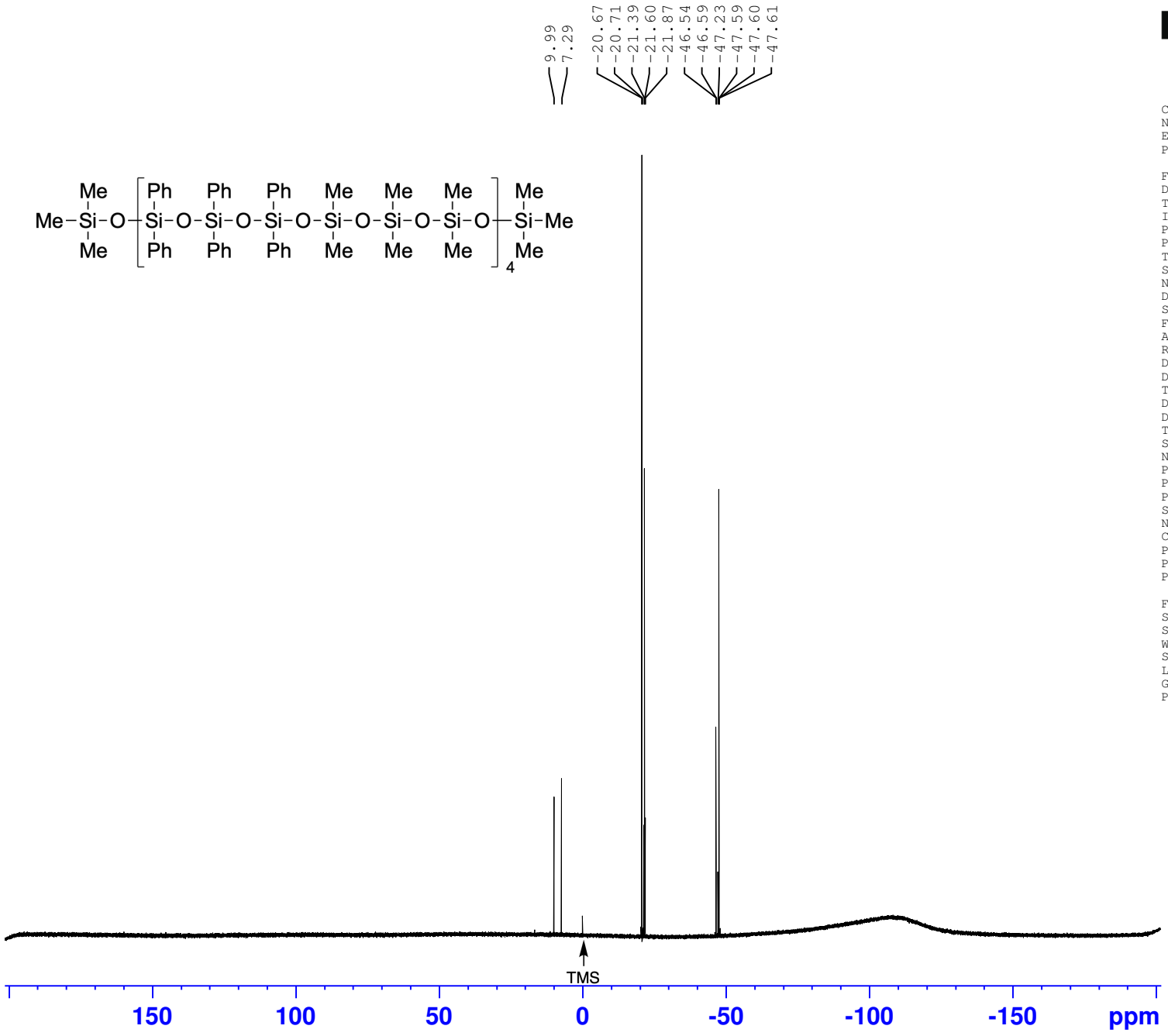


Current Data Parameters
NAME kawatsu-1343-(26mer-c)_AAABBB
EXPNO 35
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210804
Time 4.03 h
INSTRUM spect
PROBHD Z132572_0019 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 33333.332 Hz
FIDRES 1.017253 Hz
AQ 0.9830400 sec
RG 193.87
DW 15.000 usec
DE 18.14 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9430463 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 34.00000000 W
SFO2 600.2324009 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 13.00000000 W
PLW12 0.38203999 W
PLW13 0.19216000 W

F2 - Processing parameters
SI 65536
SF 150.9279629 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S42. 1,1,1,3,3,5,5,7,7,15,15,17,17,19,19,27,27,29,29,31,31,39,39,41,41,43,43,51,51,51-Triacontamethyl-9,9,11,11,13,13,21,21,23,23,25,25,33,33,35,35,37,37,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (10)
29Si NMR

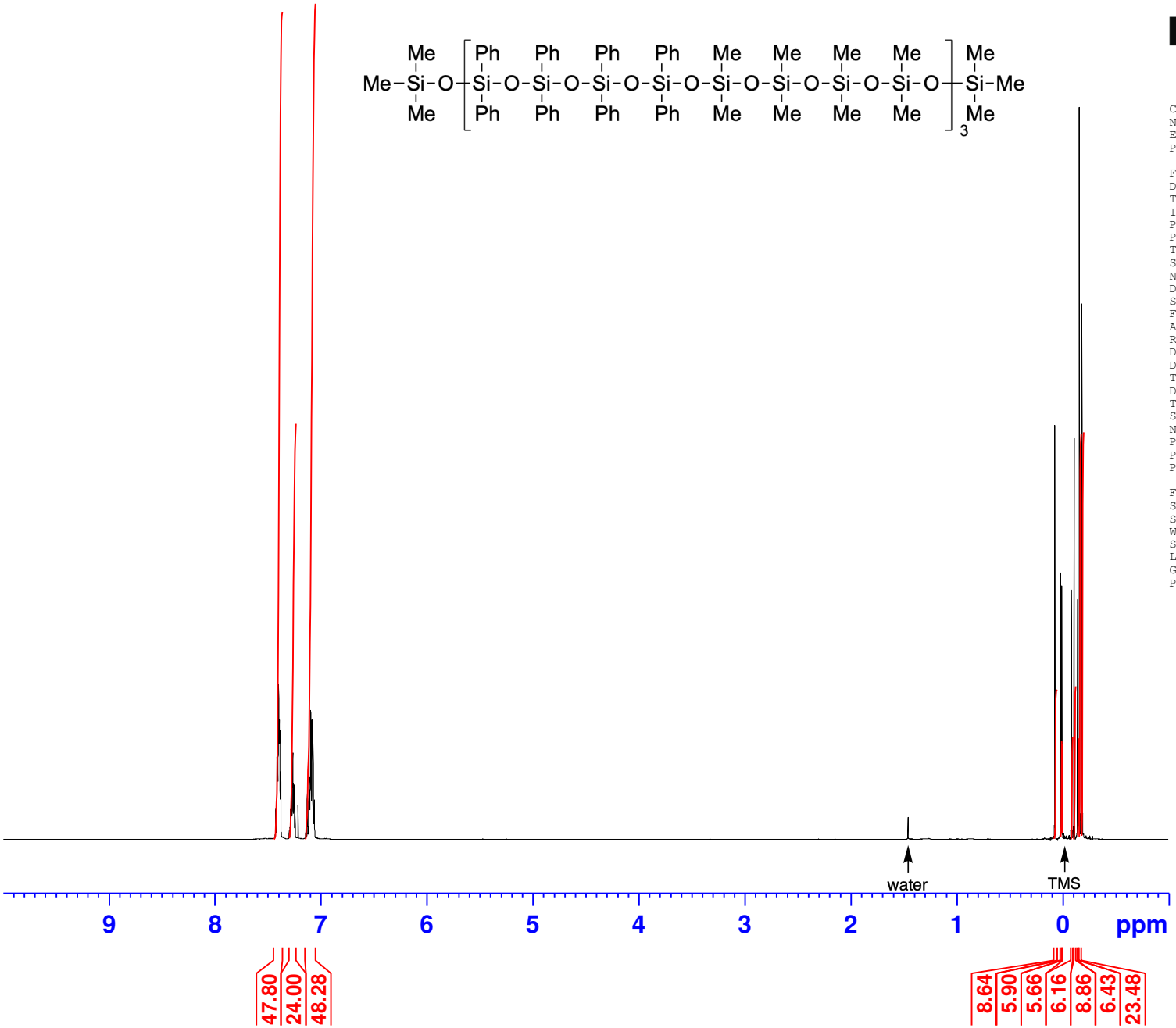


Current Data Parameters
NAME kawatsu-1343-(26mer-c)_AAABBB
EXPNO 34
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210804
Time 3.54 h
INSTRUM spect
PROBHD Z132572_0019 (
PULPROG zgig30
TD 131072
SOLVENT CDCl3
NS 512
DS 2
SWH 48076.922 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 193.87
DW 10.400 usec
DE 19.38 usec
TE 298.0 K
D1 10.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 119.2488165 MHz
NUC1 29Si
P0 4.00 usec
P1 12.00 usec
PLW1 45.00000000 W
SFO2 600.2324009 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 70.00 usec
PLW2 13.00000000 W
PLW12 0.38203999 W

F2 - Processing parameters
SI 131072
SF 119.2488192 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

Figure S43. 1,1,1,3,3,5,5,7,7,9,9,19,19,21,21,23,23,25,25,35,35,37,37,39,39,41,41,51,51,51-Triacontamethyl-11,11,13,13,15,15,17,17,27,27,29,29,31,31,33,33,43,43,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (13)
1H NMR

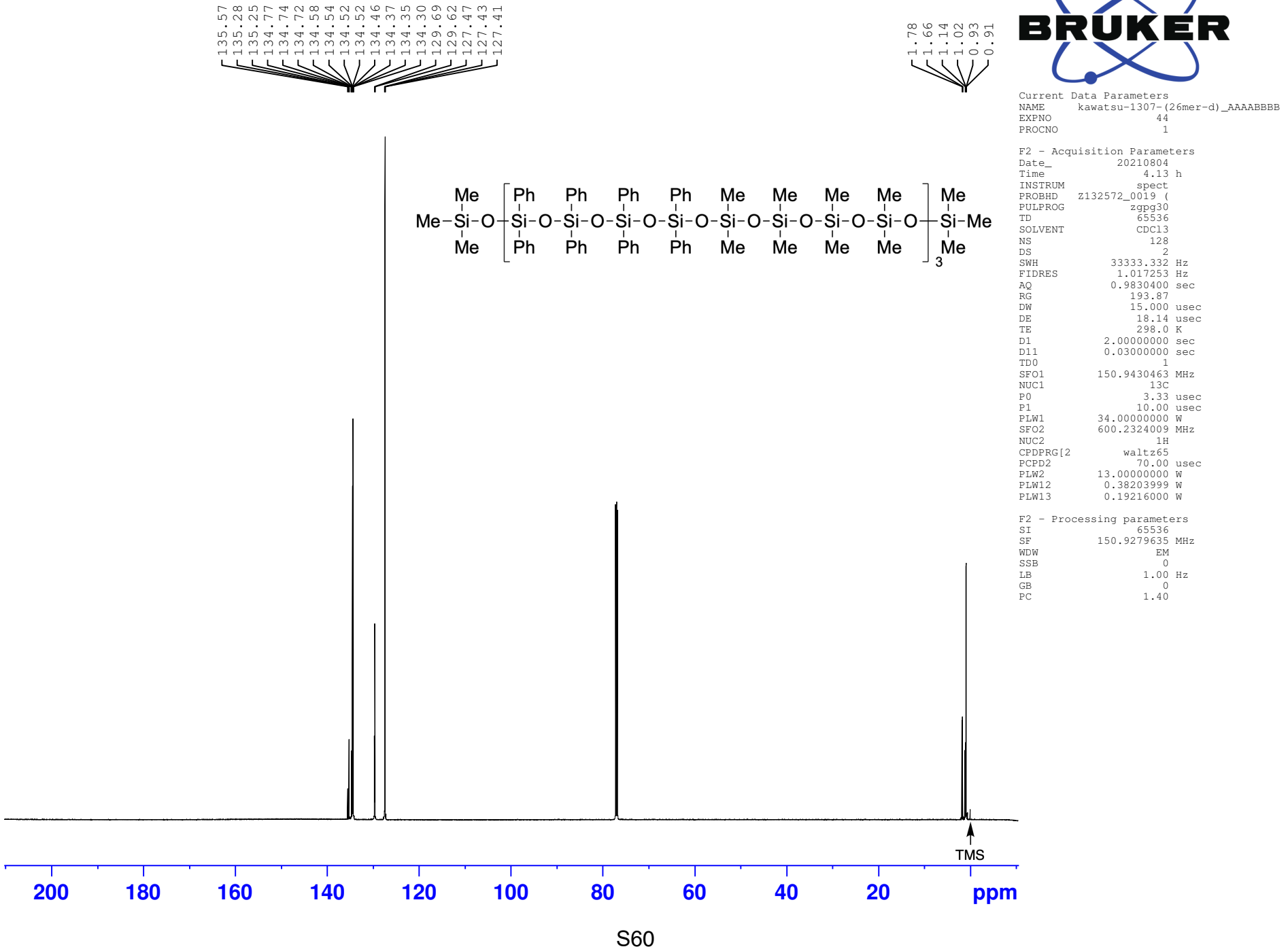



```
Current Data Parameters
NAME      kawatsu-1307-(26mer-d)_AAAABBBB
EXPNO                 43
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210804
Time               4.06 h
INSTRUM           spect
PROBHD   Z132572_0019 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12019.230 Hz
FIDRES         0.366798 Hz
AQ             2.7262976 sec
RG                16.19
DW               41.600 usec
DE                16.13 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1
SFO1         600.2330011 MHz
NUC1                 1H
P0                4.00 usec
P1               12.00 usec
PLW1        13.00000000 W

F2 - Processing parameters
SI                65536
SF           600.2300407 MHz
WDW                  EM
SSB                   0
LB                0.20 Hz
GB                    0
PC                1.00
```

Figure S44. 1,1,1,3,3,5,5,7,7,9,9,19,19,21,21,23,23,25,25,35,35,37,37,39,39,41,41,51,51,51-Triacontamethyl-11,11,13,13,15,15,17,17,27,27,29,29,31,31,33,33,43,43,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (13)
13C NMR

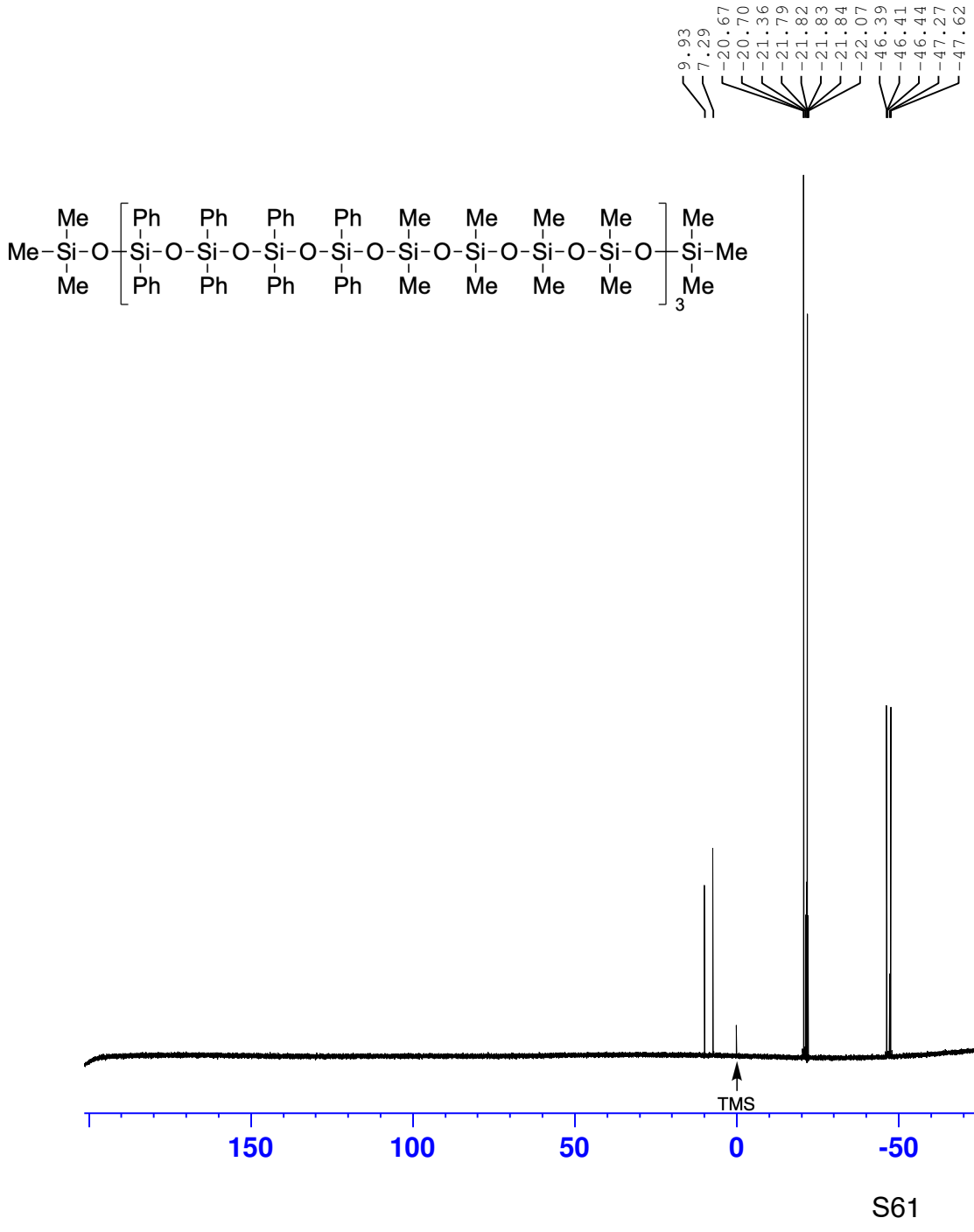



```
Current Data Parameters
NAME      kawatsu-1307-(26mer-d)_AAAABBBB
EXPNO                 44
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210804
Time               4.13 h
INSTRUM            spect
PROBHD   Z132572_0019 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    2
SWH           33333.332 Hz
FIDRES         1.017253 Hz
AQ             0.9830400 sec
RG                193.87
DW                15.000 usec
DE                18.14 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         150.9430463 MHz
NUC1                 13C
P0                 3.33 usec
P1                10.00 usec
PLW1         34.00000000 W
SFO2         600.2324009 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W
PLW13         0.19216000 W

F2 - Processing parameters
SI                65536
SF           150.9279635 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Figure S45. 1,1,1,3,3,5,5,7,7,9,9,19,19,21,21,23,23,25,25,35,35,37,37,39,39,41,41,51,51,51-Triacontamethyl-11,11,13,13,15,15,17,17,27,27,29,29,31,31,33,33,43,43,45,45,47,47,49,49-tetracosaphenylhexacosasiloxane (13)
29Si NMR




```
Current Data Parameters
NAME      kawatsu-1307-(26mer-d)_AAAABBBB
EXPNO                 45
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210804
Time               5.53 h
INSTRUM            spect
PROBHD   Z132572_0019 (
PULPROG          zgig30
TD               131072
SOLVENT            CDCl3
NS                   512
DS                     2
SWH            48076.922 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 193.87
DW                 10.400 usec
DE                 19.38 usec
TE                 298.0 K
D1           10.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1         119.2488165 MHz
NUC1                29Si
P0                  4.00 usec
P1                 12.00 usec
PLW1         45.00000000 W
SFO2         600.2324009 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2              70.00 usec
PLW2         13.00000000 W
PLW12         0.38203999 W

F2 - Processing parameters
SI                 131072
SF           119.2488192 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40
```

References

S1 T. Kawatsu, J.-C. Choi, K. Sato and K. Matsumoto, *Macromol. Rapid Commun.*, 2021, **42**, 2000593.

S2 J. A. Cella and J. C. Carpenter, *J. Organometallic Chem.*, 1994, **480**, 23–26.