Insertion Reactions of Silacyclopropanes: Evidence for a Radical-Based Mechanism

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Supporting Information

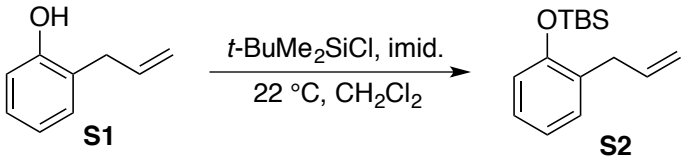
Contents:

I. **General Procedures**

^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature using Bruker AV–400 (400 and 100 MHz, respectively), AVIII-400 (400 and 100 MHz, respectively), AV–500 (500 and 125 MHz, respectively) or AVIII–600 (600 and 150 MHz, respectively) spectrometers, as indicated. ^{29}Si NMR spectra were recorded at ambient temperature using a Bruker AVIII-400 (79 MHz) spectrometer. The data are reported as follows: chemical shift in ppm from internal tetramethylsilane or referenced to residual solvent (^{1}H NMR: C_6D_6 δ 7.16; $CDCl_3$ 7.26. ^{13}C NMR: C_6D_6 δ 128.4; $CDCl_3$ δ 77.2. ^{29}Si NMR: referenced to external tetramethylsilane C_6D_6 δ 0; $CDCl_3$ δ 0.) on the δ scale, multiplicity (appar = apparent, br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Due to difficulties with purification for certain products, only distinctive peaks are listed in tabulated ^{1}H NMR and ^{13}C NMR spectral data as indicated. ^{1}H and ^{13}C peaks were assigned using a combination of COSY, NOE, HSQC, DEPT, and HMBC experiments. Multiplicity of carbon peaks was determined using a combination of HSQC and DEPT experiments. ^{1}H NMR yields were determined relative to a known concentration of internal standard, mesitylene, using a single scan. Infrared (IR) spectra were obtained using a Thermo Nicolet *AVATAR* 360 FT-IR 5000 spectrometer using either attenuated total reflectance (ATR) or a thin film on a salt plate, as indicated. High-resolution mass spectra (HRMS) were acquired on an Agilent 6224 Accurate-Mass time-of-flight spectrometer and were obtained by peak matching. Microanalyses were performed by Atlantic Microlab Inc., Norcross, GA. Melting points were reported uncorrected. Analytical thin layer chromatography was performed on Silicycle silica gel 60 Å F_{254} plates. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on Silicycle silica gel (SiO_2) 60 (230-400 mesh). Methylene chloride,

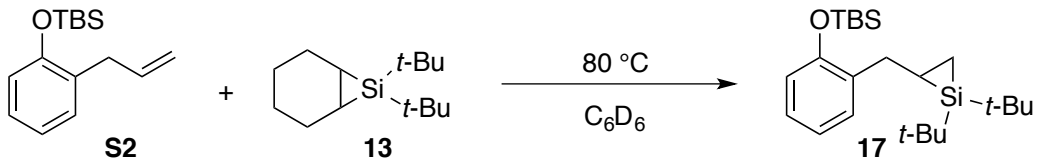
hexanes, diethyl ether, benzene, and triethylamine were dried by filtration through alumina according to the method of Grubbs.[1] C_6D_6 was dried over 3 Å molecular sieves for 48 h and degassed prior to use. Dimethyl sulfoxide was distilled over CaH_2. All reactions were run under an atmosphere of nitrogen in glassware that was flame-dried under a stream of nitrogen unless otherwise stated. Silacyclopropanes were stored and manipulated in a Vacuum Atmospheres nitrogen-atmosphere drybox. Benzaldehyde was distilled prior to use. Enone **S6**, enone **39**, 1,4 benzoquinone **11**, and 2,6-dichlorobenzoquinone **15** are commercially available and were used as received. Cyclohexene silacyclopropane **13**,[2] cis-silacyclopropane **10**,[3] trans-silacyclopropane **10**,[3] 1,1-dimethyl-di-*tert*-butylsilacyclopropane **20**,[4] diene **S3**,[5] alkene **S4,**[6] and alkene **S5**[7,8] were synthesized by known methods.

II. **Substrate Synthesis**



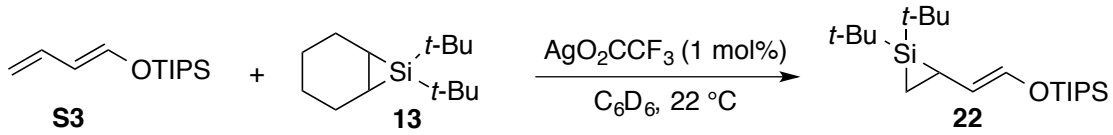
(2-Allylphenoxy)(*tert*-butyl)dimethylsilane S2. To a solution of 2-allylphenol (1.3 mL, 10 mmol) in methylene chloride (20 mL) were added imidazole (2.1 g, 31 mmol) and *t*-$BuMe_2SiCl$ (2.3 g, 15 mmol). After stirring at room temperature overnight (12 h), saturated aqueous NH_4Cl (15 mL) and H_2O (10 mL) were added. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layers were washed with H_2O (15 mL) and brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash chromatography (hexanes) provided protected allyl phenol **S2** as a colorless oil (2.5 g, 99%). The spectral data are consistent with the data reported:[9] ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.16 (dd, *J* = 7.5, 1,6, 1H), 7.11 (td, *J* = 7.7, 1.7, 1H), 6.92 (td, *J* = 7.4, 1.1, 1H), 6.82 (dd, *J* = 8.0, 1.0,

1H), 6.03–5.97 (m, 1H), 5.08 (br s, 1H), 5.06–5.04 (m, 1H), 3.41–3.39 (m, 2H), 1.04 (s, 9H), 0.26 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.5, 137.3, 130.9, 130.3, 127.2, 121.3, 118.6, 115.6, 34.6, 26.0, 18.5, –4.0; HRMS (TOF MS ES+) *m* / *z* calcd for $C_{15}H_{25}OSi$ $(M+H)^+$ 249.1669, found 249.1662.

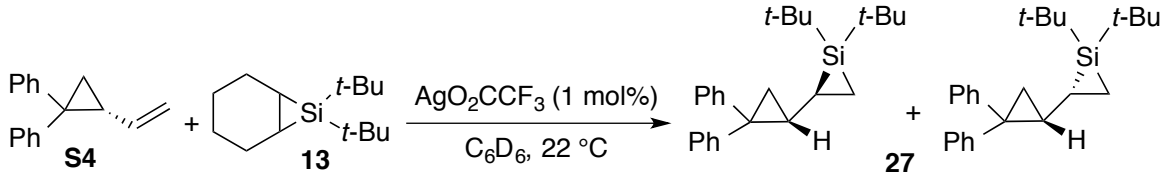


S2 **13** **17**

Silacyclopropane 17. To a solution of silyl-protected allylphenol **S2** (0.049 g, 0.20 mmol) in C_6D_6 (0.59 mL) in a J. Young NMR tube were added a solution of cyclohexene silacyclopropane **13** (0.21 mL, 1.3 M in C_6D_6, 0.27 mmol) and mesitylene (0.0020 mL, 0.014 mmol, internal standard). The reaction mixture was heated to 80 °C for 8 h. Silacyclopropane **17** was formed in 98% based on comparison of the standard peak (δ 2.17) and the methylene protons of the unpurified reaction mixture: ^{1}H NMR (500 MHz, C_6D_6) δ 7.54 (dd, *J* = 7.4, 1.6, 1H), 7.07 (td, *J* = 7.6, 1.8, 1H), 7.01 (td, *J* = 7.4, 1.2, 1H), 6.85 (dd, *J* = 7.9, 1.1, 1H), 3.26 (dd, *J* = 15.8, 6.8, 1H), 3.07 (dd, *J* = 15.7, 9.7, 1H), 1.32–1.28 (m, 1H), 1.14 (s, 9H), 1.07 (s, 9H), 1.05 (s, 9H), 0.94–0.90 (m, 1H), 0.42 (dd, *J* = 10.9, 8.9, 1H), 0.17 (s, 3H), 0.16 (s, 3H); ^{13}C NMR (125 MHz, C_6D_6) δ 154.1 (C), 135.8 (C), 130.1 (CH), 127.1 (CH), 121.9 (CH), 119.0 (CH), 32.9 (CH_2), 31.2 (CH_3), 30.2 (CH_3), 26.4 (CH_3), 19.4 (C), 18.9 (C), 18.7 (C), 13.3 (CH_2), 4.4 (CH), –3.6 (CH_3), –3.7 (CH_3).

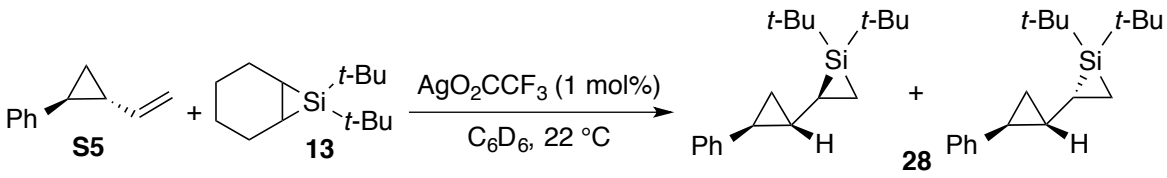


Vinylsilacyclopropane 22. To a solution of diene **S3** (0.047 g, 0.21 mmol) in C_6D_6 (0.32 mL) in a J. Young NMR tube were added a solution of cyclohexene silacyclopropane **13** (0.22 mL, 1.3 M in C_6D_6, 0.29 mmol), and mesitylene (0.0020 mL, 0.014 mmol, internal standard). Silver trifluoroacetate (0.0008 g, 0.004 mmol) was added and the unpurified reaction mixture was analyzed by NMR spectroscopy after 10 min. Vinyl silacyclopropane **22** was formed in 84% based on comparison of the standard peak (δ 6.71) and the alkene protons. The spectral data are consistent with the data reported:[5] ^{1}H NMR (500 MHz, C_6D_6) δ 6.54 (dd, J = 11.7, 1.4, 1H), 5.51 (dd, J = 11.6, 7.6, 1H), 1.70–1.64 (m, 1H), 1.12 (br s, 21H and 9H), 0.99 (s, 9H), 0.93–0.88 (m, 1H), 0.56 (dd, J = 11.0, 8.8, 1H); ^{13}C NMR (125 MHz, C_6D_6) δ 138.8, 114.9, 31.0, 30.1, 19.6, 18.8, 18.5, 12.9, 12.0, 3.3.



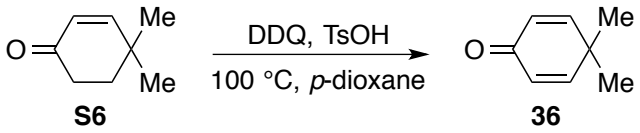
Silacyclopropanes 27. To a solution of alkene **S4** (0.210 mL, 1.05 M in C_6D_6, 0.220 mmol) in C_6D_6 (0.30 mL) in a J. Young NMR tube were added cyclohexene silacyclopropane **13** (0.055 g, 0.25 mmol) and mesitylene (0.0020 mL, 0.014 mmol, internal standard). Silver trifluoroacetate (0.008 g, 0.004 mmol) was added and the unpurified reaction mixture was analyzed by NMR spectroscopy after 20 min. Silacyclopropanes **27** were formed in 95% yield as a 74:26 mixture of diastereomers based on comparison of the standard peak (δ 6.71) and the silacyclopropane protons: ^{1}H NMR (600 MHz, C_6D_6) δ 7.54–7.52 (m, 2H), 7.44–7.43 (m, 0.7H), 7.28–7.26 (m,

2H), 7.21–7.19 (m, 3H), 7.12–7.05 (m, 4.35H), 7.01–6.96 (m, 1.35H), 1.91–1.87 (m, 0.35H), 1.76 (ddd, *J* = 10.1, 9.0, 6.2, 1H), 1.41–1.37 (m, 2.35H), 1.35–1.33 (m, 0.7H), 1.23 (dd, *J* = 6.2, 4.7, 1H), 1.18 (s, 12.2H and m, 0.35H), 0.92–0.88 (m, 1H), 0.86 (s, 3.2H), 0.84 (s, 9H), 0.65 (dd, *J* = 12.2, 11.1, 0.35H), 0.51 (dd, *J* = 10.9, 8.9, 1H), 0.44–0.40 (m, 0.35H), 0.31 (ddd, *J* = 12.1, 10.1, 8.9, 1H), 0.18–0.13 (m, 0.35H); ^{13}C NMR (150 MHz, C_6D_6, diagnostic peaks) δ 148.7 (C), 148.4 (C), 143.1 (C), 143.0 (C), 131.9 (CH), 131.7 (CH), 128.90 (CH), 128.85 (CH), 128.7 (CH), 128.6 (CH), 128.3 (CH), 127.8 (CH), 126.9 (CH), 126.7 (CH), 126.1 (CH), 126.0 (CH), 39.1 (C), 36.8 (C), 31.5 (CH), 31.20 (CH_3), 31.18 (CH_3), 28.6 (CH_3), 27.9 (CH_3), 24.9 (CH_2), 24.0 (CH_2), 19.3 (C), 19.2 (C), 18.6 (C), 18.5 (C), 16.8 (CH), 15.6 (CH), 4.9 (CH_2), 3.0 (CH_2).



Silacyclopropanes 28. To a solution of alkene **S5** (0.100 mL, 740 mM in C_6D_6, 0.0740 mmol), cyclohexene silacyclopropane **13** (0.120 mL, 1.05 M in C_6D_6, 0.126 mmol) and mesitylene (0.0020 mL, 0.014 mmol, internal standard) in C_6D_6 (0.260 mL) in a J. Young NMR tube was added a solution of AgO_2CCF_3 (0.0015 mL, 0.050 mM, 0.00074 mmol). After 15 min, the ^{1}H NMR spectrum was recorded. Silacyclopropanes **28** were formed in 70% as a 50:50 mixture of diastereomers based on comparison of the standard peak (δ 6.72) and the methine protons of the silacyclopropane ring: ^{1}H NMR (600 MHz, C_6D_6) δ 7.15–7.12 (m, 4H), 7.04–7.00 (m, 4H), 6.99–6.97 (m, 2H), 1.89–1.84 (m, 1H), 1.75–1.72 (m, 1H), 1.30–1.22 (m, 3H), 1.11 (s, 9H), 1.09 (s, 9H), 1.00 (s, 18H and m, 1H), 0.89–0.82 (m, 4H), 0.75 (dd, *J* = 12.3, 10.8, 1H), 0.64 (dt, *J* = 12.2, 8.3, 1H), 0.40 (dd, *J* = 10.9, 8.7, 1H), 0.37–0.33 (m, 1H); ^{13}C NMR (150 MHz, C_6D_6) δ 145.0 (C), 144.8 (C), 128.92 (CH), 128.90 (CH), 126.2 (CH), 126.0 (CH), 125.72 (CH), 125.66

(CH), 31.05 (CH_3), 30.95 (CH_3), 30.12 (CH_3), 30.09 (CH_3), 27.4 (CH), 27.2 (CH), 27.0 (CH), 26.7 (CH), 19.8 (CH_2), 19.5 (CH_2), 19.2 (C), 18.9 (C), 18.59 (C), 18.55 (C), 18.3 (CH), 17.0 (CH), 3.3 (CH_2), 2.1 (CH_2).



S6 **36**

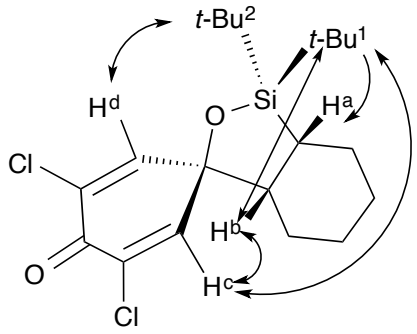
Dienone 36. To a solution of 3,3-dimethylcyclohexene-1-one (0.7 mL, 5 mmol) in *p*-dioxane (50 mL) were added DDQ (1.4 g, 6.2 mmol) and TsOH (1.0 g, 5.3 mmol) in a Schlenk tube. The reaction mixture was degassed (freeze-pump-thaw, 3 cycles) and heated to 100 °C. After 48 h, the reaction mixture was brought to room temperature. Diethyl ether (50 mL) was added and the organic layer was washed with 10% aqueous NaOH (3 x 25 mL), H_2O (3 x 25 mL), and brine (50 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Excess *p*-dioxane was removed as an azeotrope with ethanol to provide dienone **36** as an orange oil that was used without further purification (0.36 g, 56%). The spectral data are consistent with the data reported:[10,11] ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.82 (d, J = 10.0, 2H), 6.18 (d, J = 10.0, 2H), 1.25 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 186.1, 156.9, 127.5, 38.1, 26.9; HRMS (TOF MS ES+) m / z calcd for $C_8H_{11}O$ $(M+H)^+$ 123.0804, found 123.0806.

III. Stereochemical Proof

A. General Procedure for 1-D NOESY experiment

All NOE data were collected on degassed acetone-d_6 samples with a mixing time of 0.30 seconds. All peaks in the ^{1}H NMR spectra were assigned using a combination of ^{1}H NMR chemical shifts, ^{1}H/^{1}H COSY, ^{13}C NMR chemical shifts, and ^{1}H/^{13}C HSQC experiments.

B. **NOE Data**



Relevant NOE data for oxasilacyclopentane 16 (acetone-d_6)

t-Bu^1 irradiated: H^a (0.1%), H^b (0.3%), H^c (0.1%)

t-Bu^2 irradiated: H^d (0.2%)

H^b irradiated: *t*-Bu^1 (2.4%), H^c (0.2%)

H^c irradiated: *t*-Bu^1 (1.2%), H^b (1.4%)

H^d irradiated: *t*-Bu^2 (1.8%)

IV. **References**

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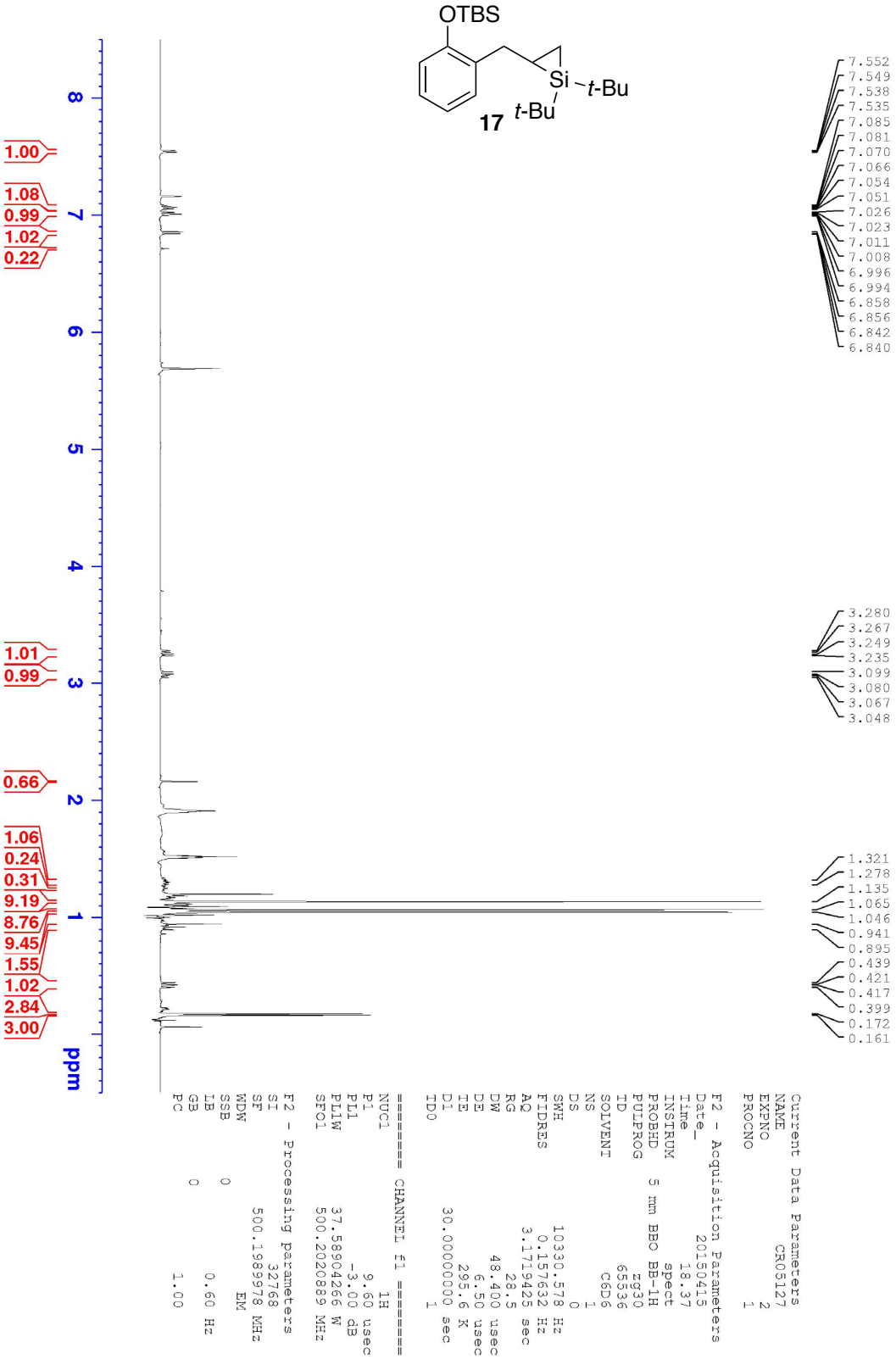
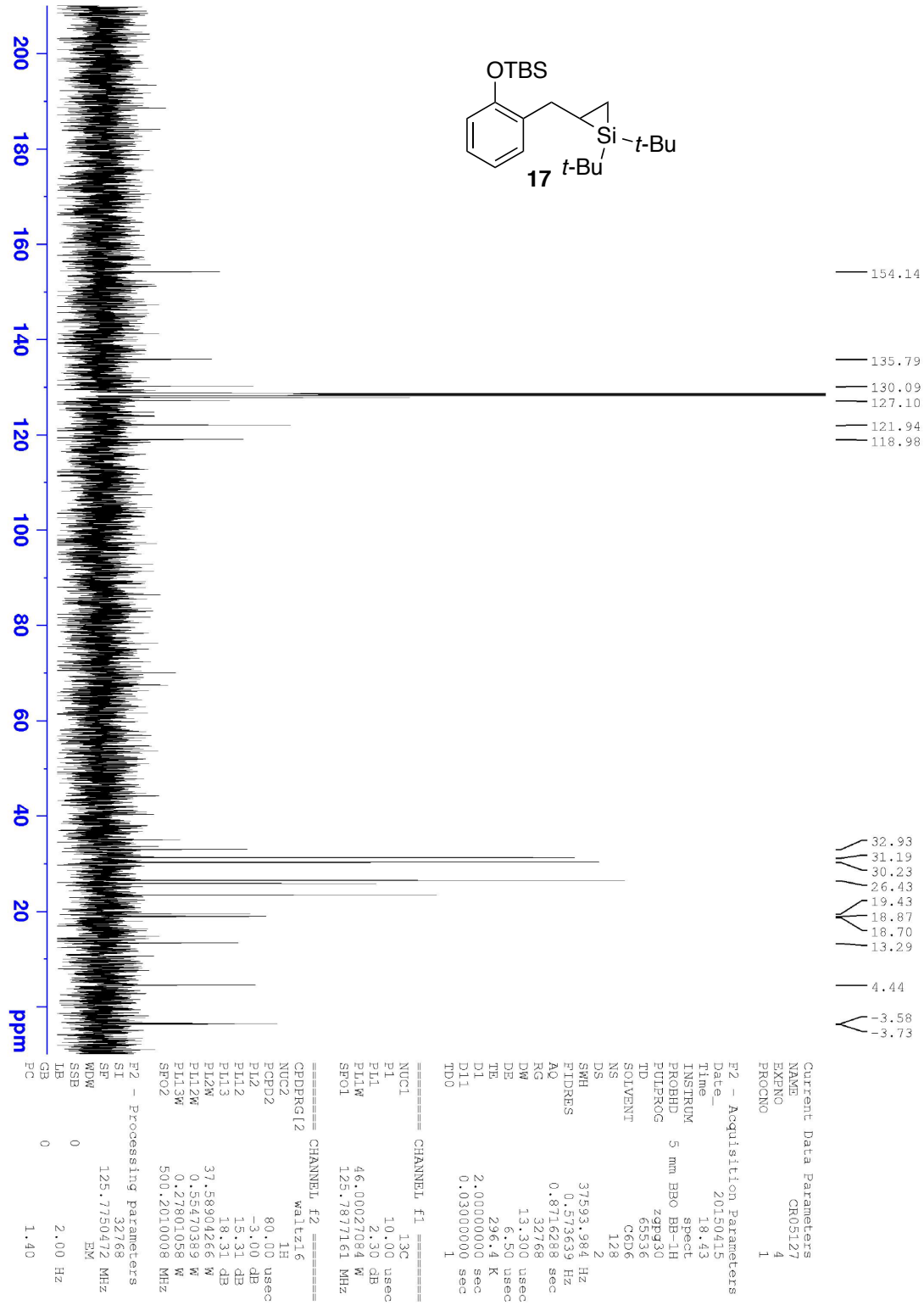
V. Selected Spectra

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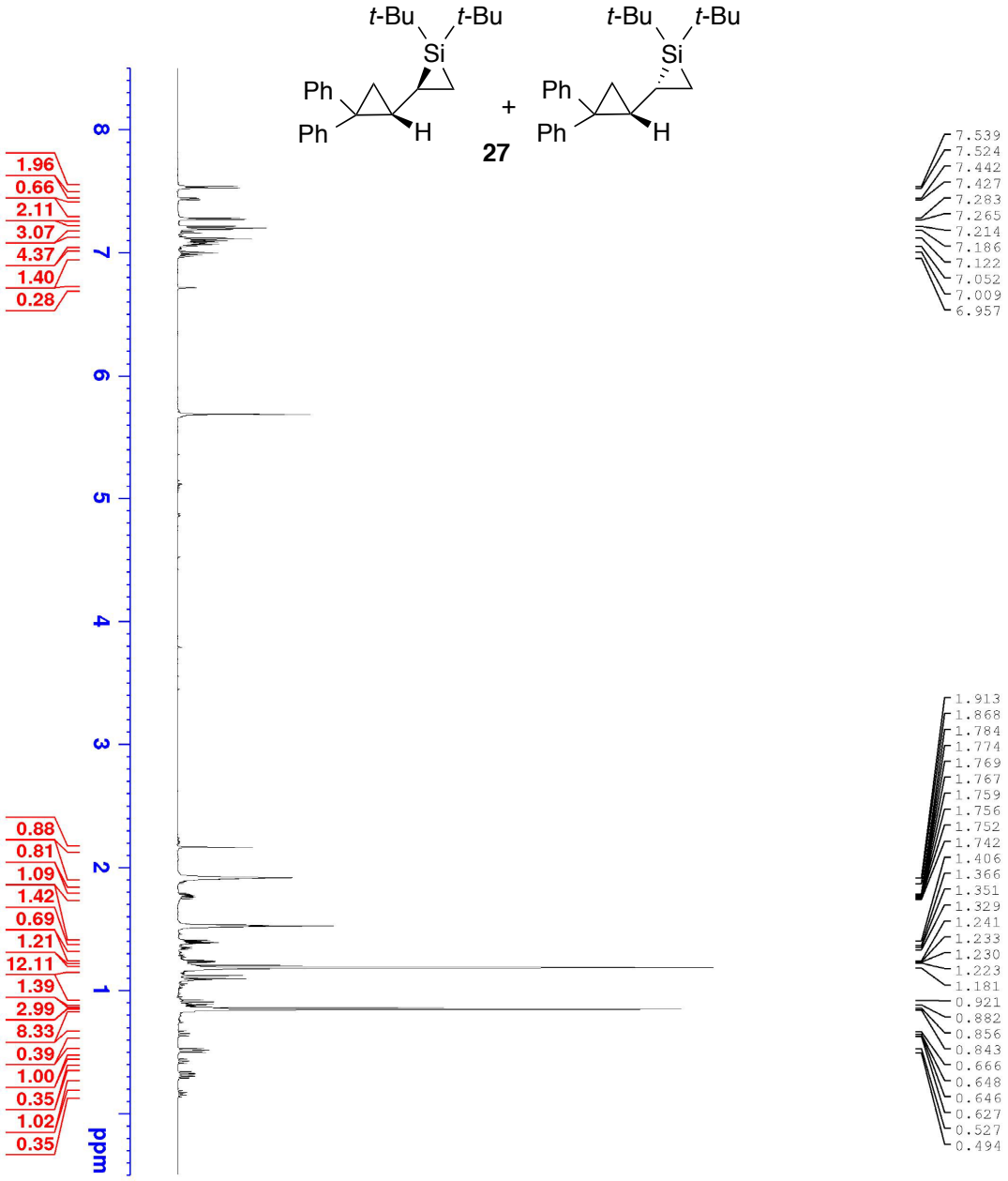
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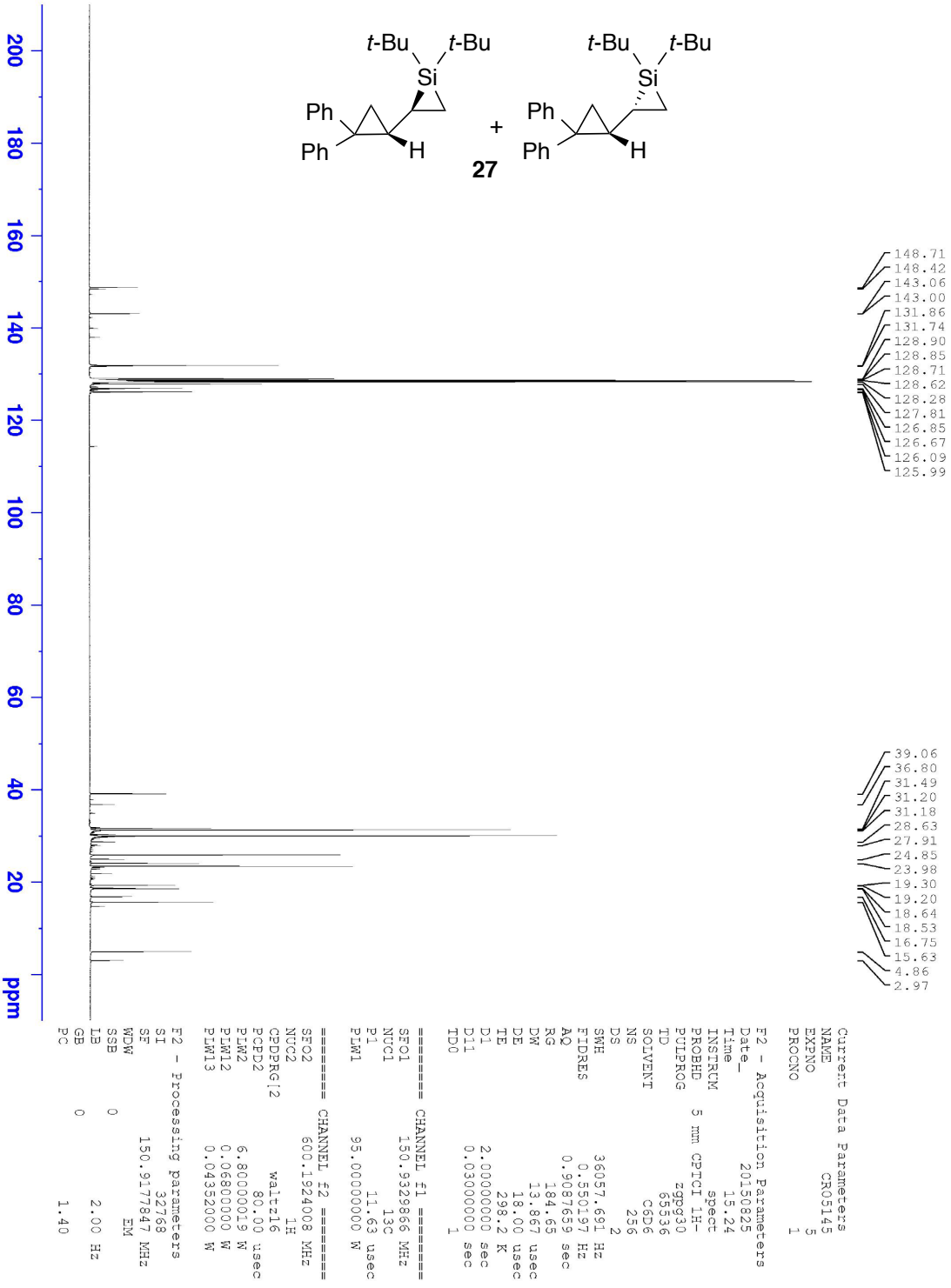
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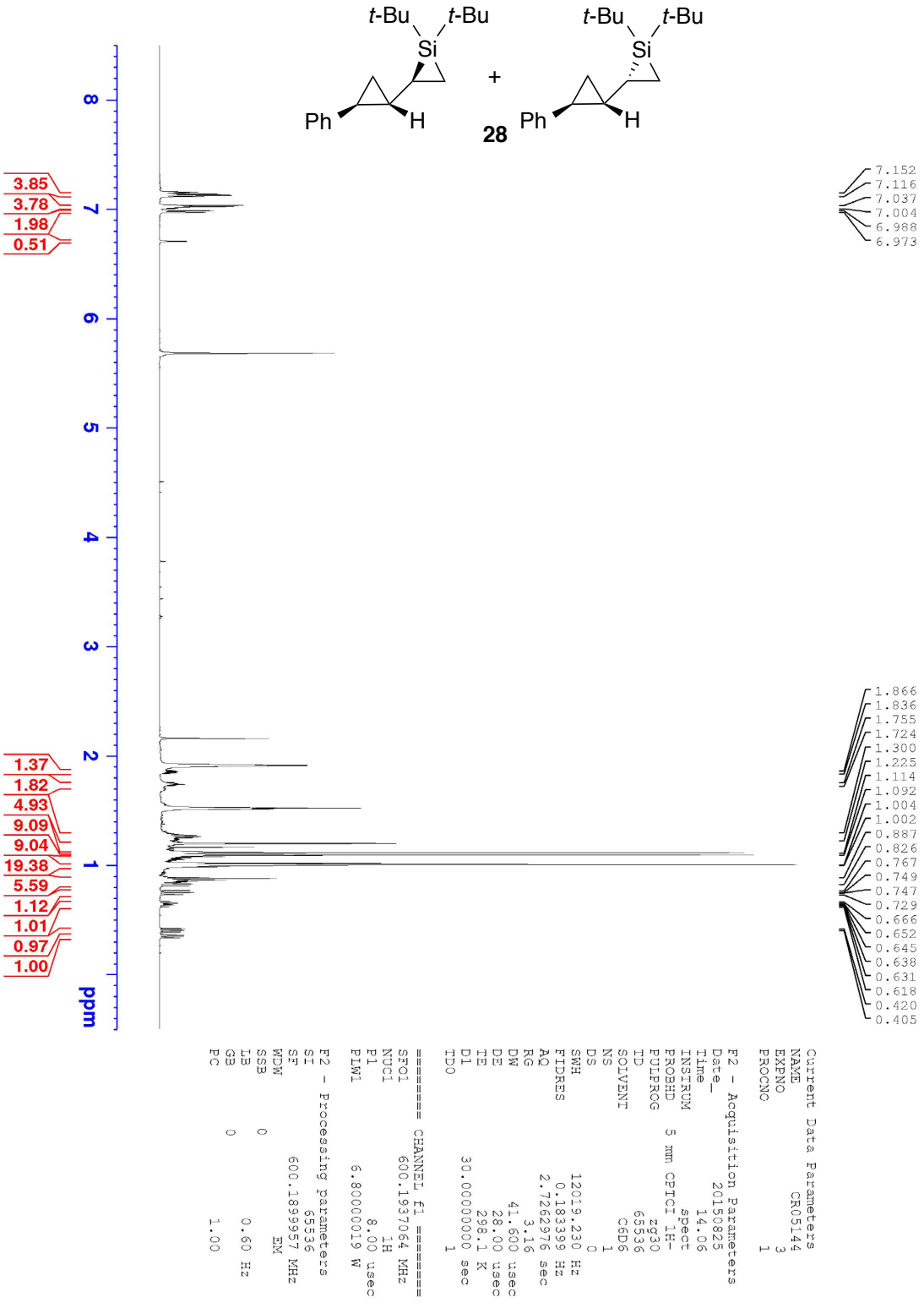
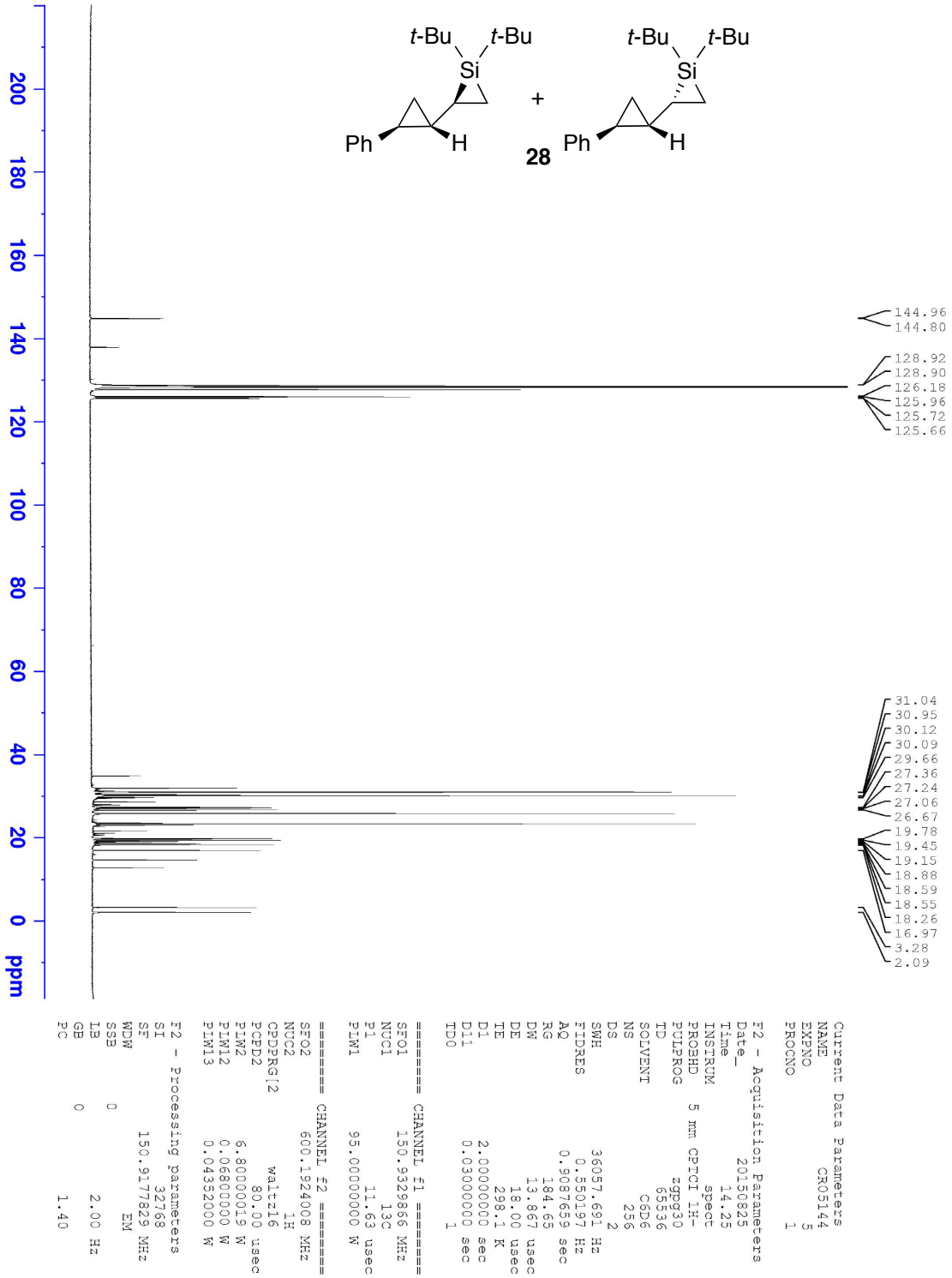
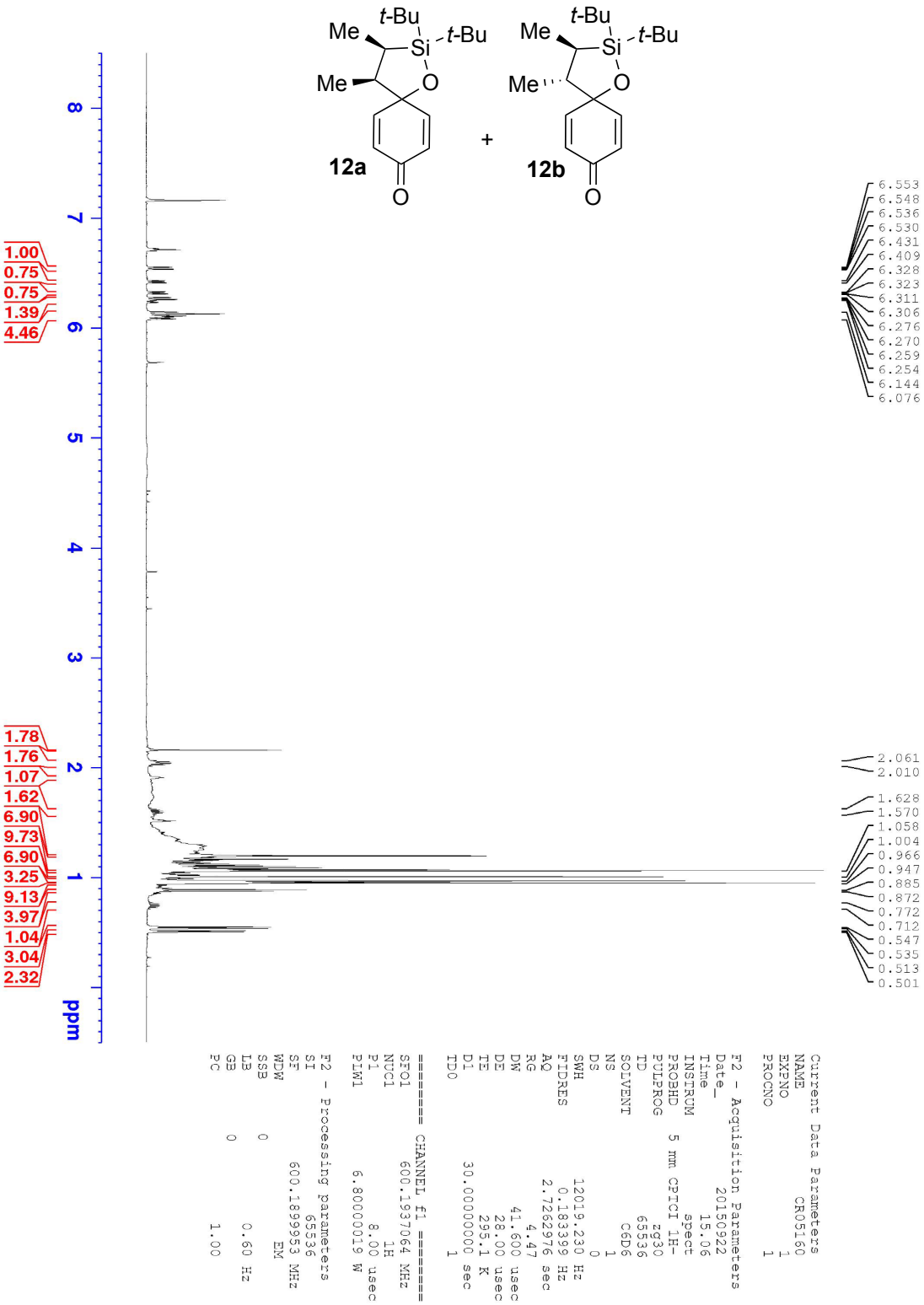
GB 0
PC 1.40

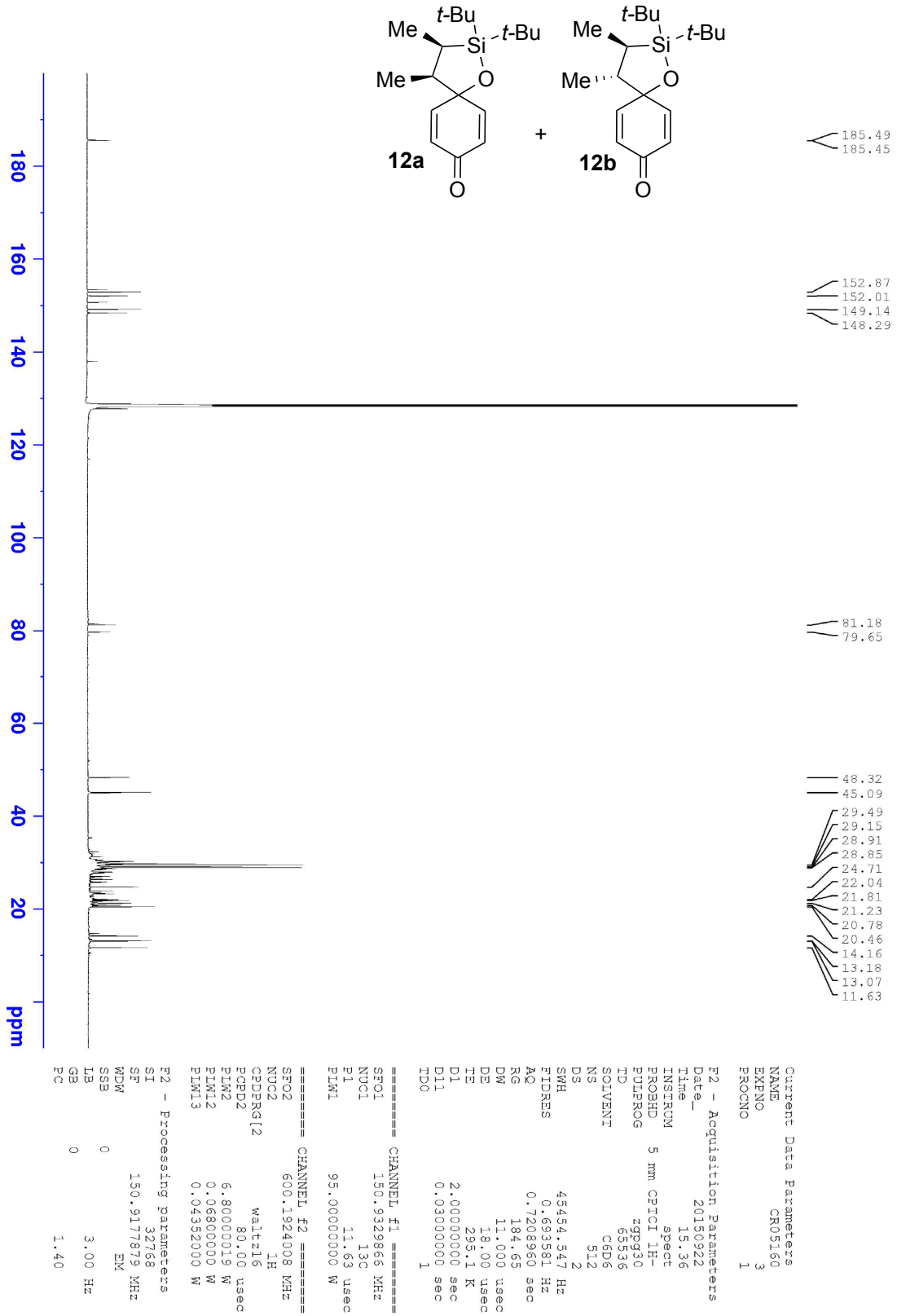

t-Bu
t-Bu
Si
Ph
H
+
28
3.85
3.78
1.98
0.51
1.37
1.82
4.93
9.09
9.04
19.38
5.59
1.12
1.01
0.97
1.00
ppm
7.152
7.116
7.037
7.004
6.988
6.973
1.866
1.836
1.755
1.724
1.300
1.225
1.114
1.092
1.004
1.002
0.887
0.826
0.767
0.749
0.747
0.729
0.666
0.652
0.645
0.638
0.631
0.618
0.420
0.405
Current Data Parameters
NAME CR05144
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150825
Time 14.06
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 3.16
DW 41.600 usec
DE 28.00 usec
TE 298.1 K
D1 30.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1937064 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.80000019 W
F2 - Processing parameters
SI 65536
SF 600.1899957 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

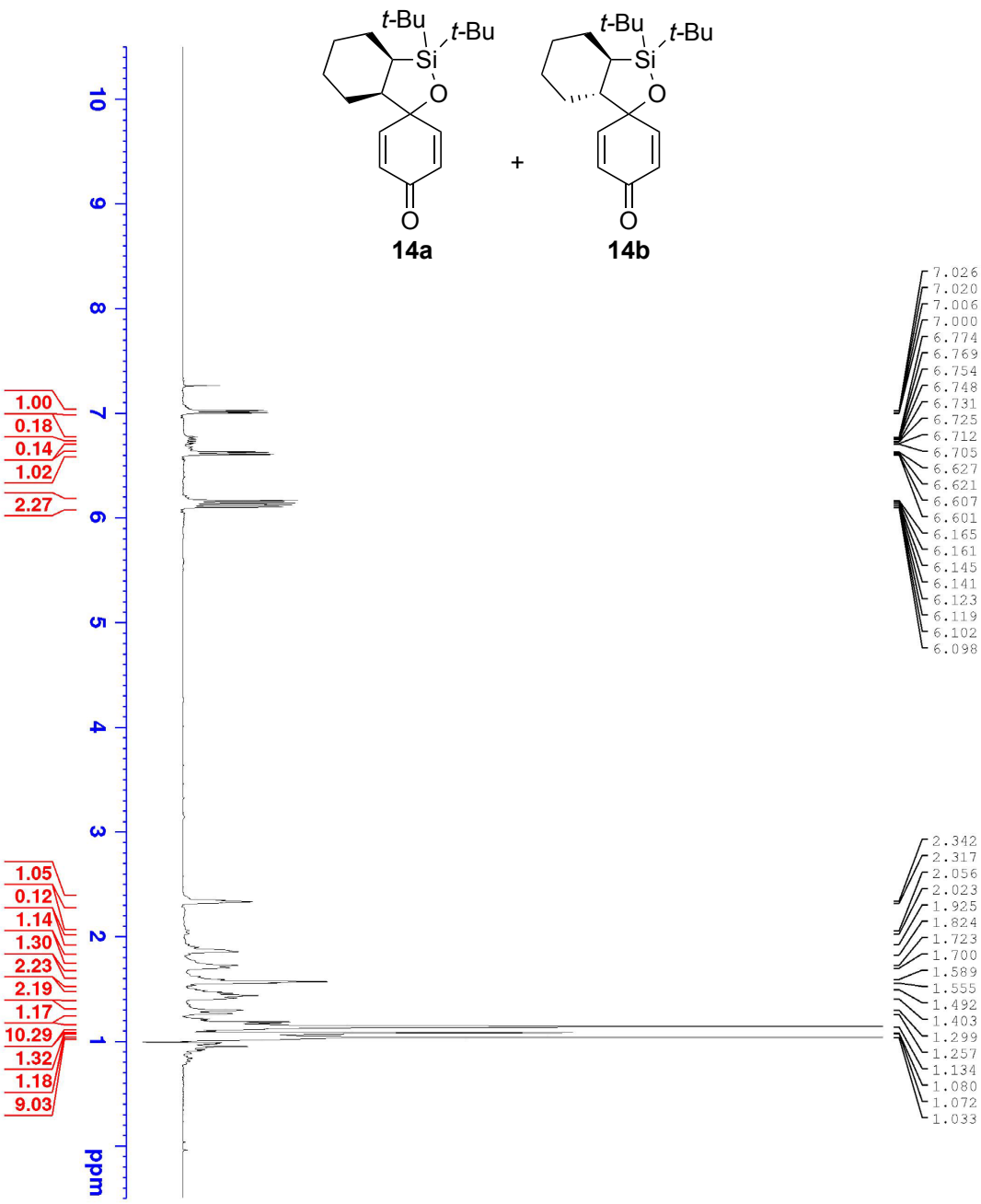


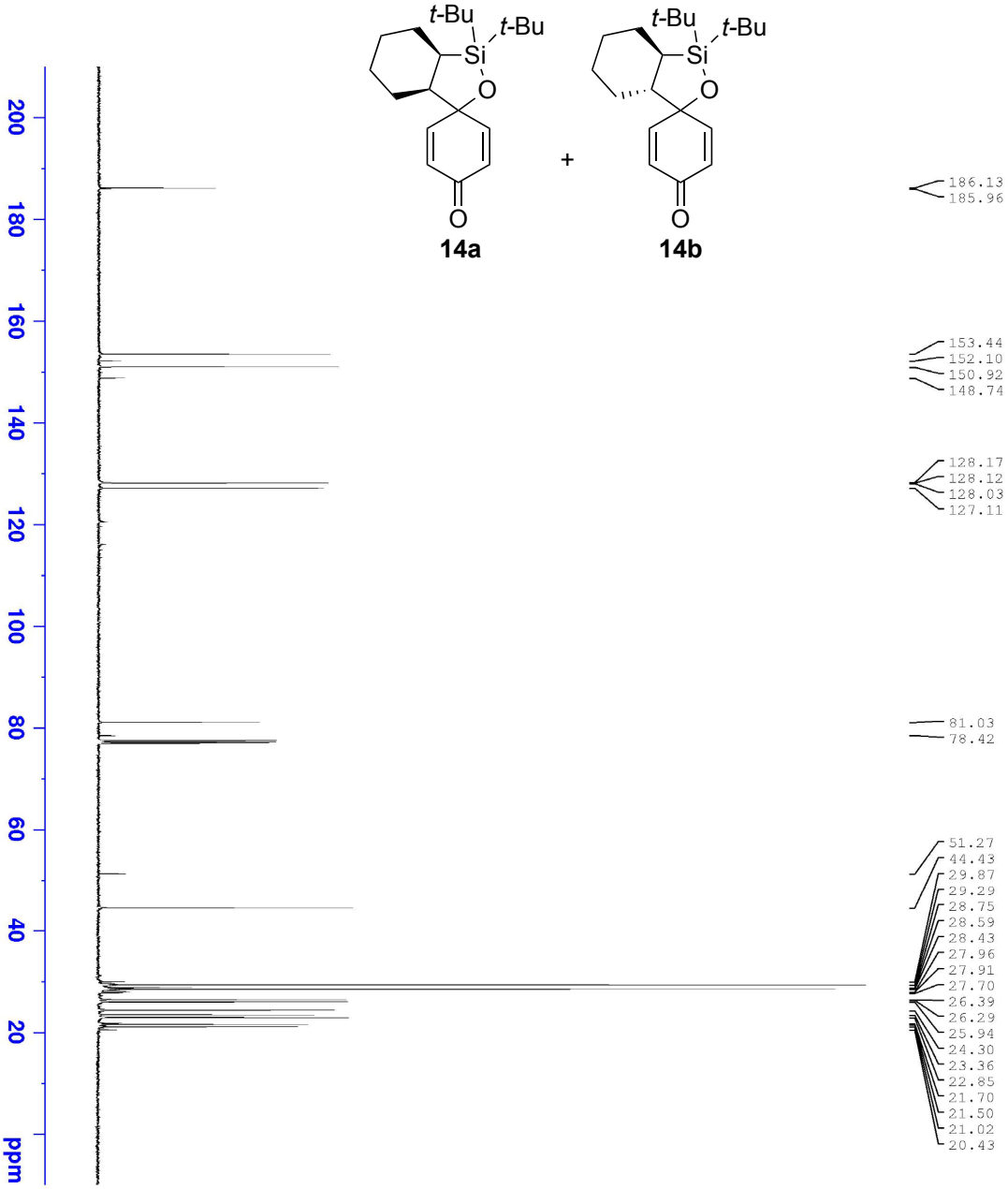

```
Current Data Parameters
NAME                   CR05144
EXPNO                        5
PROCNO                       1

F2 - Acquisition Parameters
Date_               20150825
Time                   14.25
INSTRUM                spect
PROBHD     5 mm CPTCI 1H-
PULPROG                zgpg30
TD                     65536
SOLVENT                C6D6
NS                       256
DS                         2
SWH                36057.691 Hz
FIDRES                 0.550197 Hz
AQ                 0.9087659 sec
RG                     184.65
DW                     13.867 usec
DE                     18.00 usec
TE                     298.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               150.9329866 MHz
NUC1                       13C
P1                       11.63 usec
PL1                95.00000000 W

======== CHANNEL f2 ========
SFO2               600.1924008 MHz
NUC2                       1H
CPDPRG[2                   waltz16
PCPD2                  80.00 usec
PL2                6.80000019 W
PL12               0.06800000 W
PL13               0.04352000 W

F2 - Processing parameters
SI                      32768
SF                 150.9177829 MHz
WDW                        EM
SSB                 0
LB                      2.00 Hz
GB                  0
PC                      1.40
```



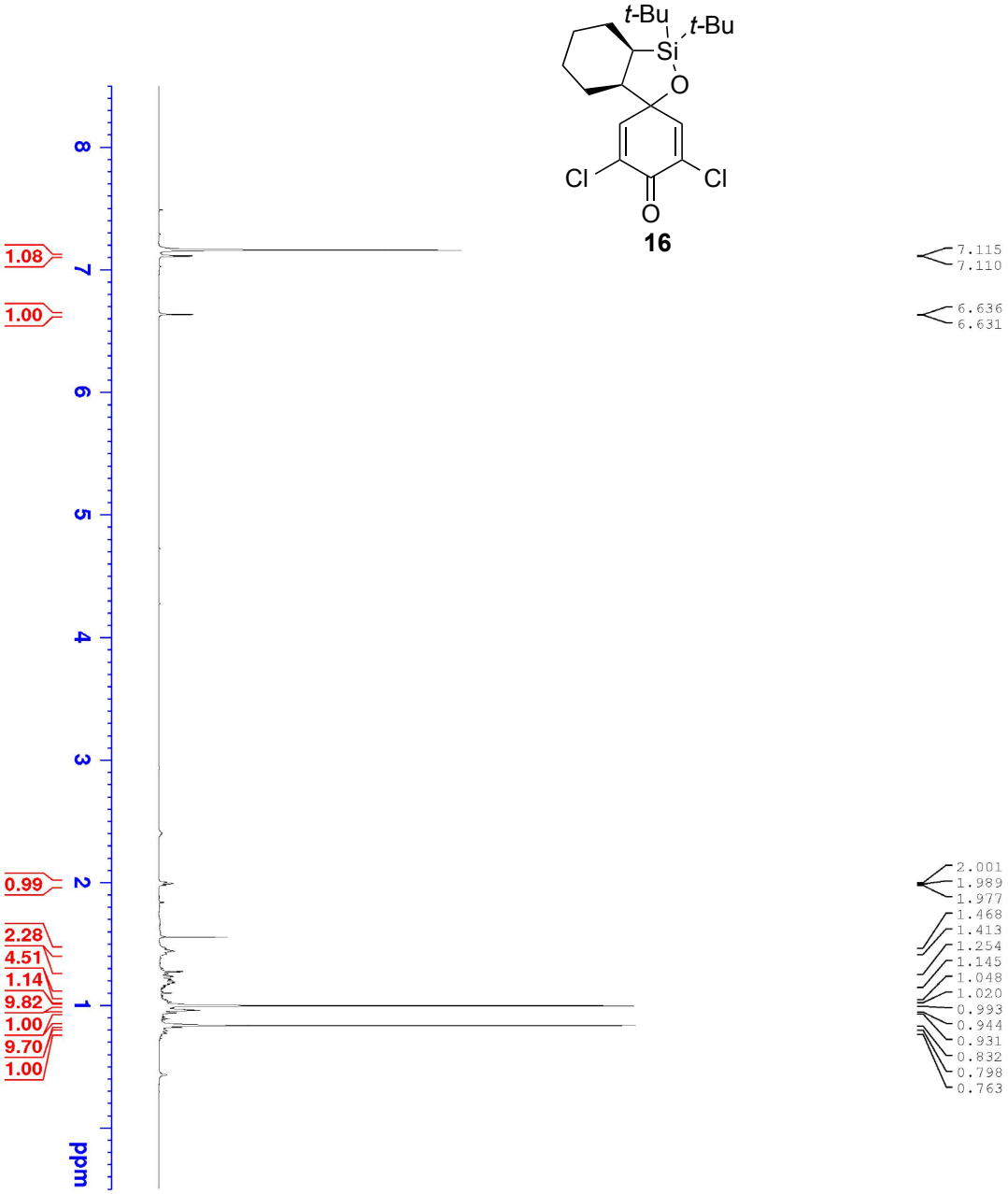

```
Current Data Parameters
NAME                  CR05160
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150922
Time                  15.06
INSTRUM                spect
PROBHD     5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT                C6D6
NS                        1
DS                        0
SWH               12019.230 Hz
FIDRES                0.183399 Hz
AQ                2.7262976 sec
RG                       4.47
DW                   41.600 usec
DE                   28.00 usec
TE                   295.1 K
D1                30.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            600.1937064 MHz
NUC1                      1H
P1                   8.00 usec
PLW1             6.80000019 W

F2 - Processing parameters
SI                    65536
SF              600.1899953 MHz
WDW                      EM
SSB                       0
LB                     0.60 Hz
GB                        0
PC                     1.00
```

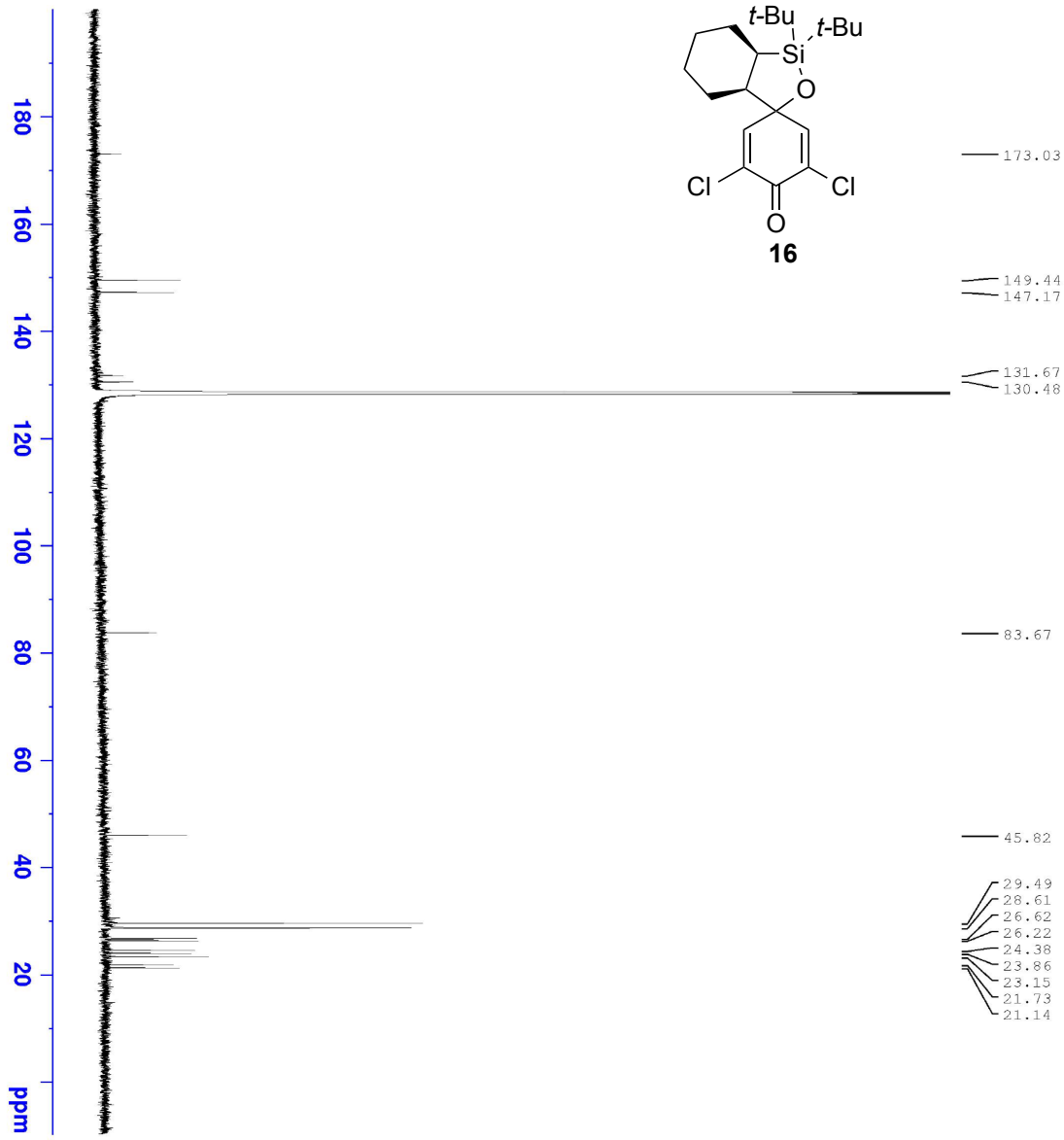

t-Bu
Me
Si
t-Bu
Me
O
12a
+
12b
185.49
185.45
152.87
152.01
149.14
148.29
81.18
79.65
48.32
45.09
29.49
29.15
28.91
28.85
24.71
22.04
21.81
21.23
20.78
20.46
14.16
13.18
13.07
11.63
ppm
Current Data Parameters
NAME CR05160
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150922
Time 15.36
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 512
DS 2
SWH 45454.547 Hz
FIDRES 0.693581 Hz
AQ 0.7208960 sec
RG 184.65
DW 11.000 usec
DE 18.00 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9329866 MHz
NUC1 13C
P1 11.63 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 600.1924008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 6.80000019 W
PLW12 0.06800000 W
PLW13 0.04352000 W
F2 - Processing parameters
SI 32768
SF 150.9177879 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40


t-Bu
t-Bu
Si
O
O
14a
+
t-Bu
t-Bu
Si
O
O
14b
1.00
0.18
0.14
1.02
2.27
1.05
0.12
1.14
1.30
2.23
2.19
1.17
10.29
1.32
1.18
9.03
ppm
7.026
7.020
7.006
7.000
6.774
6.769
6.754
6.748
6.731
6.725
6.712
6.705
6.627
6.621
6.607
6.601
6.165
6.161
6.145
6.141
6.123
6.119
6.102
6.098
2.342
2.317
2.056
2.023
1.925
1.824
1.723
1.700
1.589
1.555
1.492
1.403
1.299
1.257
1.134
1.080
1.072
1.033
Current Data Parameters
NAME CR0594
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150307
Time 14.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 4
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.17719425 sec
RG 22.6
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.60 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020989 MHz
F2 - Processing parameters
SI 32768
SF 500.1989743 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

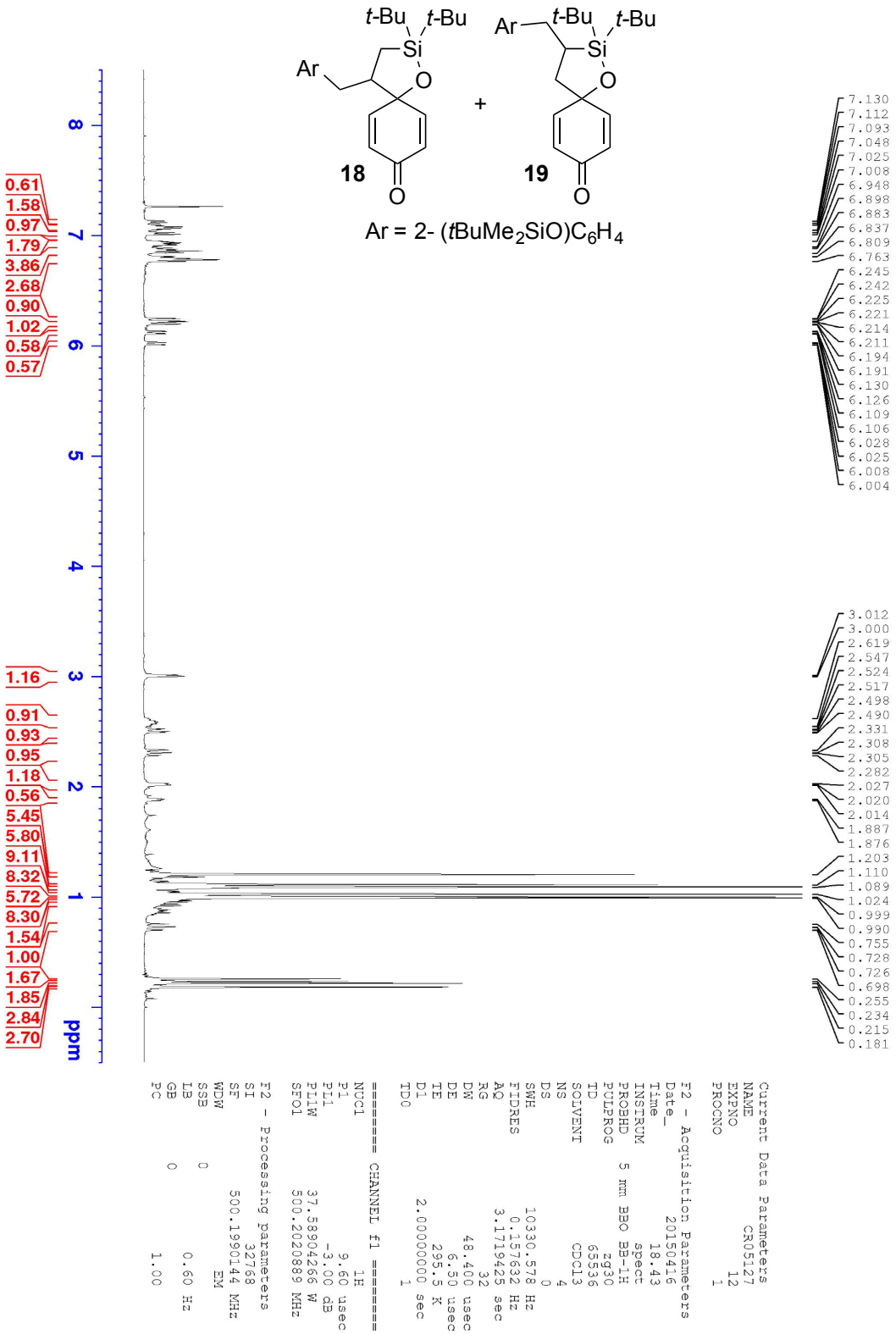


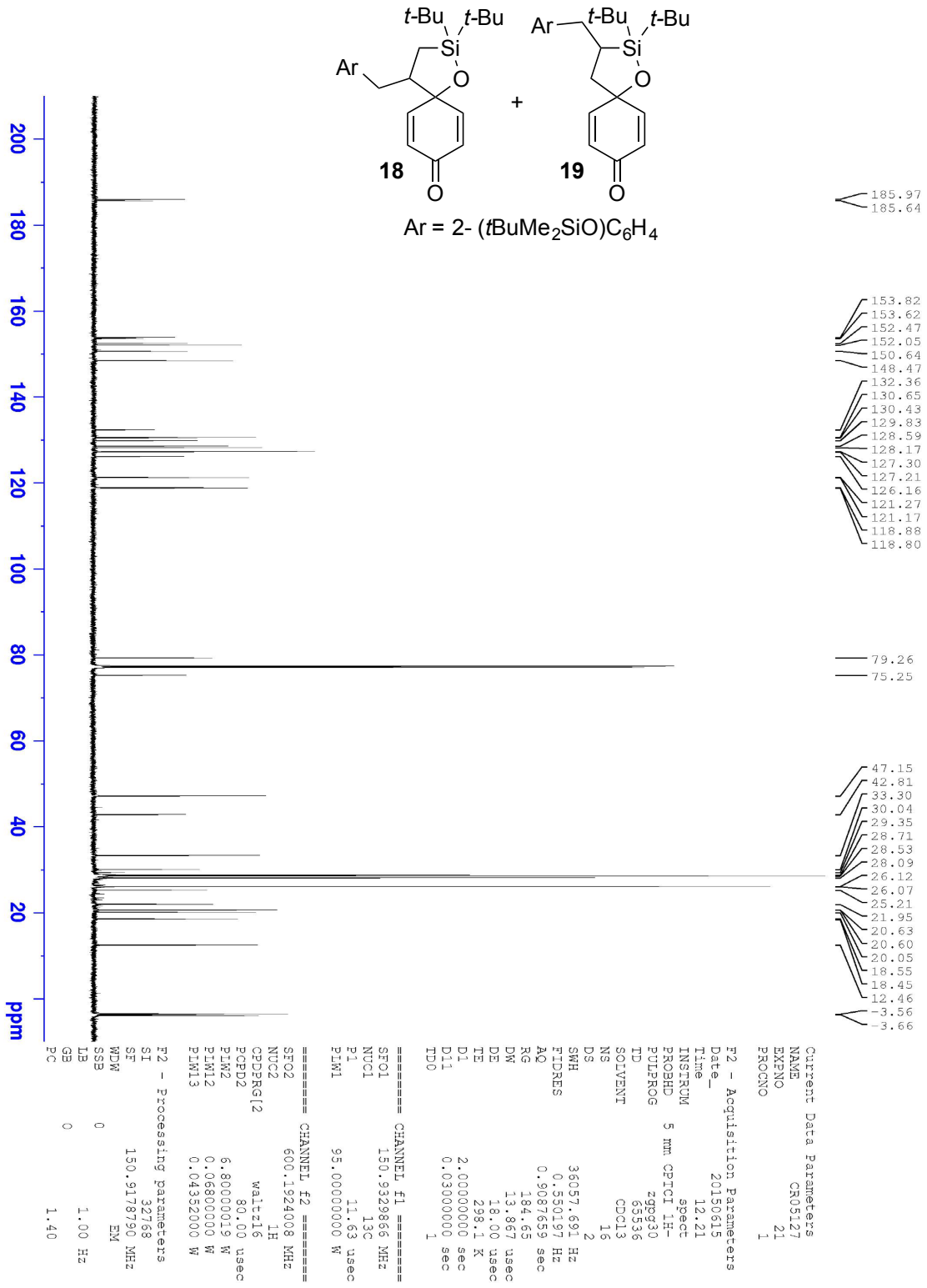

```
Current Data Parameters
NAME                  CR0594
EXPNO                    101
PROCNO                     1

F2 - Acquisition Parameters
Date_                20150307
Time                   14.17
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG                zgpg30
TD                      65536
SOLVENT                 C6D6
NS                        256
DS                          2
SWH                37593.984 Hz
FIDRES                 0.573639 Hz
AQ                    0.8716288 sec
RG                       5160.6
DW                       13.300 usec
DE                        6.50 usec
TE                       298.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.00 usec
PL1                       2.30 dB
PL1W               46.00027084 W
SFO1               125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                    -3.00 dB
PL12                   15.31 dB
PL13                   18.31 dB
PL2W               37.58904266 W
PL12W               0.55470389 W
PL13W               0.27801058 W
SFO2               500.2010008 MHz

F2 - Processing parameters
SI                         32768
SF                 125.7751189 MHz
WDW                           EM
SSB                            0
LB                          2.00 Hz
GB                             0
PC                          1.40
```

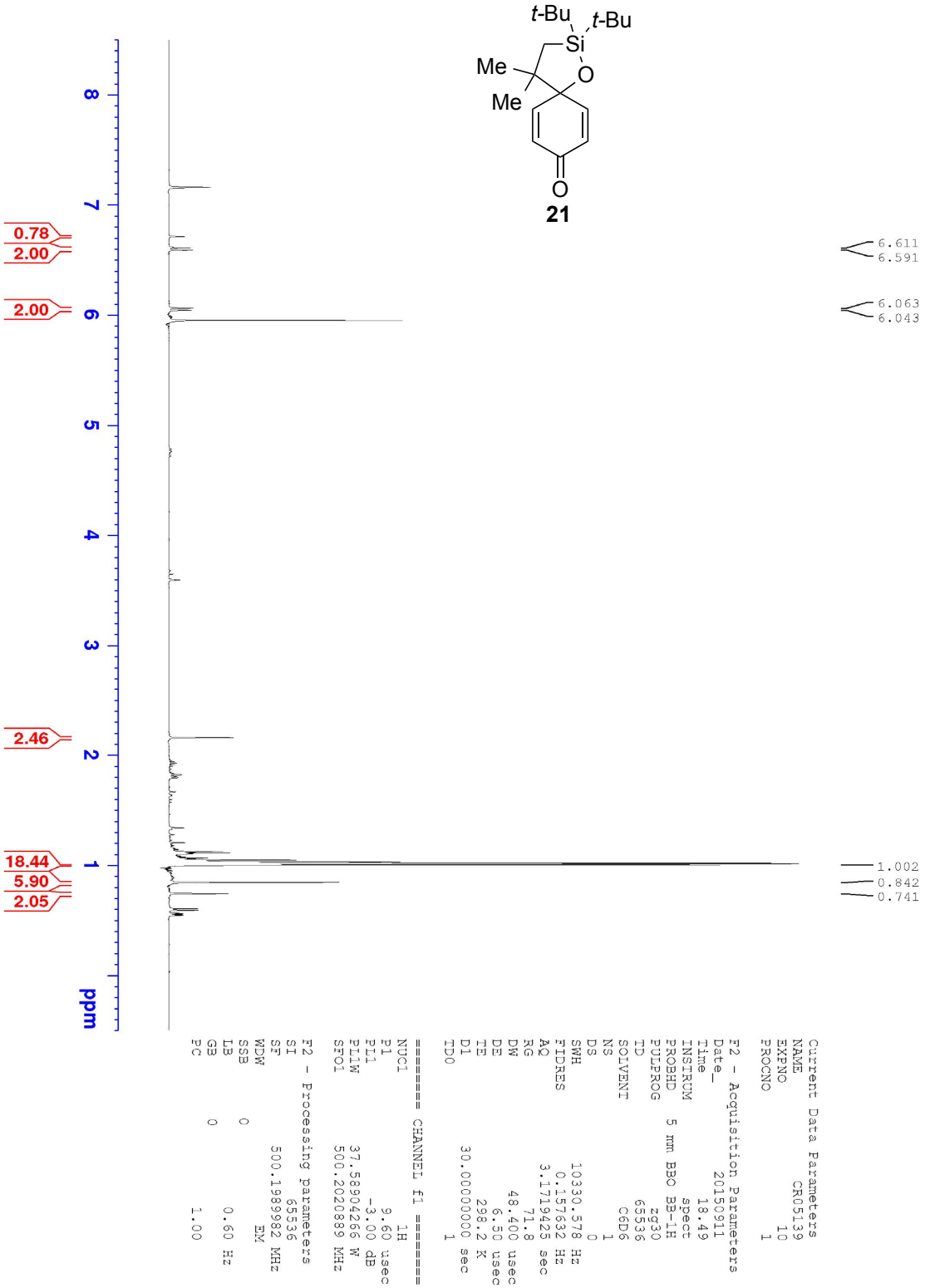

t-Bu
t-Bu
Si
O
Cl
Cl
O
16
7.115
7.110
6.636
6.631
2.001
1.989
1.977
1.468
1.413
1.254
1.145
1.048
1.020
0.993
0.944
0.931
0.832
0.798
0.763
1.08
1.00
0.99
2.28
4.51
1.14
9.82
1.00
9.70
1.00
ppm
Current Data Parameters
NAME CR0517
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150921
Time 17.46
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 4
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 33.59
DW 41.600 usec
DE 28.00 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 600.1937064 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.80000019 W
F2 - Processing parameters
SI 65536
SF 600.1899954 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

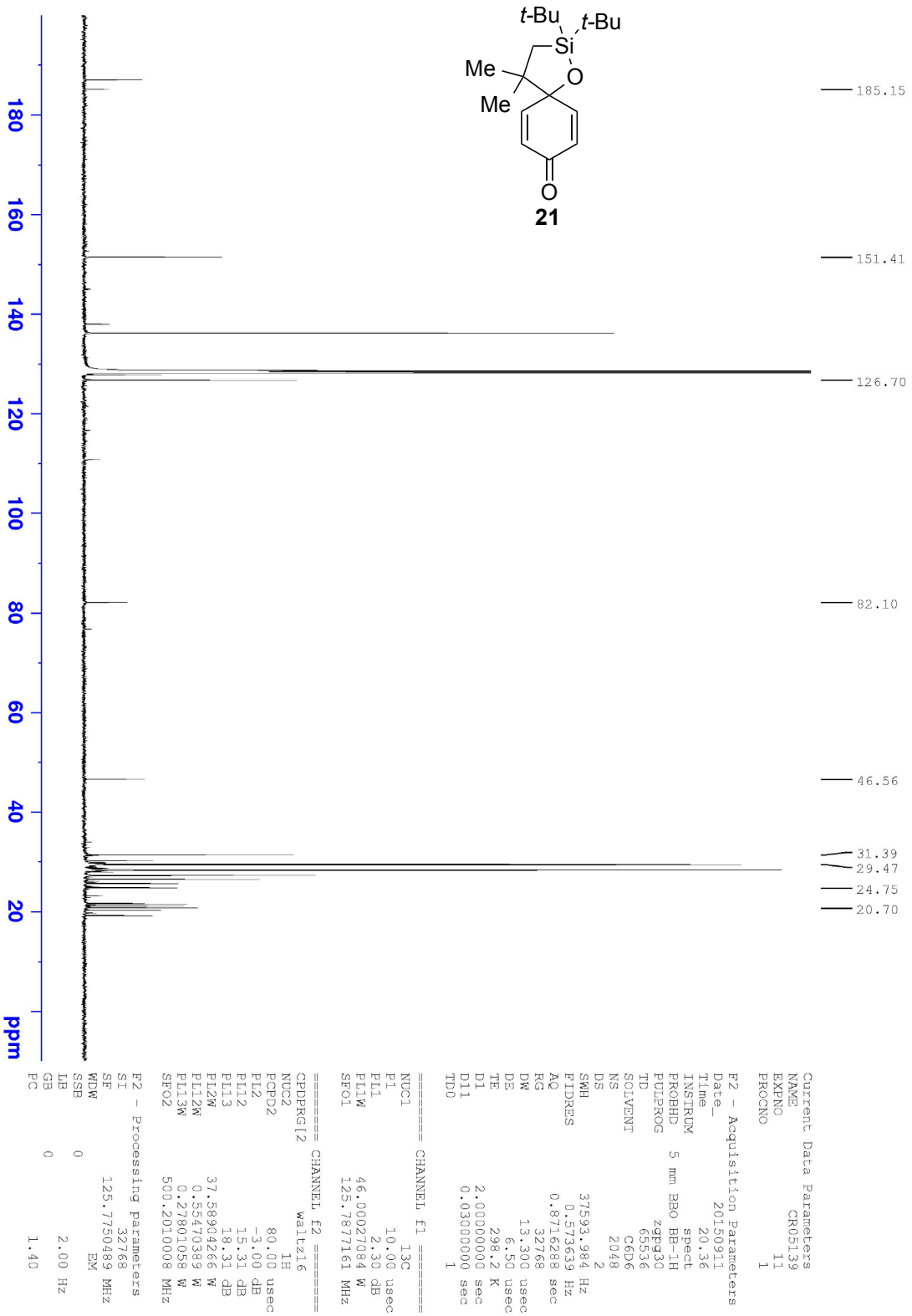


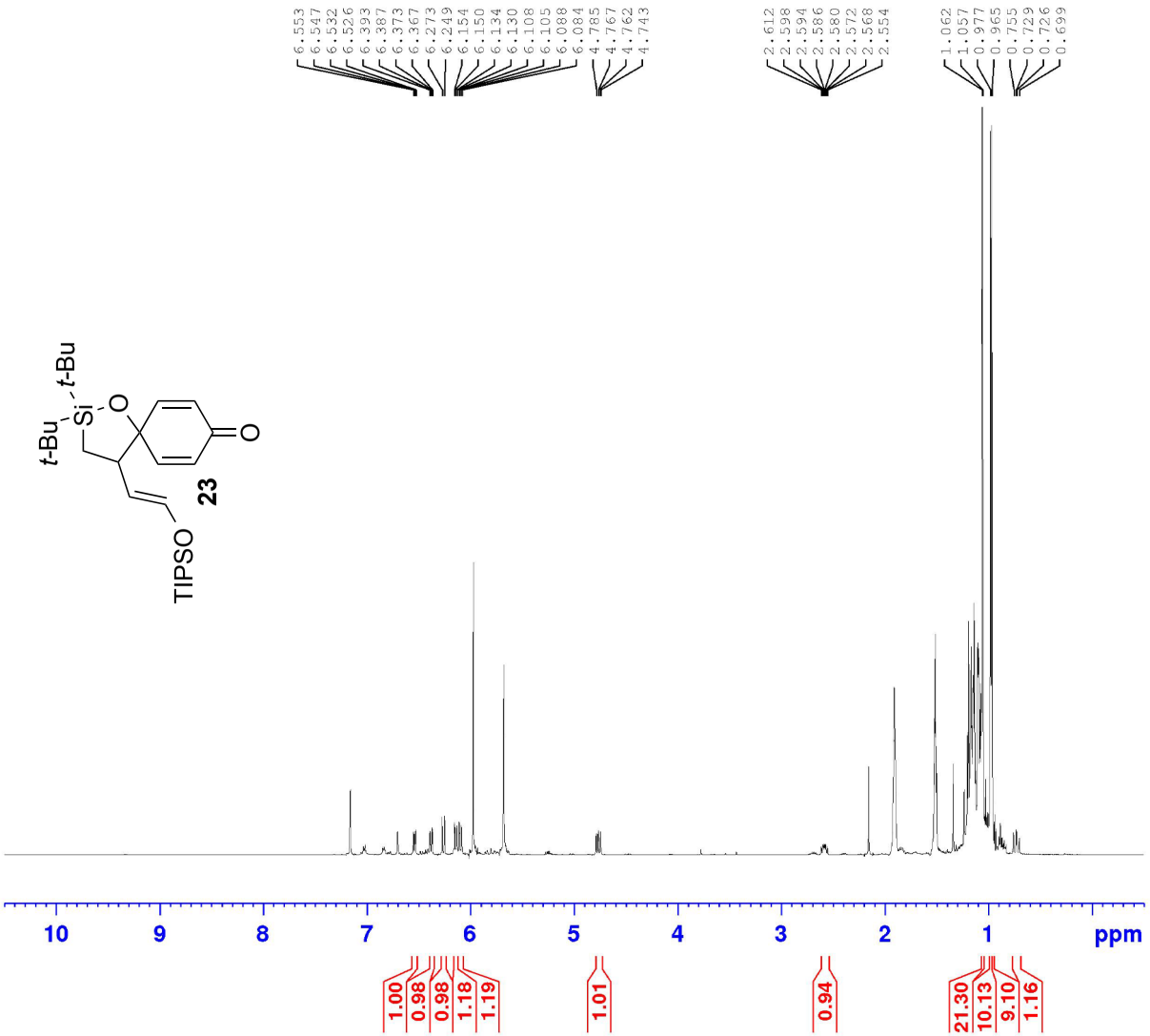

```
Current Data Parameters
NAME                  CR05157
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20150921
Time_                   17.48
INSTRUM                 spect
PROBHD     5 mm CPTCI 1H-
PULPROG             zgpg30
TD                   65536
SOLVENT                C6D6
NS                       64
DS                        2
SWH                45454.547 Hz
FIDRES                0.693581 Hz
AQ                 0.7208960 sec
RG                     184.65
DW                  11.000 usec
DE                  18.00 usec
TE                    298.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

========== CHANNEL f1 ========
SFO1               150.9329866 MHz
NUC1                        13C
P1                    11.63 usec
PLW1           95.00000000 W

========== CHANNEL f2 ========
SFO2               600.1924008 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                 80.00 usec
PLW2           6.80000019 W
PLW12          0.06800000 W
PLW13          0.04352000 W

F2 - Processing parameters
SI                       32768
SF                 150.9117893 MHz
WDW                        EM
SSB                         0
LB                       2.00 Hz
GB                          0
PC                       1.40
```


t-Bu t-Bu
Si
Ar
O
18
O
+
Ar
t-Bu t-Bu
Si
O
19
O
Ar = 2- (tBuMe2SiO)C6H4
7.130
7.112
7.093
7.048
7.025
7.008
6.948
6.898
6.883
6.837
6.809
6.763
6.245
6.242
6.225
6.221
6.214
6.211
6.194
6.191
6.130
6.126
6.109
6.106
6.028
6.025
6.008
6.004
3.012
3.000
2.619
2.547
2.524
2.517
2.498
2.490
2.331
2.308
2.305
2.282
2.027
2.020
2.014
1.887
1.876
1.203
1.110
1.089
1.024
0.999
0.990
0.755
0.728
0.726
0.698
0.255
0.234
0.215
0.181
0.61
1.58
0.97
1.79
3.86
2.68
0.90
1.02
0.58
0.57
1.16
0.91
0.93
0.95
1.18
0.56
5.45
5.80
9.11
8.32
5.72
8.30
1.54
1.00
1.67
1.85
2.84
2.70
ppm
Current Data Parameters
NAME CR05127
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150416
Time 18.43
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 32
DW 48.400 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 9.60 usec
PL1 -3.00 dB
PLIW 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1990144 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

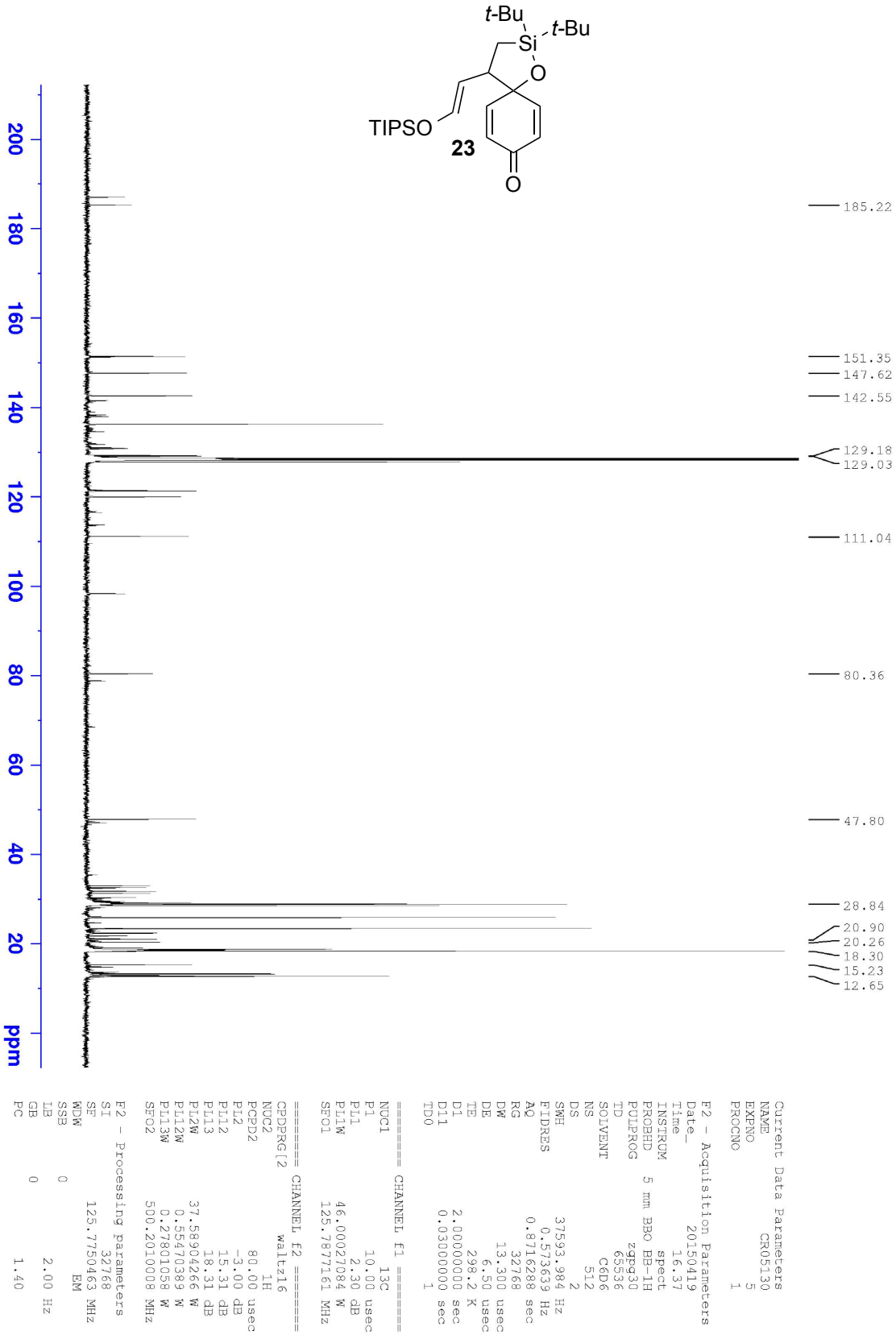

t-Bu
t-Bu
Si
O
Ar
18
19
+
Ar = 2- (tBuMe2SiO)C6H4
185.97
185.64
153.82
153.62
152.47
152.05
150.64
148.47
132.36
130.65
130.43
129.83
128.59
128.17
127.30
127.21
126.16
121.27
121.17
118.88
118.80
79.26
75.25
47.15
42.81
33.30
30.04
29.35
28.71
28.53
28.09
26.12
26.07
25.21
21.95
20.63
20.60
20.05
18.55
18.45
12.46
-3.56
-3.66
ppm


t-Bu
t-Bu
Si
O
Me
Me
O
21
0.78
2.00
2.00
2.46
18.44
5.90
2.05
ppm
6.611
6.591
6.063
6.043
1.002
0.842
0.741
Current Data Parameters
NAME CR05139
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150911
Time_ 18.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 30.0000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.60 usec
PL1 -3.00 dB
PL1W 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 65536
SF 500.1989982 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

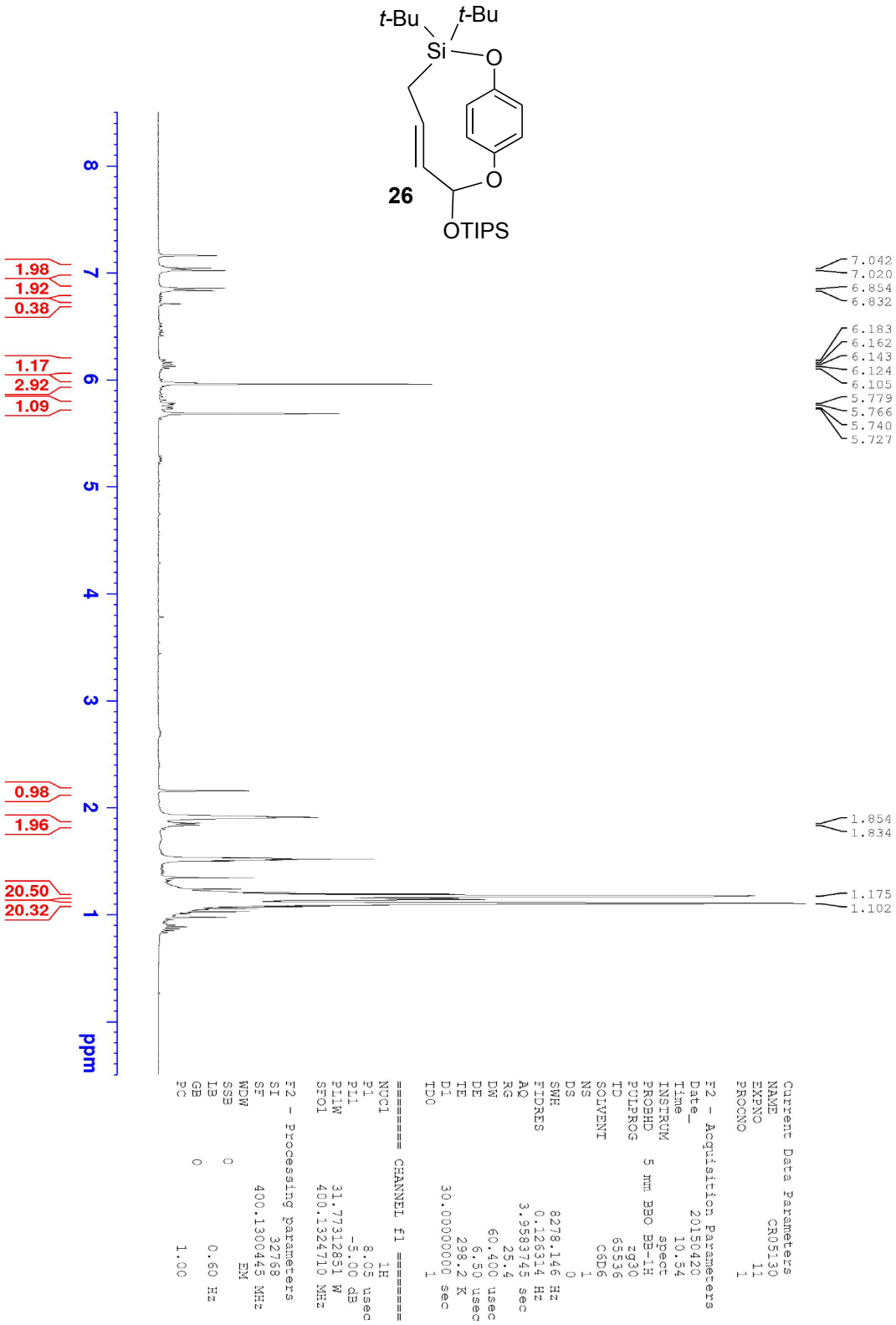

t-Bu
t-Bu
Si
Me
Me
O
O
21
185.15
151.41
126.70
82.10
46.56
31.39
29.47
24.75
20.70
ppm
Current Data Parameters
NAME CR05139
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150911
Time 20.36
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 2048
DS 2
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716288 sec
RG 32768
DW 13.300 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.30 dB
PL1W 46.00027084 W
SFO1 125.7877161 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.31 dB
PL13 18.31 dB
PL2W 37.58904266 W
PL12W 0.55470389 W
PL13W 0.27801058 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7750489 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

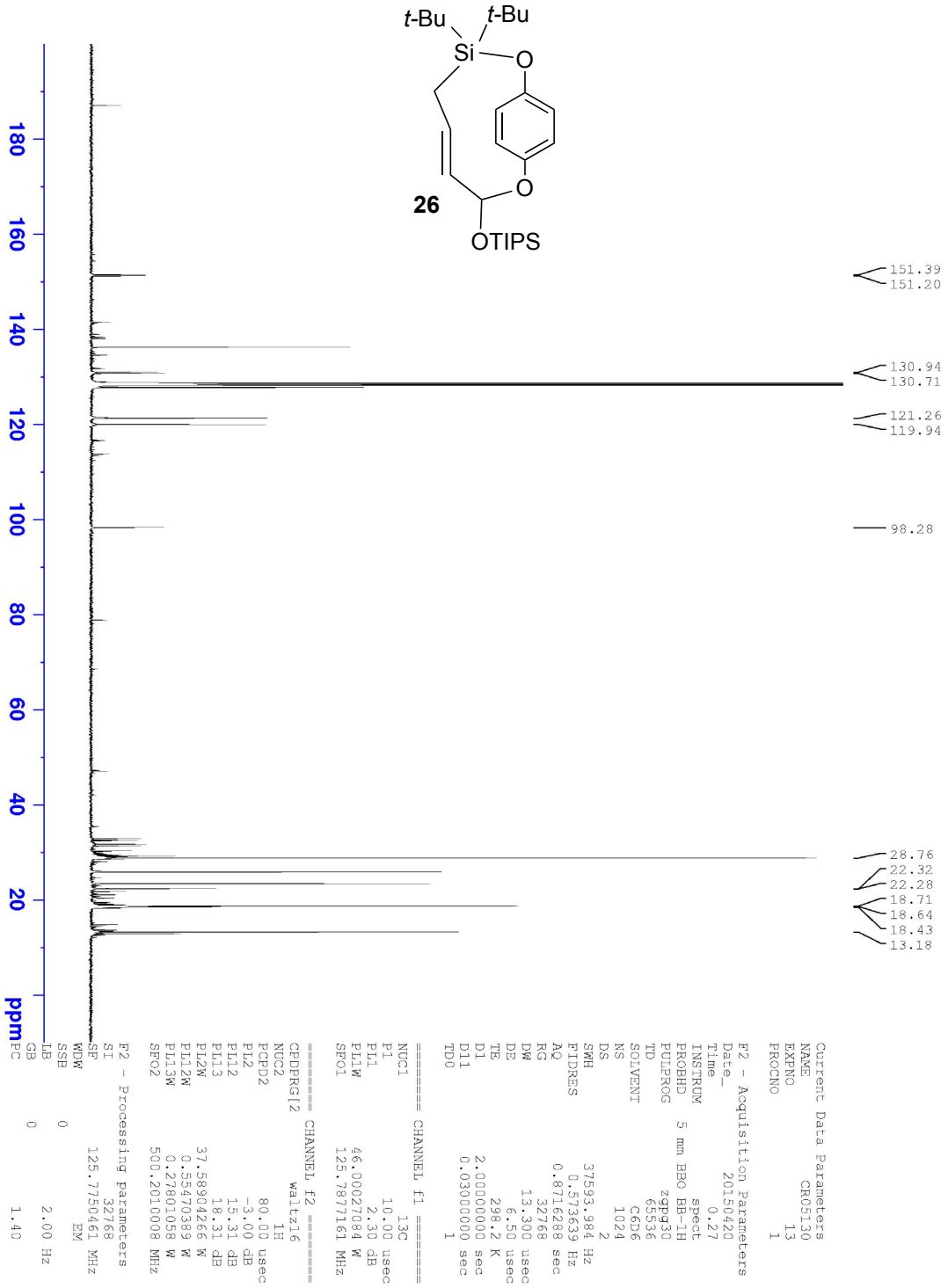


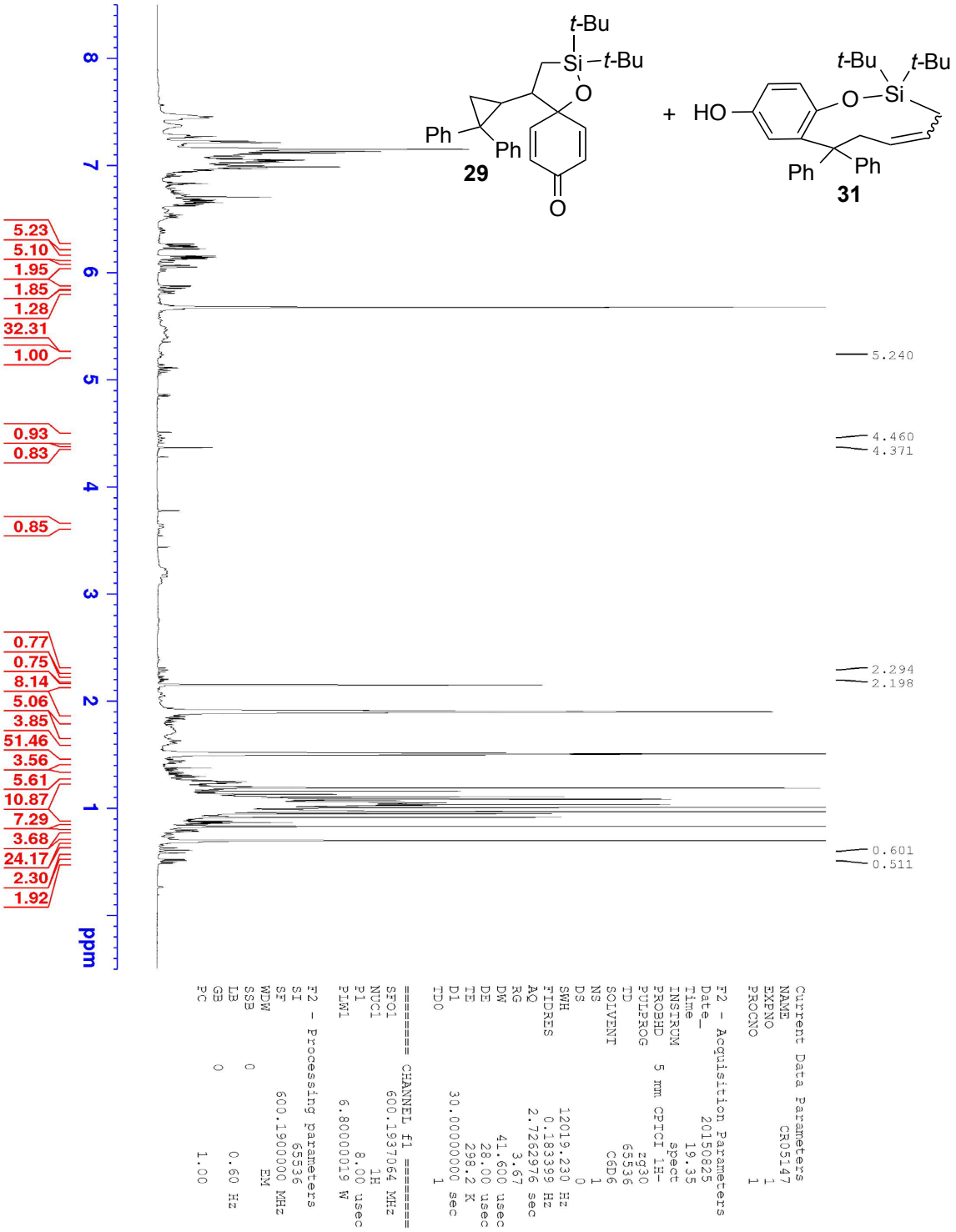

```
Current Data Parameters
NAME                  CR05130
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150419
Time                    16.00
INSTRUM                spect
PROBHD    5 mm BBO BB-1H
PULPROG                 zg30
TD                     65536
SOLVENT                 C6D6
NS                          1
DS                          0
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                       28.5
DW                     48.400 usec
DE                      6.50 usec
TE                     298.2 K
D1               30.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.60 usec
PL1                     -3.00 dB
PL1W             37.58904266 W
SFO1             500.2020889 MHz

F2 - Processing parameters
SI                      32768
SF               500.1989980 MHz
WDW                        EM
SSB          0
LB                       0.60 Hz
GB           0
PC                       1.00
```

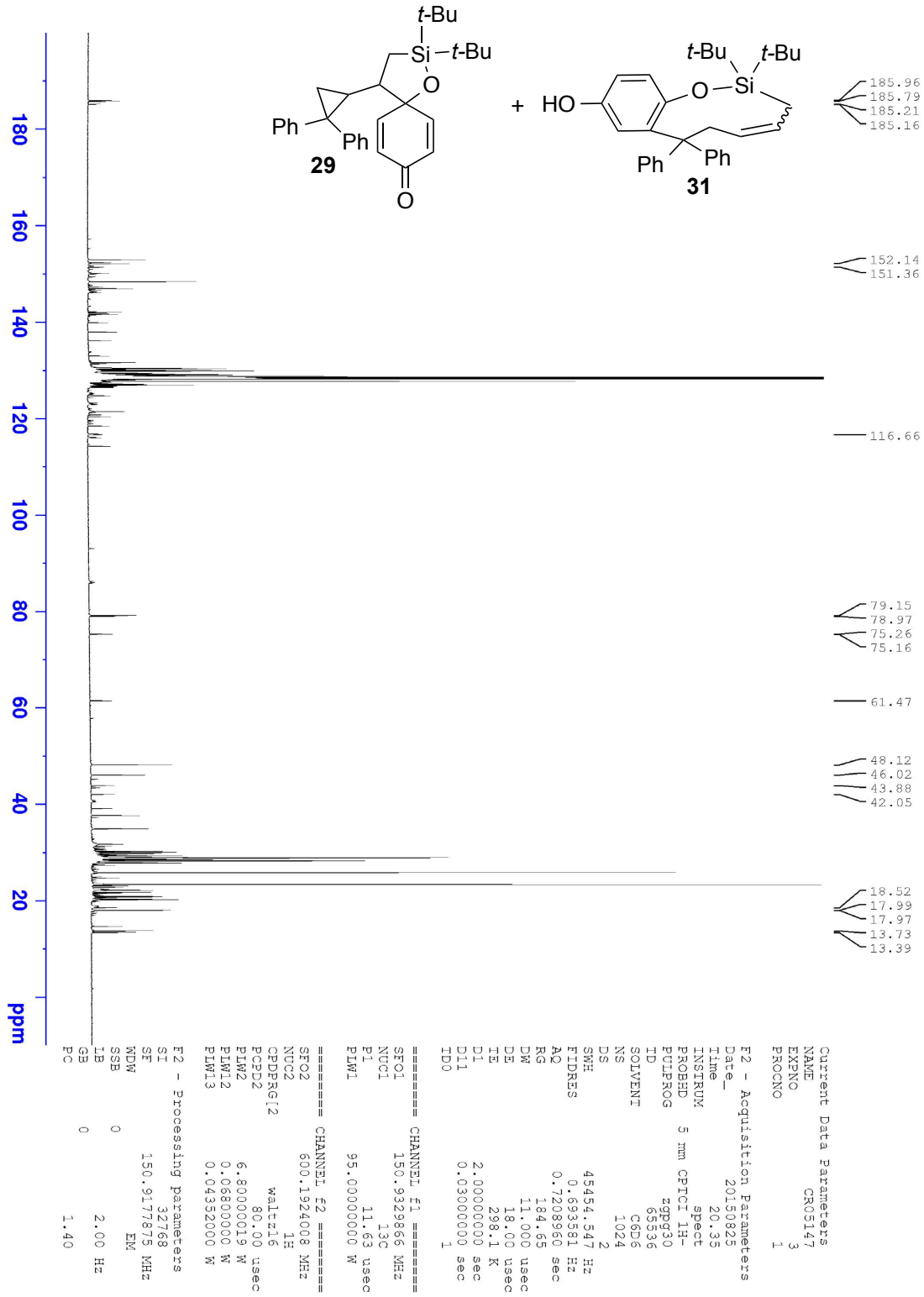


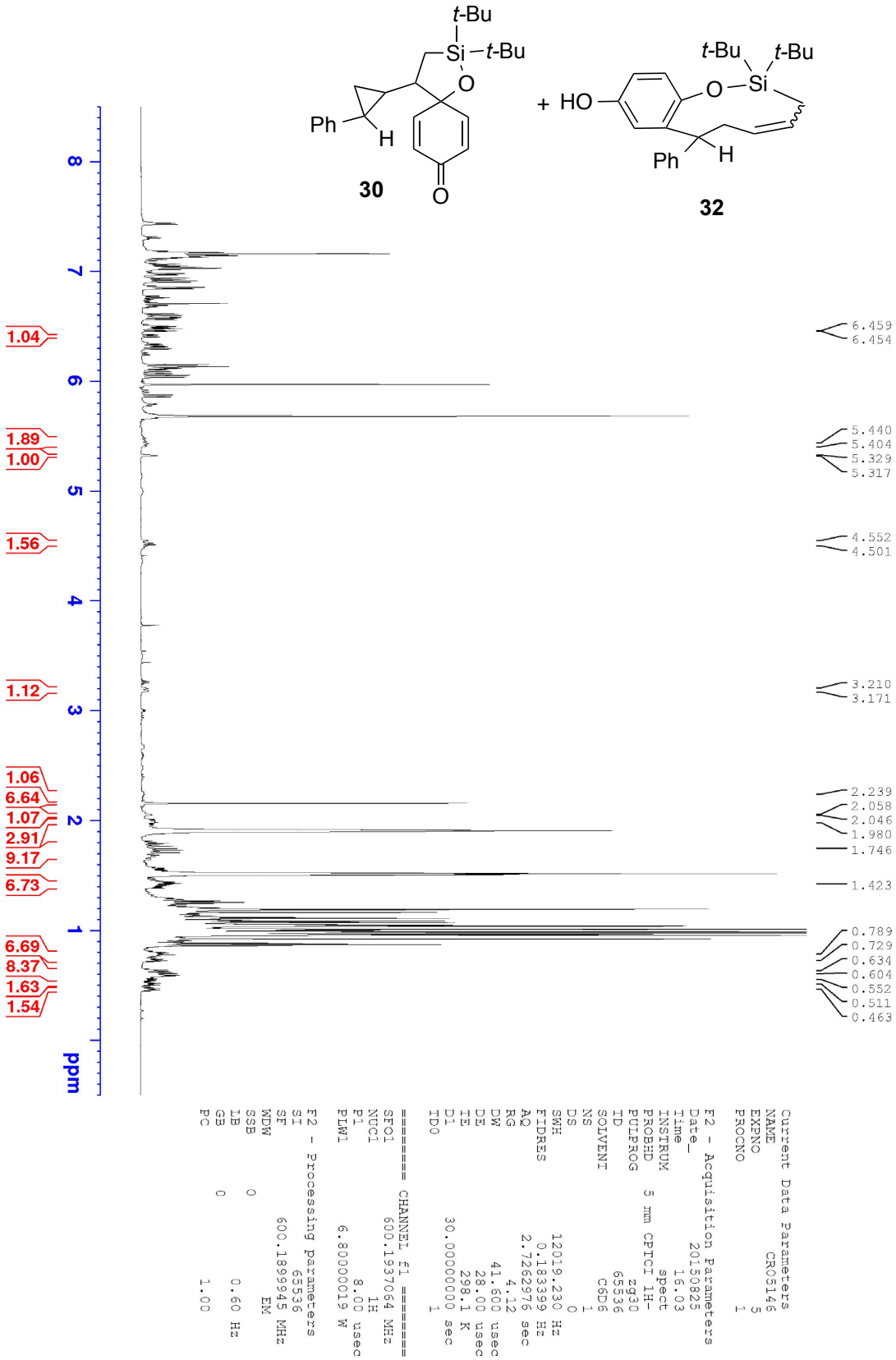

```
Current Data Parameters
NAME                  CR05130
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_               20150419
Time                   16.37
INSTRUM                spect
PROBHD     5 mm BBO BB-1H
PULPROG             zgpg30
TD                   65536
SOLVENT              C6D6
NS                    512
DS                      2
SWH                37593.984 Hz
FIDRES                0.573633 Hz
AQ                    0.8716288 sec
RG                    32768
DW                    13.300 usec
DE                     6.50 usec
TE                    298.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PL1                   2.30 dB
PL1W                46.00027084 W
SFO1               125.7877161 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -3.00 dB
PL12                  15.31 dB
PL13                  18.31 dB
PL2W                37.58904266 W
PL12W                0.55470389 W
PL13W                0.27801058 W
SFO2               500.2010008 MHz

F2 - Processing parameters
SI                      32768
SF                 125.7750463 MHz
WDW                        EM
SSB                     0
LB                      2.00 Hz
GB                      0
PC                      1.40
```

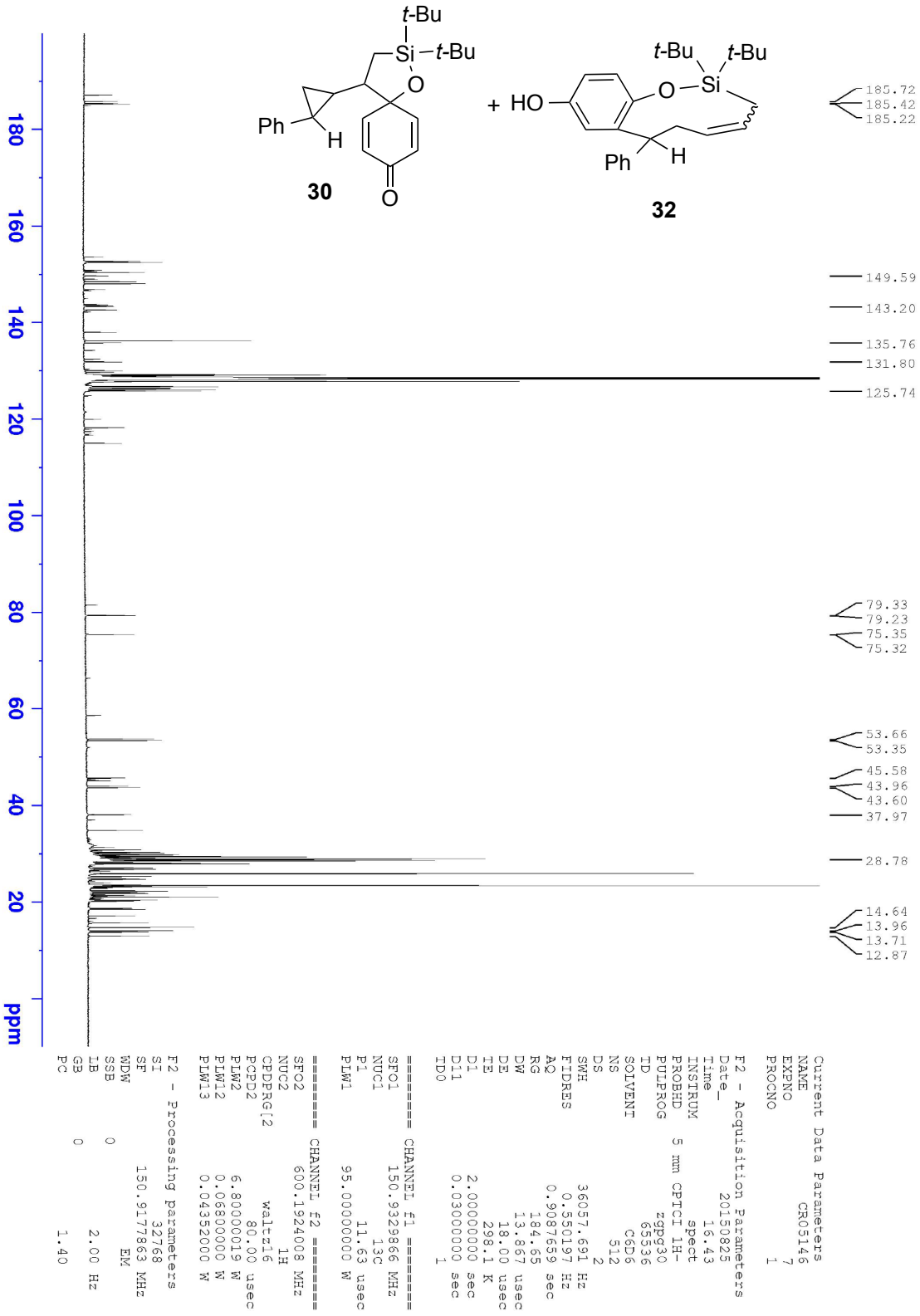

t-Bu
t-Bu
Si
O
O
26
OTIPS
7.042
7.020
6.854
6.832
6.183
6.162
6.143
6.124
6.105
5.779
5.766
5.740
5.727
1.854
1.834
1.175
1.102
1.98
1.92
0.38
1.17
2.92
1.09
0.98
1.96
20.50
20.32
ppm
Current Data Parameters
NAME CR05130
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150420
Time 10.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 25.4
DW 60.400 usec
DE 6.50 usec
TE 298.2 K
D1 30.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.05 usec
PL1 -5.00 dB
PL1W 31.77312851 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300445 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

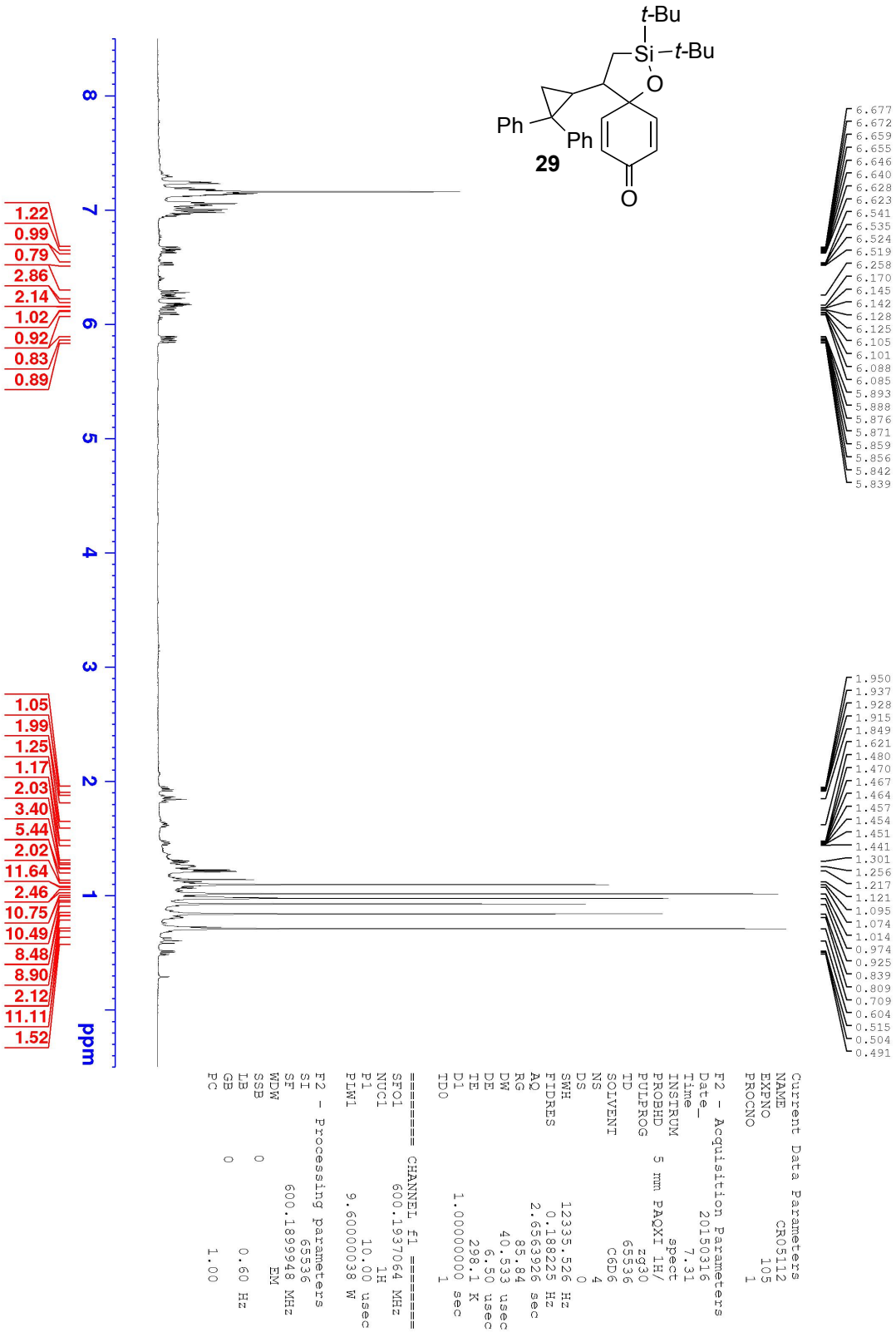

t-Bu
t-Bu
Si
O
O
26
OTIPS
151.39
151.20
130.94
130.71
121.26
119.94
98.28
28.76
22.32
22.28
18.71
18.64
18.43
13.18
ppm
180
160
140
120
100
80
60
40
20
Current Data Parameters
NAME CR05130
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150420
Time 0.27
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716288 sec
RG 32768
DW 13.300 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.30 dB
PL1W 46.00027084 W
SFO1 125.7877161 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.31 dB
PL13 18.31 dB
PL2W 37.58904266 W
PL12W 0.55470389 W
PL13W 0.27801058 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7750461 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

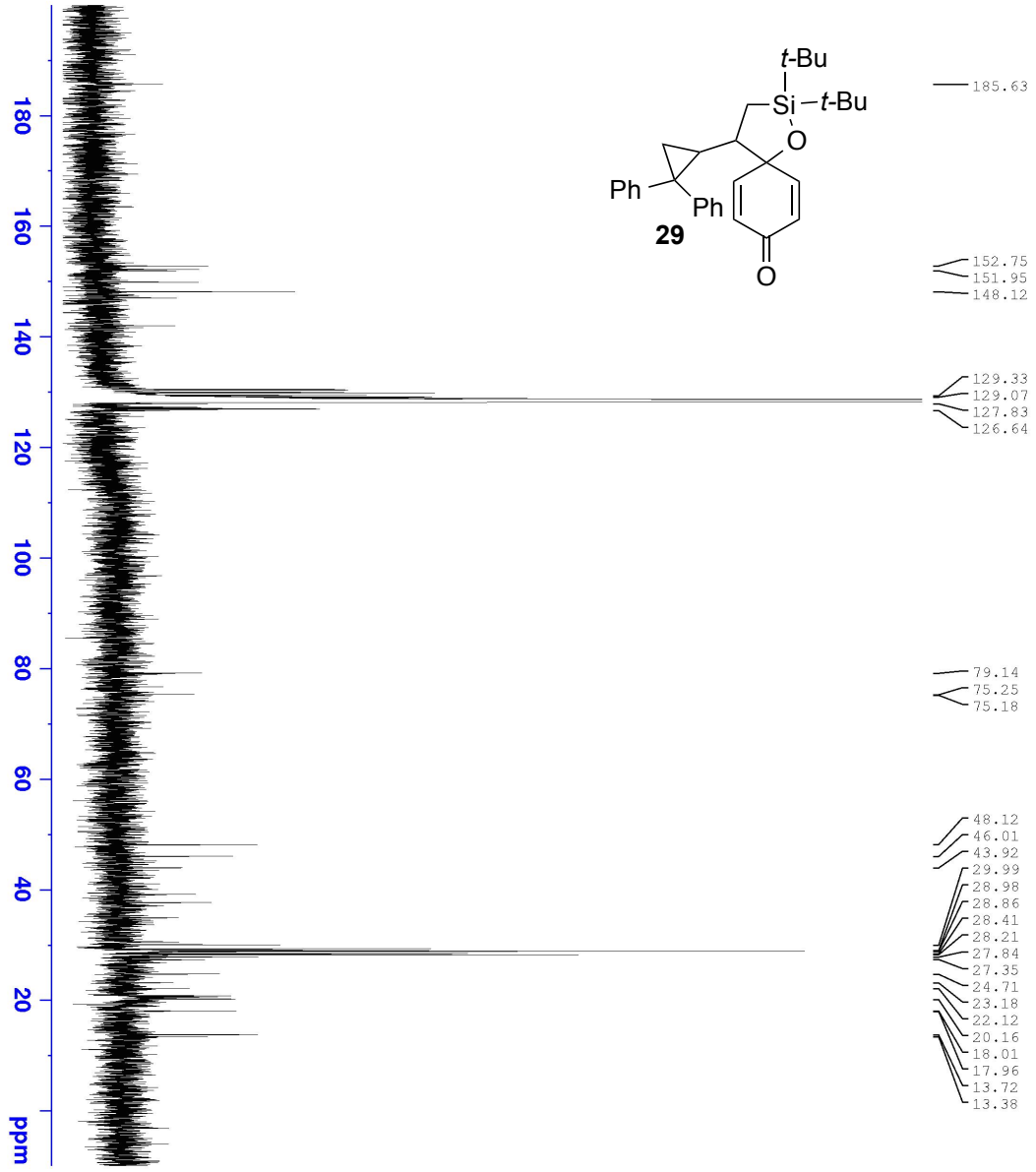

t-Bu
Si
t-Bu
O
Ph
Ph
29
O
+
HO
t-Bu
t-Bu
O
Si
Ph
Ph
31
5.23
5.10
1.95
1.85
1.28
32.31
1.00
0.93
0.83
0.85
0.77
0.75
8.14
5.06
3.85
51.46
3.56
5.61
10.87
7.29
3.68
24.17
2.30
1.92
ppm
5.240
4.460
4.371
2.294
2.198
0.601
0.511
Current Data Parameters
NAME CR05147
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150825
Time 19.35
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 3.67
DW 41.600 usec
DE 28.00 usec
TE 298.2 K
D1 30.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1937064 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.80000019 W
F2 - Processing parameters
SI 65536
SF 600.1900000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00


t-Bu
Si
t-Bu
O
Ph
Ph
29
O
+
HO
t-Bu
t-Bu
O
Si
Ph
Ph
31
185.96
185.79
185.21
185.16
152.14
151.36
116.66
79.15
78.97
75.26
75.16
61.47
48.12
46.02
43.88
42.05
18.52
17.99
17.97
13.73
13.39
ppm
180
160
140
120
100
80
60
40
20
Current Data Parameters
NAME CR05147
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150825
Time_ 20.35
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1024
DS 2
SWH 45454.547 Hz
FIDRES 0.693581 Hz
AQ 0.7208960 sec
RG 184.65
DW 11.000 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 150.9329866 MHz
NUC1 13C
P1 11.63 usec
PLW1 95.00000000 W
========== CHANNEL f2 ==========
SFO2 600.1924008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 6.80000019 W
PLW12 0.06800000 W
PLW13 0.04352000 W
F2 - Processing parameters
SI 32768
SF 150.9177875 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

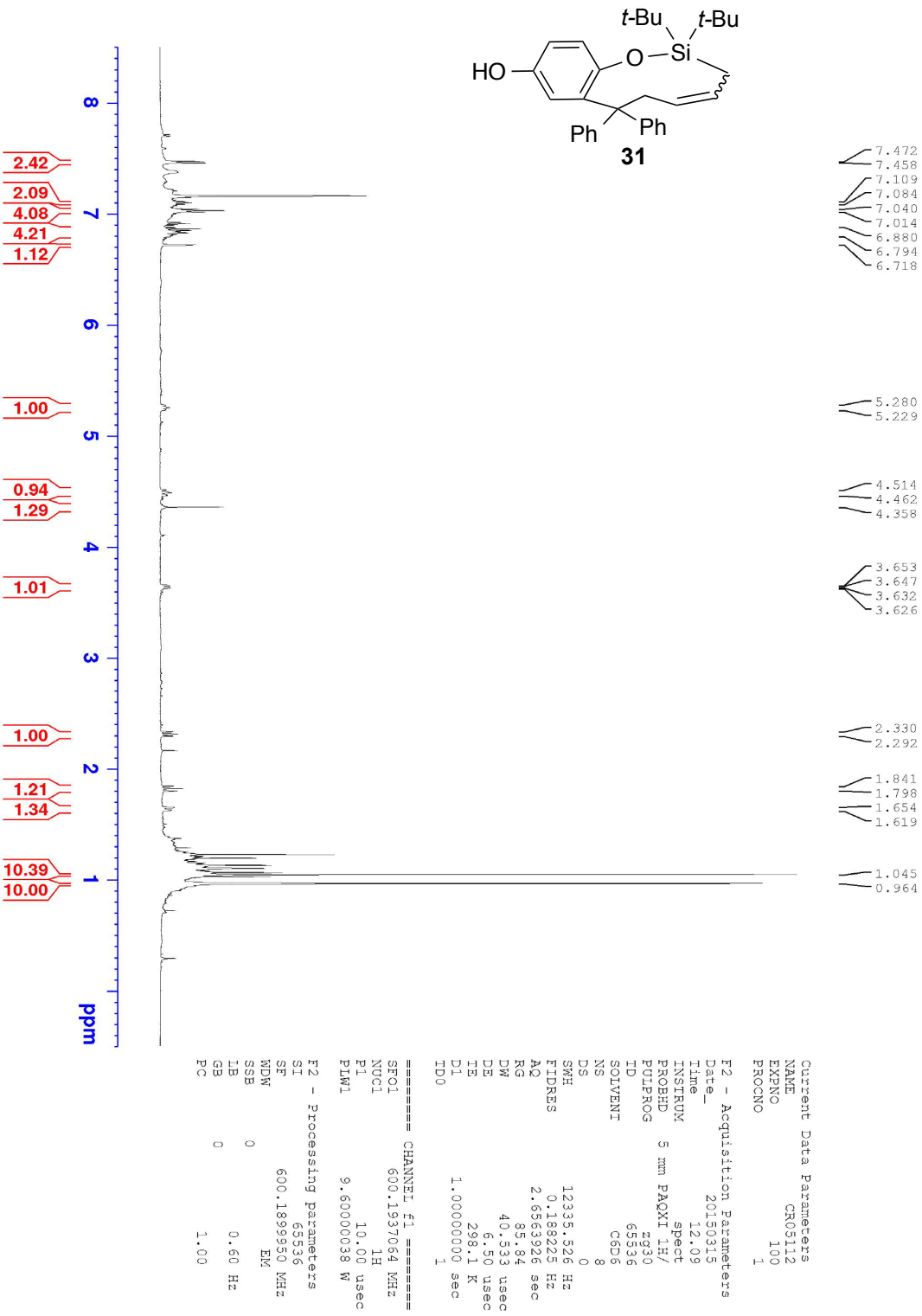

t-Bu
Si
t-Bu
O
Ph
H
O
30
+ HO
t-Bu
t-Bu
Si
O
Ph
H
32
ppm
1.04
1.89
1.00
1.56
1.12
1.06
6.64
1.07
2.91
9.17
6.73
6.69
8.37
1.63
1.54
6.459
6.454
5.440
5.404
5.329
5.317
4.552
4.501
3.210
3.171
2.239
2.058
2.046
1.980
1.746
1.423
0.789
0.729
0.634
0.604
0.552
0.511
0.463
Current Data Parameters
NAME CR05146
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150825
Time 16.03
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 4.12
DW 41.600 usec
DE 28.00 usec
TE 298.1 K
D1 30.00000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 600.1937064 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.80000019 W
F2 - Processing parameters
SI 65536
SF 600.1899945 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00


t-Bu
Si
t-Bu
O
Ph
H
O
30
+ HO
t-Bu
t-Bu
O
Si
Ph
H
32
185.72
185.42
185.22
149.59
143.20
135.76
131.80
125.74
79.33
79.23
75.35
75.32
53.66
53.35
45.58
43.96
43.60
37.97
28.78
14.64
13.96
13.71
12.87
ppm
Current Data Parameters
NAME CR05146
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150825
Time 16.43
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 512
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 184.65
DW 13.867 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
ID0 1
========== CHANNEL f1 ==========
SFO1 150.9329866 MHz
NUC1 13C
P1 11.63 usec
PLW1 95.00000000 W
========== CHANNEL f2 ==========
SFO2 600.1924008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 6.80000019 W
PLW12 0.06800000 W
PLW13 0.04352000 W
F2 - Processing parameters
SI 32768
SF 150.9177863 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

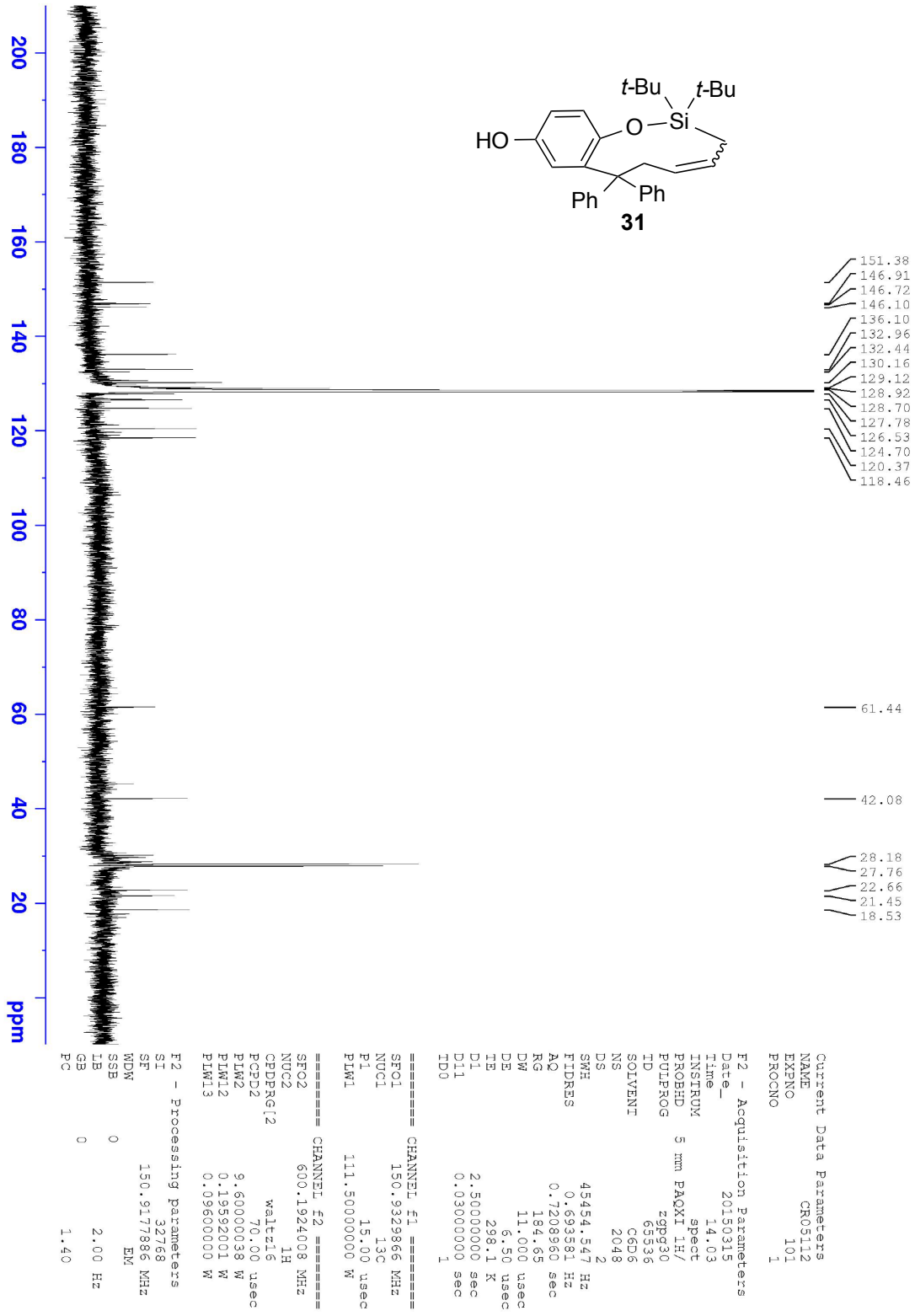

t-Bu
Si
t-Bu
O
Ph
Ph
29
O
ppm
1.22
0.99
0.79
2.86
2.14
1.02
0.92
0.83
0.89
1.05
1.99
1.25
1.17
2.03
3.40
5.44
2.02
11.64
2.46
10.75
10.49
8.48
8.90
2.12
11.11
1.52
6.677
6.672
6.659
6.655
6.646
6.640
6.628
6.623
6.541
6.535
6.524
6.519
6.258
6.170
6.145
6.142
6.128
6.125
6.105
6.101
6.088
6.085
5.893
5.888
5.876
5.871
5.859
5.856
5.842
5.839
1.950
1.937
1.928
1.915
1.849
1.621
1.480
1.470
1.467
1.464
1.457
1.454
1.451
1.441
1.301
1.256
1.217
1.121
1.095
1.074
1.014
0.974
0.925
0.839
0.809
0.709
0.604
0.515
0.504
0.491
Current Data Parameters
NAME CR05112
EXPNO 105
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150316
Time 7.31
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 4
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6563926 sec
RG 85.84
DW 40.533 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 600.1937064 MHz
NUC1 1H
P1 10.00 usec
PLW1 9.60000038 W
F2 - Processing parameters
SI 65536
SF 600.1899948 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00

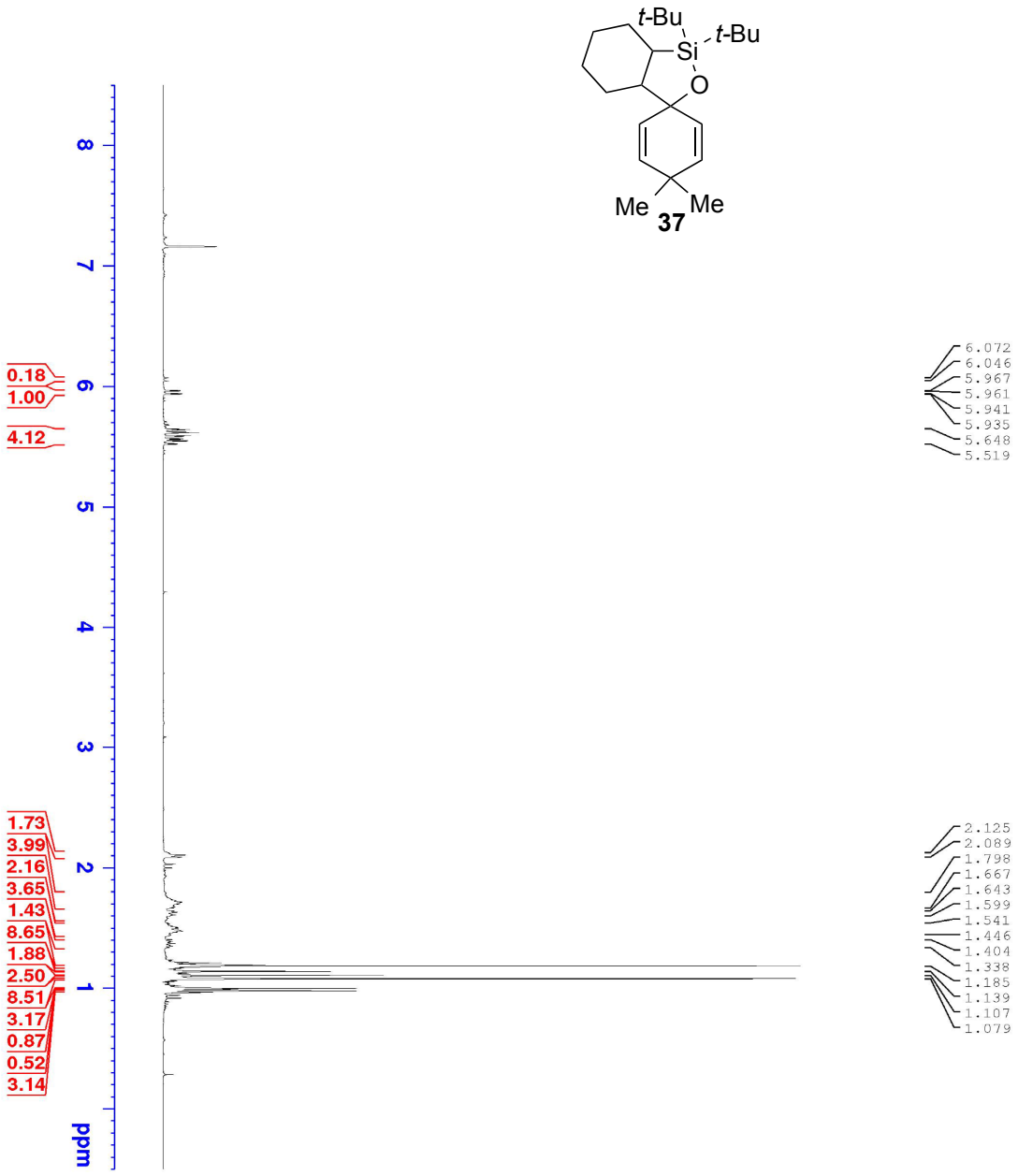


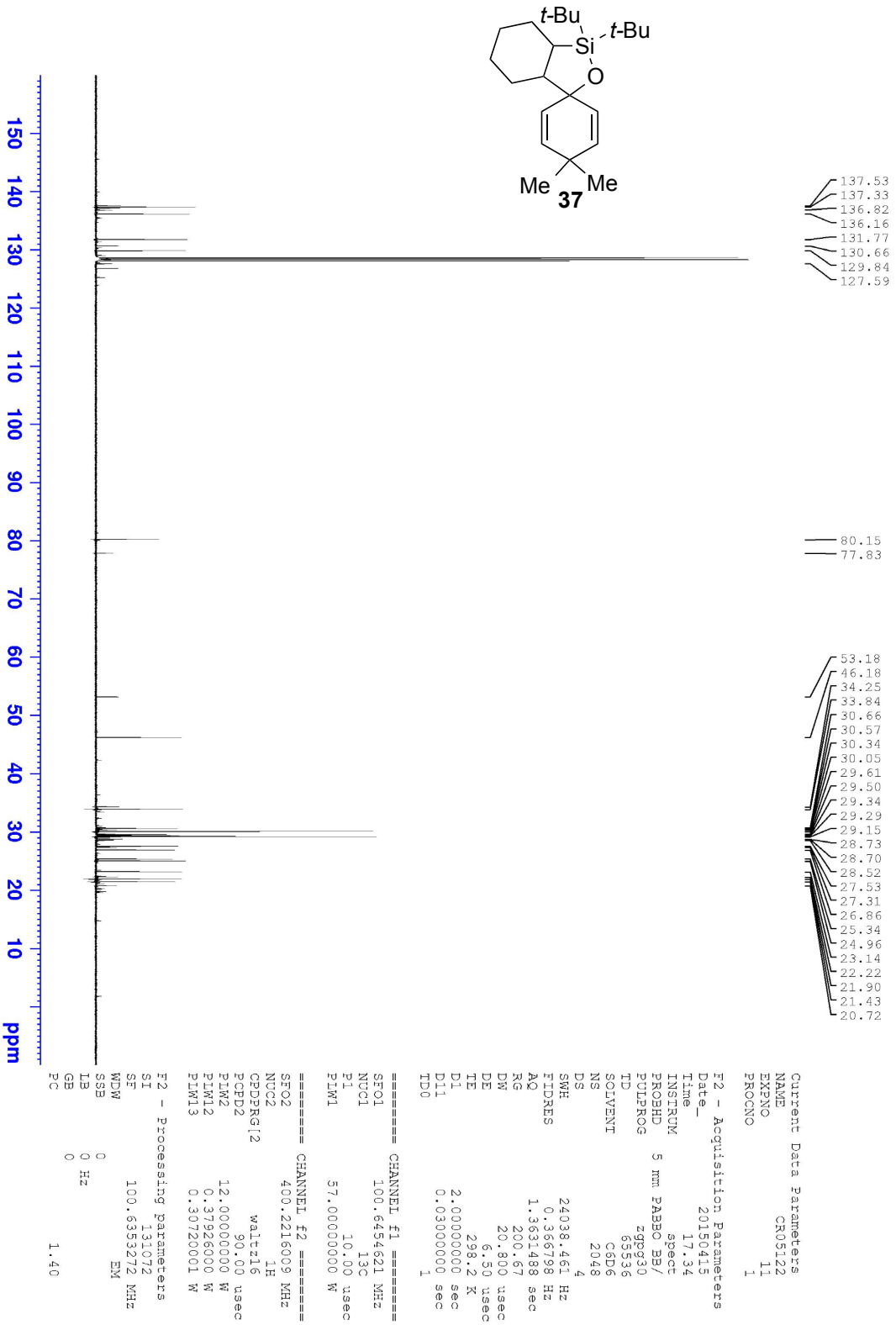

```
Current Data Parameters
NAME                  CR05112
EXPNO                     106
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150316
Time                     8.20
INSTRUM                  spect
PROBHD     5 mm PAQXI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 C6D6
NS                      1024
DS                         2
SWH                45454.547 Hz
FIDRES                 0.693581 Hz
AQ                     0.7208960 sec
RG                       184.65
DW                     11.000 usec
DE                      6.50 usec
TE                     298.1 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                        1

========== CHANNEL f1 ========
SFO1            150.9329873 MHz
NOC1                       13C
P1                  15.00 usec
PLW1           111.50000000 W

========== CHANNEL f2 ========
SFO2            600.1924008 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                   70.00 usec
PLW2           9.60000038 W
PLW12          0.19592001 W
PLW13          0.09600000 W

F2 - Processing parameters
SI                        32768
SF              150.9177889 MHz
WDW                        EM
SSB                         0
LB                       2.00 Hz
GB                          0
PC                       1.40
```

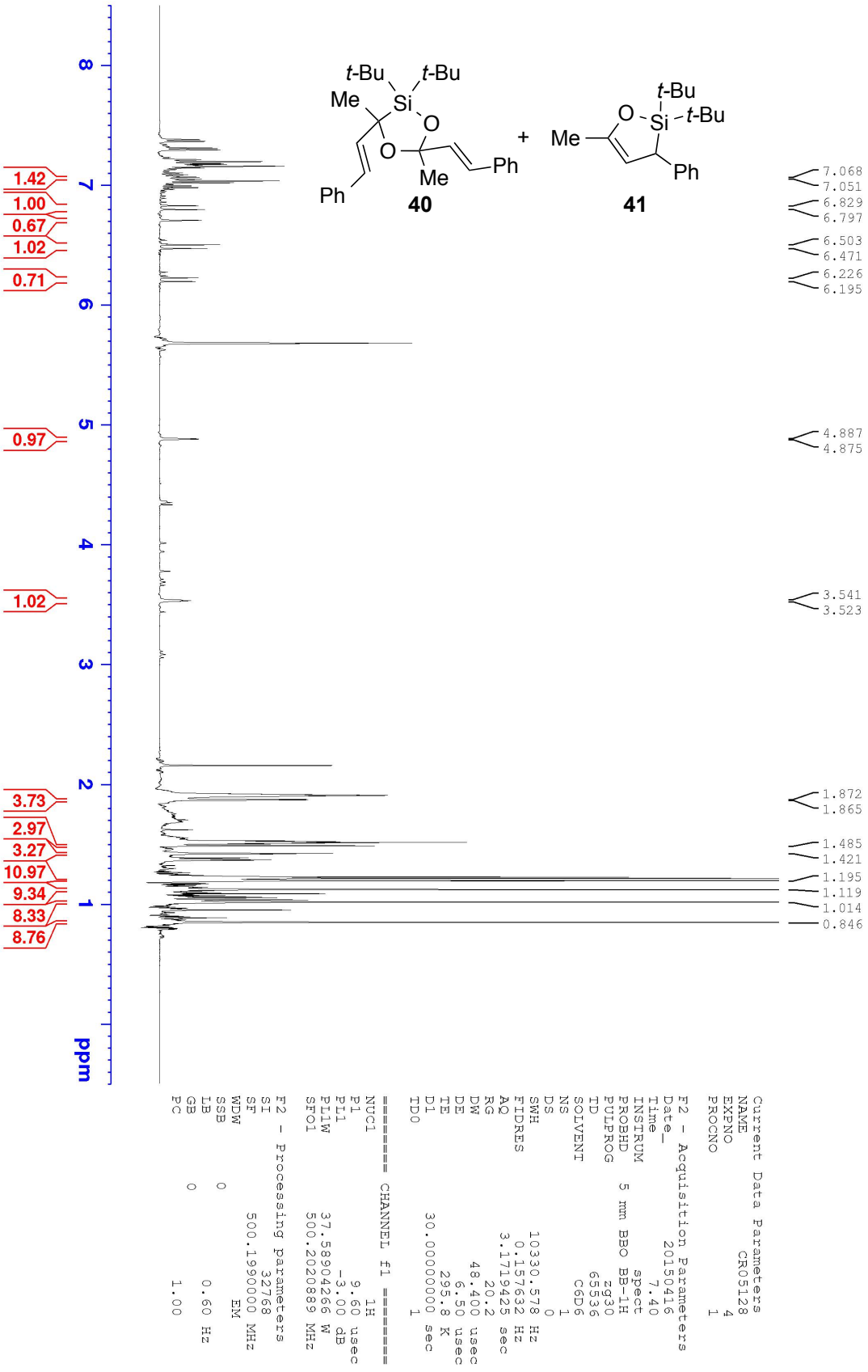


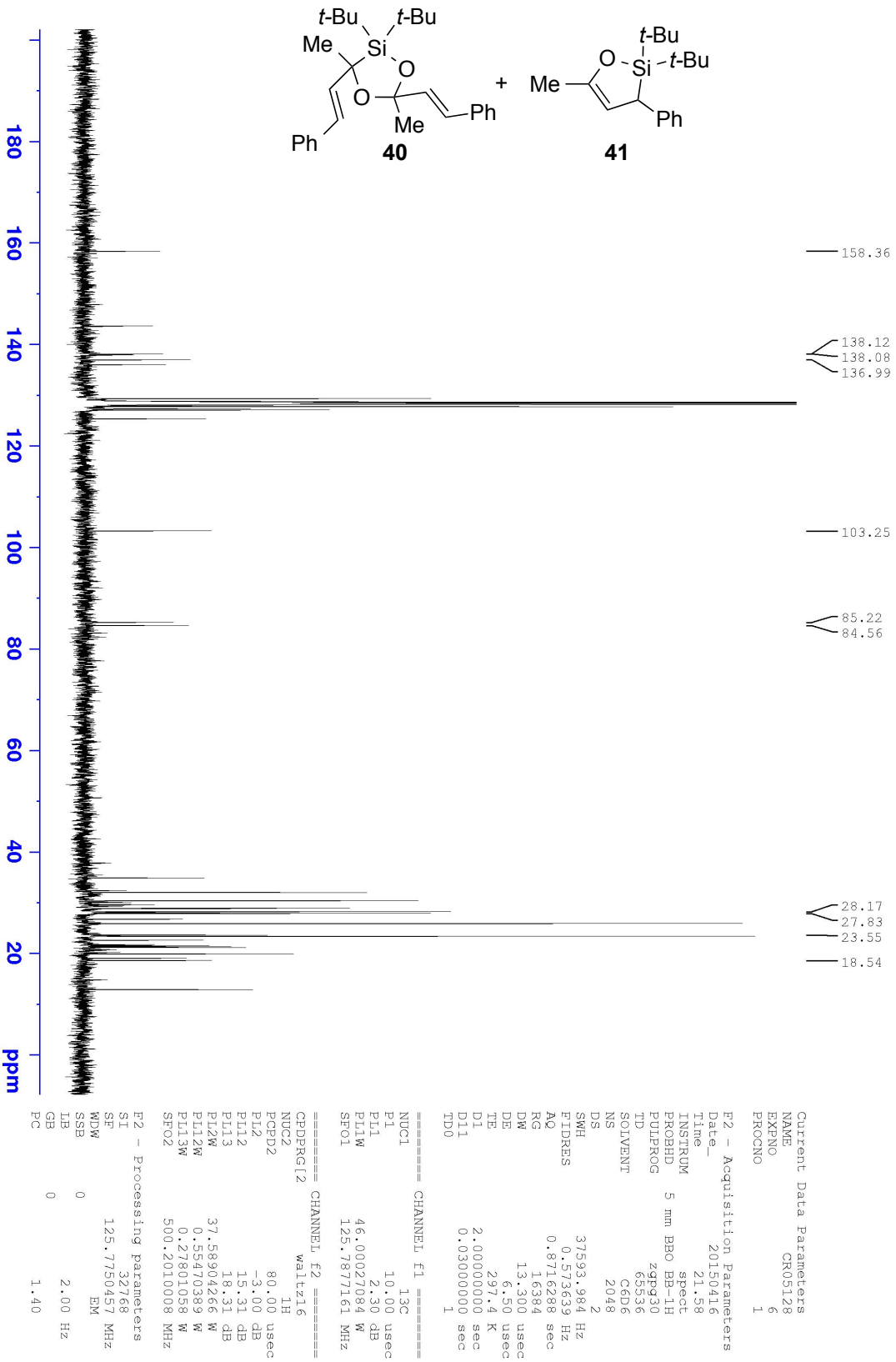

```
Current Data Parameters
NAME                  CR05112
EXPNO                      100
PROCNO                       1

F2 - Acquisition Parameters
Date_               20150315
Time                   12.09
INSTRUM                 spect
PROBHD     5 mm PAQXI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               C6D6
NS                        8
DS                        0
SWH               12335.526 Hz
FIDRES            0.188225 Hz
AQ                2.6563926 sec
RG                   85.84
DW                40.533 usec
DE                    6.50 usec
TE                   298.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1              600.1937064 MHz
NUC1                      1H
P1                    10.00 usec
PLW1              9.60000038 W

F2 - Processing parameters
SI                    65536
SF                600.1899950 MHz
WDW                       EM
SSB                       0
LB                     0.60 Hz
GB                        0
PC                     1.00
```


t-Bu
t-Bu
HO
O
Si
Ph
Ph
31
151.38
146.91
146.72
146.10
136.10
132.96
132.44
130.16
129.12
128.92
128.70
127.78
126.53
124.70
120.37
118.46
61.44
42.08
28.18
27.76
22.66
21.45
18.53
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR05112
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150315
Time 14.03
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 2048
DS 2
SWH 45454.547 Hz
FIDRES 0.693581 Hz
AQ 0.7208960 sec
RG 184.65
DW 11.000 usec
DE 6.50 usec
TE 298.1 K
D1 2.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9329866 MHz
NUC1 13C
P1 15.00 usec
PLW1 111.50000000 W
======== CHANNEL f2 ========
SFO2 600.1924008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 9.60000038 W
PLW12 0.19592001 W
PLW13 0.09600000 W
F2 - Processing parameters
SI 32768
SF 150.9177886 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40


t-Bu
t-Bu
Si
O
Me
Me
37
6.072
6.046
5.967
5.961
5.941
5.935
5.648
5.519
2.125
2.089
1.798
1.667
1.643
1.599
1.541
1.446
1.404
1.338
1.185
1.139
1.107
1.079
0.18
1.00
4.12
1.73
3.99
2.16
3.65
1.43
8.65
1.88
2.50
8.51
3.17
0.87
0.52
3.14
ppm
Current Data Parameters
NAME CR05122
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150415
Time 15.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 46.71
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
SFO1 400.2224715 MHz
NUC1 1H
P1 15.00 usec
PLW1 14.25000000 W
F2 - Processing parameters
SI 65536
SF 400.2199958 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


t-Bu
Si
t-Bu
O
Me
Me
37
137.53
137.33
136.82
136.16
131.77
130.66
129.84
127.59
80.15
77.83
53.18
46.18
34.25
33.84
30.66
30.57
30.34
30.05
29.61
29.50
29.34
29.29
29.15
28.73
28.70
28.52
27.53
27.31
26.86
25.34
24.96
23.14
22.22
21.90
21.43
20.72
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME CR05122
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150415
Time 17.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 200.67
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6454621 MHz
NUC1 13C
P1 10.00 usec
PLW1 57.00000000 W
======== CHANNEL f2 ========
SFO2 400.2216009 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.37926000 W
PLW13 0.30720001 W
F2 - Processing parameters
SI 131072
SF 100.6353272 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.40


t-Bu
t-Bu
Me
Si
O
O
Me
Ph
Ph
40
+
t-Bu
O
Si
t-Bu
Me
Ph
41
1.42
1.00
0.67
1.02
0.71
0.97
1.02
3.73
2.97
3.27
10.97
9.34
8.33
8.76
ppm
7.068
7.051
6.829
6.797
6.503
6.471
6.226
6.195
4.887
4.875
3.541
3.523
1.872
1.865
1.485
1.421
1.195
1.119
1.014
0.846
Current Data Parameters
NAME CR05128
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150416
Time 7.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 1
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 20.2
DW 48.400 usec
DE 6.50 usec
TE 295.8 K
D1 30.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 9.60 usec
PL1 -3.00 dB
PLIW 37.58904266 W
SFO1 500.2020889 MHz
F2 - Processing parameters
SI 32768
SF 500.1990000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00


t-Bu
t-Bu
Me
Si
O
O
Me
Ph
Ph
40
+
t-Bu
O
Si
t-Bu
Me
Ph
41
158.36
138.12
138.08
136.99
103.25
85.22
84.56
28.17
27.83
23.55
18.54
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME CR05128
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150416
Time 21.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 2048
DS 2
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716288 sec
RG 16384
DW 13.300 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.30 dB
PL1W 46.00027084 W
SFO1 125.78771161 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.31 dB
PL13 18.31 dB
PL2W 37.58904266 W
PL12W 0.55470389 W
PL13W 0.27801058 W
SFO2 500.2010008 MHz
F2 - Processing parameters
SI 32768
SF 125.7750457 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40