

## Magnesium-mediated intramolecular reductive coupling: A stereoselective synthesis of C<sub>2</sub>-symmetric 3,4-bis-silyl-substituted adipic acid derivatives

Pintu K. Kundu and Sunil K. Ghosh\*

Bio-Organic Division, Bhabha Atomic Research Centre

Trombay, Mumbai 400085, India

E-mail: [ghsunil@barc.gov.in](mailto:ghsunil@barc.gov.in)

### Table of Contents

Entry	Description	Page
1	Figure-S1. <sup>1</sup> H NMR Spectrum of <b>2a</b>	3
2	Figure-S2. <sup>13</sup> C NMR Spectrum of <b>2a</b>	4
3	Figure-S3. <sup>1</sup> H NMR Spectrum of <i>trans</i> - <b>1a</b>	5
4	Figure-S4. <sup>13</sup> C NMR Spectrum of <i>trans</i> - <b>1a</b>	6
5	Figure-S5. <sup>1</sup> H NMR Spectrum of <i>cis</i> - <b>1a</b>	7
6	Figure-S6. <sup>13</sup> C NMR Spectrum of <i>cis</i> - <b>1a</b>	8
7	Figure-S7. <sup>1</sup> H NMR Spectrum of <b>5a</b>	9
8	Figure-S8. <sup>13</sup> C NMR Spectrum of <b>5a</b>	10
9	Figure-S9. <sup>1</sup> H NMR Spectrum of <b>9a</b>	11
10	Figure-S10. <sup>1</sup> H NMR Spectrum of <b>13a</b>	12
11	Figure-S11. <sup>13</sup> C NMR Spectrum of <b>11a</b>	13
12	Figure-S12. <sup>1</sup> H NMR Spectrum of <b>11b</b>	14
13	Figure-S13. <sup>13</sup> C NMR Spectrum of <b>11b</b>	15
14	Figure-S14. <sup>1</sup> H NMR Spectrum of <b>10a</b>	16
15	Figure-S15. <sup>13</sup> C NMR Spectrum of <b>10a</b>	17
16	Figure-S16. <sup>1</sup> H NMR Spectrum of <b>13a</b>	18
17	Figure-S17. <sup>13</sup> C NMR Spectrum of <b>13a</b>	19
18	Figure-S18. <sup>1</sup> H NMR Spectrum of <i>trans</i> - <b>14a</b>	20
19	Figure-S19. <sup>13</sup> C NMR Spectrum of <i>trans</i> - <b>14a</b>	21
20	Figure-S20. <sup>1</sup> H NMR Spectrum of <i>trans</i> - <b>15a</b>	22
21	Figure-S21. <sup>13</sup> C NMR Spectrum of <i>trans</i> - <b>15a</b>	23
22	Figure-S22. <sup>1</sup> H NMR Spectrum of <i>cis</i> - <b>14a</b>	24
23	Figure-S23. <sup>13</sup> C NMR Spectrum of <i>cis</i> - <b>14a</b>	25
24	Figure-S24. <sup>1</sup> H NMR Spectrum of <b>10b</b>	26
25	Figure-S25. <sup>13</sup> C NMR Spectrum of <b>10b</b>	27
26	Figure-S26. <sup>1</sup> H NMR Spectrum of <b>13b</b>	28
27	Figure-S27. <sup>13</sup> C NMR Spectrum of <b>13b</b>	29
28	Figure-S28. <sup>1</sup> H NMR Spectrum of <i>trans</i> - <b>15b</b>	30

29	Figure-S29. $^{13}\text{C}$ NMR Spectrum of <i>trans</i> - <b>15b</b>	31
30	Figure-S30. $^1\text{H}$ NMR Spectrum of <b>10c</b>	32
31	Figure-S31. $^{13}\text{C}$ NMR Spectrum of <b>10c</b>	33
32	Figure-S32. $^1\text{H}$ NMR Spectrum of <b>13c</b>	34
33	Figure-S33. $^{13}\text{C}$ NMR Spectrum of <b>13c</b>	35
34	Figure-S34. $^1\text{H}$ NMR Spectrum of <i>trans</i> - <b>14c</b>	36
35	Figure-S35. $^1\text{H}$ NMR Spectrum of <i>trans</i> - <b>15c</b>	37
36	Figure-S36. $^{13}\text{C}$ NMR Spectrum of <i>trans</i> - <b>15c</b>	38
37	Figure-S37. $^1\text{H}$ NMR Spectrum of (-)- <b>16</b>	39
38	Figure-S38. $^{13}\text{C}$ NMR Spectrum of (-)- <b>16</b>	40
39	Figure-S39. View of crystals of (a) <i>trans</i> - <b>14a</b> ; (b) <i>trans</i> - <b>15a</b>	41

Figure-S1. <sup>1</sup>H NMR of **2a**

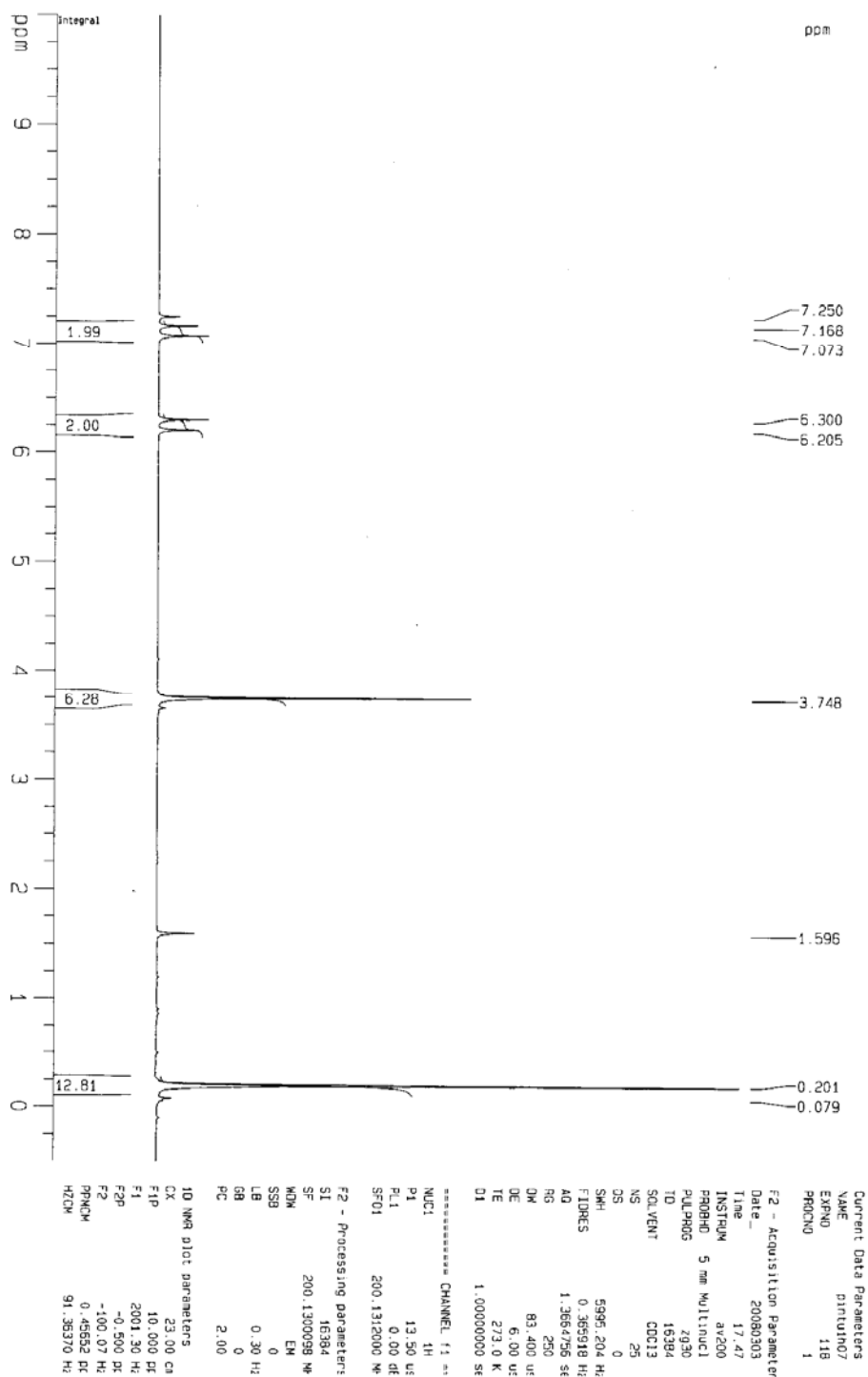
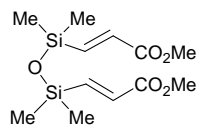
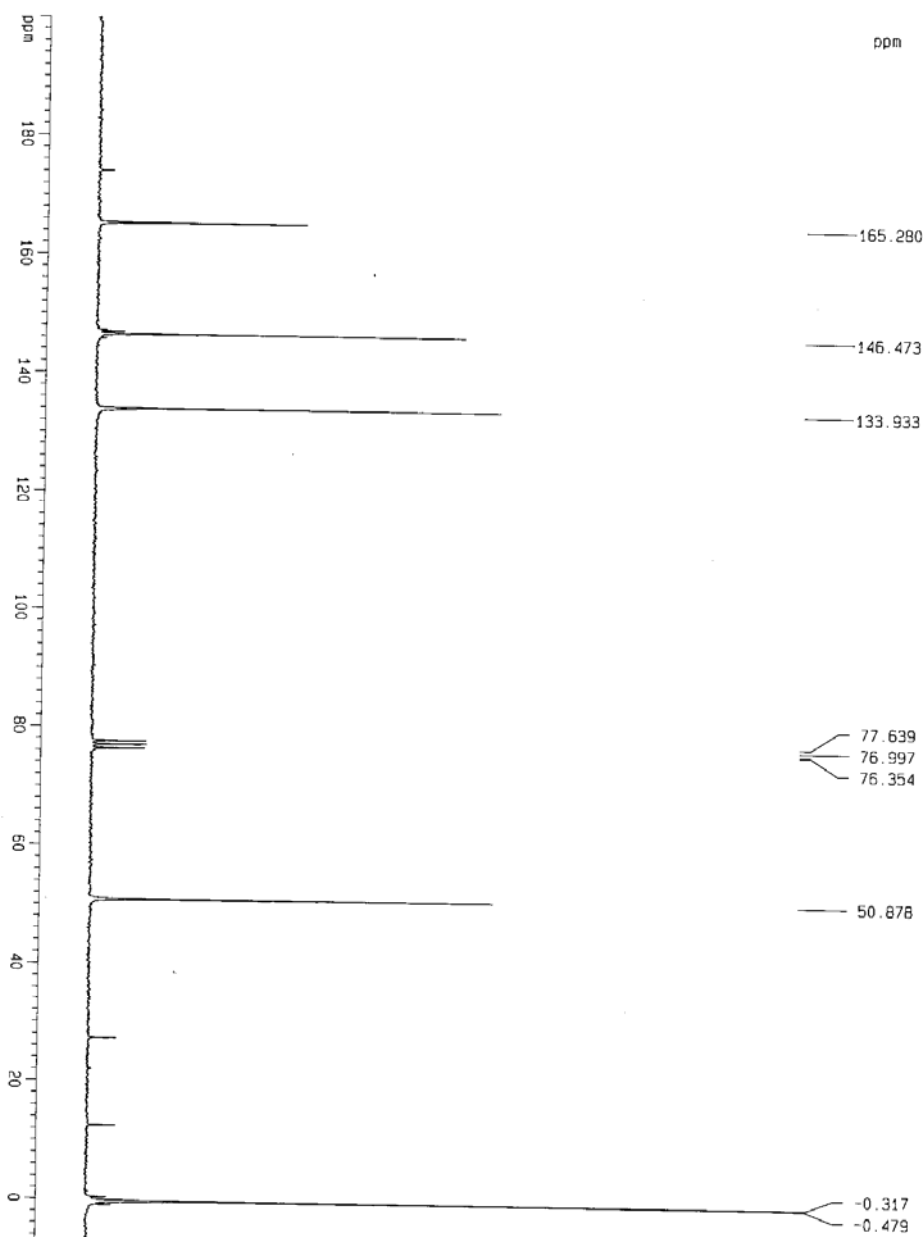
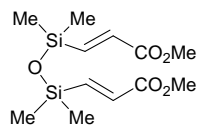


Figure-S2. <sup>13</sup>C NMR of 2a



Current Data Parameters  
 Name: p1rc013507  
 Date\_ Time: 20071029 13:06  
 ExpNO: 4  
 F2 - Acquisition Parameters  
 Date\_ Time: 20071029 13:06  
 INSTRUM: spect  
 PROBRD: 5 mm NAL114PC  
 PULPROG: zgpg30  
 TO: 32768  
 SOLVENT: CDCl3  
 NS: 539  
 DS: 4  
 SFO1: 15060.241 Hz  
 SFO2: 0.459602 Hz  
 AQ: 1.0673475 sec  
 RG: 32768  
 DM: 33.200 usec  
 DE: 5.00 usec  
 TE: 300.0 K  
 O1: 2.00000000 sec  
 d11: 0.03000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: <sup>13</sup>C  
 P1: 6.20 usec  
 PL1: -3.00 dB  
 SF01: 50.3282445 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CHOPRS2: MALTZ16  
 NUC2: <sup>1</sup>H  
 P2: 100.00 usec  
 PL2: 120.00 dB  
 PL12: 16.00 dB  
 SF02: 200.1312008 MHz

F2 - Processing parameters  
 SI: 32768  
 SF: 50.3282447 MHz  
 VOP: EM  
 SSB: 0  
 LB: 3.00 Hz  
 GB: 0  
 PC: 0.20

ID (NMR) plot parameters:  
 CX: 23.00 cm  
 F1P: 200.000 GHz  
 F1: 10064.55 MHz  
 F2P: -10.000 GHz  
 F2: -503.23 Hz  
 PPMCHN: 9.13043 ppm/cm  
 HZCHN: 459.48854 Hz/cm

Figure-S3.  $^1\text{H}$  NMR of *trans*-**1a**

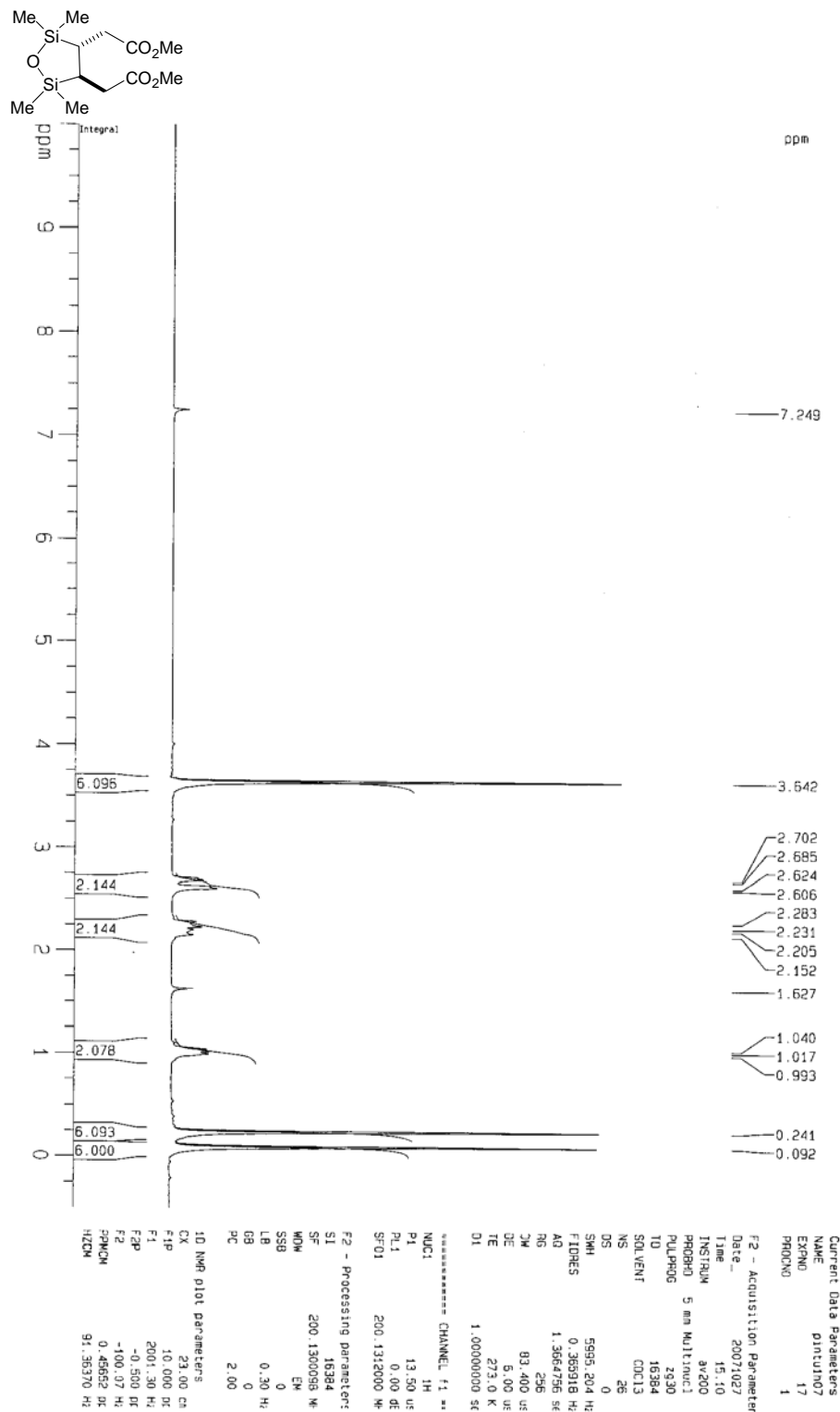


Figure-S4.  $^{13}\text{C}$  NMR of *trans*-**1a**

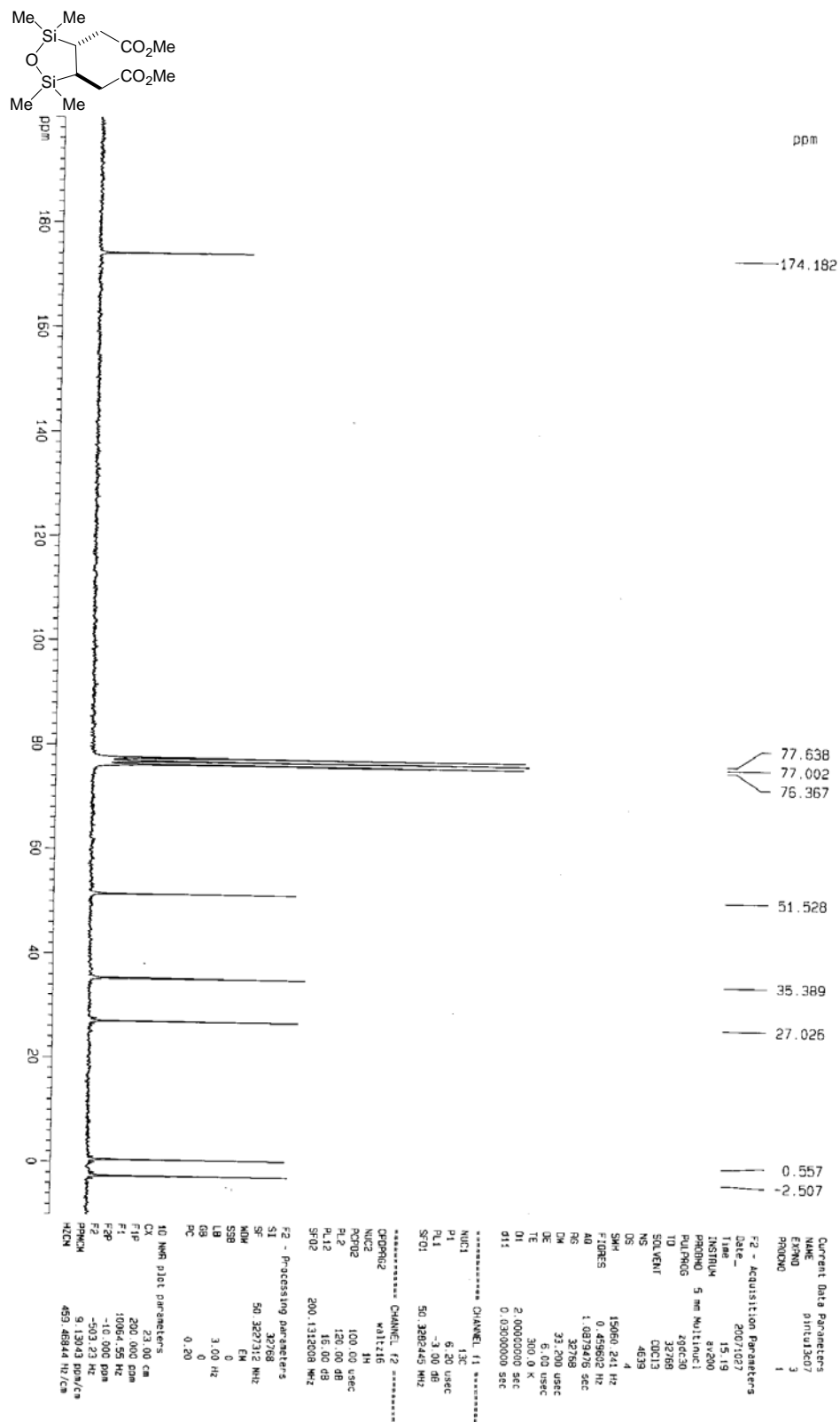
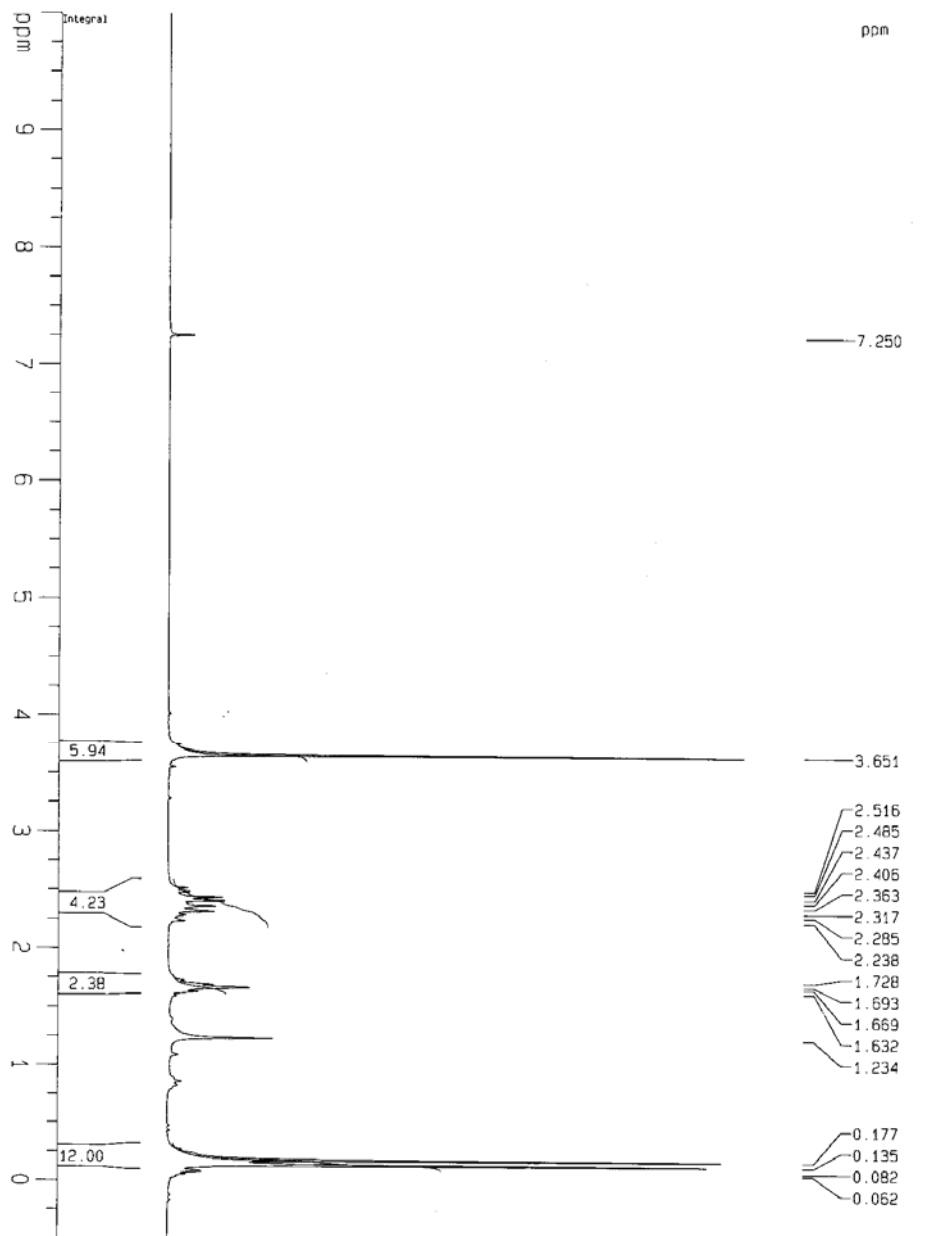
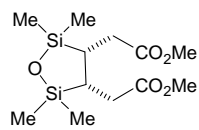


Figure-S5. <sup>1</sup>H NMR of *cis-1a*



```

Current Data Parameters
NAME      product7
EXPNO    284
PROCNO   1

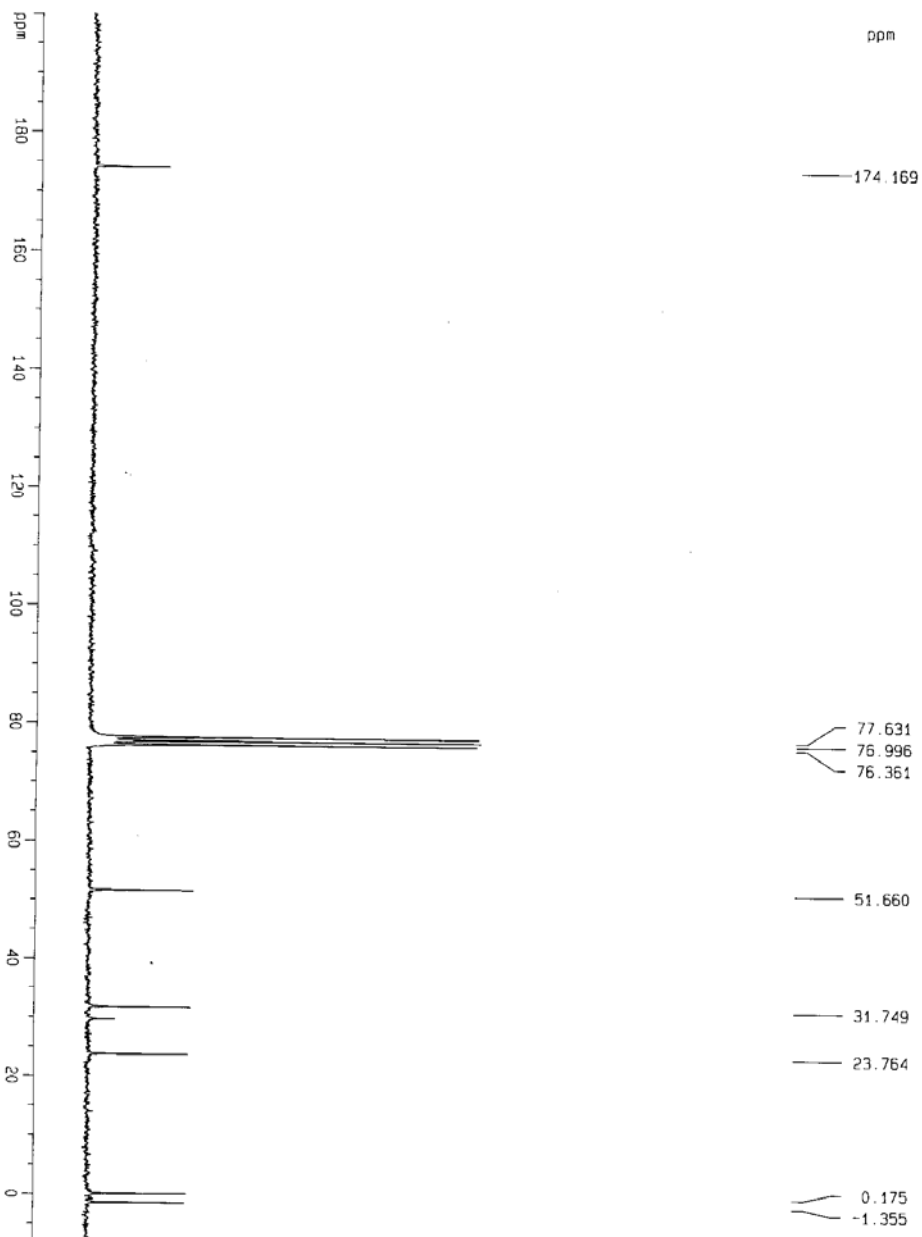
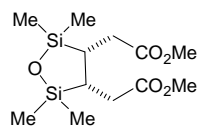
F2 - Acquisition Parameters
Date_    20080814
Time     18.33
INSTRUM  av200
PROBHD   5 mm Multinucl
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        30
DS        0
SHH       5995.204 Hz
FIDRES    0.365918 Hz
AQ         1.3664756 sF
RG         256
DM         63.400 uF
DE         6.00 uF
TE         273.0 K
D1         1.00000000 sF

***** CHANNEL f1 **
NUC1      1H
P1        13.50 uF
PL1       0.00 dB
SFO1     200.1312000 MHz

F2 - Processing parameters
SI         16384
SF         200.1300098 MHz
EN         0
SSB        0
LB         0.30 Hz
GB         0
PC         2.00

1D NMR plot parameters
CX         23.00 cm
FAP        10.000 Hz
F1         2001.30 Hz
F2         -0.500 Hz
PPH1CM    -100.07 Hz
PPH2CM    0.45552 Hz
HZCM      91.56370 Hz
    
```

Figure-S6.  $^{13}\text{C}$  NMR of *cis*-**1a**



Current Data Parameters  
 NAME pmt13c07  
 EXPNO 35  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080818  
 Time 11:27  
 INSTRUM av200  
 PROBHD 5 mm NLI11nc1  
 PULPROG zgpg30  
 ID 32768  
 SOLVENT CDCl3  
 NS 3931  
 DS 4  
 SM 15060.241 Hz  
 FLORES 0.459602 Hz  
 AQ 1.0819476 sec  
 RG 32768  
 DM 31.5000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1  $^{13}\text{C}$   
 P1 6.30 usec  
 PL1 -3.00 dB  
 SFO1 50.3282445 MHz

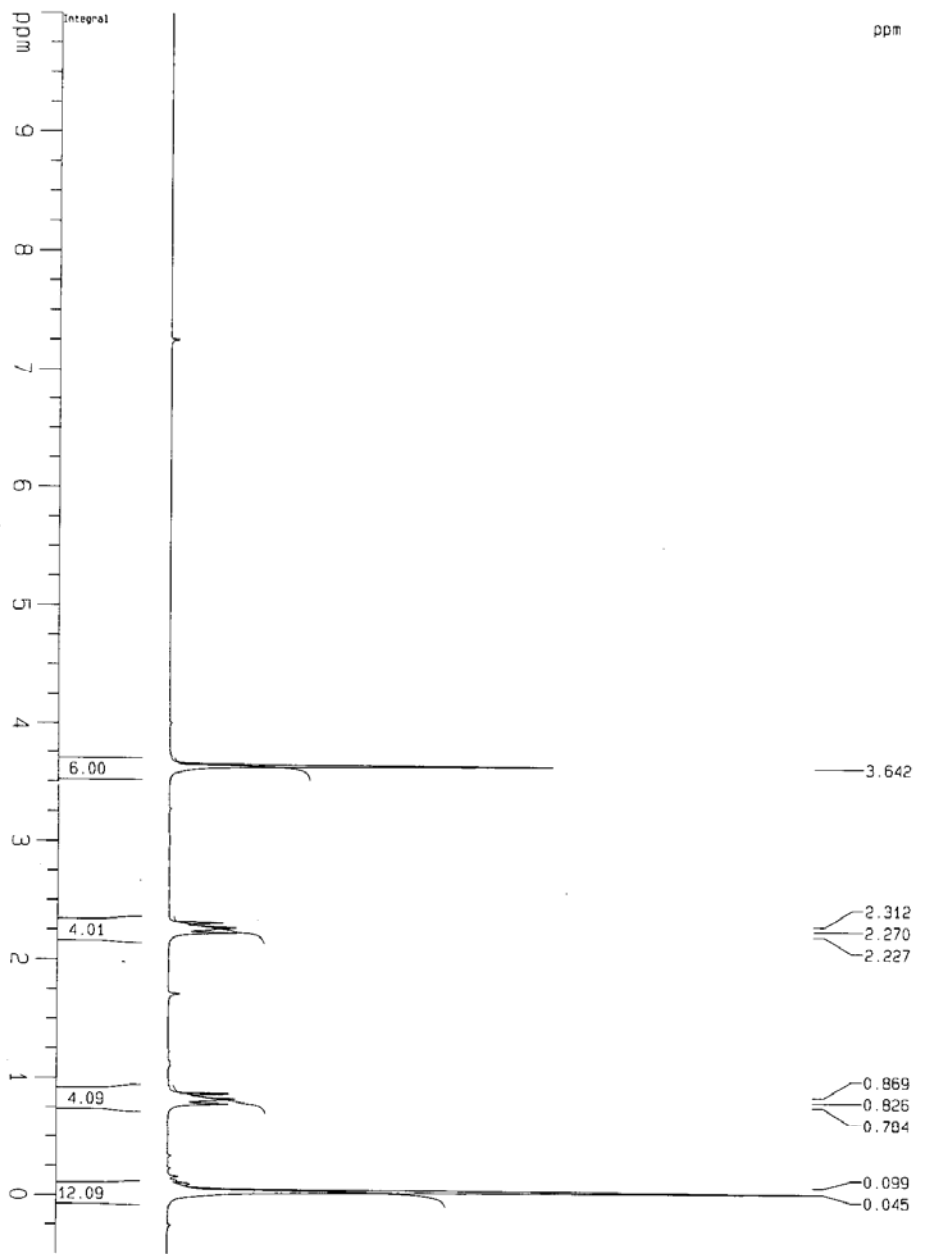
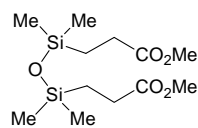
\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CHPROG2 waltz16  
 NUC2  $^1\text{H}$   
 P2 100.00 usec  
 PL2 120.00 dB  
 SFO2 50.3282445 MHz

F2 - Processing parameters  
 SI 32768  
 SF 50.3282716 MHz  
 KW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 0.20

1D NMR plot parameters  
 CX 23.00 cm  
 F1P 200.000 ppm  
 F1 10064.95 Hz  
 F2P -503.23 Hz  
 F2 9.18043 ppm/cm  
 PPM0H 459.46844 Hz/cm  
 NCH



Figure-S7. <sup>1</sup>H NMR of 5a



Current Data Parameters  
 NAME p1nuh07  
 EXPNO 9  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071022  
 Time 12.53  
 INSTRUM av200  
 PROBRG 5 mm Multinuc1  
 PULPROG zg30  
 TD 65536  
 SFO1 16384  
 SOLVENT DMS-D6  
 NS 37  
 DS 0  
 SMH 5995.204 Hz  
 FIDRES 0.365918 Hz  
 AQ 1.3654756 sec  
 RG 16  
 DM 83.400 us  
 DE 6.00 us  
 TE 273.0 K  
 D1 1.00000000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*  
 NUC1 1H  
 P1 13.50 us  
 PL1 0.00 dB  
 SFO1 200.1312000 MHz

F2 - Processing parameters  
 SI 16384  
 SF 200.1300099 MHz  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

1D NMR d10c parameters  
 CX 23.00 ca  
 F1P 10.000 pf  
 F1 2001.30 Hz  
 F2P -0.500 pf  
 F2 -100.07 Hz  
 PPGCM 0.46682 pf  
 HZM 51.56370 Hz

Figure-S8.  $^{13}\text{C}$  NMR of **5a**

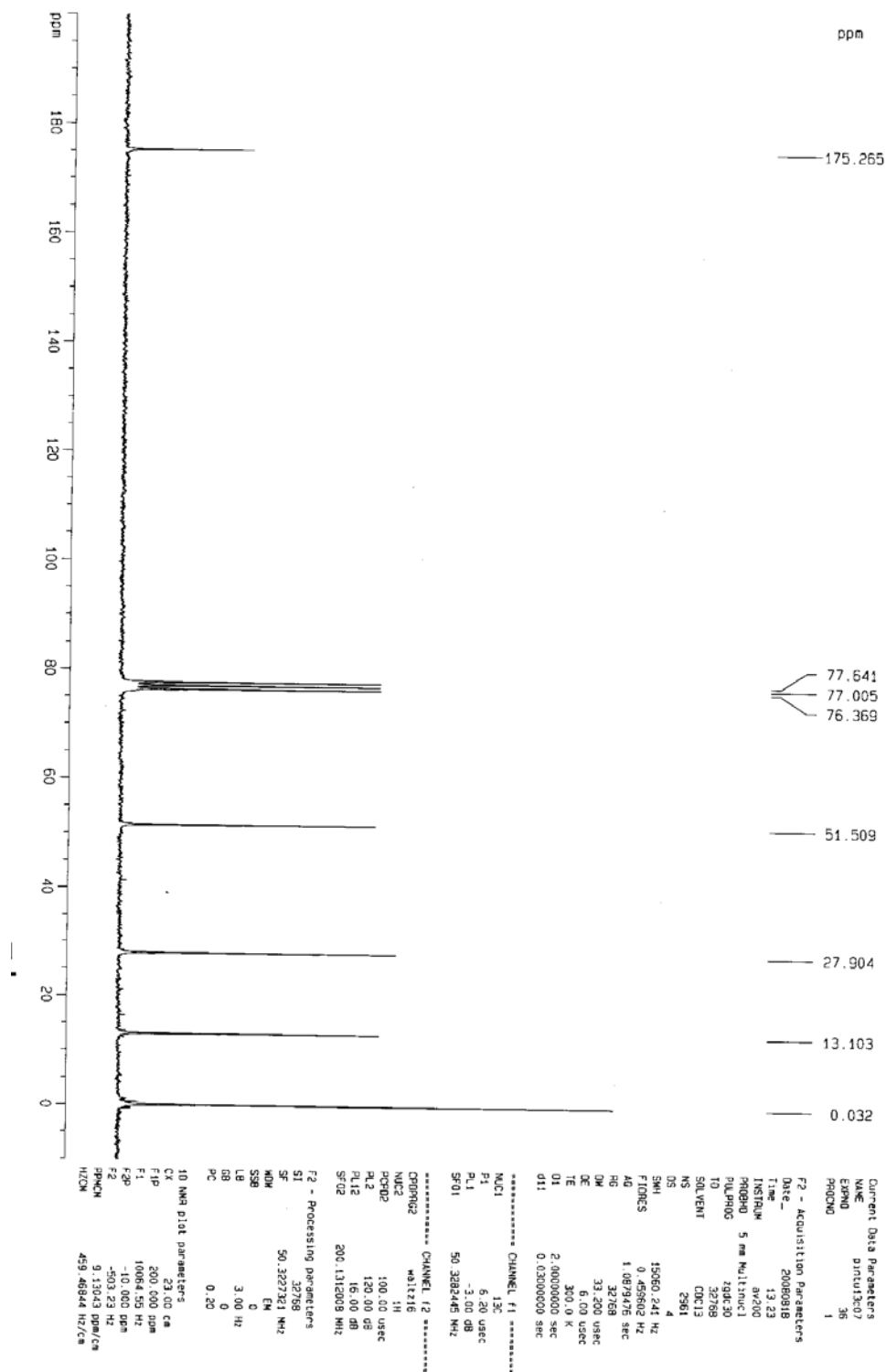
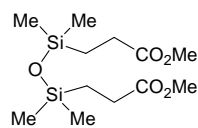


Figure-S9. <sup>1</sup>H NMR of 9a

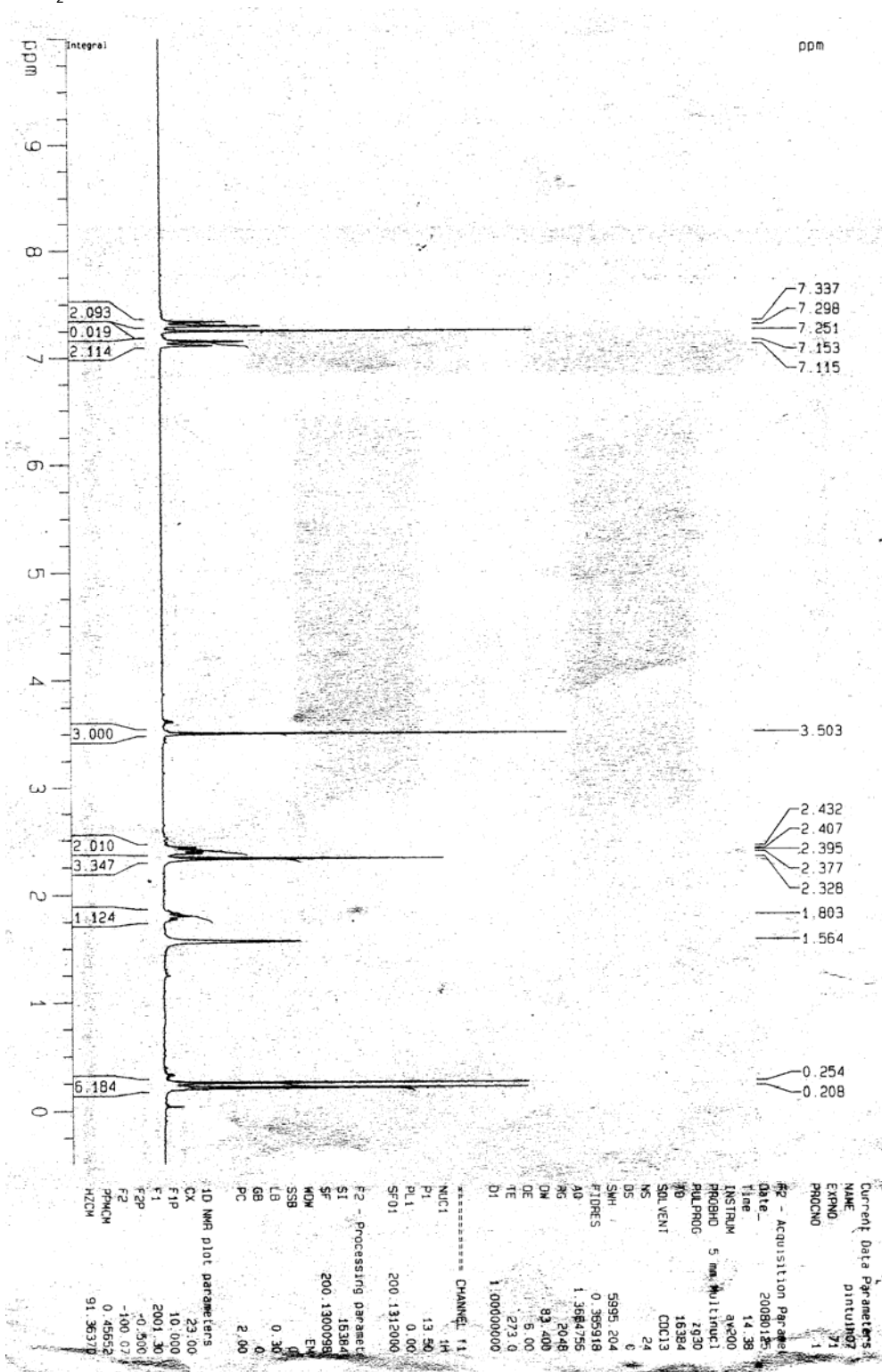
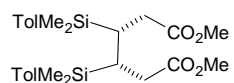


Figure-S10. <sup>1</sup>H NMR of **11a**

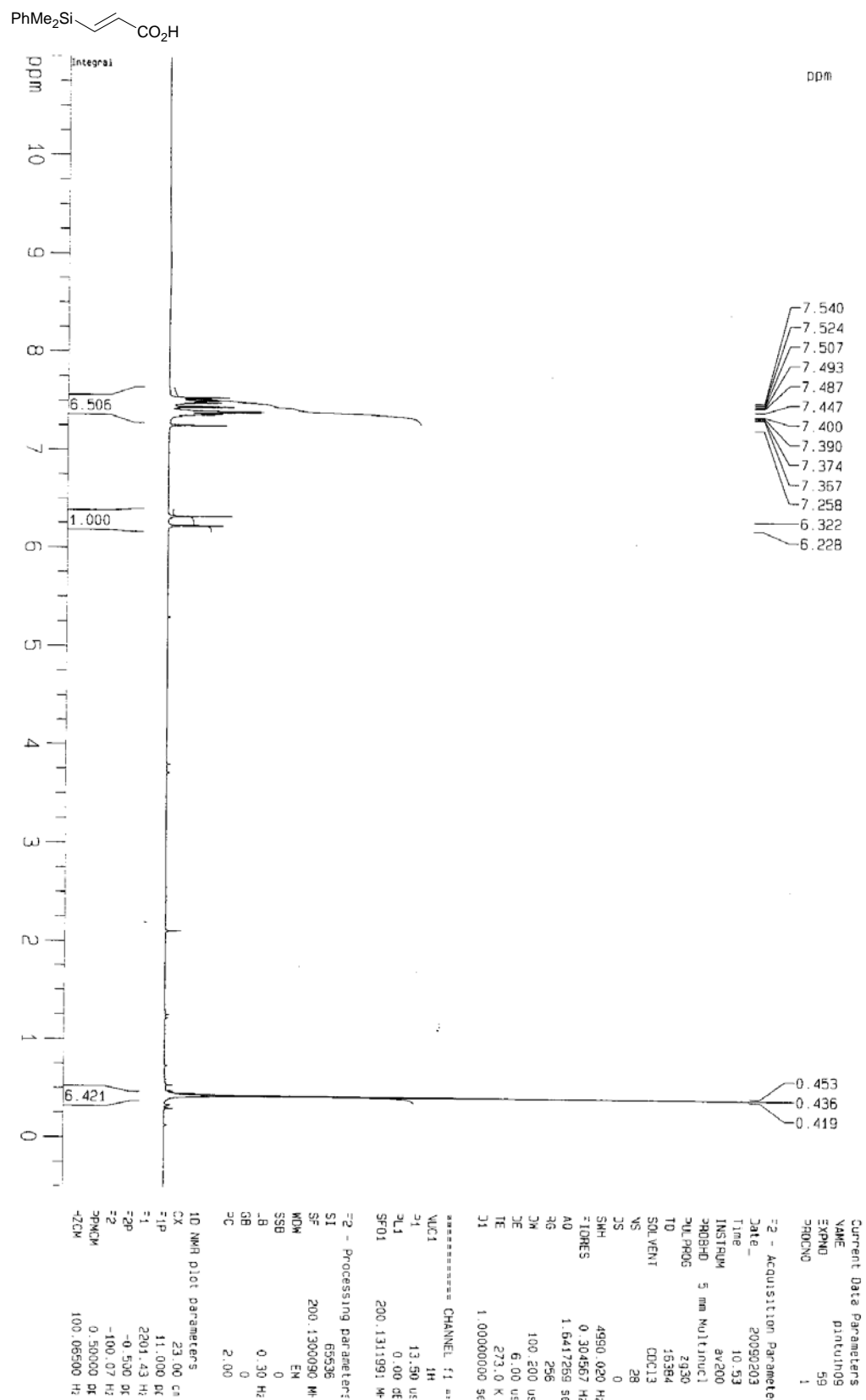


Figure-S11. <sup>13</sup>C NMR of **11a**

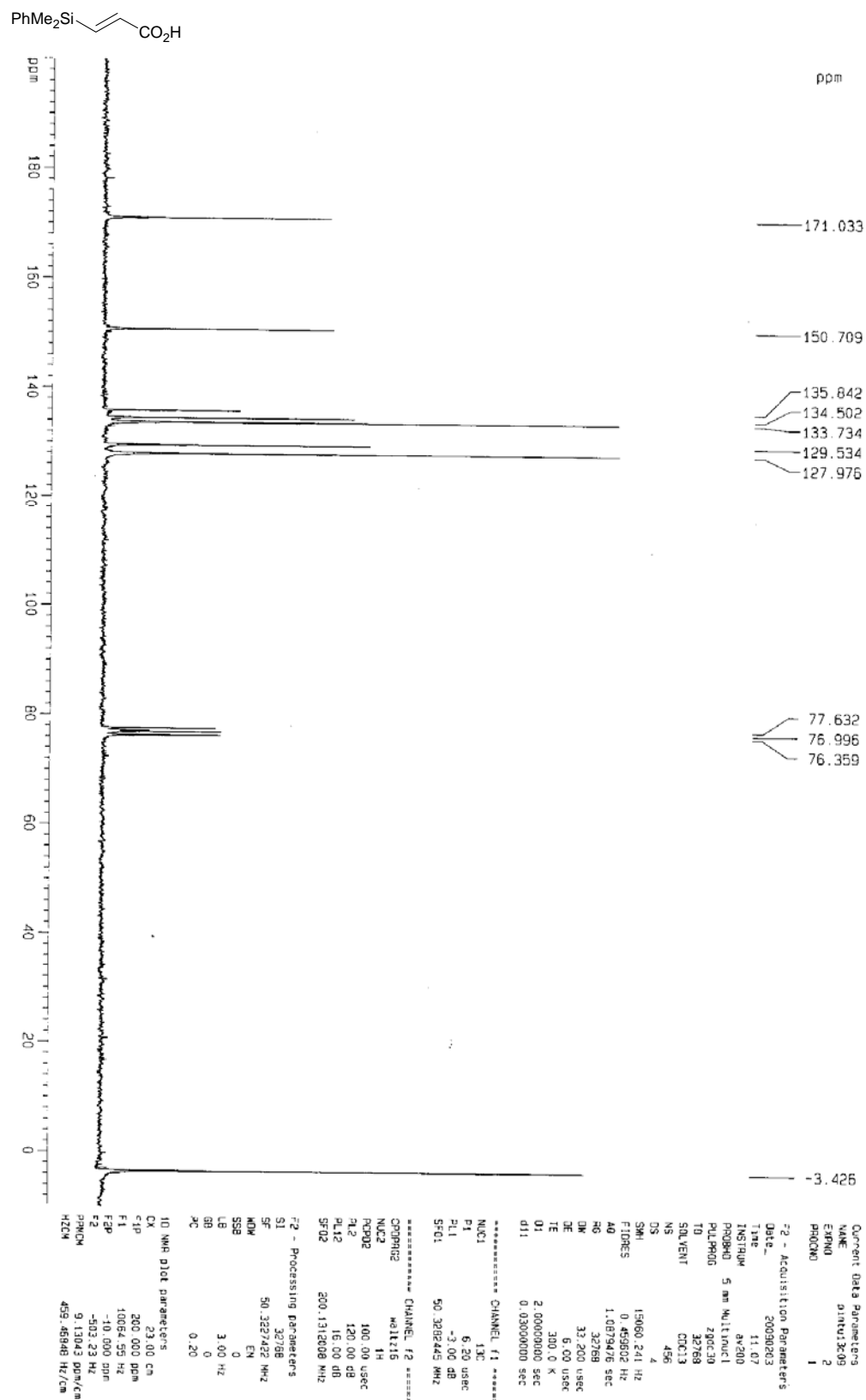


Figure-S12. <sup>1</sup>H NMR of **11b**

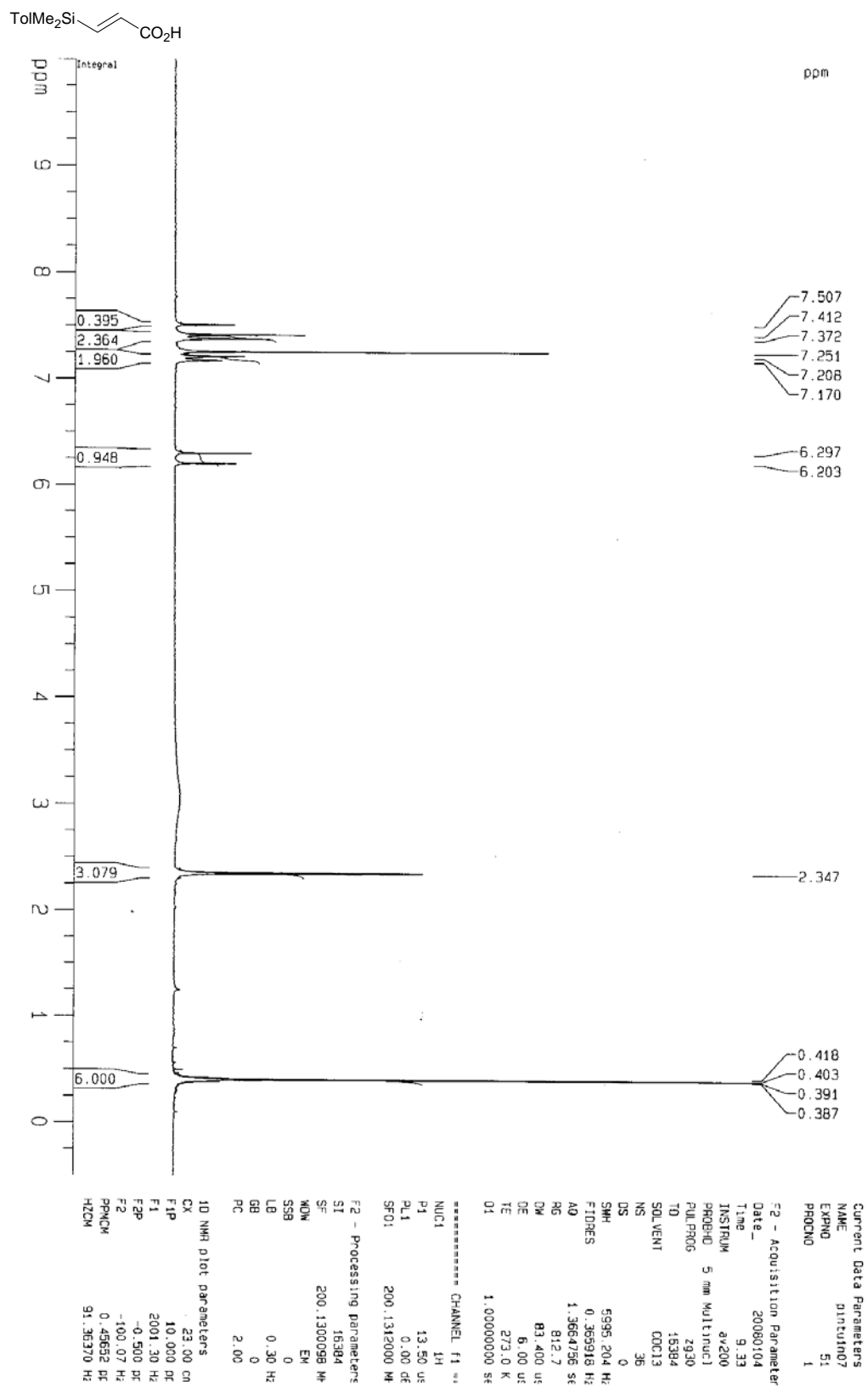


Figure-S13.  $^{13}\text{C}$  NMR of **11b**

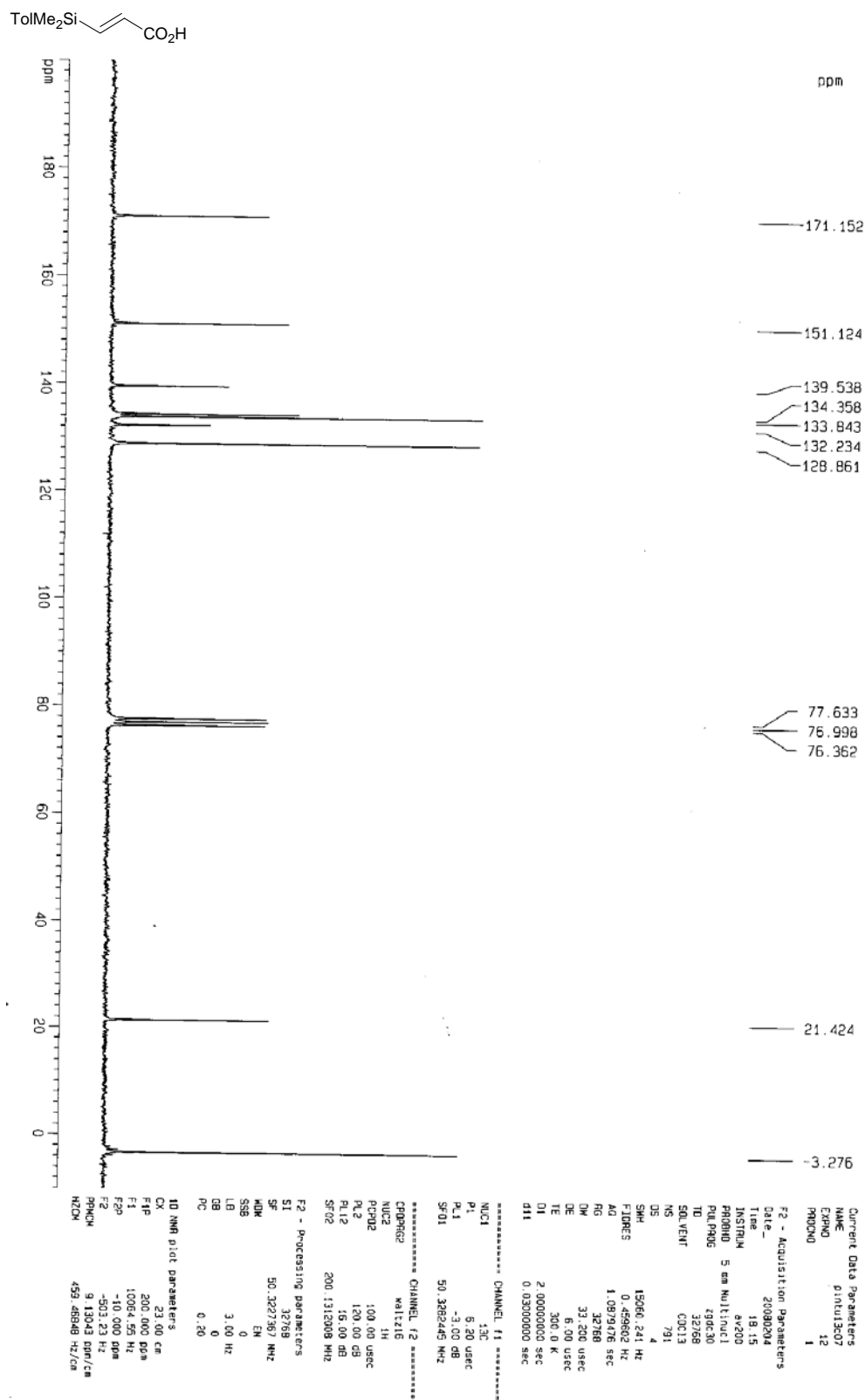


Figure-S14.  $^1\text{H}$  NMR of **10a**

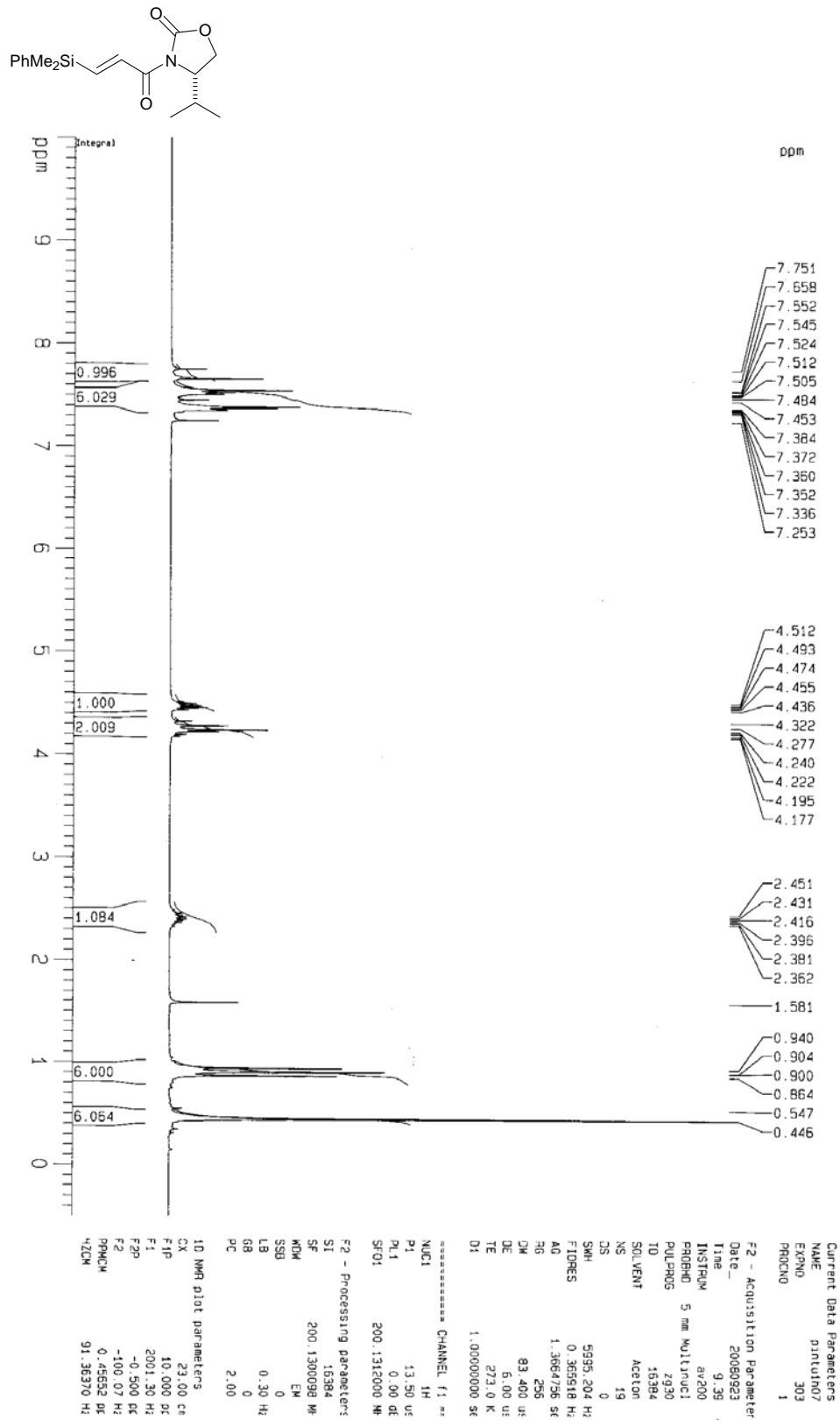




Figure-S15.  $^{13}\text{C}$  NMR of **10a**

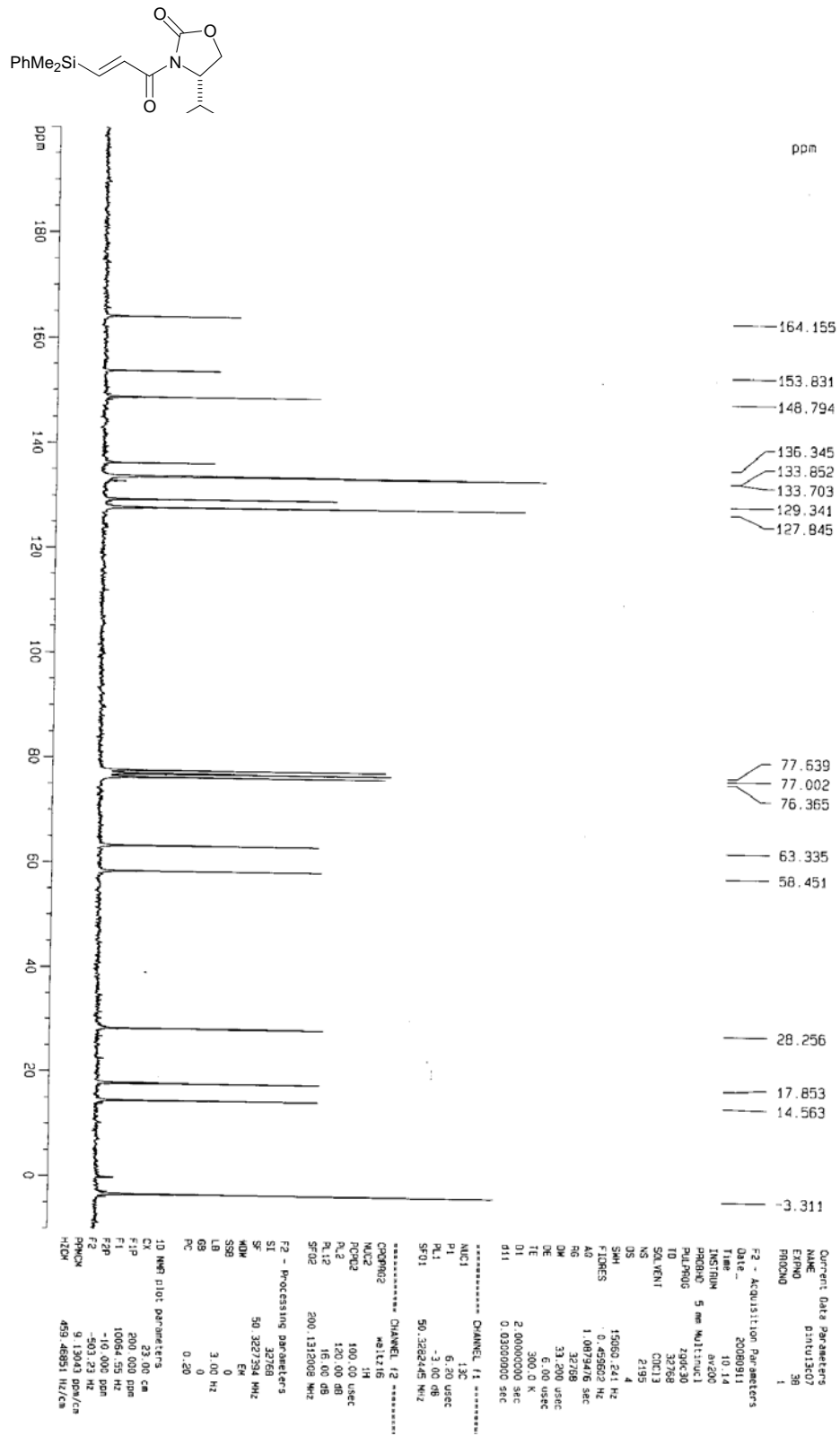


Figure-S16. <sup>1</sup>H NMR of **13a**

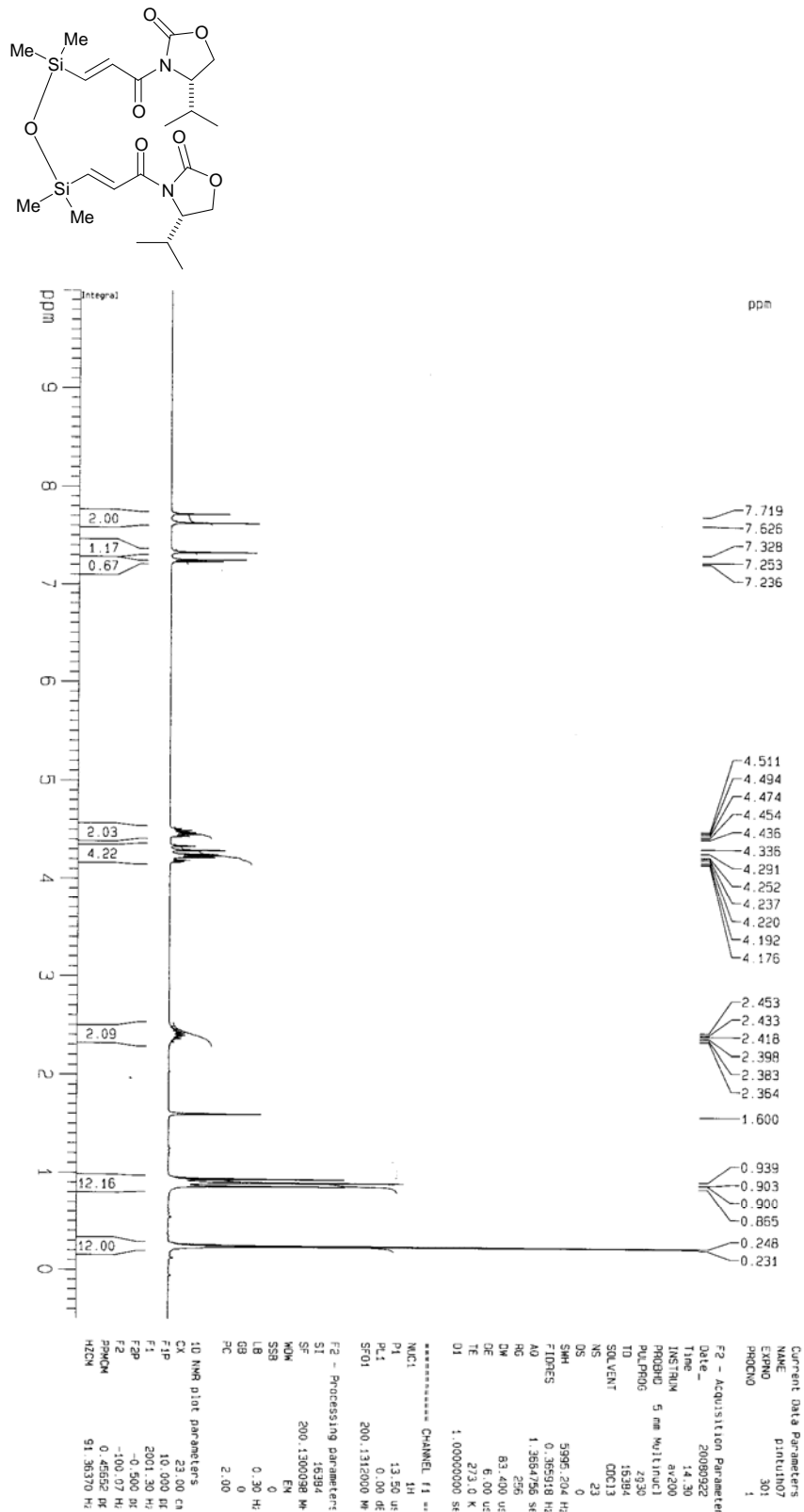


Figure-S17. <sup>13</sup>C NMR of **13a**

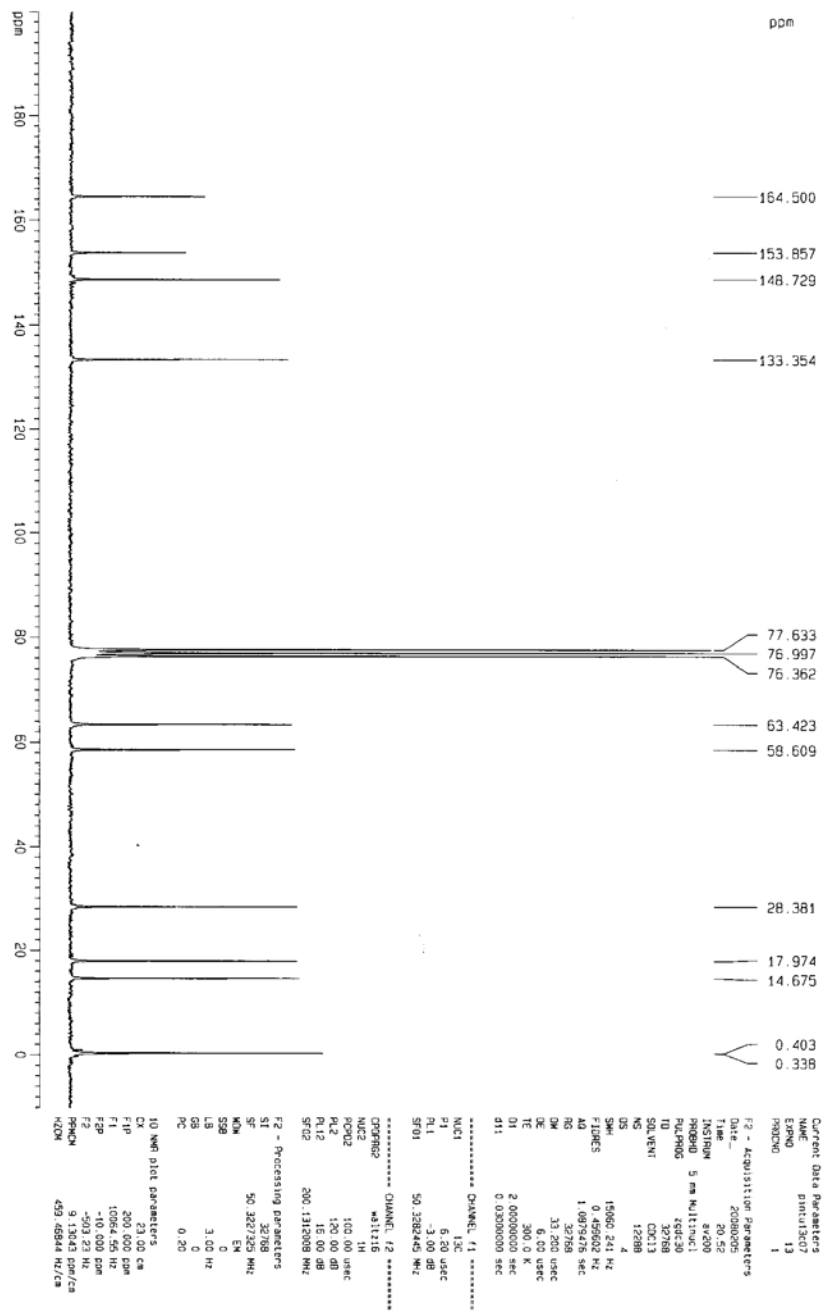
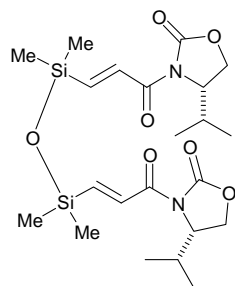






Figure-S20. <sup>1</sup>H NMR of *trans*-15a

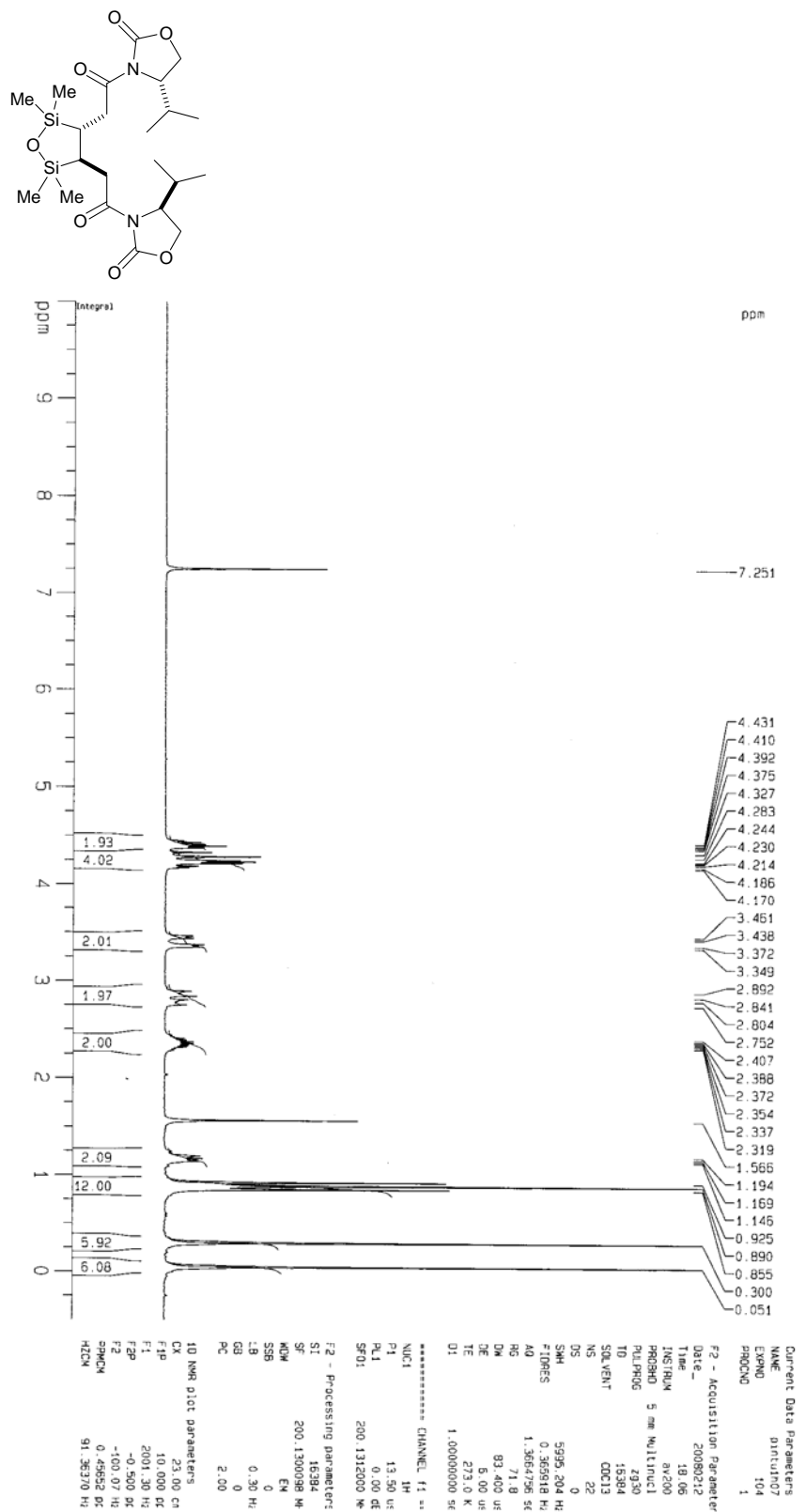


Figure-S21.  $^{13}\text{C}$  NMR of *trans*-15a

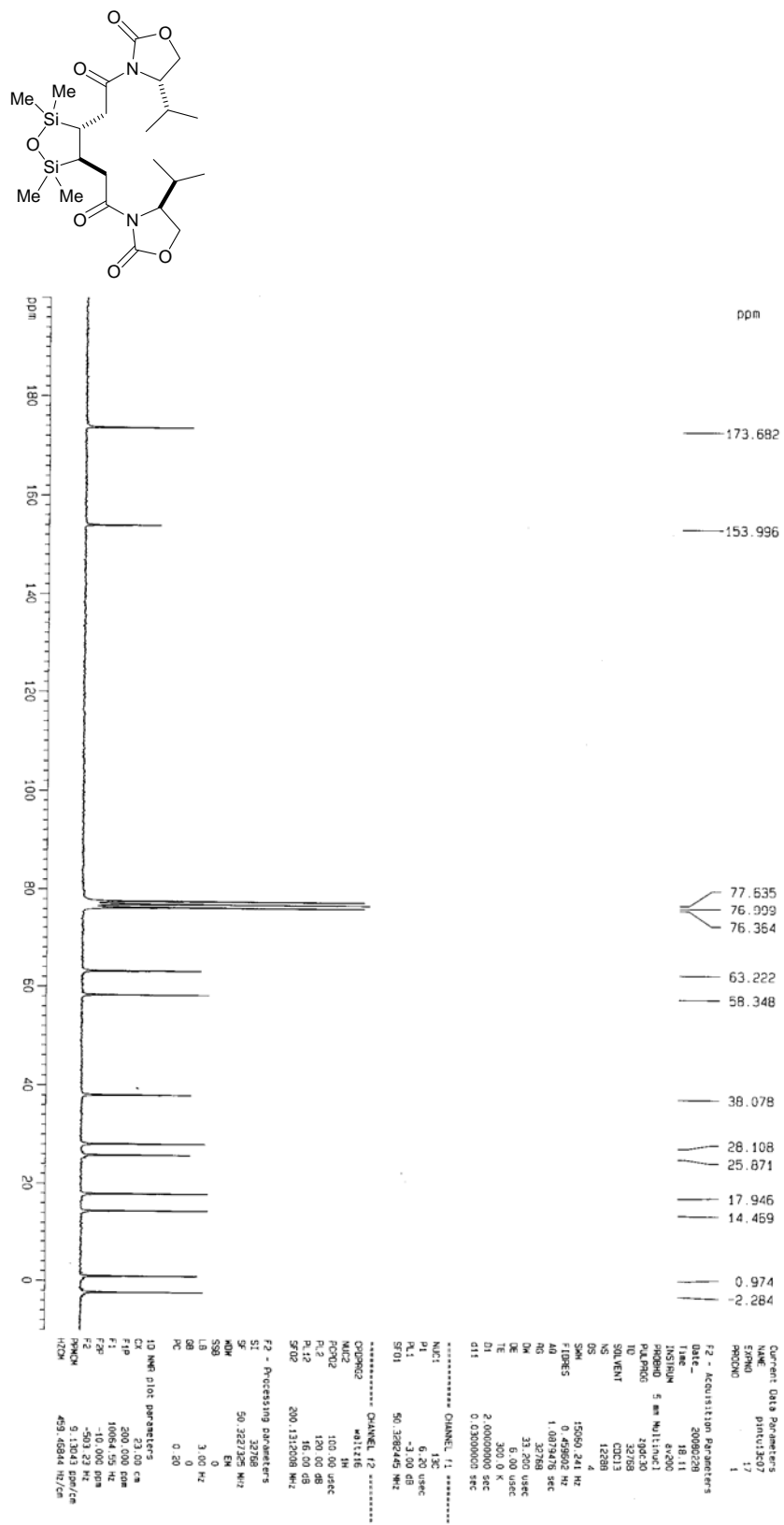


Figure-S22.  $^1\text{H}$  NMR of *cis*-**14a**

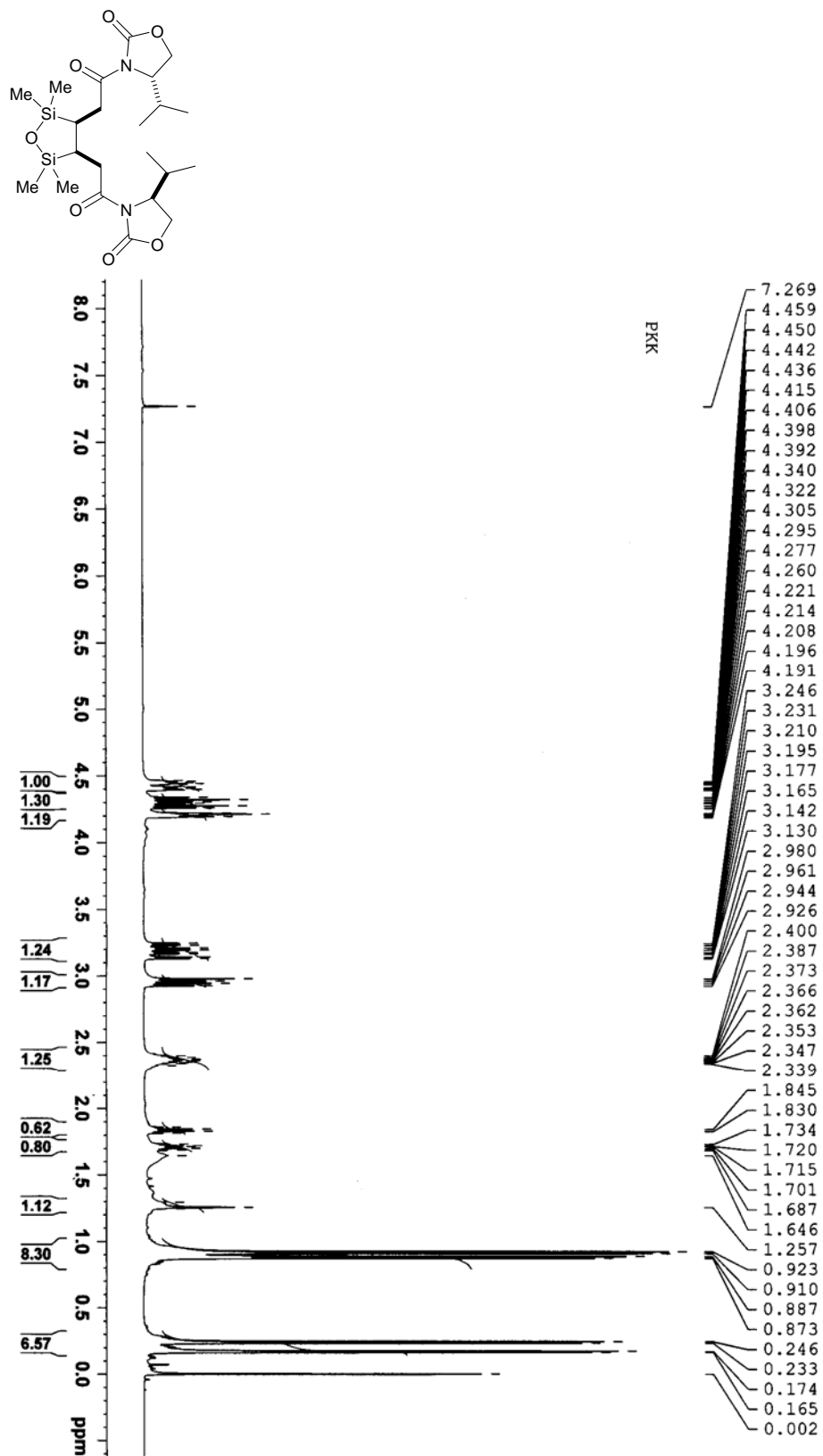




Figure-S23.  $^{13}\text{C}$  NMR of *cis*-**14a**

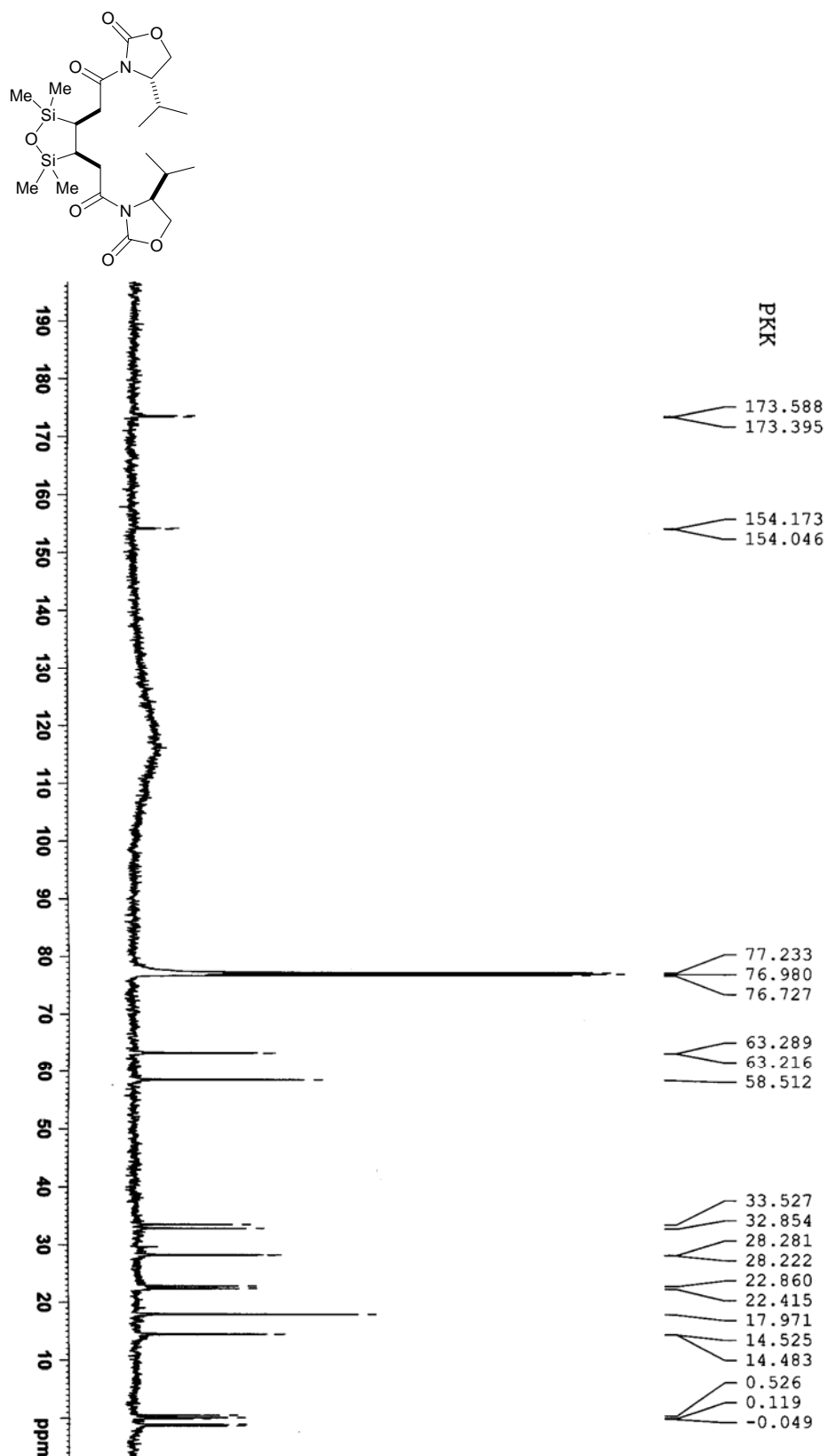


Figure-S24. <sup>1</sup>H NMR of **10b**

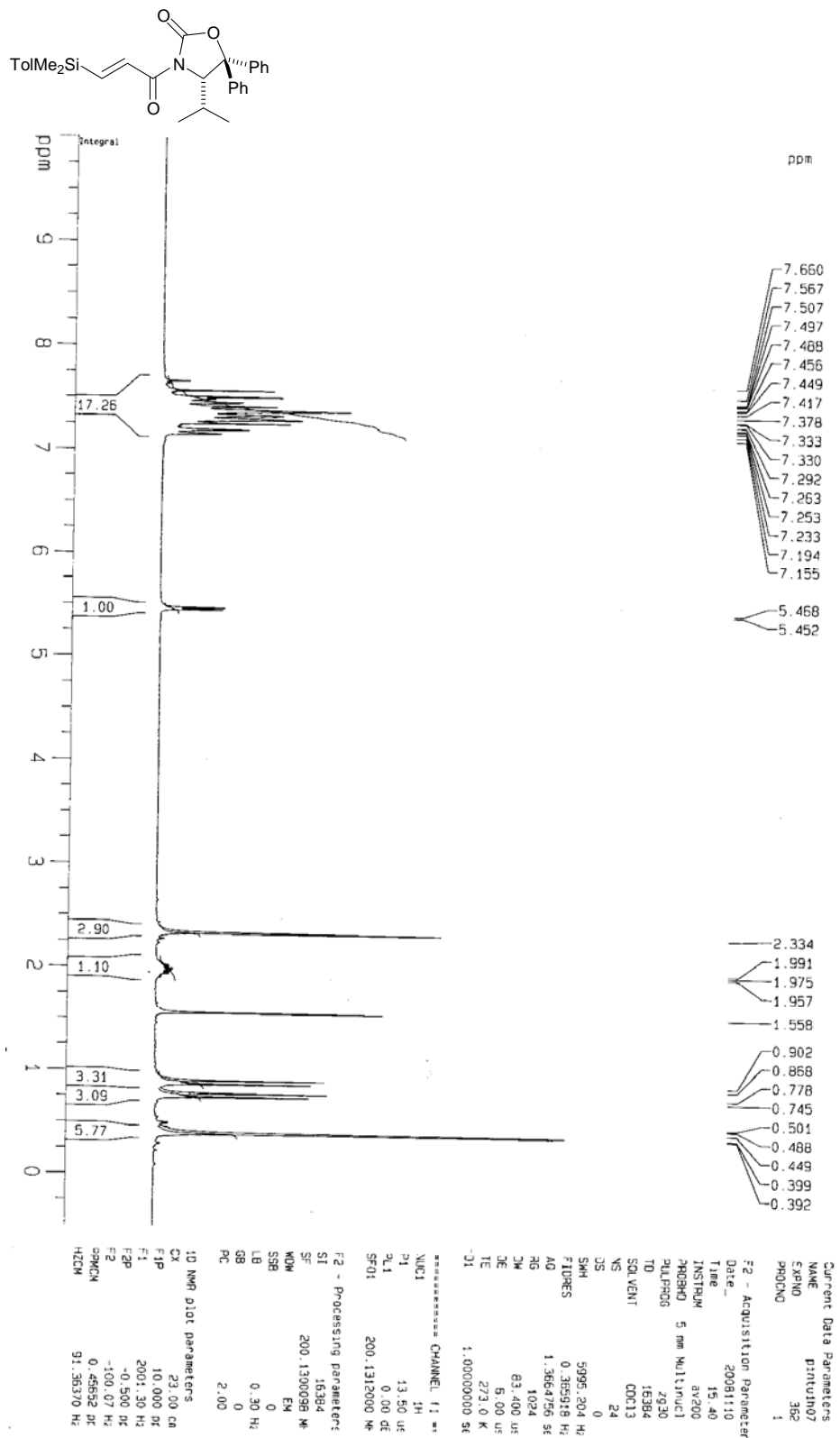


Figure-S25. <sup>13</sup>C NMR of **10b**

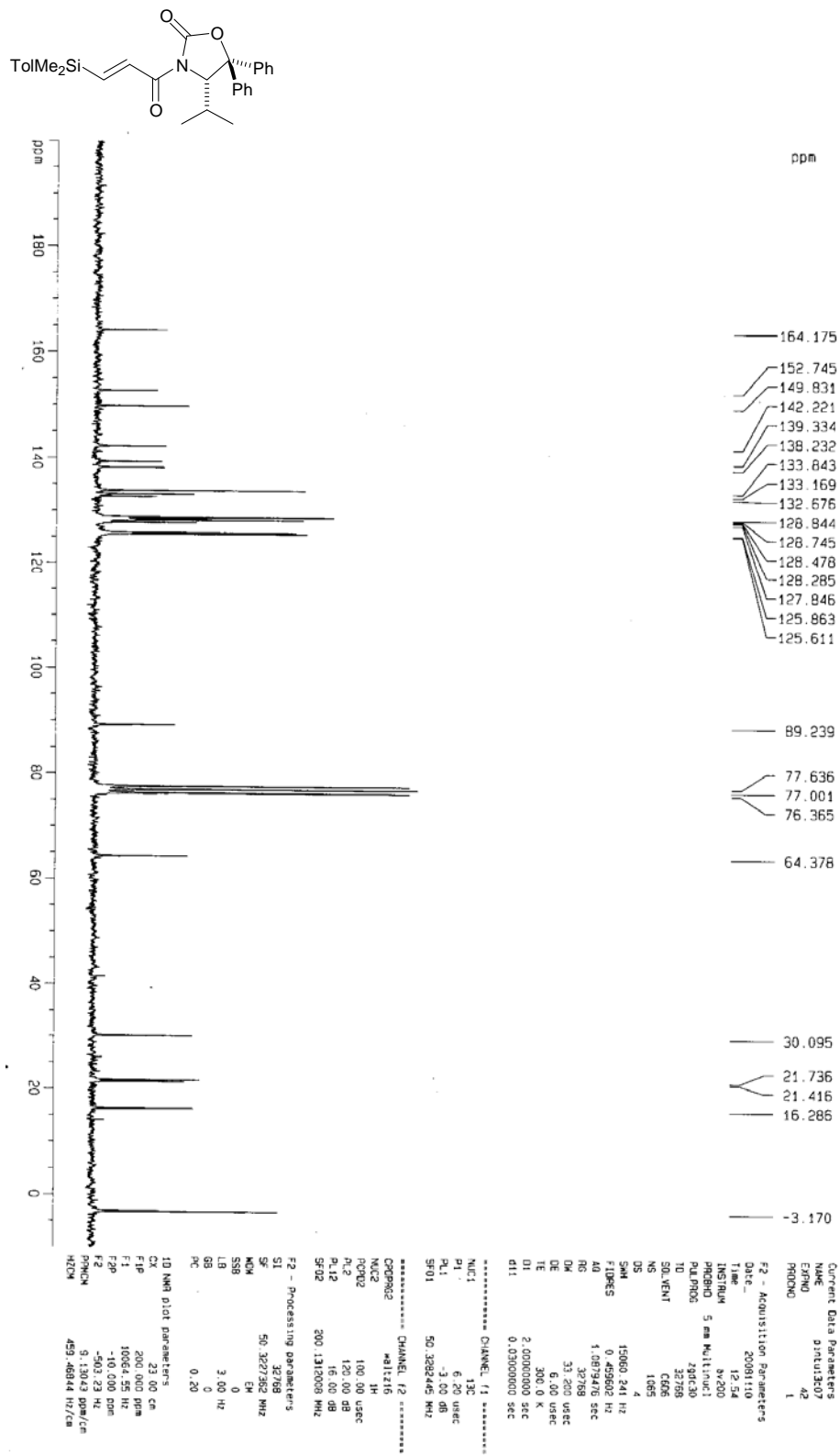


Figure-S26.  $^1\text{H}$  NMR of **13b**

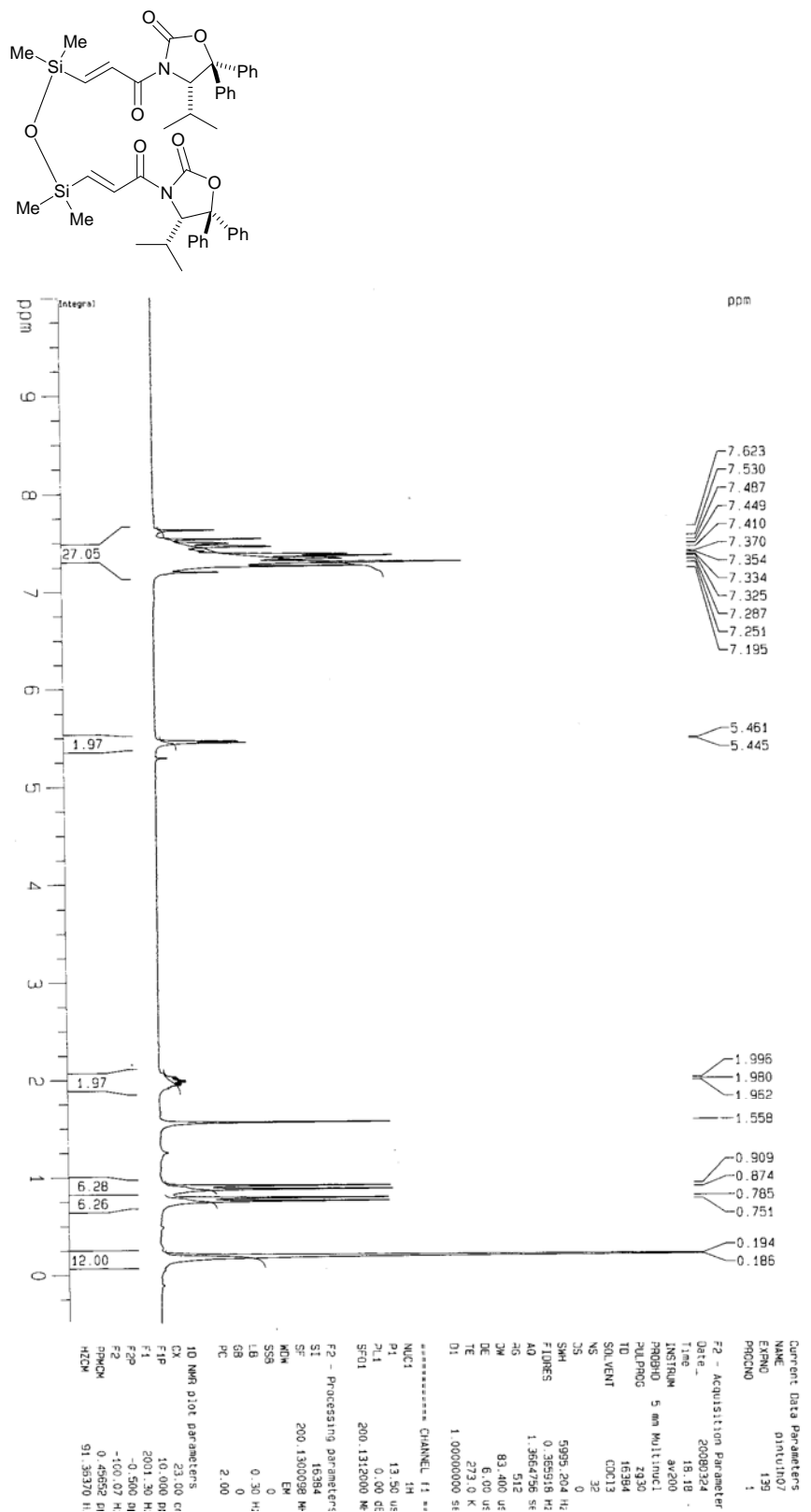


Figure-S27.  $^{13}\text{C}$  NMR of **13b**

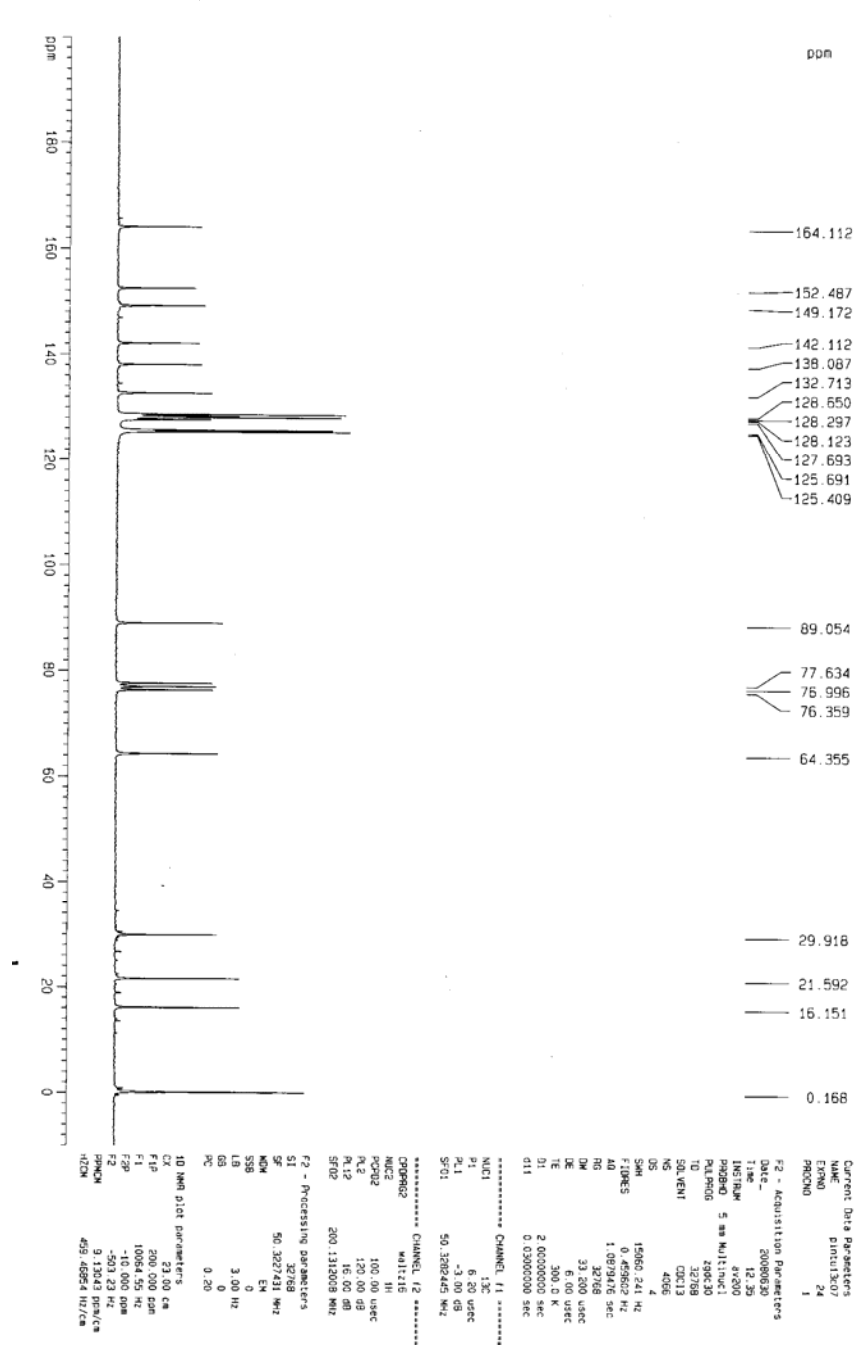
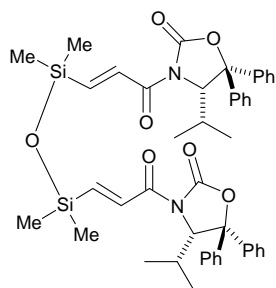


Figure-S28.  $^1\text{H}$  NMR of *trans*-**15b**

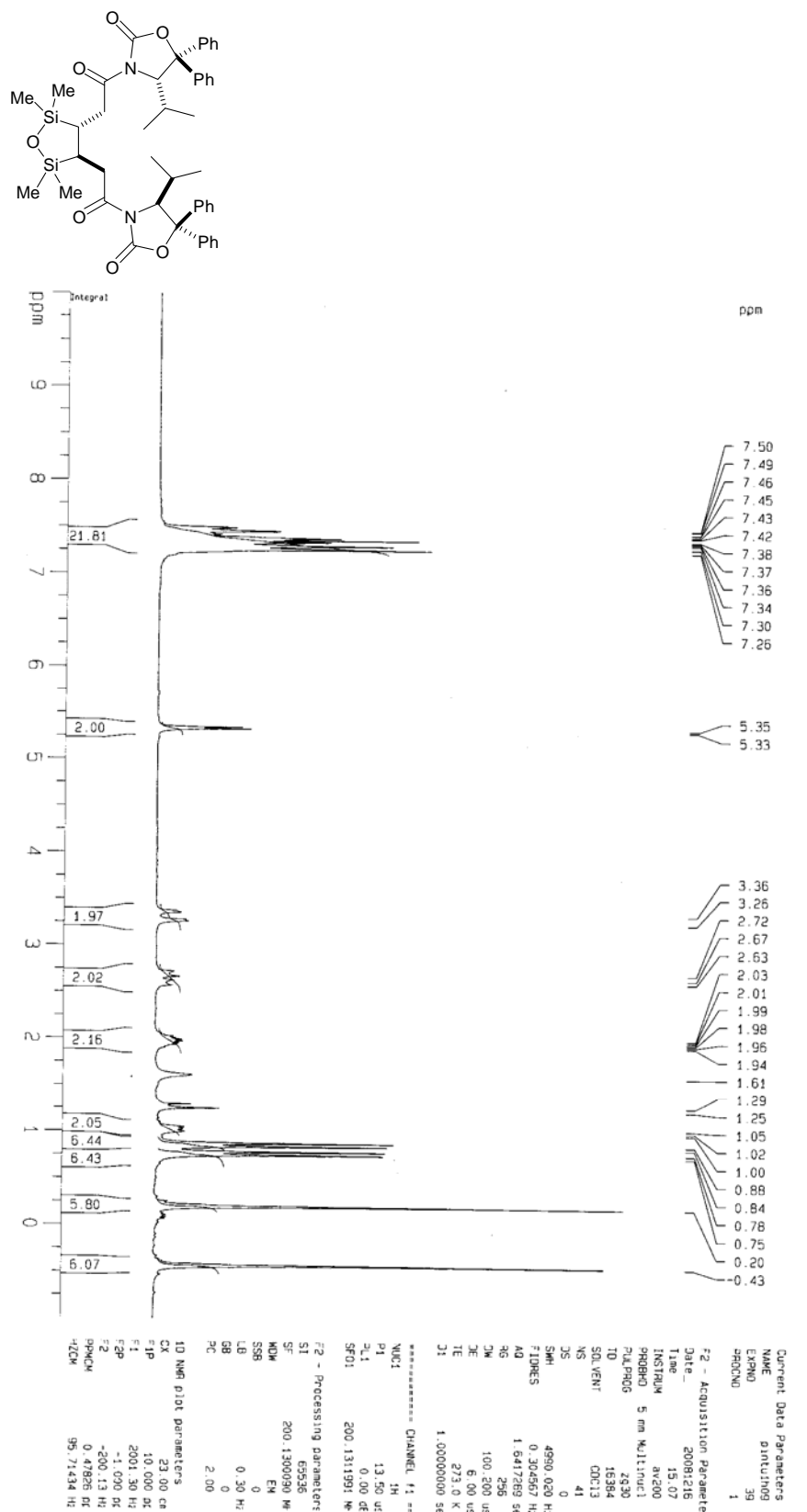




Figure-S30.  $^1\text{H}$  NMR of **10c**

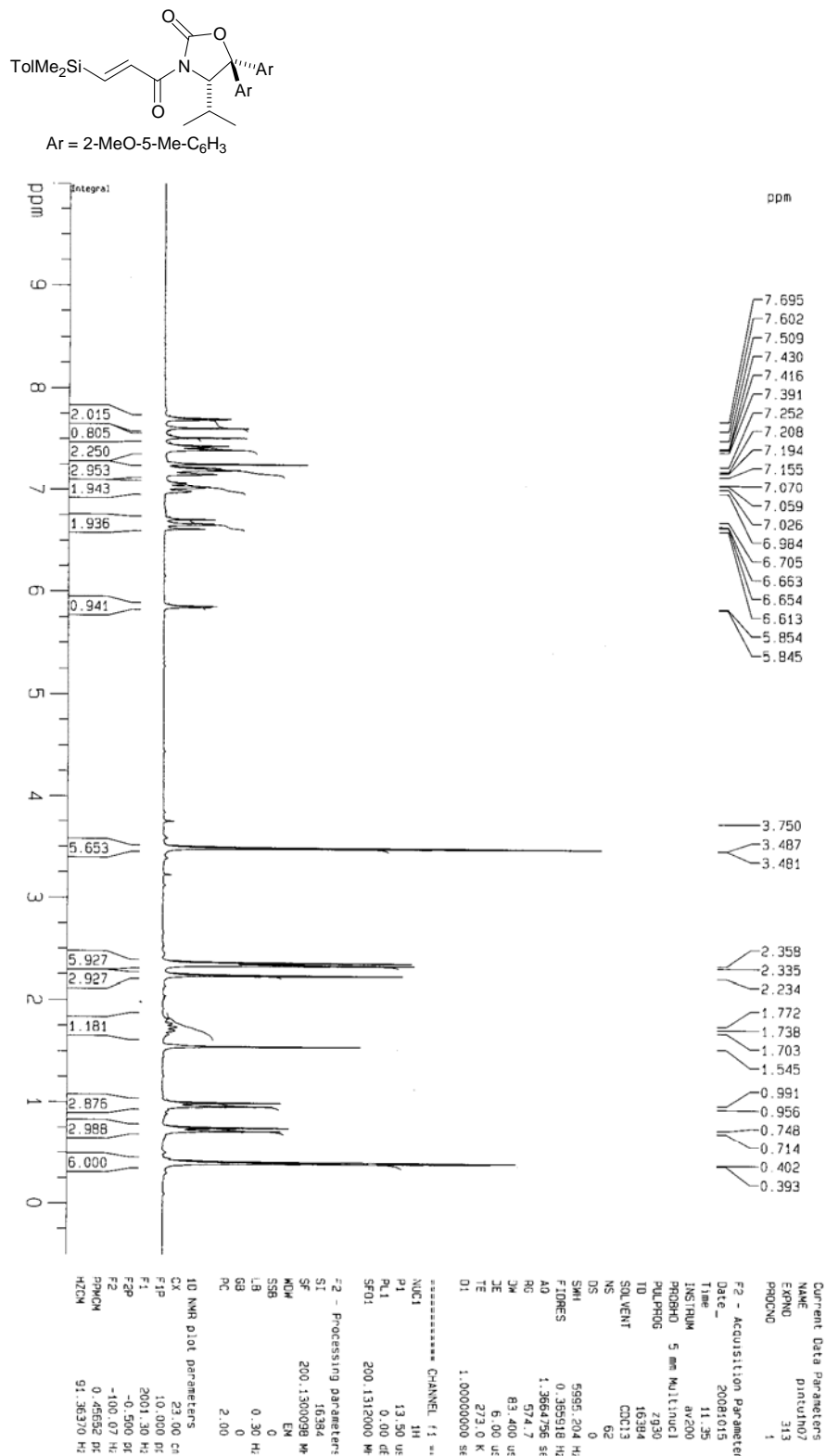




Figure-S31. <sup>13</sup>C NMR of **10c**

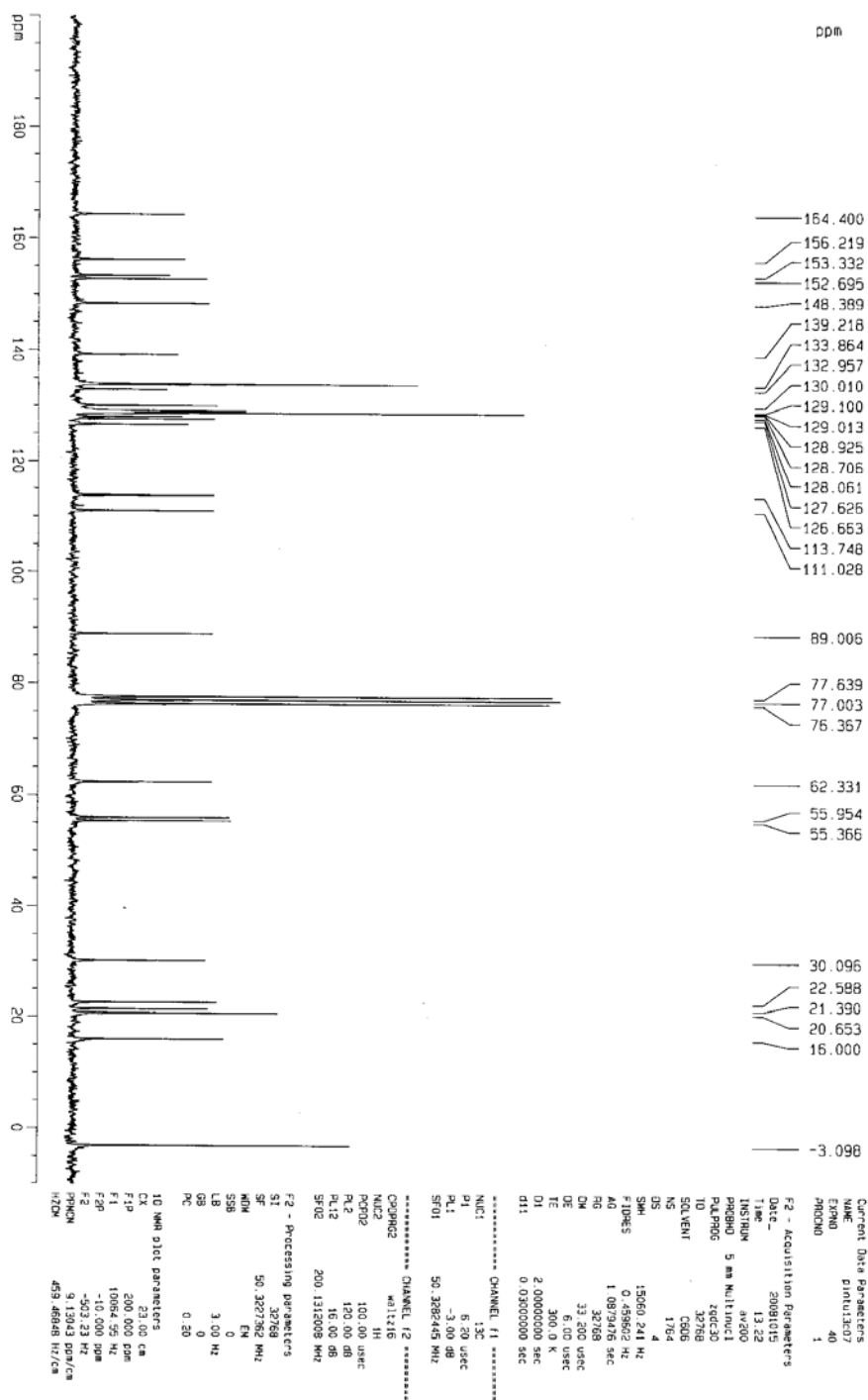
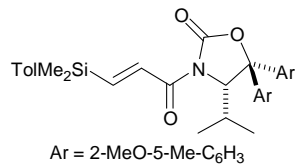


Figure-S32.  $^1\text{H}$  NMR of **13c**

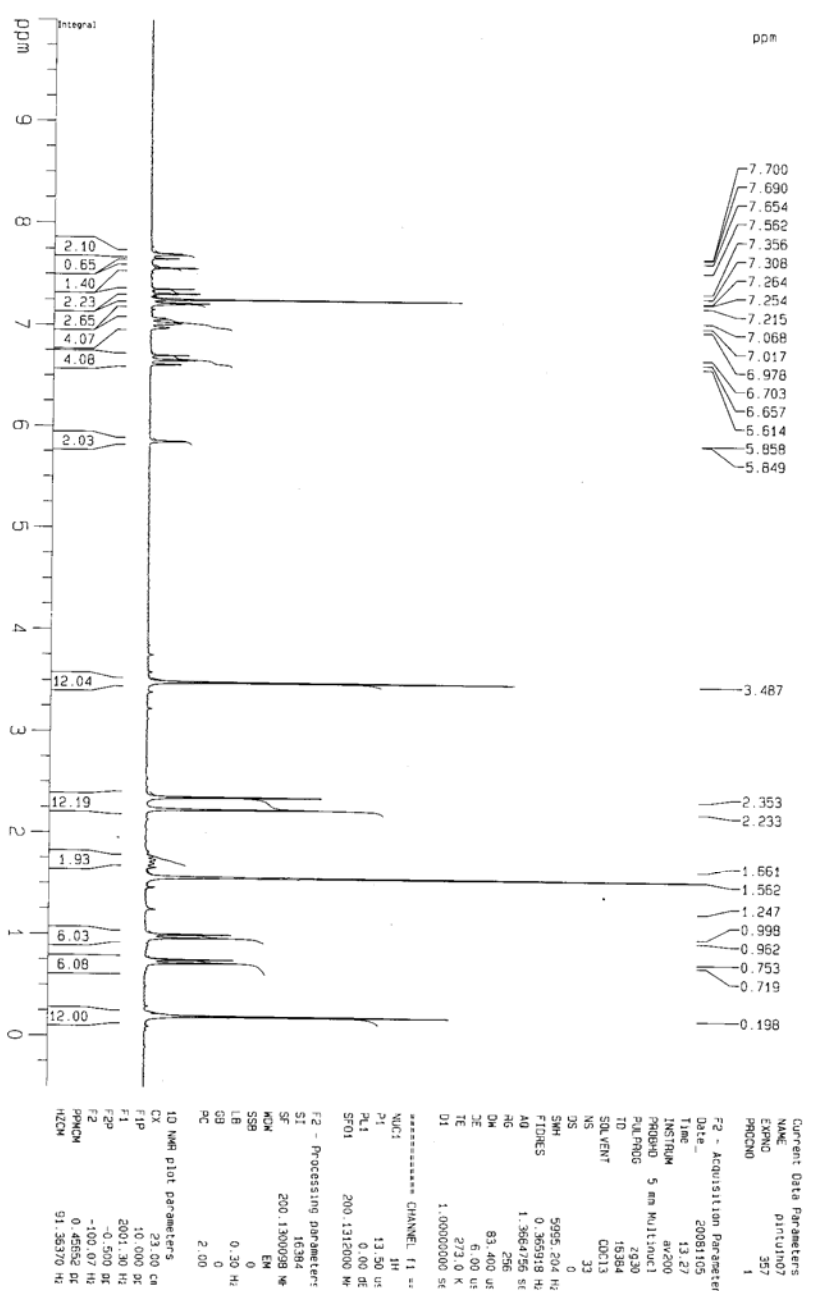
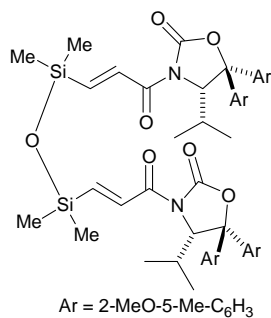


Figure-S33. <sup>13</sup>C NMR of **13c**

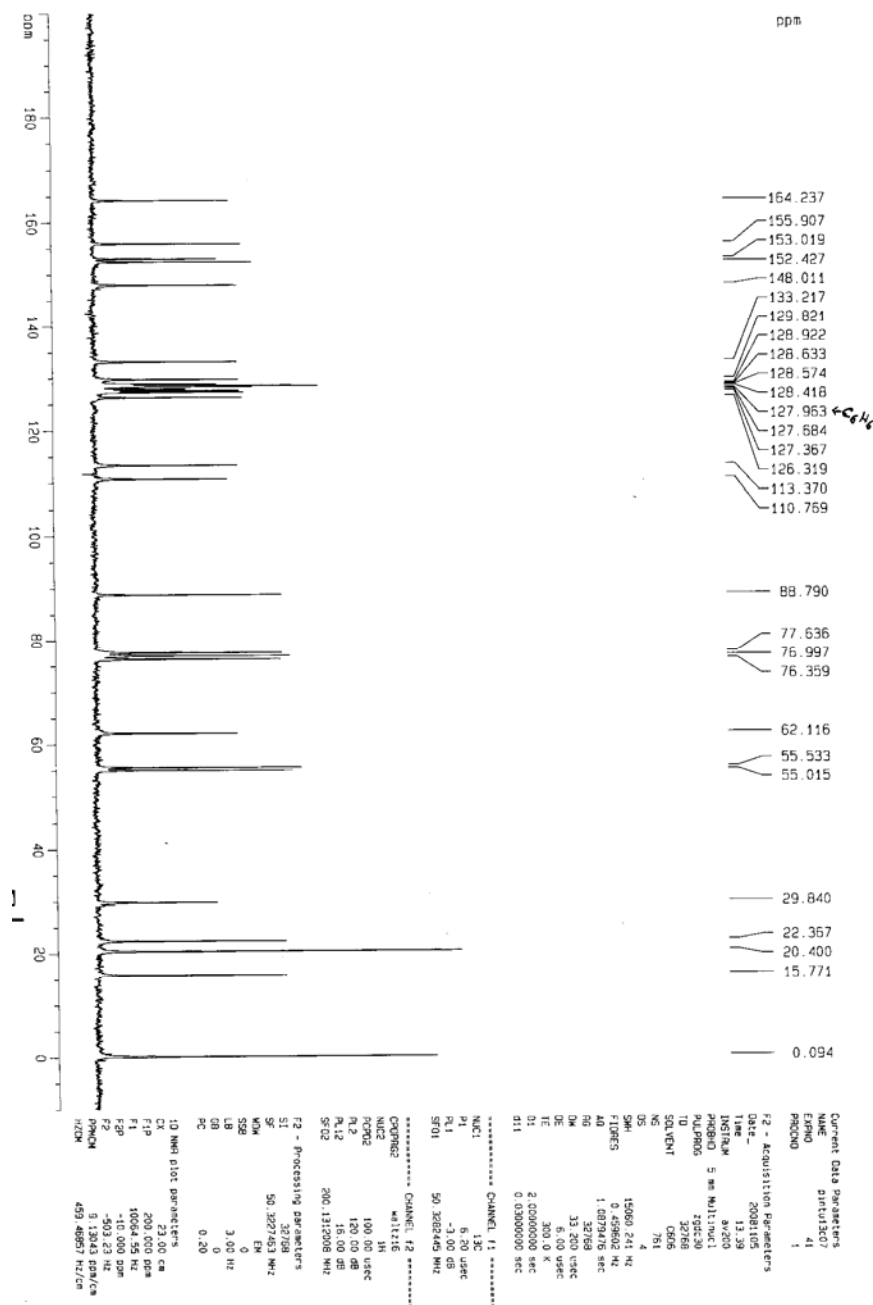
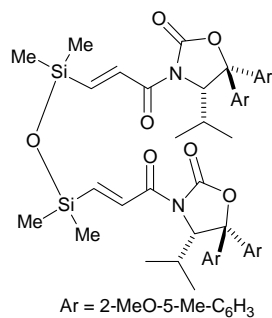


Figure-S34.  $^1\text{H}$  NMR of *trans*-**14c**

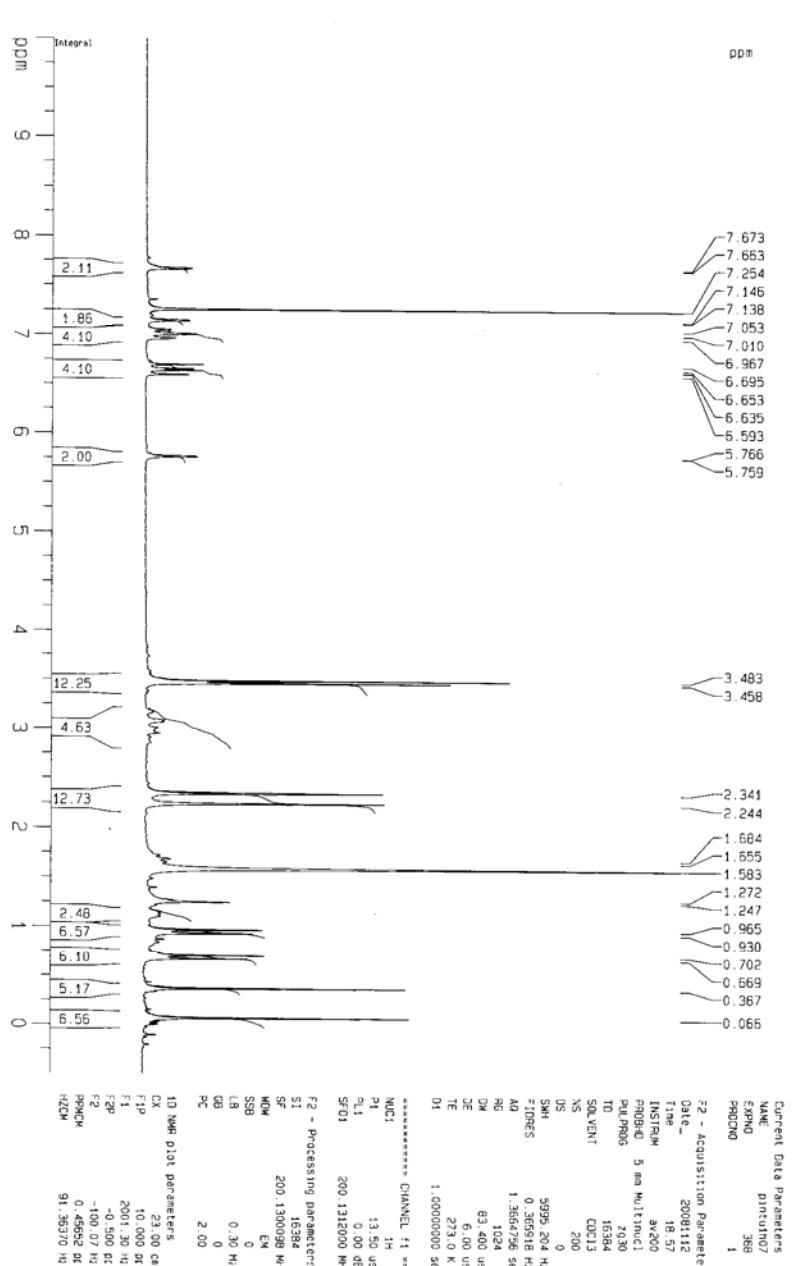
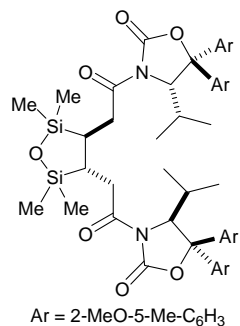
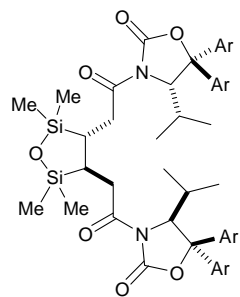


Figure-S35.  $^1\text{H}$  NMR of *trans*-**15c**



Ar = 2-MeO-5-Me-C<sub>6</sub>H<sub>3</sub>

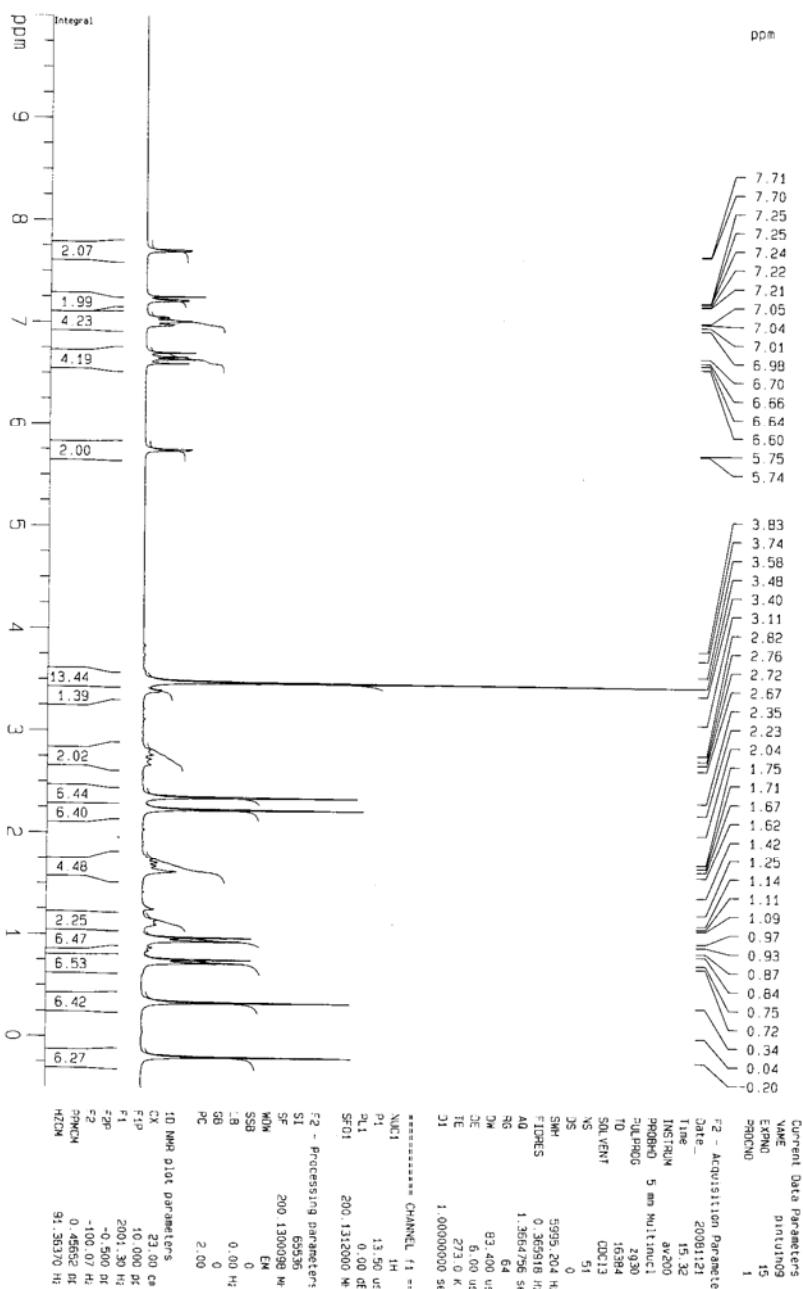


Figure-S36.  $^{13}\text{C}$  NMR of *trans*-15c

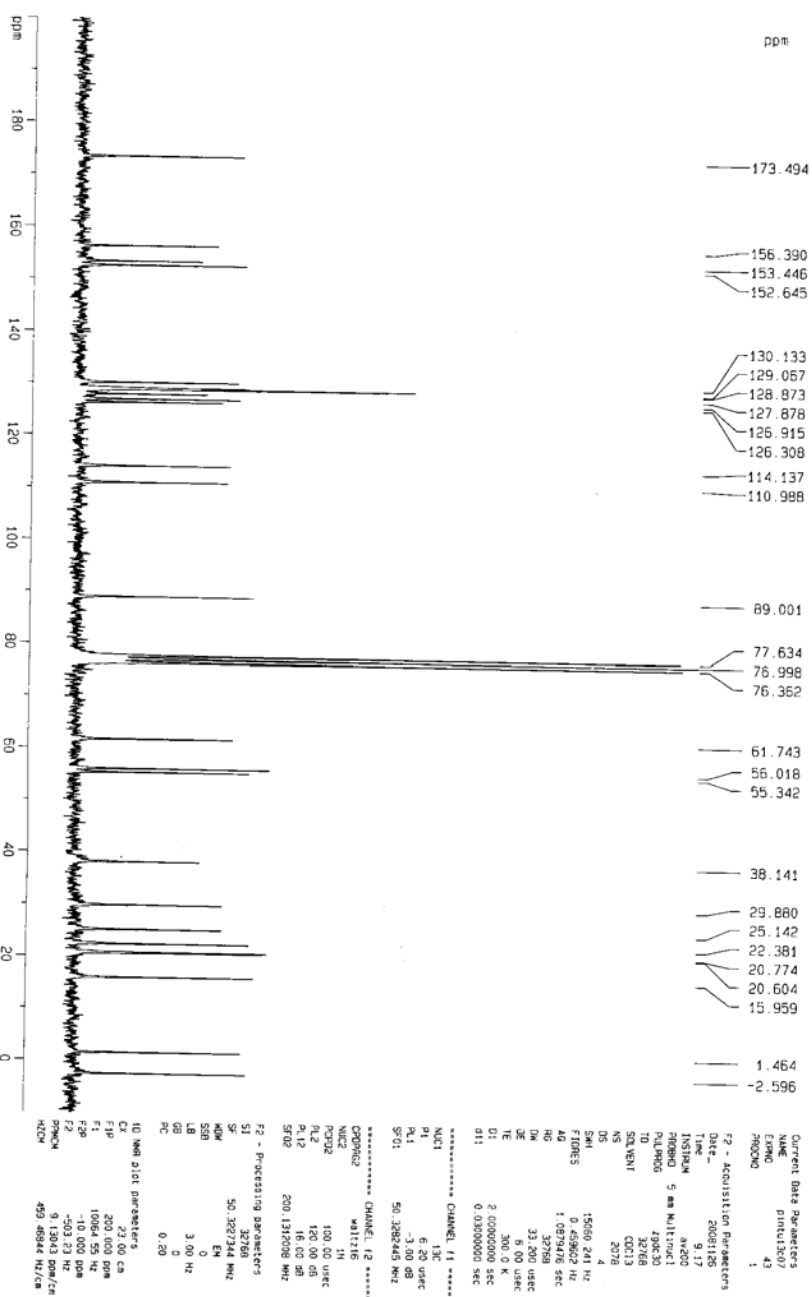
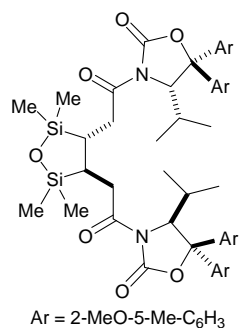


Figure-S37. <sup>1</sup>H NMR of (-)-16

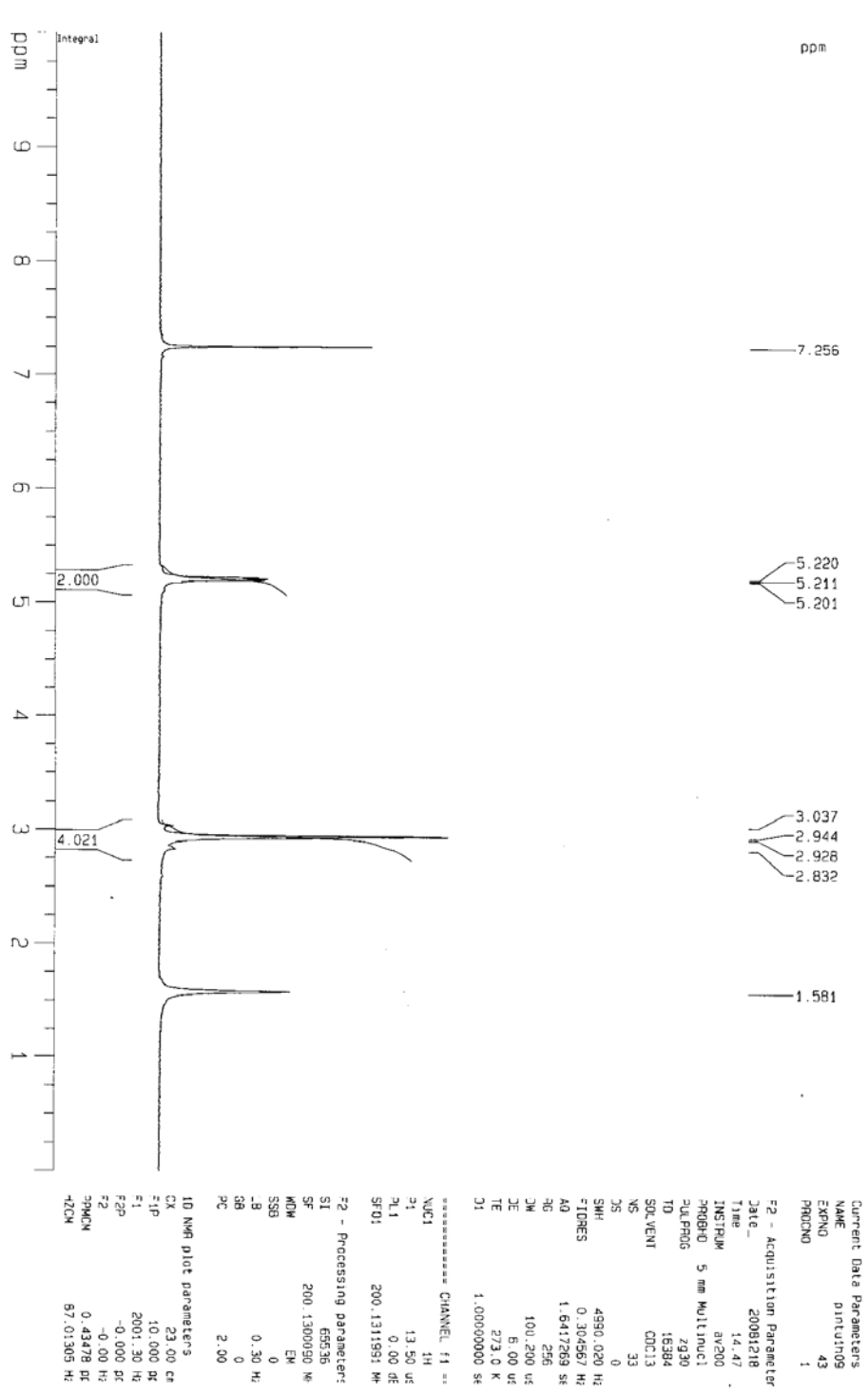
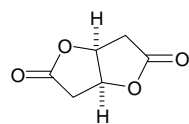
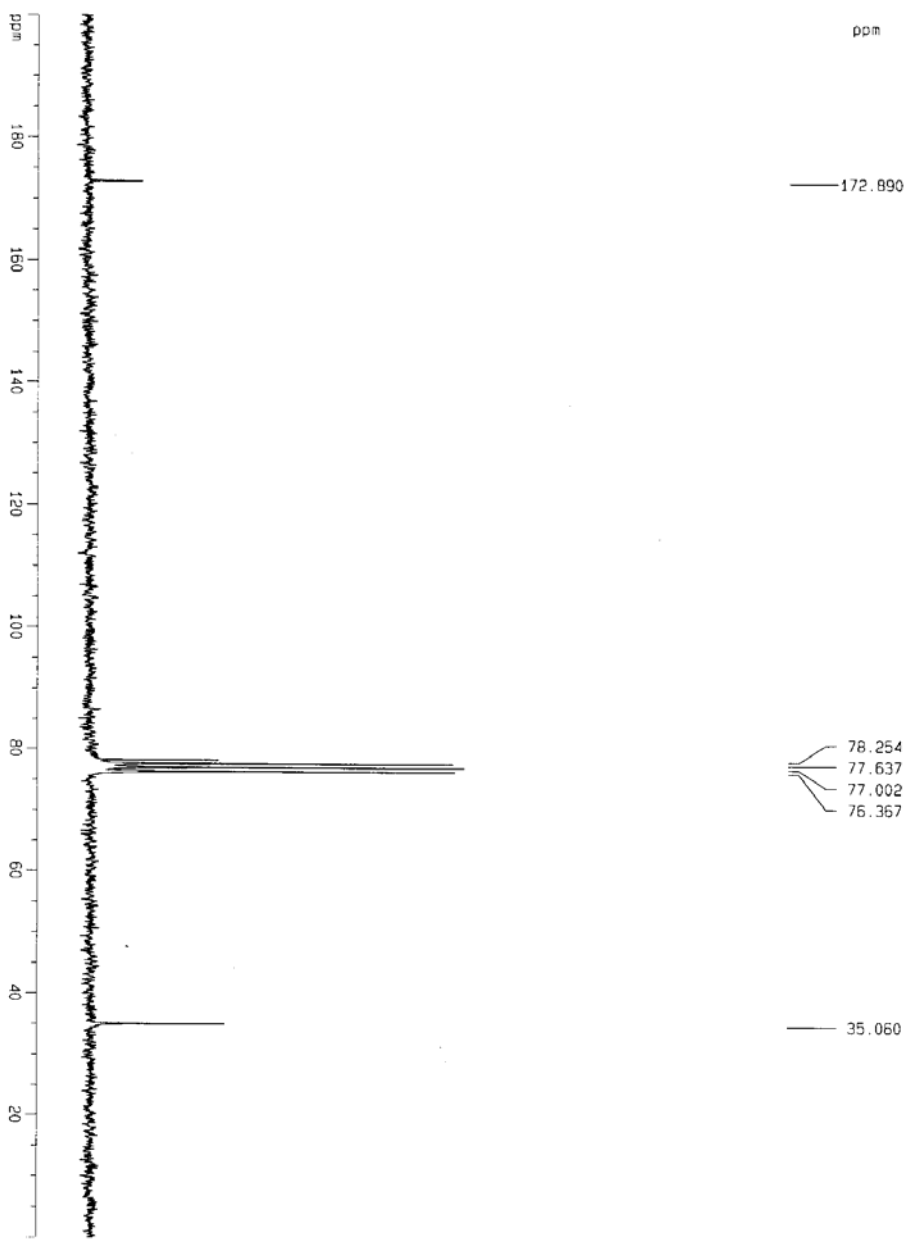
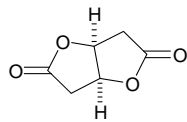


Figure-S38.  $^{13}\text{C}$  NMR of (-)-16



```

Current Data Parameters
NAME          prfu13c07
EXPNO        45
PROCNO       1

F2 - Acquisition Parameters
Date_        20081218
Time         15:00
INSTRUM     av200
PROBHD      5 mm Mlti1nuC1
PULPROG     zgpg30
TD           32768
SOLVENT     CDCl3
NS           954
DS           4
SWH          15060.241 Hz
FIDRES       0.455602 Hz
AQ           1.0879476 sec
RG           32768
DM           33.200 usec
DE           6.00 usec
TE           300.0 K
D1           2.0000000 sec
d11          0.0300000 sec

***** CHANNEL f1 *****
NUC1          13C
P1            6.20 usec
PL1           -3.00 dB
SFO1         50.3082465 MHz

***** CHANNEL f2 *****
CPDPRG2      meltz16
NUC2          1H
PCPD2        100.00 usec
PL2           120.00 dB
PL12         16.00 dB
SFO2         200.1312908 MHz

F2 - Processing parameters
SI            32768
SF            50.3287325 MHz
KCM           EM
SSB           0
LB            3.00 Hz
GB            0
PC            0.20

1D NMR plot parameters
F2           50.3287325 MHz
F1           200.1312908 MHz
F2P          19084.55 ppm
F1P          -0.000 ppm
PCMCX        8.65965 ppm/cm
PCMCY        437.58902 Hz/cm
    
```



Figure-S39. View of crystals of (a) *trans*-**14a**; (b) *trans*-**15a**

(a) *trans*-**14a**



(b) *trans*-**15a**

