

SUPPORTING INFORMATION, Part 1

Synthesis of Phenolic Components of Grains of Paradise

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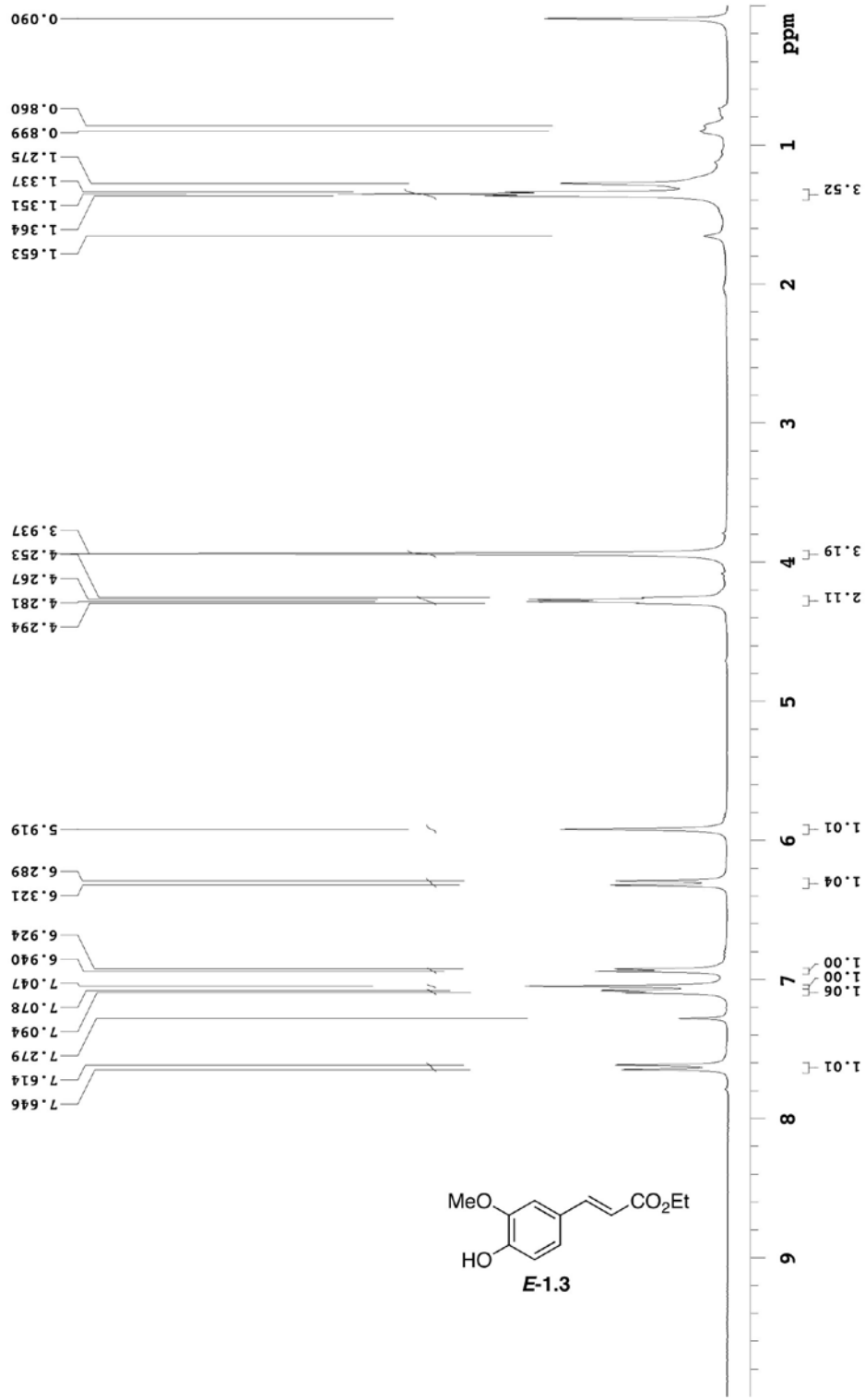
Recorded on: **ibds**, Apr 3 2018
Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
Digital Res.(Hz/pp): **0.09**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **20.76**

Relaxation Delay(s): **0.1**
Completed Scans **16**

498.118 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
temp 26.9 C -> actual temp = 27.0 C, autoxdb probe



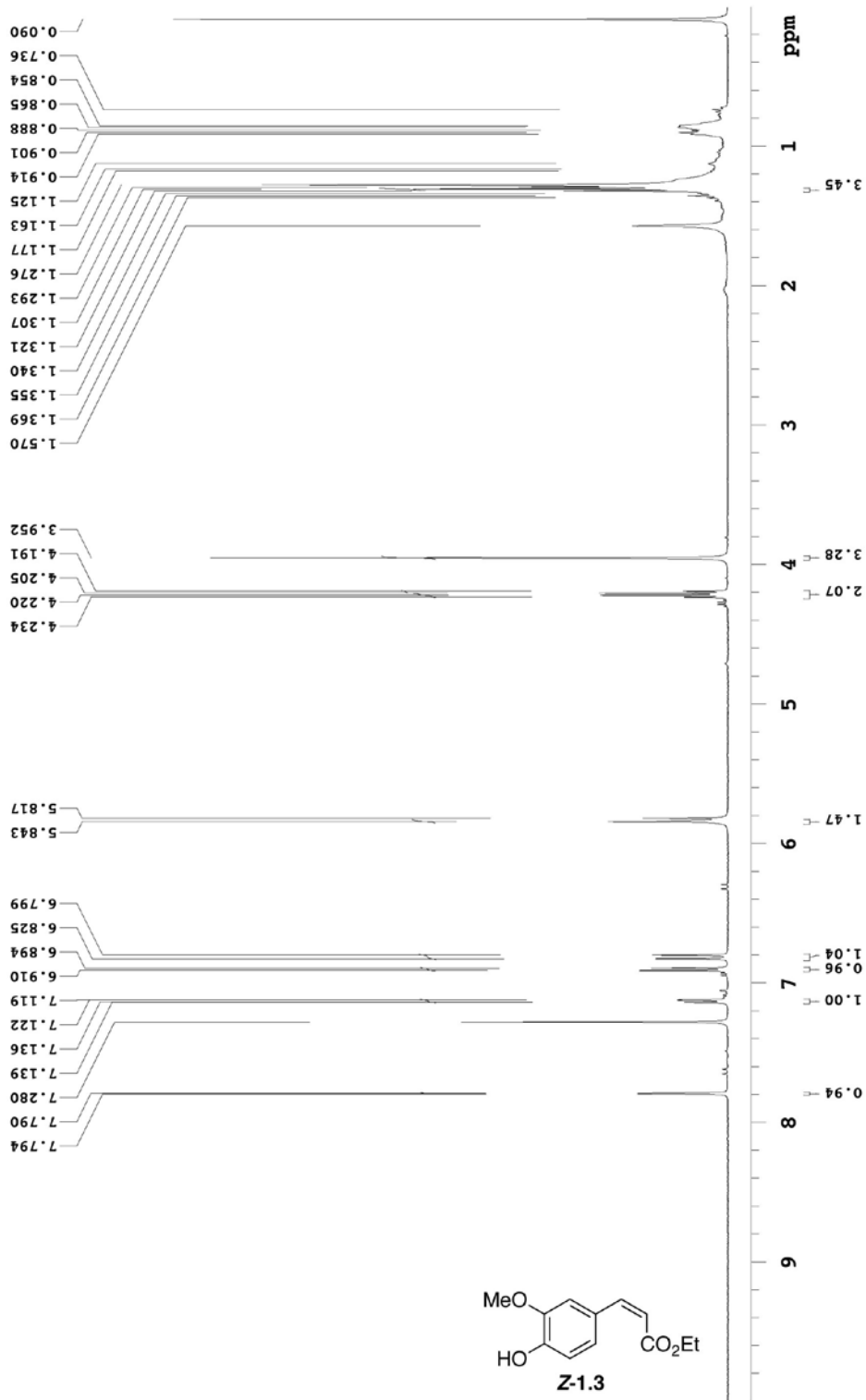
Recorded on: **1bd5, Apr 3 2018**
 Pulse Sequence: **s2pul**

Sweep Width(Hz): **6000.6**
 Digital Res.(Hz/pt): **0.09**

Acquisition Time(s): **5**
 Hz per mm(Hz/mm): **20.74**

Relaxation Delay(s): **0.1**
 Completed Scans: **16**

498.118 MHz ¹H 1D in cdcl₃ (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxdb probe





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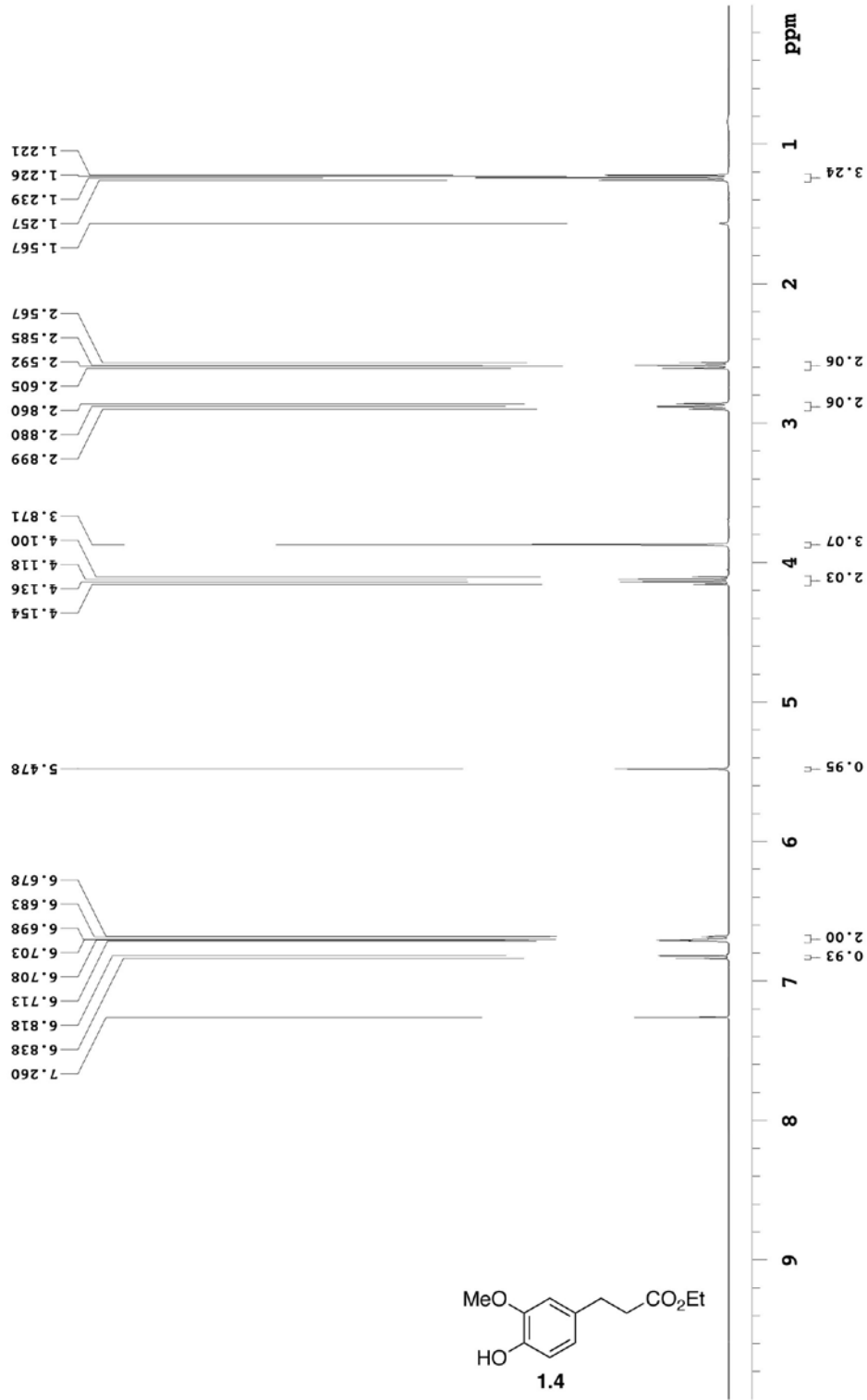
Recorded on: **mr400_Apr17 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/ppm): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **16.66**

Relaxation Delay(s): **0.1**
Completed Scans: **16**

399.978 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
temp 25.9 C -> actual temp = 27.0 C, onenmr probe





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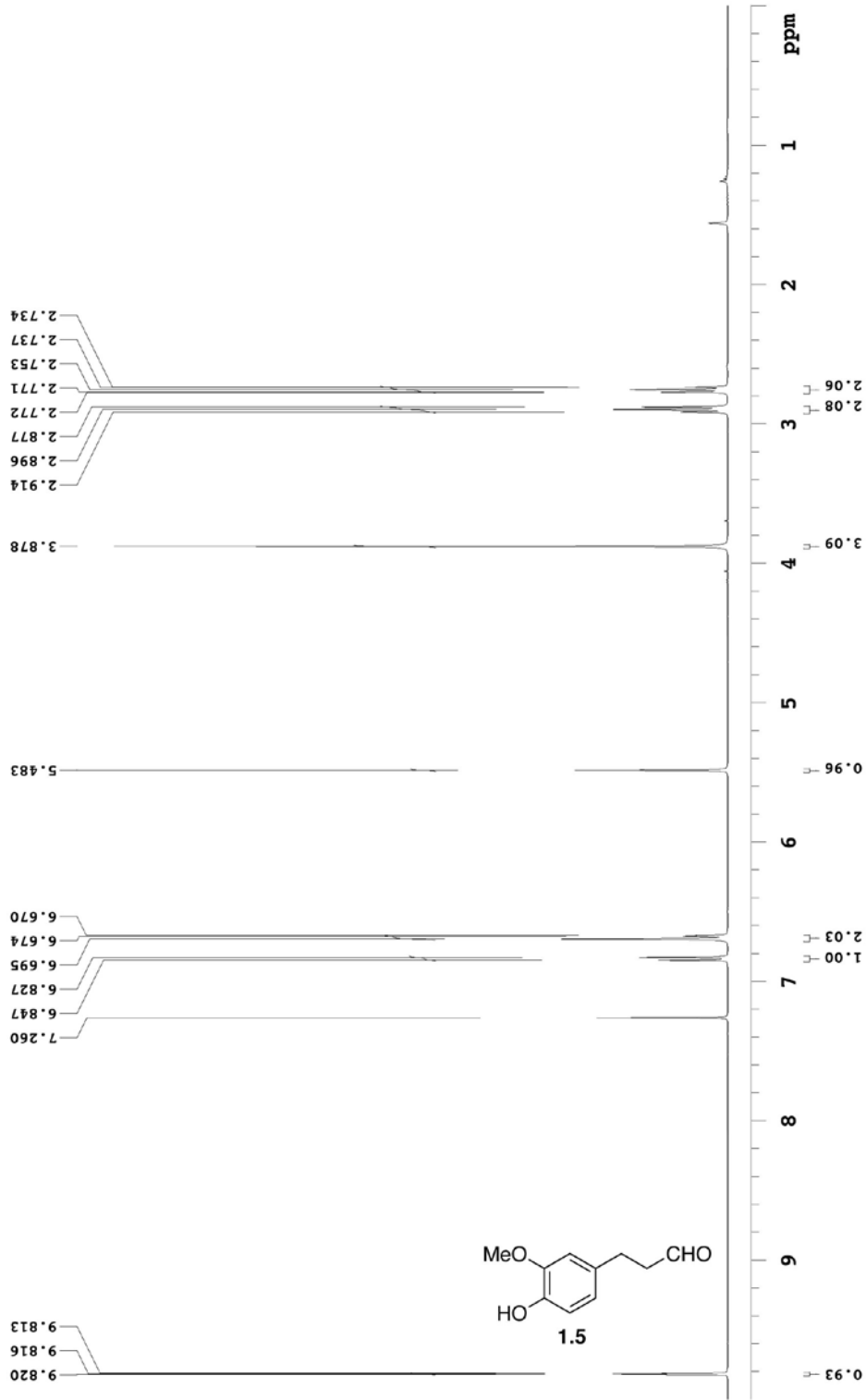
Recorded on: **mr400, Apr 12 2018**
Pulse Sequence: **s2pul**

Sweep Width(Hz): **4807.69**
Digital Res.(Hz/pp): **0.07**

Acquisition Time(s): **5**
Hz per mm(Hz/mm): **16.67**

Relaxation Delay(s): **0.1**
Completed Scans: **16**

399.978 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
temp 25.9 C -> actual temp = 27.0 C, onenmr probe

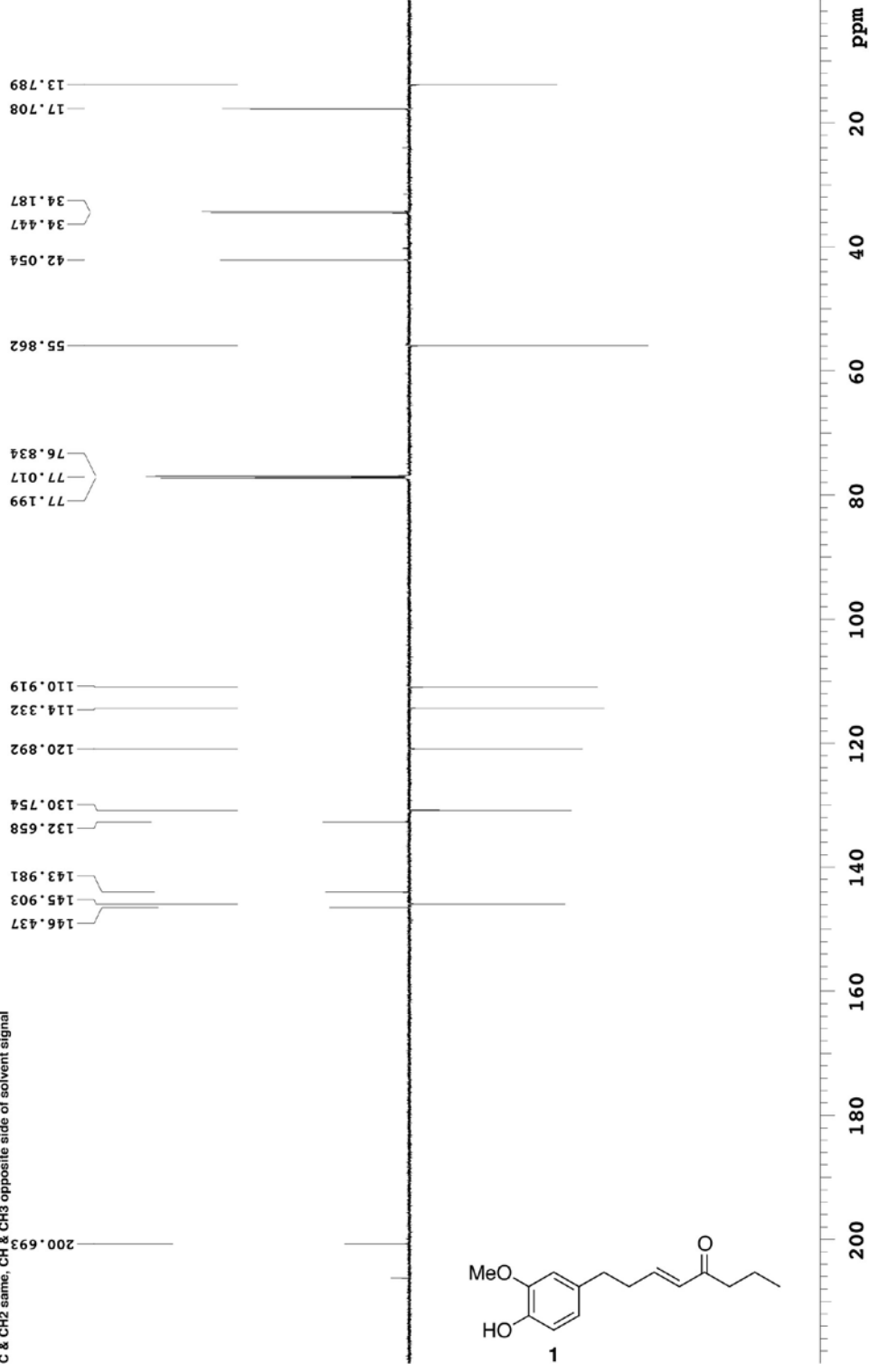




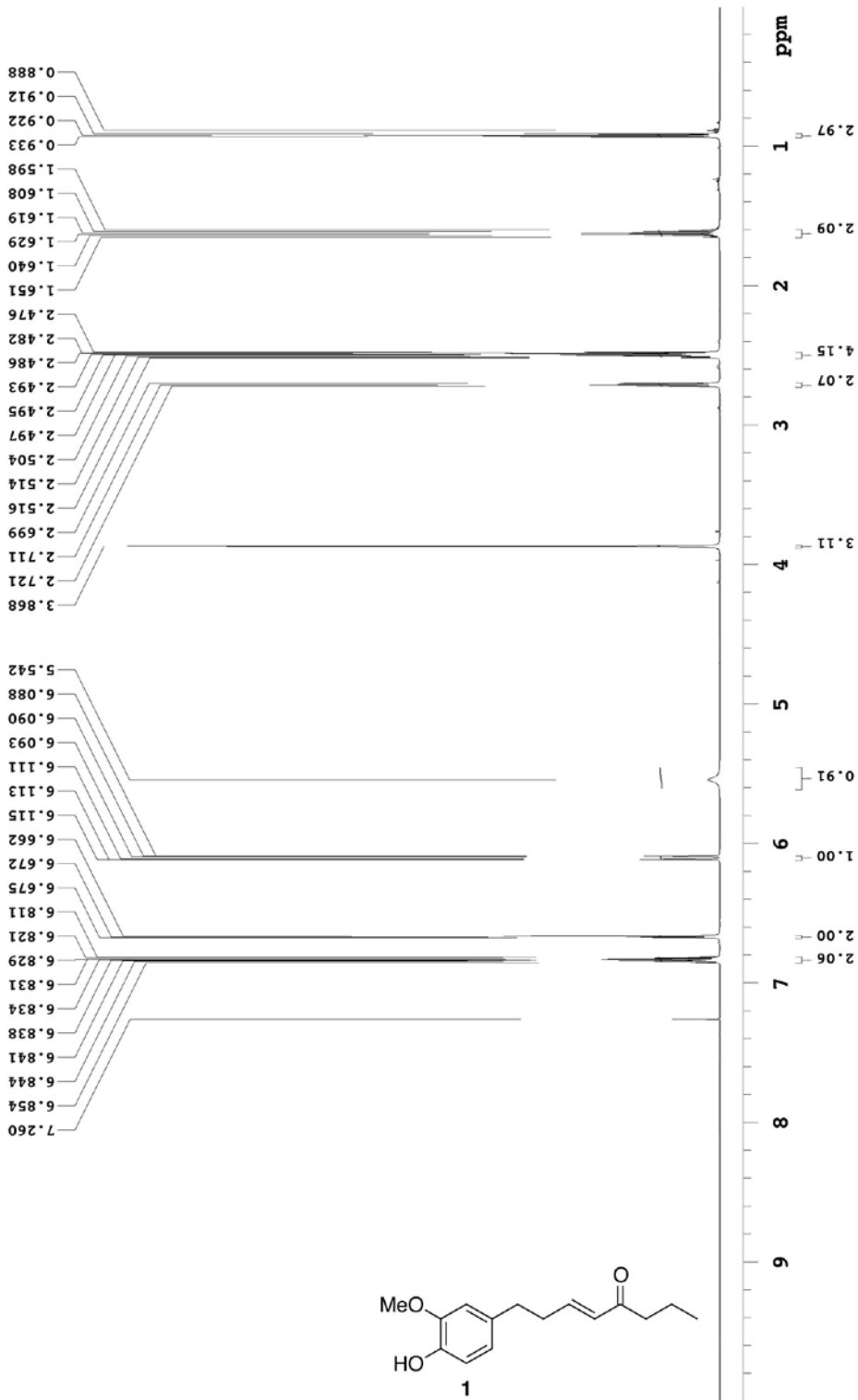
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Recorded on: **v700, Dec 20 2018** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **APT ad** Digital Res. (Hz/pp): **0.37** Hz per mm(Hz/mm): **161.17** Completed Scans: **92**

Hiroyuki, Compound_J
 175.975 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.5 C -> actual temp = 27.0 C, coldid probe
 C & CH2 same, CH & CH3 opposite side of solvent signal



Hiroyuki, Compound_J
 699.762 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
 temp 27.5 C -> actual temp = 27.0 C, coldid probe

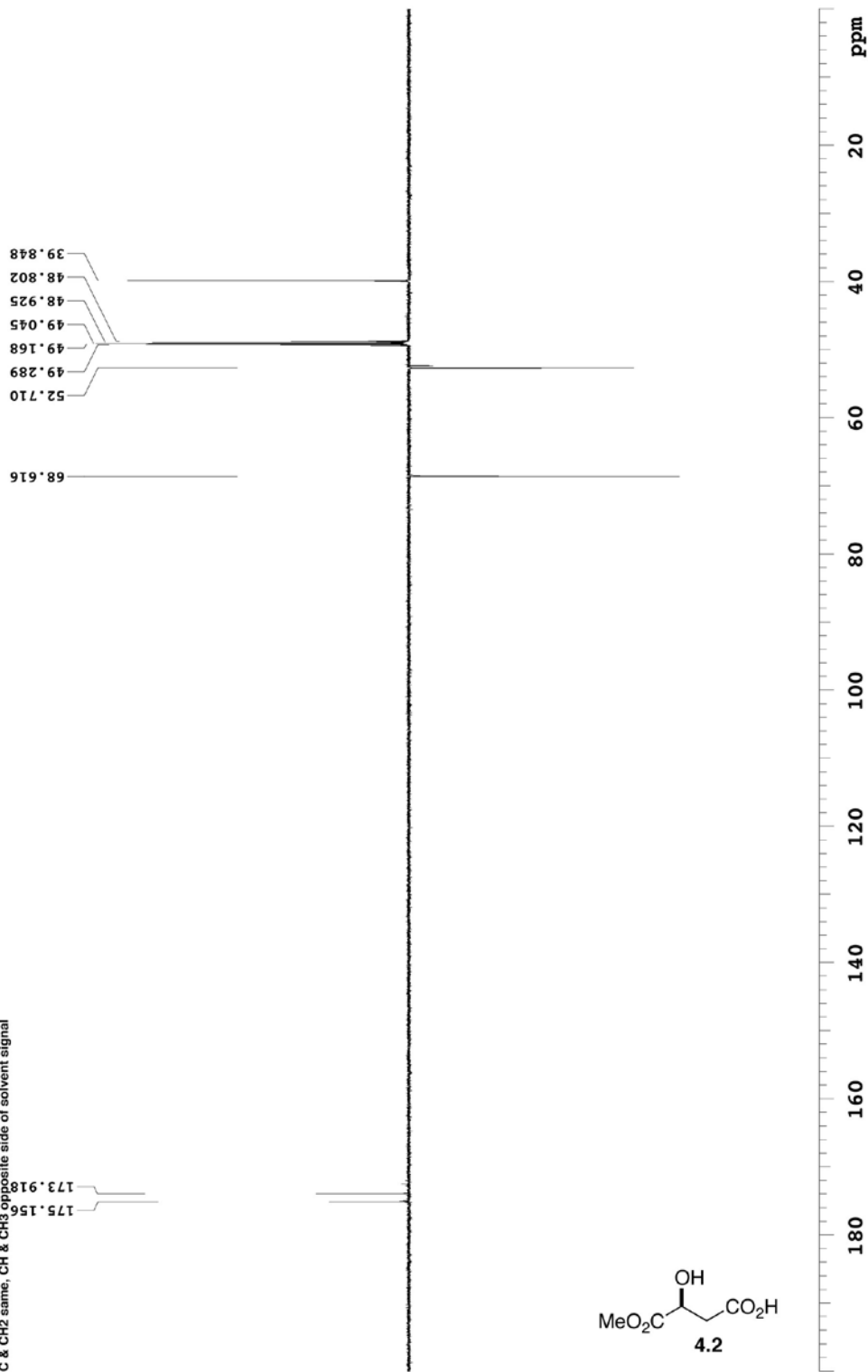


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Recorded on: **v700, Nov 30 2018** Sweep Width(Hz): **48076.9** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **APT_ad** Digital Res.(Hz/pp): **0.37** Hz per mm(Hz/mm): **146.68** Completed Scans: **8**

Hiroiyuki, HH-2-173
 175.975 MHz C13 APT_ad in cd3od (ref. to CD3OD @ 49.0 ppm)
 temp 27.5 C -> actual temp = 27.0 C, coldid probe
 C & CH2 same, CH & CH3 opposite side of solvent signal

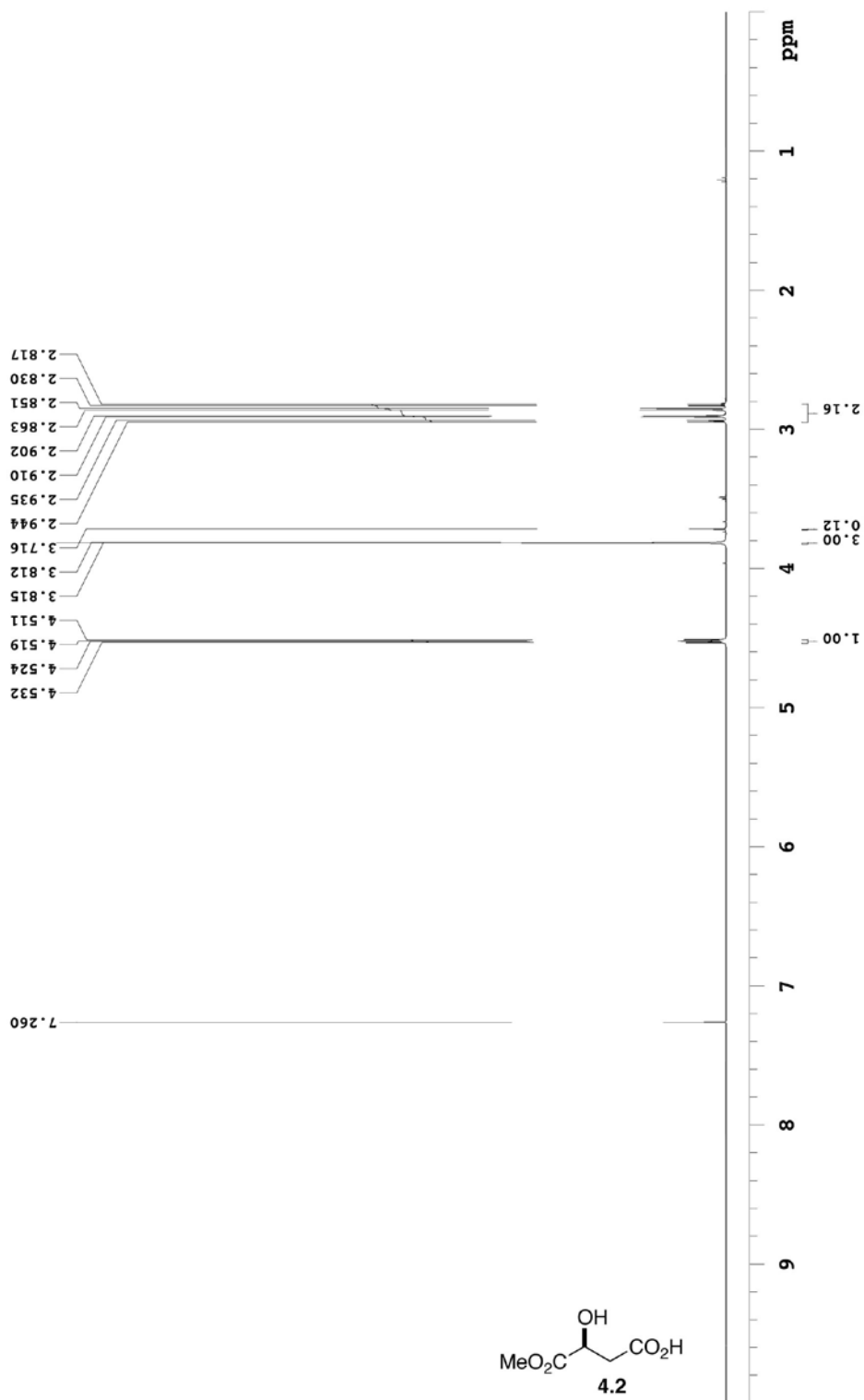


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HH-2-167 (Ibd5 NMR)
 498.118 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxdo probe

Recorded on: **Ibd5, Nov 21 2018** Sweep Width(Hz): **6000.6** Acquisition Time(s): **5** Relaxation Delay(s): **0.1**
 Pulse Sequence: **s2pul** Digital Res.(Hz/ppm): **0.09** Hz per mm(Hz/mm): **20.76** Completed Scans: **16**



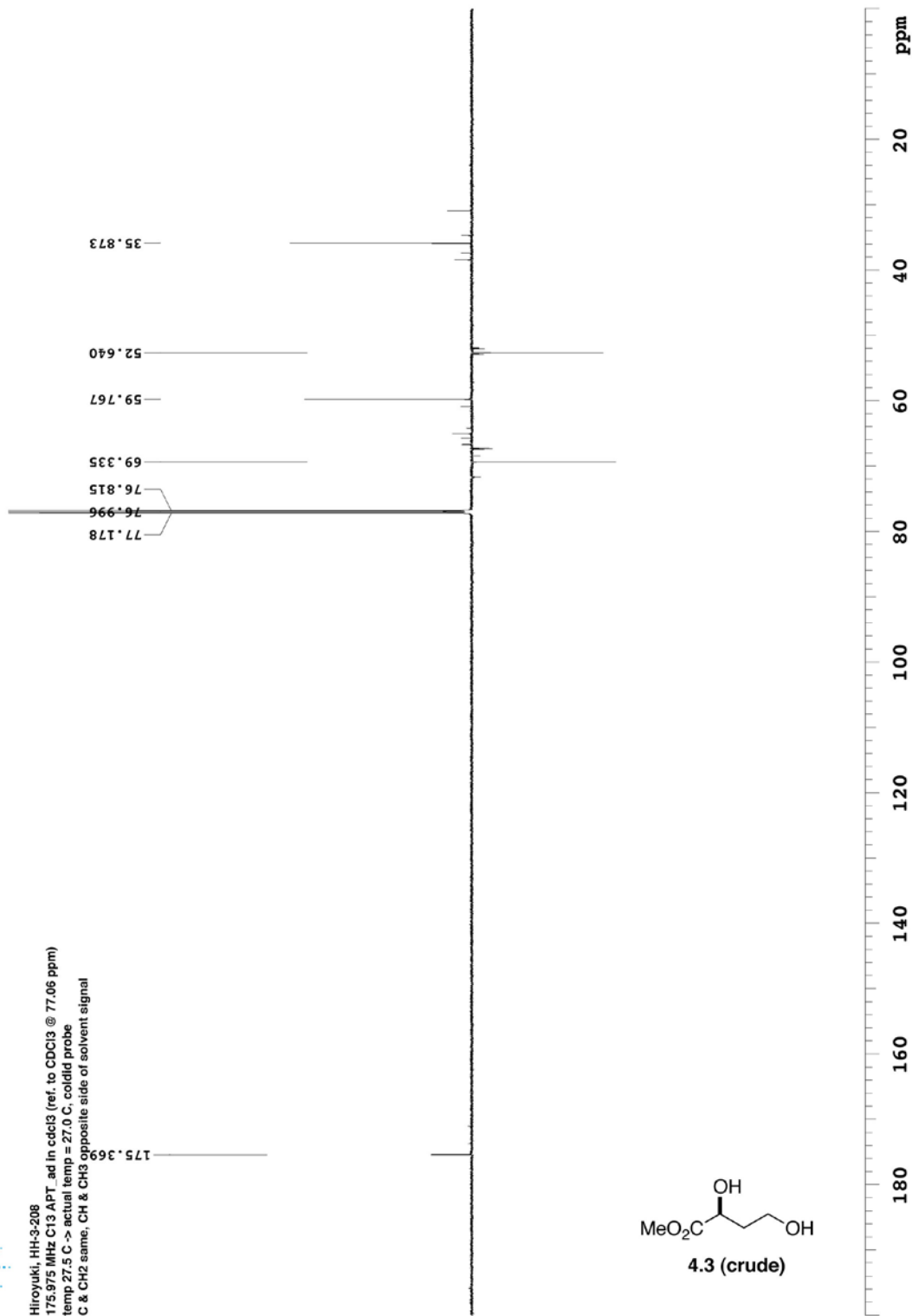
File: /mnt/d600/home14/civenmfr/mrdata/hattori/2018.11.21.15_JHH-2-167_H1_1D



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Recorded on: **v700, Jan 27 2019** Sweep Width(Hz): **48076.9** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **APT ad** Digital Res.(Hz/pp): **0.37** Hz per mm(Hz/mm): **146.68** Completed Scans: **572**

Hiroiyuki, HH-3-208
 175.975 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.5 C -> actual temp = 27.0 C, coldid probe
 C & CH2 same, CH & CH3 opposite side of solvent signal

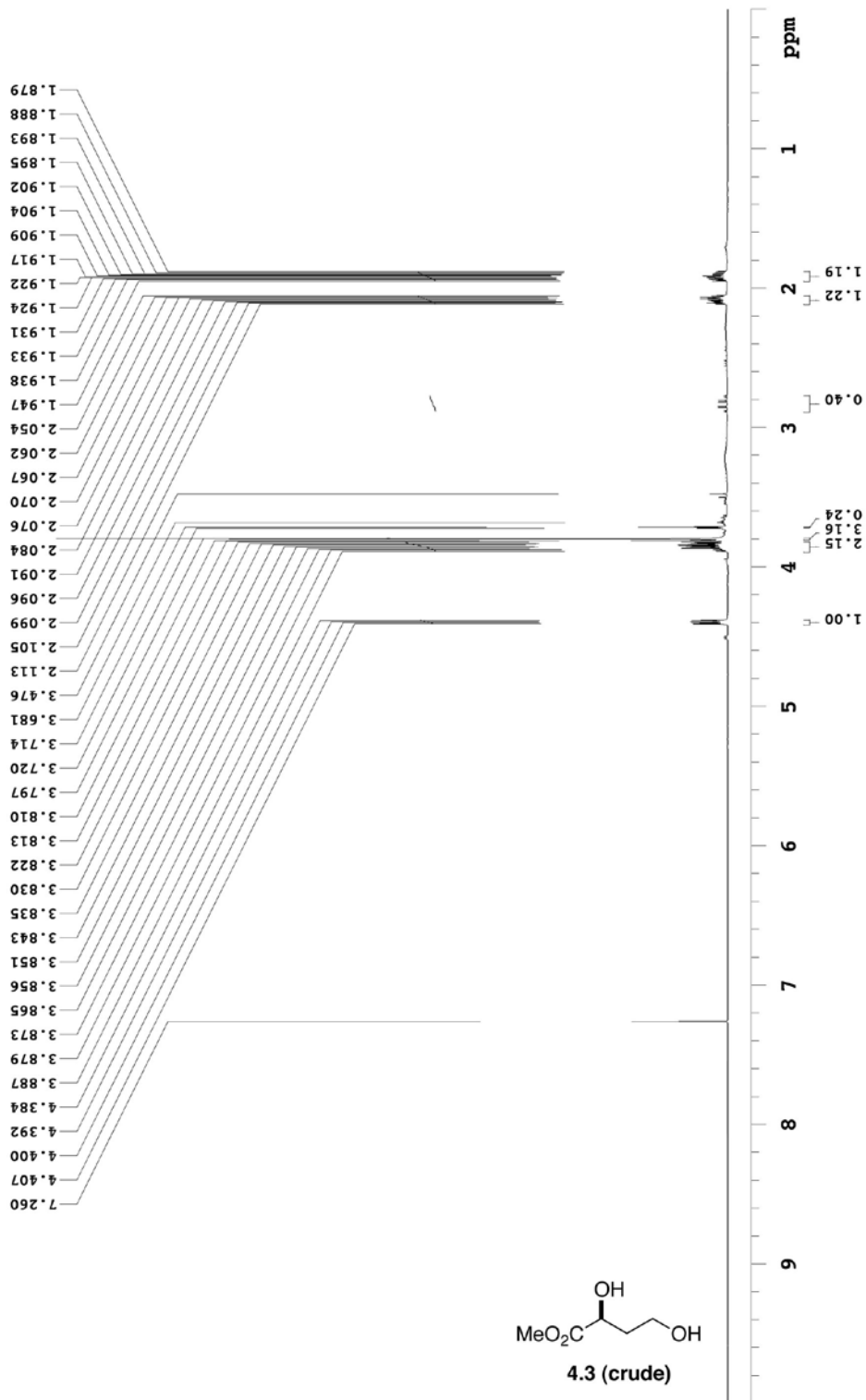


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Recorded on: **ibd5, Oct 3 2018** Sweep Width(Hz): **6000.6** Acquisition Time(s): **5** Relaxation Delay(s): **0.1**
 Pulse Sequence: **s2pul** Digital Res.(Hz/pt): **0.09** Hz per mm(Hz/mm): **20.76** Completed Scans: **16**

HH-2-126 crude (ibd5 NMR)
 498.118 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxtdb probe

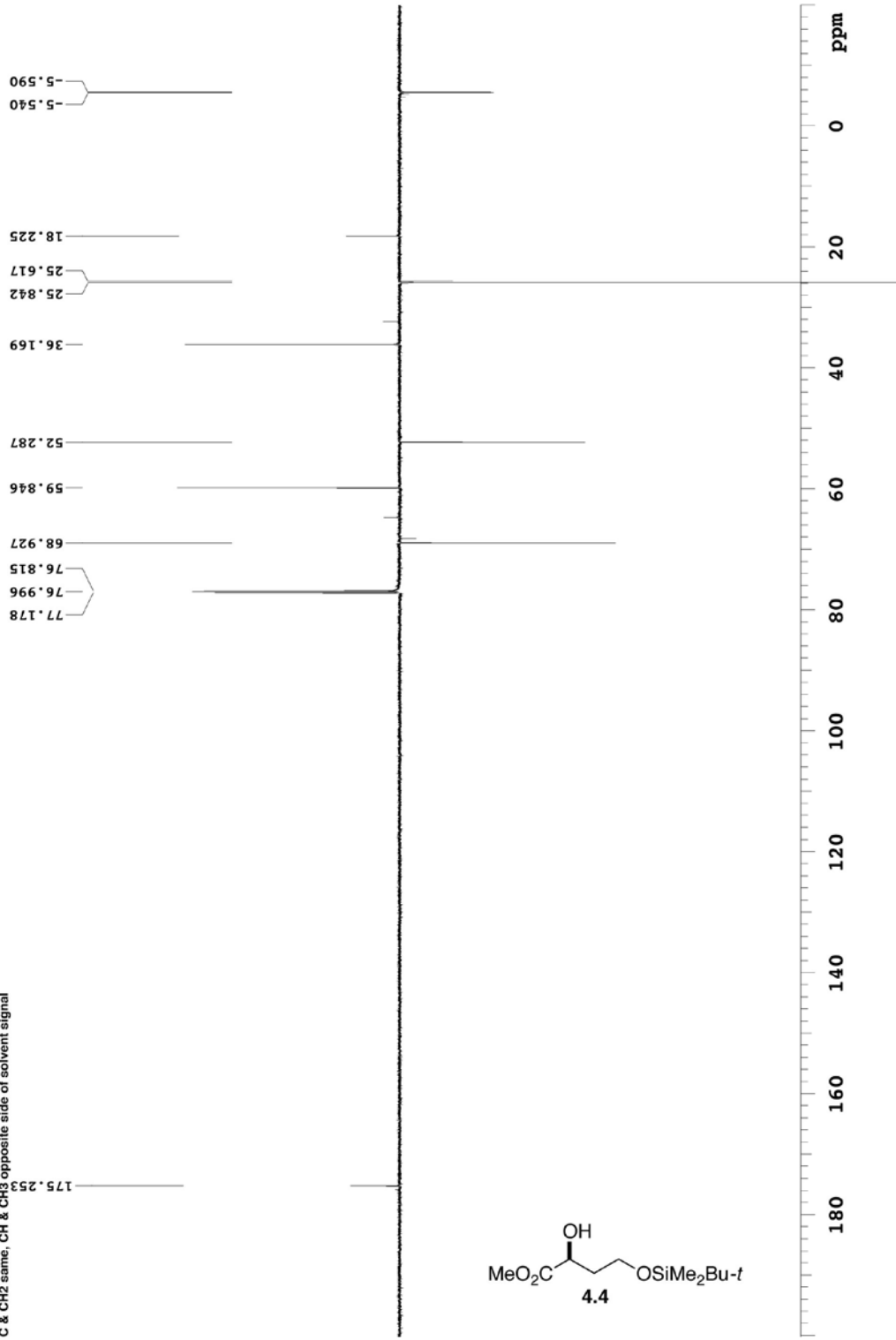


File: /mnt/d600/home14/civenm/nmrdata/hattori/2018.10.03.15_JH-2-126_crude_H1_1D



Recorded on: **v700, Nov 30 2018** Sweep Width(Hz): **48076.9** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **APT ad** Digital Res.(Hz/pp): **0.37** Hz per mm(Hz/mm): **161.43** Completed Scans: **72**

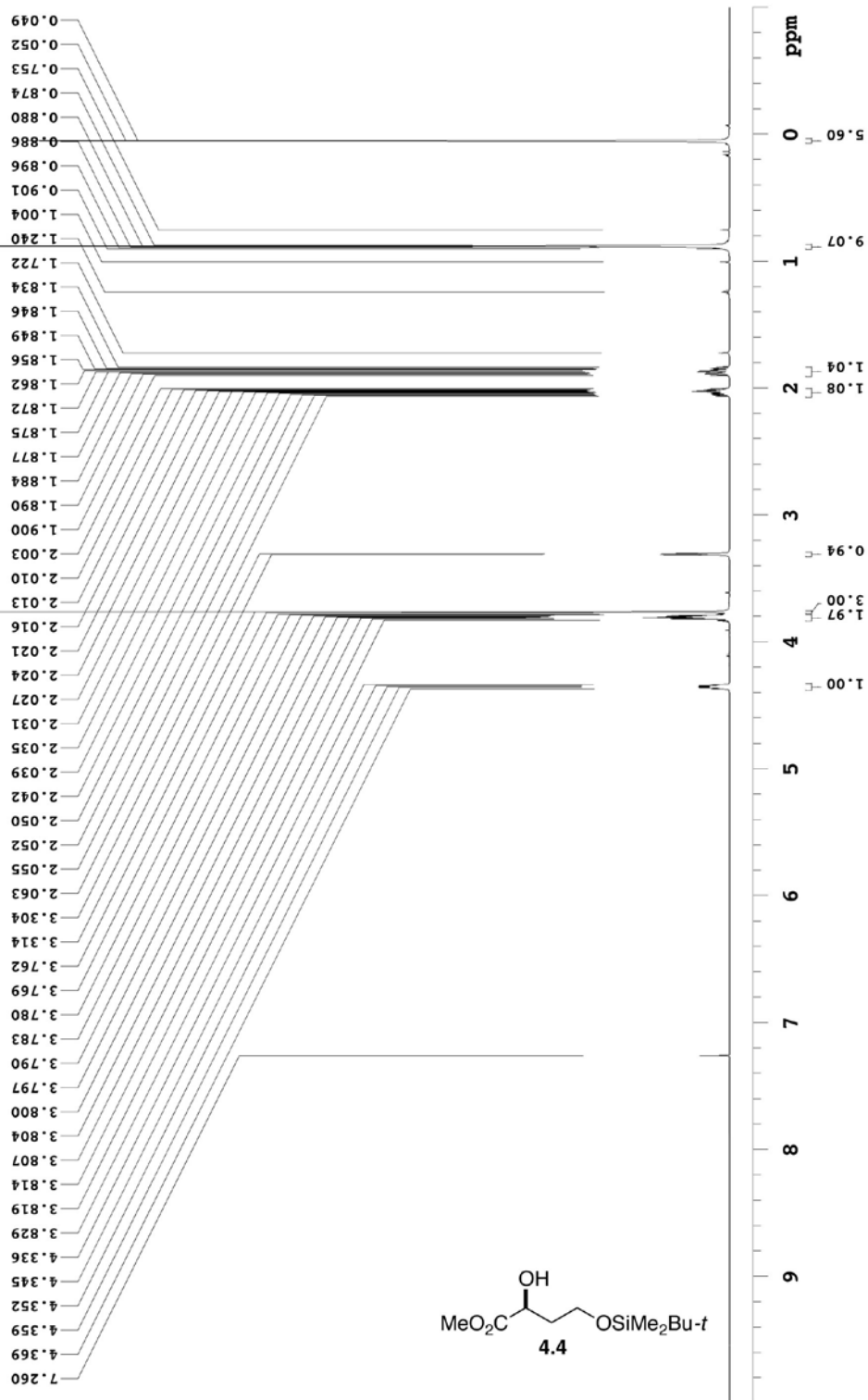
Hiroyuki, HH-2-175
 175.975 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.5 C -> actual temp = 27.0 C, coldid probe
 C & CH2 same, CH & CH3 opposite side of solvent signal



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Recorded on: **ibds, Oct 4 2018**
Pulse Sequence: **s2pul**Sweep Width(Hz): **6000.6**
Digital Res.(Hz/mm): **22.83**Acquisition Time(s): **5**
Relaxation Delay(s): **0.1**
Completed Scans: **16**

HH-2-127B (16d5 NMR)
498.118 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm)
temp 26.9 C -> actual temp = 27.0 C, autoxtdb probe



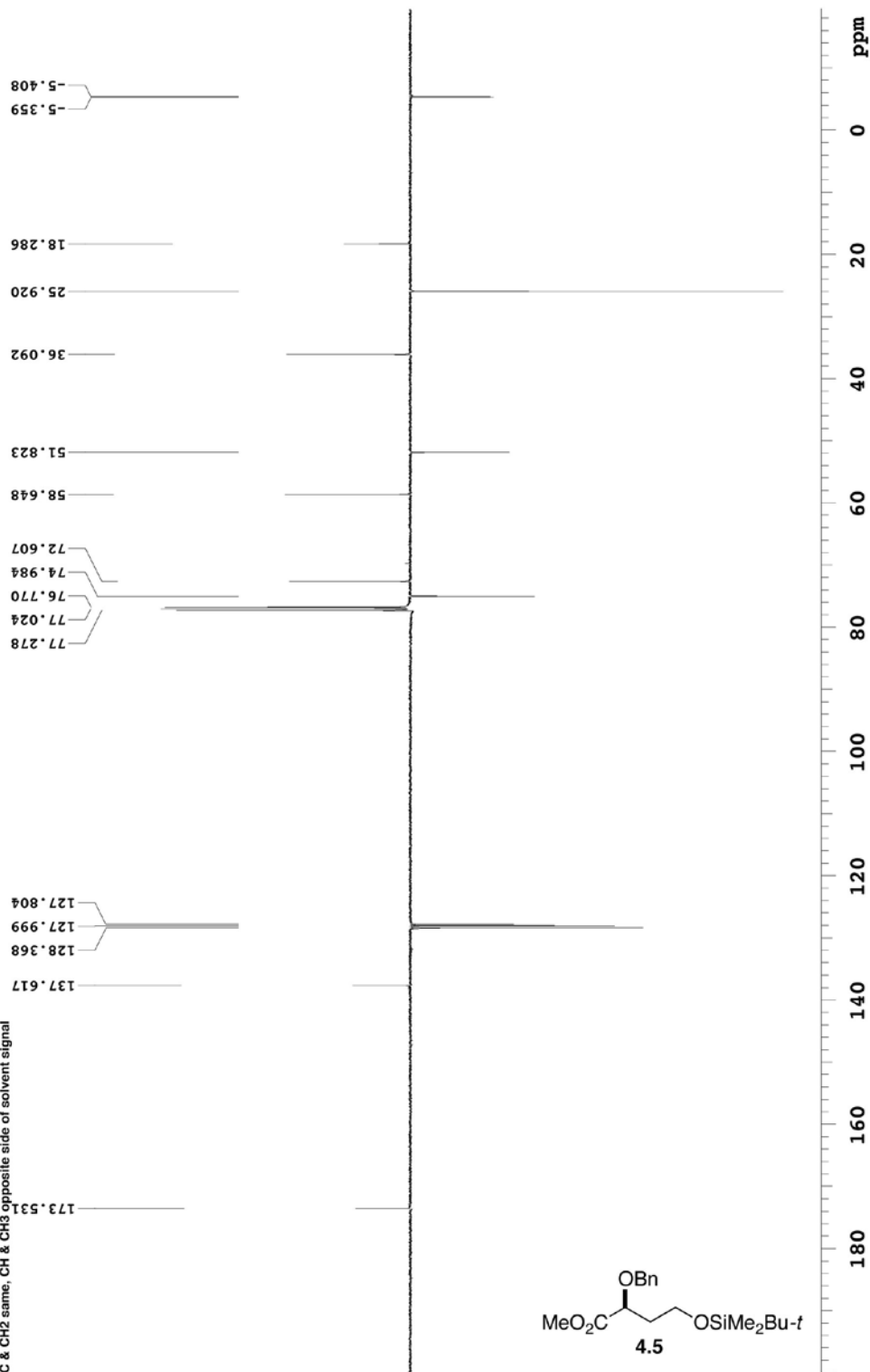
File: /mnt/d600/home14/civenm/nmr/data/hattori/2018.10.04.15_JH-2-127B_H1_1D

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Recorded on: **u500, Oct 9 2018** Sweep Width(Hz): **33783.8** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **-APT.ad** Digital Res.(Hz/ppm): **0.26** Hz per mm(Hz/mm): **115** Completed Scans: **84**

Hiroiyuki, HH-2-129C
 125.688 MHz C13 APT.ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.7 C -> actual temp = 27.0 C, coldlual probe
 C & CH2 same, CH & CH3 opposite side of solvent signal



File: /mnt/d600/home14/ctivenmr/nmrdata/129C_102018_10.09.u5_HH-2-129C_loc1_17.48_C13_APT.ad

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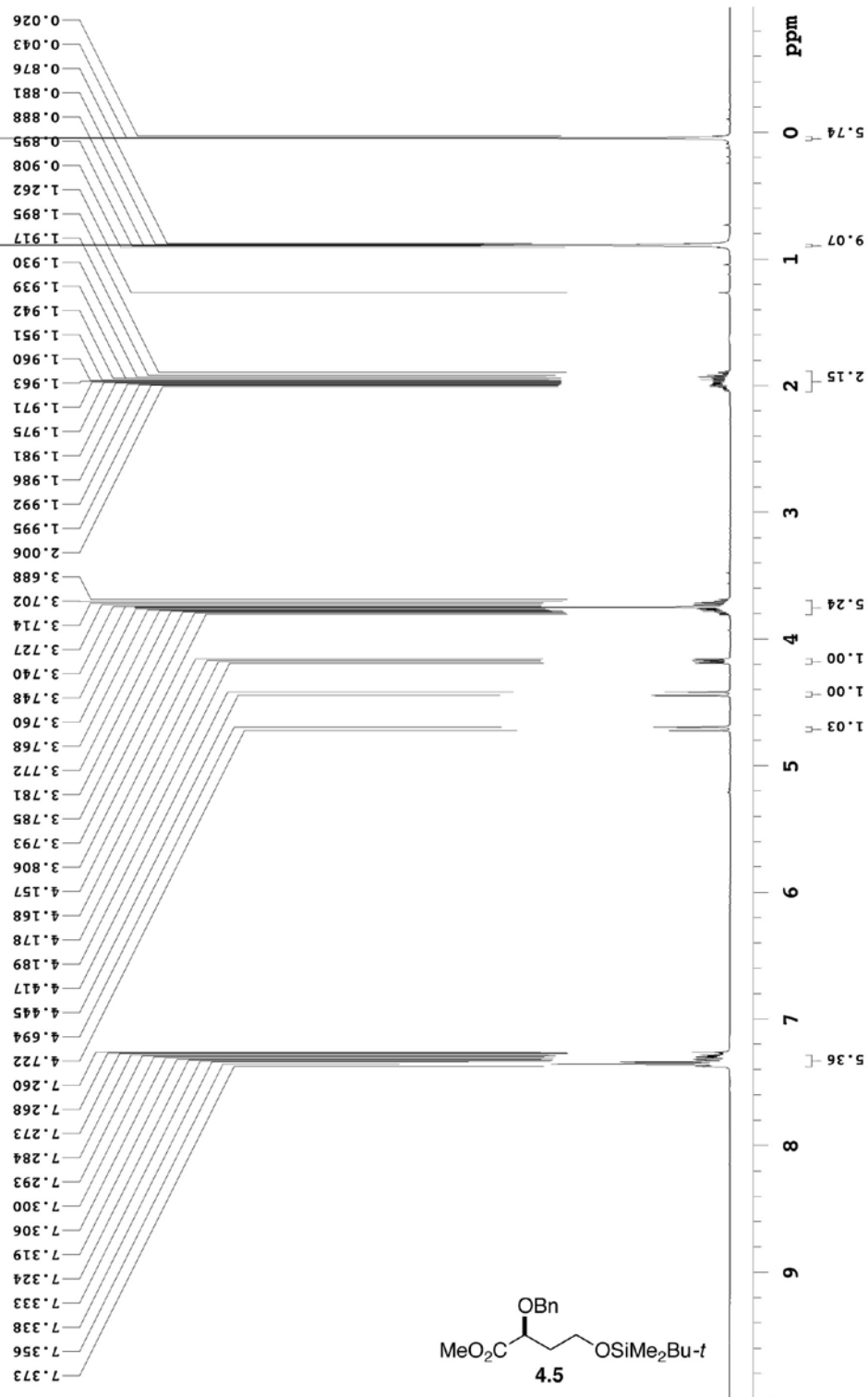
Recorded on: **m400, Nov 20 2018**
 Pulse Sequence: **PRESAT**

Sweep Width(Hz): **4803.07**
 Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **4.997**
 Hz per mm(Hz/mm): **18.35**

Relaxation Delay(s): **0.1**
 Completed Scans: **32**

Hiroyuki, HH-2-166_check
 400.369 MHz ¹H 1D in cdcl₃ (ref. to CDC13 @ 7.26 ppm)
 temp 27.0 C -> actual temp = 27.0 C, m400gz probe



File: /mnt/d600/home14/ci/venmr/nmrdata/1D_DATA_FROM_NMRSERVICE/Hiroyuki/2018.11/2018.11.20.m4_JHH-2-166_check_loc9_13.56_H1_1D

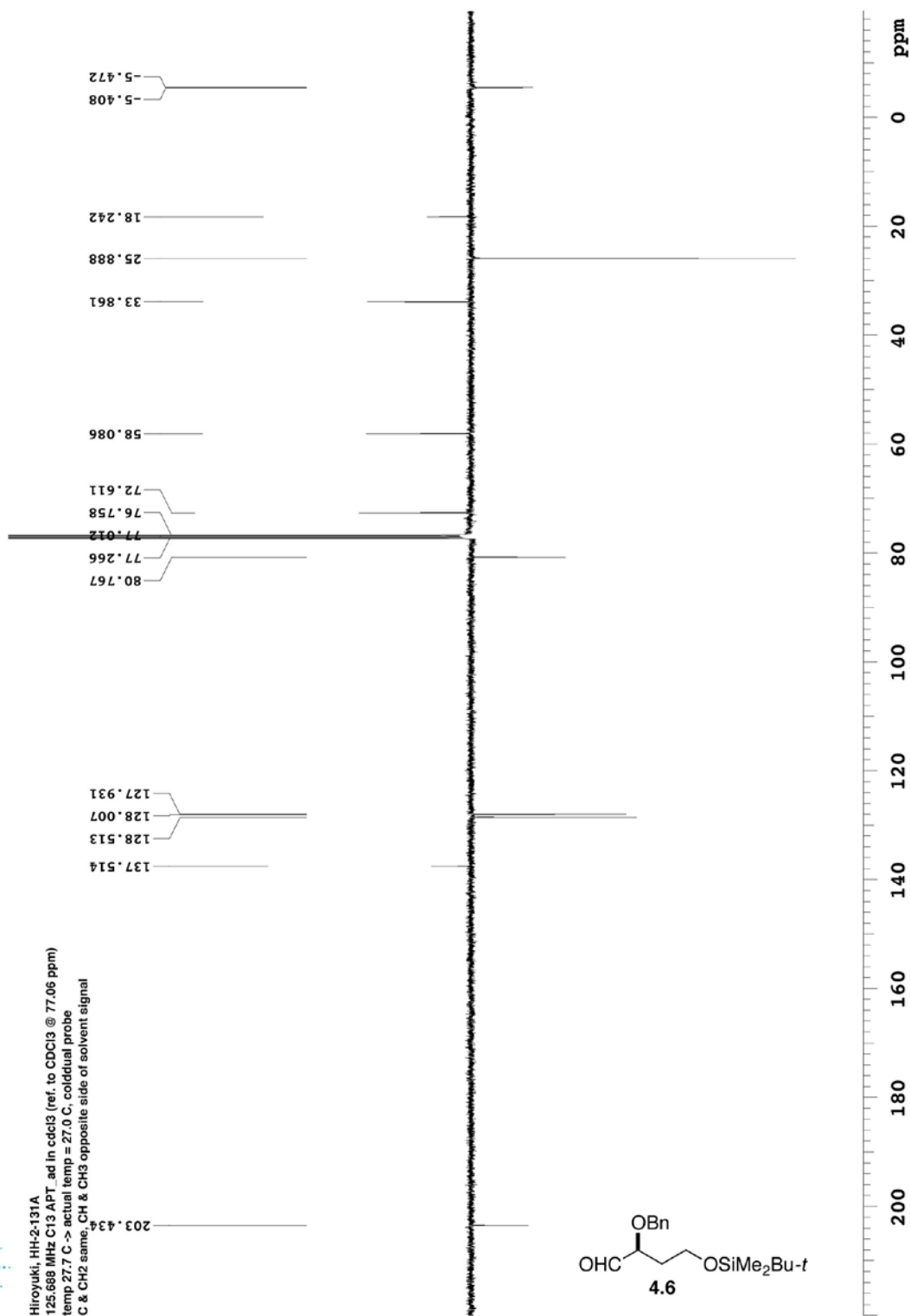
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Recorded on: u500, Oct 9 2018
Pulse Sequence: APT_adSweep Width(Hz): 33783.8
Digital Res.(Hz/pp): 0.26Acquisition Time(s): 1
Hz per mm(Hz/mm): 125.45Relaxation Delay(s): 1
Completed Scans: 128

Hiroyuki, HH-2-131A
125.688 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
temp 27.7 C -> actual temp = 27.0 C, coldlual probe
C & CH2 same, CH & CH3 opposite side of solvent signal



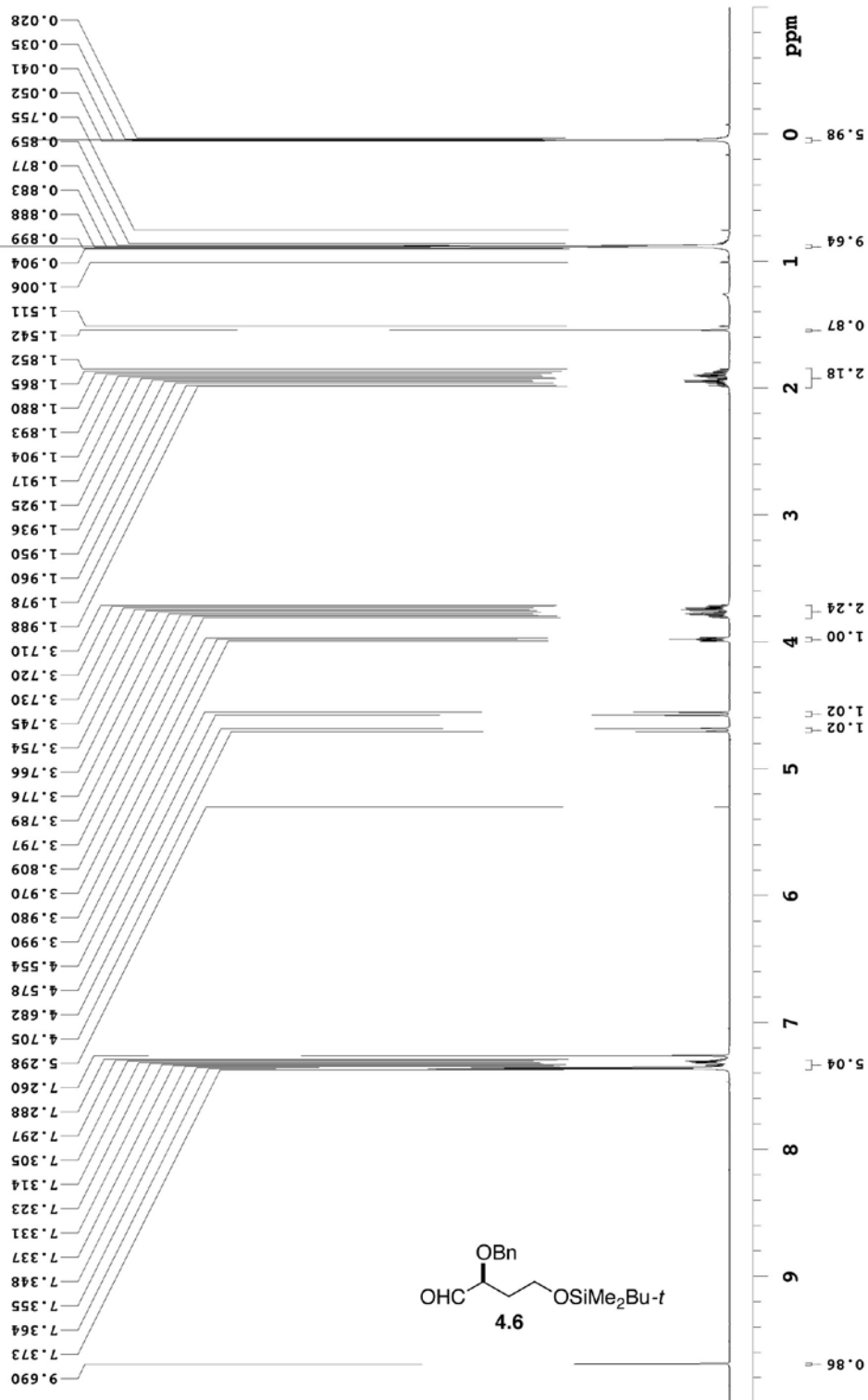
File: /mnt/d600/home14/ci/venmfr/mrdata/131A_14.36_C13_APT_ad

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 Recorded on: **ibd5, Oct 9 2018**
 Pulse Sequence: **s2pul**

 Acquisition Time(s): **5**
 Hz per mm(Hz/mm): **22.83**

 Relaxation Delay(s): **0.1**
 Completed Scans: **16**

 HH-2-131A (ibd5 NMR)
 498.118 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxtdb probe


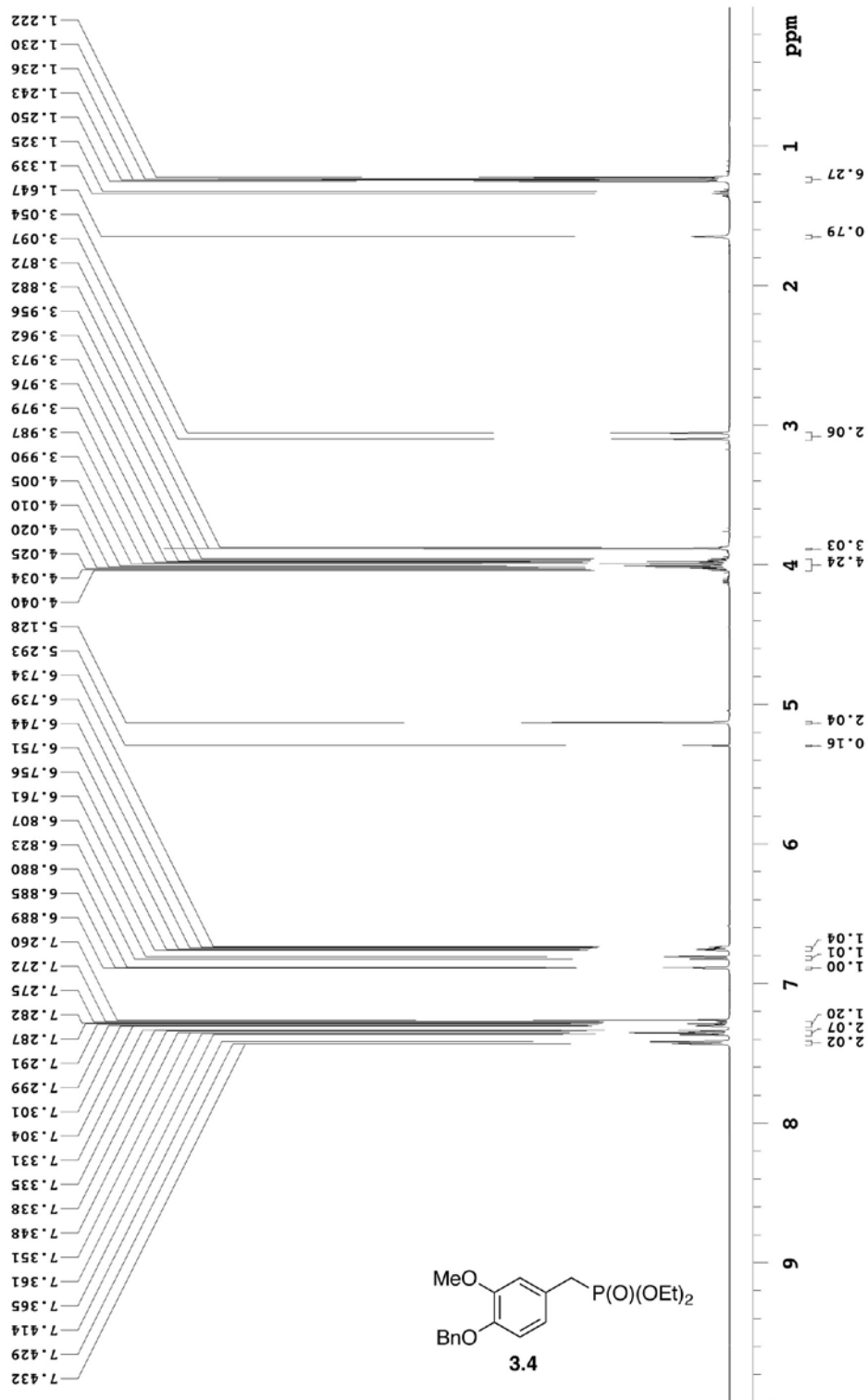
File: /mnt/d600/home14/ci/enmr/nmrdata/hattori/2018_10_09_05_JH-2-131A_H1_1D

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Recorded on: **ibd5, Oct 12 2018** Sweep Width(Hz): **6000.6** Relaxation Delay(s): **0.1**
 Pulse Sequence: **s2pul** Digital Res.(Hz/ppm): **20.76** Hz per mm(Hz/mm): **20.76** Completed Scans **16**

HH-2-117_1 check (ibd5 NMR)
 498.118 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxtdb probe



File: /mnt/d600/home14/civer/mr/mrdata/hattori/2018.10.12.15_HH-2-117_1_check_H1_1D

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 Recorded on: **V700, Nov 5 2018**
 Pulse Sequence: **APT ad**

 Sweep Width(Hz): **48076.9**
 Digital Res.(Hz/pt): **0.37**

 Acquisition Time(s): **1**
 Hz per mm(Hz/mm): **146.68**

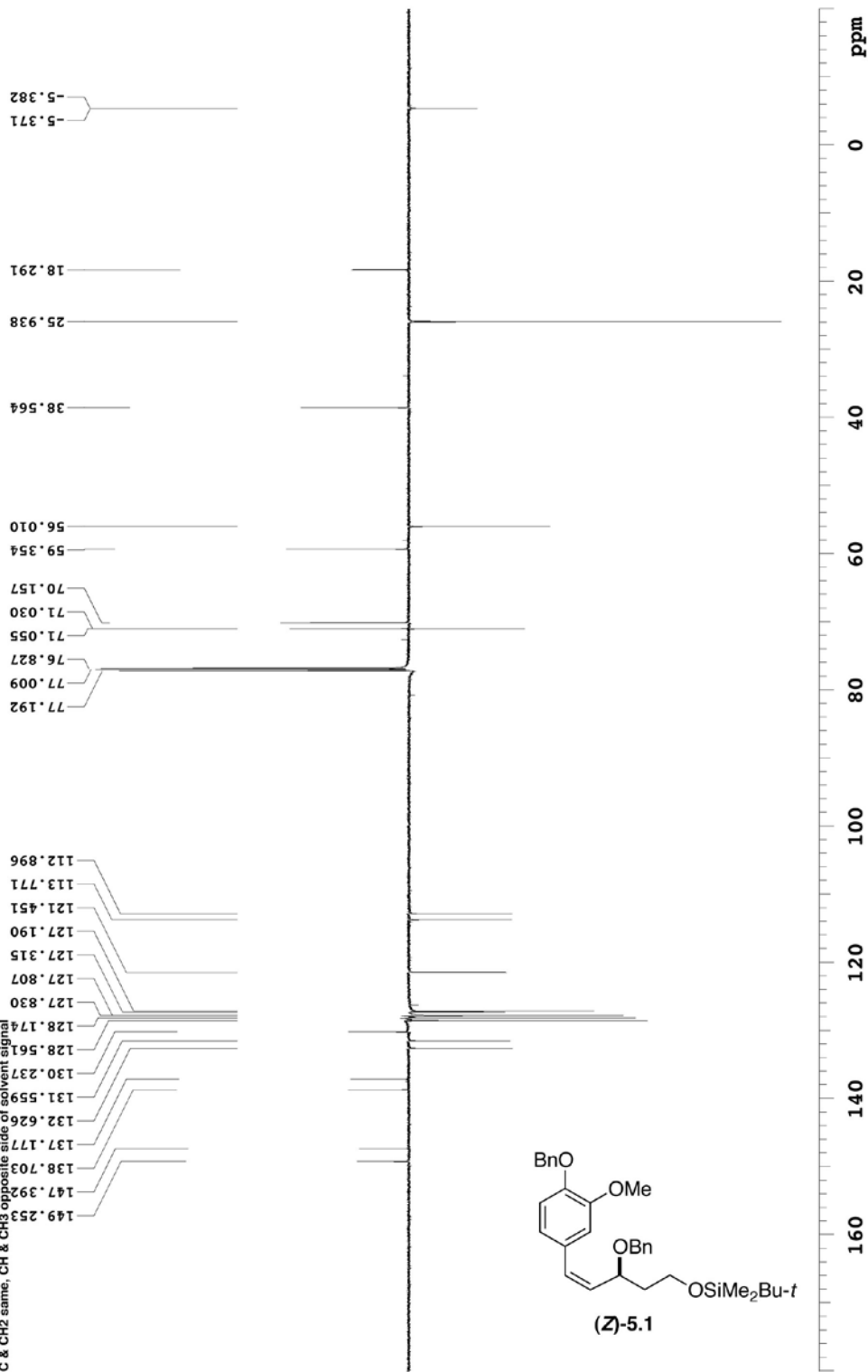
 Relaxation Delay(s): **1**
 Completed Scans: **208**

Hiroyuki, HH-2-149C

175.975 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)

temp 27.5 C -> actual temp = 27.0 C, coldid probe

C & CH2 same, CH & CH3 opposite side of solvent signal



File: /mnt/d600/home14/ci/venmr/nmrdata/149C_2018_11_05/v7_HH-2-149C_6c40_12_12_C13_APT_ad

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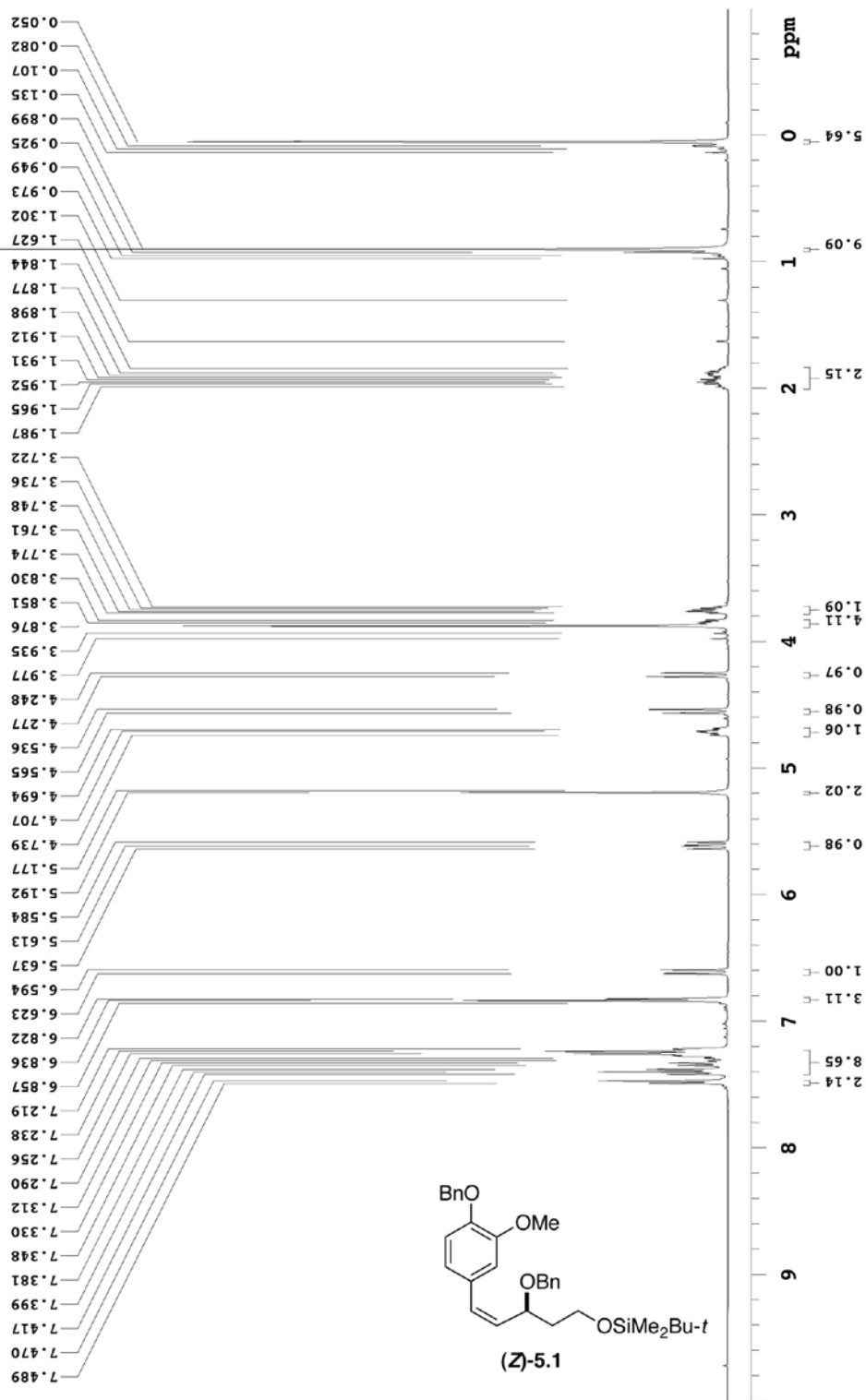
Recorded on: **m400, Nov 3 2018**
 Pulse Sequence: **PRESAT**

Sweep Width(Hz): **4803.07**
 Digital Res.(Hz/pt): **0.07**

Acquisition Time(s): **4.997**
 Hz per mm(Hz/mm): **18.35**

Relaxation Delay(s): **0.1**
 Completed Scans: **32**

Hiroyuki, HH-2-149C
 400.369 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
 temp 27.0 C -> actual temp = 27.0 C, m400qz probe



File: /mnt/d600/home14/ctivenmr/mrdata/149C_12.50_H1_1D

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 Recorded on: **V700, Nov 5 2018**
 Pulse Sequence: **APT_ad**

 Acquisition Time(s): **1**
 Hz per mm(Hz/mm): **146.81**

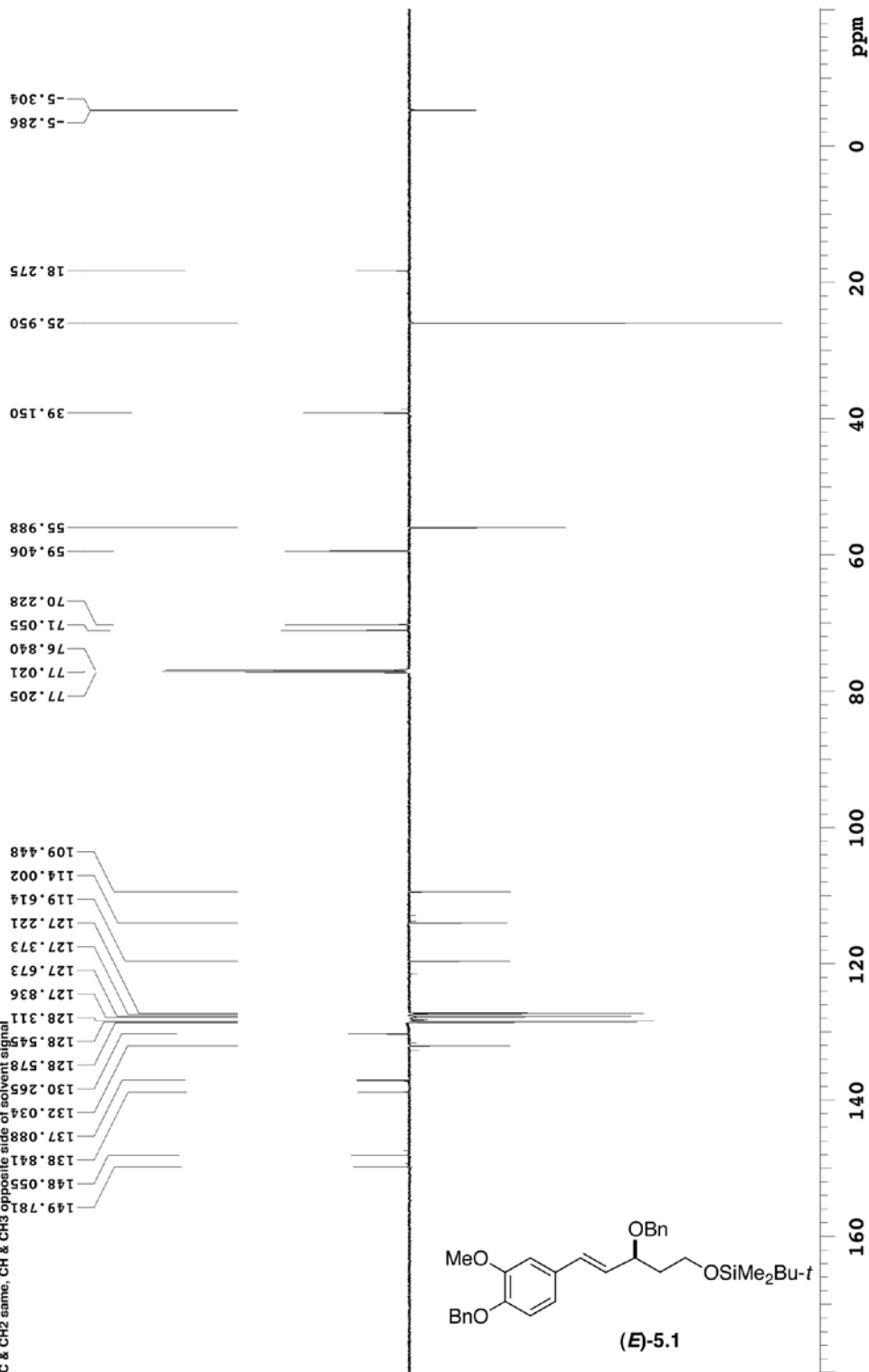
 Relaxation Delay(s): **1**
 Completed Scans: **128**

Hiroyuki, HH-2-149D

175.975 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)

temp 27.5 C -> actual temp = 27.0 C, coldid probe

C & CH2 same, CH & CH3 opposite side of solvent signal



File: /mnt/d600/home14/cive/mr/mrdata/149D/175.975 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm) 2018.11.05.v7_HH-2-149D_12.23_C13_APT_ad

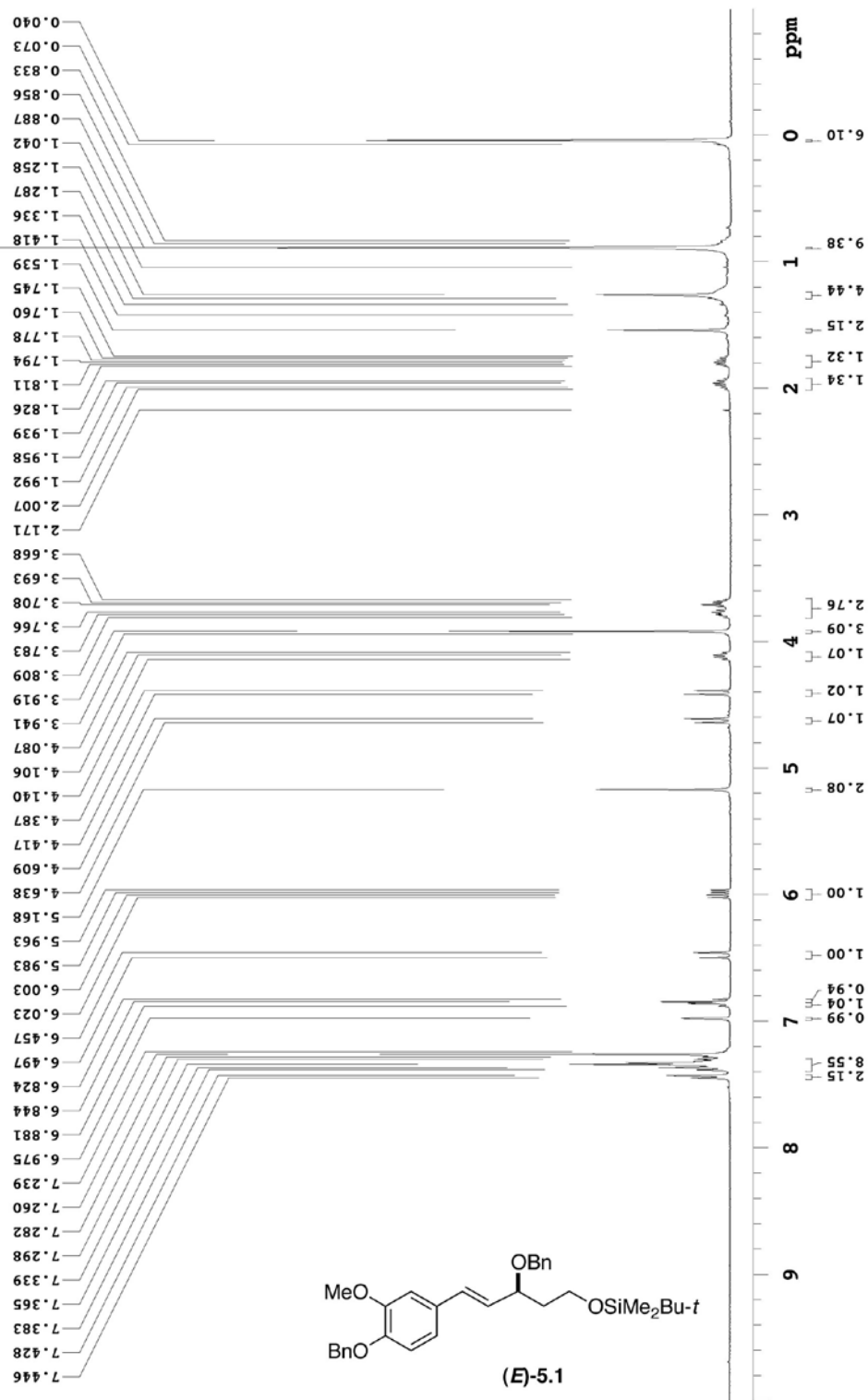
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 Recorded on: **m400, Oct 10 2018**
 Pulse Sequence: **PRESAT**

 Sweep Width(Hz): **4803.07**
 Digital Res.(Hz/pt): **0.07**

 Acquisition Time(s): **4.997**
 Hz per mm(Hz/mm): **18.35**

 Relaxation Delay(s): **0.1**
 Completed Scans: **32**

 HH-2-132C (m400 NMR)
 400.369 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm)
 temp 27.0 C -> actual temp = 27.0 C, m400gz probe


File: /mnt/d600/home14/c1venmr/nmrdata/102018_10_10.m4_HH-2-132C_loc5_22.14_H1_1D

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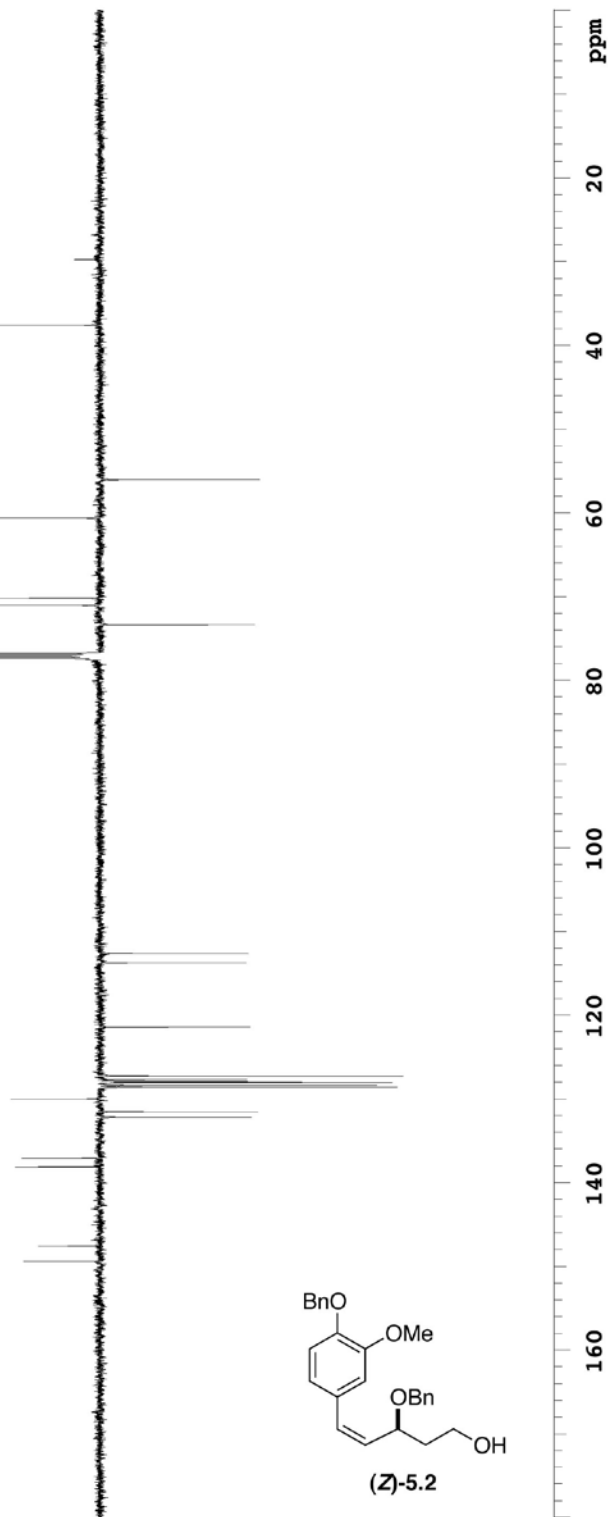


Recorded on: **u500, Oct 26 2018** Sweep Width(Hz): **33783.8** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **-APT_ad** Digital Res.(Hz/pp): **0.26** Hz per mm(Hz/mm): **94.36** Completed Scans: **128**

Hiroyuki, HH-2-143A
 125.688 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.7 C -> actual temp = 27.0 C, coldlual probe
 C & CH2 same, CH & CH3 opposite side of solvent signal

149.357
 147.542
 138.084
 137.071
 132.162
 131.514
 129.982
 128.583
 128.329
 127.990
 127.892
 127.652
 127.238
 121.395
 113.715
 112.602

77.276
 77.022
 76.768
 73.347
 71.024
 70.169
 60.610
 56.010
 37.540



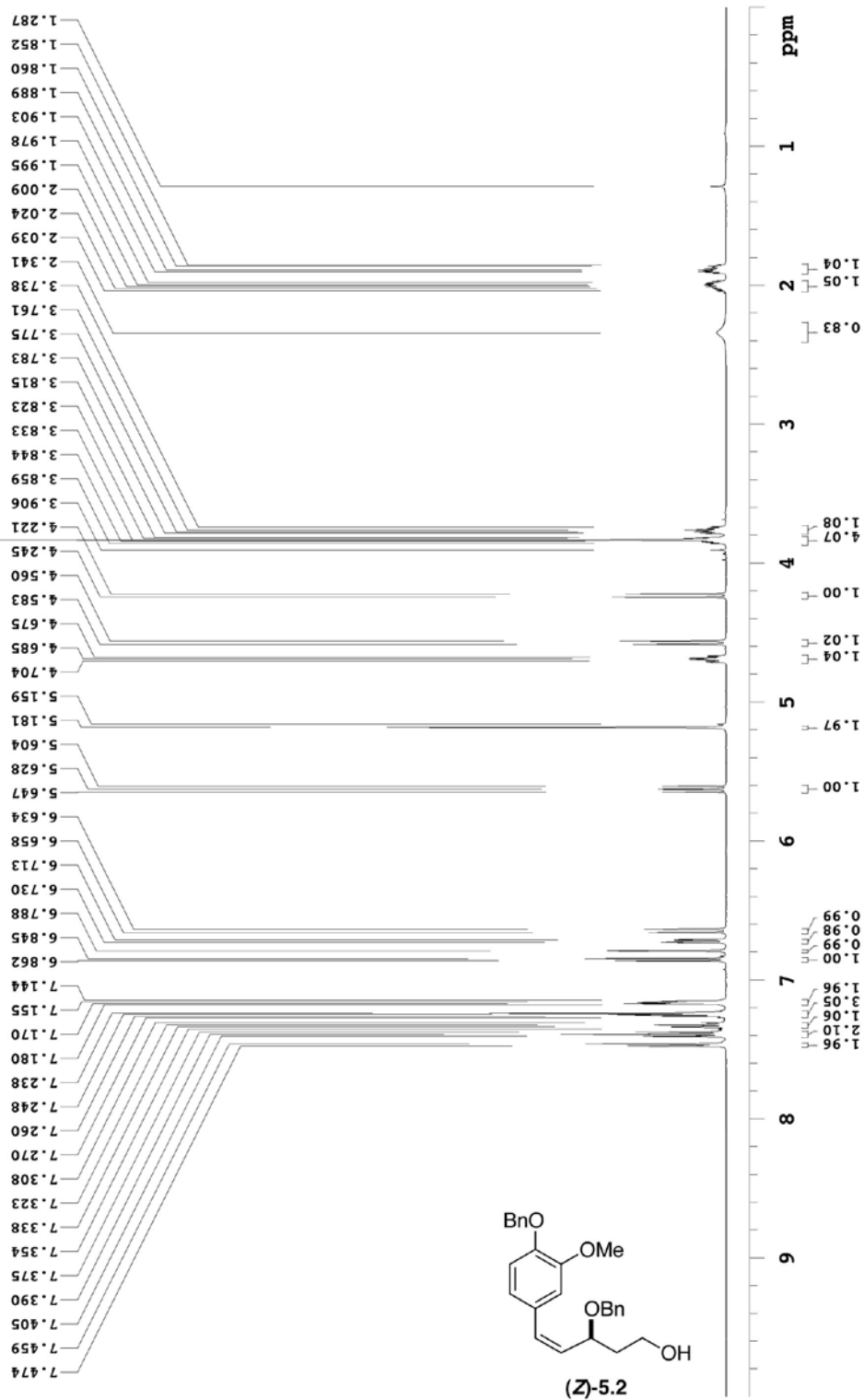
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Recorded on: **ibd5, Nov 6 2018** Sweep Width(Hz): **6000.6** Acquisition Time(s): **5** Relaxation Delay(s): **0.1**
 Pulse Sequence: **s2pul** Digital Res.(Hz/mm): **20.76** Hz per mm(Hz/mm): **20.76** Completed Scans: **16**

HH-2-151B (ibd5 NMR)
 498.118 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxdb probe



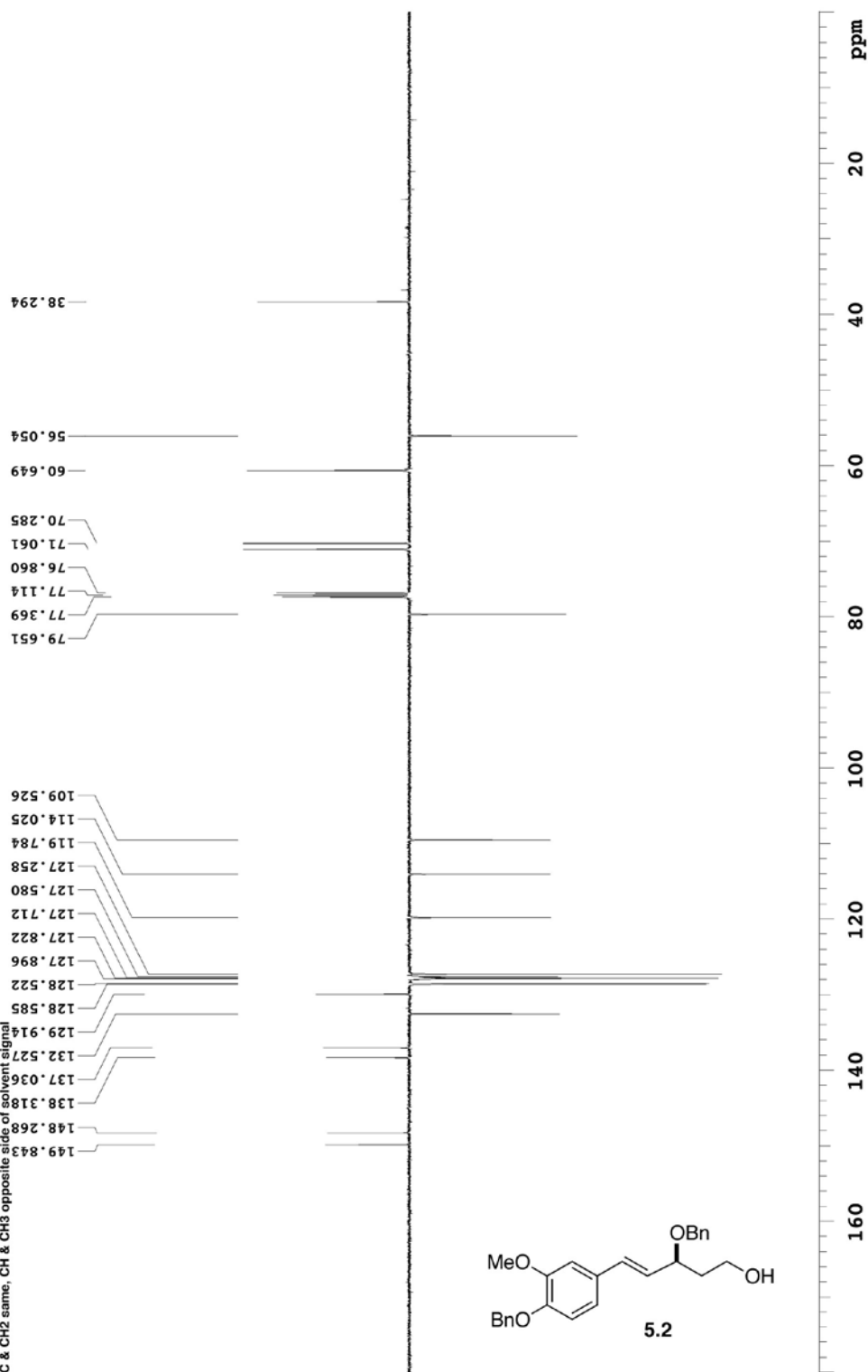
File: /mnt/d600/home14/civer/nmr/mrdata/hattori/2018.11.06.05_JH-2-151B_H1_1D

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Recorded on: **u500, Oct 26 2018** Sweep Width(Hz): **33783.8** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **-APT ad** Digital Res.(Hz/pp): **94.36** Hz per mm(Hz/mm): **94.36** Completed Scans: **20**

Hiroyuki, HH-2-143B
 125.688 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.7 C -> actual temp = 27.0 C, coldlual probe
 C & CH2 same, CH & CH3 opposite side of solvent signal

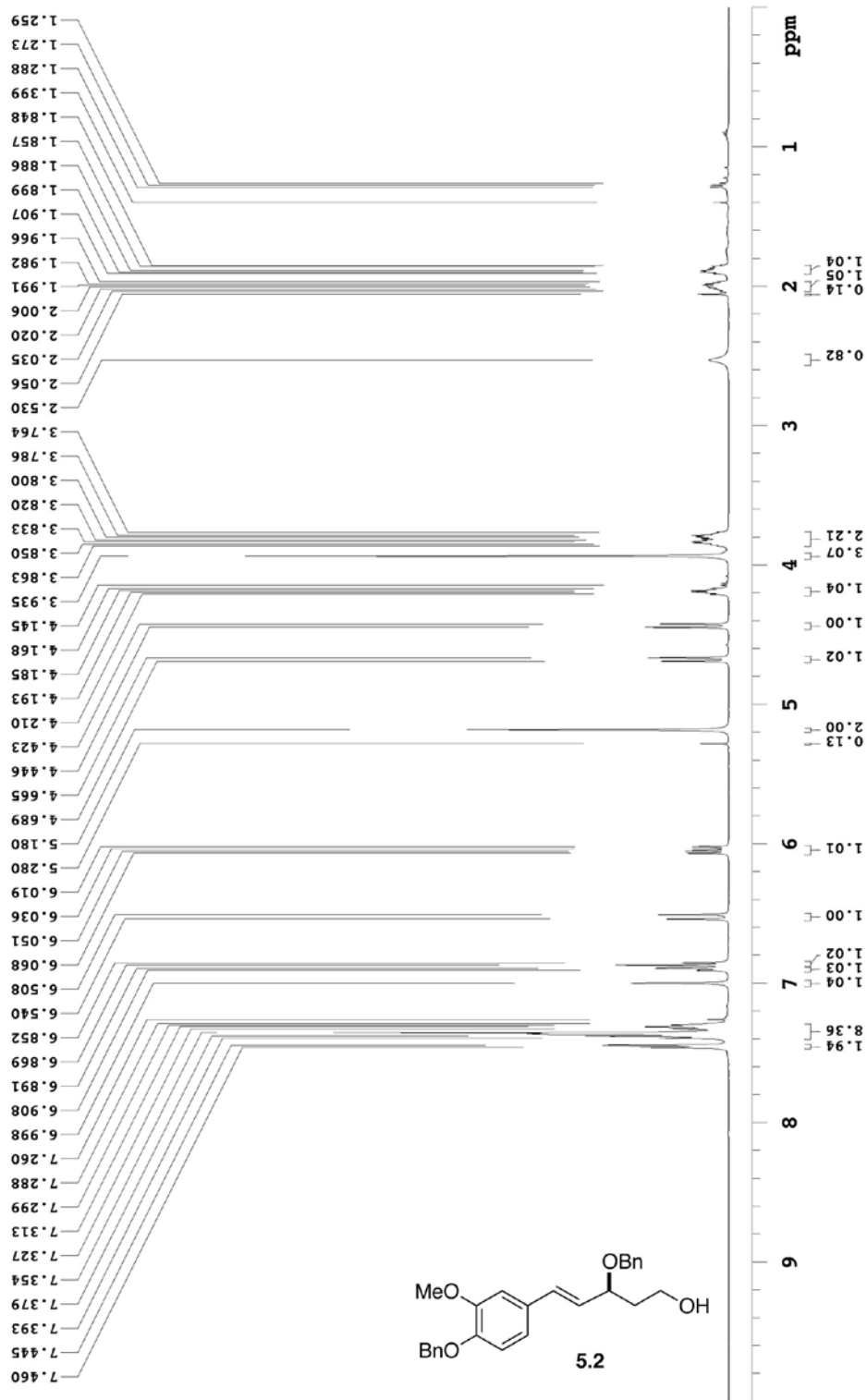


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Recorded on: **ibd5, Oct 25 2018** Sweep Width(Hz): **6000.6** Acquisition Time(s): **5** Relaxation Delay(s): **0.1**
 Pulse Sequence: **s2pul** Digital Res.(Hz/mm): **20.76** Hz per mm(Hz/mm): **20.76** Completed Scans: **16**

HH-2-143B (ibd5 NMR)
 498.118 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm)
 temp 26.9 C -> actual temp = 27.0 C, autoxdb probe



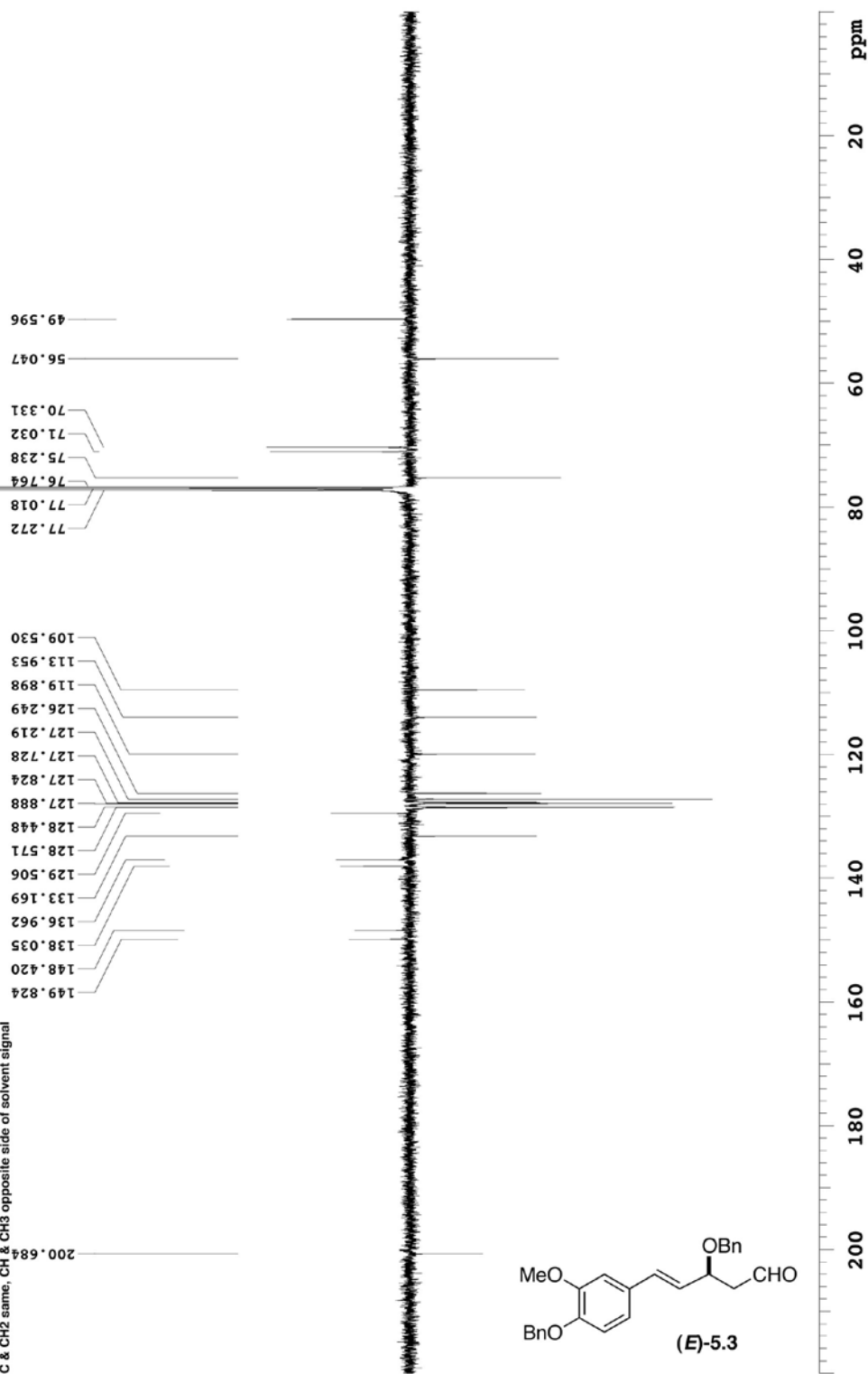
File: /mnt/d600/home14/civenm/nmr/data/hattori/2018.10.25.05_JHH-2-143B_H1_1D

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Recorded on: **u500, Nov 1 2018** Sweep Width(Hz): **33783.8** Acquisition Time(s): **1** Relaxation Delay(s): **1**
 Pulse Sequence: **APT ad** Digital Res.(Hz/pp): **0.26** Hz per mm(Hz/mm): **115.27** Completed Scans: **128**

Hiroyuki, HH-2-148B
 125.688 MHz C13 APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm)
 temp 27.7 C -> actual temp = 27.0 C, coldludal probe
 C & CH2 same, CH & CH3 opposite side of solvent signal



File: /mnt/d600/home14/ci/venmfr/mrdata/148B_11/2018.11.01.u5_HH-2-148B_fc1_06.23_C13_APT_ad

