

## Supplementary Information

### Synthesis and LC-MS/MS Analysis of Desmosine-CH<sub>2</sub>, a Potential Internal Standard for the Degraded Elastin Biomaker Desmosine

Yuko Murakami,<sup>a</sup> Rina Suzuki,<sup>a</sup> Hiroto Yanuma,<sup>a</sup> Jiantao He,<sup>b</sup> Shuren Ma,<sup>b</sup> Gerard M.  
Turino,<sup>b</sup> Yong Y. Lin<sup>b</sup> and Toyonobu Usuki\*<sup>a</sup>

<sup>a</sup>*Department of Materials and Life Sciences, Faculty of Science and Technology, Sophia  
University, 7-1 Kioicho, Chiyoda-ku, Tokyo 102-8554, Japan*

<sup>b</sup>*Roosevelt Hospital Center, Mount Sinai School of Medicine, 432W 58th St, New York,  
NY 10019, USA*

\* Corresponding author.

Tel.: +81 3 3238 3446

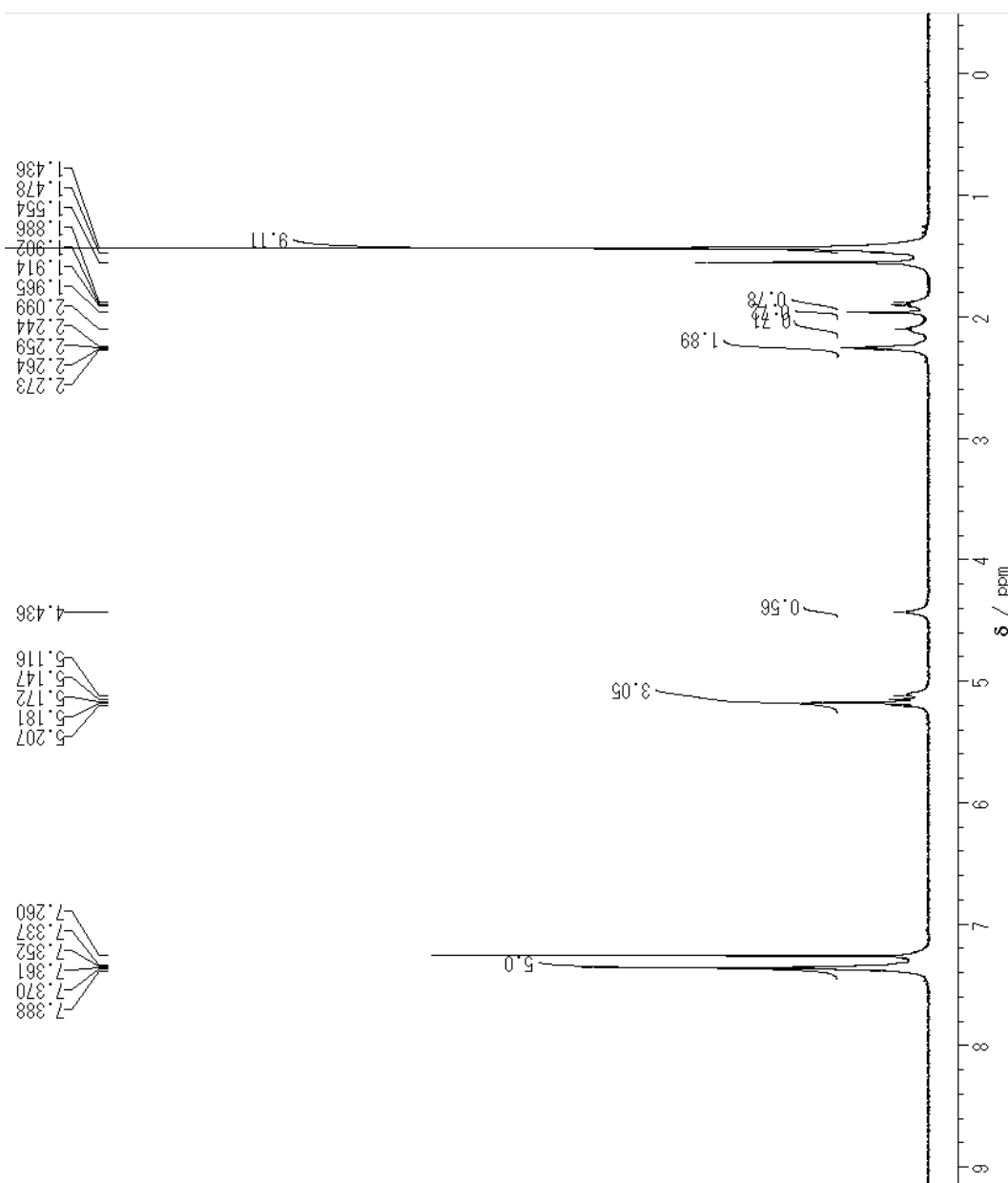
Fax: +81 3 3238 3361

*E-mail address:* [t-usuki@sophia.ac.jp](mailto:t-usuki@sophia.ac.jp) (T. Usuki)

**<sup>1</sup>H and <sup>13</sup>C NMR spectra of synthetic compounds**

Supplementary Data 1

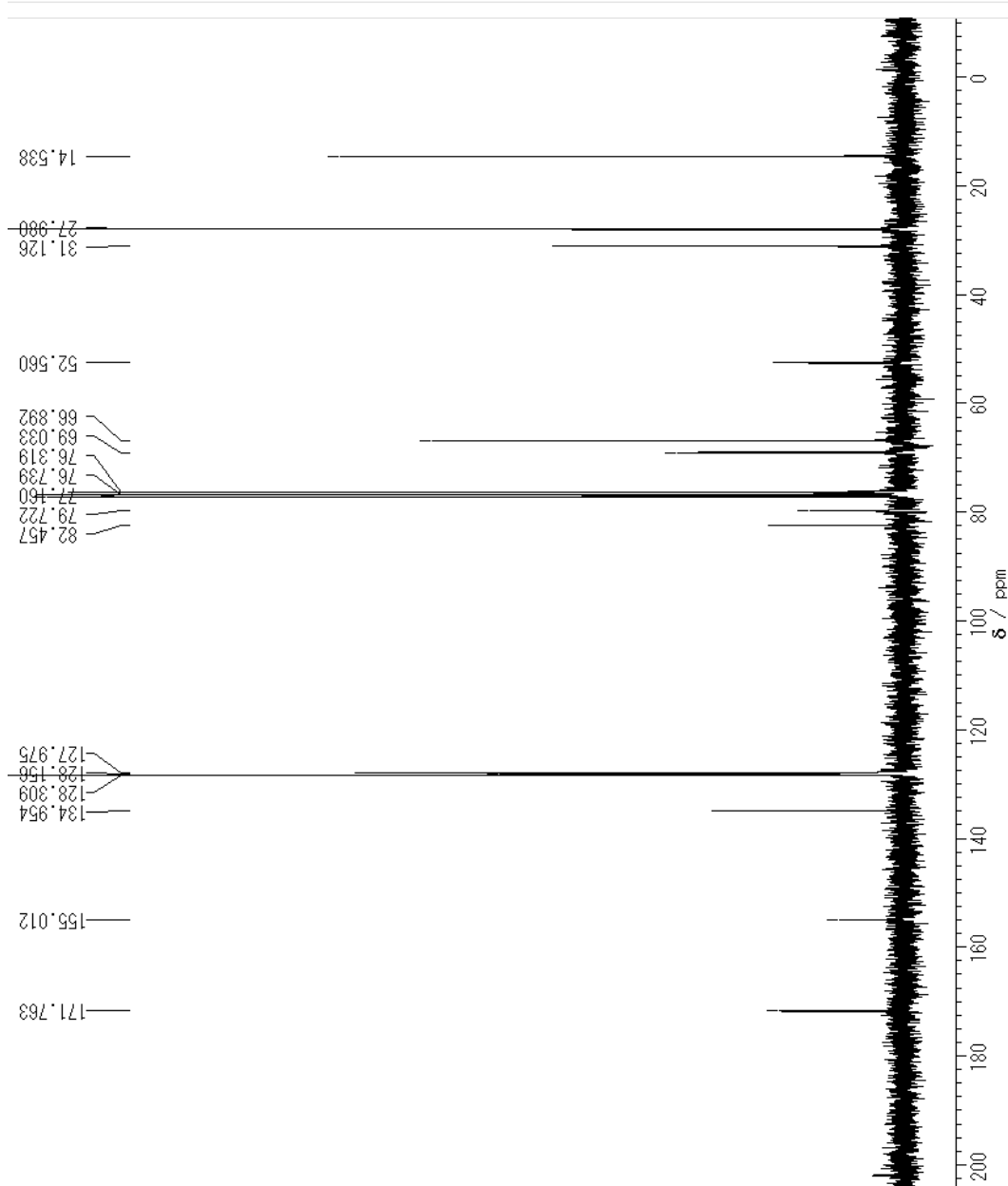
File D:\研究室\NMR\CH2-DES\MURAKAMI145\_P  
 ROTON-1\_145  
 測定時の77-値名  
 Date 25-06-2012 15:48:36  
 Comment 34/murakami145  
 ObsMod 1H  
 ExMode single\_pulse.ex2  
 ObsFreq 500.16 MHz  
 ObsSret 2.41 kHz  
 ObsSet 6.0082 Hz  
 ObsFine  
 Point 13107  
 Frequency(Span) 7507.393 Hz  
 Scan 16  
 AcqTime 1.7459 s  
 PD 4.0 s  
 Pulse 3.9  $\mu$ s  
 Temperature 23.5  $^{\circ}$ C  
 Solvent CDCl<sub>3</sub>  
 Reference 7.26 ppm  
 Broad.Factor 0.2864 Hz  
 RGain 82  
 Printed 2014/Jan/20 13:55:07  
 Operator



S1-1. <sup>1</sup>H NMR spectrum of 8.

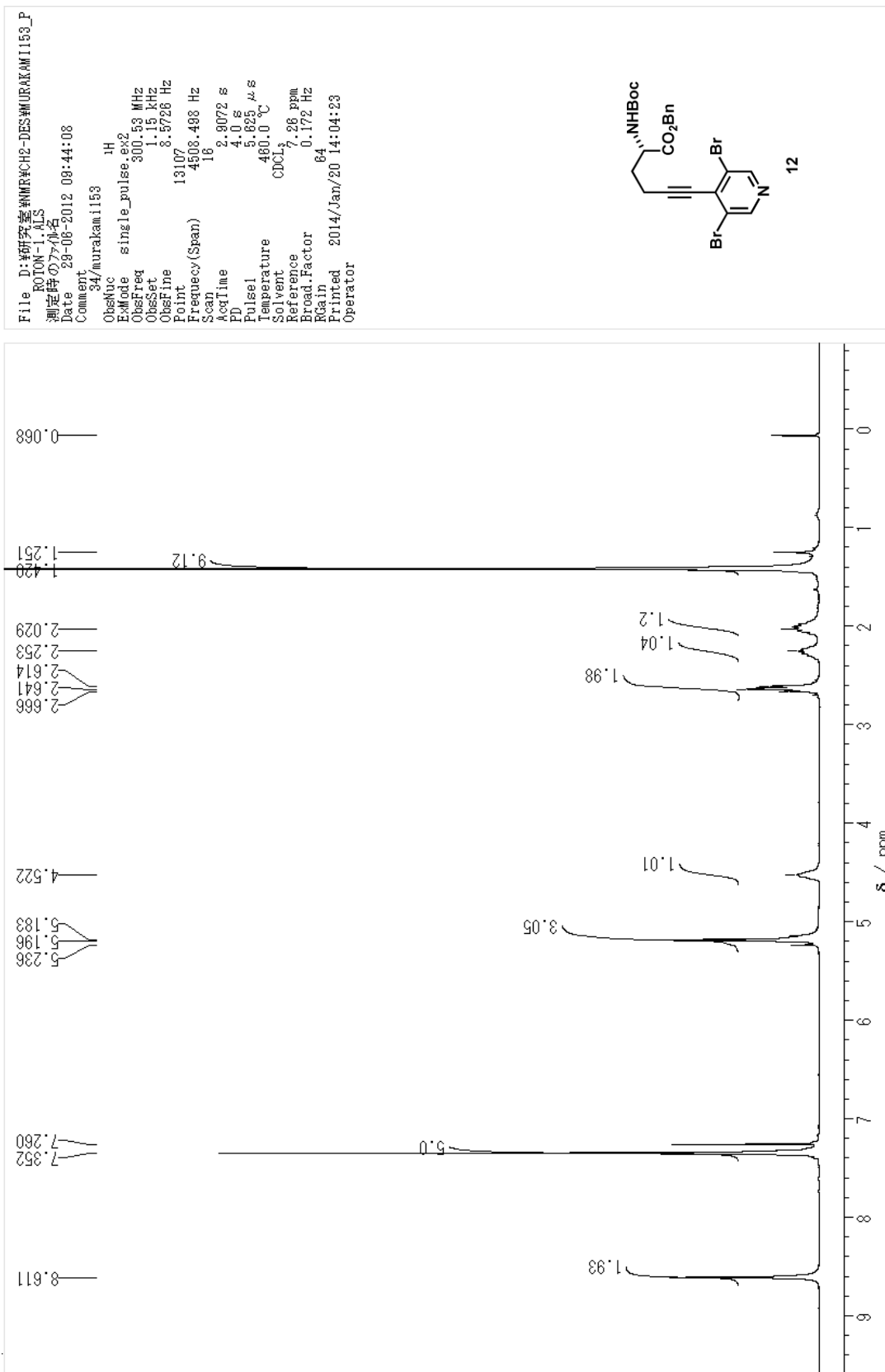
Supplementary Data 1

File D:\研究室\NMR\CH2-DES\MURAKAMI154\_C  
 ARBON-13.ALLS  
 測定時のファイル名  
 Date 02-07-2012 20:30:52  
 Comment  
 34/murakami154  
 ObsNuc 13C  
 ExpMode single\_pulse\_dec  
 ObsFreq 75.57 MHz  
 ObsSet 5.79 kHz  
 ObsFine 1.0834 Hz  
 Point 26214  
 Frequency(Span) 18939.11 Hz  
 Scan 256  
 AcqTime 1.3841 s  
 PD 2.0 s  
 Pulse 3.0 μs  
 Temperature 24.0 °C  
 Solvent CDCl<sub>3</sub>  
 Reference 77.18 ppm  
 Broad.Factor 0.3612 Hz  
 RGain 80  
 Printed 2014/Jan/20 13:58:55  
 Operator



S1-2. <sup>13</sup>C NMR spectrum of **8**.

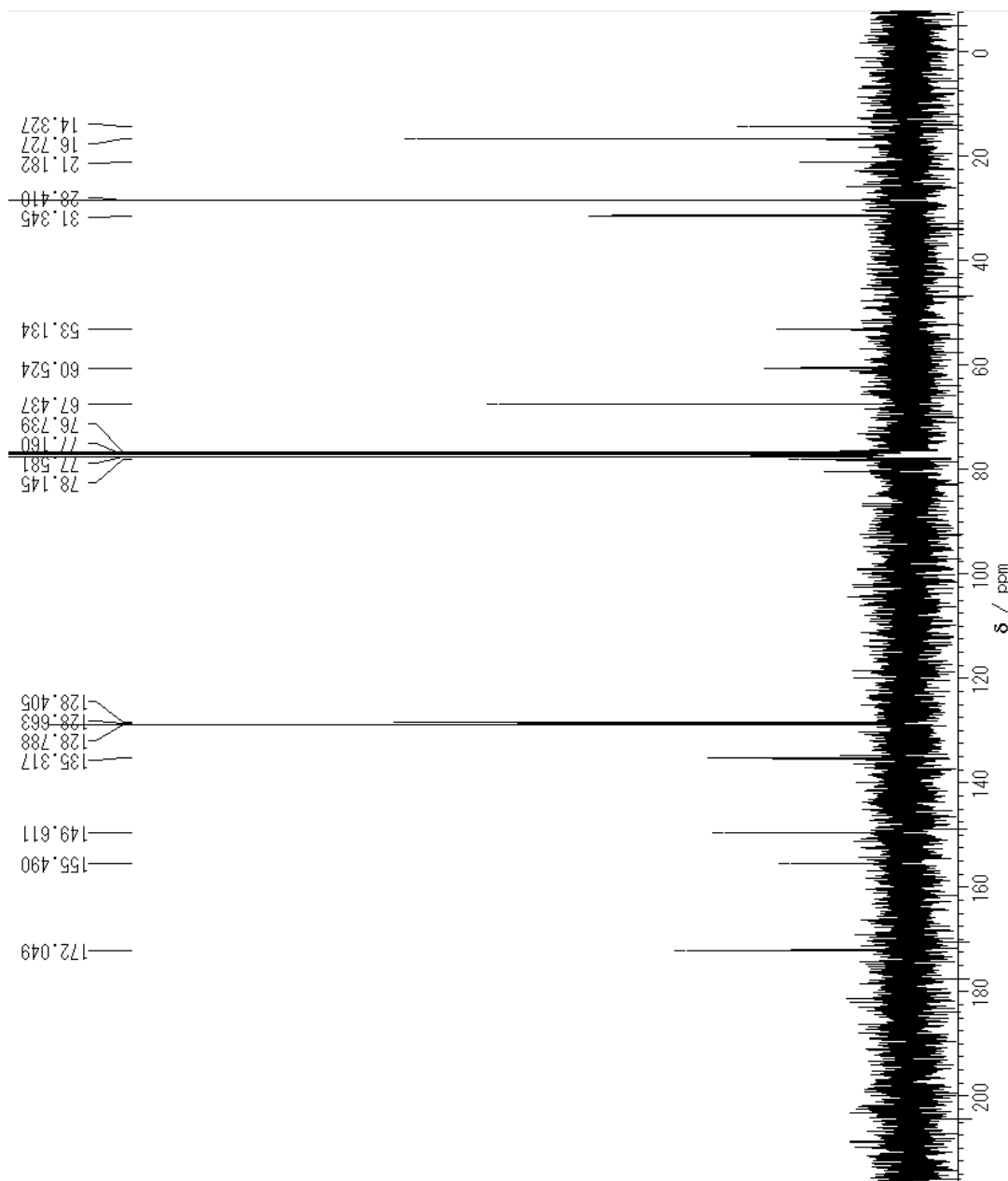
Supplementary Data 1



SI-3. <sup>1</sup>H NMR spectrum of **12**.

Supplementary Data 1

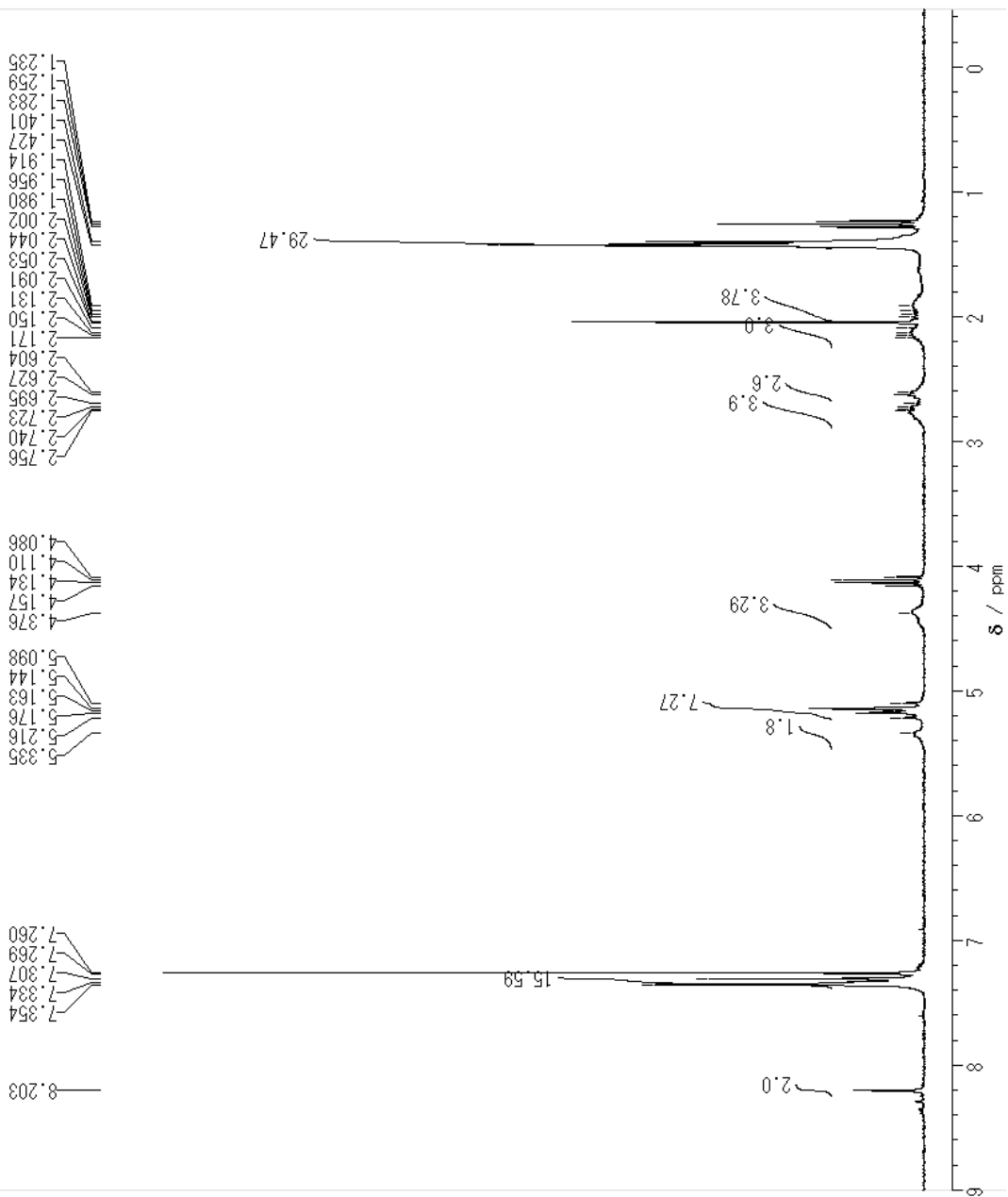
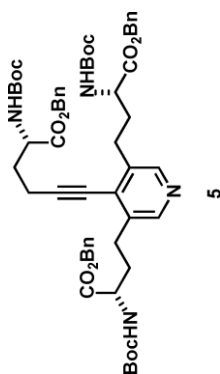
File D:\研究室\NMR\CH2-DES\MURAKAMI157\_C  
 ARBON-13ALS  
 測定時の7名  
 Date 03-07-2012 14:08:32  
 Comment 34/murakami157  
 ObsType 13C  
 EMode single\_pulse\_dec  
 ObsFreq 75.57 MHz  
 ObsSet 5.78 kHz  
 ObsFlne 1.0834 Hz  
 Point 26214  
 Frequency(Span) 18838.11 Hz  
 Scan 256  
 AcqTime 1.3841 s  
 PD 2.0 s  
 Pulse 3.0 μs  
 Temperature 24.0 °C  
 Solvent CDCl<sub>3</sub>  
 Reference 77.16 ppm  
 Broad.Factor 0.3612 Hz  
 Gain 60  
 Printed 2014/Jan/20 14:08:20  
 Operator



SI-4. <sup>13</sup>C NMR spectrum of 12.

Supplementary Data 1

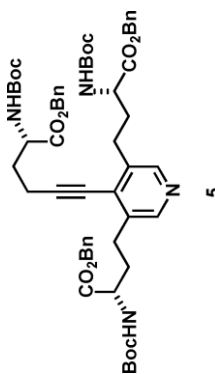
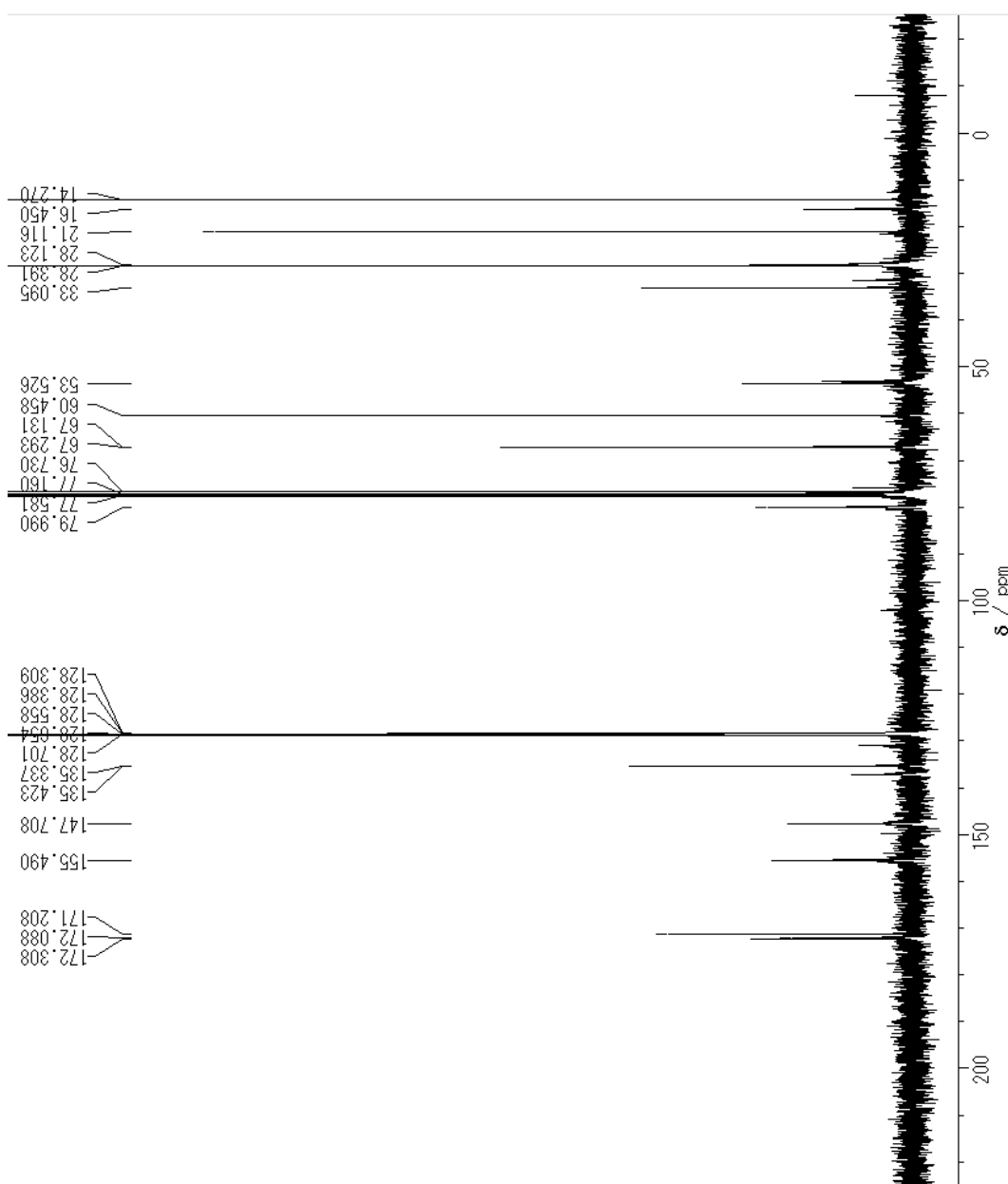
File D:\研究室\NMR\CH2-DES\MURAKAMI219\_P  
 ROTON-1 ALS  
 測定時の文件名  
 Date 28-08-2012 11:36:39  
 Comment 34/murakami219  
 ObsNuc <sup>1</sup>H  
 ExpMode single\_pulse.ex2  
 ObsFreq 300.53 MHz  
 ObsSet 1.15 kHz  
 ObsFine 8.5728 Hz  
 Point 13107  
 Frequency(Span) 4508.498 Hz  
 Scan 16  
 AcqTime 2.9072 s  
 PD 4.0 s  
 Pulse 5.875 μs  
 Temperature 23.7 °C  
 Solvent CDCl<sub>3</sub>  
 Reference 7.26 ppm  
 Broad.Factor 0.172 Hz  
 RGain 70  
 Printed 2014/Jan/20 14:17:52  
 Operator



S1-5. <sup>1</sup>H NMR spectrum of 5.

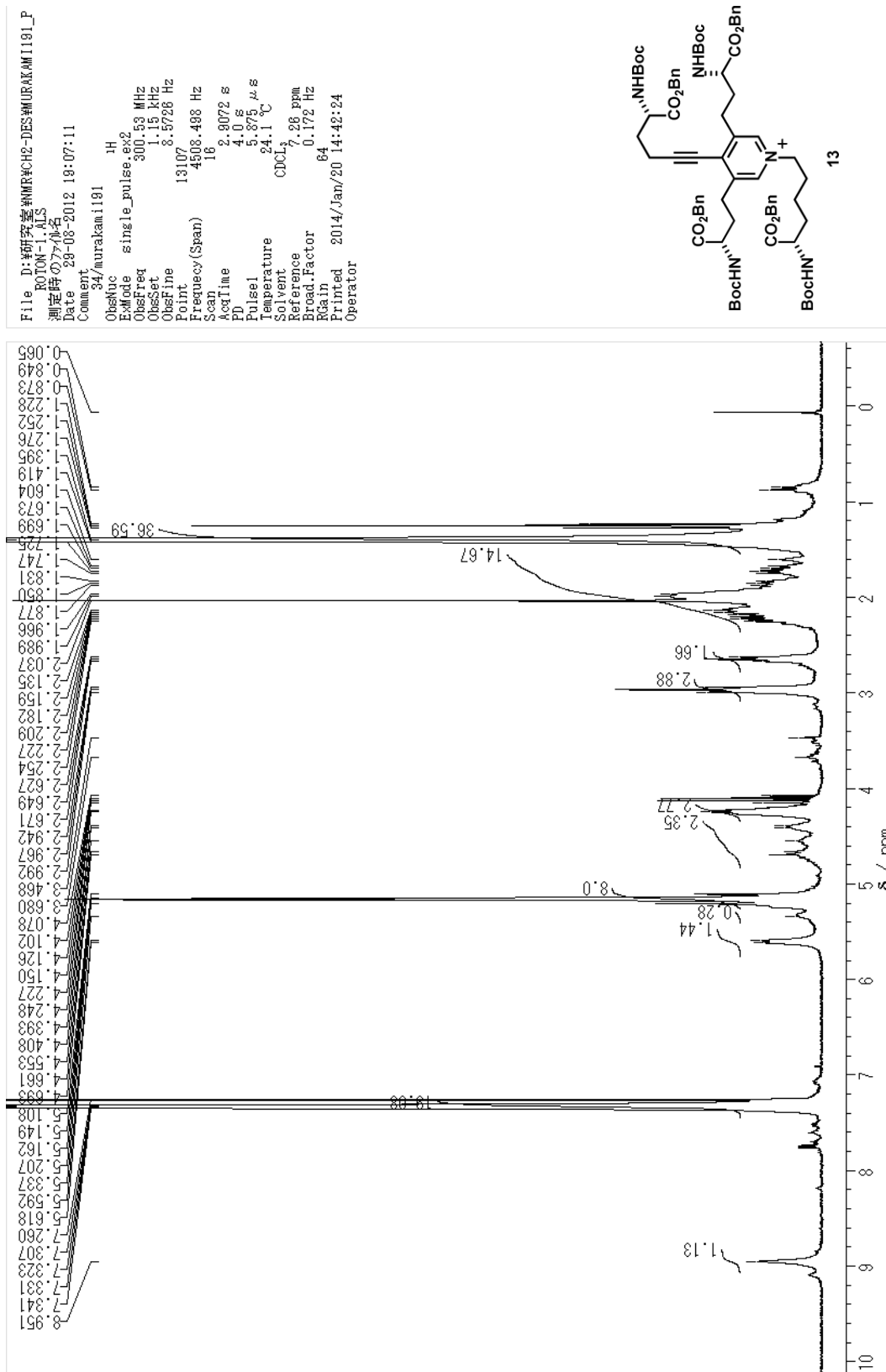
Supplementary Data 1

File D:\研究室\NMR\CH2-DES\MURAKAMI248\_C  
 ARBON-13ALS  
 測定時の77-値石  
 Date 15-11-2012 08:18:10  
 Comment 34/murakami248  
 ObsKuc 13C  
 ExMode single\_pulse\_dec  
 ObsFreq 75.57 MHz  
 ObsSet 5.79 kHz  
 ObsFine 1.0834 Hz  
 Point 28214  
 Frequency(Span) 18938.11 Hz  
 Scan 512  
 AcqTime 1.3841 s  
 PD 2.0 s  
 Pulse1 3.0 s  
 Temperature 22.4 °C  
 Solvent CDCl<sub>3</sub>  
 Reference 77.16 ppm  
 Broad.Factor 0.3612 Hz  
 RGain 60  
 Printed 2014/Jan/20 14:18:22  
 Operator



S1-6. <sup>13</sup>C NMR spectrum of 5.

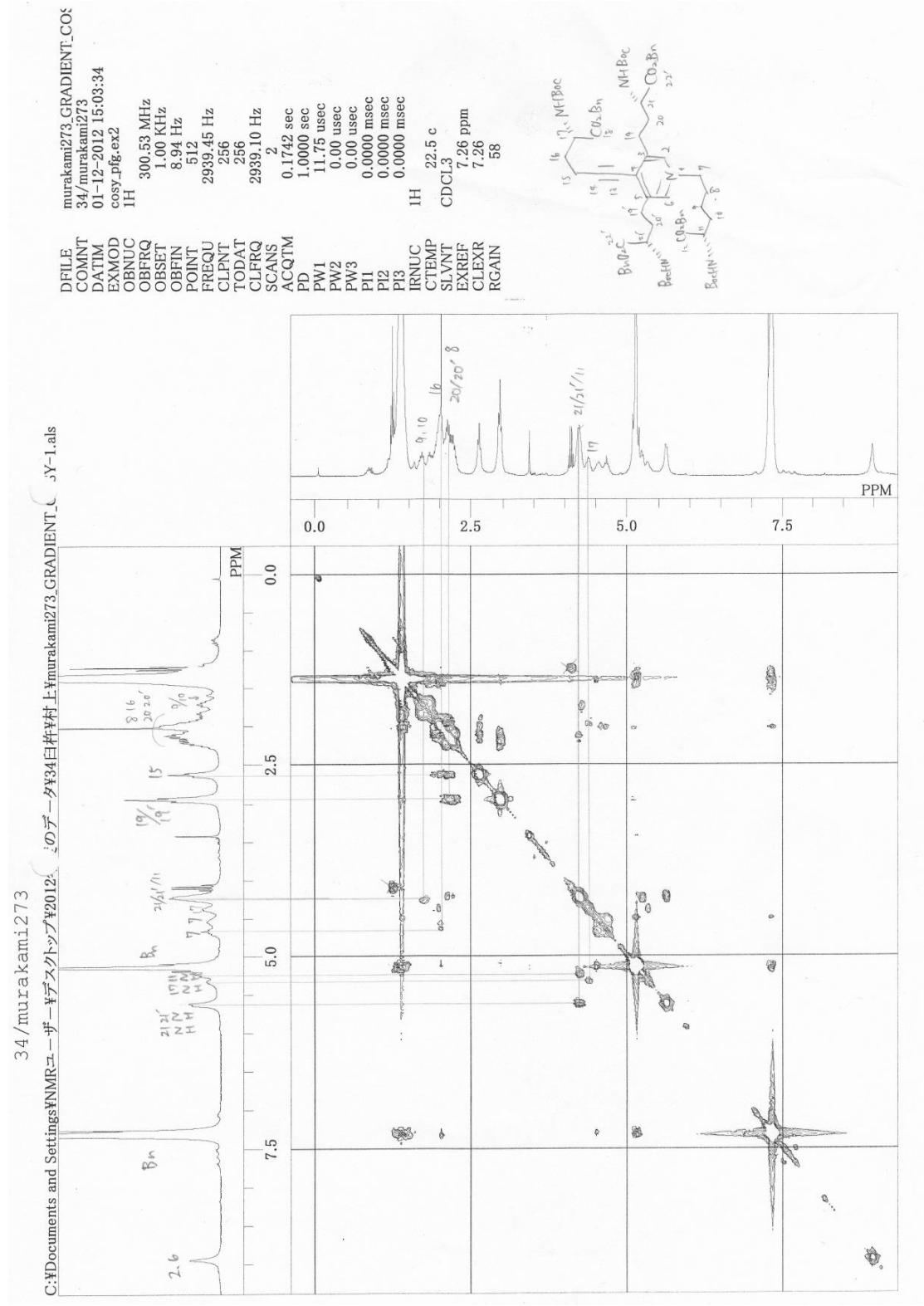
Supplementary Data 1



S1-7. <sup>1</sup>H NMR spectrum of 13.



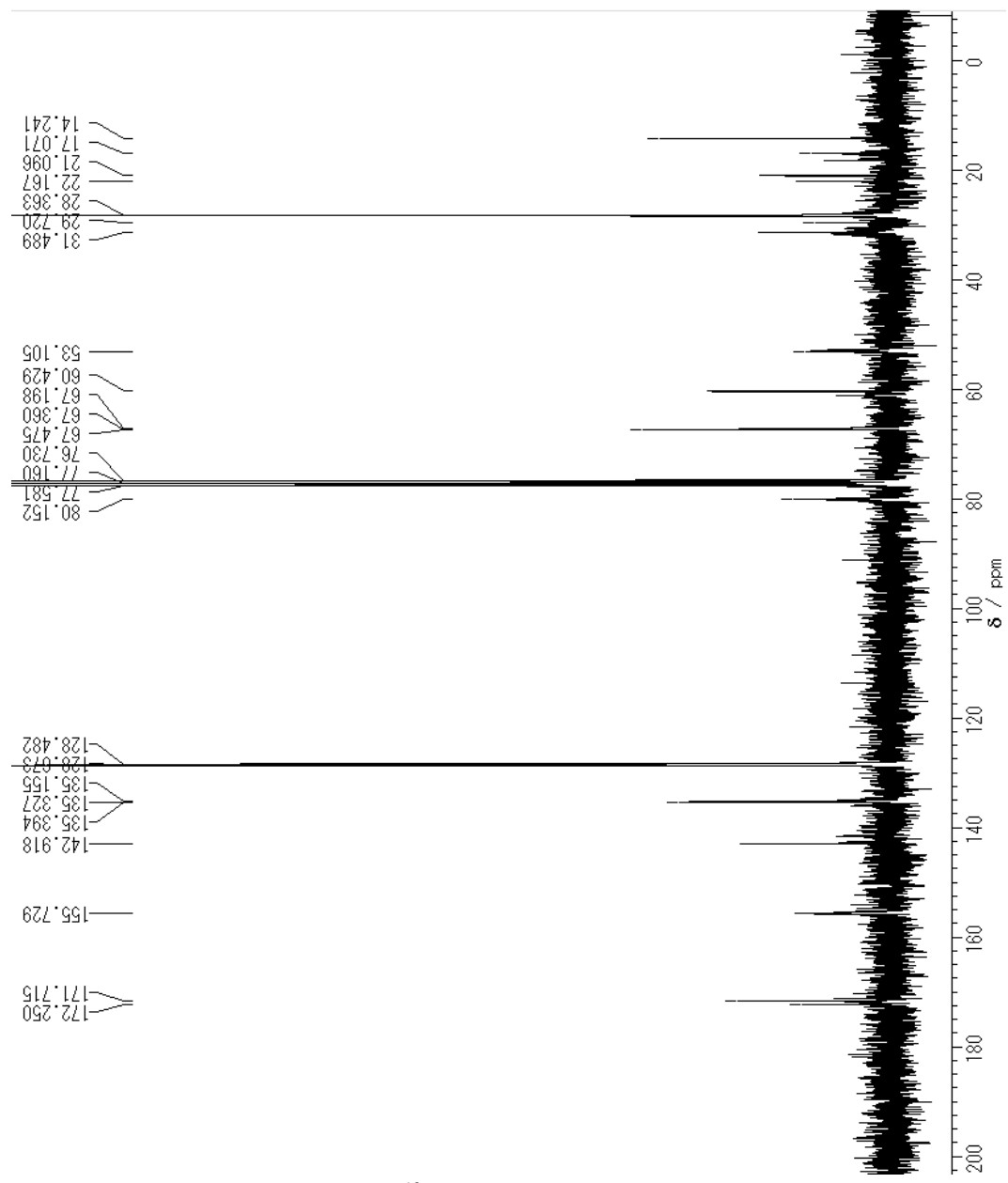
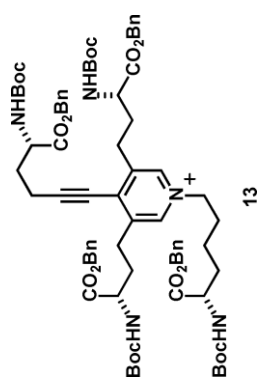
Supplementary Data 1



S1-8. HH-COSY spectrum of 13.

Supplementary Data 1

File D:\研究室\NMR\CH2-DES\MURAKAMI267\_C  
 ARDON-1.ALS  
 測定時の名前  
 Date 27-11-2012 08:18:13  
 Comment 34/murakami267  
 Obskuc 13C  
 ExpMode single\_pulse\_dec  
 ObsFreq 75.57 MHz  
 ObsSet 5.79 kHz  
 ObsFine 1.0834 Hz  
 Point 28214  
 Frequency(Span) 18939.11 Hz  
 Scan 256  
 AcqTime 1.3841 s  
 PD 9.0 s  
 Pulse1 3.0 s  
 Temperature 22.3 °C  
 Solvent CDCl<sub>3</sub>  
 Reference 77.16 ppm  
 Broad.Factor 0.3612 Hz  
 RGain 80  
 Printed 2014/Jan/20 14:43:48  
 Operator

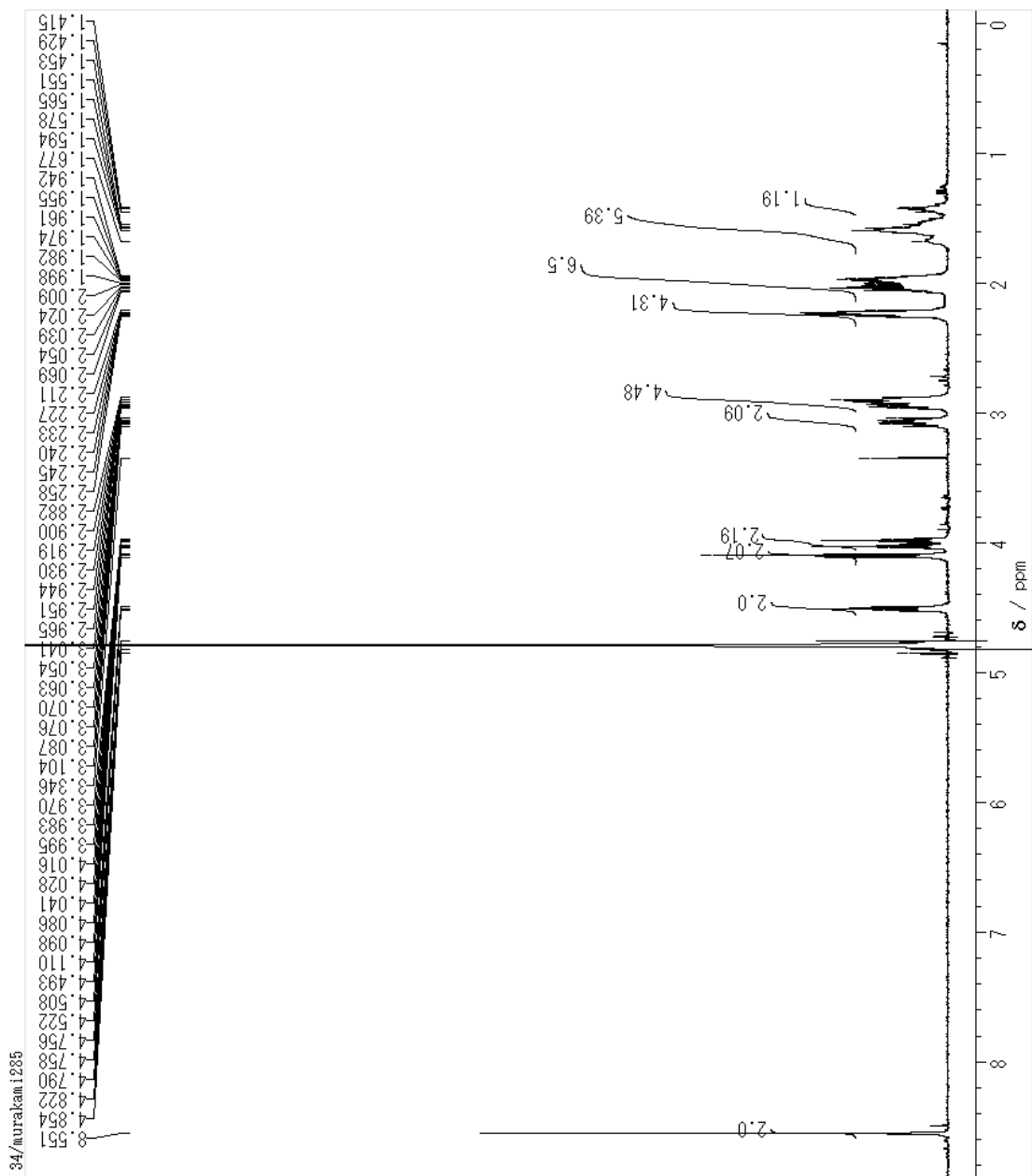
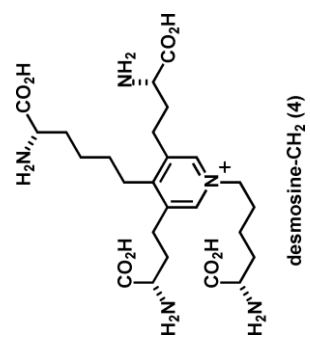


S1-9. <sup>13</sup>C NMR spectrum of 13.

Supplementary Data 1

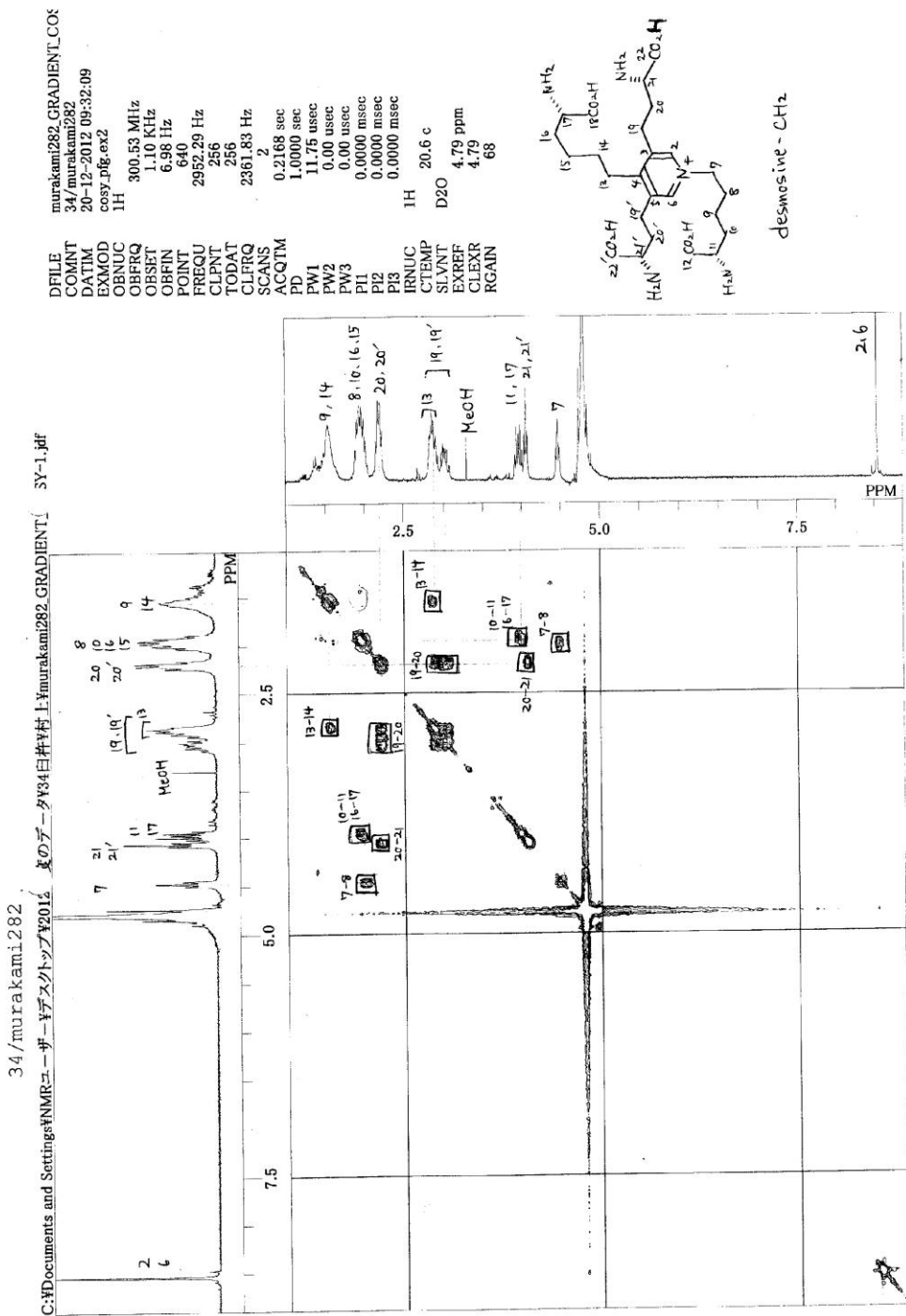
File C:\USERS\村上祐子\DESKTOP\研究資料\NM  
 R#CH2-DESMOSIMURAKAMI285\_PROTON-1\_ALS  
 測定時の7>小名  
 Date 2012-12-20 13:54:11  
 Comment 34/murakami285

ObsNuc 1H  
 ExMode single\_pulse.ex2  
 ObsFreq 500.16 MHz  
 ObsSet 2.41 kHz  
 ObsFine 6.0082 Hz  
 Poinc 13107  
 Frequency(Span) 7507.393 Hz  
 Scan 16  
 AcqTime 1.7459 s  
 PD 4.0 s  
 Pulse 5.5 μs  
 Temperature 23.8 °C  
 Solvent D<sub>2</sub>O  
 Reference 4.79 ppm  
 Broad.Factor 0.2864 Hz  
 RGain 50  
 Printed 2014/Feb/14 08:12:49  
 Operator



S1-10. <sup>1</sup>H NMR spectrum of 4.

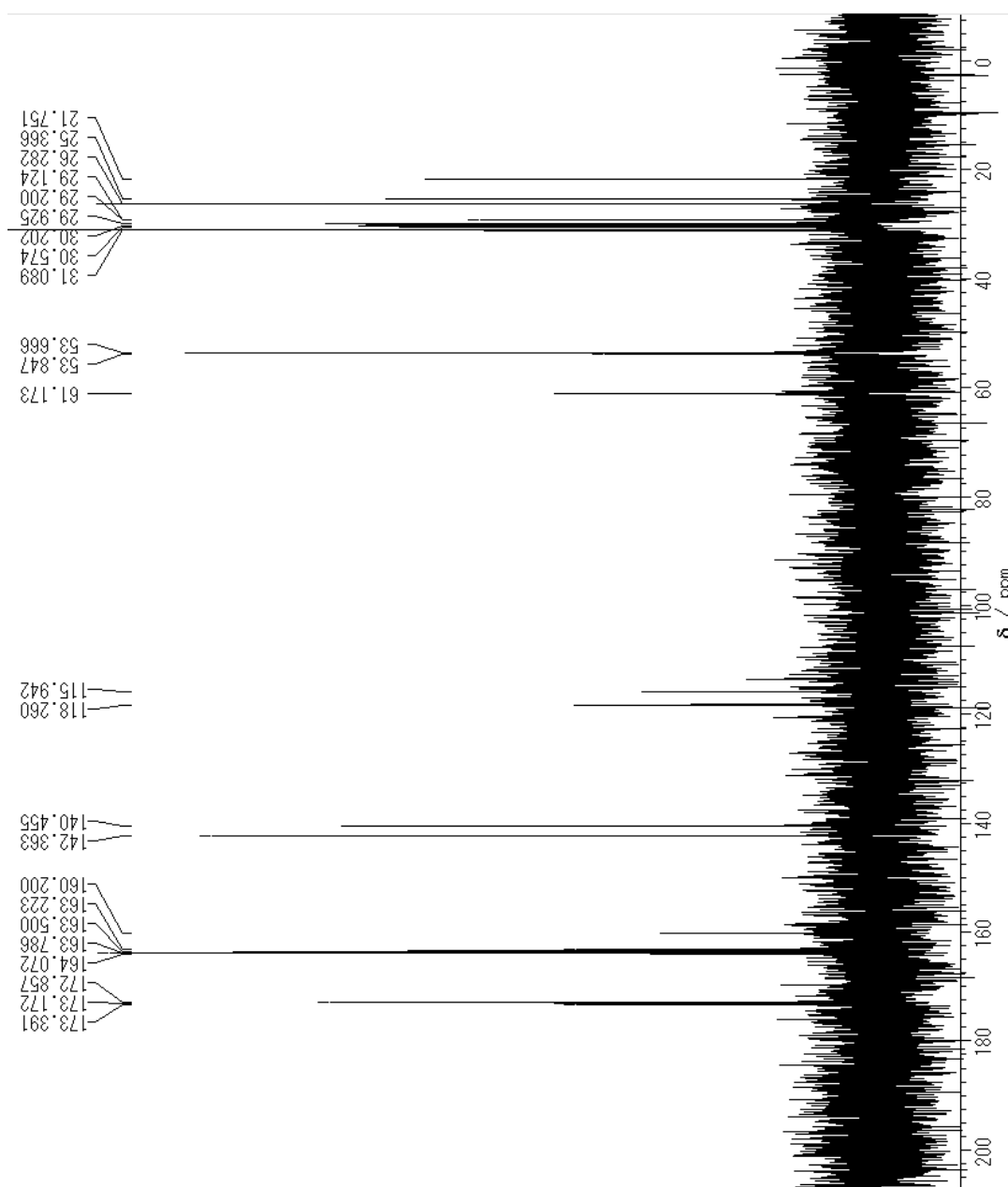
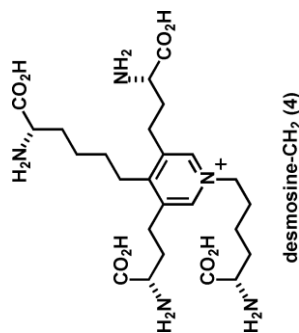
Supplementary Data 1



S1-11. HH-COSY spectrum of 4.

Supplementary Data 1

File D:\研究室\NMR\CH2-DES\MURAKAMI28562  
 CARBON-4-2.ALS  
 測定時の77名  
 Date 27-12-2012 08:19:04  
 Comment 84/murakami28562 uc  
 Obsfuc single\_pulse\_dec  
 ExMode 128.77 MHz  
 ObsFreq 7.87 kHz  
 ObsSet 4.2131 Hz  
 Obsfline 26214  
 Point 31446.08 Hz  
 Frequency(Span) 20000  
 Scan 0.8336 s  
 AcqTime 2.0 s  
 PD 3.6667 μs  
 Pulse Temperature 23.5 °C  
 Solvent D<sub>2</sub>O  
 Reference 168.5 ppm  
 Broad.Factor 0.5888 Hz  
 Gain 54  
 Printed 2014/Jan/20 14:48:32  
 Operator



SI-12. <sup>13</sup>C NMR spectrum of 4.