

*Supplementary information for*

**Synthesis of lactam-bridged cyclic peptides using sequential olefin metathesis and diimide reduction reactions**

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Contents:

Copies of <sup>1</sup>H, <sup>13</sup>C NMR charts: **1, 3, 4, 7, 9, 10, 11, 12, 19** and **20**.....S1–S21

4-Allylcarbamoyl-2-(9H-fluoren-9-ylmethoxycarbonylamino)-butyric acid (1).

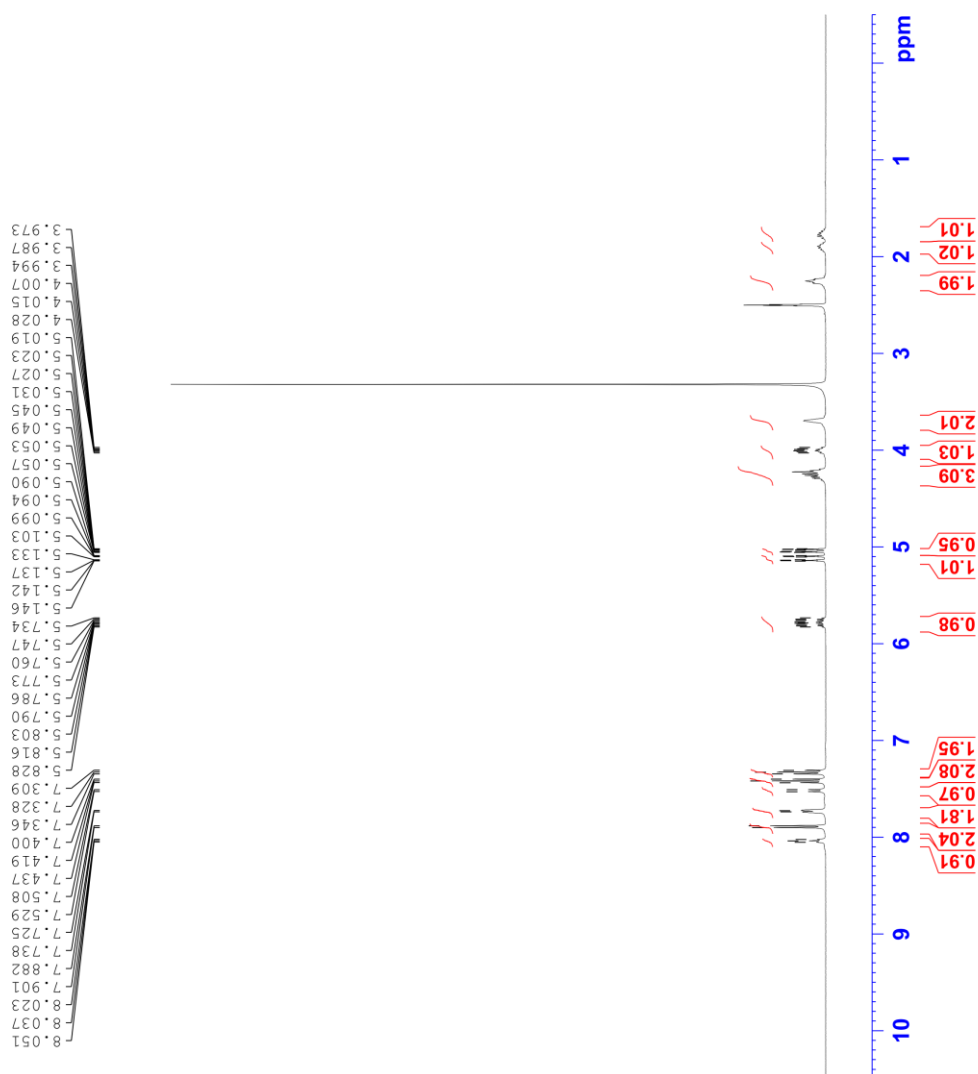
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PROCNO   1

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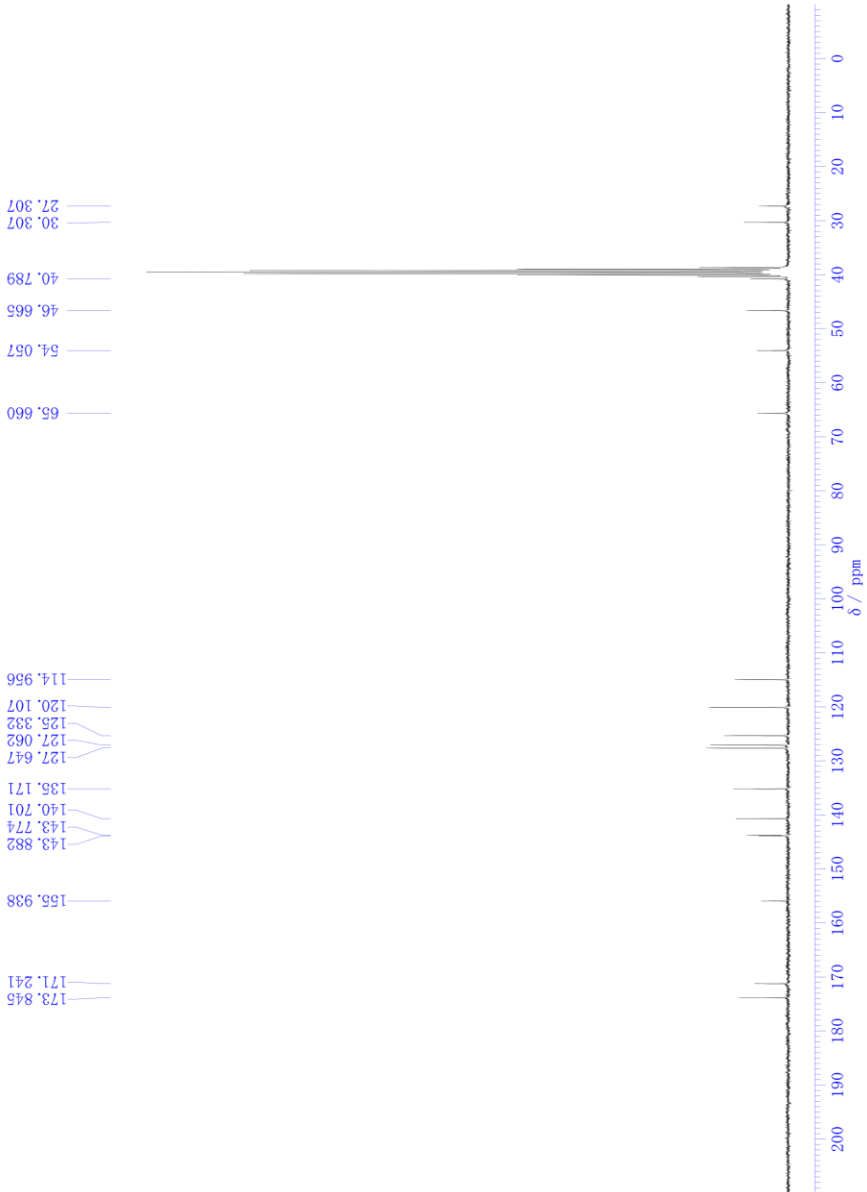
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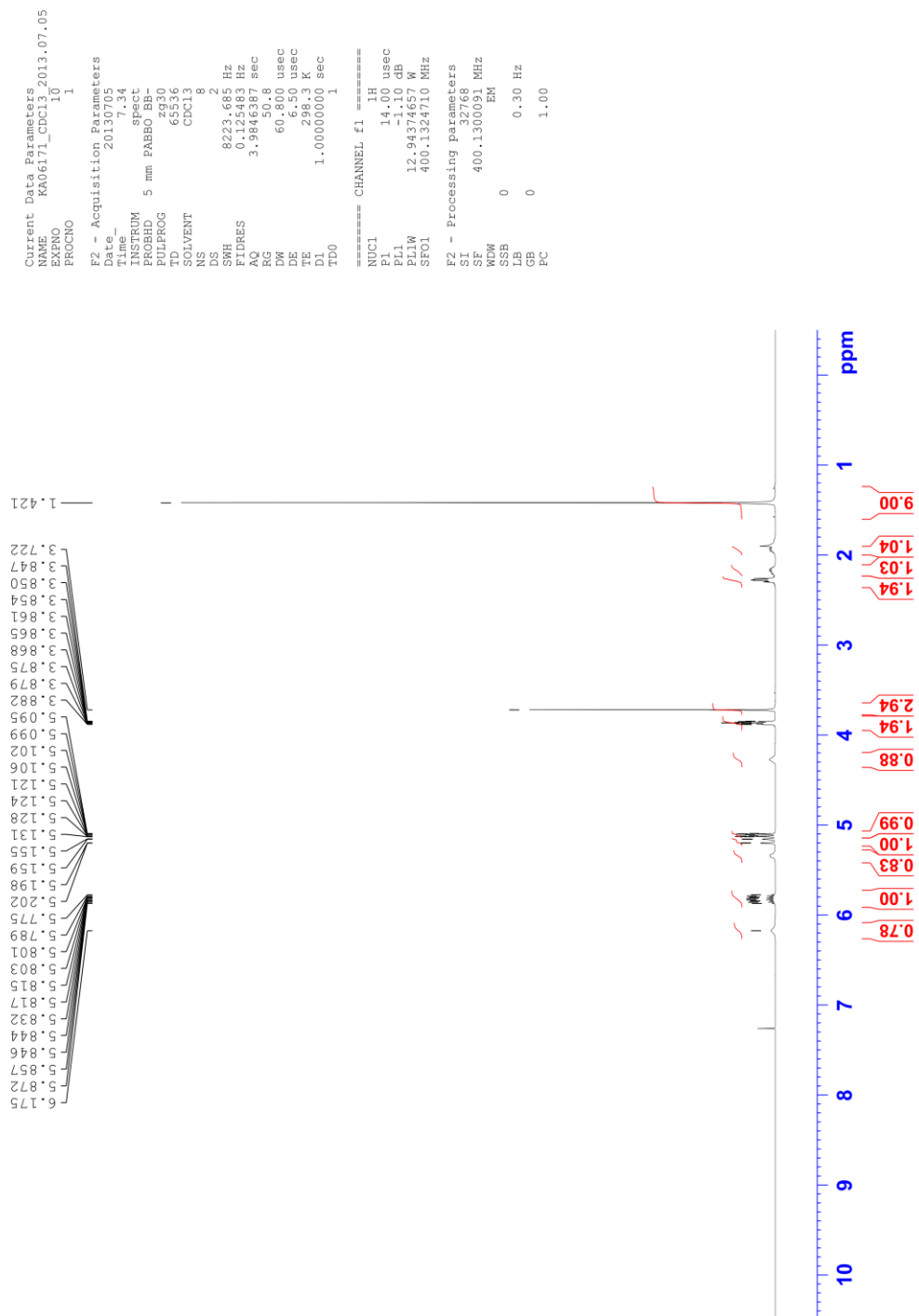
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 Treatment  
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 RGain 24

Operator

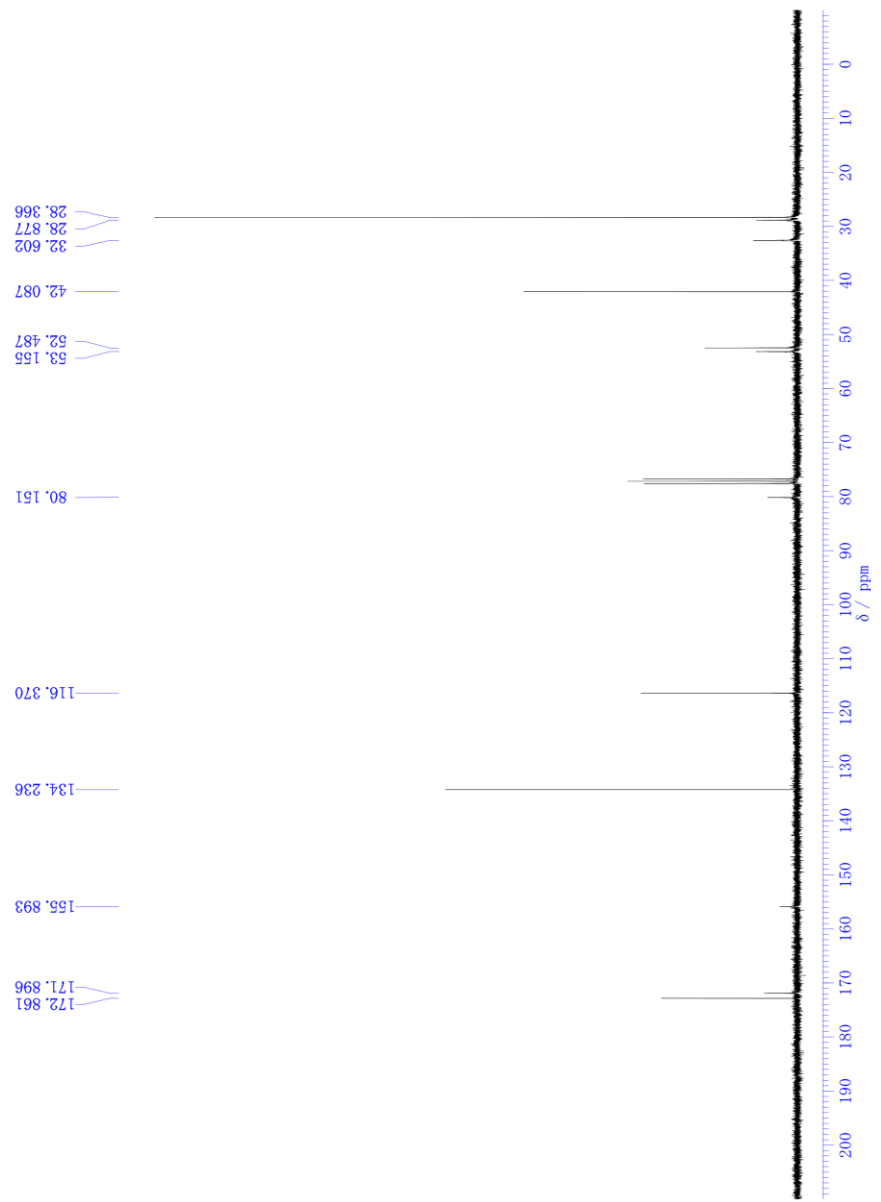


4-Allylcarbamoyl-2-tert-butoxycarbonylamino-butyric acid methyl ester (3)

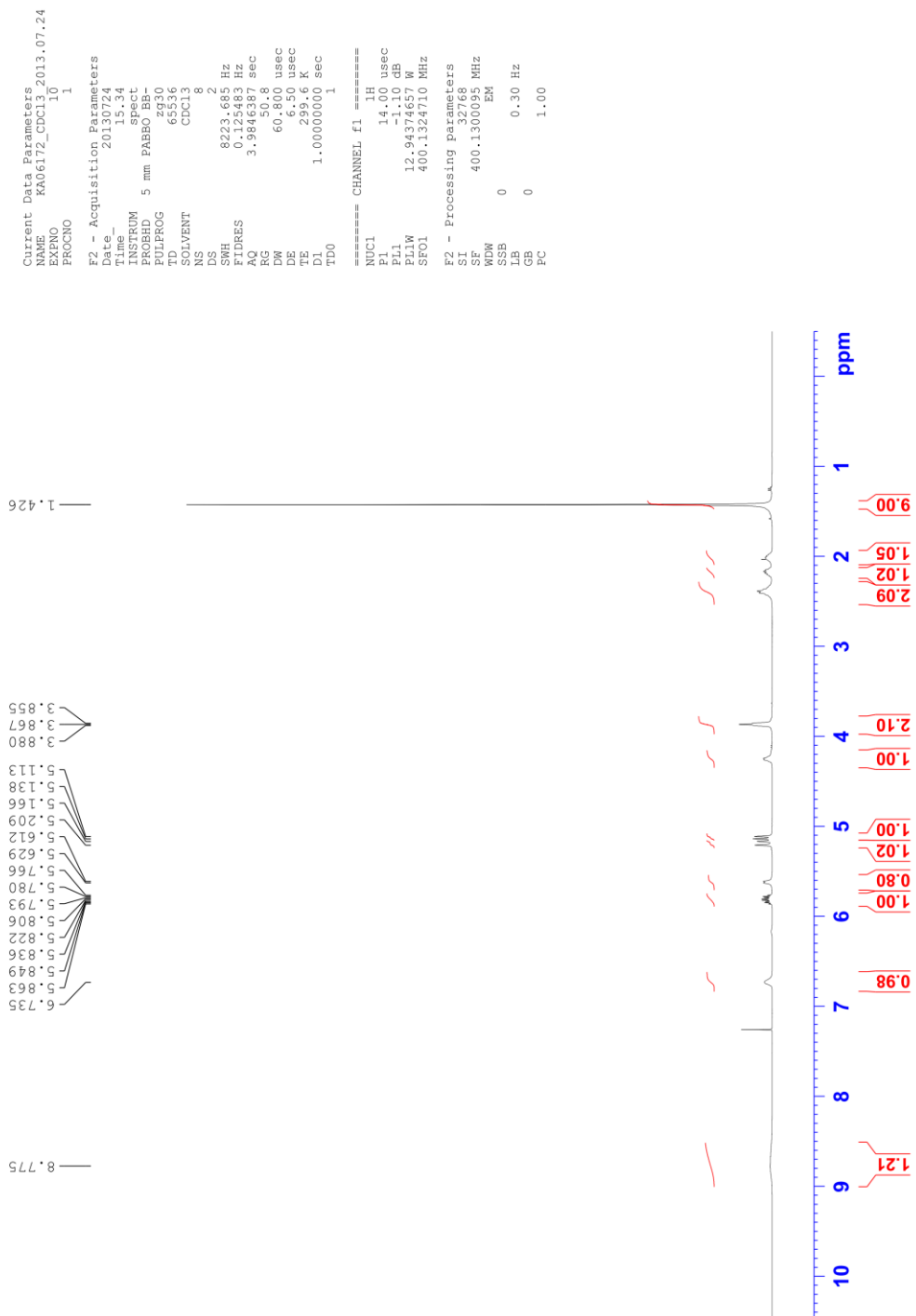


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 PrnMod 1H  
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 Window Exponential  
 RGain 24  
 Operator

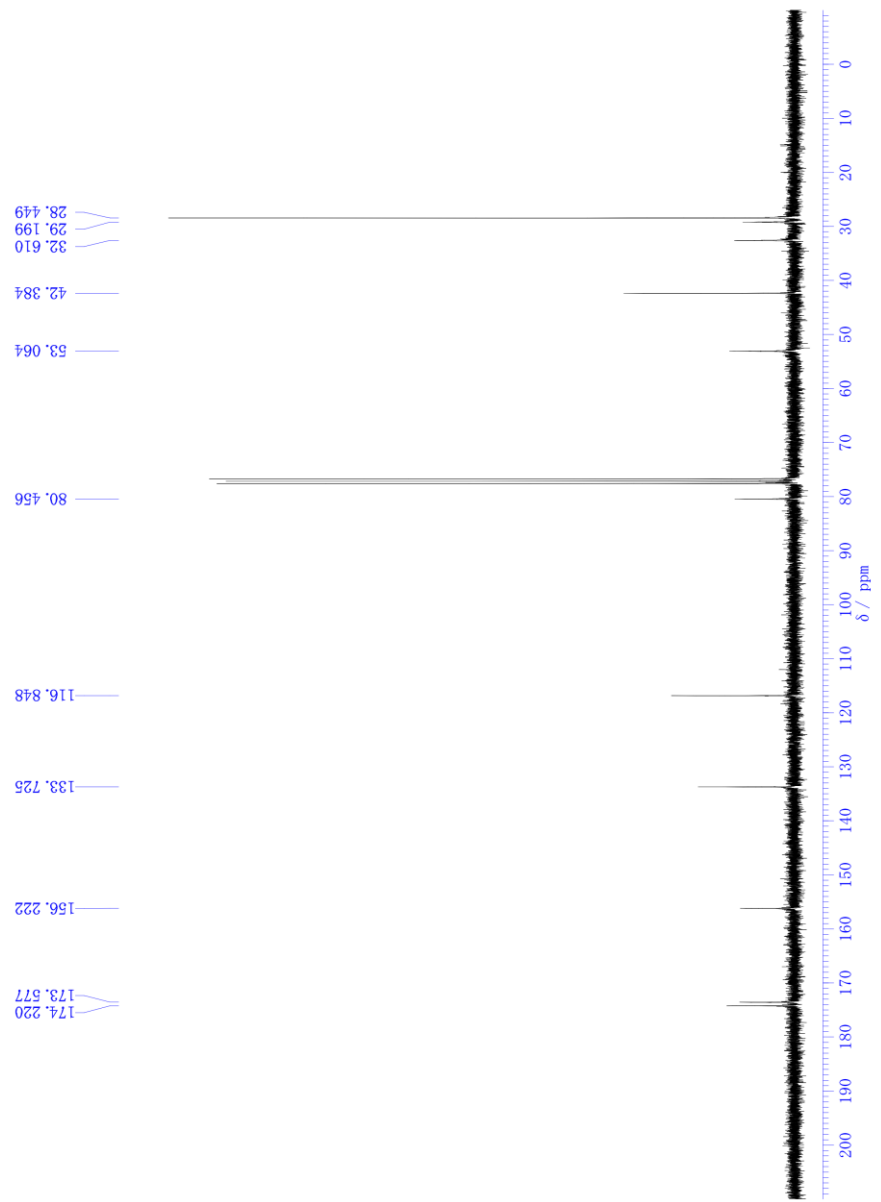


4-Allylcarbamoyl-2-tert-butoxycarbonylamino-butyric acid (4)



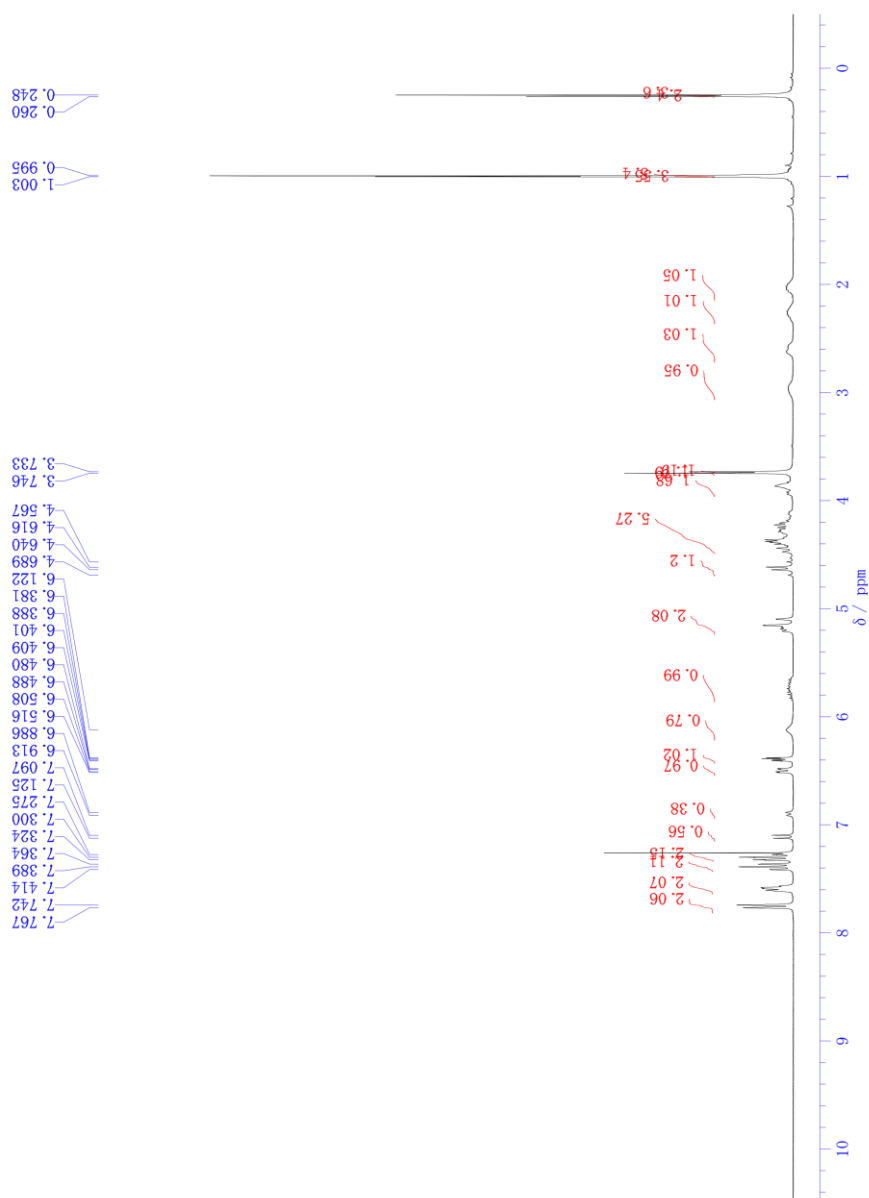
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 PrnMod IH  
 Treatment  
 PulseProgram BCM  
 GradientPRG  
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 Solvent CDCL3  
 Reference 77.16 ppm  
 BF 0.3114 Hz  
 Window Exponential  
 RGain 27  
 Operator



4-{Allyl-[2-(tert-butyl-dimethyl-silanyloxy)-4-methoxy-benzyl]-carbamoyl}-2-(9H-fluorene-  
n-9-ylmethoxycarbonylamino)-butyric acid (7)

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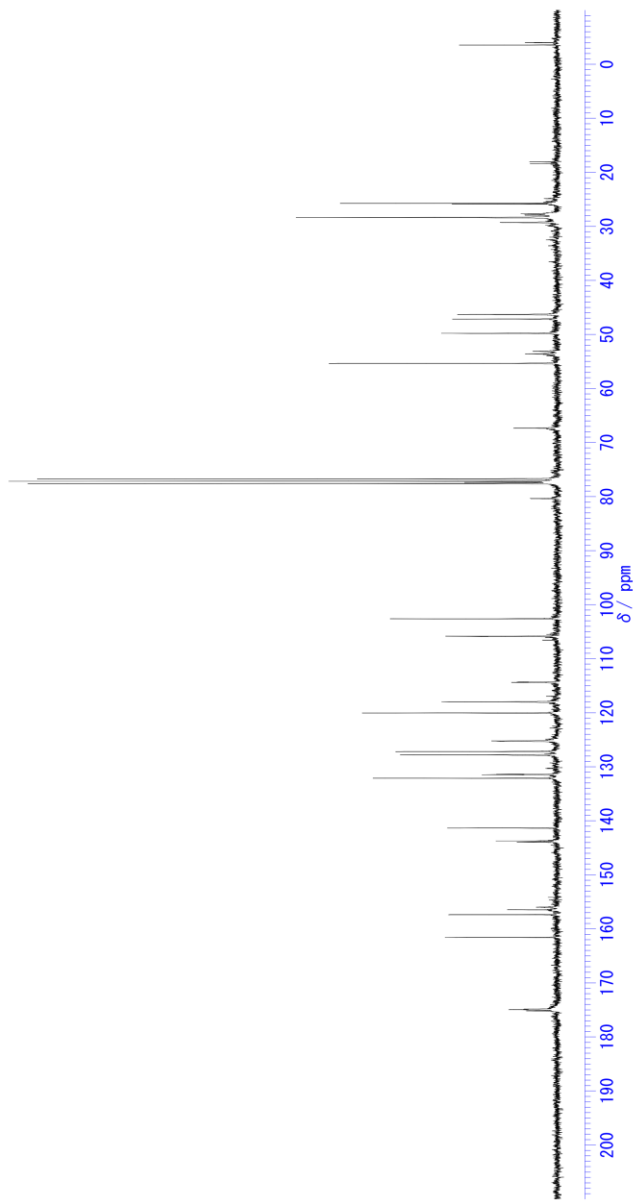




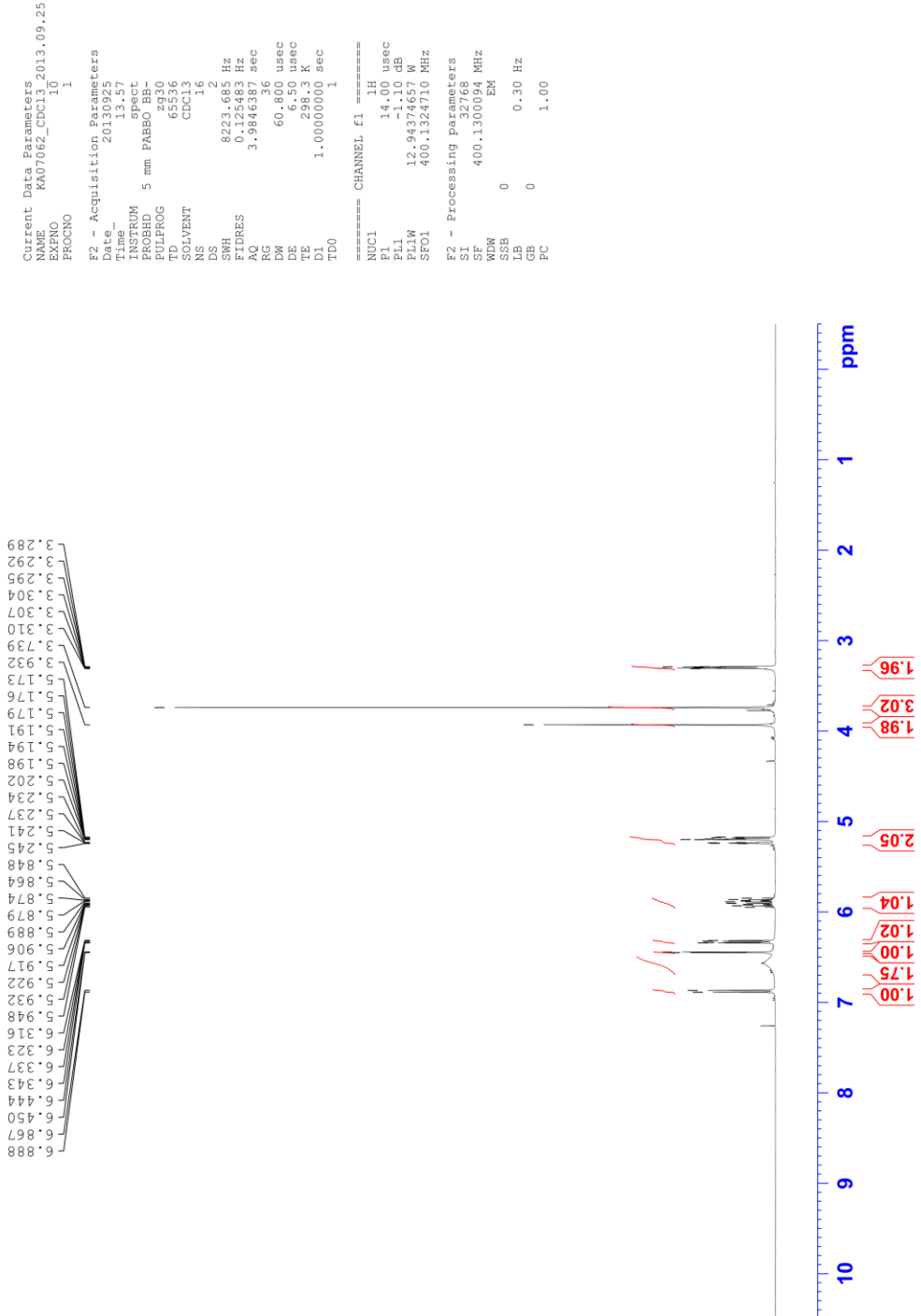
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 Scan 3682  
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 P1 1.394 s  
 Pulse 4.5  $\mu$ s  
 ProbHD 1H  
 Instrument  
 PulseProgram BCM  
 GradientPRG  
 Temp. 30.0  $^{\circ}$ C  
 Solvent CDCL3  
 Reference 77.16 ppm  
 BF 0.3114 Hz  
 Window Exponential  
 RGain 24  
 Operator

- 175.176
- 174.937
- 161.562
- 157.368
- 156.470
- 155.983
- 143.935
- 143.721
- 141.339
- 132.126
- 131.385
- 131.475
- 127.816
- 127.190
- 125.278
- 125.220
- 120.045
- 117.993
- 114.392
- 114.318
- 105.846
- 102.616
- 67.329
- 55.347
- 53.583
- 53.089
- 49.784
- 47.147
- 46.290
- 29.264
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- 3.567
- 4.004
- 4.045

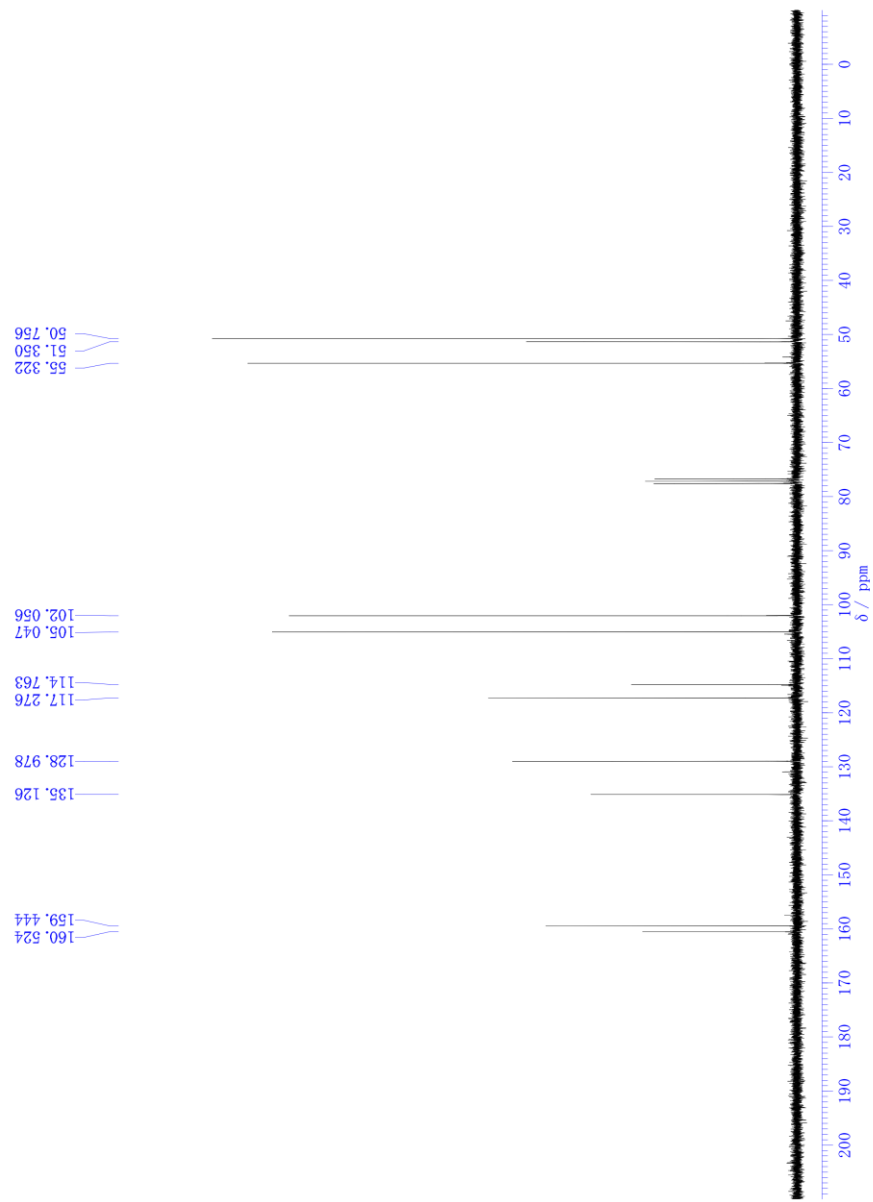


2-Allylaminomethyl-5-methoxy-phenol (9)

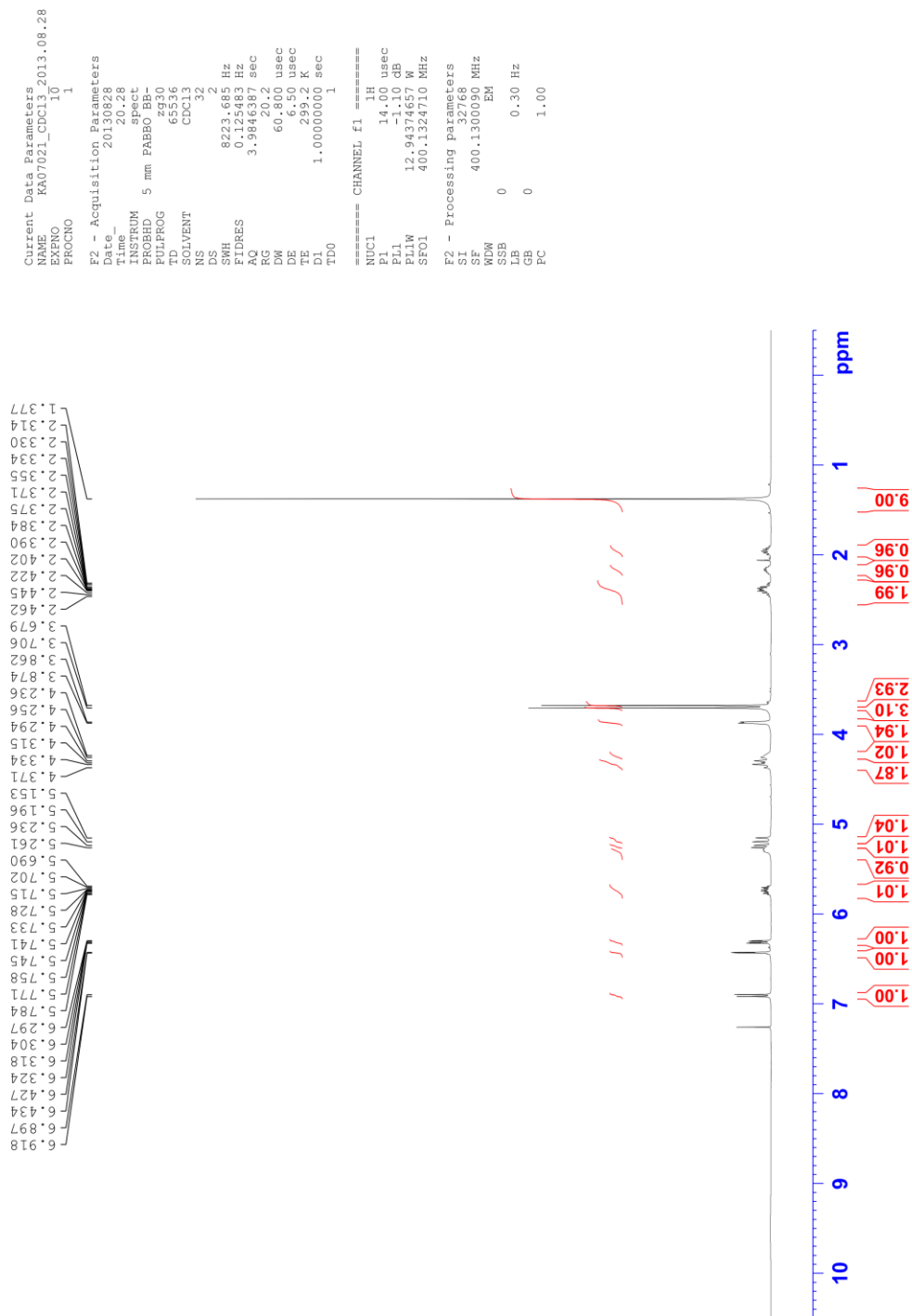


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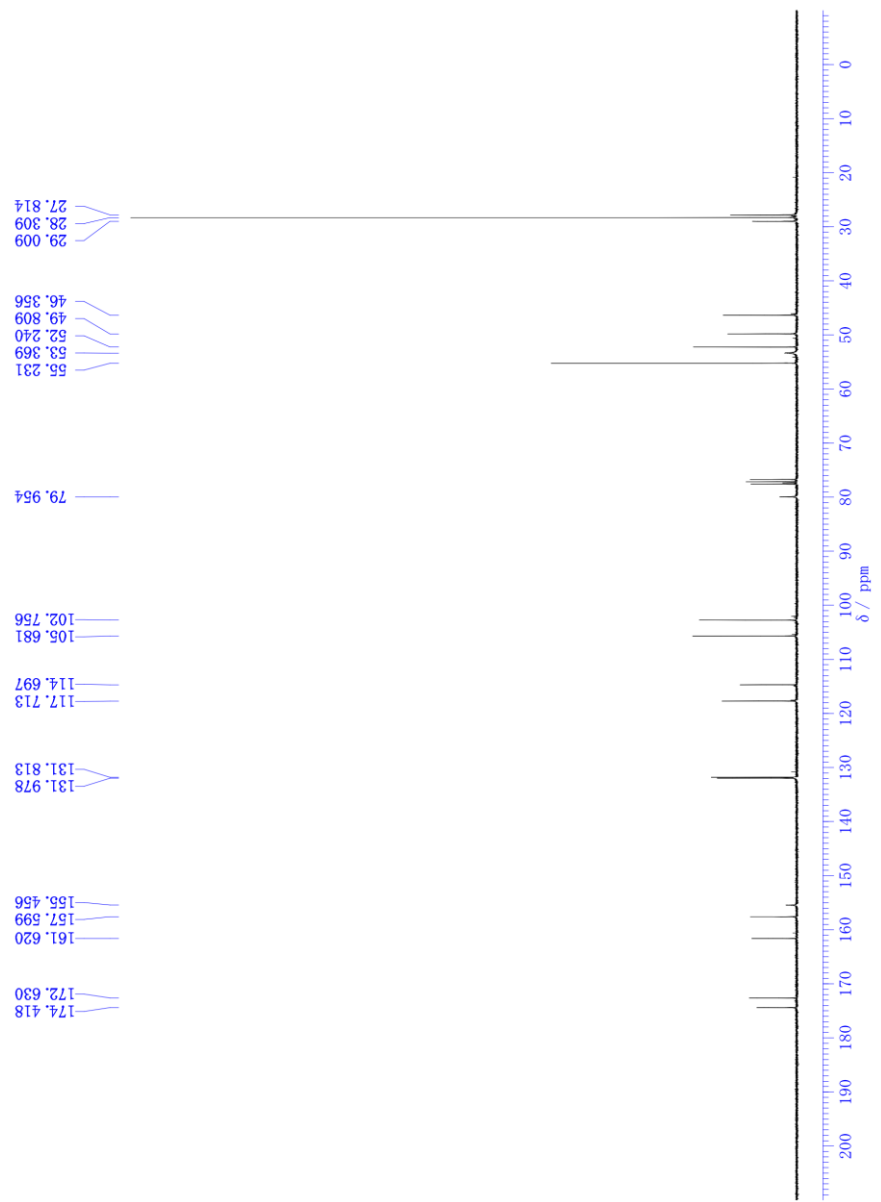
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 Pulse2 1H  
 Program  
 Instrument BCM  
 PulseProgram BCM  
 GradientPRG  
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 BF 0.3114 Hz  
 Window Exponential  
 RGain 24  
 Operator



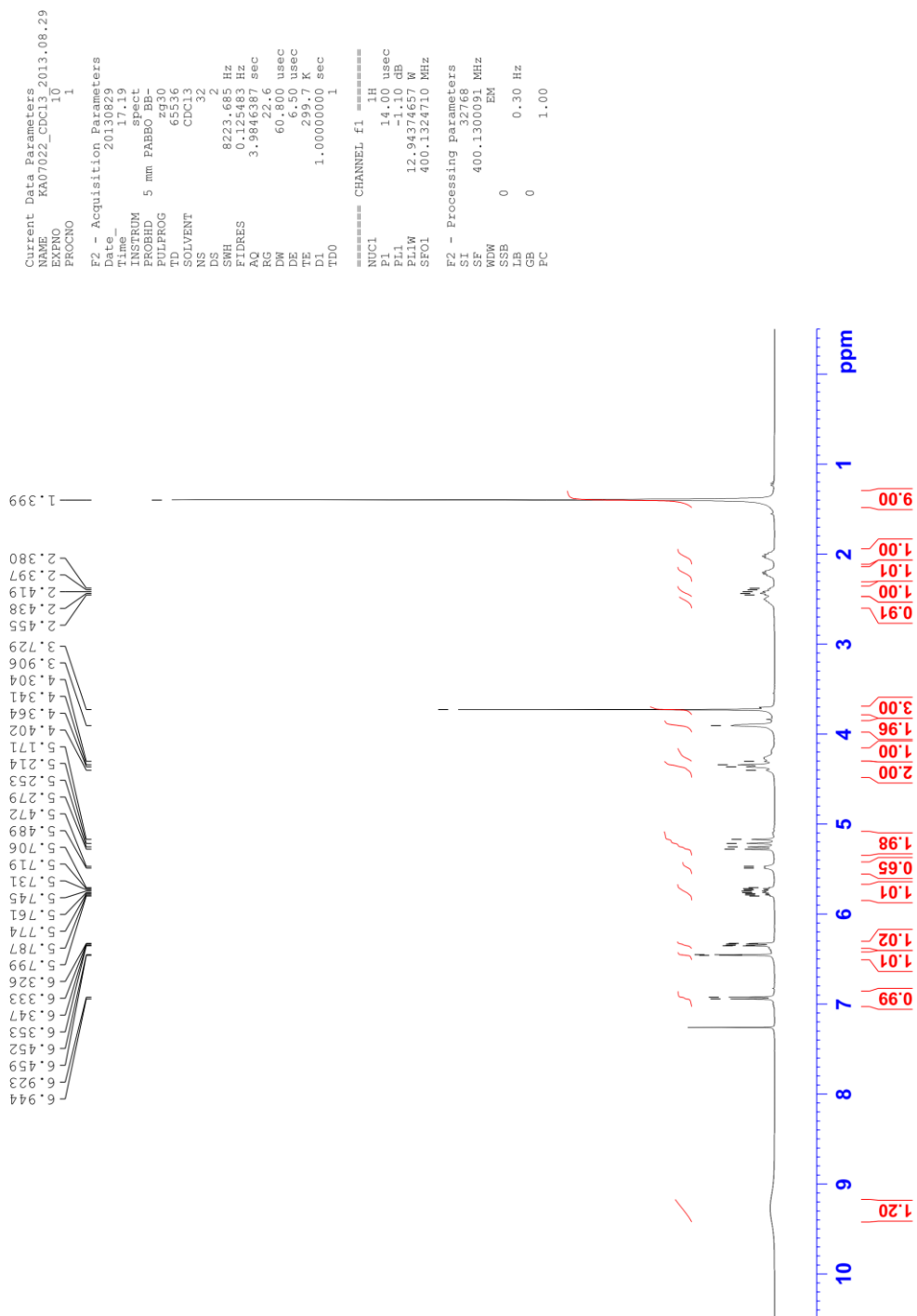
4-[Allyl-(2-hydroxy-4-methoxy-benzyl)-carbamoyl]-2-tert-butoxycarbonylamino-butyric acid methyl ester (10)



File YFLANDISK-AOYNRYAIHARAYUURONY  
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 Comment  
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 PrnMod 1H  
 Program  
 Instrument BCM  
 PulseProgram BCM  
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 Window Exponential  
 RGain 24  
 Operator

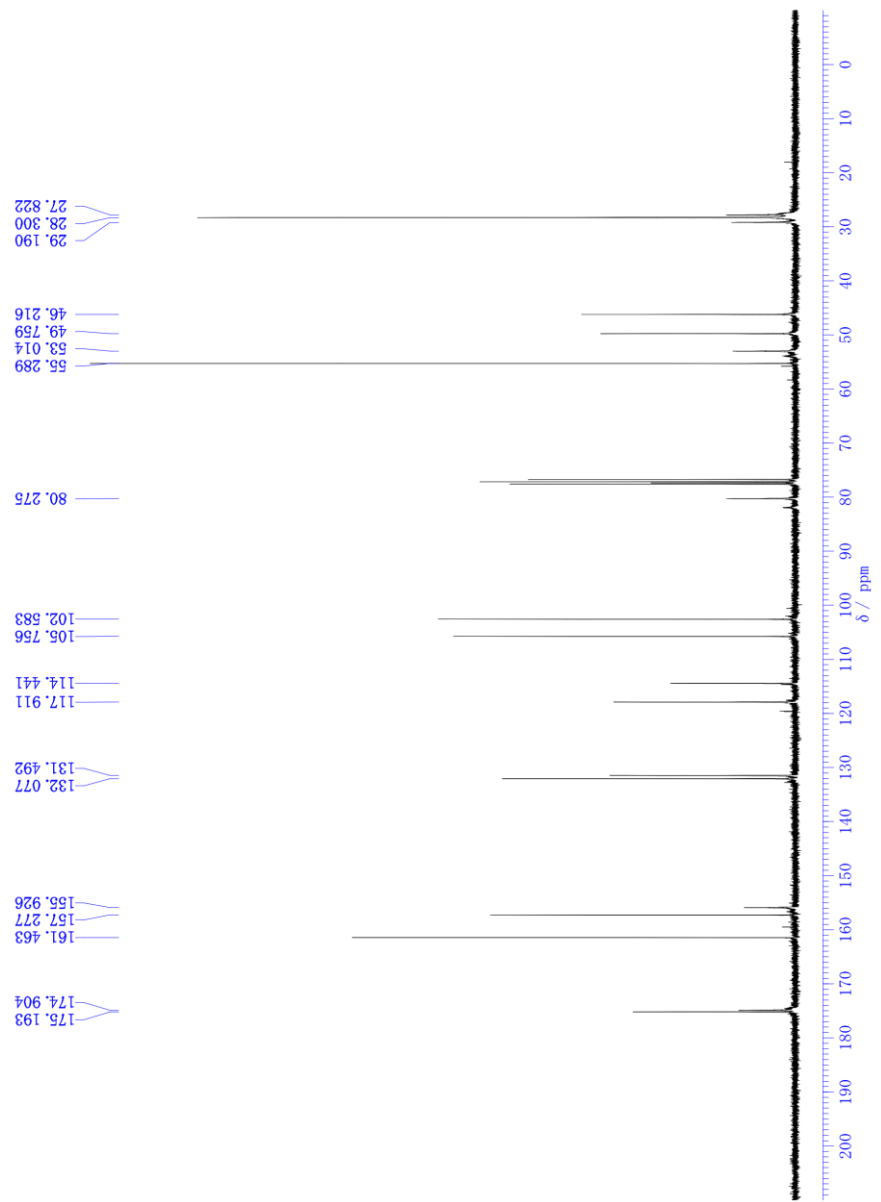


4-[Allyl-(2-hydroxy-4-methoxy-benzyl)-carbamoyl]-2-tert-butoxycarbonylamino-butyric acid (**11**)



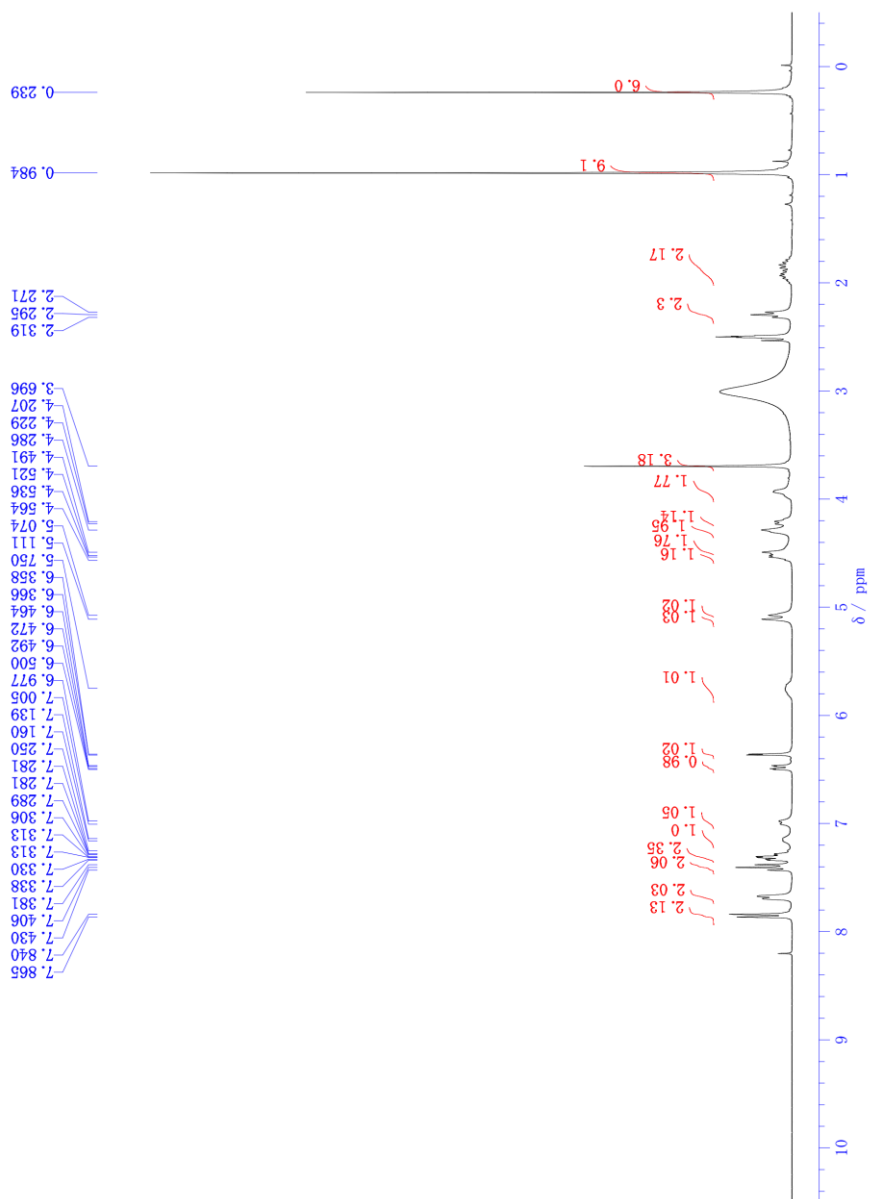
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 Comment Thu Aug 29 19:15:38 2013

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 ObsFine 1840.0 Hz  
 Point 32768  
 Frequency 20408.1 Hz  
 Scan 2000  
 AcqTime 1.6056 s  
 PD 1.39 s  
 Pulse1 3.0  $\mu$ s  
 Pulse2 1H  
 Program  
 Treatment BCM  
 PulseProgram BCM  
 GradientPRG  
 Temp 50.0  $^{\circ}$ C  
 Solvent CDCL3  
 Reference 77.16 ppm  
 BF 0.3114 Hz  
 Window Exponential  
 RGain 24  
 Operator



4-{Allyl-[2-(tert-butyl-dimethyl-silanyloxy)-4-methoxy-benzyl]-carbamoyl}-4-(9H-fluoren-9-ylmethoxycarbonylamino)-butyric acid (**12**)

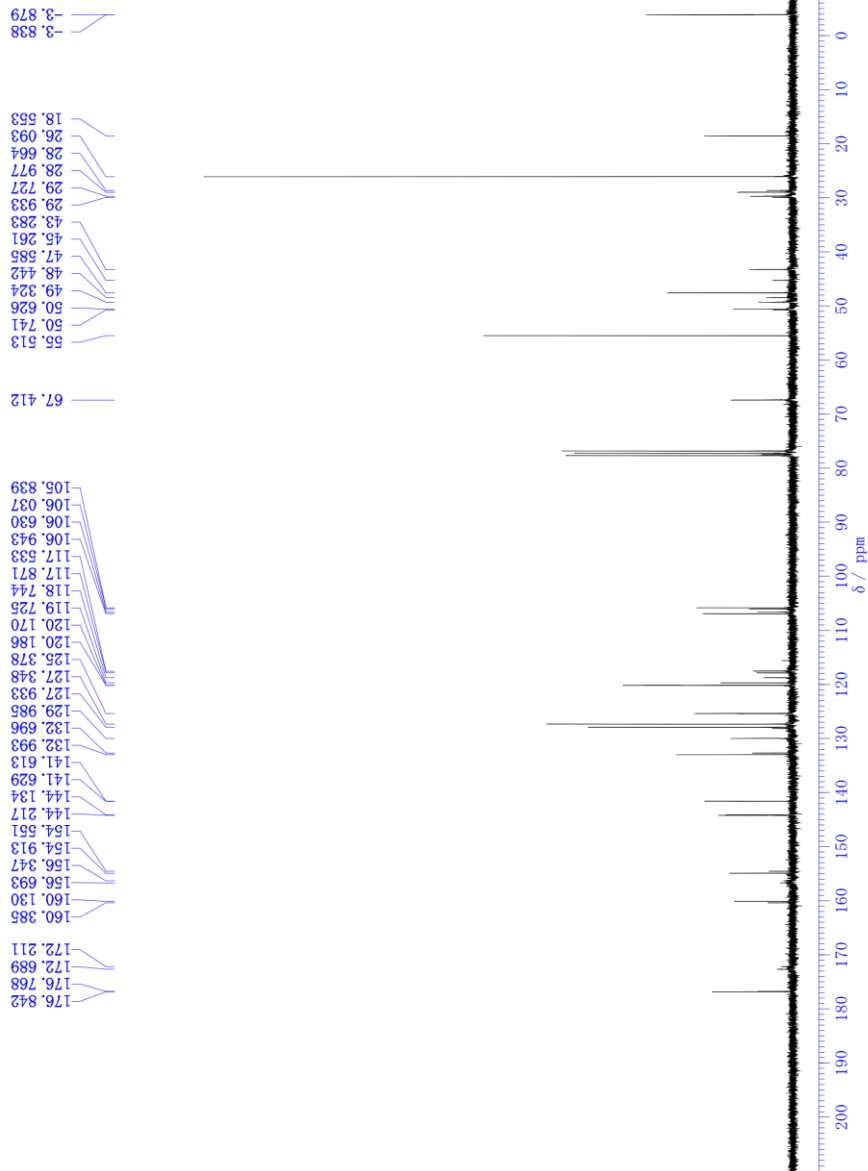
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 ObsSet 130.0 kHz  
 ObsFlne 130.0 Hz  
 Point 32708  
 Frequency 6006.0 Hz  
 Scan 64  
 AcqTime 3.4559 s  
 P1 1.544 s  
 Pulse 3.0  $\mu$ s  
 Prb 1H  
 Instrument  
 PulseProgram NON  
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 Solvent DMSO  
 Reference 2.5 ppm  
 BF 0.0916 Hz  
 Window Exponential  
 RGain 18  
 Operator



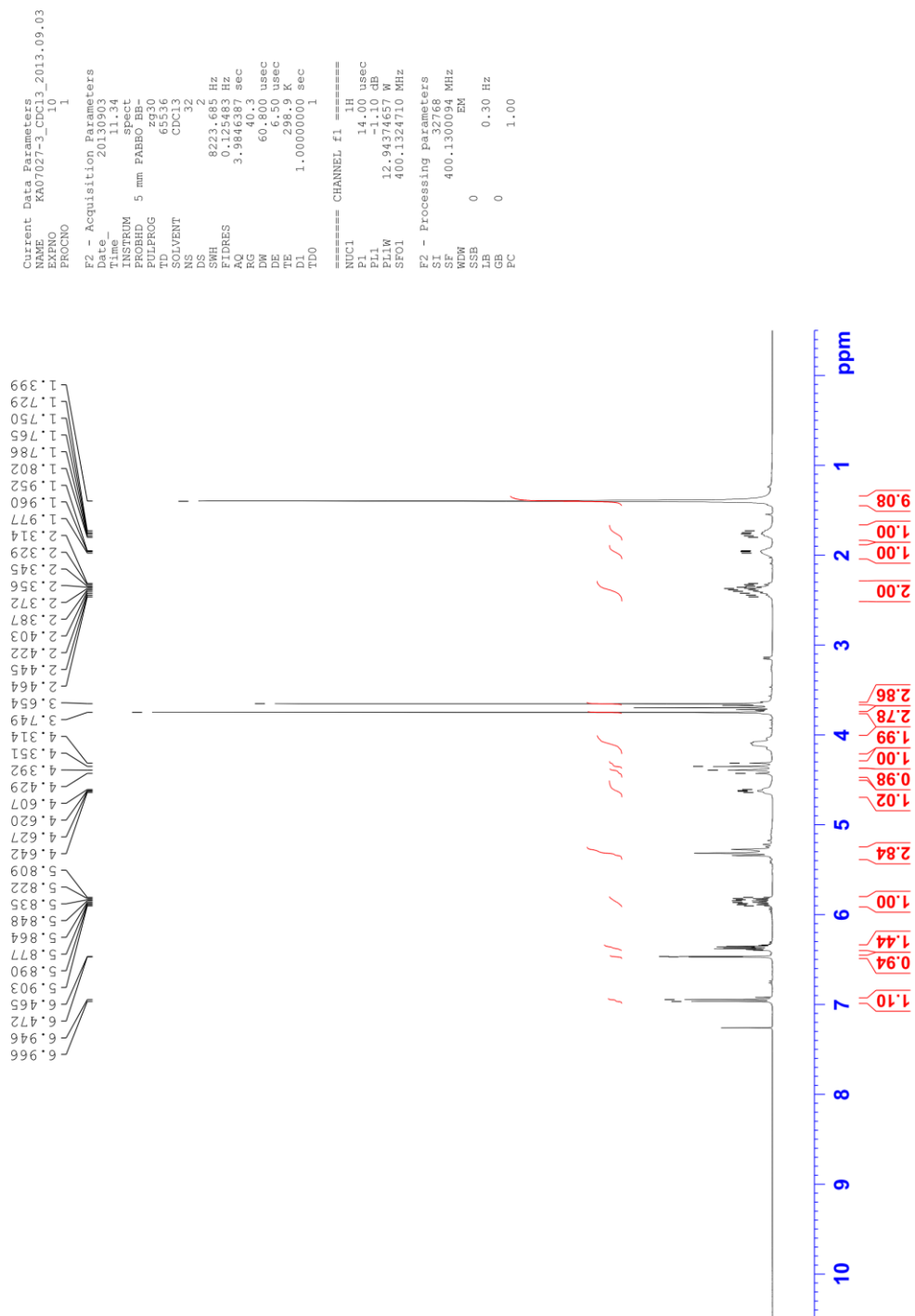


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 Thu Sep 05 22:51:00 2013

Date  
 Comment  
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 ObsFlne 1840.0 Hz  
 Point 32768  
 Frequency 20408.1 Hz  
 Scan 2000  
 AcqTime 1.6056 s  
 Pulse 1.39 s  
 IrrNuc 5.0  $\mu$ s  
 ProbeHD IH  
 Instrument  
 PulseProgram BCM  
 GradientPRG  
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 BF 0.3114 Hz  
 Window Exponential  
 RGain 24  
 Operator

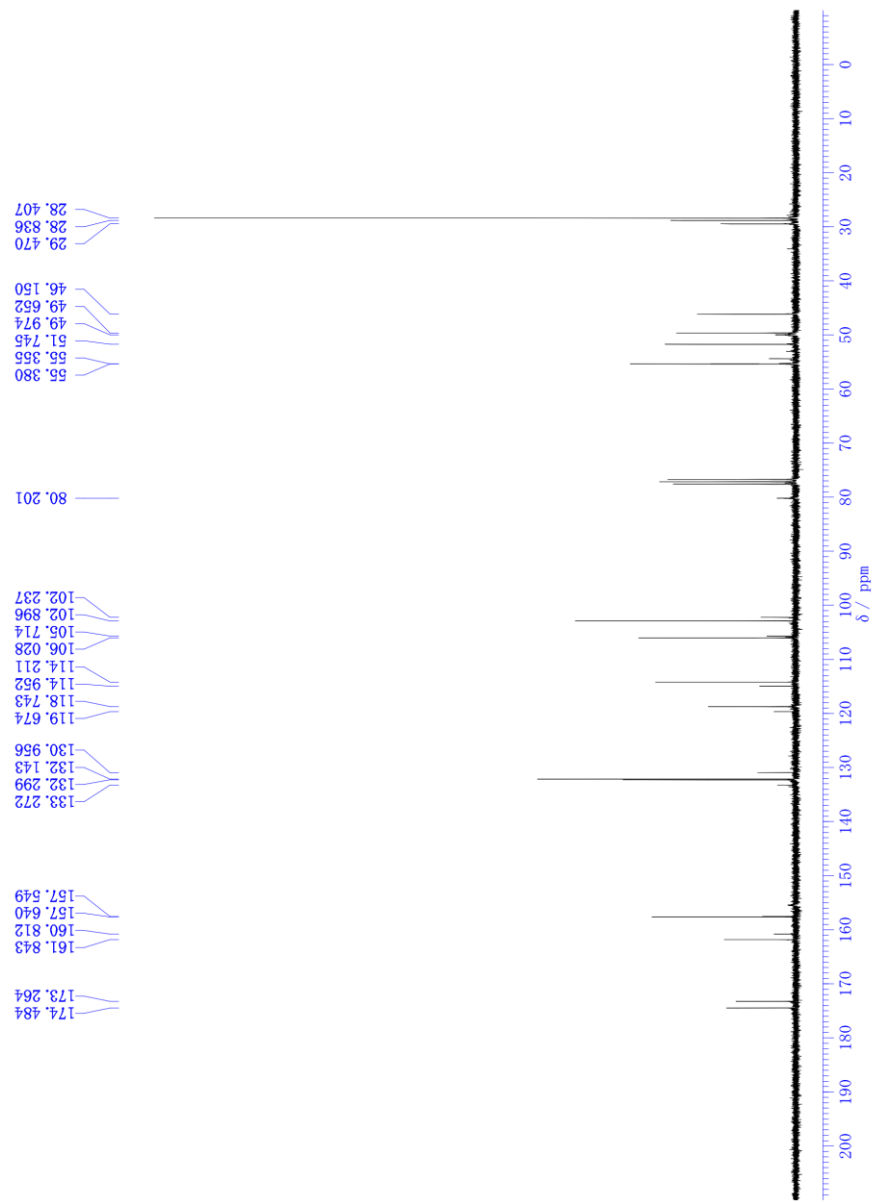


4-[Allyl-(2-hydroxy-4-methoxy-benzyl)-carbamoyl]-4-tert-butoxycarbonylamino-butyric acid methyl ester (**19**)

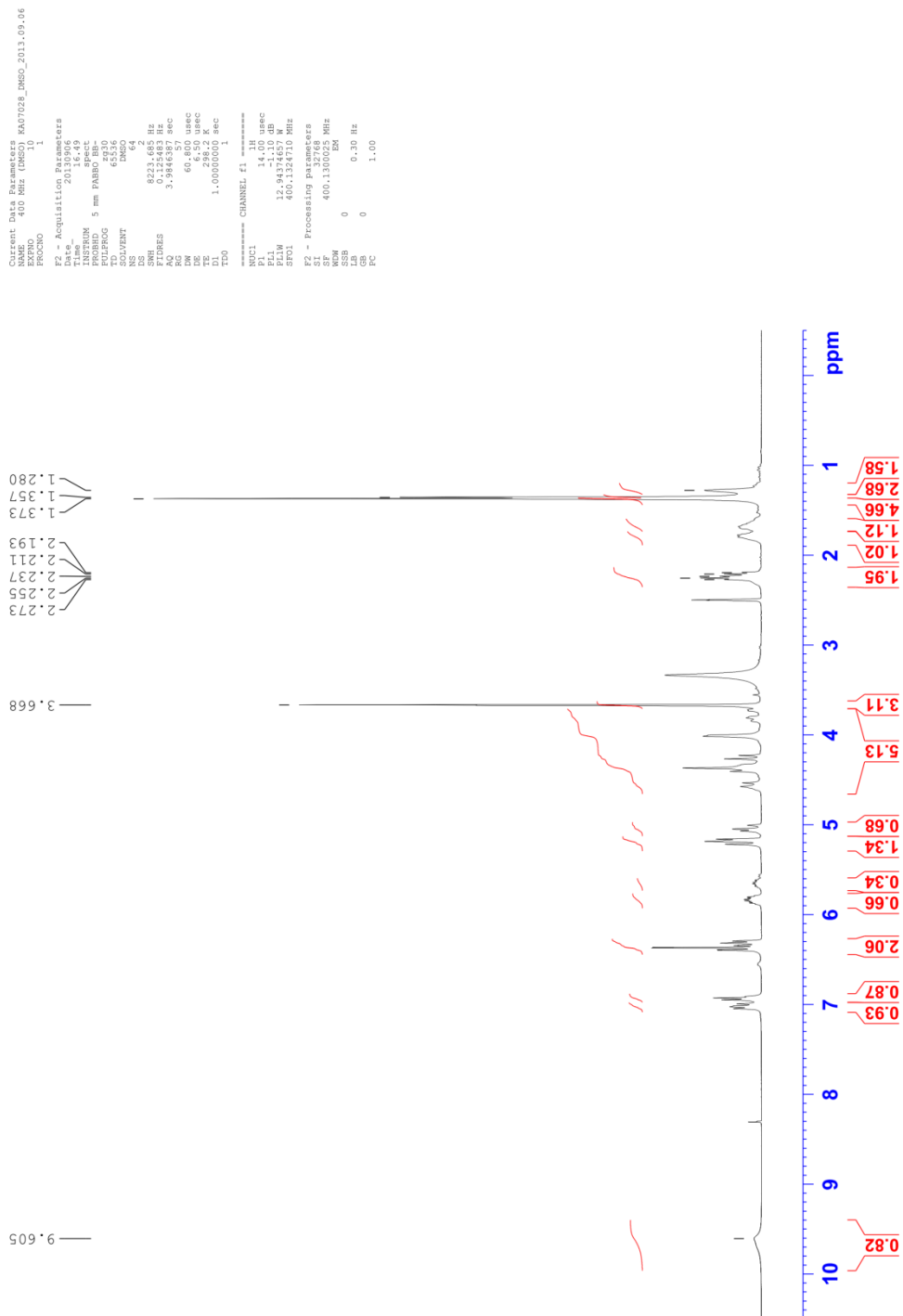


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 Scan 1000  
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 Instrument  
 PulseProgram BCM  
 GradientPRG  
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 Solvent CDCl3  
 Reference 77.16 ppm  
 BF 0.3114 Hz  
 Window Exponential  
 RGain 24  
 Operator



4-[Allyl-(2-hydroxy-4-methoxy-benzyl)-carbamoyl]-4-tert-butoxycarbonylamino-butyric acid (20)



File YFLANDISK-AOVNRYAIHARAYUURONY  
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 Fri Sep 06 23:25:25 2013

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 Scan 2000  
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 ProbMfD 1H  
 Instrument  
 PulseProgram BCM  
 GradientPRG  
 Temp. 100.0  $^{\circ}$ C  
 Solvent DMSO  
 Reference 39.52 ppm  
 BF 0.3114 Hz  
 Window Exponential  
 RGain 24  
 Operator

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 159.506  
 156.276  
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 117.140  
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 115.311  
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 101.202  
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 49.631  
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 46.516  
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 42.973  
 29.895  
 29.771  
 29.712  
 28.172  
 27.876  
 27.076  
 26.821

