

Ligations of O-acyl threonine units to give native peptides *via* 5-, 8-, 9- and 10-membered cyclic transition states

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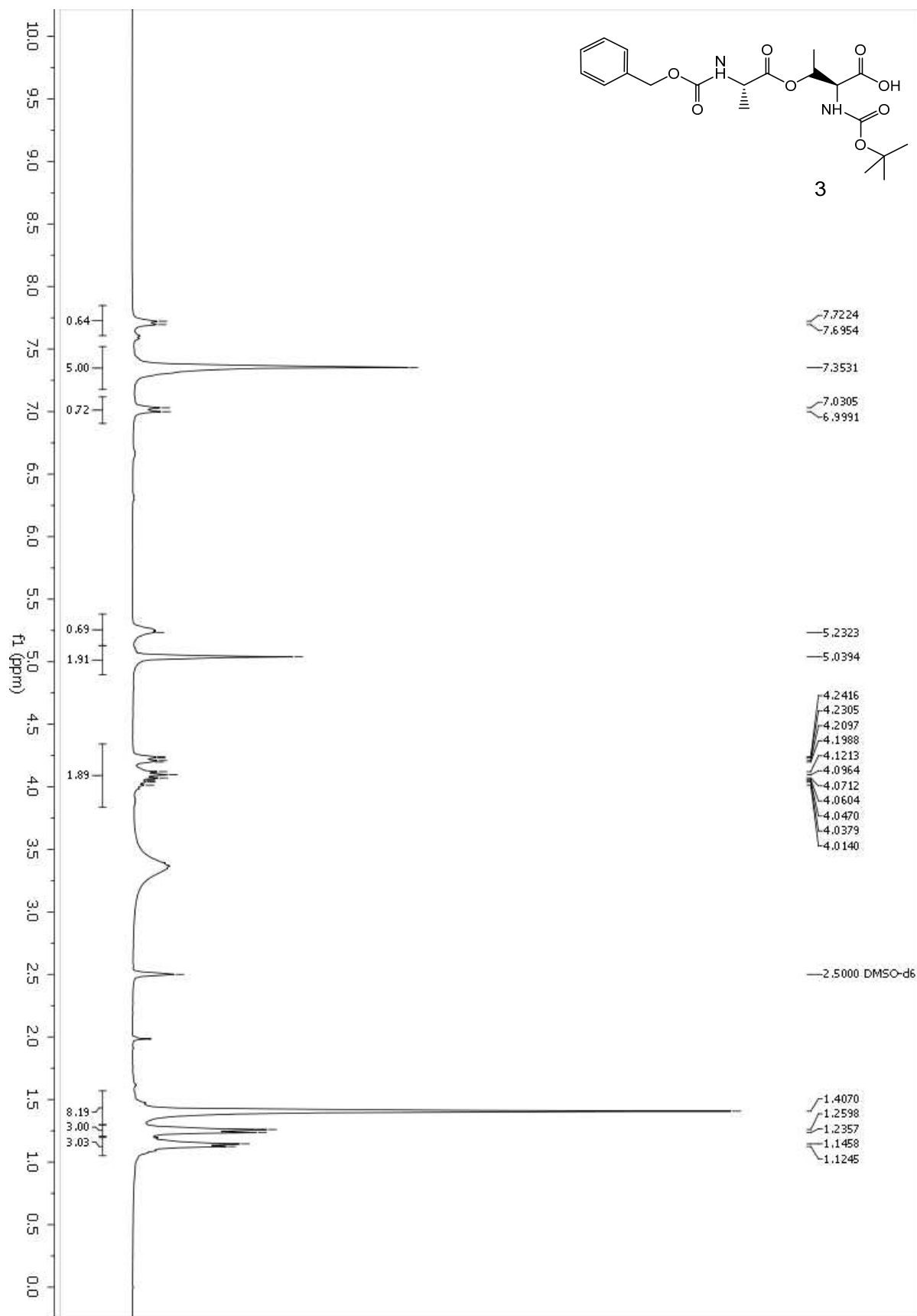
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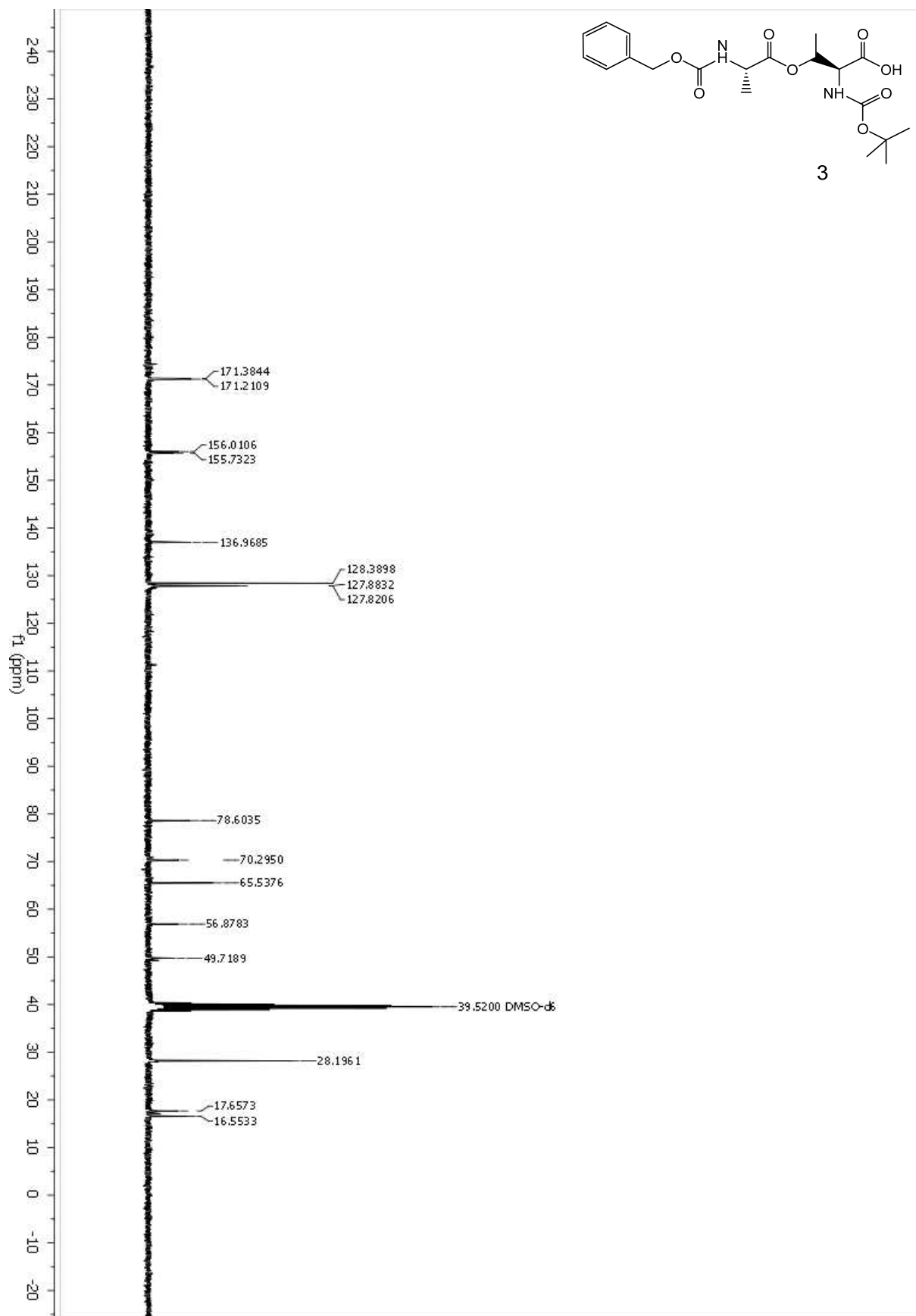
E-mail: sspanda12@gmail.com, charlesdennishall@gmail.com

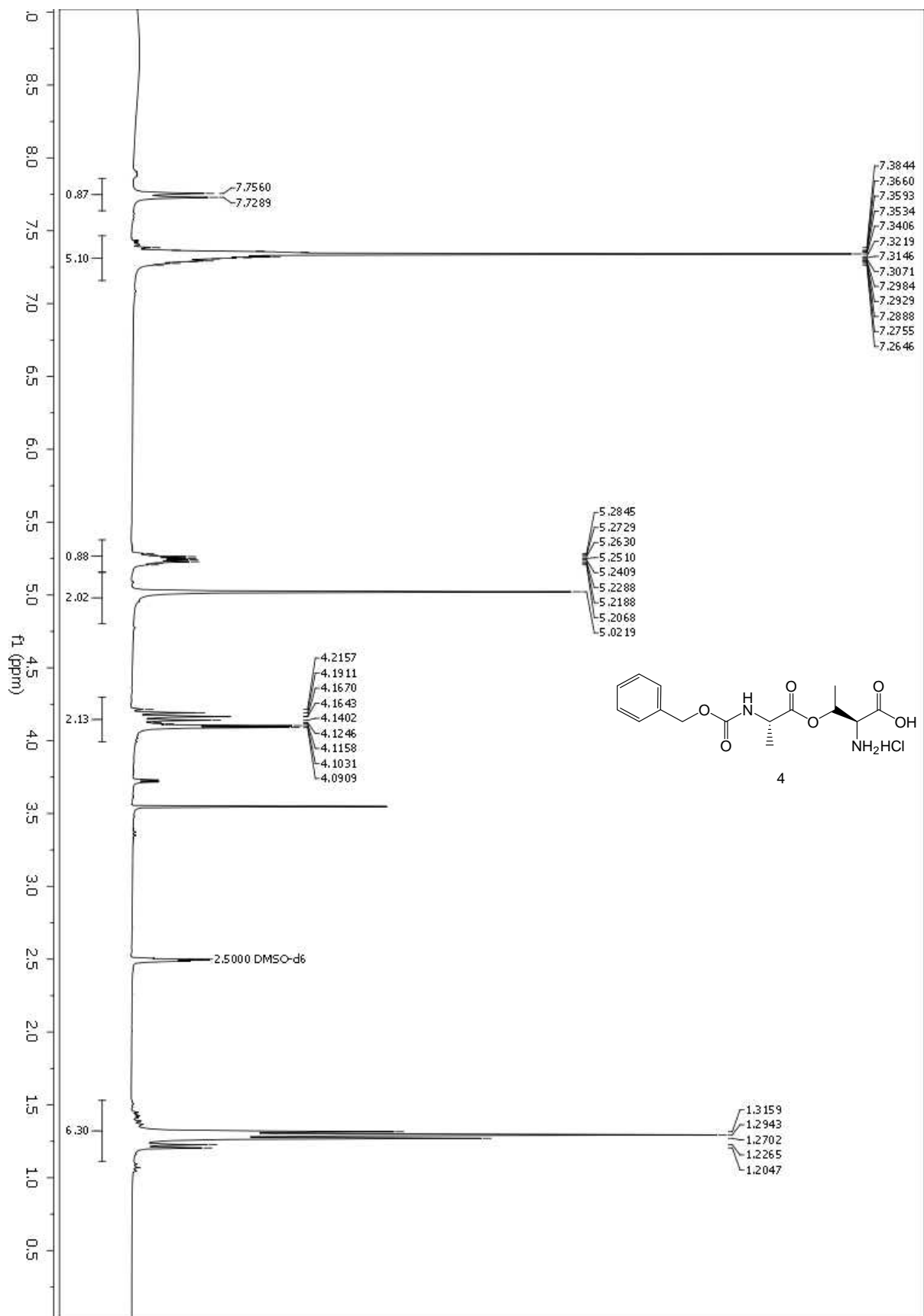
[†]*Deceased on February 10, 2014*

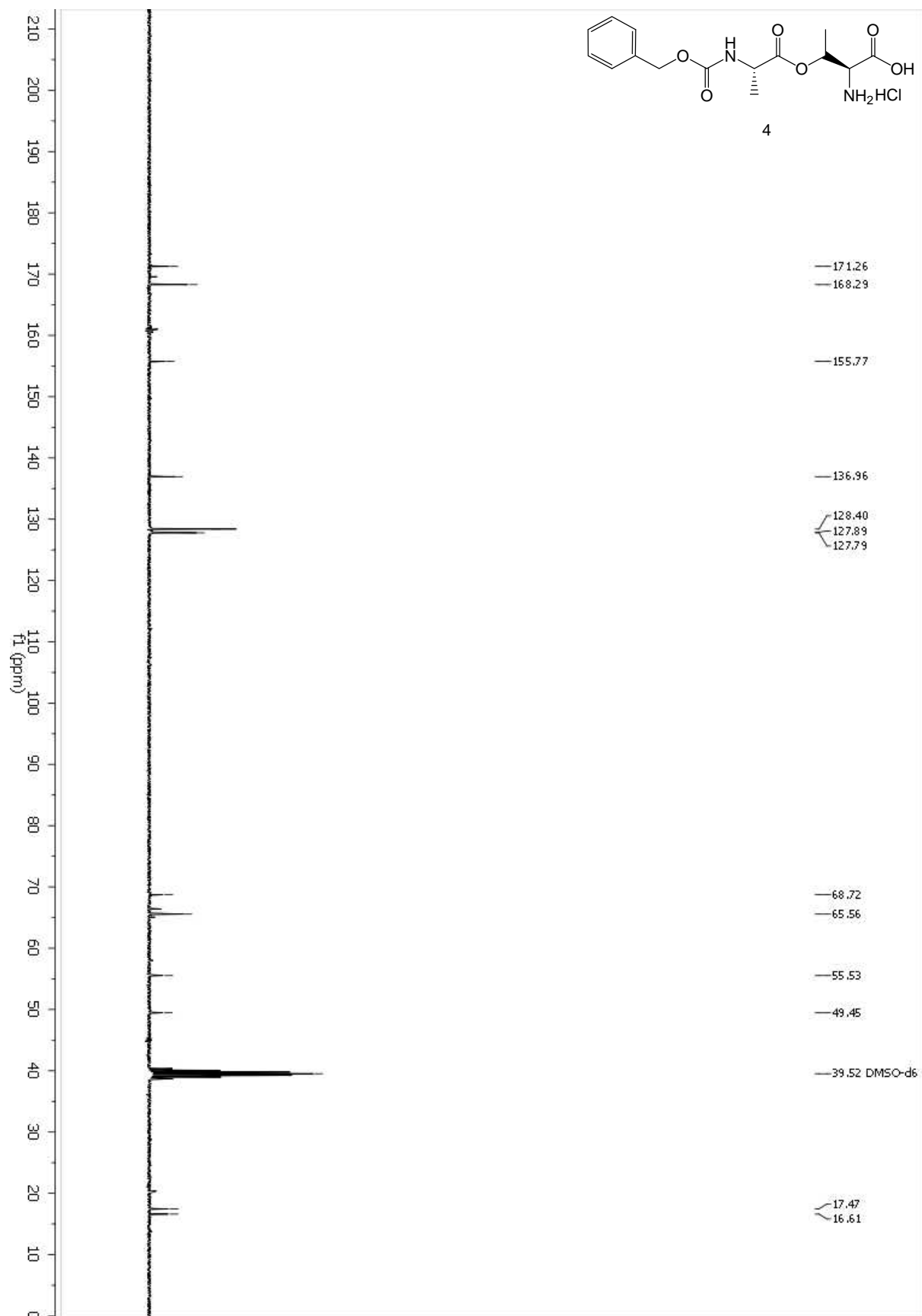
SUPPORTING INFORMATION

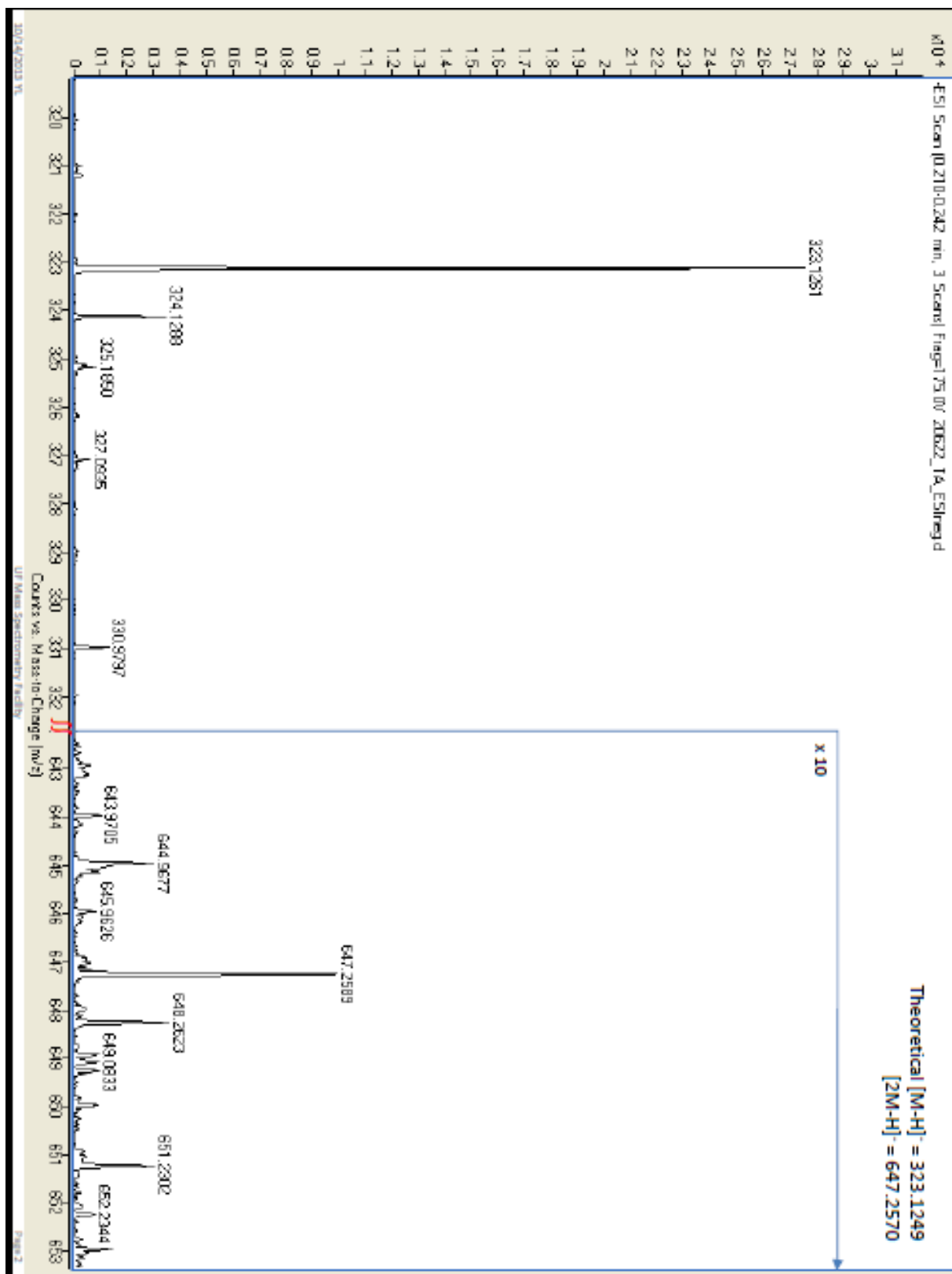
- | | |
|---|-----|
| 1. Copy of ¹ H, ¹³ C NMR and CHN/HRMS spectra for compounds 3 , 4 , 8a-c , and 9a-c | S2 |
| 2. HPLC-MS spectra for compounds 4-6 , 9b , 10b and 11b | S25 |

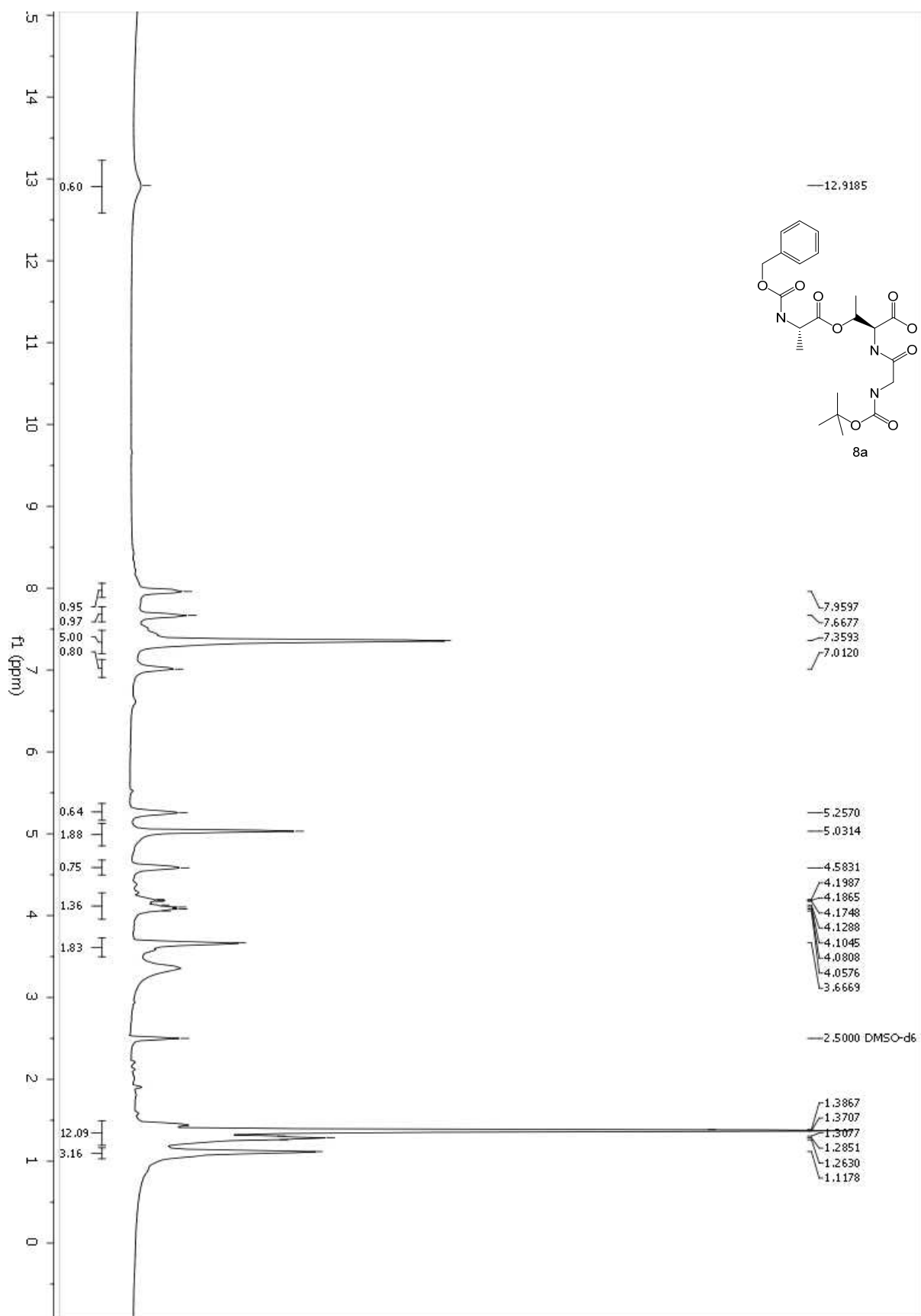


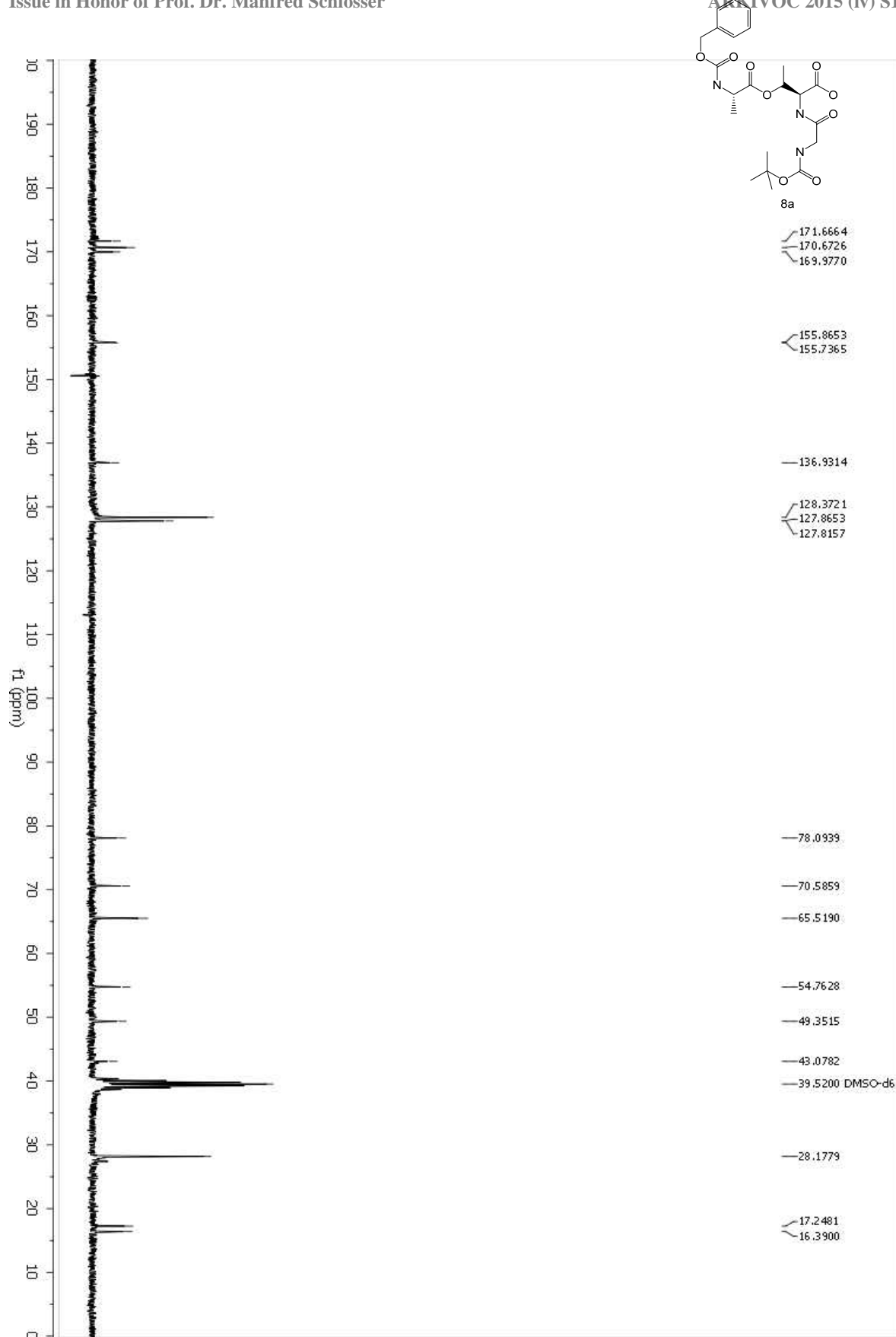


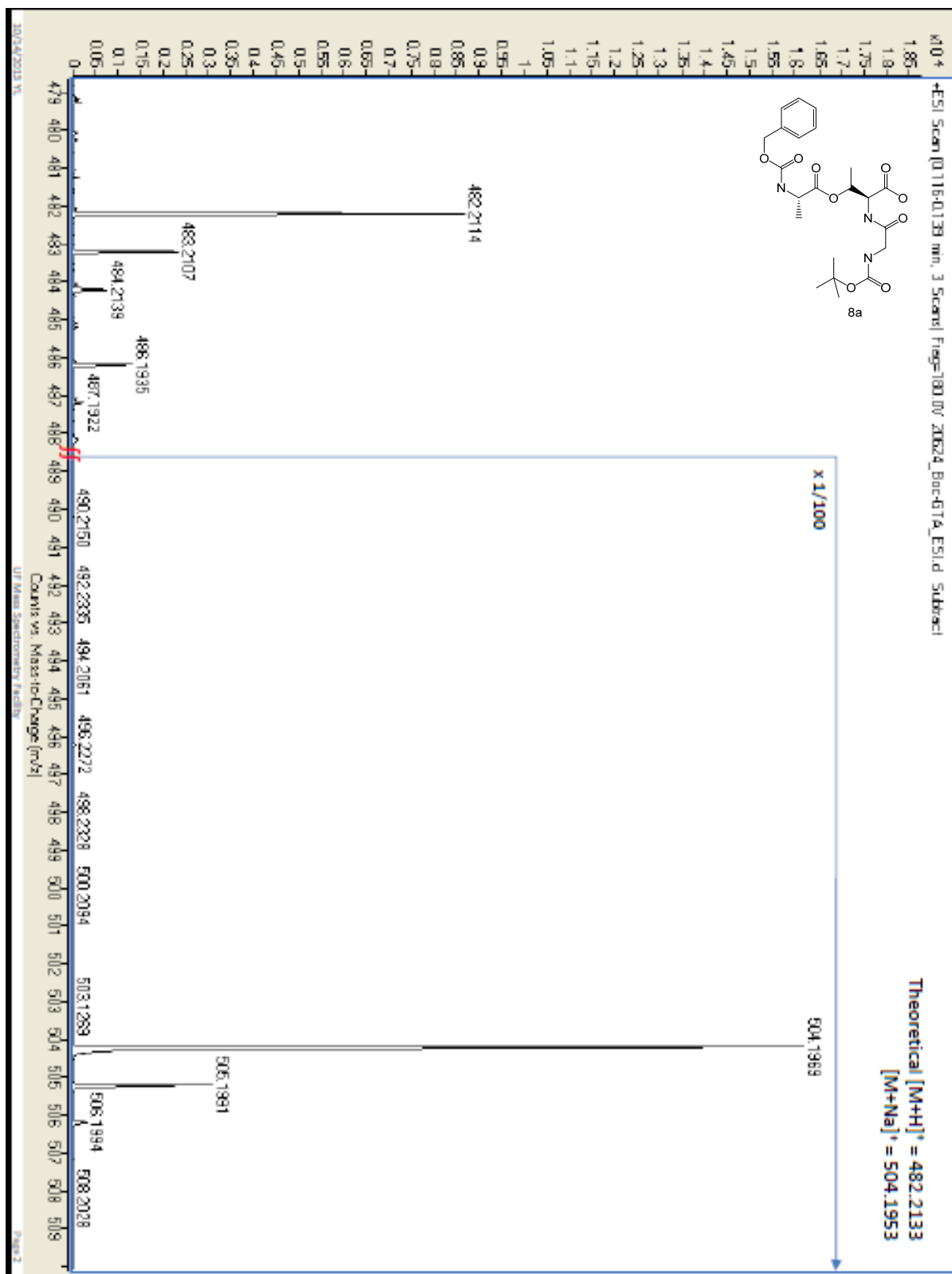


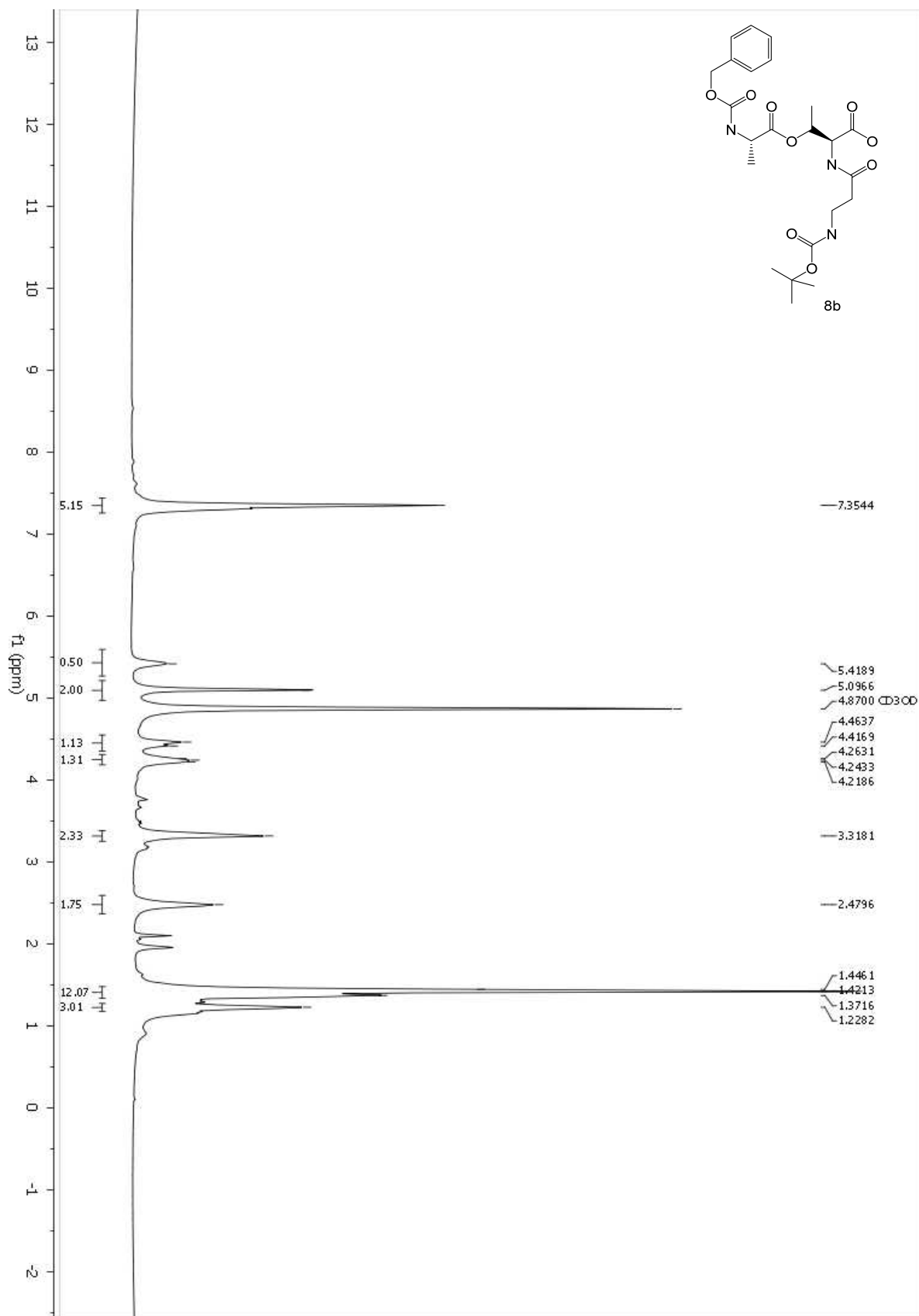


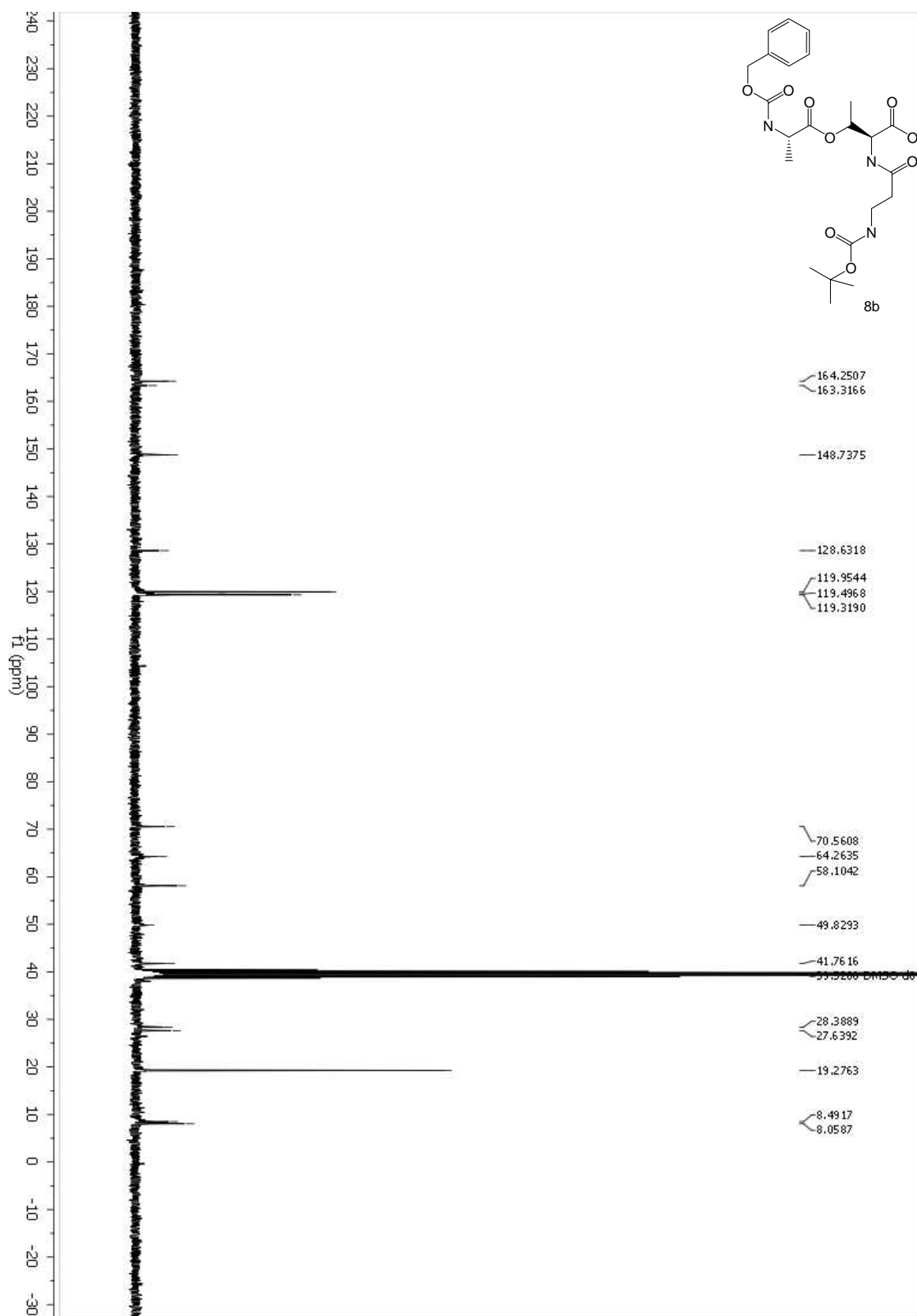


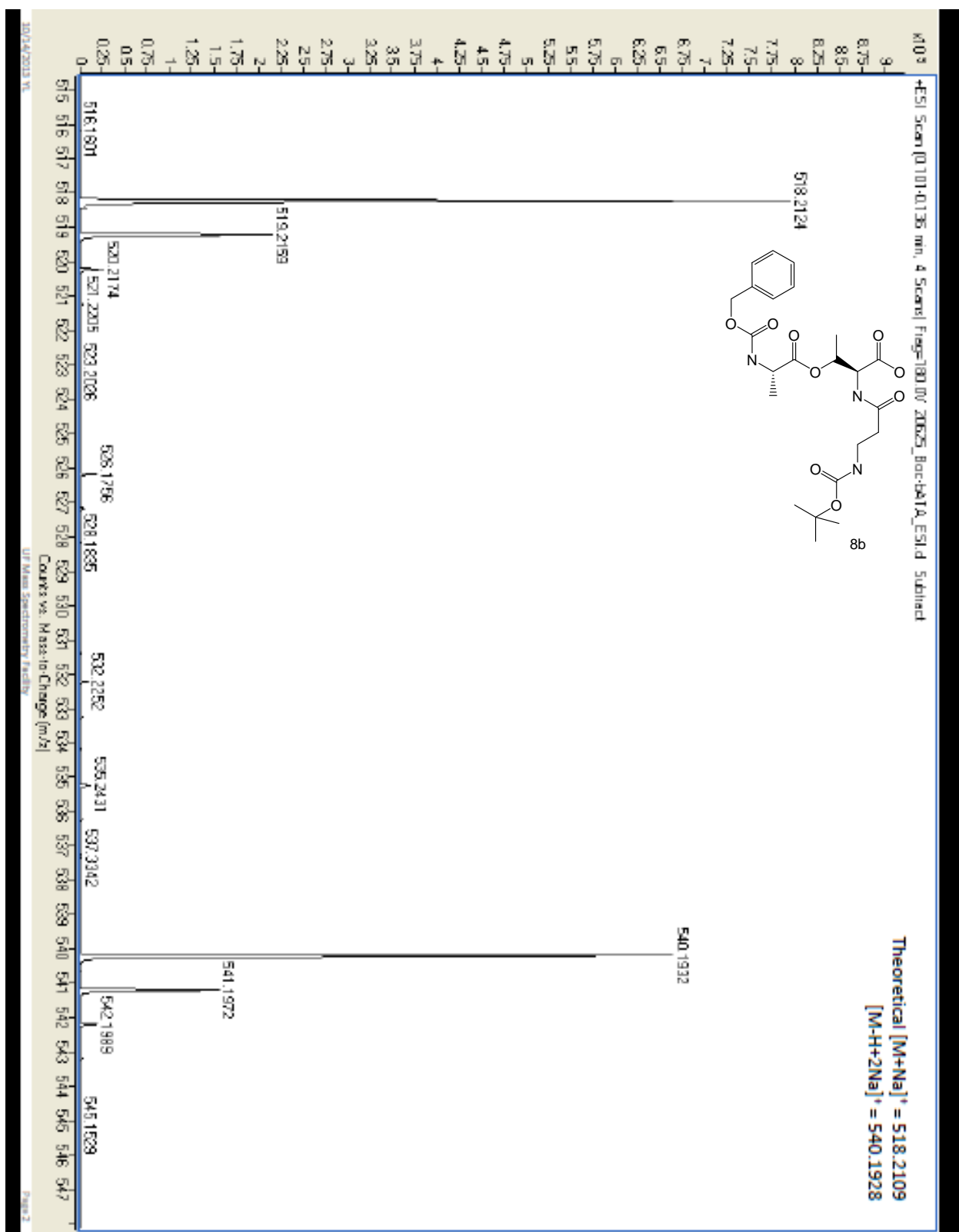


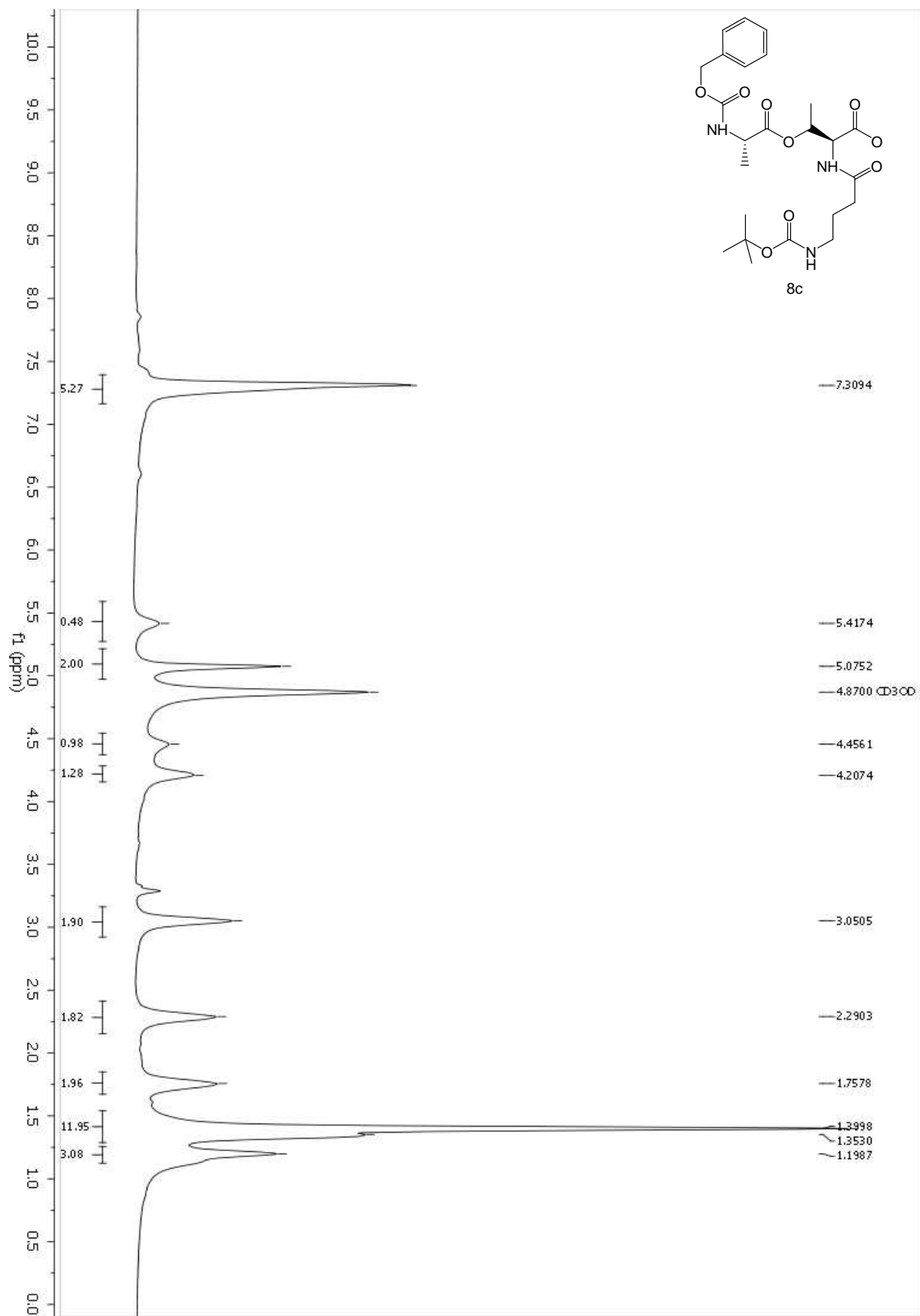


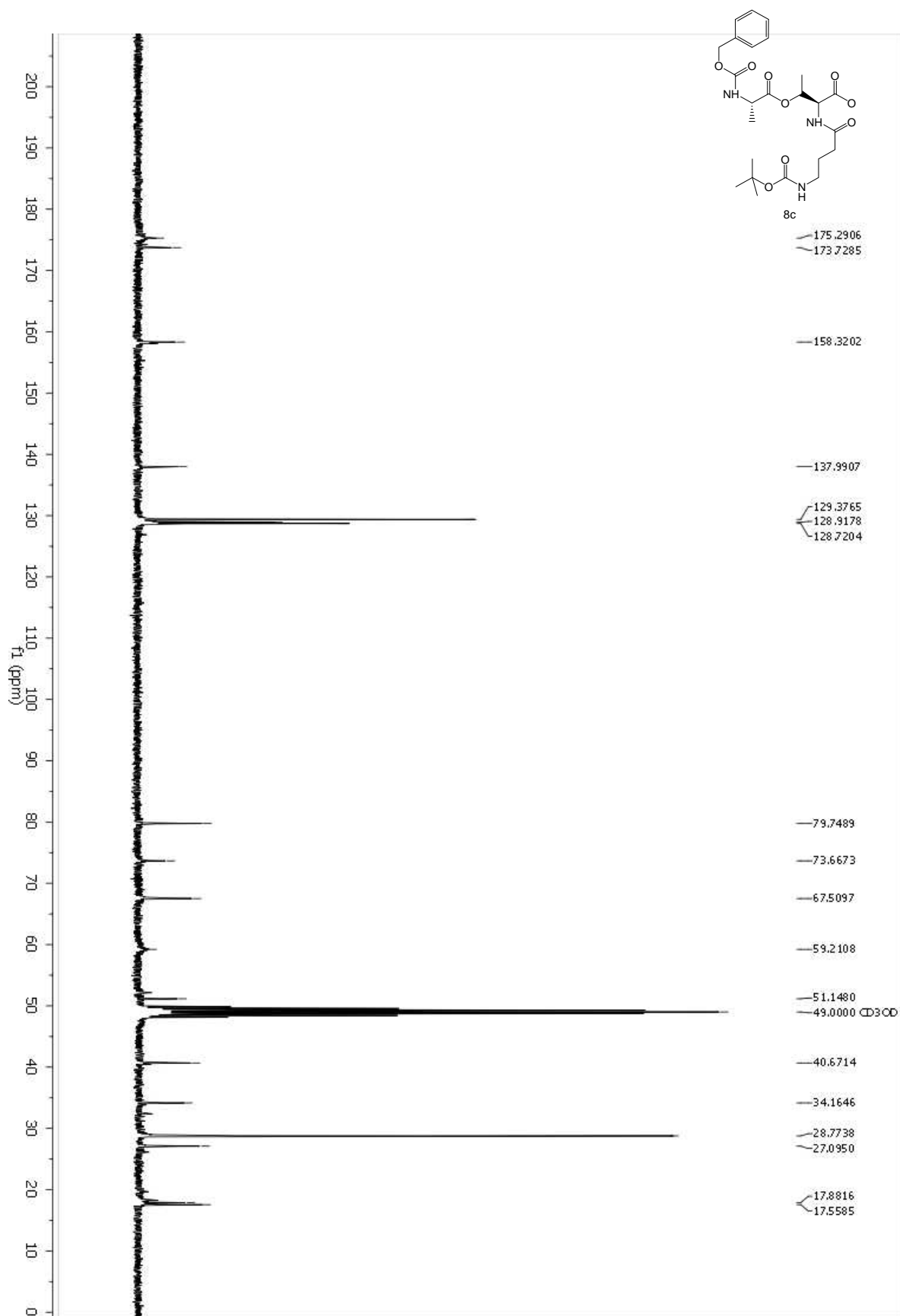


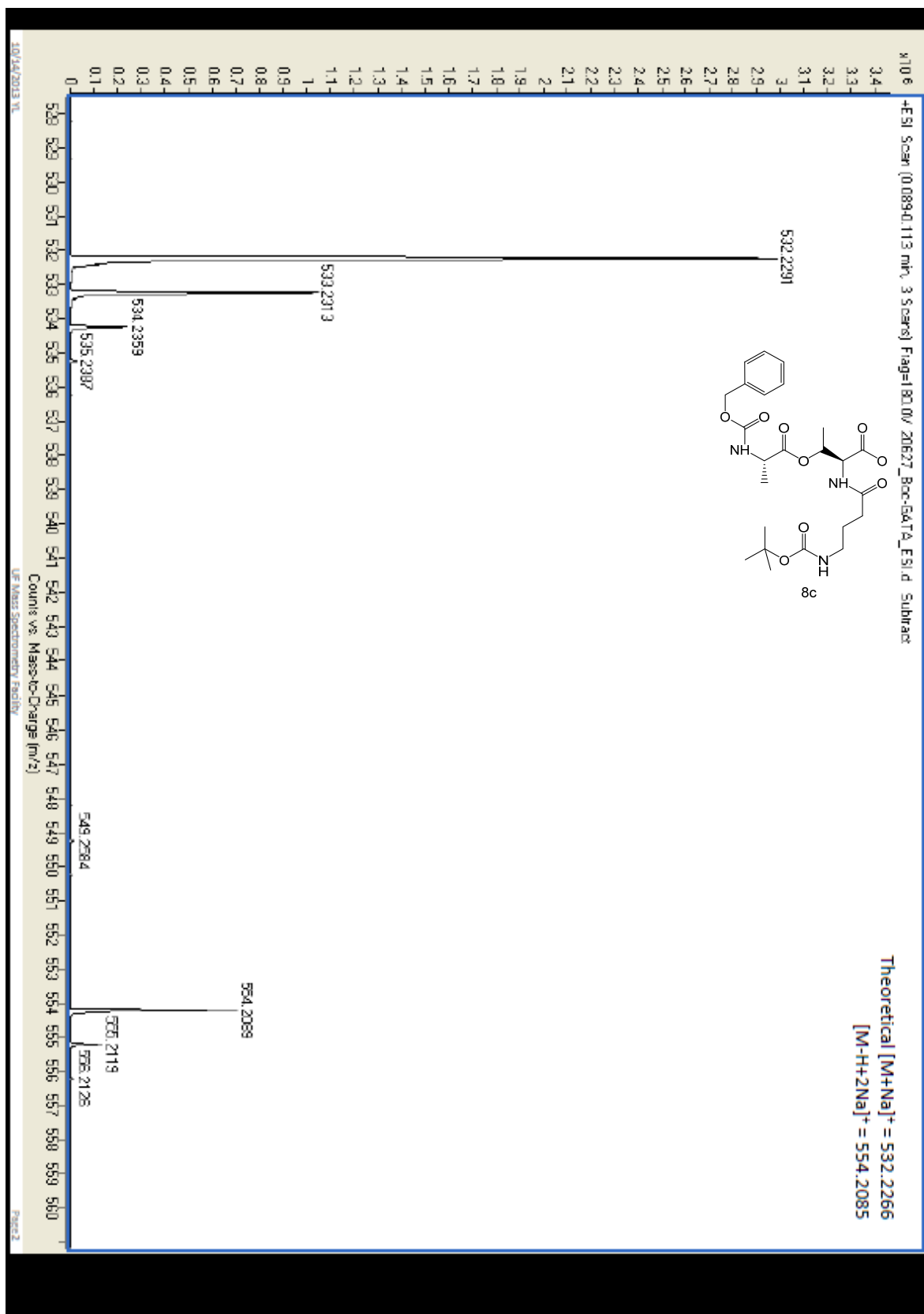


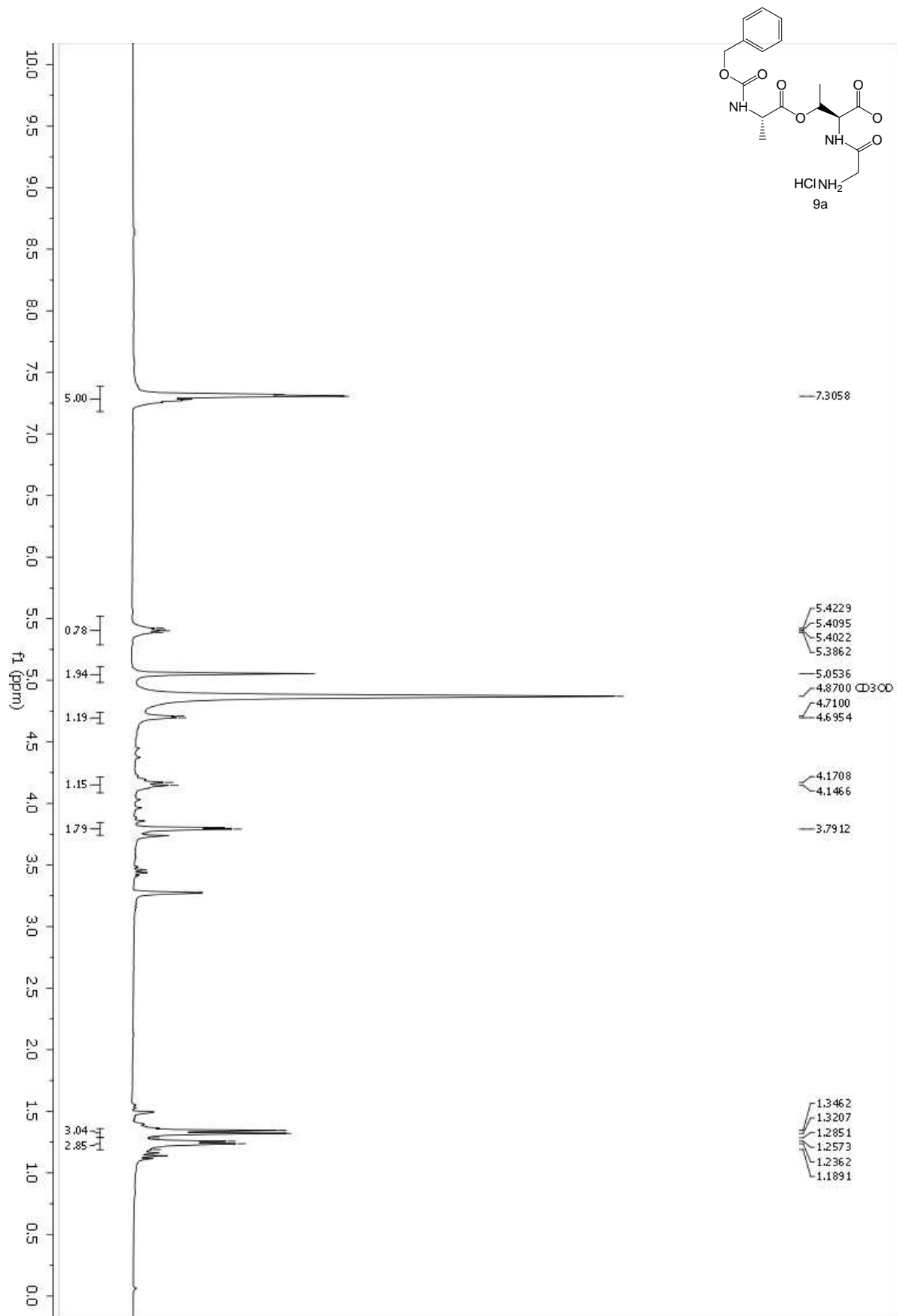


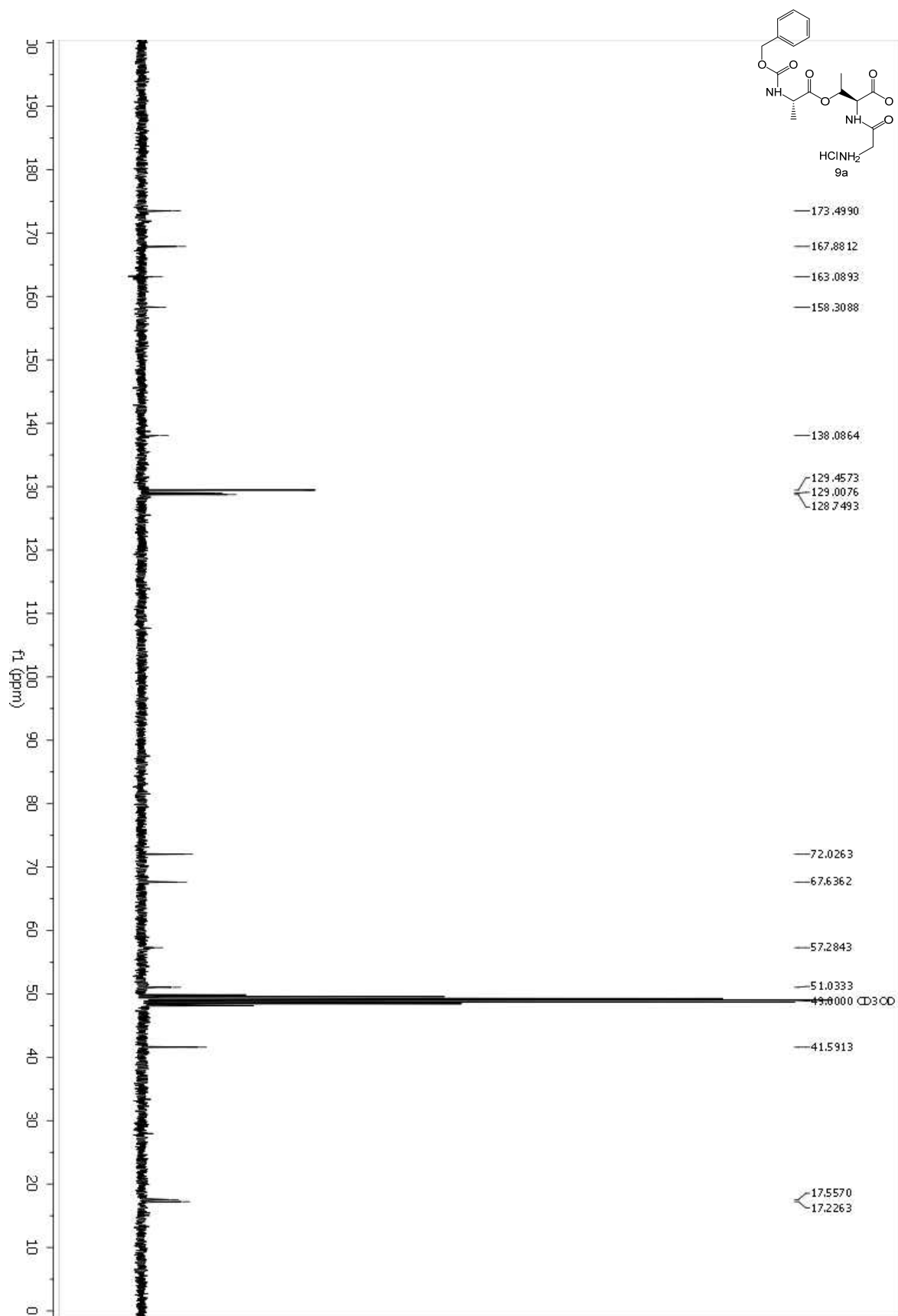


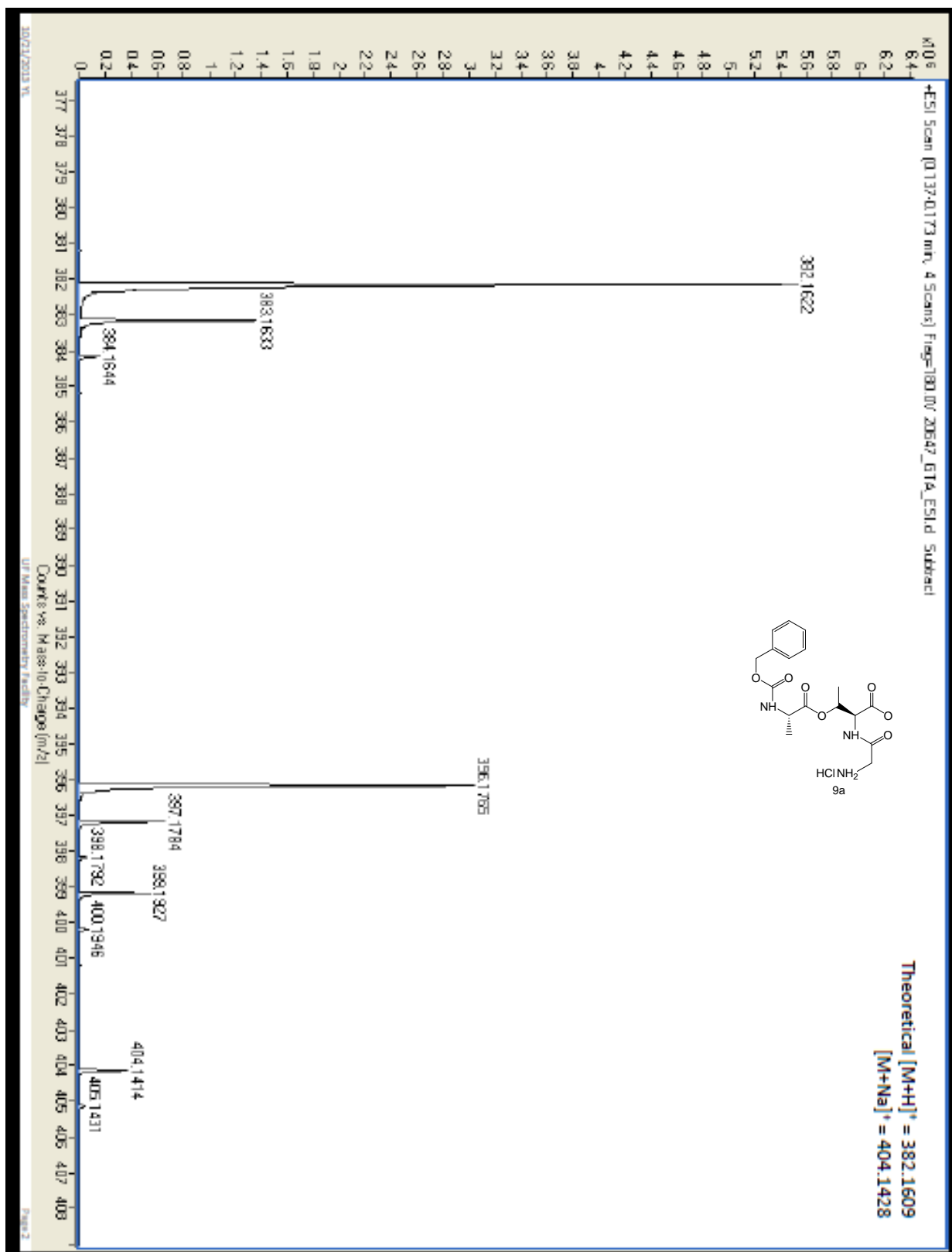


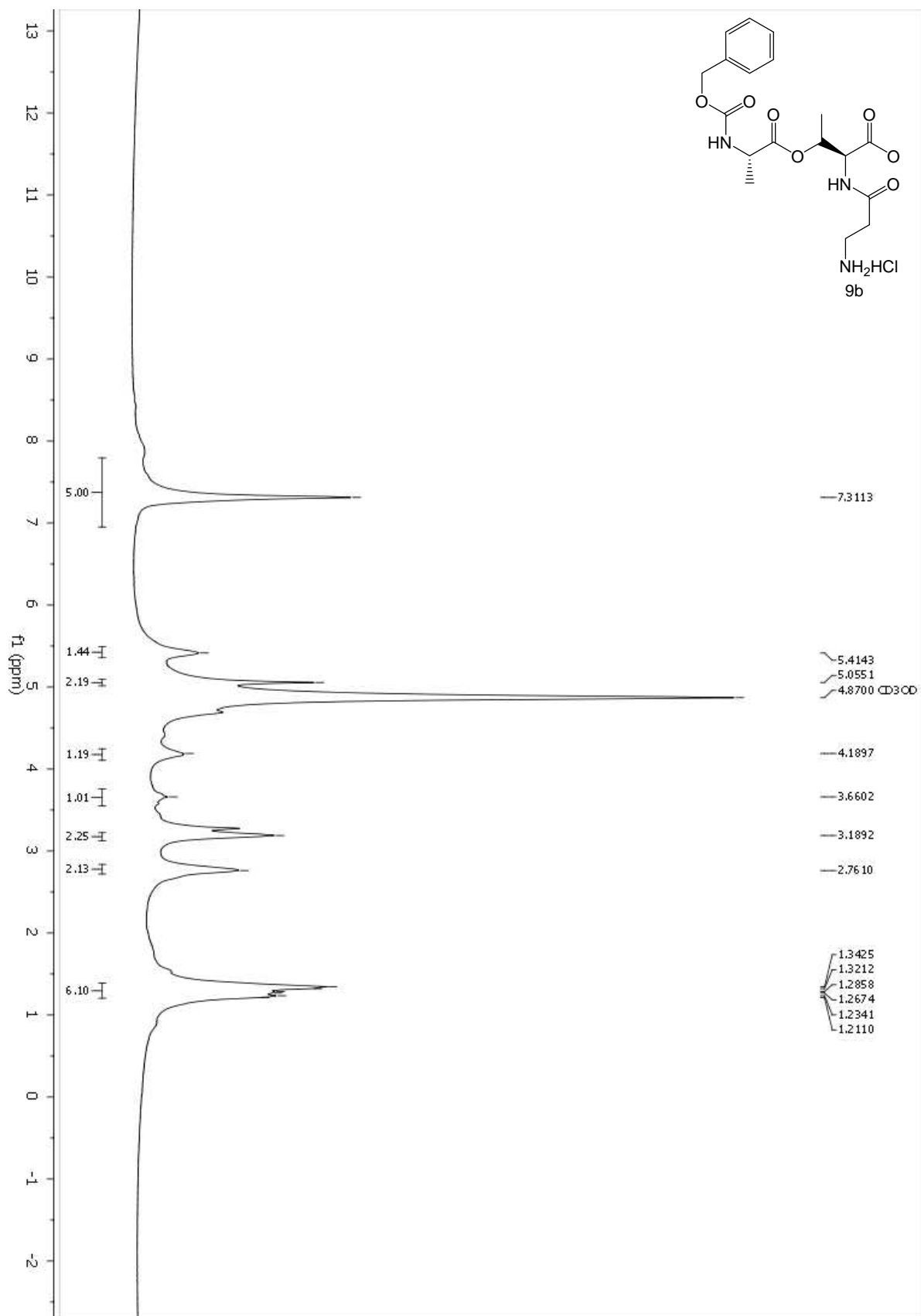


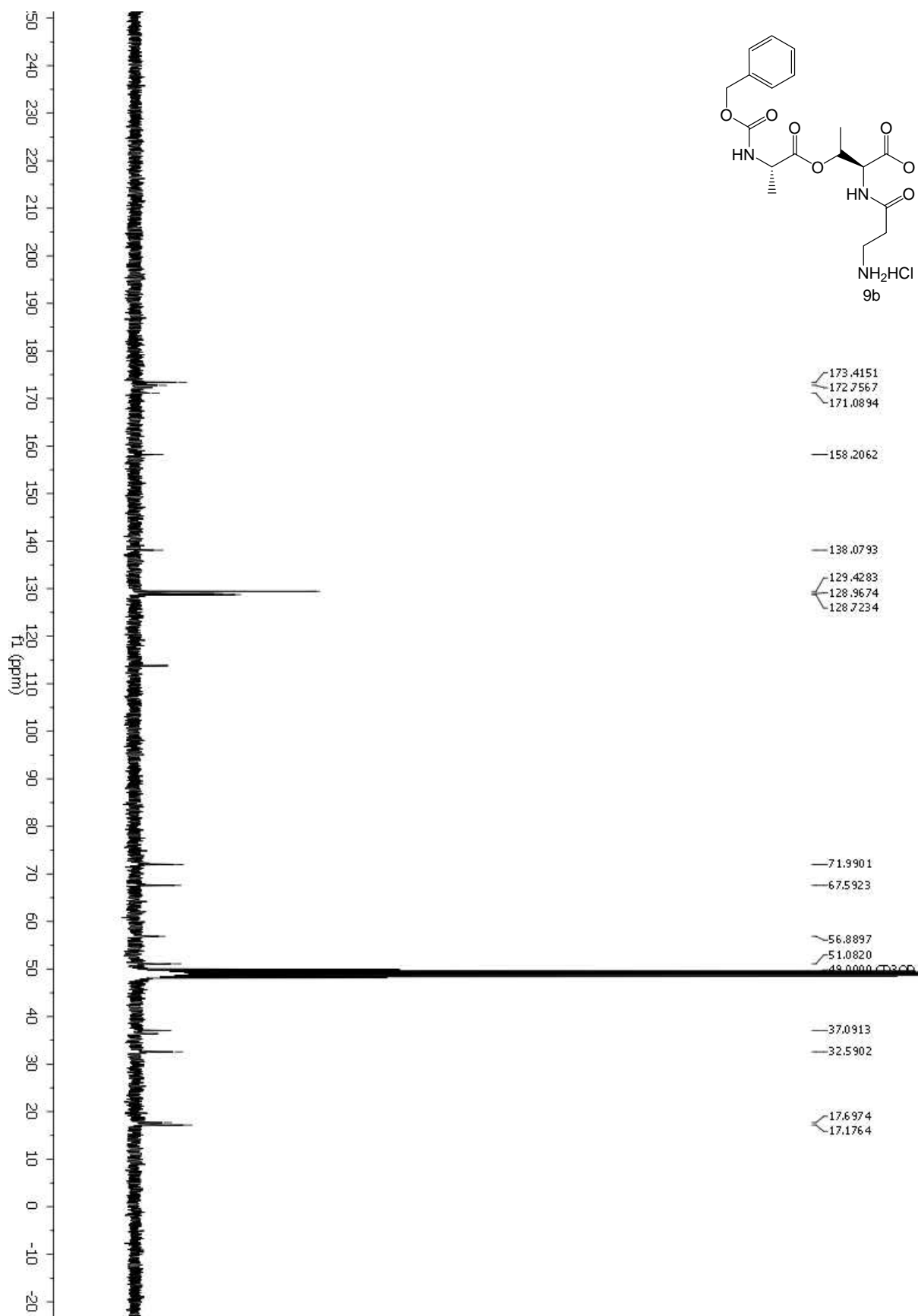


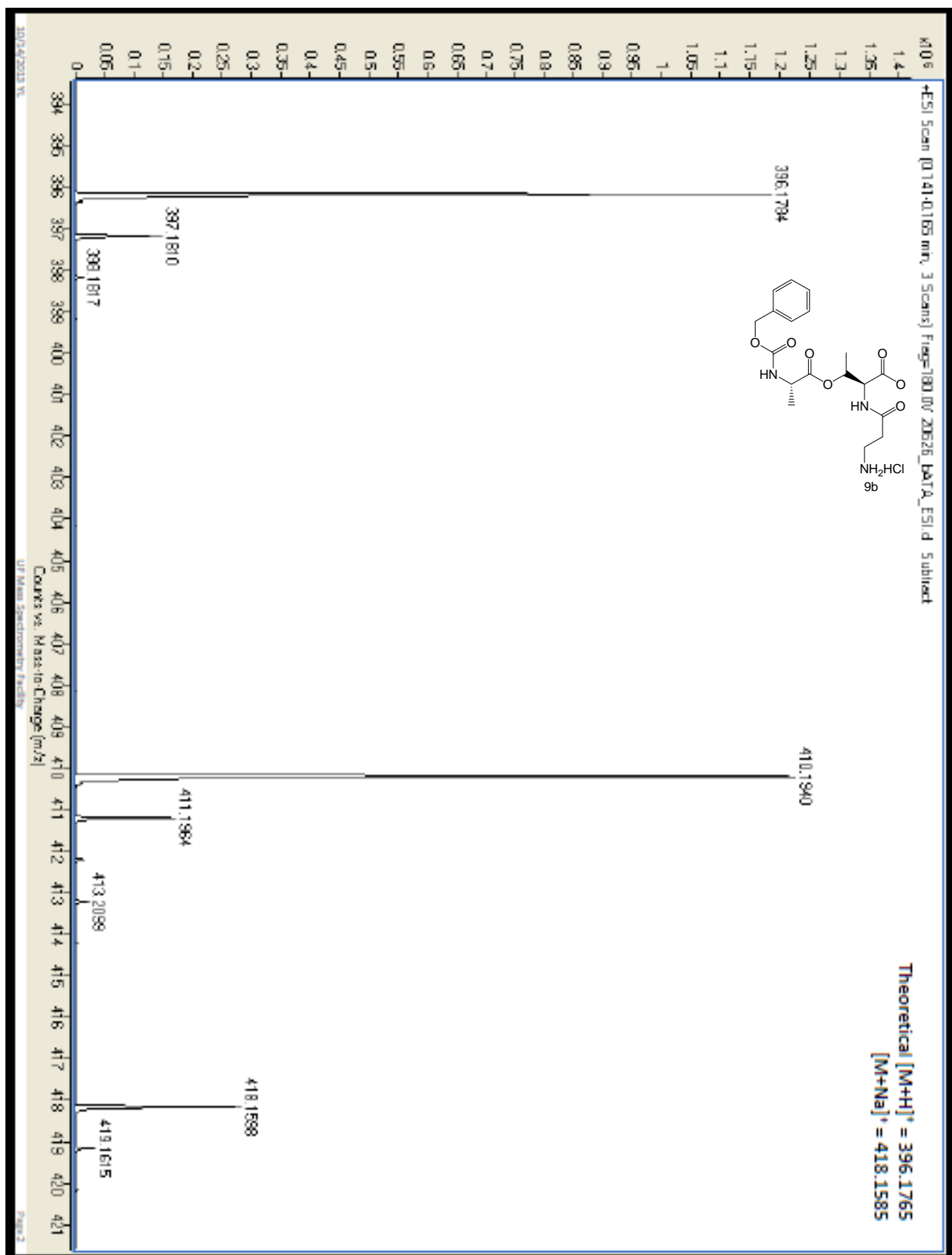


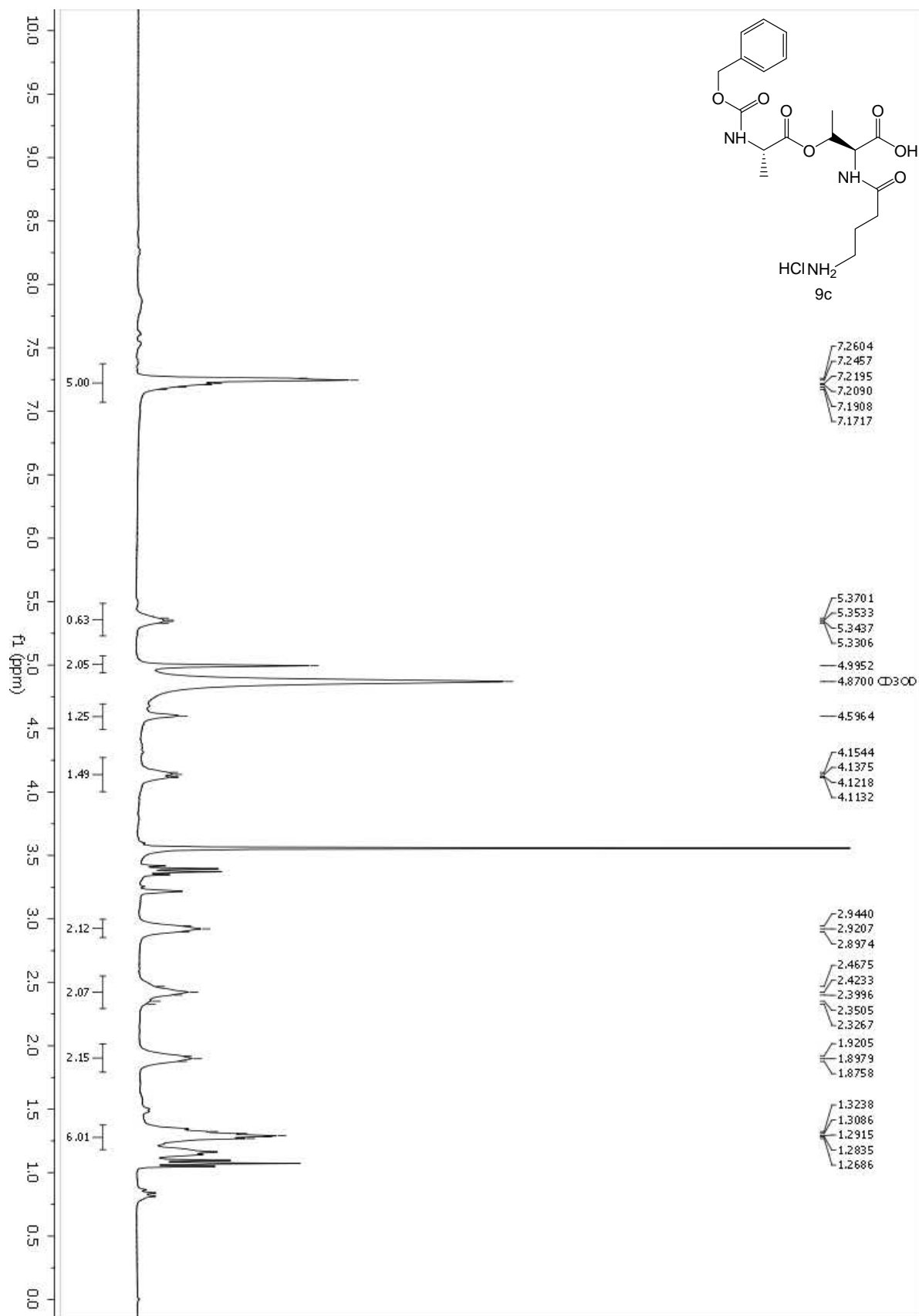


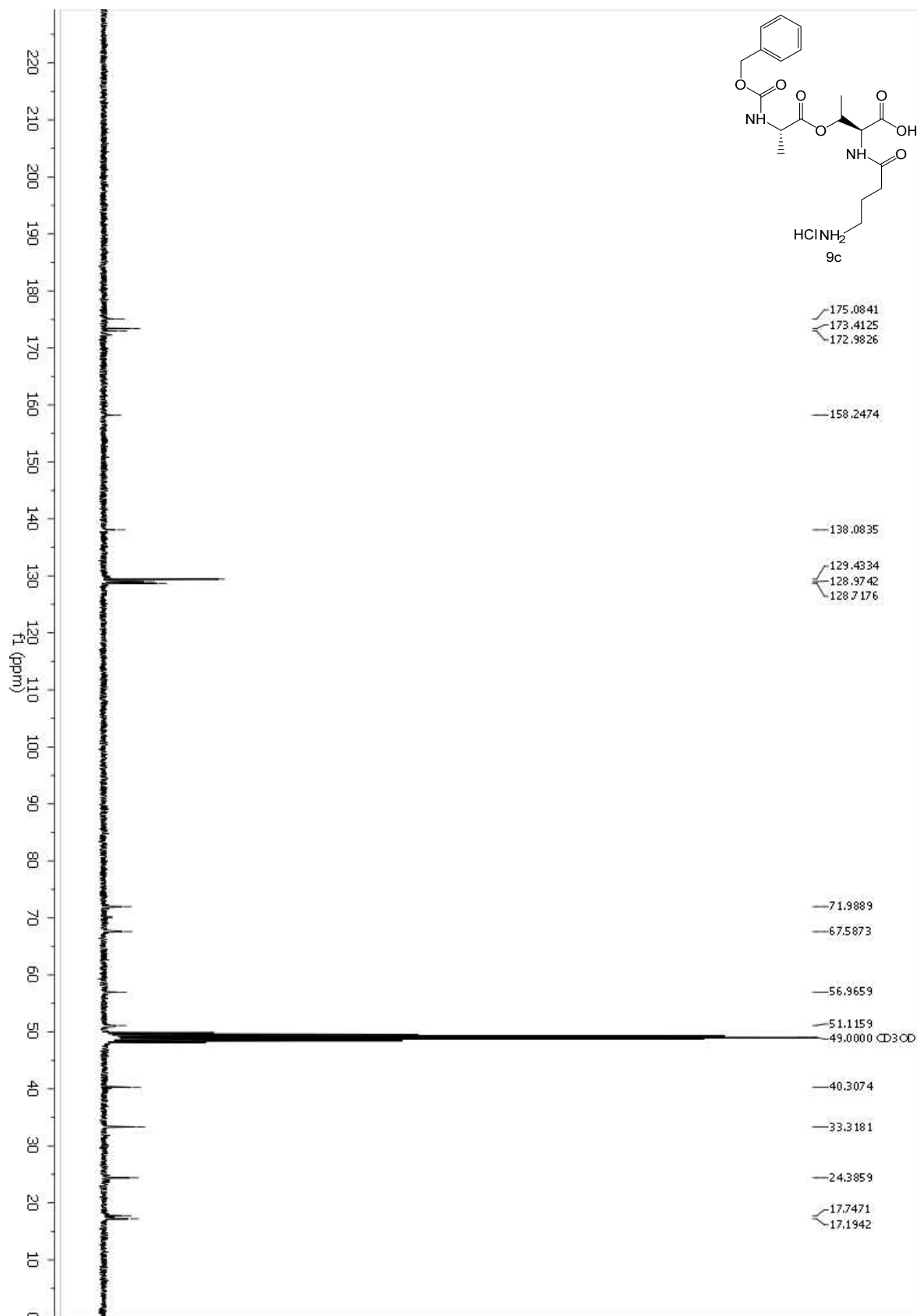


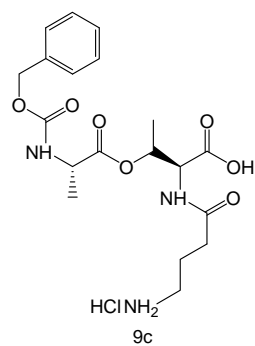
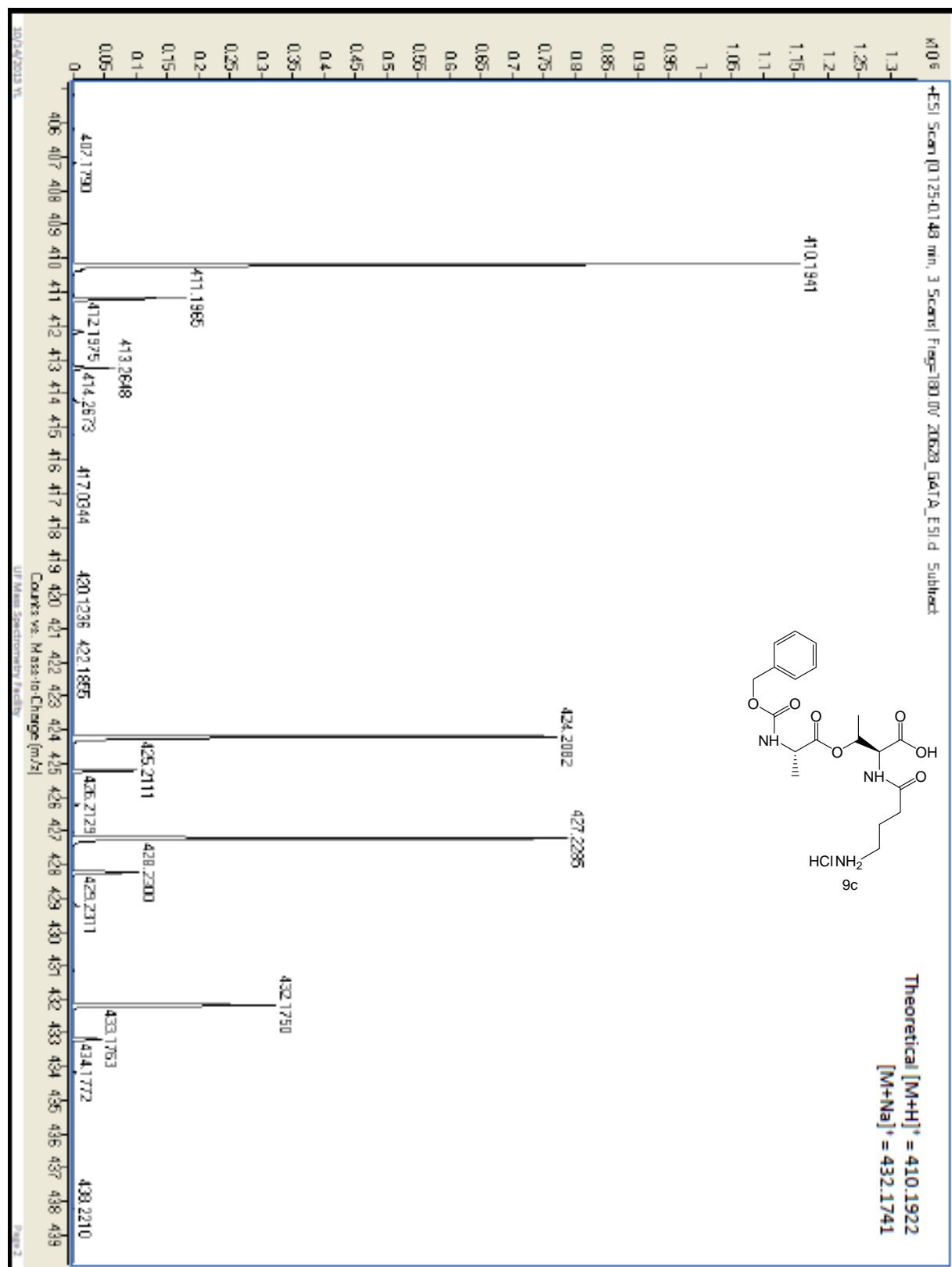






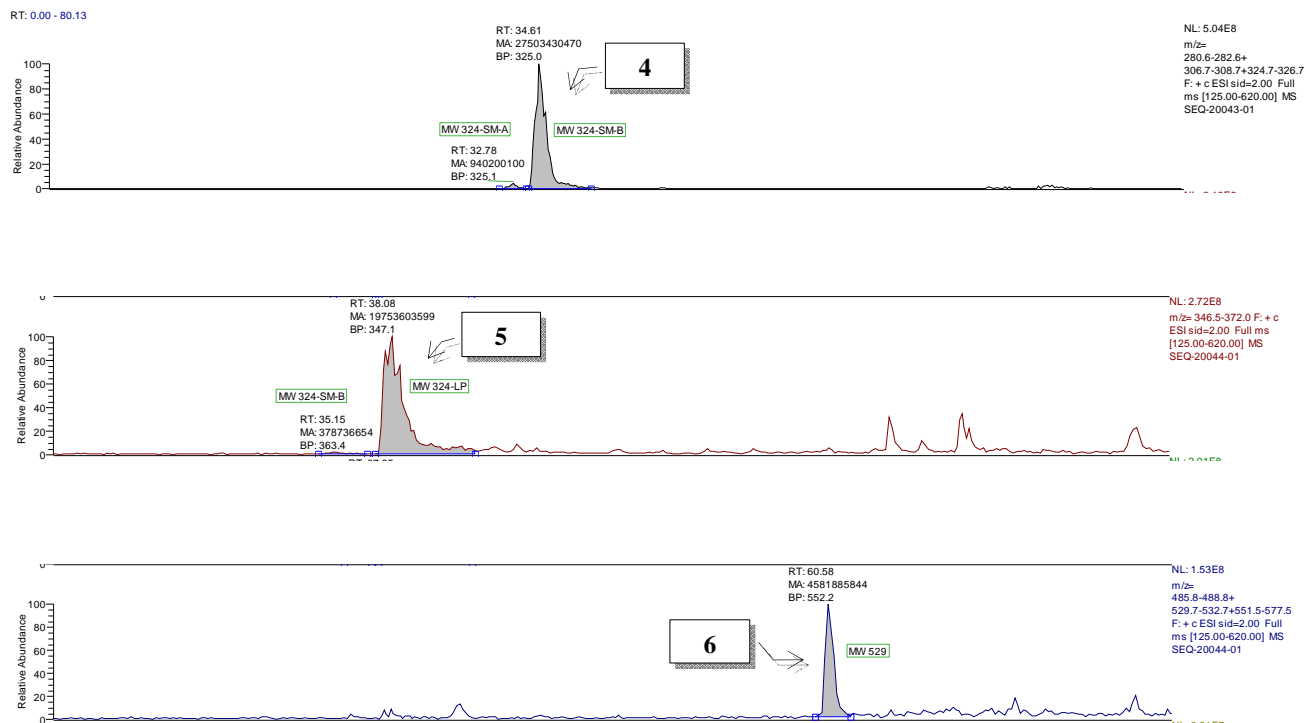






HPLC chromatograms and ESI-MS for chemical ligation of 4 in buffer

Summary Chromatogram: HPLC/(+)ESI-MS integrated ion-peaks for the **4** (top, rt 34.61), **5** (middle, rt 38.08), and **6** (bottom, rt 60.58).



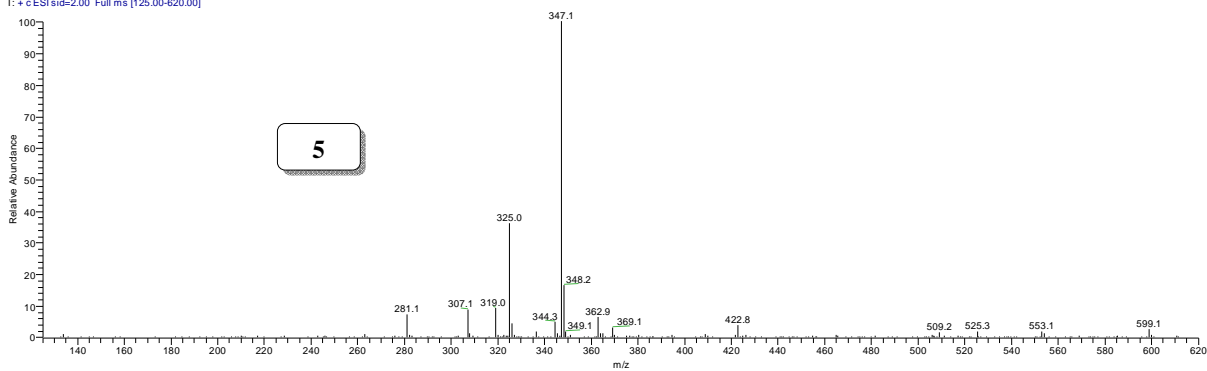
MW 324-LP **5** (top) and MW 324-SM **4** (bottom): (+)ESI-MS.

SEQ-20044-01
Xerra MS C18:0.15:95:5(0)>65:35(10)>25:75(50)>5:95(60-75)

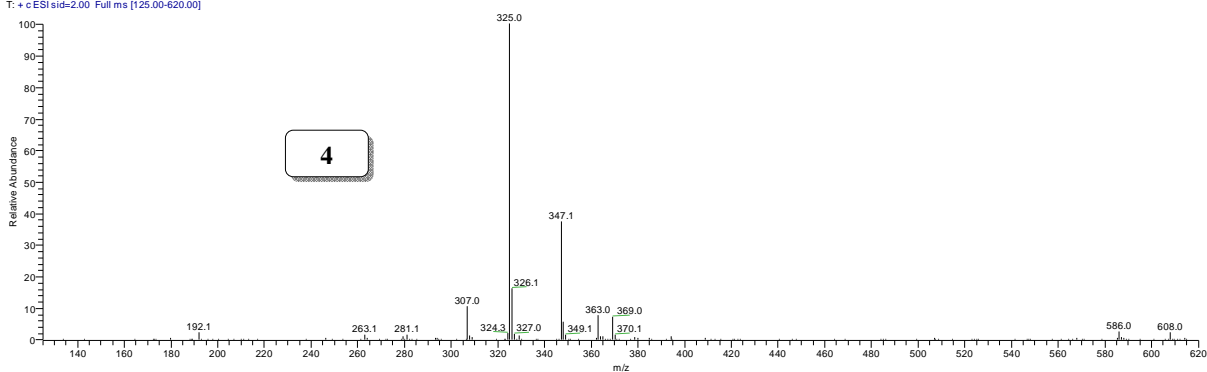
7/18/2013 6:47:02 PM

2181-5RS-LP; 5 uL

SEQ-20044-01 #1654-1726 RT: 37.60-38.56 AV: 9 SB: 4 36.90-37.18, 40.25-40.69 NL: 1.51E8
T: + c ESI:sd=2.00 Full ms [125.00-620.00]

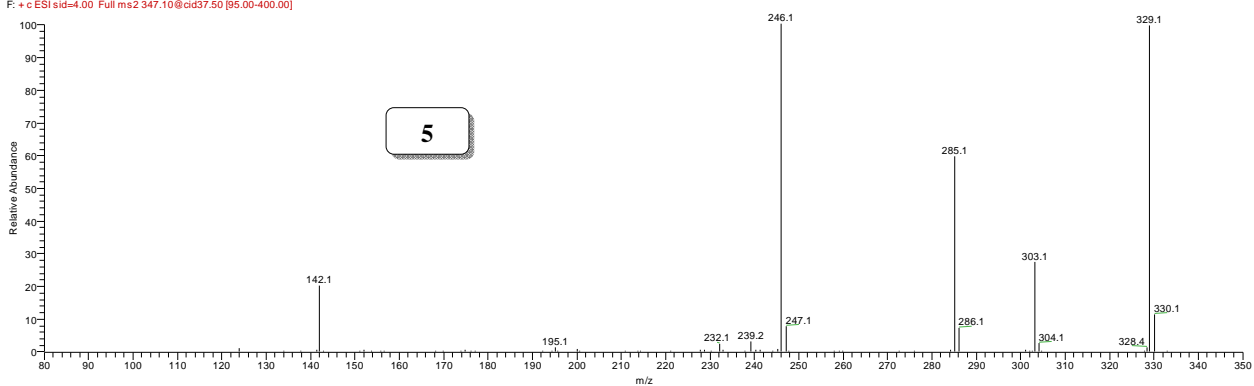


SEQ-20043-01 #1603-1660 RT: 34.40-35.04 AV: 7 SB: 4 33.64-33.79, 36.03-36.31 NL: 2.76E8
T: + c ESI:sd=2.00 Full ms [125.00-620.00]

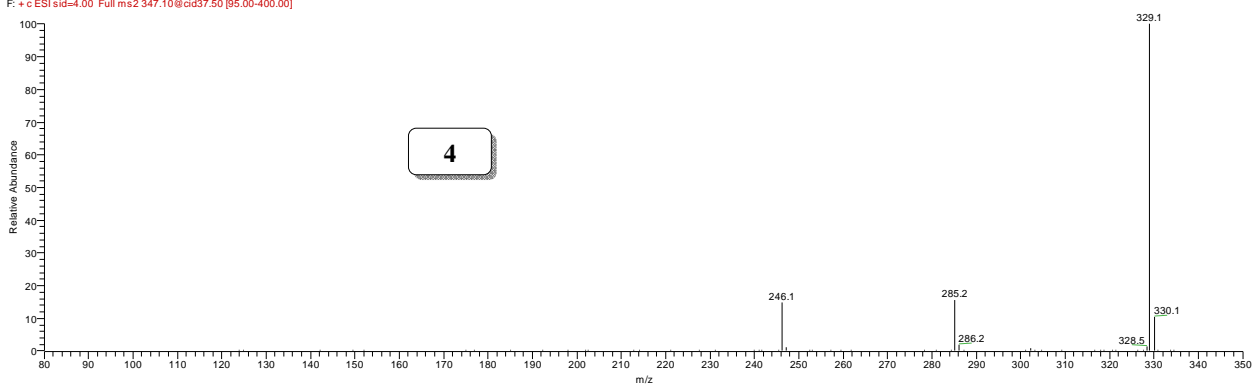


MW 324-LP **5** (top) and MW 324-SM **4** (bottom): (+)ESI-MS/MS of the m/z 347 $[M+Na]^+$ ions.

SEQ-20044-01 7/18/2013 6:47:02 PM 2181-5RS-LP; 5 μ L
Xterra MS C18.0.15.95.5(0)>65.35(10)>25.75(60)>5.95(60-75)
SEQ-20044-01 #1650-1742 RT: 37.59-38.79 AV: 11 SB: 1 36.96-37.18 NL: 1.62E7
F: + c ESI sid=4.00 Full ms2 347.10@cid37.50 [95.00-400.00]



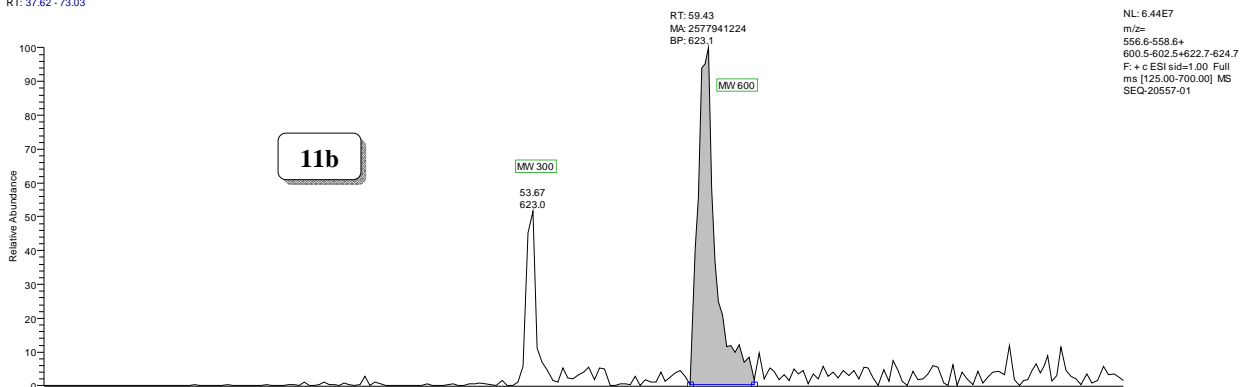
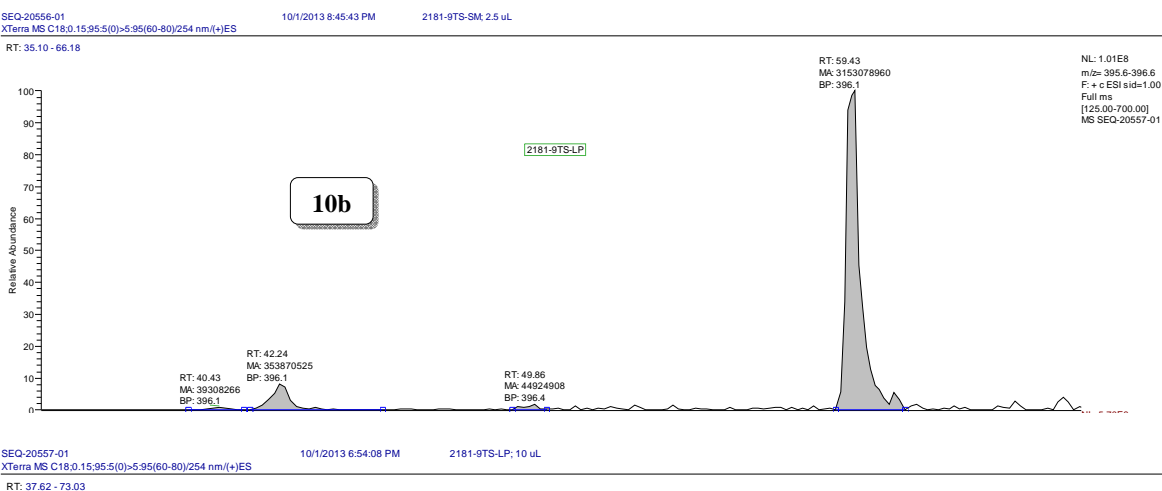
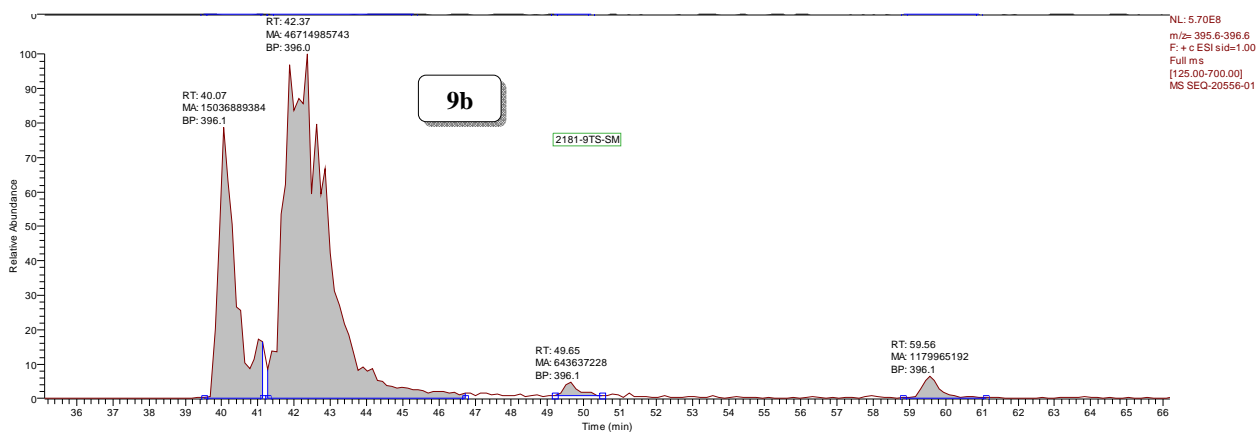
SEQ-20043-01 #1589-1678 RT: 34.17-35.25 AV: 11 SB: 2 33.61-33.88 NL: 5.63E7
F: + c ESI sid=4.00 Full ms2 347.10@cid37.50 [95.00-400.00]

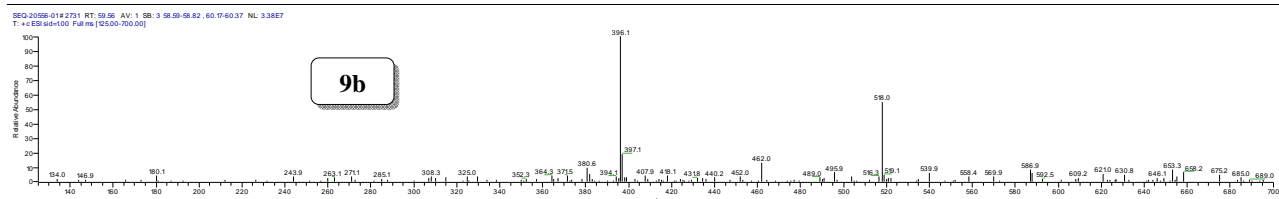
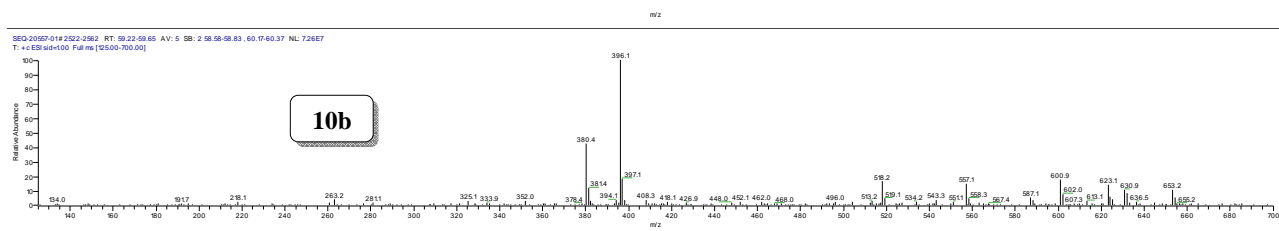


MW 324-LP **5** (top two) and MW 324-SM **4** (bottom two): (+)ESI-MS/MS (top and 3rd) and -MS/MS/MS (2nd and bottom) of the m/z 325 [M+H]⁺ ions.



Summary Chromatogram: HPLC/(+)ESI-MS integrated ion-peaks for the **4** (top, rt 34.61), **5** (middle, rt 38.08), and **6** (bottom, rt 60.58).



MW 395 isomers: (+)ESI-MS for RT 59.5 min LP **10b** (top) and SM **9b** (bottom).MW 395, RT 59.5 min: (+)ESI-MS/MS of m/z 396 [M+H]⁺ ions for LP (top) and SM (bottom).