Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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# Supporting Information

For

# 'Clickable' 2,5-diketopiperazines as scaffolds for ligation of biomolecules: use in Aβ inhibitor's assembly

E. Dufour, L. Moni, L. Bonnat, S. Chierici*, and J. Garcia

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## S1. Kinetic studies of $A\beta_{40}$ fibril formation in presence of DKPs

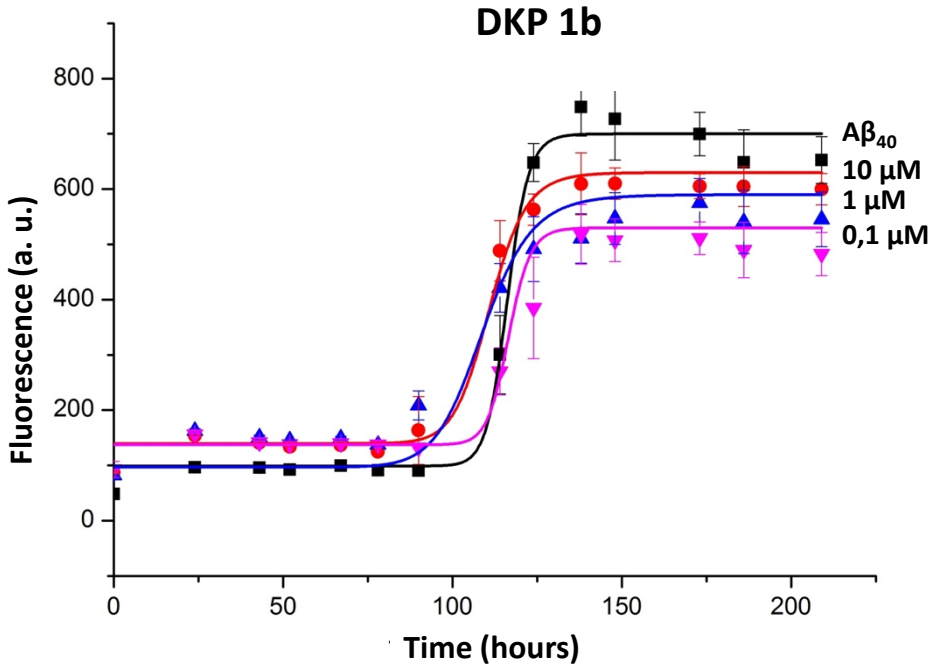
### Materials and methods

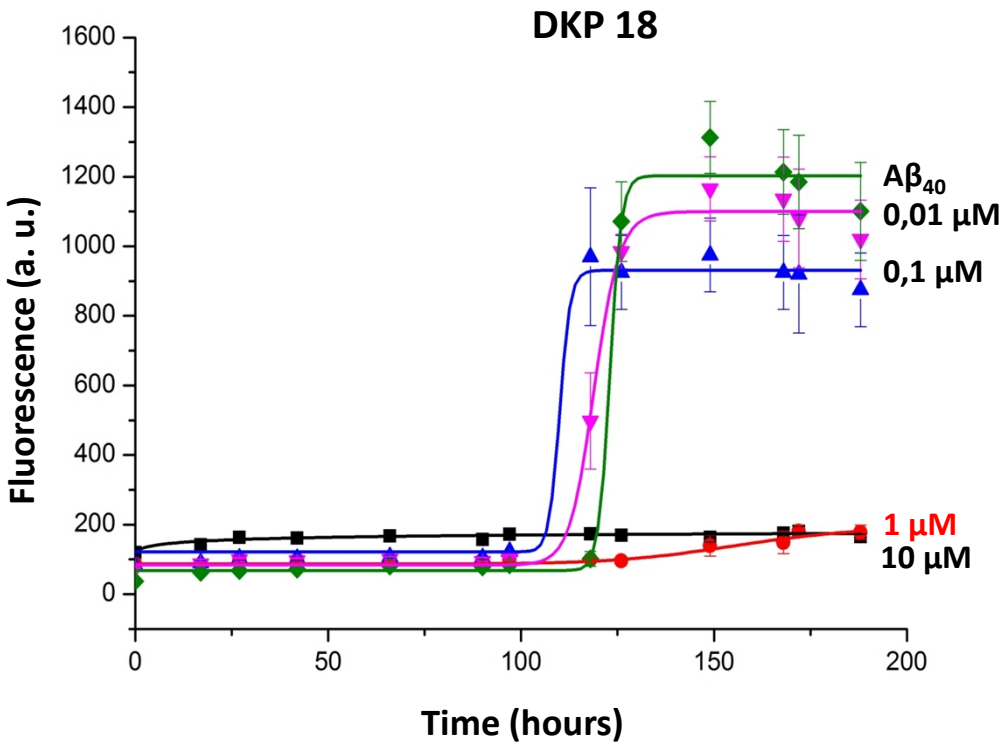
We used for the kinetic assays the same materials and conditions as we have previously described (G. T. Dolphin, S. Chierici, M. Ouberai, P. Dumy and J. Garcia, *ChemBioChem*, 2008, **9**, 952. These conditions are mentioned below.

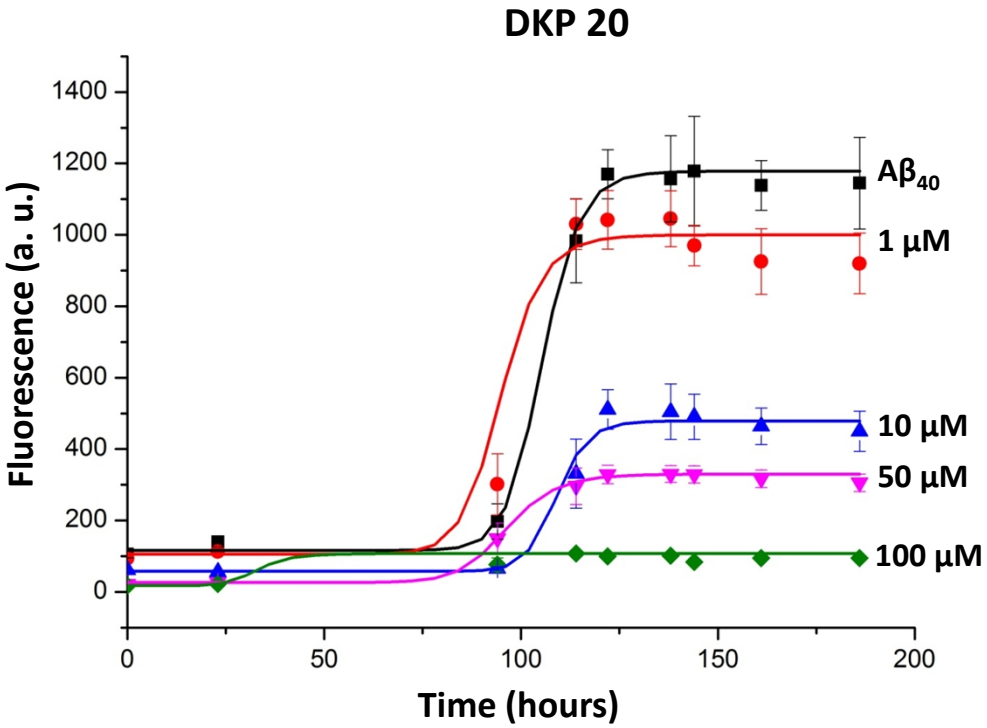
*Preparation of synthetic $A\beta_{40}$ peptide:* $A\beta_{40}$ was synthesized on an Applied Biosystems 433A peptide synthesizer using Fmoc-Val-Novasyn-TGA resin (loading 0.24 mmol/g). The peptide was assembled using standard solid phase methods but coupling reaction times of 60 min were used and difficult residues were coupled twice. Removal of protecting groups and cleavage from the resin were carried out with a mixture of TFA/TIS/$H_2O$/EDT (94:2:2:2), with swirling for 2 h. After filtration, TFA was removed under *vacuum* and the peptide was precipitated in $Et_2O$. $A\beta_{40}$ peptide was obtained after purification by RP-HPLC (C5, 214 nm, 10-90% B in 30 min) and lyophilization, and was stored at -20 °C. RP-HPLC (C18, 214 nm, 5-100% solvent B in 20 min) $t_R$ = 12 min. ESI-MS calcd 4328, found 4329.7.

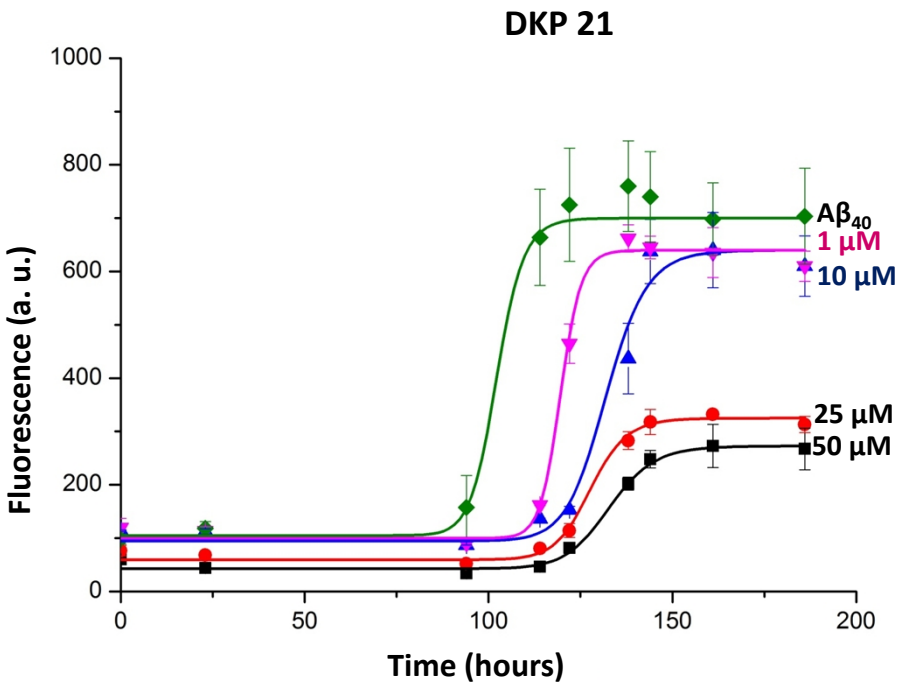
*Preparation of inhibitor stock solutions:* Inhibitors were dissolved in DMSO/$H_2O$ (1/1). Stock solutions at 5 mM were first prepared, thereafter they were diluted with DMSO/$H_2O$ (1/1) to afford a concentration range of 2.5 mM to 0.5 µM. Final concentrations of DMSO in inhibition studies were less than 2%.

*Aggregation measurement of Aβ40:* A solution of $A\beta_{40}$ was prepared as follows: 2.7 mg were dissolved in 200 µL of 1,1,1,3,3,3-hexafluoro-2-propanol to disassemble preformed aggregates, thereafter it was lyophilized. One mL of pure water was added to the lyophilized peptide and the solution was centrifuged at 12 000 g to remove eventual aggregates. The concentration of $A\beta_{40}$ was 500 µM. Aggregation of $A\beta_{40}$ was performed in 96-well black polypropylene microplates (Sterilin). To each well an aliquot of the $A\beta_{40}$ peptide solution was mixed into the aggregation buffer giving a final composition of $A\beta_{40}$ (50 µM) and ThT (10 µM) in sodium phosphate (50 mM) and NaCl (100 mM) pH 7.4. Aliquots of 2 µL of the inhibitor compounds were added, giving the aggregation mixture a total volume of 100 µL. The microplates were sealed with a plastic sheet and incubated at 37 °C. The ThT fluorescence intensity was recorded once or twice daily using bandpass filters of 445 nm for excitation and 485 nm for emission, and a cutoff filter of 475 nm, using a Molecular Devices Spectra MAX Gemini XS microplate reader. The data are the result of three experiments. Kinetic data were fitted with the stretched exponential function: $F(t)=F(\infty)-\Delta F\exp(-(kt)^n)$, where F(t) is the fluorescence at time t, $F(\infty)$ is the fluorescence after complete fibril formation, $\Delta F$ is the difference in fluorescence between t(0) and $t(\infty)$, k is the rate constant, and values larger than 1 for the parameter n indicate a sigmoidal transition with an initial lag-phase.

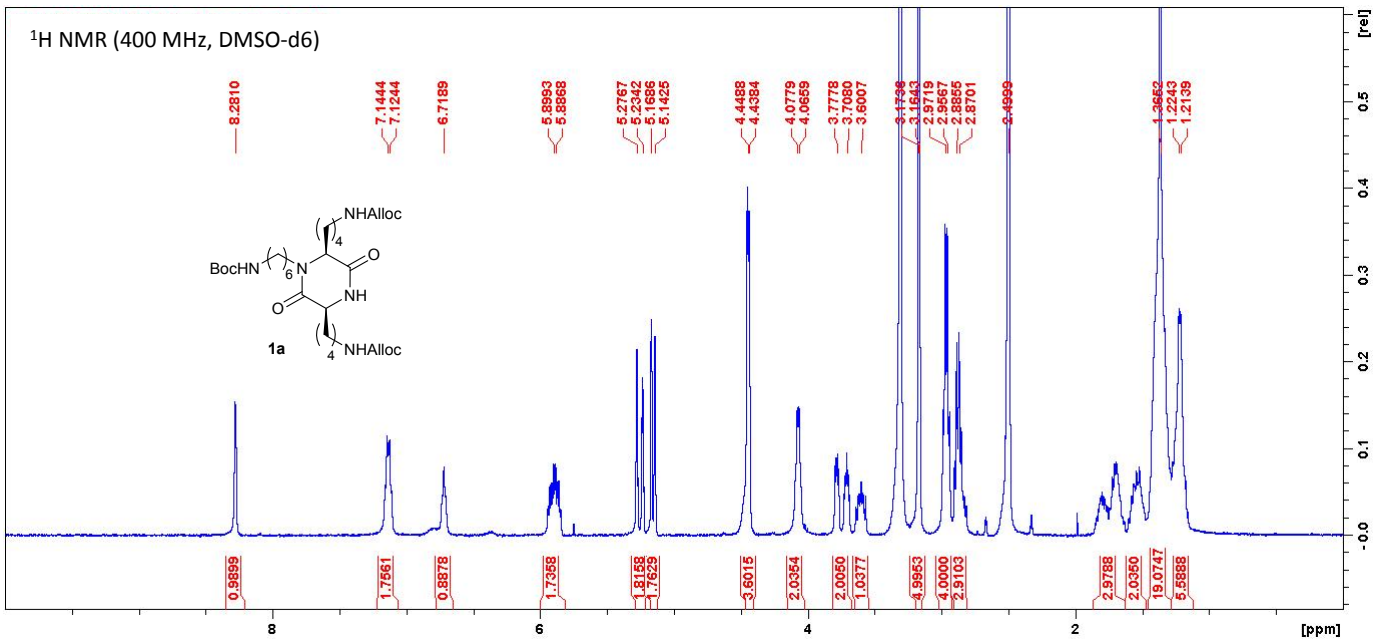


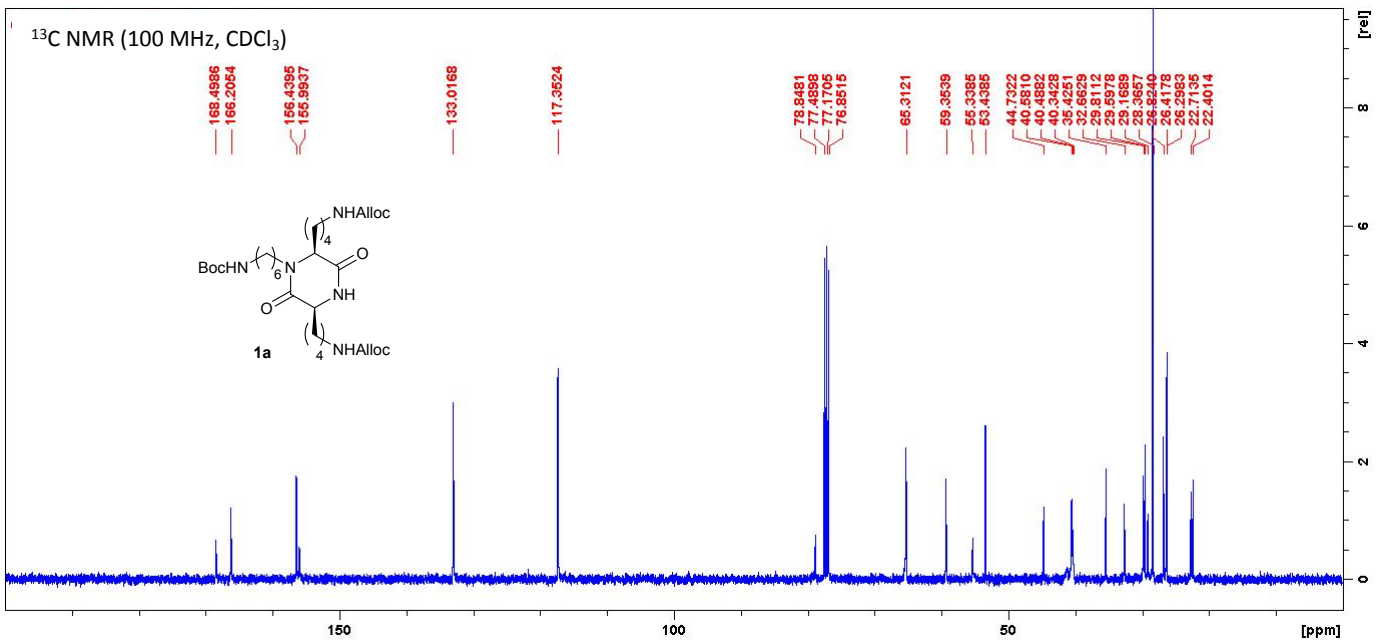



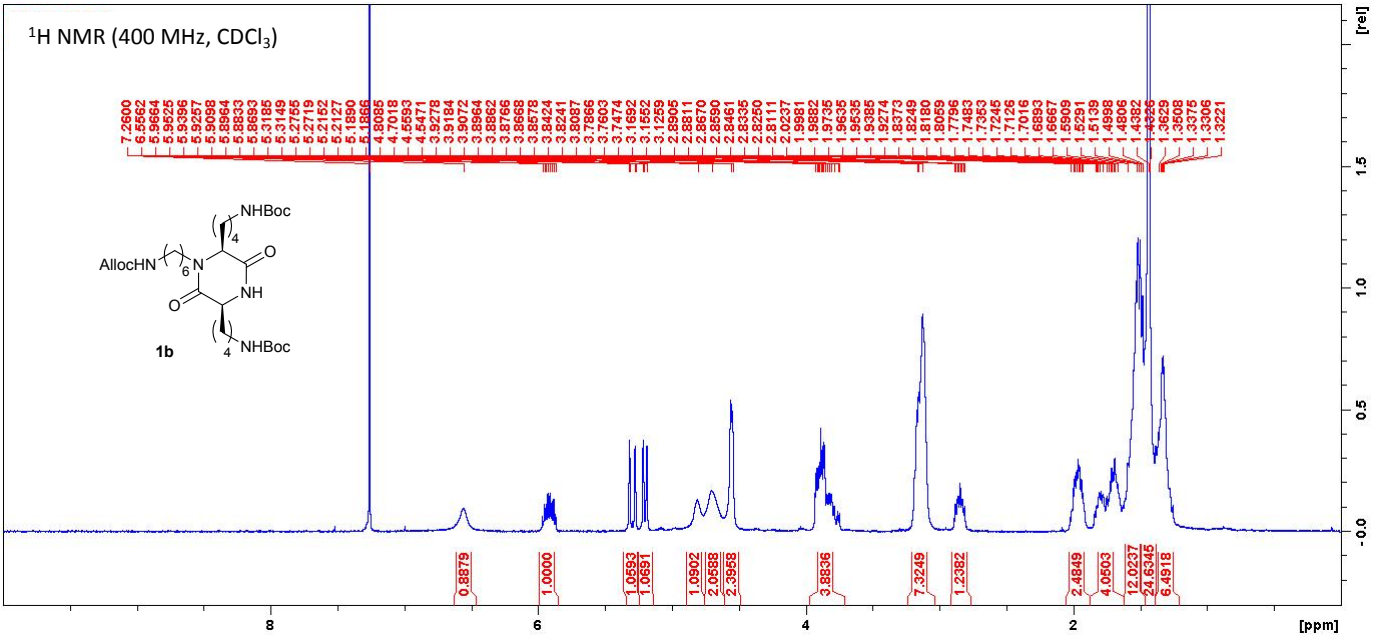


# S2. Copies of NMR spectra

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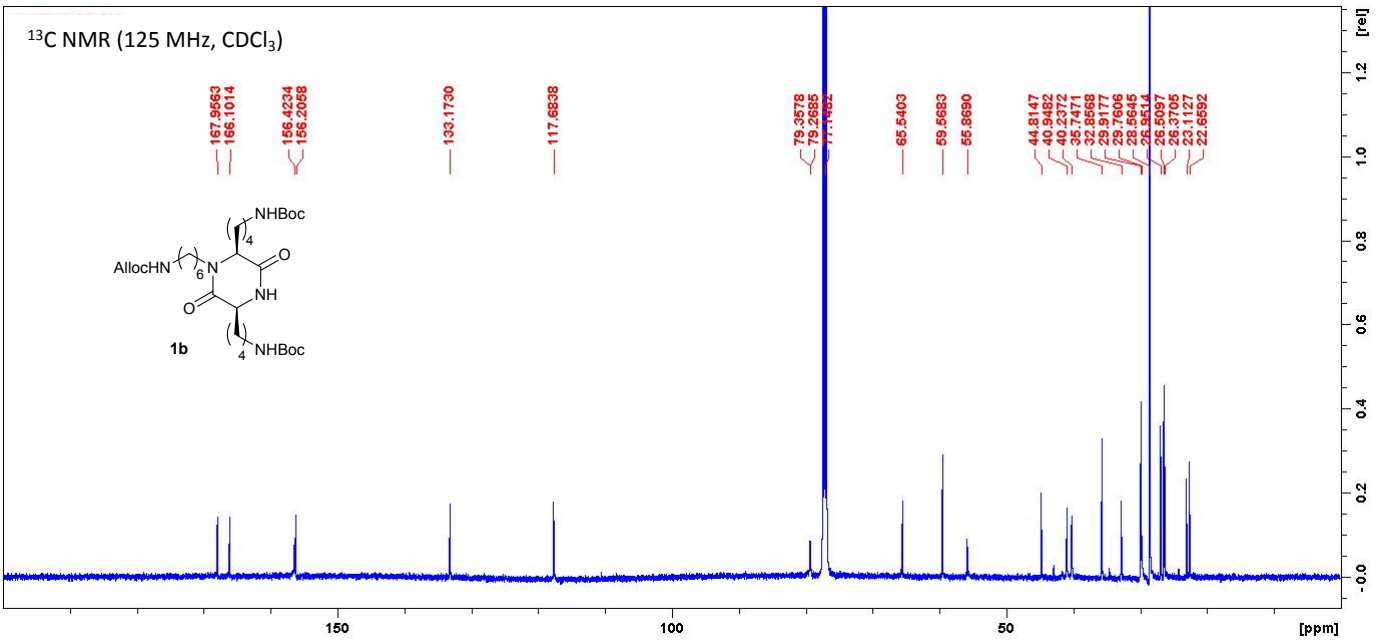
[rel]
[ppm]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NH
NHBoc
1b
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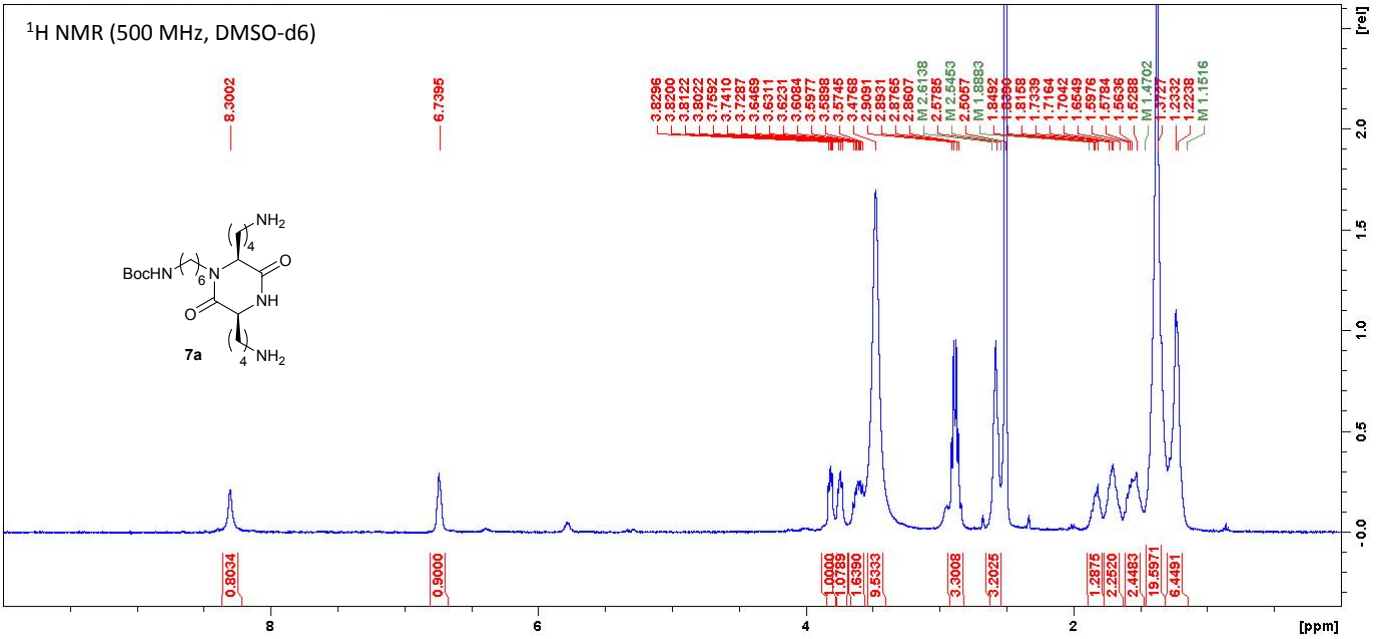
[ppm]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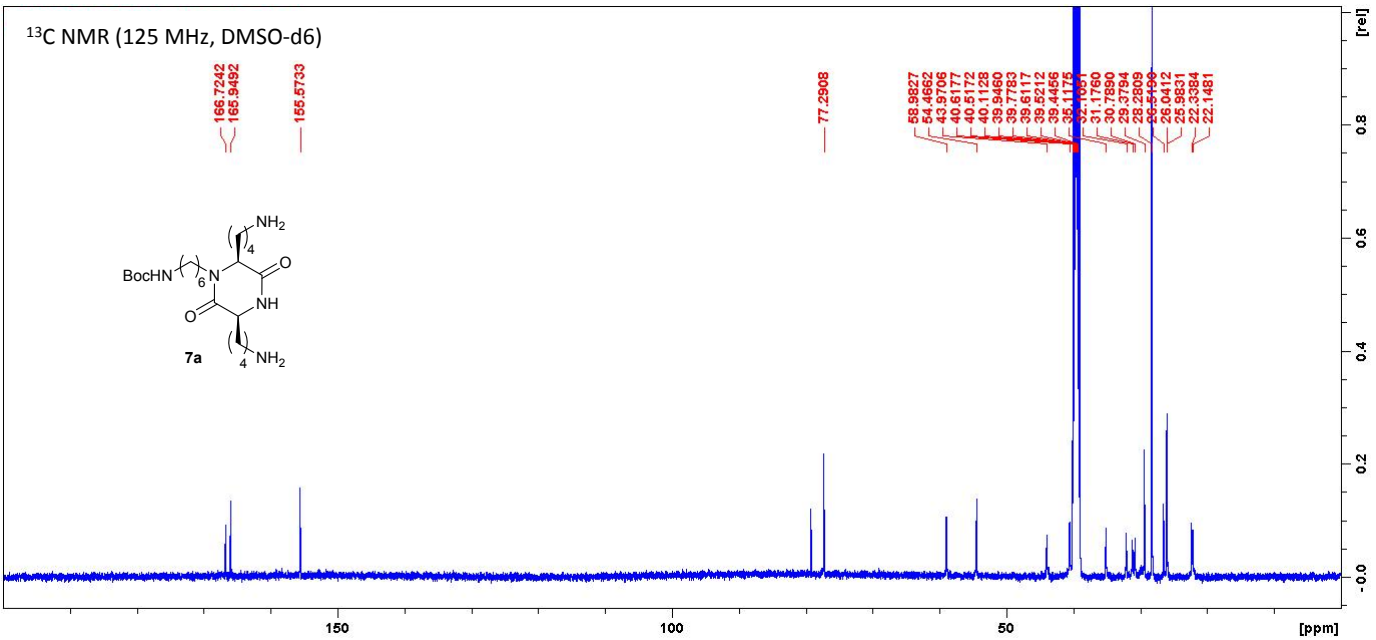
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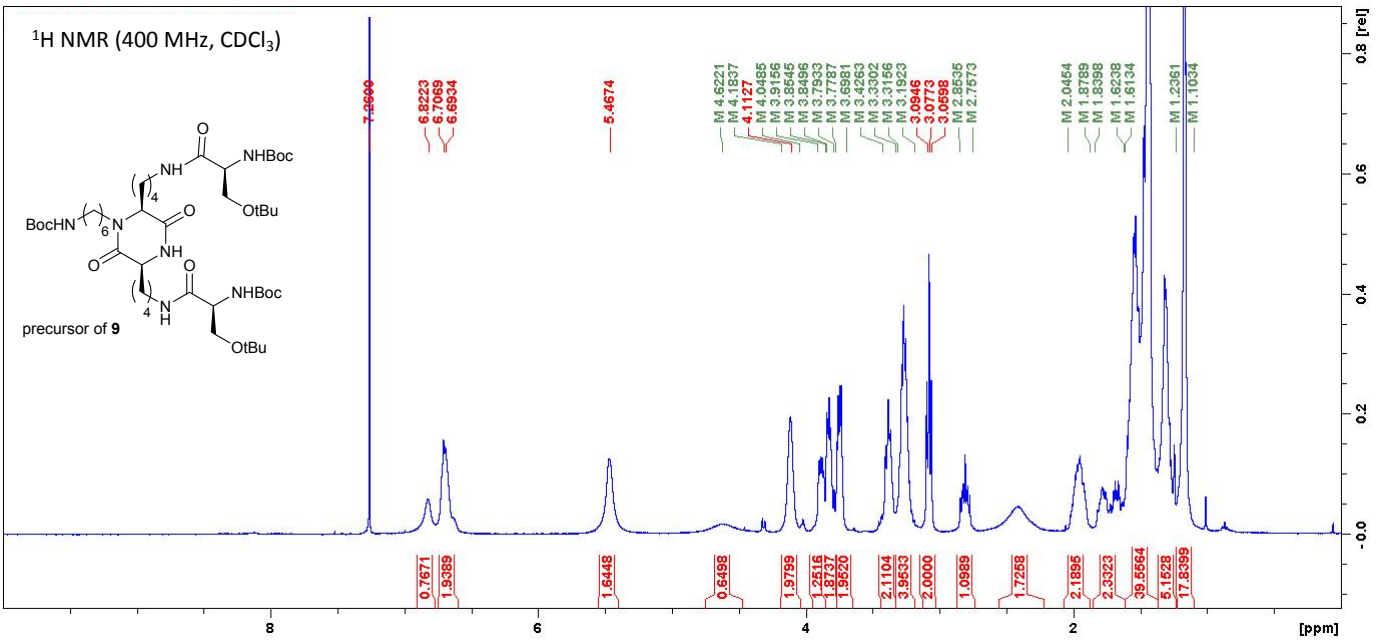
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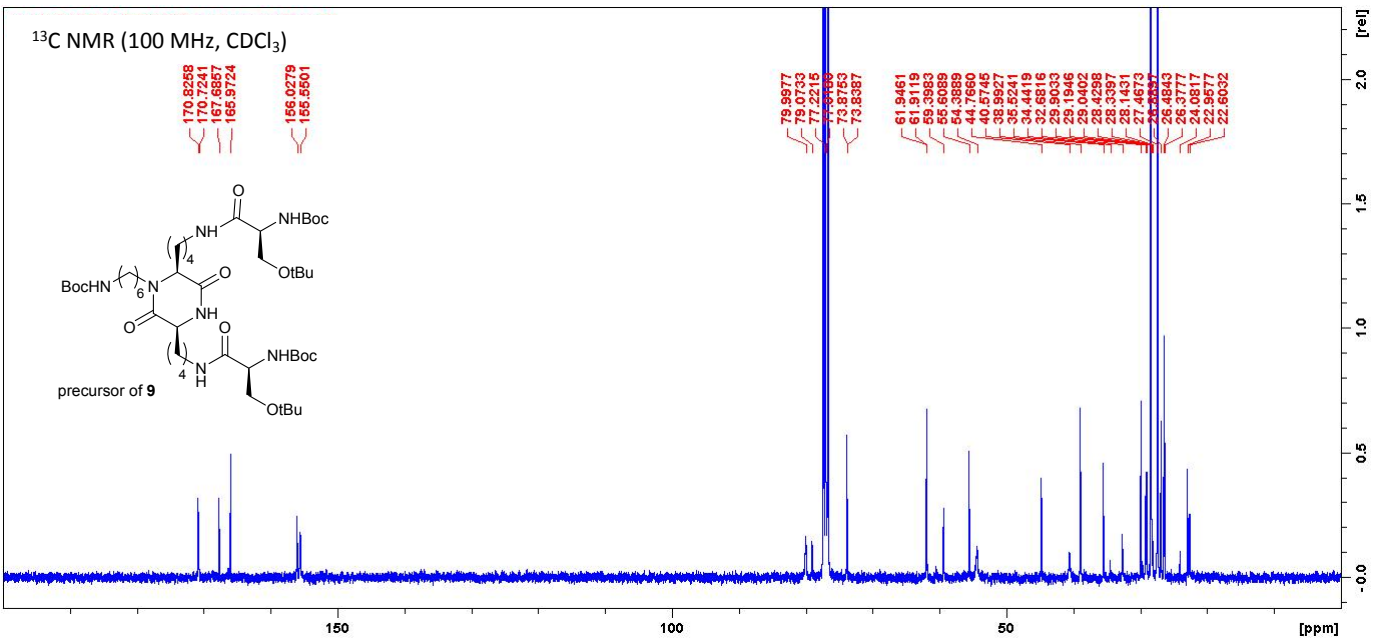
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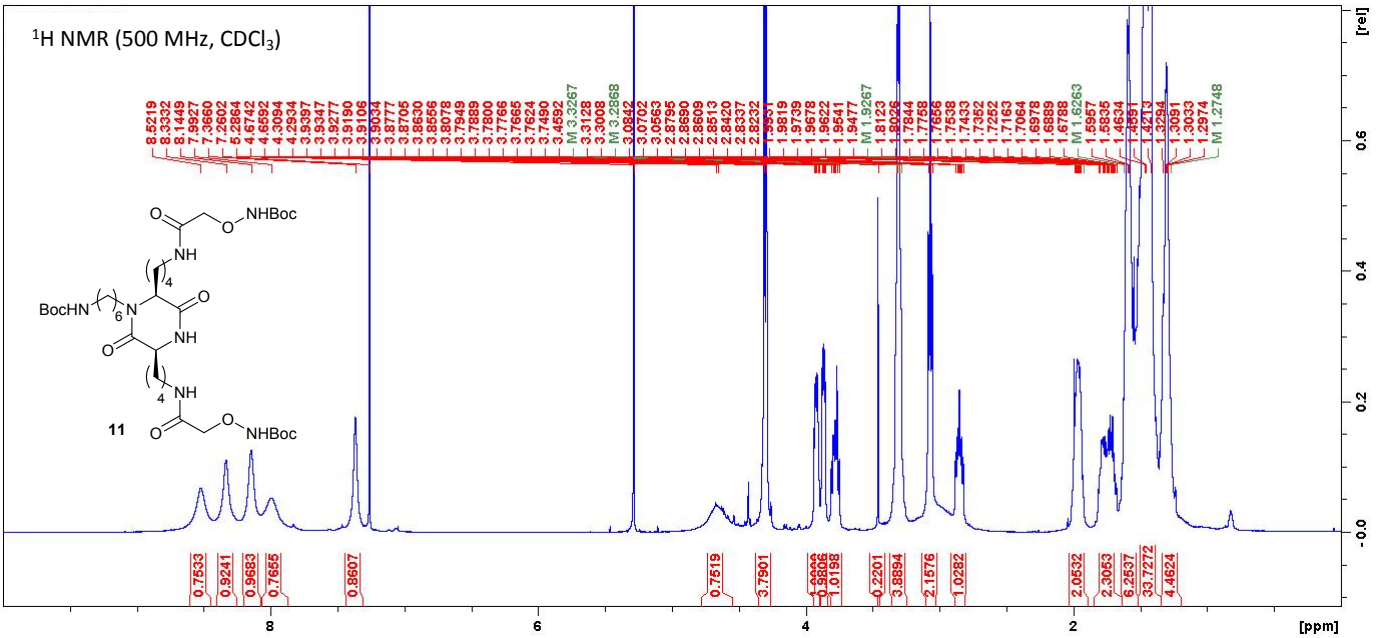
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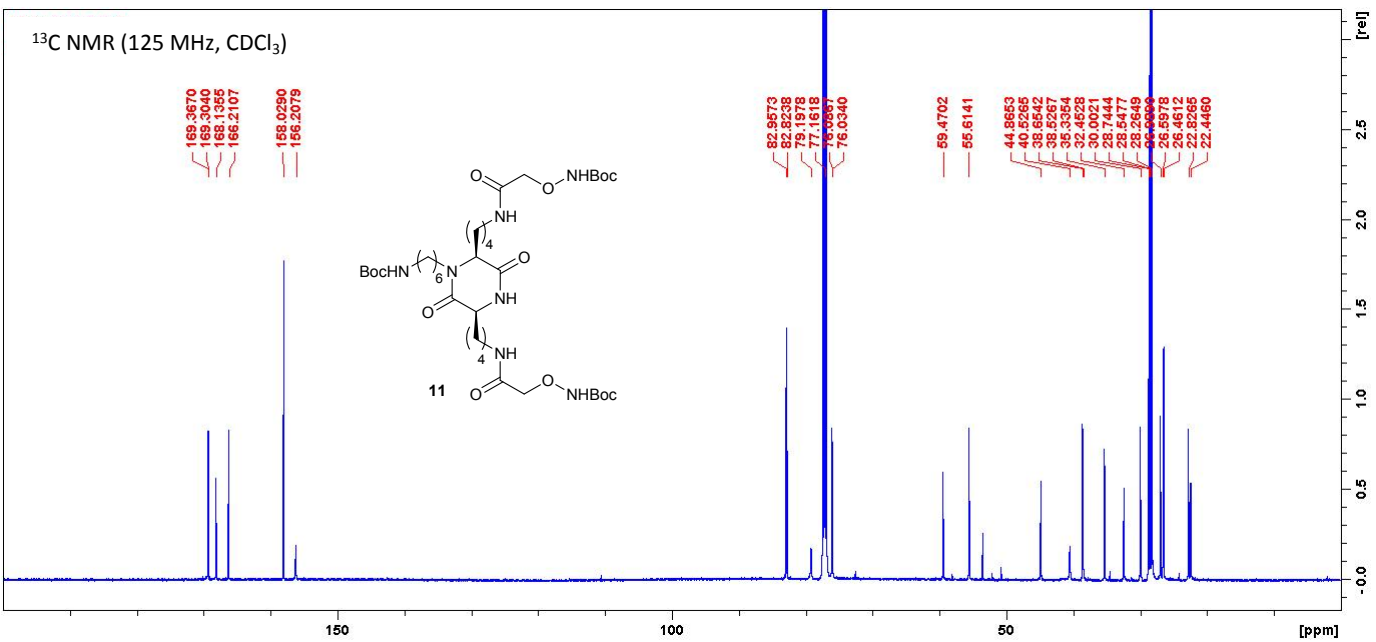
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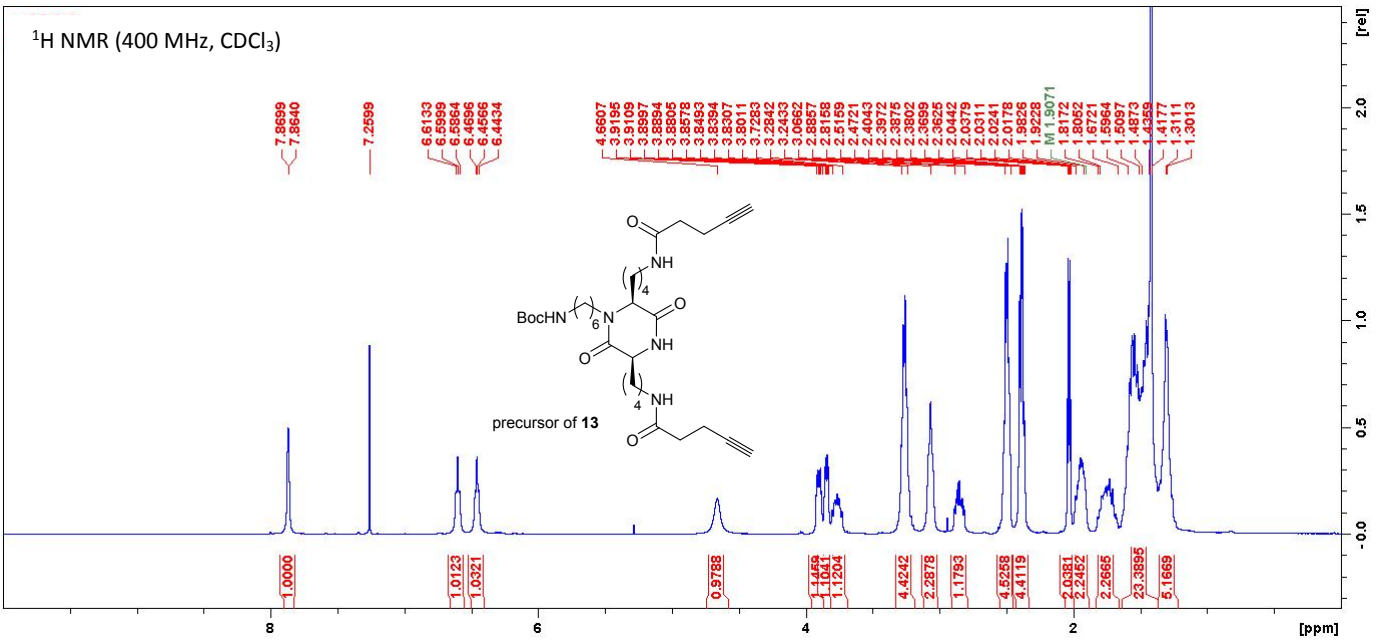
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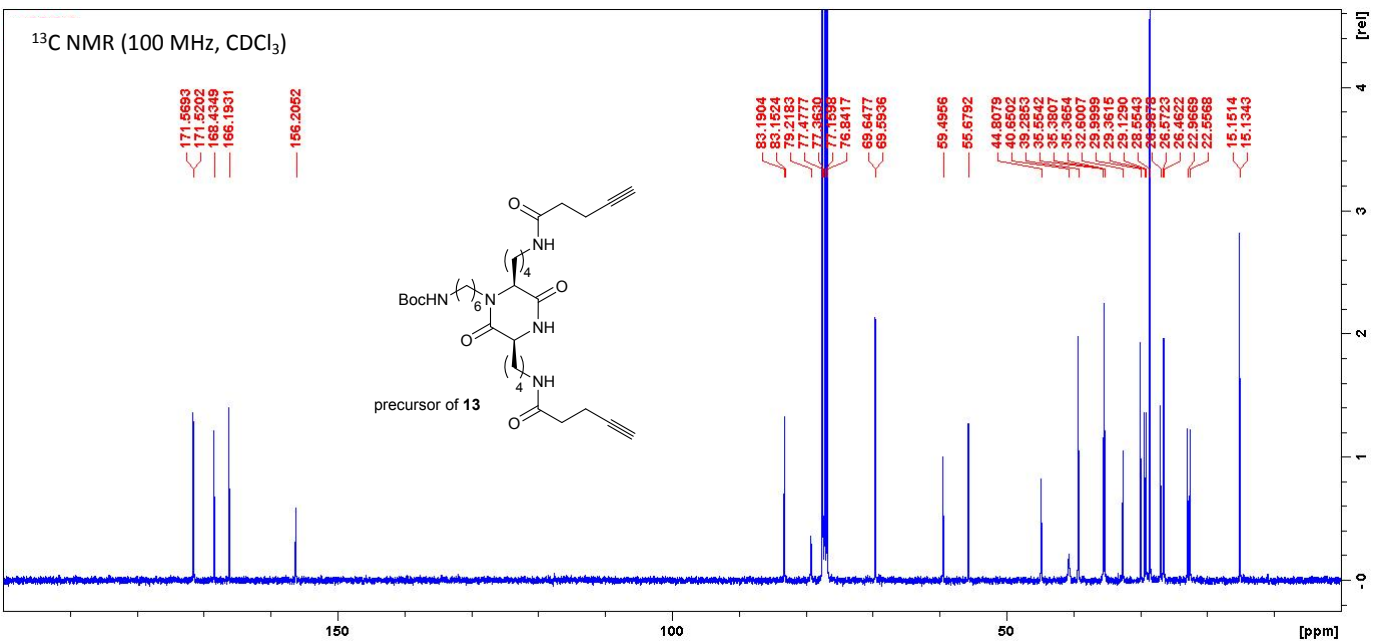
[rel]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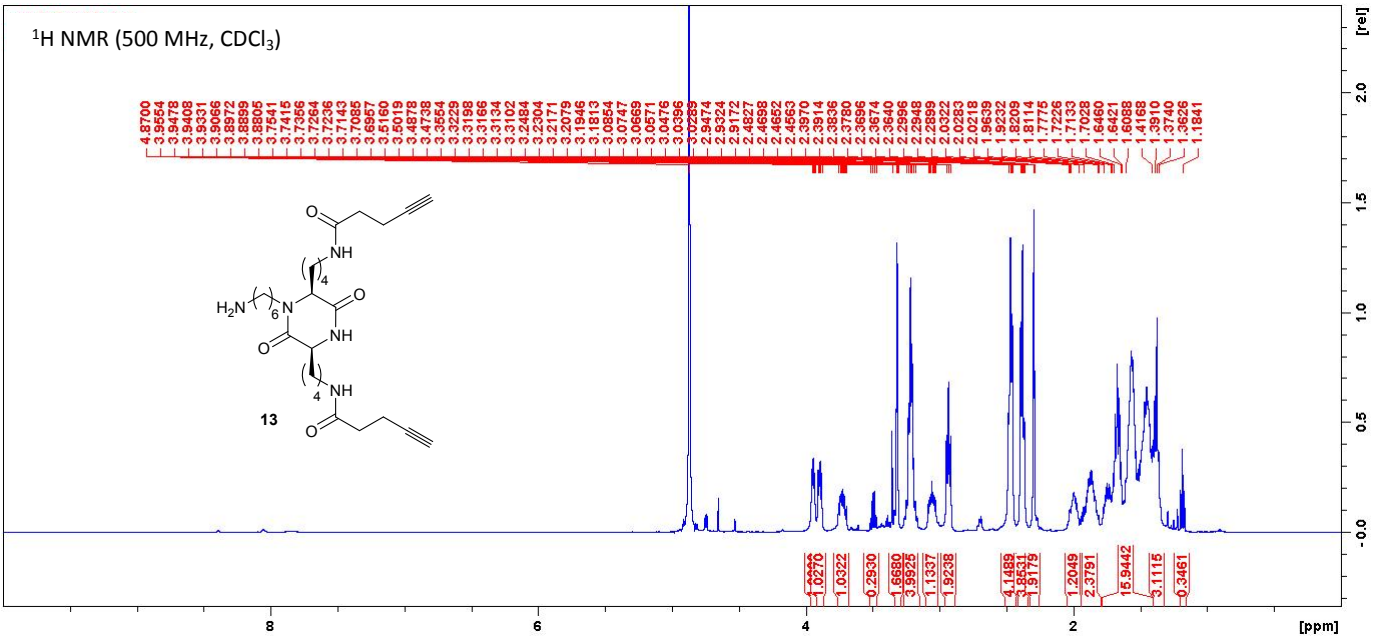
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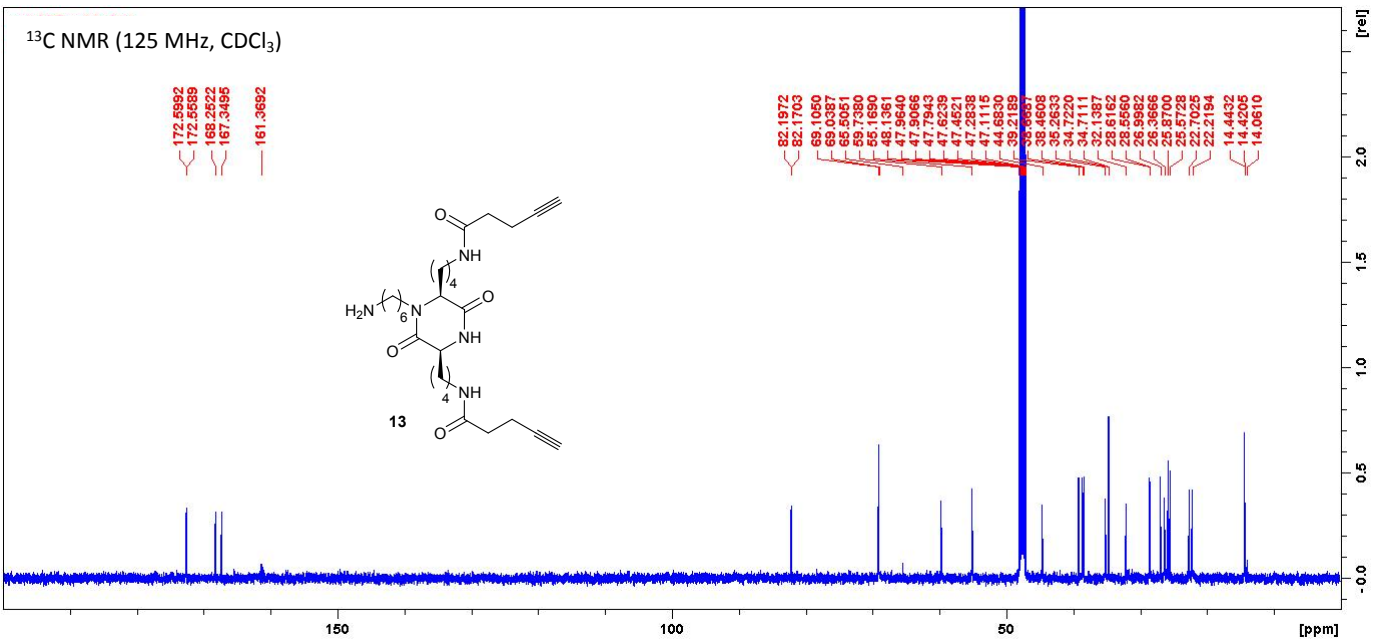
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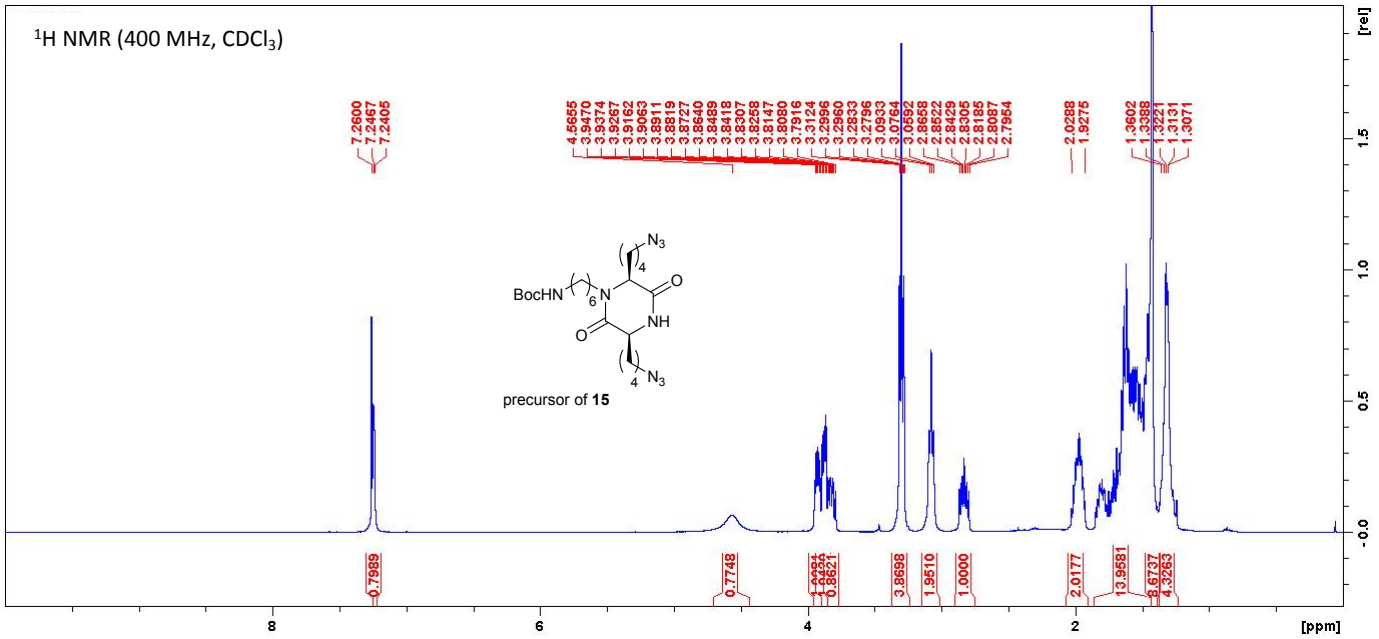
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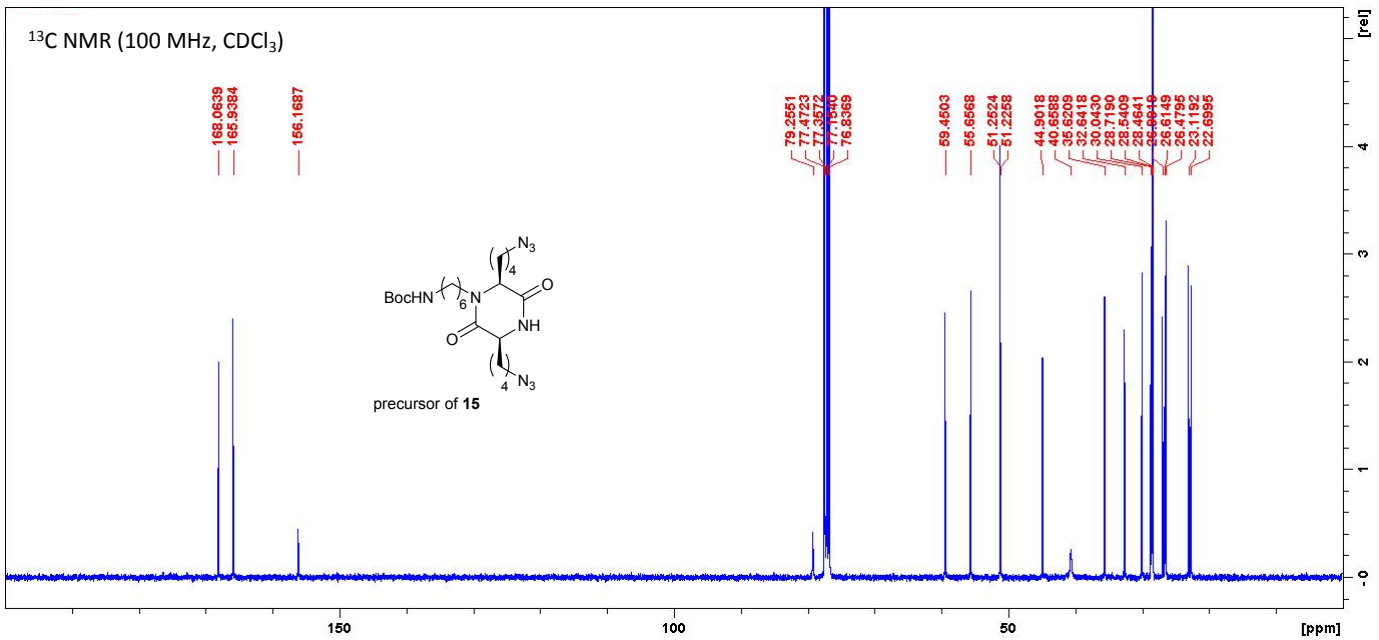
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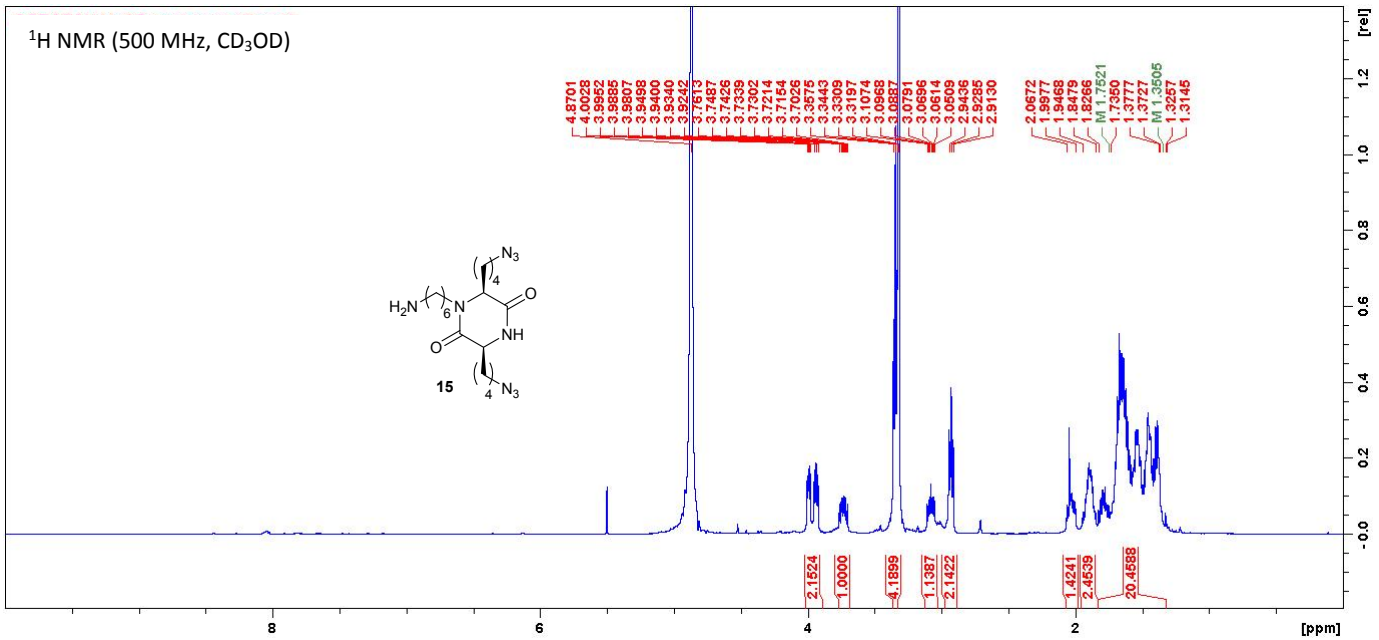
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3.9242
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3.7302
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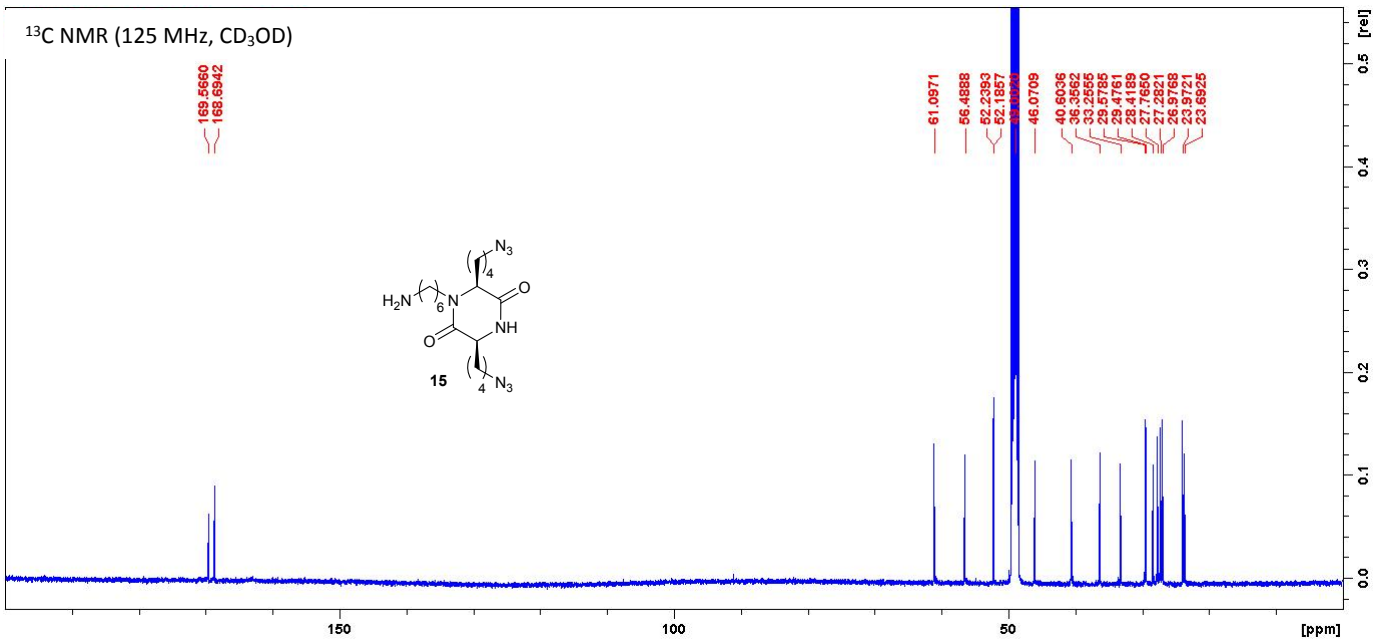
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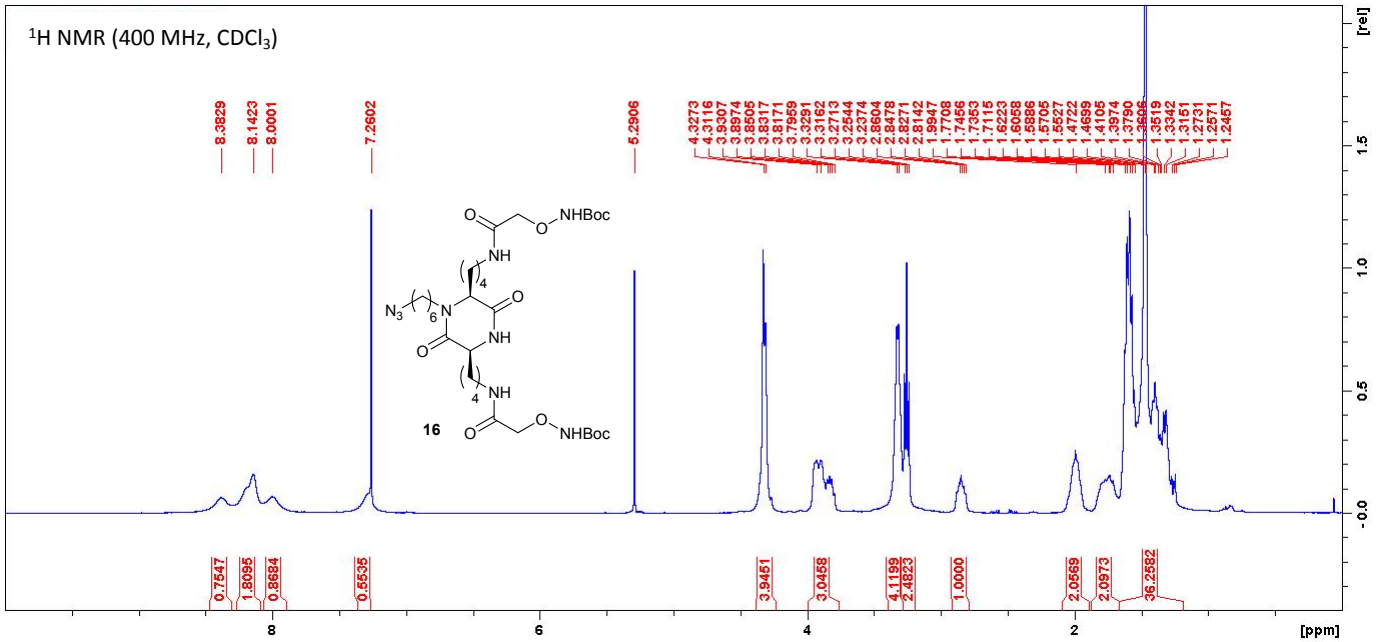
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1.2457
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O
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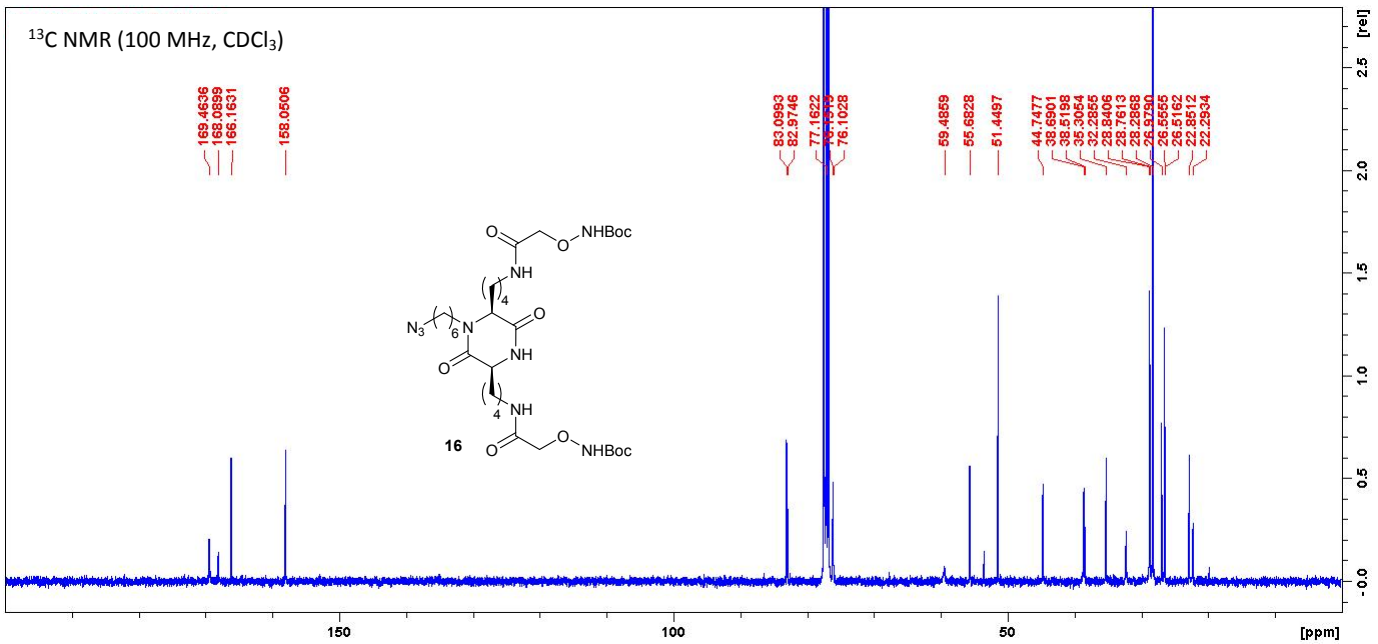
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28.2868
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26.5555
26.5162
22.8512
22.2934
NHBoc
NH
N3
N
O
16
[rel]
[ppm]

# S3. Copies of MS and HRMS spectra

Compound **17**, HRMS (ESI)

Fédération de Recherche Physique et Chimie du Vivant (FR2708 : CBM/ICOA)
Plate-forme de Spectrométrie de Masse Haute Résolution

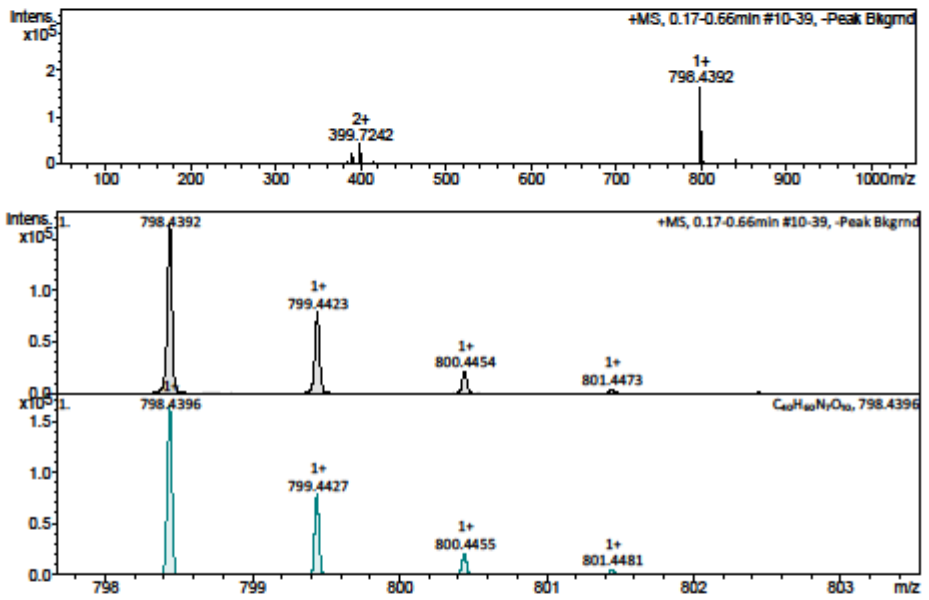
**HRMS**

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | OBC17 | Acquisition Date | 16/04/2014 21:56:12 |
| Analysis Name | X013762CYC_ACN.d | Laboratory | |
| Method | Positif.m | Instrument / Ser# | maXis 255552.00086 |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.6 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 7.0 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 1000.0 Vpp | Set Divert Valve | Waste |




| Meas. m/z | z | # | Ion Formula | m/z | err [ppm] | mSigma | rdb | e¯ Conf |
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| | 2+ | 2 | C41H55N11O5 | 390.718833 | -0.2 | 15.0 | 20.0 | even |
| | 2+ | 3 | C44H63NO11 | 390.719507 | 1.5 | 15.1 | 14.0 | even |
| | 2+ | 4 | C42H51N15O | 390.719502 | 1.5 | 26.8 | 25.0 | even |
| | 2+ | 5 | C57H55N3 | 390.719251 | -0.9 | 83.7 | 32.0 | even |
| 399.724227 | 2+ | 1 | C40H61N7O10 | 399.723447 | 2.0 | 3.6 | 14.0 | even |
| | 2+ | 2 | C41H57N11O6 | 399.724116 | 0.3 | 13.2 | 19.0 | even |
| | 2+ | 3 | C44H65NO12 | 399.724790 | -1.4 | 13.9 | 13.0 | even |
| | 2+ | 4 | C38H49N21 | 399.723442 | -2.0 | 16.7 | 25.0 | even |
| | 2+ | 5 | C42H53N15O2 | 399.724784 | -1.4 | 26.4 | 24.0 | even |
| | 2+ | 6 | C57H57N3O | 399.724533 | 0.8 | 82.7 | 31.0 | even |
| 780.428893 | 1+ | 1 | C41H54N11O5 | 780.430390 | 1.9 | 12.9 | 20.5 | even |
| | 1+ | 2 | C40H58N7O9 | 780.429053 | -0.2 | 18.3 | 15.5 | even |
| | 1+ | 3 | C37H50N17O3 | 780.427705 | 1.5 | 20.8 | 21.5 | even |
| | 1+ | 4 | C39H62N3O13 | 780.427715 | 1.5 | 27.5 | 10.5 | even |
| 798.439181 | 1+ | 1 | C40H60N7O10 | 798.439618 | 0.5 | 2.6 | 14.5 | even |

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printed: 16/04/2014 23:12:03
Page 1 of 2

Compound **17**, Mass spectra (ESI)

# Display Report

**Analysis Info**

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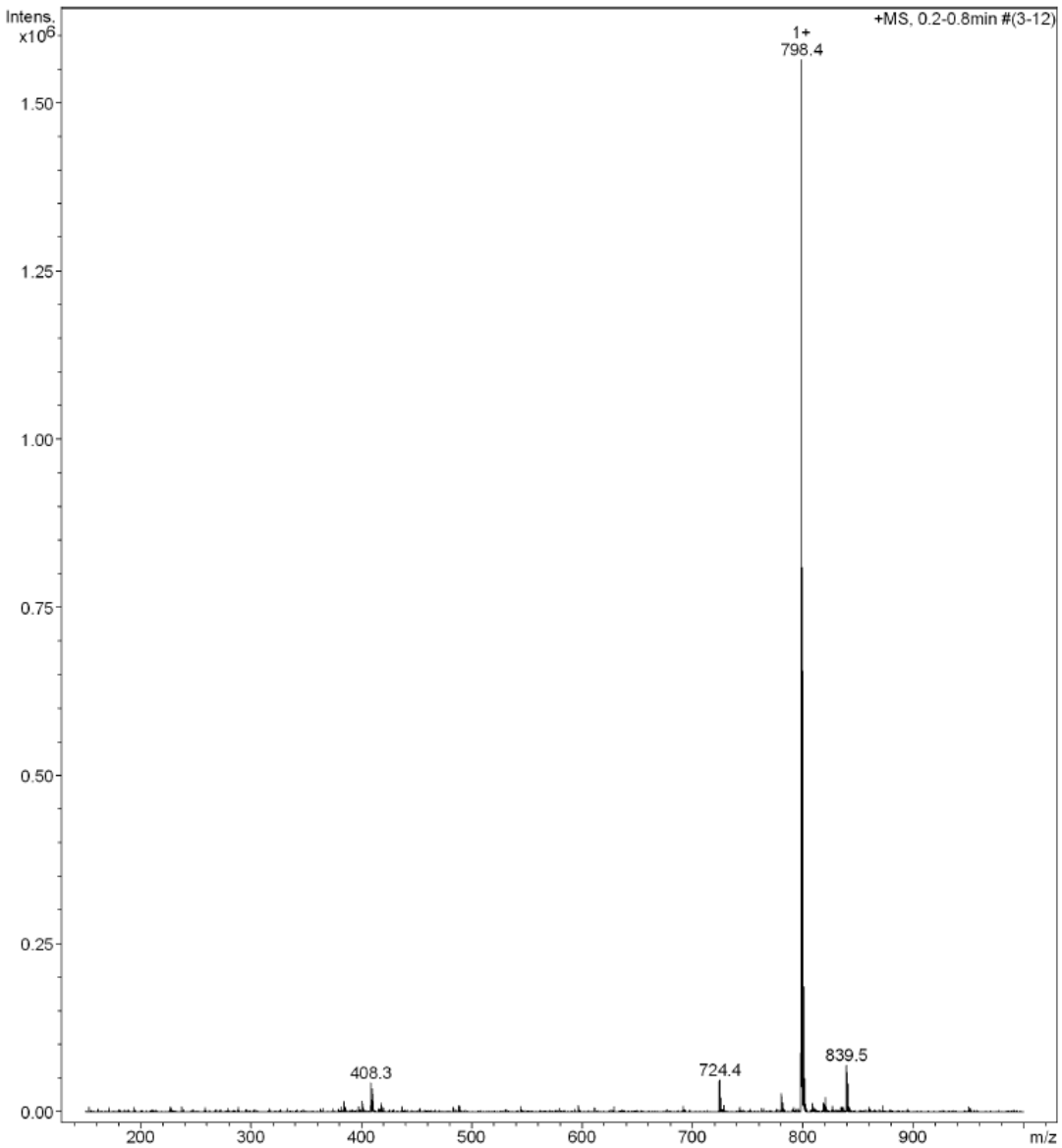
| Analysis Name | D:\Data\I2BM\E_14105_12.d | Acquisition Date | 4/15/2014 4:54:23 PM |
| Method | peptide_mw_1800_2500.m | Operator | Rodolphe |
| Sample Name | chierici | Instrument | esquire3000 plus |
| Comment | OBC17(779)<br>dilue 1/100 eau/acn/hcooh | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Ion Source Type | ESI | Ion Polarity | Positive | Alternating Ion Polarity | off |
| Mass Range Mode | Std/Enhanced | Scan Begin | 150 m/z | Scan End | 1000 m/z |
| Capillary Exit | 136.0 Volt | Skim 1 | 40.0 Volt | Trap Drive | 83.1 |
| Accumulation Time | 511 µs | Averages | 20 Spectra | Auto MS/MS | off |




Bruker Daltonics DataAnalysis 3.3 printed: 4/15/2014 4:57:00 PM Page 1 of 1

Compound **18**, HRMS (ESI)

Fédération de Recherche Physique et Chimie du Vivant (FR2708 : CBM/ICOA)
Plate-forme de Spectrométrie de Masse Haute Résolution

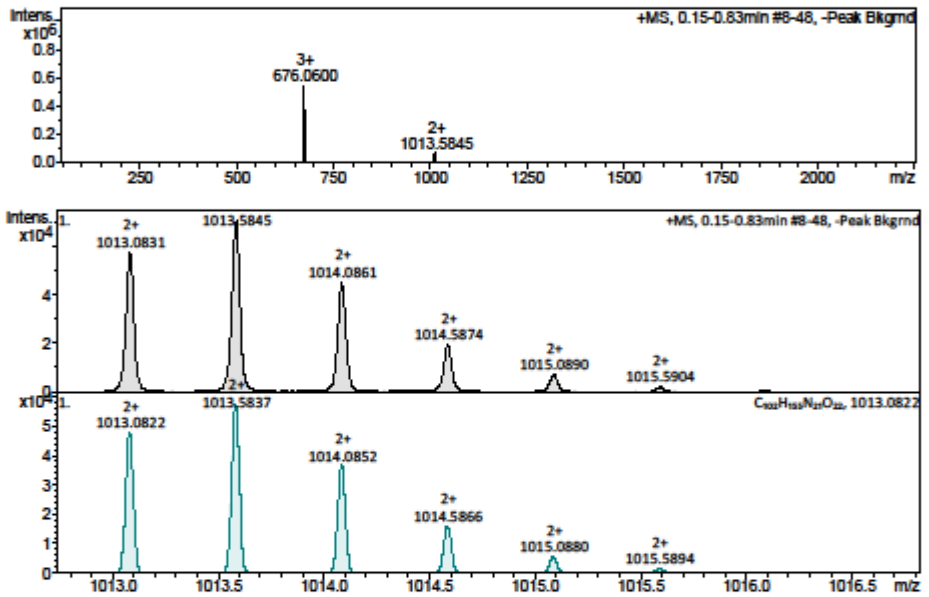
**HRMS**

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | **OBC18** | Acquisition Date | 16/04/2014 15:55:02 |
| Analysis Name | X013759CYC.d | Laboratory | |
| Method | Positif.m | Instrument / Ser# | maXis 255552.00086 |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.6 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 7.0 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 1000.0 Vpp | Set Divert Valve | Waste |




| Meas. m/z | z | # | Ion Formula | m/z | err [ppm] | mSigma | rdb | e¯ Conf |
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| | 3+ | 2 | C107H156N19O20 | 675.725253 | 0.6 | 27.0 | 39.5 | even |
| | 3+ | 3 | C100H160N19O25 | 675.727210 | -2.3 | 27.5 | 30.5 | even |
| | 3+ | 4 | C103H152N25O18 | 675.724357 | -1.9 | 28.3 | 40.5 | even |
| 1013.083140 | 2+ | 1 | C103H151N25O18 | 1013.082898 | 0.2 | 6.2 | 41.0 | even |
| | 2+ | 2 | C102H155N21O22 | 1013.082229 | -0.9 | 6.8 | 36.0 | even |
| | 2+ | 3 | C101H159N17O26 | 1013.081561 | -1.6 | 11.6 | 31.0 | even |
| | 2+ | 4 | C107H155N19O20 | 1013.084241 | -1.1 | 12.3 | 40.0 | even |

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Compound **18**, Mass spectra (ESI)

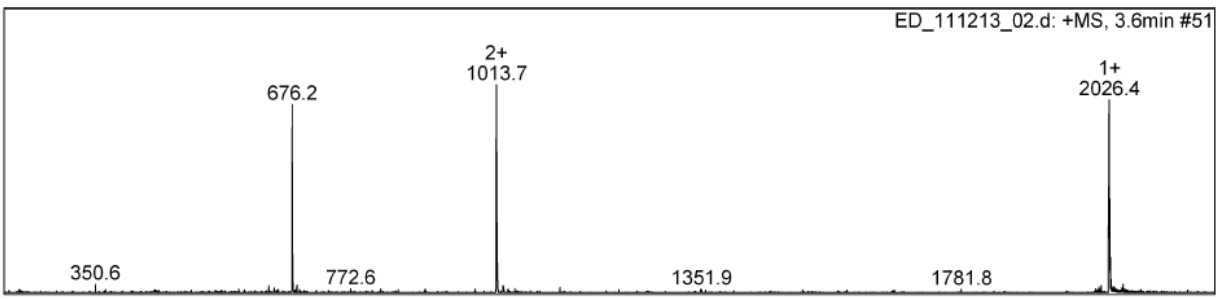
# Mass Spectrum Deconvolution Report

**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\I2BM\ED_111213_02.d | Acquisition Date | 12/13/2011 7:14:50 PM |
| Method | Standby.m | Operator | Rodolphe |
| Sample Name | emilie dufour | Instrument | esquire3000 plus |
| Comment | edufB39 tr=15.9 | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Ion Source Type | ESI | Ion Polarity | Positive | Alternating Ion Polarity | off |
| Mass Range Mode | Std/Normal | Scan Begin | 200 m/z | Scan End | 2200 m/z |
| Capillary Exit | 151.0 Volt | Skim 1 | 40.0 Volt | Trap Drive | 101.1 |
| Accumulation Time | 42 µs | Averages | 20 Spectra | Auto MS/MS | off |




| Component | Molecular Mass | Molecule | | Absolute Abundance | Relative Abundance |
|---|---|---|---|---|---|
| A | 2025.4 | 2026.7 | [M + H]+ | 5242157 | 100.00 |

Component A Detail

| Actual Peak | Charge | Isotopic Mass([M + H]+) | Predicted Peak |
|---|---|---|---|
| 1013.3 | 2+ | 2025.5 | 1013.2 |
| 1013.7 | 2+ | 2026.4 | 1013.7 |
| 1014.2 | 2+ | 2027.3 | 1014.2 |
| 1014.7 | 2+ | 2028.3 | 1014.7 |
| 1015.2 | 2+ | 2029.4 | 1015.2 |
| 1015.6 | 2+ | 2030.3 | 1015.7 |
| 1016.2 | 2+ | 2031.4 | 1016.2 |
| 1016.6 | 2+ | 2032.2 | 1016.7 |
| 2025.5 | 1+ | 2025.5 | 2025.4 |
| 2026.4 | 1+ | 2026.4 | 2026.4 |
| 2027.3 | 1+ | 2027.3 | 2027.4 |
| 2028.3 | 1+ | 2028.3 | 2028.4 |
| 2029.4 | 1+ | 2029.4 | 2029.4 |

Molecular Mass ([M + H]+): 2025.4 Std. Deviation: 0.0918918
Average Mass ([M + H]+): 2026.7

Compound **20**, HRMS (ESI)

For this analysis, an available solution of 20 at 500 μM in a water/DMSO mixture has been used justifying the background at low mass

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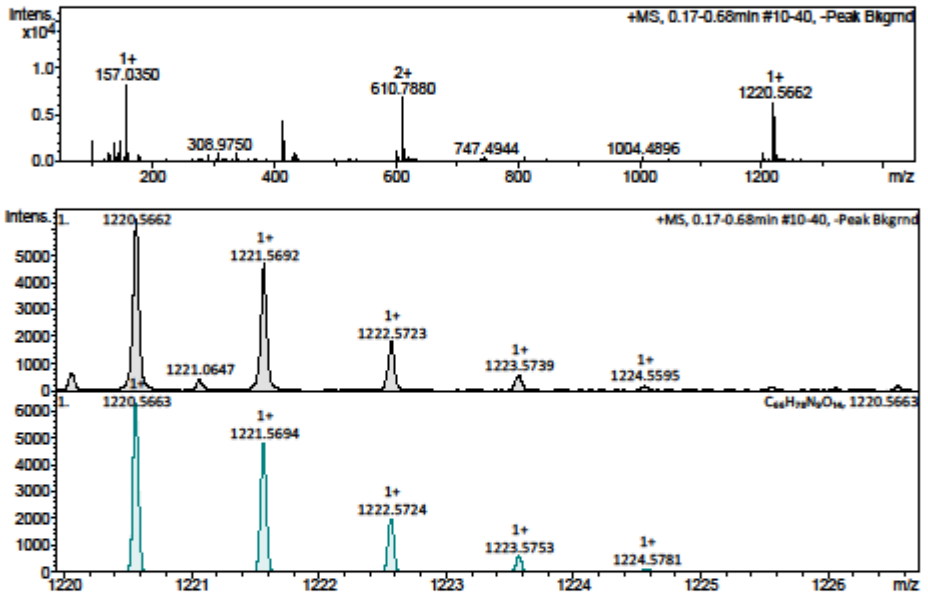
**HRMS**

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | **OBC20** | Acquisition Date | 16/04/2014 21:49:03 |
| Analysis Name | X013761CYC_22971.d | Laboratory | |
| Method | Positif.m | Instrument / Ser# | maXis 255552.00086 |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.6 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 7.0 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 1000.0 Vpp | Set Divert Valve | Waste |




| Meas. m/z | z | # | Ion Formula | m/z | err [ppm] | mSigma | rdb | e¯ Conf |
|---|---|---|---|---|---|---|---|---|
| 610.788045 | 2+ | 1 | C66H79N9O14 | 610.786776 | 2.1 | 5.0 | 32.0 | even |
| | 2+ | 2 | C67H75N13O10 | 610.787444 | -1.0 | 11.1 | 37.0 | even |
| | 2+ | 3 | C70H83N3O16 | 610.788118 | -0.1 | 15.5 | 31.0 | even |
| | 2+ | 4 | C71H79N7O12 | 610.788787 | 1.2 | 24.4 | 36.0 | even |
| | 2+ | 5 | C82H79NO9 | 610.787193 | -1.4 | 75.7 | 44.0 | even |
| | 2+ | 6 | C83H75N5O5 | 610.787862 | 0.3 | 86.4 | 49.0 | even |
| | 2+ | 7 | C84H71N9O | 610.788530 | 0.8 | 97.3 | 54.0 | even |
| 1220.566181 | 1+ | 1 | C65H82N5O18 | 1220.564937 | -1.0 | 8.4 | 27.5 | even |
| | 1+ | 2 | C66H78N9O14 | 1220.566275 | 0.1 | 13.8 | 32.5 | even |
| | 1+ | 3 | C67H74N13O10 | 1220.567612 | 1.2 | 23.0 | 37.5 | even |
| | 1+ | 4 | C78H74N7O7 | 1220.564424 | -1.4 | 73.2 | 45.5 | even |
| | 1+ | 5 | C79H70N11O3 | 1220.565762 | 0.3 | 83.9 | 50.5 | even |
| | 1+ | 6 | C82H78NO9 | 1220.567110 | 0.8 | 87.4 | 44.5 | even |
| | 1+ | 7 | C83H74N5O5 | 1220.568447 | -1.9 | 98.1 | 49.5 | even |

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Compound **20**, Mass spectra (ESI)

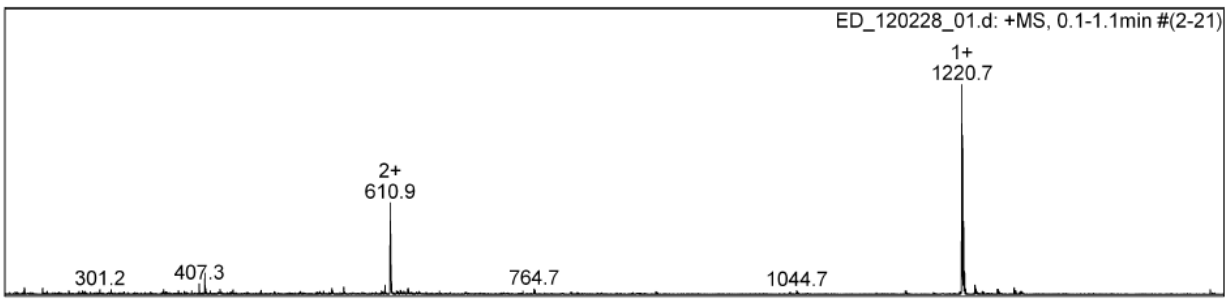
# Mass Spectrum Deconvolution Report

**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\I2BM\ED_120228_01.d | Acquisition Date | 2/28/2012 9:27:47 AM |
| Method | Standby.m | Operator | Laure F |
| Sample Name | emilie dufour | Instrument | esquire3000 plus |
| Comment | edufB70 tr=20.4 | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Ion Source Type | ESI | Ion Polarity | Positive | Alternating Ion Polarity | off |
| Mass Range Mode | Std/Normal | Scan Begin | 200 m/z | Scan End | 1500 m/z |
| Capillary Exit | 163.0 Volt | Skim 1 | 40.0 Volt | Trap Drive | 117.0 |
| Accumulation Time | 860 µs | Averages | 20 Spectra | Auto MS/MS | off |




| Component | Molecular Mass | Molecule | | Absolute Abundance | Relative Abundance |
|---|---|---|---|---|---|
| A | 1220.7 | 1221.6 | [M + H]+ | 955211 | 100.00 |
| B | 413.4 | 413.8 | [M + H]+ | 67748 | 7.09 |

Component A Detail

| Actual Peak | Charge | Isotopic Mass([M + H]+) | Predicted Peak |
|---|---|---|---|
| 610.9 | 2+ | 1220.8 | 610.9 |
| 611.4 | 2+ | 1221.8 | 611.4 |
| 611.9 | 2+ | 1222.8 | 611.9 |
| 612.4 | 2+ | 1223.8 | 612.4 |
| 612.8 | 2+ | 1224.7 | 612.9 |
| 613.4 | 2+ | 1225.8 | 613.4 |
| 1220.7 | 1+ | 1220.7 | 1220.7 |
| 1221.7 | 1+ | 1221.7 | 1221.7 |
| 1222.7 | 1+ | 1222.7 | 1222.7 |
| 1223.7 | 1+ | 1223.7 | 1223.7 |
| 1224.7 | 1+ | 1224.7 | 1224.8 |
| 1225.7 | 1+ | 1225.7 | 1225.8 |
| 1227.0 | 1+ | 1227.0 | 1226.8 |

Molecular Mass ([M + H]+): 1220.7 Std. Deviation: 0.0968051
Average Mass ([M + H]+): 1221.6

Compound **21**, HRMS (ESI)

For this analysis, an available solution of 21 at 500 µM in a water/DMSO mixture has been used justifying the background at low mass

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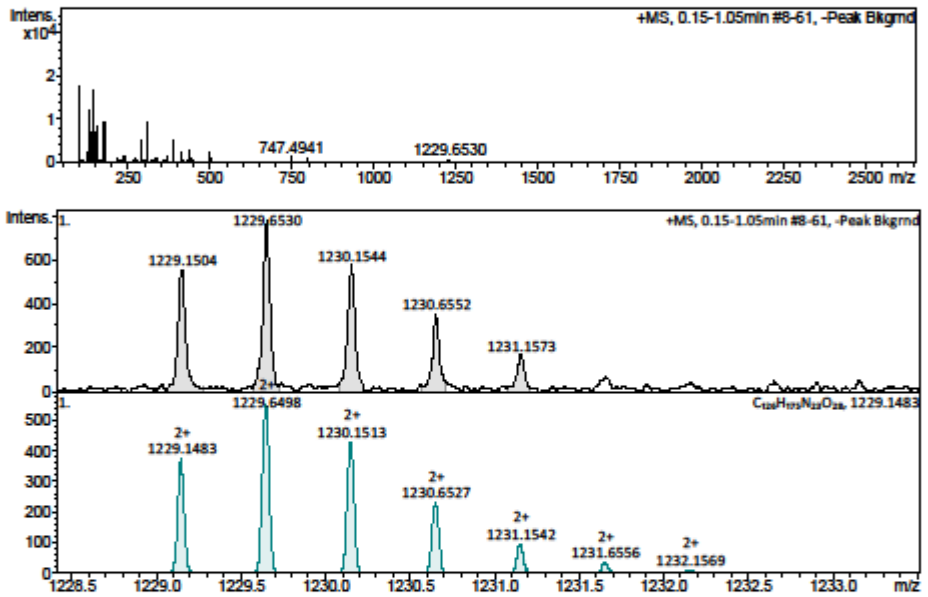
**HRMS**

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | **OBC21** | Acquisition Date | 16/04/2014 22:52:46 |
| Analysis Name | X013760CYC_22983.d | Laboratory | |
| Method | Positif.m | Instrument / Ser# | maXis 255552.00086 |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.6 Bar |
| Focus | Not active | Set Capillary | 4500 V | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 7.0 l/min |
| Scan End | 3000 m/z | Set Collision Cell RF | 1000.0 Vpp | Set Divert Valve | Waste |




| Meas. m/z | z | # | Ion Formula | m/z | err [ppm] | mSigma | rdb | e¯ Conf |
|---|---|---|---|---|---|---|---|---|
| 1229.150439 | 2+ | 1 | C126H175N23O28 | 1229.148297 | -1.7 | 35.0 | 51.0 | even |
| | 2+ | 2 | C127H171N27O24 | 1229.148966 | -1.2 | 36.6 | 56.0 | even |
| | 2+ | 3 | C131H175N21O26 | 1229.150309 | -0.1 | 38.5 | 55.0 | even |

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Compound **21**, Mass spectra (ESI)

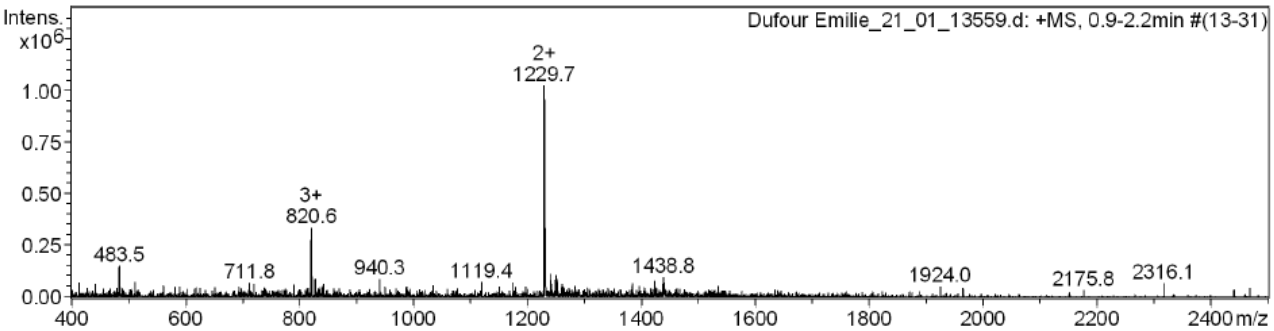
# Mass Spectrum Deconvolution Report

**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\i2bm\Dufour Emilie_21_01_13559.d | Acquisition Date | 6/14/2012 11:28:30 AM |
| Method | 13559.m | Operator | LF |
| Sample Name | Dufour Emilie | Instrument | esquire3000 plus |
| Comment | edufB98 pic2(2459)<br>conc hplc | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Ion Source Type | ESI | Ion Polarity | Positive | Alternating Ion Polarity | off |
| Mass Range Mode | Std/Normal | Scan Begin | 400 m/z | Scan End | 2500 m/z |
| Capillary Exit | 163.0 Volt | Skim 1 | 40.0 Volt | Trap Drive | 122.4 |
| Accumulation Time | 746 µs | Averages | 20 Spectra | Auto MS/MS | off |




| Component | Molecular Mass | Molecule | | Absolute Abundance | Relative Abundance |
|---|---|---|---|---|---|
| A | 2457.4 | 2458.9 | [M + H]+ | 1311460 | 100.00 |

Component A Detail

| Actual Peak | Charge | Isotopic Mass([M + H]+) | Predicted Peak |
|---|---|---|---|
| 820.0 | 3+ | 2458.0 | 820.2 |
| 820.2 | 3+ | 2458.6 | 820.2 |
| 820.6 | 3+ | 2459.7 | 820.5 |
| 1229.2 | 2+ | 2457.4 | 1229.2 |
| 1229.7 | 2+ | 2458.4 | 1229.7 |
| 1230.2 | 2+ | 2459.5 | 1230.2 |
| 1230.7 | 2+ | 2460.5 | 1230.7 |
| 1231.1 | 2+ | 2461.3 | 1231.2 |

Molecular Mass ([M + H]+): 2457.4 Std. Deviation: 0.202472
Average Mass ([M + H]+): 2458.9