

# **New eremophilane derivatives produced by the marine-derived fungus *Emericellopsis maritima* BC17 in liquid culture media**

Jorge R. Virués-Segovia<sup>1,2</sup>, Cristina Pinedo<sup>1,2</sup>, David Zorrilla<sup>3</sup>, Jesús Sánchez-Márquez<sup>3</sup>, Pilar Sánchez<sup>4</sup>, María C. Ramos<sup>4</sup>, Mercedes de la Cruz<sup>4</sup>, Josefina Aleu<sup>1,2,\*</sup>, and Rosa Durán-Patrón<sup>1,2,\*</sup>

<sup>1</sup> *Departamento de Química Orgánica, Facultad de Ciencias, Universidad de Cádiz, Puerto Real, 11510 Cádiz, Spain.*

<sup>2</sup> *Instituto de Investigación en Biomoléculas (INBIO), Universidad de Cádiz, Puerto Real, 11510 Cádiz, Spain.*

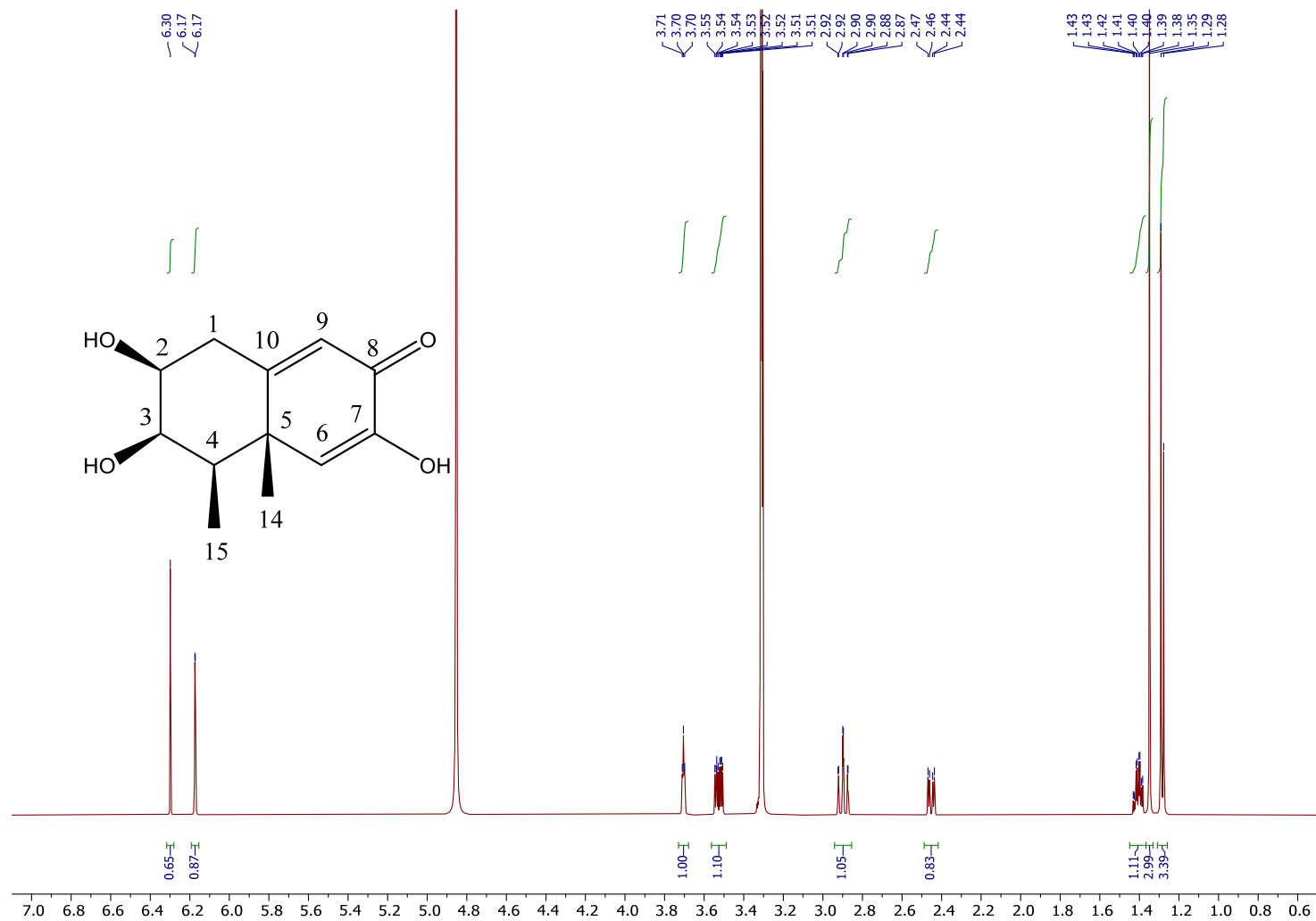
<sup>2</sup> *Departamento de Química Física, Facultad de Ciencias, Universidad de Cádiz, Puerto Real, 11510 Cádiz, Spain.*

<sup>3</sup> *Centro de Excelencia en Investigación de Medicamentos Innovadores en Andalucía, Fundación MEDINA, 18016 Granada, Spain.*

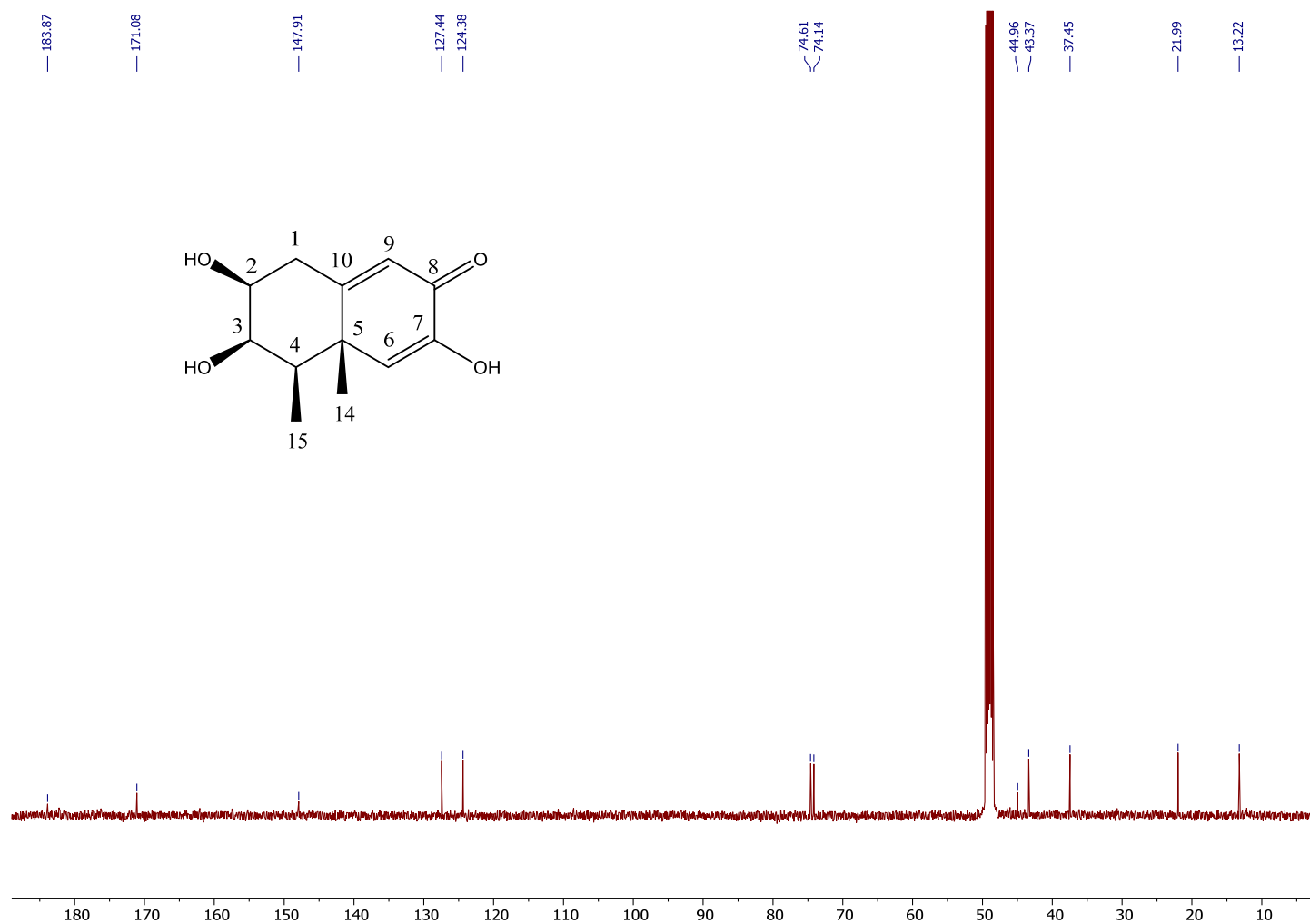
\* Correspondence: [josefina.aleu@uca.es](mailto:josefina.aleu@uca.es) (J.A.), [rosa.duran@uca.es](mailto:rosa.duran@uca.es) (R.D.-P.)

## Table of contents

<b>Figure S1.</b> $^1\text{H}$ NMR spectrum (500 MHz, $\text{CD}_3\text{OD}$ ) of compound <b>1</b> .....	3
<b>Figure S2.</b> $^{13}\text{C}$ NMR spectrum (125 MHz, $\text{CD}_3\text{OD}$ ) of compound <b>1</b> .....	4
<b>Figure S3.</b> gCOSY spectrum of compound <b>1</b> .....	5
<b>Figure S4.</b> gHSQC spectrum of compound <b>1</b> .....	6
<b>Figure S5.</b> gHMBC spectrum of compound <b>1</b> .....	7
<b>Figure S6a-g.</b> 1D NOESY spectra of compound <b>1</b> .....	8
<b>Figure S7.</b> HRESIMS spectrum of compound <b>1</b> .....	15
<b>Figure S8.</b> $^1\text{H}$ NMR spectrum (400 MHz, $\text{CDCl}_3$ ) of compound <b>2</b> .....	16
<b>Figure S9.</b> $^{13}\text{C}$ NMR spectrum (125 MHz, $\text{CDCl}_3$ ) of compound <b>2</b> .....	17
<b>Figure S10.</b> gCOSY spectrum of compound <b>2</b> .....	18
<b>Figure S11.</b> gHSQC spectrum of compound <b>2</b> .....	19
<b>Figure S12.</b> gHMBC spectrum of compound <b>2</b> .....	20
<b>Figure S13a-f.</b> 1D NOESY spectra of compound <b>2</b> .....	21
<b>Figure S14.</b> HRESIMS spectrum of compound <b>2</b> .....	27
<b>Figure S15.</b> $^1\text{H}$ NMR spectrum (500 MHz, $\text{CDCl}_3$ ) of compound <b>3</b> .....	28
<b>Figure S16.</b> $^{13}\text{C}$ NMR spectrum (125 MHz, $\text{CDCl}_3$ ) of compound <b>3</b> .....	29
<b>Figure S17.</b> gCOSY spectrum of compound <b>3</b> .....	30
<b>Figure S18.</b> gHSQC spectrum of compound <b>3</b> .....	31
<b>Figure S19.</b> gHMBC spectrum of compound <b>3</b> .....	32
<b>Figure S20a-e.</b> 1D NOESY spectra of compound <b>3</b> .....	33
<b>Figure S21.</b> HRESIMS spectrum of compound <b>3</b> .....	38

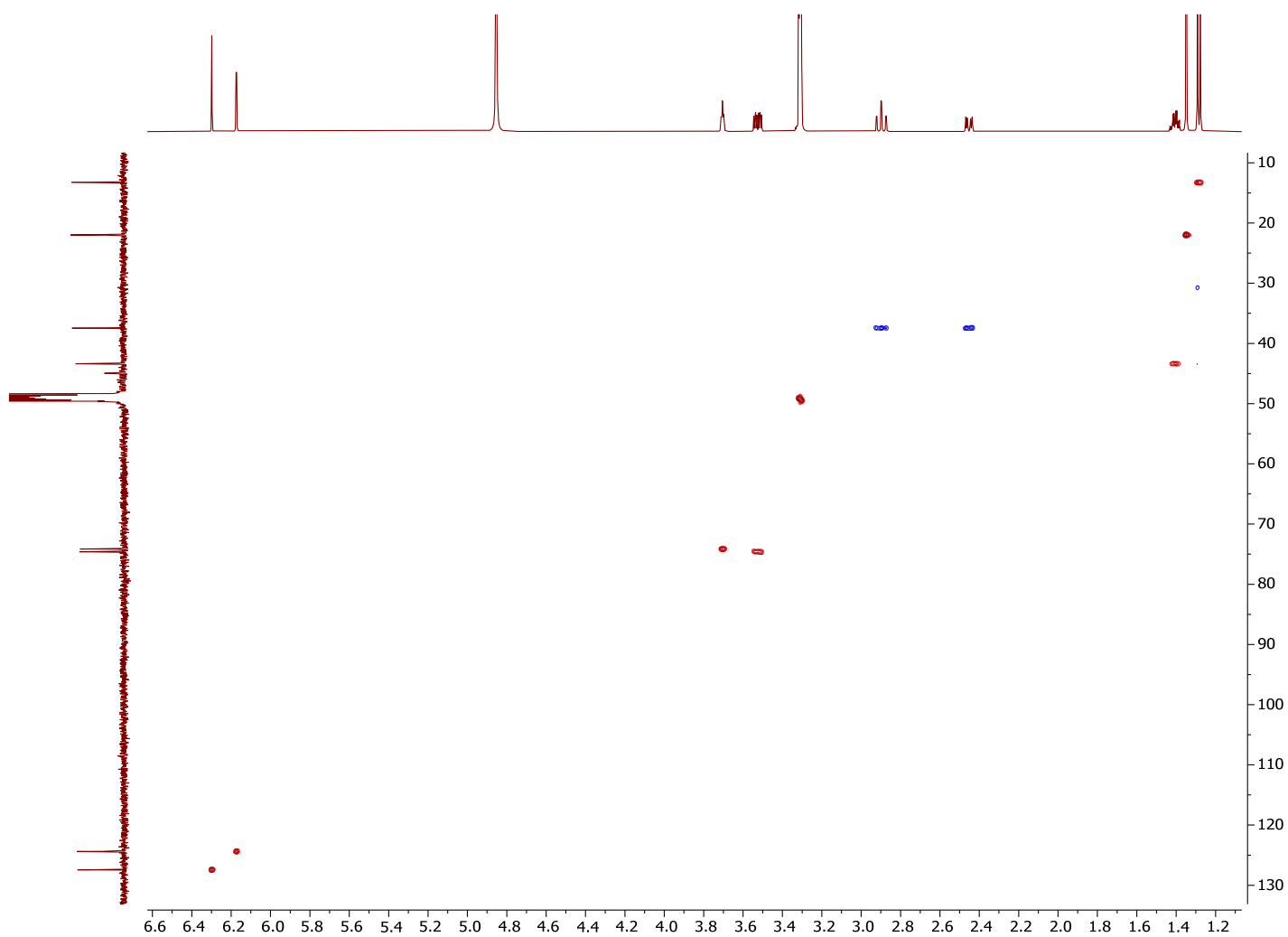


**Figure S1.**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{CD}_3\text{OD}$ ) of compound 1.

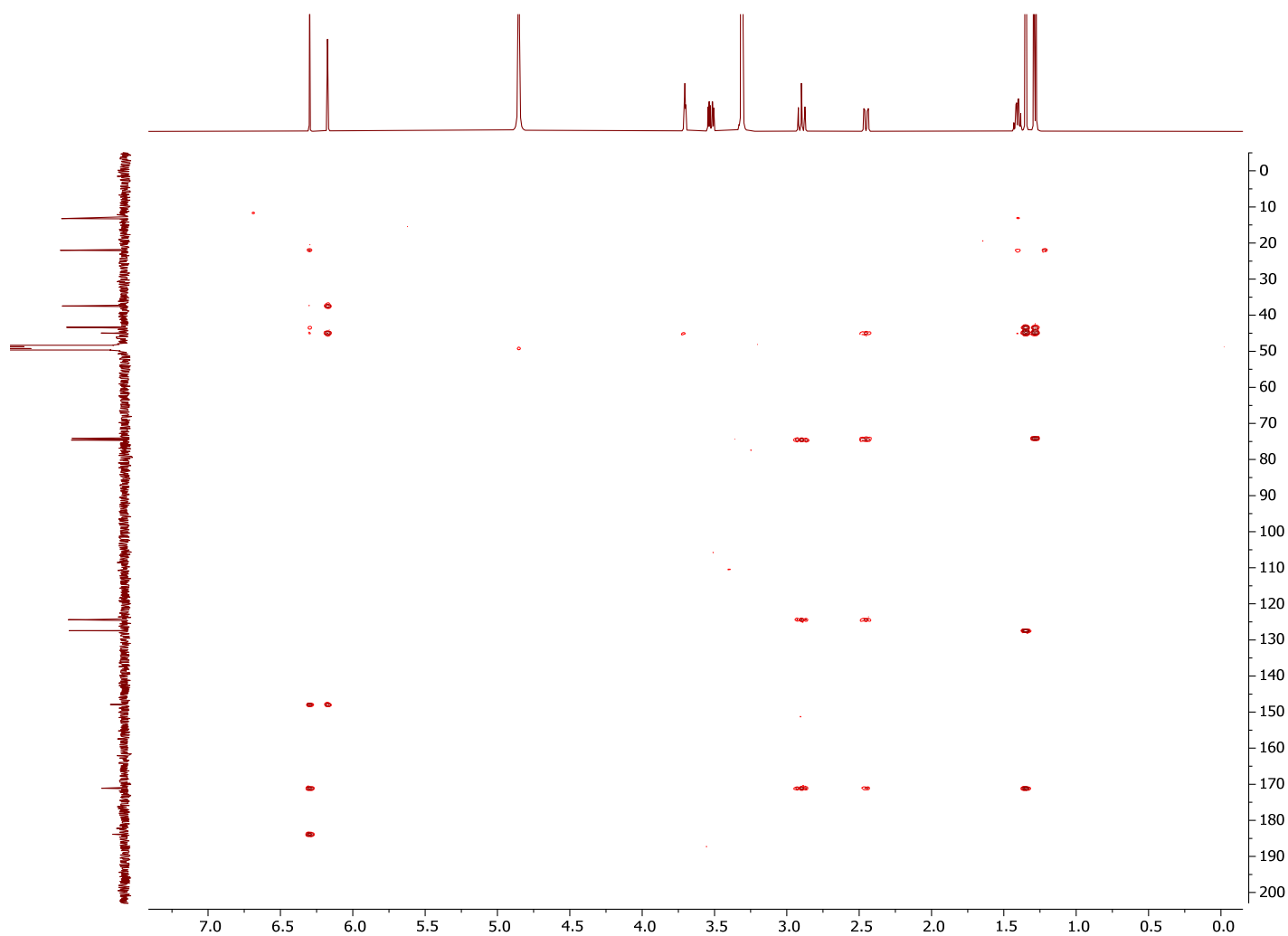


**Figure S2.** <sup>13</sup>C NMR spectrum (125 MHz, CD<sub>3</sub>OD) of compound **1**.

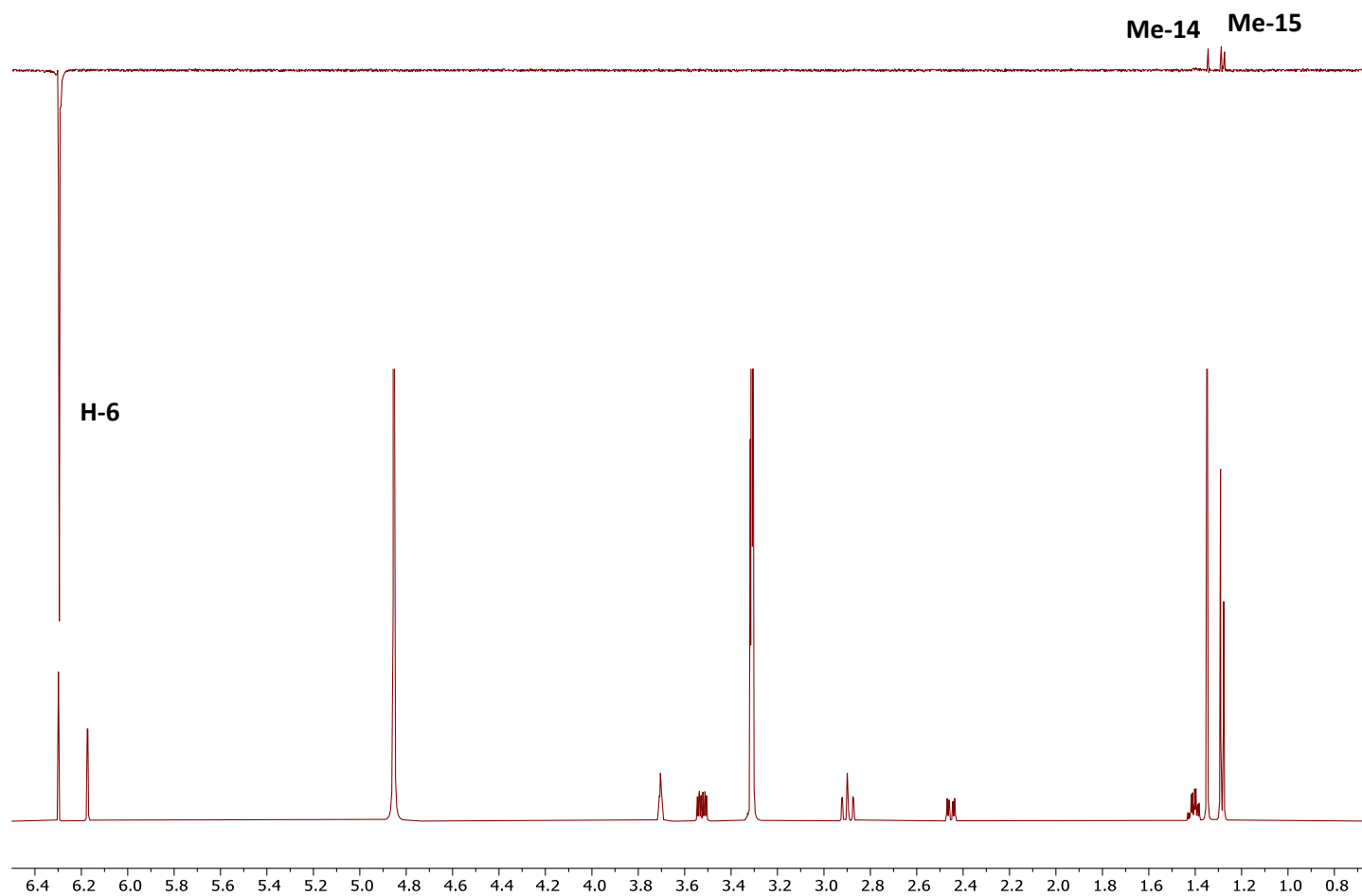




**Figure S4.** gHSQC spectrum of compound **1**.

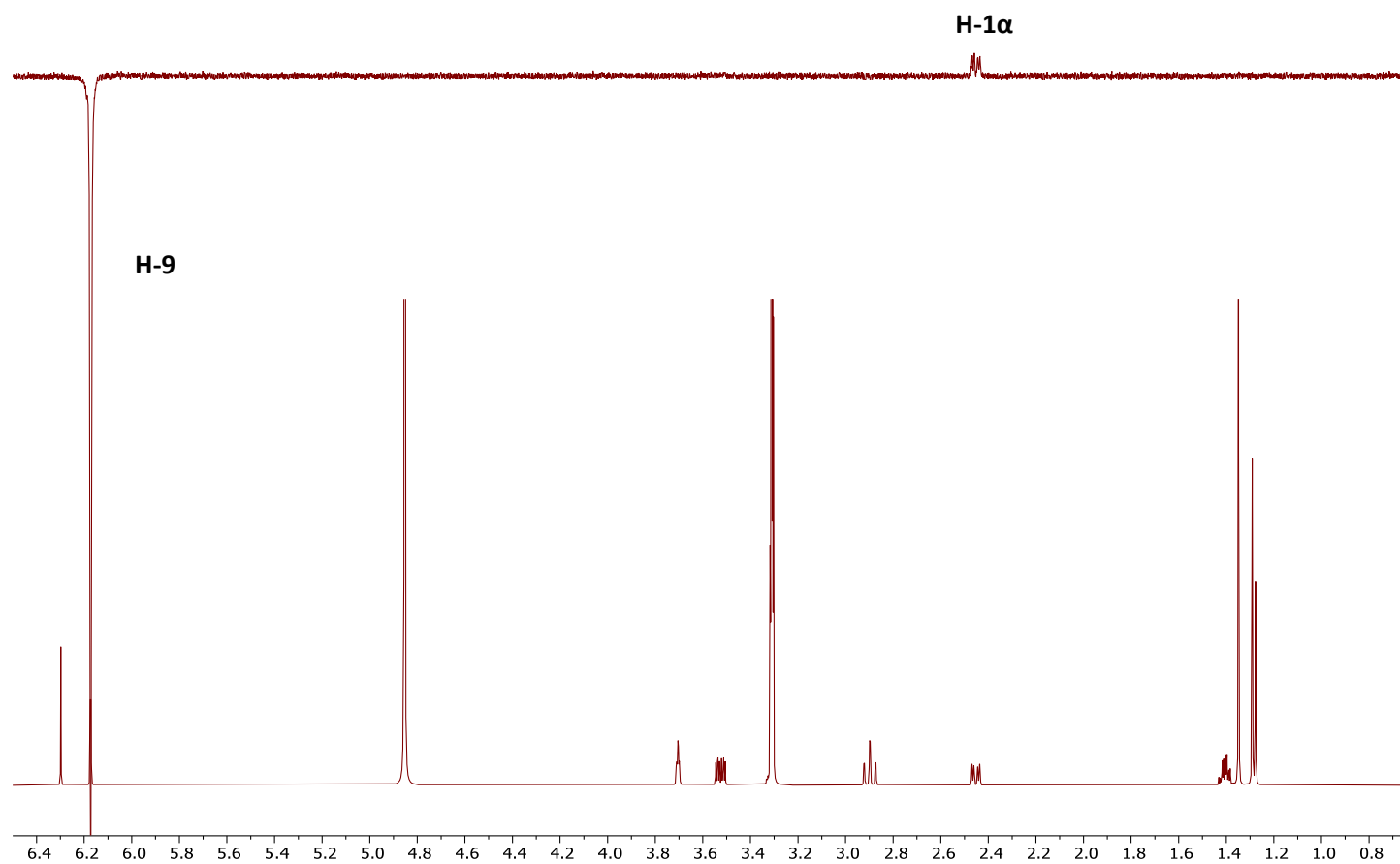


**Figure S5.** gHMBC spectrum of compound **1**.

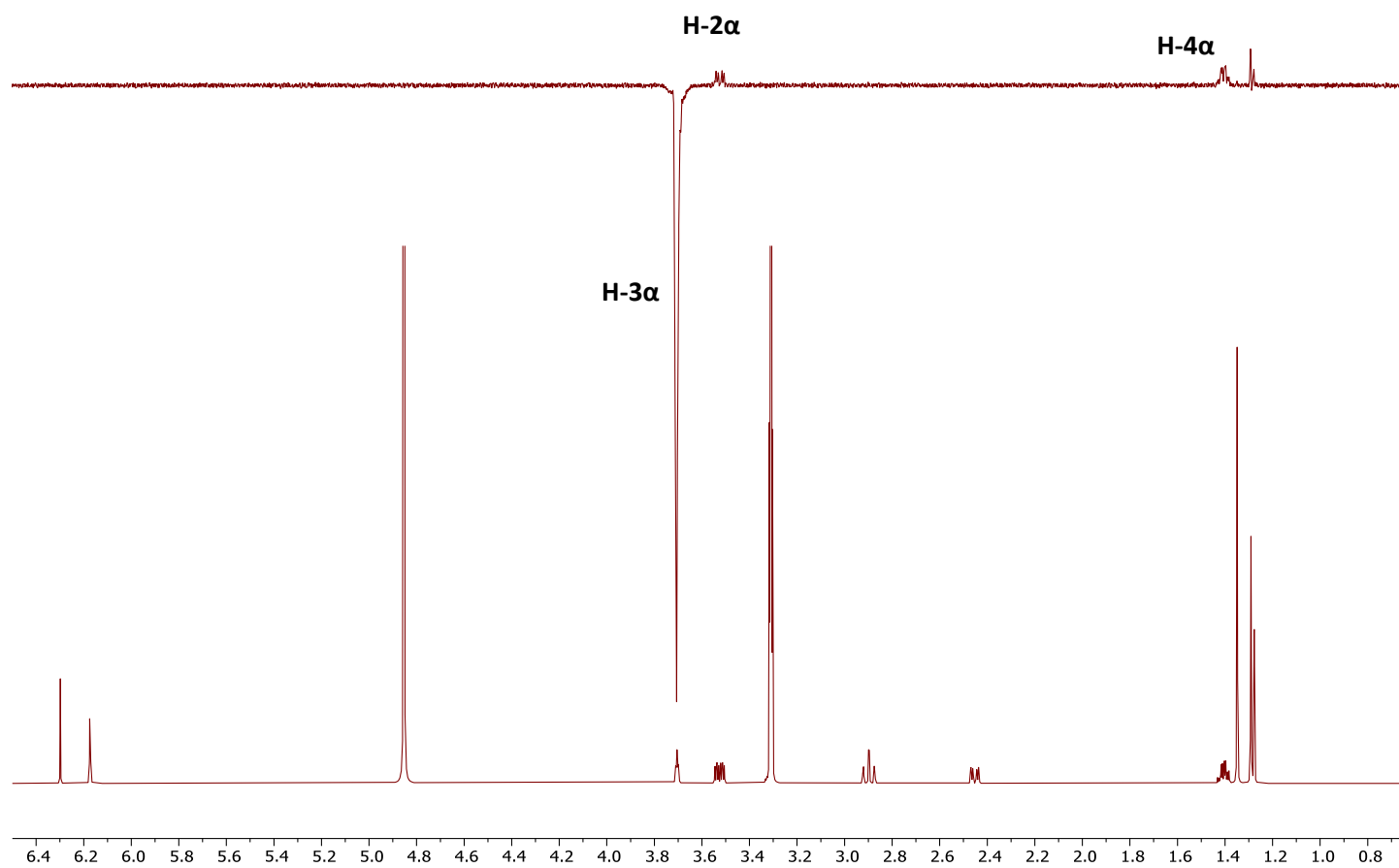


**Figure S6a.** 1D NOESY spectrum of compound **1**.

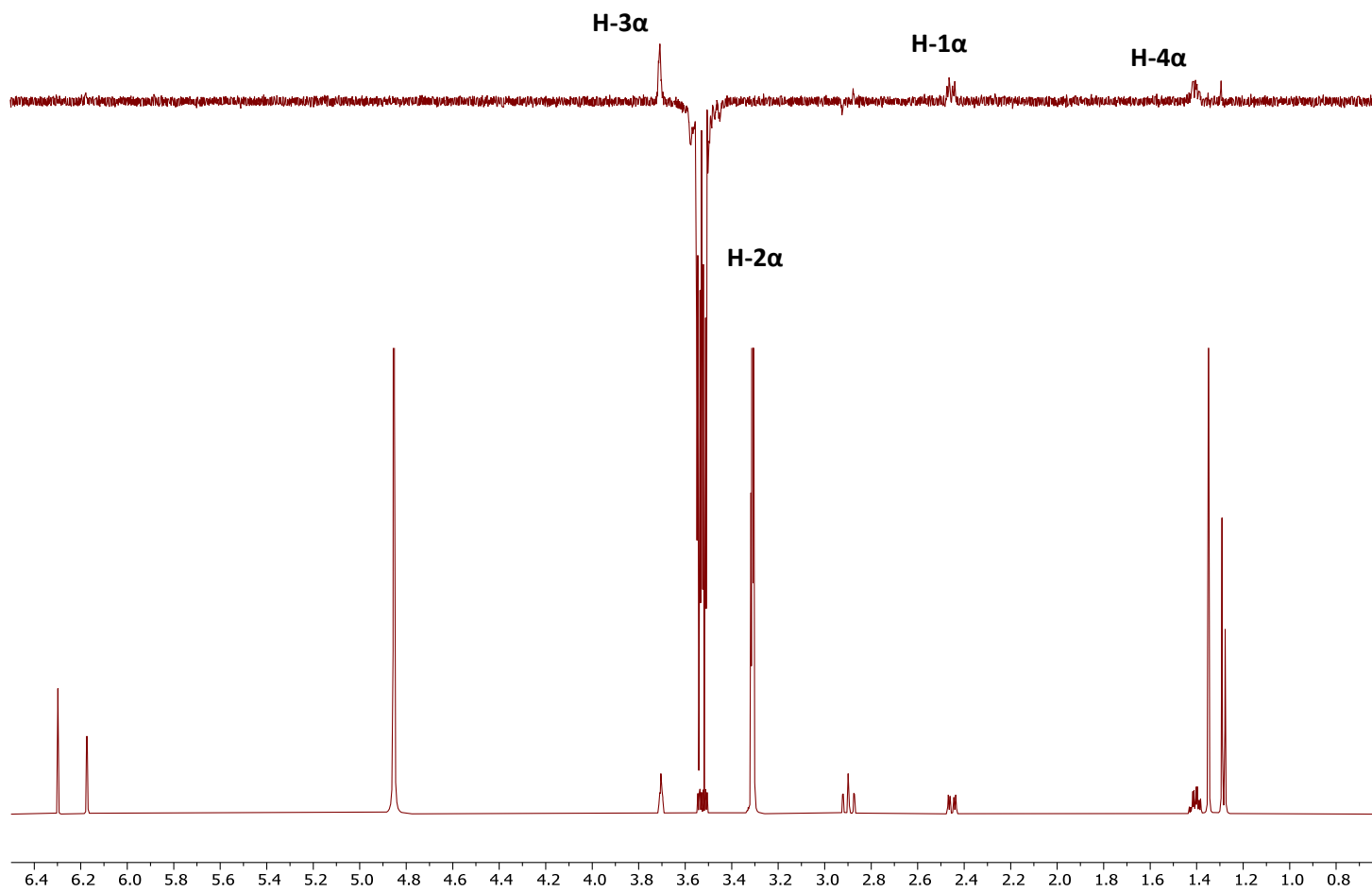




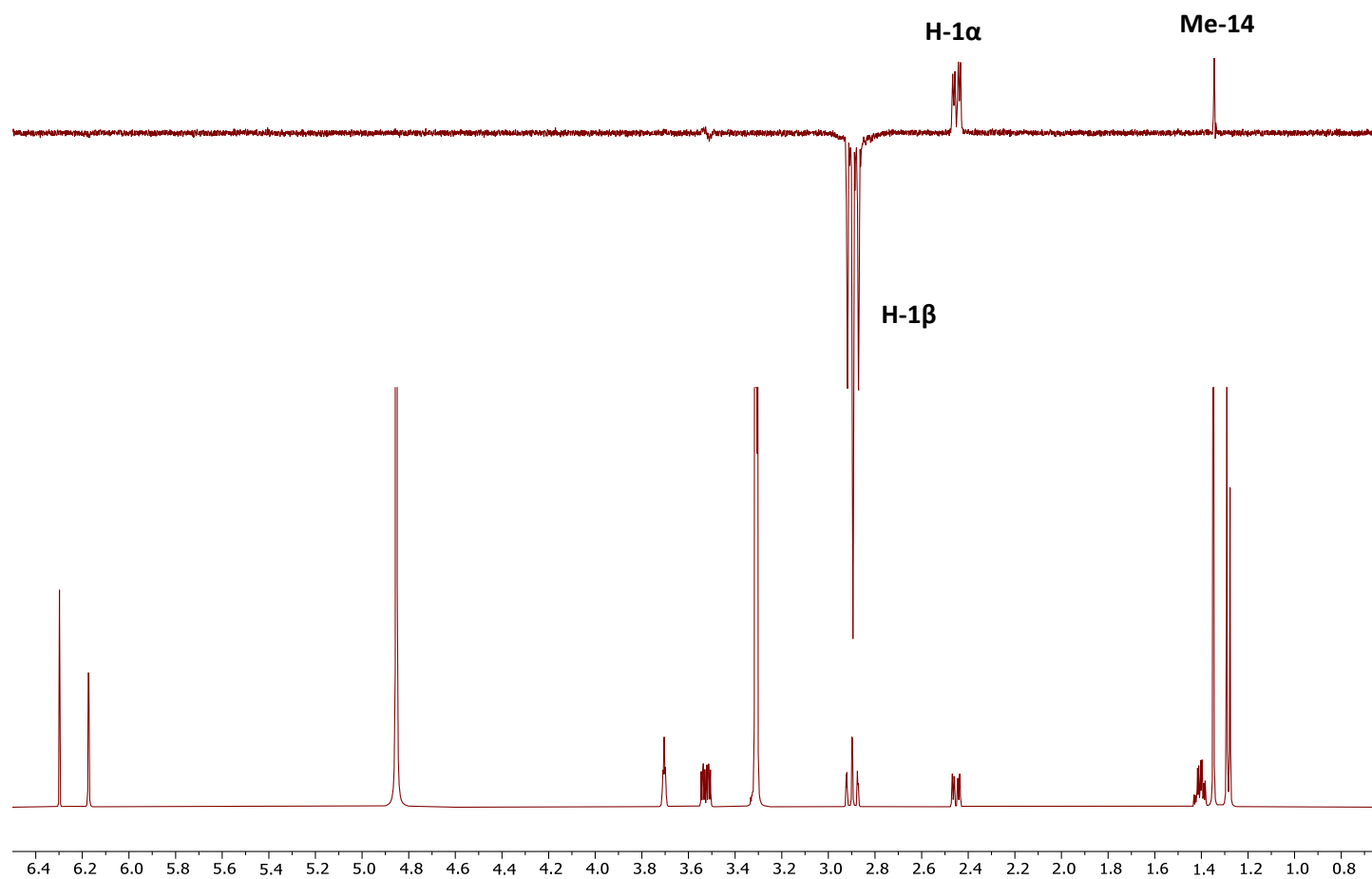
**Figure S6b.** 1D NOESY spectrum of compound **1**.



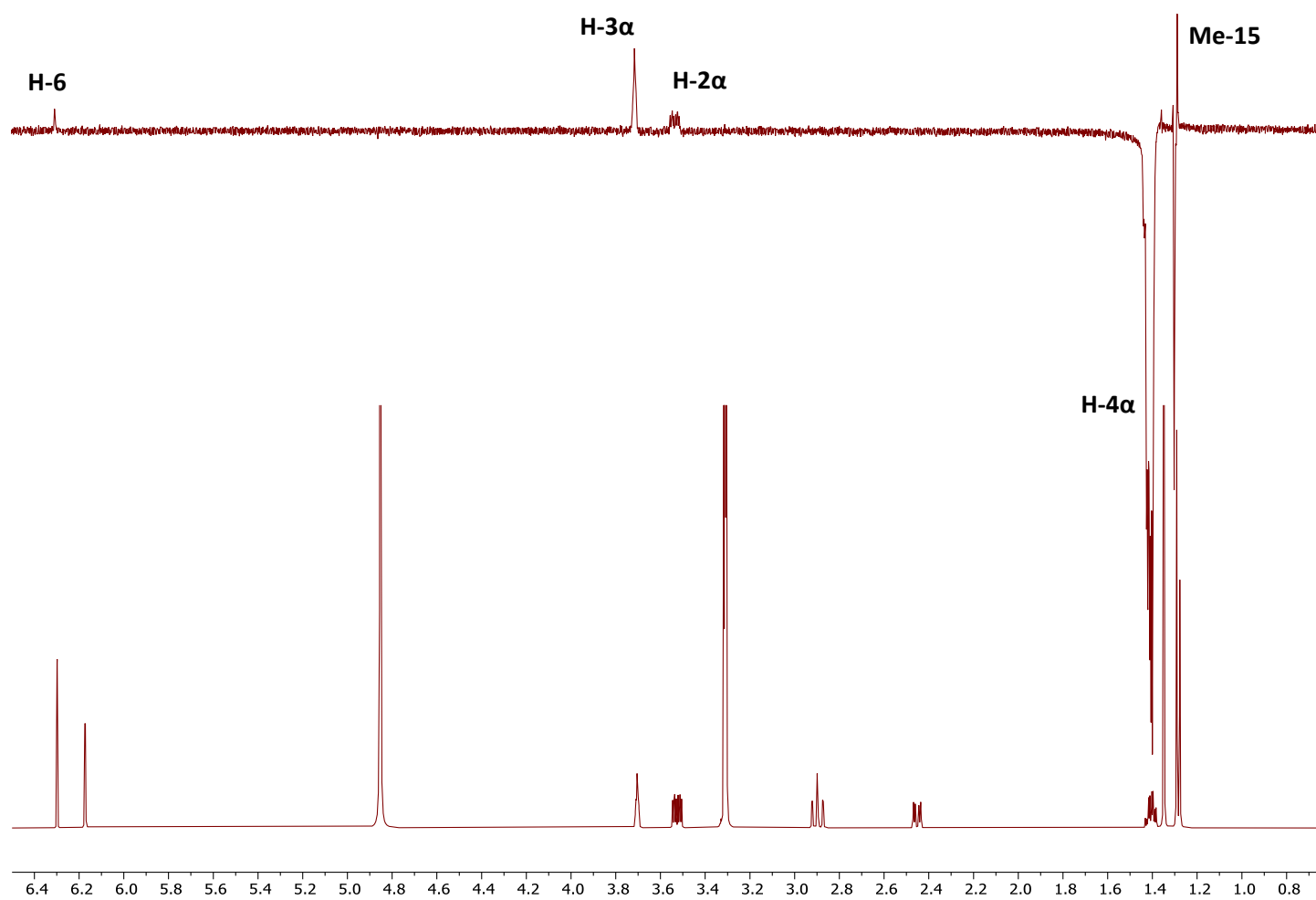
**Figure S6c.** 1D NOESY spectrum of compound **1**.



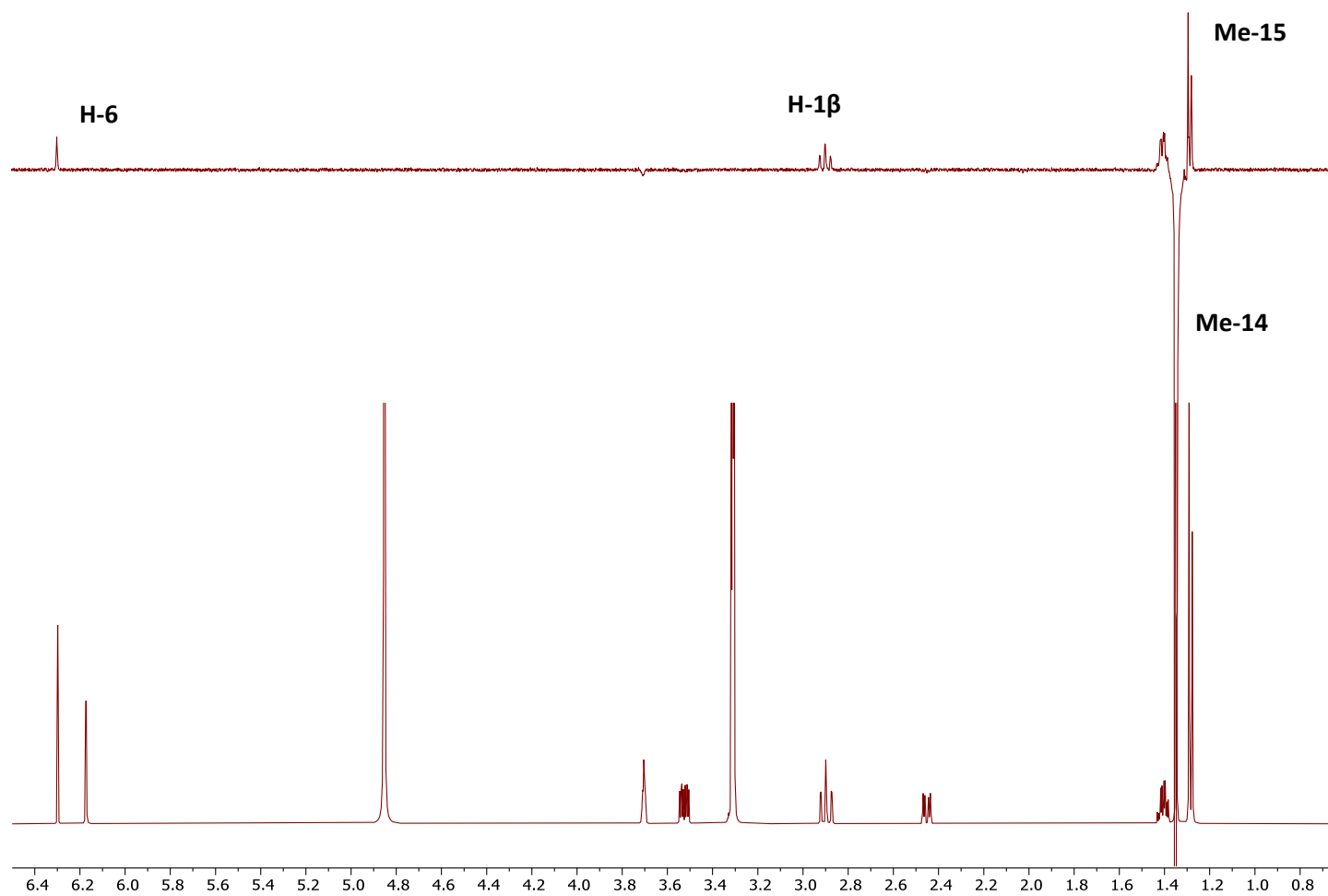
**Figure S6d.** 1D NOESY spectrum of compound 1.



**Figure S6e.** 1D NOESY spectrum of compound **1**.



**Figure S6f.** 1D NOESY spectrum of compound **1**.



**Figure S6g.** 1D NOESY difference spectrum of compound **1**.

### Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

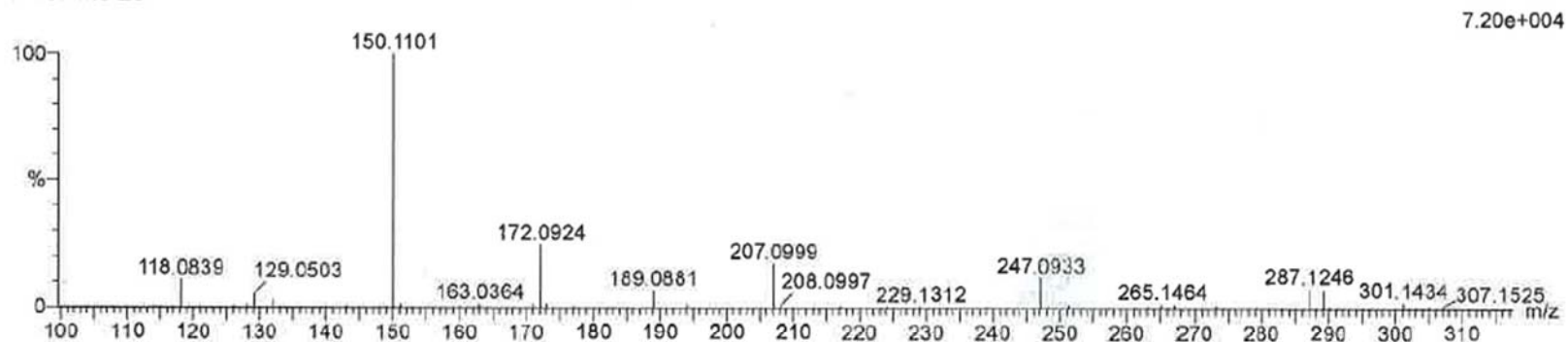
76 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-500 H: 0-1000 O: 0-200 Na: 0-1

CDI-80-P8 26 (0.489)

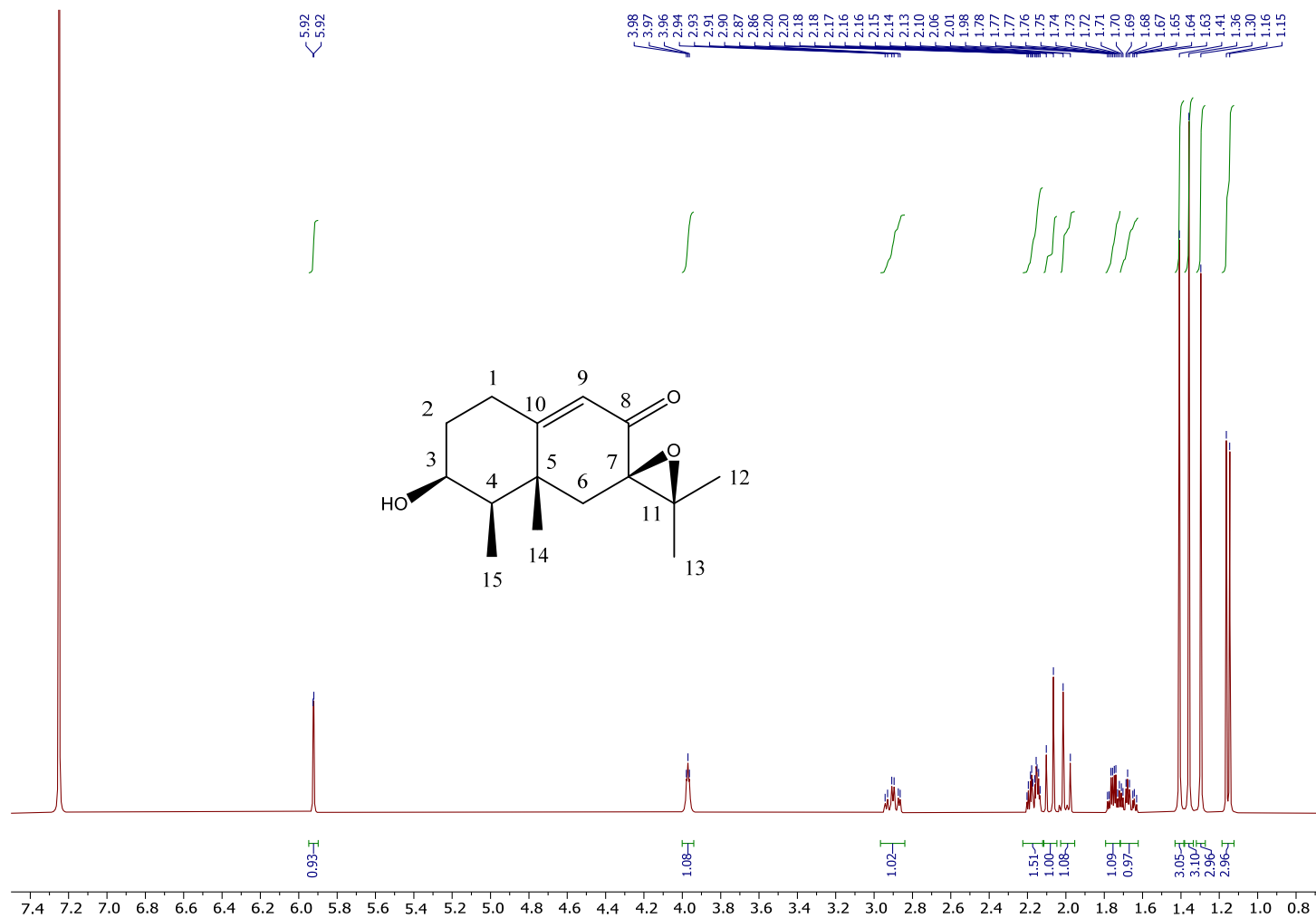
1: TOF MS ES+



Minimum: -1.5  
Maximum: 5.0 10.0 50.0

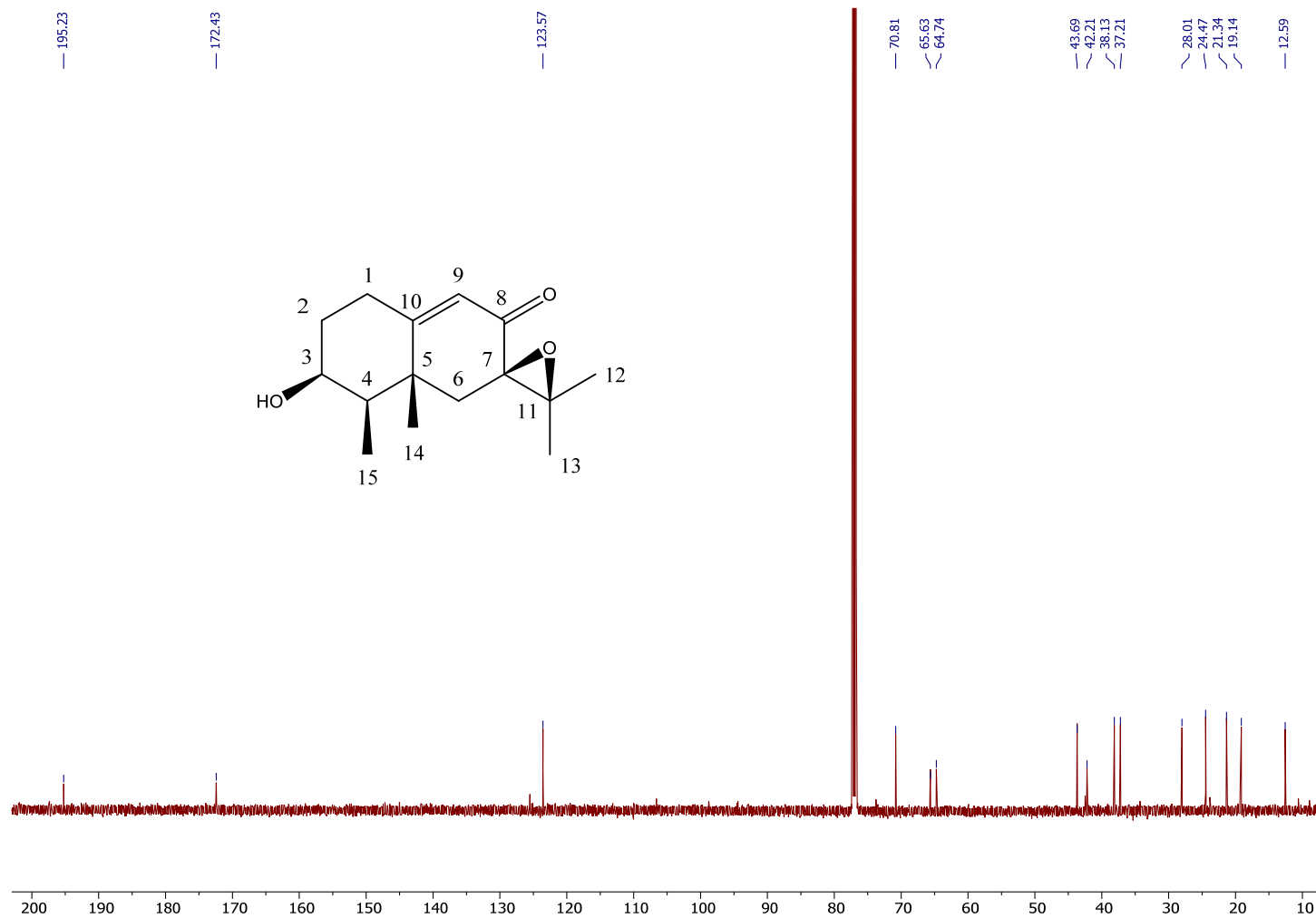
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
247.0933	247.0946	-1.3	-5.3	4.5	323.1	0.316	72.93	C12 H16 O4 Na
	247.0970	-3.7	-15.0	7.5	324.1	1.307	27.07	C14 H15 O4

Figure S7. HRESIMS spectrum of compound 1.

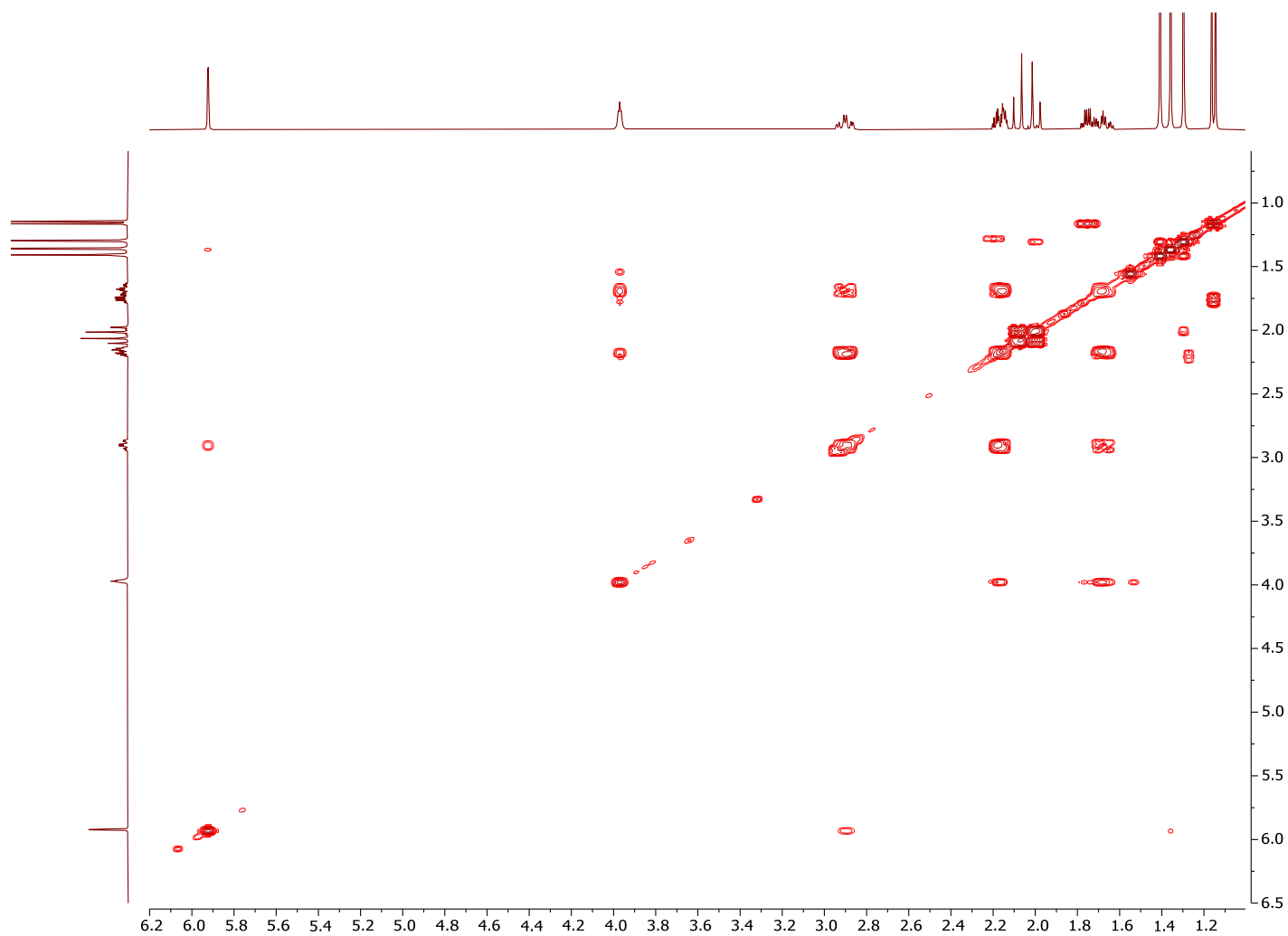


**Figure S8.** <sup>1</sup>H NMR spectrum (400 MHz, CDCl<sub>3</sub>) of compound 2.

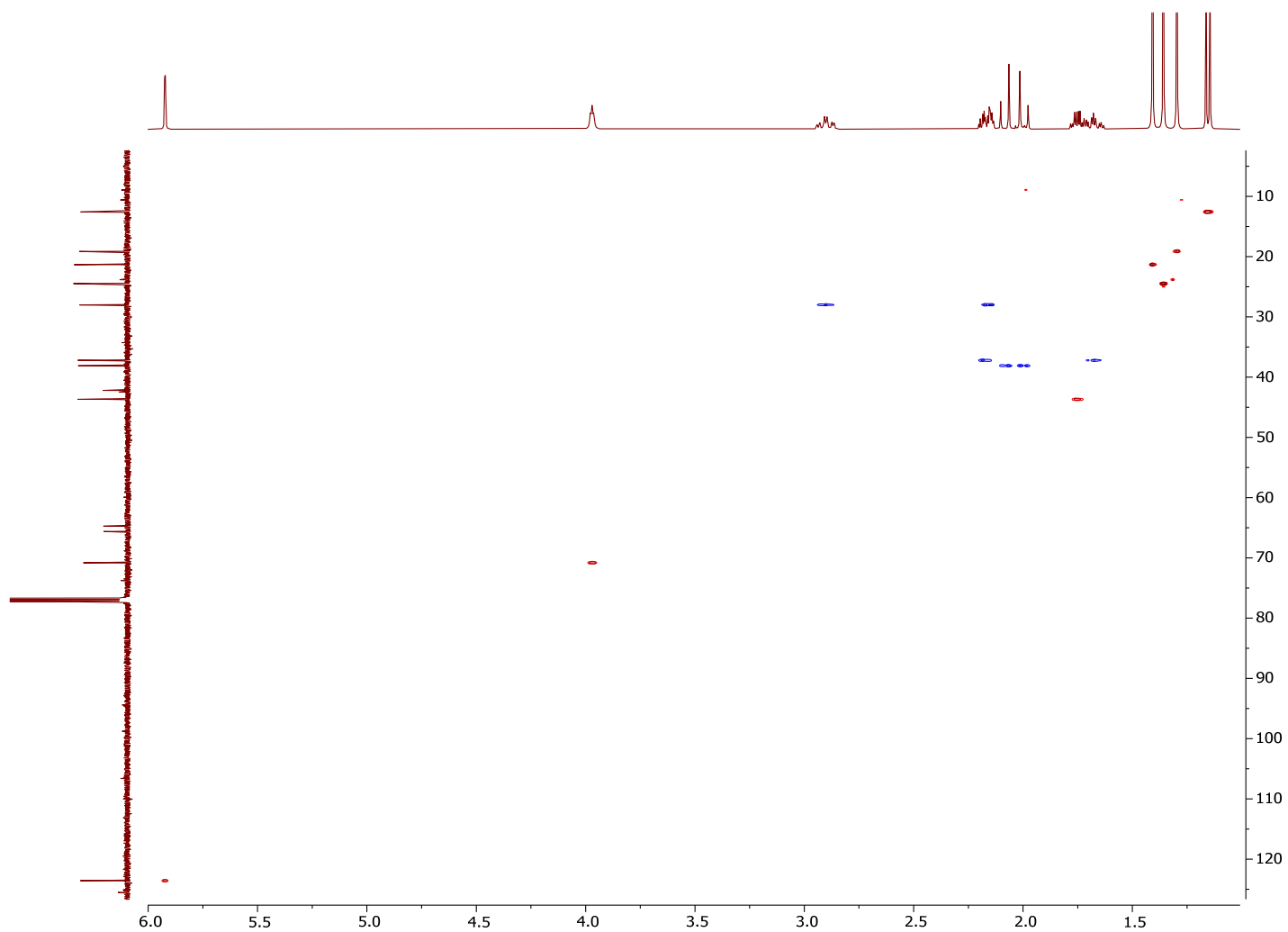




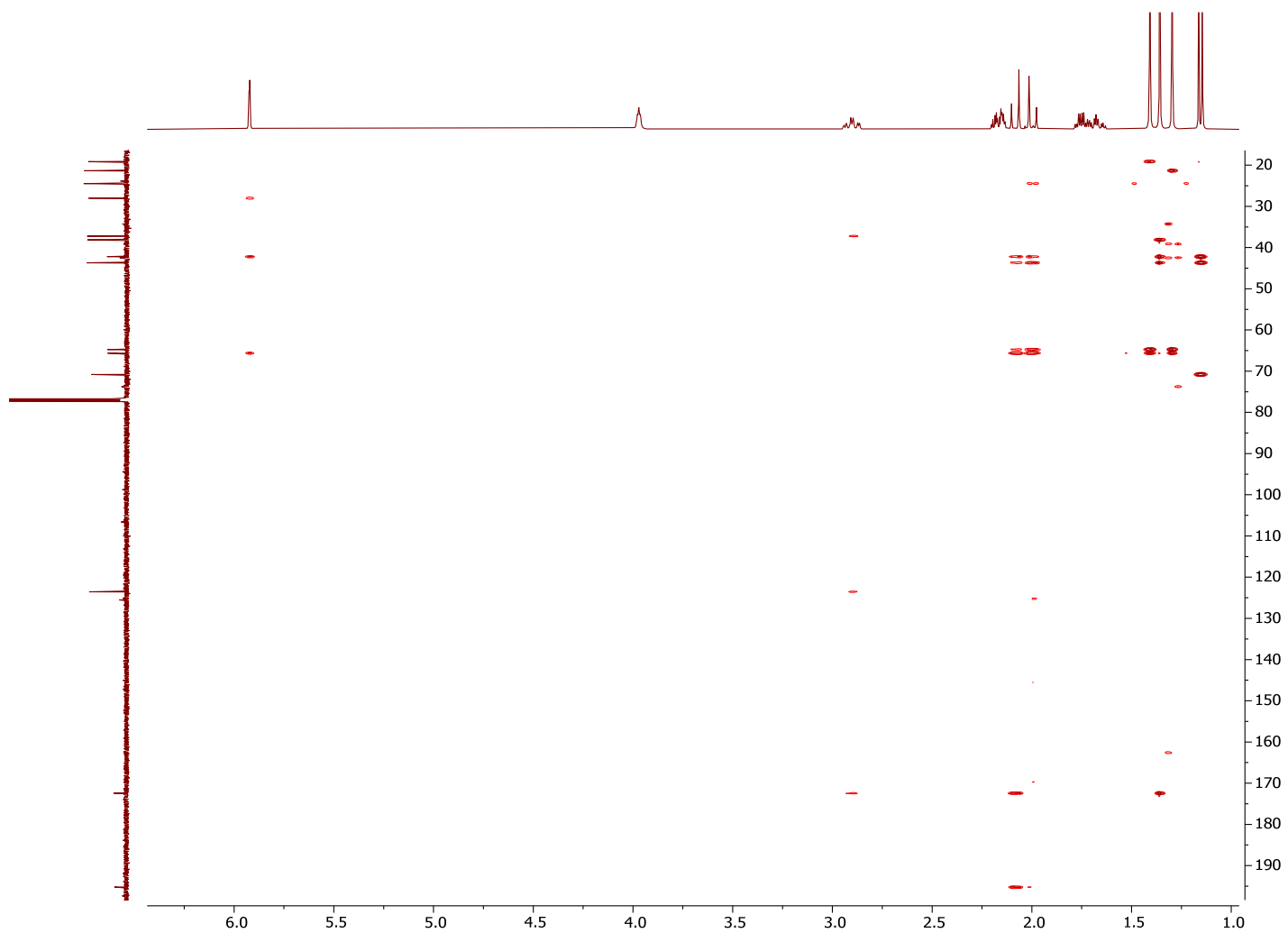
**Figure S9.** <sup>13</sup>C NMR spectrum (125 MHz, CDCl<sub>3</sub>) of compound 2.



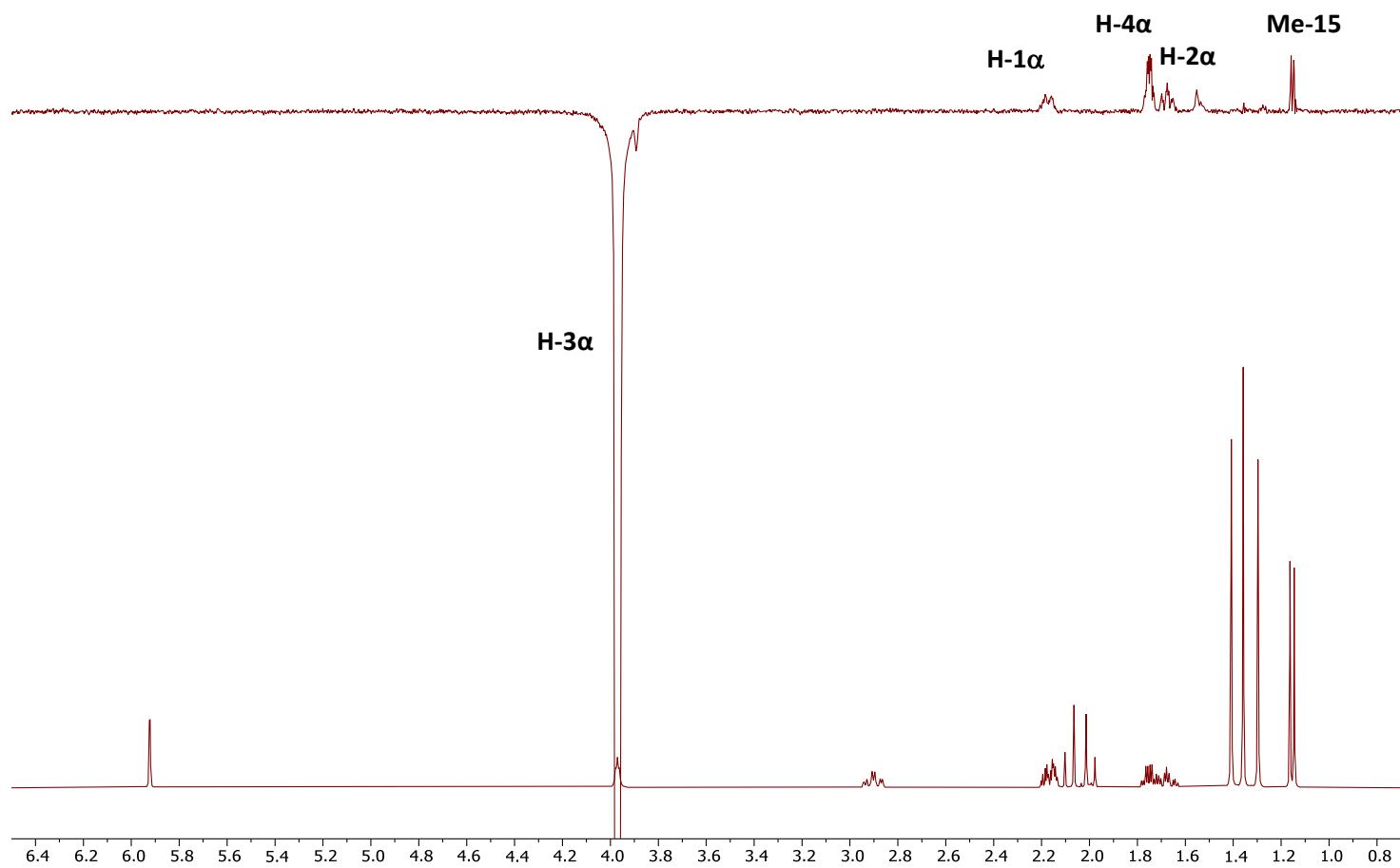
**Figure S10.** gCOSY spectrum of compound **2**.



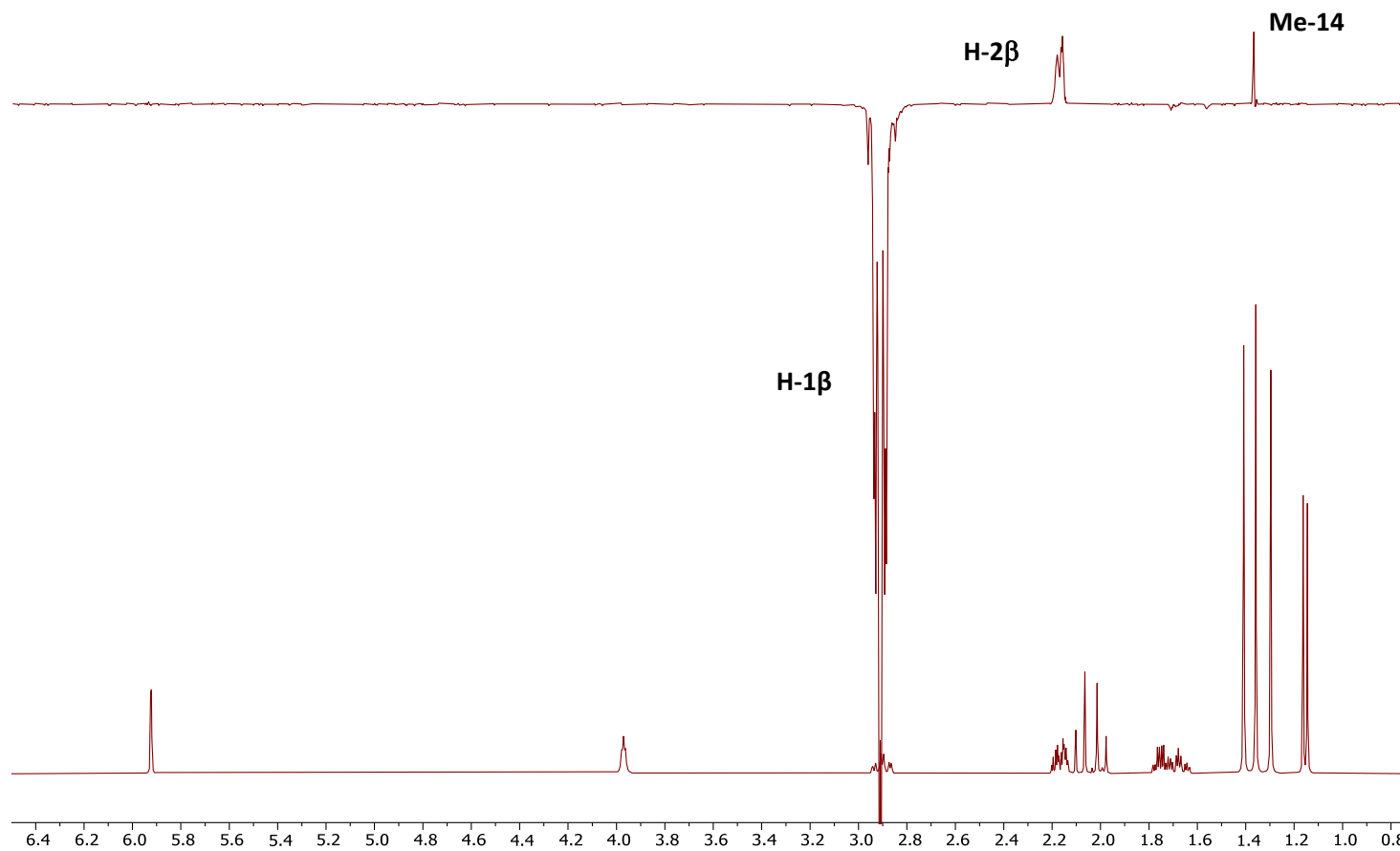
**Figure S11.** gHSQC spectrum of compound **2**.



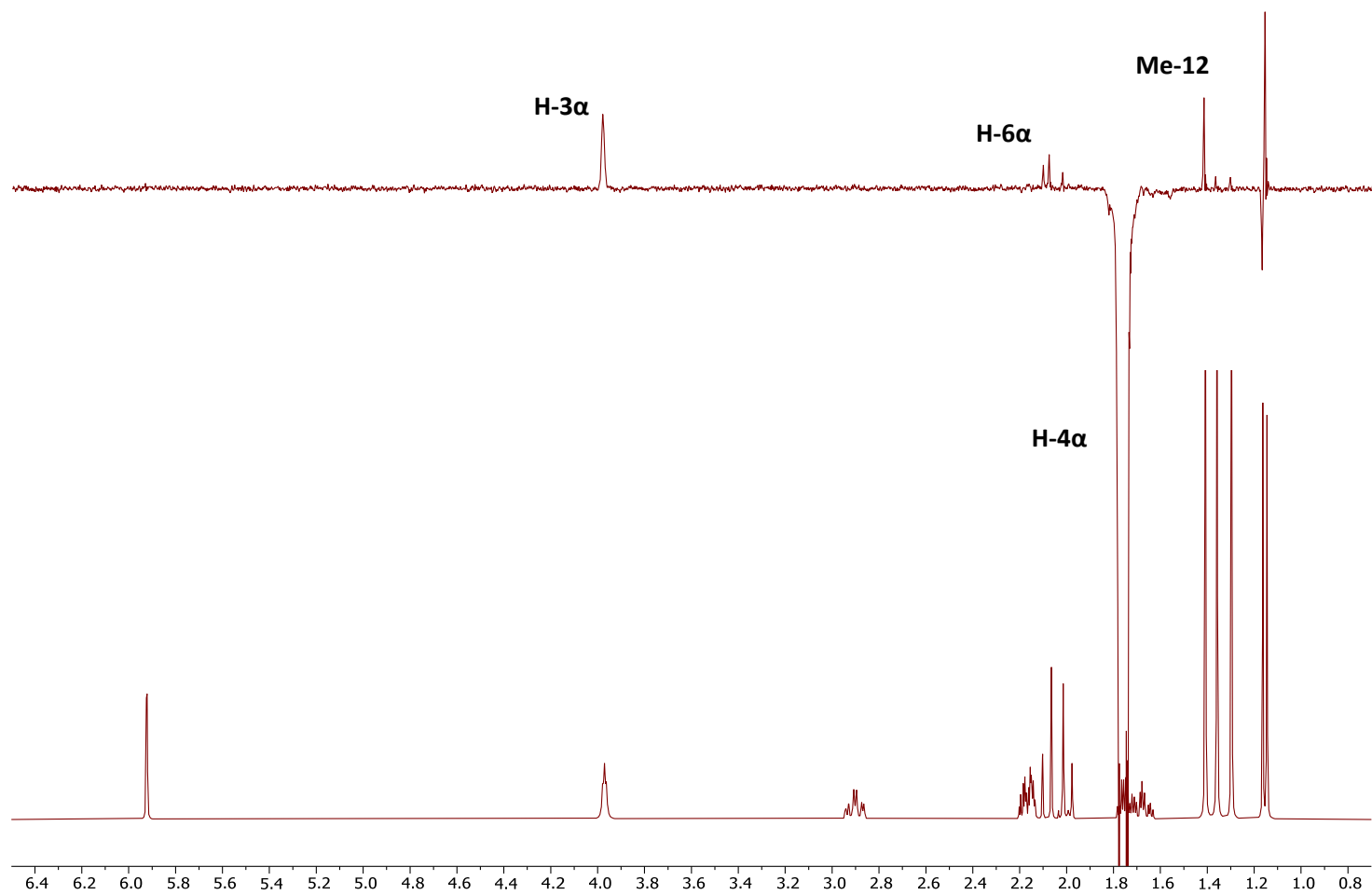
**Figure S12.** gHMBC spectrum of compound **2**.



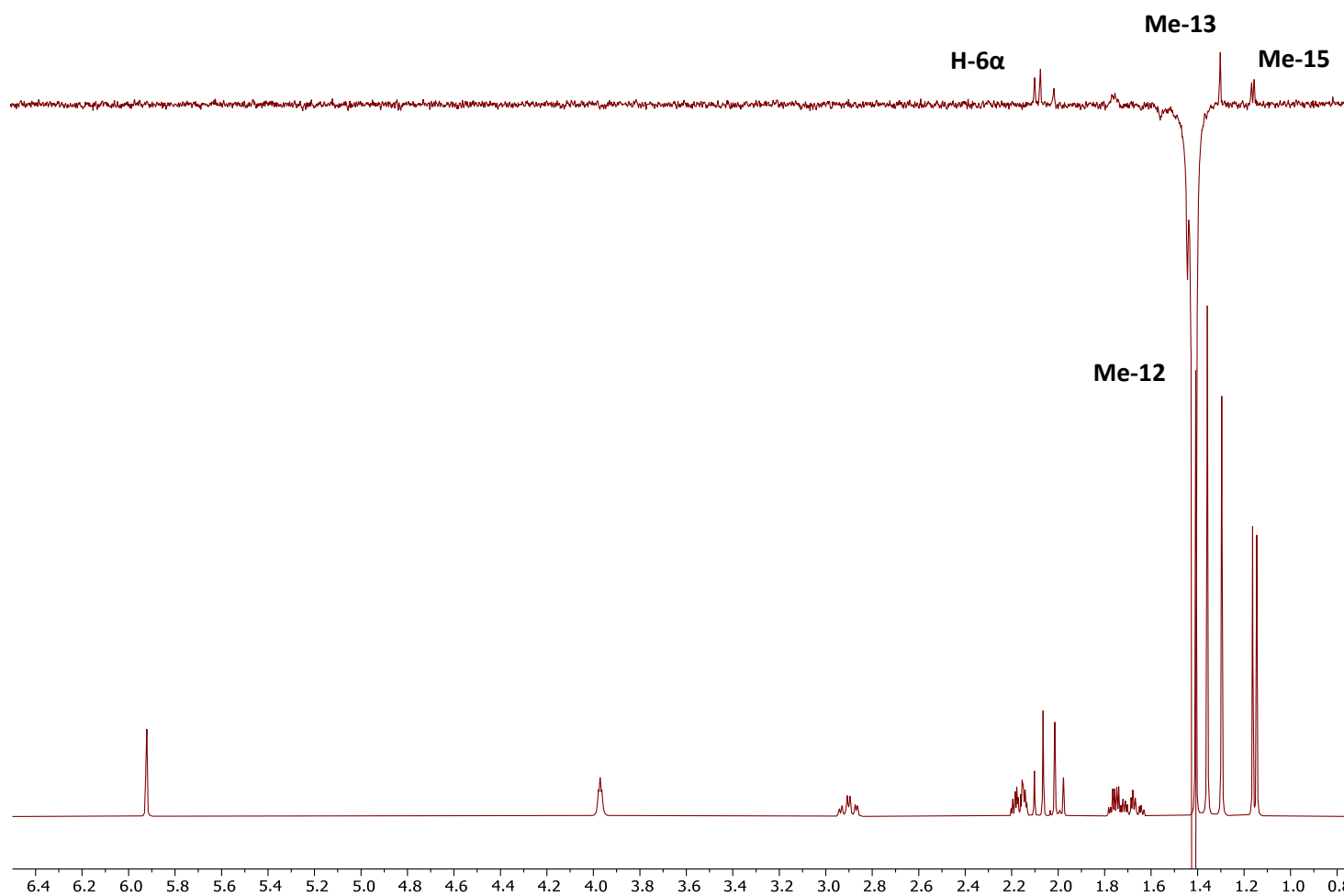
**Figure S13a.** 1D NOESY spectrum of compound **2**.



**Figure S13b.** 1D NOESY spectrum of compound **2**.

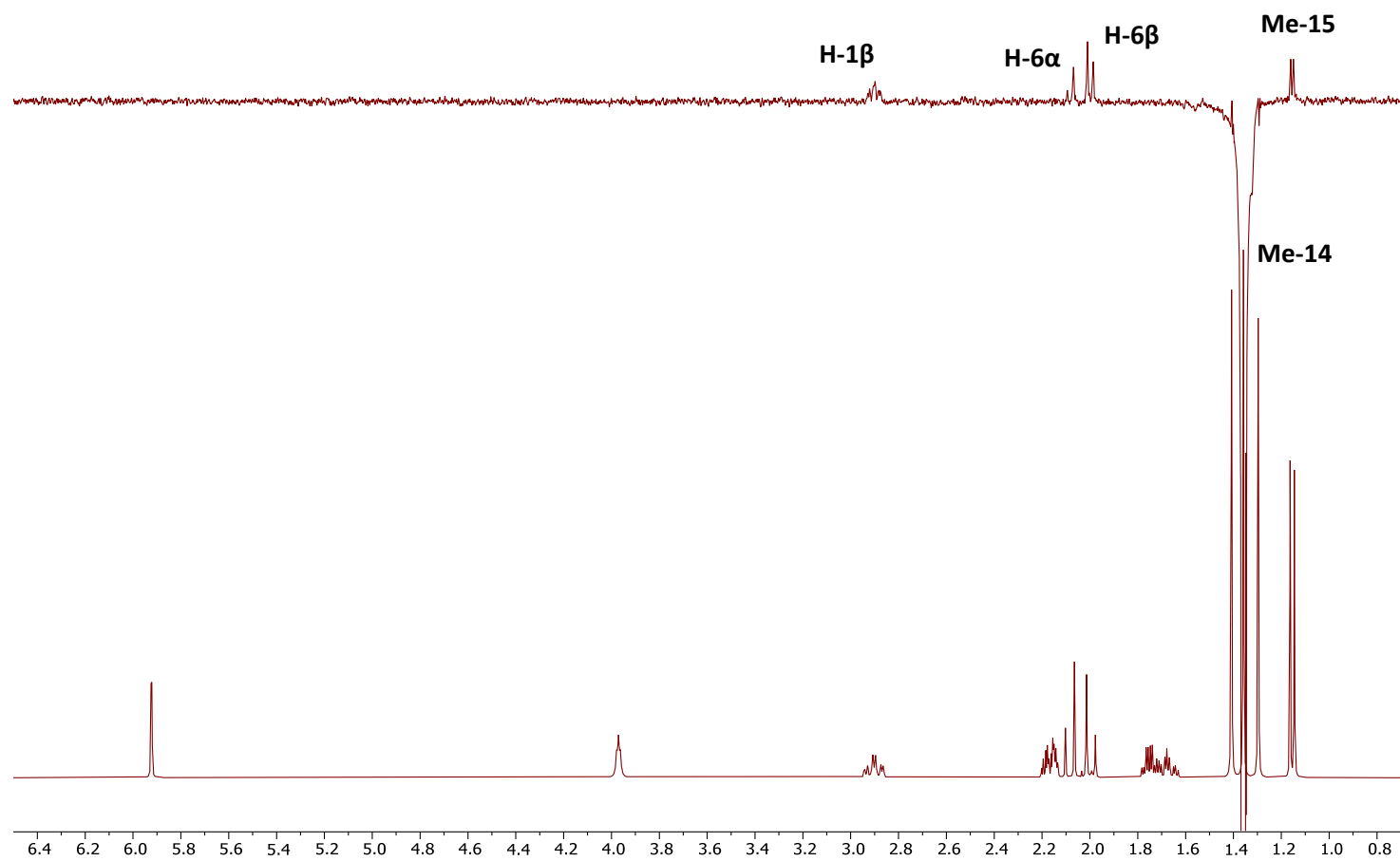


**Figure S13c.** 1D NOESY spectrum of compound **2**.

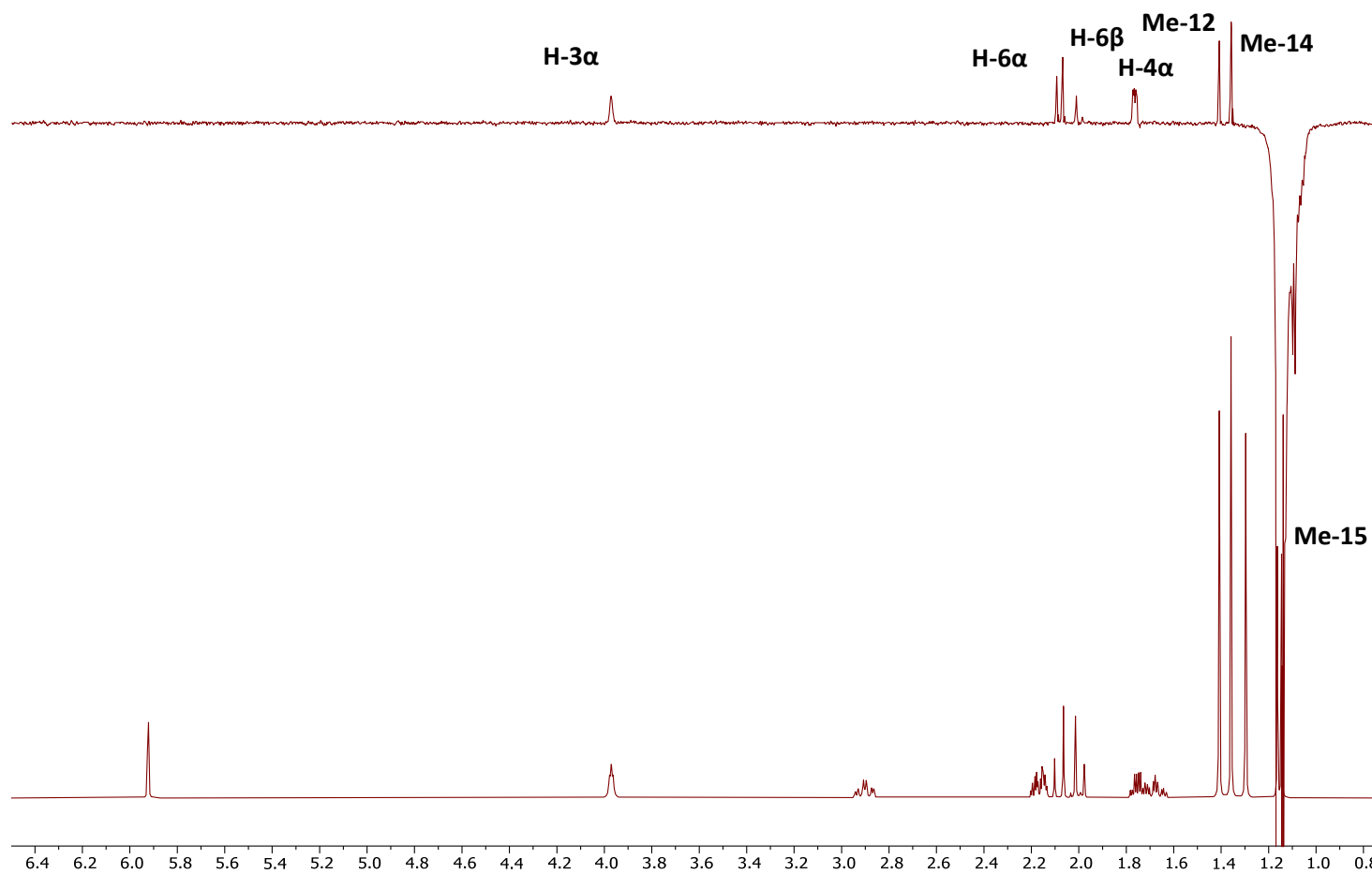


**Figure S13d.** 1D NOESY spectrum of compound **2**.





**Figure S13e.** 1D NOESY spectrum of compound **2**.



**Figure S13f.** 1D NOESY spectrum of compound **2**.

### Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

83 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

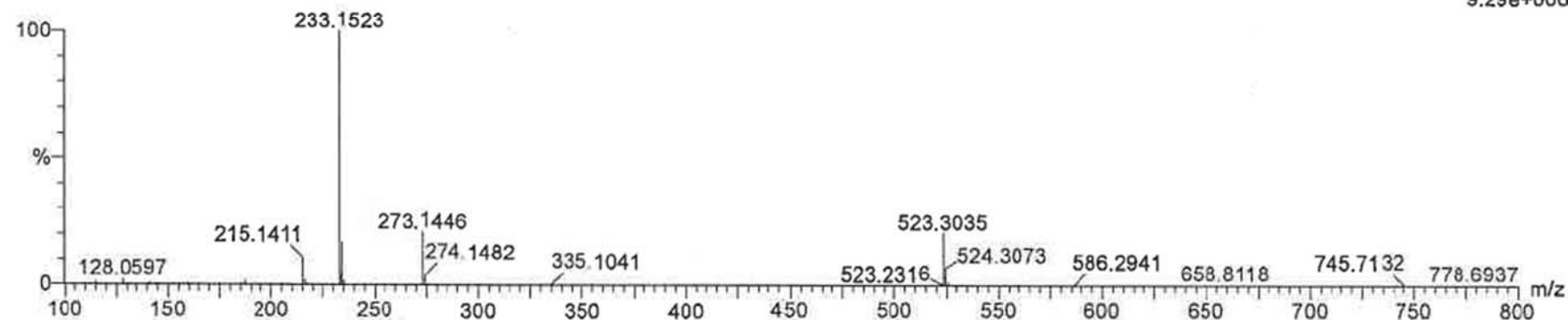
Elements Used:

C: 0-500 H: 0-1000 O: 0-200 Na: 0-1

CD2-80-P6 43 (0.803)

1: TOF MS ES+

9.29e+006



Minimum:

Maximum: 5.0 10.0 -1.5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
273.1446	273.1467	-2.1	-7.7	4.5	685.4	0.150	86.07	C15 H22 O3 Na
	273.1491	-4.5	-16.5	7.5	687.2	1.971	13.93	C17 H21 O3

Figure S14. HRESIMS spectrum of compound 2.

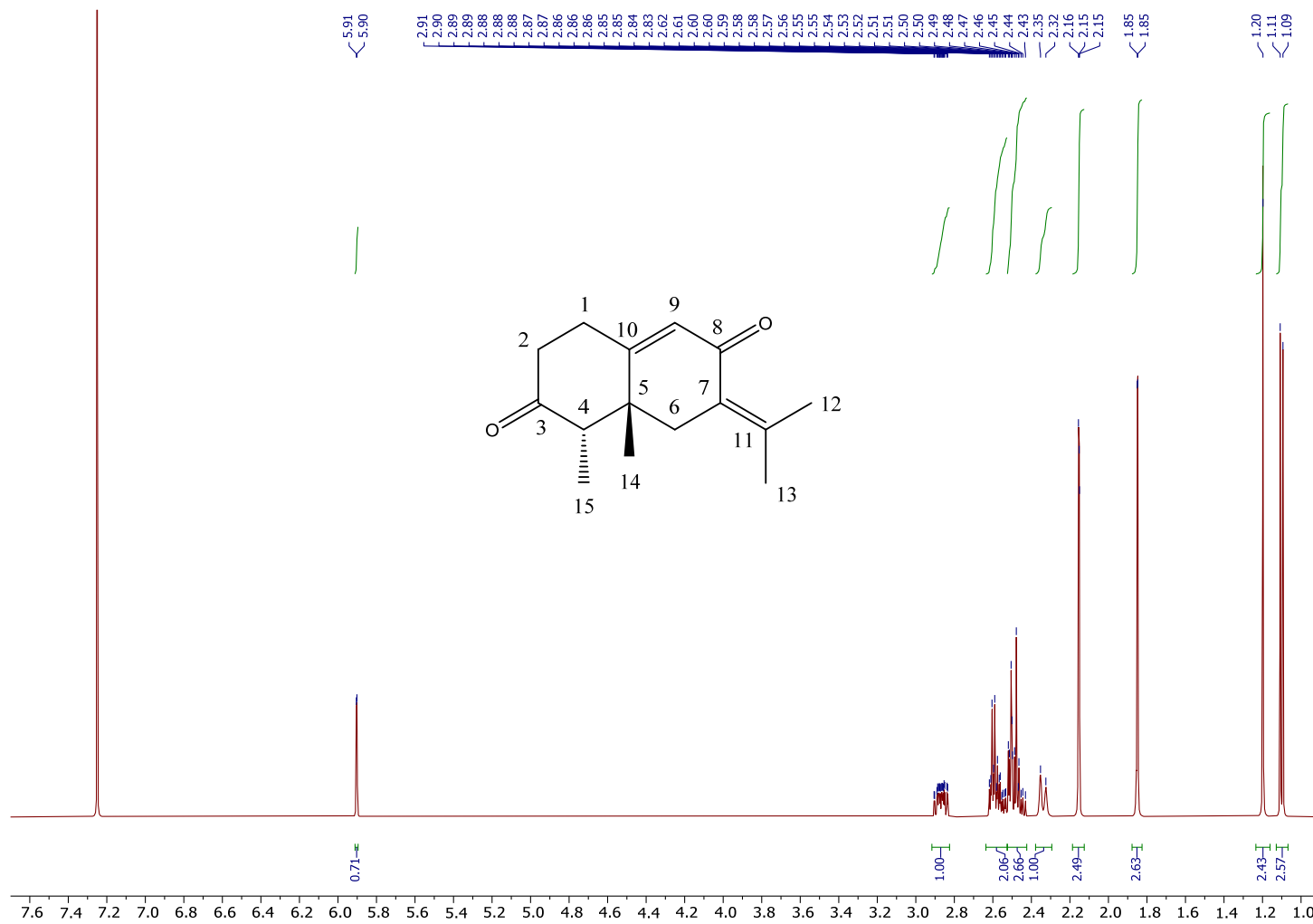
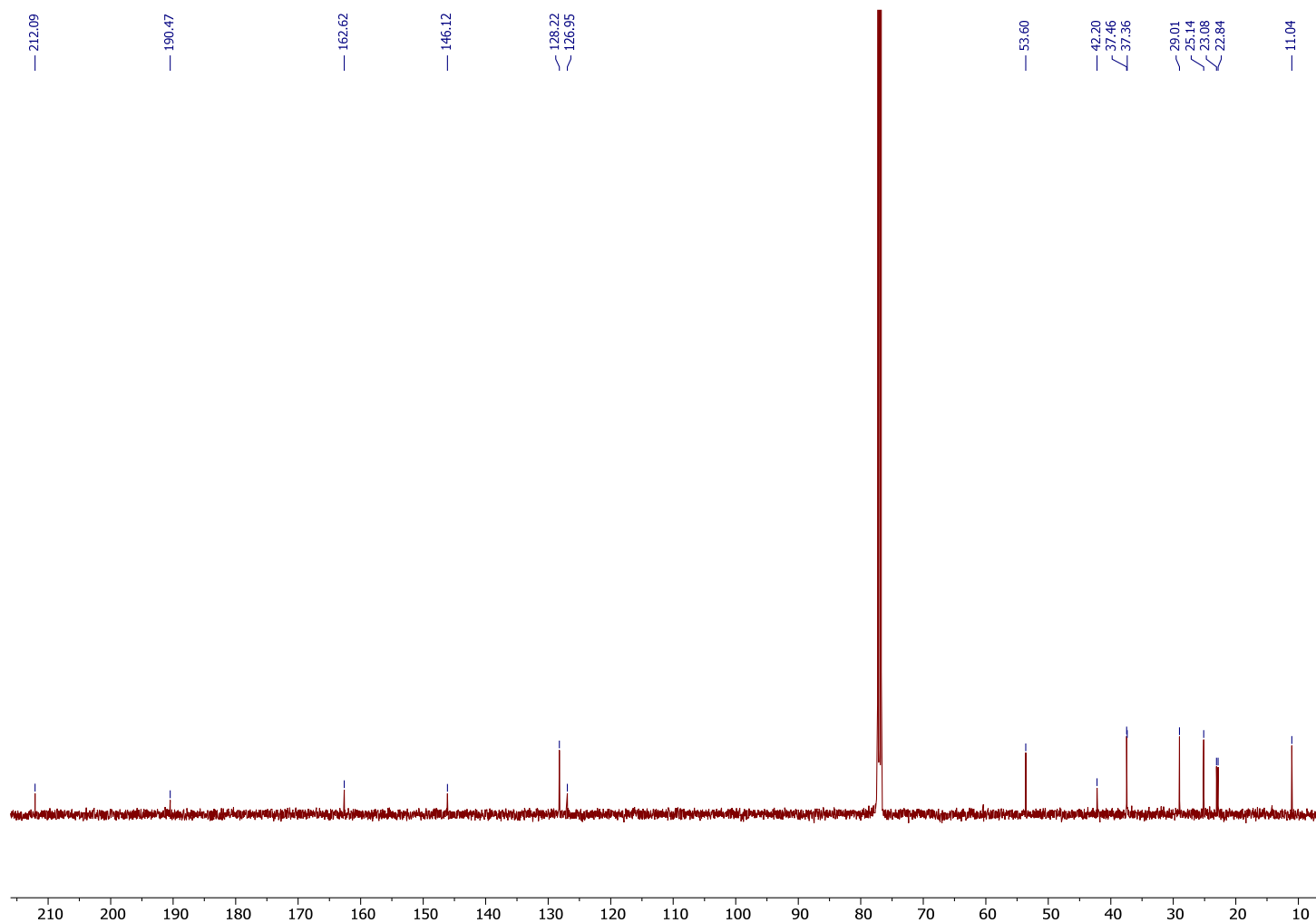
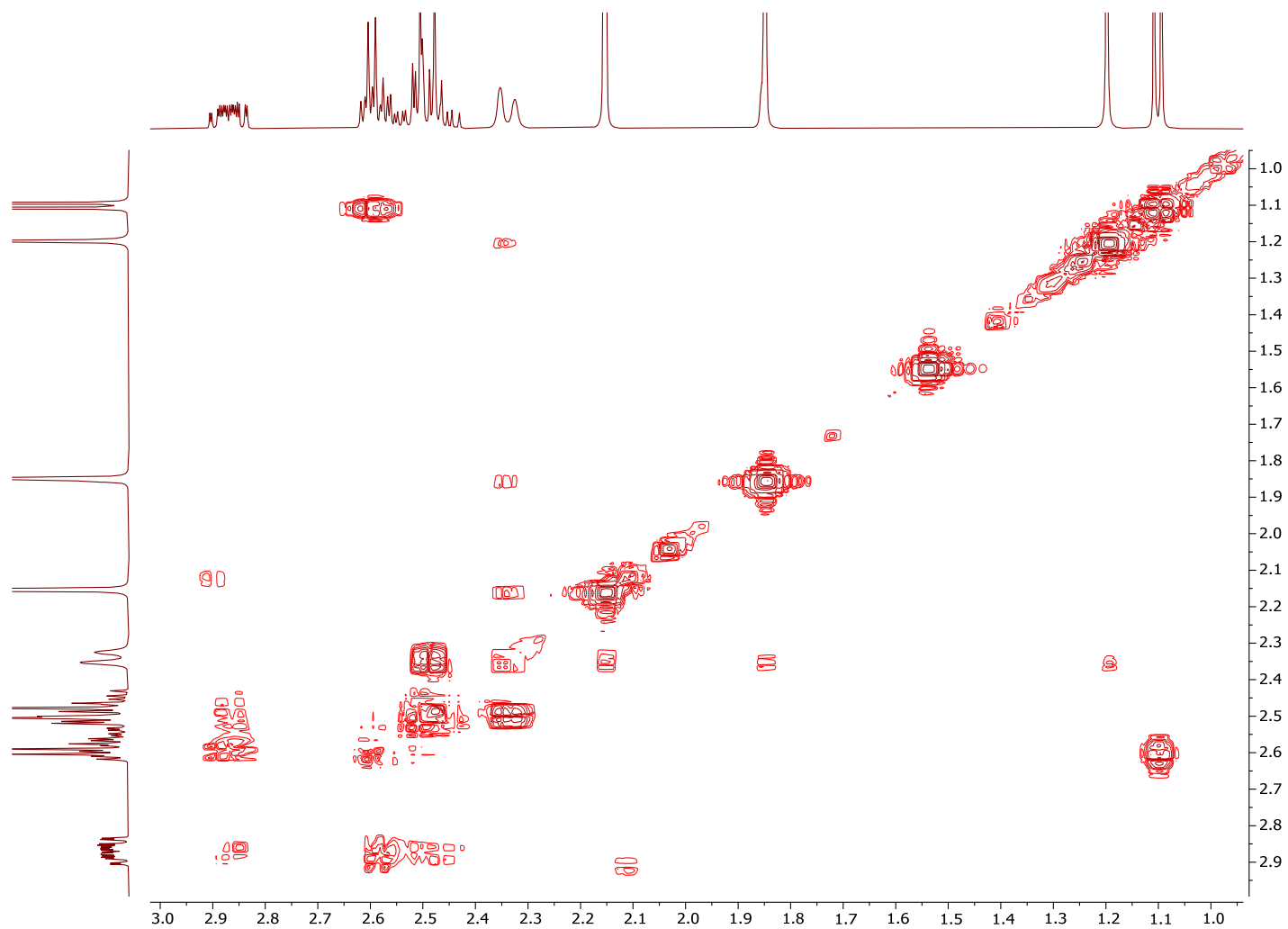


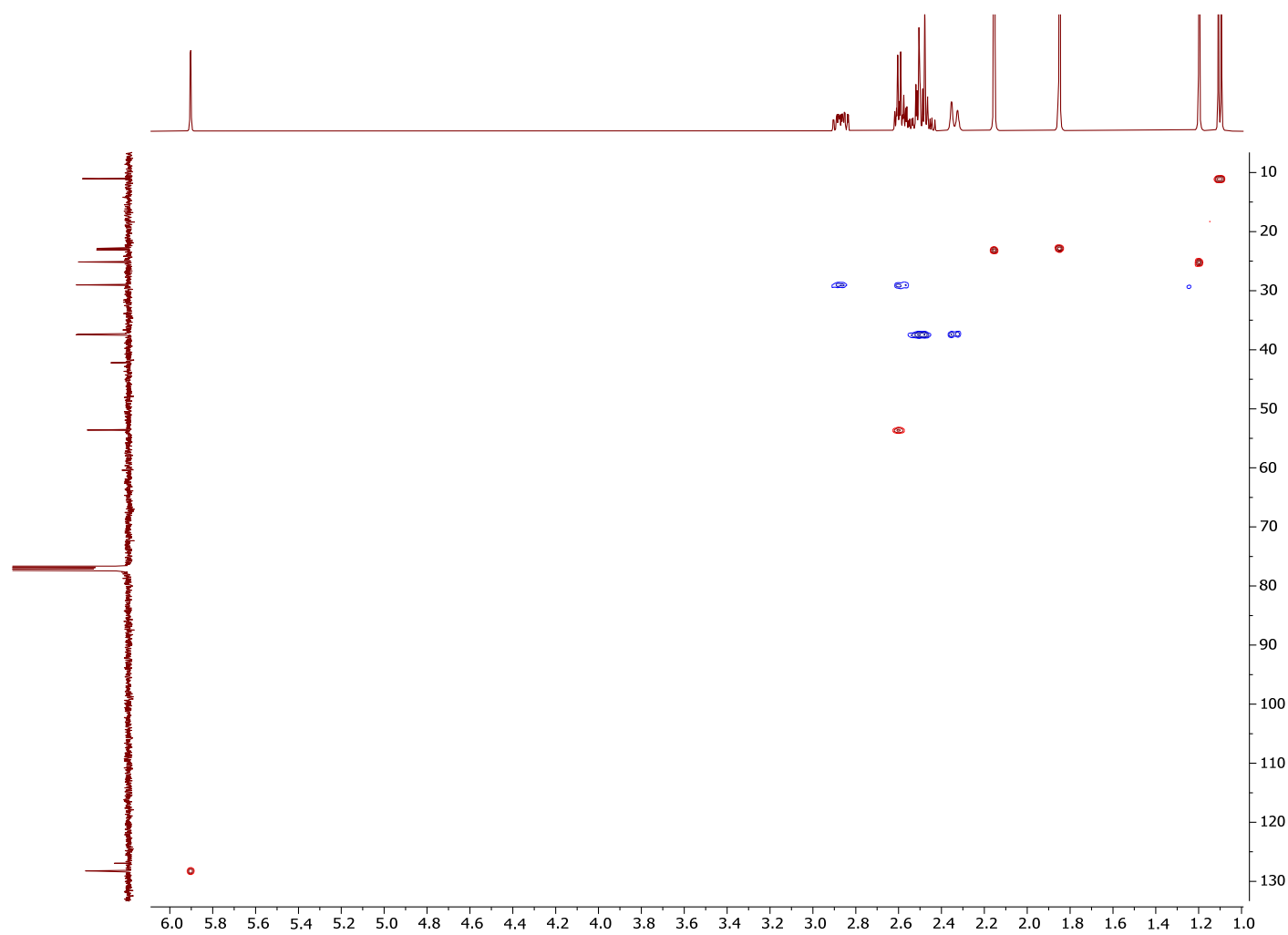
Figure S15. <sup>1</sup>H NMR spectrum (500 MHz, CDCl<sub>3</sub>) of compound 3.



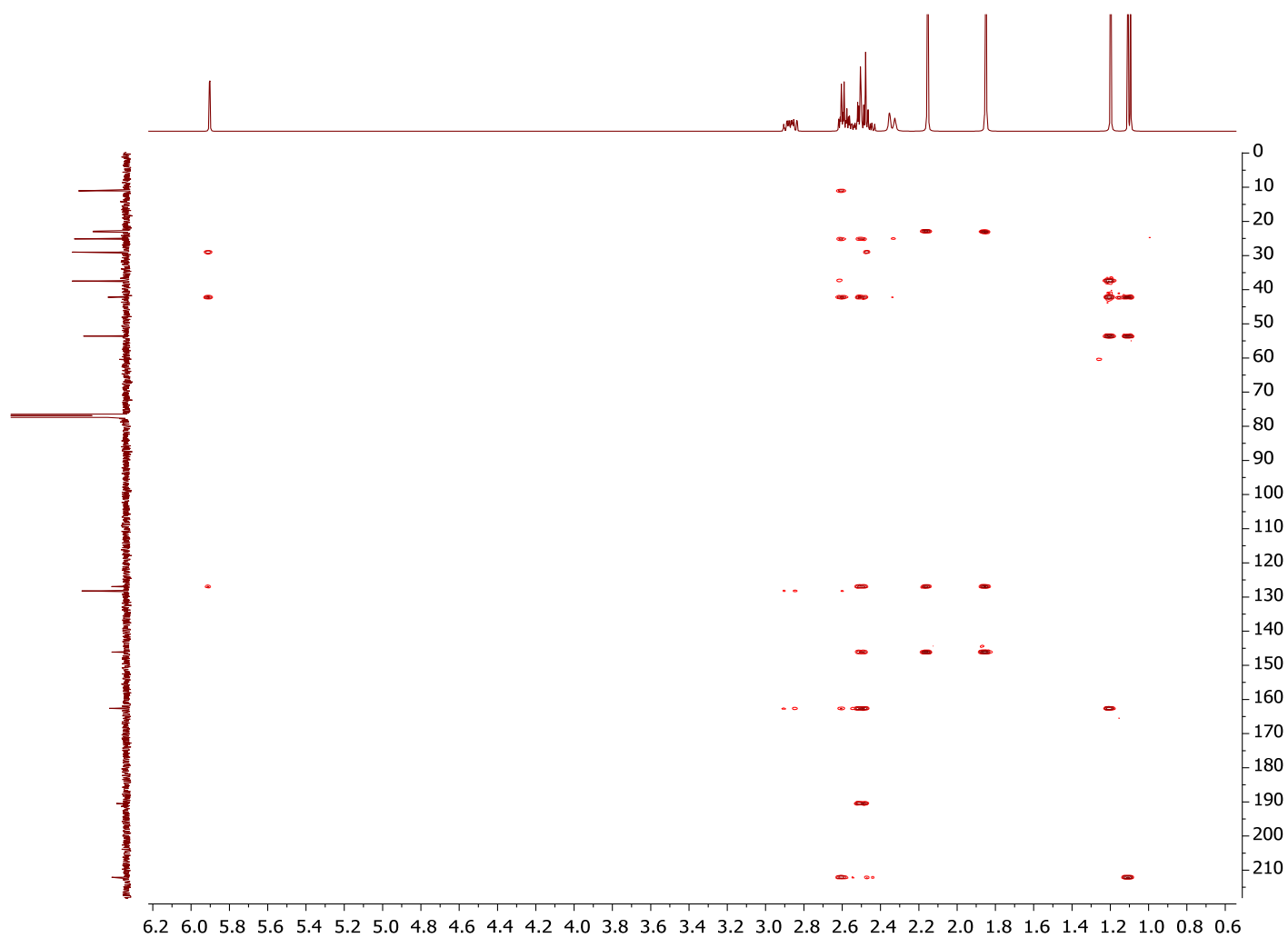
**Figure S16.**  $^{13}\text{C}$  NMR spectrum (125 MHz,  $\text{CDCl}_3$ ) of compound **3**.



**Figure S17.** gCOSY spectrum of compound **3**.

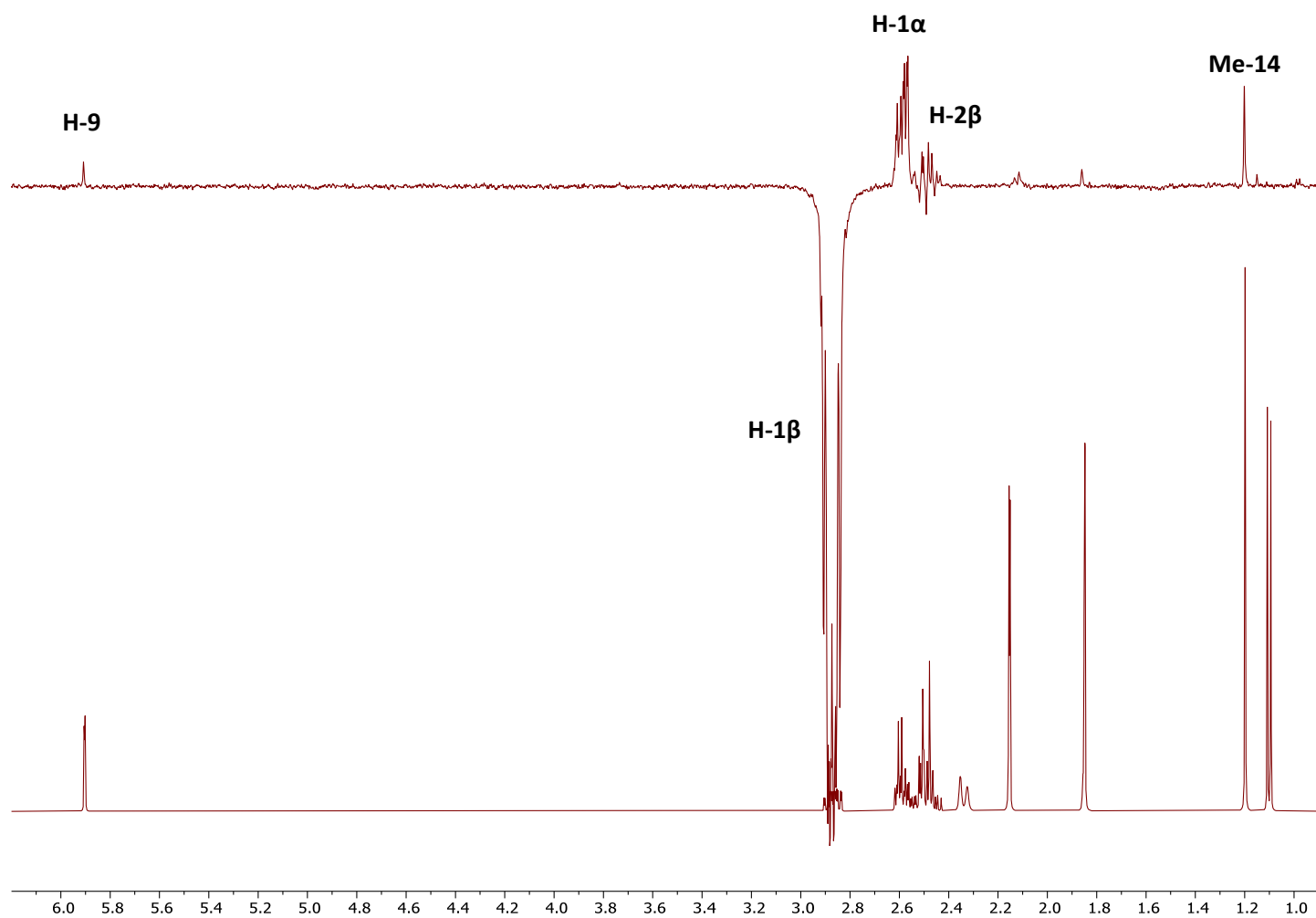


**Figure S18.** gHSQC spectrum of compound **3**.

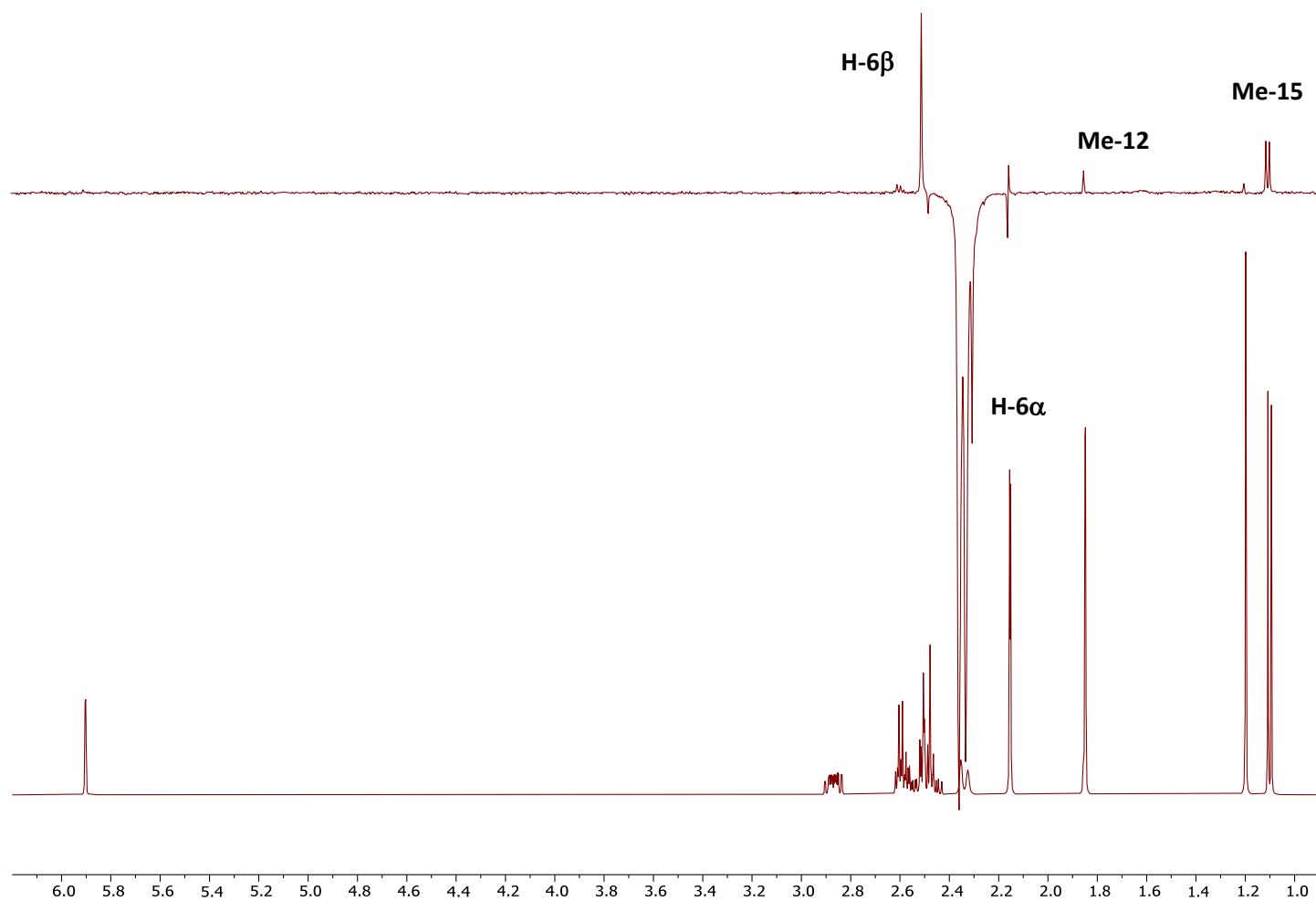


**Figure S19.** gHMBC spectrum of compound 3.

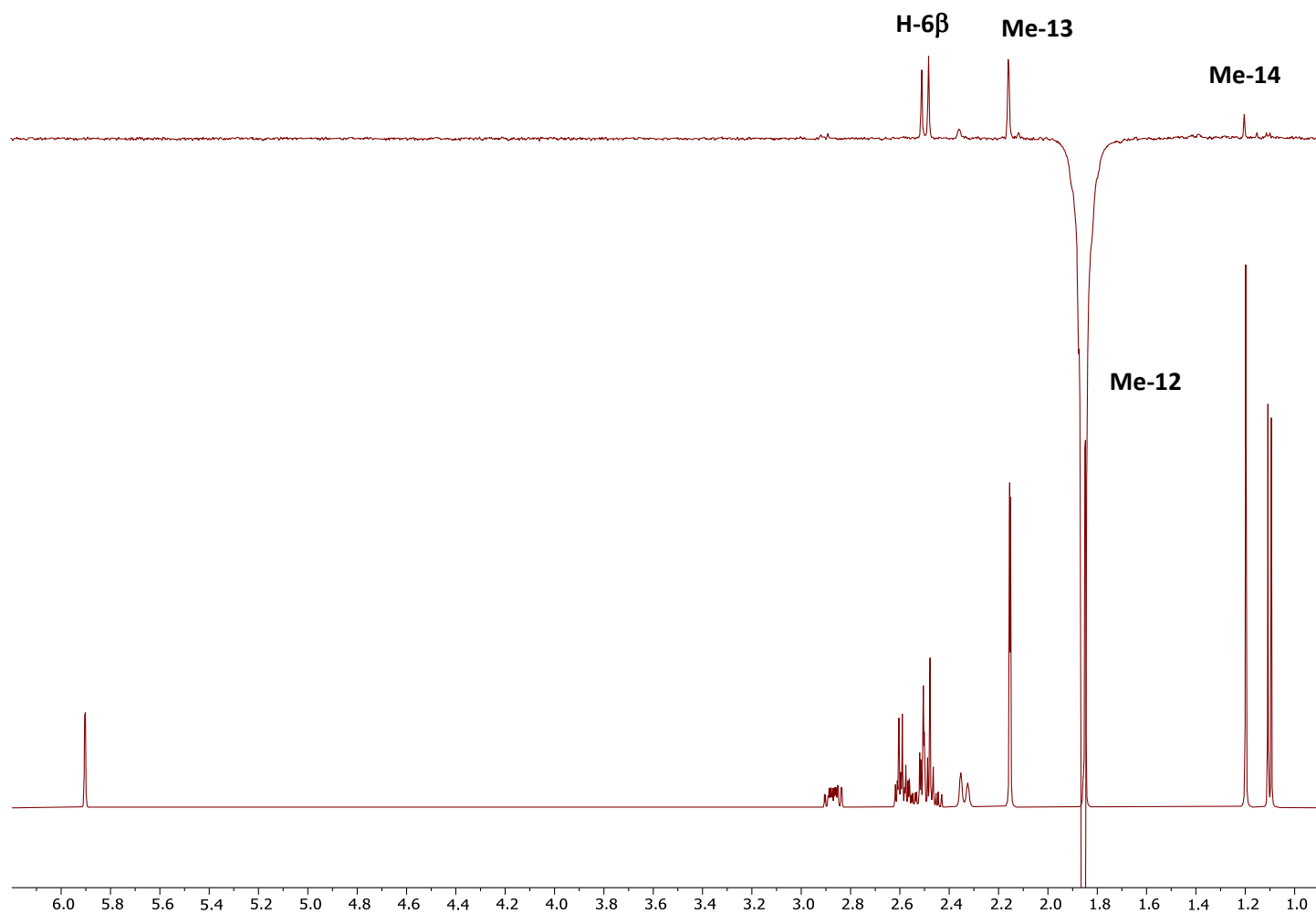




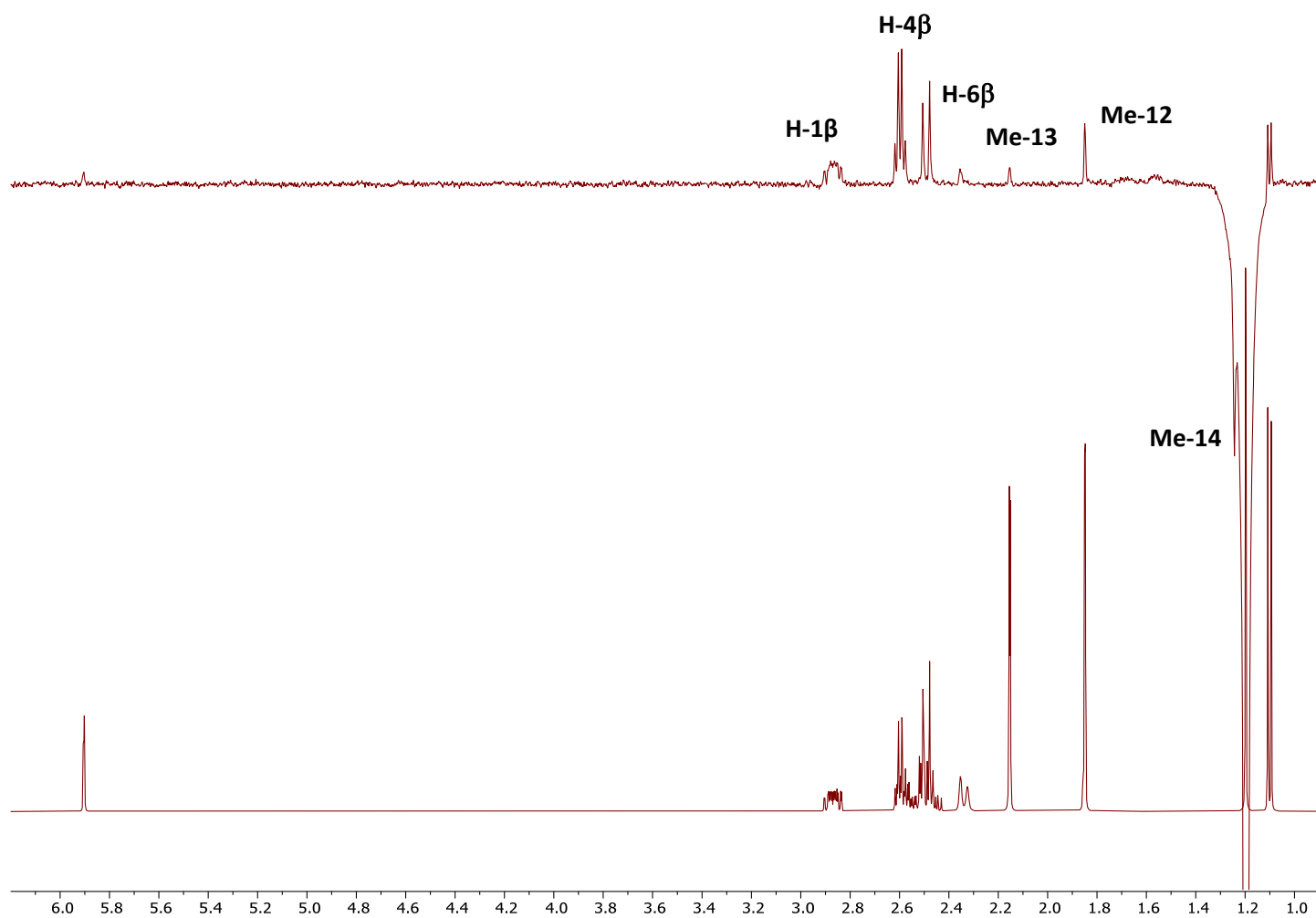
**Figure S20a.** 1D NOESY spectrum of compound **3**.



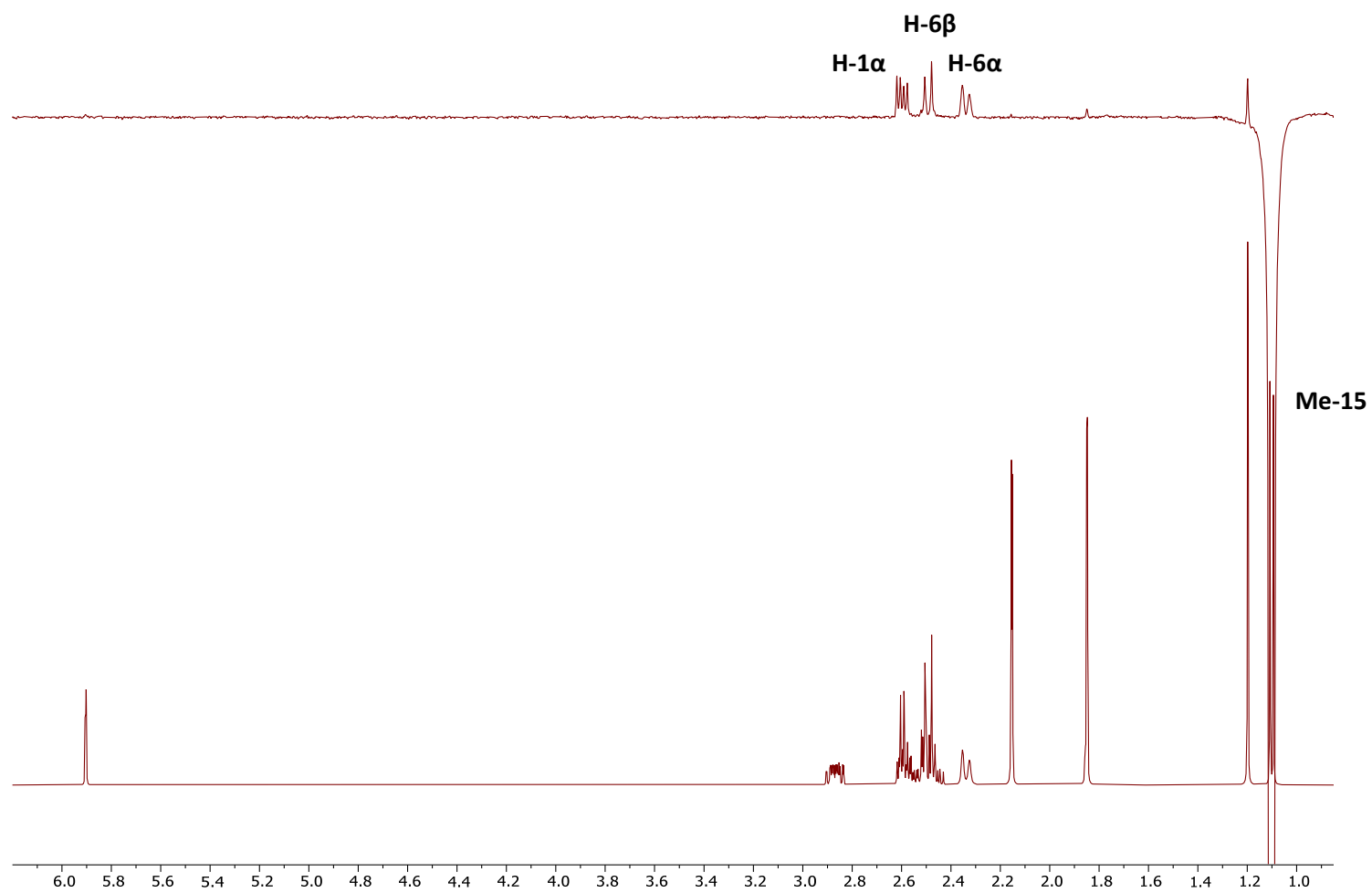
**Figure S20b.** 1D NOESY spectrum of compound **3**.



**Figure S20c.** 1D NOESY spectrum of compound **3**.



**Figure S20d.** 1D NOESY spectrum of compound **3**.



**Figure S20e.** 1D NOESY spectrum of compound **3**.

## Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

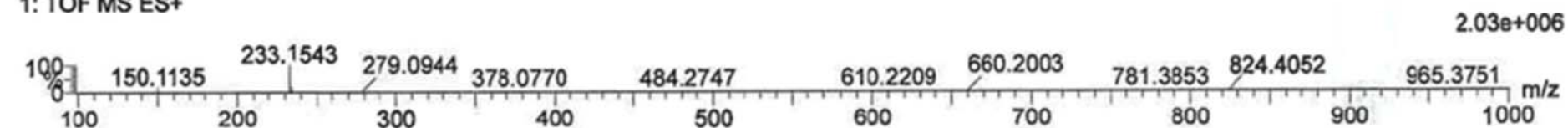
36 formula(e) evaluated with 1 results within limits (up to 5 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-200 O: 0-50

CD1-40-P8 288 (2.670)

1: TOF MS ES+



Minimum: -1.5  
Maximum: 5.0 10.0 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
233.1543	233.1542	0.1	0.4	5.5	1387.8	n/a	n/a	C15 H21 O2

Figure S21. HRESIMS spectrum of compound 3.