

ELECTRONIC SUPPLEMENTARY INFORMATION

New Haliclamines E and F from the Arctic Sponge *Haliclona viscosa*

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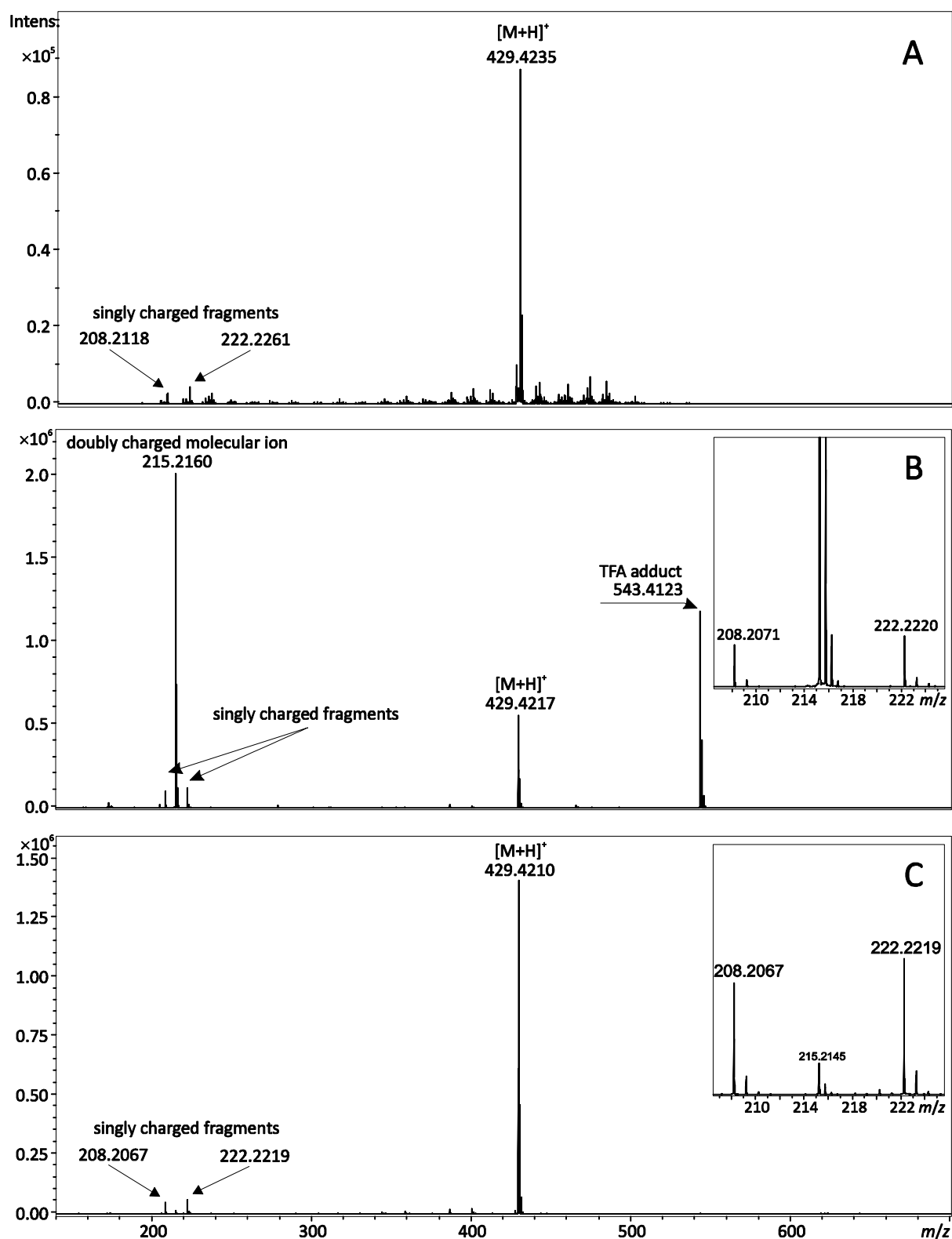


Figure SI-1. Comparison of mass spectra of the natural and synthetic compound **1**. – (A). Mass trace obtained from an LCMS experiment of the crude extract of *Haliclona viscosa*. – (B). Synthetic compound under standard ESI conditions. – (C). Synthetic compound under API-CID-MS/MS conditions. The sections show details of the doubly charged molecular ion and the singly charged fragments.

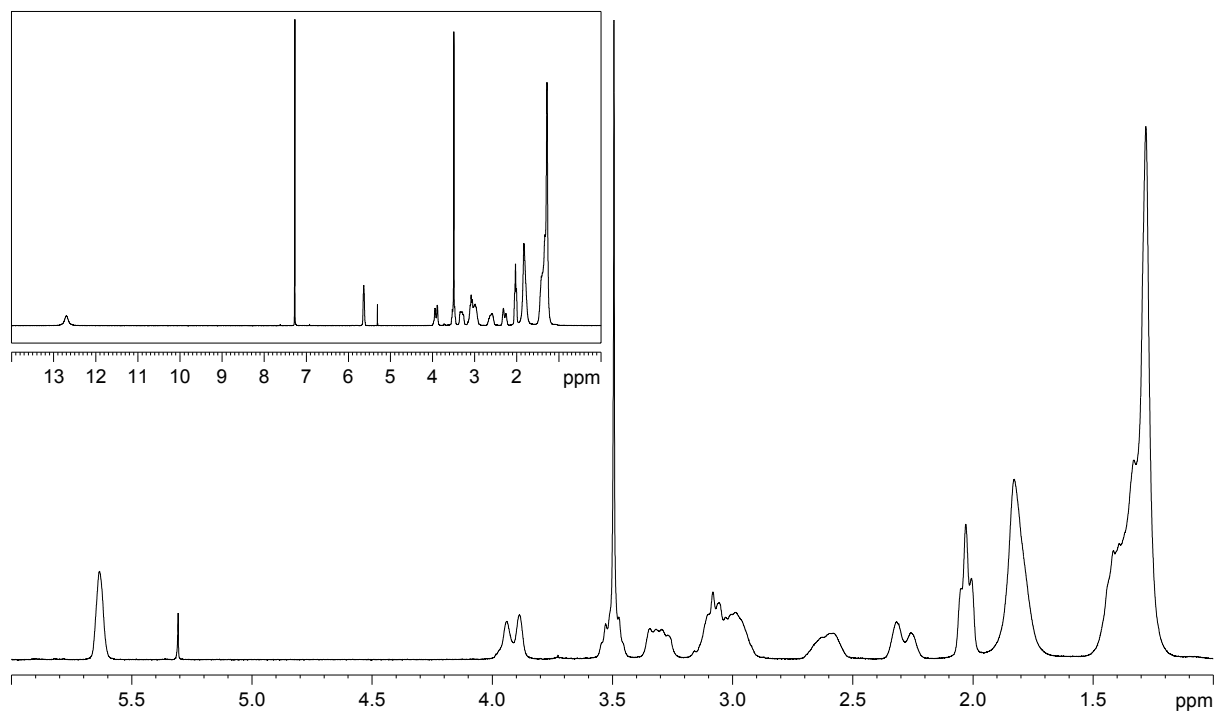


Figure SI-2. Detail and full scan (box) of a $^1\text{H-NMR}$ spectrum (300 MHz, CDCl_3) of the synthetic haliclamine E (**1**).

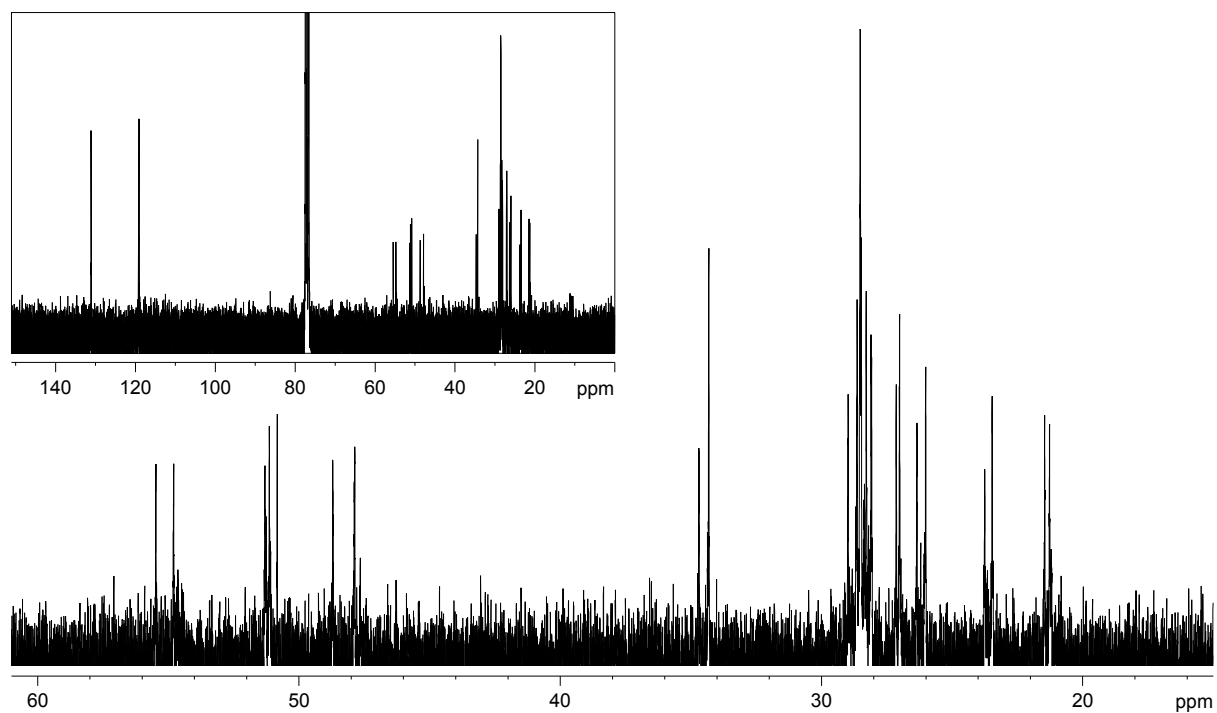


Figure SI-3. Detail and full scan (box) of a $^{13}\text{C-NMR}$ spectrum (75 MHz, CDCl_3) of the synthetic haliclamine E (**1**).

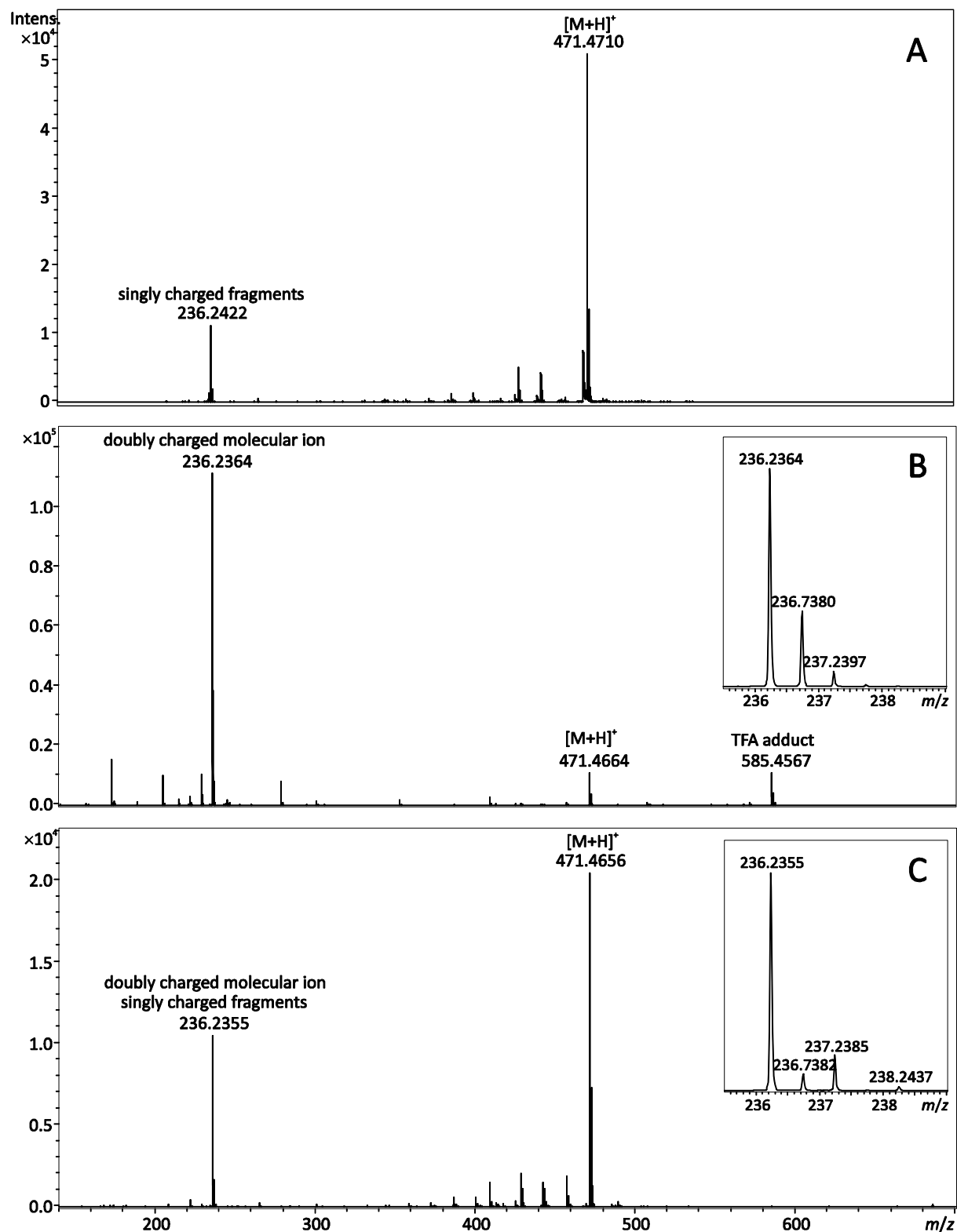


Figure SI-4. Comparison of mass spectra of the natural and synthetic compound **2**. – (A). Mass trace obtained from an LCMS experiment of the crude extract of *Haliclona viscosa*. – (B). Synthetic compound under standard ESI conditions. The section shows a detail of the doubly charged molecular ion. – (C). Synthetic compound under API-CID-MS/MS conditions. The section shows a detail of the doubly charged molecular ion superimposed by the singly charged fragment.

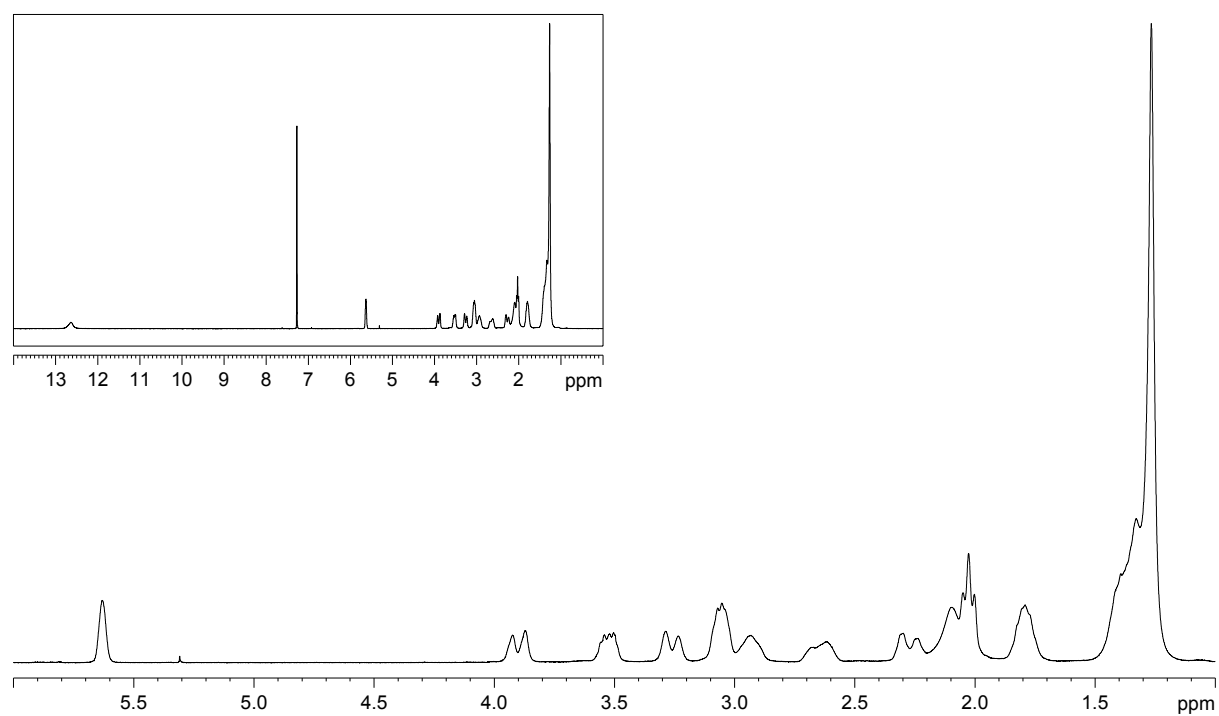


Figure SI-5. Detail and full scan (box) of a ¹H-NMR spectrum (300 MHz, CDCl₃) of the synthetic haliclamine F (**2**).

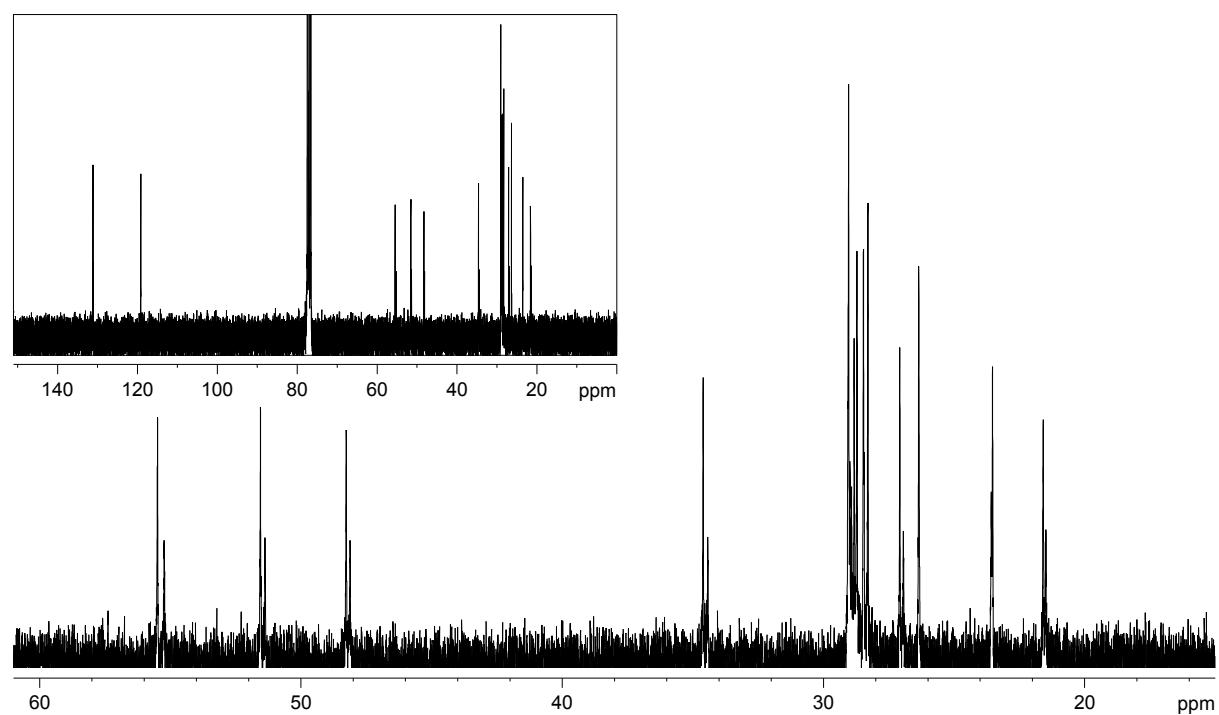
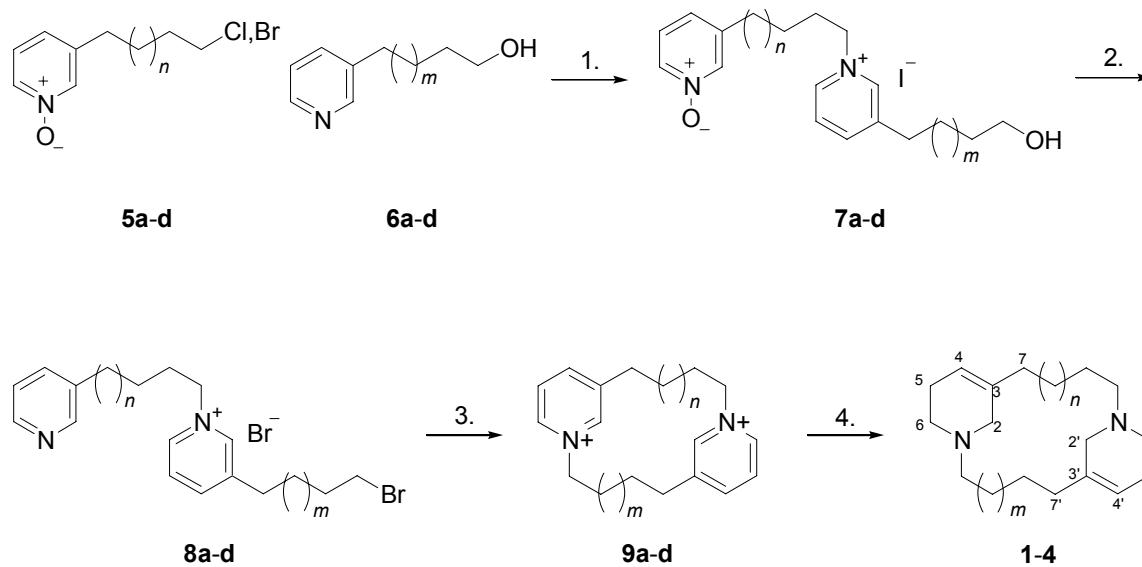


Figure SI-6. Detail and full scan (box) of a ¹³C-NMR spectrum (75 MHz, CDCl₃) of the synthetic haliclamine F (**2**).

Table SI-1. ^1H and ^{13}C NMR data of Haliclamines E (**1**) and F (**2**) in CDCl_3 . The compounds in the solution were present as TFA-salts.

Haliclamine E (1)			Haliclamine F (2)		
pos.	δH	δC	pos.	δH	δC
NH, NH'	12.62 (br. s, 2 H)		NH, NH'	12.62 (s, 2 H)	
2	3.90 (d, 2 H, 15.7 Hz)	51.1	2	3.89 (d, 2 H, 15.8 Hz)	51.5
2'	3.21-3.37 (m, 2 H)	50.8	2'	3.25 (d, 2 H, 16.0 Hz)	51.4
3, 3'		131.1	3, 3'		131.1
4, 4'	5.62 (s, 2 H)	119.1	4, 4'	5.62 (s, 2 H)	119.1
5	2.51-2.69 (m, 2 H)	21.5	5	2.55-2.72 (m, 2 H)	21.6
5'	2.27 (d, 2 H, 17.3 Hz)	21.8	5'	2.26 (d, 2 H, 18.5 Hz)	21.5
6	3.34-3.55 (m, 2 H)	48.7	6	3.46-3.58 (m, 2 H)	48.3
6'	2.87-3.16 (m, 6 H) ^a	47.9	6'	2.85-3.11 (m, 6 H) ^b	48.1
7, 7'	2.20 (t, 4 H, 6.6 Hz)	34.3, 34.7	7, 7'	2.01 (t, 4 H, 7.1 Hz)	34.4, 34.6
8-13, 8'-14'	1.18-1.49 (m, 26 H)	26.0, 26.4, 27.0, 27.1, 27.2, 28.0- 28.7, 29.0	8-15, 8'-15'	1.18-1.49 (m, 32 H)	26.4, 27.0, 27.1, 28.3- 29.0
14, 15'	1.72-1.89 (4 H, m)	23.5, 23.8	16, 16'	1.70-1.85 (m, 4 H)	23.5
15, 16'	2.87-3.16 (m, 6 H) ^a	54.8, 55.5	17, 17'	2.85-3.11 (m, 6 H) ^b	55.2, 55.5

^a Assignments are interchangeable. ^b Assignments are interchangeable.



a = 1: $n = 6, m = 7$; **b = 2:** $n = 8, m = 8$; **c = 3:** $n = 6, m = 8$; **d = 4:** $n = 7, m = 8$

Figure SI-7. Synthetic pathway of haliclamine E (**1**), F (**2**), C (**3**) and D (**4**). – 1. Synthesis of *N*-oxide dimers. – 2./3. Synthesis of cyclostelletamines. – 4. Synthesis of haliclamine (refer to paragraphs in the text).