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Guaianolides and a seco-eudesmane from the resinous exudates of cushion bush (*Leucophyta brownii*) and evaluation of their cytostatic and anti-inflammatory activity

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Supporting Information

Guaianolides and a seco-Eudesmane from the Resinous Exudates of Cushion Bush (*Leucophyta brownii*) and Evaluation of Their Cytostatic and Anti-inflammatory Activity

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Supporting Information Contents

Supporting information contains an analytical HPLC chromatogram of the dichloromethane extract of fresh aerial parts of *Leucophyta brownii* at 210 nm showing the separation and t_R of the isolated compounds **1–10**, ¹H, ¹³C, DEPT, and 2D NMR (¹H–¹H COSY, NOESY, HSQC, and HMBC) spectra of compounds **1–10**, crystal structures (ORTEP diagrams) of the sesquiterpene lactones **2–4**, **7**, and **8** with displacement ellipsoids at 50% probability for non-H atoms, and a description of physical (melting point, optical activity), spectroscopic (UV, IR) and spectrometric (APCIMS, HRESIMS) data of compounds **1–4** and **7–9** (page 5 to 92).

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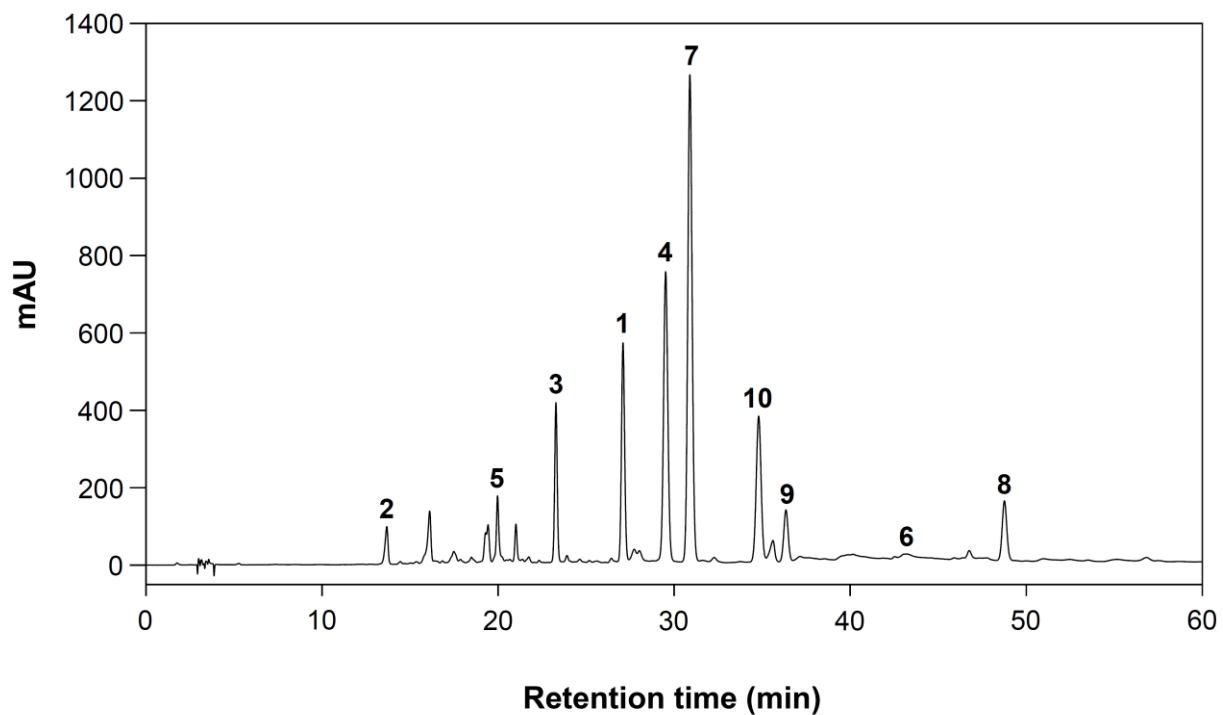
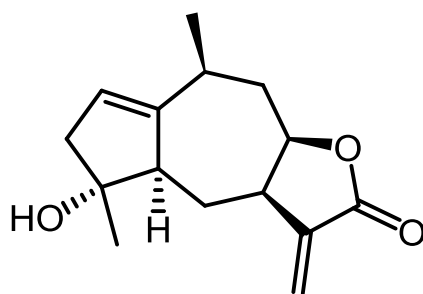


Fig. S1. Analytical HPLC chromatogram of the dichloromethane extract of fresh aerial parts of *Leucophyta brownii* at 210 nm (Peak numbers 1–10 in the HPLC chromatogram refer to compound numbers).

4 α -Hydroxy-5 α H,10 α H-1,11(13)-guaidien-8 β ,12-olide (1)

Colorless resin-like oil; $[\alpha]_D^{25} +153$ (c 0.4, MeOH); UV (MeOH) λ_{\max} (log ϵ) 205 (3.66) nm; IR (KBr) ν_{\max} 3338, 2926, 1771, 1660, 1110 cm^{-1} APCIMS: m/z 249 $[\text{M} + \text{H}]^+$ (14), 231 $[\text{M} + \text{H} - \text{H}_2\text{O}]^+$ (100); HRESIMS m/z 249.1489 (calcd for $\text{C}_{15}\text{H}_{21}\text{O}_3$, 249.1491).



1

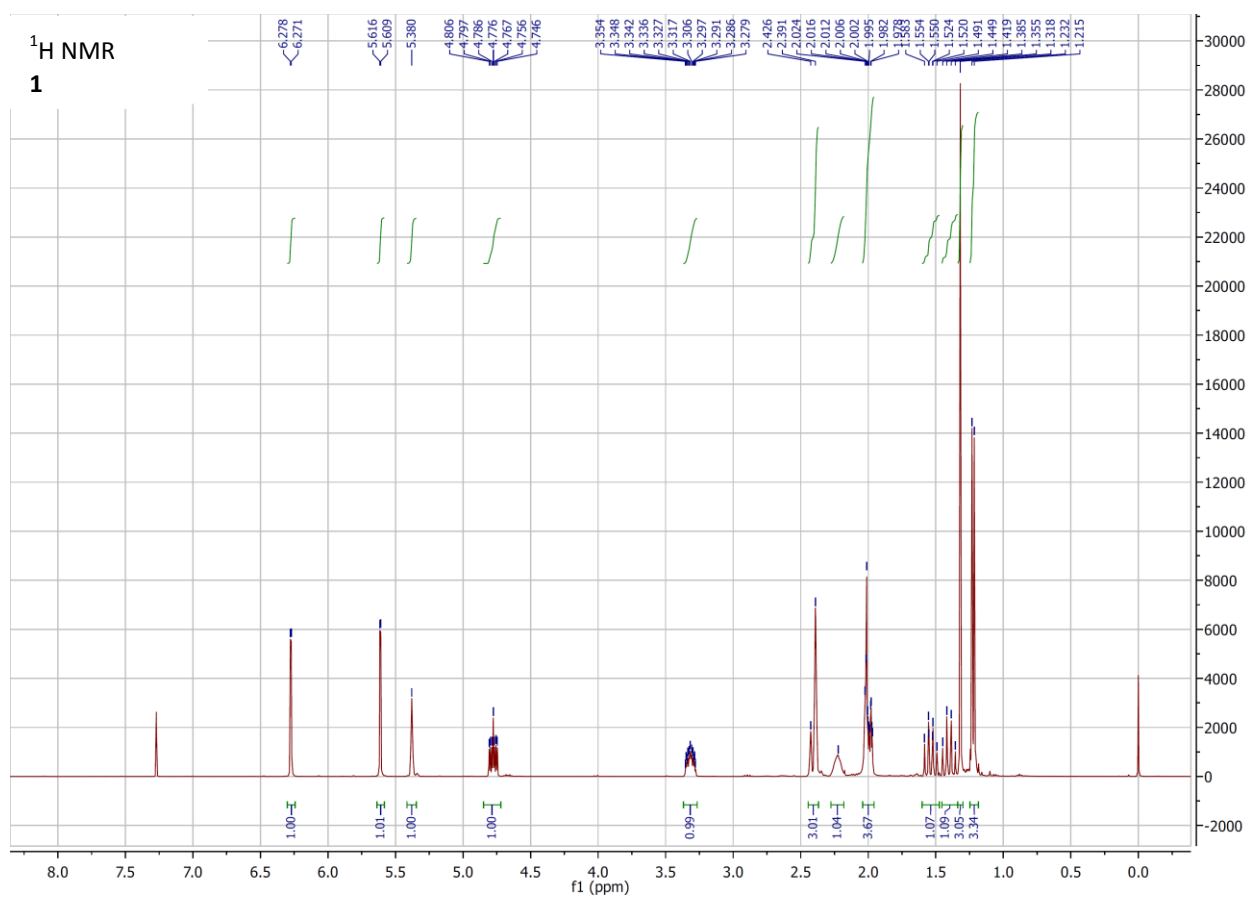


Fig. S2. ¹H NMR (400 MHz, CDCl₃) spectrum of compound 1.

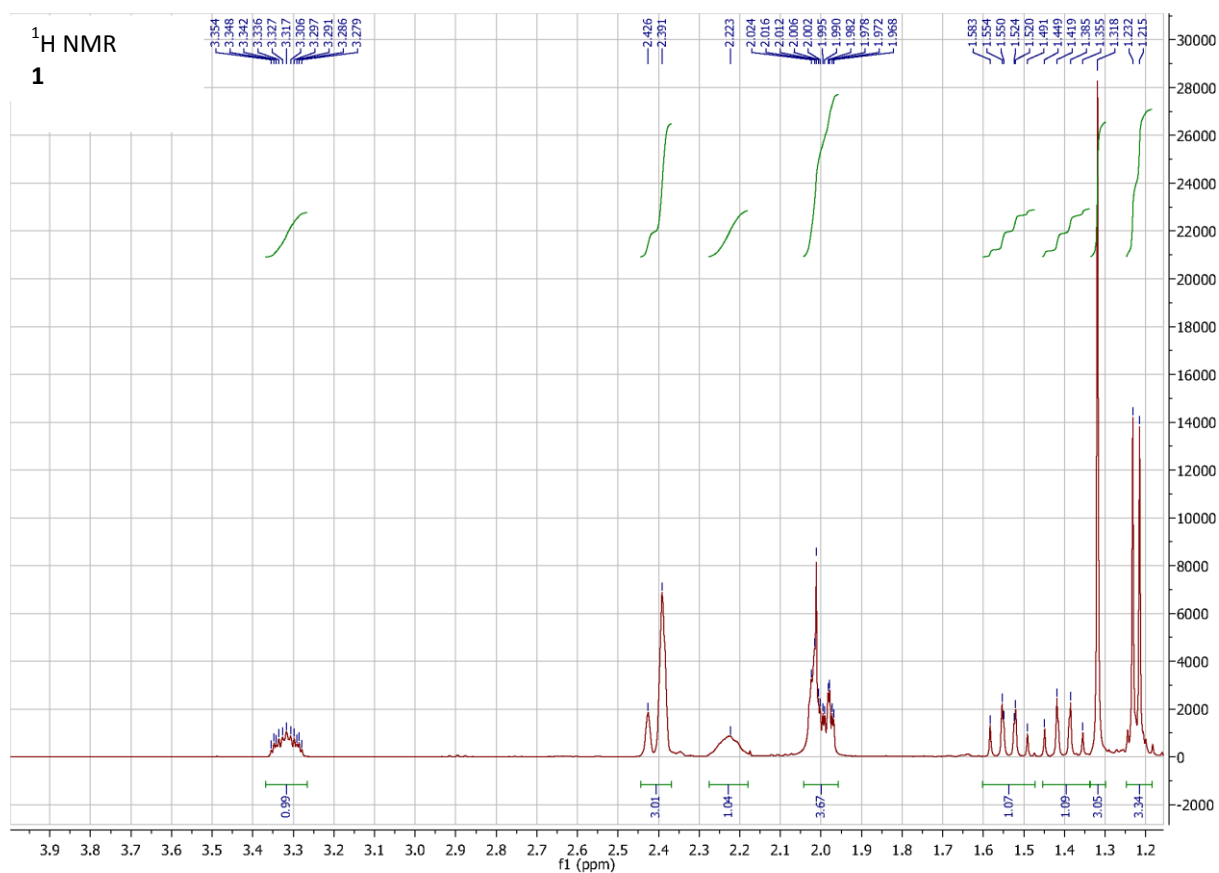


Fig. S3. ¹H NMR (400 MHz, CDCl₃) spectrum of compound **1** expanded in the region 1.1–4.0 ppm.

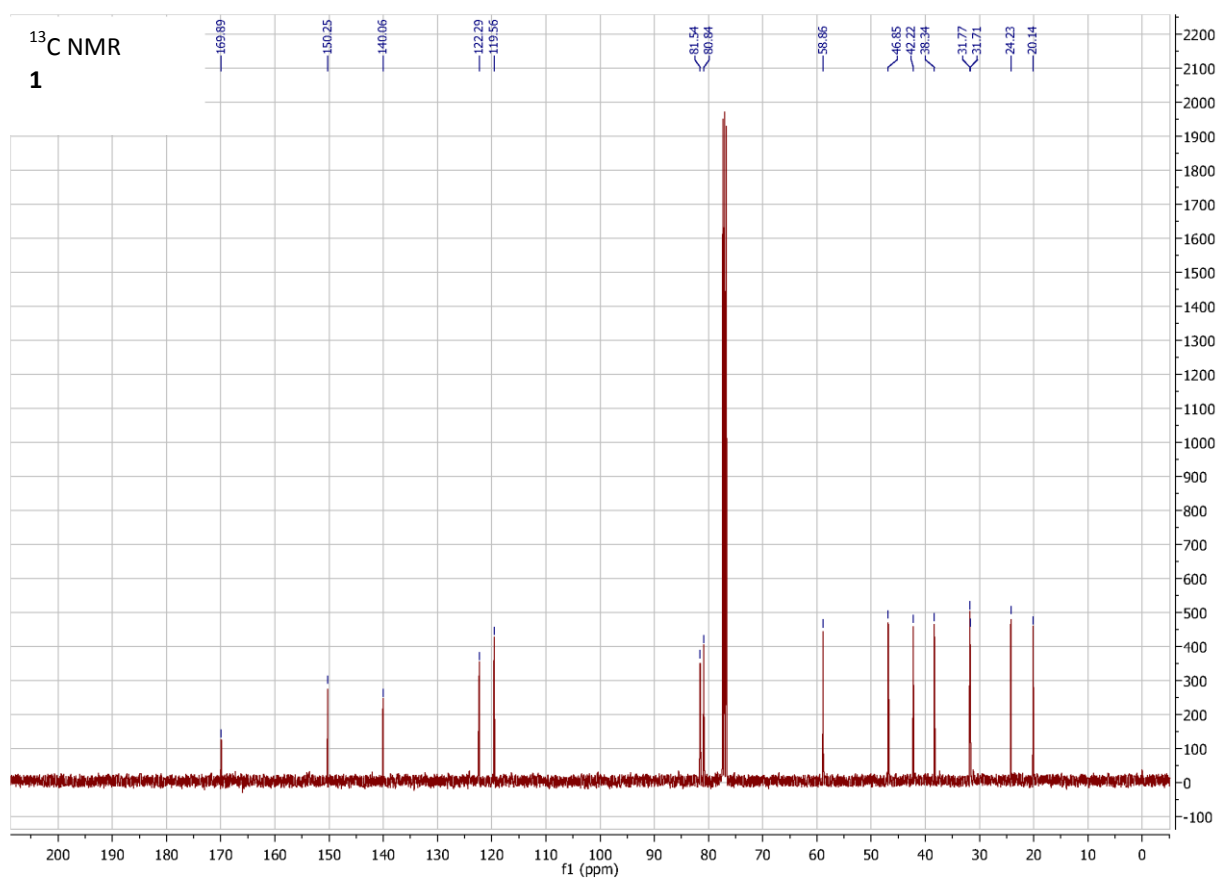


Fig. S4. ¹³C NMR (100 MHz, CDCl₃) spectrum of compound **1**.

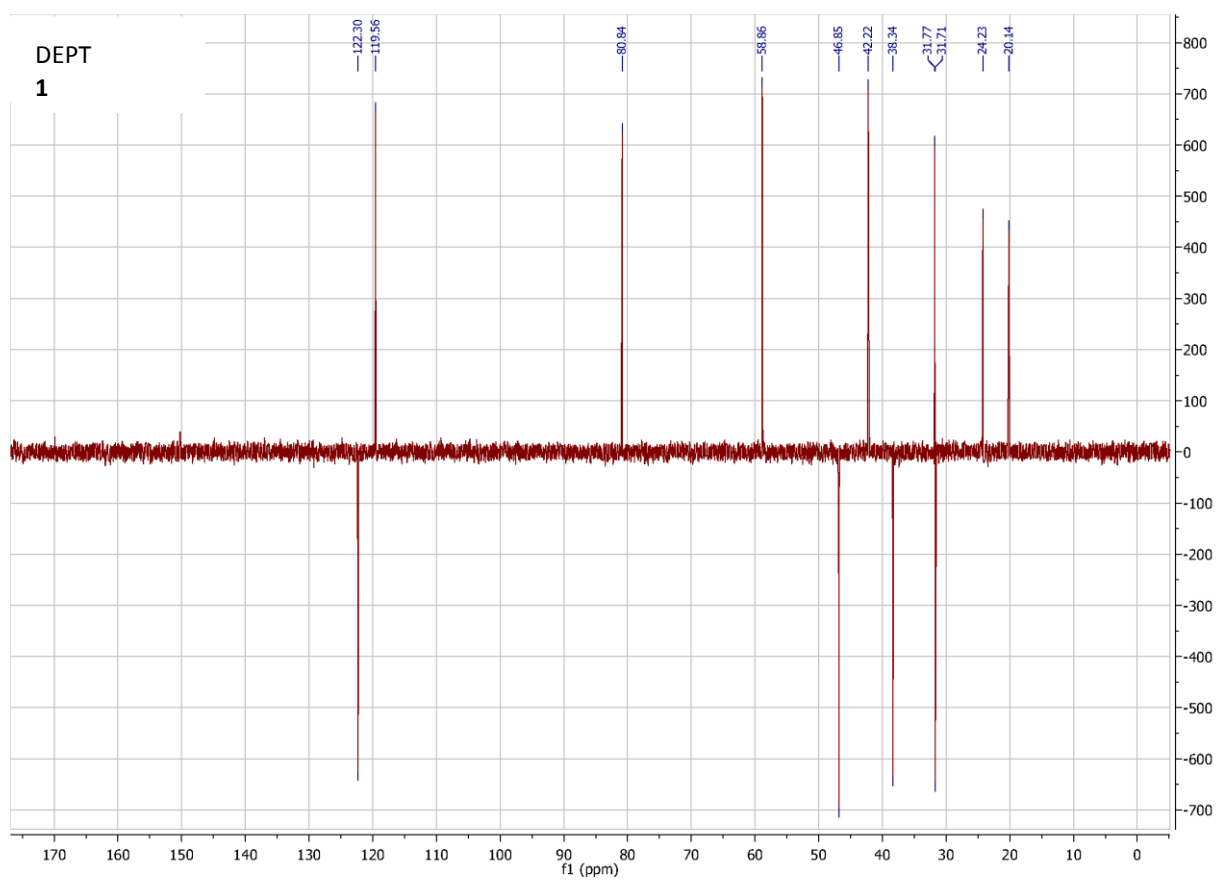


Fig. S5. DEPT spectrum of compound **1**.

COSY
1

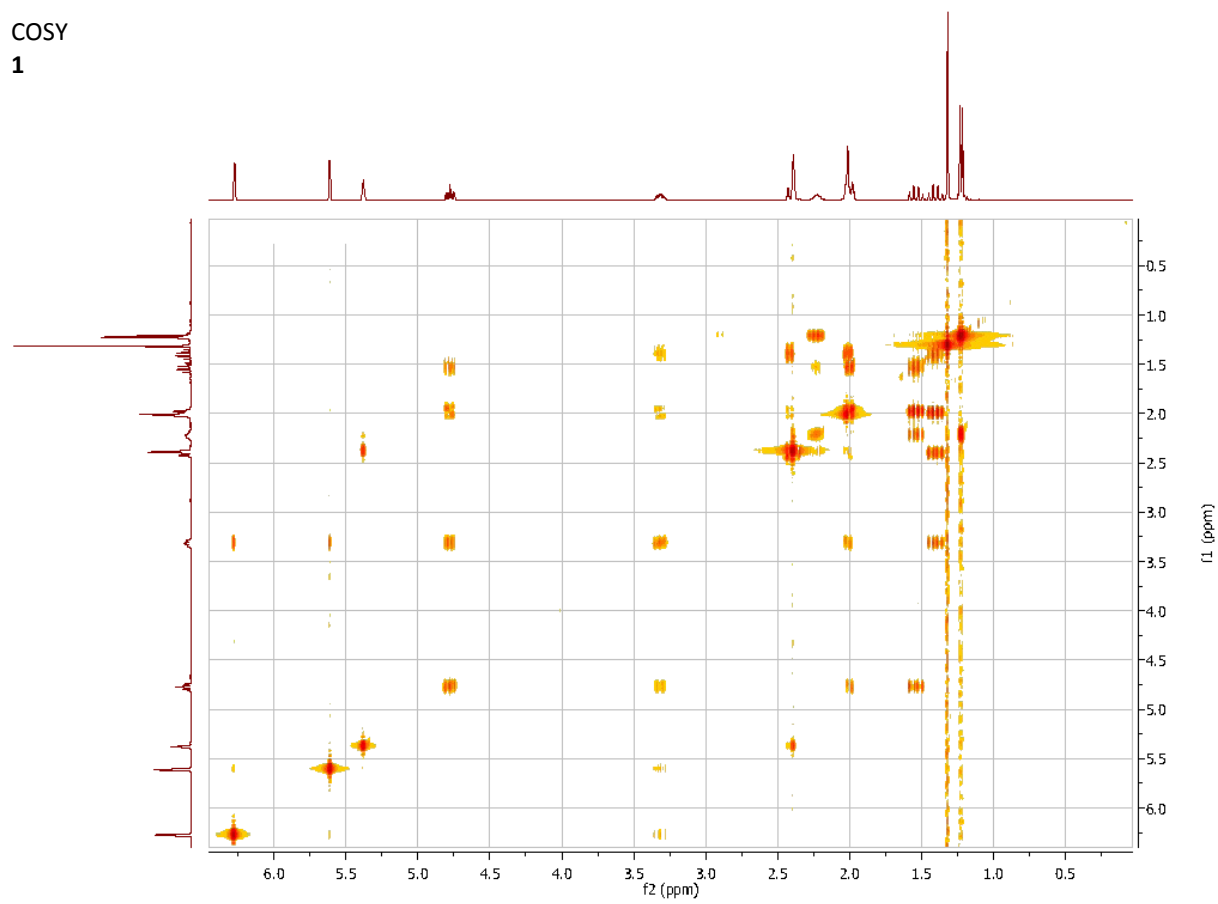


Fig. S6. ^1H – ^1H COSY spectrum of compound **1**.

NOESY
1

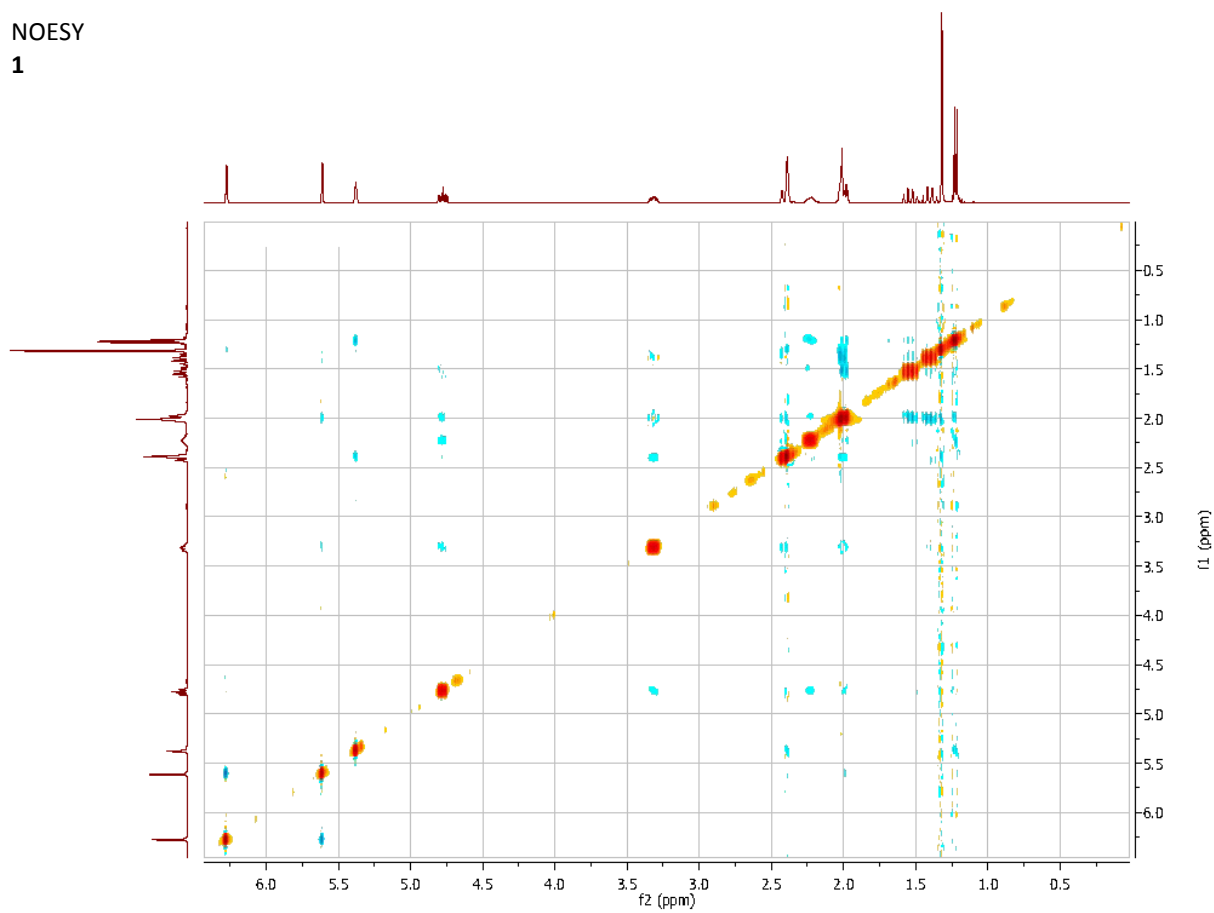


Fig. S7. NOESY spectrum of compound **1**.

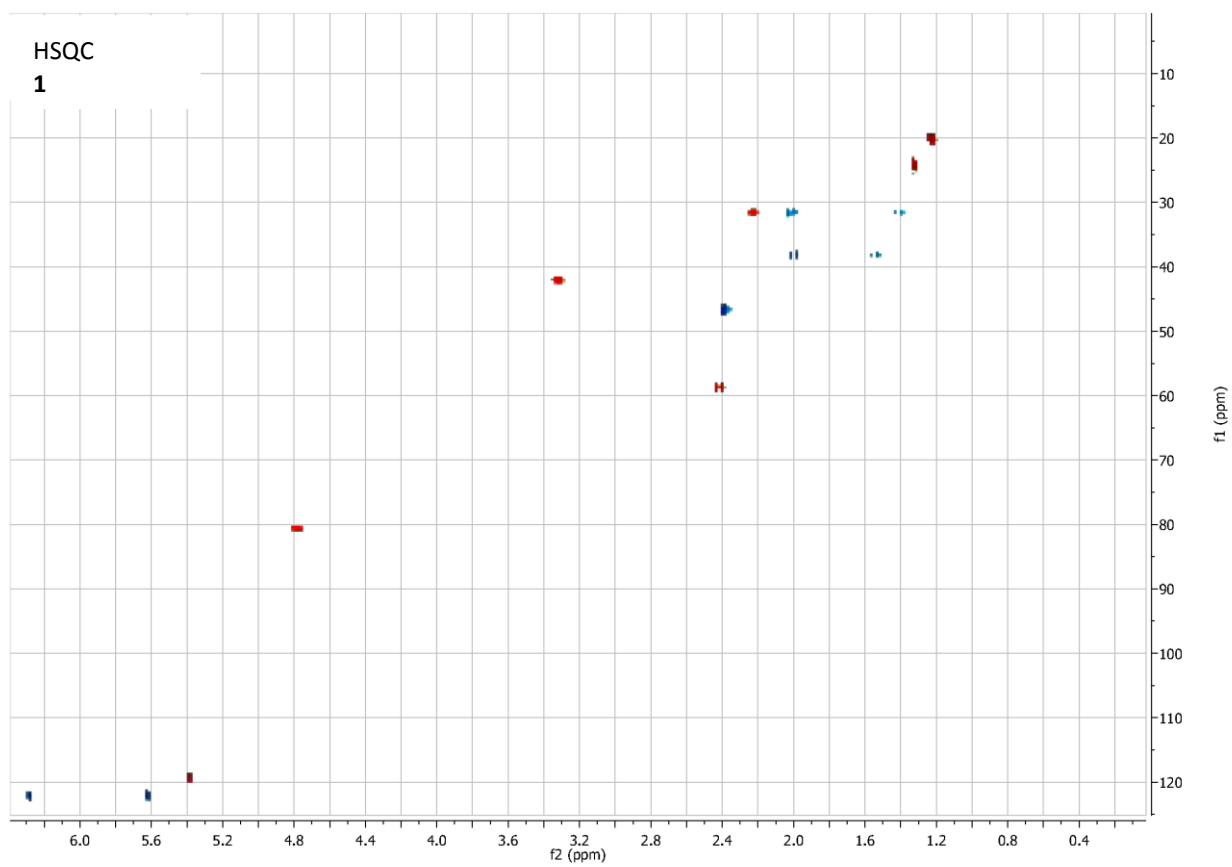


Fig. S8. HSQC spectrum of compound **1**.

HMBC
1

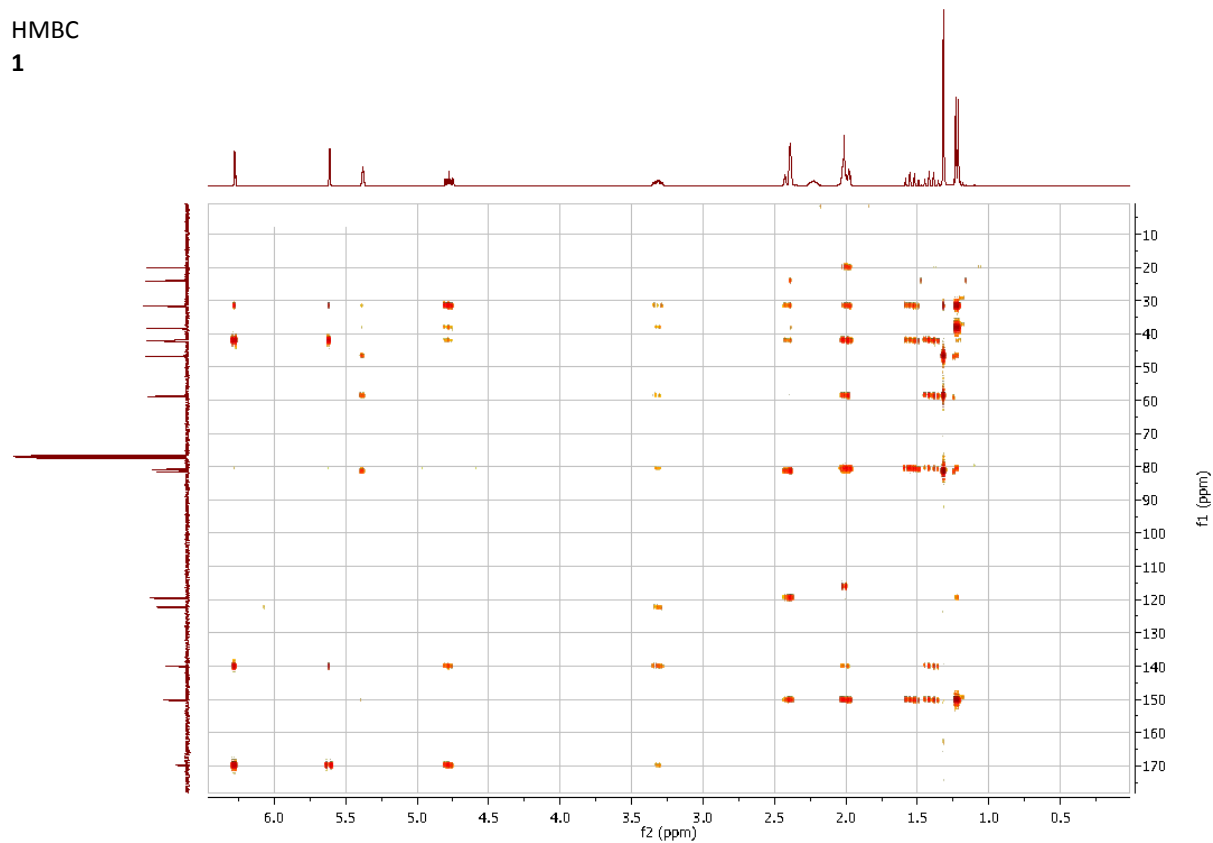


Fig. S9. HMBC spectrum of compound **1**.

4 α ,10 β -Dihydroxy-5 α H-1,11(13)-guaidien-8 β ,12-olide (2)

Colorless needle-like crystals; mp 169–170 °C; $[\alpha]_D^{25} +34$ (c 0.4, MeOH); UV (MeOH) λ_{\max} (log ϵ) 209 (3.84) nm; IR (KBr) ν_{\max} 3338, 2941, 1758, 1655, 1380, 1273, 1169 cm^{-1} . APCIMS: m/z 265 $[\text{M} + \text{H}]^+$ (8), 247 $[\text{M} - \text{H}_2\text{O} + \text{H}]^+$ (51), 229 $[\text{M} - 2 \text{H}_2\text{O} + \text{H}]^+$ (100); HRESIMS m/z 265.1441 (calcd for $\text{C}_{15}\text{H}_{21}\text{O}_4$, 265.1440).

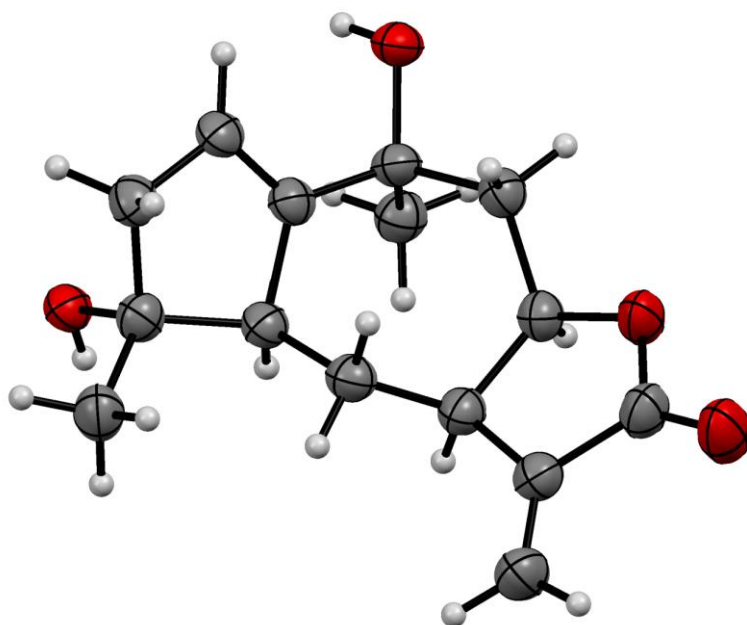
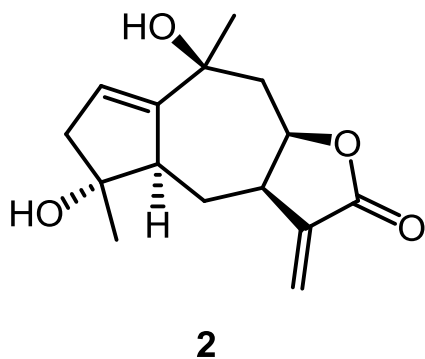


Fig. S10. Crystal structure of compound **2** (ORTEP diagram) with displacement ellipsoids at 50% probability for non-H atoms.

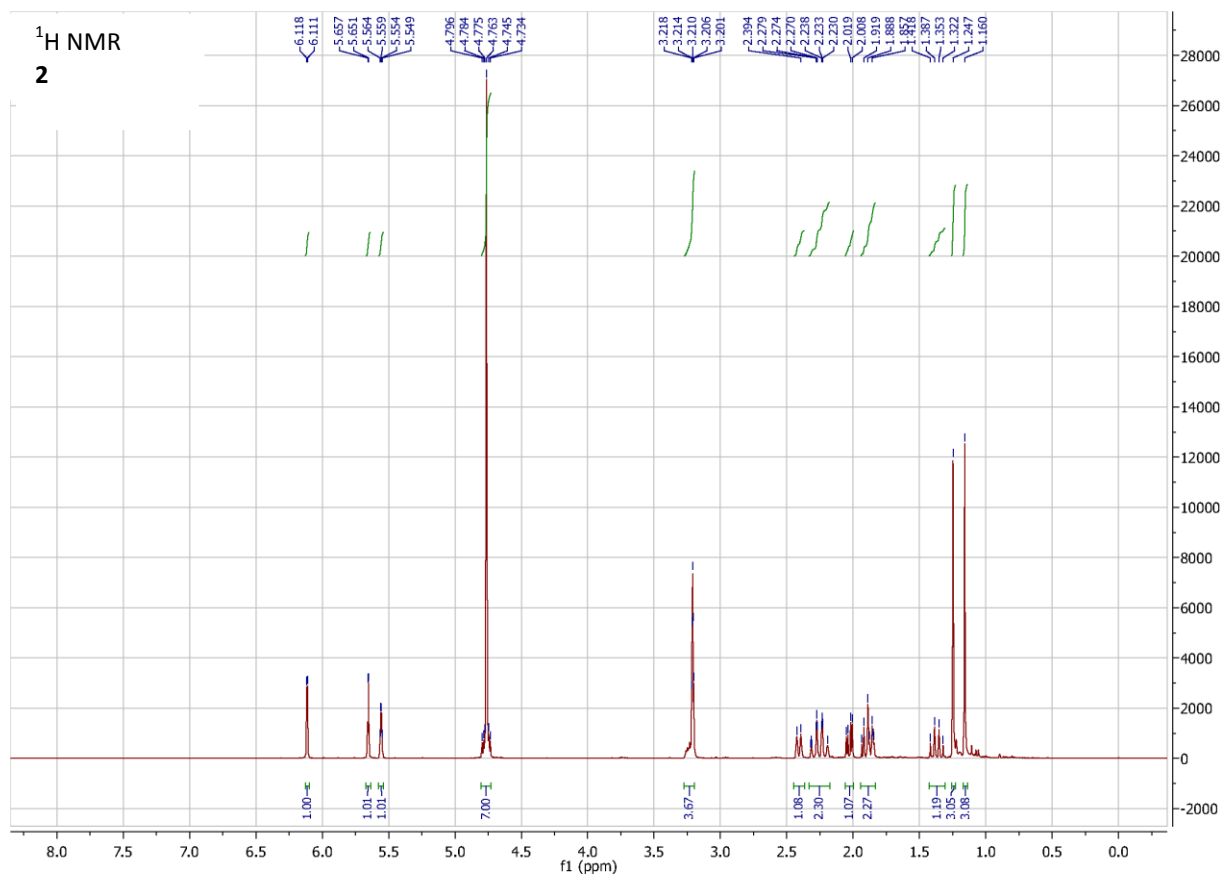


Fig. S11. ¹H NMR (400 MHz, methanol-*d*₄) spectrum of compound 2.

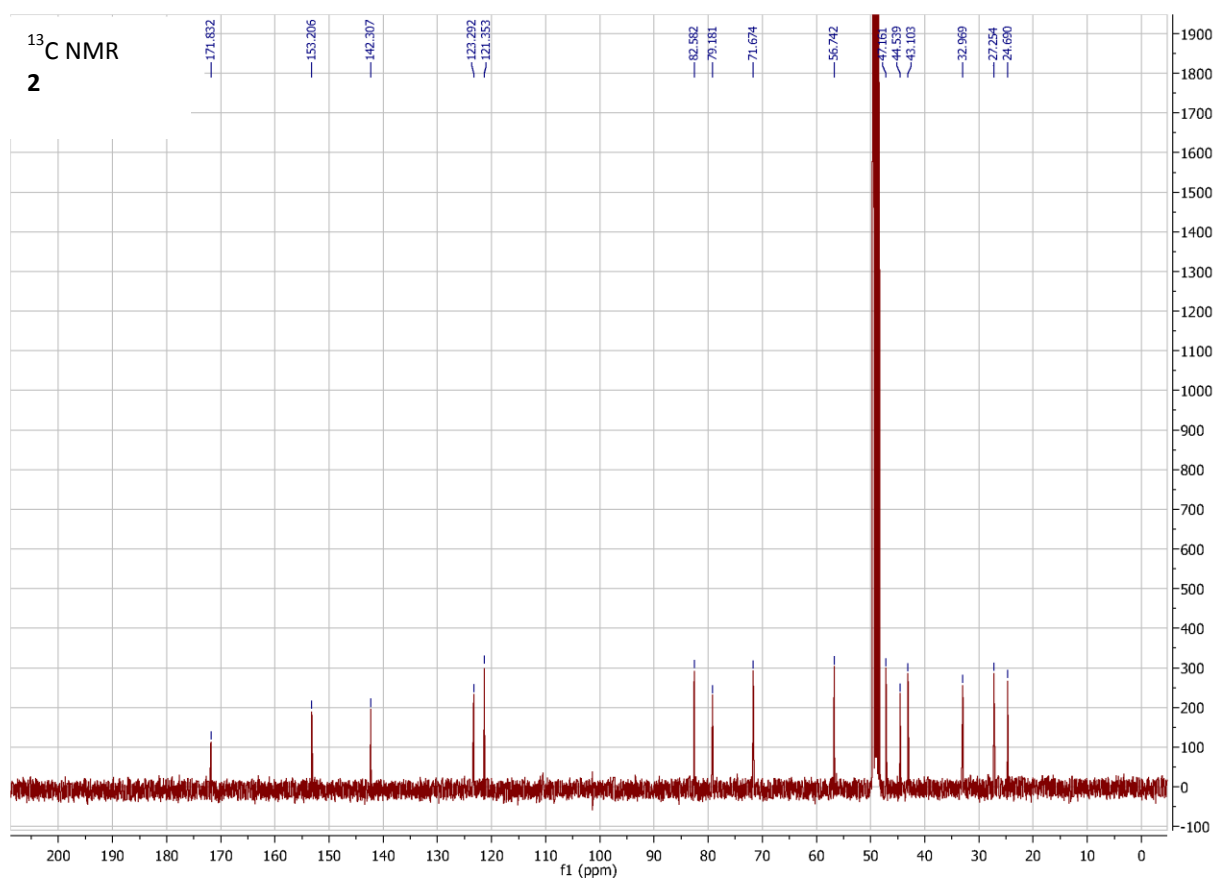


Fig. S12. ¹³C NMR (100 MHz, methanol-*d*₄) spectrum of compound 2.

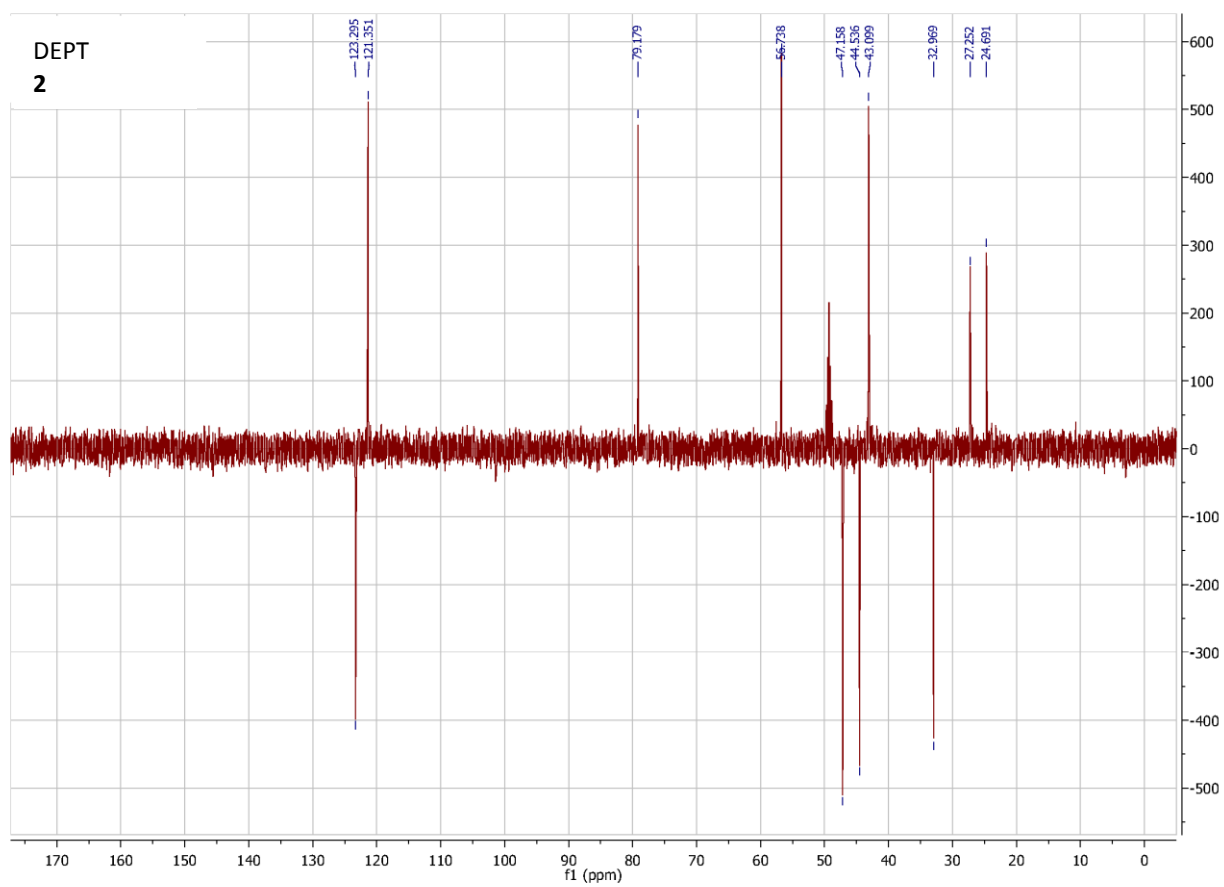


Fig. S13. DEPT spectrum of compound 2.

COSY
2

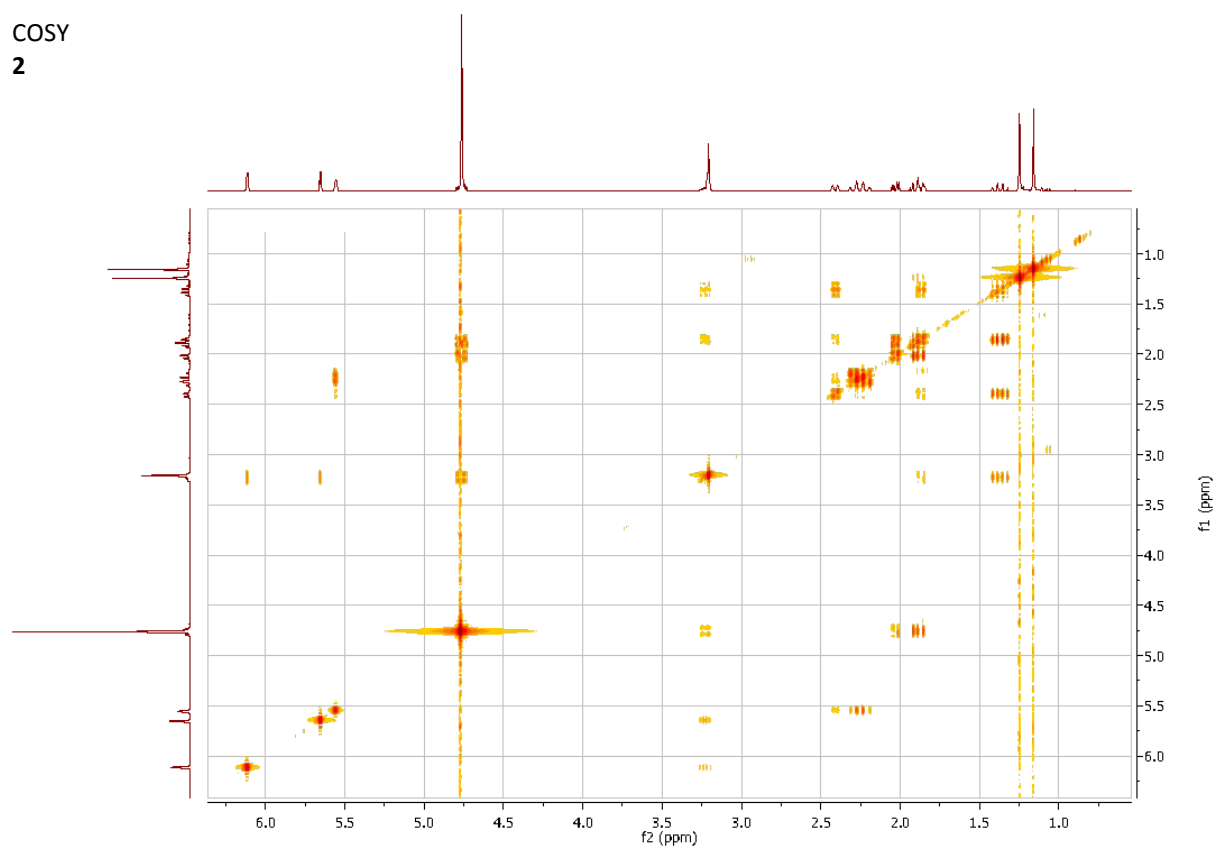


Fig. S14. ^1H - ^1H COSY spectrum of compound **2**.

NOESY
2

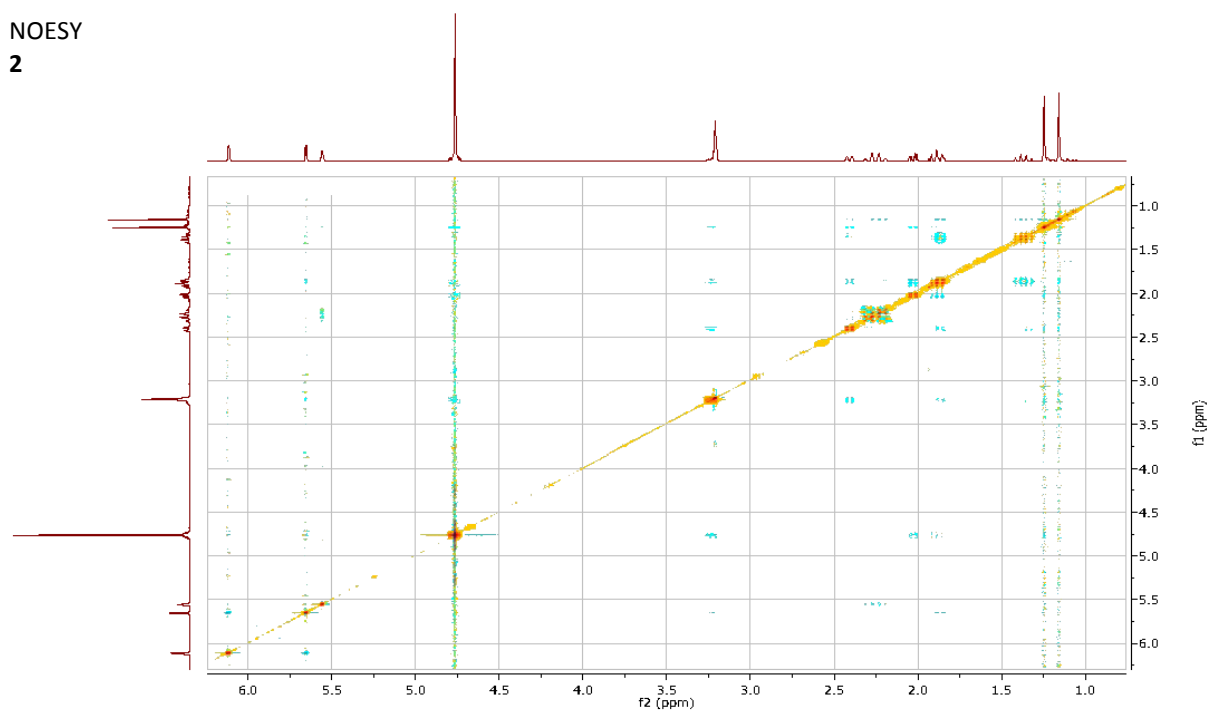


Fig. S15. NOESY spectrum of compound 2.

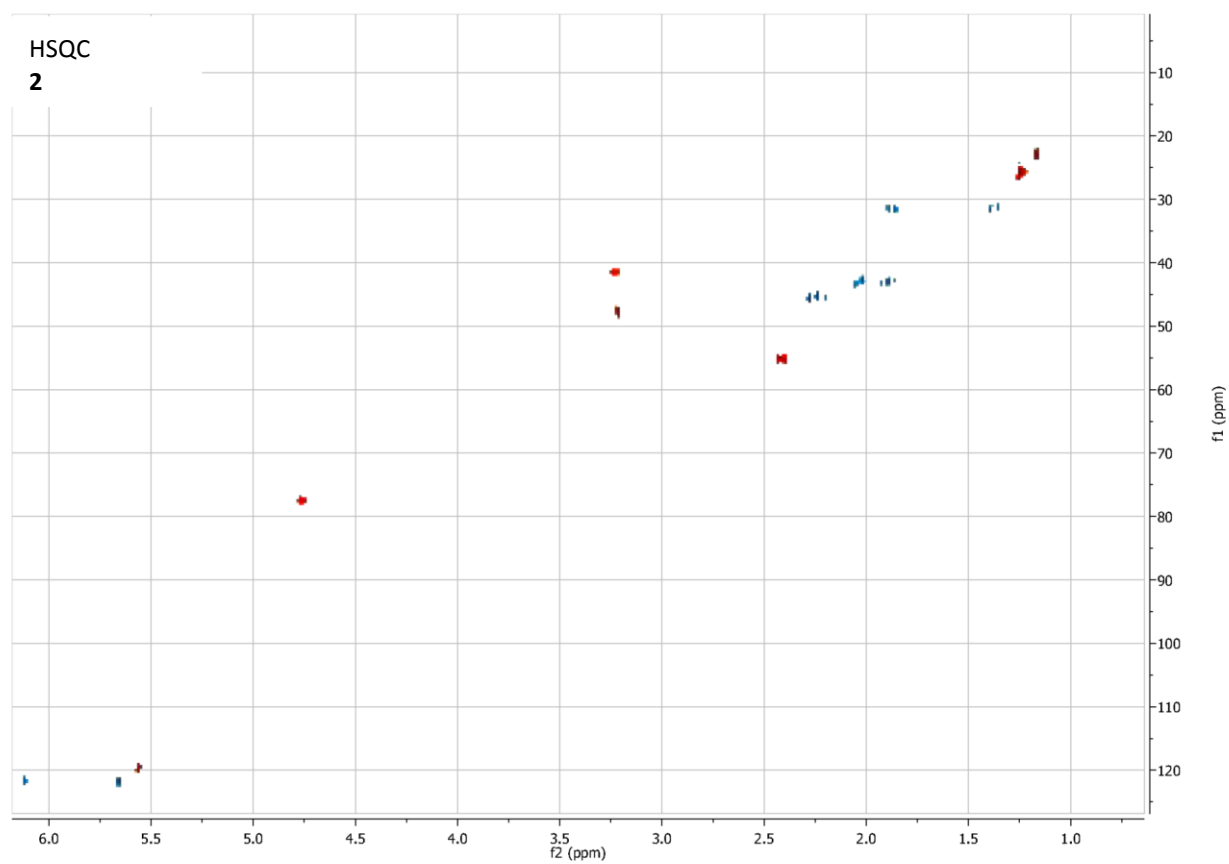


Fig. S16. HSQC spectrum of compound **2**.

HMBC
2

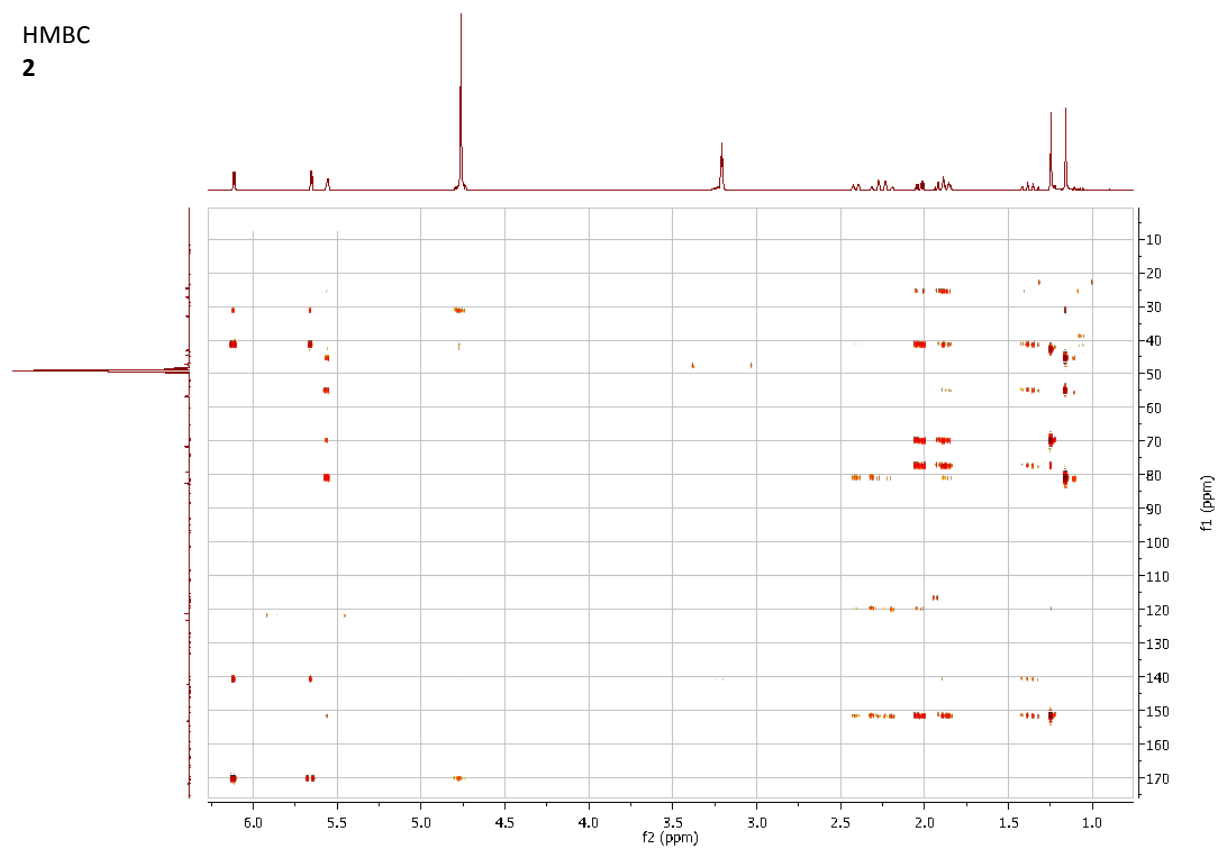
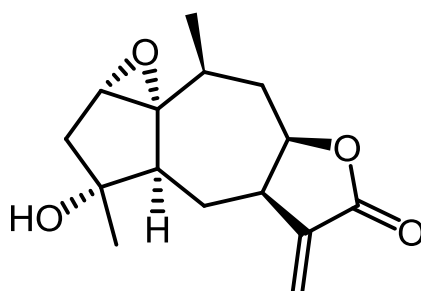


Fig. S17. HMBC spectrum of compound **2**.

4 α -Hydroxy-1 α ,2 α -epoxy-5 α H,10 α H-11(13)-guaian-8 β ,12-olide (3)

Colorless crystals; mp 168–169 °C; $[\alpha]_D^{25} +31$ (c 0.4, CHCl₃); UV (MeOH) λ_{\max} (log ϵ) 210 (4.11) nm; IR (KBr) ν_{\max} 3497, 2949, 1757, 1658, 1398, 1271, 1131 cm⁻¹; APCIMS: m/z 265 [M + H]⁺ (13), 247 [M – H₂O + H]⁺ (39), 229 [M – 2 H₂O + H]⁺ (100); HRESIMS m/z 265.1440 (calcd for C₁₅H₂₁O₄, 265.1440).



3

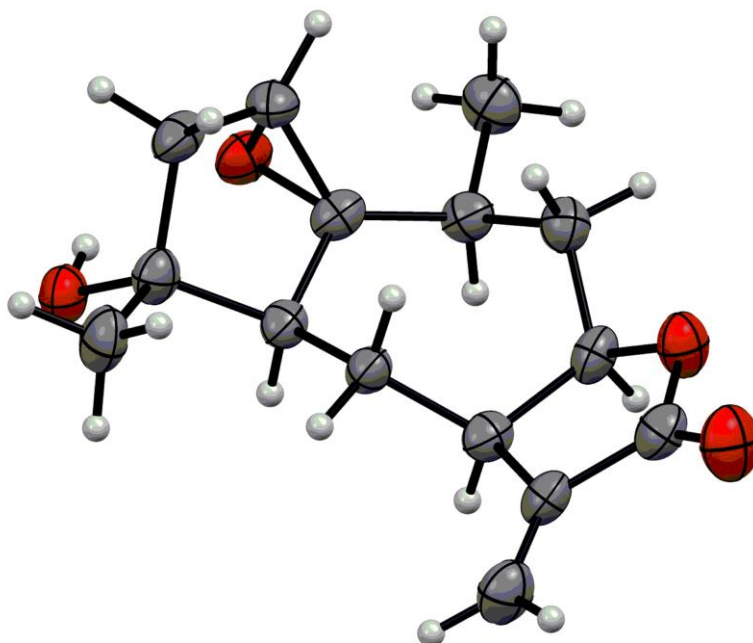


Fig. S18. Crystal structure of compound **3** (ORTEP diagram) with displacement ellipsoids at 50% probability for non-H atoms.

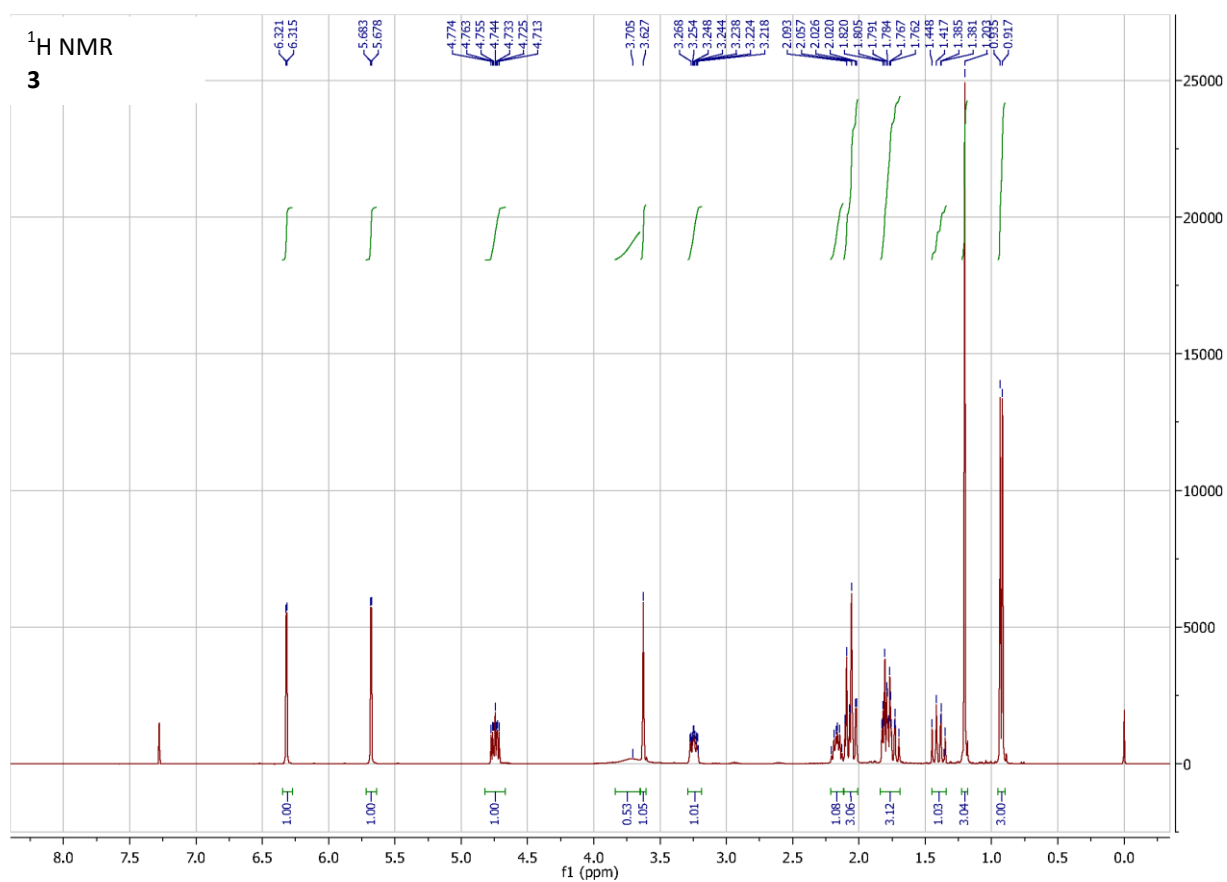


Fig. S19. ¹H NMR (400 MHz, CDCl₃) spectrum of compound **3**.

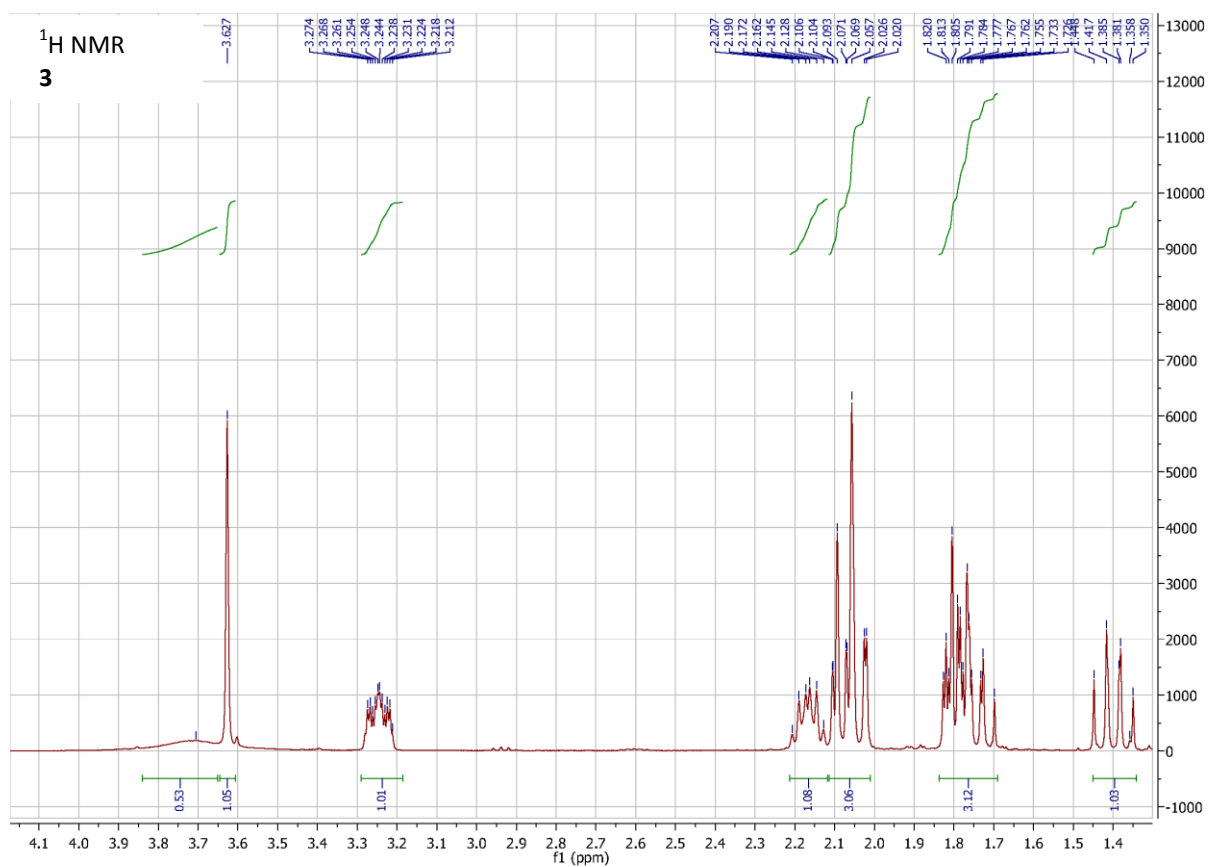


Fig. S20. ¹H NMR (400 MHz, CDCl₃) spectrum of compound **3** expanded in the region 1.3–4.2 ppm.

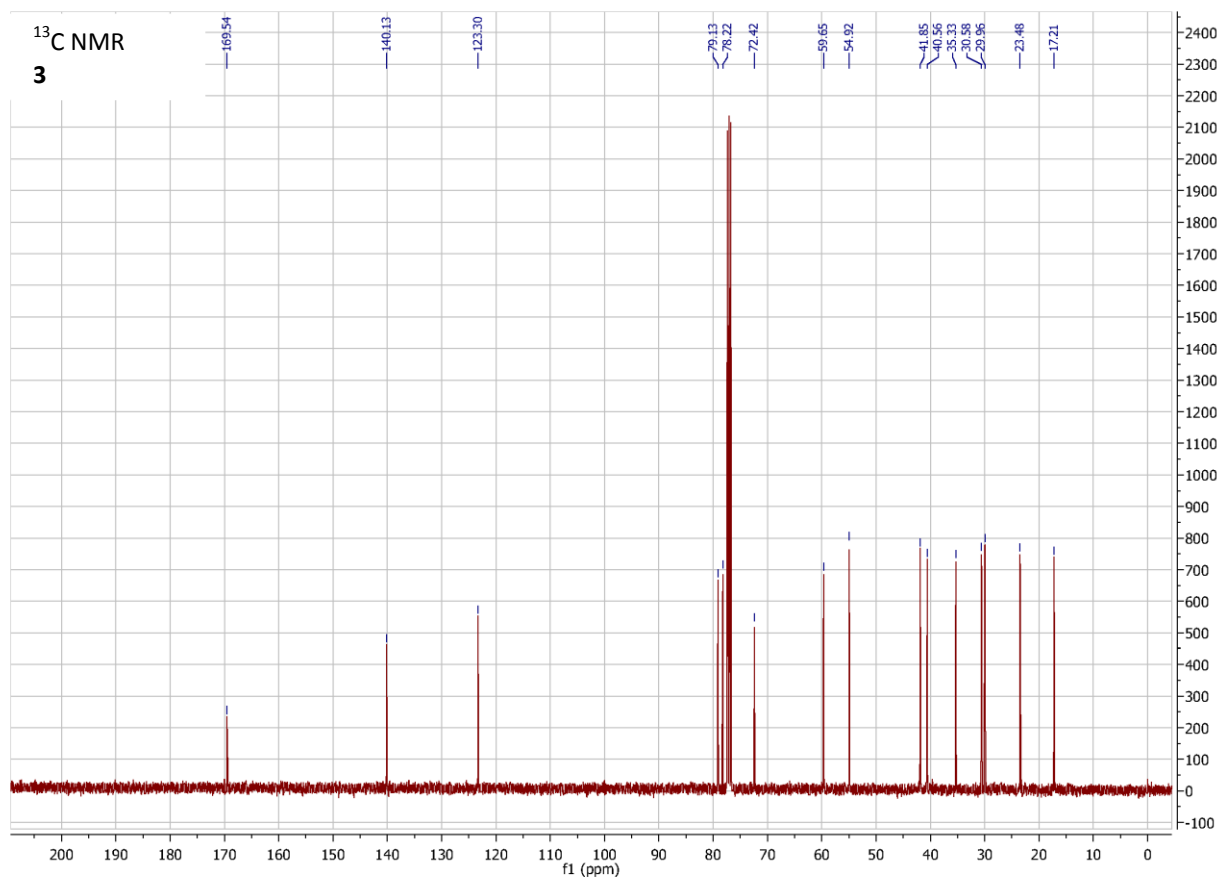


Fig. S21. ¹³C NMR (100 MHz, CDCl₃) spectrum of compound 3.

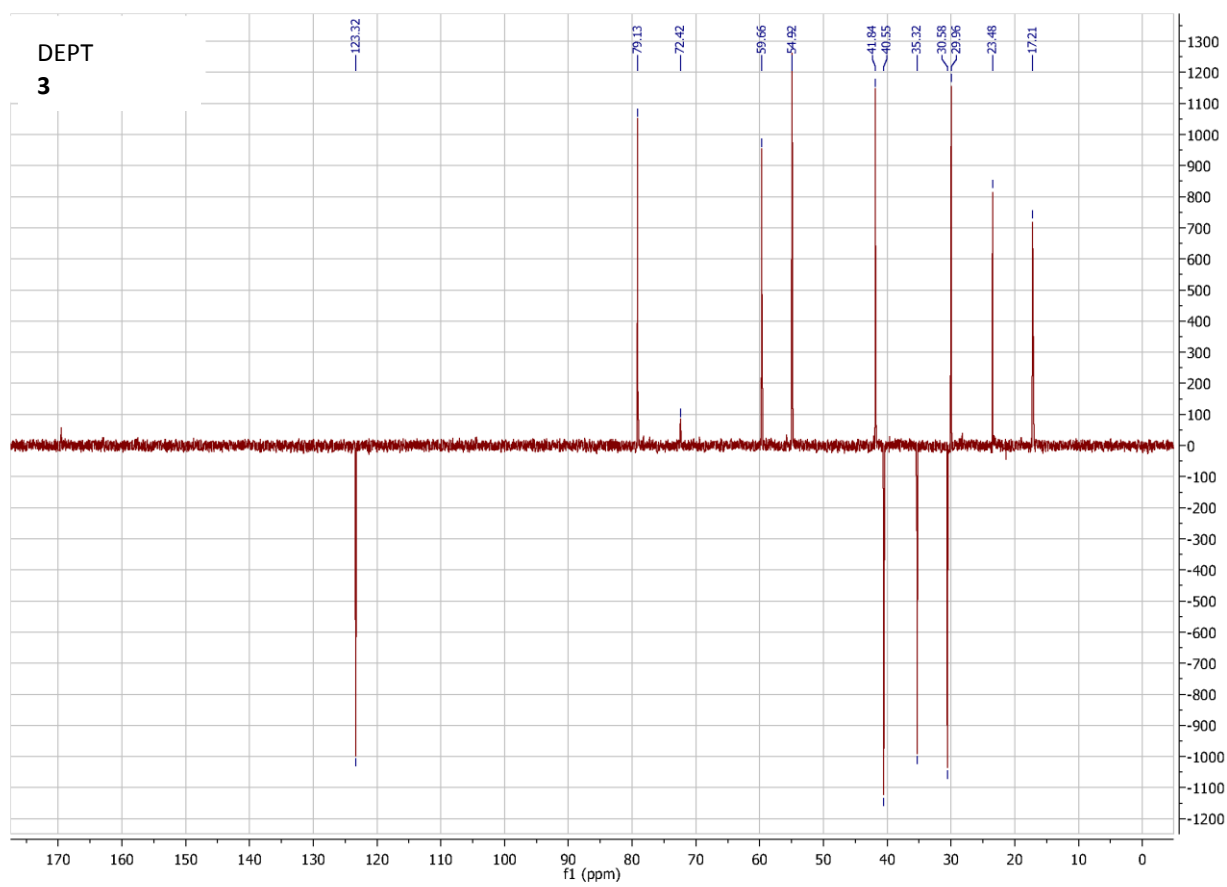


Fig. S22. DEPT spectrum of compound 3.

COSY
3

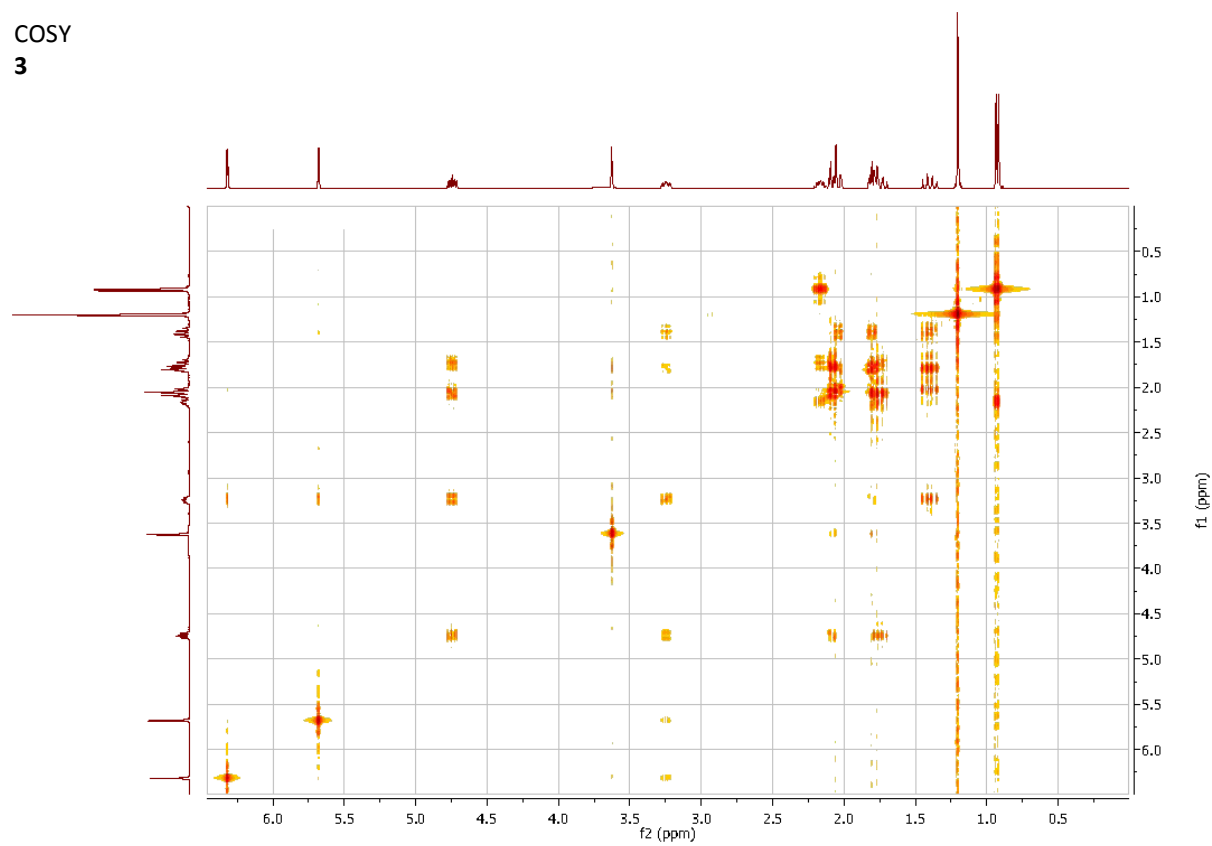


Fig. S23. ¹H–¹H COSY spectrum of compound 3.

NOESY
3

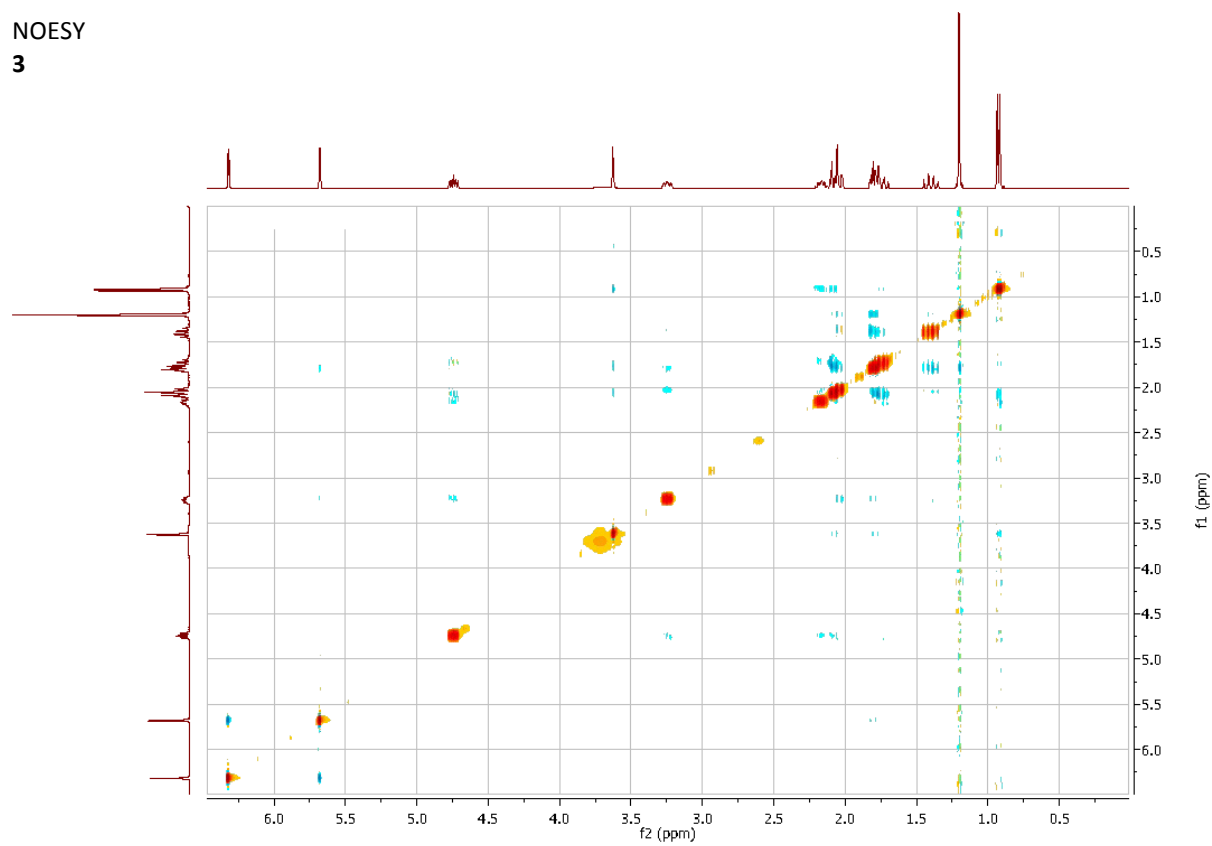


Fig. S24. NOESY spectrum of compound 3.

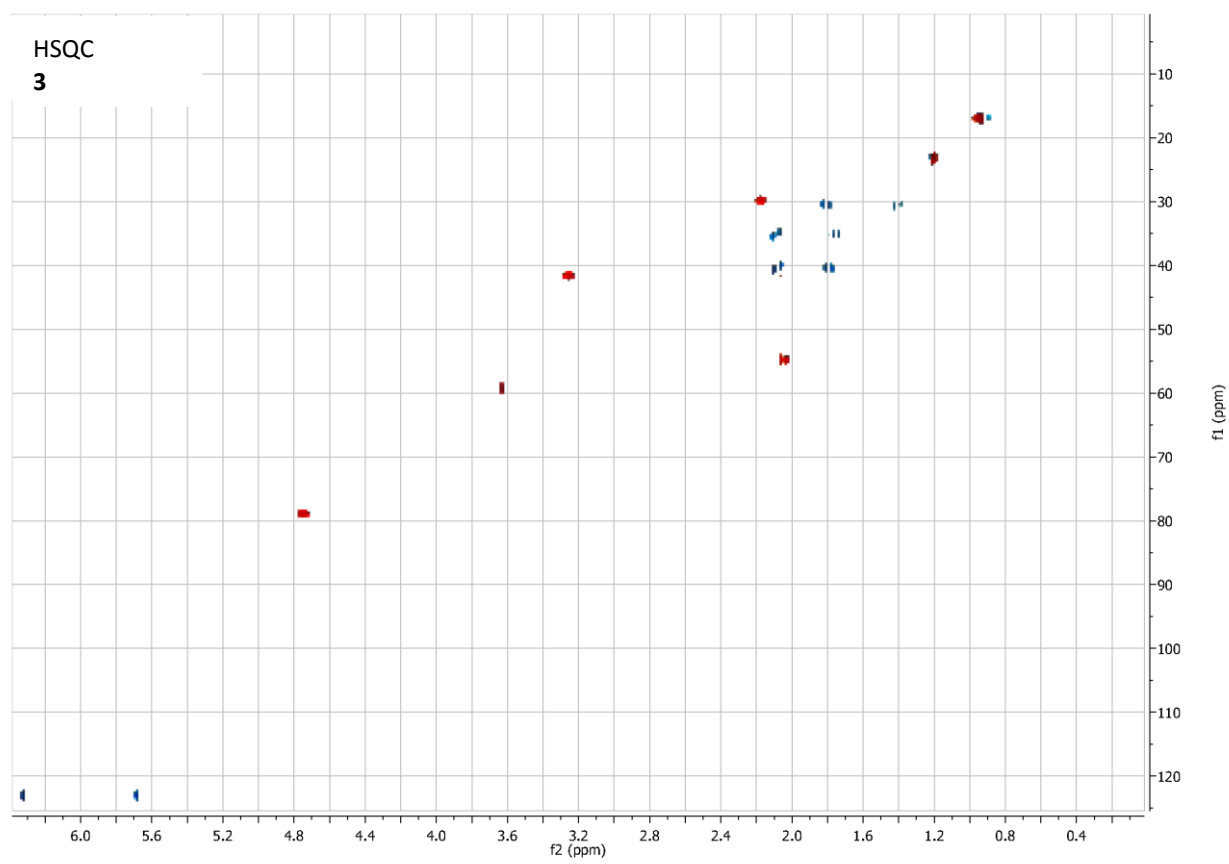


Fig. S25. HSQC spectrum of compound **3**.

HMBC
3

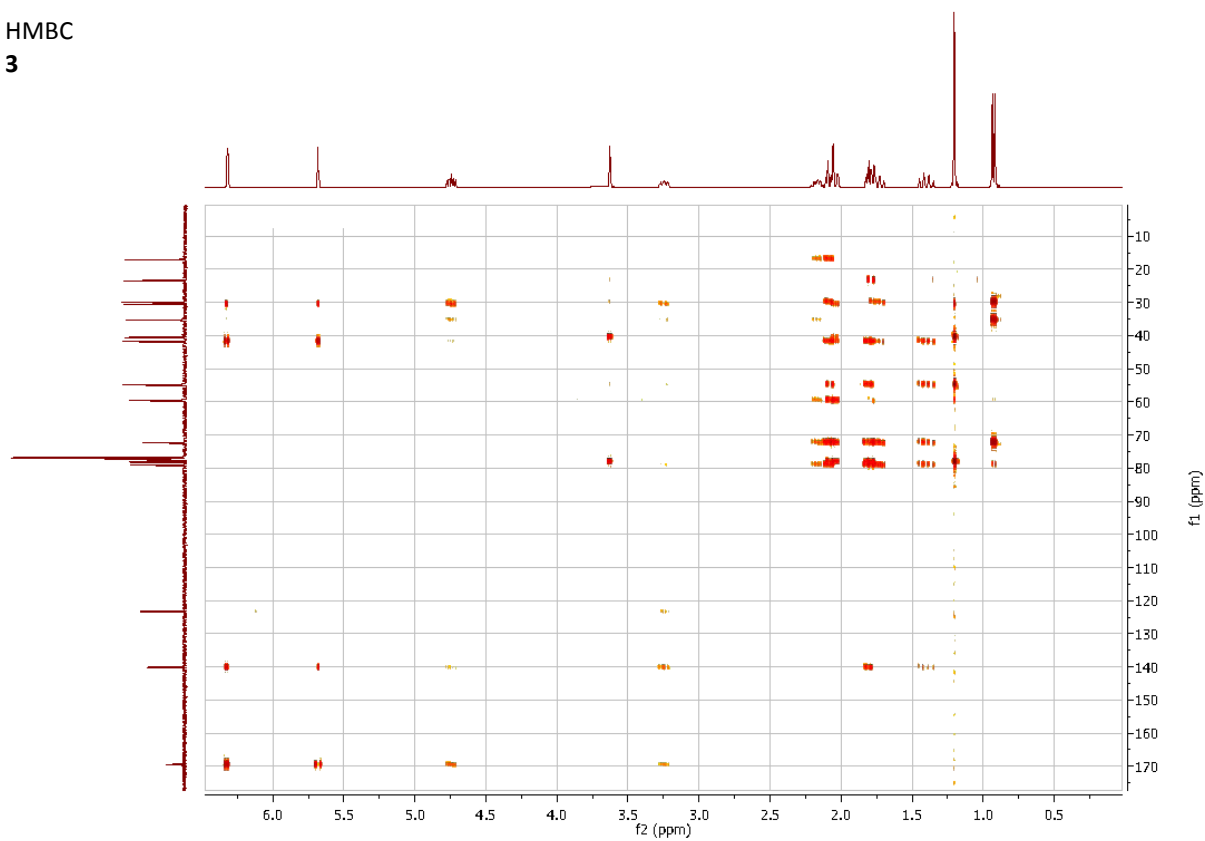
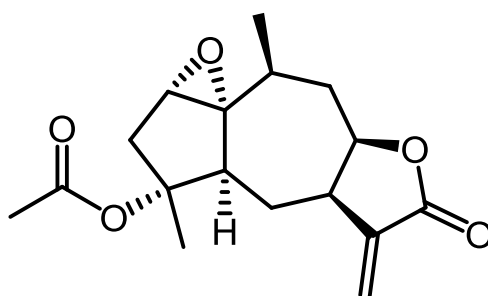


Fig. S26. HMBC spectrum of compound 3.

Calocephalin [*4* α -acetoxy-1 α ,2 α -epoxy-5 α H,10 α H-11(13)-guaian-8 β ,12-olide] (**4**)

Colorless crystals; mp 125–126 °C; $[\alpha]_D^{25} +14$ (c 0.4, CHCl₃); UV (MeOH) (log ϵ) 212 (4.12) nm; IR (KBr) ν_{\max} 3425, 2920, 1746, 1716, 1657, 1459, 1370, 1263, 1115, 1007 cm⁻¹; APCIMS: m/z 307 [M + H]⁺ (23), 264 [M + H – Ac]⁺ (16), 247 [M + H – HOAc]⁺ (100), 229 [M + H – HOAc – H₂O]⁺ (93); HRESIMS m/z 307.1540 (calcd for C₁₇H₂₃O₅, 307.1546).



Calocephalin (**4**)

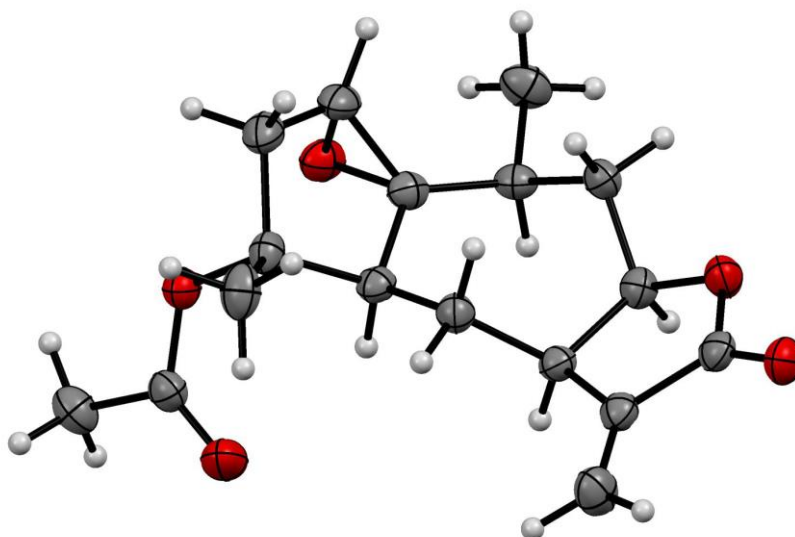


Fig. S27. Crystal structure of calocephalin (**4**) [ORTEP diagram] with displacement ellipsoids at 50% probability for non-H atoms.

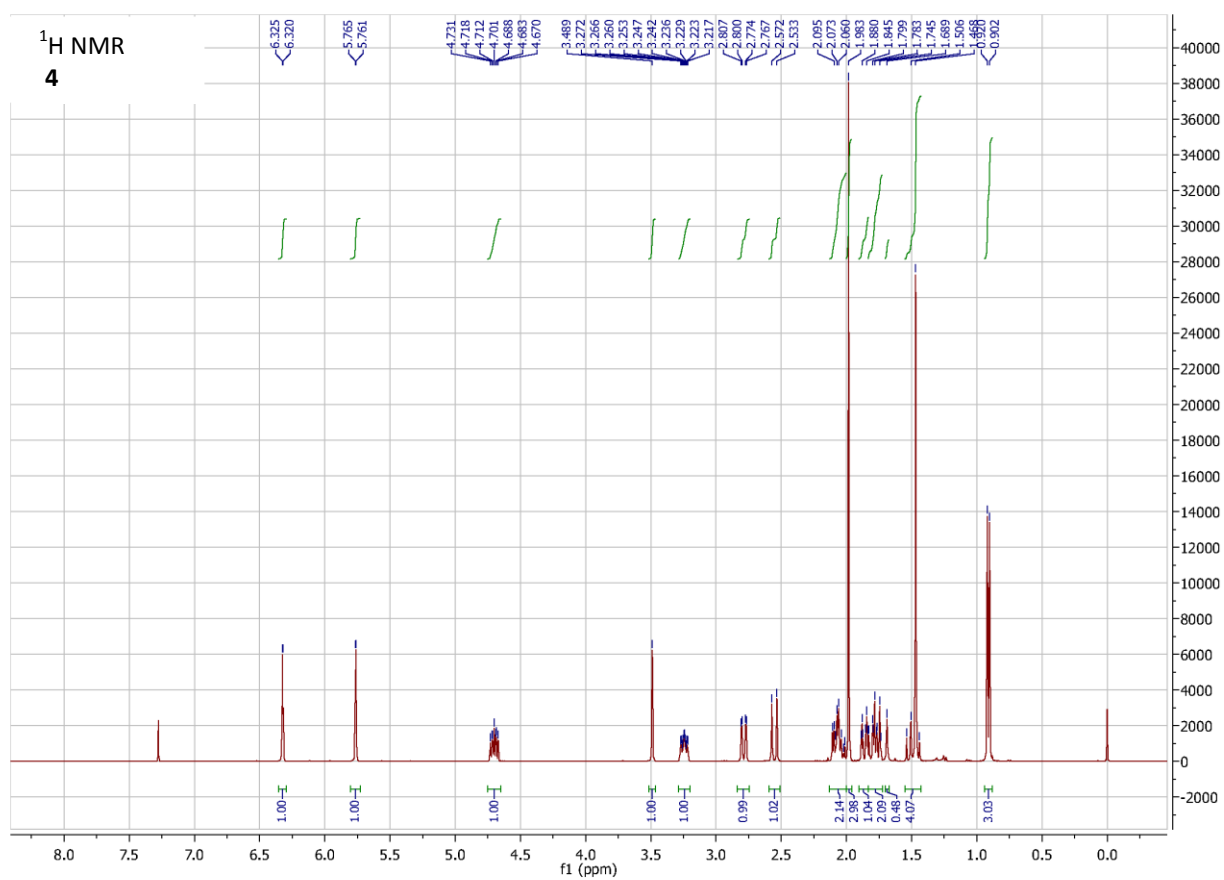


Fig. S28. ¹H NMR (400 MHz, CDCl₃) spectrum of calocephalin (4).

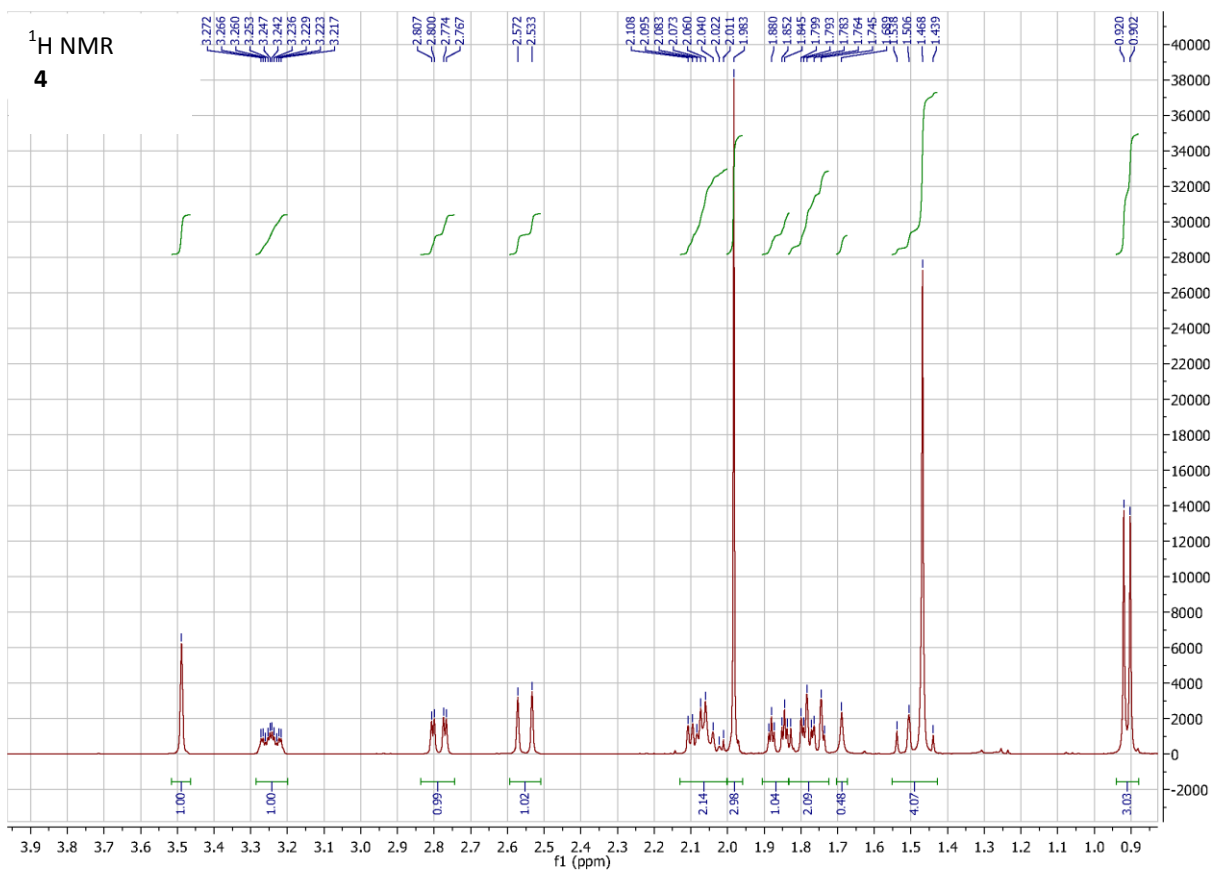


Fig. S29. ¹H NMR (400 MHz, CDCl₃) spectrum of calocephalin (**4**) expanded in the region 0.83–3.97 ppm.

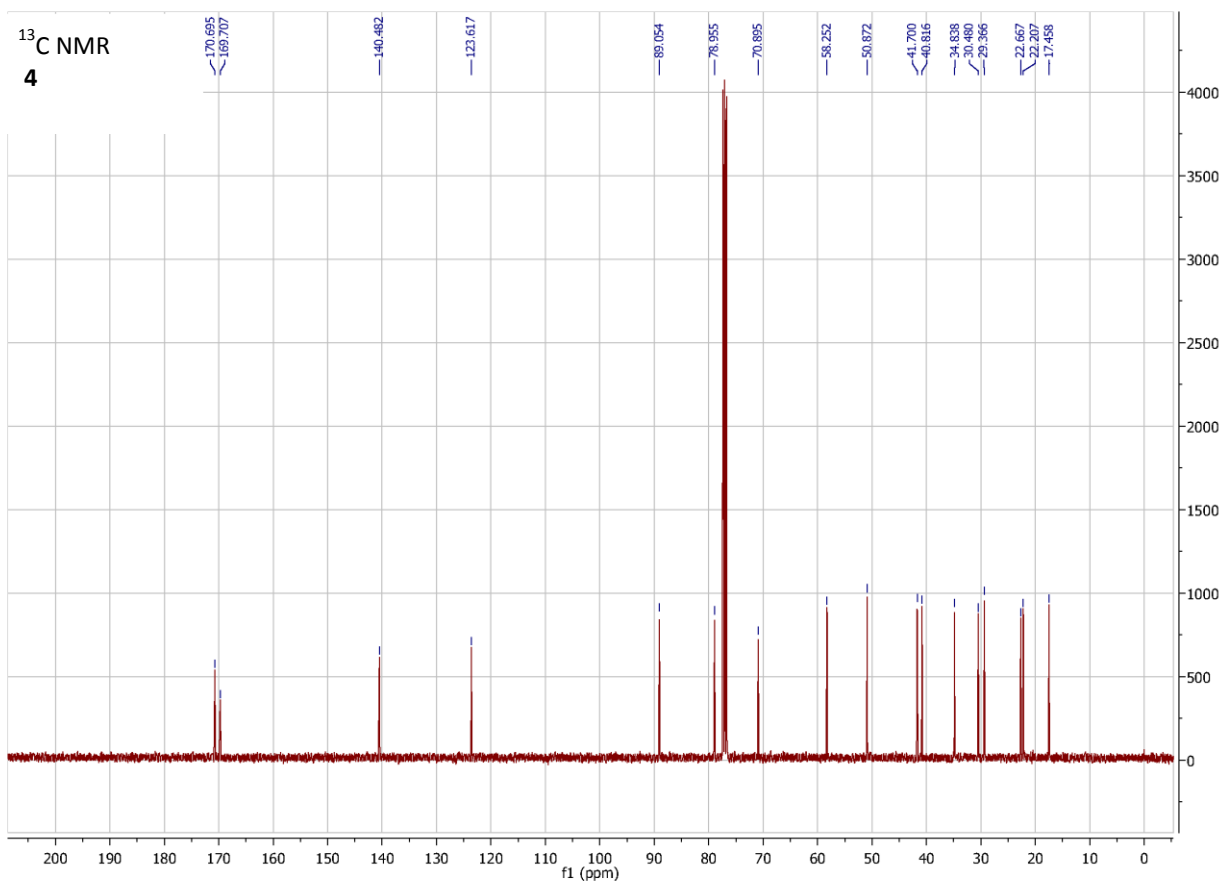


Fig. S30. ¹³C NMR (100 MHz, CDCl₃) spectrum of calcephalin (**4**).

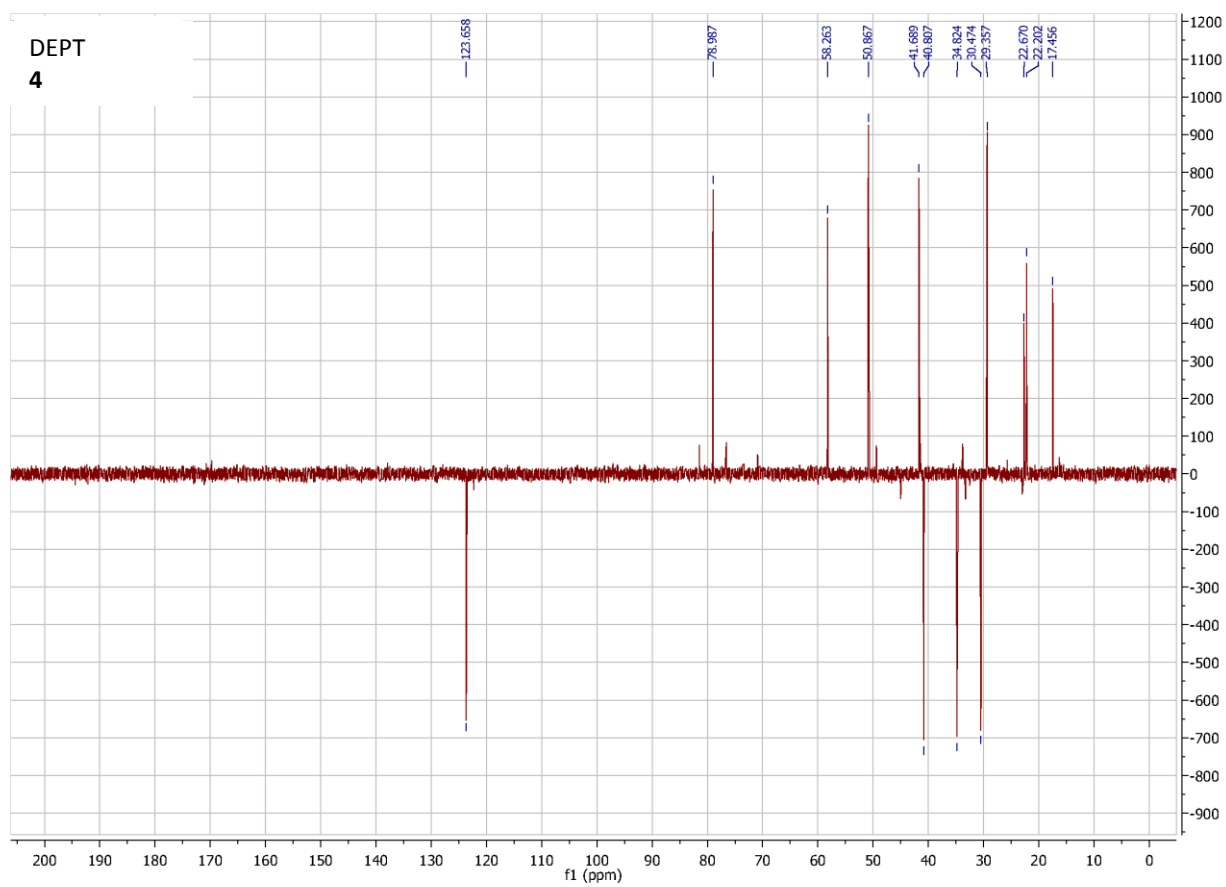


Fig. S31. DEPT spectrum of calocephalin (4).

COSY
4

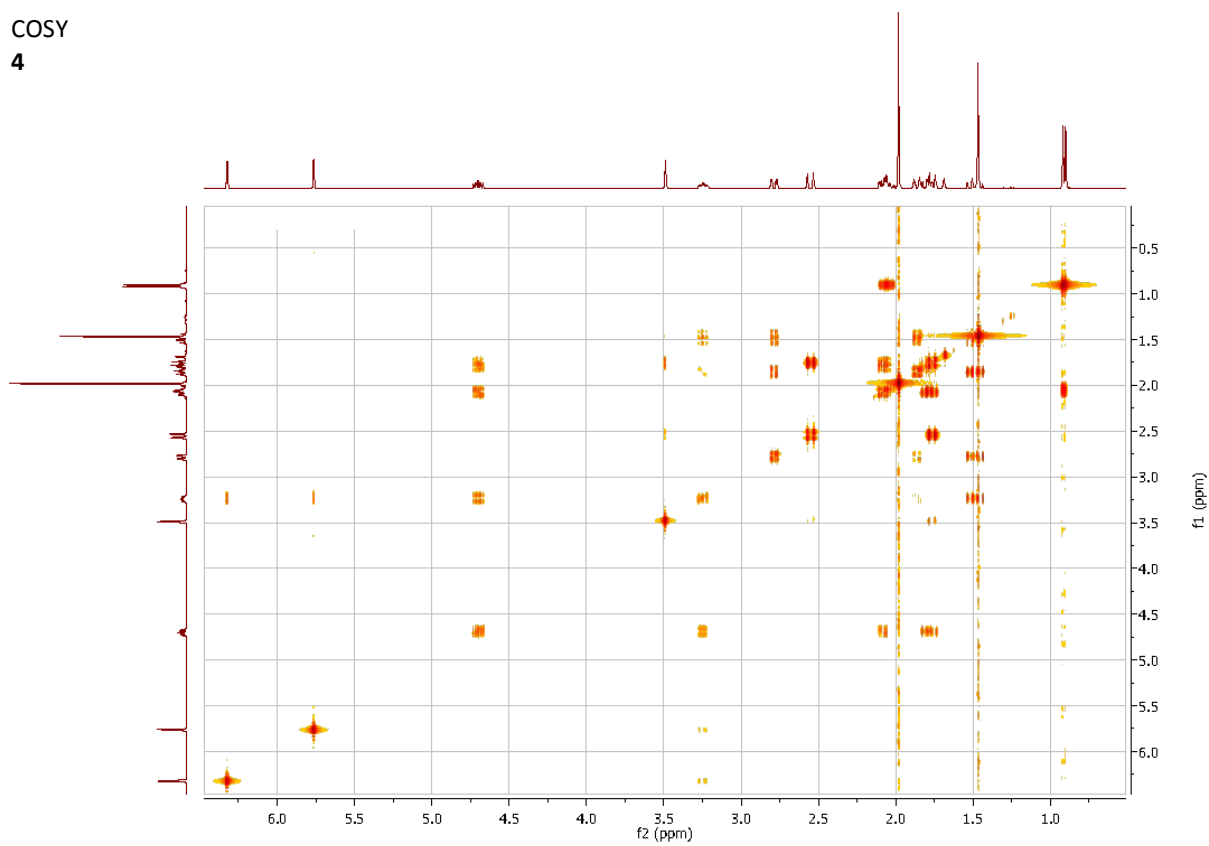


Fig. S32. ^1H - ^1H COSY spectrum of calocephalin (**4**).

NOESY
4

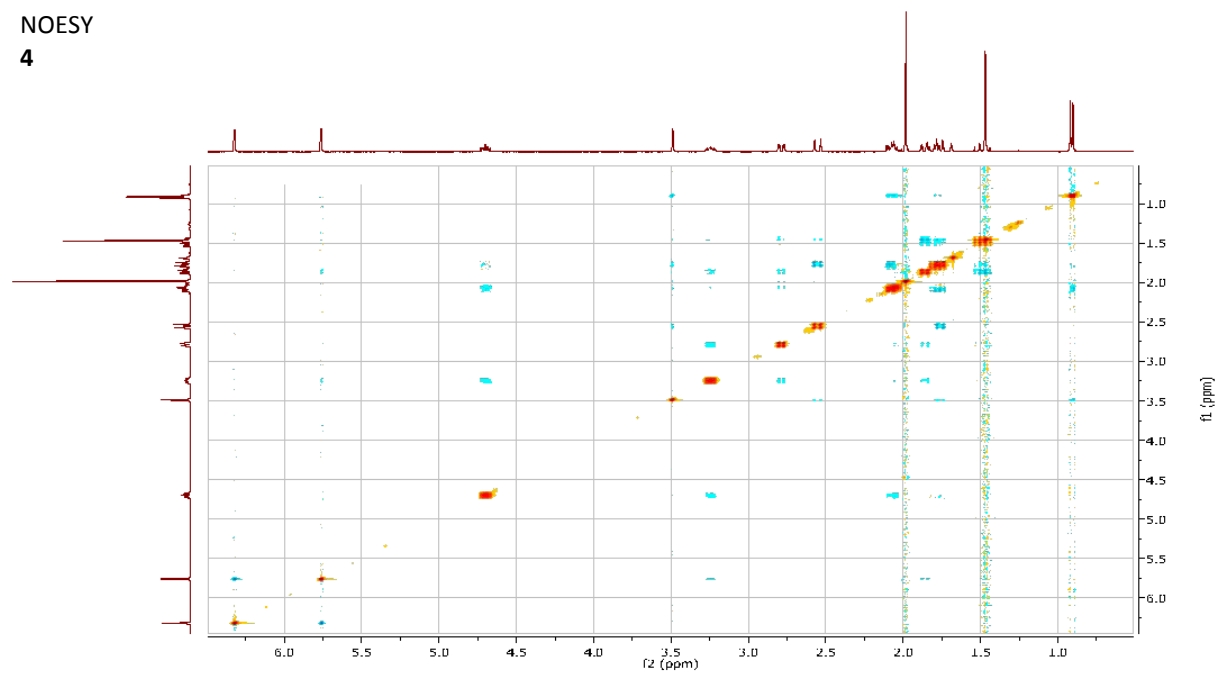


Fig. S33. NOESY spectrum of calocephalin (**4**).

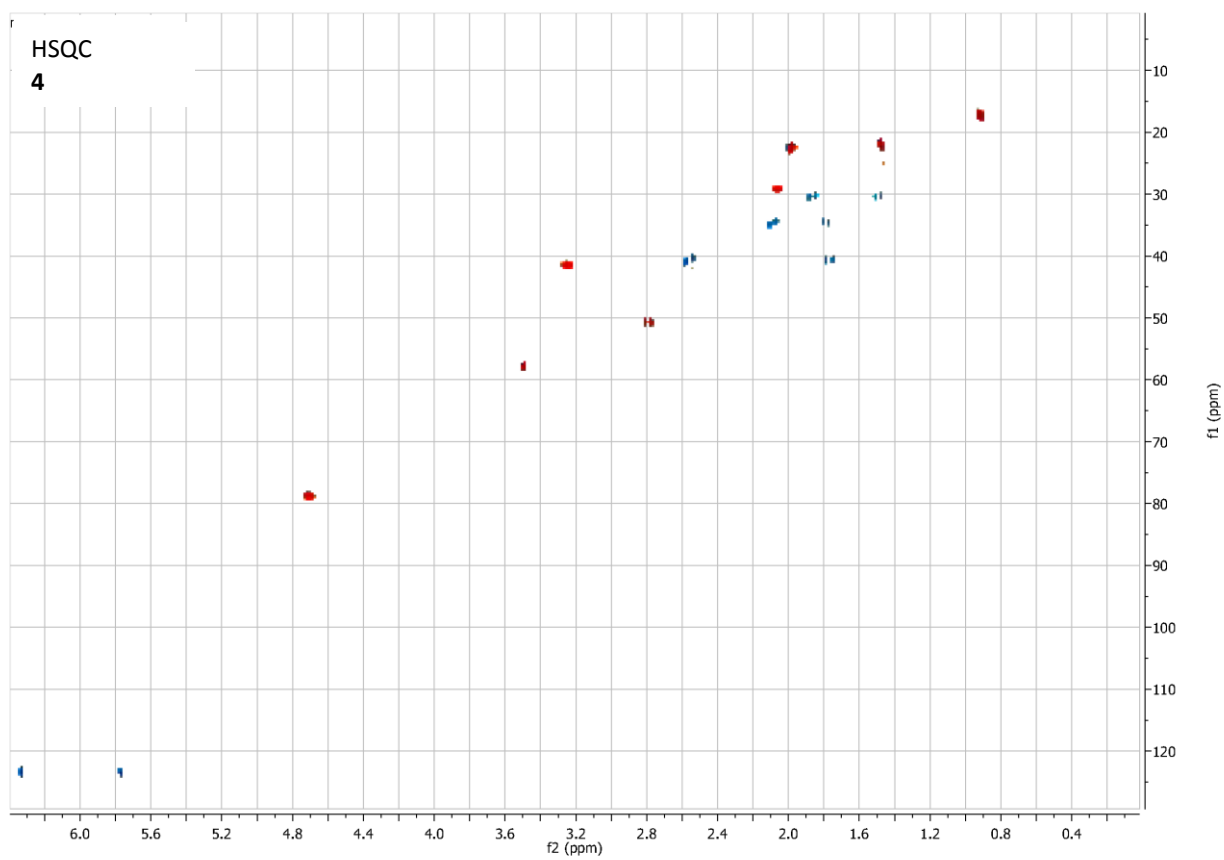


Fig. S34. HSQC spectrum of calocephalin (**4**).

HMBC
4

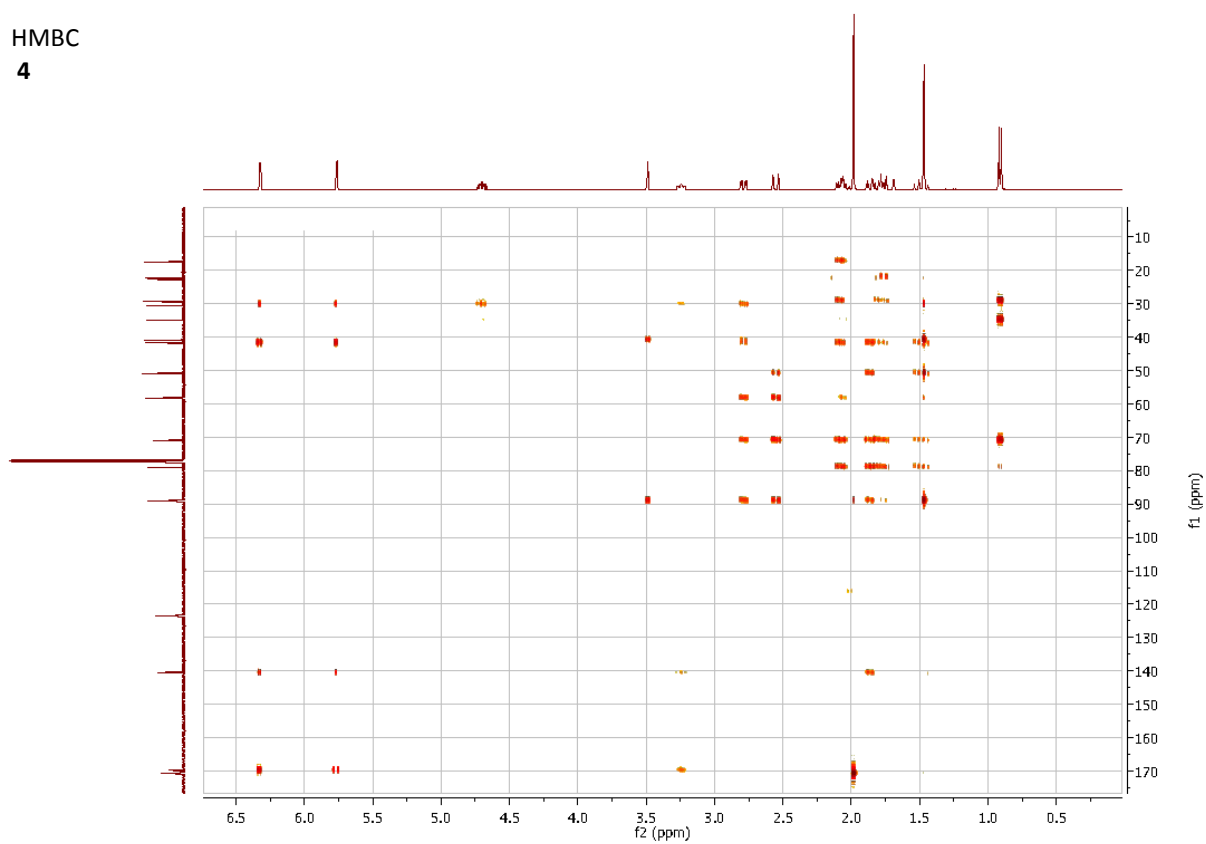


Fig. S35. HMBC spectrum of calocephalin (4).

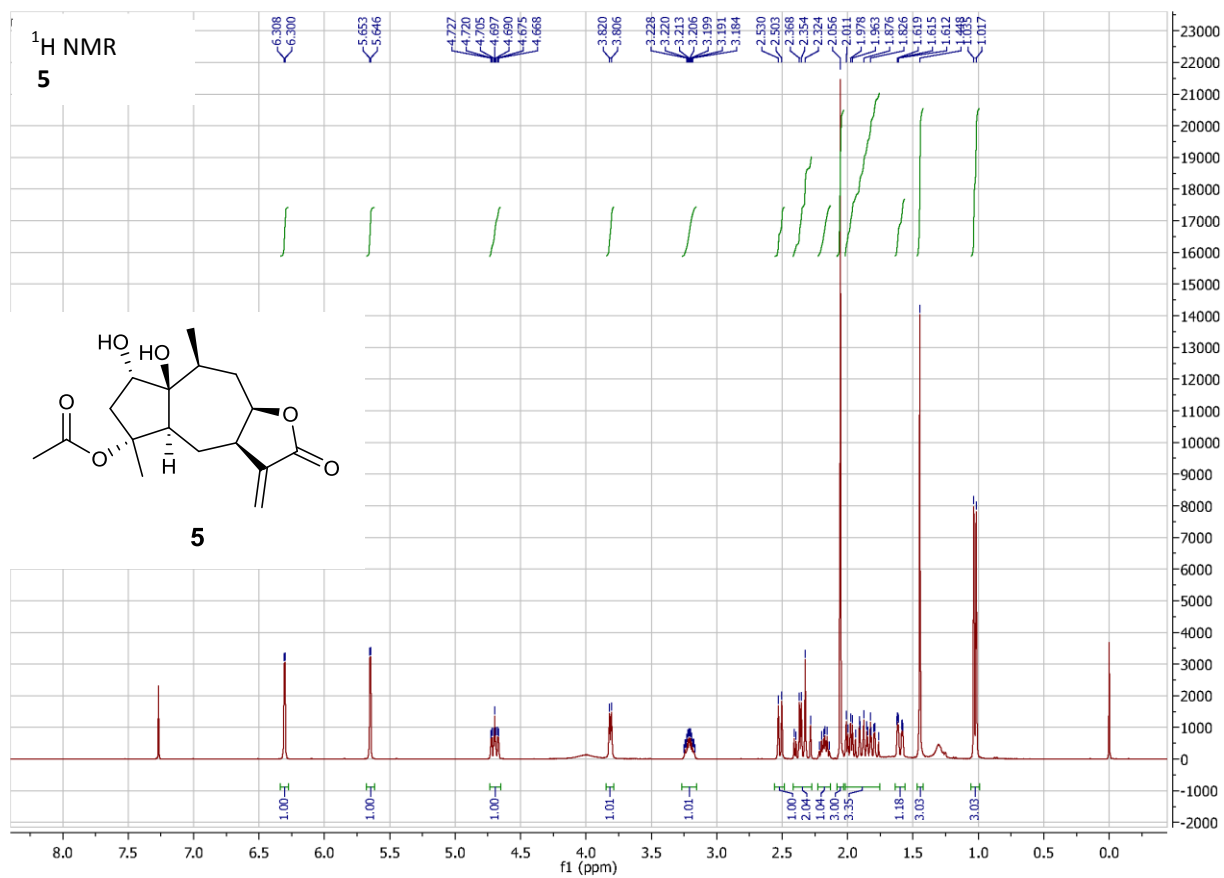


Fig. S36. ¹H NMR (400 MHz, CDCl₃) spectrum of the new compound 5.

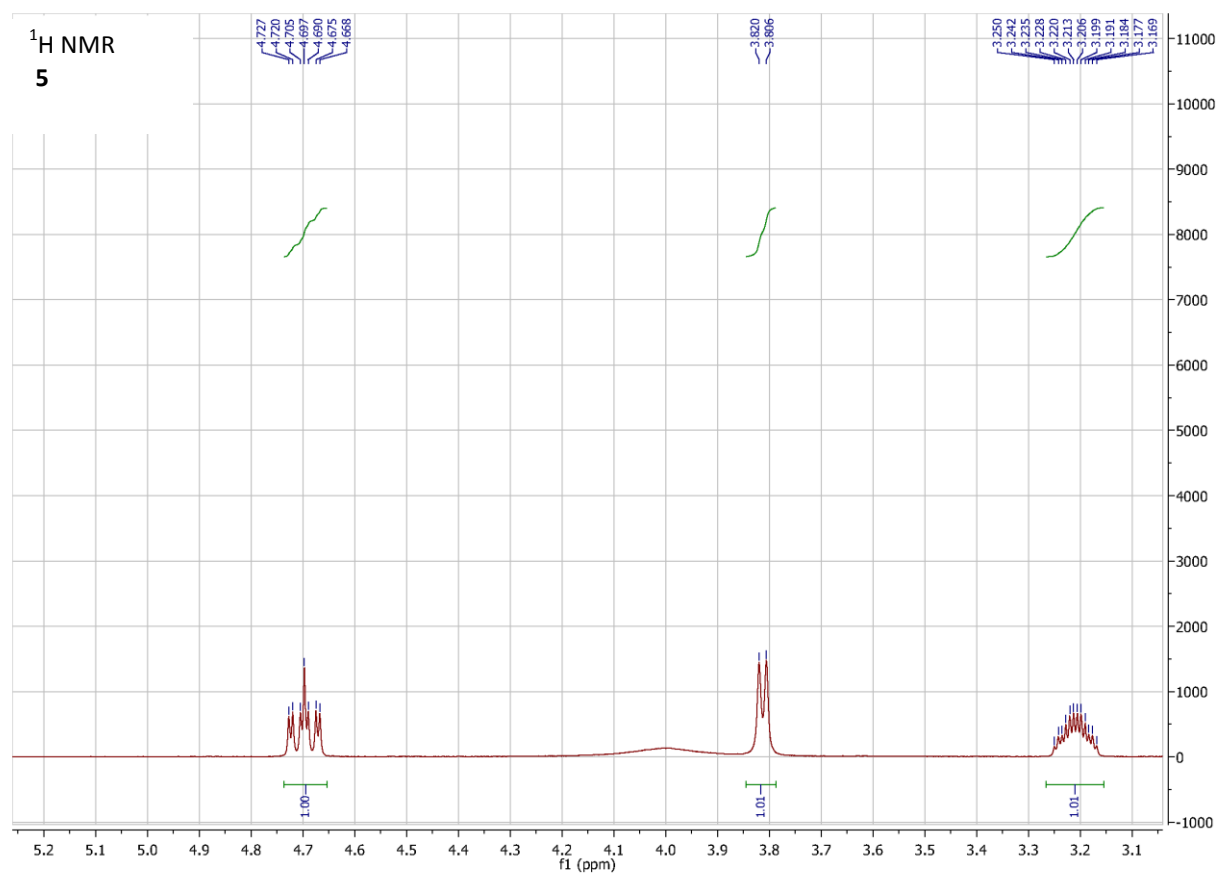


Fig. S37. ¹H NMR (400 MHz, CDCl₃) spectrum of the new compound **5** expanded in the region 3.04–5.26 ppm.

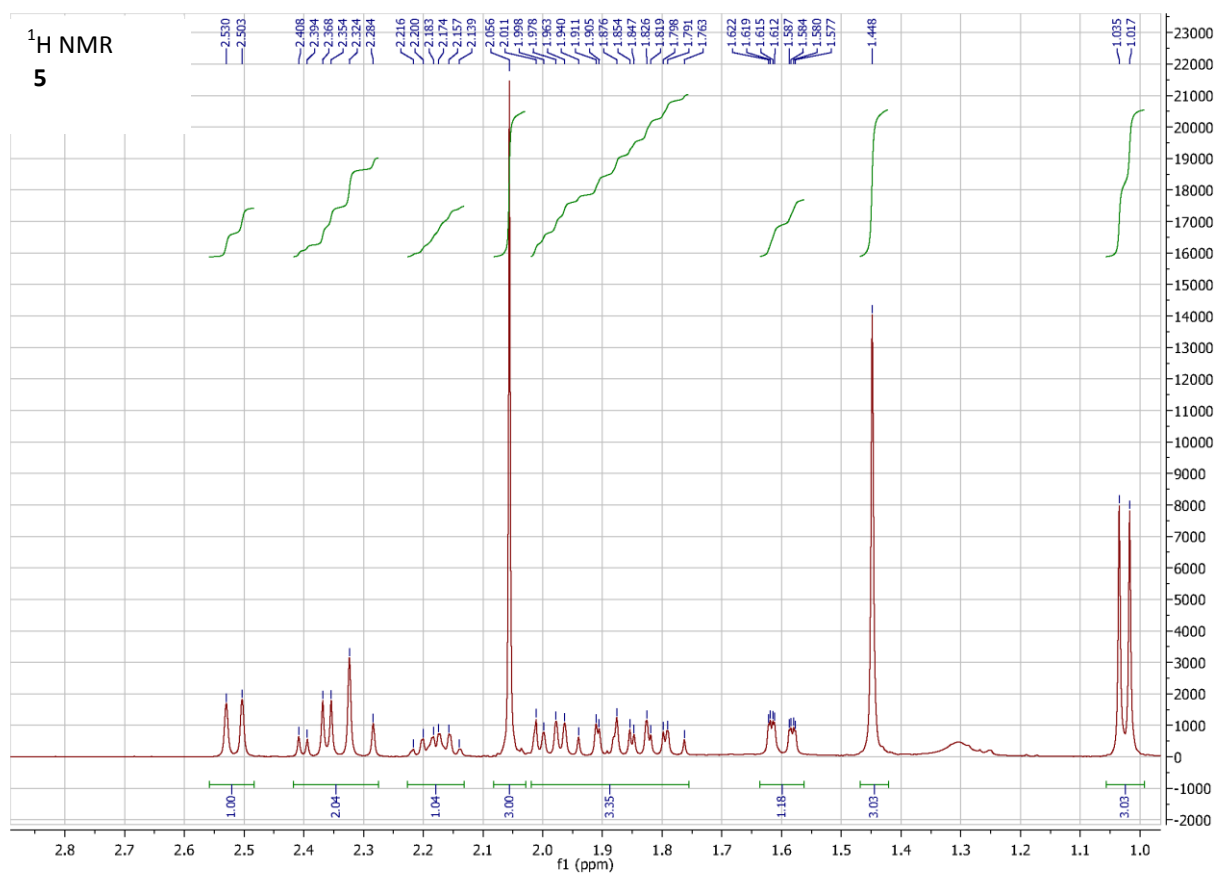


Fig. S38. ¹H NMR (400 MHz, CDCl₃) spectrum of the new compound **5** expanded in the region 0.9–3.0 ppm.

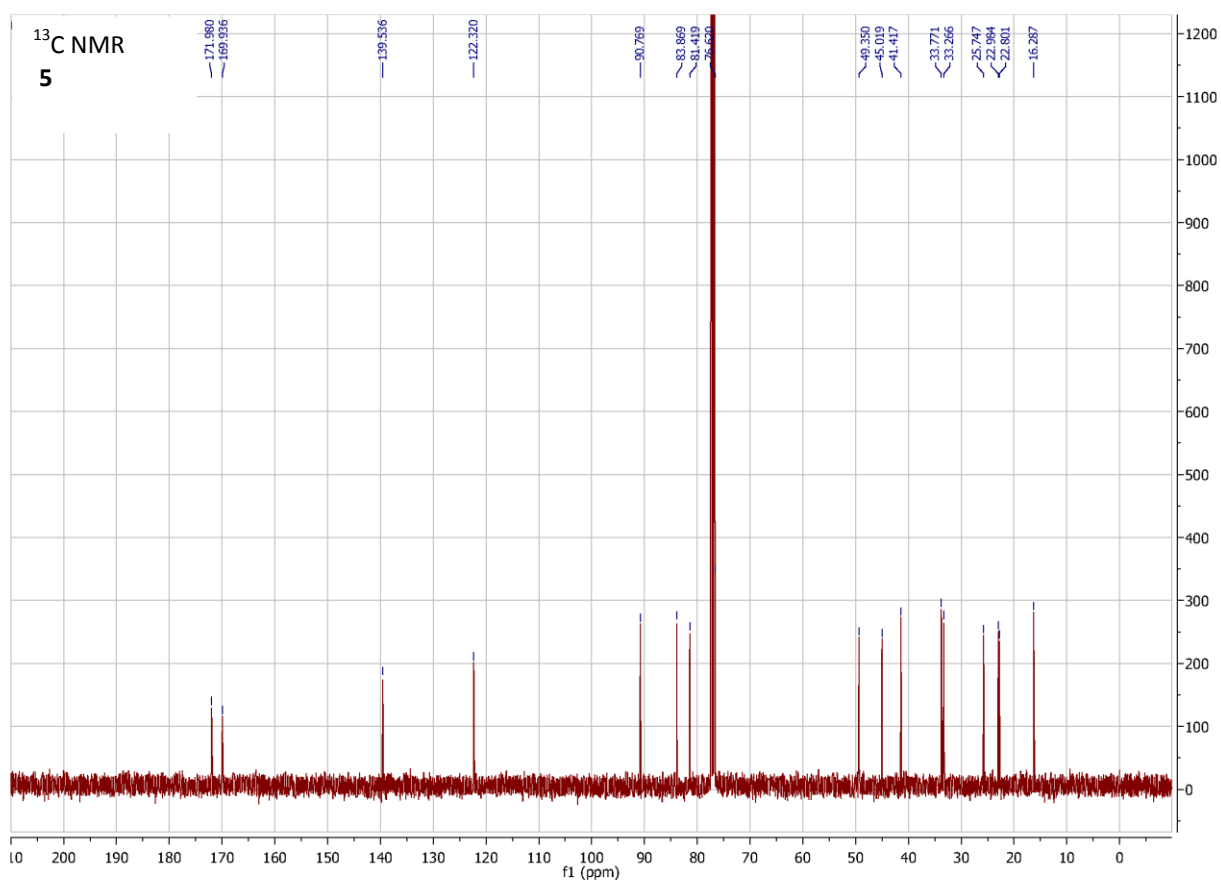


Fig. S39. ¹³C NMR (100 MHz, CDCl₃) spectrum of the new compound 5.

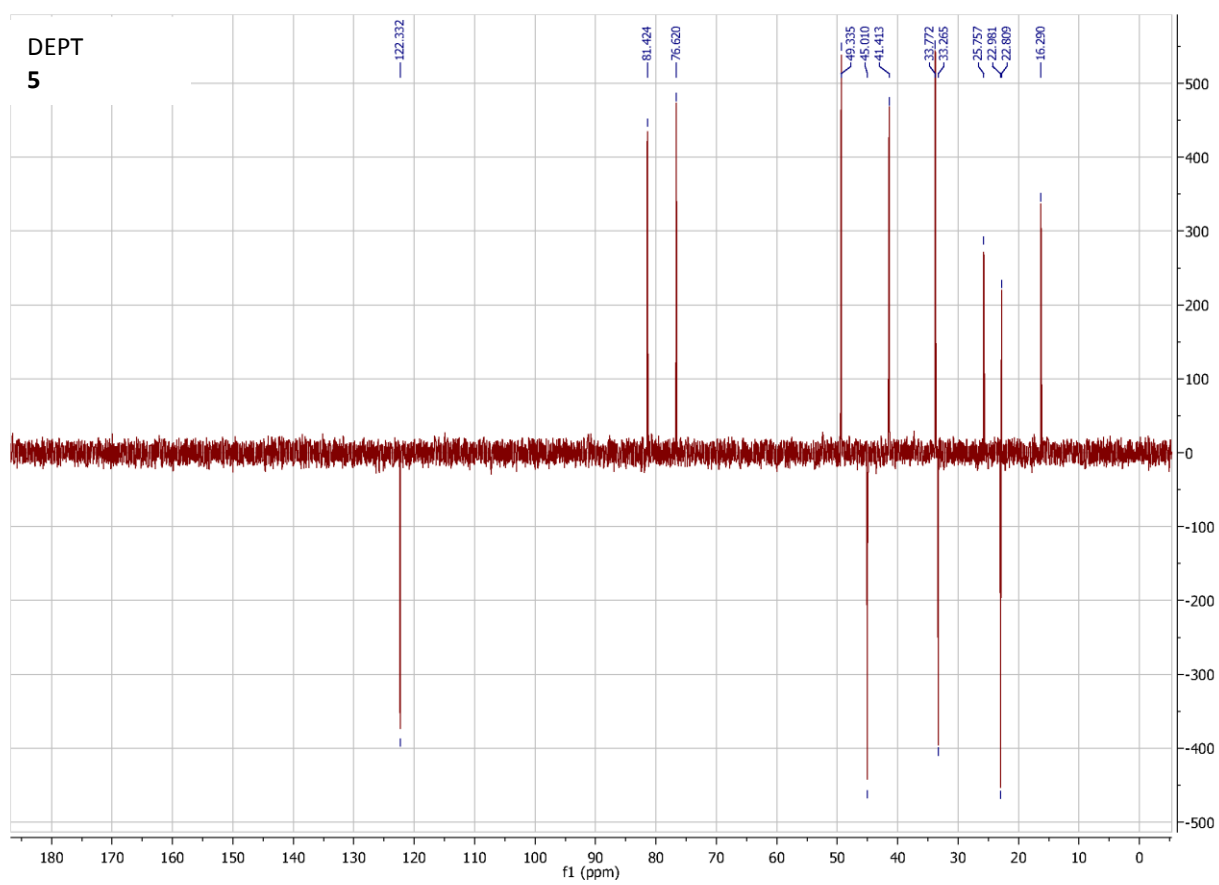


Fig. S40. DEPT spectrum of the new compound 5.

COSY
5

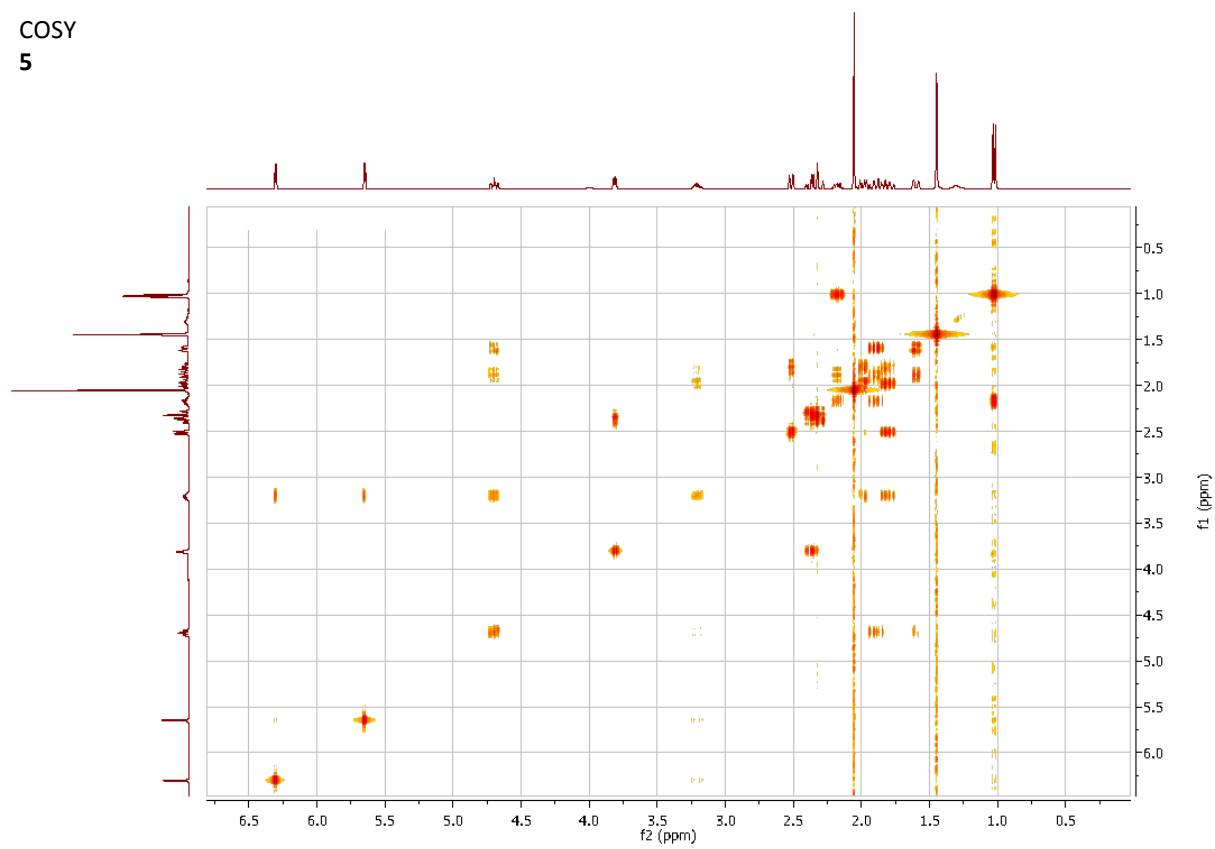


Fig. S41. ^1H - ^1H COSY spectrum of the new compound **5**.

NOESY
5

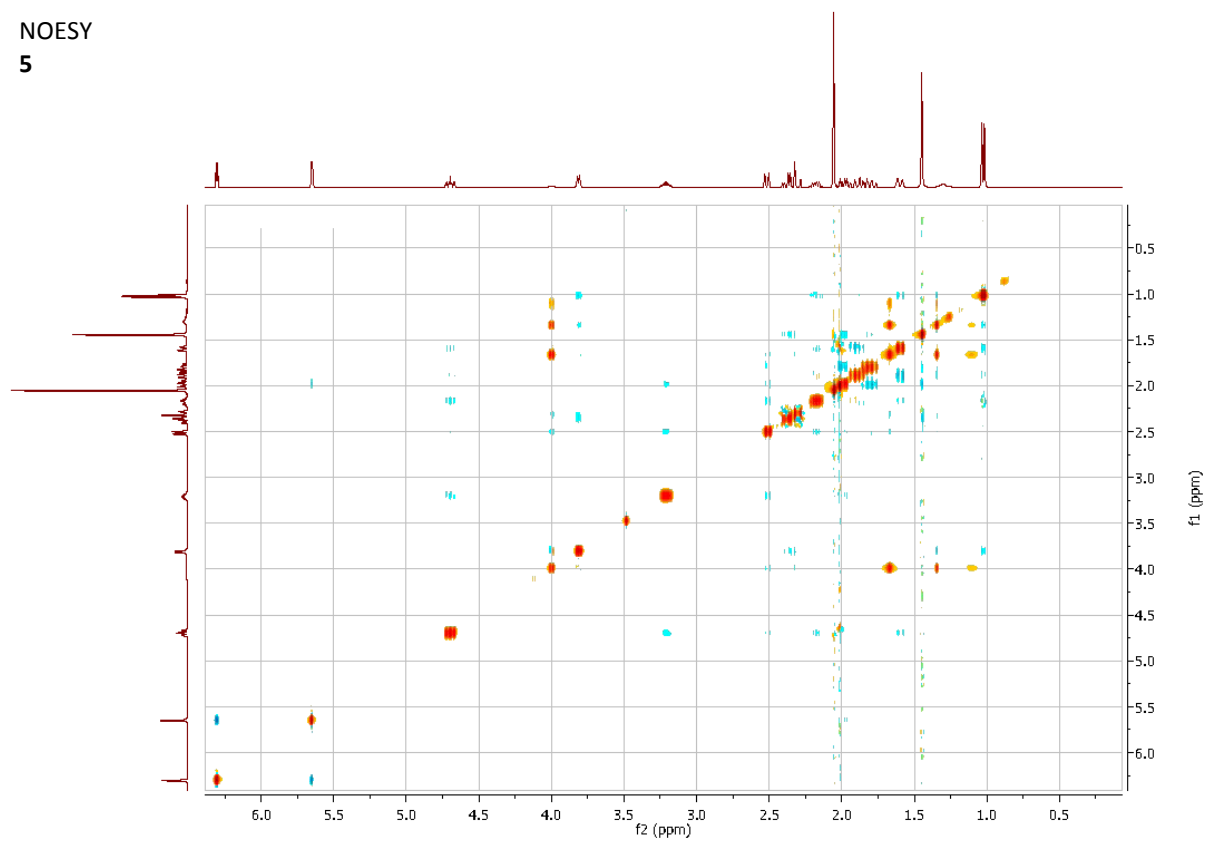


Fig. S42. NOESY spectrum of the new compound 5.

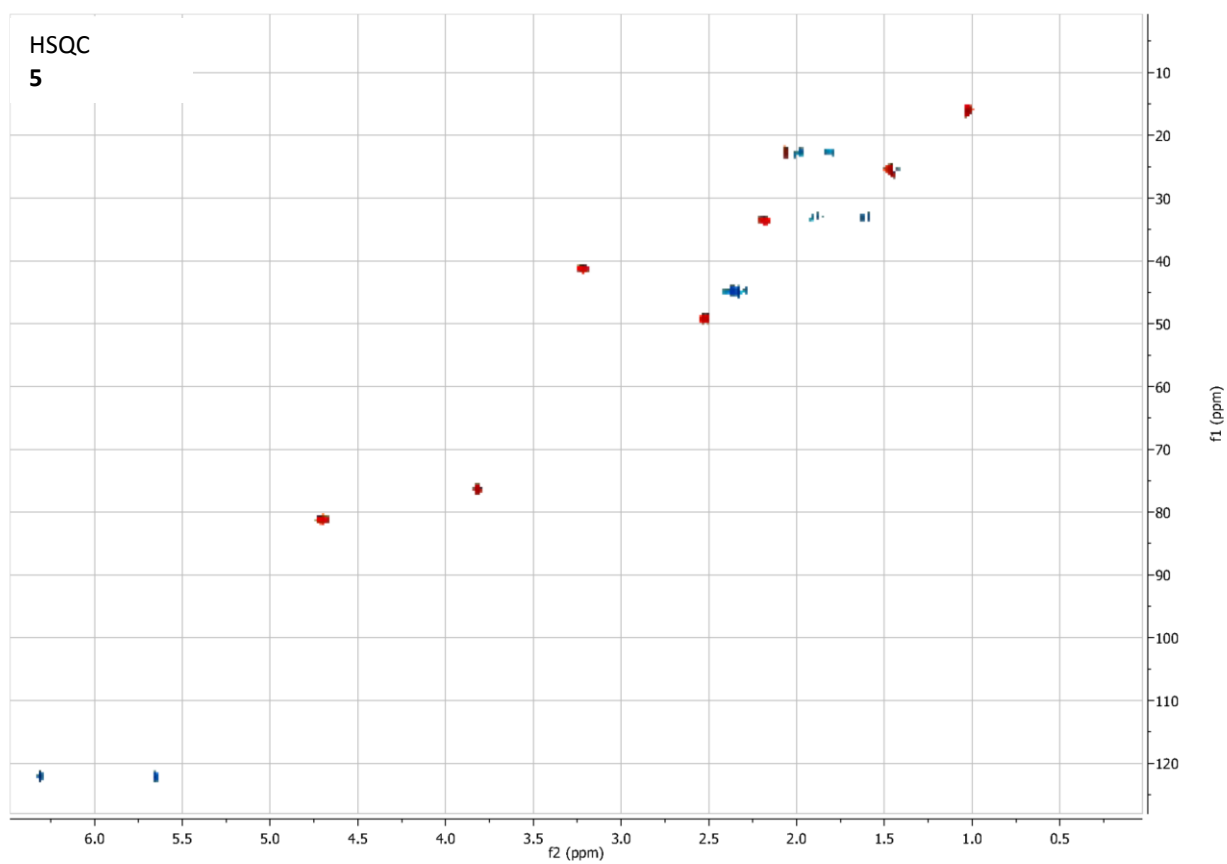


Fig. S43. HSQC spectrum of the new compound **5**.

HMBC
5

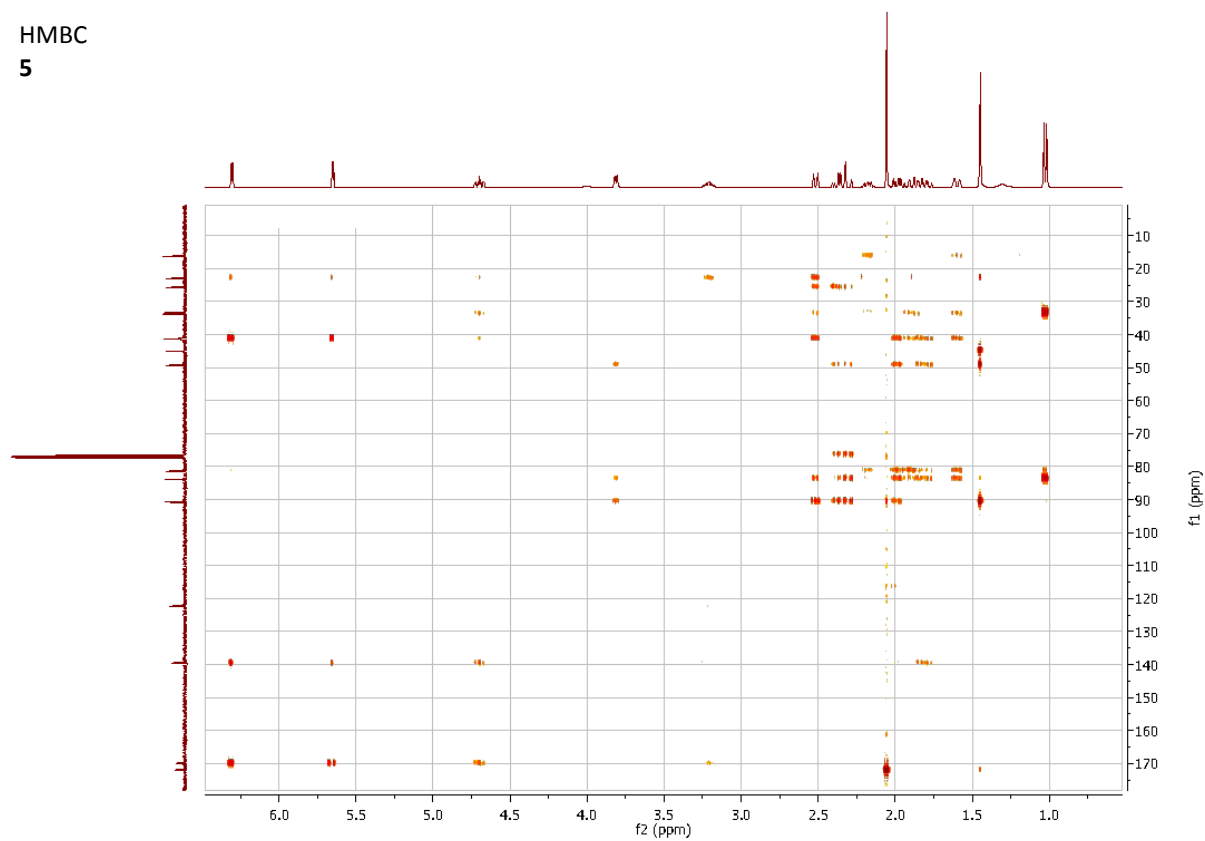


Fig. S44. HMBC spectrum of the new compound 5.

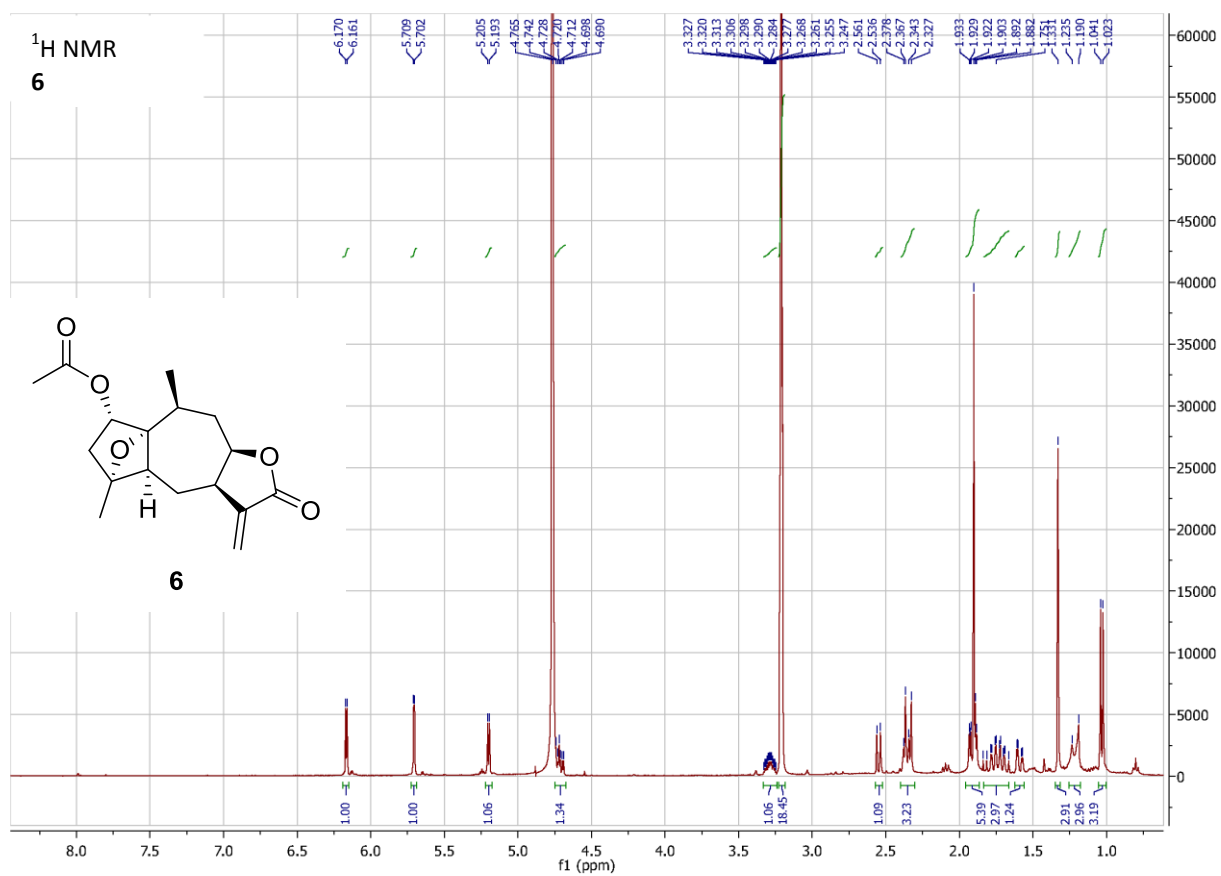


Fig. S45. ¹H NMR (400 MHz, methanol-*d*₄) spectrum of the new compound 6.

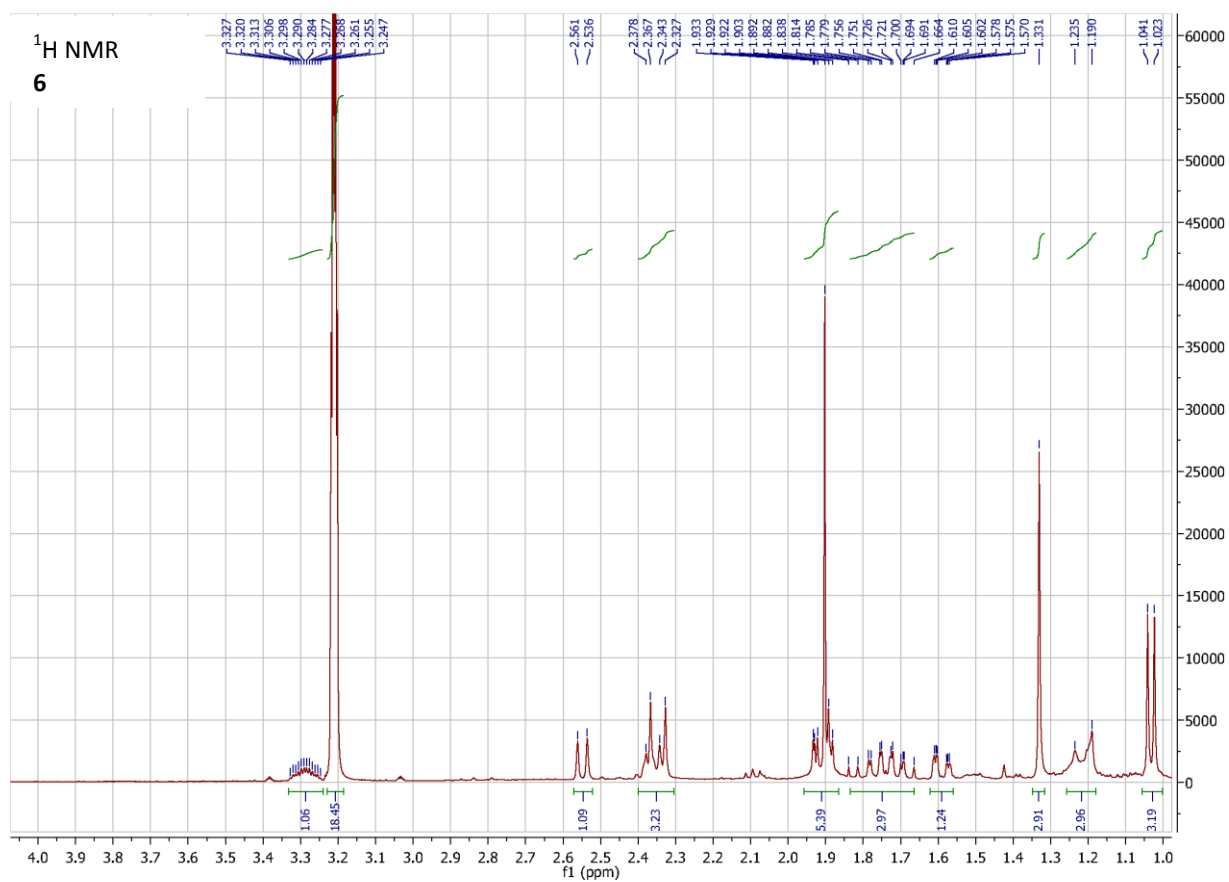


Fig. S46. ¹H NMR (400 MHz, methanol-*d*₄) spectrum of the new compound **6** expanded in the region 0.95–4.15 ppm.

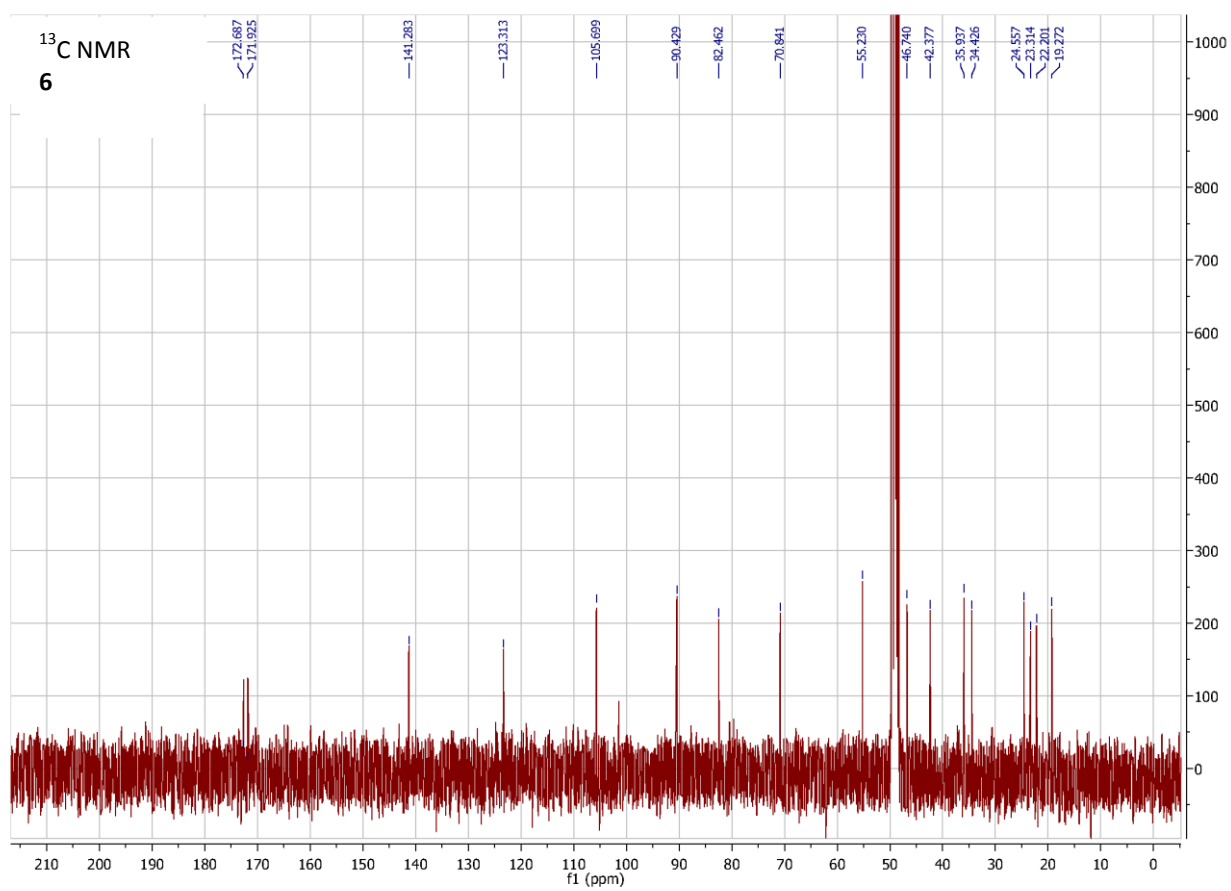


Fig. S47. ¹³C NMR (100 MHz, methanol-*d*₄) spectrum of the new compound **6**.

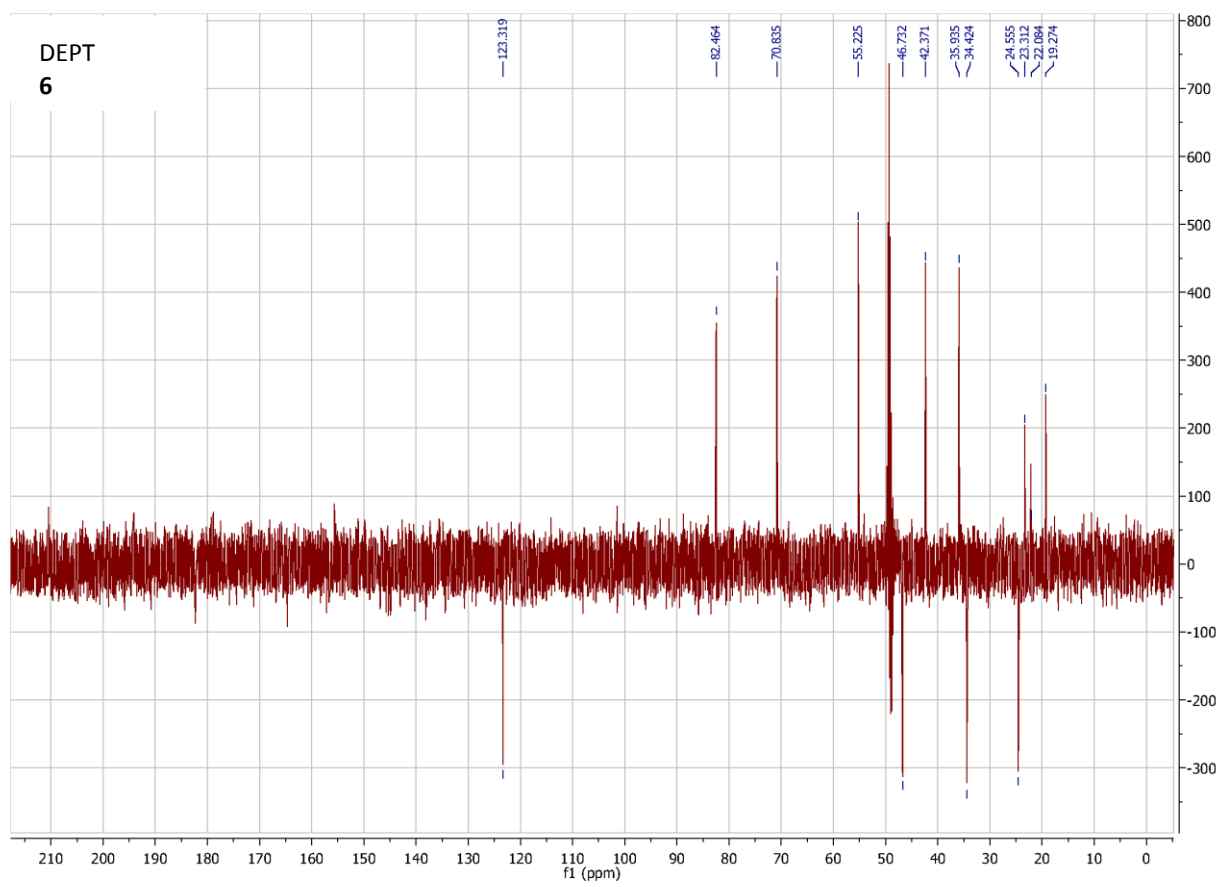


Fig. S48. DEPT spectrum of the new compound **6**.

COSY
6

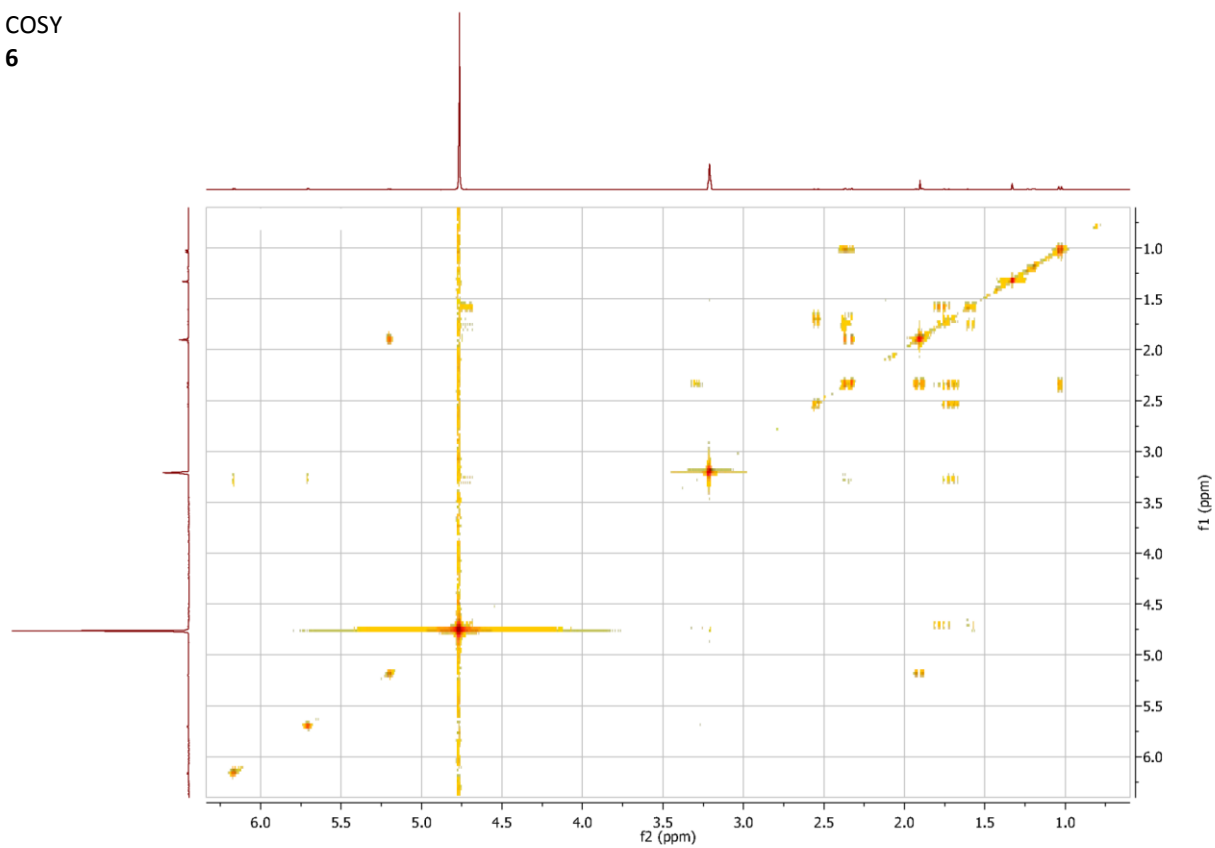


Fig. S49. ^1H - ^1H COSY spectrum of the new compound **6**.

NOESY
6

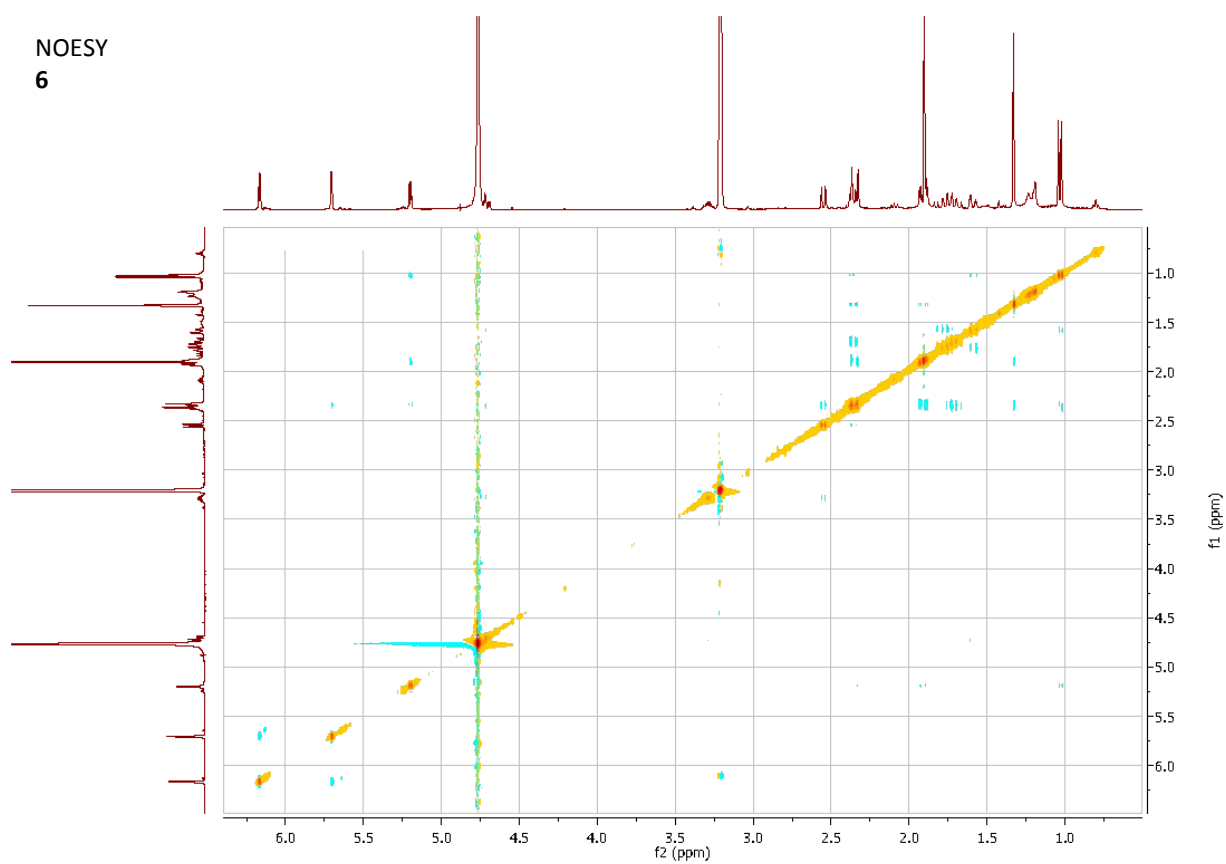


Fig. S50. NOESY spectrum of the new compound **6**.

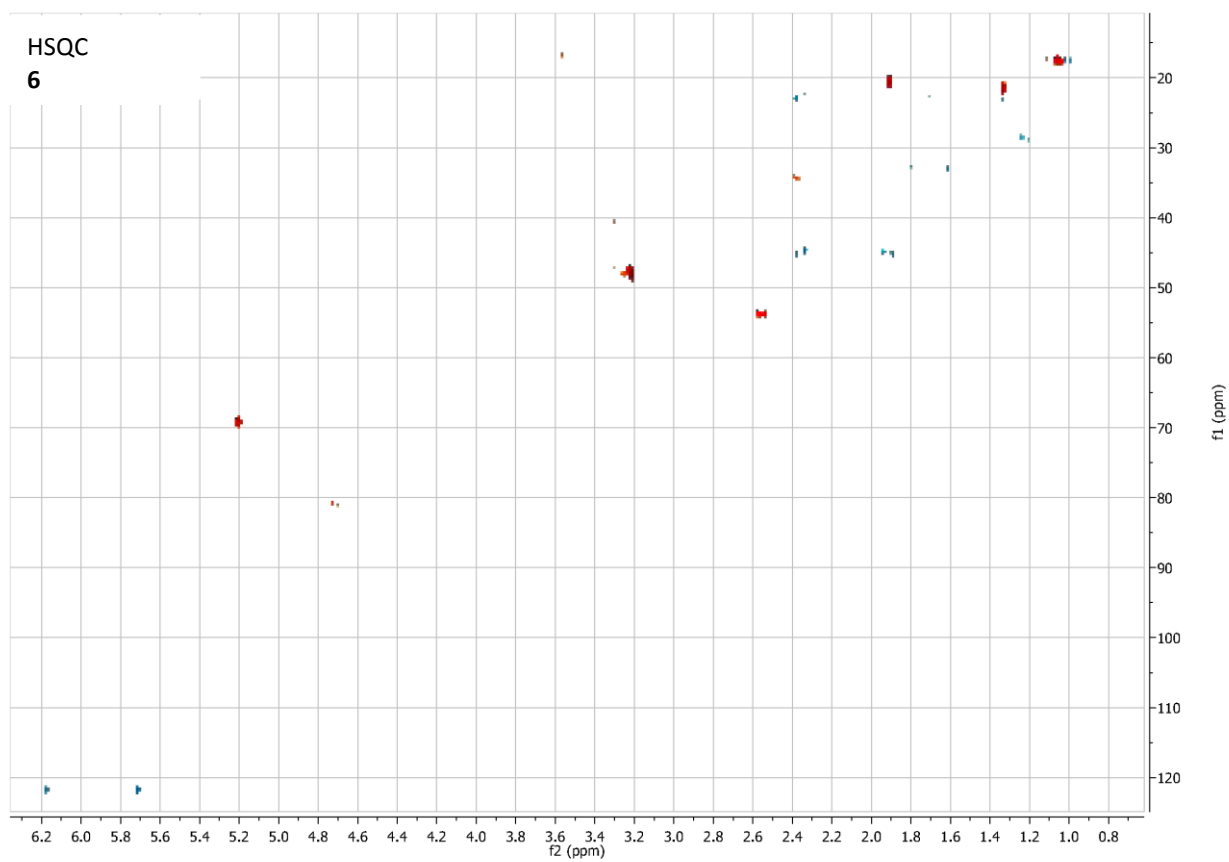


Fig. S51. HSQC spectrum of the new compound **6**.

HMBC
6

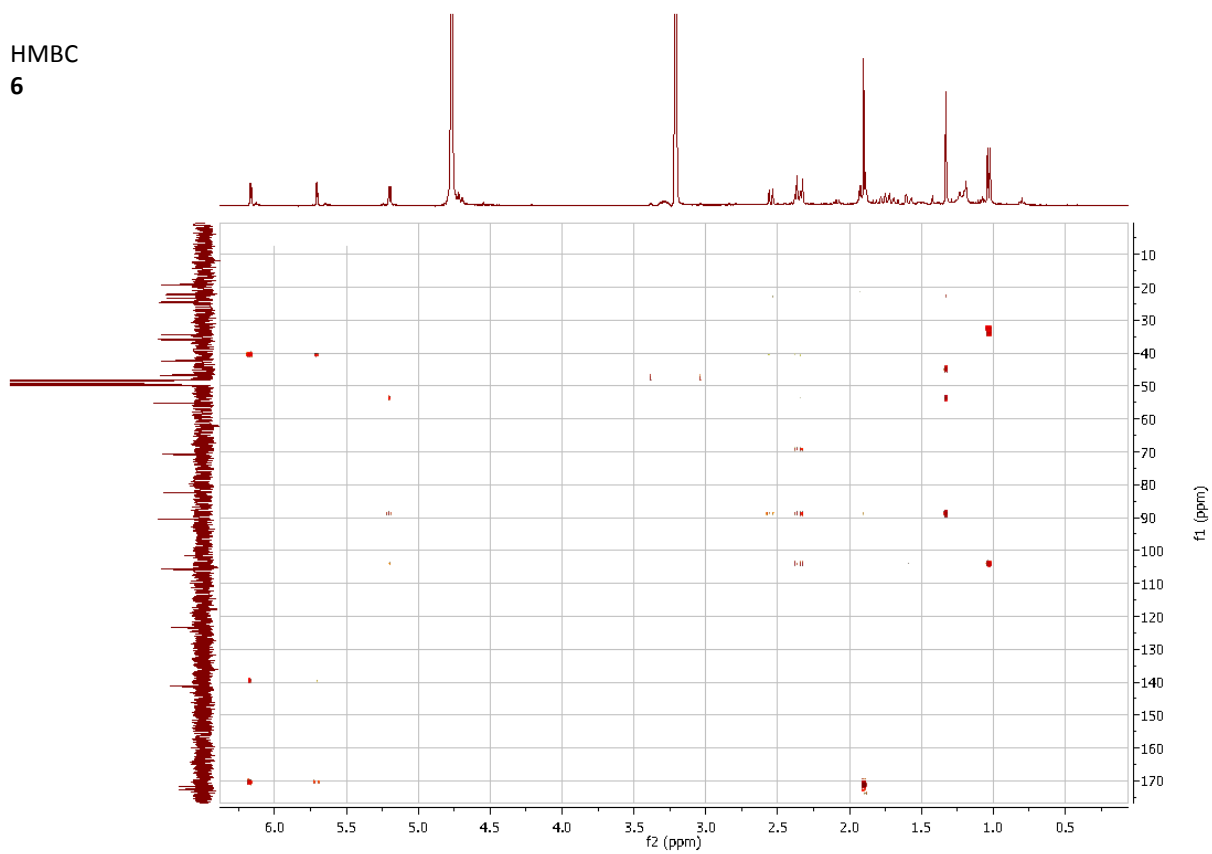
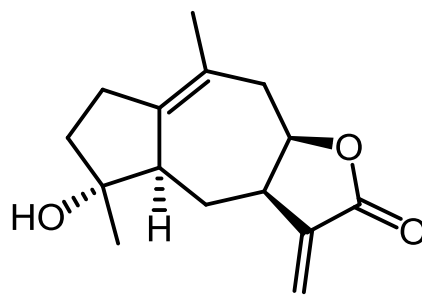


Fig. S52. HMBC spectrum of the new compound 6.

Pseudoivalin [*4*α-*hydroxy-5*α*H-10,11(13)-guaidien-8*β,*12-olide*] (**7**)

Colorless crystals; mp 121–122 °C; $[\alpha]_D^{25} -146$ (*c* 0.4, CHCl₃); UV (MeOH) λ_{\max} (log ϵ) 214 (4.15) nm; IR (KBr) ν_{\max} 3578, 3342, 2971, 1772, 1754, 1640, 1308, 1105 cm⁻¹; APCIMS: *m/z* 249 [M + H]⁺ (21), 231 [M + H – H₂O]⁺ (100); HRESIMS *m/z* 249.1487 (calcd for C₁₅H₂₁O₃, 249.1491).



Pseudoivalin (**7**)

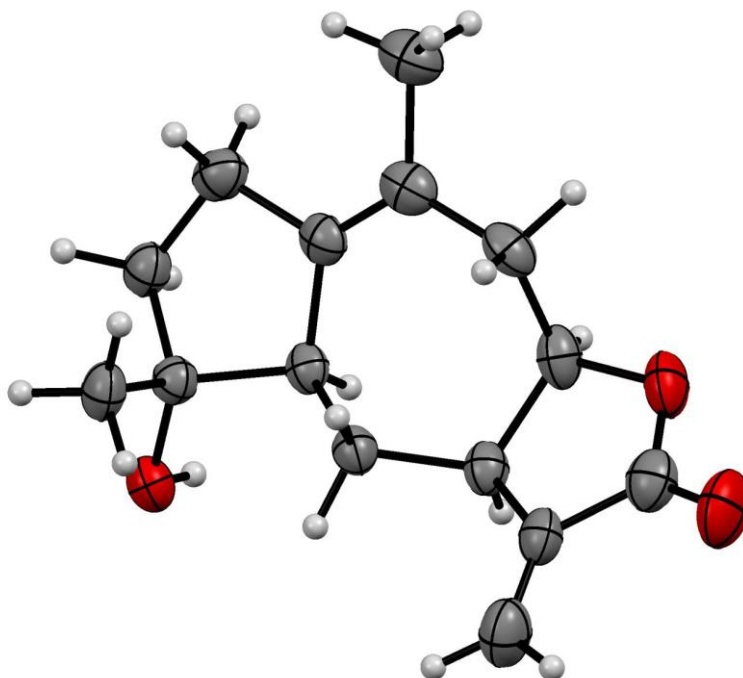


Fig. S53. Crystal structure of pseudoivalin (**7**) [ORTEP diagram] with displacement ellipsoids at 50% probability for non-H atoms.

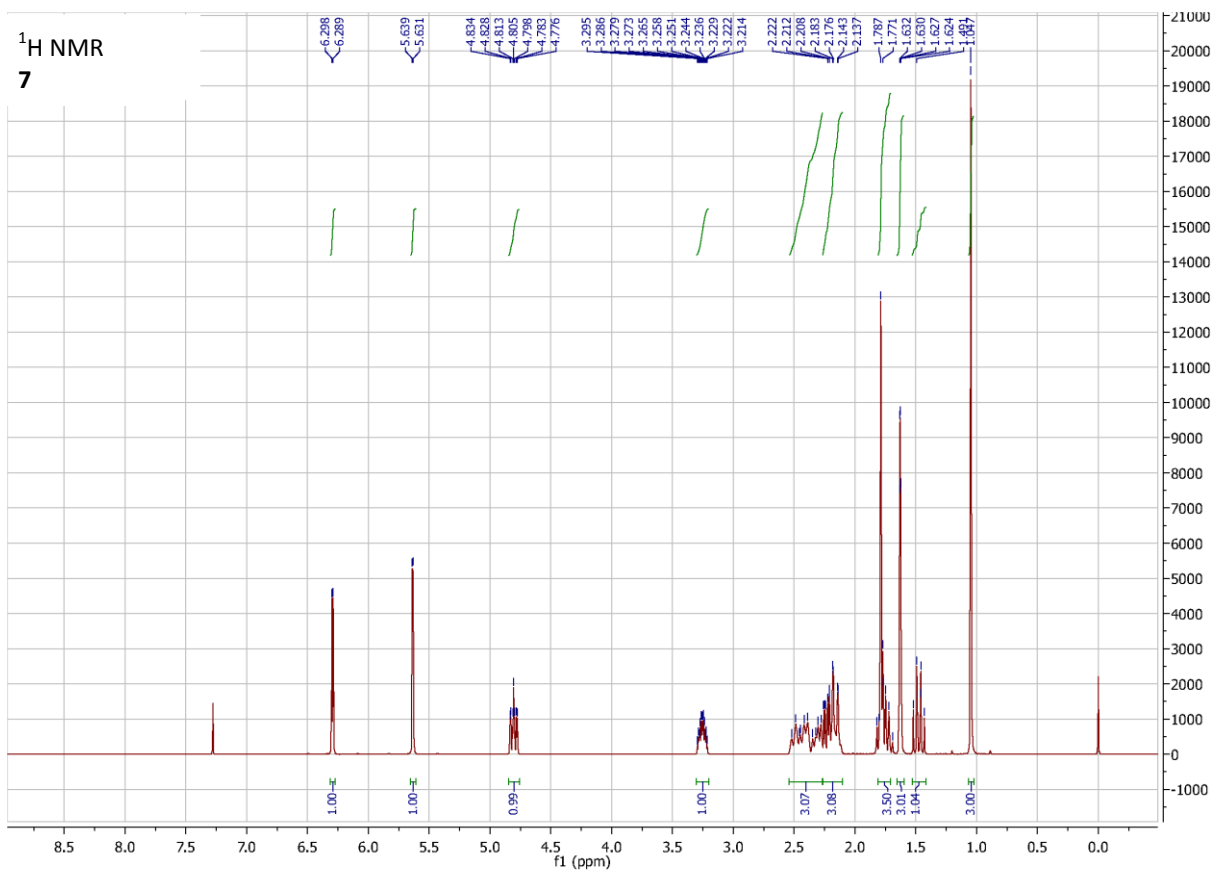


Fig. S54. ¹H NMR (400 MHz, CDCl₃) spectrum of pseudoivalin (7).

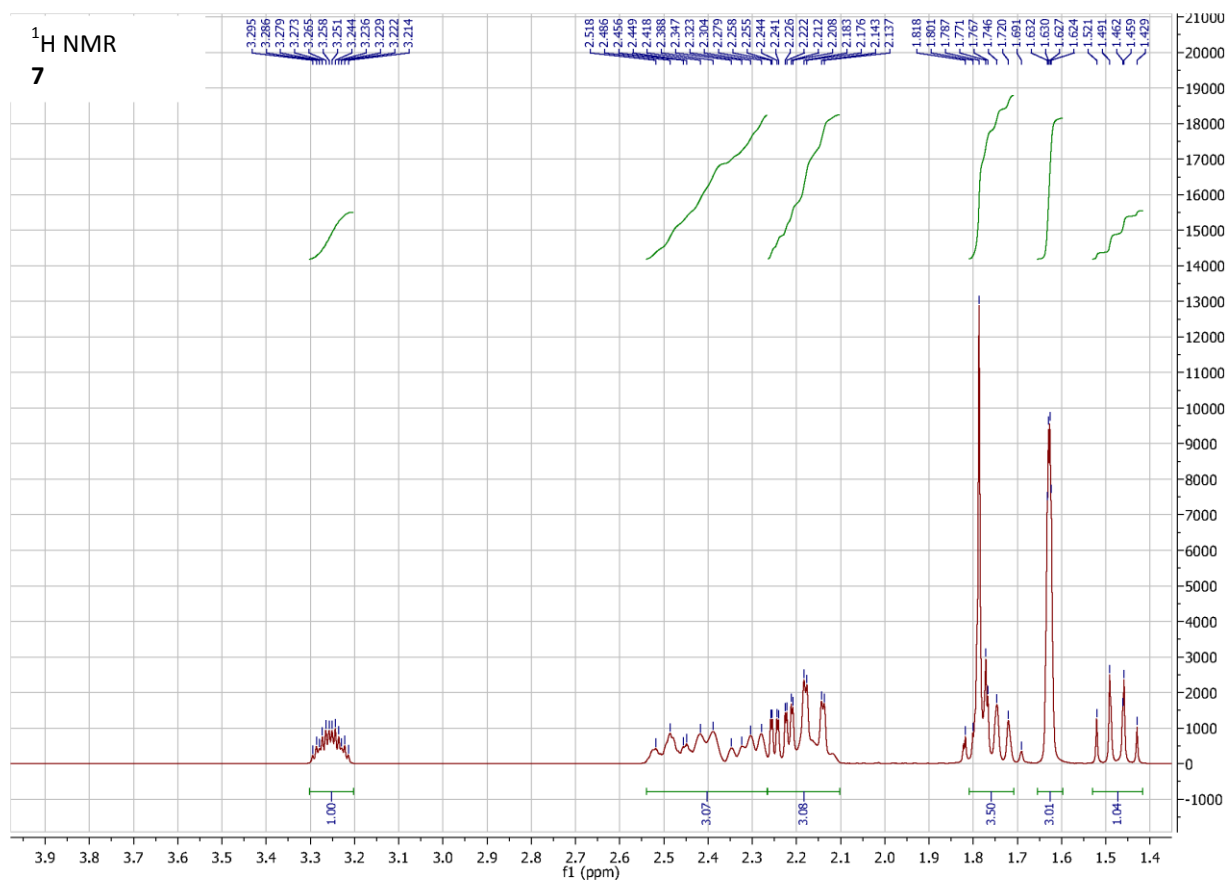


Fig. S55. ¹H NMR (400 MHz, CDCl₃) spectrum of pseudoivalin (**7**) expanded in the region 1.35–3.98 ppm.

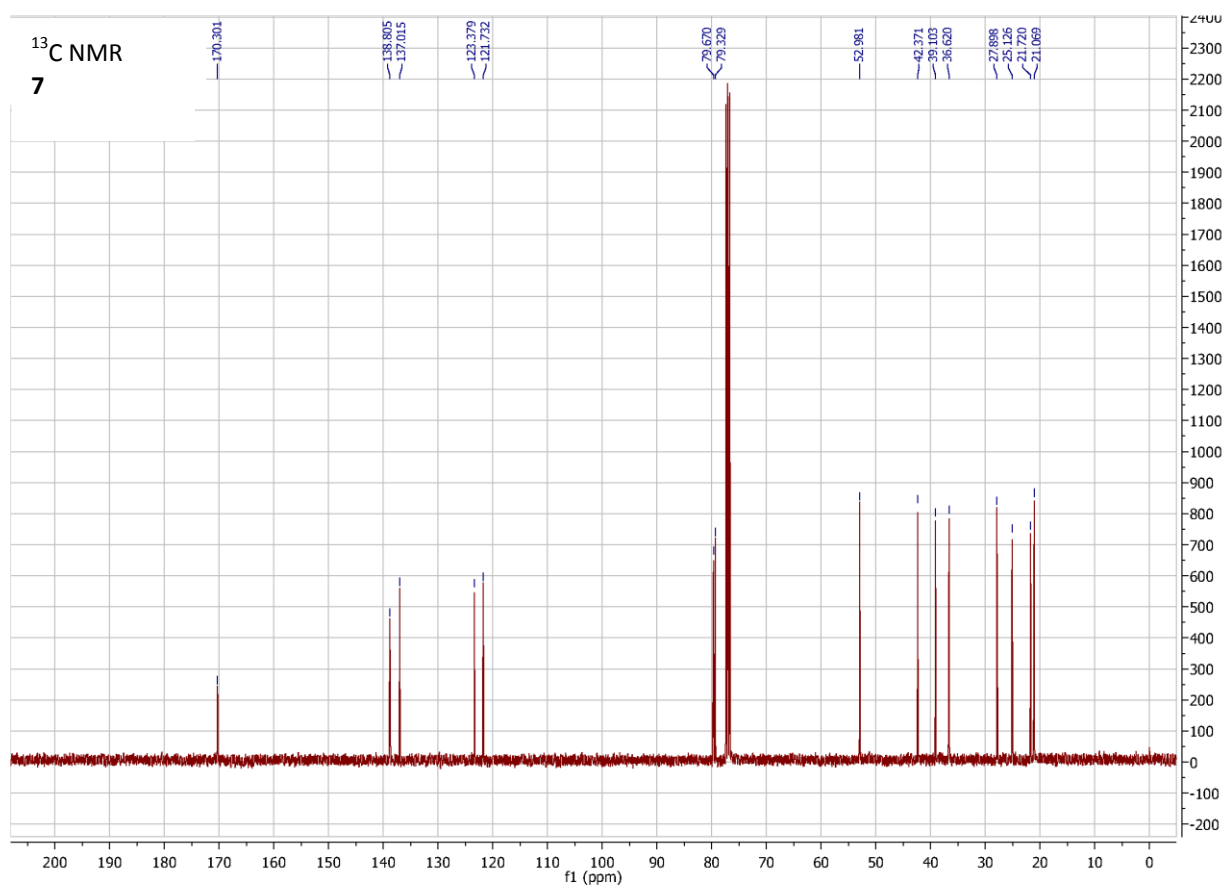


Fig. S56. ¹³C NMR (100 MHz, CDCl₃) spectrum of pseudoivalin (**7**).

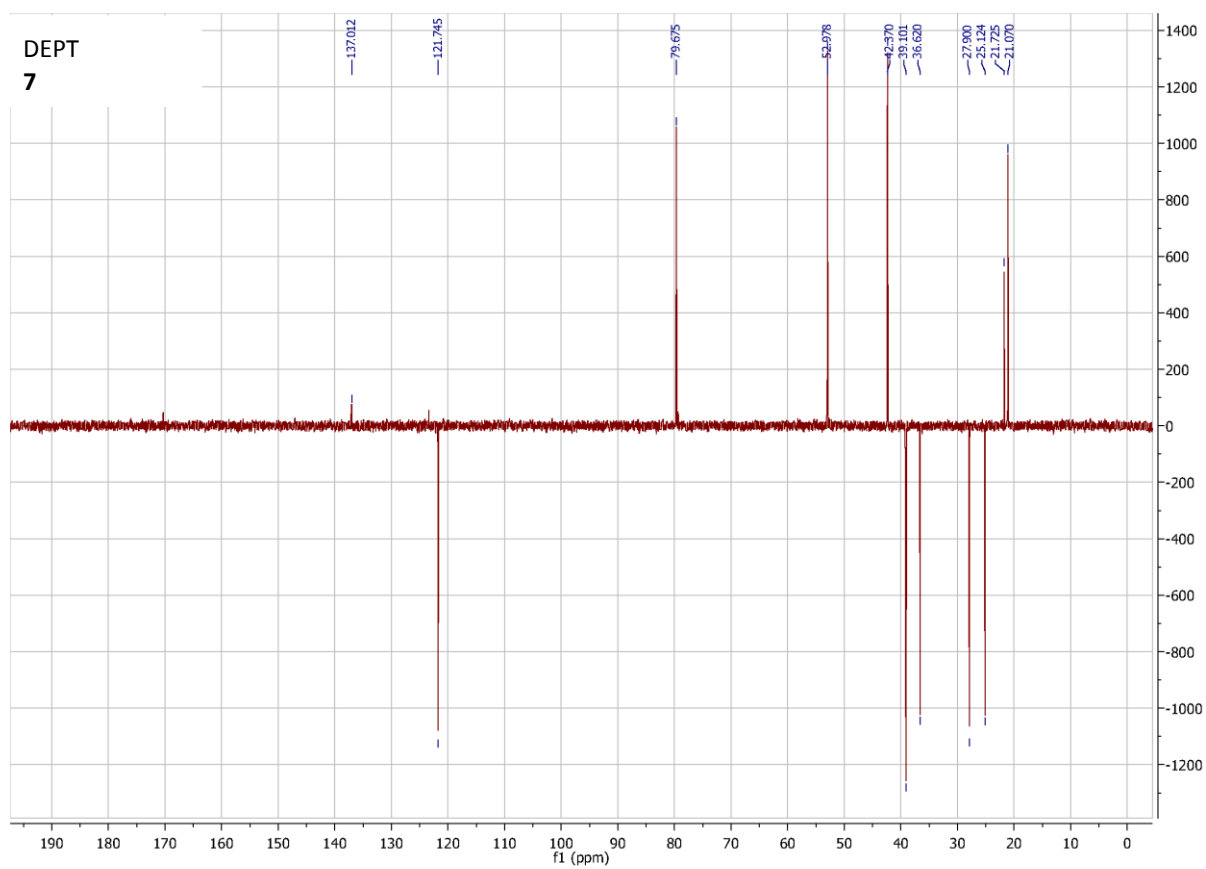


Fig. S57. DEPT spectrum of pseudoivalin (**7**).

NOESY
7

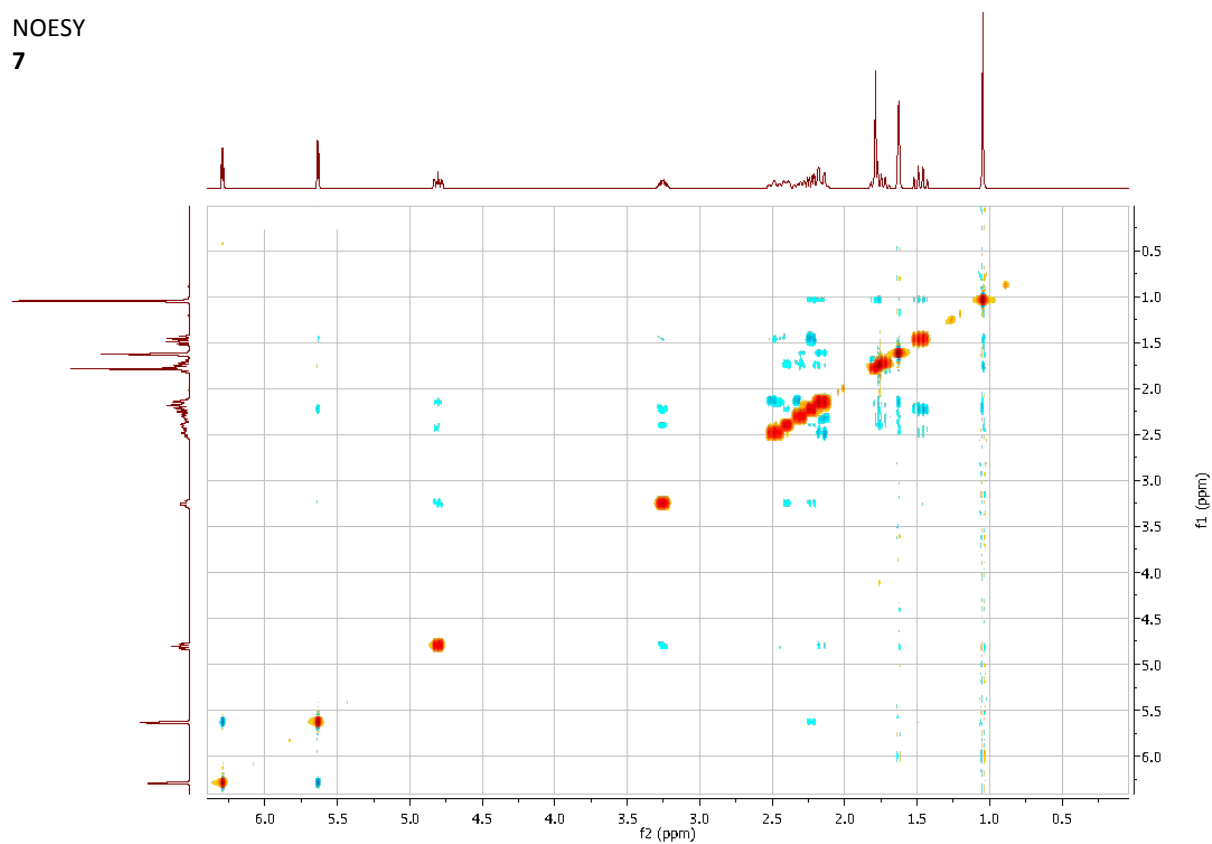


Fig. S59. NOESY spectrum of pseudoivalin (**7**).

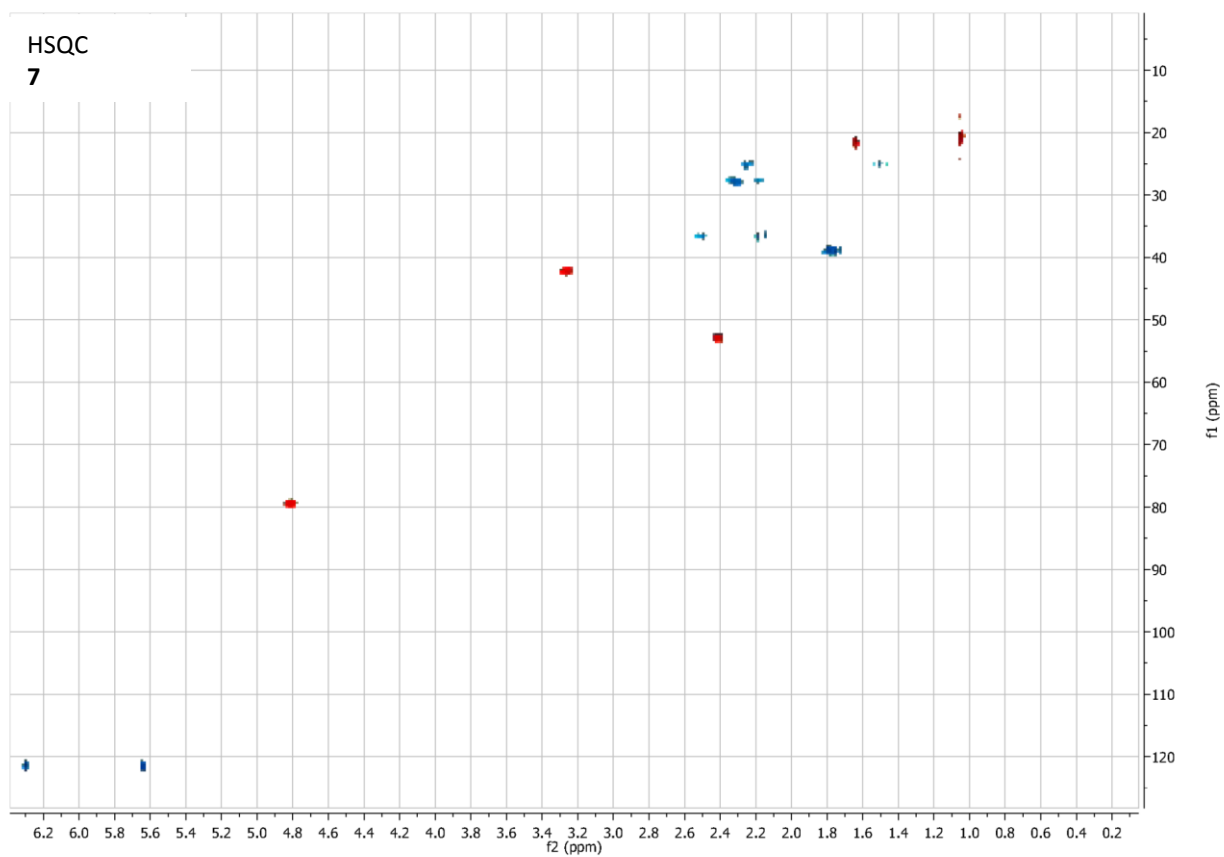


Fig. S60. HSQC spectrum of pseudoivalin (**7**).

HMBC
7

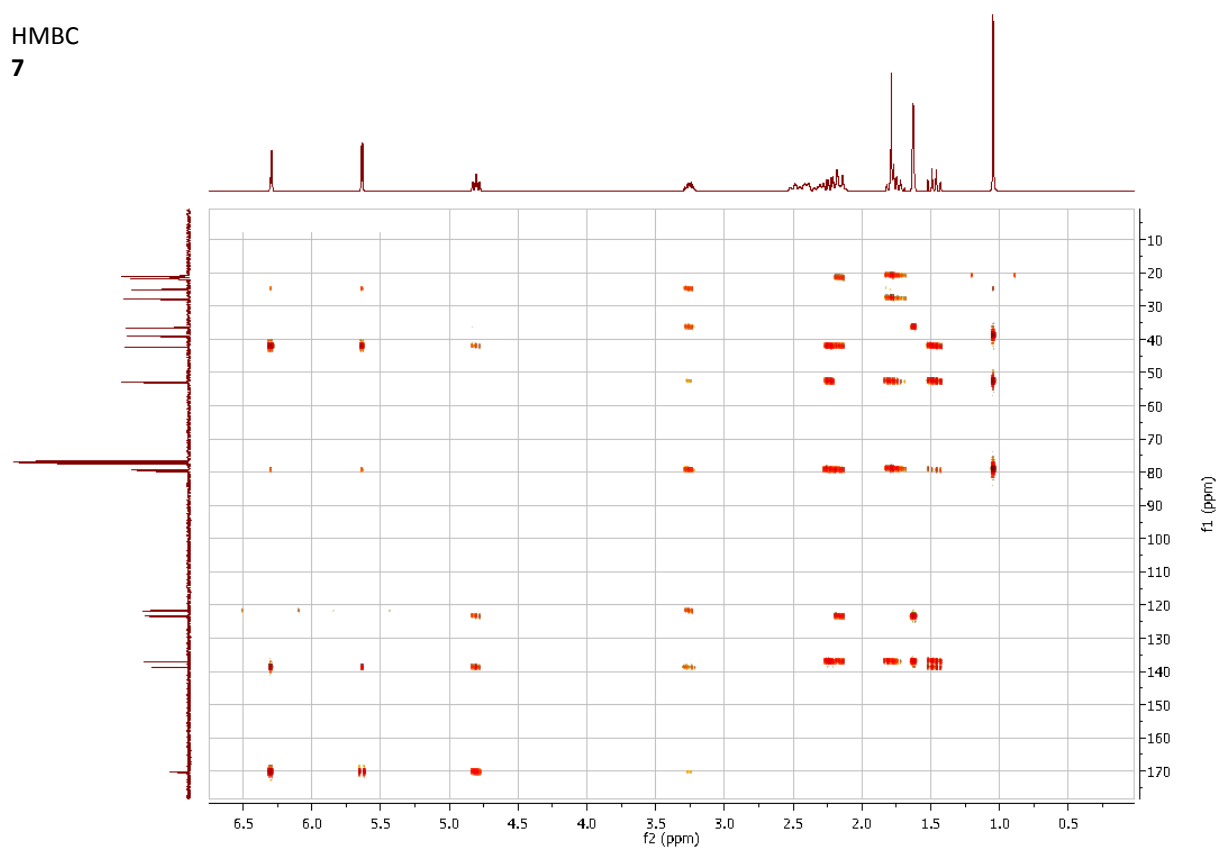
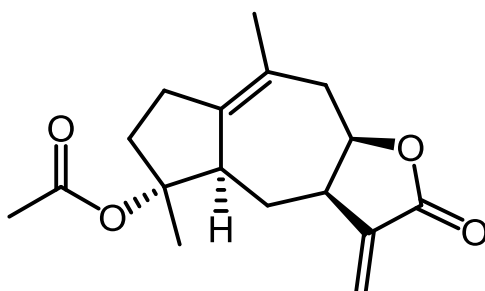


Fig. S61. HMBC spectrum of pseudoivalin (**7**).

Pseudoivalin acetate [*4*α-*acetoxy-5*α*H-10,11(13)-guaidien-8*β,*12-olide*] (**8**)

Colorless crystals; mp 160–161 °C; $[\alpha]_D^{25} -164$ (*c* 0.4, CHCl₃); UV (MeOH) λ_{\max} (log ϵ) 207 (4.05) nm; IR (KBr) ν_{\max} 3430, 2942, 1766, 1725, 1664, 1373, 1260, 1117, 1060 cm⁻¹; APCIMS: *m/z* 248 [M + H – Ac]⁺ (21), 231 [M + H – HOAc]⁺ (100); HRESIMS *m/z* 313.1408 (calcd for C₁₇H₂₂O₄Na, 313.1416).



Pseudoivalin acetate (**8**)

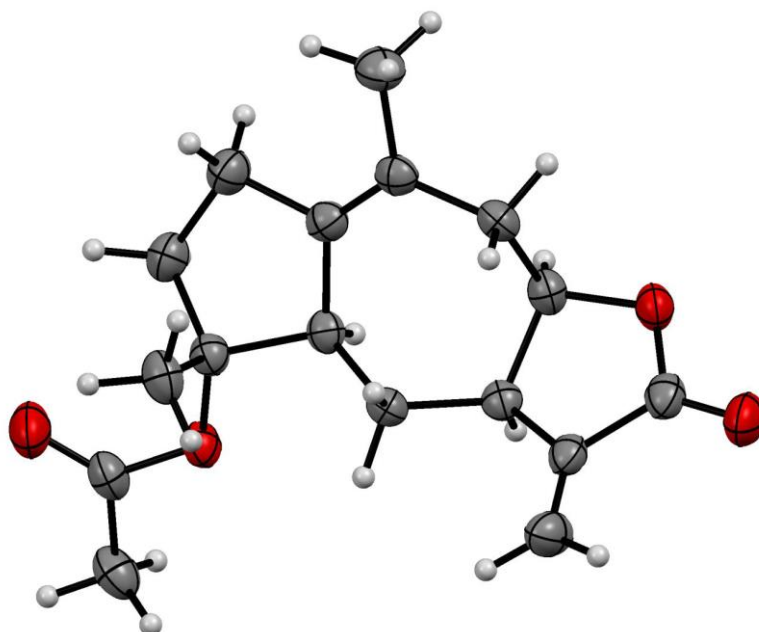


Fig. S62. Crystal structure of pseudoivalin acetate (**8**) [ORTEP diagram] with displacement ellipsoids at 50% probability for non-H atoms.

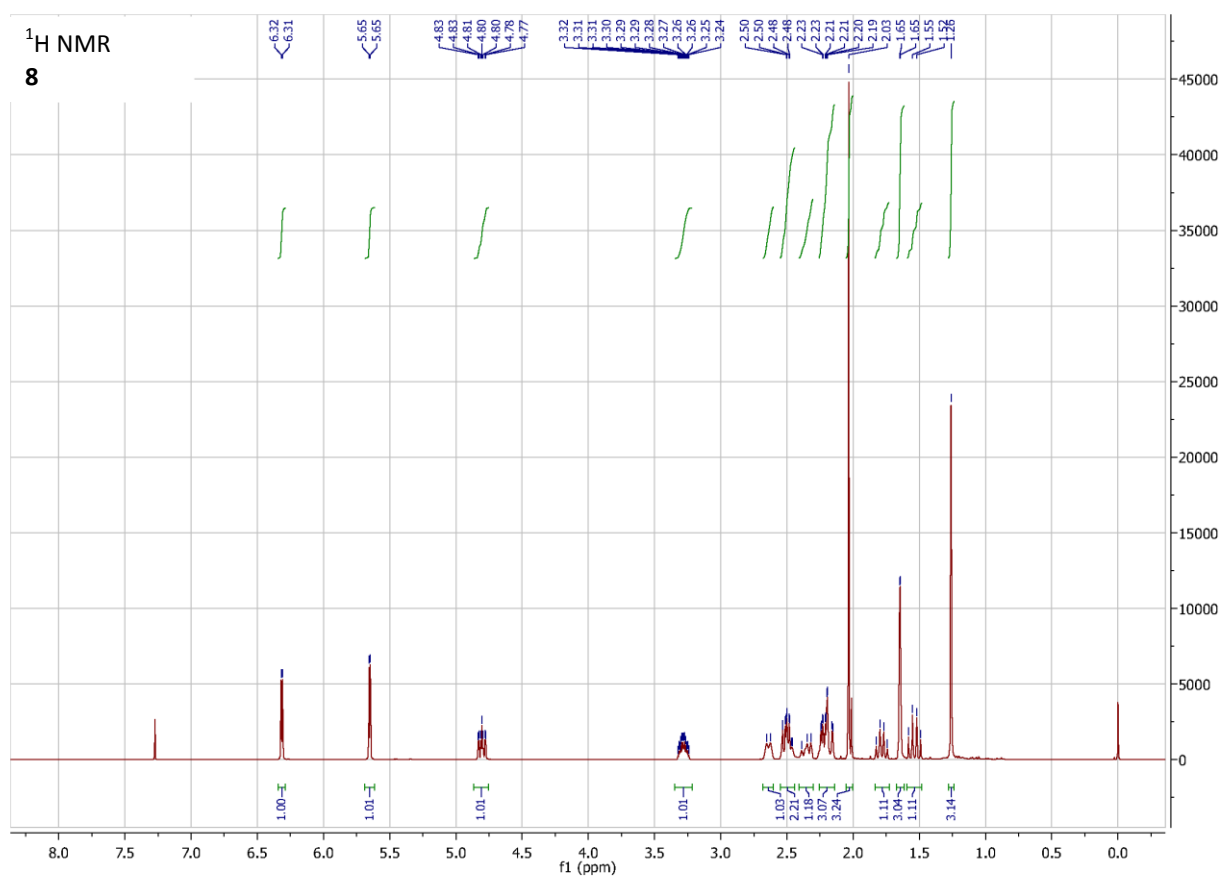


Fig. S63. ¹H NMR (400 MHz, CDCl₃) spectrum of pseudoivalin acetate (**8**).

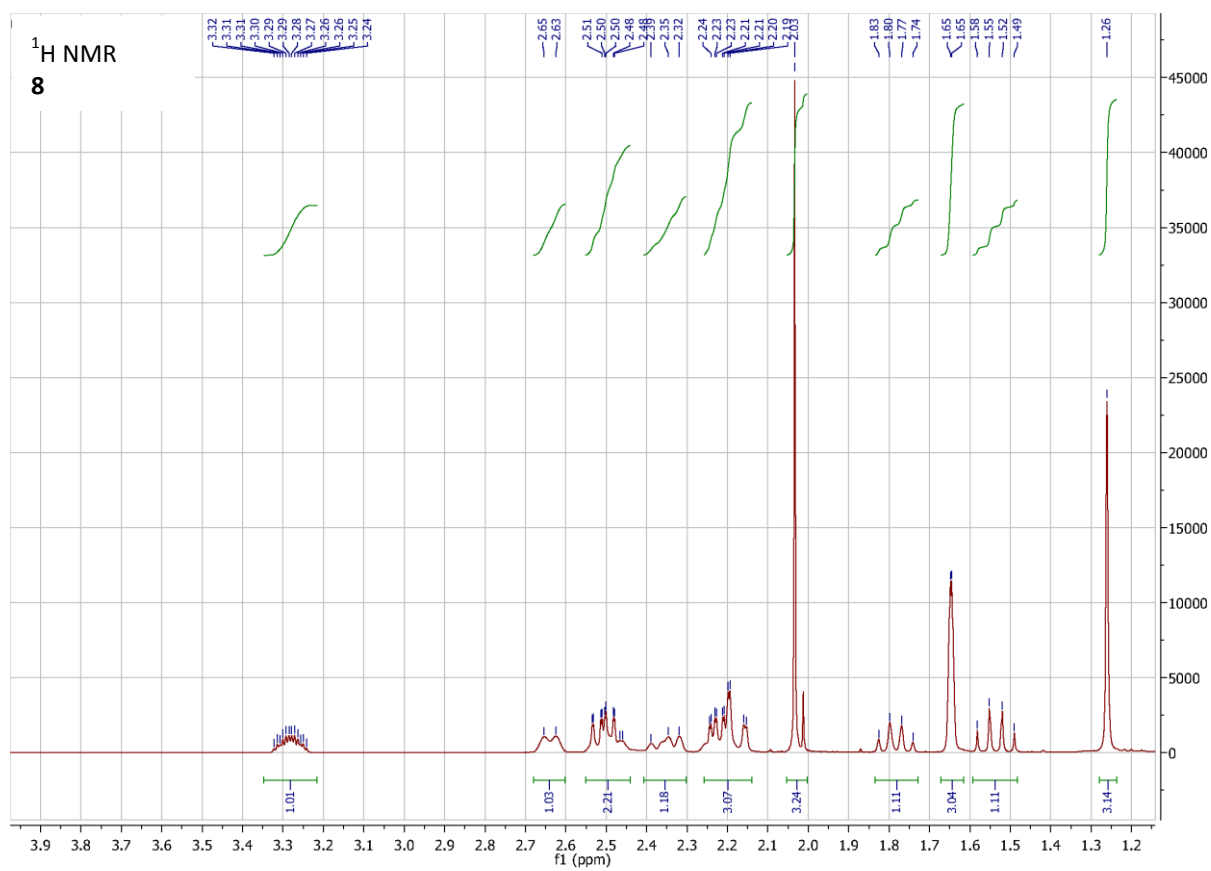


Fig. S64. ¹H NMR (400 MHz, CDCl₃) spectrum of pseudoivalin acetate (**8**) expanded in the region 1.03–3.98 ppm.

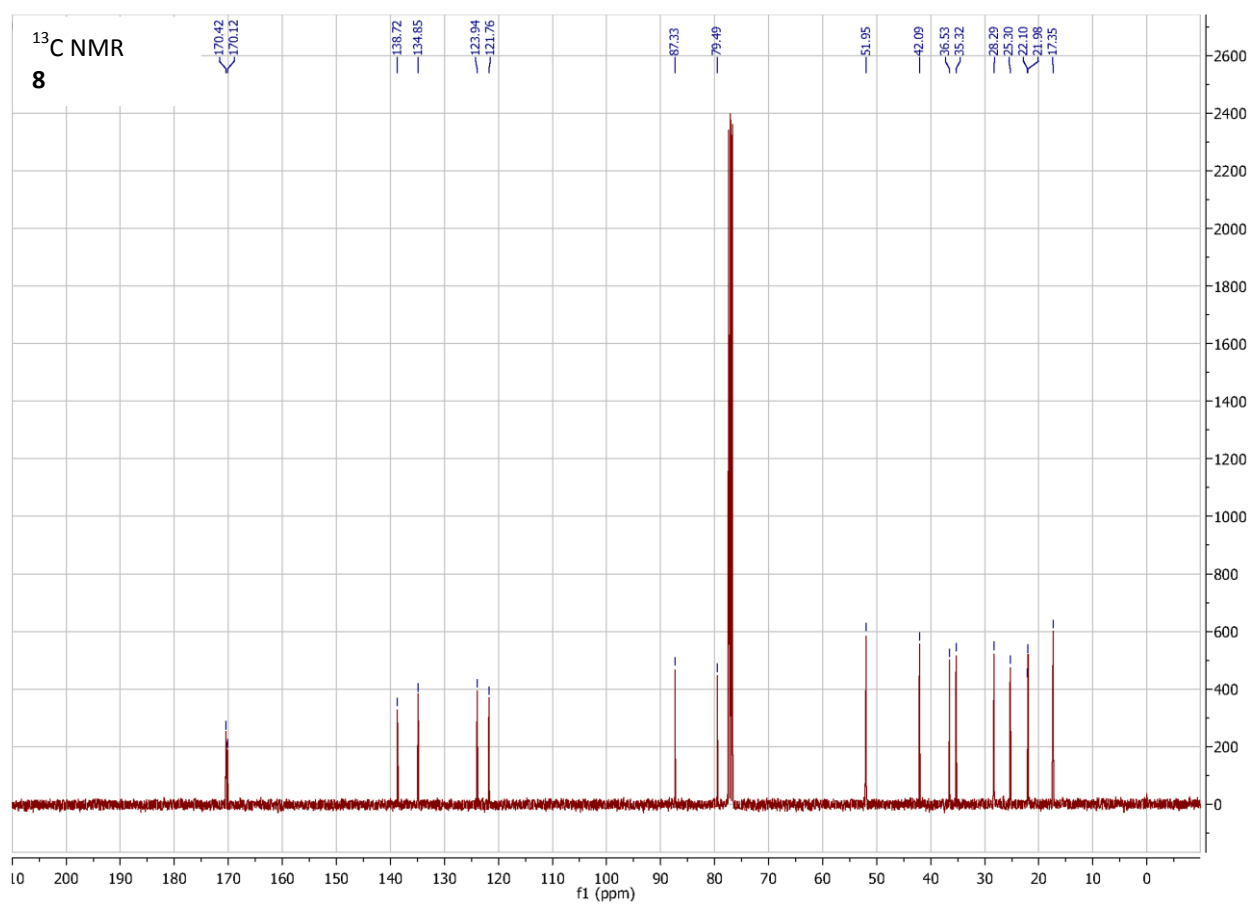


Fig. S65. ¹³C NMR (100 MHz, CDCl₃) spectrum of pseudoivalin acetate (**8**).

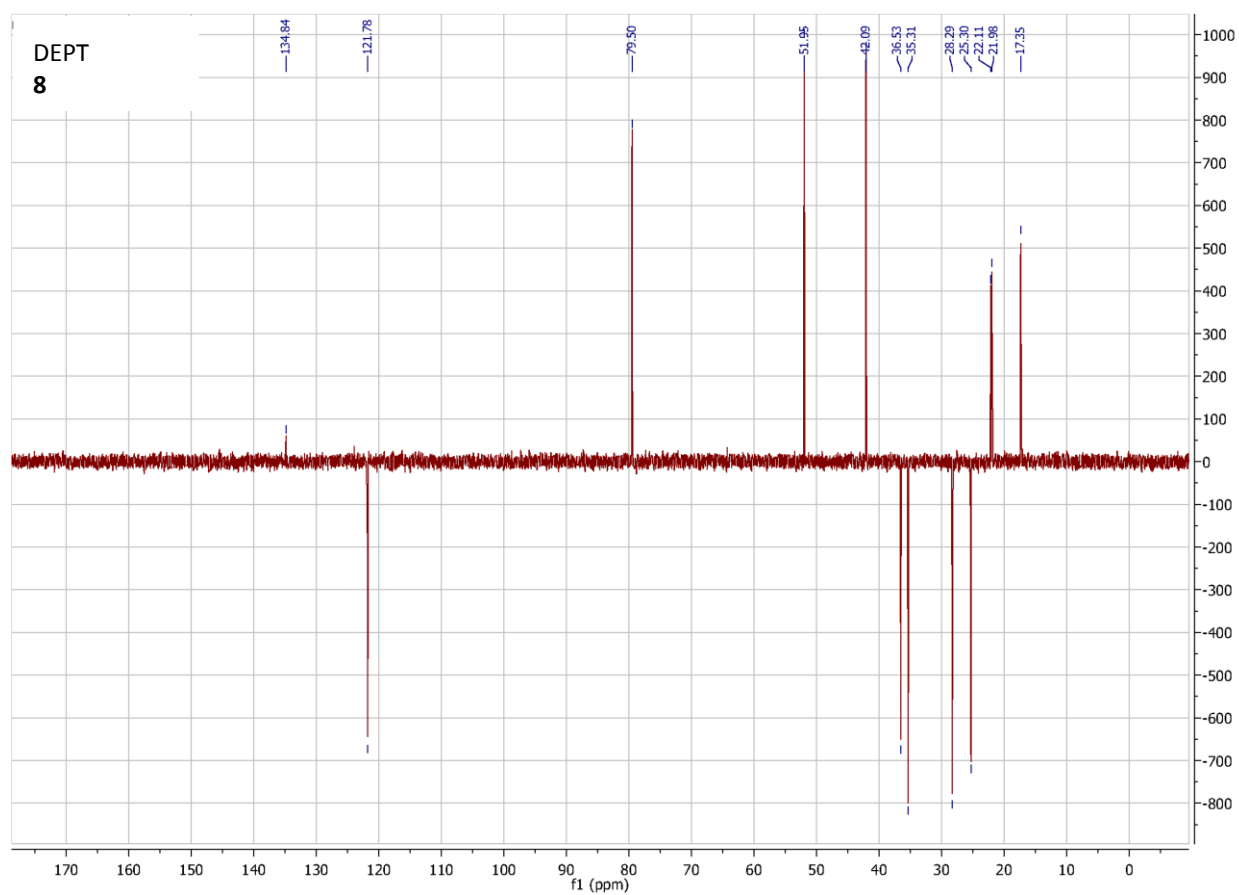


Fig. S66. DEPT spectrum of pseudoivalin acetate (**8**).

COSY
8

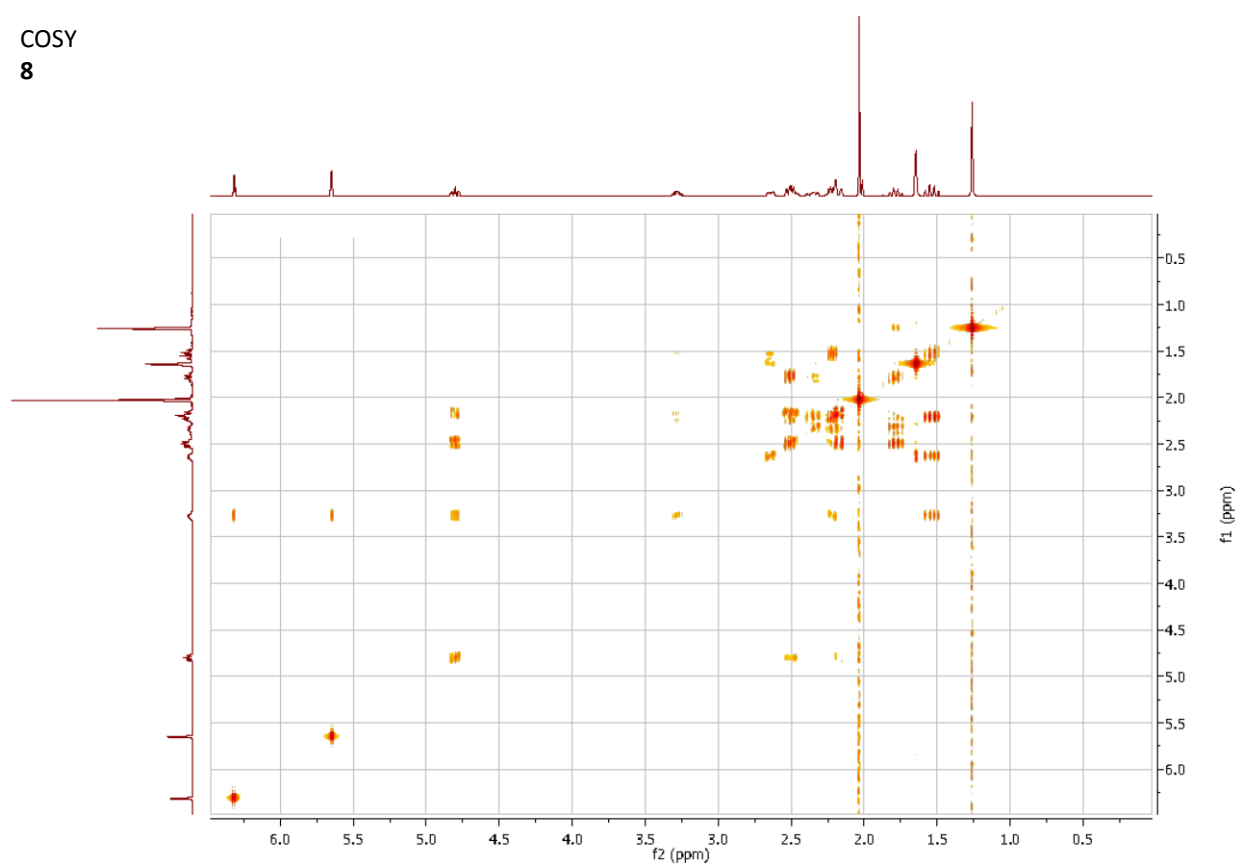


Fig. S67. ¹H–¹H COSY spectrum of pseudoivalin acetate (**8**).

NOESY
8

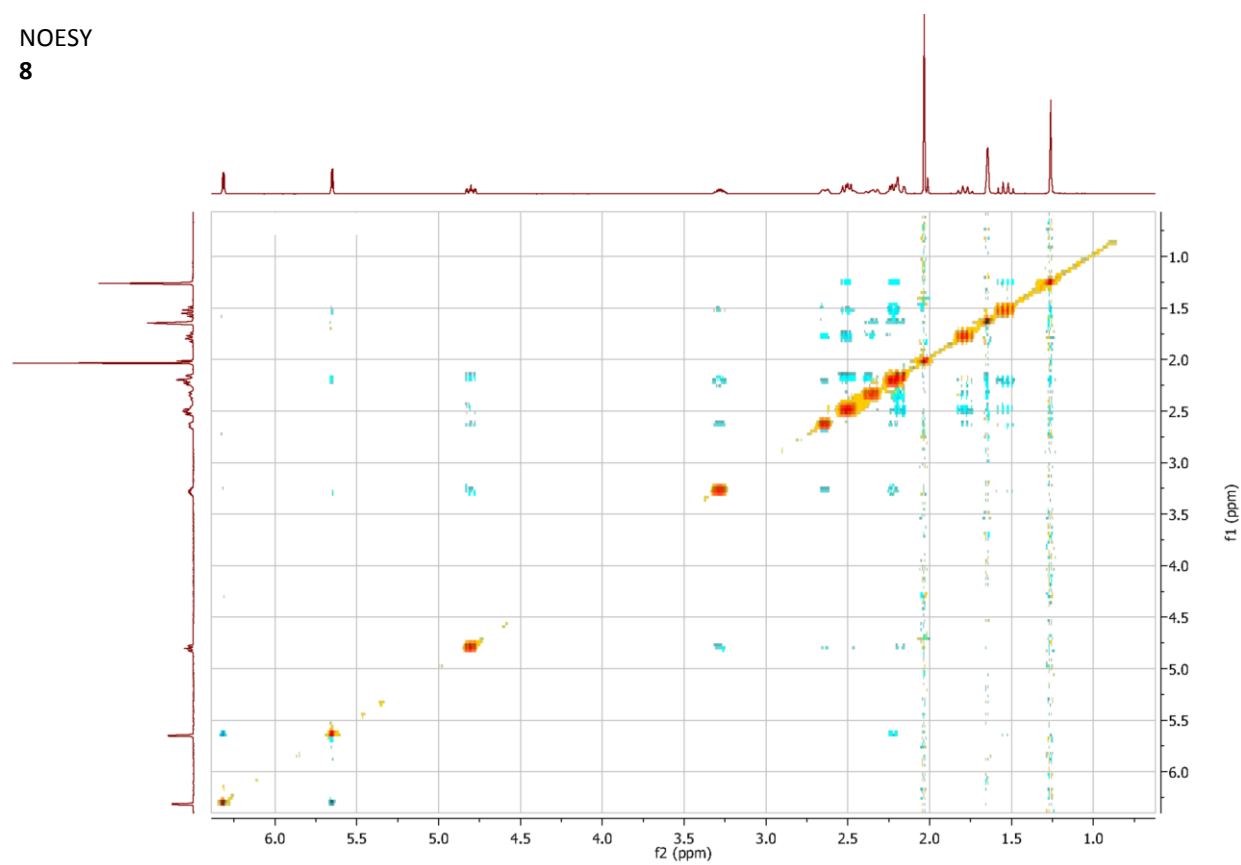


Fig. S68. NOESY spectrum of pseudoivalin acetate (**8**).

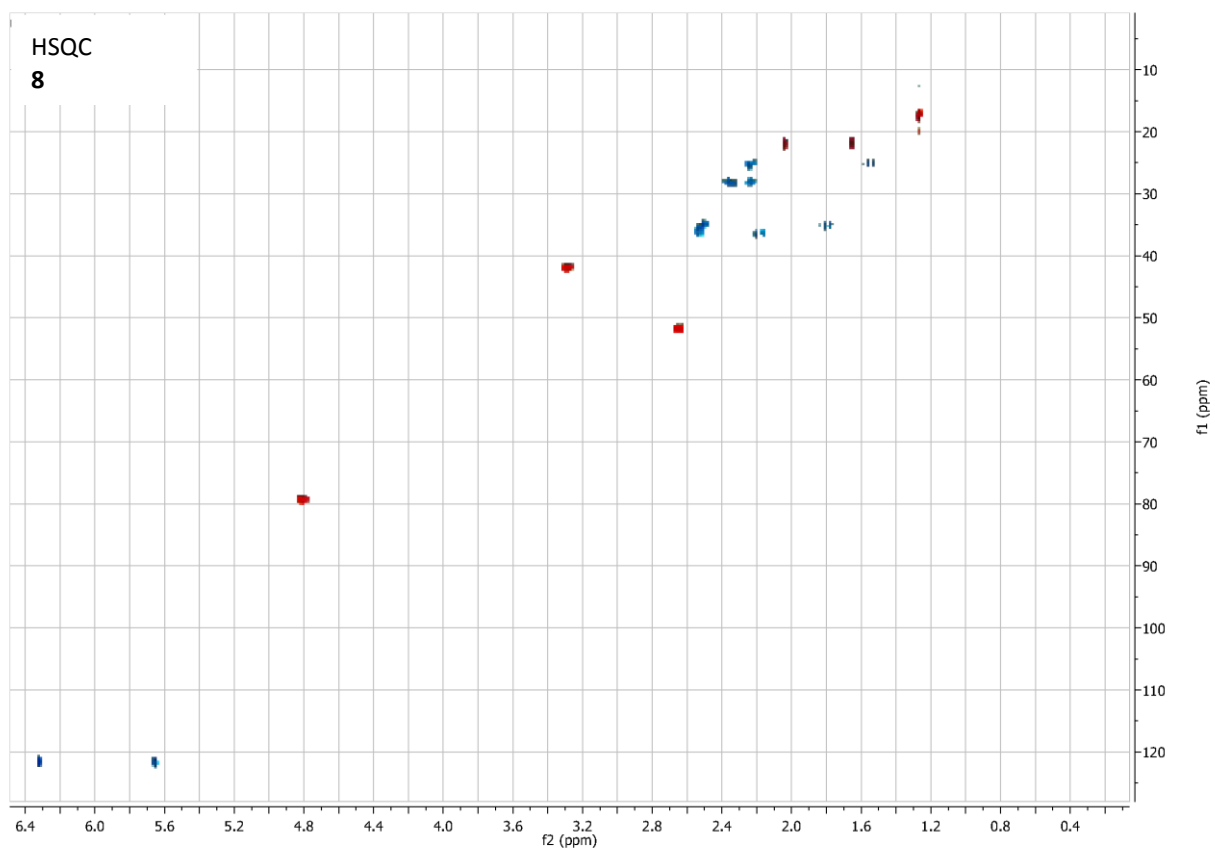


Fig. S69. HSQC spectrum of pseudoivalin acetate (**8**).

HMBC
8

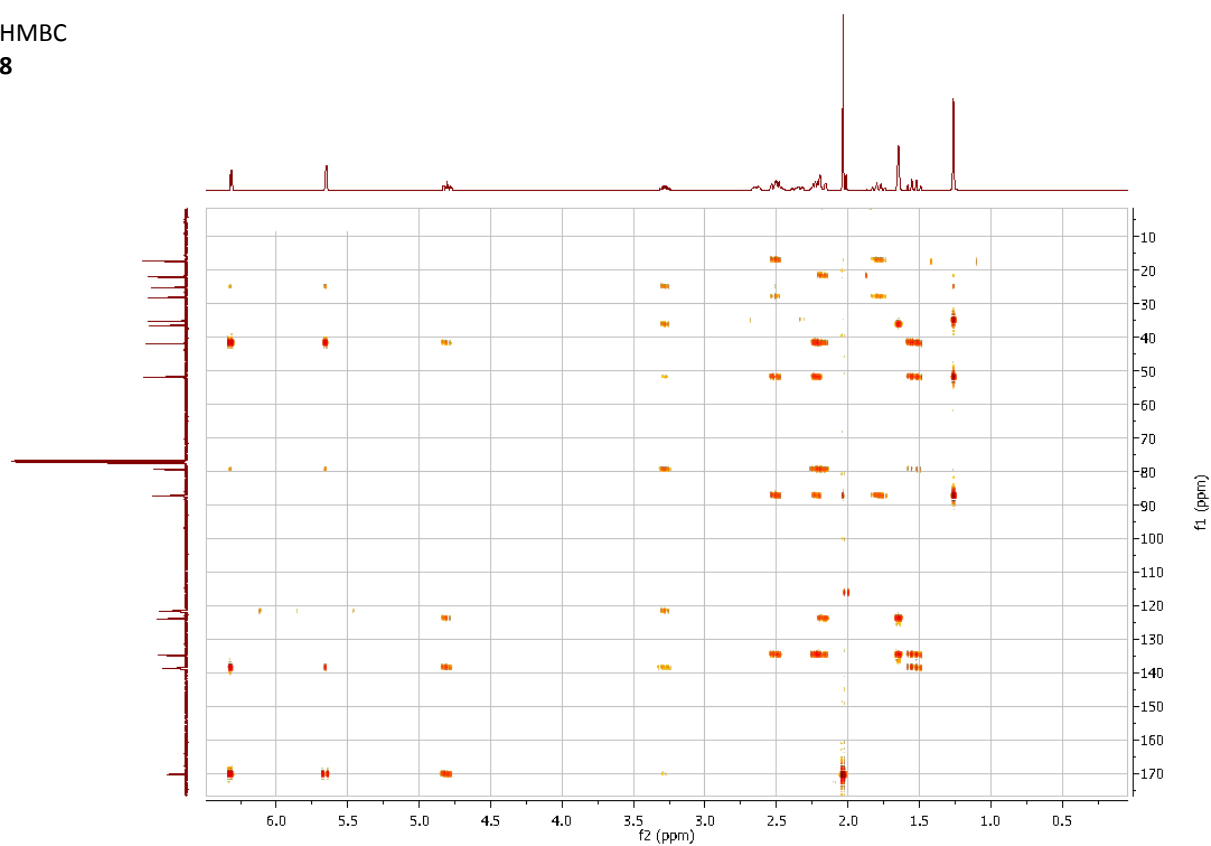
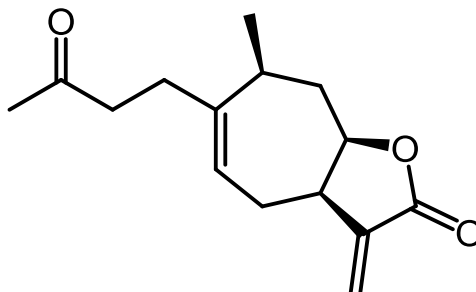


Fig. S70. HMBC spectrum of pseudoivalin acetate (8).

Tomentosin (9)

Colorless oil; $[\alpha]_D^{25} +8$ (c 0.4, CHCl_3); UV (MeOH) λ_{max} ($\log \epsilon$) 215 (4.12) nm; APCIMS: m/z 249

$[\text{M} + \text{H}]^+$ (100), 231 $[\text{M} + \text{H} - \text{H}_2\text{O}]^+$ (65); HRESIMS m/z 249.1490 (calcd for $\text{C}_{15}\text{H}_{21}\text{O}_3$, 249.1491).



Tomentosin (**9**)

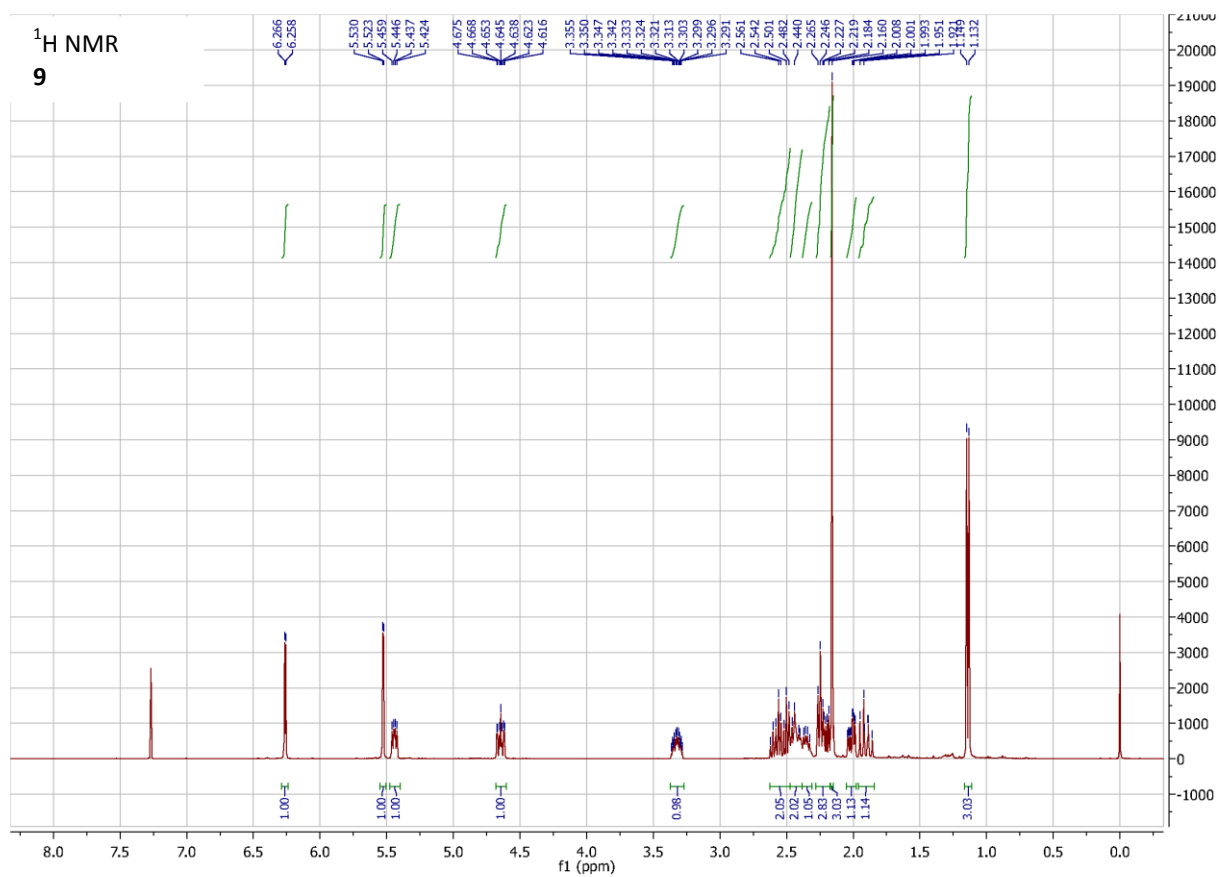


Fig. S71. ¹H NMR (400 MHz, CDCl₃) spectrum of tomentosin (9).

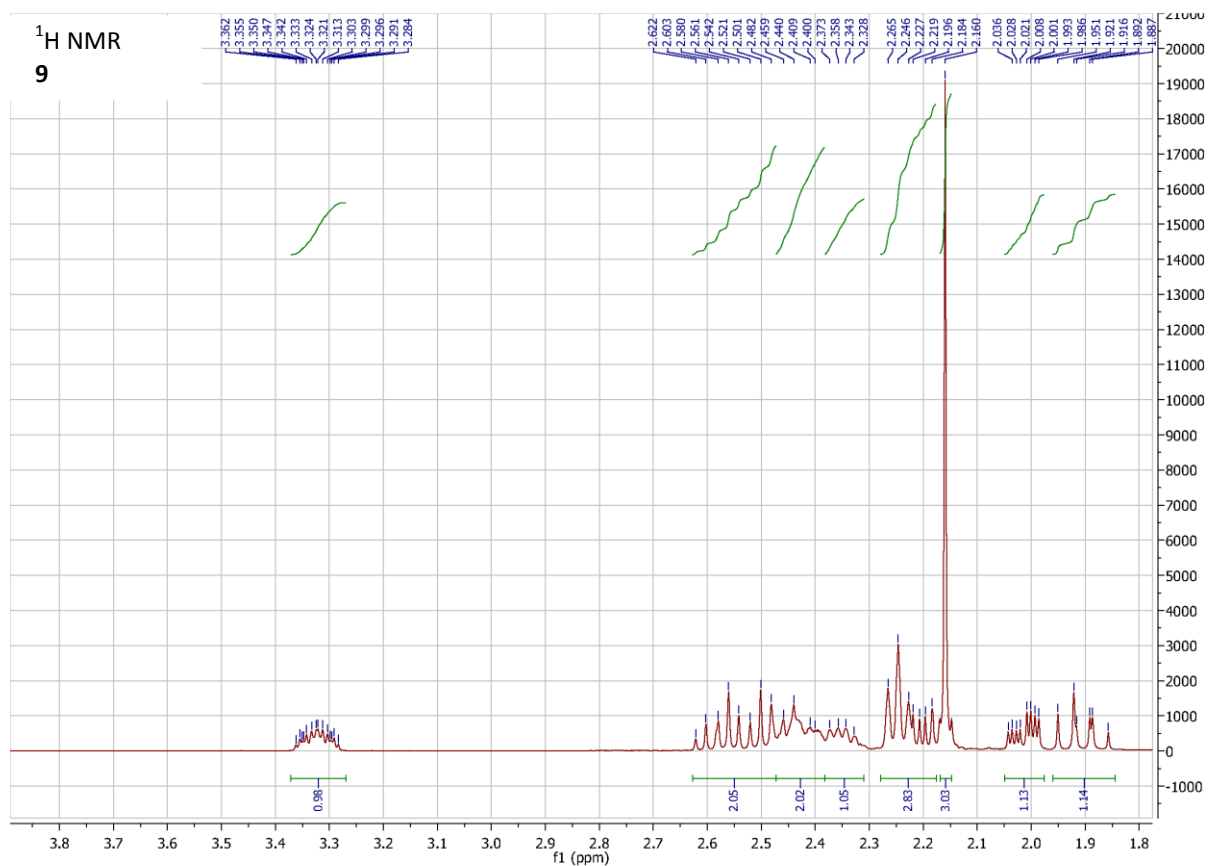


Fig. S72. ¹H NMR (400 MHz, CDCl₃) spectrum of tomentosin (**9**) expanded in the region 1.78–3.98 ppm.

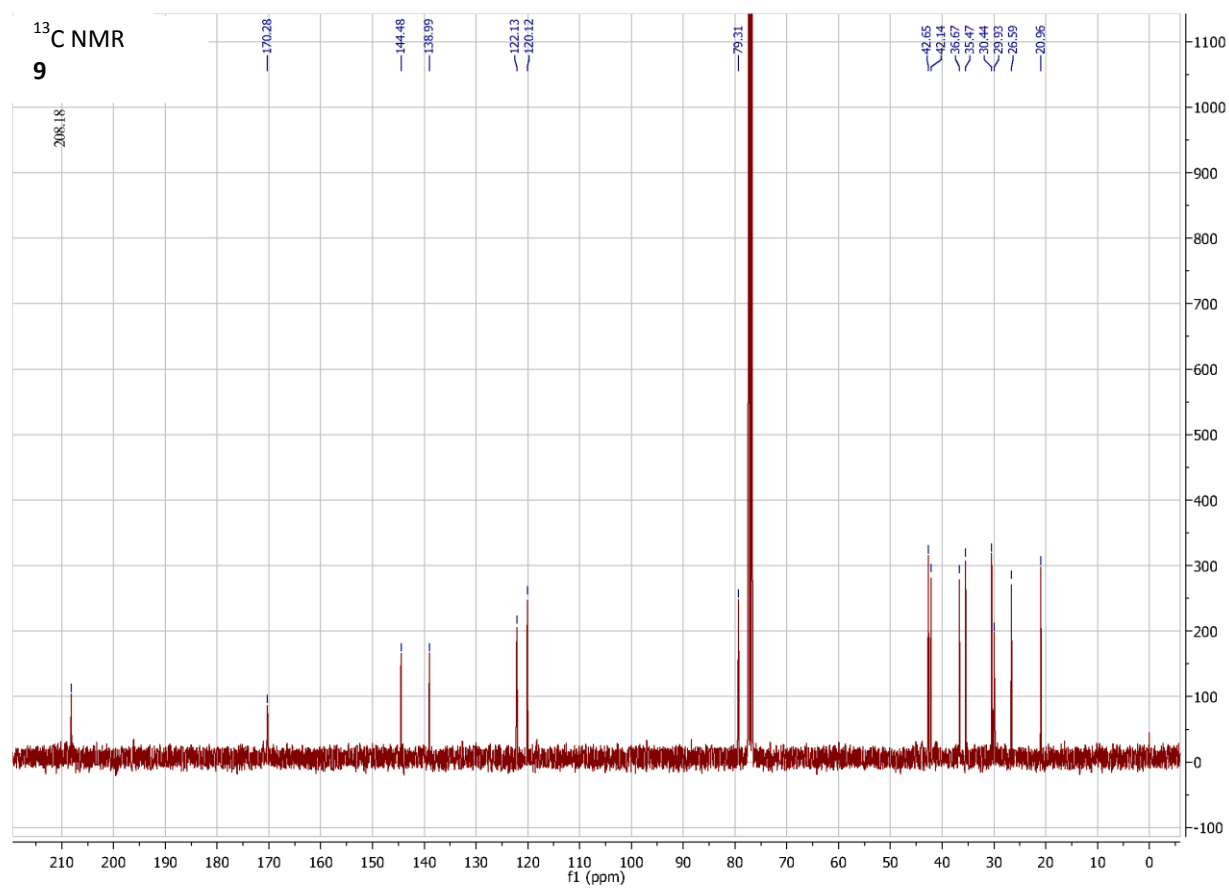


Fig. S73. ¹³C NMR (100 MHz, CDCl₃) spectrum of tomentosin (9).

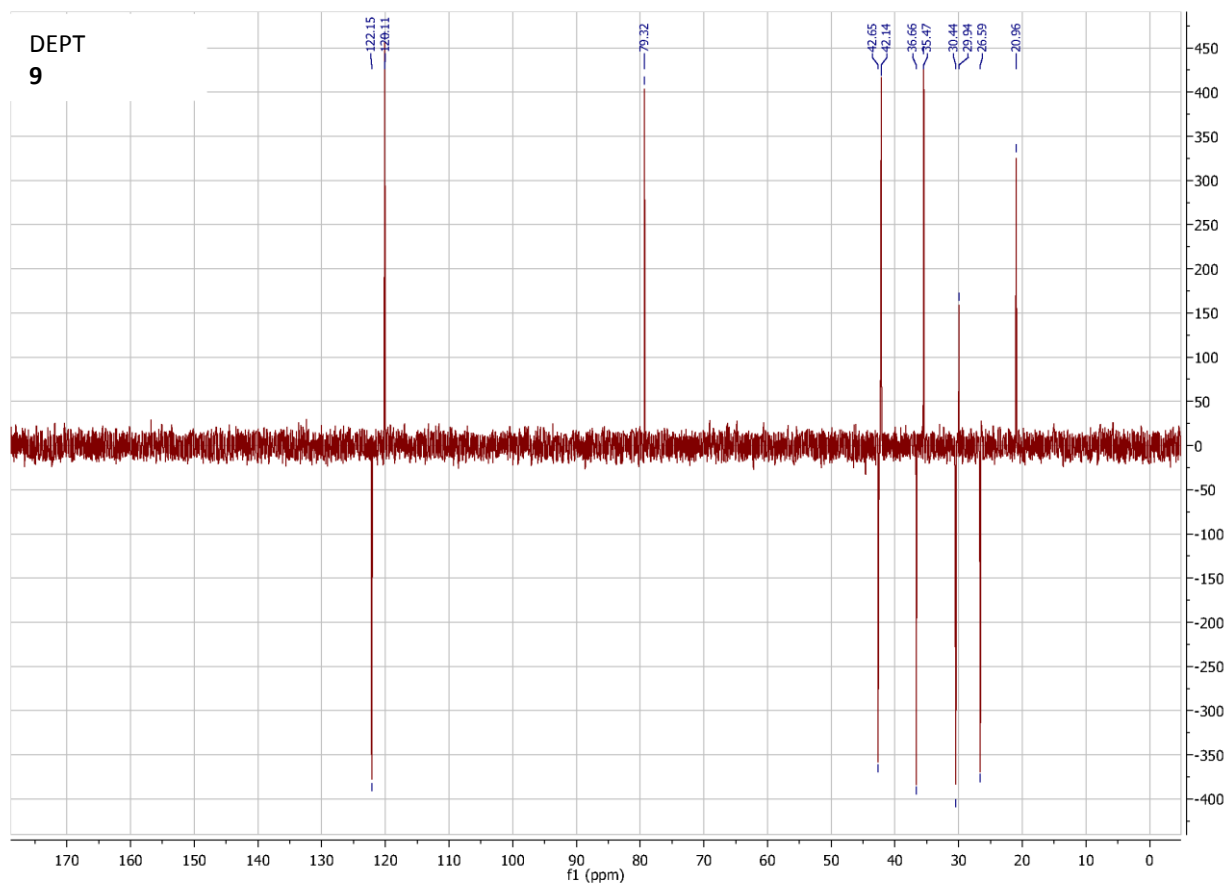


Fig. S74. DEPT spectrum of tomentosin (9).

COSY
9

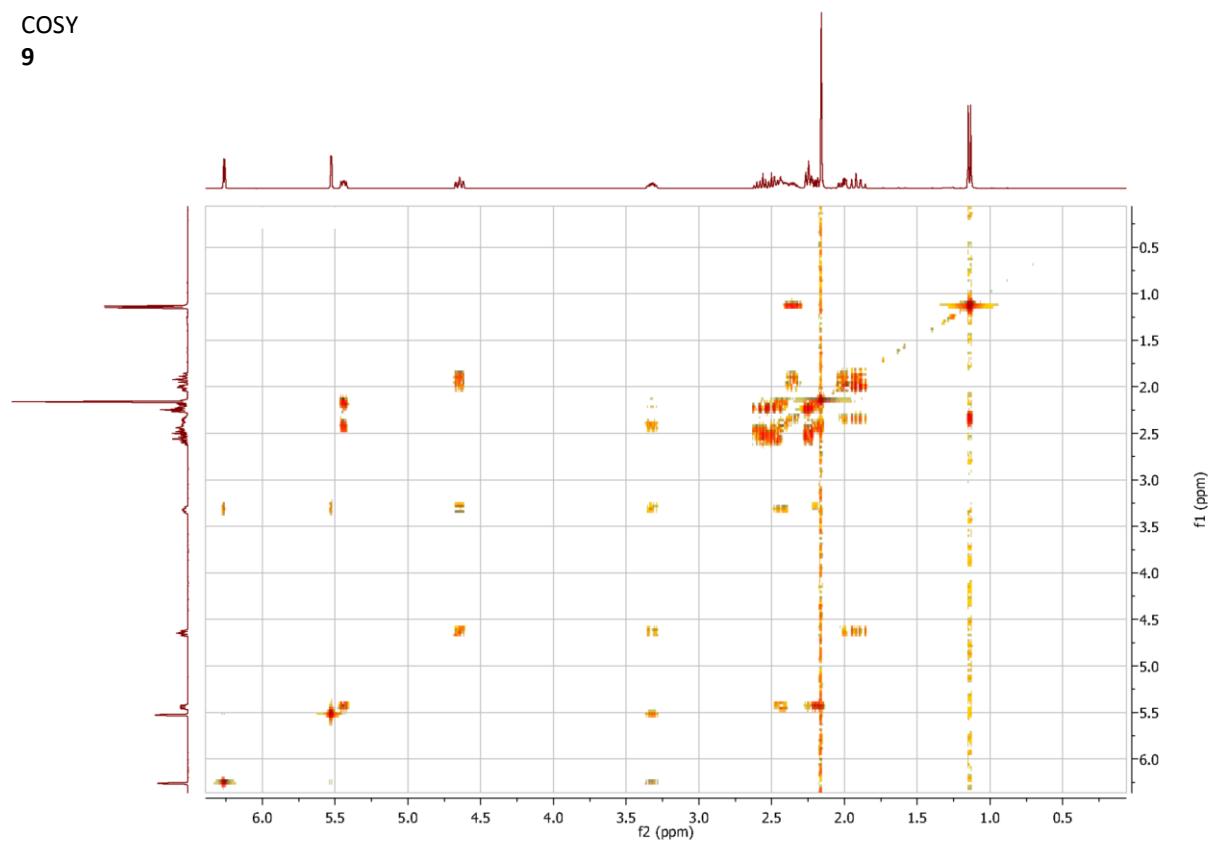


Fig. S75. ¹H–¹H COSY spectrum of tomentosin (9).

NOESY
9

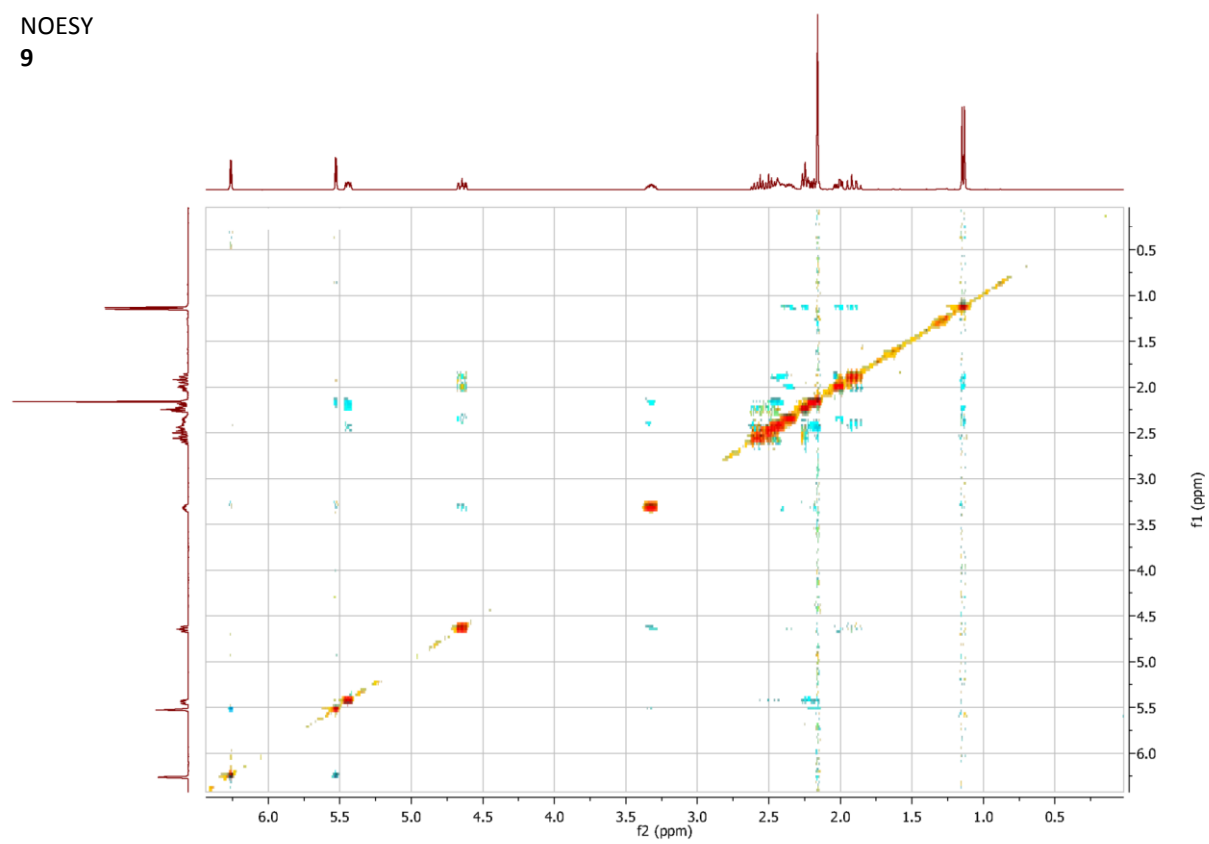


Fig. S76. NOESY spectrum of tomentosin (9).

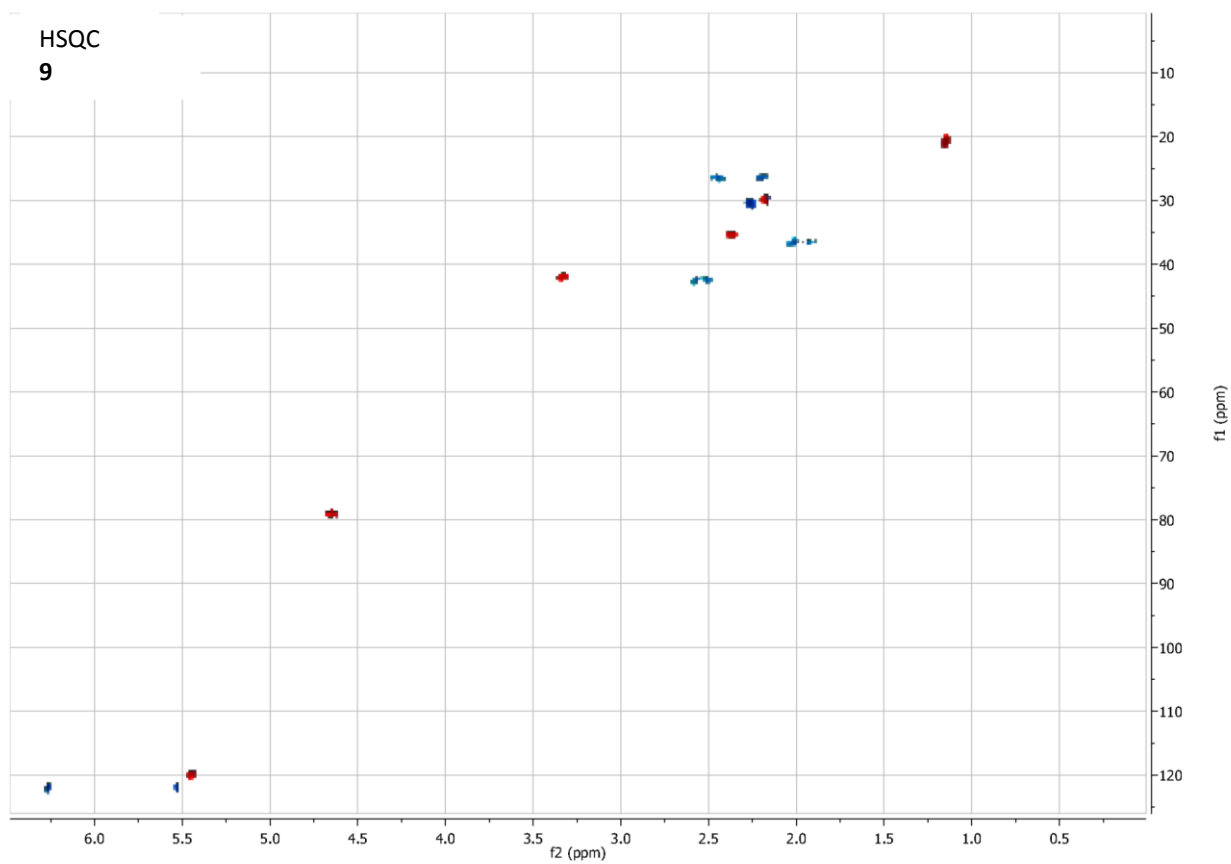


Fig. S77. HSQC spectrum of tomentosin (**9**).

HMBC
9

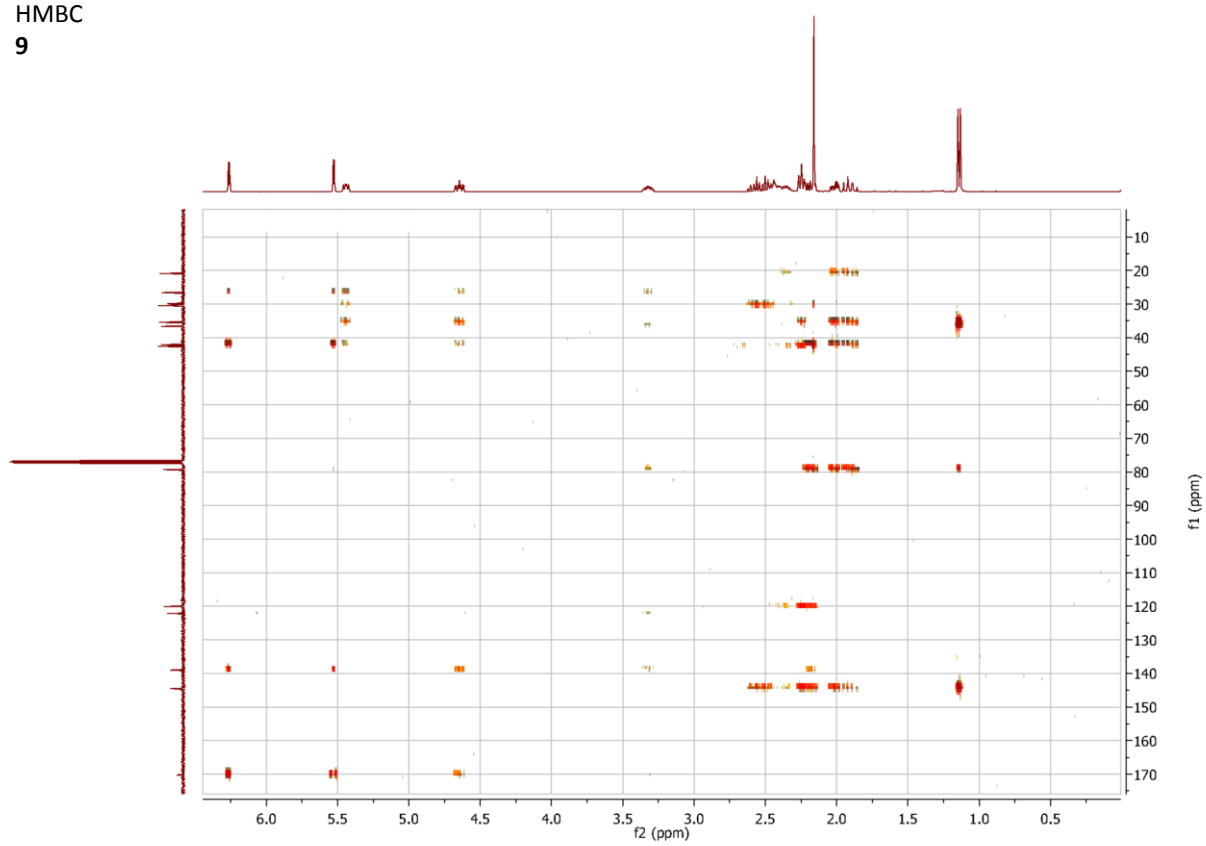


Fig. S78. HMBC spectrum of tomentosin (9).

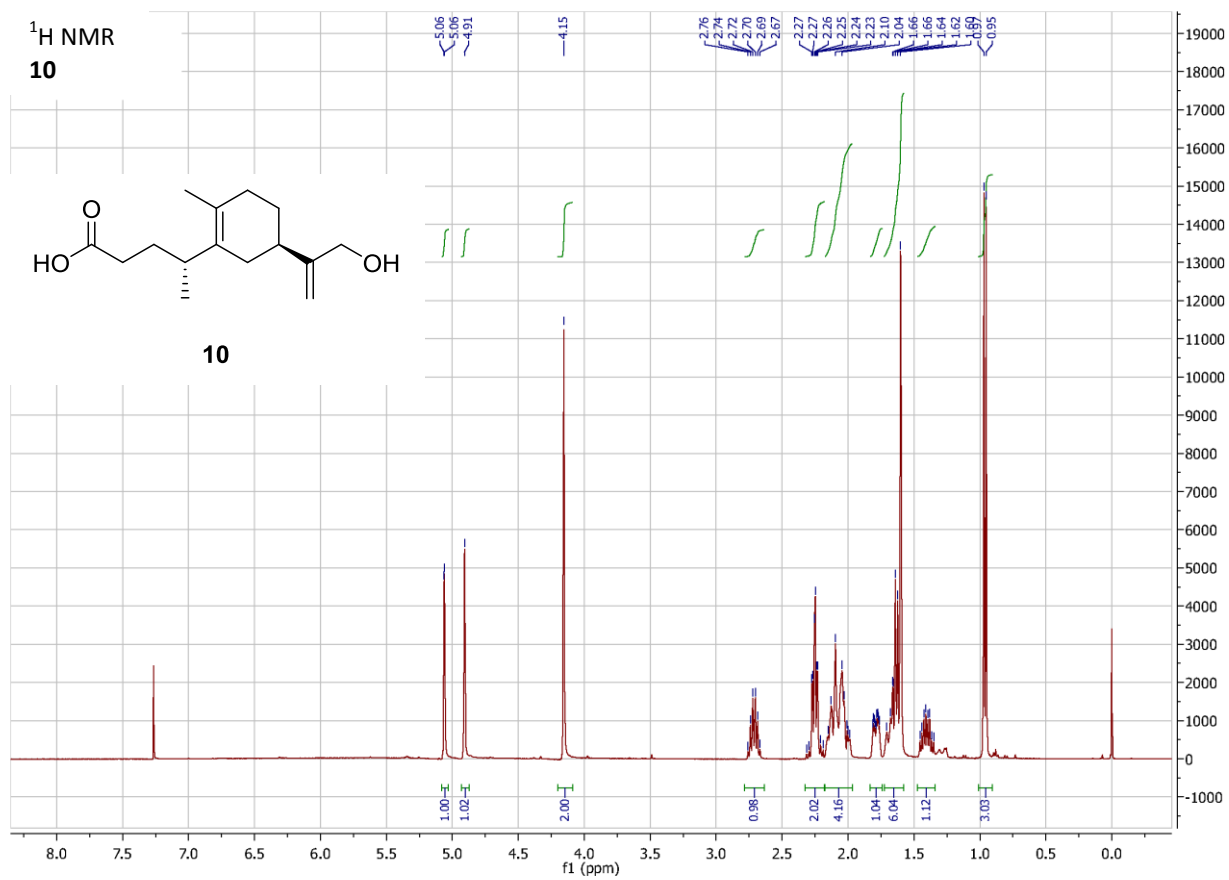


Fig. S79. ¹H NMR (400 MHz, CDCl₃) spectrum of the new compound **10**.

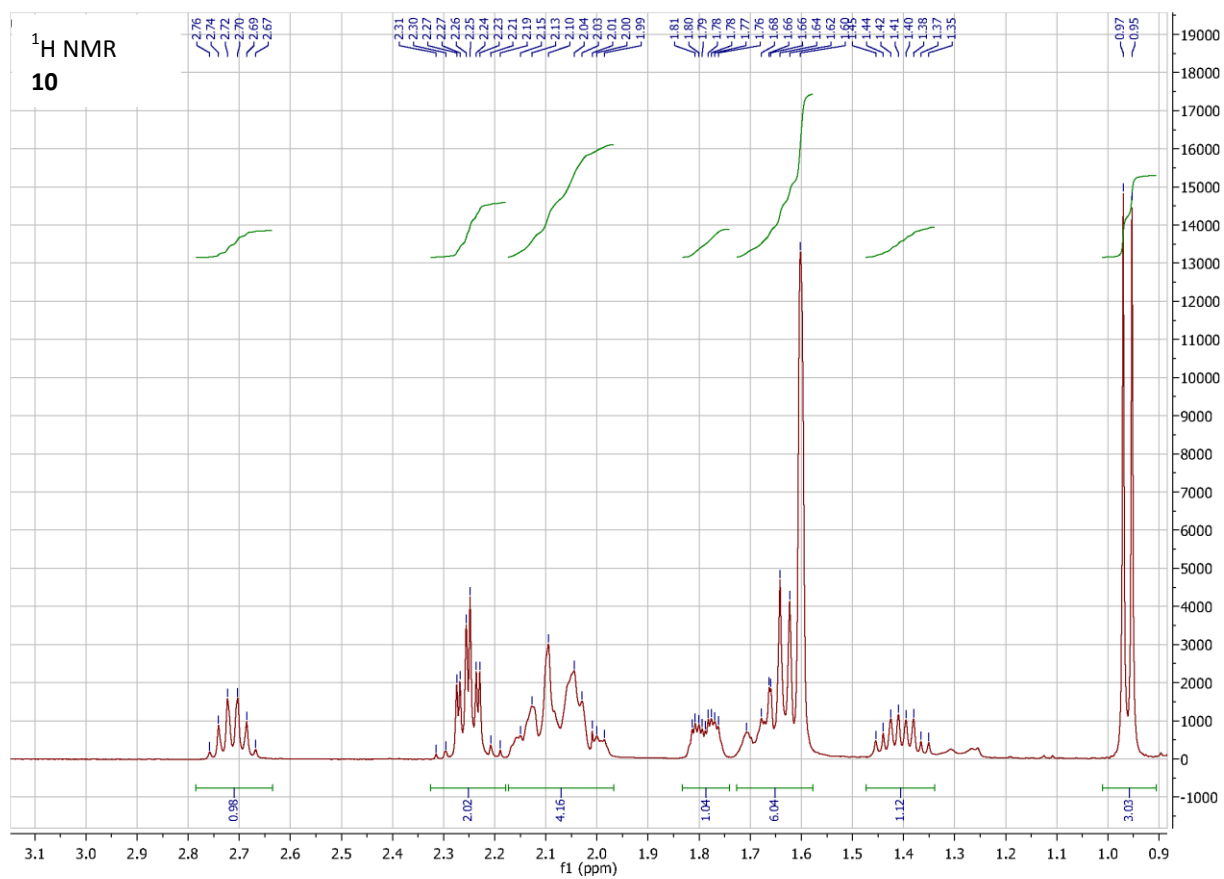


Fig. S80. ¹H NMR (400 MHz, CDCl₃) spectrum of the new compound **10** expanded in the region 0.88–3.15 ppm.

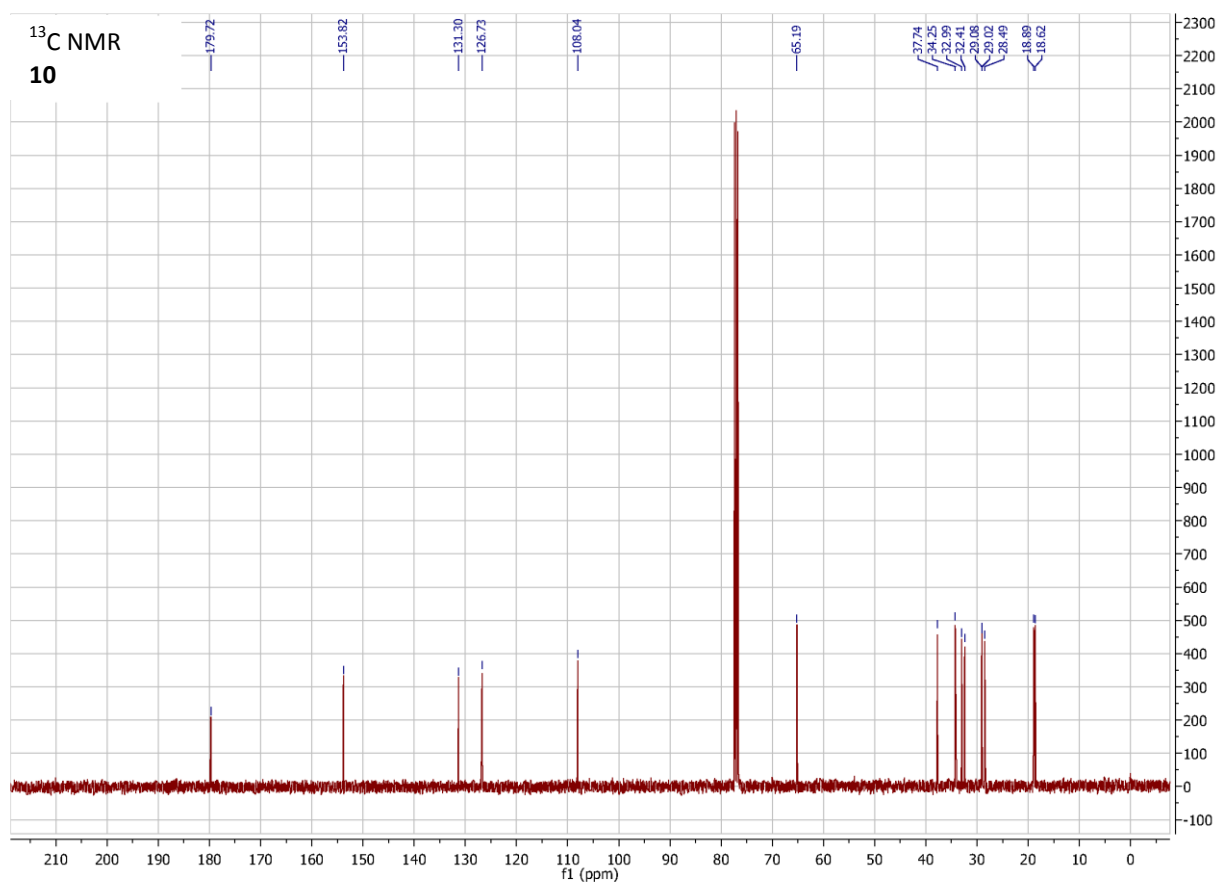


Fig. S81. ¹³C NMR (100 MHz, CDCl₃) spectrum of the new compound **10**.

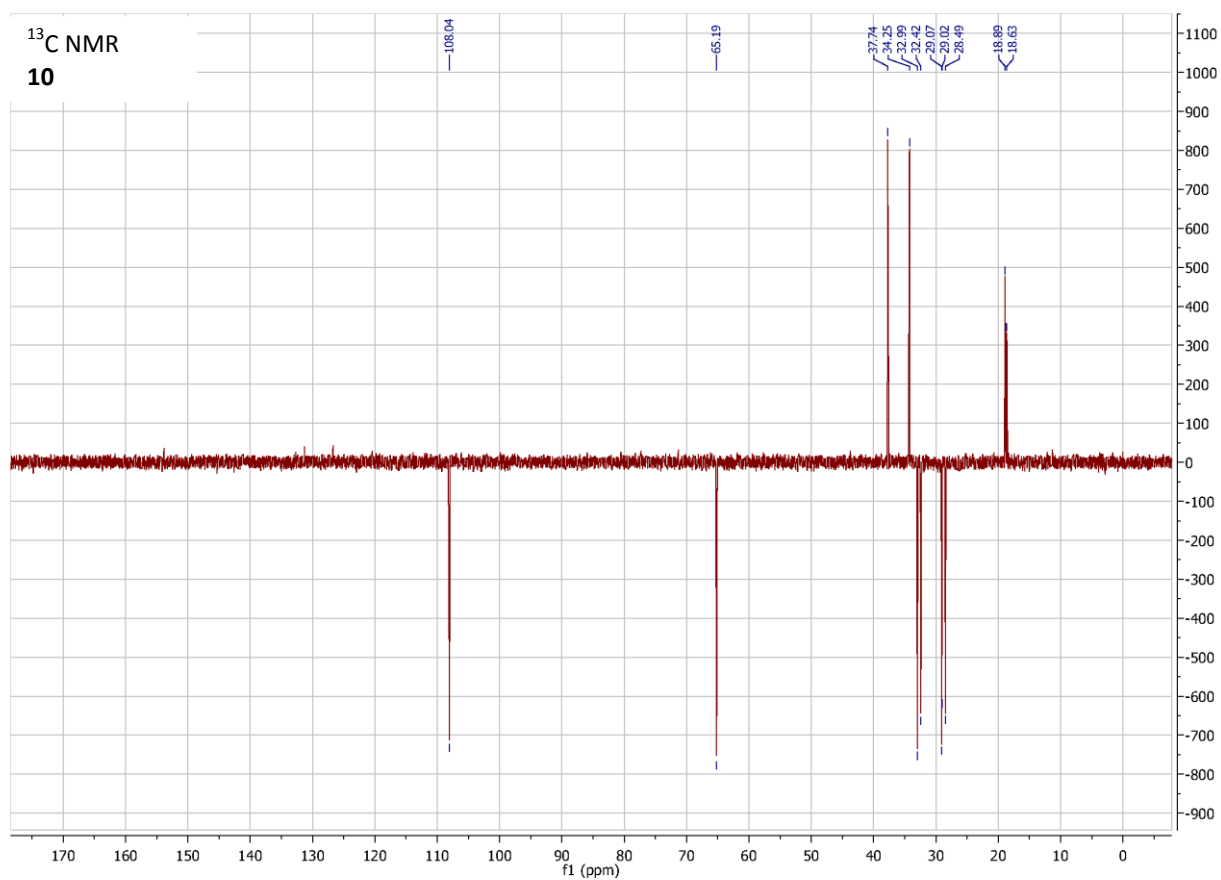


Fig. S82. DEPT spectrum of the new compound **10**.

COSY
10

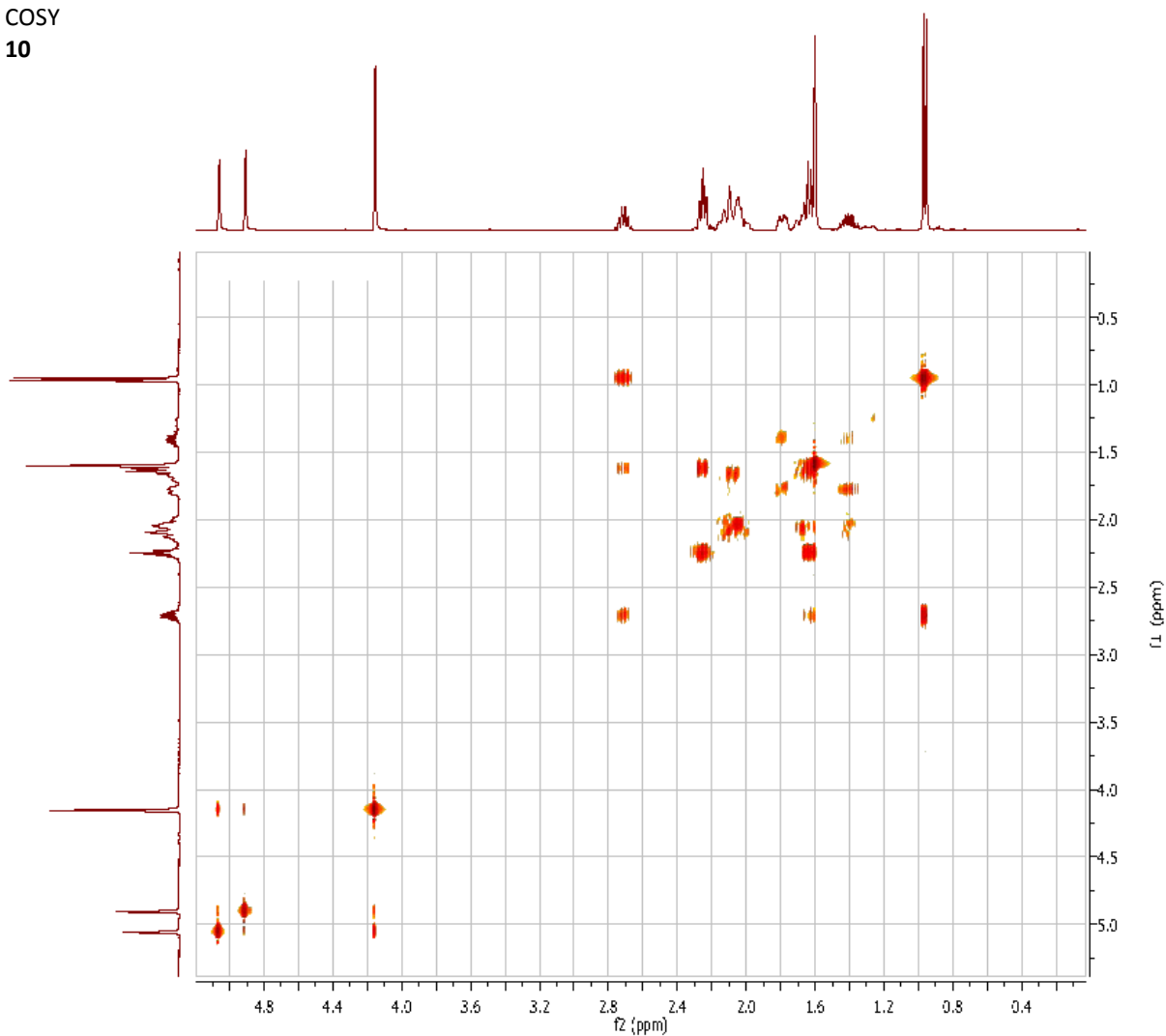


Fig. S83. ^1H - ^1H COSY spectrum of the new compound **10**.

NOESY
10

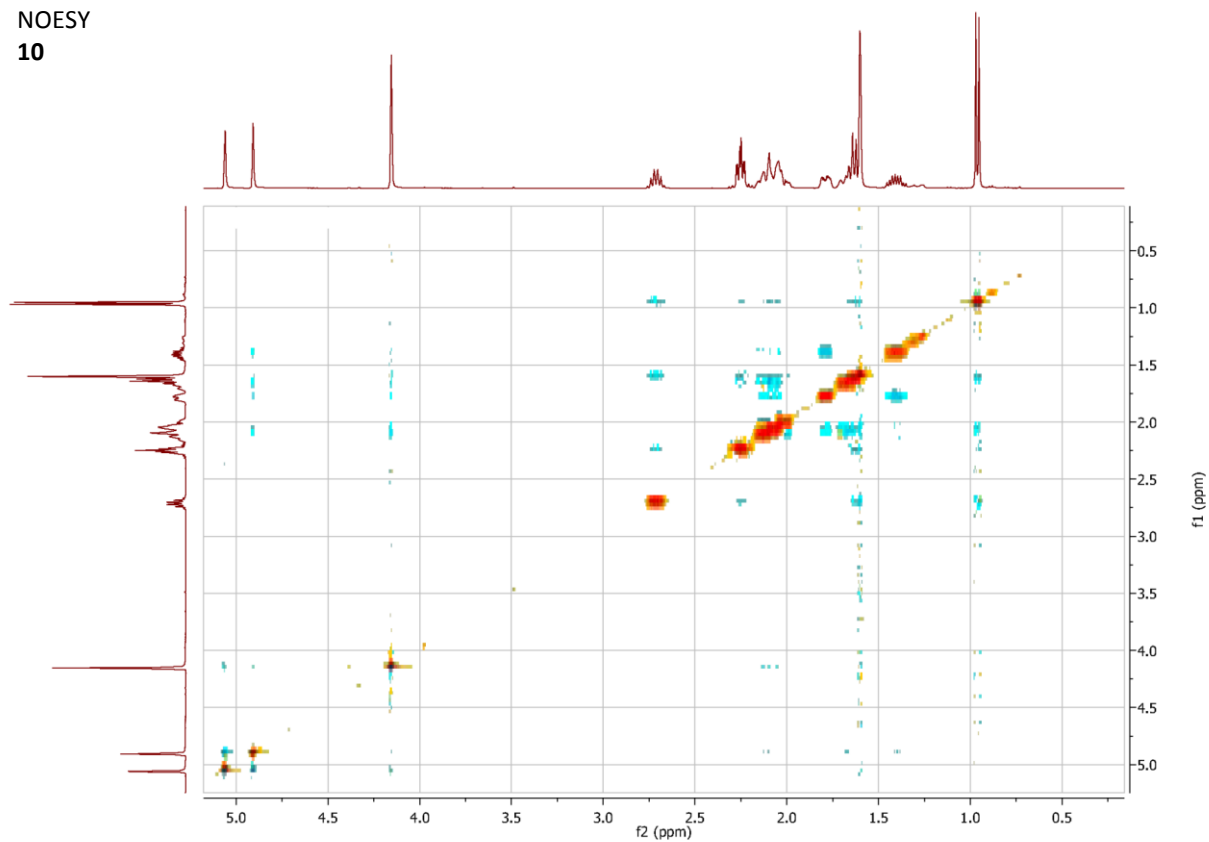


Fig. S84. NOESY spectrum of the new compound **10**.

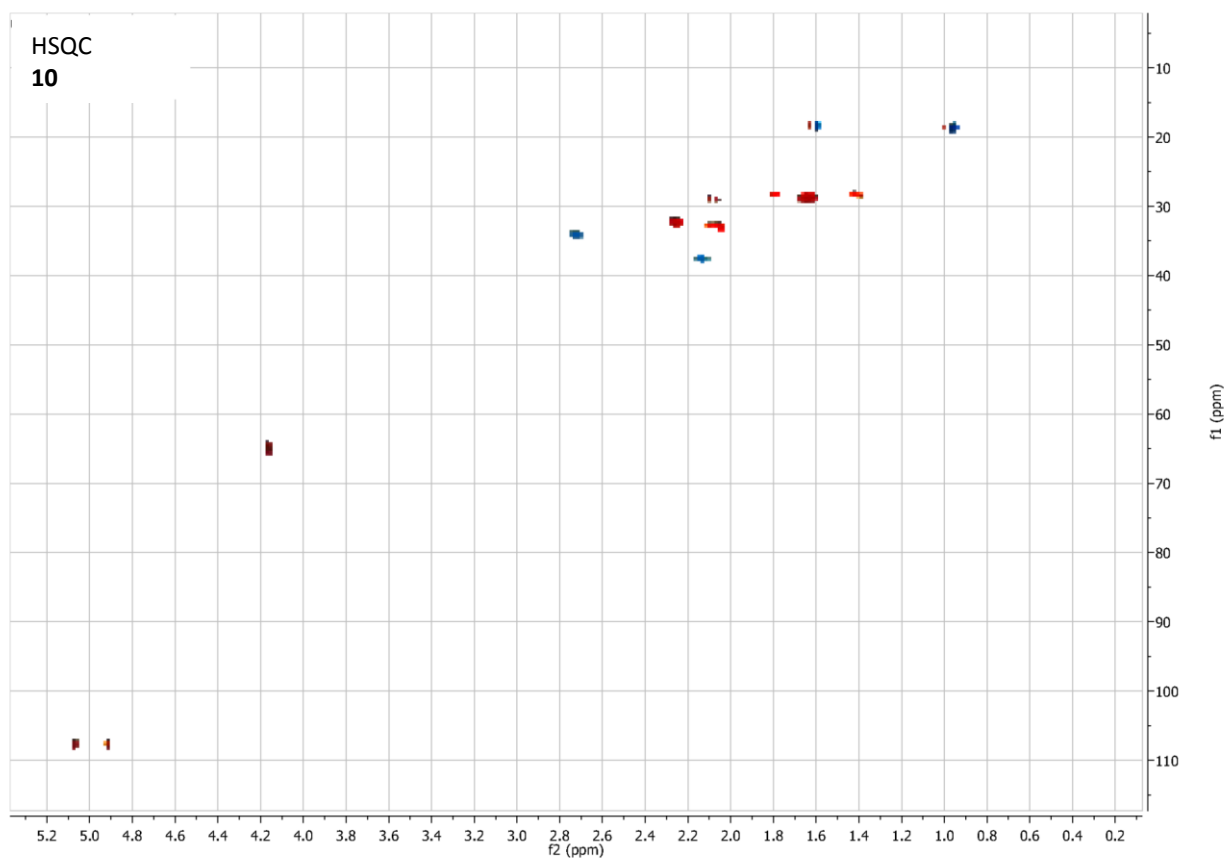


Fig. S85. HSQC spectrum of the new compound **10**.

HMBC
10

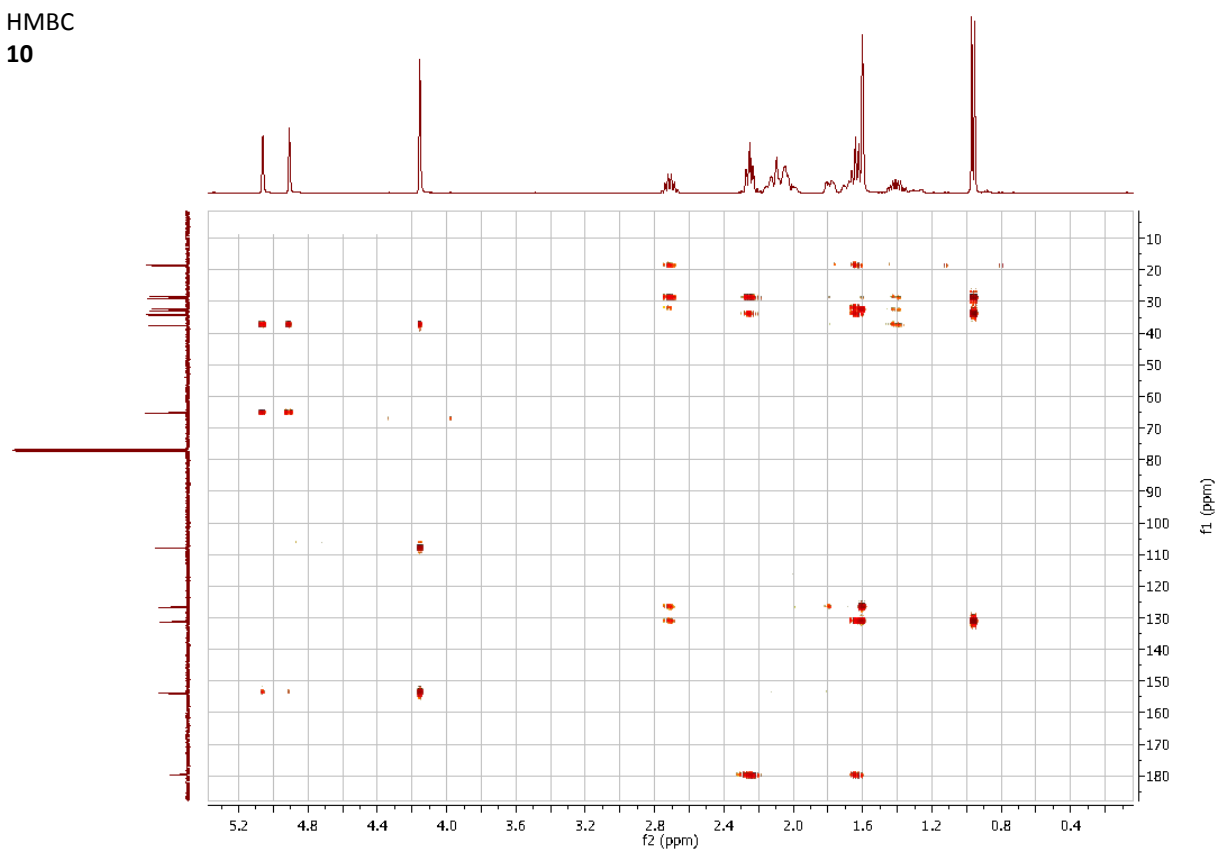


Fig. S86. HMBC spectrum of the new compound 10.