

## **Supplementary Materials**

# ***In Vitro Anticancer and Cancer-Preventive Activity of New Triterpene Glycosides from the Far Eastern Starfish *Solaster pacificus****

**Timofey V. Malyarenko<sup>1,2\*</sup>, Olesya S. Malyarenko<sup>1</sup>, Alla A. Kicha<sup>1</sup>, Anatoly I. Kalinovsky<sup>1</sup>, Pavel S. Dmitrenok<sup>1</sup>, and Natalia V. Ivanchina<sup>1\*</sup>**

<sup>1</sup> G.B. Elyakov Pacific Institute of Bioorganic Chemistry, Far Eastern Branch of the Russian Academy of Sciences, Pr. 100-let Vladivostoku 159, 690022 Vladivostok, Russia; malyarenko.os@gmail.com (O.S.M.); kicha@piboc.dvo.ru (A.A.K.); kaaniw@piboc.dvo.ru (A.I.K.); paveldmt@piboc.dvo.ru (P.S.D.)

<sup>2</sup> Department of Bioorganic Chemistry and Biotechnology, School of Natural Sciences, Far Eastern Federal University, Russky Island, Ajax Bay, 10, 690922, Vladivostok, Russia

\* Correspondence: malyarenko-tv@mail.ru (T.V.M.); ivanchina@piboc.dvo.ru (N.V.I.). Tel.: +7-423-2312-360; Fax: +7-423-2314-050

## List

**Figure S1.** HRESIMS spectrum of pacificusoside D (**1**).

**Figure S2.** IR spectrum of pacificusoside D (**1**) in KBr.

**Figure S3.**  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S4.** Expansion №1 of  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S5.** Expansion №2 of  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S6.** Expansion №3 of  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S7.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S8.** Expansion №1 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S9.** Expansion №2 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S10.** Expansion №3 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S11.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S12.** HSQC spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S13.** HMBC spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S14.** ROESY spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S15.** UV spectrum of pacificusoside D (**1**) in MeOH.

**Figure S16.** HRESIMS spectrum of pacificusoside E (**2**).

**Figure S17.** IR spectrum of pacificusoside E (**2**) in KBr.

**Figure S18.**  $^1\text{H}$ -NMR spectrum of pacificusoside E (**2**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S19.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside E (**2**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S20.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside E (**2**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S21.** HSQC spectrum of pacificusoside E (**2**) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S22.** HMBC spectrum of pacificusoside E (**2**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S23.** ROESY spectrum of pacificusoside E (**2**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S24.** HRESIMS spectrum of pacificusoside F (**3**).

**Figure S25.** IR spectrum of pacificusoside F (**3**) in KBr.

**Figure S26.** <sup>1</sup>H-NMR spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S27.** <sup>13</sup>C-NMR spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S28.** <sup>1</sup>H-<sup>1</sup>H COSY spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S29.** HSQC spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S30.** HMBC spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S31.** ROESY spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S32.** HRESIMS spectrum of pacificusoside G (**5**).

**Figure S33.** IR spectrum of pacificusoside G (**5**) in KBr.

**Figure S34.** <sup>1</sup>H-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S35.** Expansion №1 of <sup>1</sup>H-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S36.** Expansion №2 of <sup>1</sup>H-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S37.** Expansion №3 of <sup>1</sup>H-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S38.** <sup>13</sup>C-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S39.** Expansion №1 of <sup>13</sup>C-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S40.** Expansion №2 of <sup>13</sup>C-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S41.** Expansion №3 of <sup>13</sup>C-NMR spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S42.** UV spectrum of pacificusoside G (**5**) in MeOH.

**Figure S43.** <sup>1</sup>H-<sup>1</sup>H COSY spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S44.** HSQC spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S45.** HMBC spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S46.** ROESY spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S47.** HRESIMS spectrum of pacificusoside H (**6**).

**Figure S48.** IR spectrum of pacificusoside H (**6**) in KBr.

**Figure S49.** <sup>1</sup>H-NMR spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S50.** <sup>13</sup>C-NMR spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S51.** <sup>1</sup>H-<sup>1</sup>H COSY spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S52.** HSQC spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S53.** HMBC spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S54.** ROESY spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S55.** HRESIMS spectrum of pacificusoside I (**7**).

**Figure S56.** IR spectrum of pacificusoside I (**7**) in KBr.

**Figure S57.** <sup>1</sup>H-NMR spectrum of pacificusoside I (**7**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S58.** <sup>13</sup>C-NMR spectrum of pacificusoside I (**7**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S59.** <sup>1</sup>H-<sup>1</sup>H COSY spectrum of pacificusoside I (**7**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S60.** HSQC spectrum of pacificusoside I (**7**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S61.** HMBC spectrum of pacificusoside I (**7**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S62.** ROESY spectrum of pacificusoside I (**7**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S63.** HRESIMS spectrum of pacificusoside J (**8**).

**Figure S64.** IR spectrum of pacificusoside J (**8**) in KBr.

**Figure S65.** <sup>1</sup>H-NMR spectrum of pacificusoside J (**8**) in C<sub>5</sub>D<sub>5</sub>N.

**Figure S66.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S67.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S68.** HSQC spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S69.** HMBC spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S70.** ROESY spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S71.** HRESIMS spectrum of pacificusoside K (9).

**Figure S72.** IR spectrum of pacificusoside K (9) in KBr.

**Figure S73.**  $^1\text{H}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S74.** Expansion №1 of  $^1\text{H}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S75.** Expansion №2 of  $^1\text{H}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S76.** Expansion №3 of  $^1\text{H}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S77.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S78.** Expansion №1 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S79.** Expansion №2 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S80.** Expansion №3 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

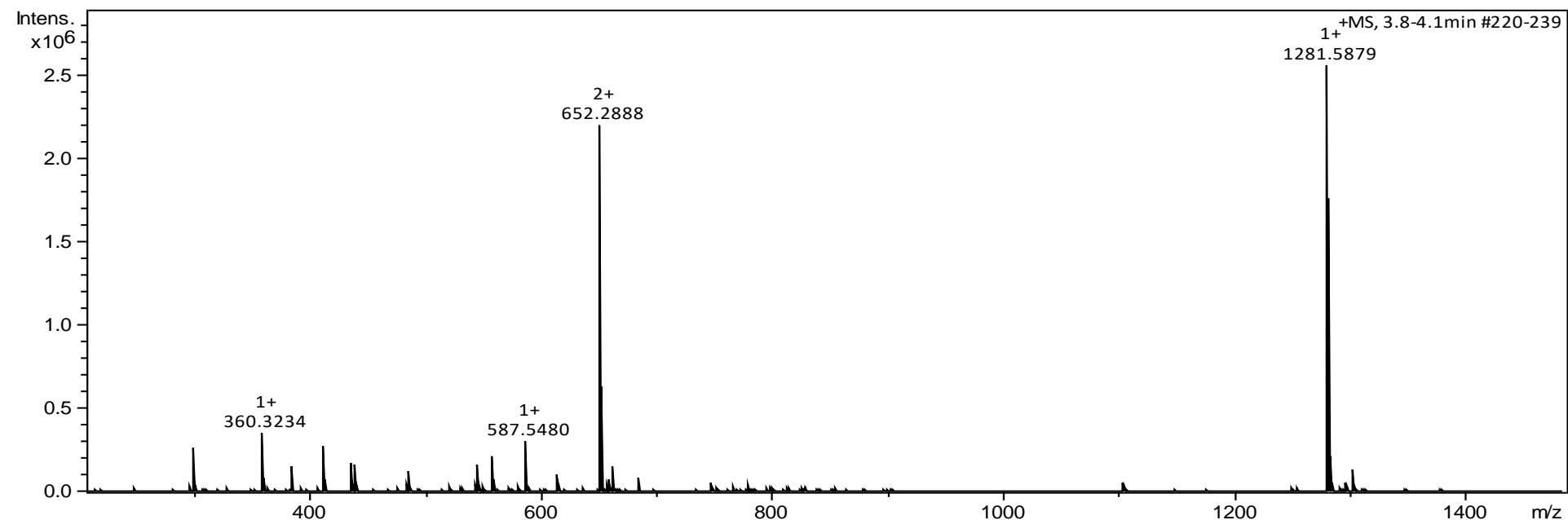
**Figure S81.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S82.** HSQC spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

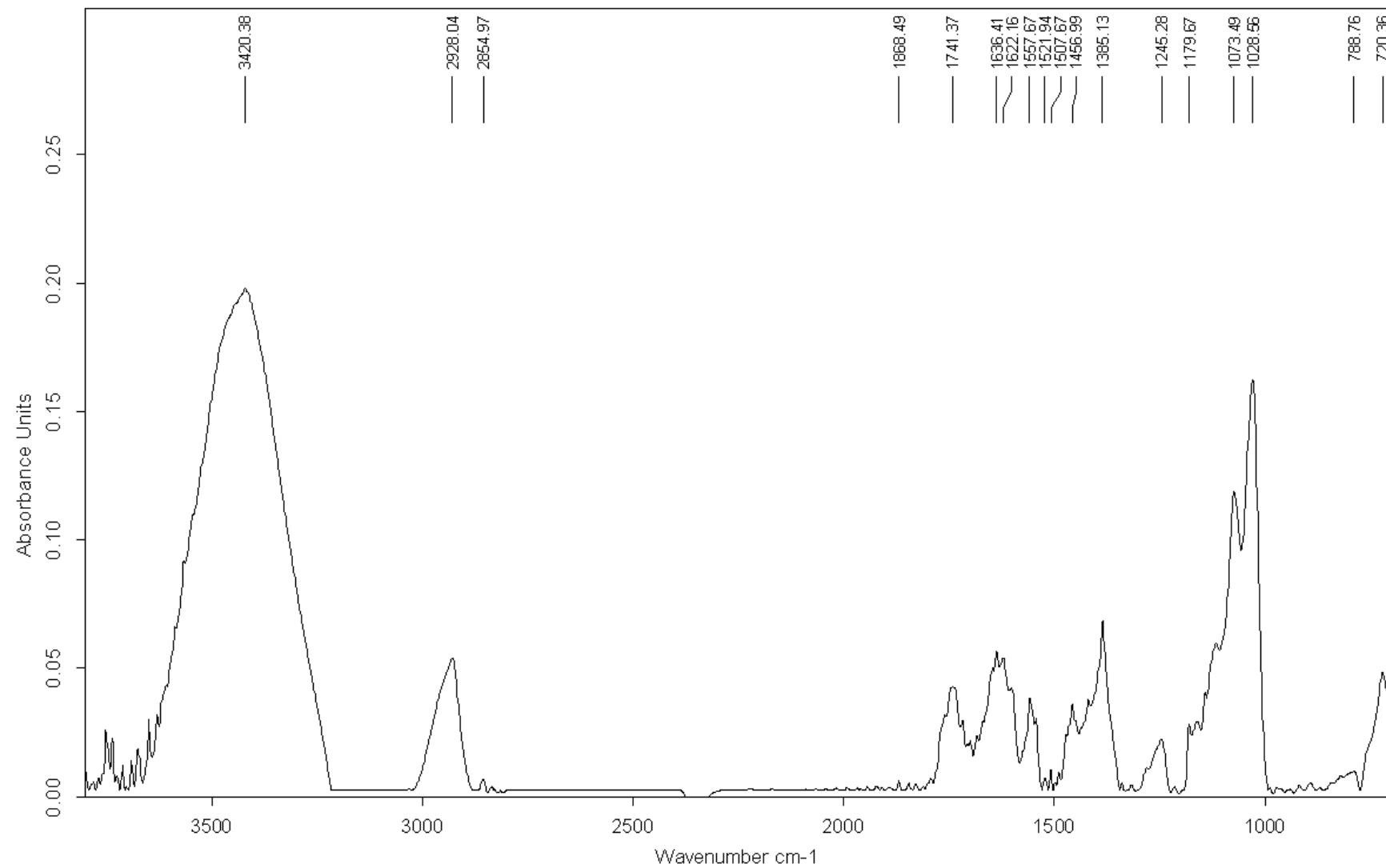
**Figure S83.** HMBC spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

**Figure S84.** ROESY spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .

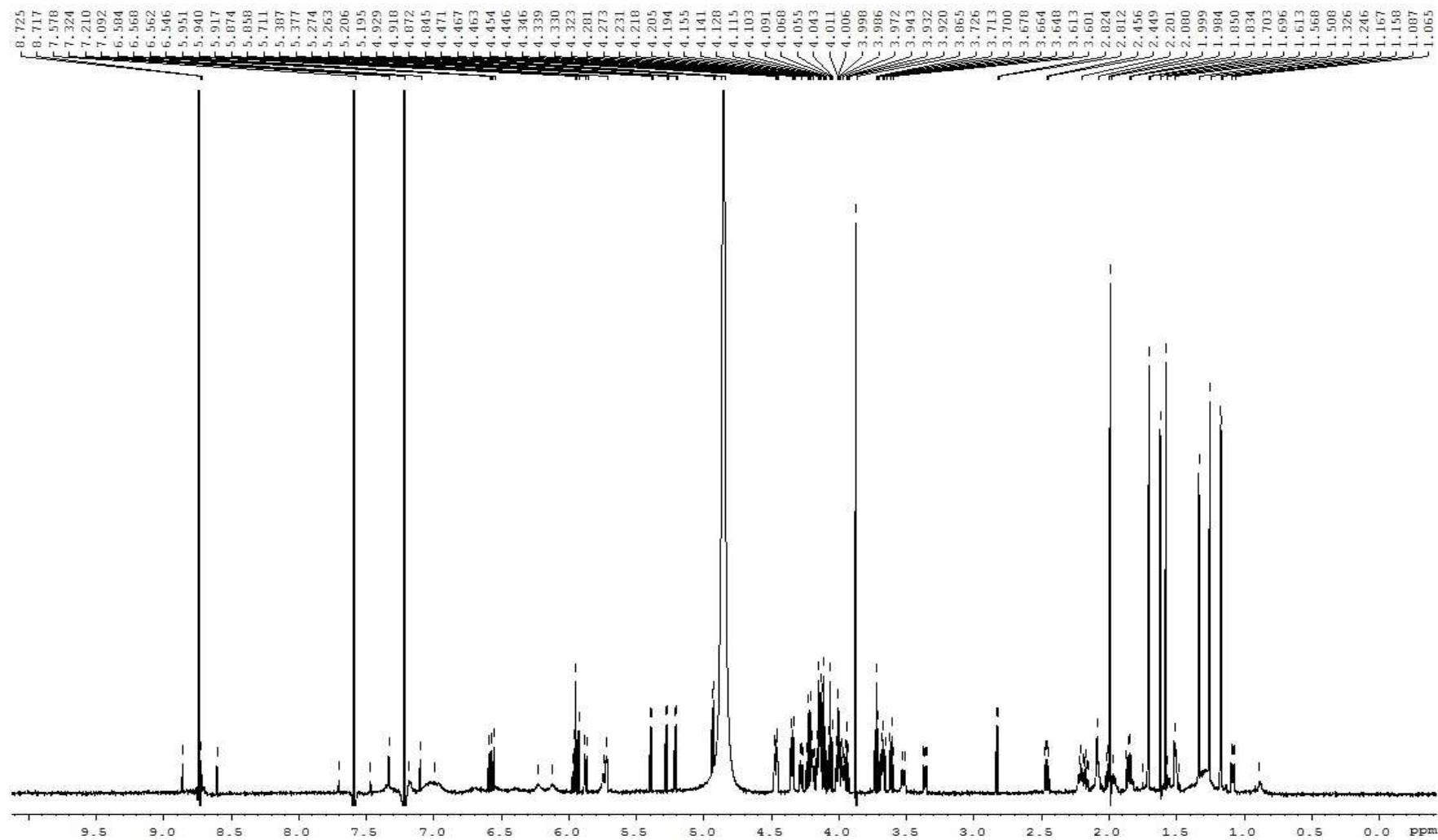
**Figure S1.** HRESIMS spectrum of pacificusoside D (**1**).



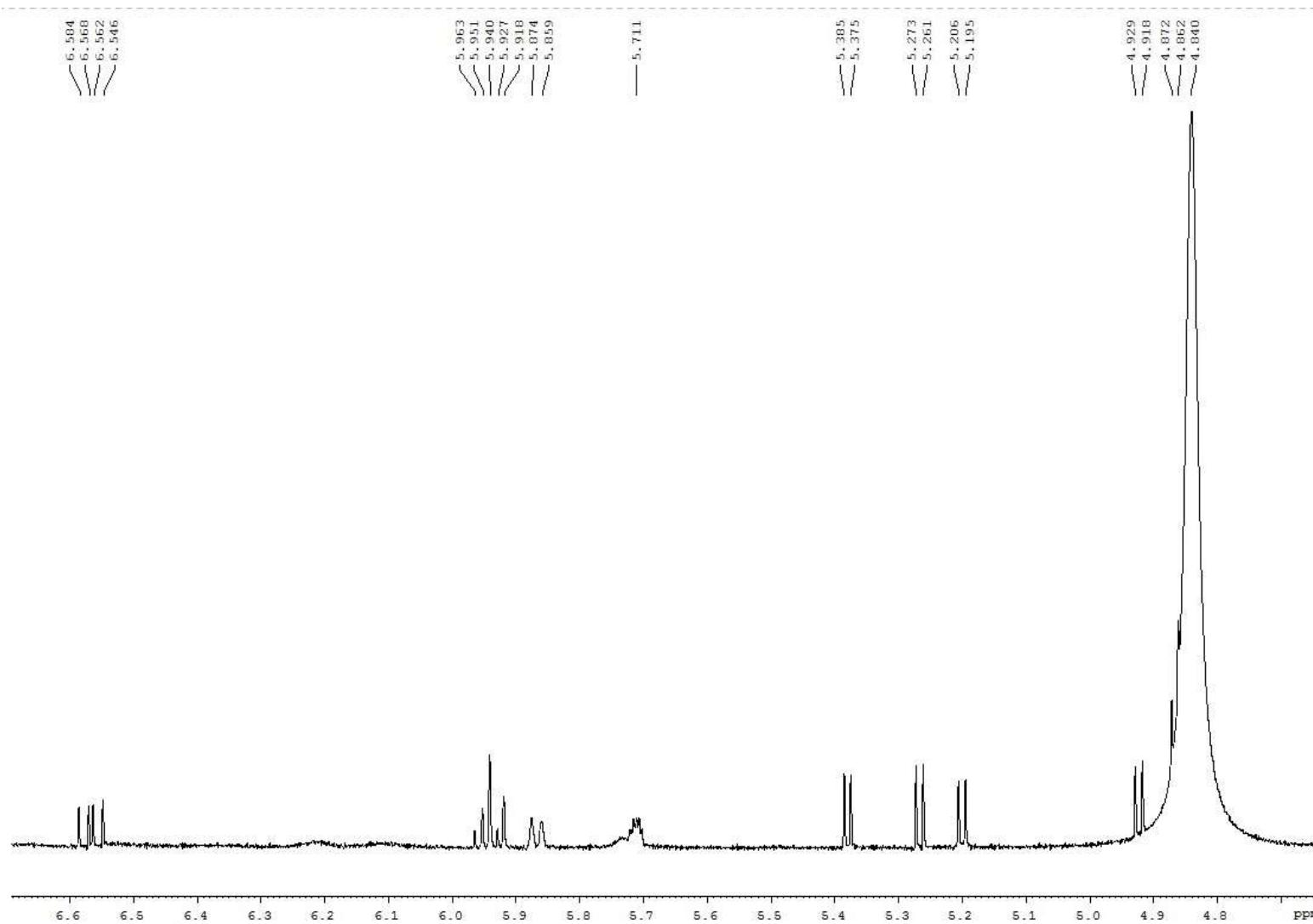
**Figure S2.** IR spectrum of pacificusoside D (**1**) in KBr.



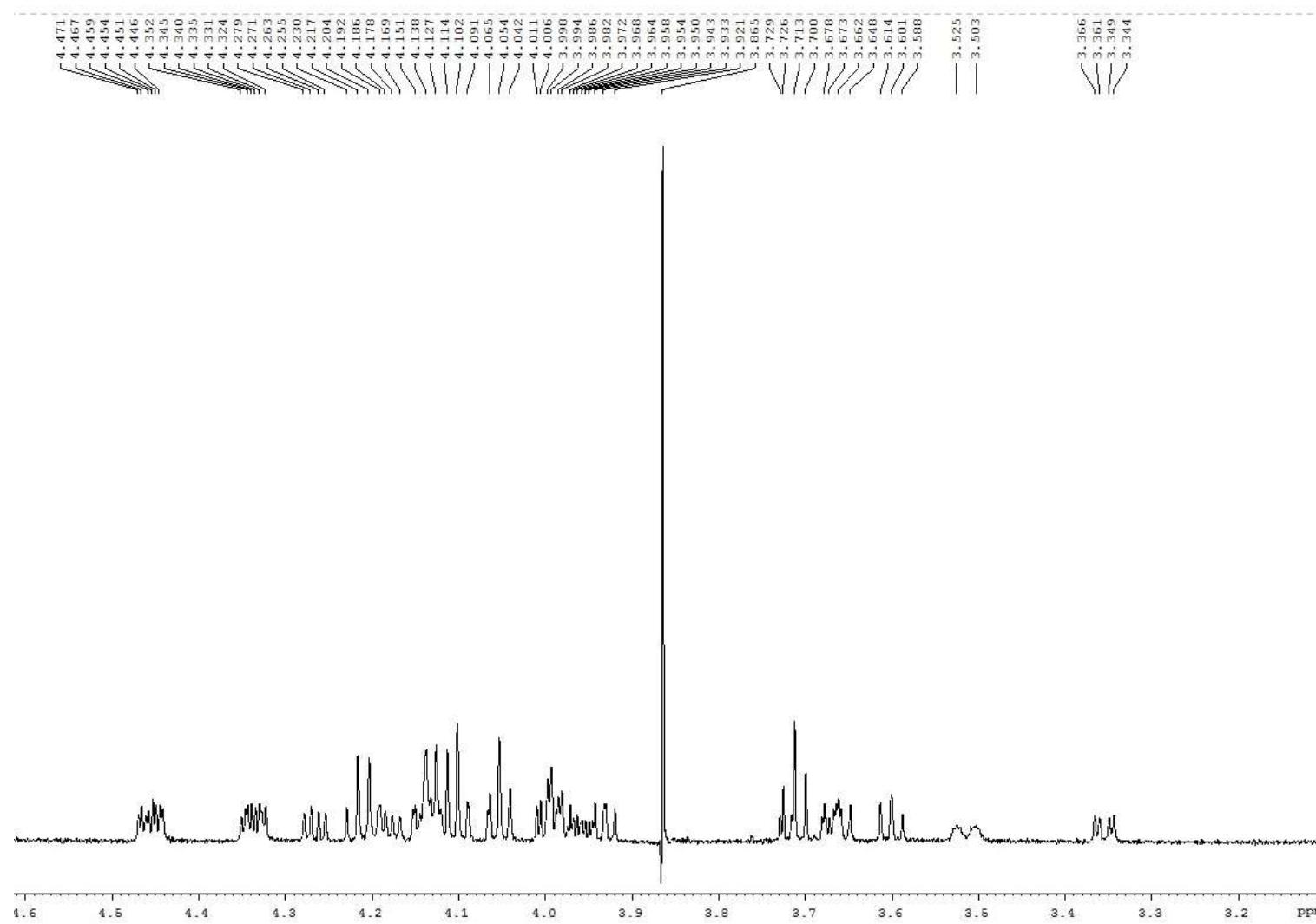
**Figure S3.**  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



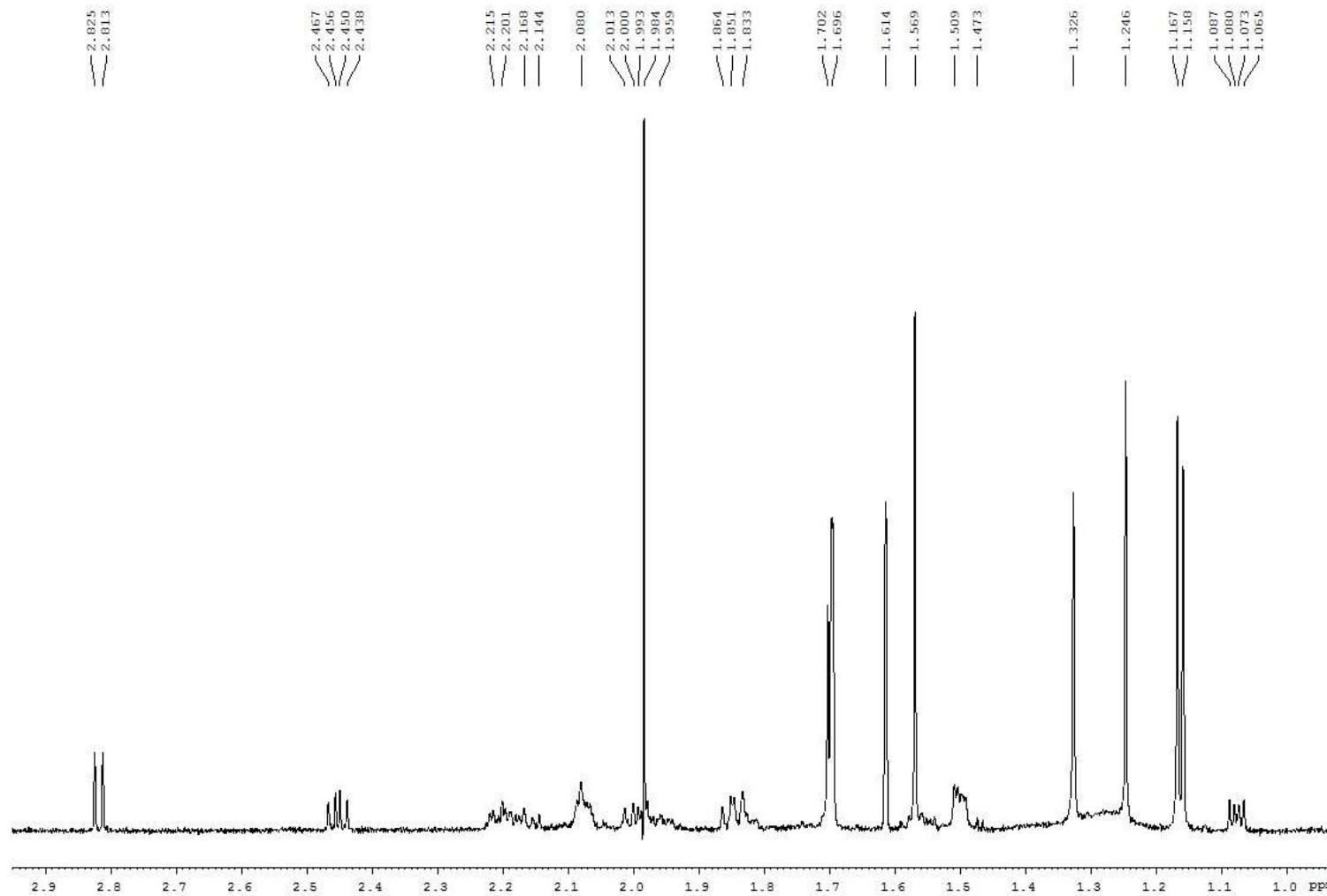
**Figure S4.** Expansion №1 of  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



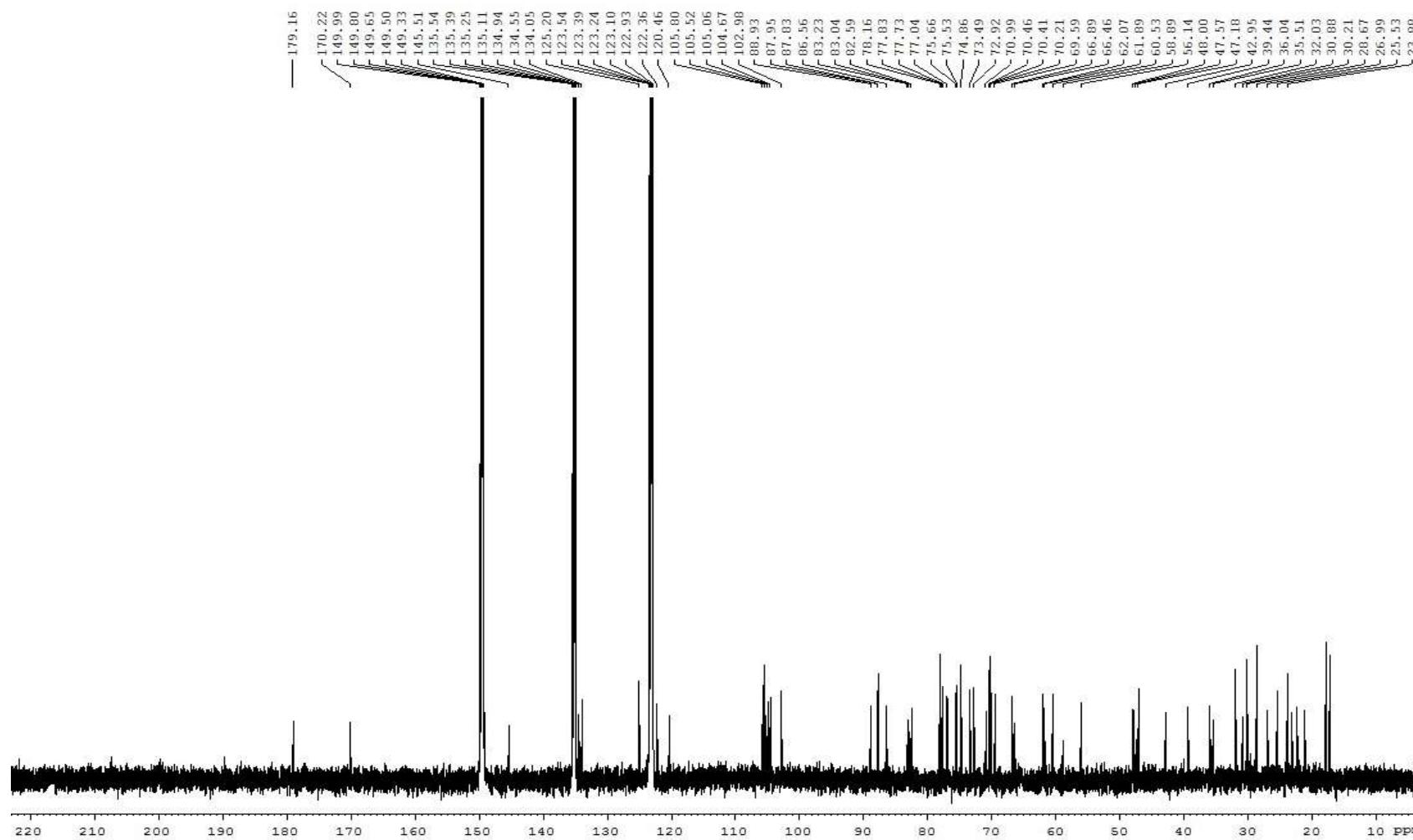
**Figure S5.** Expansion №2 of  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



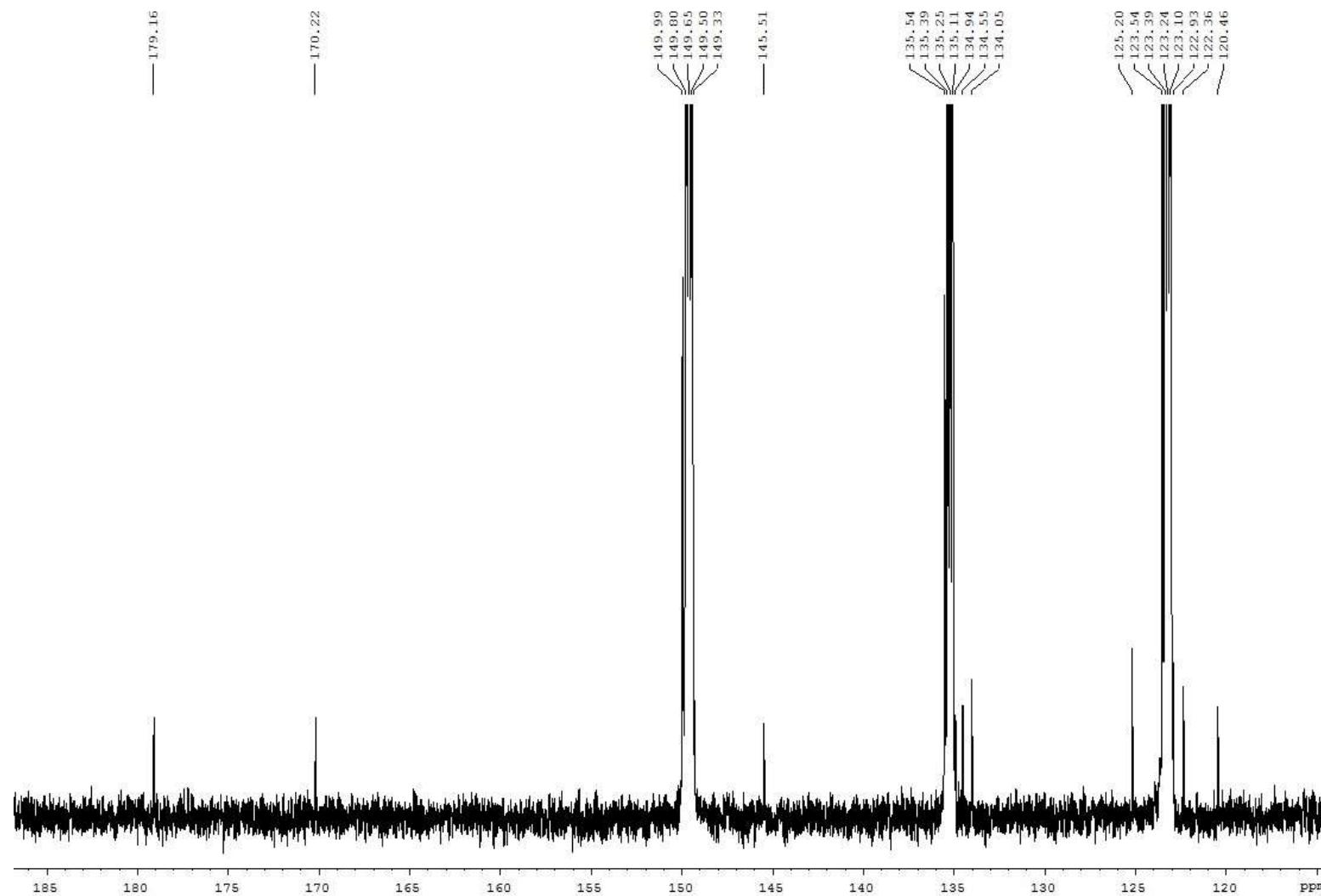
**Figure S6.** Expansion №3 of  $^1\text{H}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



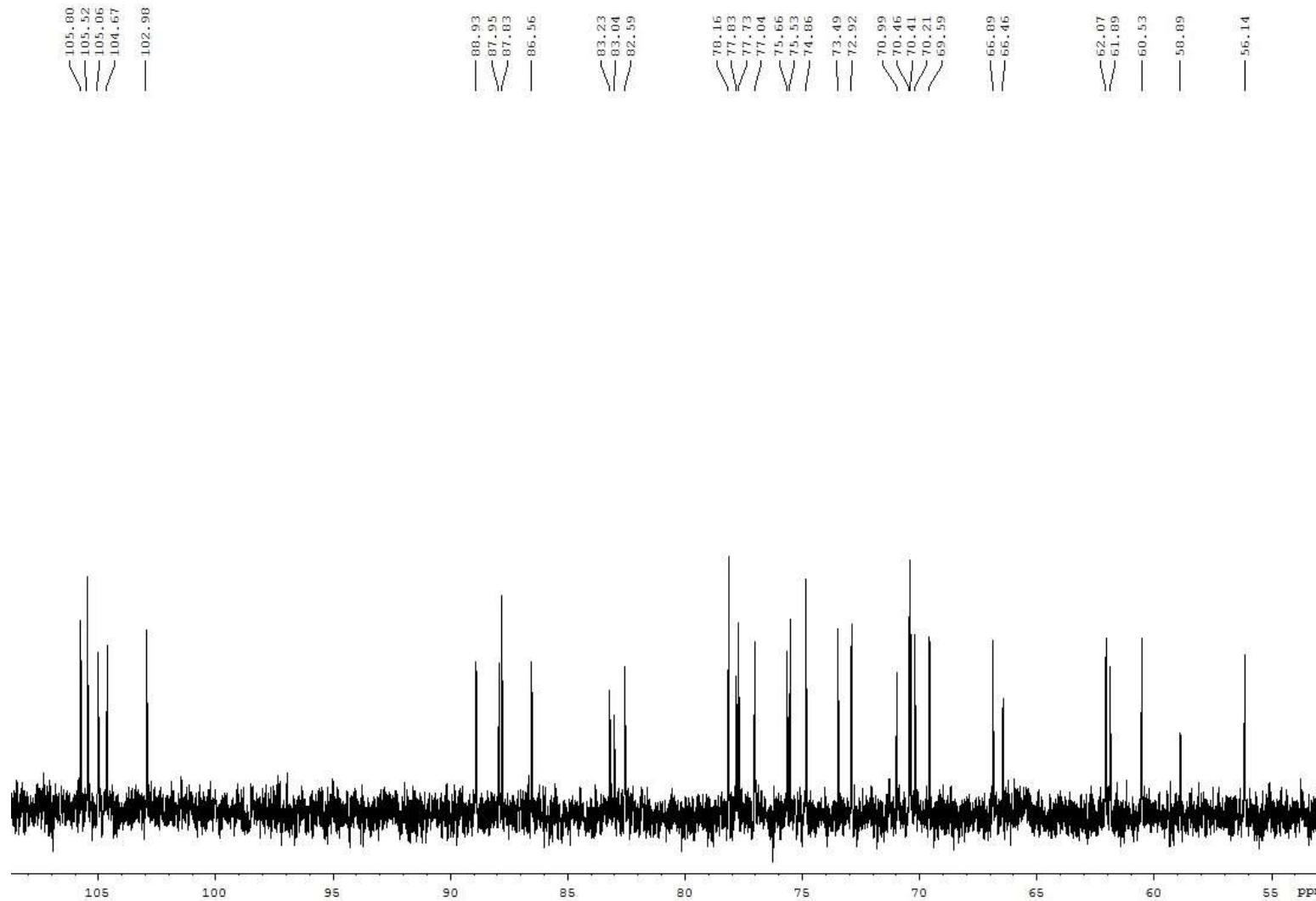
**Figure S7.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



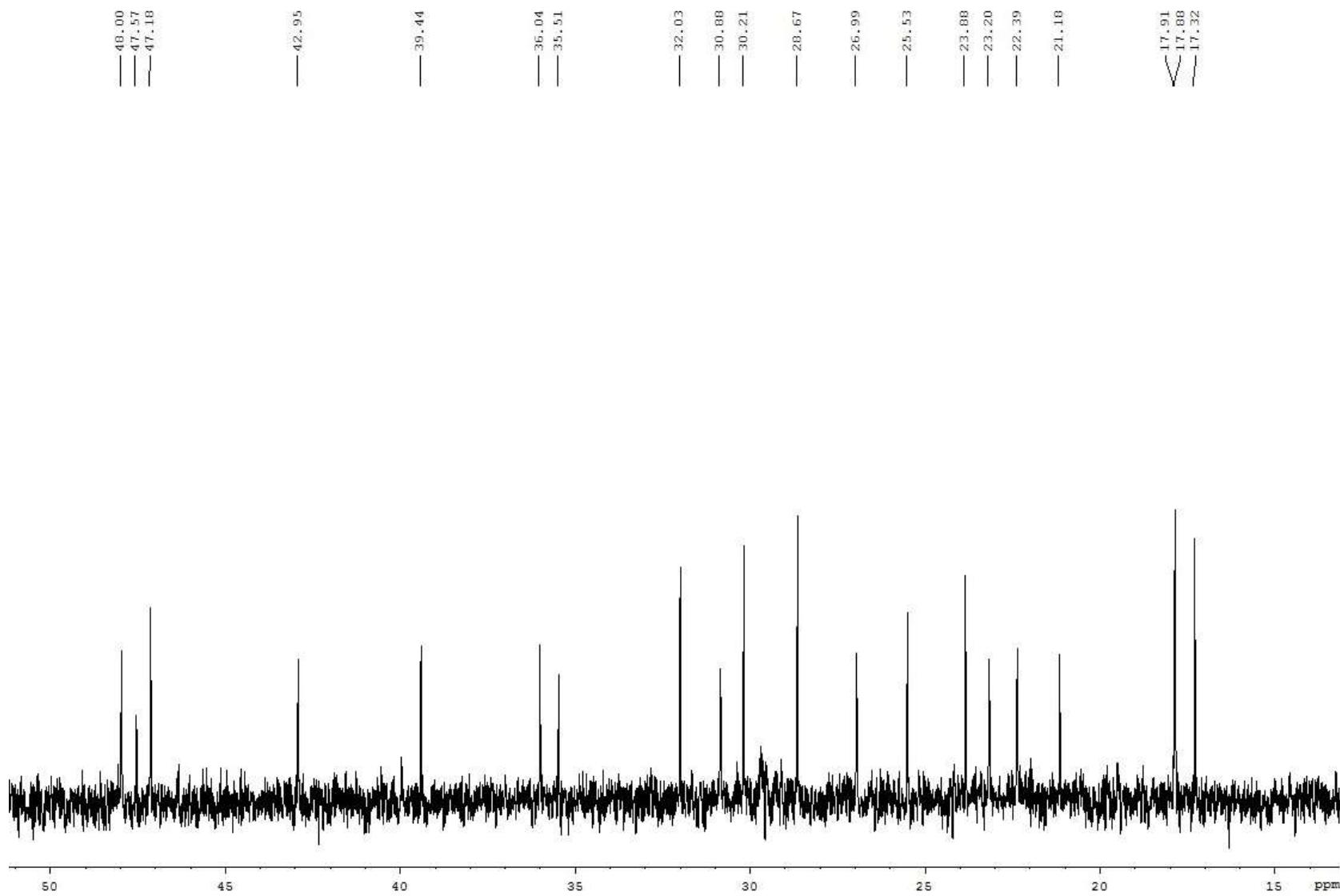
**Figure S8.** Expansion №1 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



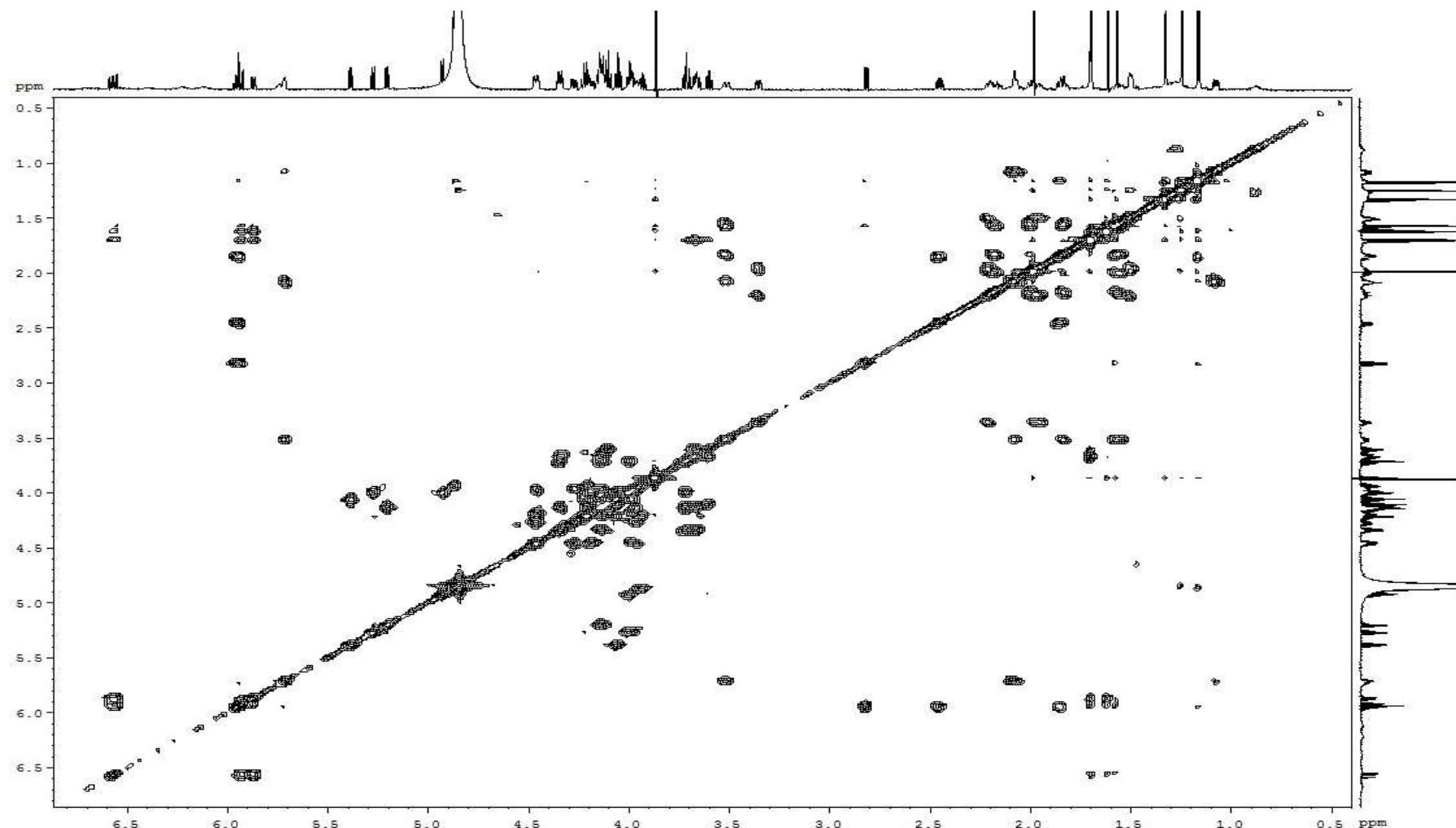
**Figure S9.** Expansion №2 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



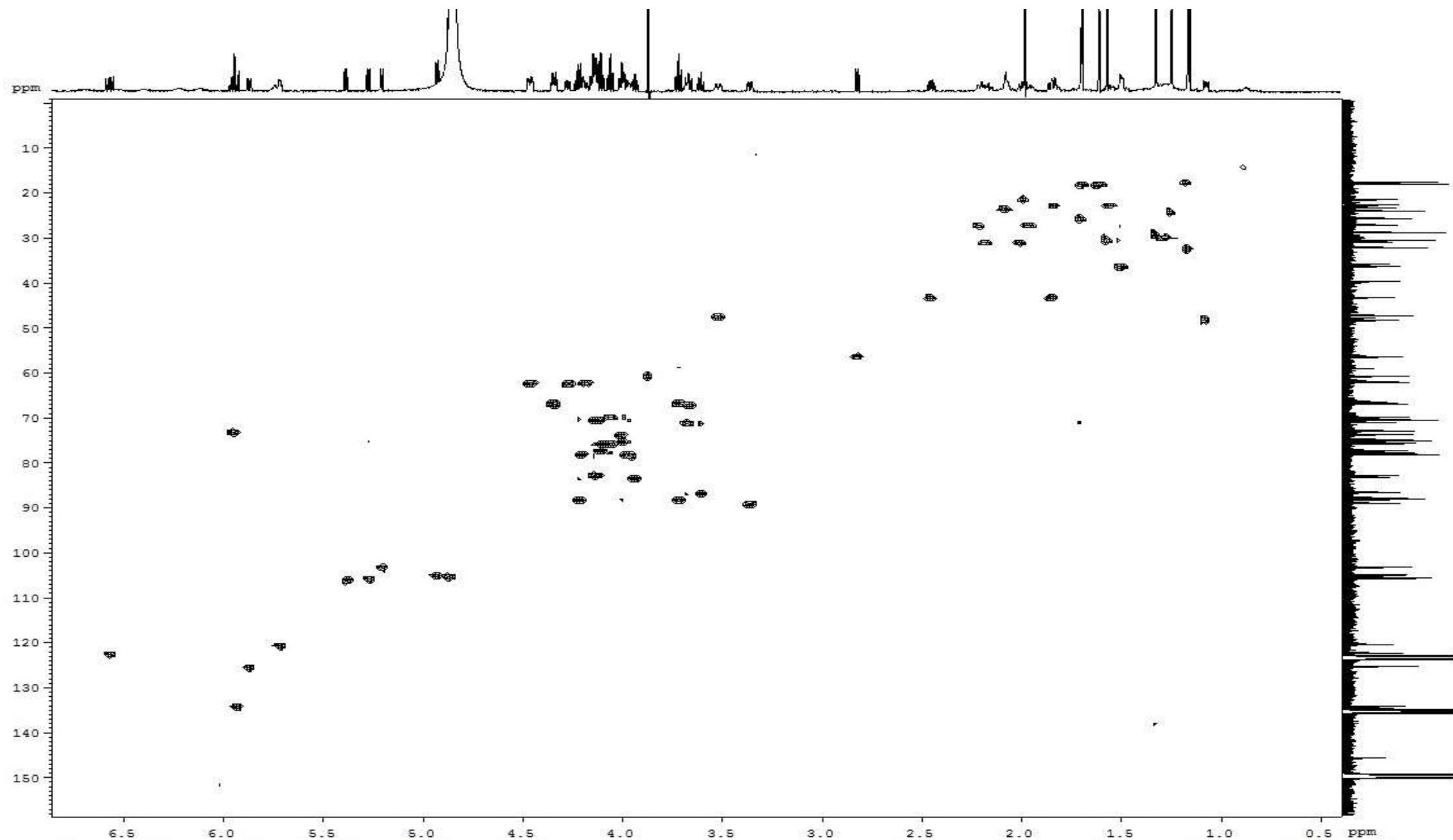
**Figure S10.** Expansion №3 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



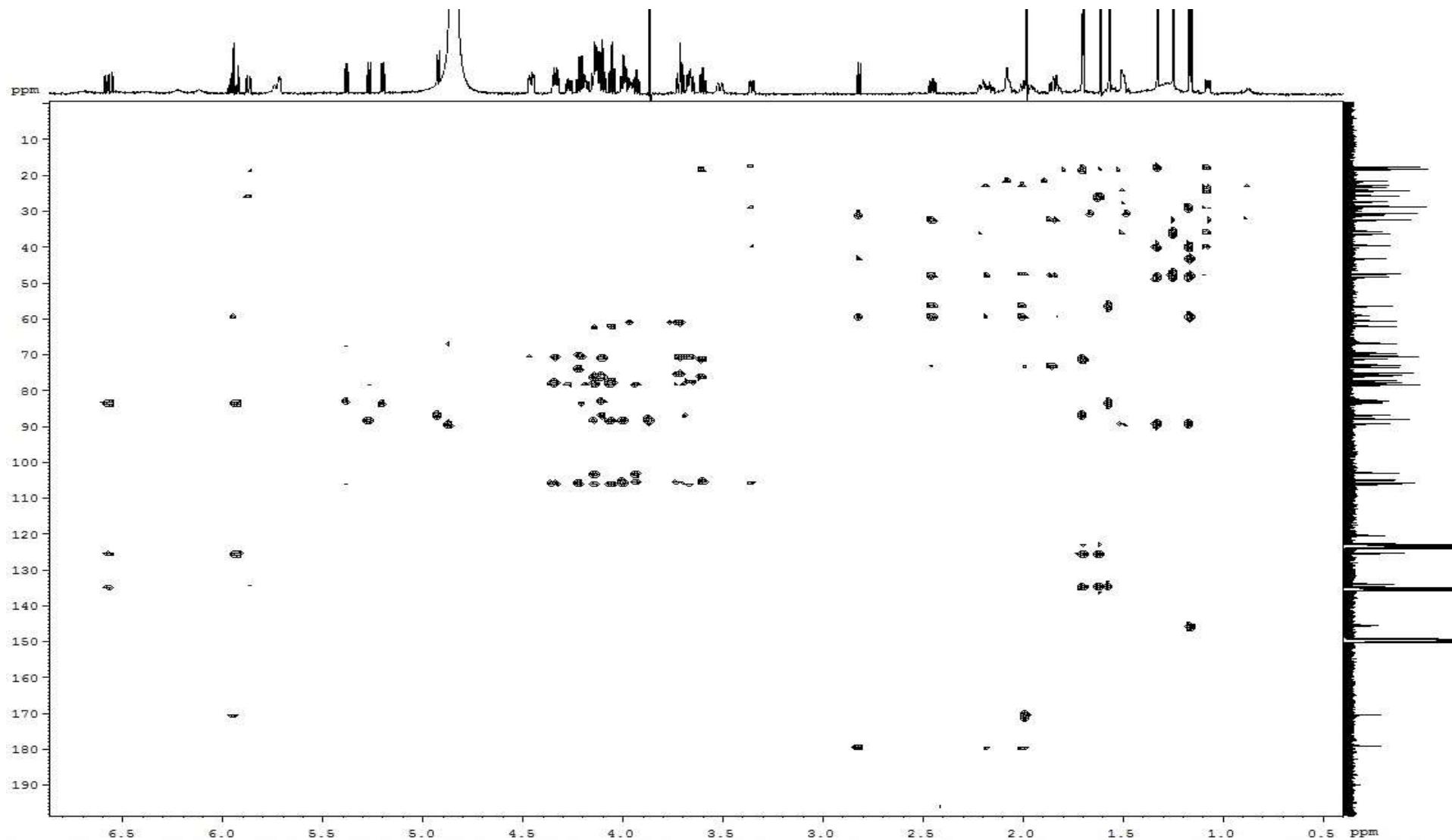
**Figure S11.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside D (**1**) in  $\text{C}_5\text{D}_5\text{N}$ .



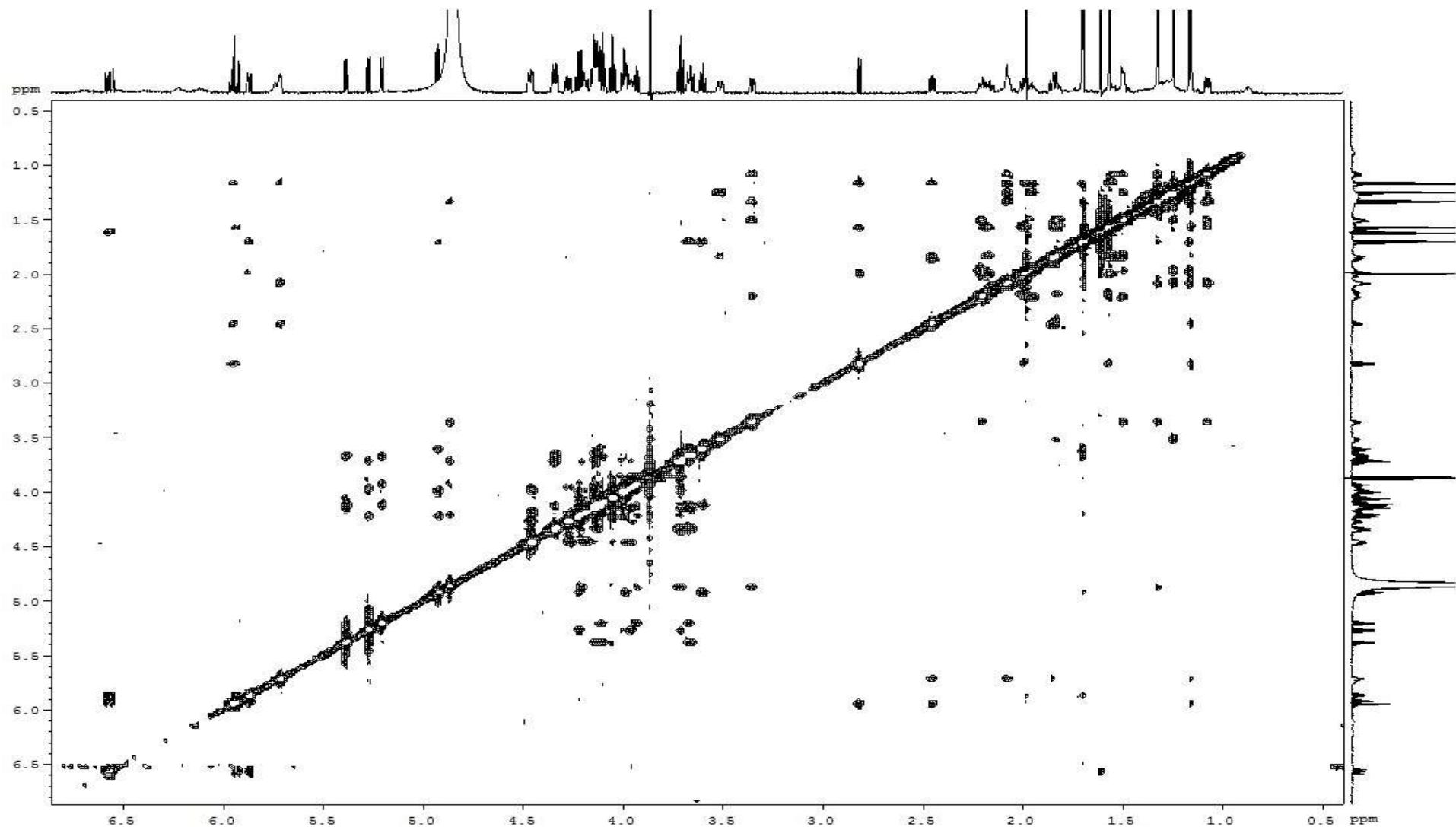
**Figure S12.** HSQC spectrum of pacificusoside D (**1**) in C<sub>5</sub>D<sub>5</sub>N.



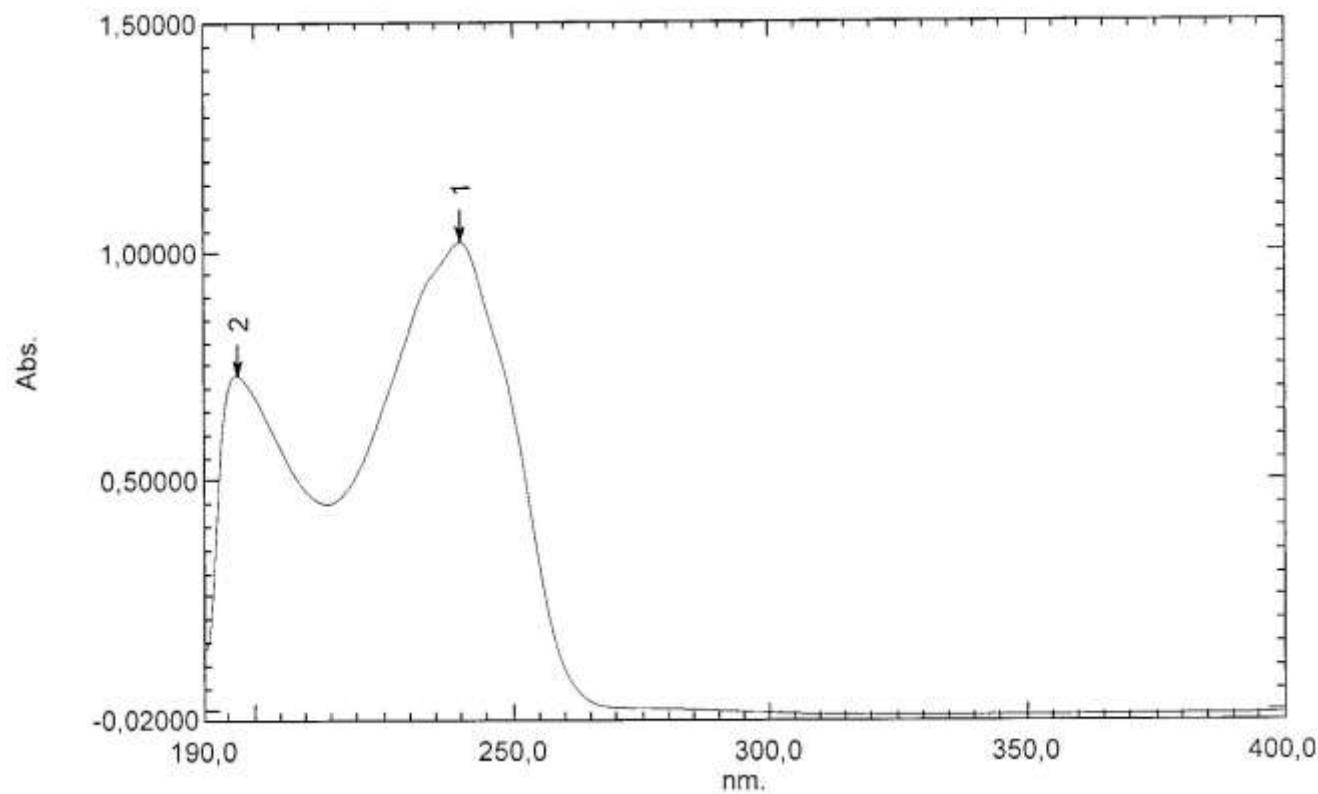
**Figure S13.** HMBC spectrum of pacificusoside D (**1**) in C<sub>5</sub>D<sub>5</sub>N.



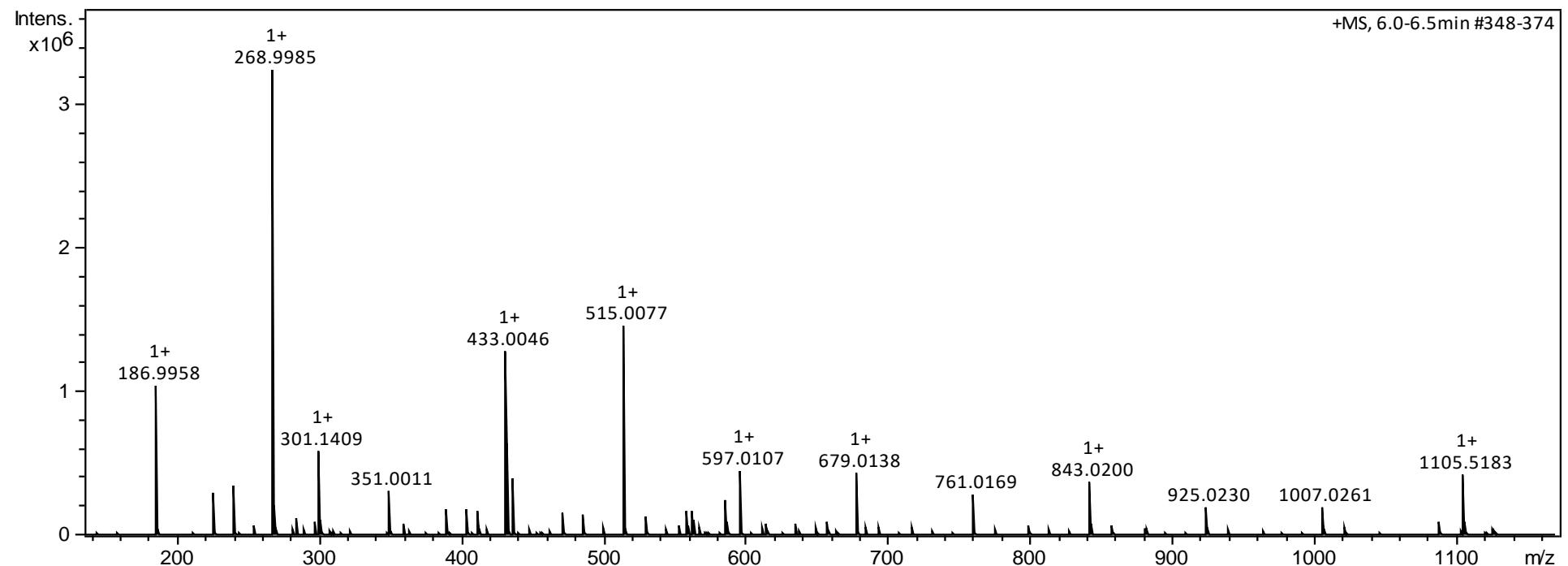
**Figure S14.** ROESY spectrum of pacificusoside D (**1**) in C<sub>5</sub>D<sub>5</sub>N.



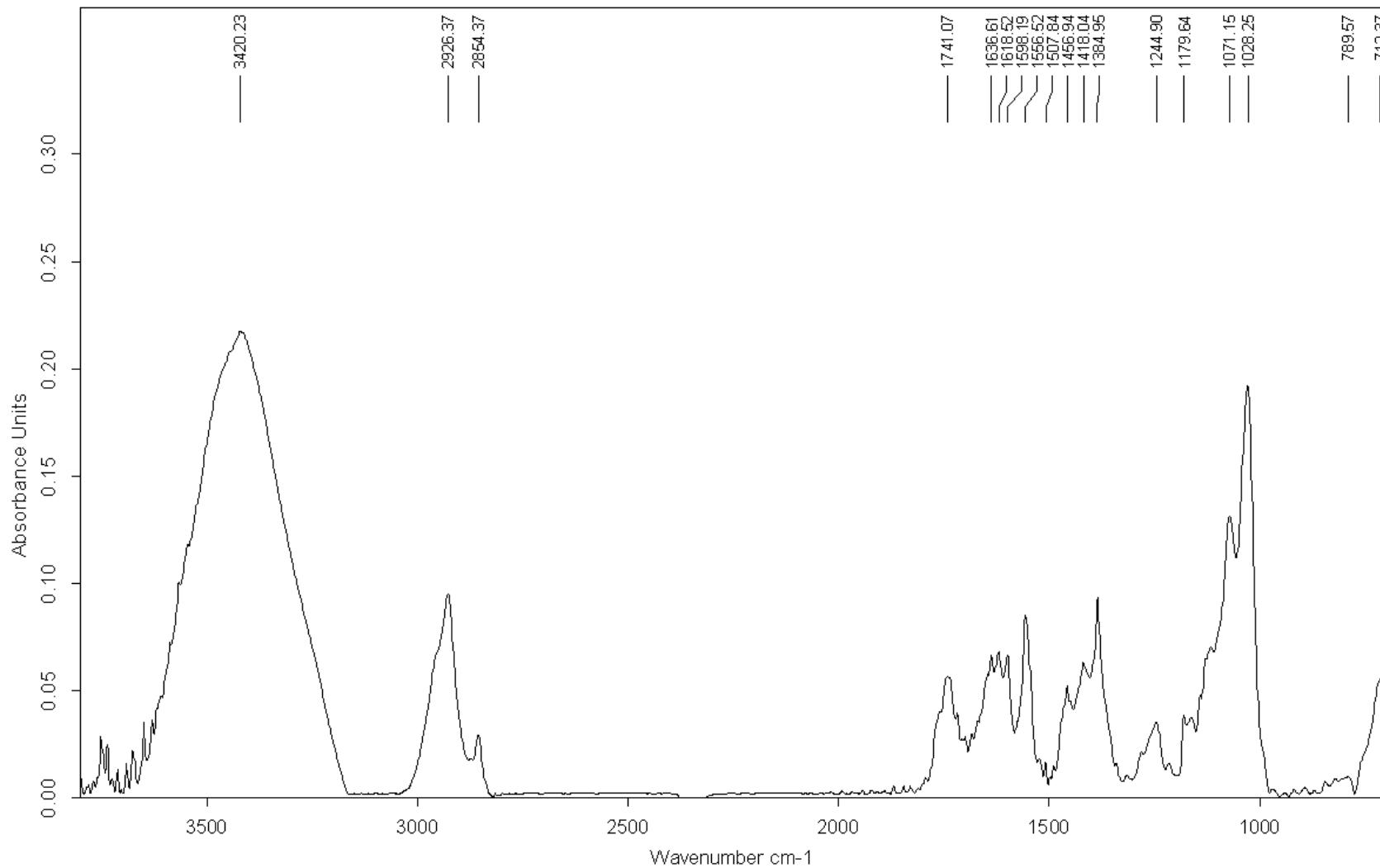
**Figure S15.** UV spectrum of pacificusoside D (**1**) in MeOH.



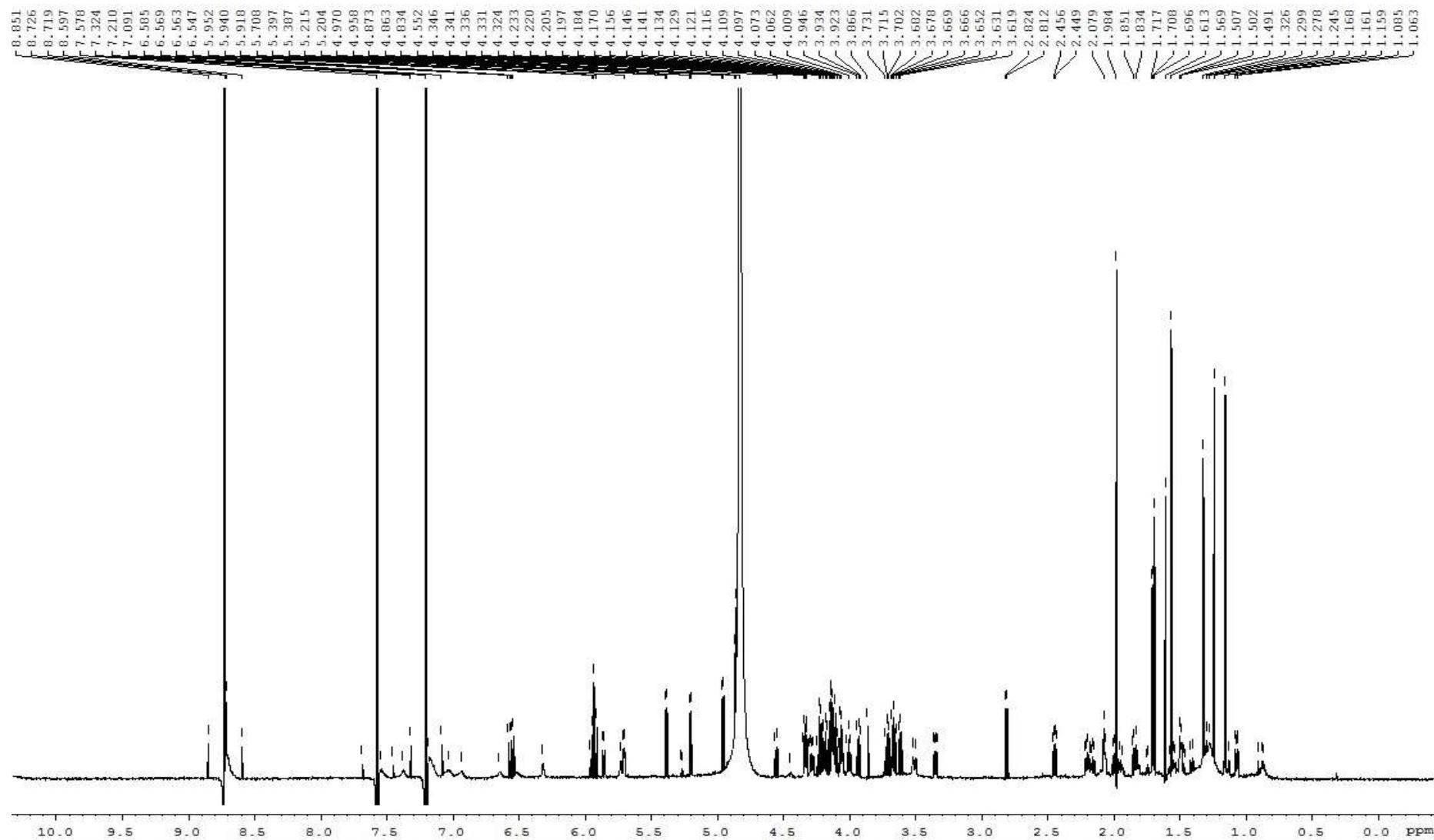
**Figure S16.** HRESIMS spectrum of pacificusoside E (**2**).



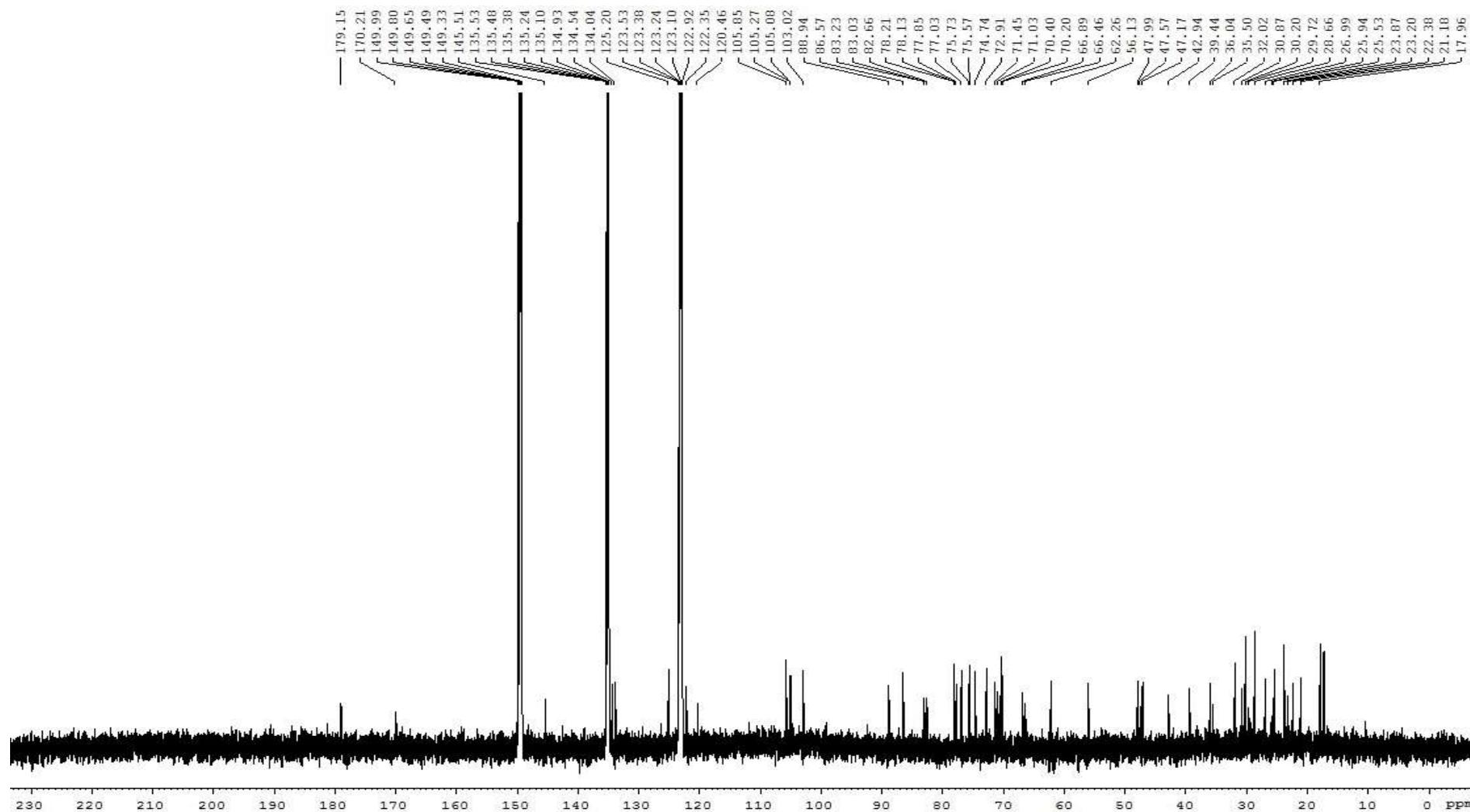
**Figure S17.** IR spectrum of pacificusoside E (**2**) in KBr.



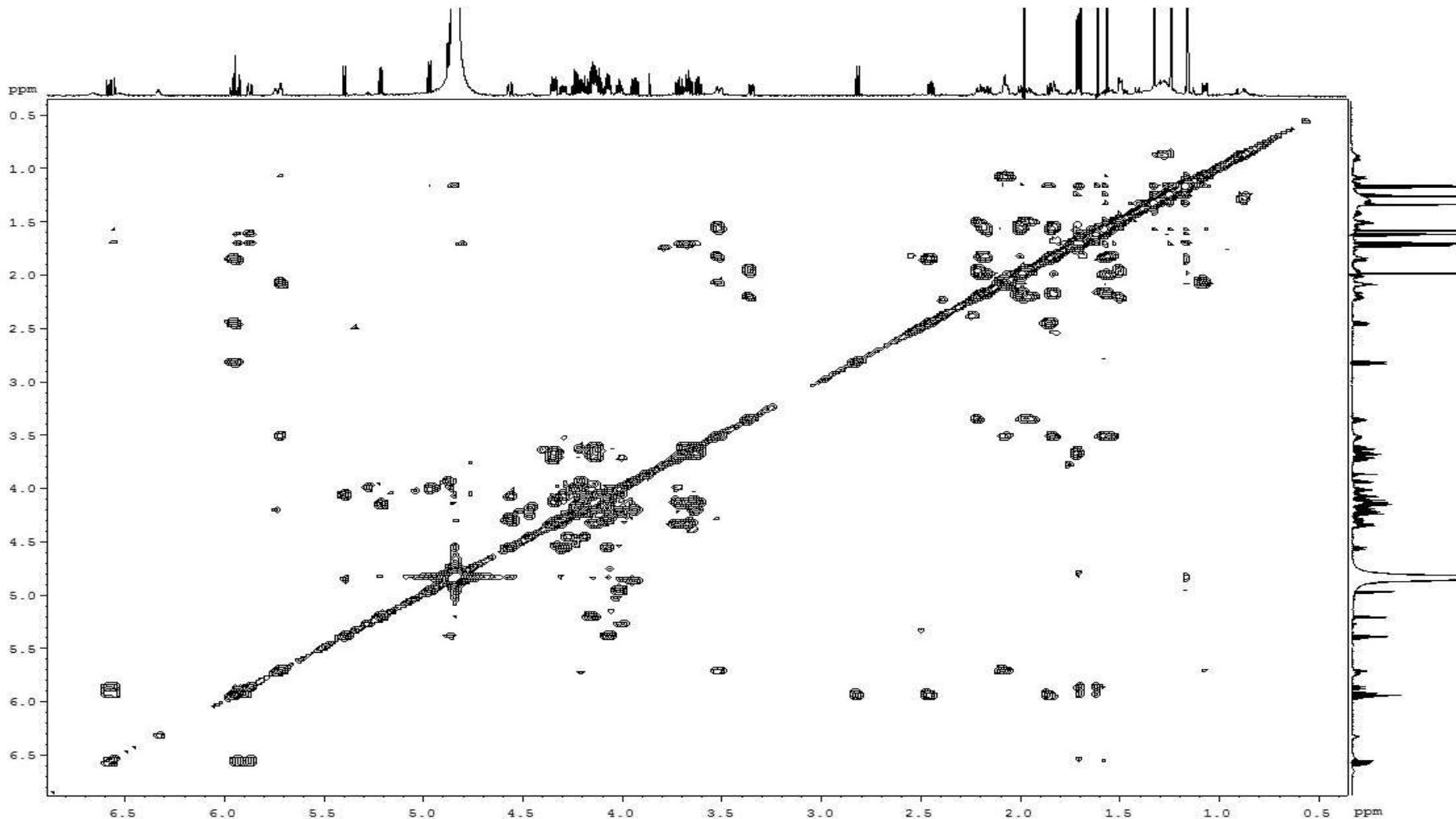
**Figure S18.**  $^1\text{H}$ -NMR spectrum of pacificusoside E (**2**) in  $\text{C}_5\text{D}_5\text{N}$ .



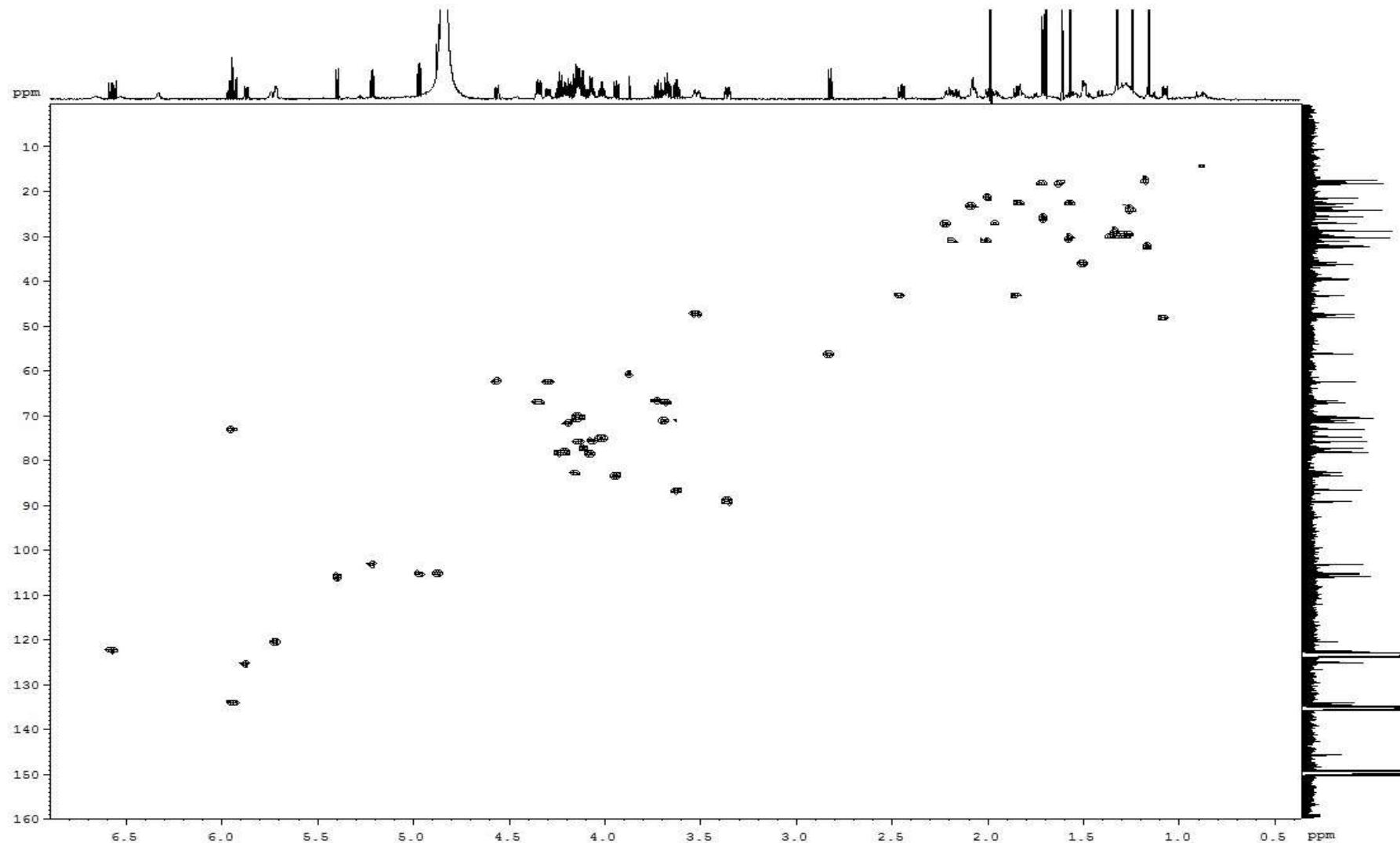
**Figure S19.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside E (2) in  $\text{C}_5\text{D}_5\text{N}$ .



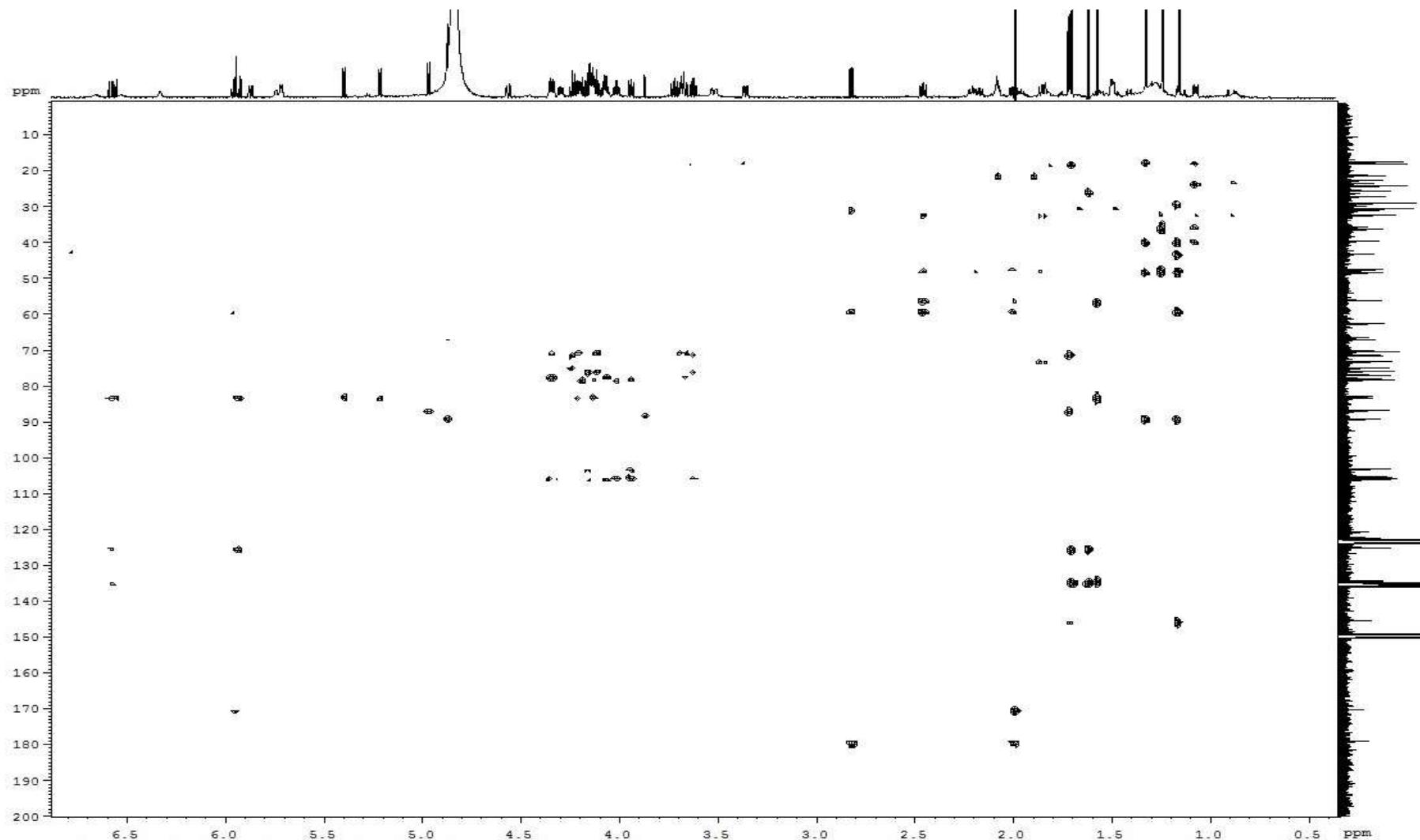
**Figure S20.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside E (2) in  $\text{C}_5\text{D}_5\text{N}$ .



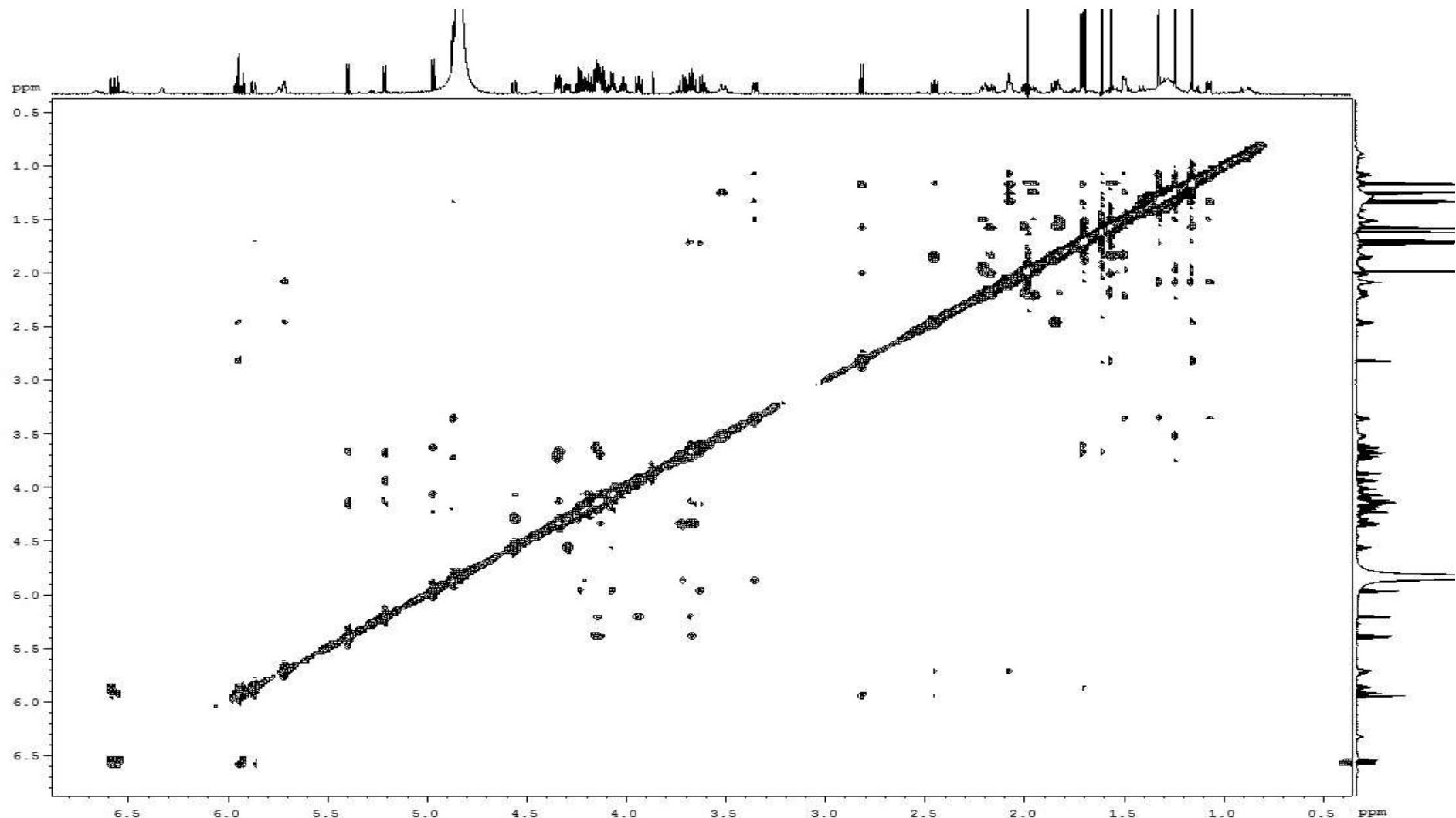
**Figure S21.** HSQC spectrum of pacificusoside E (**2**) in C<sub>5</sub>D<sub>5</sub>N.



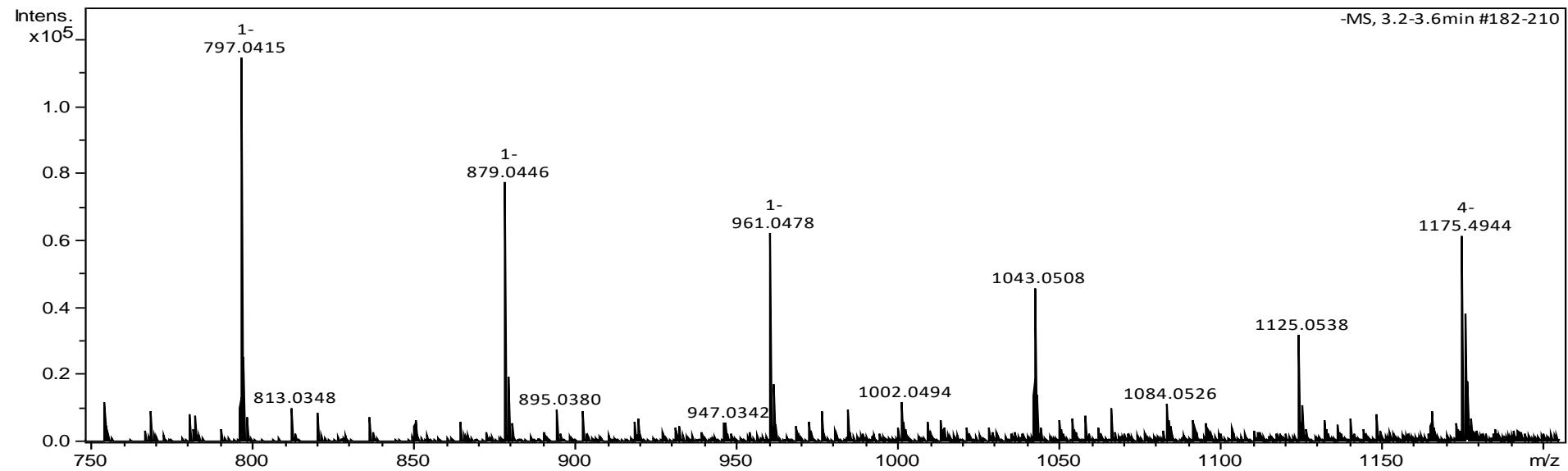
**Figure S22.** HMBC spectrum of pacificusoside E (**2**) in C<sub>5</sub>D<sub>5</sub>N.



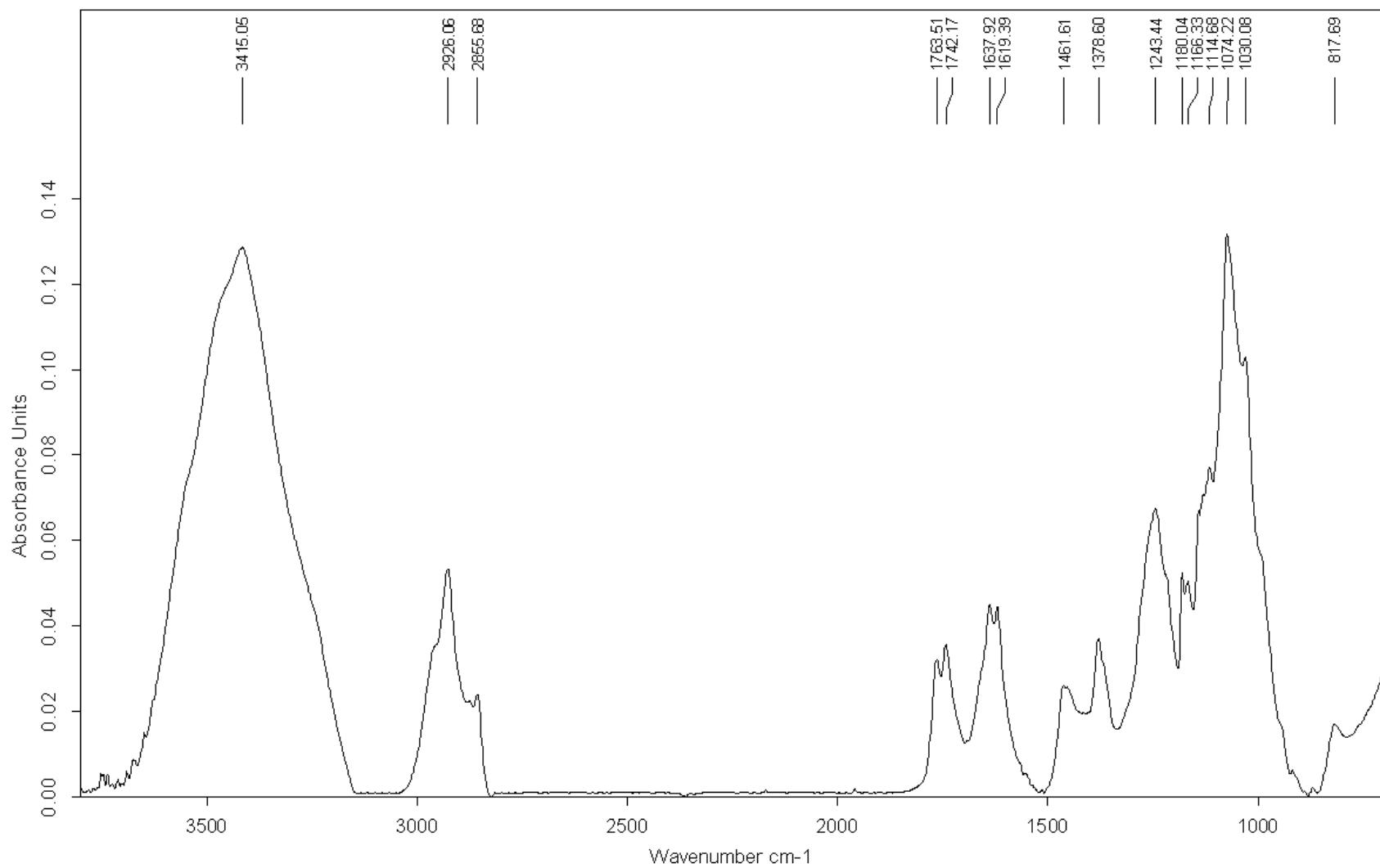
**Figure S23.** ROESY spectrum of pacificusoside E (**2**) in C<sub>5</sub>D<sub>5</sub>N.



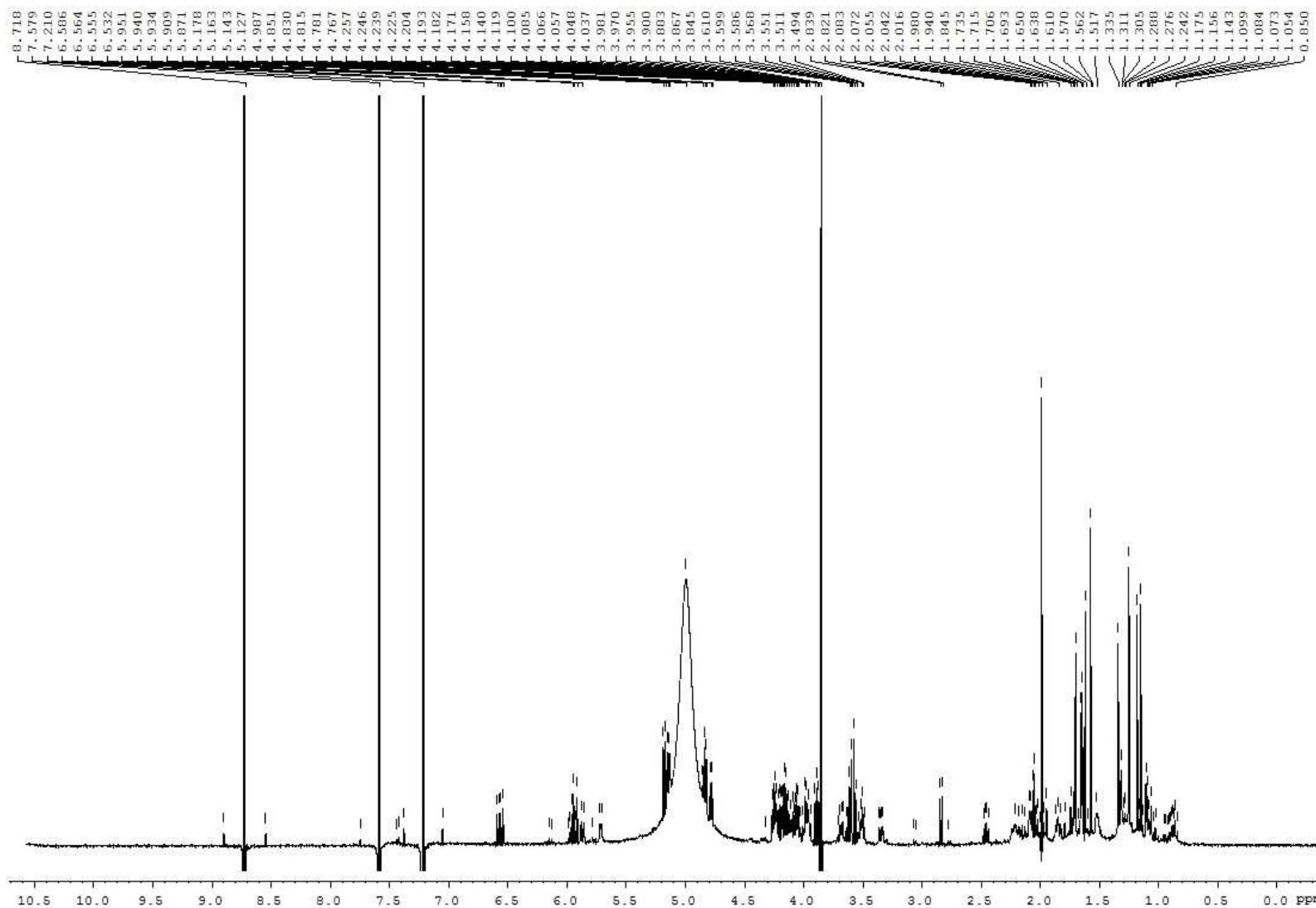
**Figure S24.** HRESIMS spectrum of pacificusoside F (3).



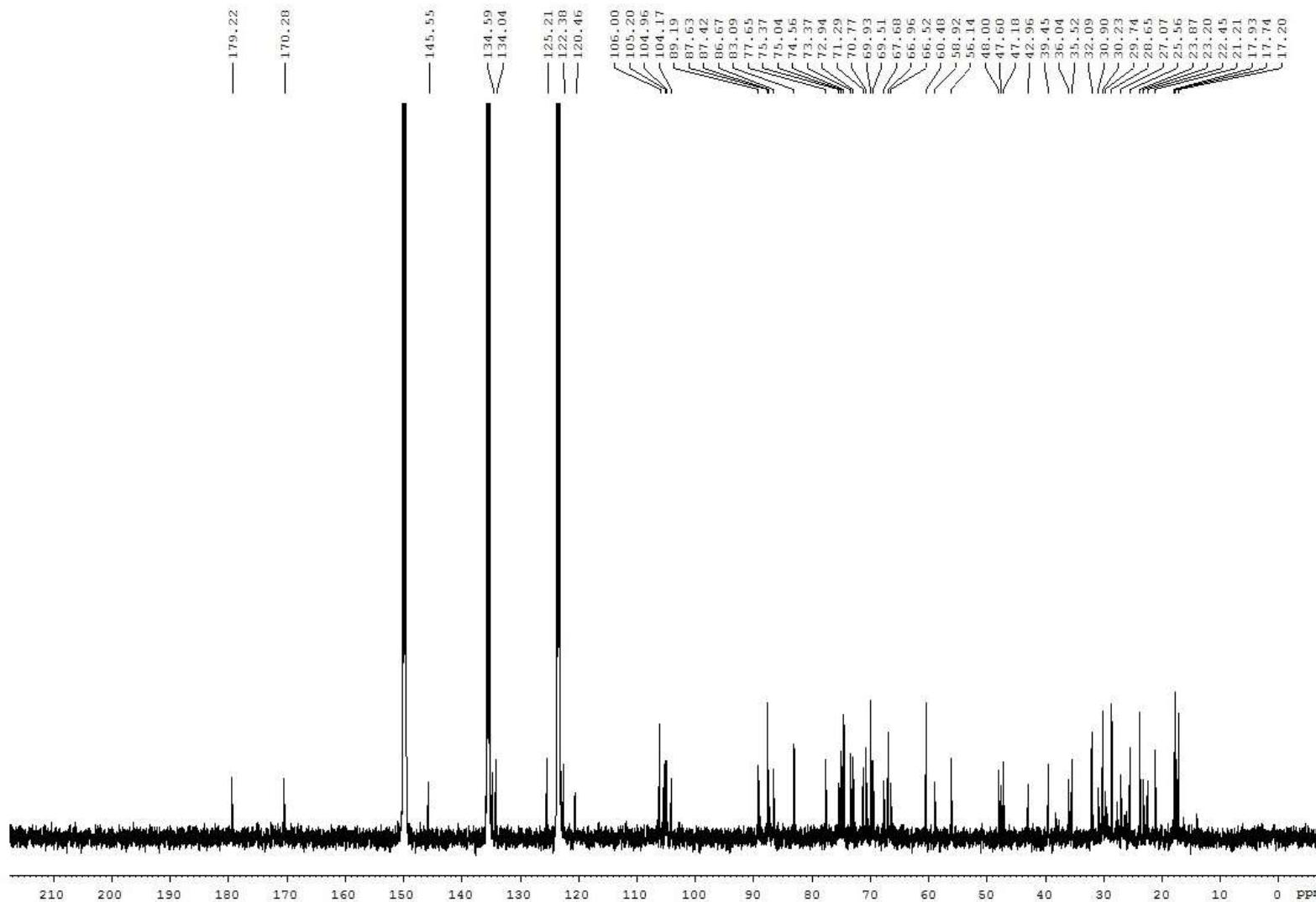
**Figure S25.** IR spectrum of pacificusoside F (**3**) in KBr.



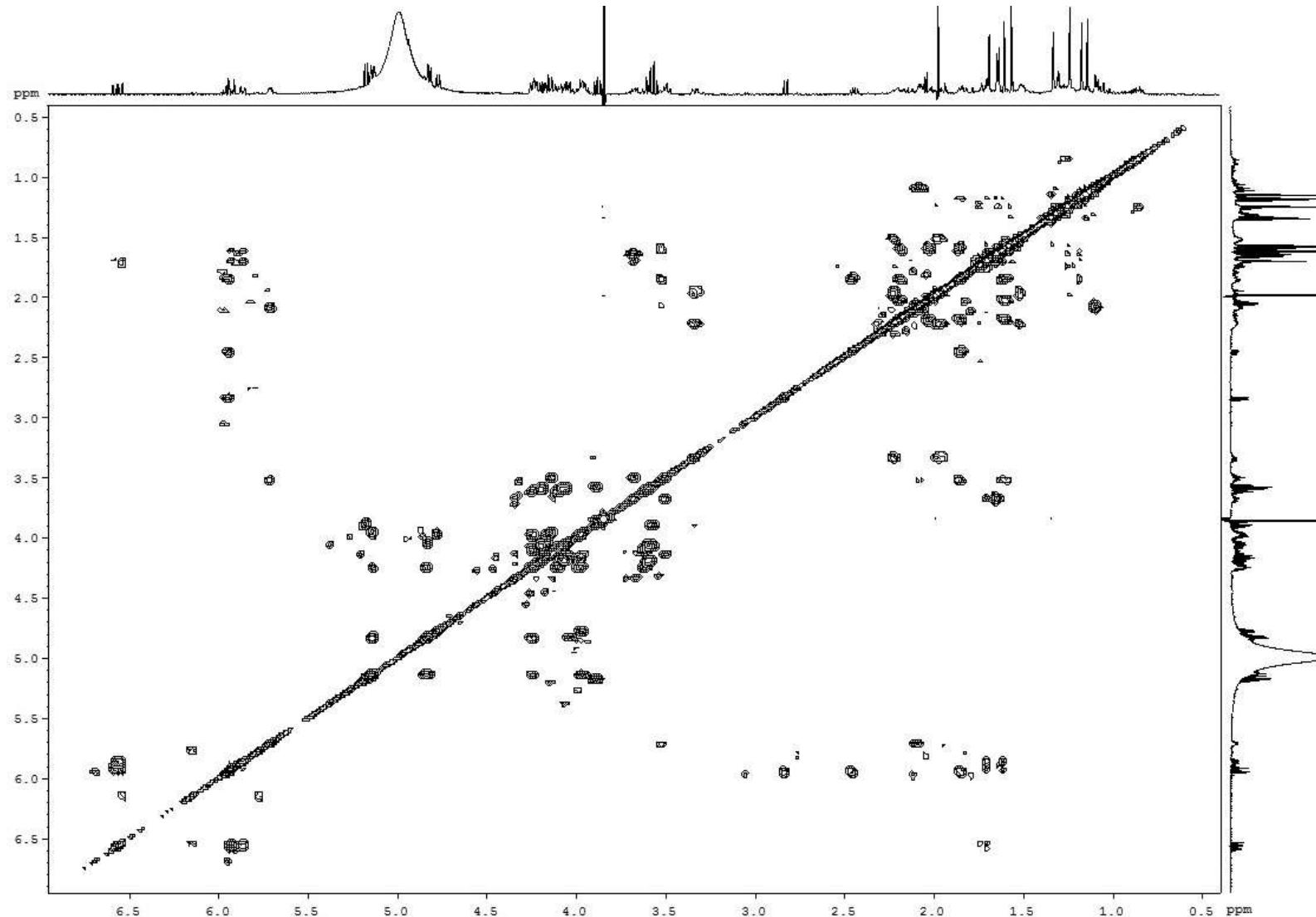
**Figure S26.**  $^1\text{H}$ -NMR spectrum of pacificusoside F (**3**) in  $\text{C}_5\text{D}_5\text{N}$ .



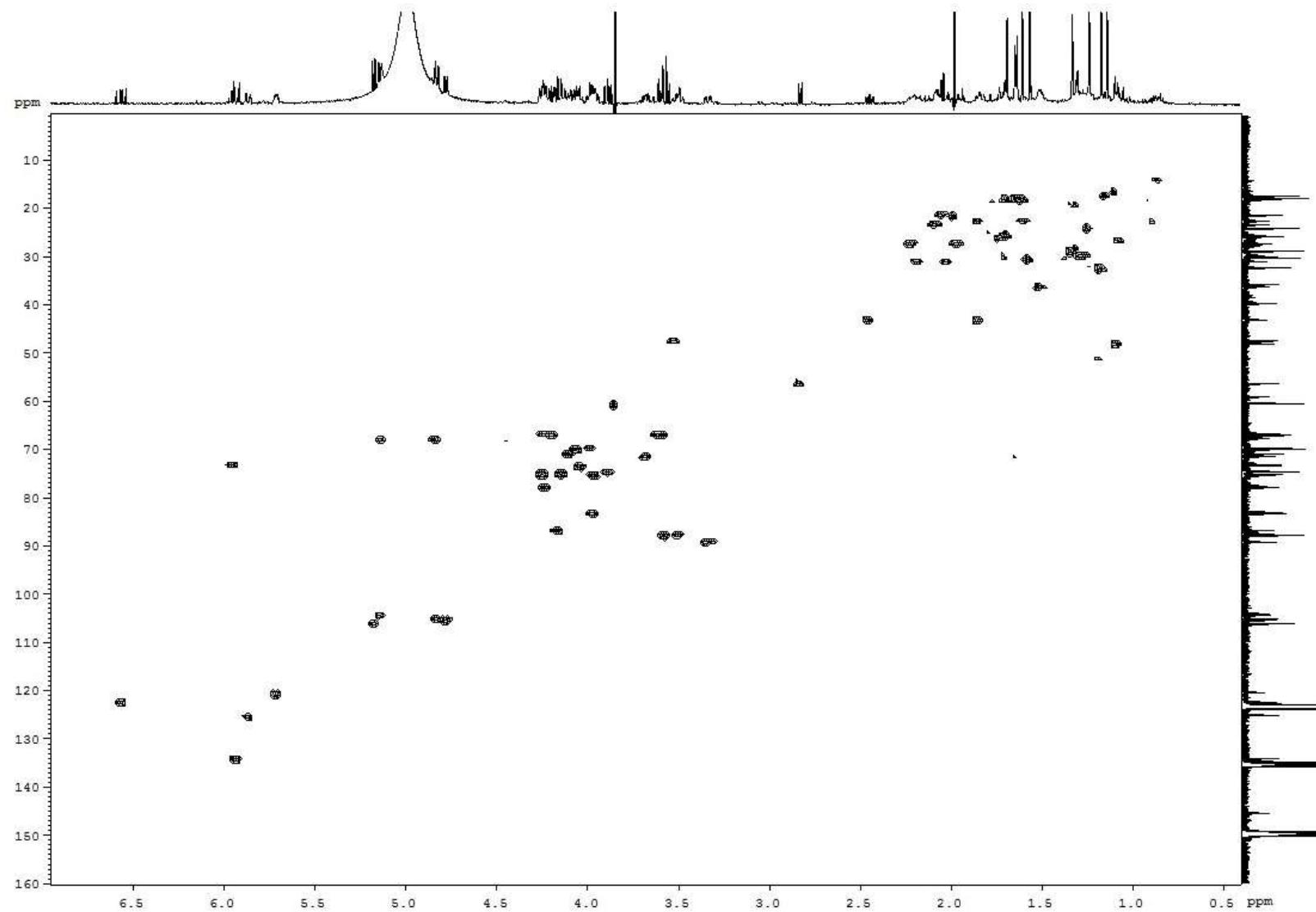
**Figure S27.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside F (**3**) in  $\text{C}_5\text{D}_5\text{N}$ .



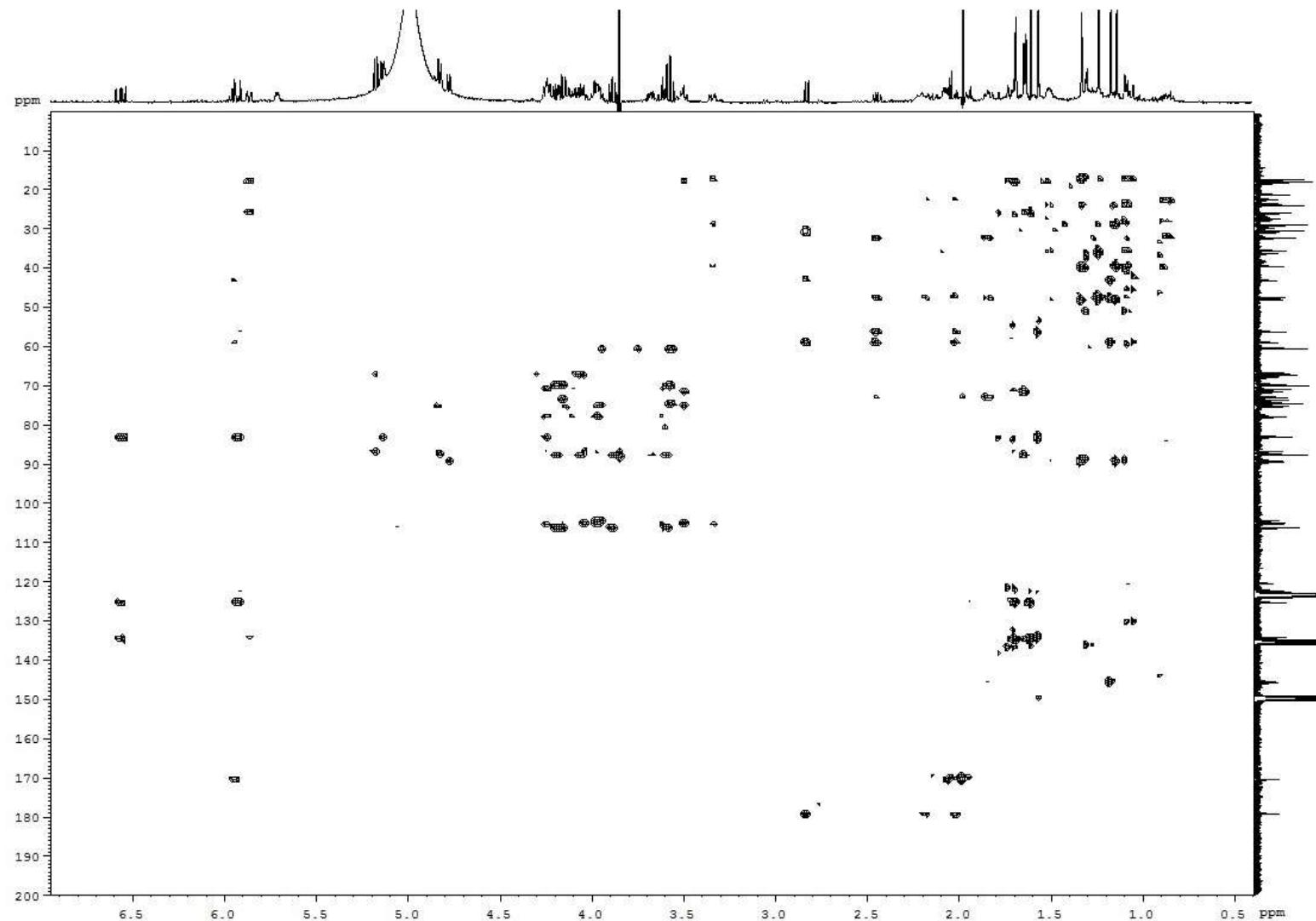
**Figure S28.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside F (**3**) in  $\text{C}_5\text{D}_5\text{N}$ .



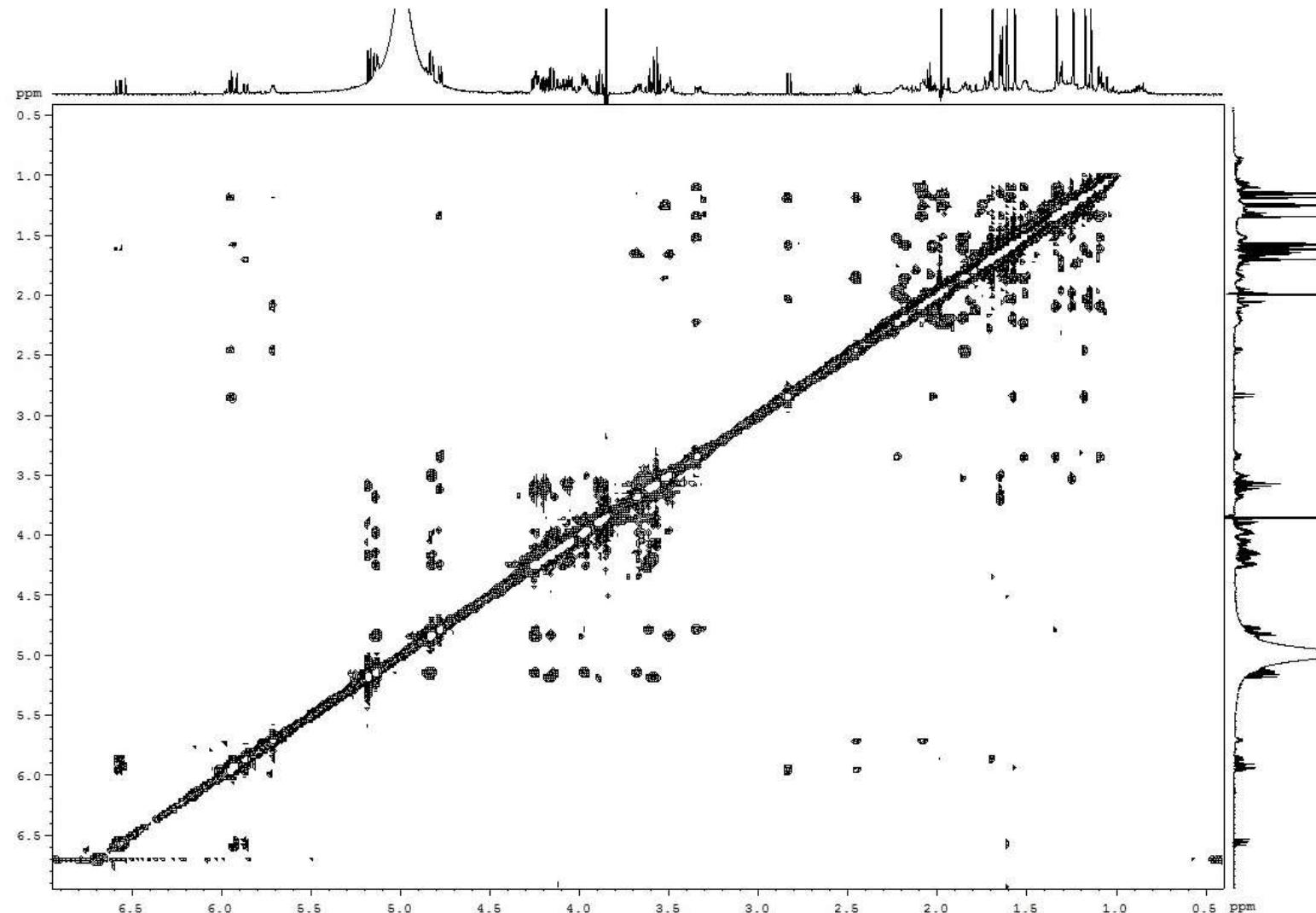
**Figure S29.** HSQC spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.



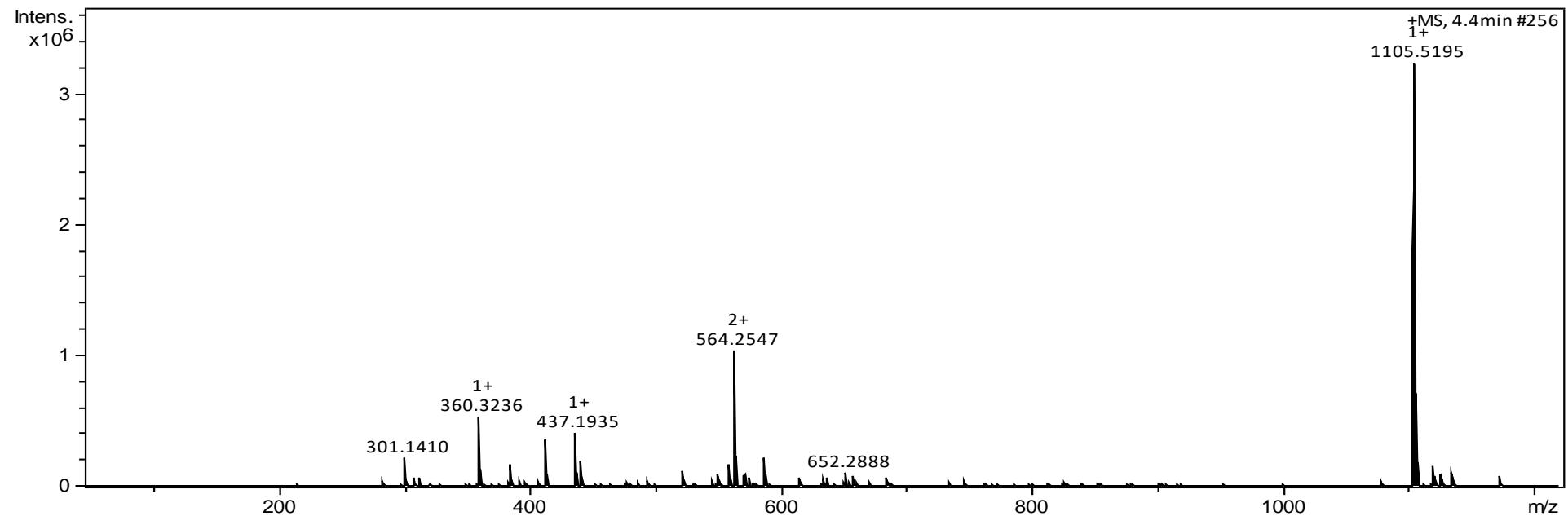
**Figure S30.** HMBC spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.



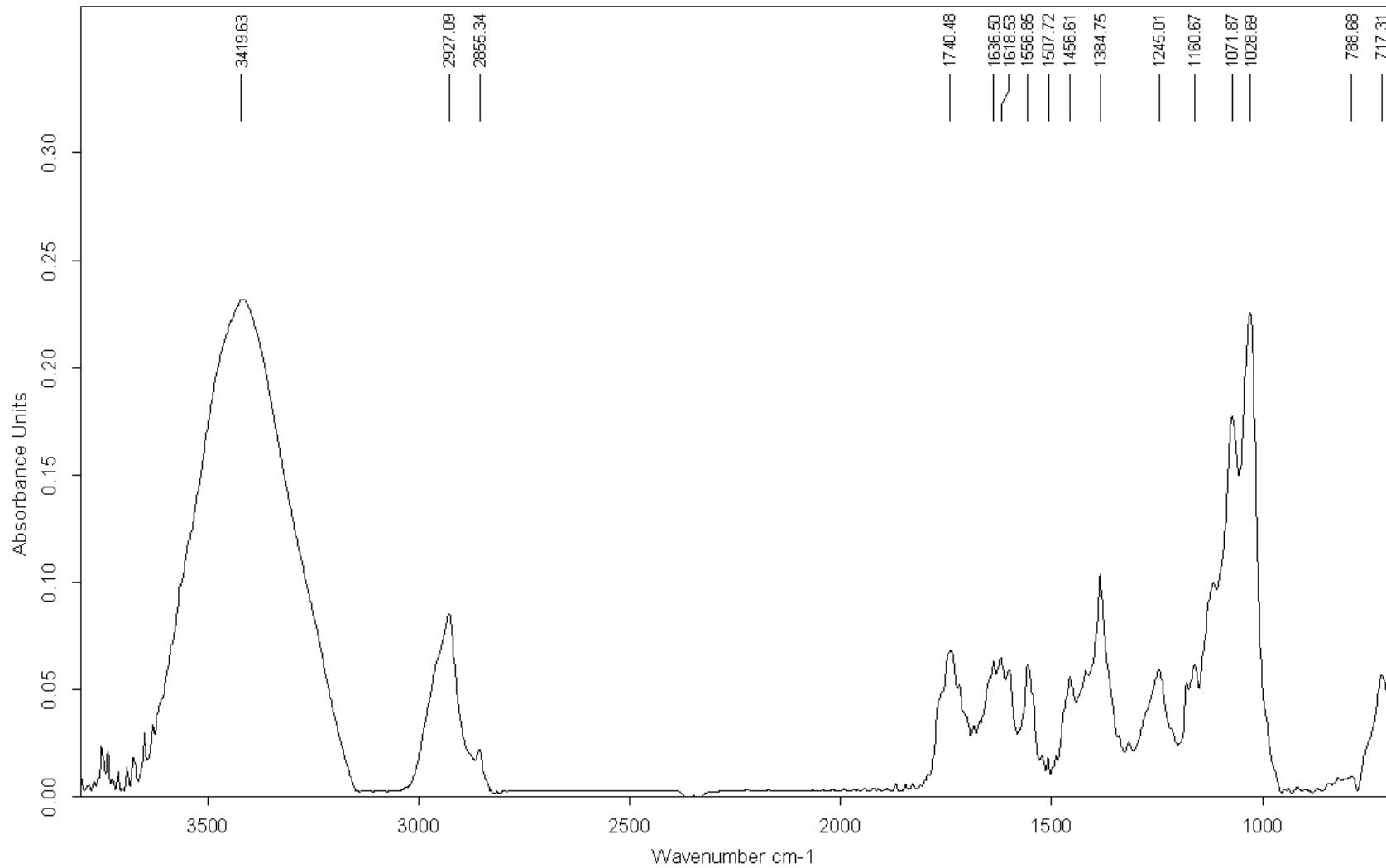
**Figure S31.** ROESY spectrum of pacificusoside F (**3**) in C<sub>5</sub>D<sub>5</sub>N.



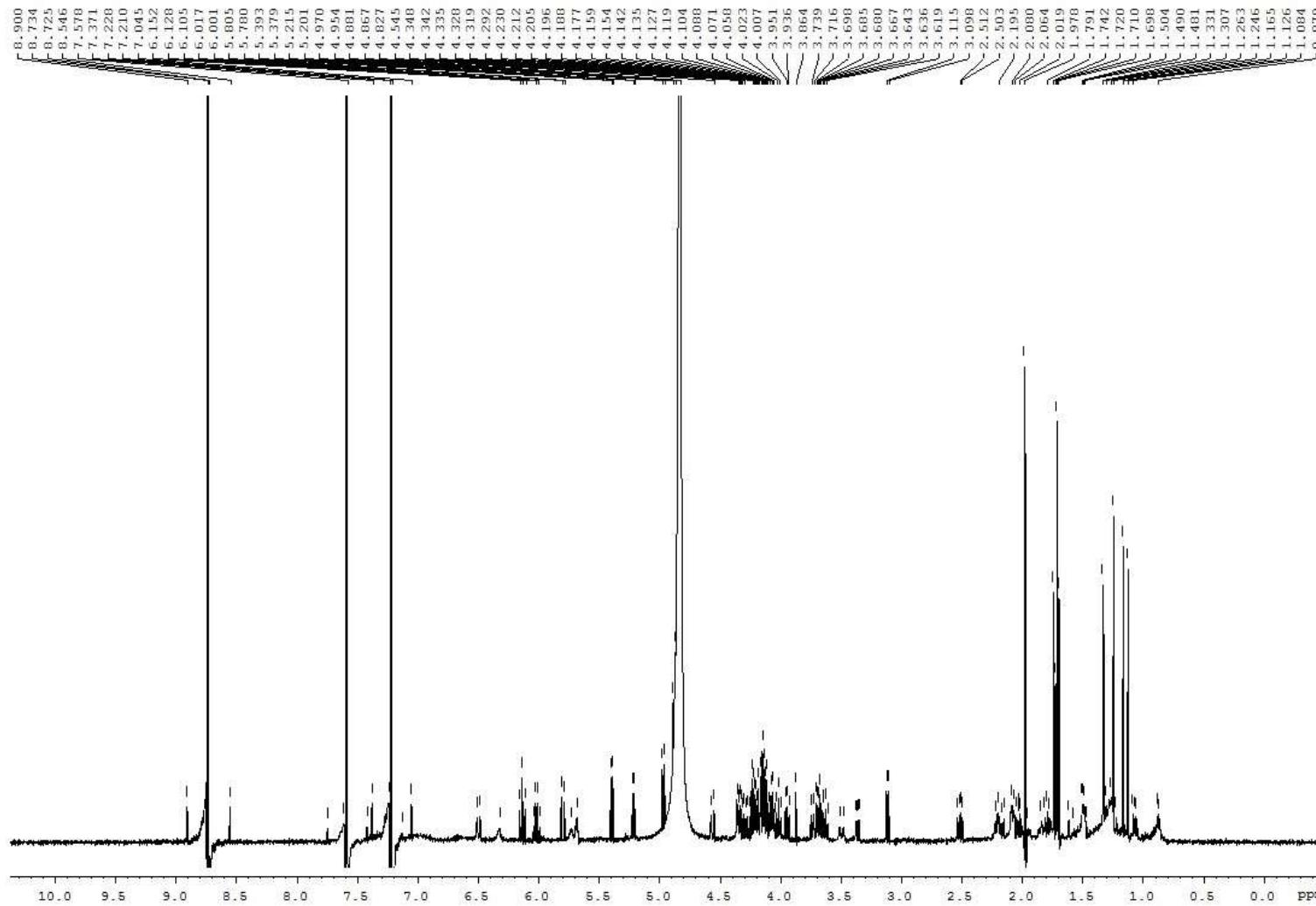
**Figure S32.** HRESIMS spectrum of pacificusoside G (5).



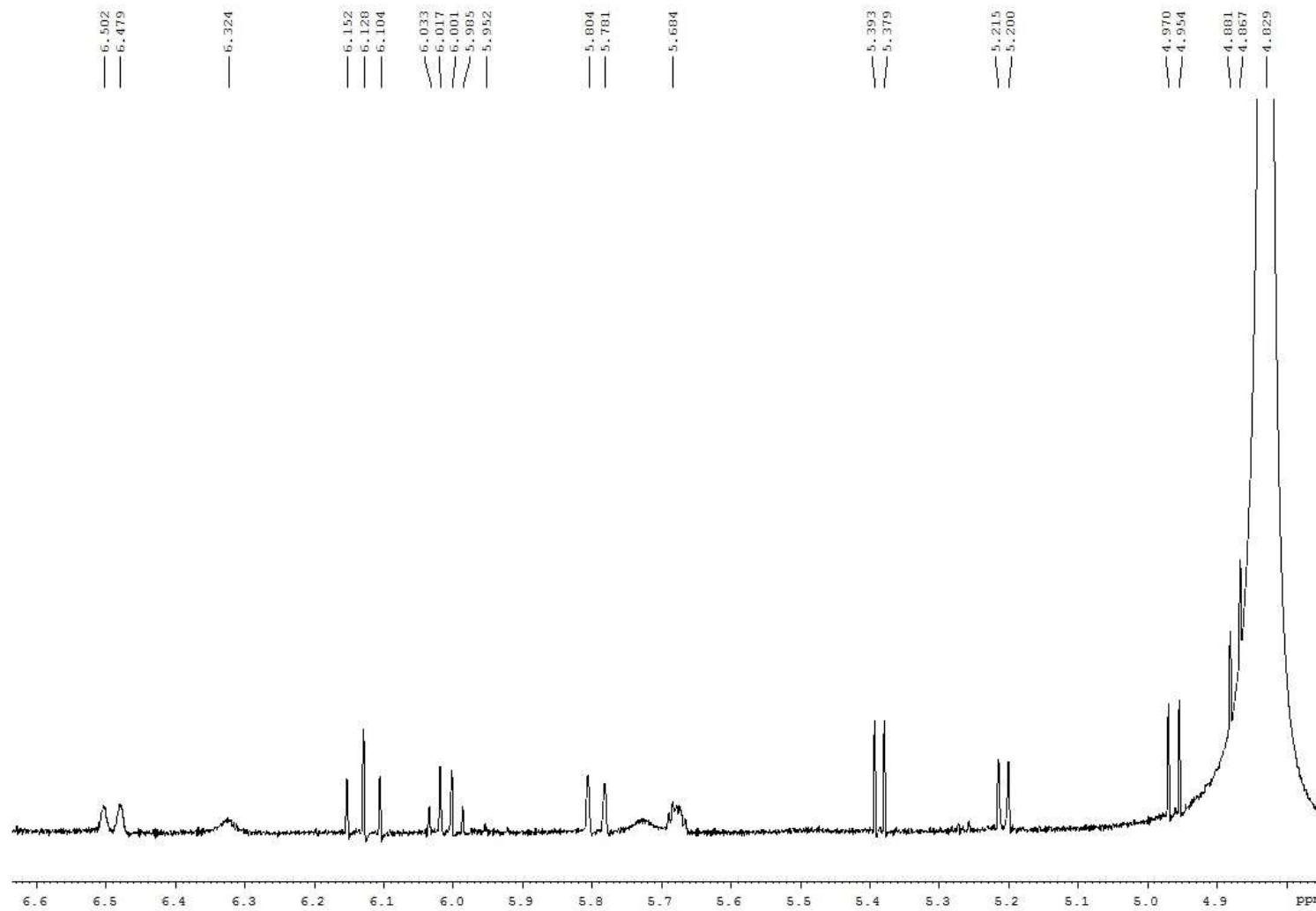
**Figure S33.** IR spectrum of pacificusoside G (5) in KBr.



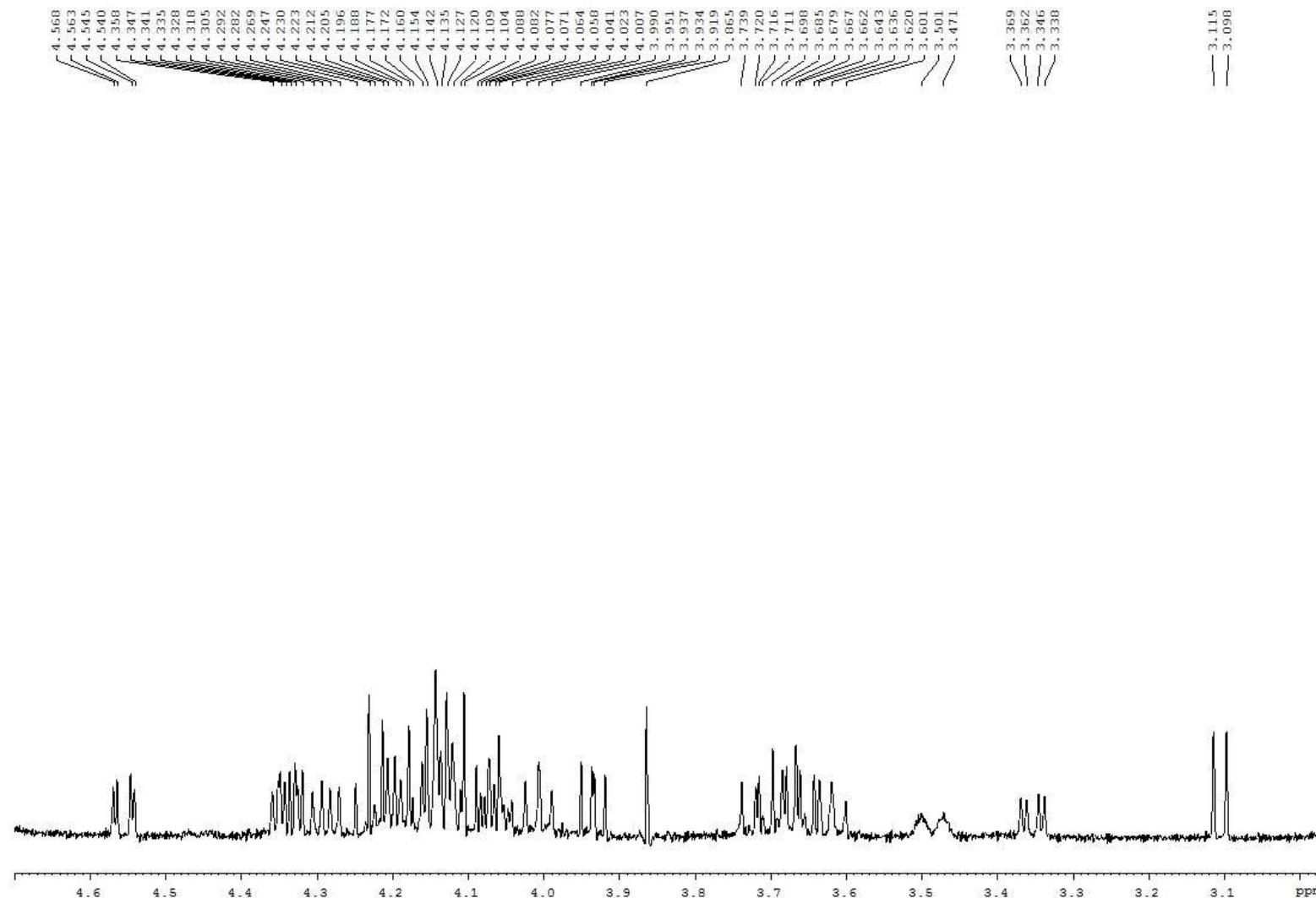
**Figure S34.**  $^1\text{H}$ -NMR spectrum of pacificusoside G (**5**) in  $\text{C}_5\text{D}_5\text{N}$ .



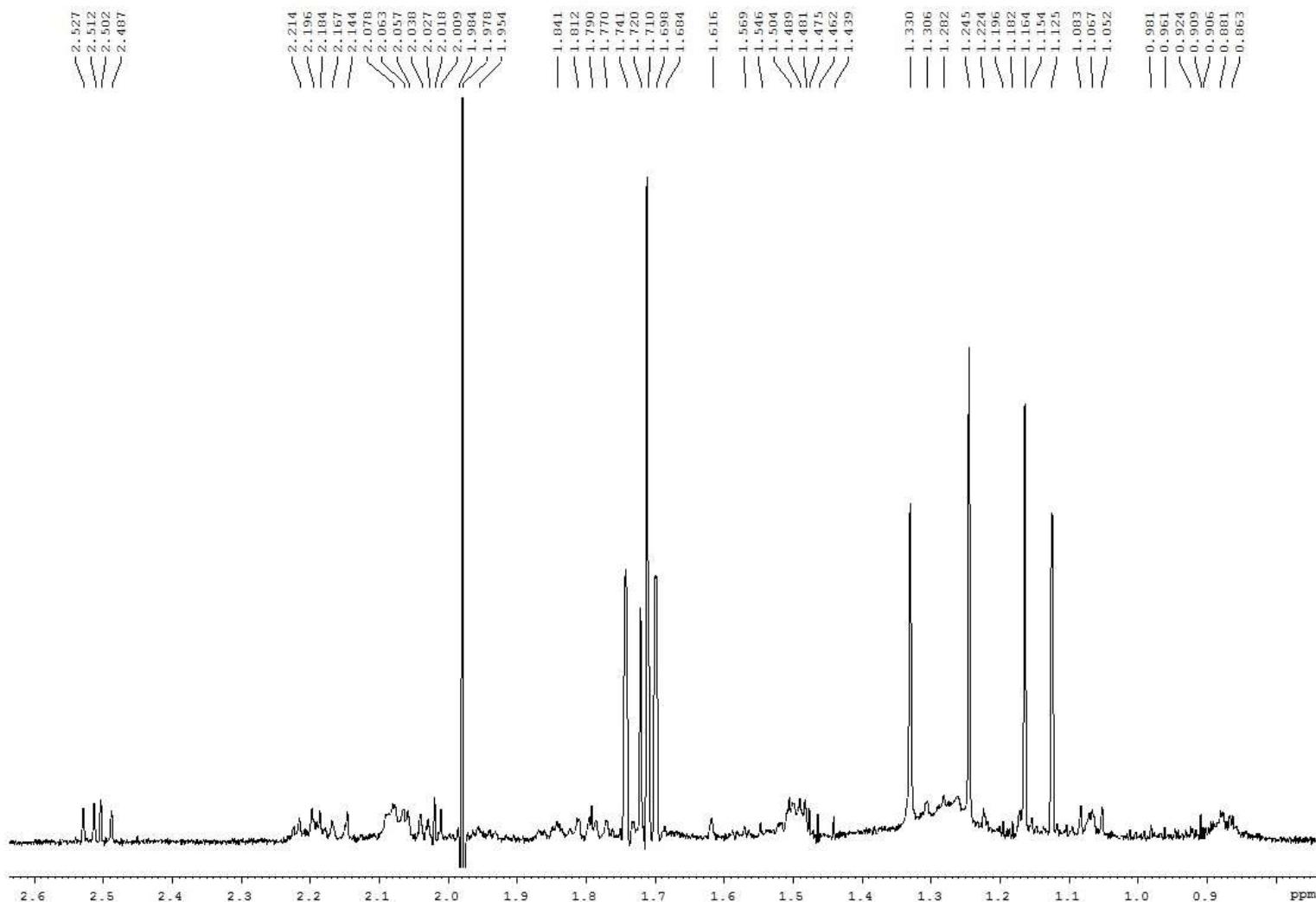
**Figure S35.** Expansion №1 of  $^1\text{H}$ -NMR spectrum of pacificusoside G (**5**) in  $\text{C}_5\text{D}_5\text{N}$ .



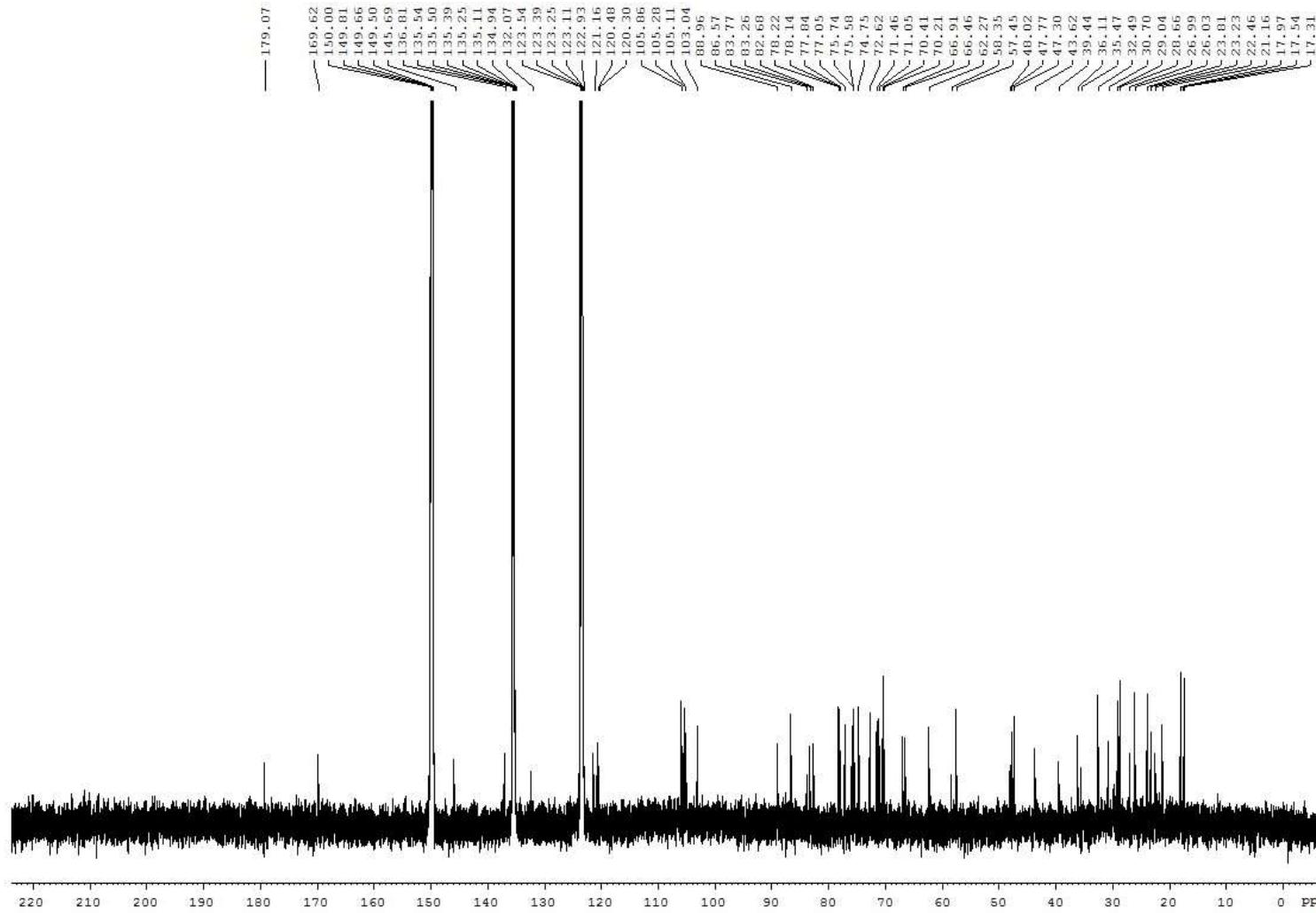
**Figure S36.** Expansion №2 of  $^1\text{H}$ -NMR spectrum of pacificusoside G (**5**) in  $\text{C}_5\text{D}_5\text{N}$ .



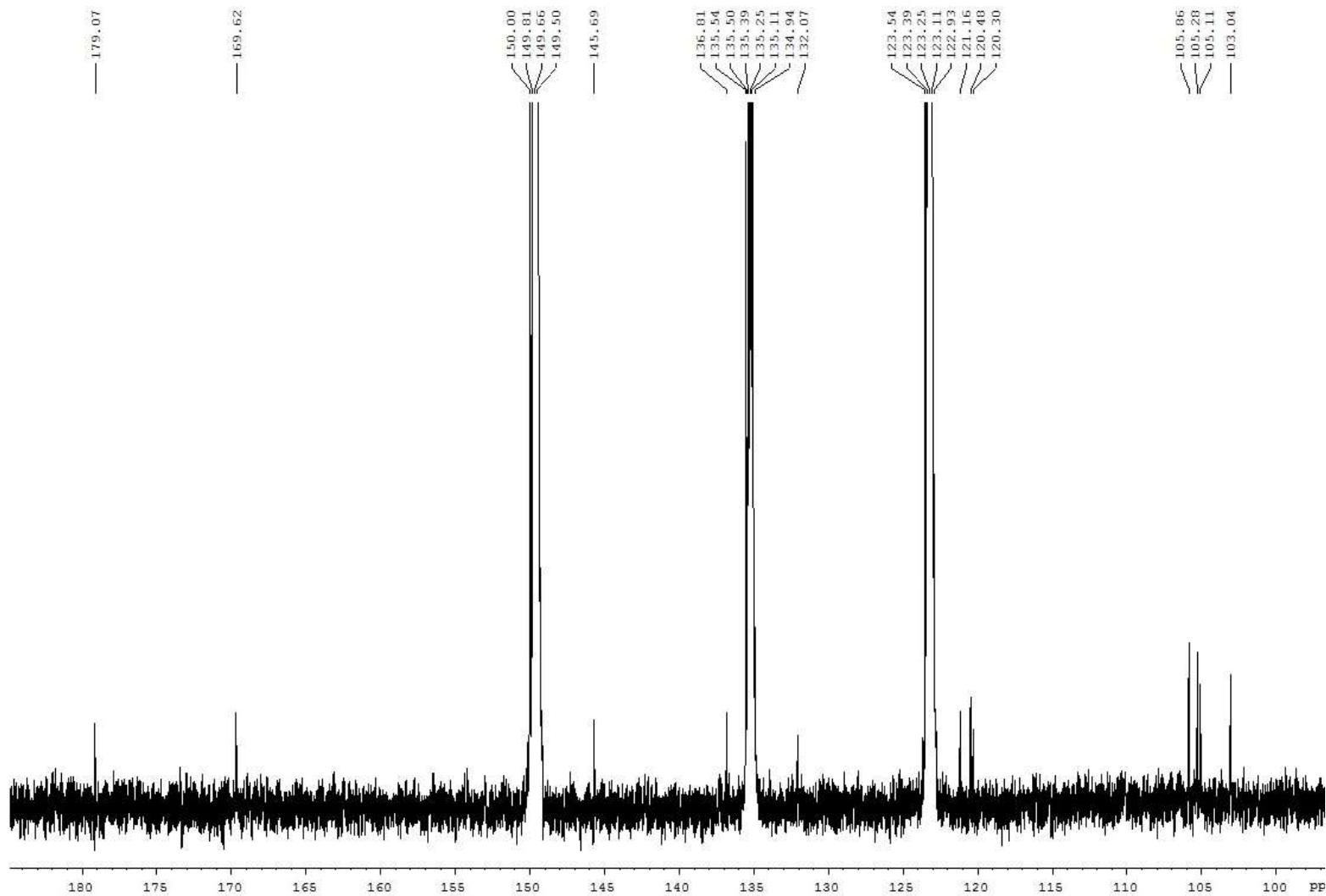
**Figure S37.** Expansion №3 of  $^1\text{H}$ -NMR spectrum of pacificusoside G (**5**) in  $\text{C}_5\text{D}_5\text{N}$ .



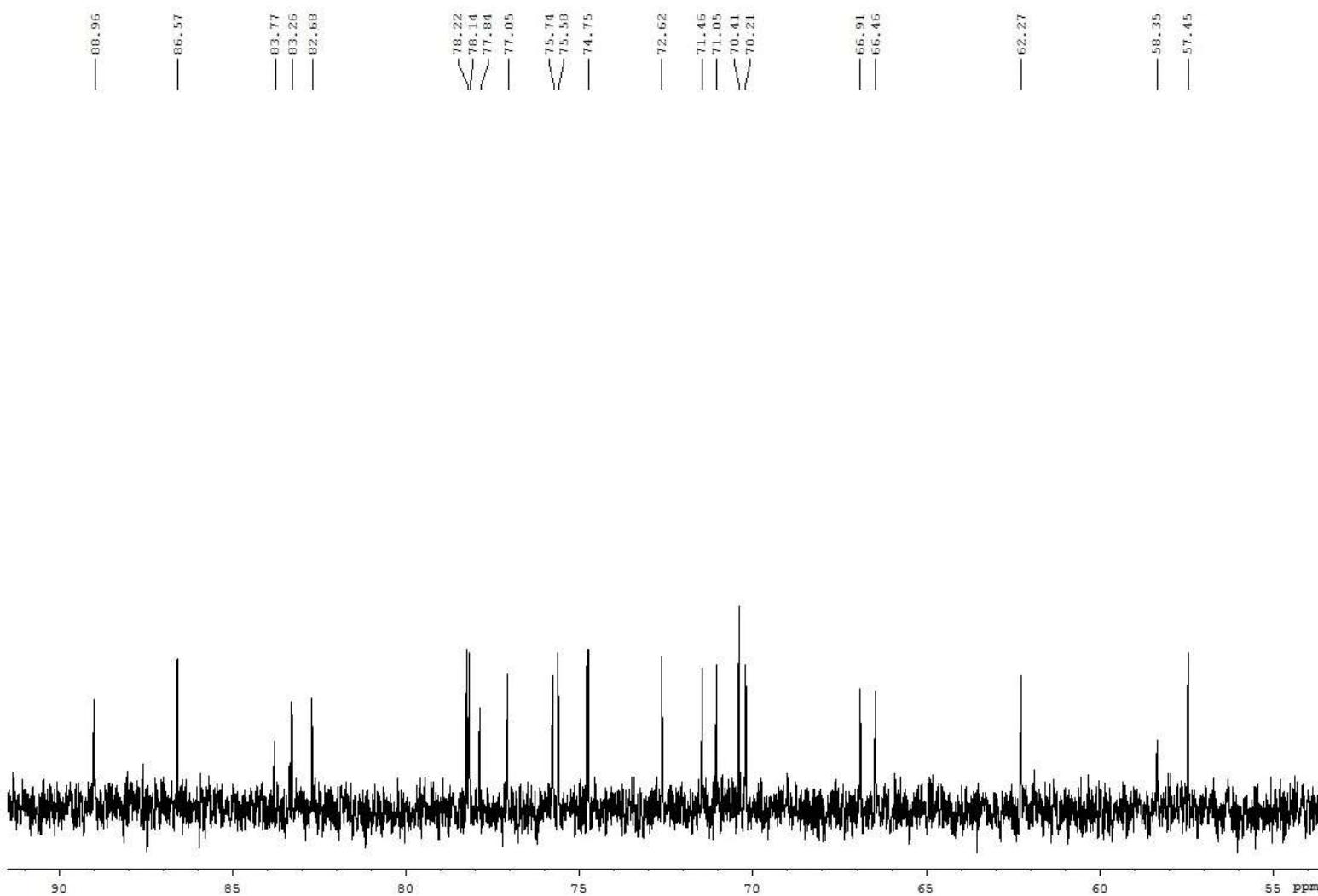
**Figure S38.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside G (5) in  $\text{C}_5\text{D}_5\text{N}$ .



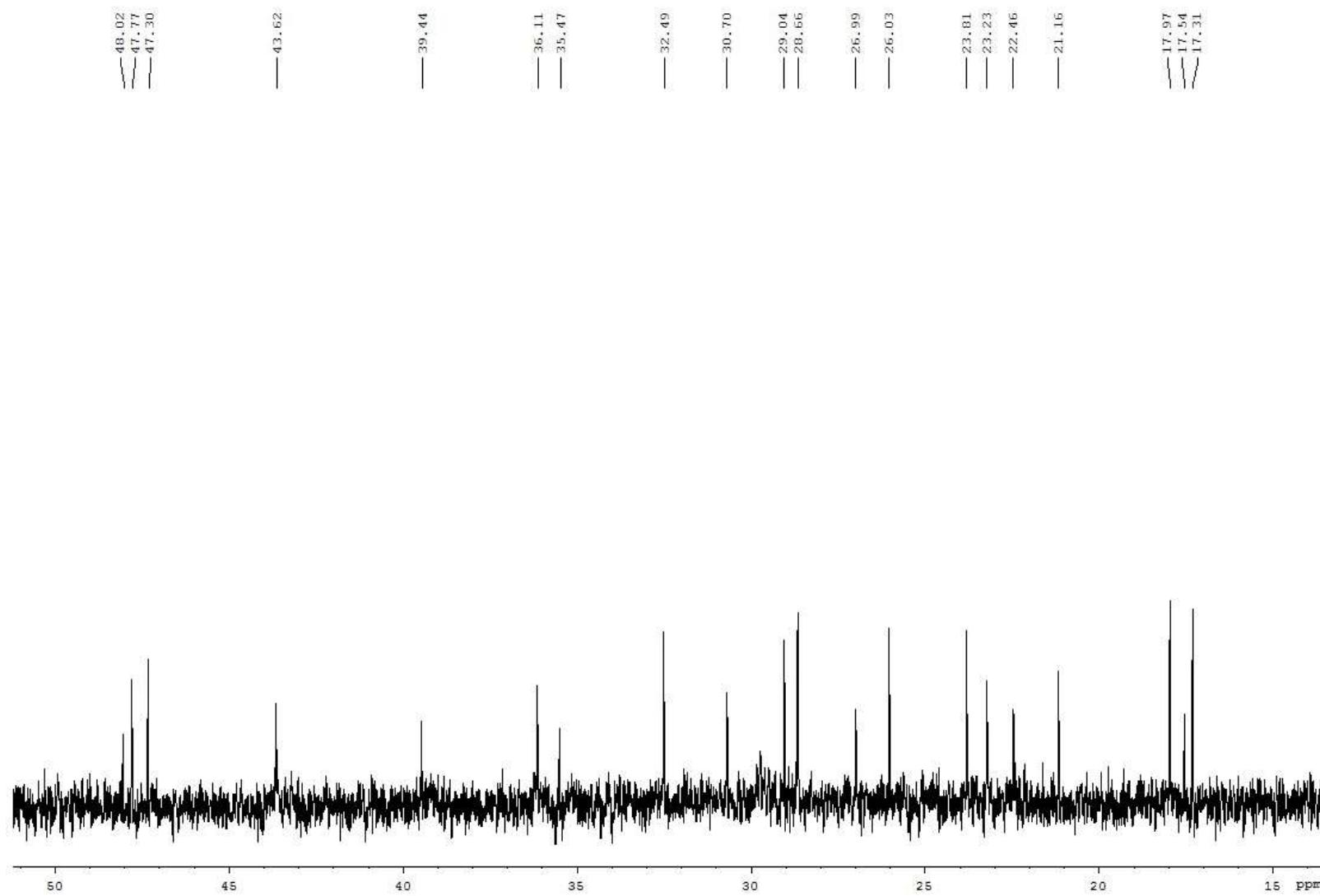
**Figure S39.** Expansion №1 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside G (5) in  $\text{C}_5\text{D}_5\text{N}$ .



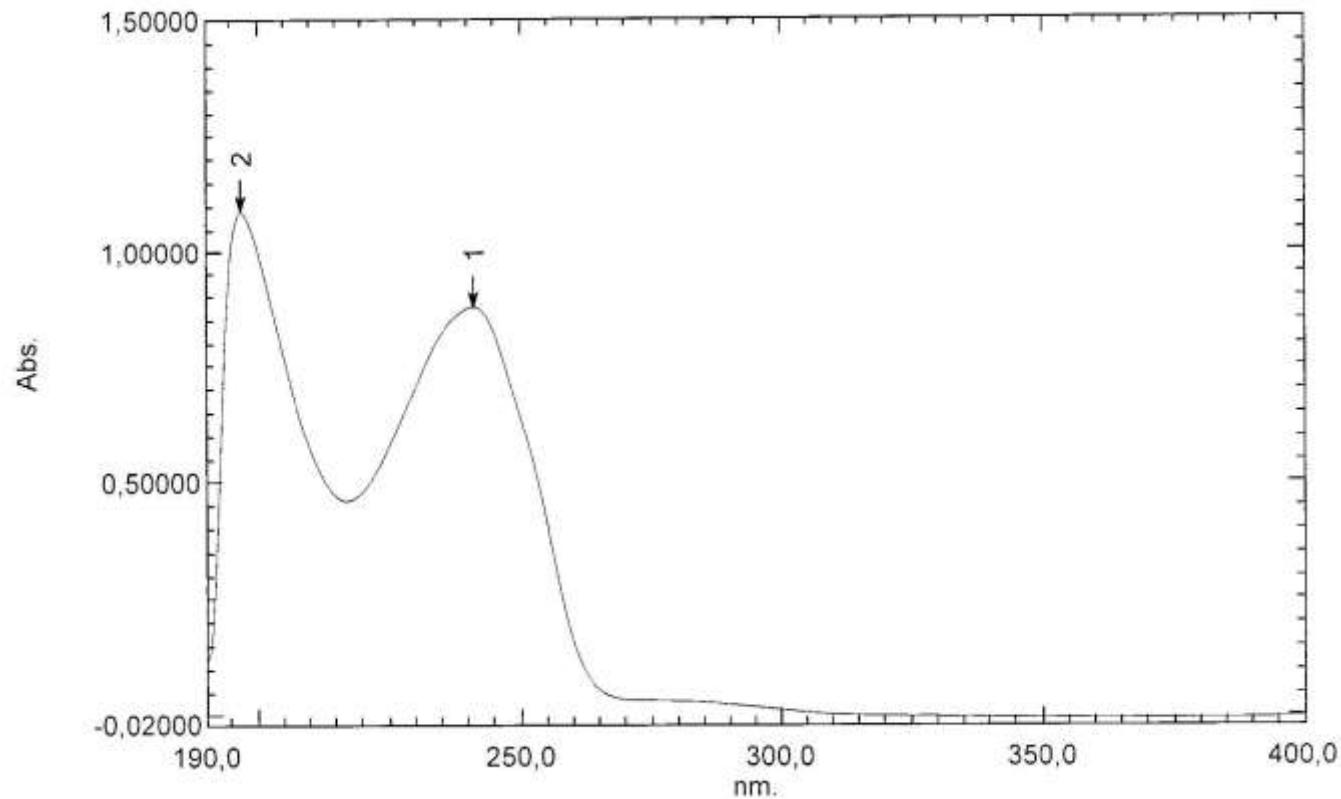
**Figure S40.** Expansion №2 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside G (5) in  $\text{C}_5\text{D}_5\text{N}$ .



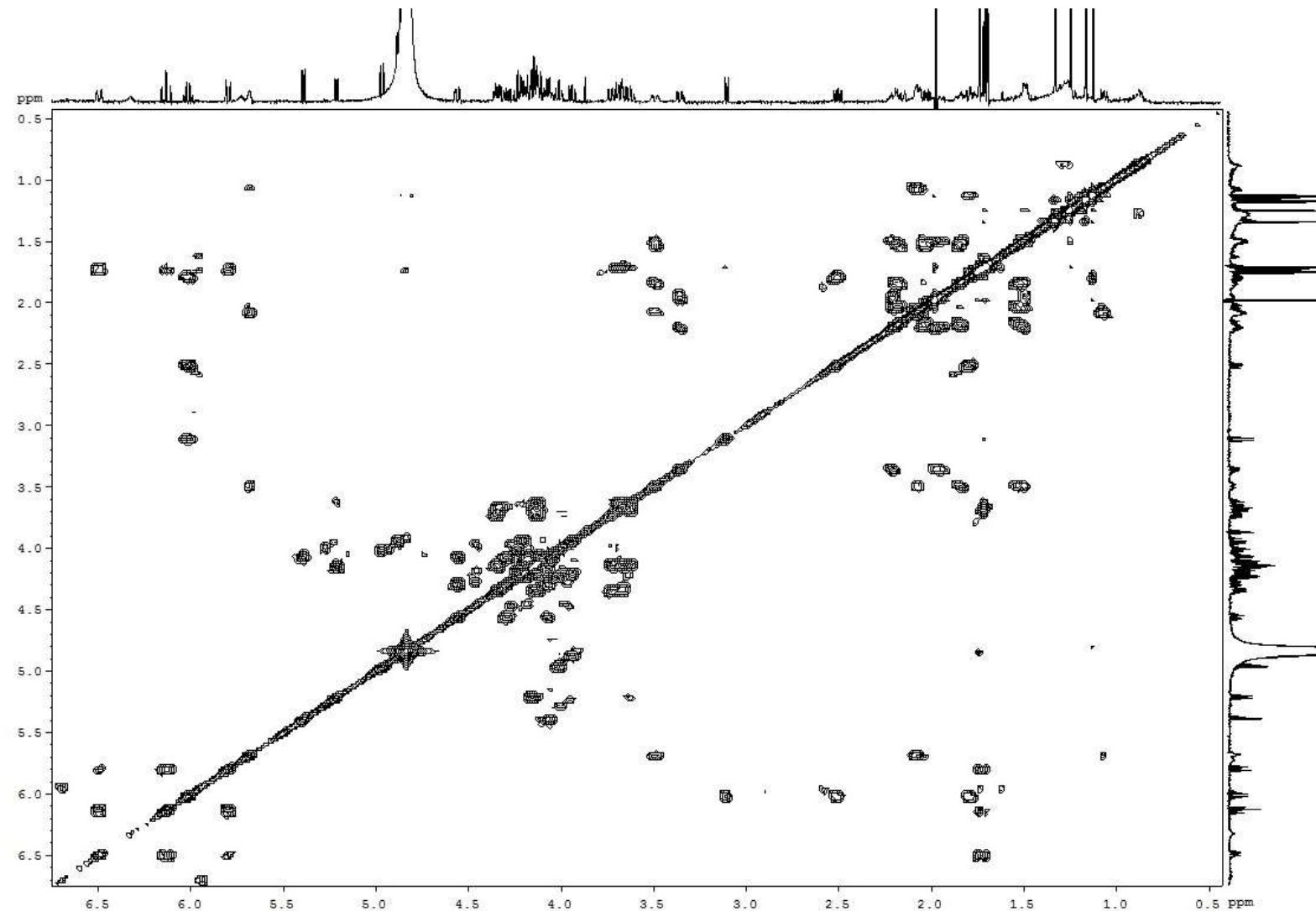
**Figure S41.** Expansion №3 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside G (5) in  $\text{C}_5\text{D}_5\text{N}$ .



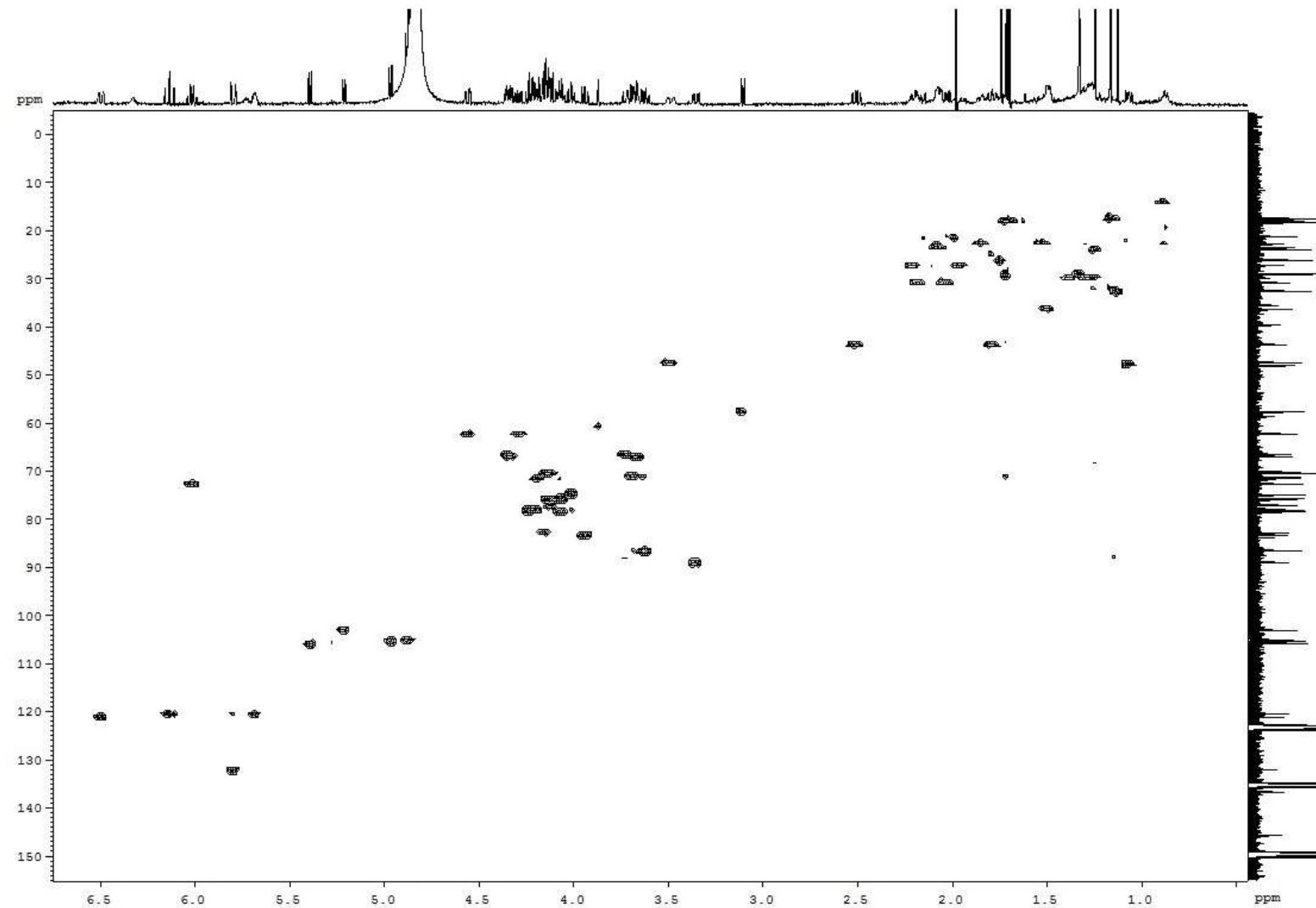
**Figure S42.** UV spectrum of pacificusoside G (5) in MeOH.



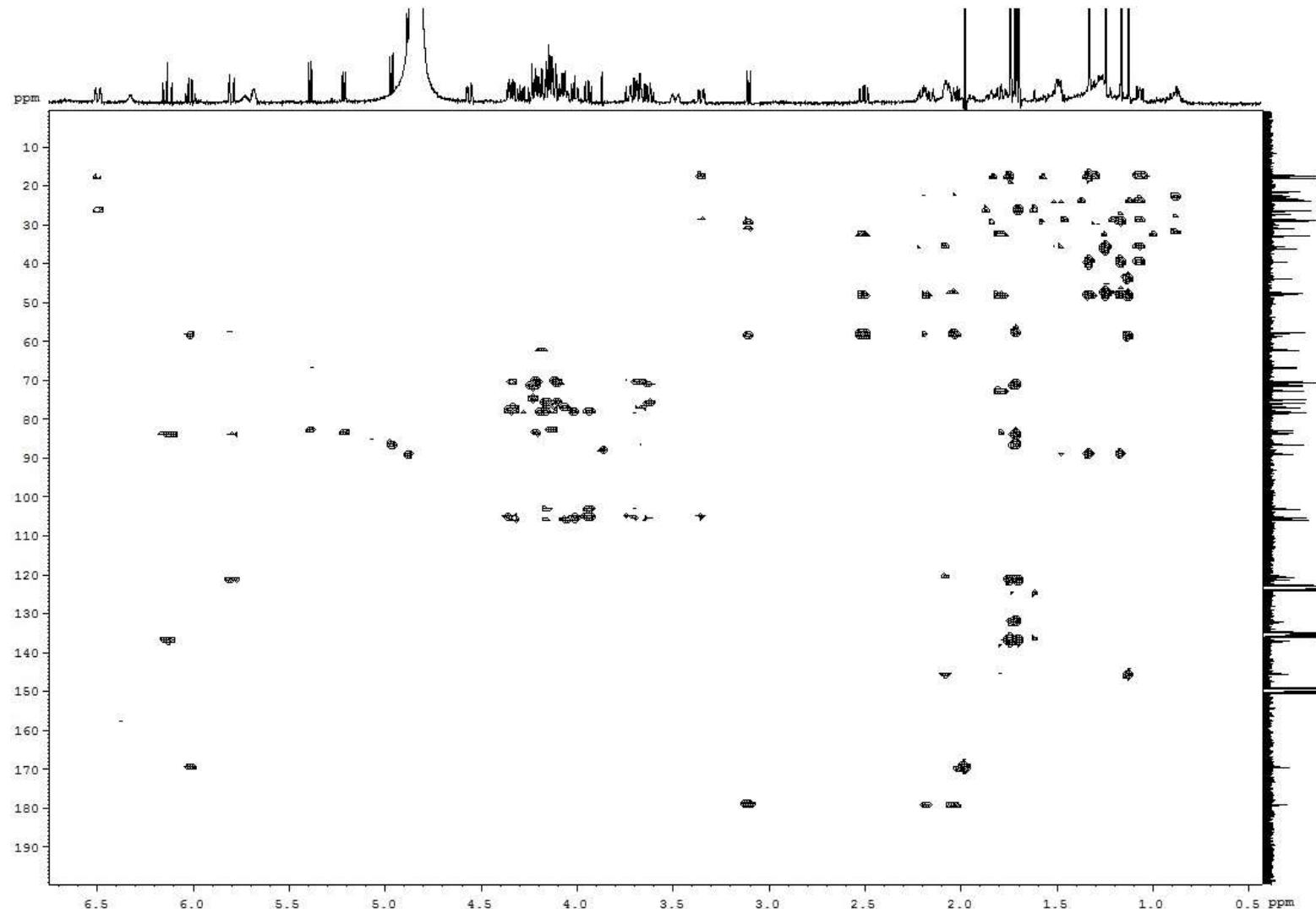
**Figure S43.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside G (**5**) in  $\text{C}_5\text{D}_5\text{N}$ .



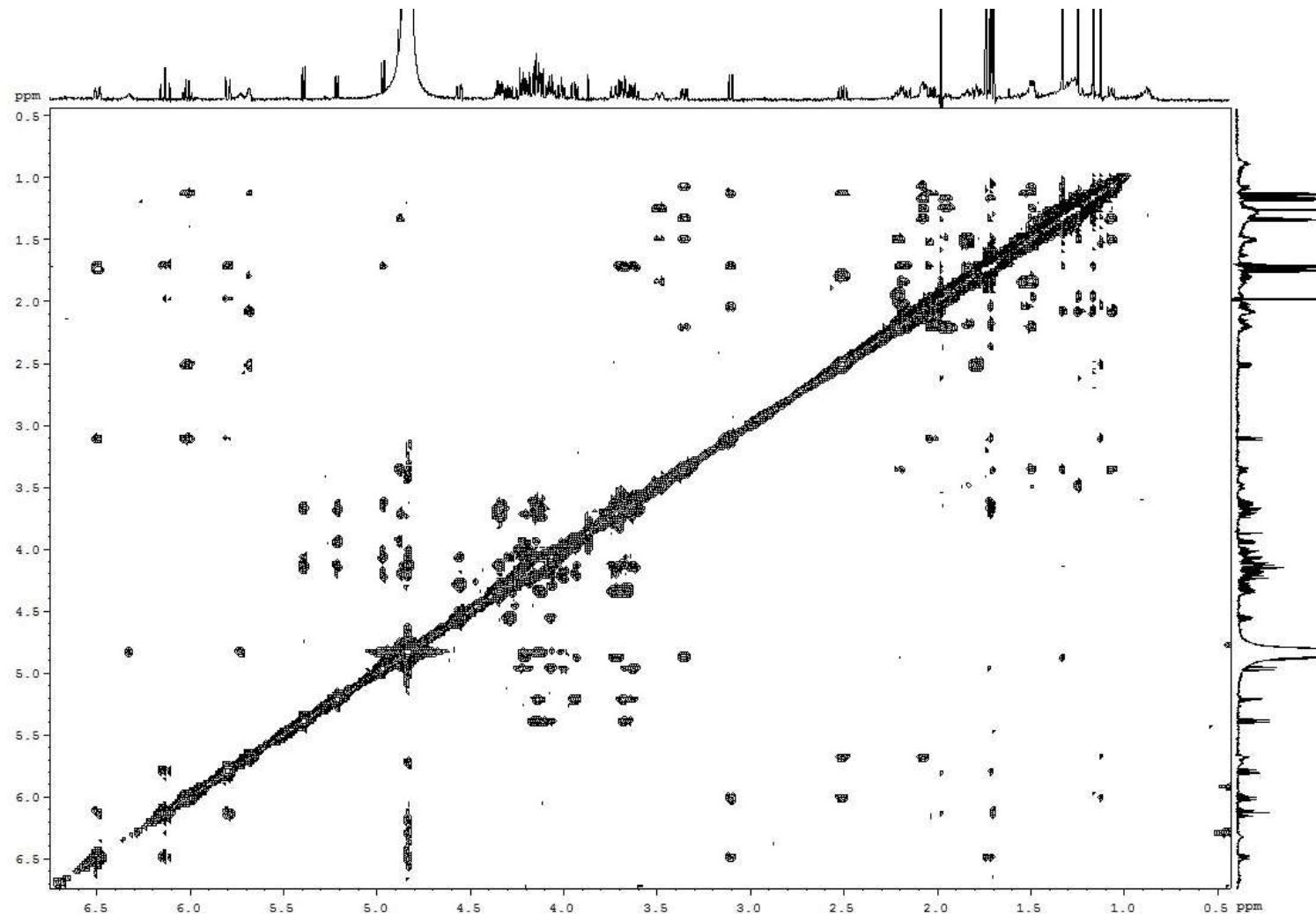
**Figure S44.** HSQC spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.



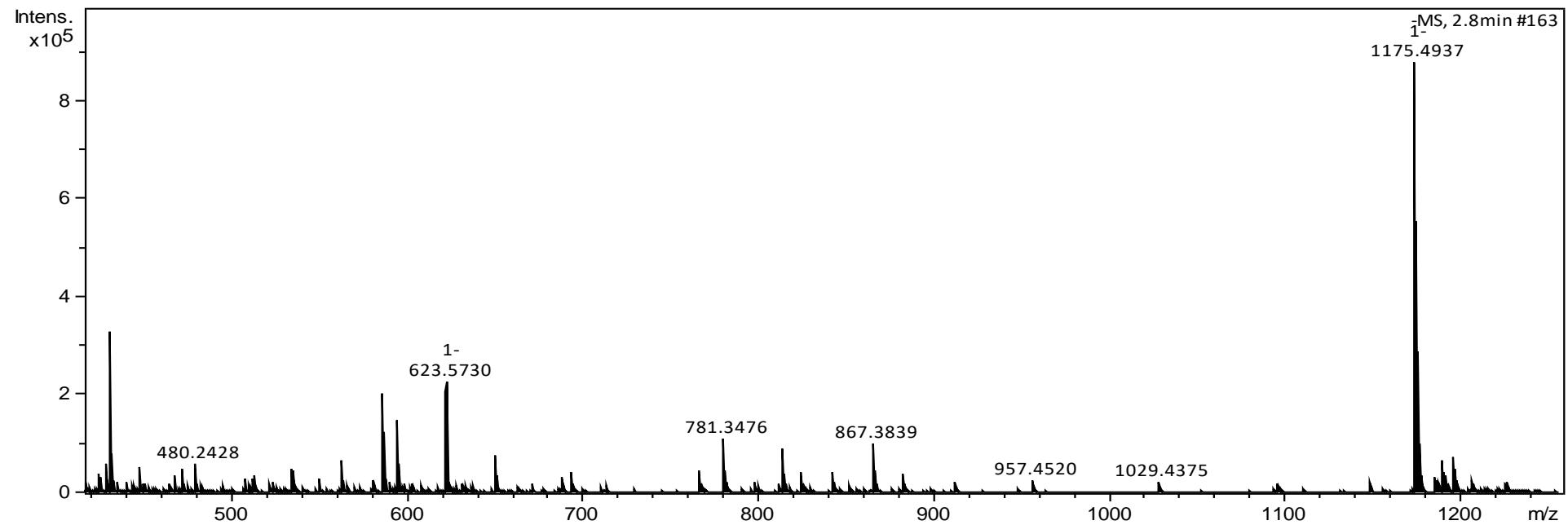
**Figure S45.** HMBC spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.



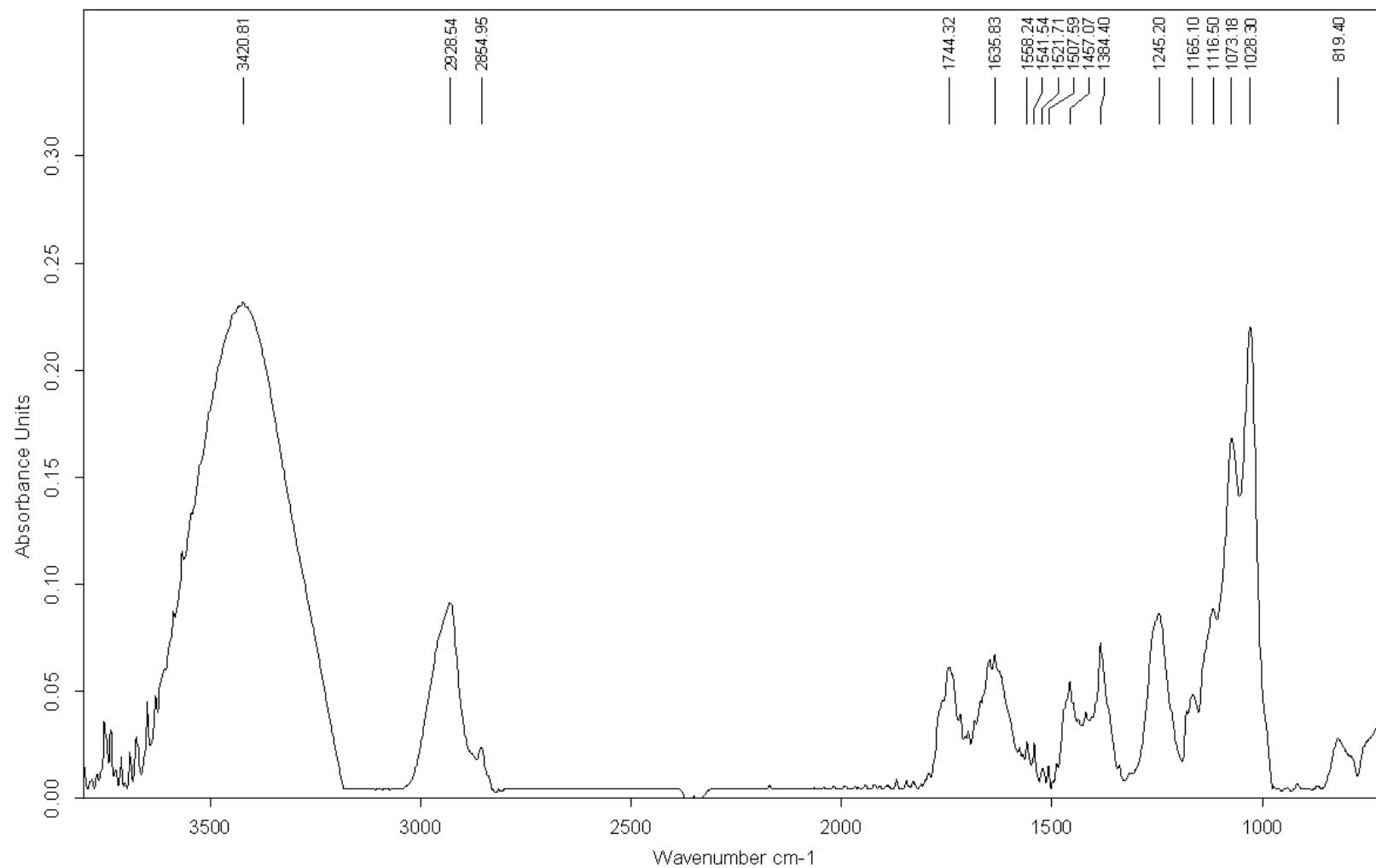
**Figure S46.** ROESY spectrum of pacificusoside G (**5**) in C<sub>5</sub>D<sub>5</sub>N.



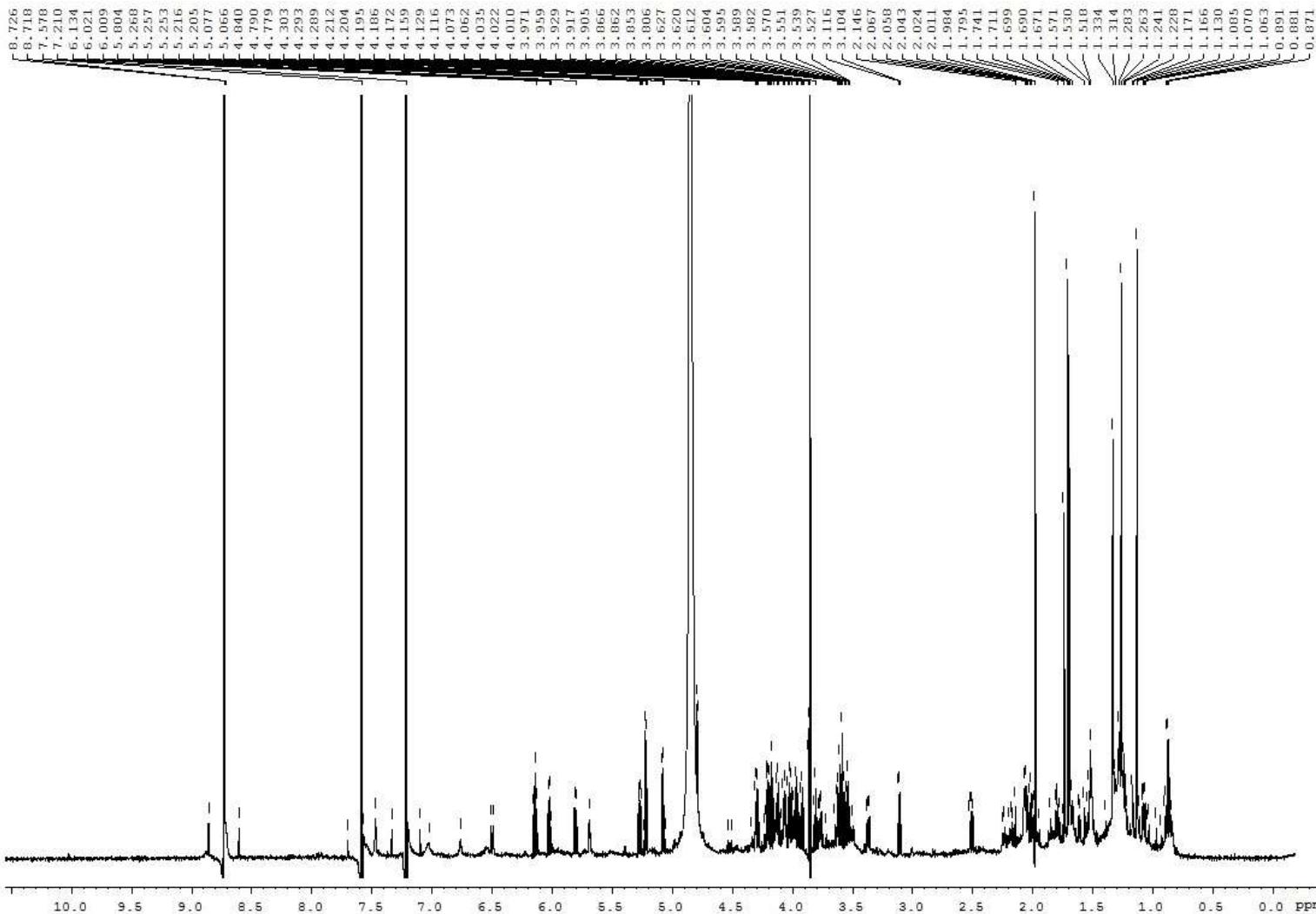
**Figure S47.** HRESIMS spectrum of pacificusoside H (**6**).



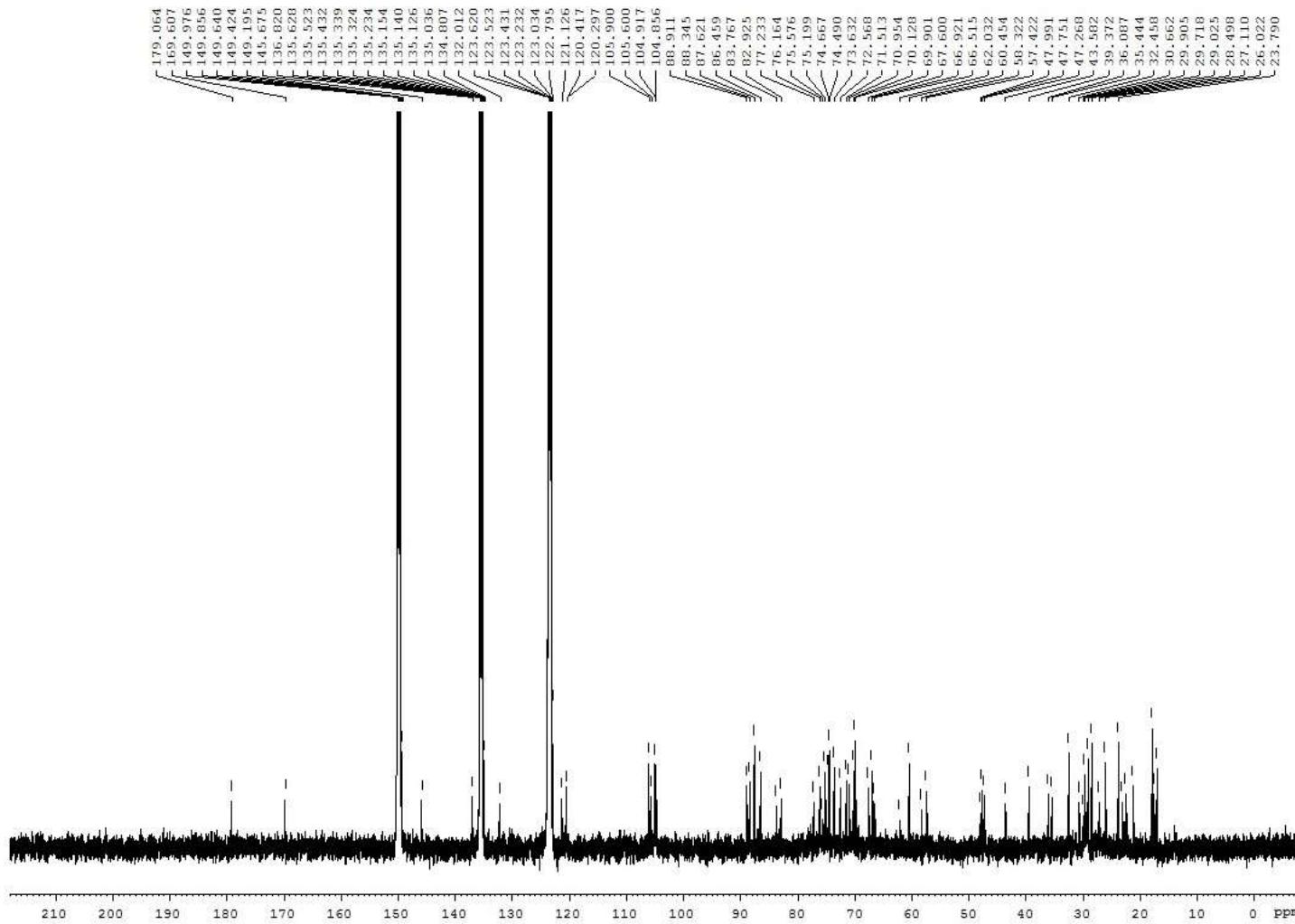
**Figure S48.** IR spectrum of pacificusoside H (**6**) in KBr.



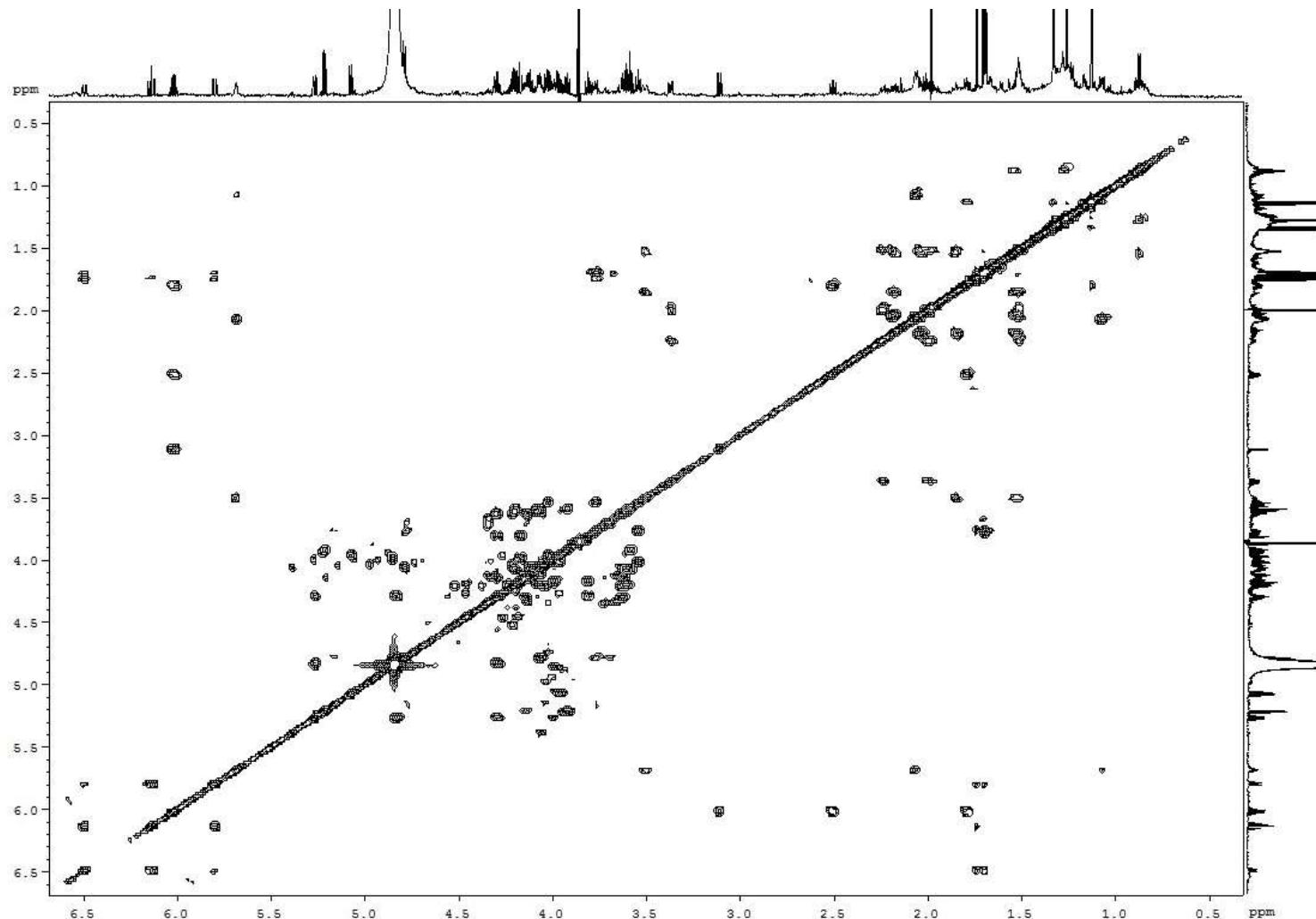
**Figure S49.**  $^1\text{H}$ -NMR spectrum of pacificusoside H (**6**) in  $\text{C}_5\text{D}_5\text{N}$ .



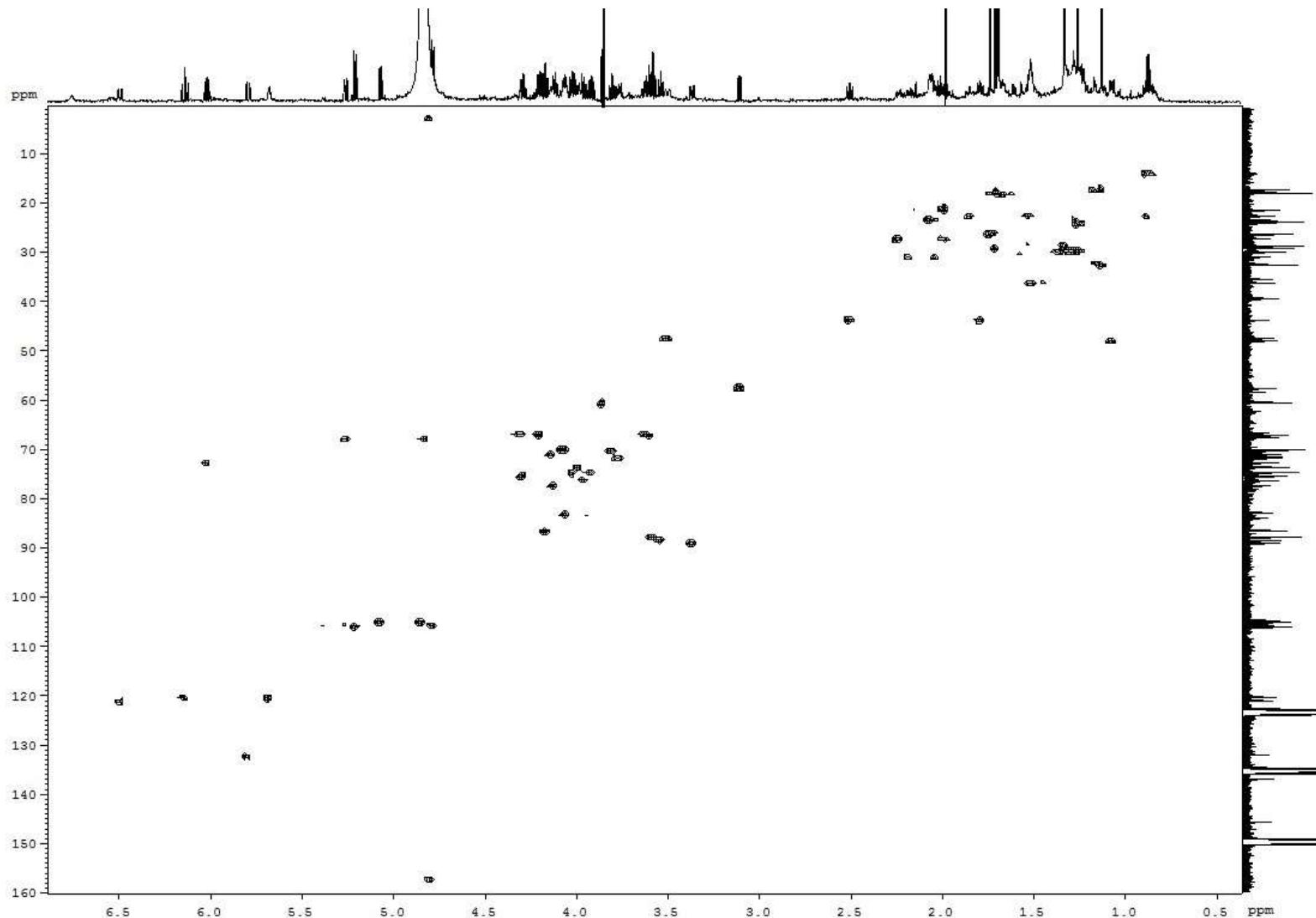
**Figure S50.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside H (**6**) in  $\text{C}_5\text{D}_5\text{N}$ .



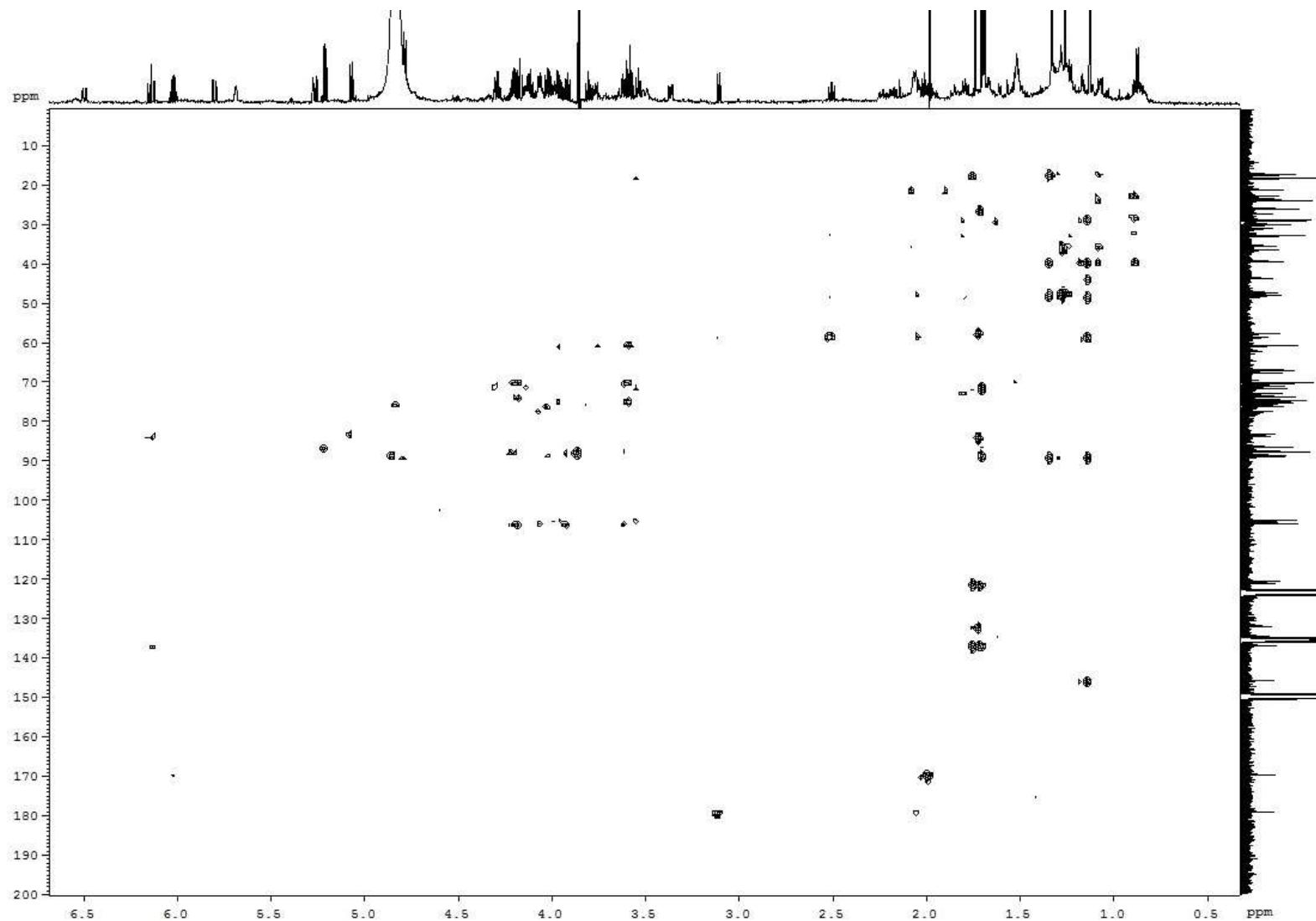
**Figure S51.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside H (**6**) in  $\text{C}_5\text{D}_5\text{N}$ .



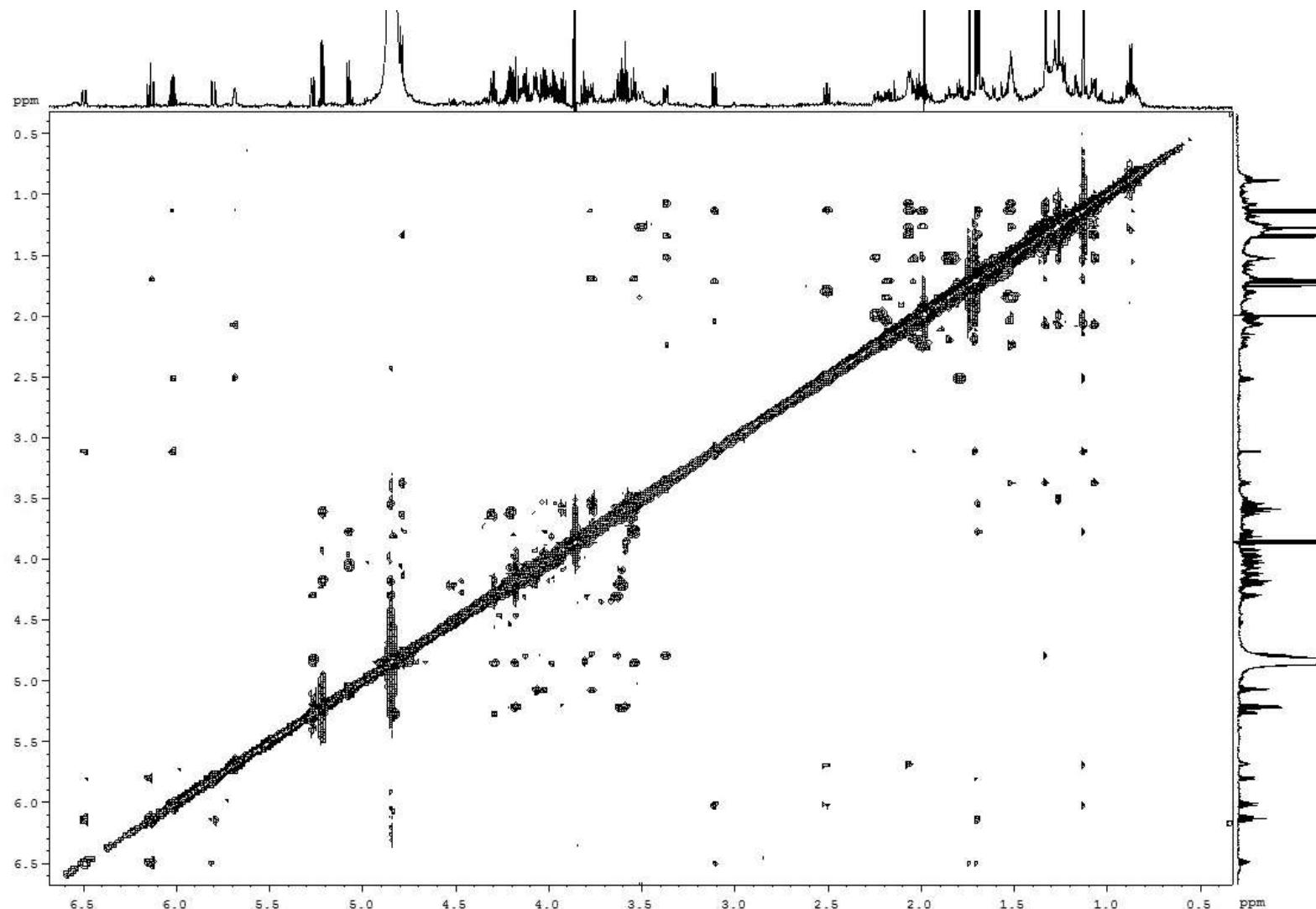
**Figure S52.** HSQC spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.



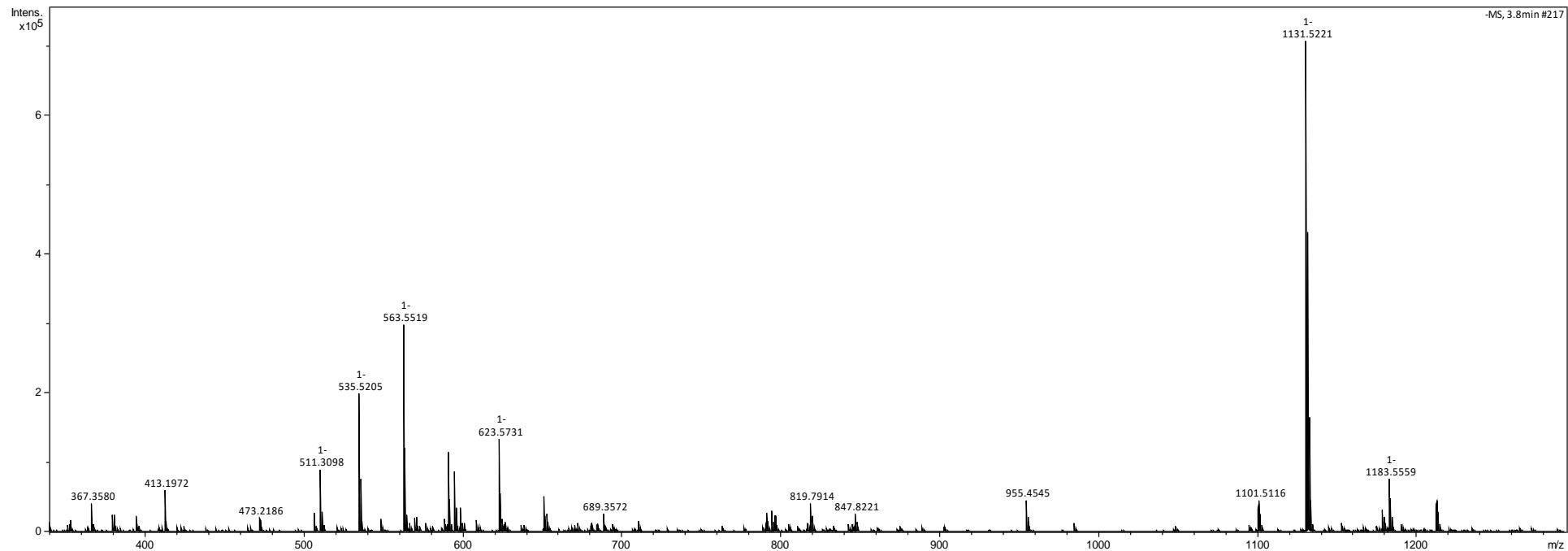
**Figure S53.** HMBC spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.



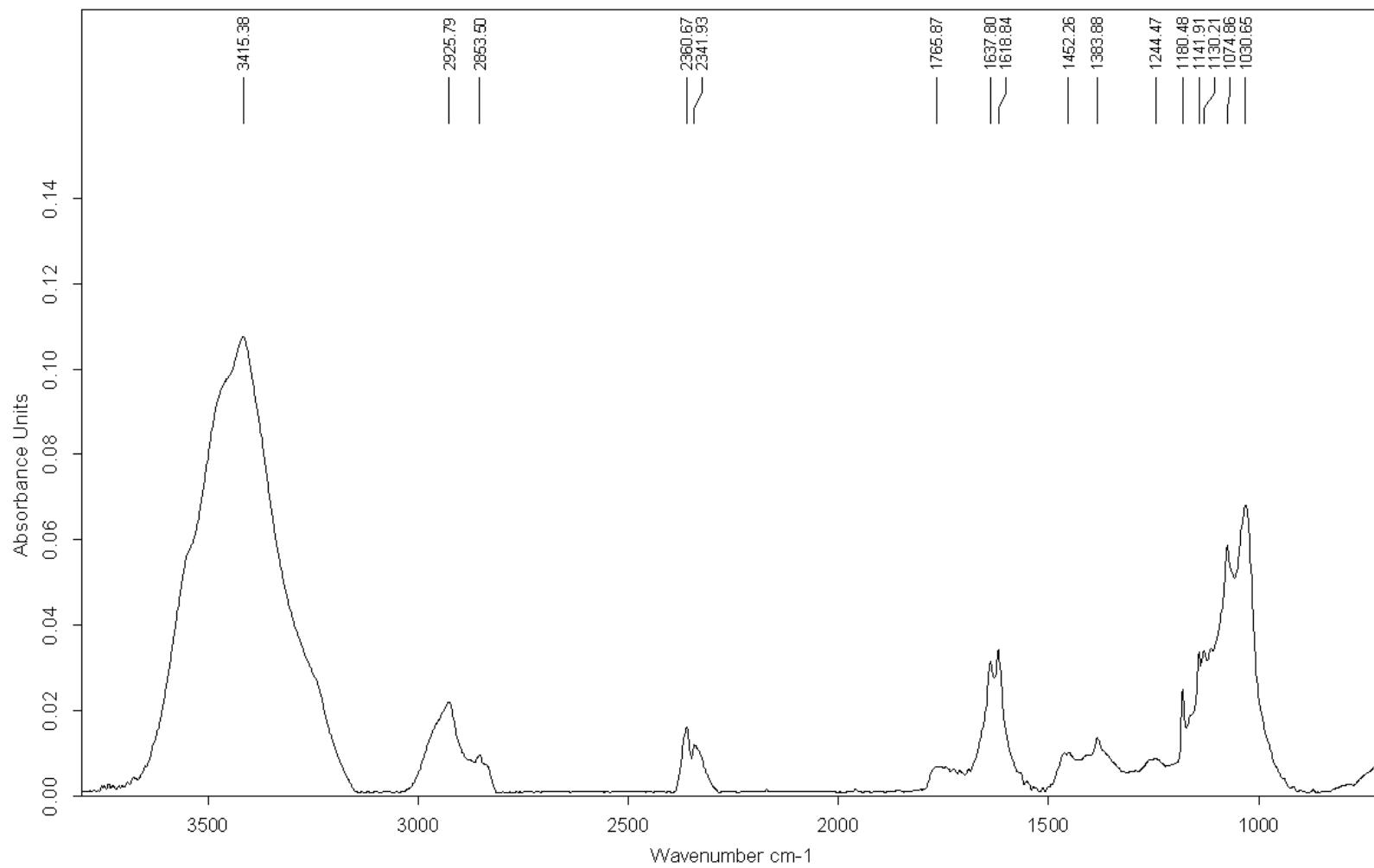
**Figure S54.** ROESY spectrum of pacificusoside H (**6**) in C<sub>5</sub>D<sub>5</sub>N.



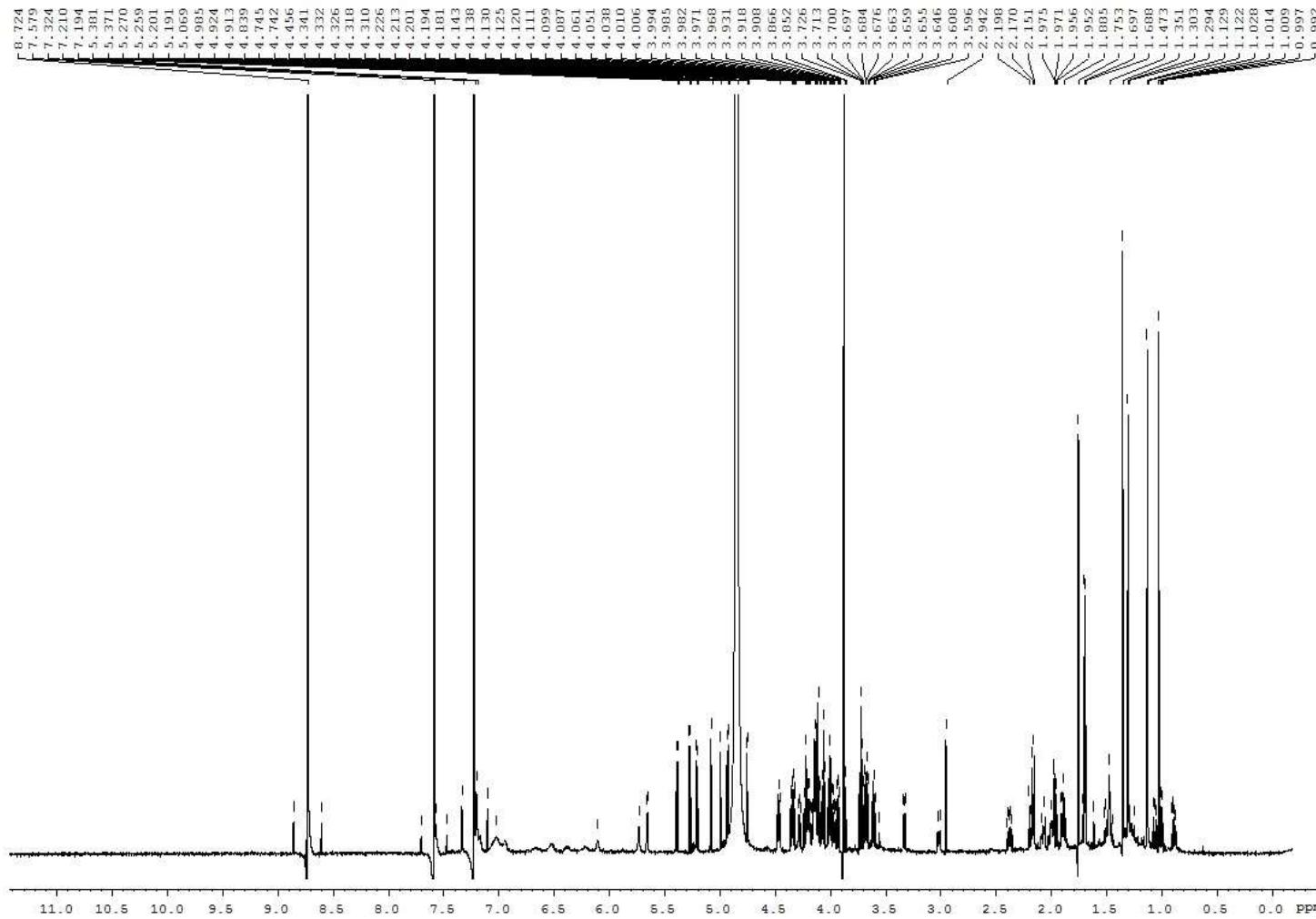
**Figure S55.** HRESIMS spectrum of pacificusoside I (7).



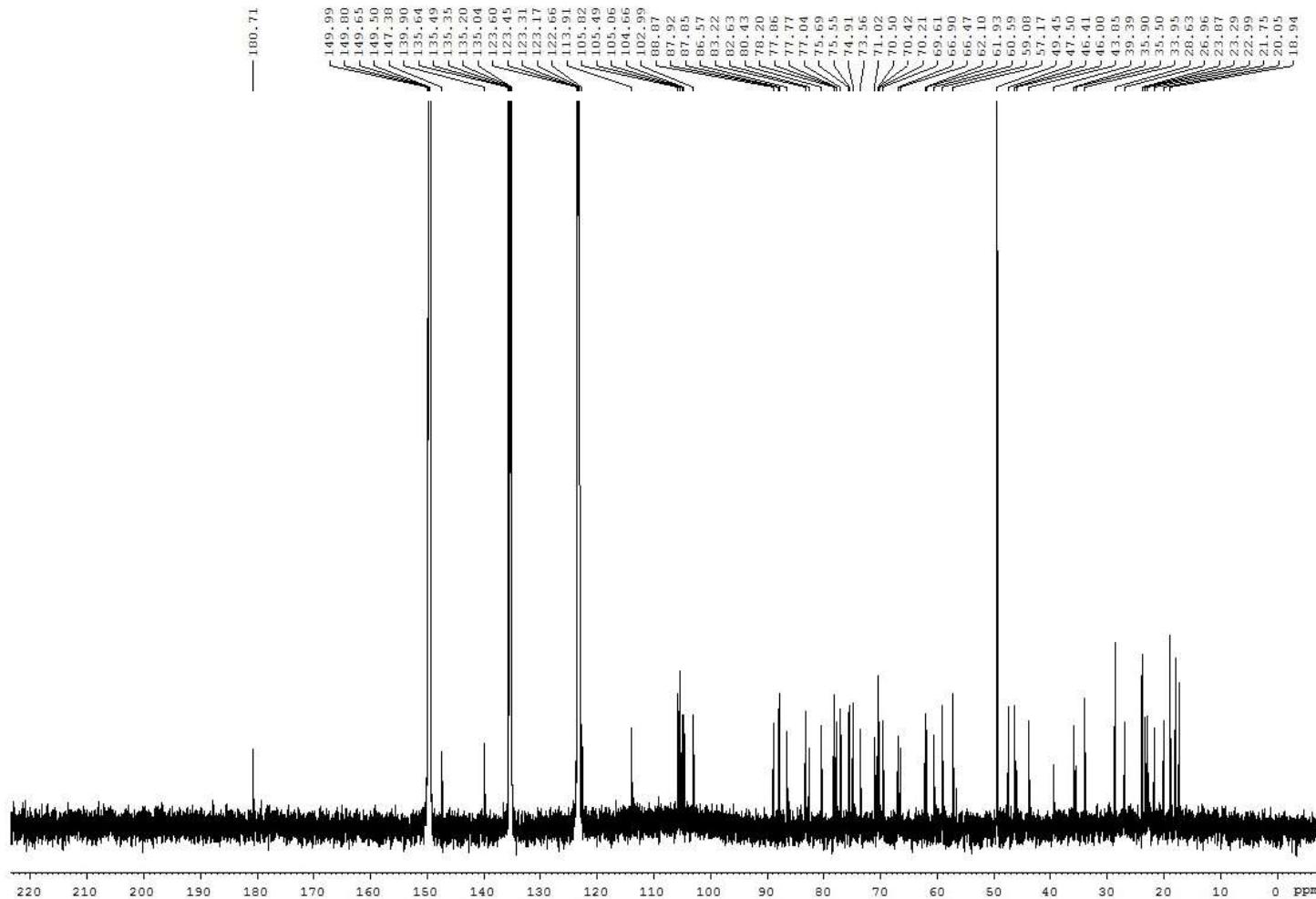
**Figure S56.** IR spectrum of pacificusoside I (7) in KBr.



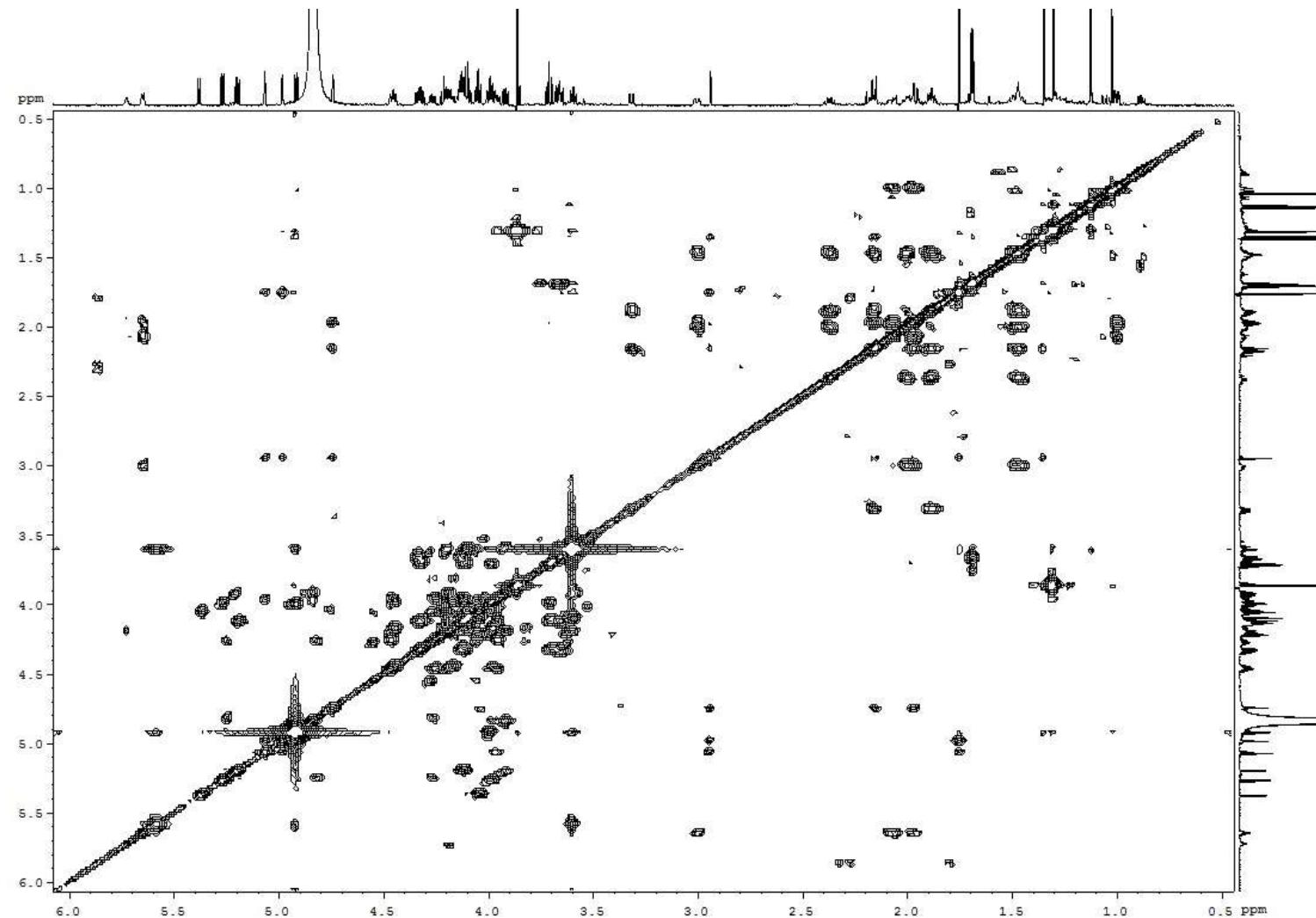
**Figure S57.**  $^1\text{H}$ -NMR spectrum of pacificusoside I (7) in  $\text{C}_5\text{D}_5\text{N}$ .



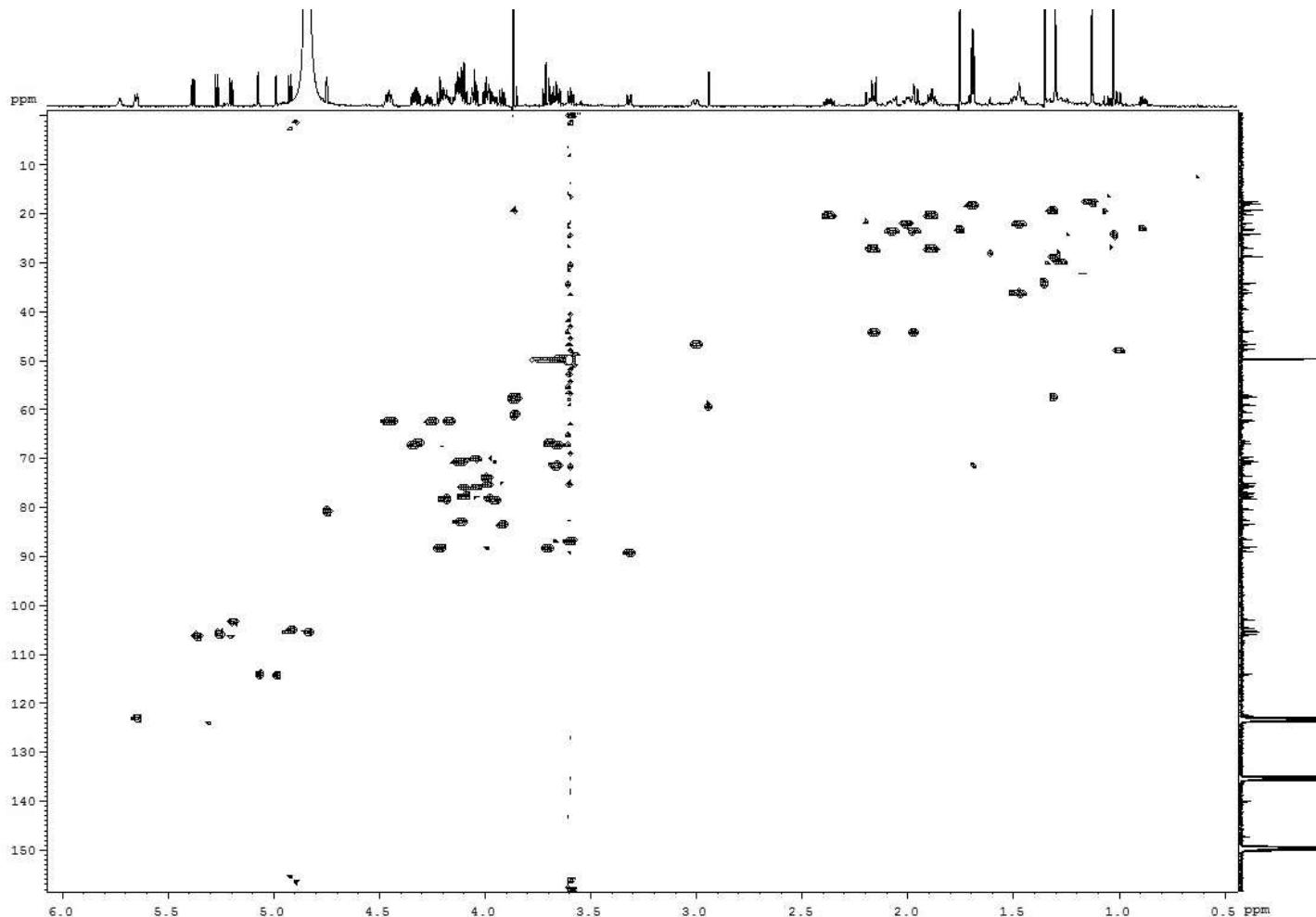
**Figure S58.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside I (7) in  $\text{C}_5\text{D}_5\text{N}$ .



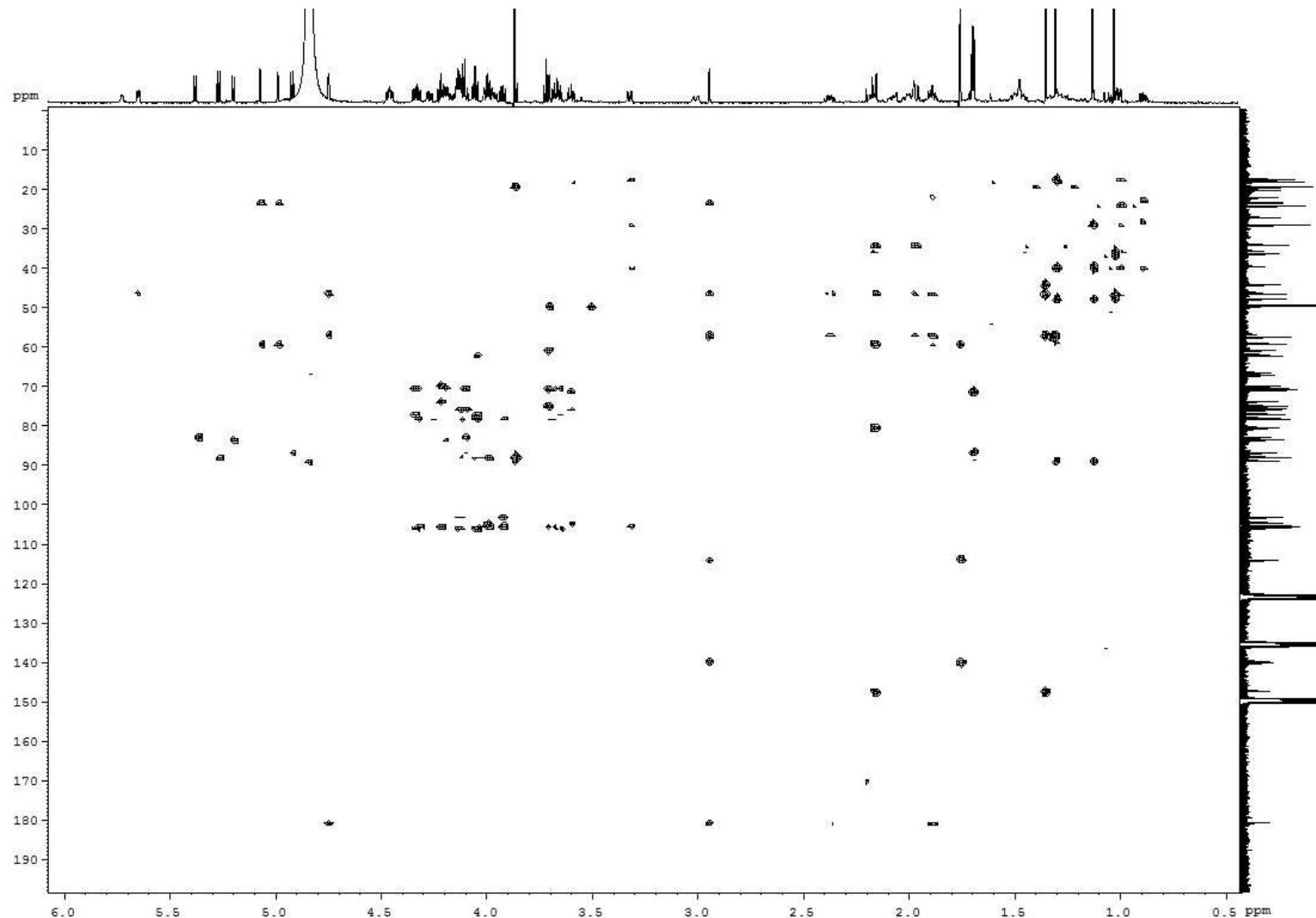
**Figure S59.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside I (7) in  $\text{C}_5\text{D}_5\text{N}$ .



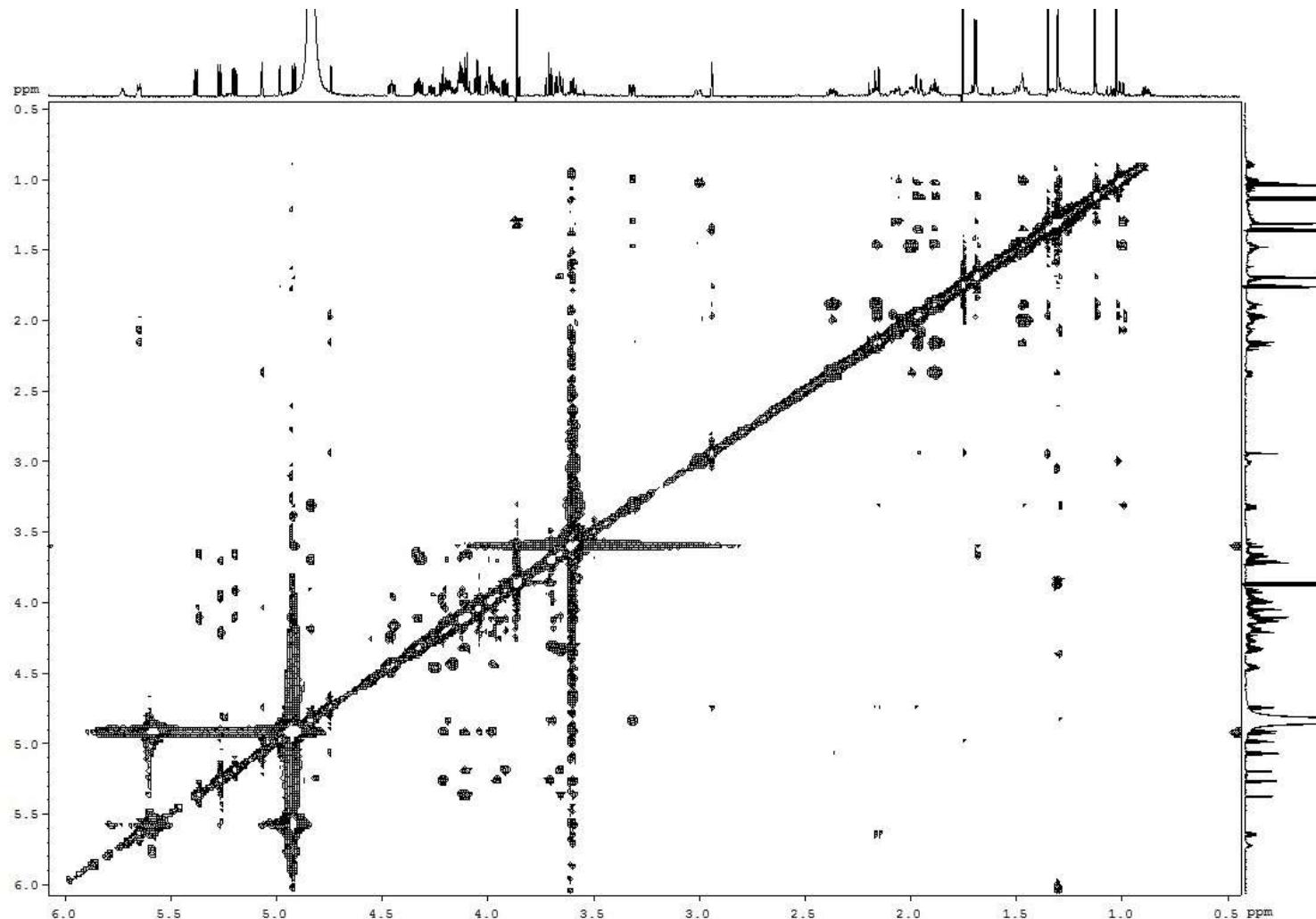
**Figure S60.** HSQC spectrum of pacificusoside I (7) in C<sub>5</sub>D<sub>5</sub>N.



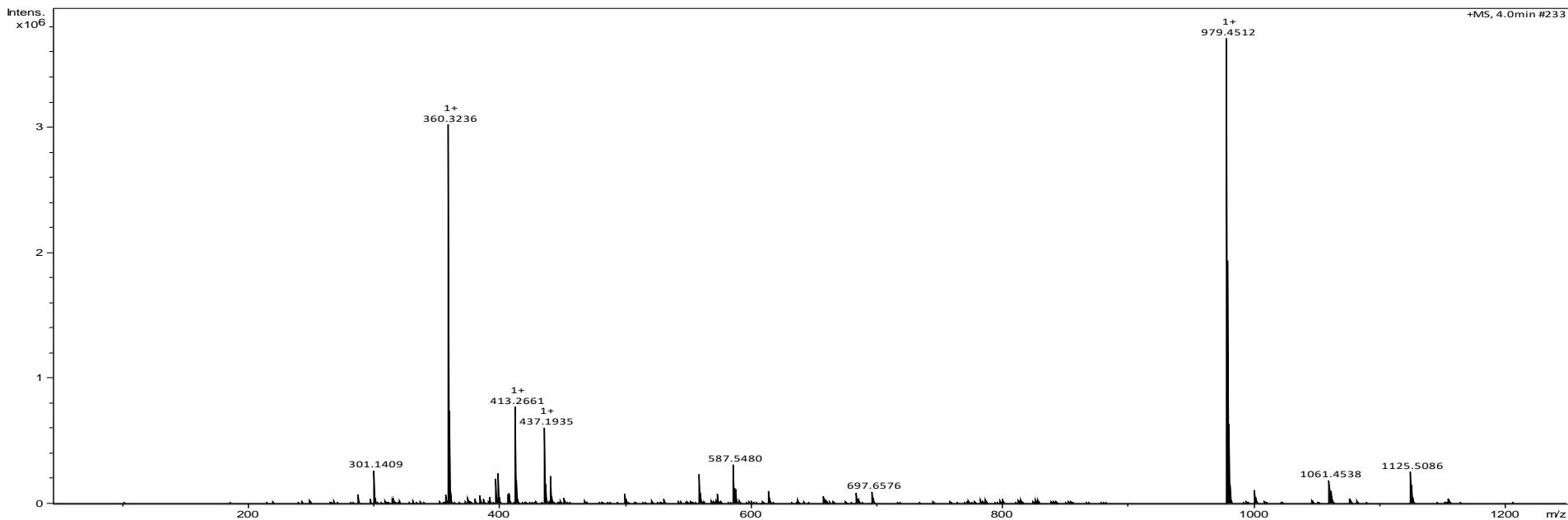
**Figure S61.** HMBC spectrum of pacificusoside I (7) in C<sub>5</sub>D<sub>5</sub>N.



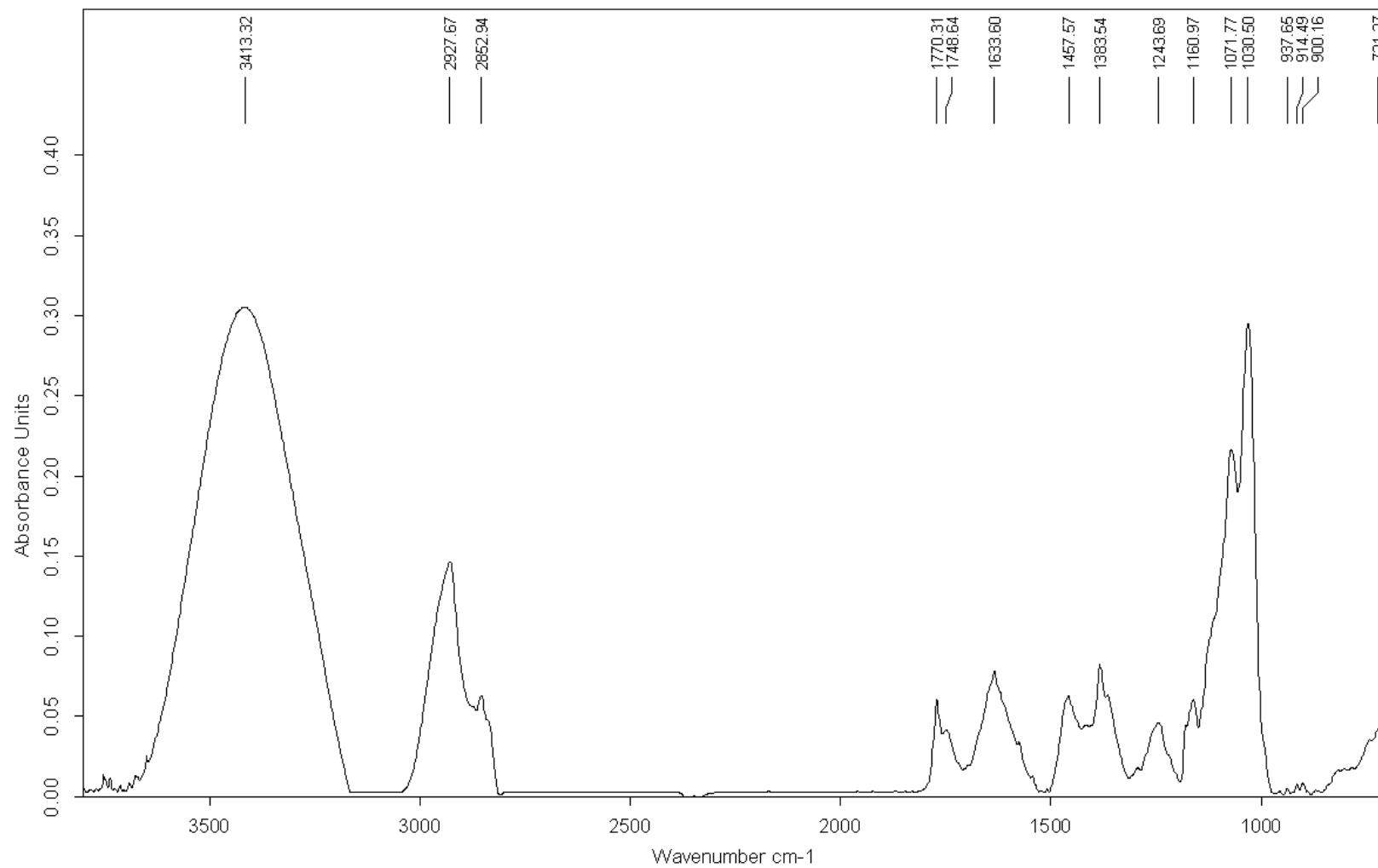
**Figure S62.** ROESY spectrum of pacificusoside I (7) in C<sub>5</sub>D<sub>5</sub>N.



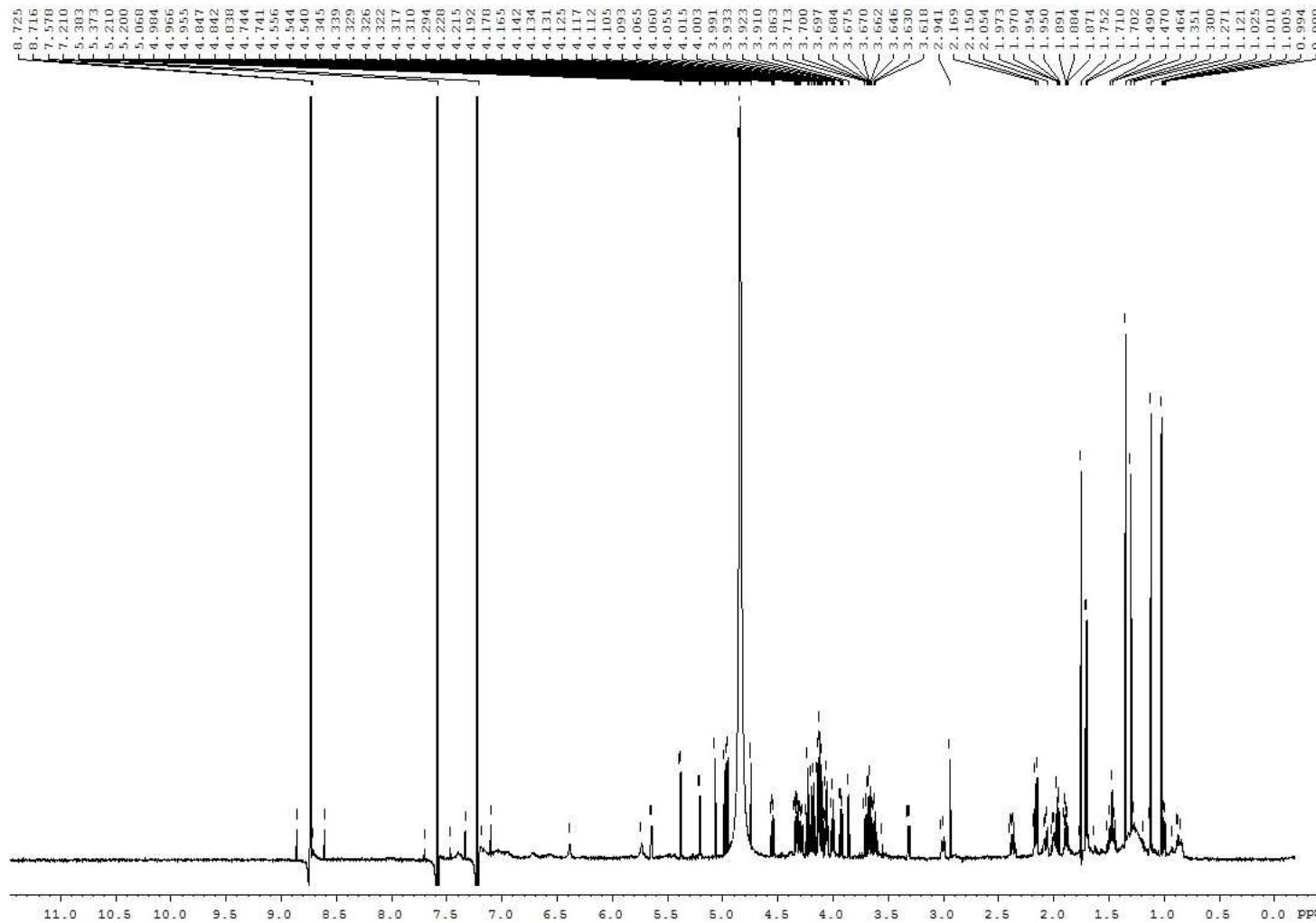
**Figure S63.** HRESIMS spectrum of pacificusoside J (8).



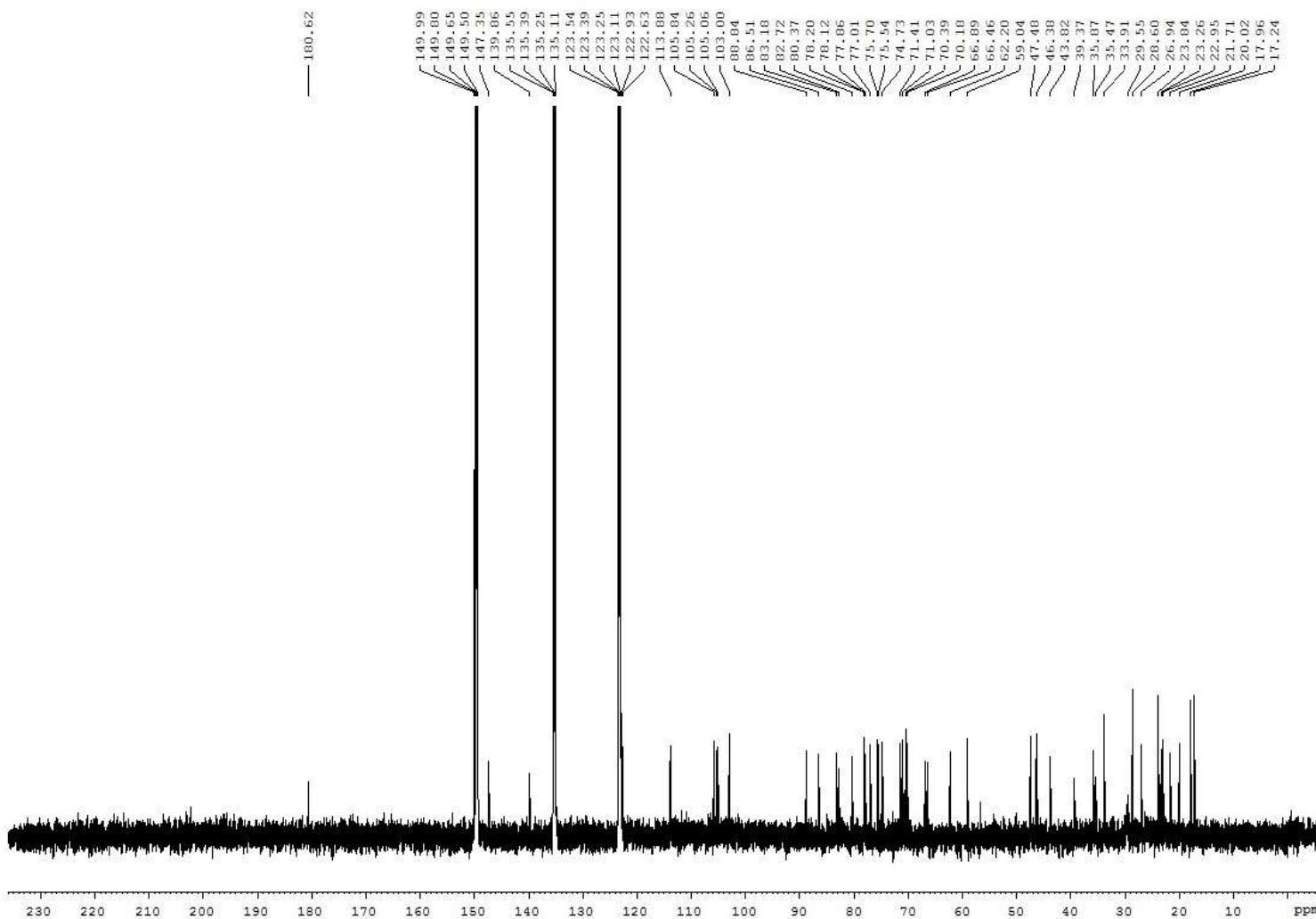
**Figure S64.** IR spectrum of pacificusoside J (8) in KBr.



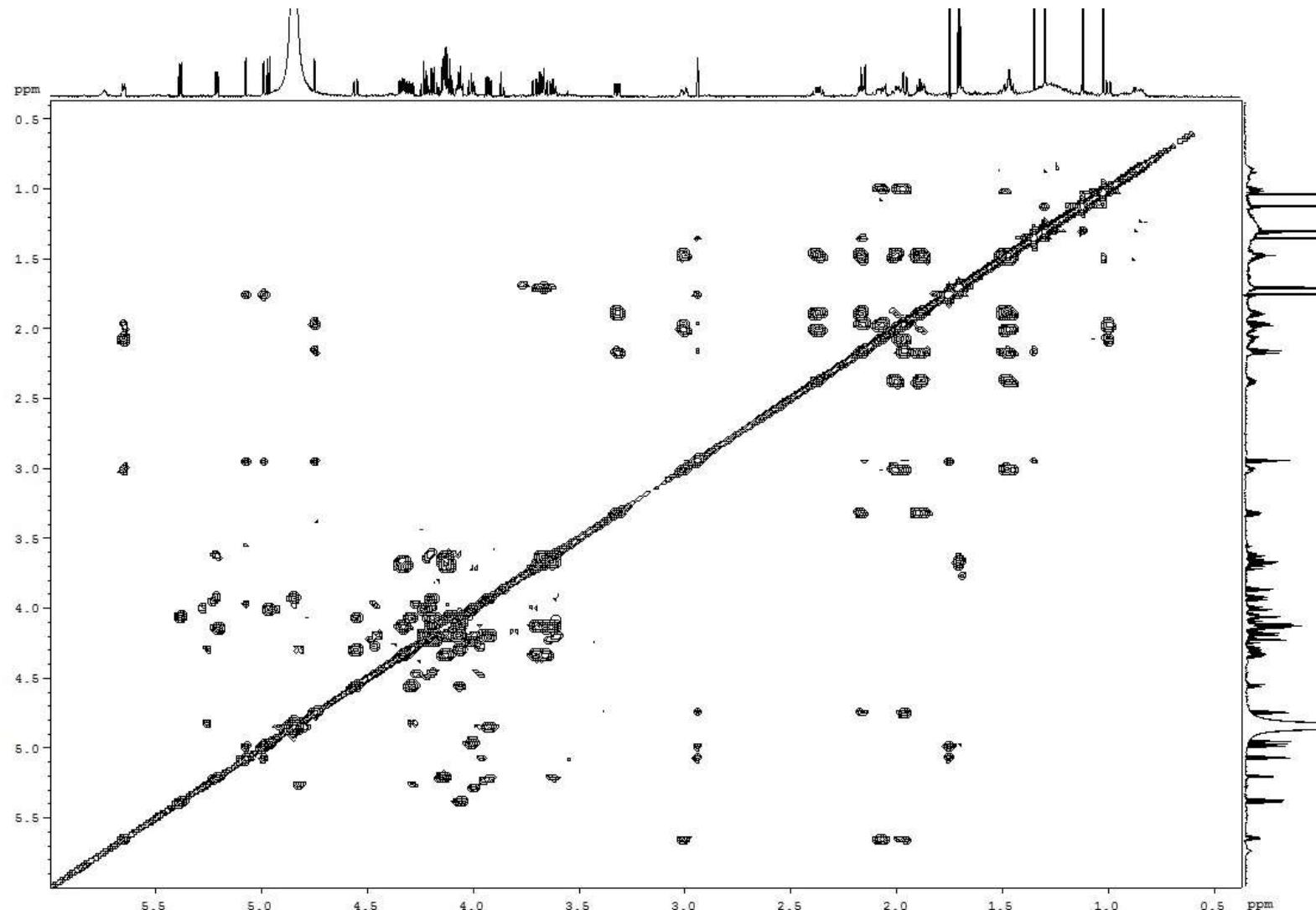
**Figure S65.**  $^1\text{H}$ -NMR spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .



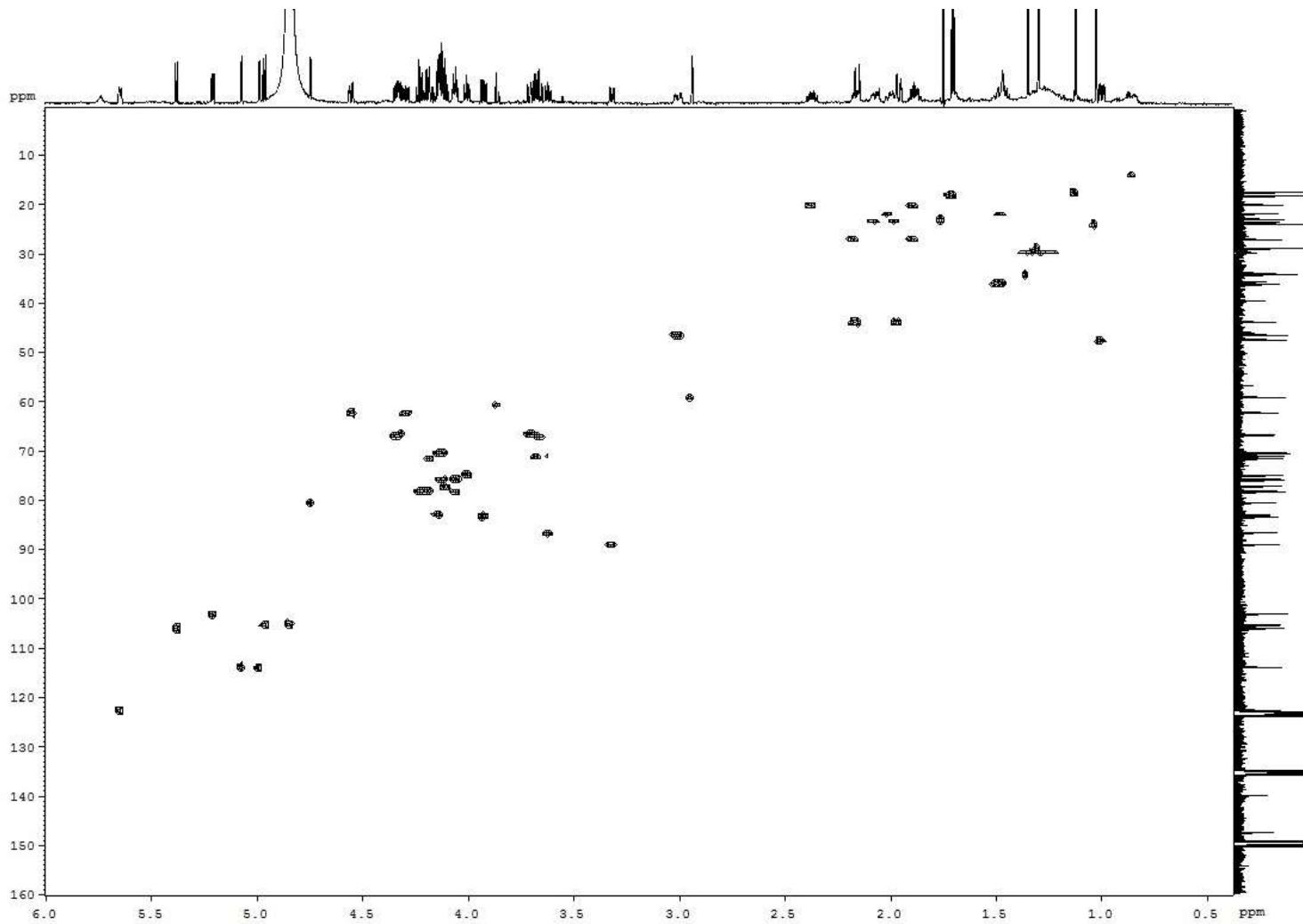
**Figure S66.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .



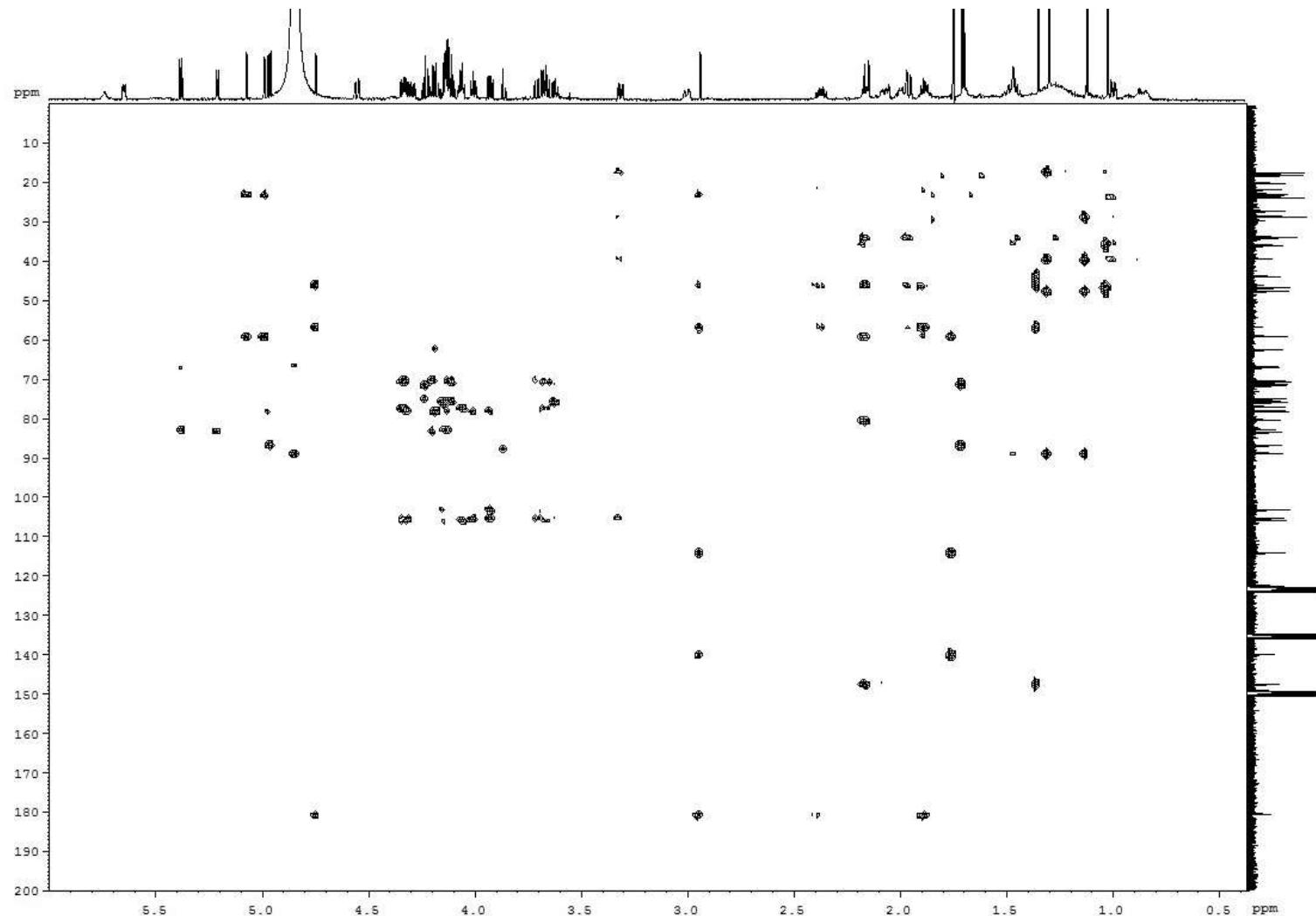
**Figure S67.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside J (8) in  $\text{C}_5\text{D}_5\text{N}$ .



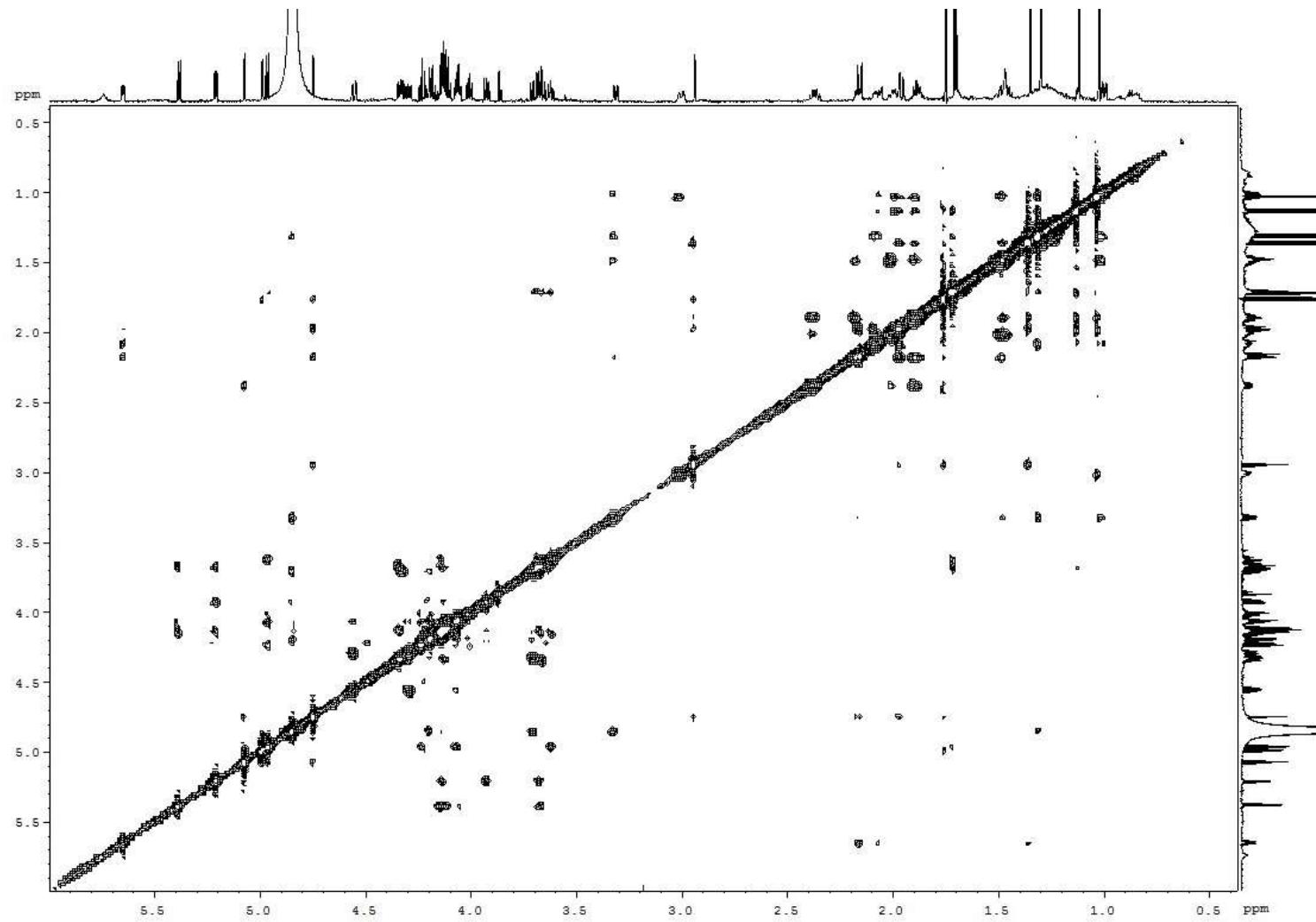
**Figure S68.** HSQC spectrum of pacificusoside J (8) in C<sub>5</sub>D<sub>5</sub>N.



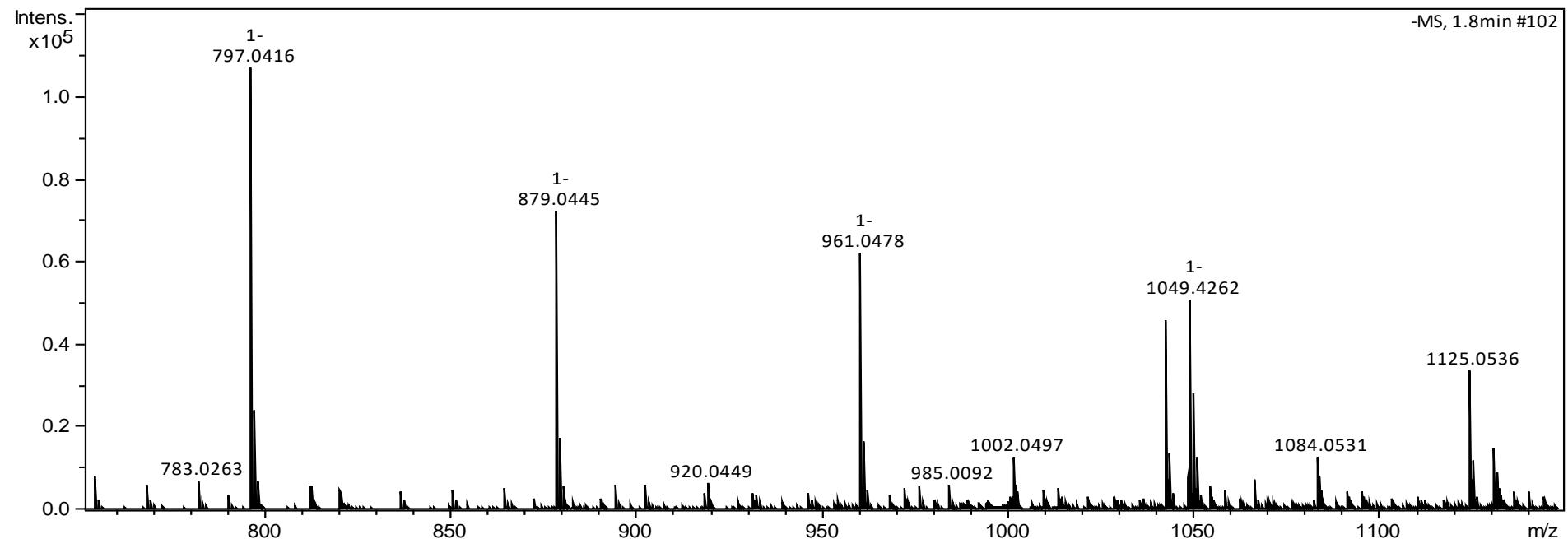
**Figure S69.** HMBC spectrum of pacificusoside J (8) in C<sub>5</sub>D<sub>5</sub>N.



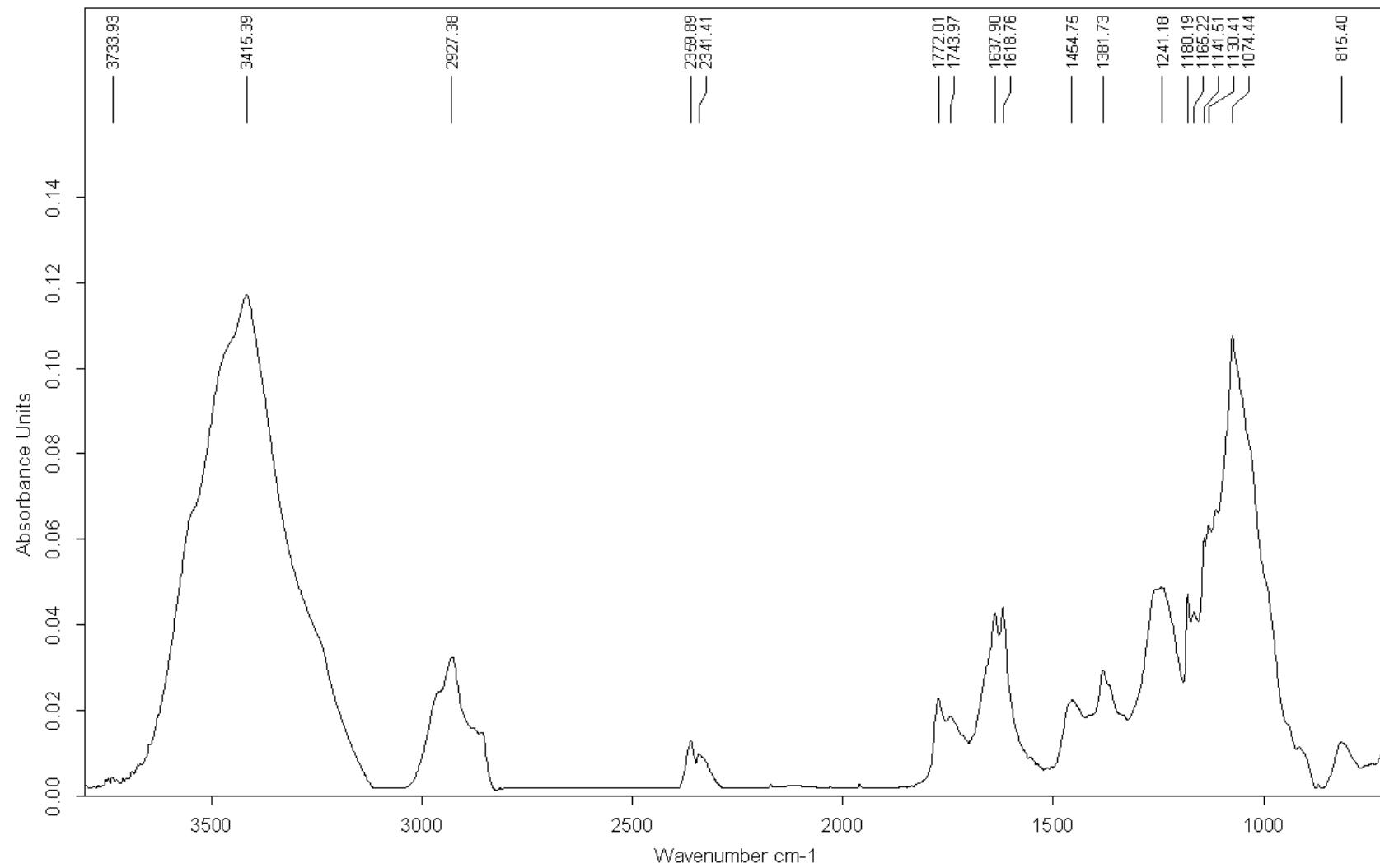
**Figure S70.** ROESY spectrum of pacificusoside J (8) in C<sub>5</sub>D<sub>5</sub>N.



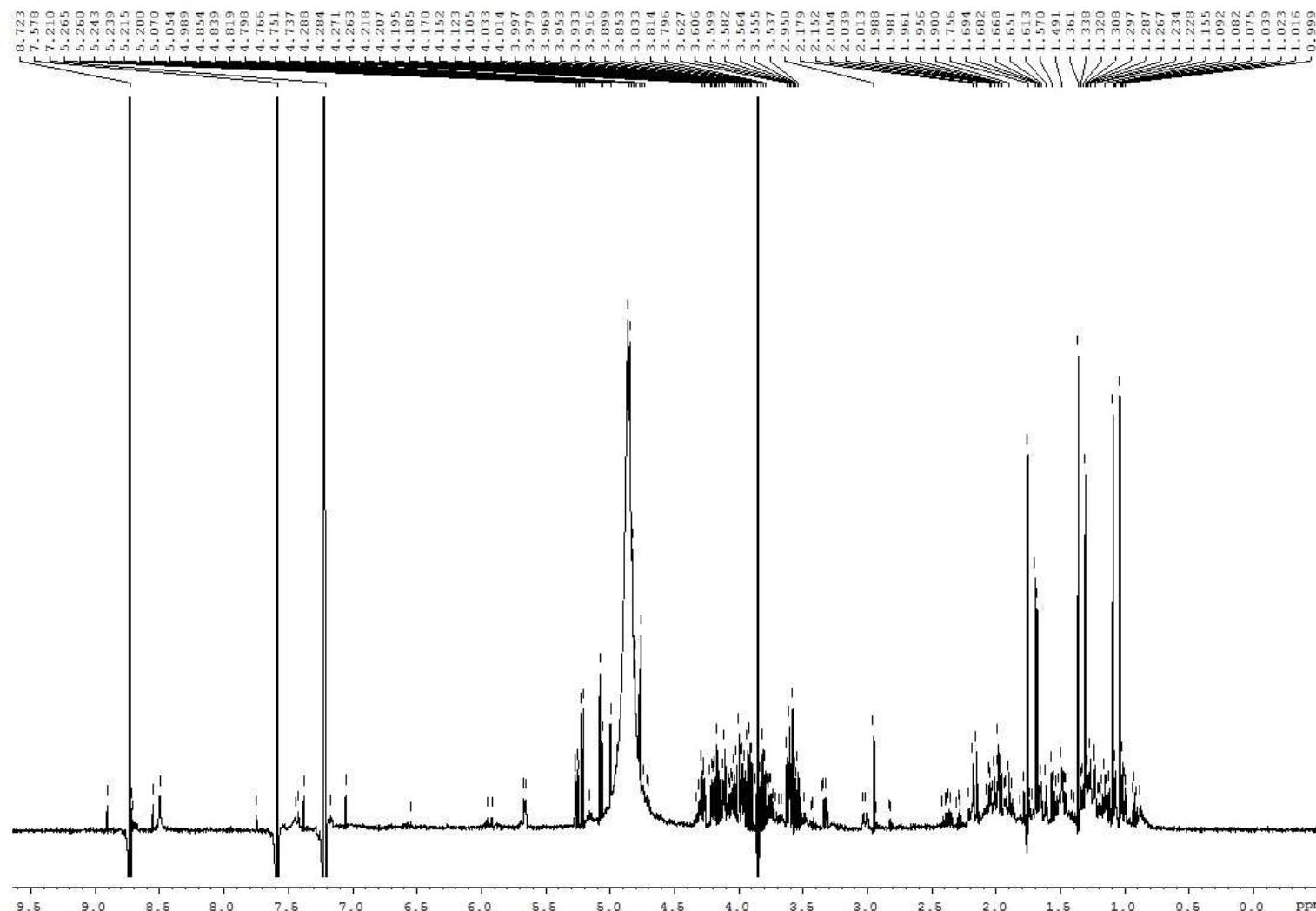
**Figure S71.** HRESIMS spectrum of pacificusoside K (9).



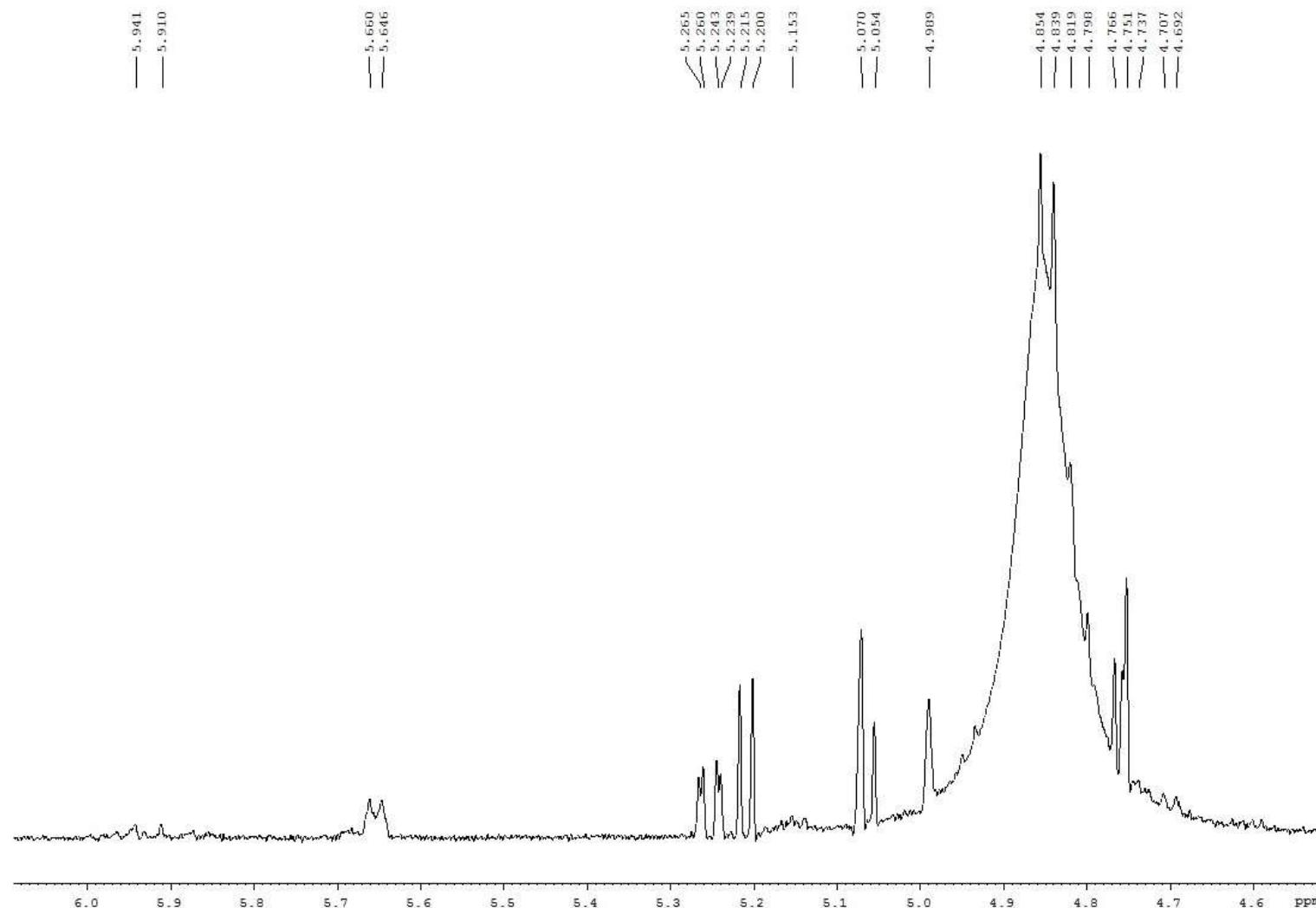
**Figure S72.** IR spectrum of pacificusoside K (9) in KBr.



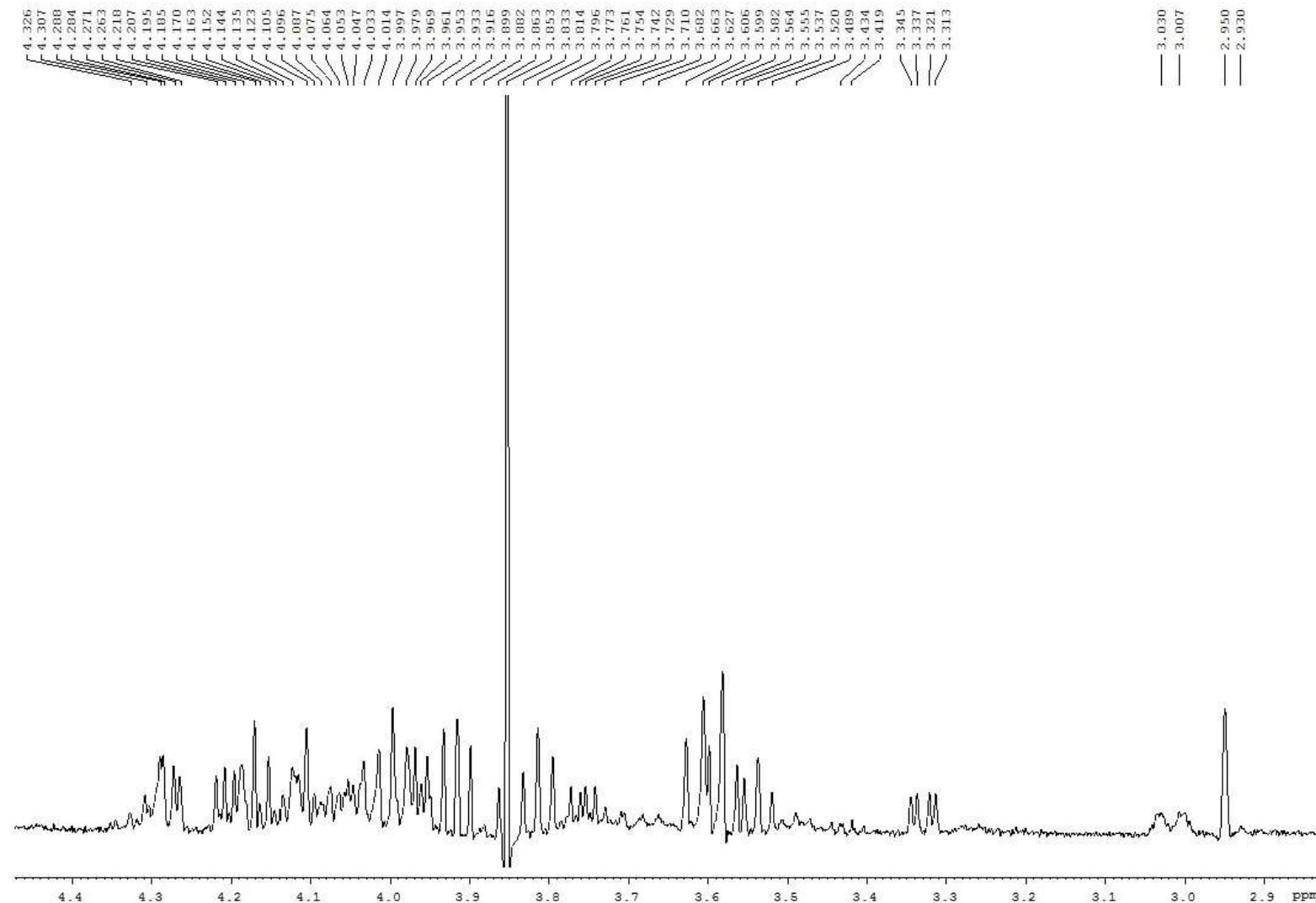
**Figure S73.**  $^1\text{H}$ -NMR spectrum of pacificusoside K (**9**) in  $\text{C}_5\text{D}_5\text{N}$ .



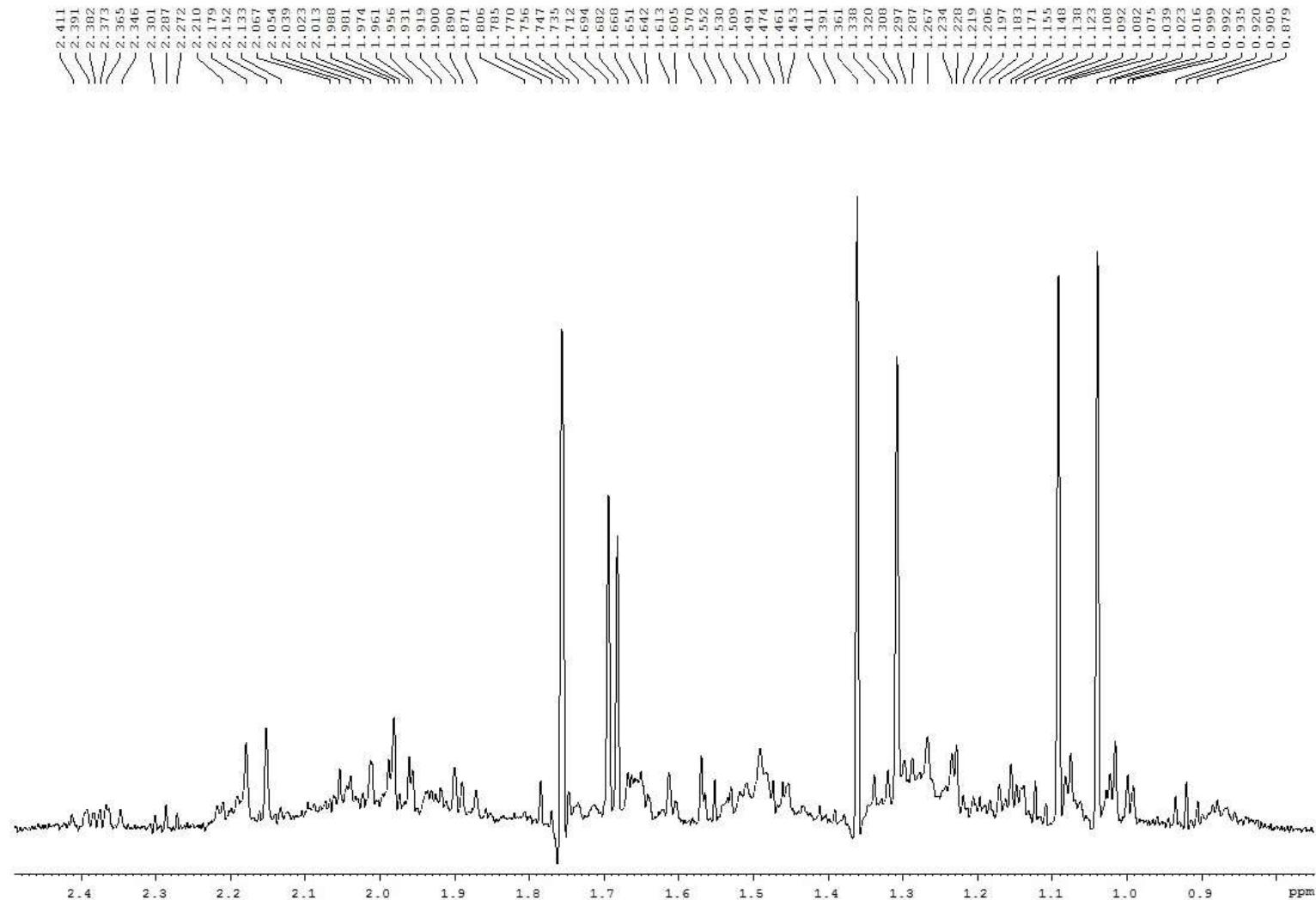
**Figure S74.** Expansion №1 of  $^1\text{H}$ -NMR spectrum of pacificusoside K (**9**) in  $\text{C}_5\text{D}_5\text{N}$ .



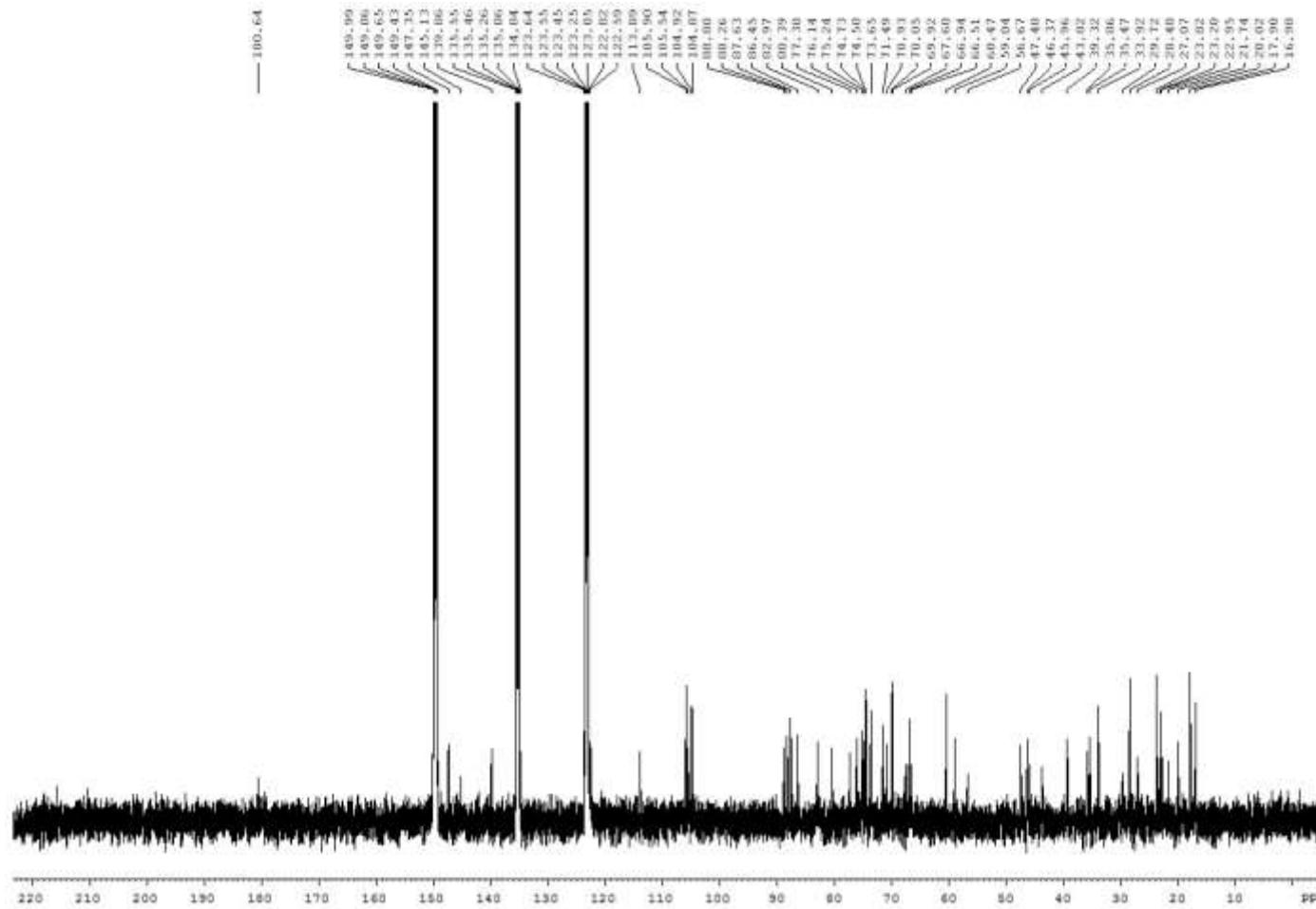
**Figure S75.** Expansion №2 of  $^1\text{H}$ -NMR spectrum of pacificusoside K (**9**) in  $\text{C}_5\text{D}_5\text{N}$ .



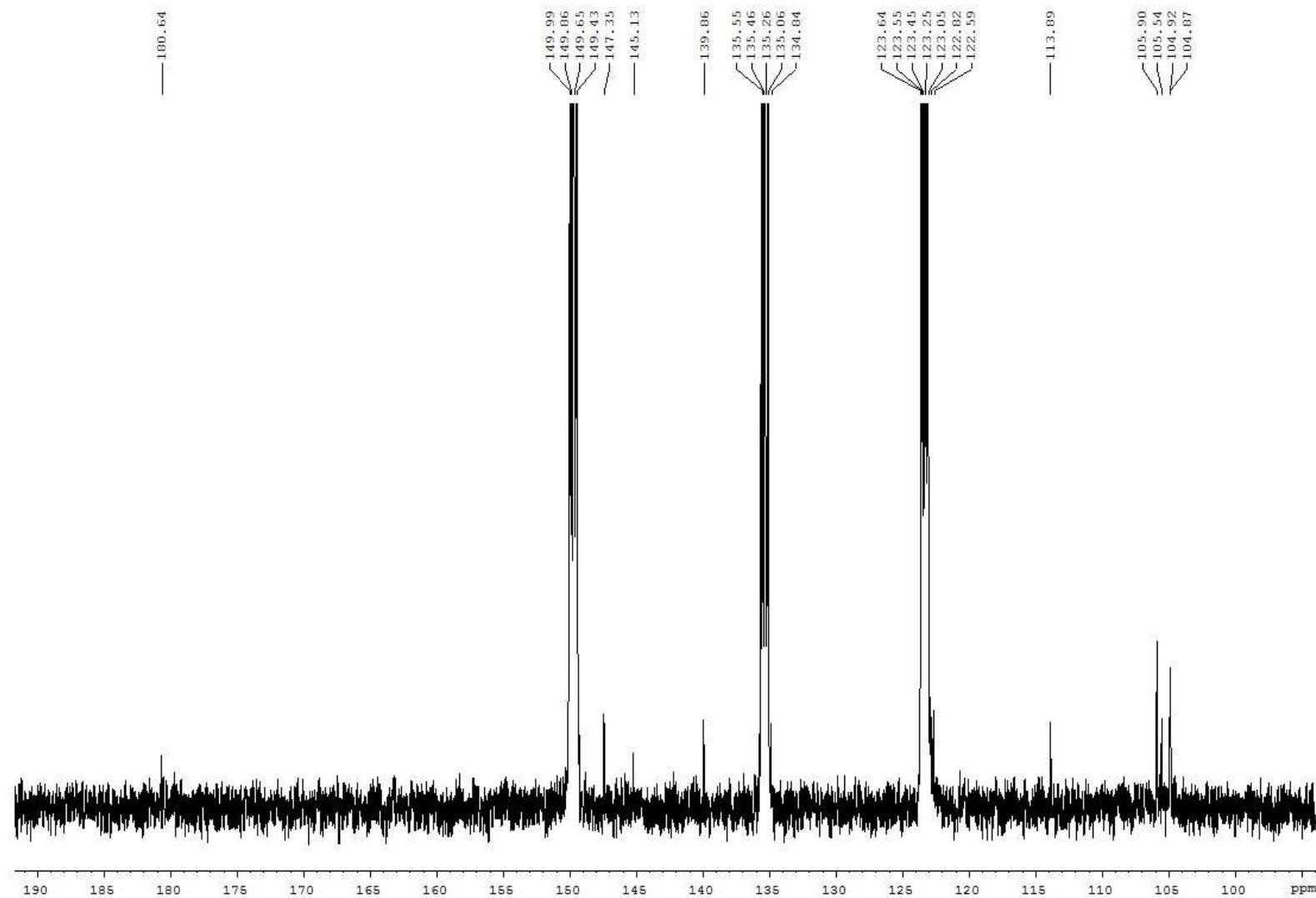
**Figure S76.** Expansion №3 of  $^1\text{H}$ -NMR spectrum of pacificusoside K (**9**) in  $\text{C}_5\text{D}_5\text{N}$ .



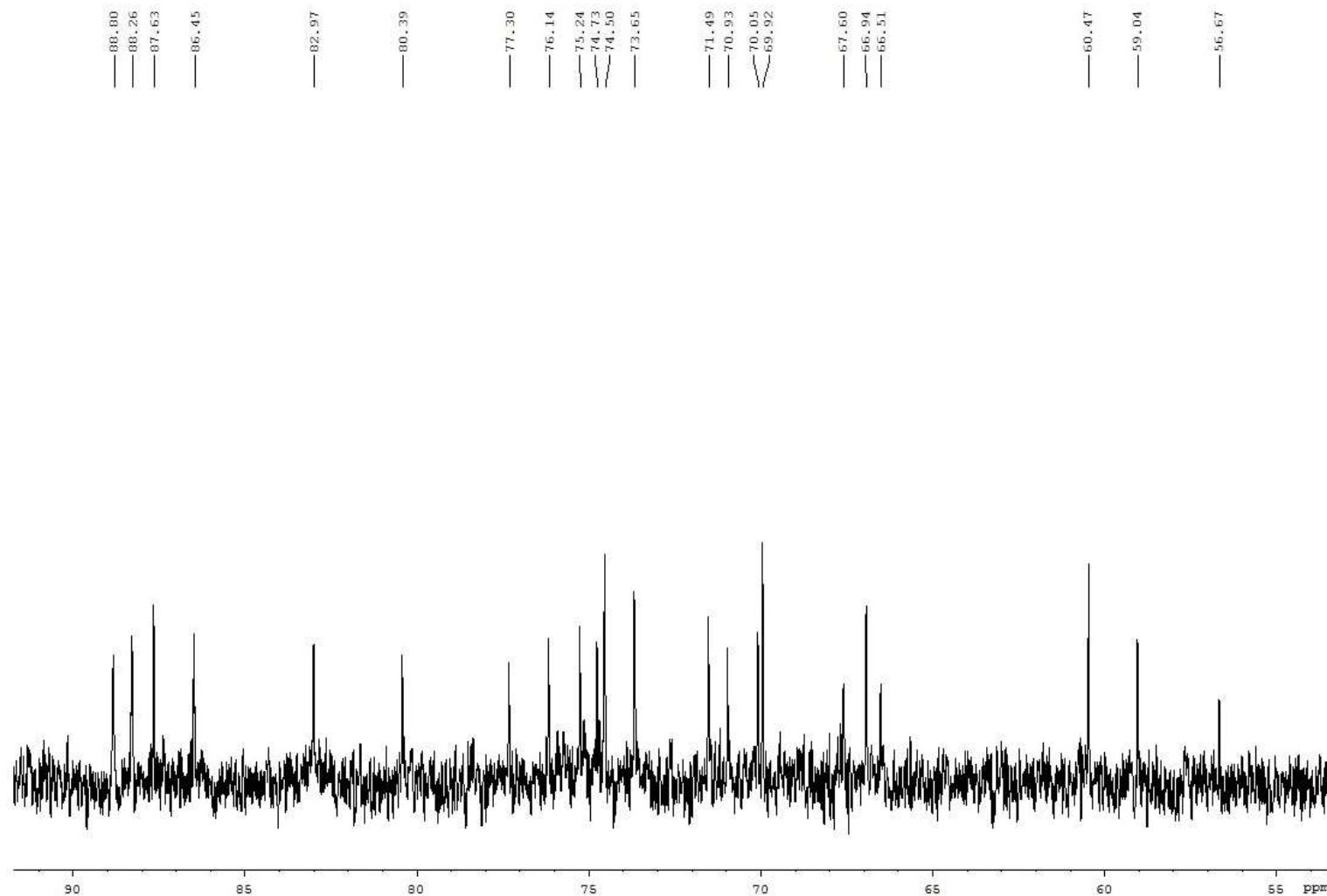
**Figure S77.**  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .



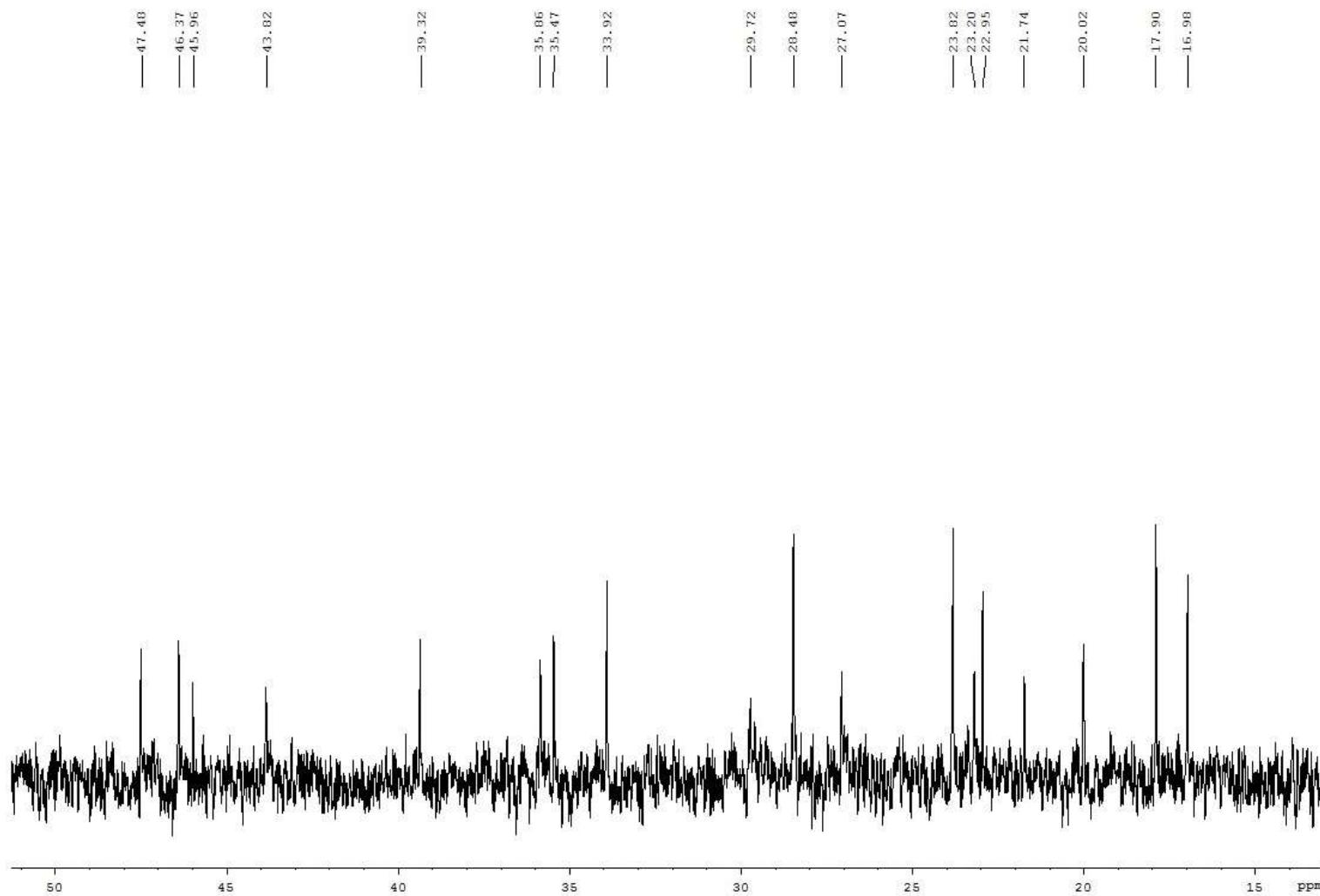
**Figure S78.** Expansion N°1 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .



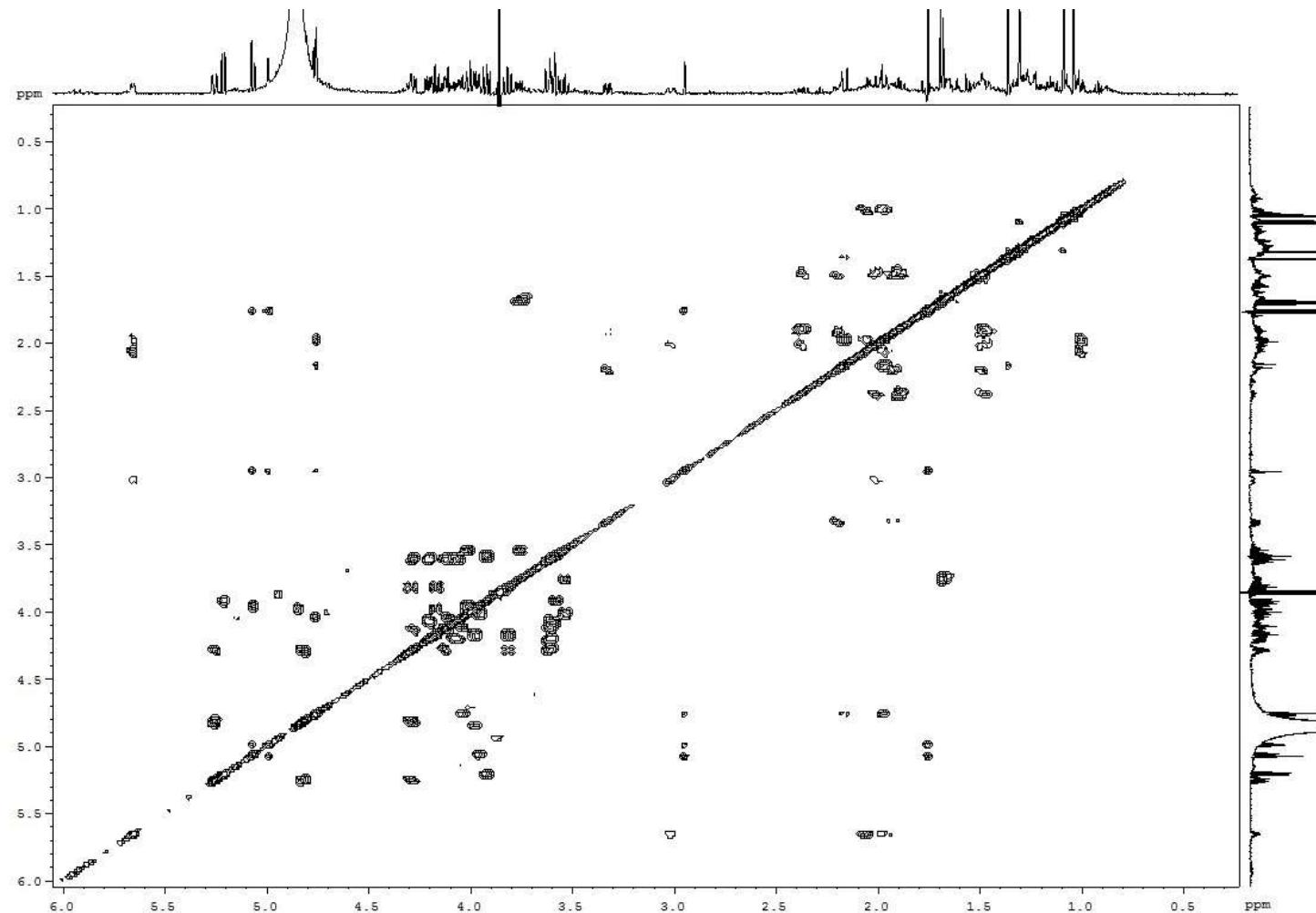
**Figure S79.** Expansion №2 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .



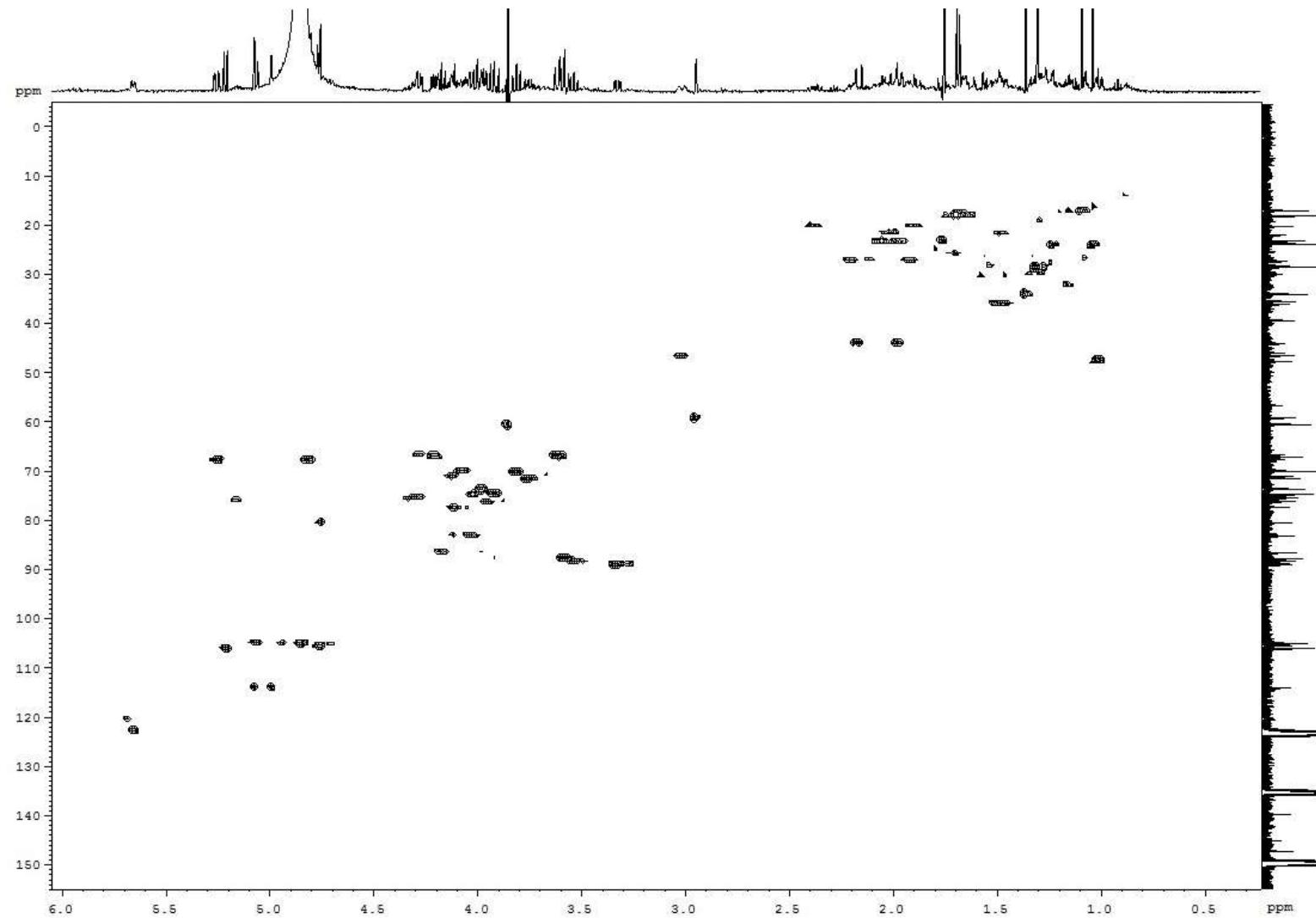
**Figure S80.** Expansion №3 of  $^{13}\text{C}$ -NMR spectrum of pacificusoside K (9) in  $\text{C}_5\text{D}_5\text{N}$ .



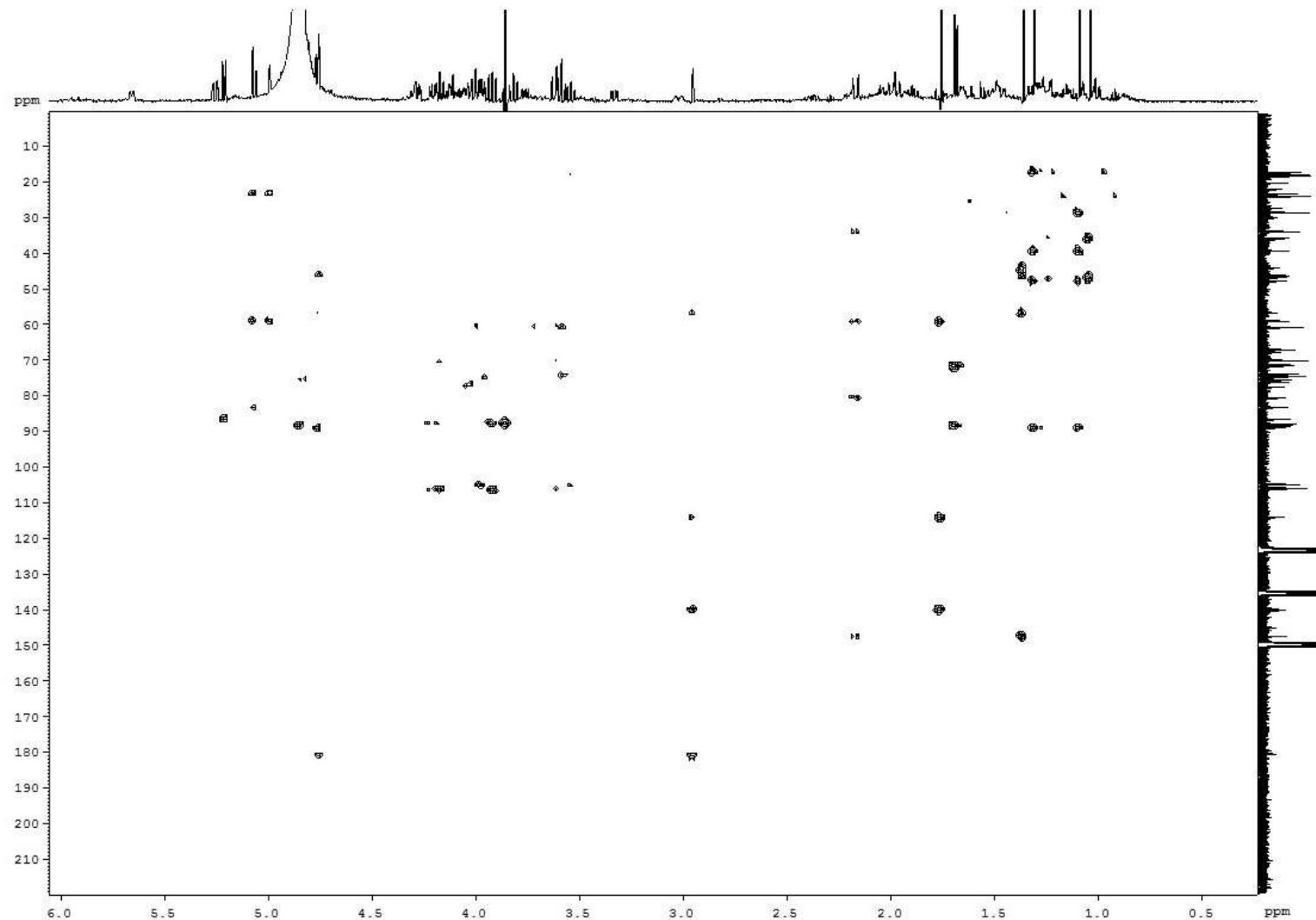
**Figure S81.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of pacificusoside K (**9**) in  $\text{C}_5\text{D}_5\text{N}$ .



**Figure S82.** HSQC spectrum of pacificusoside K (**9**) in C<sub>5</sub>D<sub>5</sub>N.



**Figure S83.** HMBC spectrum of pacificusoside K (**9**) in C<sub>5</sub>D<sub>5</sub>N.



**Figure S84.** ROESY spectrum of pacificusoside K (**9**) in C<sub>5</sub>D<sub>5</sub>N.

