

## Supplementary Materials

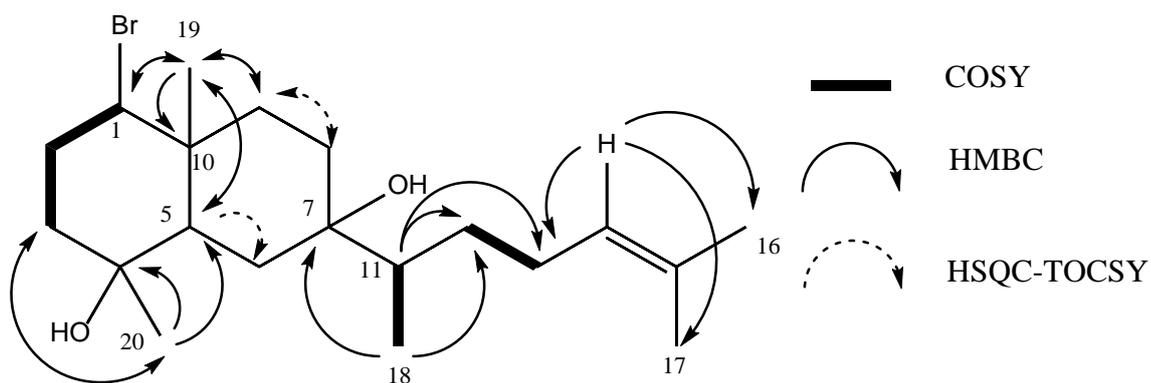


Figure S1. Key 2D NMR correlations of compound 1.

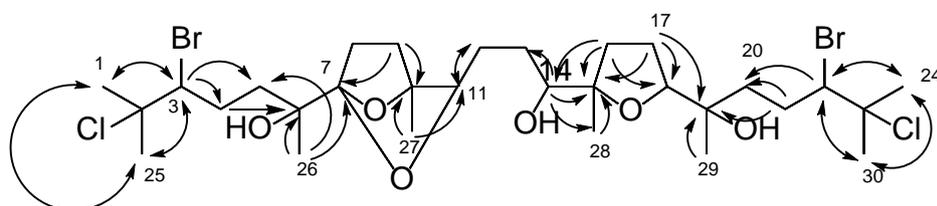


Figure S2. Key 2D NMR correlations of compound 2.

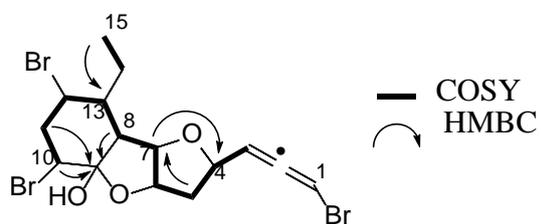


Figure S3. Key 2D NMR correlations of compound 3.

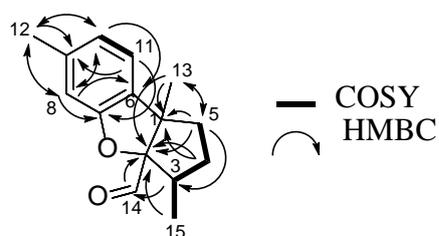


Figure S4. Key 2D NMR correlations of compound 5.

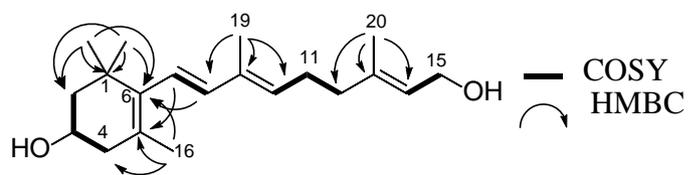


Figure S5. Key 2D NMR correlations of compound 6.

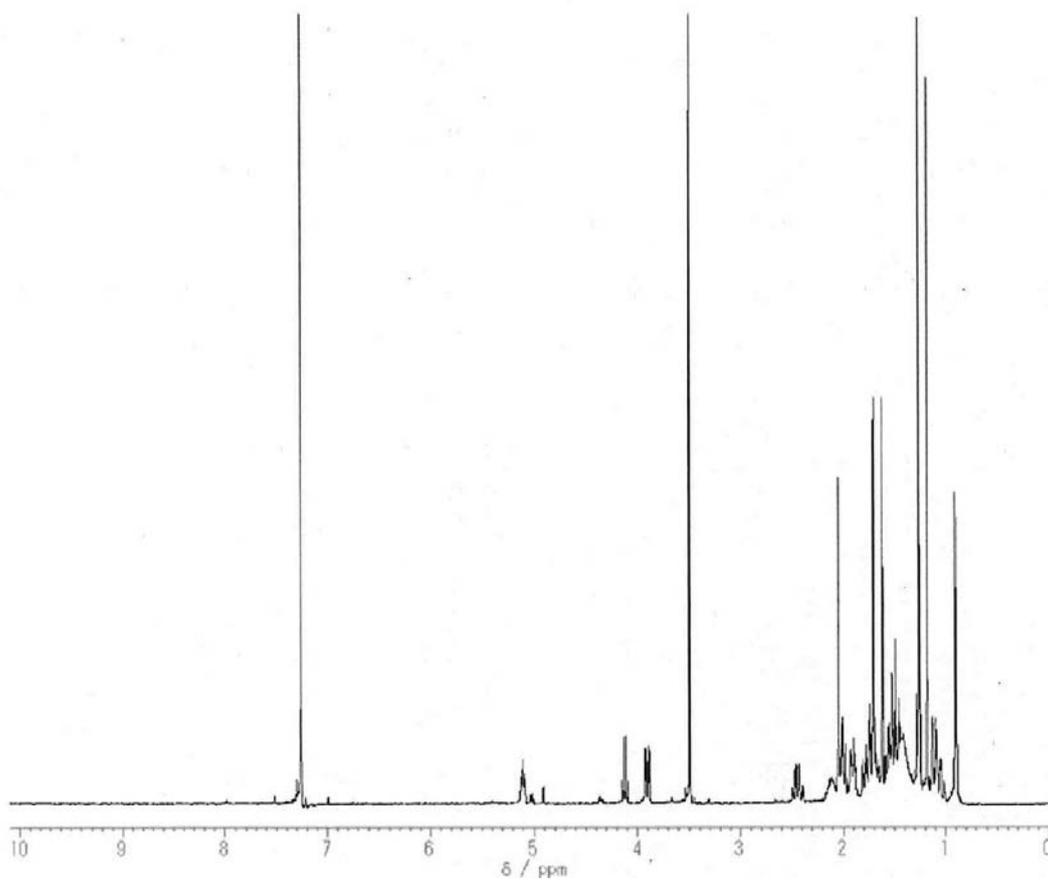
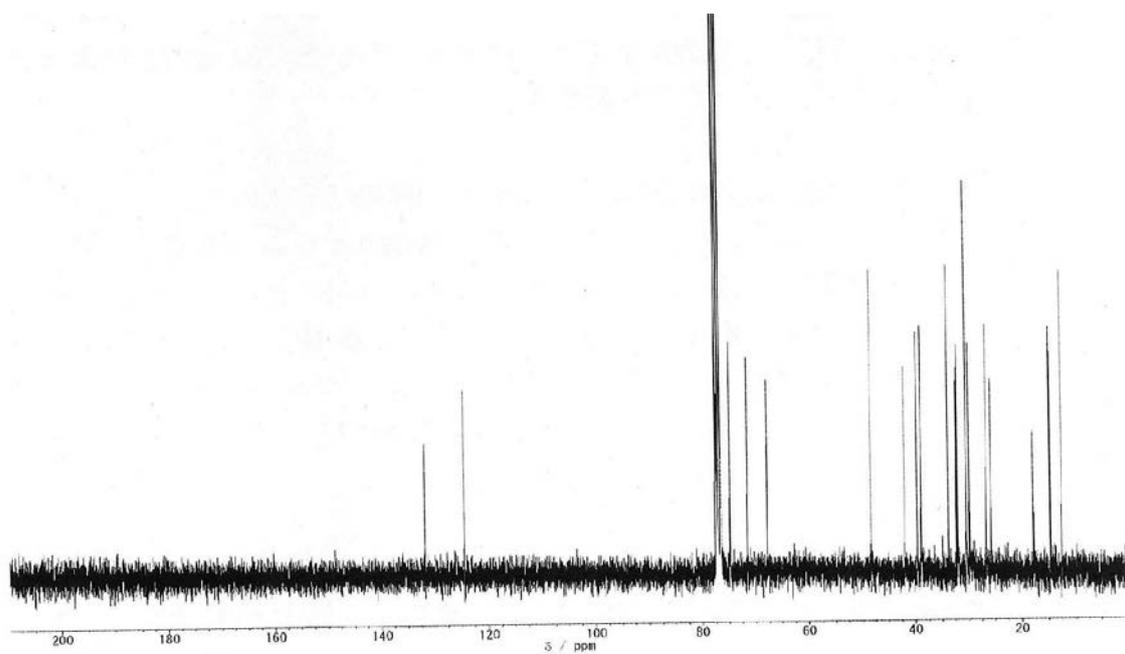
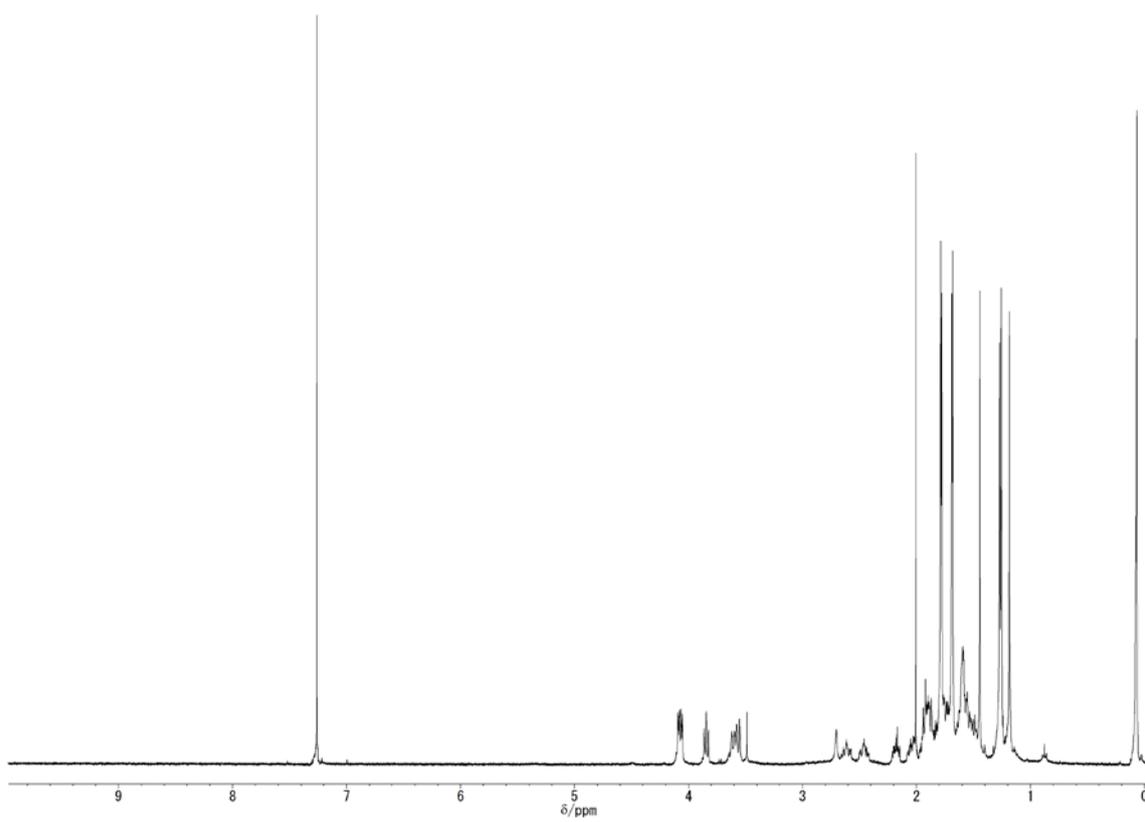


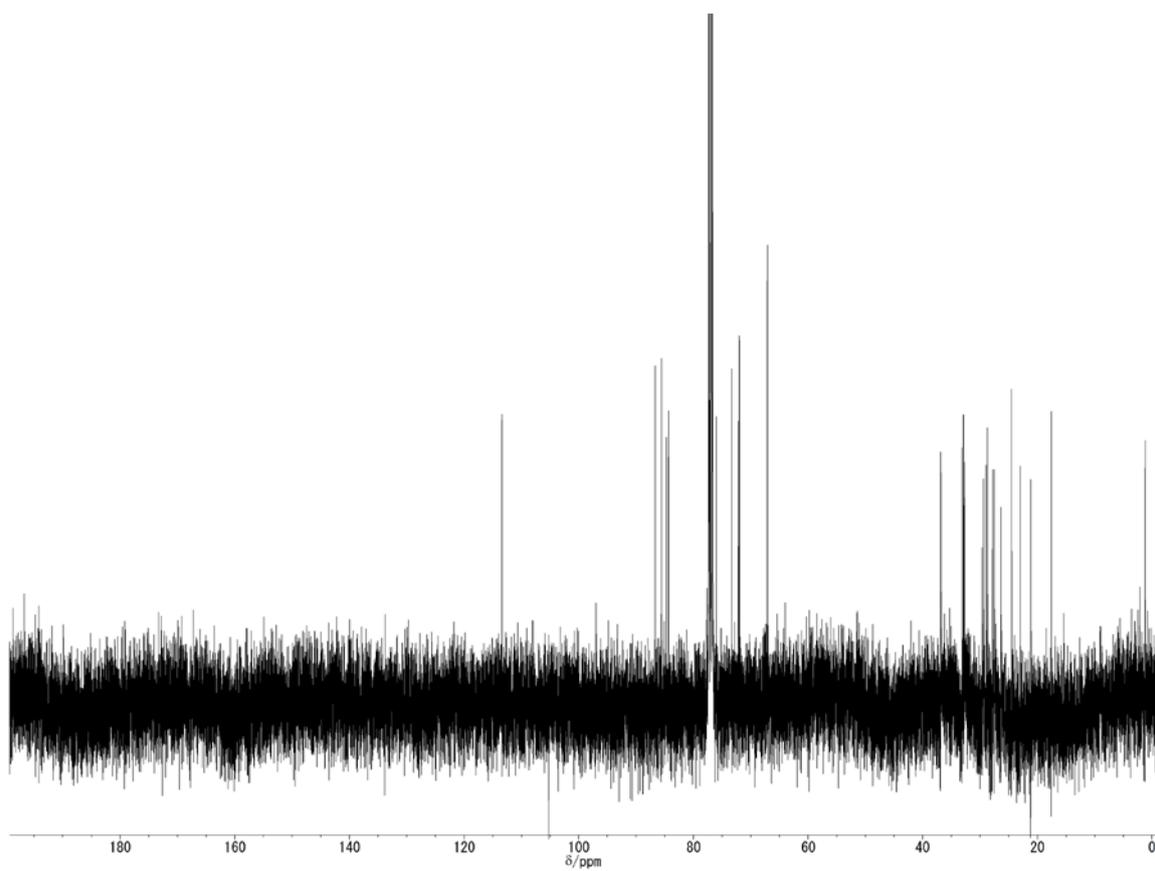
Figure S6.  $^1\text{H}$  NMR spectrum of omaezol (1) in  $\text{CDCl}_3$ .



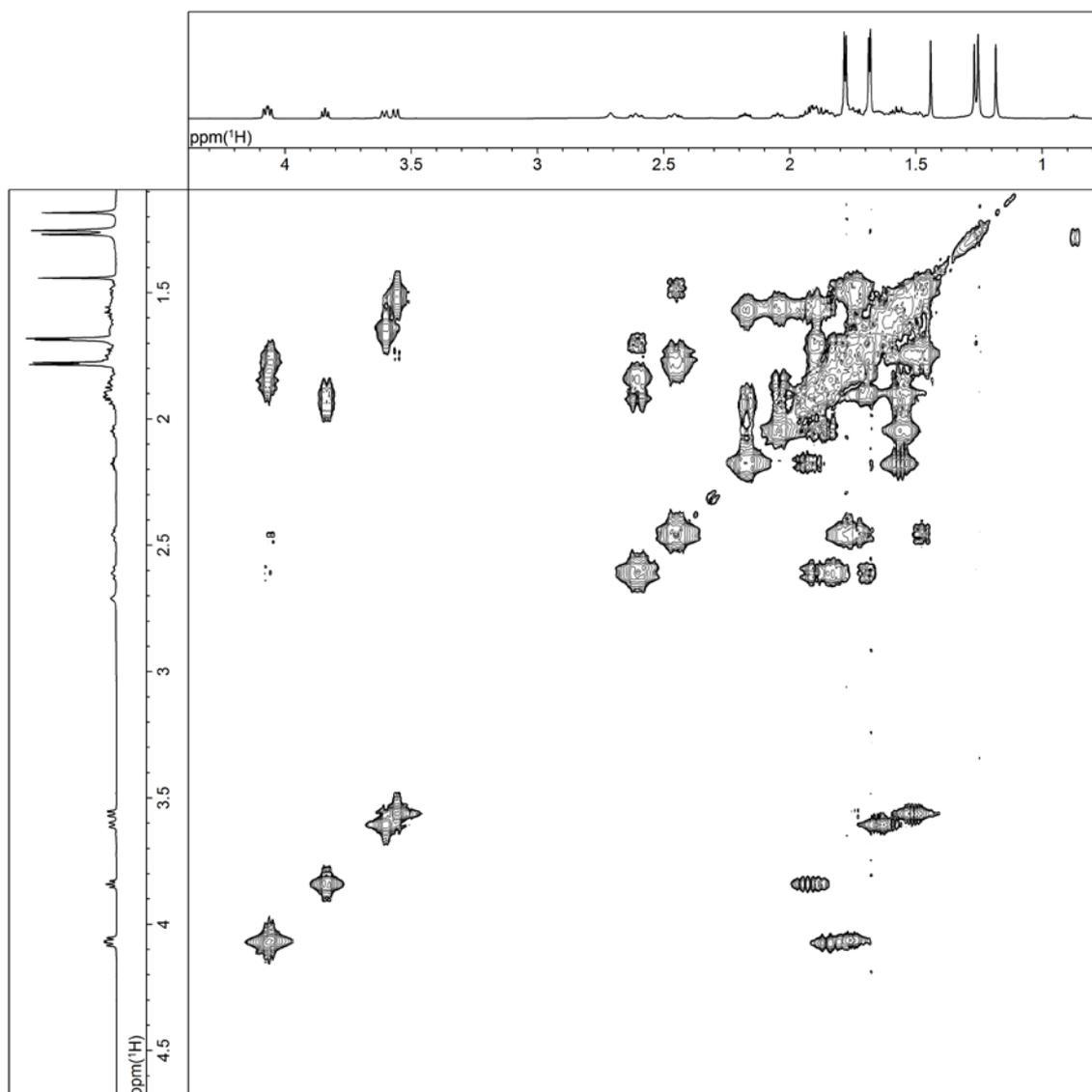
**Figure S7.**  $^{13}\text{C}$  NMR spectrum of omaezol (**1**) in  $\text{CDCl}_3$ .



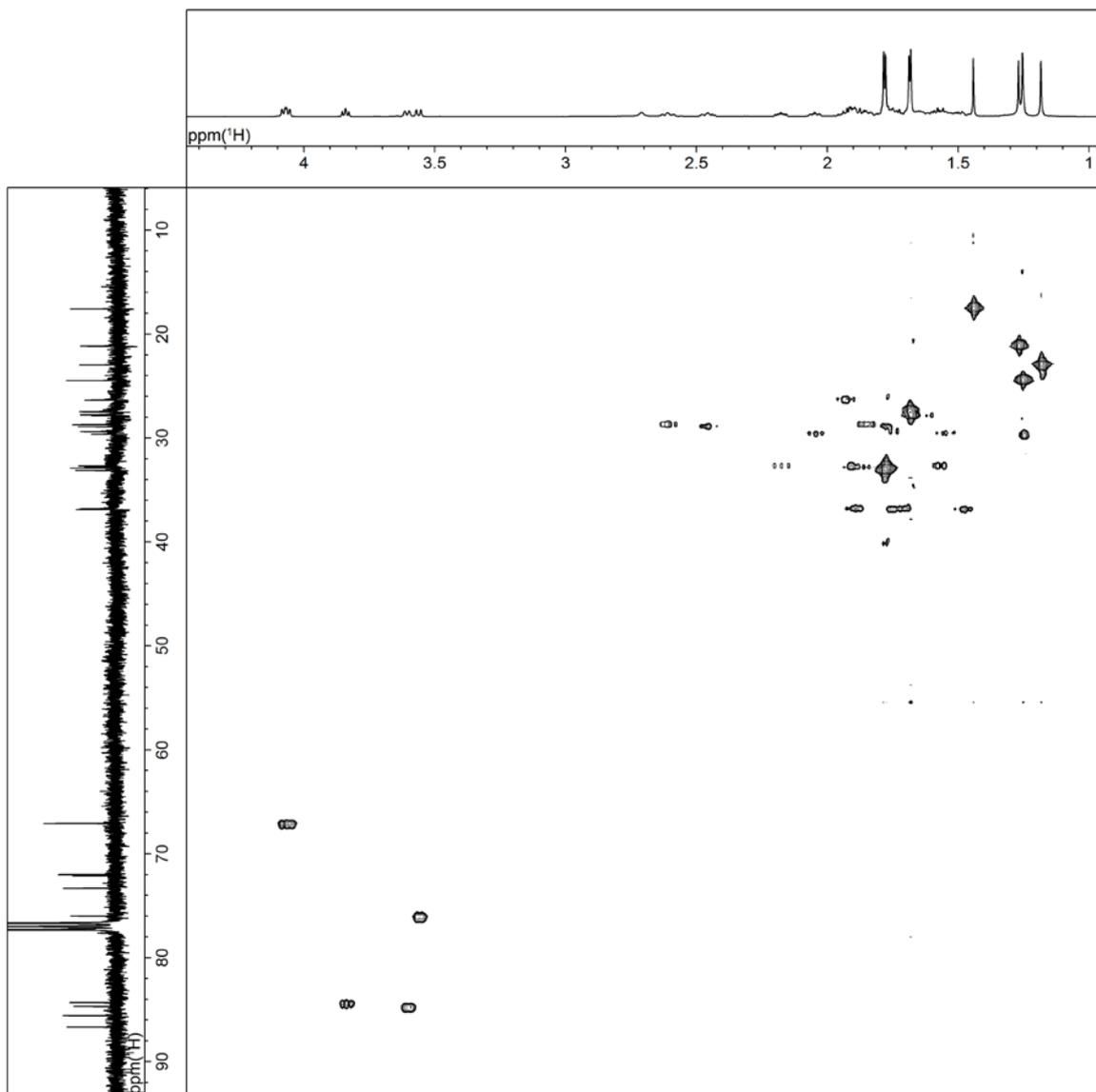
**Figure S8.**  $^1\text{H}$  NMR spectrum of intricatriol (**2**) in  $\text{CDCl}_3$ .



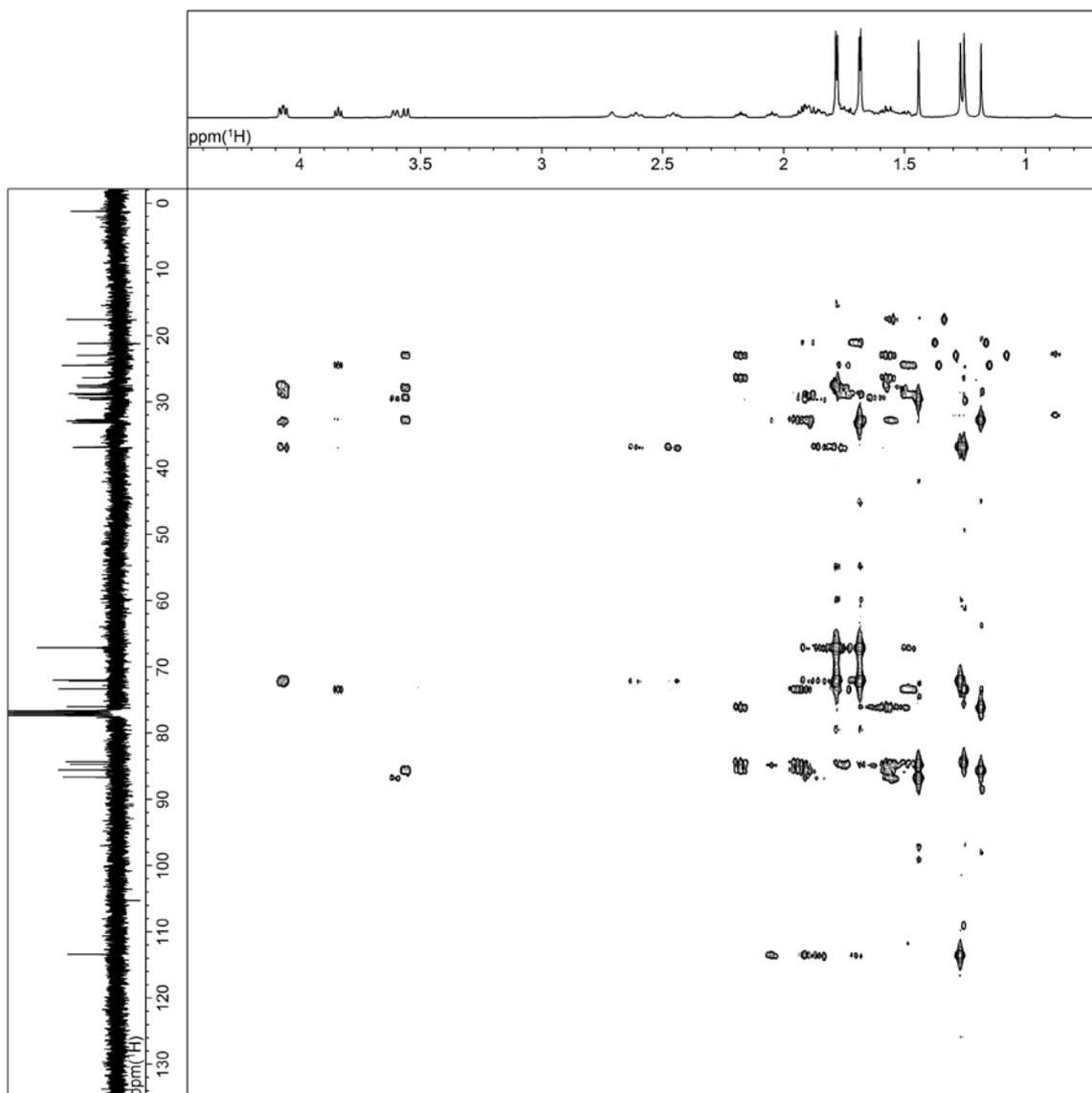
**Figure S9.**  $^{13}\text{C}$  NMR spectrum of intricatriol (**2**) in  $\text{CDCl}_3$ .



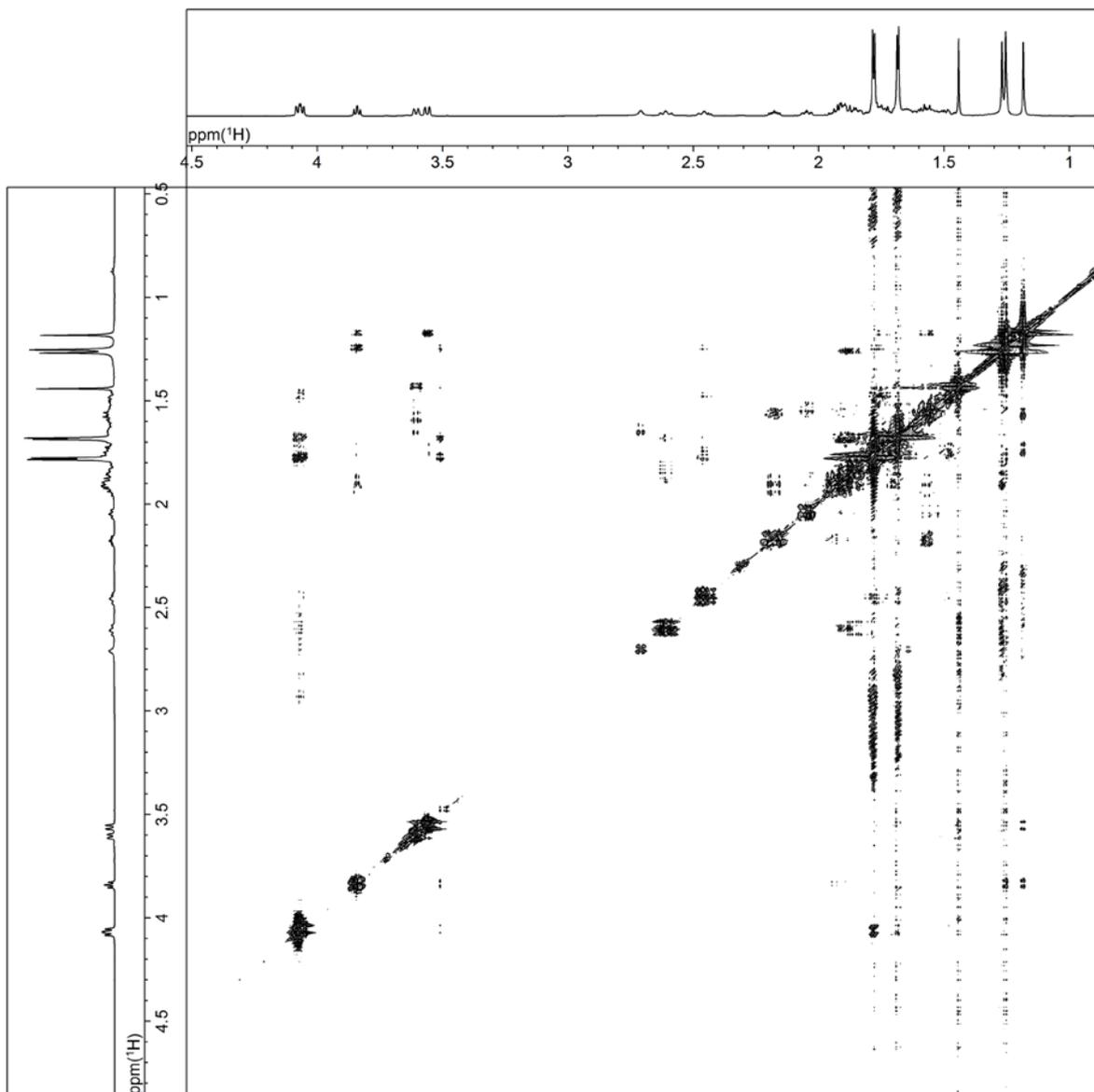
**Figure S10.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of intricatriol (**2**) in  $\text{CDCl}_3$ .



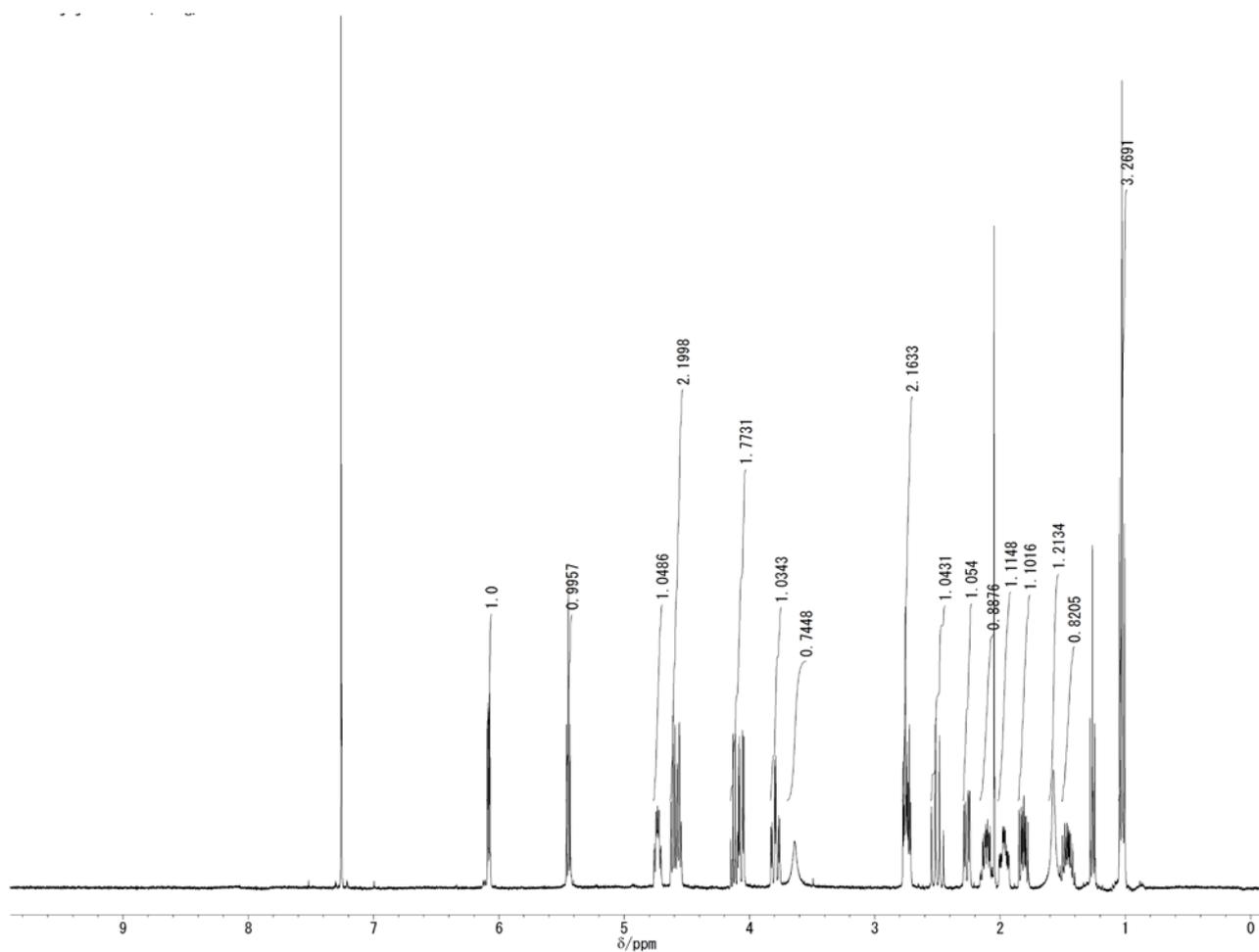
**Figure S11.** HSQC spectrum of intricatriol (**2**) in CDCl<sub>3</sub>.



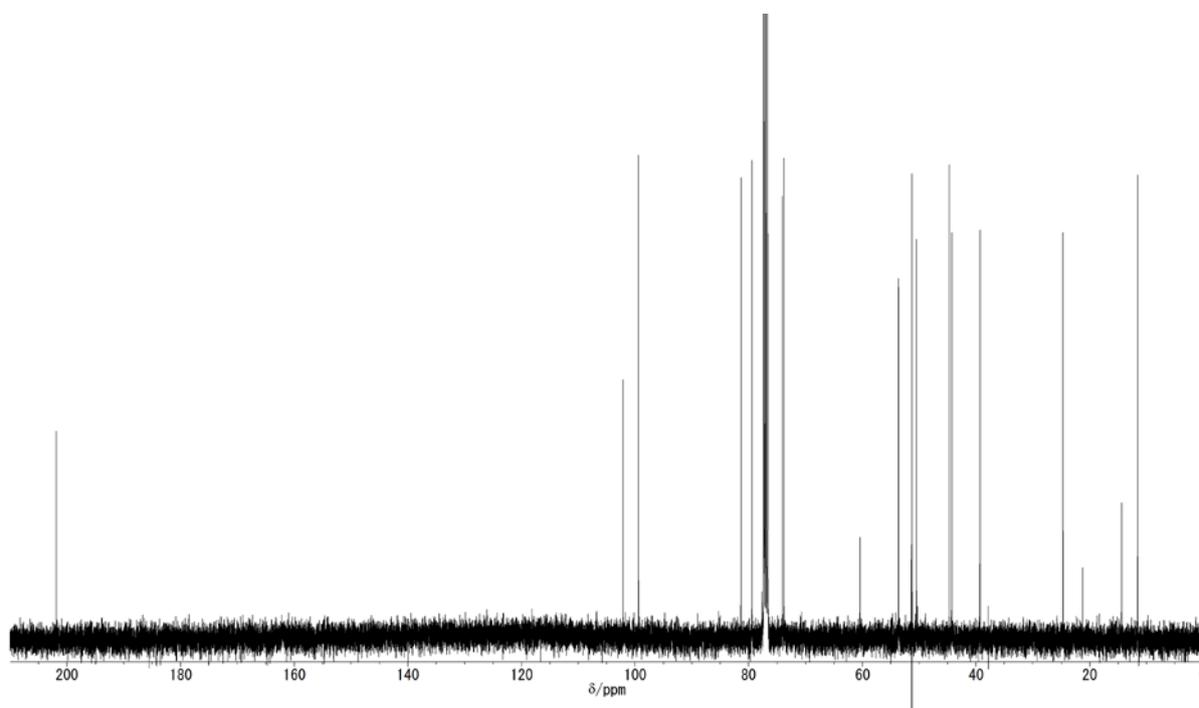
**Figure S12.** HMBC spectrum of intricatriol (**2**) in CDCl<sub>3</sub>.



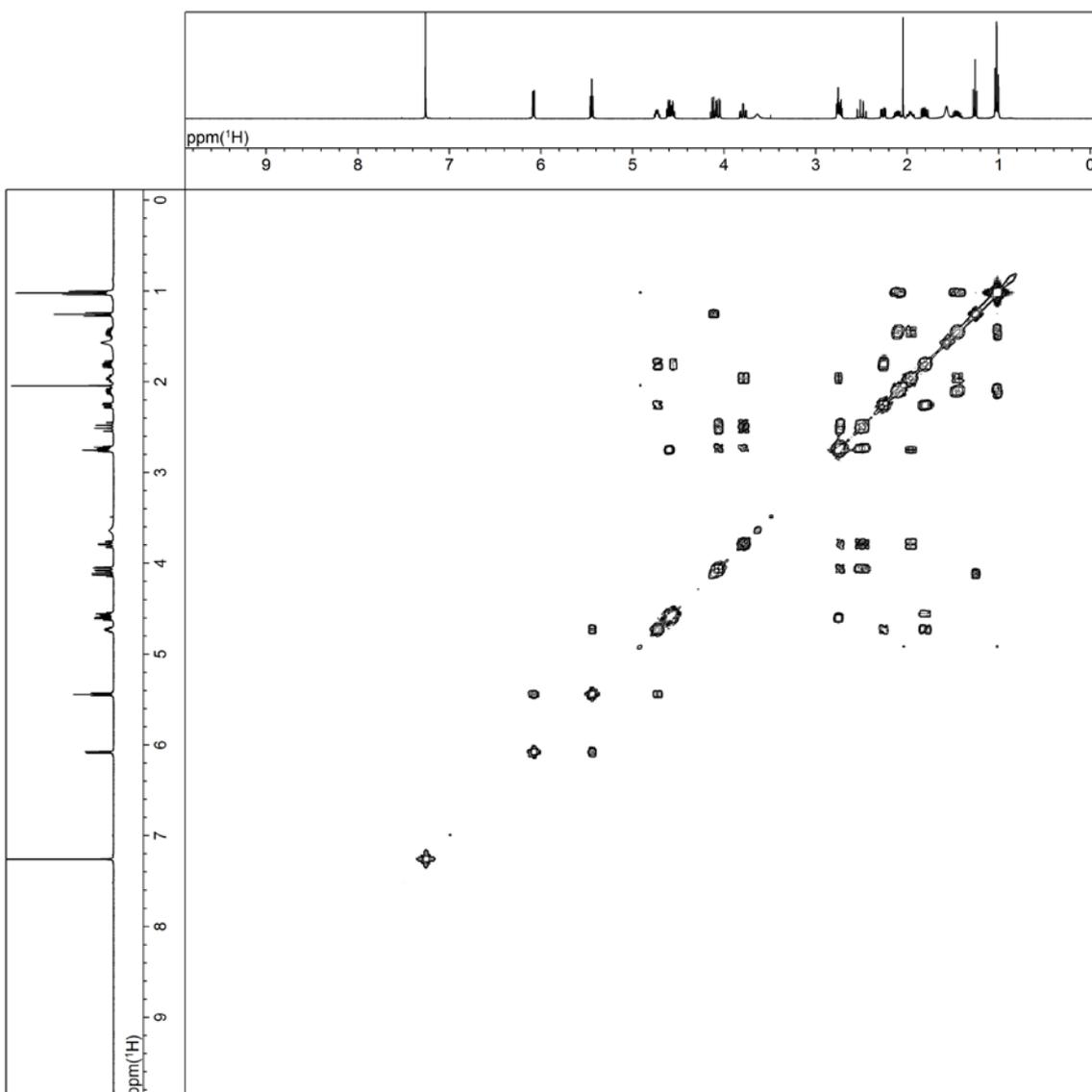
**Figure S13.** NOESY spectrum of intricatriol (**2**) in CDCl<sub>3</sub>.



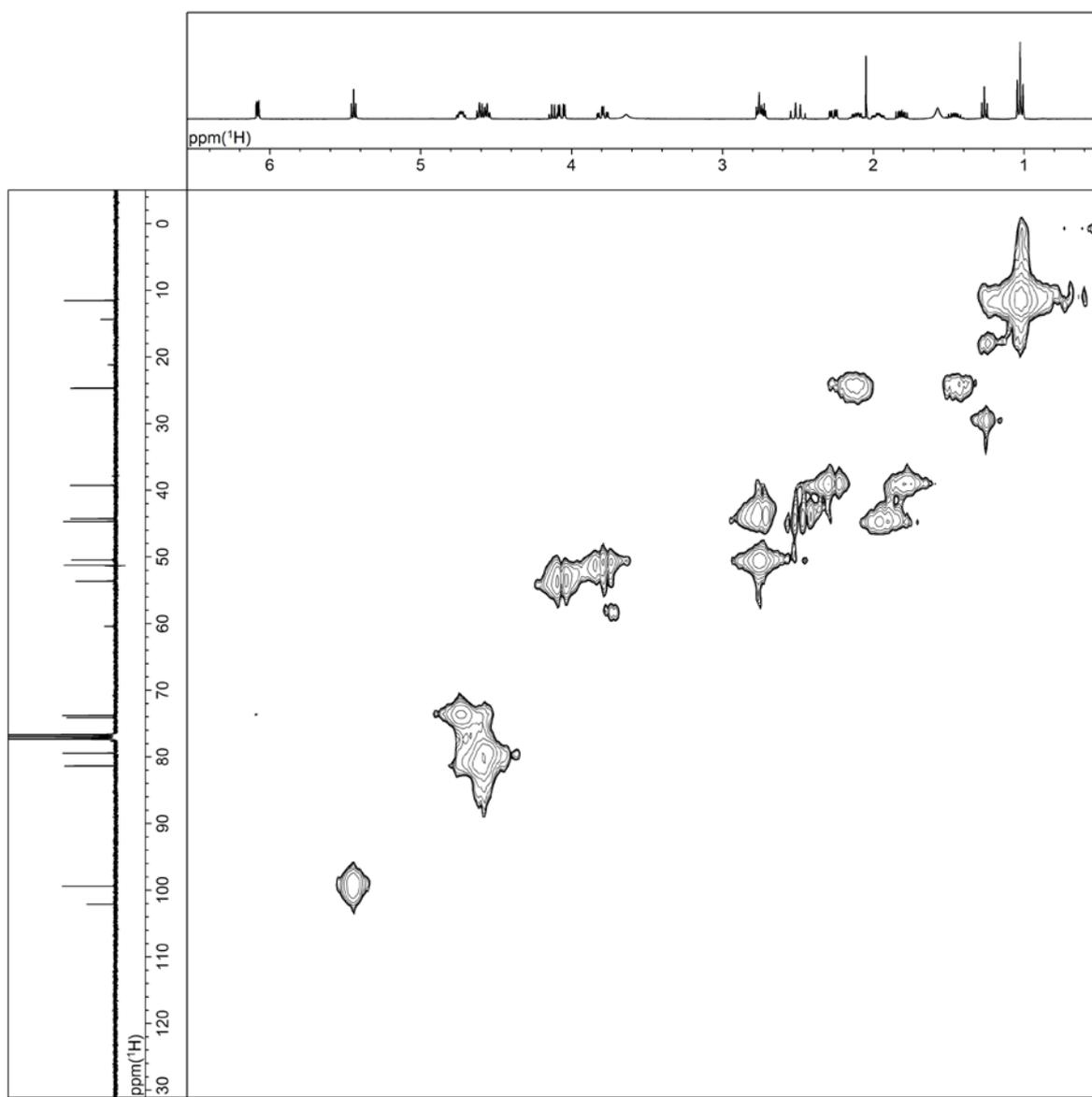
**Figure S14.**  $^1\text{H}$  NMR spectrum of hachijojimallene A (**3**) in  $\text{CDCl}_3$ .



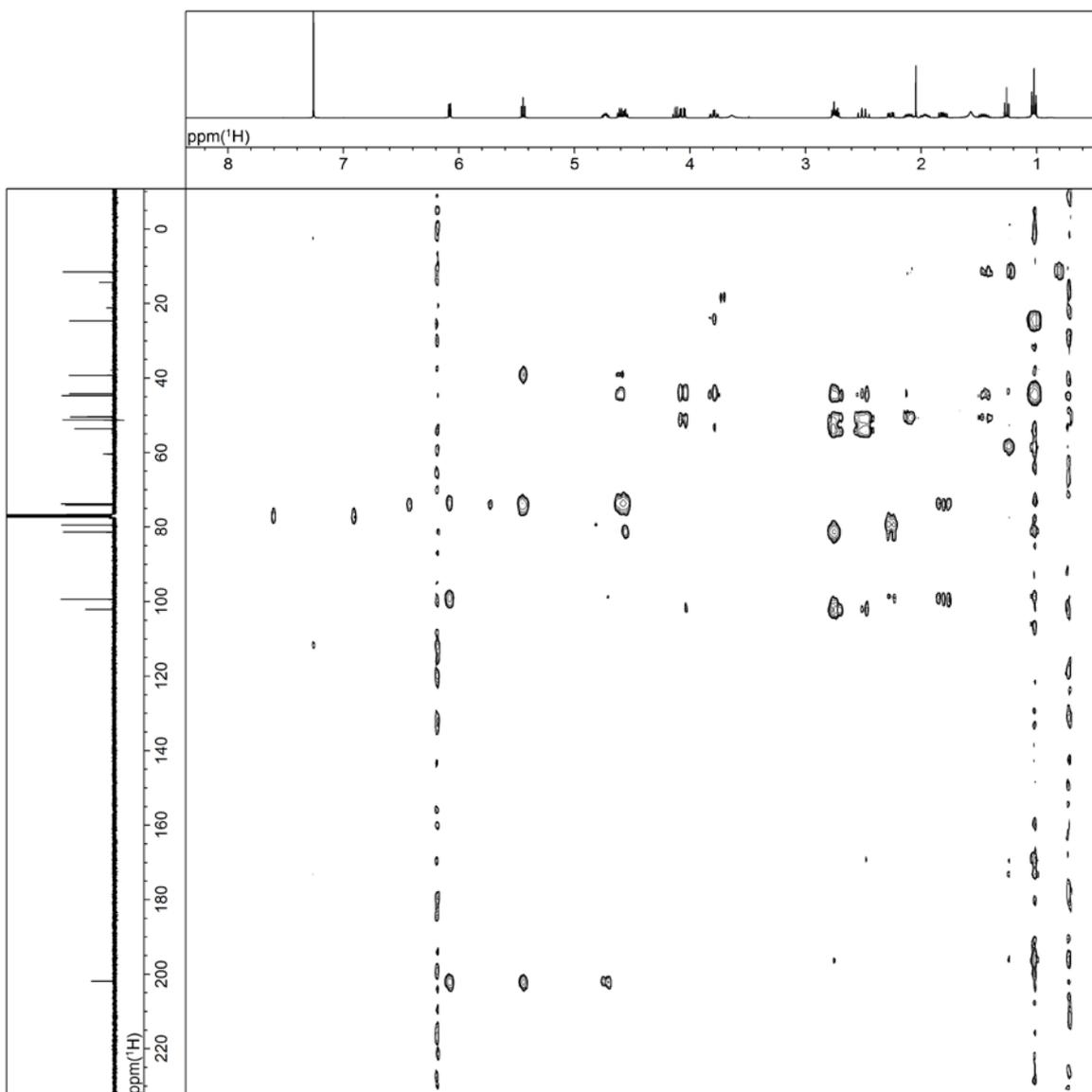
**Figure S15.**  $^{13}\text{C}$  NMR spectrum of hachijojimallene A (**3**) in  $\text{CDCl}_3$ .



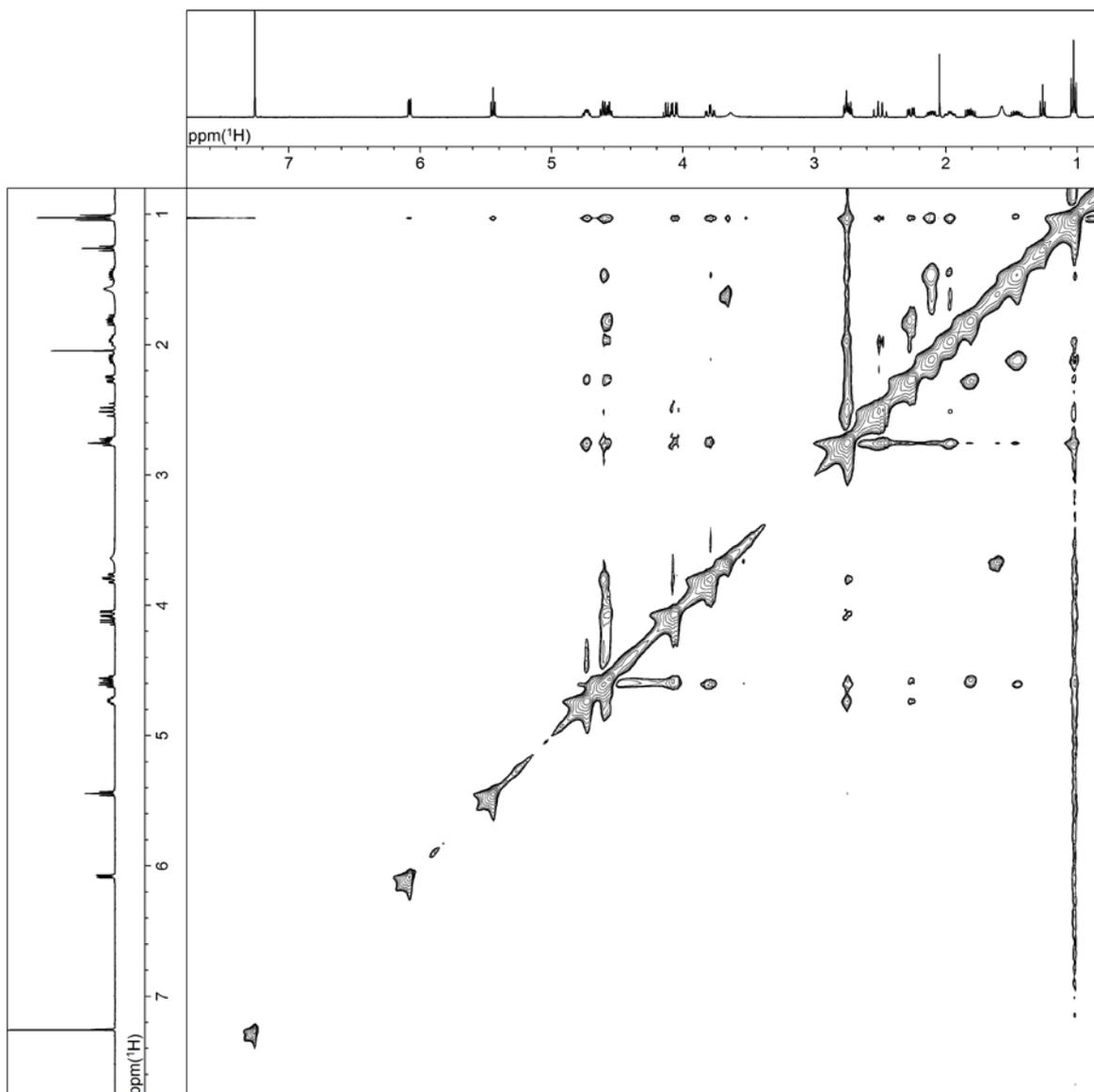
**Figure S16.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of hachijojimallene A (**3**) in  $\text{CDCl}_3$ .



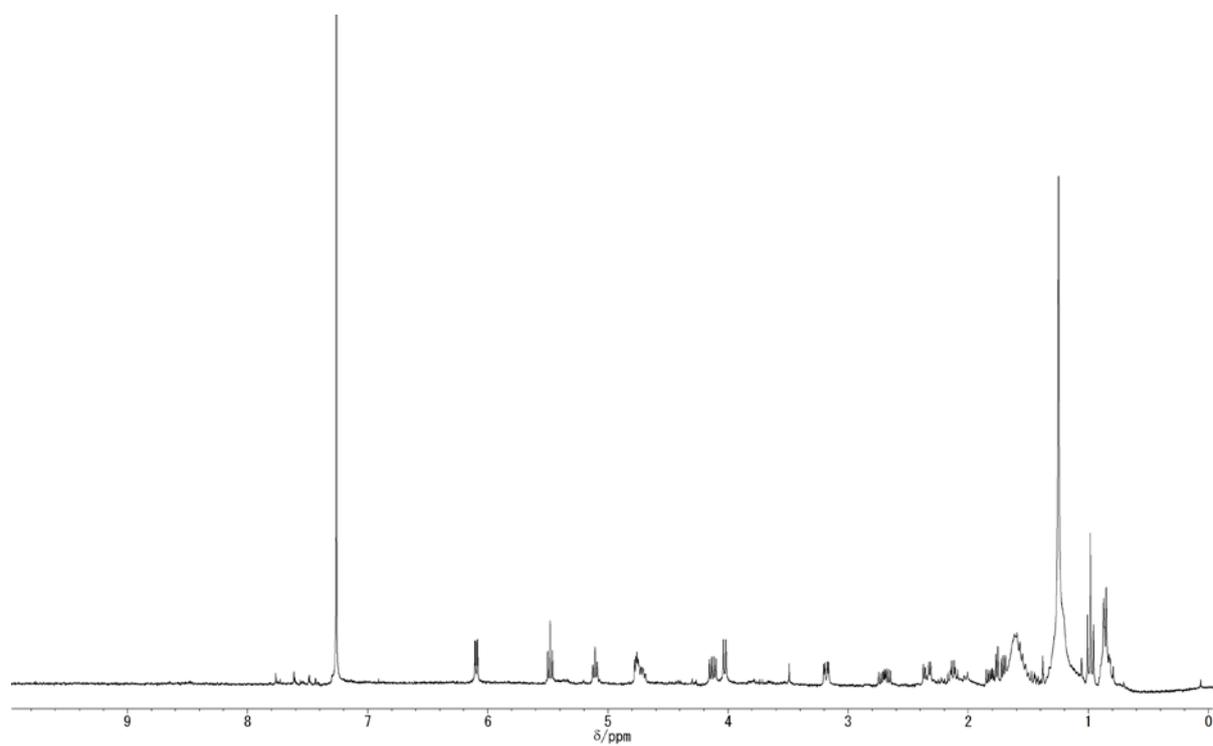
**Figure S17.** HMPC spectrum of hachijojimallene A (**3**) in CDCl<sub>3</sub>.



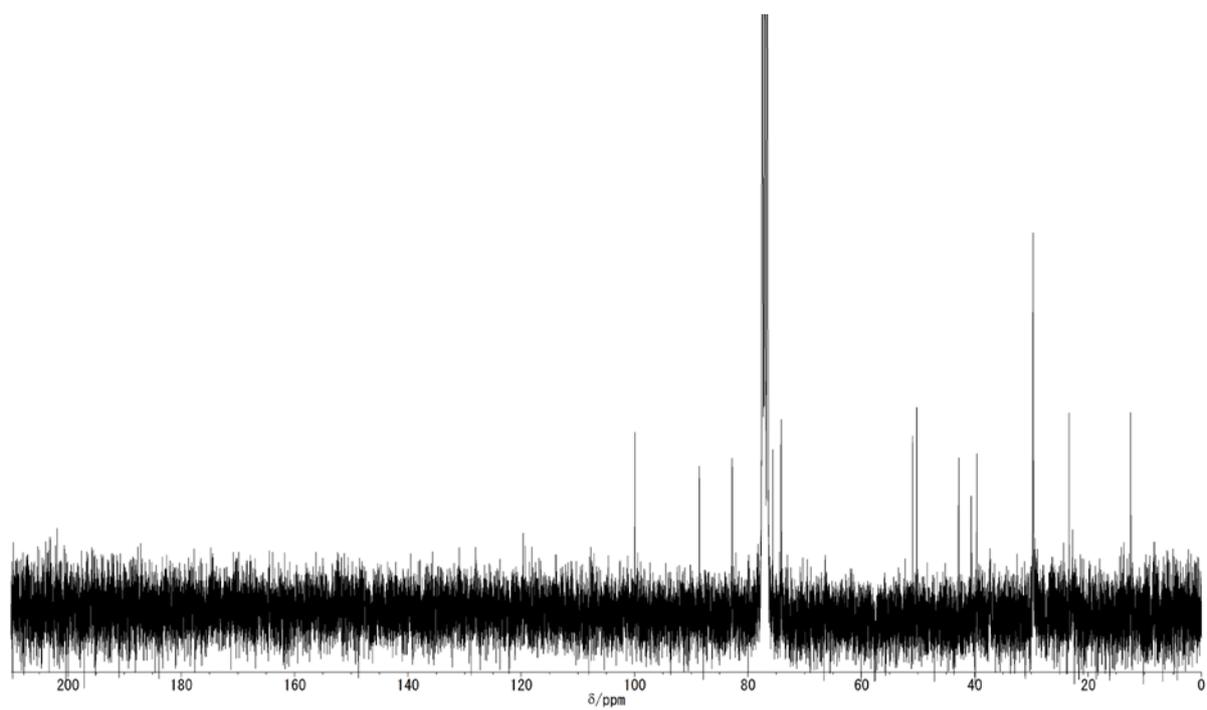
**Figure S18.** HMBC spectrum of hachijojimallene A (**3**) in CDCl<sub>3</sub>.



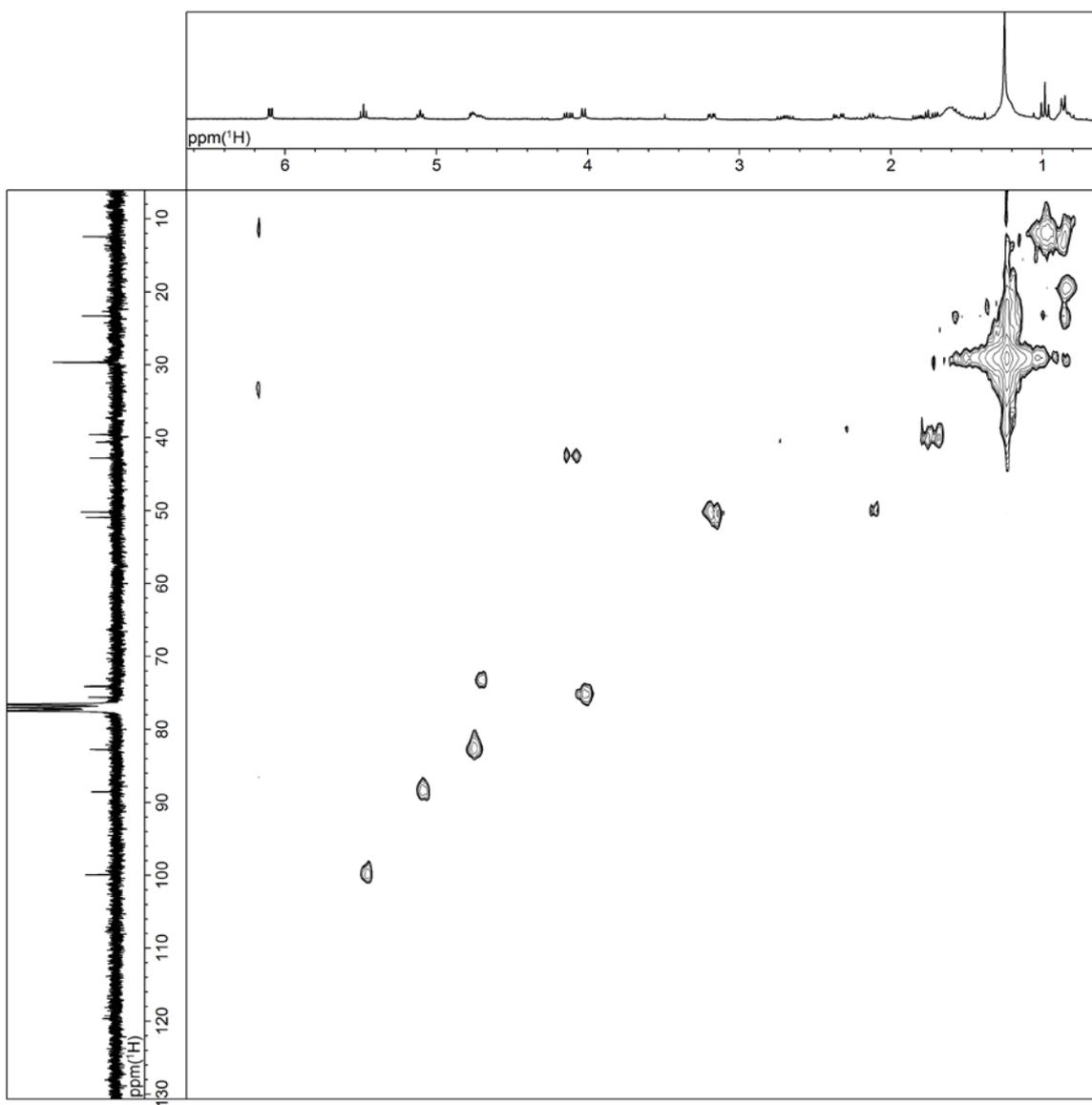
**Figure S19.** NOESY spectrum of hachijojimallene A (**3**) in  $CDCl_3$ .



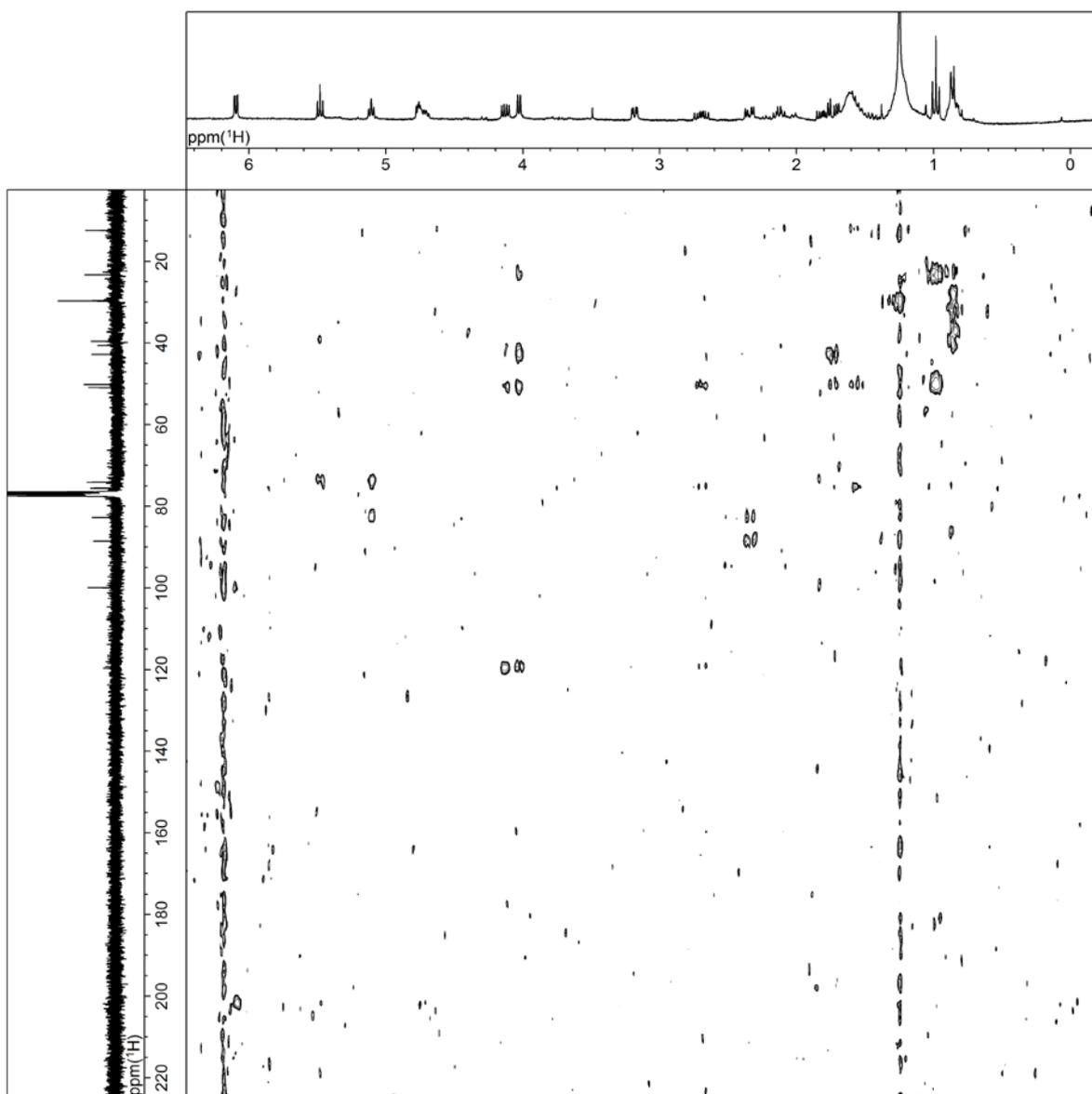
**Figure S20.** <sup>1</sup>H NMR spectrum of hachijojimallene B (**4**) in CDCl<sub>3</sub>.



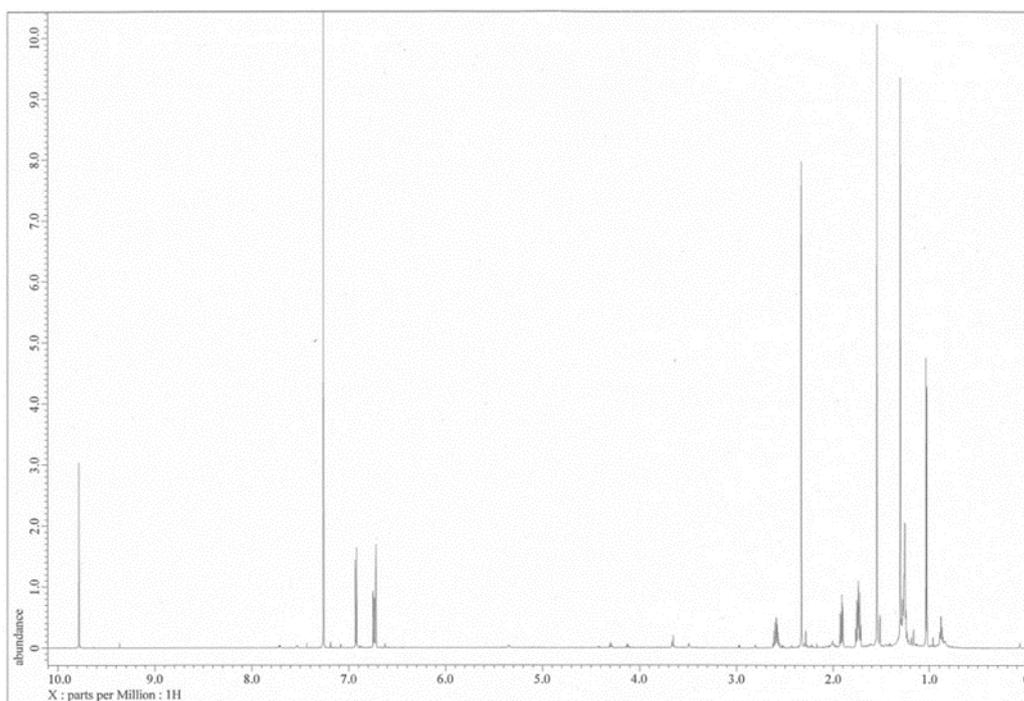
**Figure S21.** <sup>13</sup>C NMR spectrum of hachijojimallene B (**4**) in CDCl<sub>3</sub>.



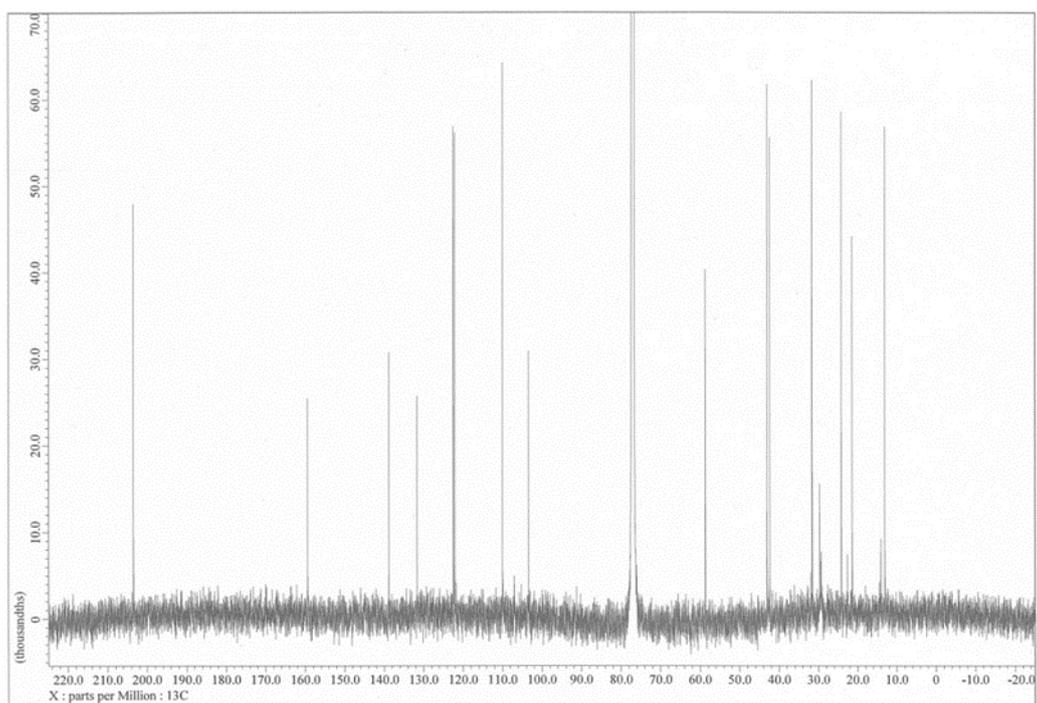
**Figure S22.** HMQC spectrum of hachijojimallene B (4) in CDCl<sub>3</sub>.



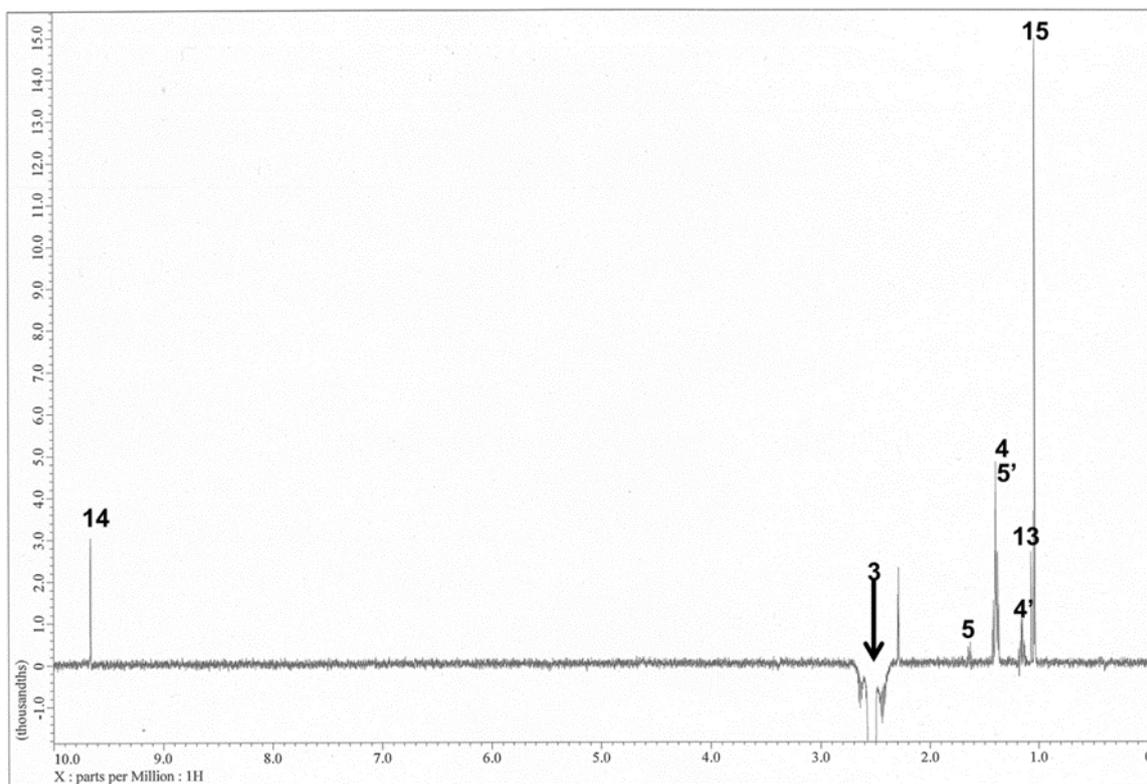
**Figure S23.** HMBC spectrum of hachijojimallene B (**4**) in CDCl<sub>3</sub>.



**Figure S24.** <sup>1</sup>H NMR spectrum of debromoaplysinal (**5**) in CDCl<sub>3</sub>.



**Figure S25.** <sup>13</sup>C NMR spectrum of debromoaplysinal (**5**) in CDCl<sub>3</sub>.



**Figure S26.** DPGSE 1D NOE spectrum of debromoaplysinal (**5**) in  $CDCl_3$ .

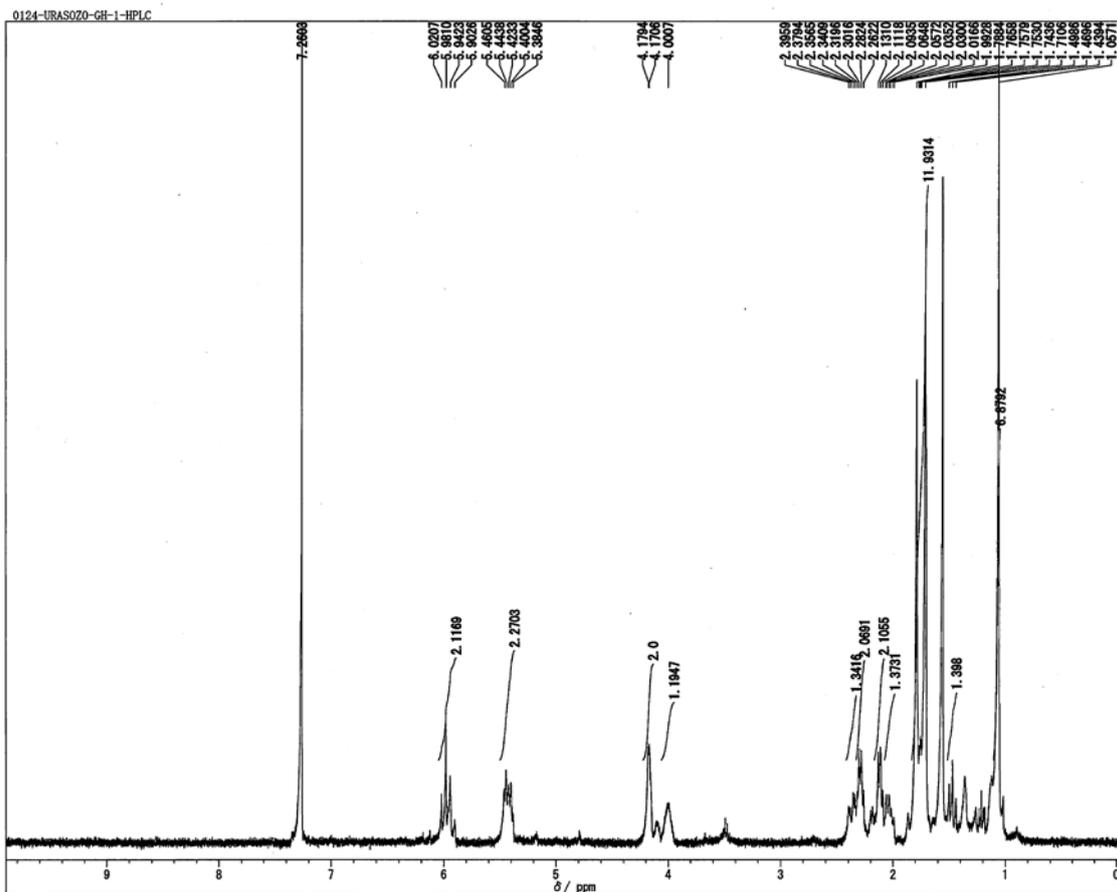


Figure S27.  $^1\text{H}$  NMR spectrum of 11,12-dihydro-3-hydroxyretinol (**6**) in  $\text{CDCl}_3$ .

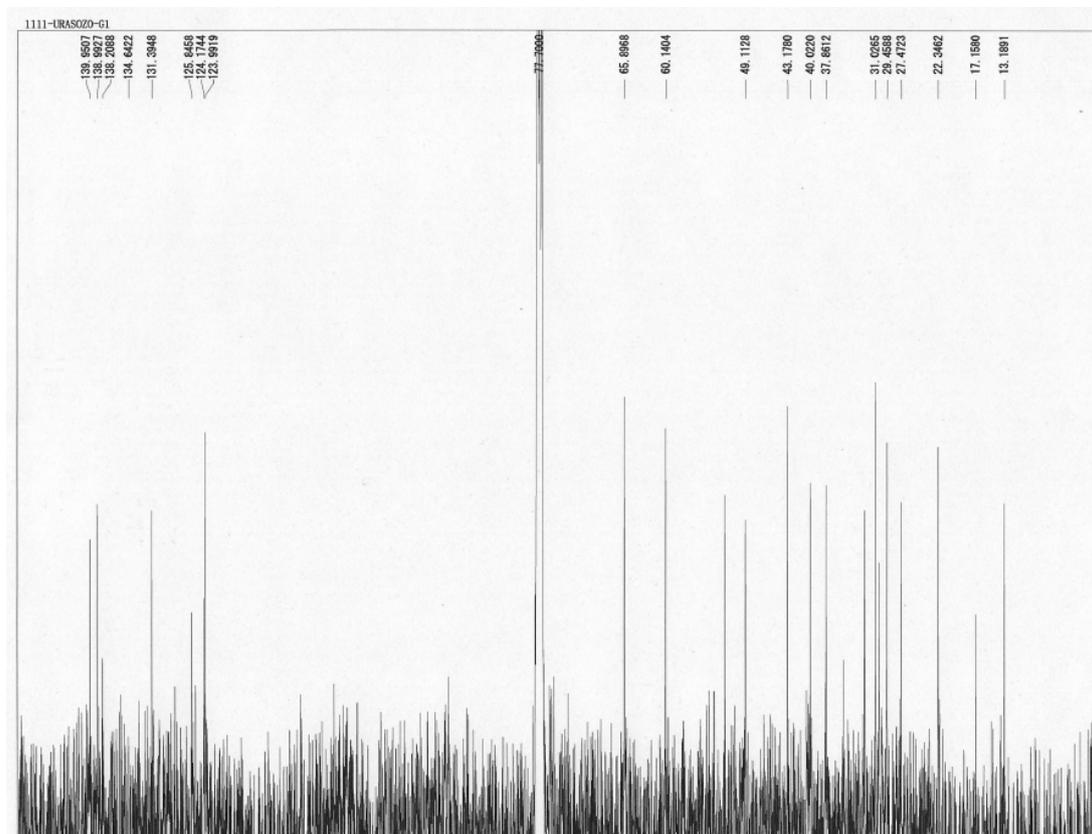


Figure S28.  $^{13}\text{C}$  NMR spectrum of 11,12-dihydro-3-hydroxyretinol (**6**) in  $\text{CDCl}_3$ .