

# Supplementary Materials: Three New Cytotoxic Polyhydroxysteroidal Glycosides from the Starfish *Craspidaster hesperus*

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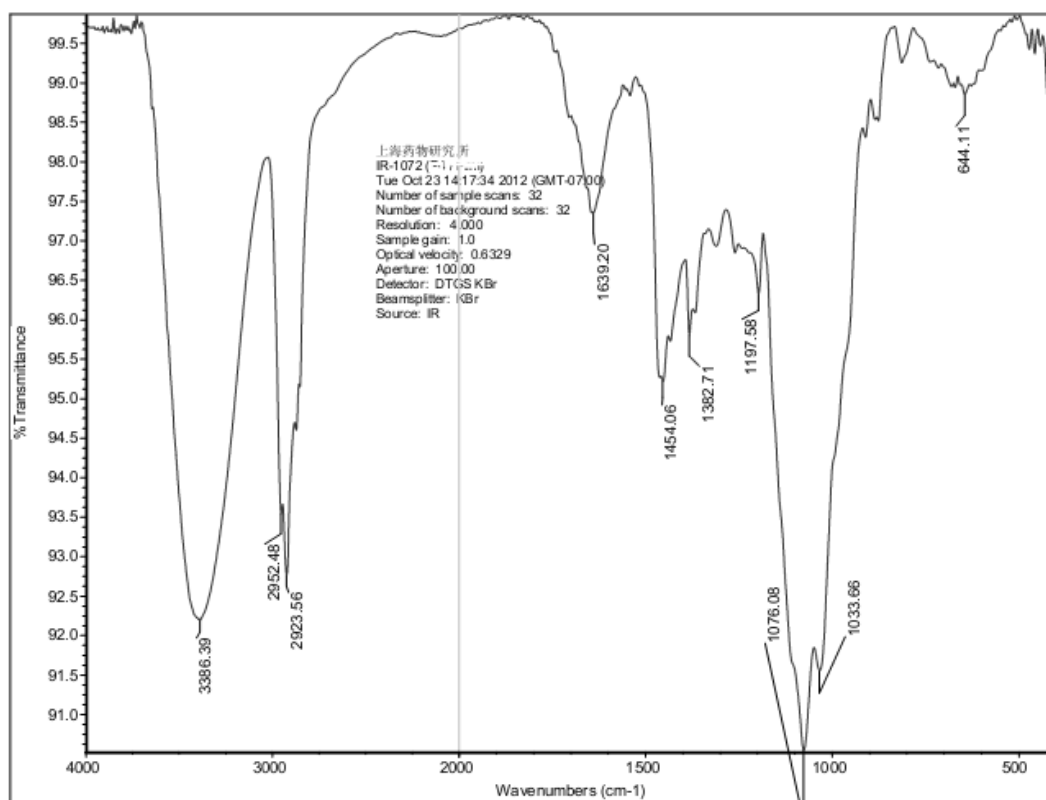
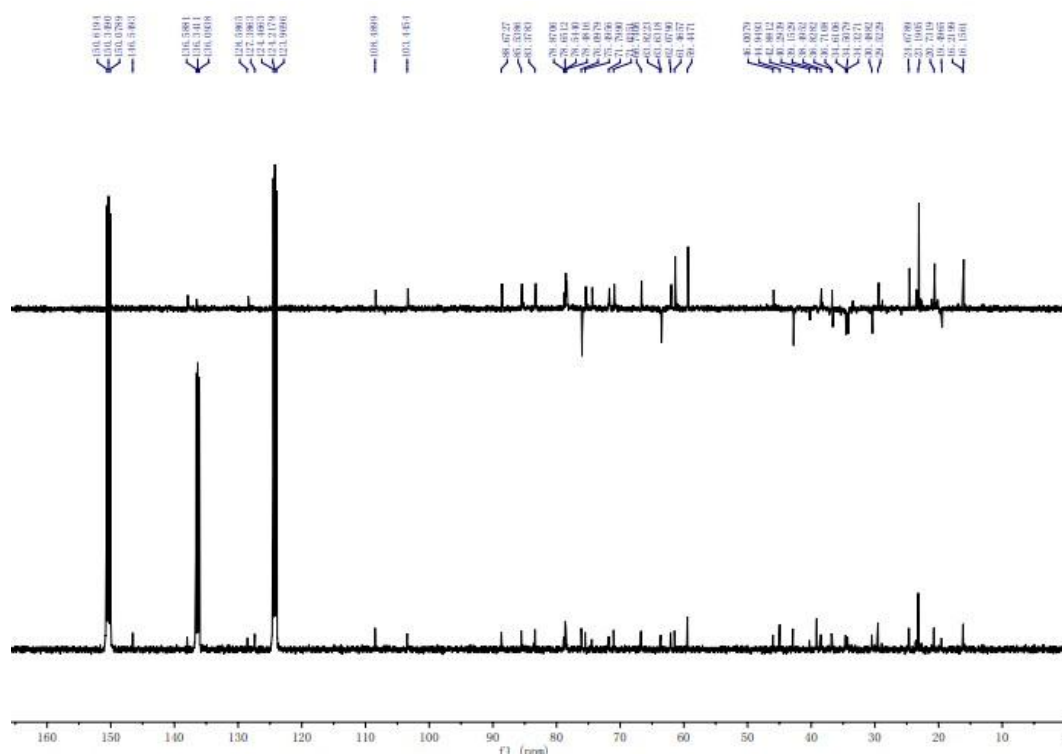


Figure S1. IR spectrum of hesperuside A (1).

Figure S2.  $^{13}\text{C}$  NMR and DEPT spectrum of hesperuside A (1) in pyridine- $d_5$ , 150MHz.

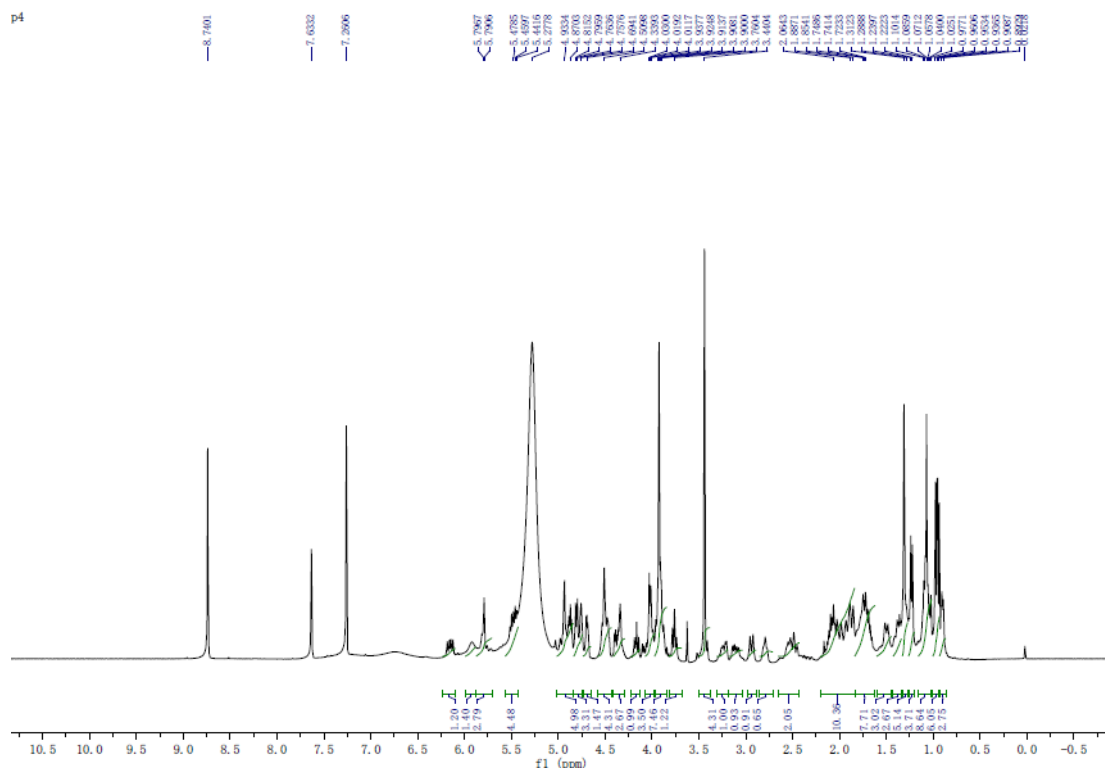


Figure S3. <sup>1</sup>H NMR spectrum of hesperuside A (1) in pyridine-*d*<sub>5</sub>, 600MHz.

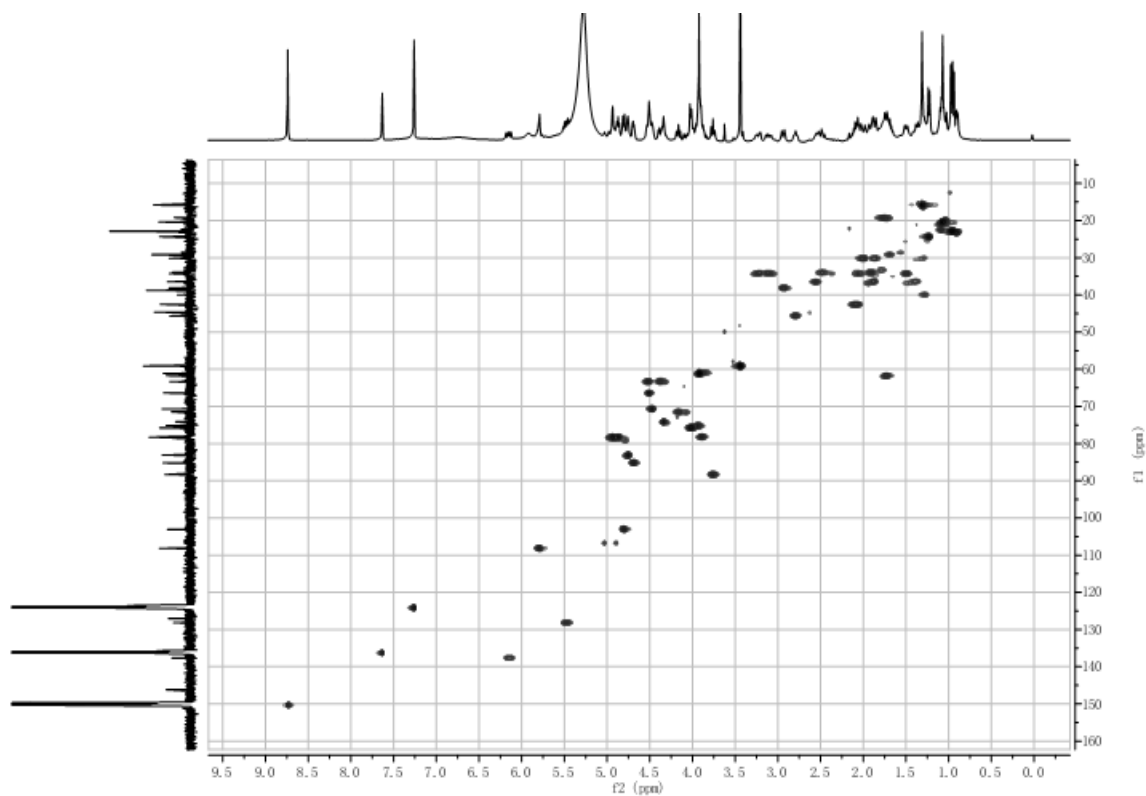


Figure S4. HMQC spectrum of hesperuside A (1).

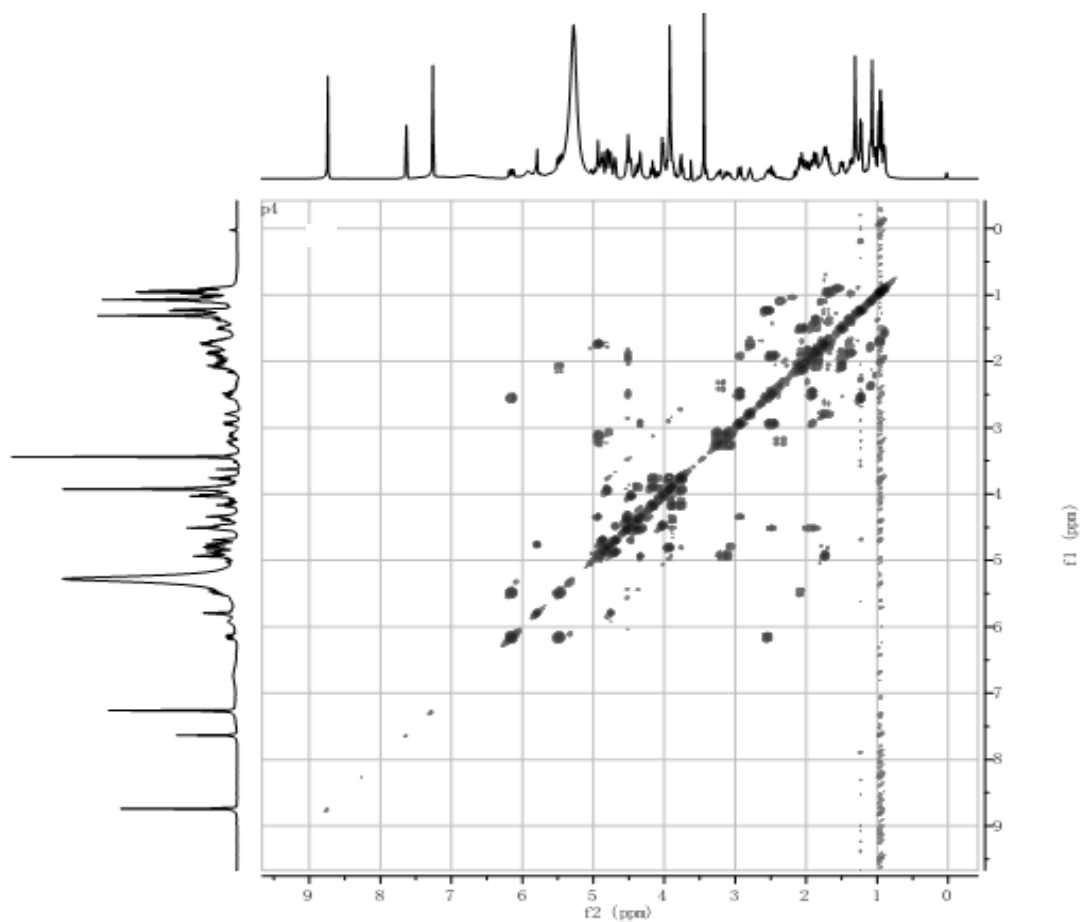


Figure S5.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of hesperuside A (1).

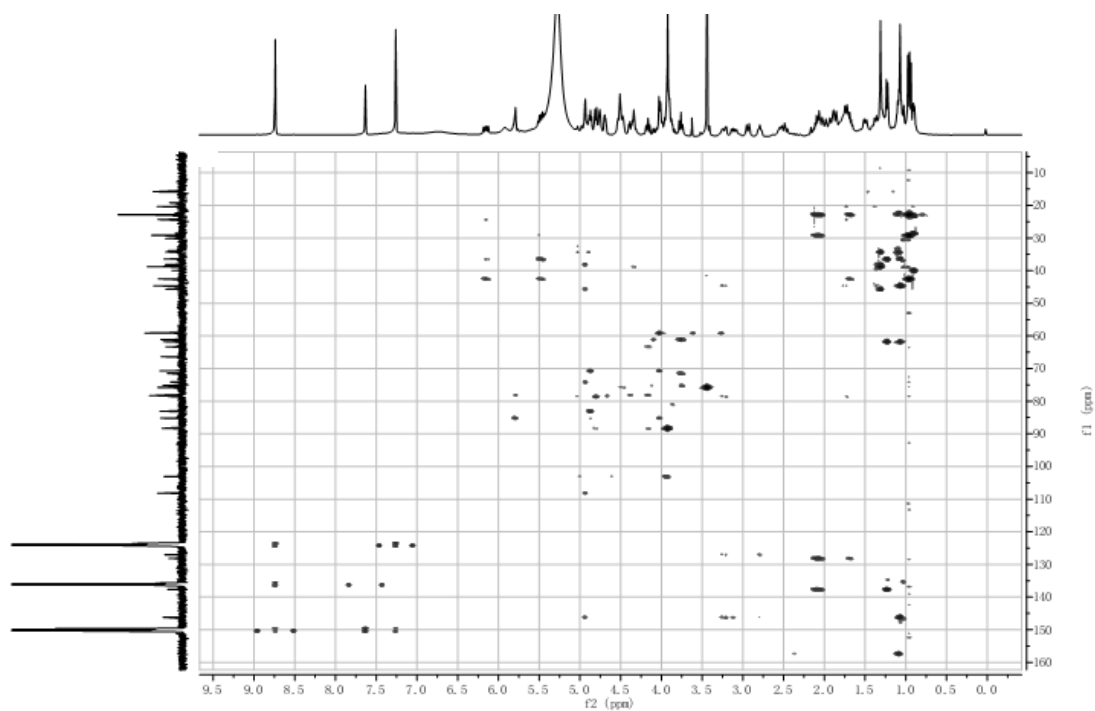


Figure S6. HMBC spectrum of hesperuside A (1).

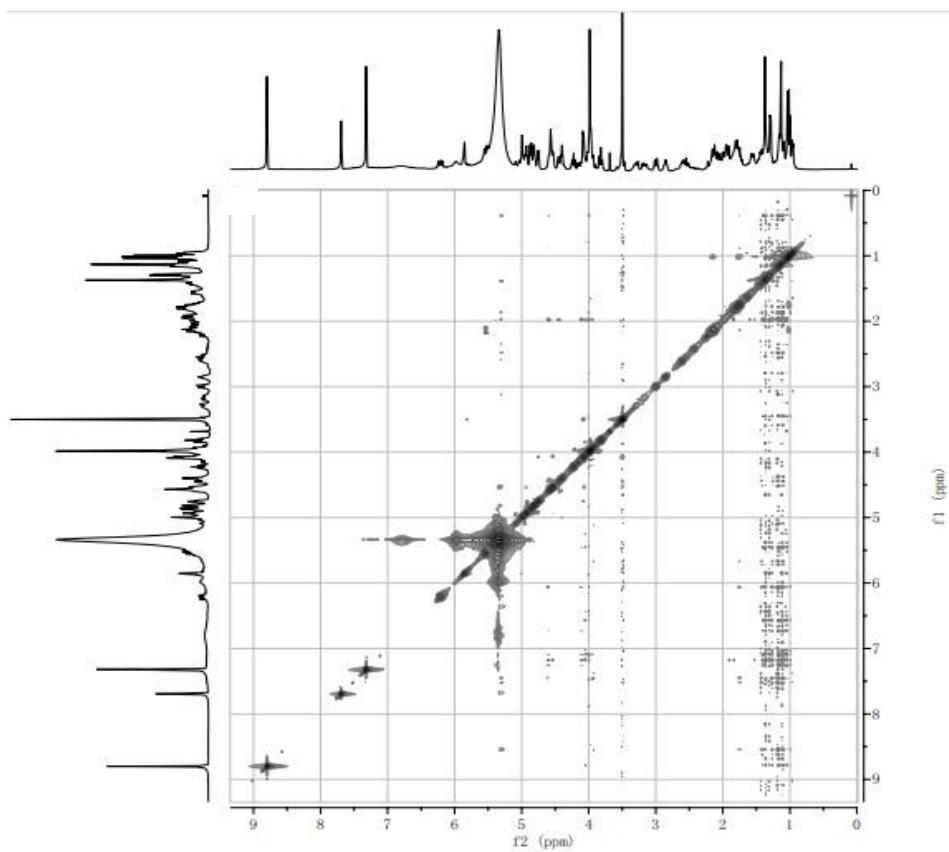


Figure S7. NOESY spectrum of hesperuside A (1).

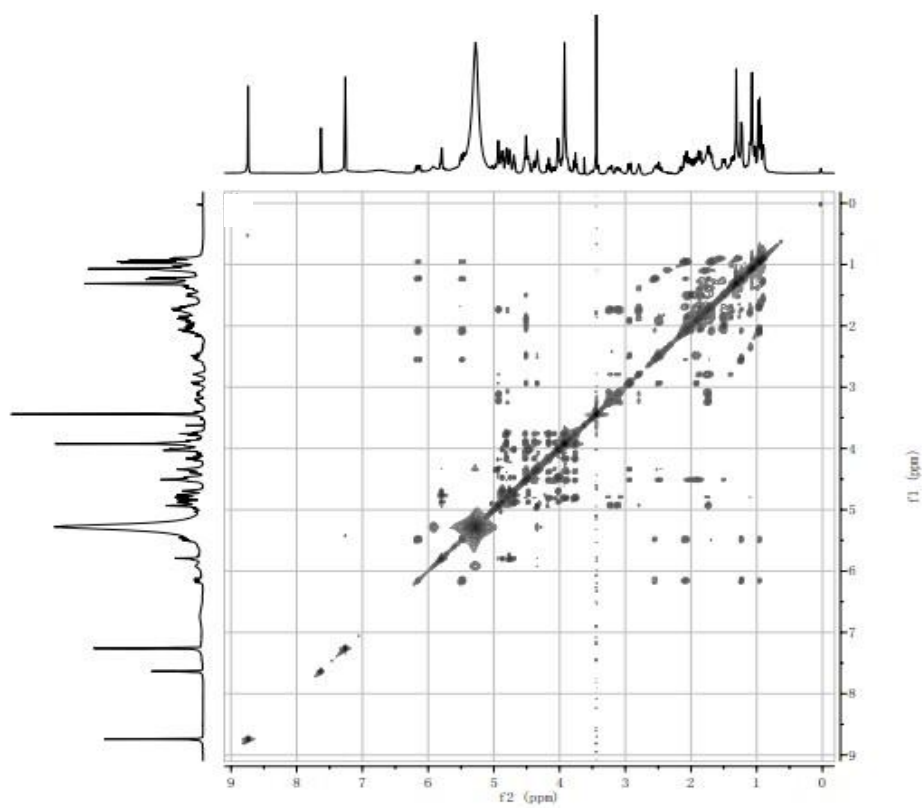


Figure S8. TOCSY spectrum of hesperuside A (1).

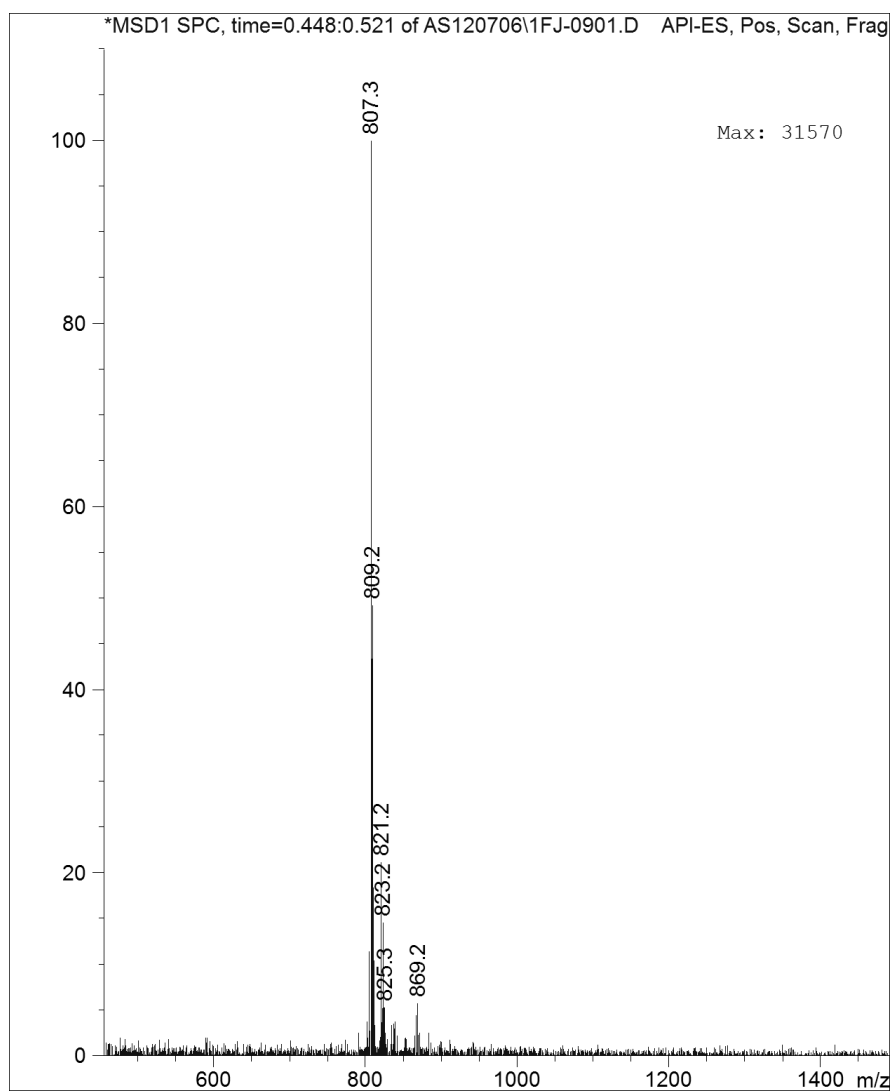


Figure S9. EI-MS spectrum of hesperuside A (1).

Ion Mass (Measured) 807.45341

Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e <sup>-</sup>
C <sub>44</sub> H <sub>66</sub> N <sub>1</sub> Na <sub>1</sub> O <sub>11</sub>	0.088	807.45281	-0.75	-1.70	-0.81	12.00	ok	odd
C <sub>41</sub> H <sub>68</sub> Na <sub>1</sub> O <sub>14</sub>	0.091	807.45013	-4.07	-5.62	-3.29	7.50	ok	even

Figure S10. HR-EI-MS spectrum of hesperuside A (1).

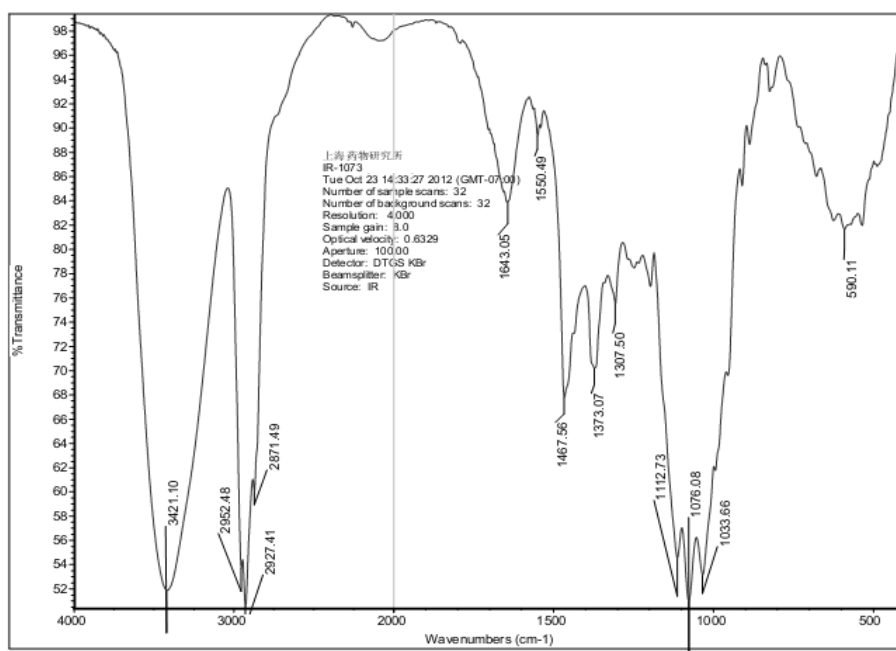
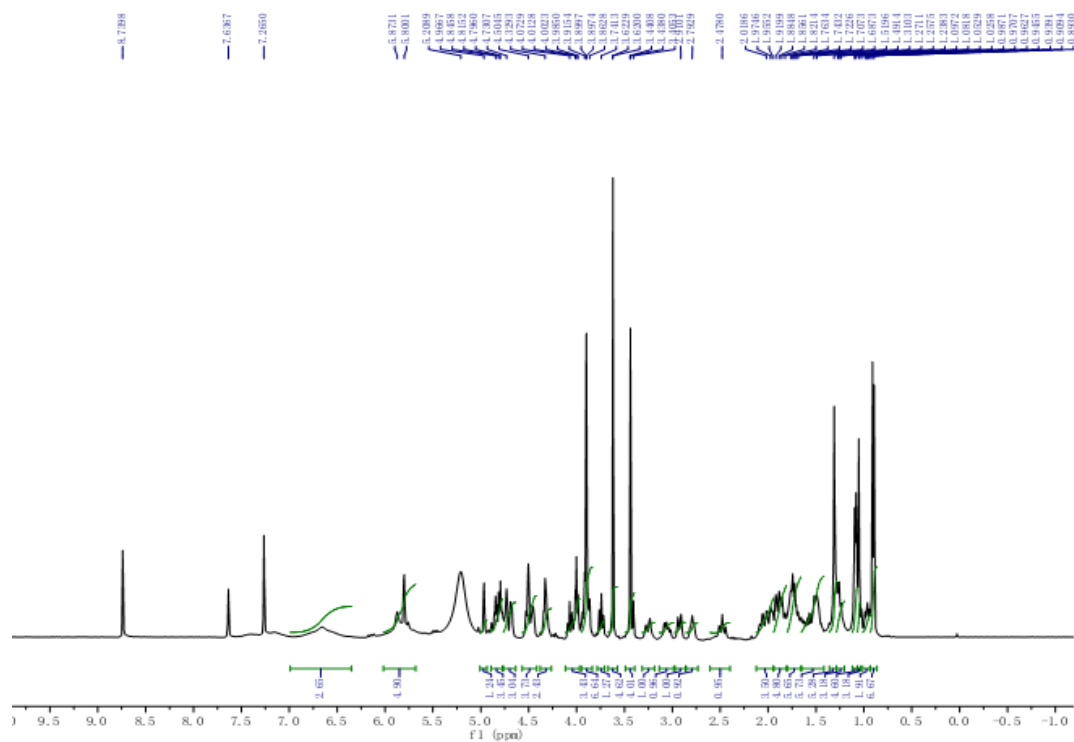


Figure S11. IR spectrum of hesperuside B (2).

Figure S12.  $^1\text{H}$  NMR spectrum of hesperuside B (2) in pyridine- $d_5$ , 600MHz.

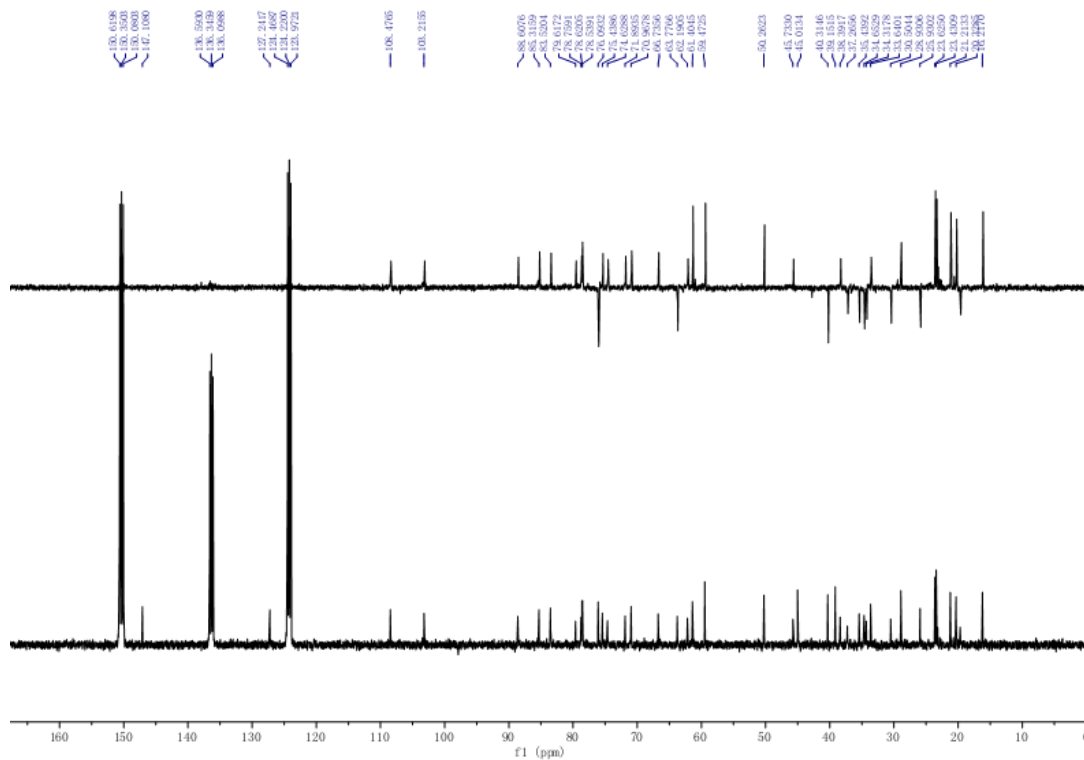


Figure S13. <sup>13</sup>C NMR and DEPT spectrum of hesperuside B (2) in pyridine-*d*<sub>5</sub>, 150MHz.

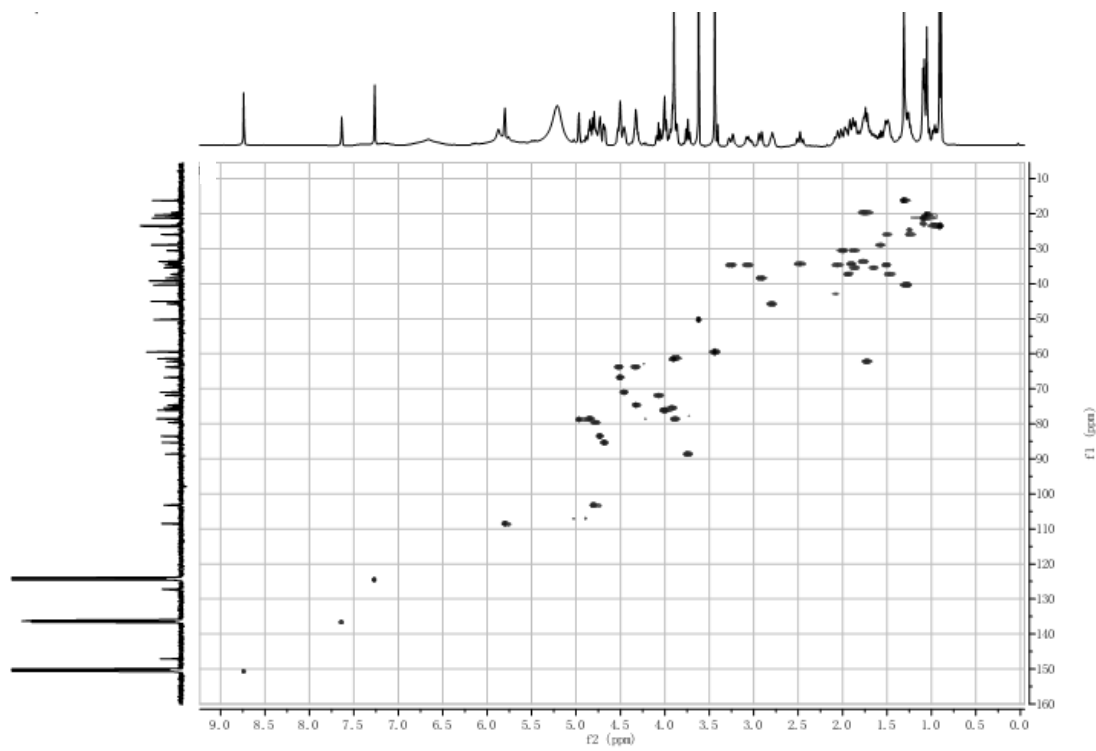


Figure S14. HMQC spectrum of hesperuside B (2).



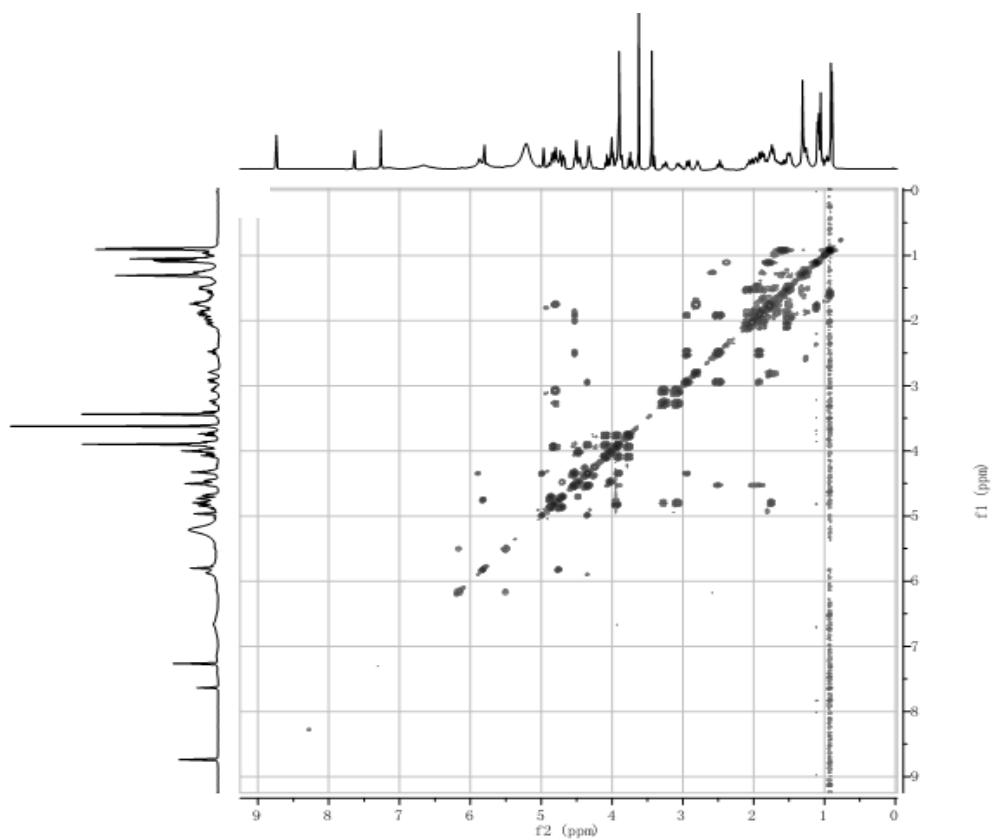


Figure S15.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of hesperuside B (2).

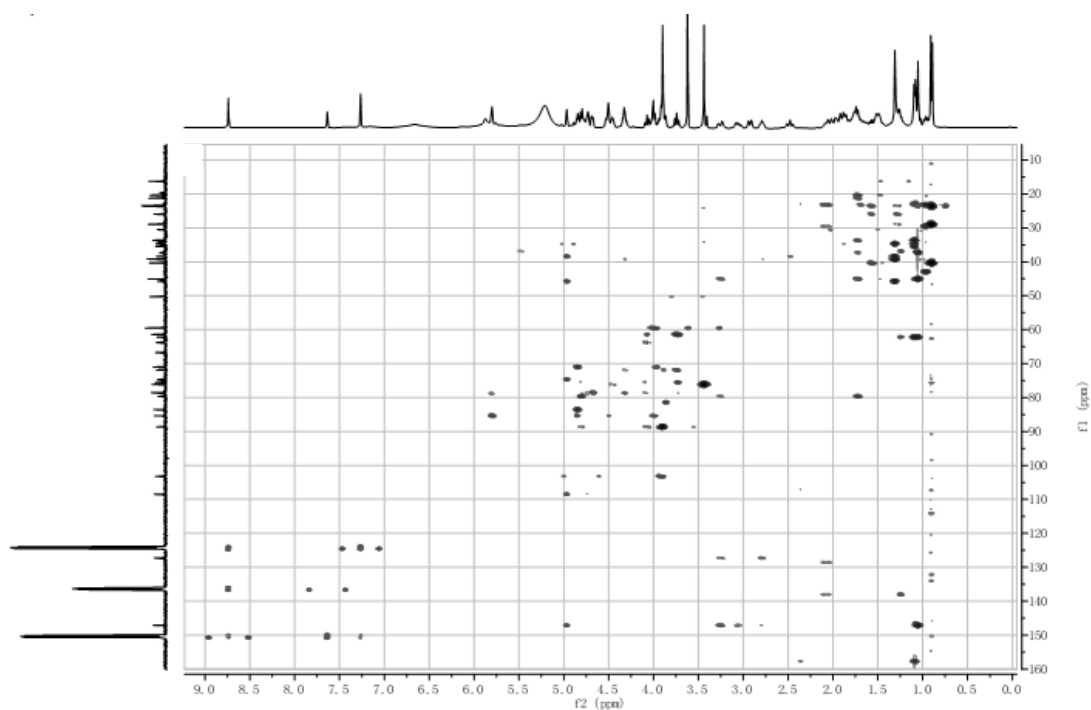


Figure S16. HMBC spectrum of hesperuside B (2).

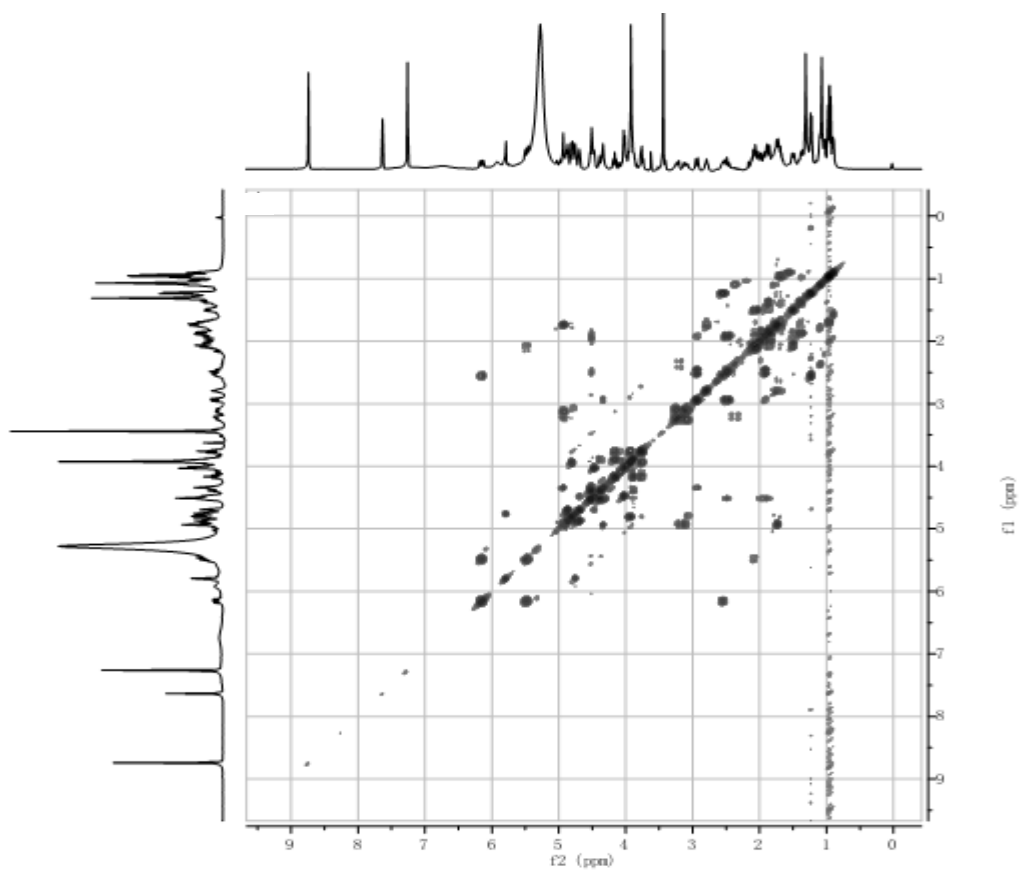


Figure S17. NOESY spectrum of hesperuside B (2).

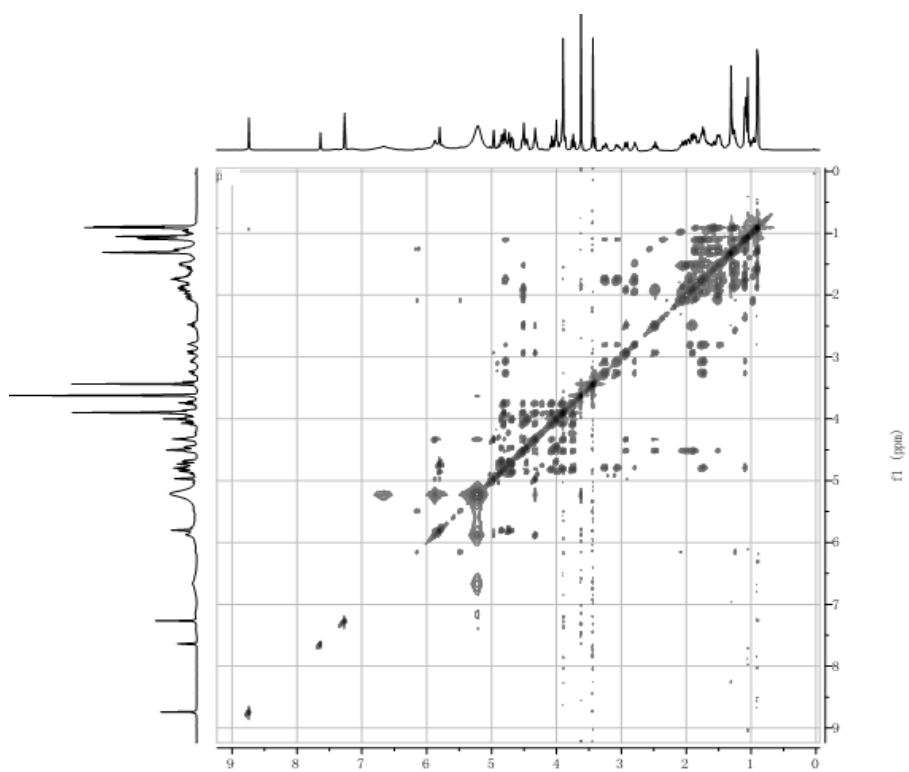


Figure S18. TOCSY spectrum of hesperuside B (2).

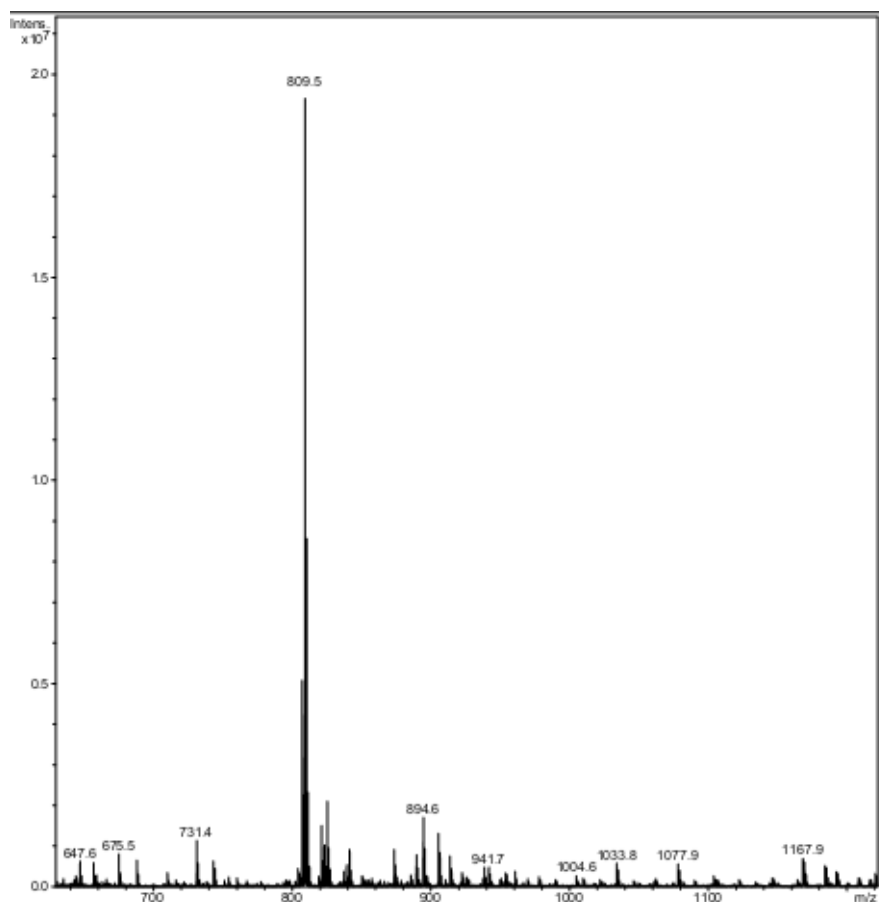


Figure S19. EI-MS spectrum of hesperuside B (2).

Ionization Mode ESI-Positive

Ion Mass (Measured) 809.46384

Sum	Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e <sup>-</sup>
C 41 H 70 Na 1 O 14		0.010	809.46578	2.39	2.23	1.94	6.50	ok	even
C 48 H 66 Na 1 O 9		0.049	809.45990	-4.86	-5.06	-3.94	15.50	ok	even
C 51 H 64 N 1 Na 1 O 6		0.068	809.46258	-1.55	-1.79	-1.26	20.00	ok	odd
C 54 H 62 N 2 Na 1 O 3		0.086	809.46526	1.76	1.48	1.42	24.50	ok	even

Figure S20. HR-EI-MS spectrum of hesperuside B (2).

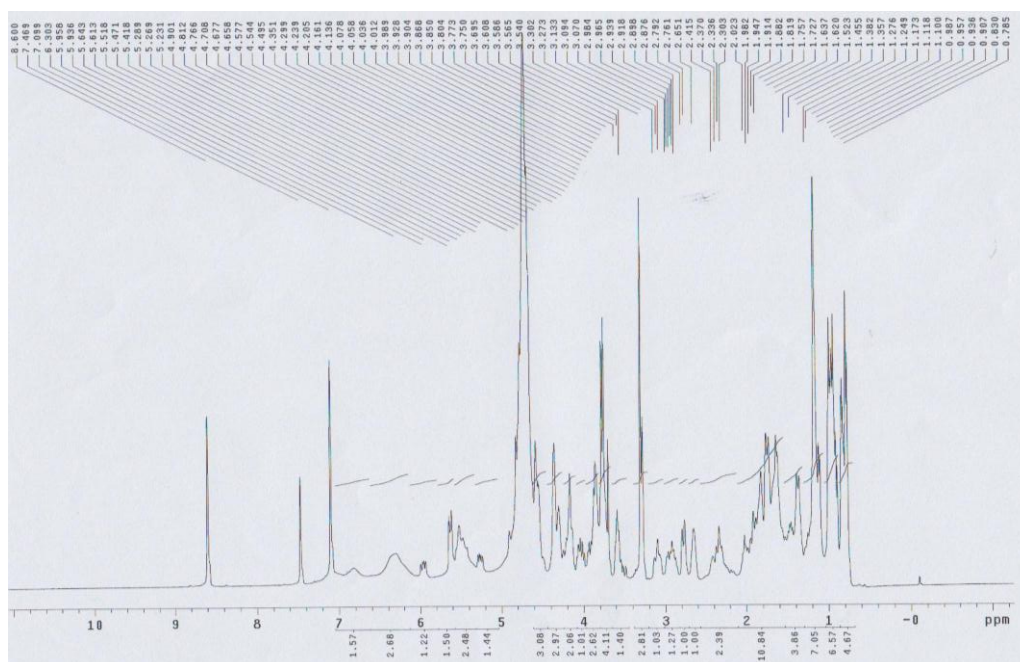


Figure S21. <sup>1</sup>H NMR spectrum of hesperuside C (3) in pyridine-*d*<sub>5</sub>, 600MHz.

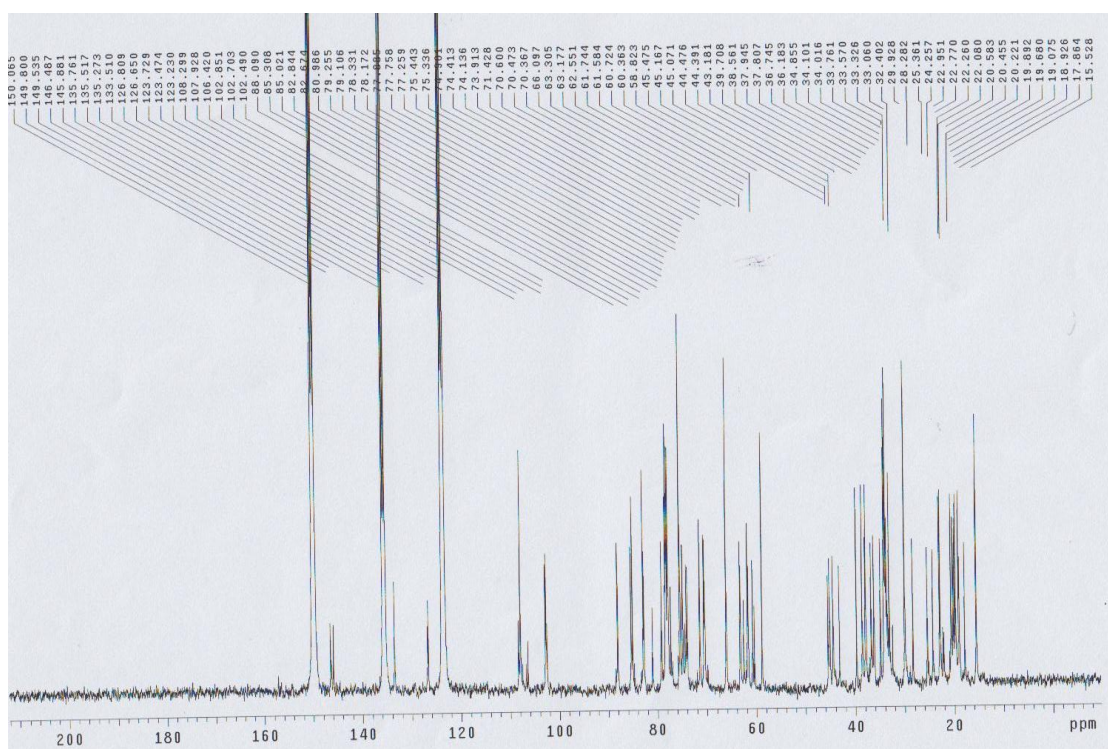


Figure S22. <sup>13</sup>C NMR spectrum of hesperuside C (3) in pyridine-*d*<sub>5</sub>, 150MHz.

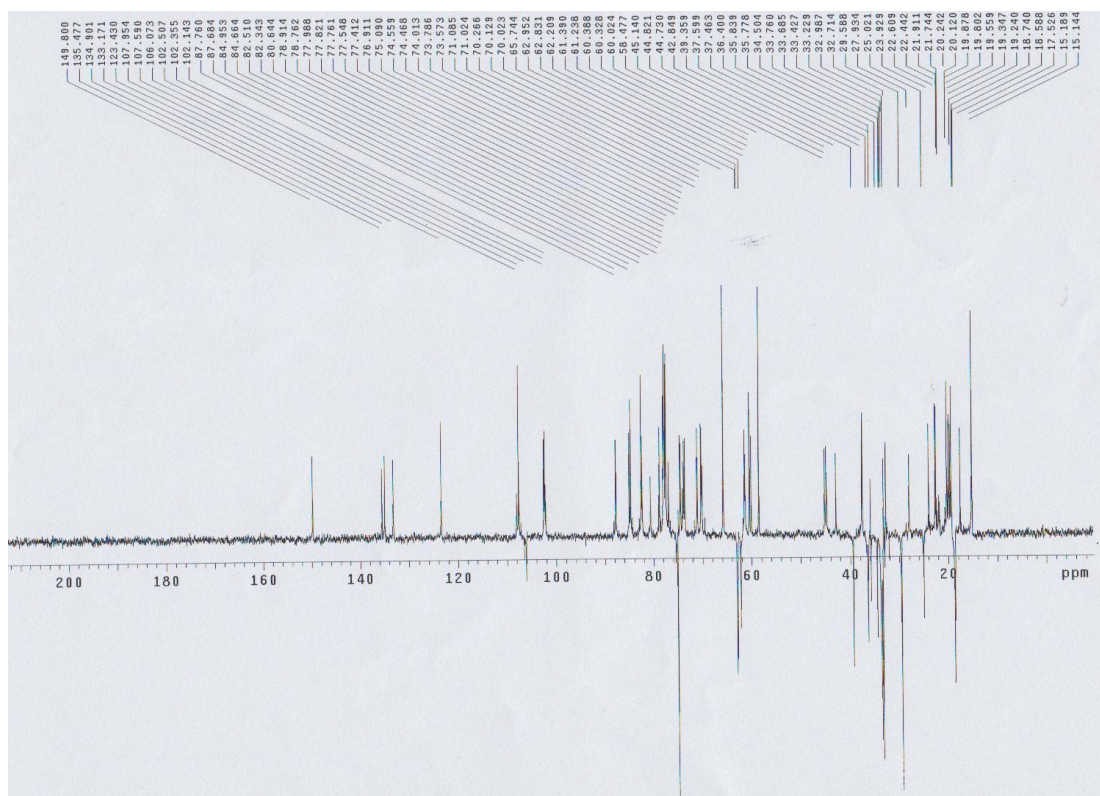


Figure S23. DEPT spectrum of hesperuside C (3).

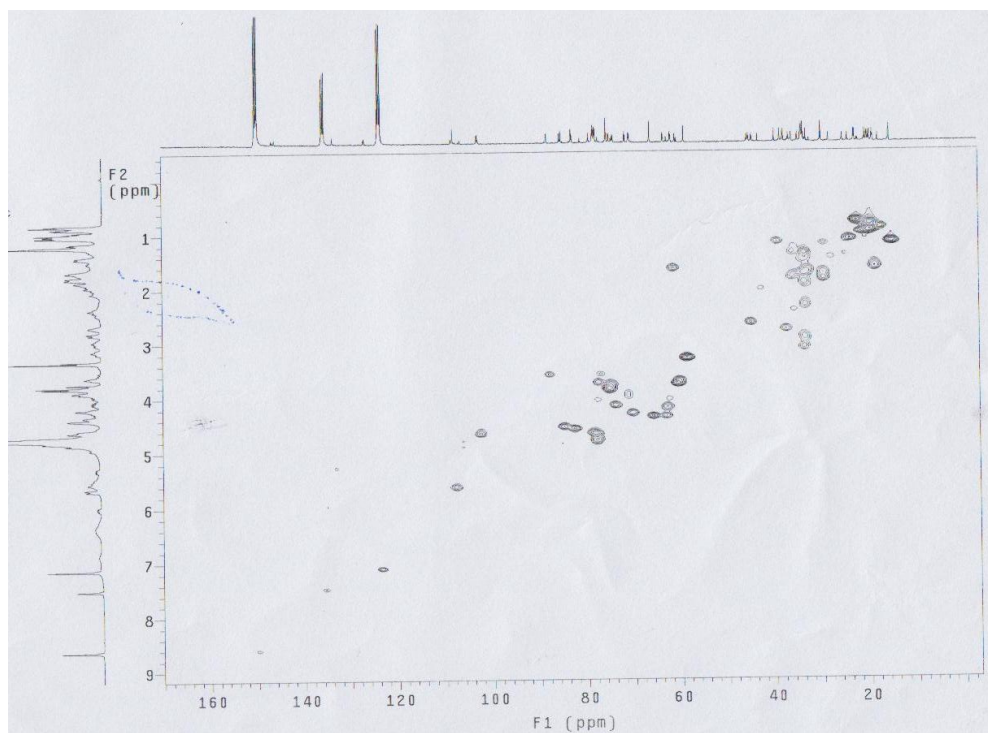


Figure S24. HMQC spectrum of hesperuside C (3).



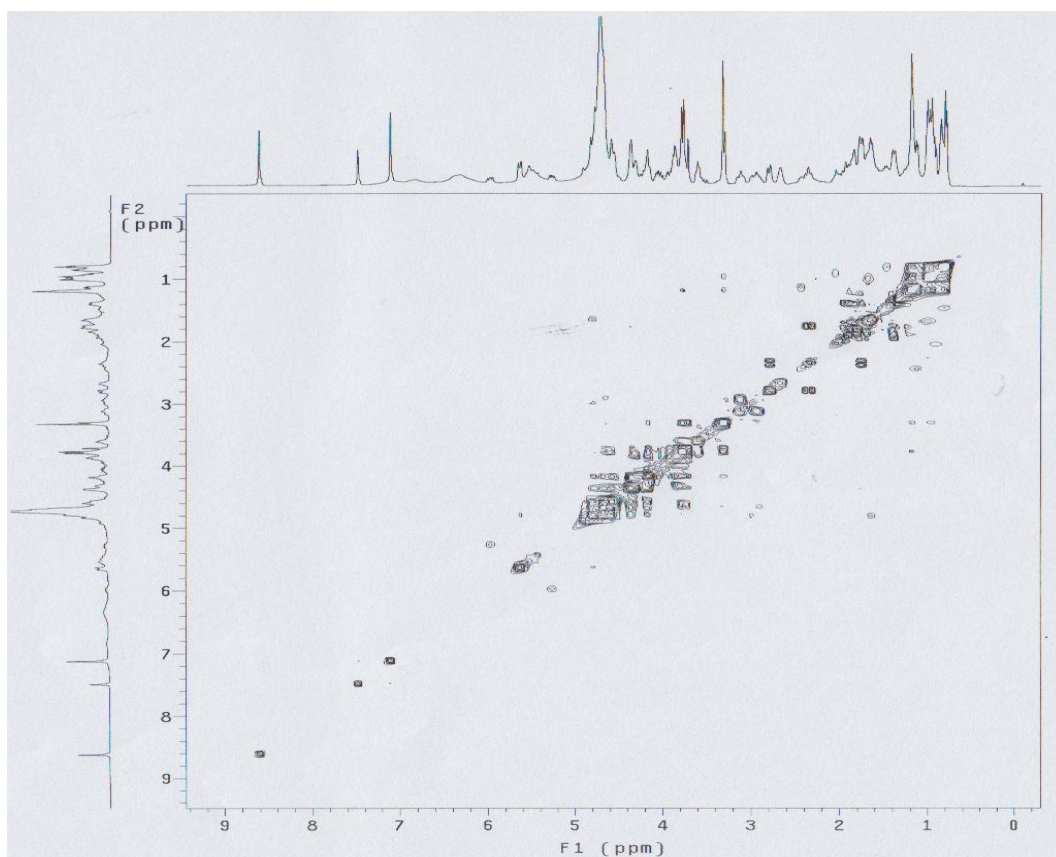


Figure S25. <sup>1</sup>H-<sup>1</sup>H COSY spectrum of hesperuside C (3).

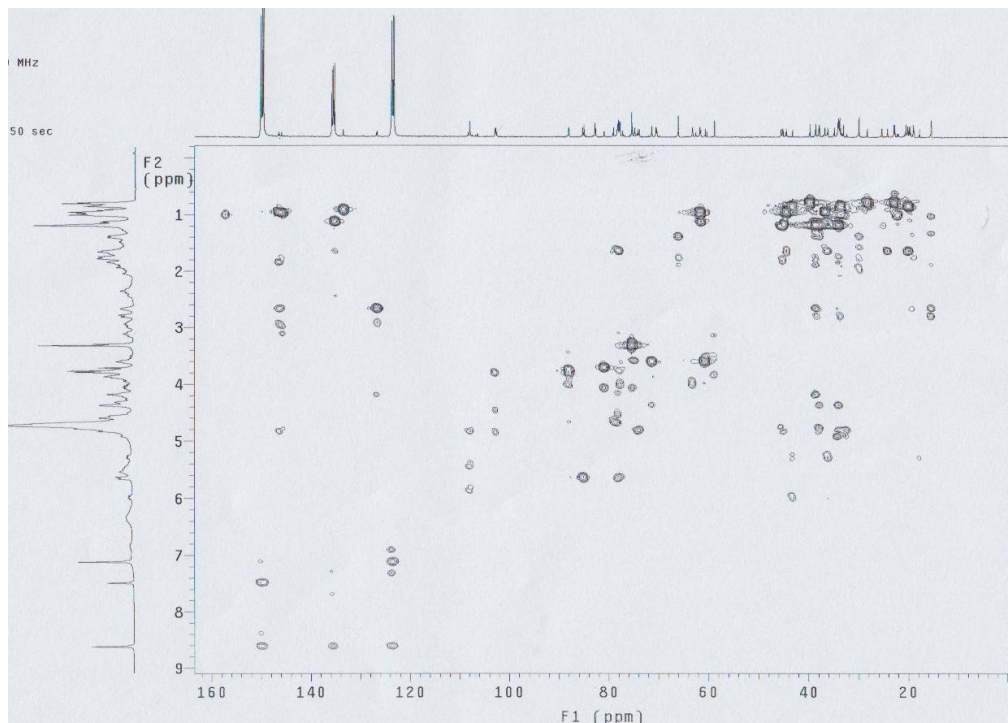


Figure S26. HMBC spectrum of hesperuside C (3).

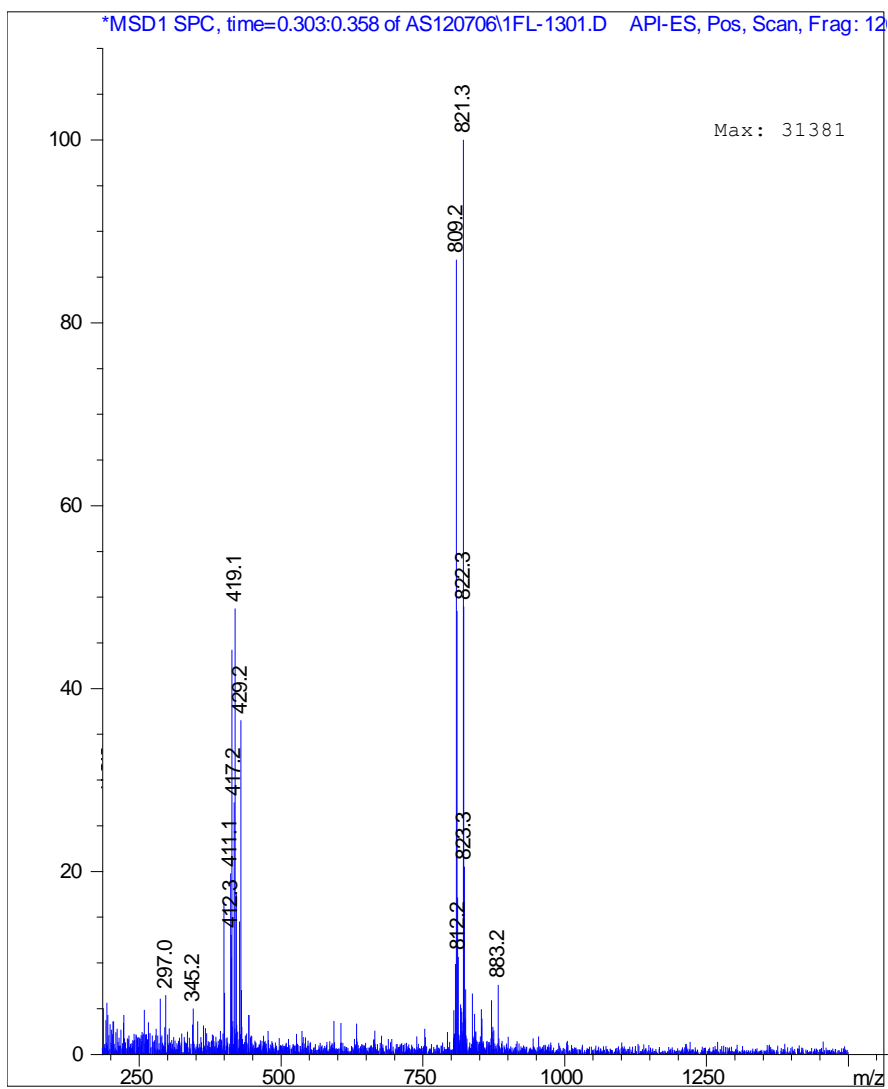


Figure S27. EI-MS spectrum of hesperuside C (3).