

# Supplementary Information

## Piperamides from *Piper ottonoides* by NMR and GC-MS Based Mixture Analysis

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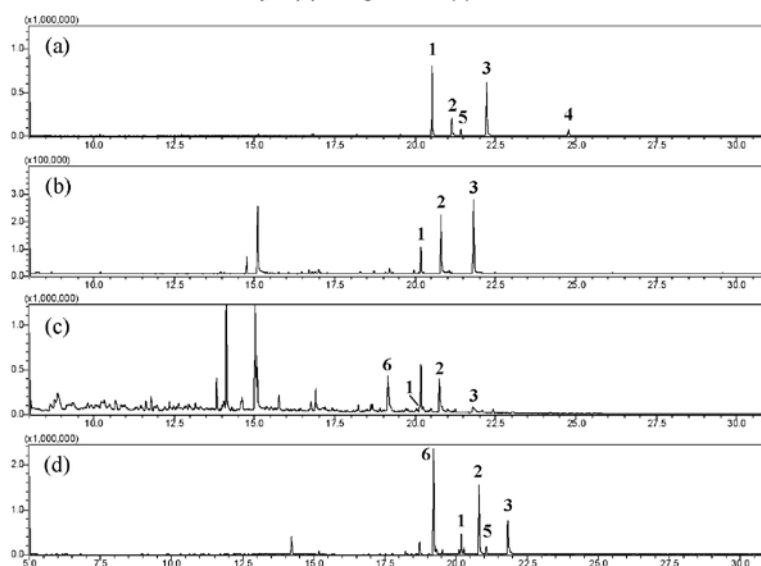
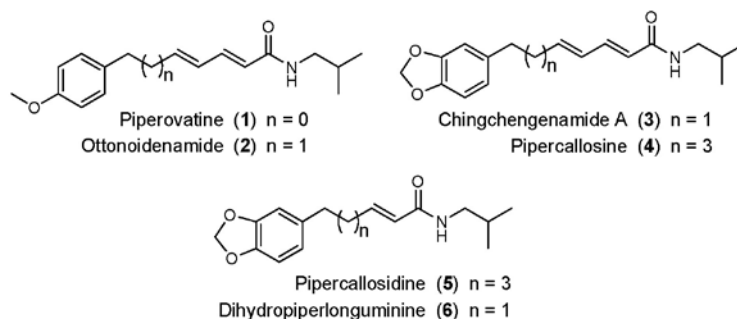
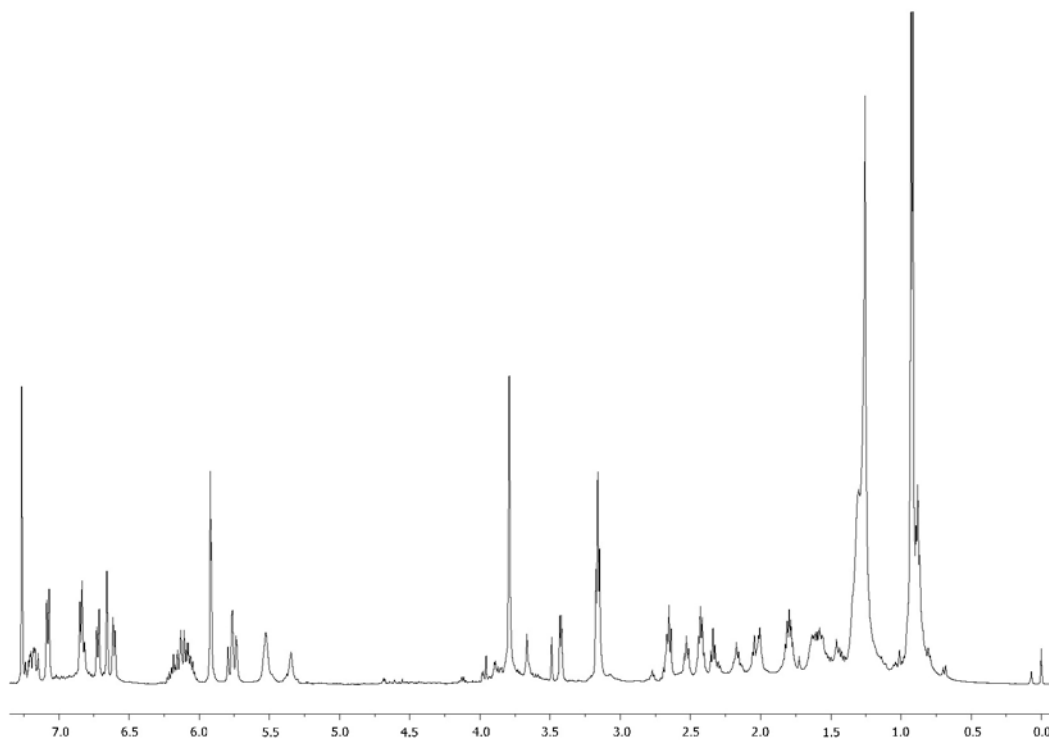
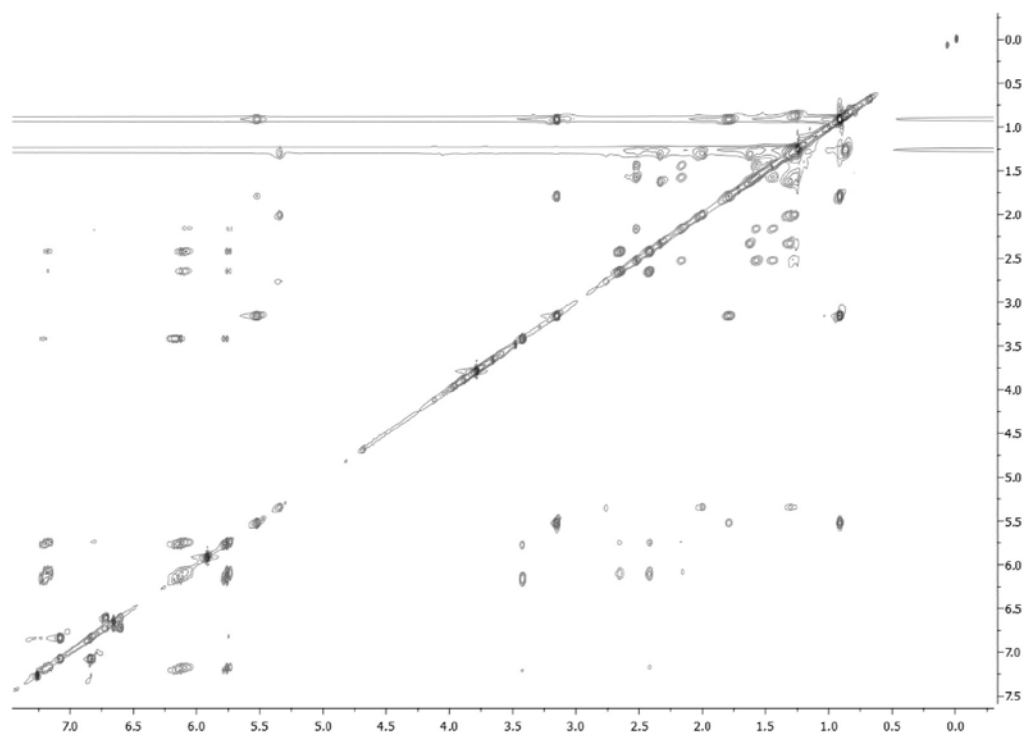


Figure S1. GC-MS chromatograms of *Piper ottonoides* semi-purified fractions from: (a) root; (b) stem; (c) leaf and (d) fruit.

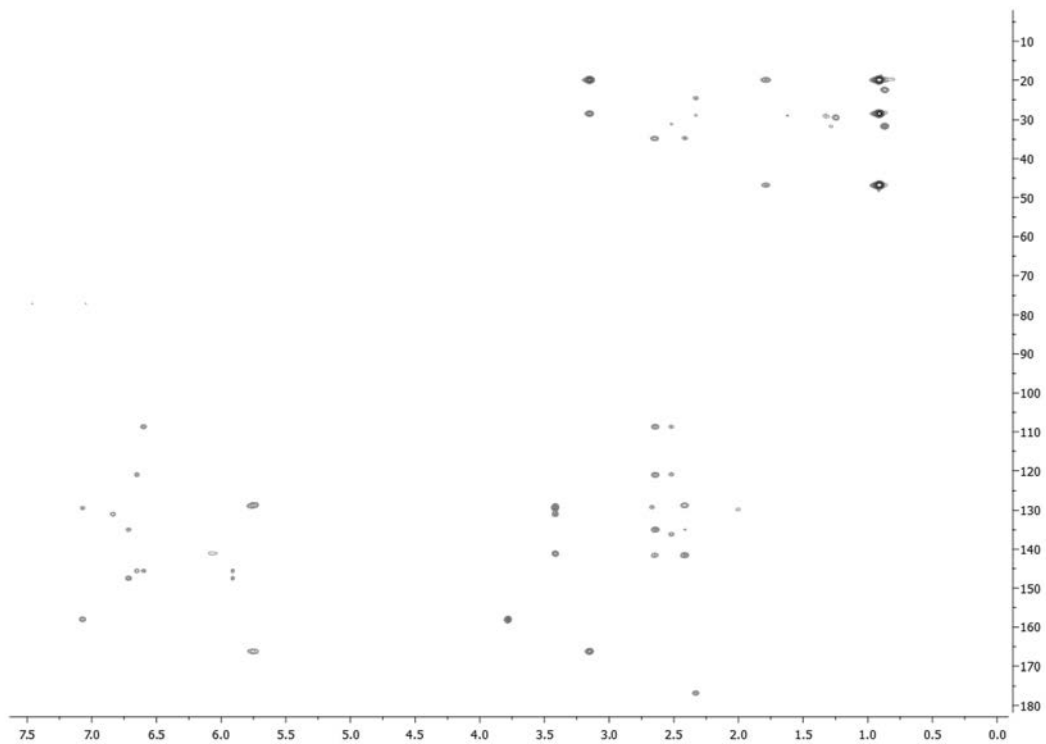
\*e-mail: valente@iq.ufrj.br



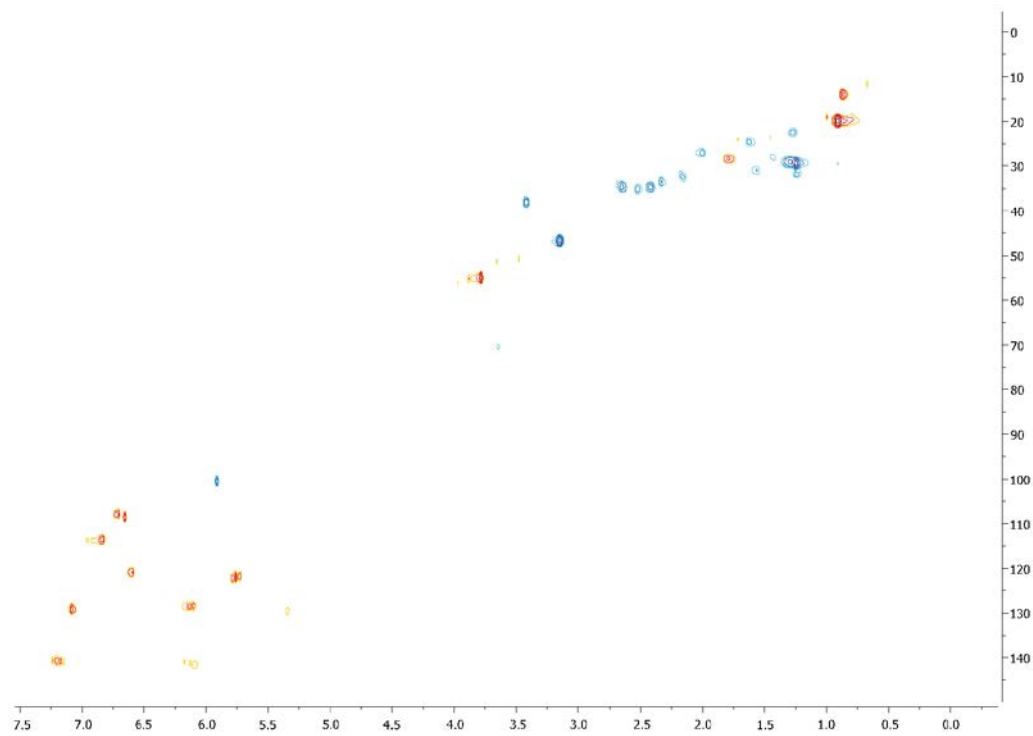
**Figure S2.**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* root fraction.



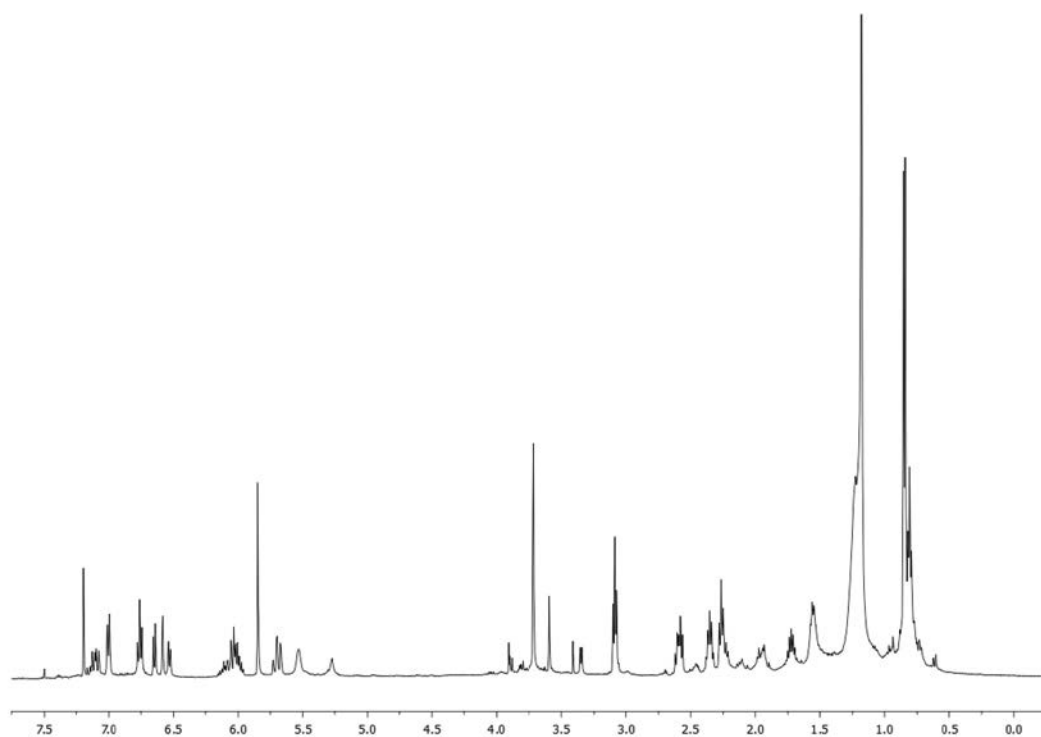
**Figure S3.** TOCSY 2D spectrum of *Piper ottonoides* root fraction.



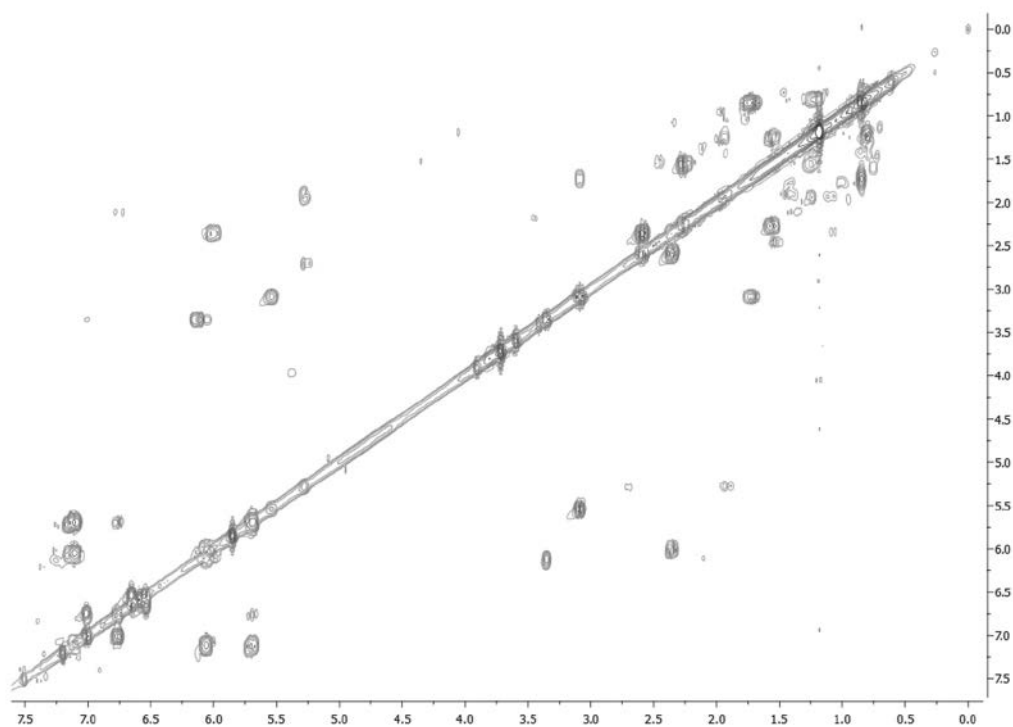
**Figure S4.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC spectrum of *Piper ottonoides* root fraction.



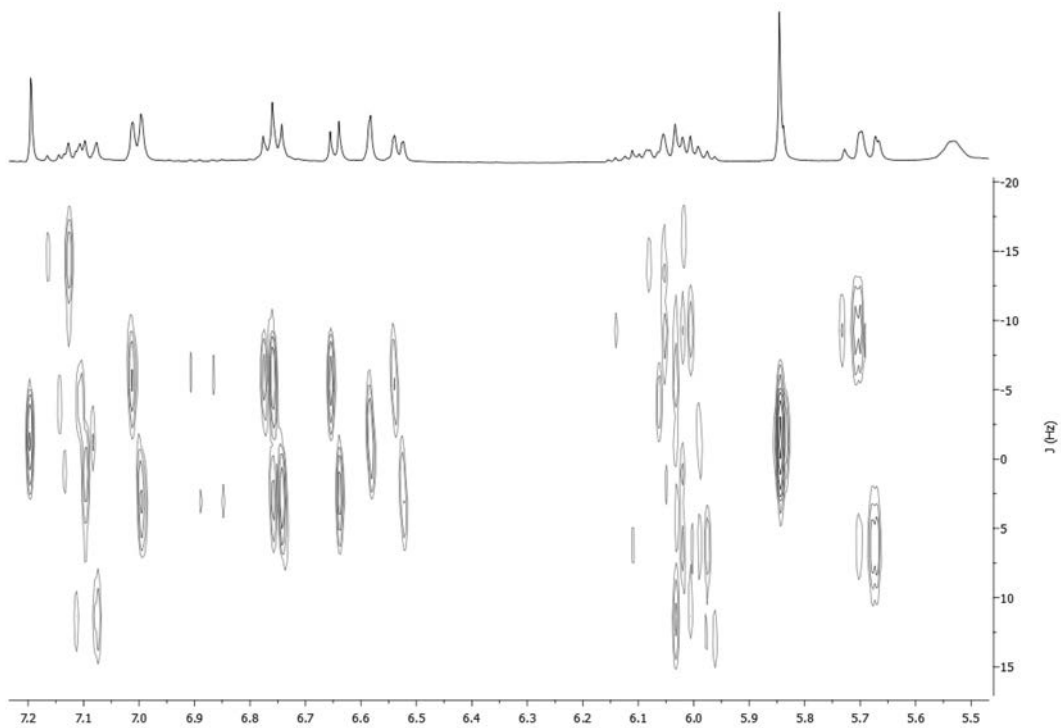
**Figure S5.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC spectrum of *Piper ottonoides* root fraction.



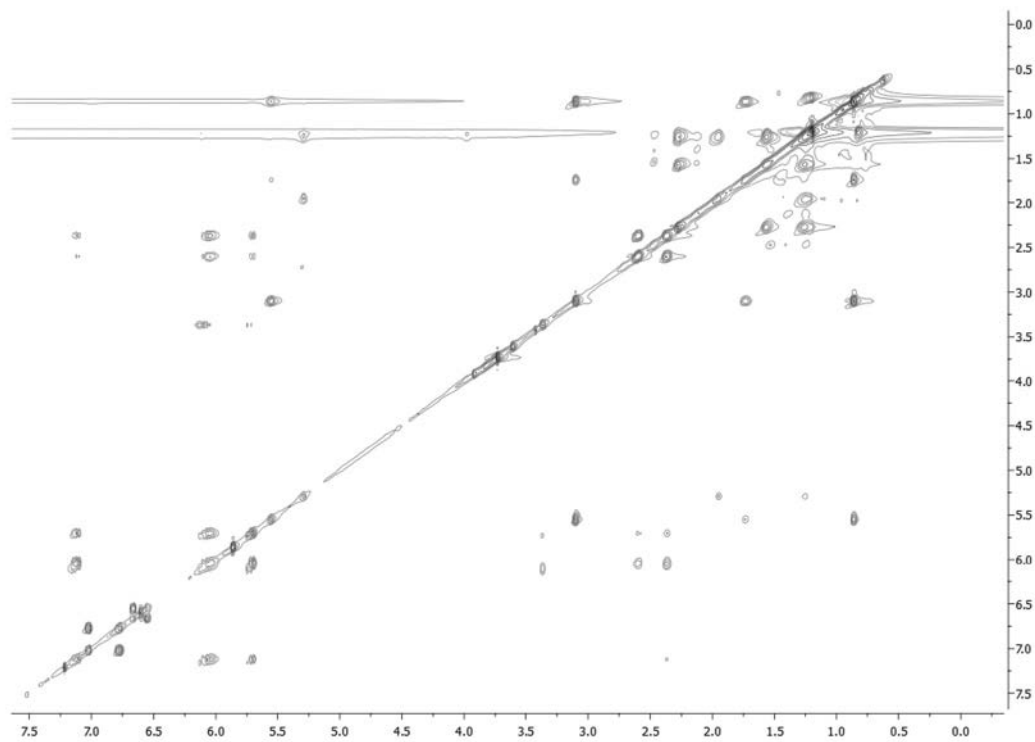
**Figure S6.**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* stem fraction.



**Figure S7.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* stem fraction.



**Figure S8.** J-RES spectrum of *Piper ottonoides* stem fraction.



**Figure S9.** TOCSY 2D spectrum of *Piper ottonoides* stem fraction.

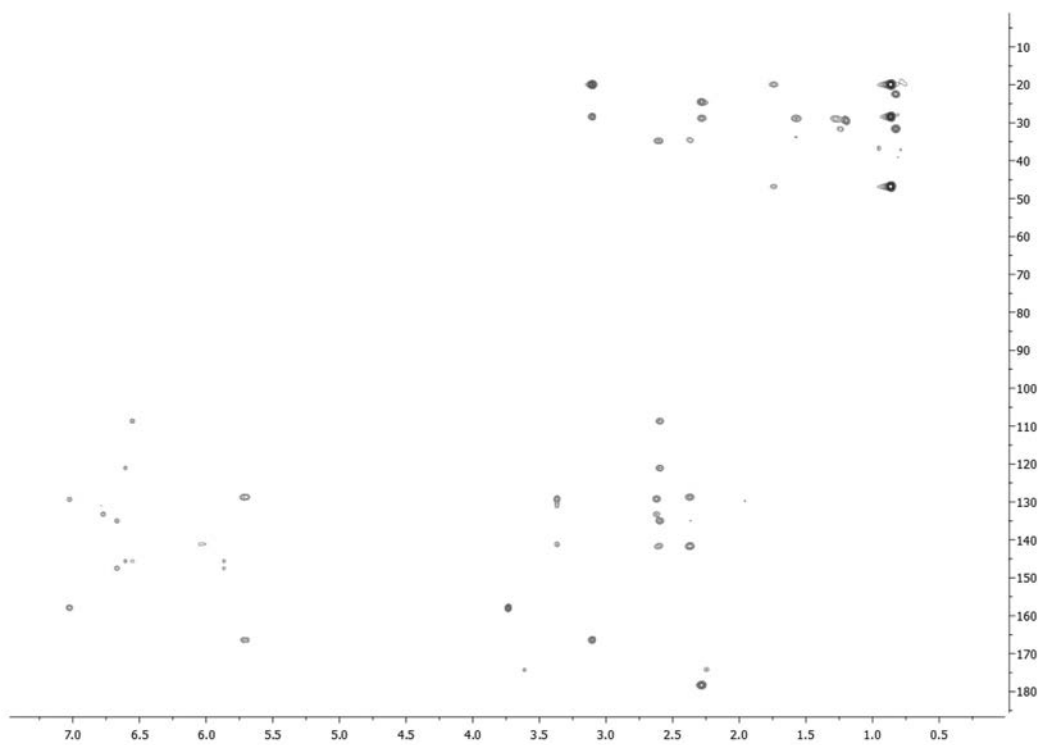


Figure S10.  $^1\text{H}$ - $^{13}\text{C}$  HMBC spectrum of *Piper ottonoides* stem fraction.

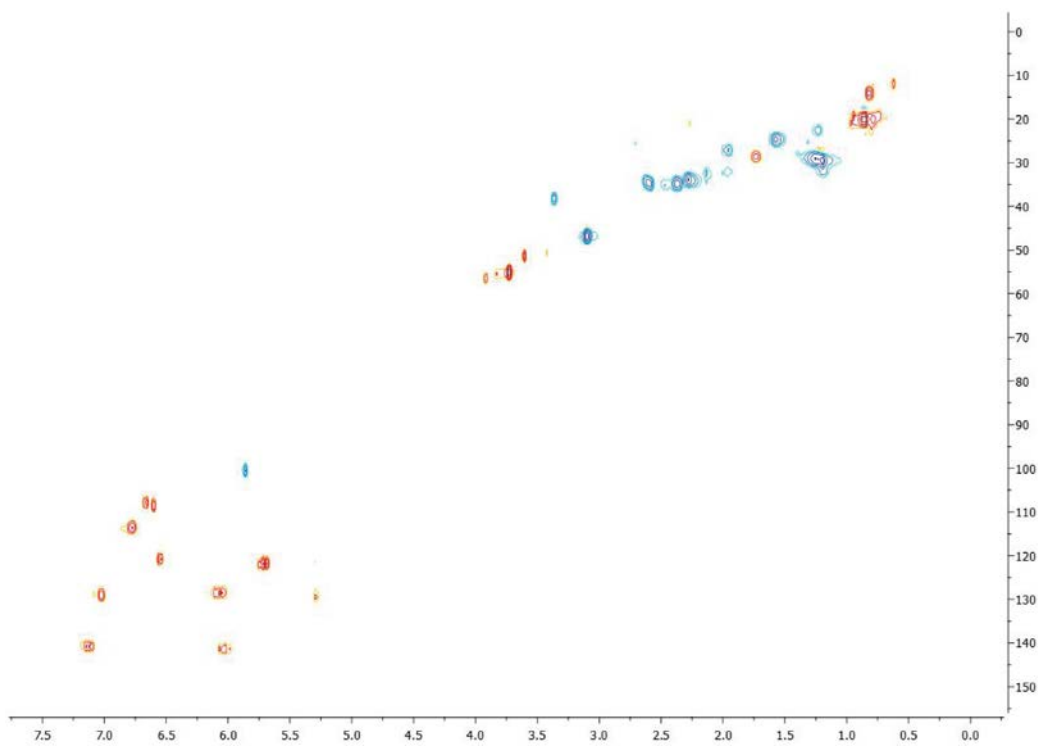
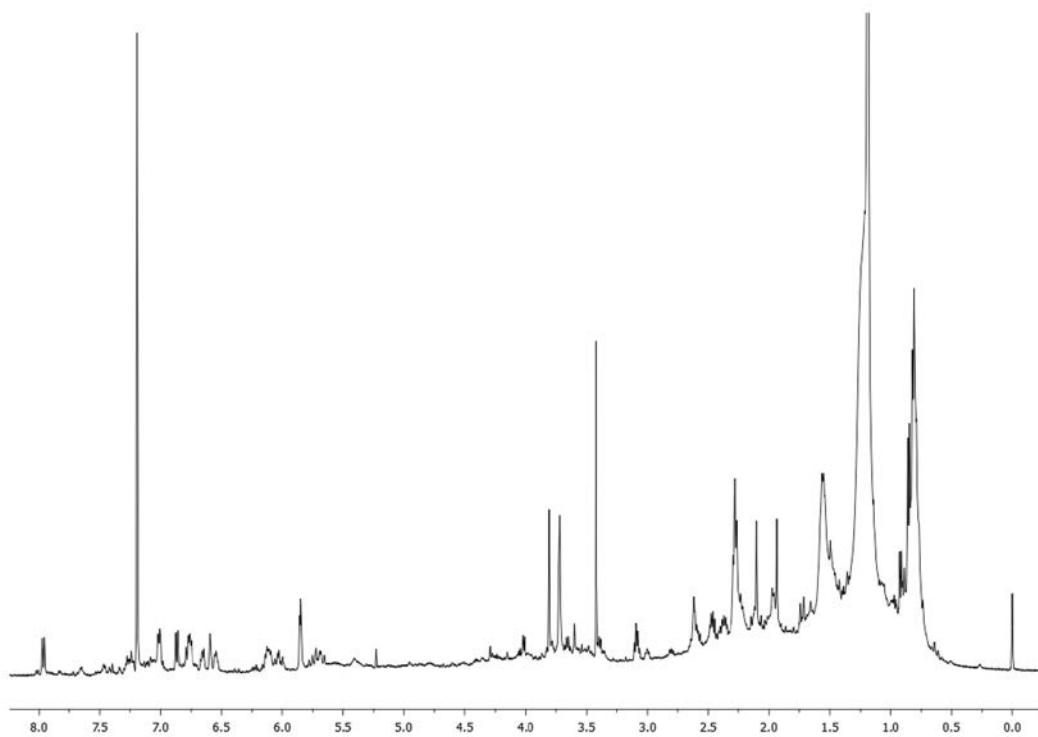
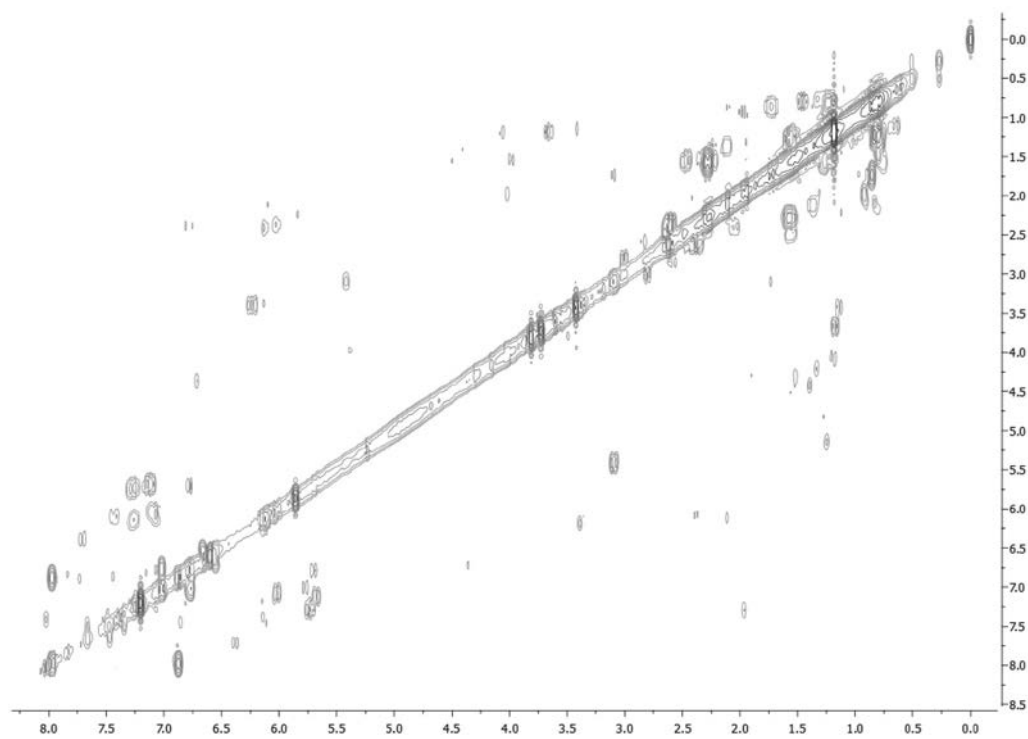


Figure S11.  $^1\text{H}$ - $^{13}\text{C}$  HSQC spectrum of *Piper ottonoides* stem fraction.



**Figure S12.**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* leaf fraction.



**Figure S13.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* leaf fraction.

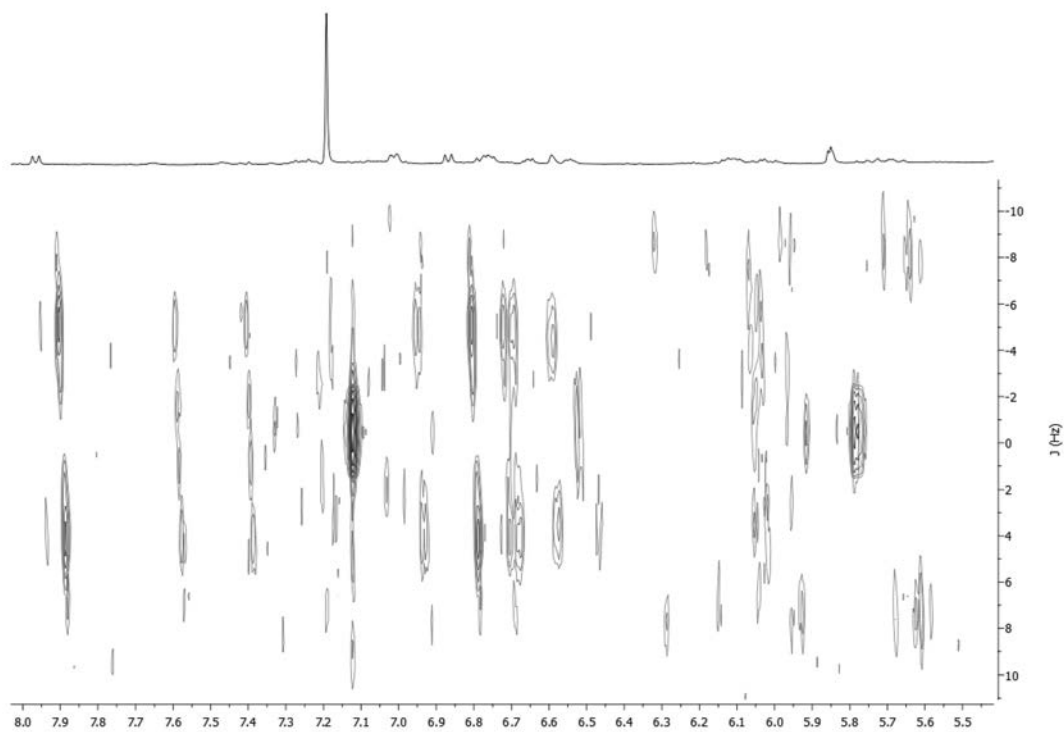


Figure S14. J-RES spectrum of *Piper ottonoides* leaf fraction.

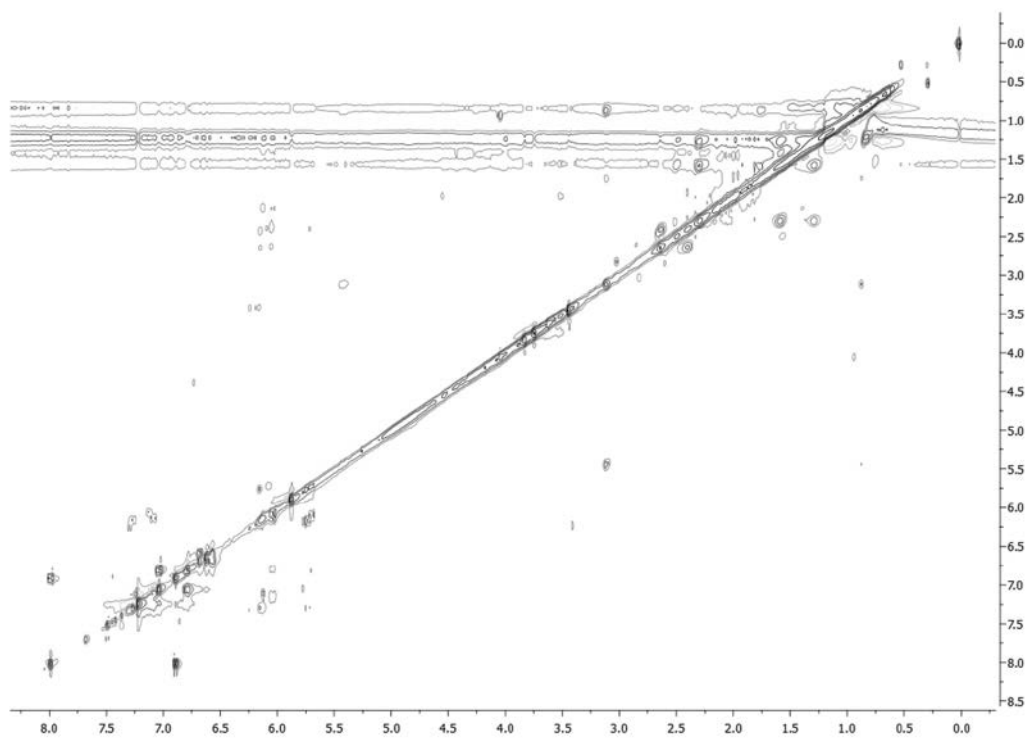
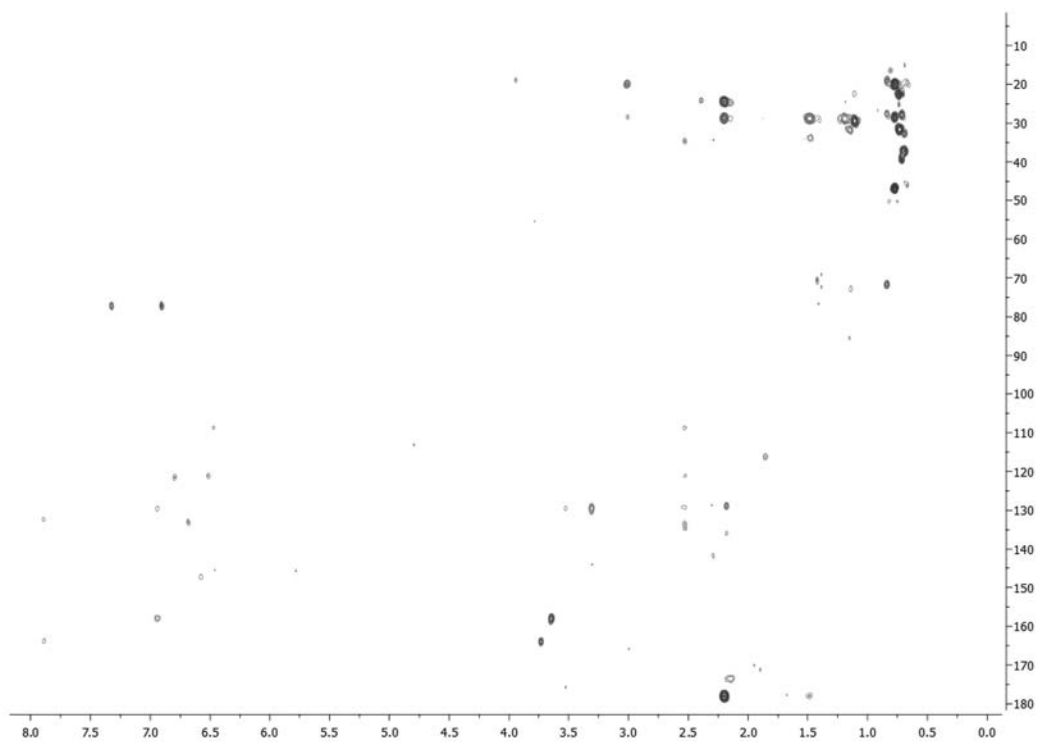
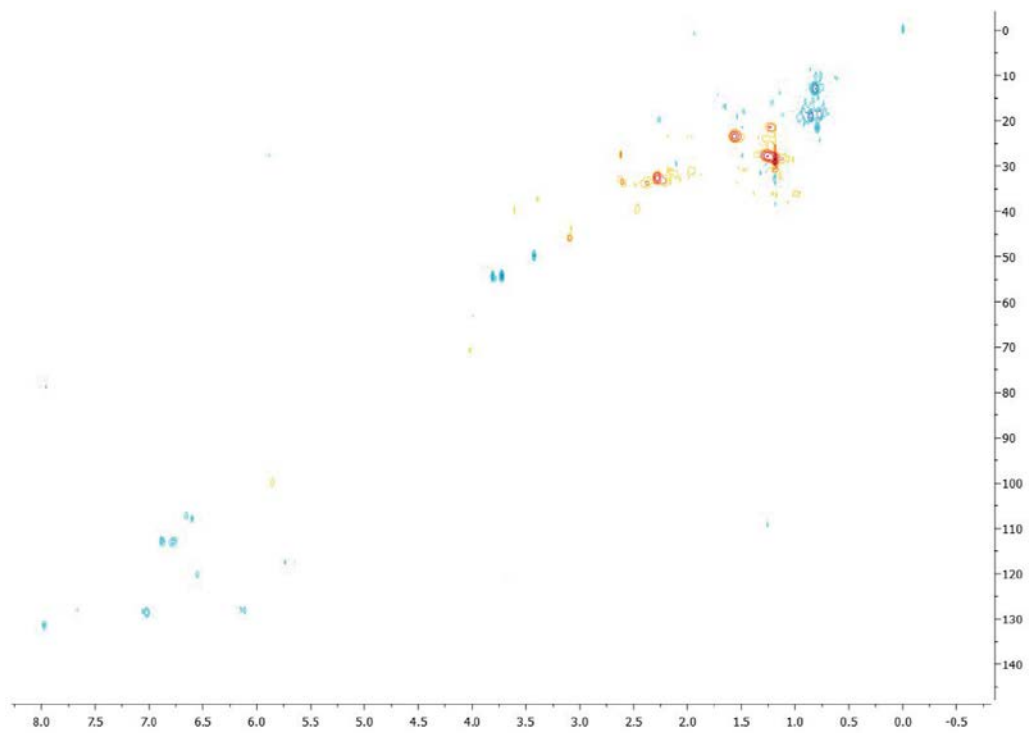


Figure S15. TOCSY 2D spectrum of *Piper ottonoides* leaf fraction.

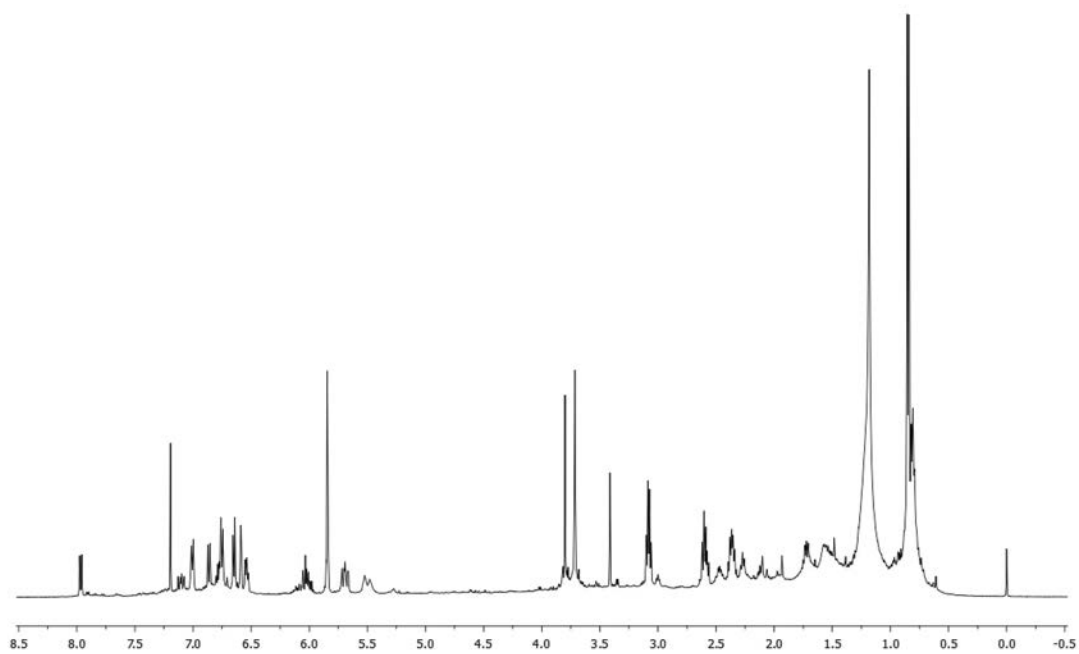




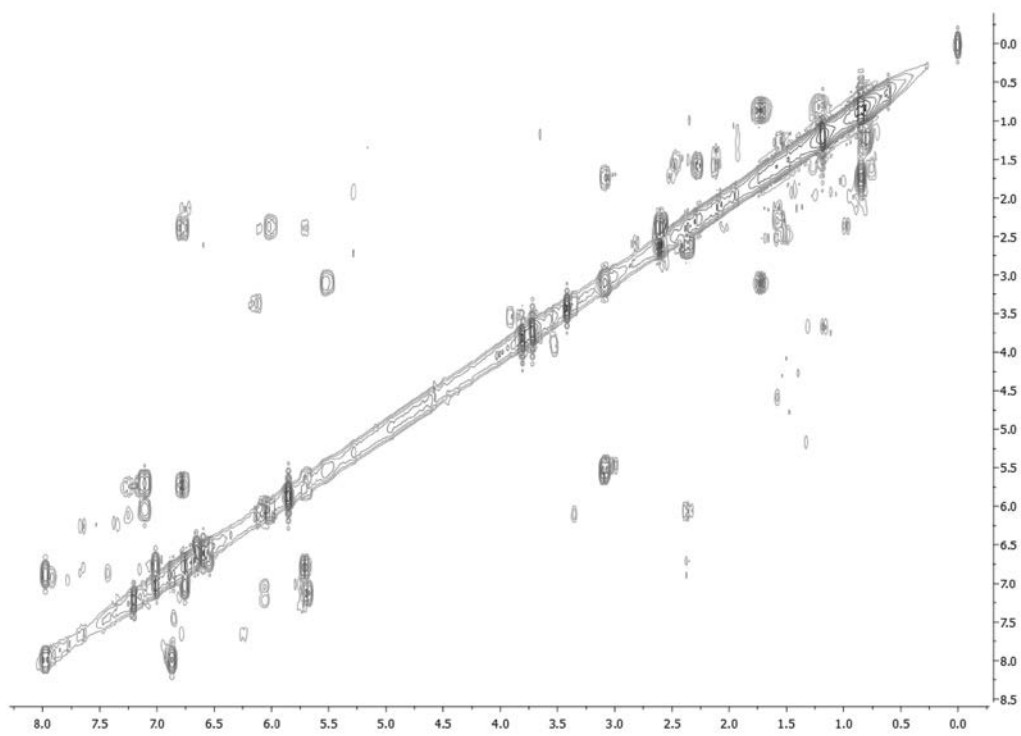
**Figure S16.**  $^1\text{H}$ - $^{13}\text{C}$  HMBC spectrum of *Piper ottonoides* leaf fraction.



**Figure S17.**  $^1\text{H}$ - $^{13}\text{C}$  HSQC spectrum of *Piper ottonoides* leaf fraction.



**Figure S18.**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* fruit fraction.



**Figure S19.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum (500 MHz,  $\text{CDCl}_3$ ) of *Piper ottonoides* fruit fraction.

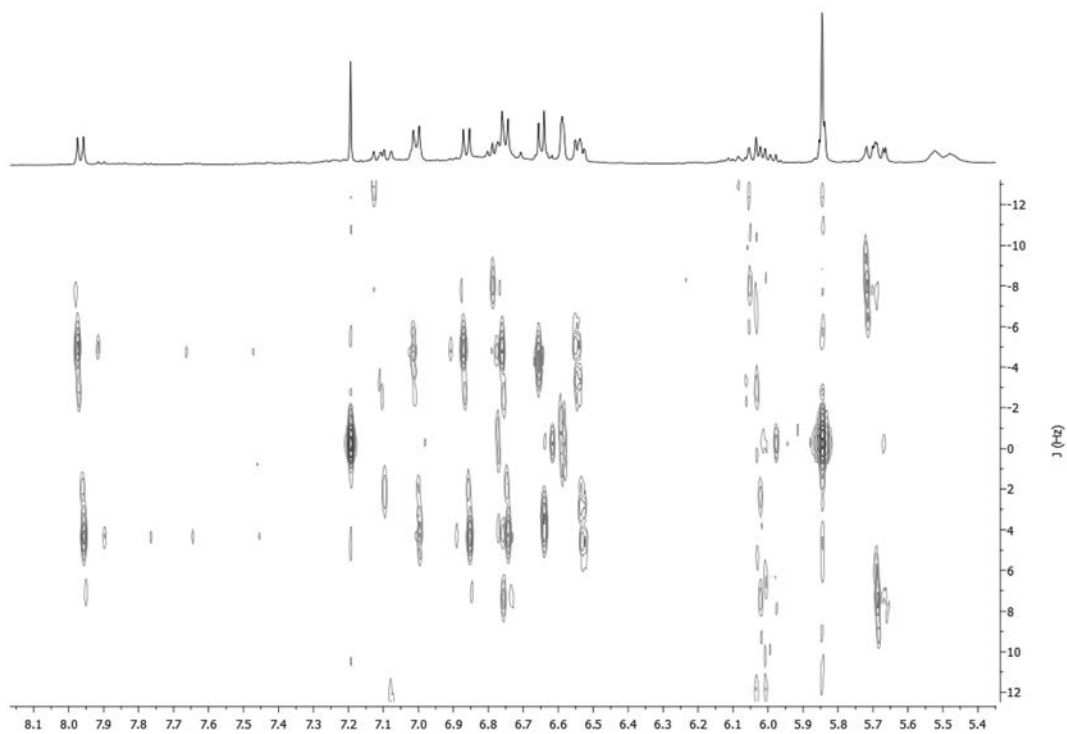


Figure S20. J-RES spectrum of *Piper ottonoides* fruit fraction.

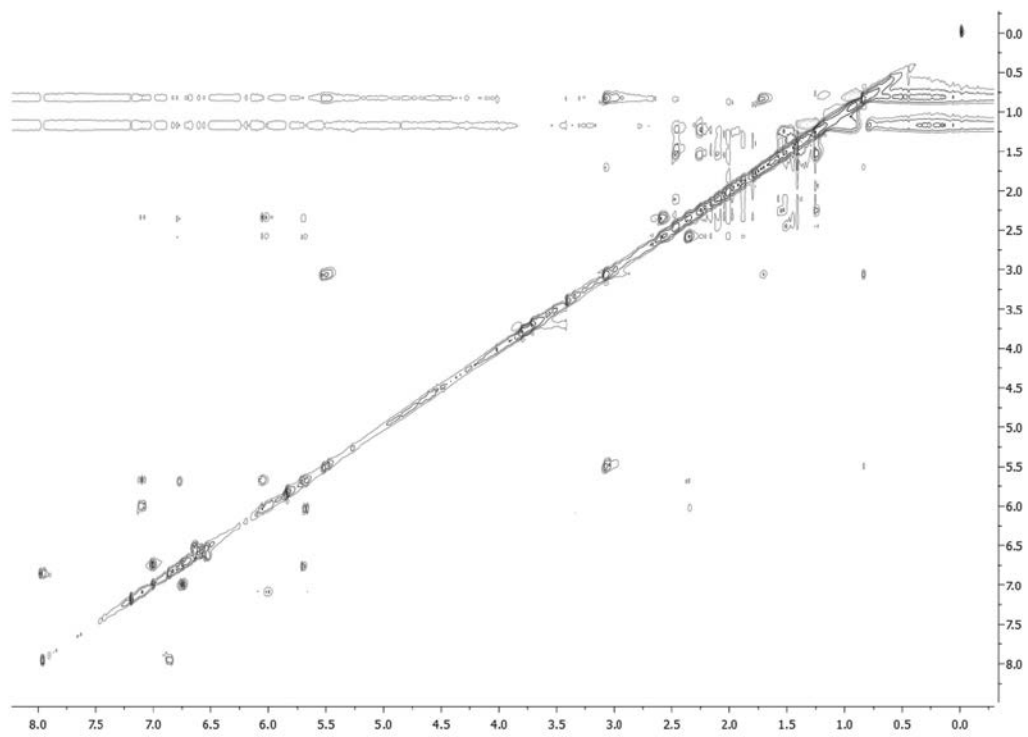


Figure S21. TOCSY 2D spectrum of *Piper ottonoides* fruit fraction.

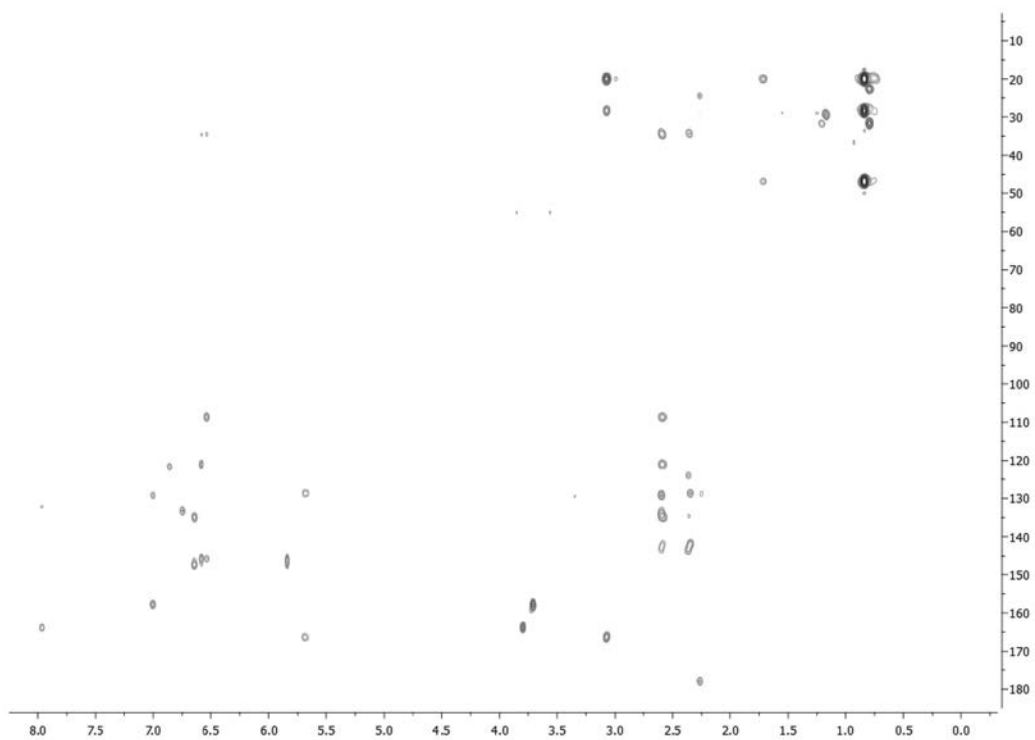


Figure S22.  $^1\text{H}$ - $^{13}\text{C}$  HMBC spectrum of *Piper ottonoides* fruit fraction.

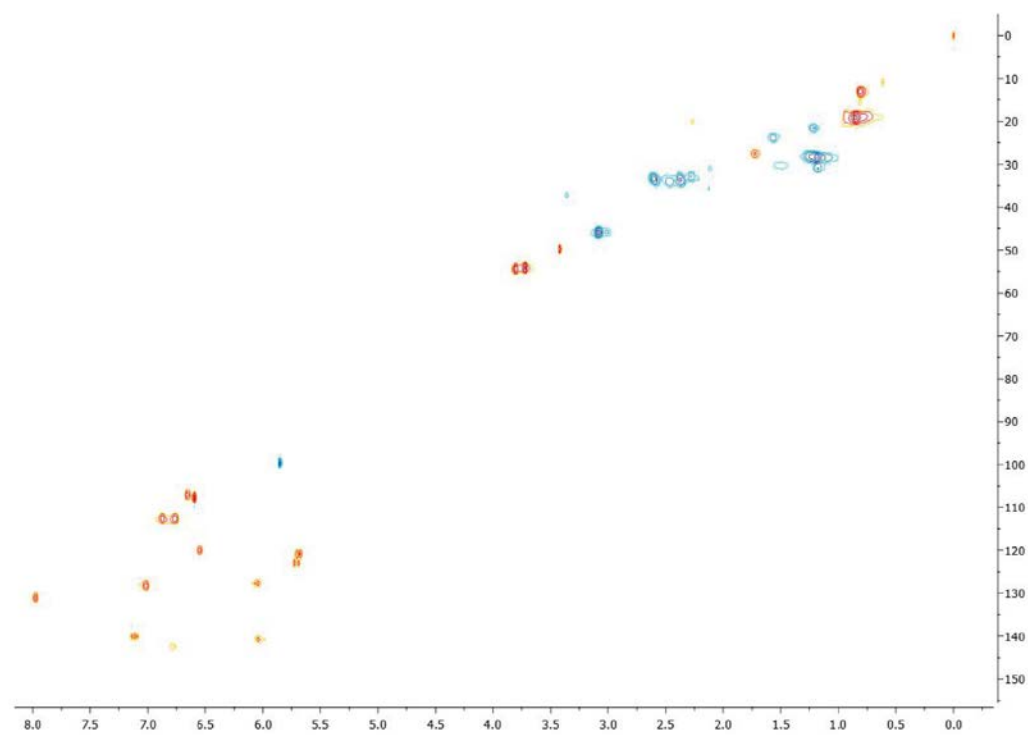
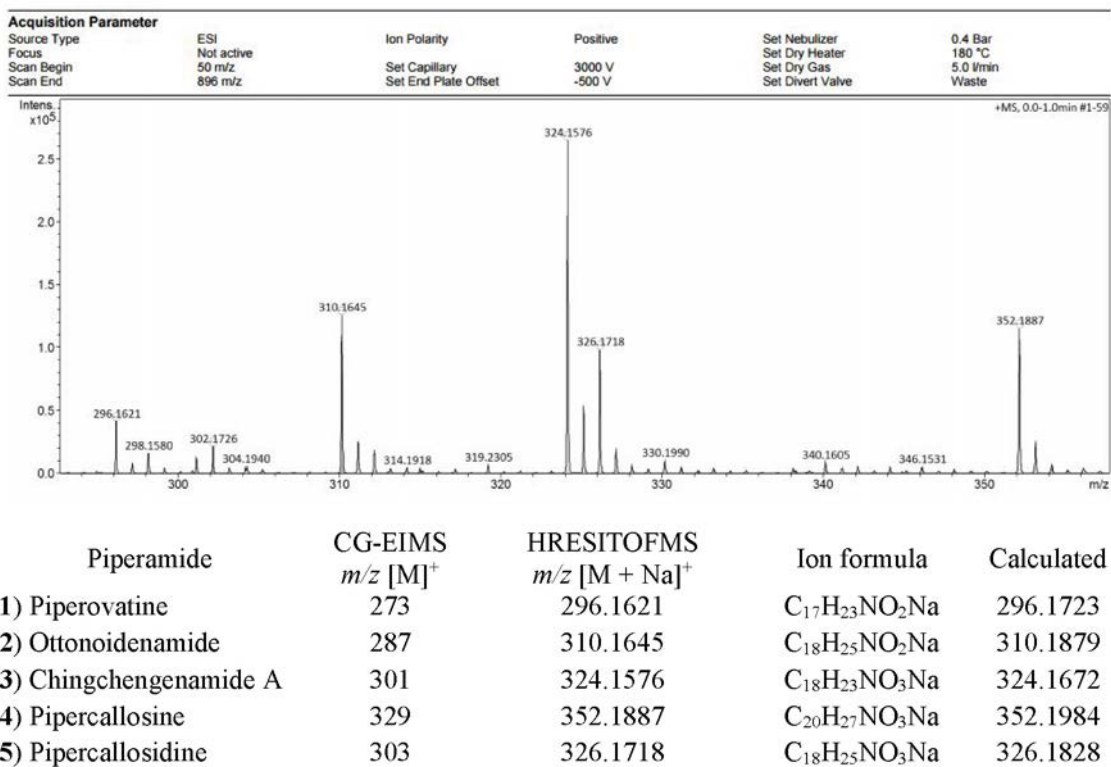


Figure S23.  $^1\text{H}$ - $^{13}\text{C}$  HSQC spectrum of *Piper ottonoides* fruit fraction.



**Figure S24.** Positive HRESITOFMS analysis of the root fraction.

**Table S1.** <sup>1</sup>H (500 MHz) and <sup>13</sup>C (by HSQC and HMBC) NMR data of compounds **1**, **3-6** in CDCl<sub>3</sub>

Position	<sup>1</sup> H [δ in ppm, mult. (J in Hz)]					<sup>13</sup> C [δ in ppm]				
	(1)	(3)	(4)	(5)	(6)	(1)	(3)	(4)	(5)	(6)
1	–	–	–	–	–	166.4	166.4	166.4	166.4	166.3
2	5.78 d (15.8)	5.78 d (15.8)	5.78 d (15.8)	5.75 d (13.2)	5.70 d (15.0)	122.2	122.2	122.2	122.0	123.1
3	7.19 dd (15.6 and 10.9)	7.19 dd (15.6 and 10.9)	7.19 dd (15.6 and 10.9)	6.81 m	6.78 m	141.0	141.0	141.0	144.4	142.8
4	6.12 m	6.12 m	6.12 m	2.18 m	2.36 m	128.9	128.9	128.9	32.7	33.8
5	6.12 m	6.12 m	6.12 m	1.49 m	2.61 m	141.8	141.8	141.8	23.6	33.8
6	3.42 d (6.2)	2.42 q (6.9)	2.17 m	1.59 m	–	38.4	34.9	32.4	31.1	–
7	–	2.65 t (7.2)	1.45 m	2.52 t (7.4)	–	–	34.9	28.7	35.2	–
8	–	–	1.59 m	–	–	–	–	31.2	–	–
9	–	–	2.50 t (7.4)	–	–	–	–	35.4	–	–
1'	–	–	–	–	–	129.4	134.5	136.2	136.2	135.0
2'	7.08 d (8.2)	6.66 bs	6.66 bs	6.66 bs	6.59 bs	129.4	108.8	108.8	108.8	107.8
3'	6.84 d (8.2)	–	–	–	–	113.9	147.6	147.6	147.6	147.4
4'	–	–	–	–	–	158.0	145.1	145.1	145.1	145.7
5'	6.84 d (8.2)	6.72 d (7.8)	6.72 d (7.8)	6.72 d (7.8)	6.65 d (7.9)	113.9	108.2	108.2	108.2	107.3
6'	7.08 d (8.2)	6.60 d (7.7)	6.60 d (7.7)	6.60 d (7.7)	6.54 d (7.1)	129.4	121.2	121.2	121.2	120.2
1''	3.16 t (6.4)	3.16 t (6.4)	3.16 t (6.4)	3.16 t (6.4)	3.08 t (6.8)	47.1	47.1	47.1	47.1	46.0
2''	1.80 m	1.80 m	1.80 m	1.80 m	1.73 m	28.6	28.6	28.6	28.6	27.7
3''	0.92 d (6.5)	0.92 d (6.5)	0.92 d (6.5)	0.92 d (6.5)	0.85 d (6.7)	20.1	20.1	20.1	20.1	19.1
4''	0.92 d (6.5)	0.92 d (6.5)	0.92 d (6.5)	0.92 d (6.5)	0.85 d (6.7)	20.1	20.1	20.1	20.1	19.1
OCH <sub>3</sub>	3.79 s	–	–	–	–	55.3	–	–	–	–
OCH <sub>2</sub> O	–	5.92 s	5.92 s	5.92 s	5.85 s	–	100.8	100.8	100.8	99.8
NH	5.53 bs	5.53 bs	5.53 bs	5.53 bs	5.50 bs	–	–	–	–	–