

Supplementary Files

# Isolation and HPLC quantitative determination of 5 $\alpha$ -reductase inhibitors from *Tectona grandis* L.f. leaf extract

Kamonlak Insumrong <sup>1</sup>, Kornkanok Ingkaninan <sup>2</sup>, Neti Waranuch <sup>3</sup>, Nuchaninad Tanuphol <sup>2</sup>,  
Wudtichai Wisuitiprot <sup>4</sup>, Trinop Promgool <sup>3</sup>, and Nungruthai Suphrom <sup>1,5,\*</sup>

<sup>1</sup> Department of Chemistry, Faculty of Science, Naresuan University, Phitsanulok 65000, Thailand; kamonlaki59@nu.ac.th

<sup>2</sup> Centre of Excellence in Cannabis Research, Department of Pharmaceutical Chemistry and Pharmacognosy, Faculty of Pharmaceutical Sciences and Center of Excellence for Innovation in Chemistry, Naresuan University, Phitsanulok 65000, Thailand; k\_ingkaninan@yahoo.com (K.I.); nuchaninadt62@nu.ac.th (N.T.)

<sup>3</sup> Cosmetics and Natural Products Research Center and Center of Excellence for Innovation in Chemistry, Department of Pharmaceutical Technology, Faculty of Pharmaceutical Sciences, Naresuan University, Phitsanulok 65000, Thailand; netiw@nu.ac.th (N.W.); tpromgool@gmail.com (T.P.)

<sup>4</sup> Department of Thai Traditional Medicine, Sirindhorn College of Public Health, Phitsanulok 65130, Thailand; wisuitiprot@hotmail.com

<sup>5</sup> Department of Chemistry, Faculty of Science and Center of Excellence for Innovation in Chemistry, Naresuan University, Phitsanulok 65000, Thailand; suphrom.n1@gmail.com

\* Correspondence: suphrom.n1@gmail.com; Tel.: +66-92-257-31-45; Fax: +66-55-96-34-01

## Supplementary Materials

Figure S1:  $^1\text{H}$ -NMR spectrum of **1** (400 MHz,  $\text{CDCl}_3$ )

Figure S2:  $^{13}\text{C}$ -NMR spectrum of **1** (100 MHz,  $\text{CDCl}_3$ )

Figure S3: HMBC spectrum of **1** (400 MHz for  $^1\text{H}$  and 100 MHz, for  $^{13}\text{C}$ ,  $\text{CDCl}_3$ )

Figure S4: FT-IR (ATR mode) spectrum of **1**

Figure S5: HRESI-MS (negative ion mode) spectrum of **1**

Figure S6:  $^1\text{H}$ -NMR spectrum of **2** (400 MHz,  $\text{CDCl}_3$ )

Figure S7:  $^{13}\text{C}$ -NMR spectrum of **2** (100 MHz,  $\text{CDCl}_3$ )

Figure S8: HMBC spectrum of **2** (400 MHz for  $^1\text{H}$  and 100 MHz, for  $^{13}\text{C}$ ,  $\text{CDCl}_3$ )

Figure S9: FT-IR (ATR mode) spectrum of **2**

Figure S10: HRESI-MS (negative ion mode) spectrum of **2**

Figure S11:  $^1\text{H}$ -NMR spectrum of **3** (400 MHz,  $\text{CDCl}_3$ )

Figure S12:  $^{13}\text{C}$ -NMR spectrum of **3** (100 MHz,  $\text{CDCl}_3$ )

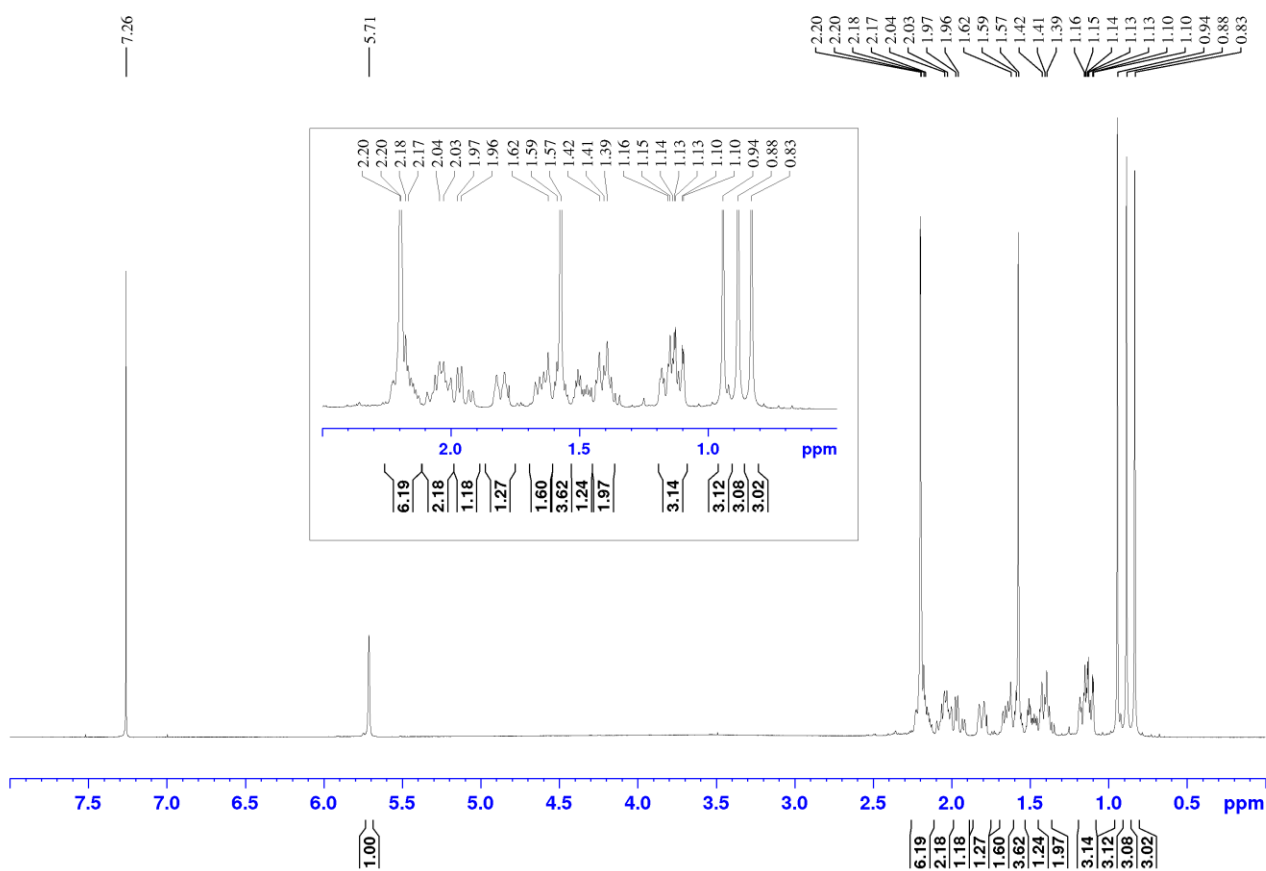
Figure S13: FT-IR (ATR mode) spectrum of **3** and an expanded region of the spectrum (a) chemical shift at 35.0-60.0 ppm and (b) chemical shift at 12.0-32.0 ppm

Figure S14: EI-MS spectrum of **3**

Table S1: NMR data of **1** and **2** (in  $\text{CDCl}_3$ ) measured at 100 ( $^{13}\text{C}$ ) and 400 ( $^1\text{H}$ ) MHz

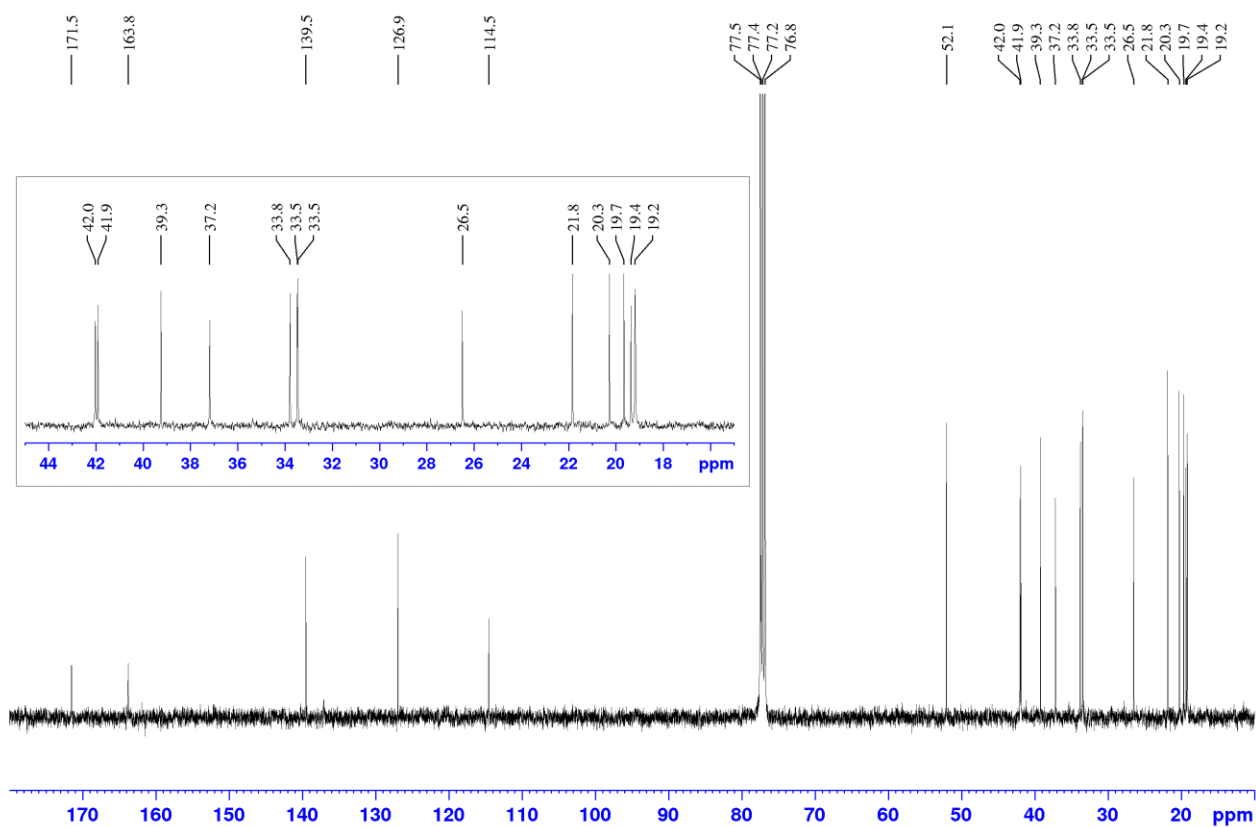
Table S2: NMR data of **3** (in  $\text{CDCl}_3$ ) measured at 100 ( $^{13}\text{C}$ ) and 400 ( $^1\text{H}$ ) MHz

<sup>1</sup>H Compound 1 in CDCl<sub>3</sub> 07/10/2020 (exp 1)



**Figure S1.** <sup>1</sup>H-NMR spectrum of **1** (400 MHz, CDCl<sub>3</sub>)

<sup>13</sup>C Compound 1 in CDCl<sub>3</sub> 07/10/2020 (exp 2) ns-2k



**Figure S2.** <sup>13</sup>C-NMR spectrum of **1** (100 MHz, CDCl<sub>3</sub>)

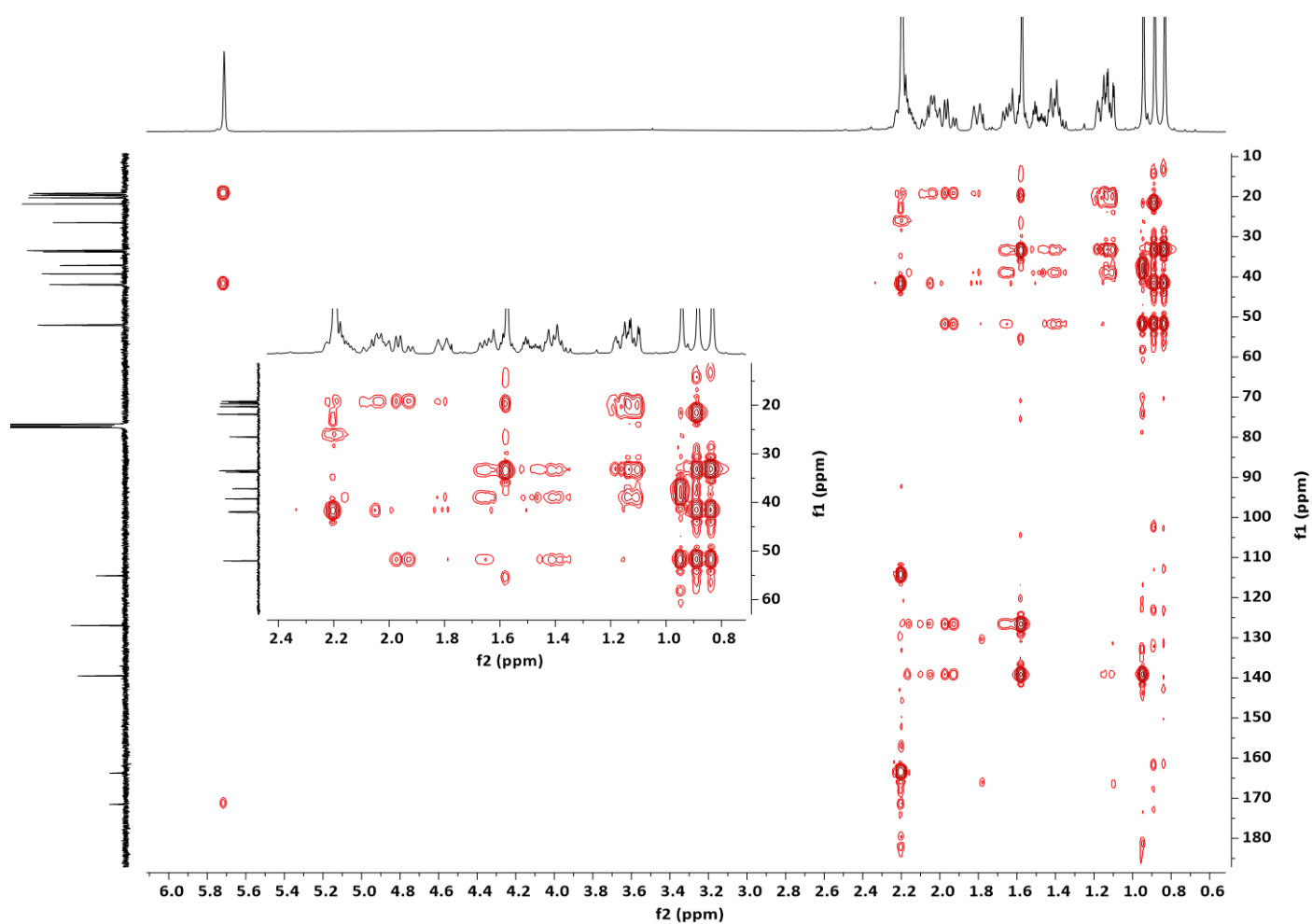


Figure S3: HMBC spectrum of **1** (400 MHz for  $^1\text{H}$  and 100 MHz, for  $^{13}\text{C}$ ,  $\text{CDCl}_3$ )

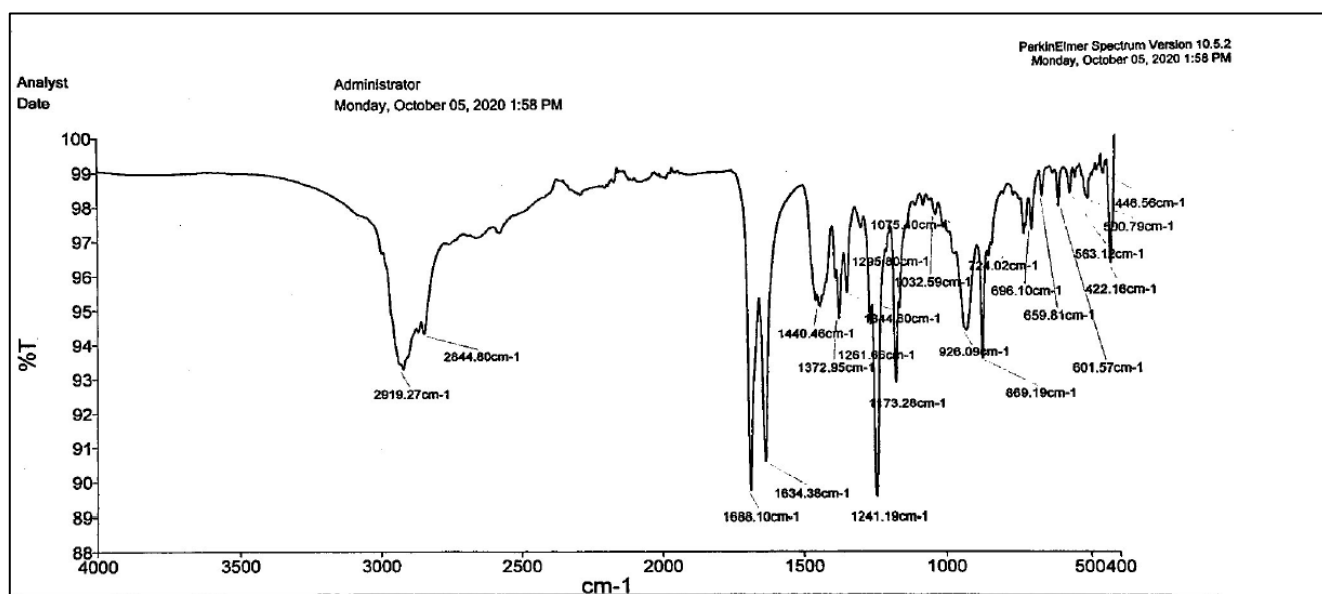


Figure S4: FT-IR (ATR mode) spectrum of 1

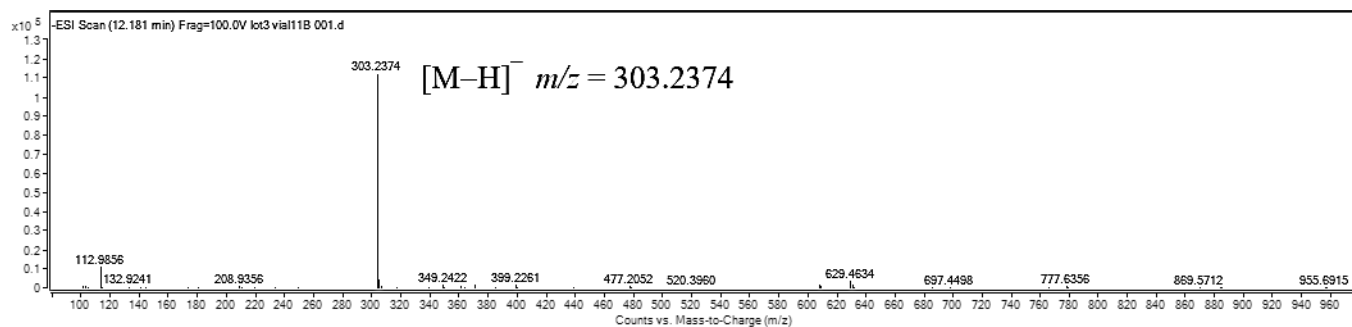


Figure S5: HRESI-MS (negative ion mode) spectrum of 1

<sup>1</sup>H Compound 2 in CDCl<sub>3</sub> 07/10/2020 (exp 7)

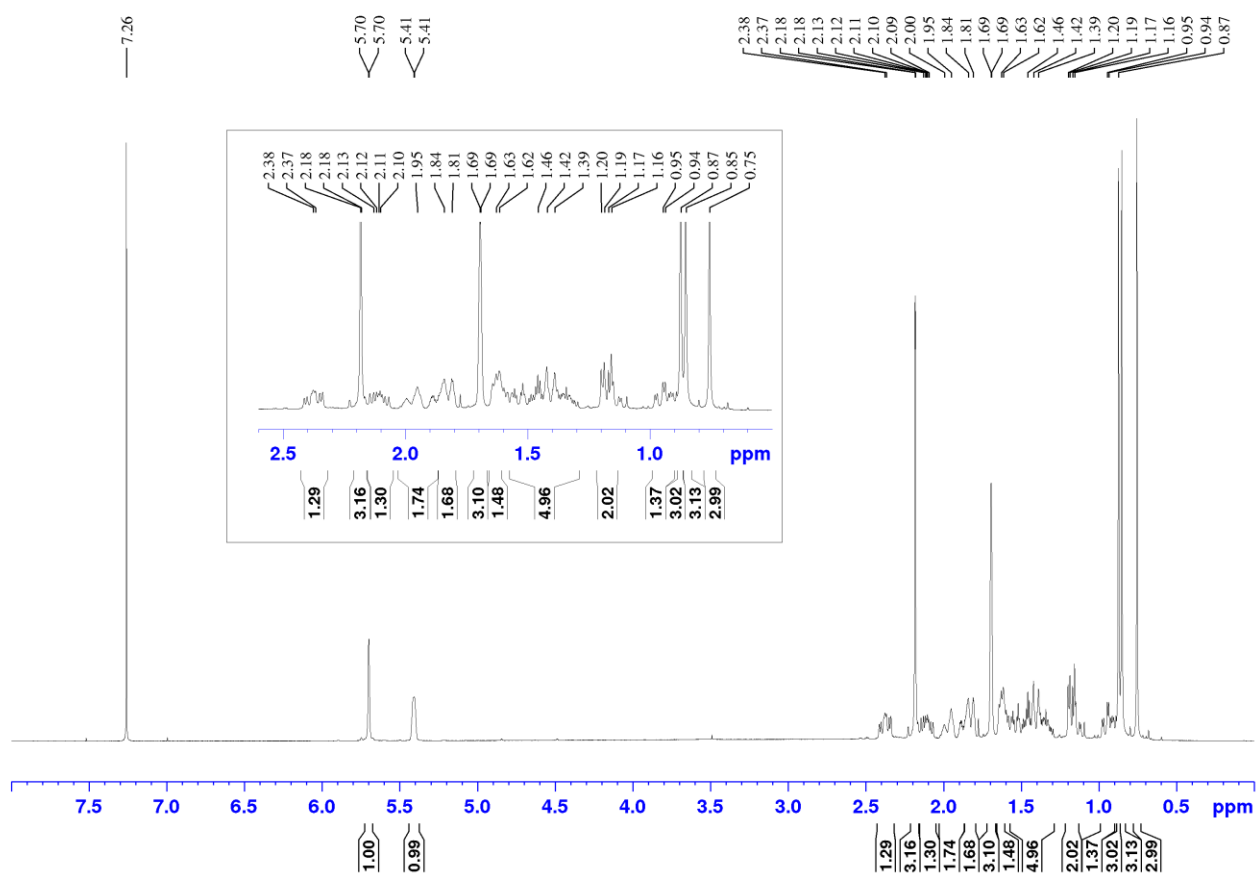
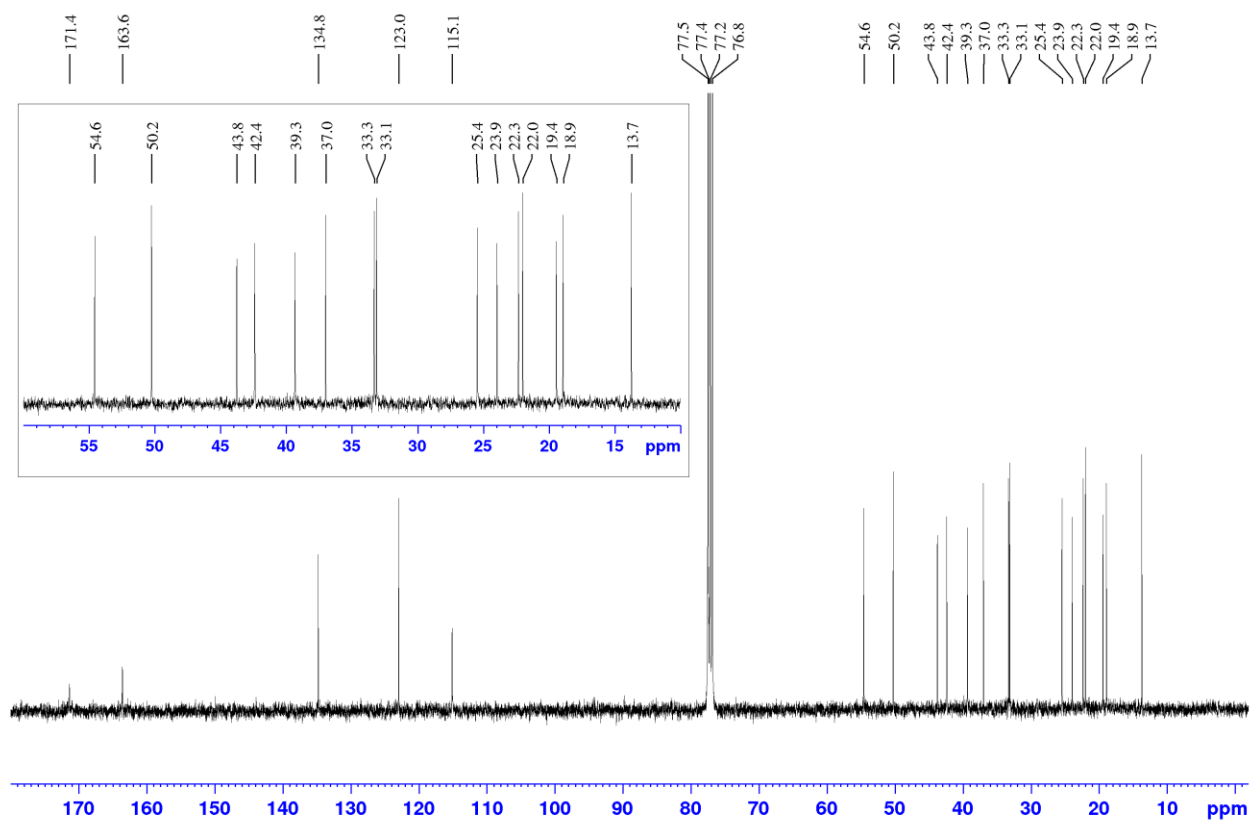


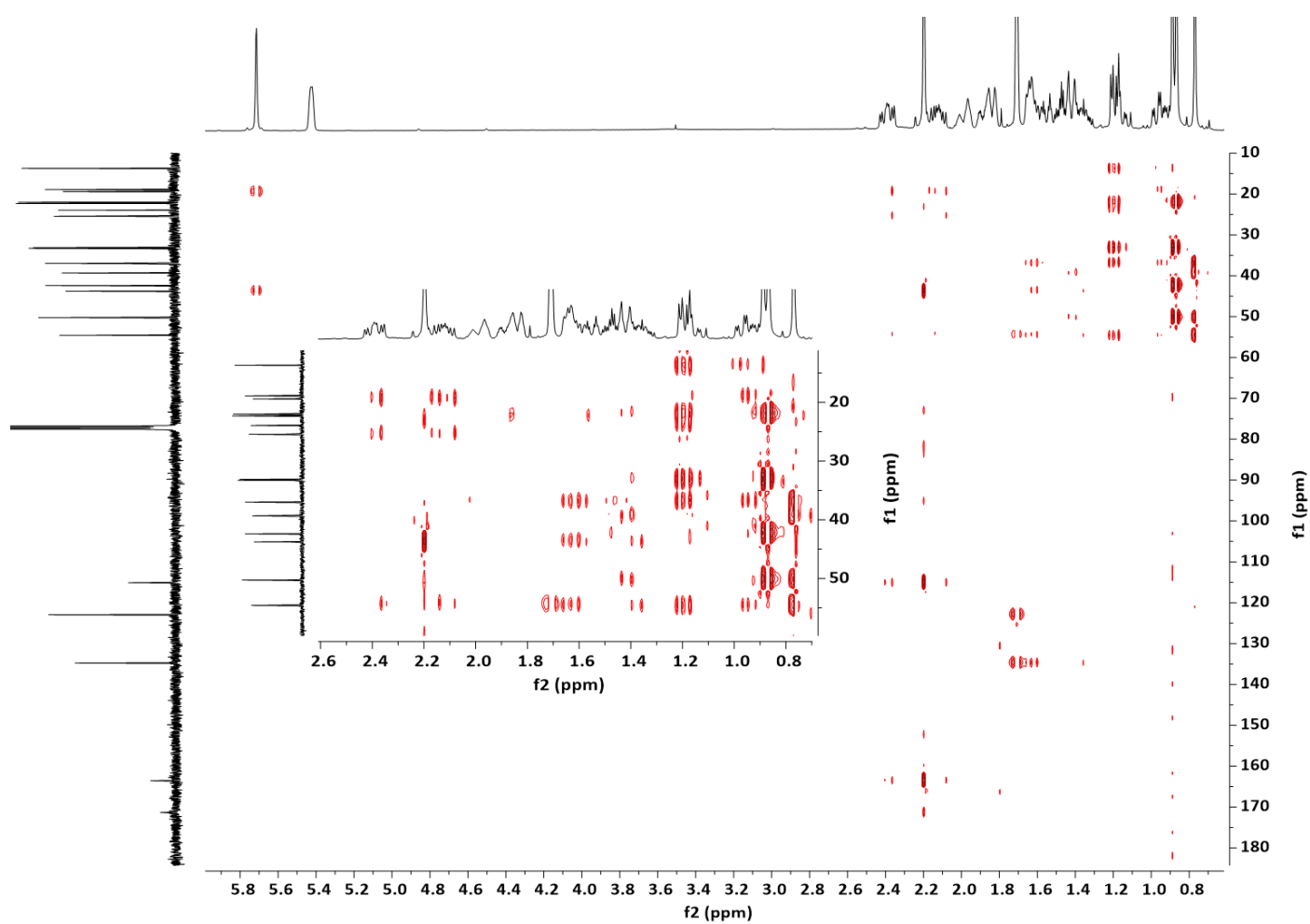
Figure S6. <sup>1</sup>H-NMR spectrum of 2 (400 MHz, CDCl<sub>3</sub>)

<sup>13</sup>C Compound 2 in CDCl<sub>3</sub> 07/10/2020 (exp 8) ns-2k



**Figure S7.** <sup>13</sup>C-NMR spectrum of 2 (100 MHz, CDCl<sub>3</sub>)





**Figure S8:** HMBC spectrum of **2** (400 MHz for  $^1\text{H}$  and 100 MHz, for  $^{13}\text{C}$ ,  $\text{CDCl}_3$ )

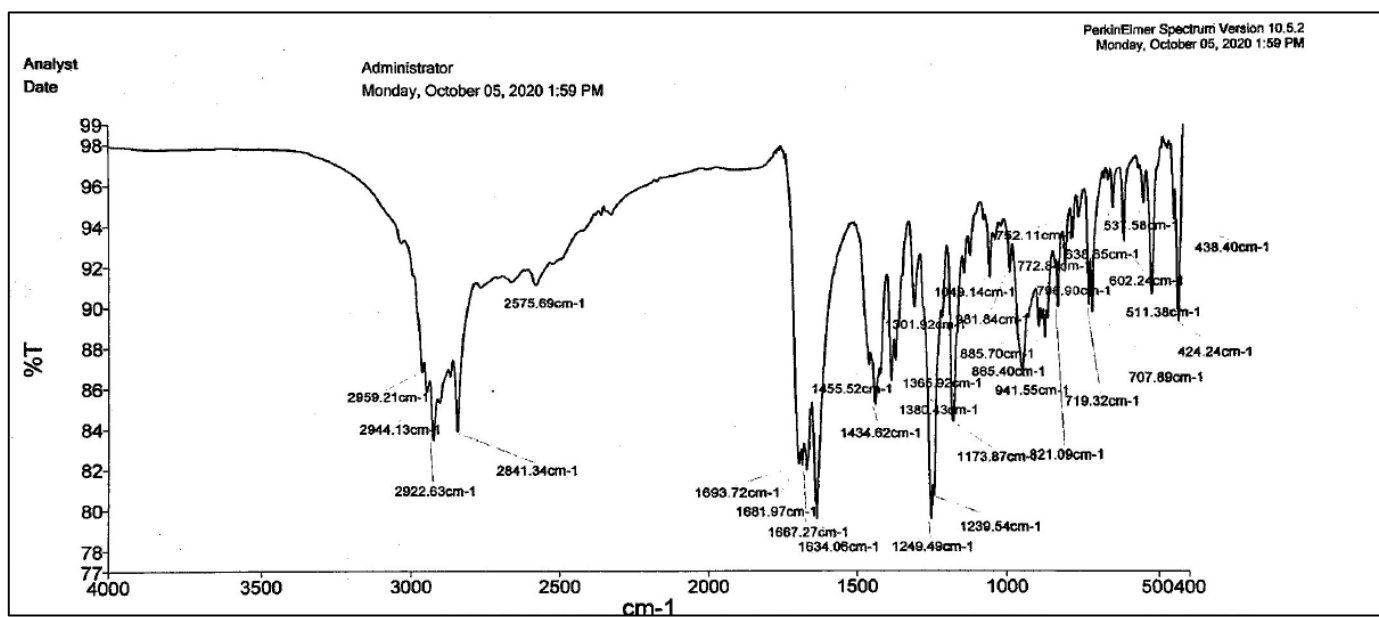


Figure S9: FT-IR (ATR mode) spectrum of 2

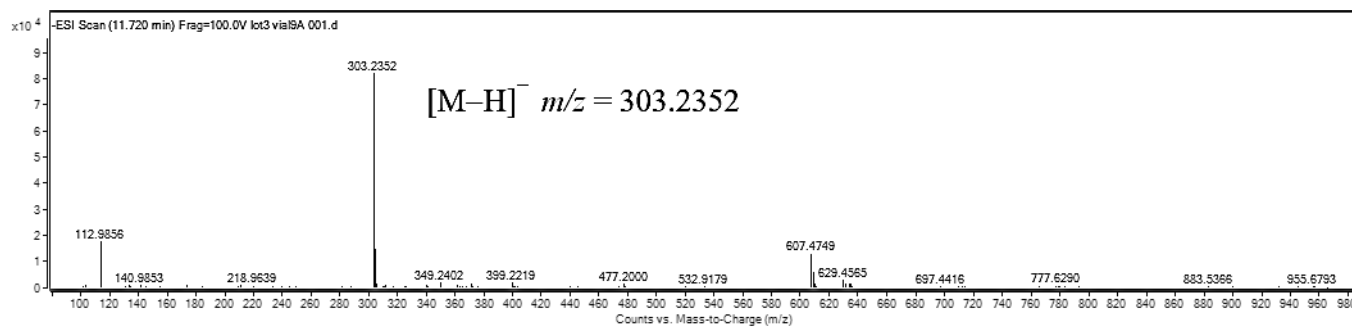


Figure S10: HRESI-MS (negative ion mode) spectrum of 2

<sup>1</sup>H Compound3 in CDCl<sub>3</sub> 21/05/2020 (exp 4)

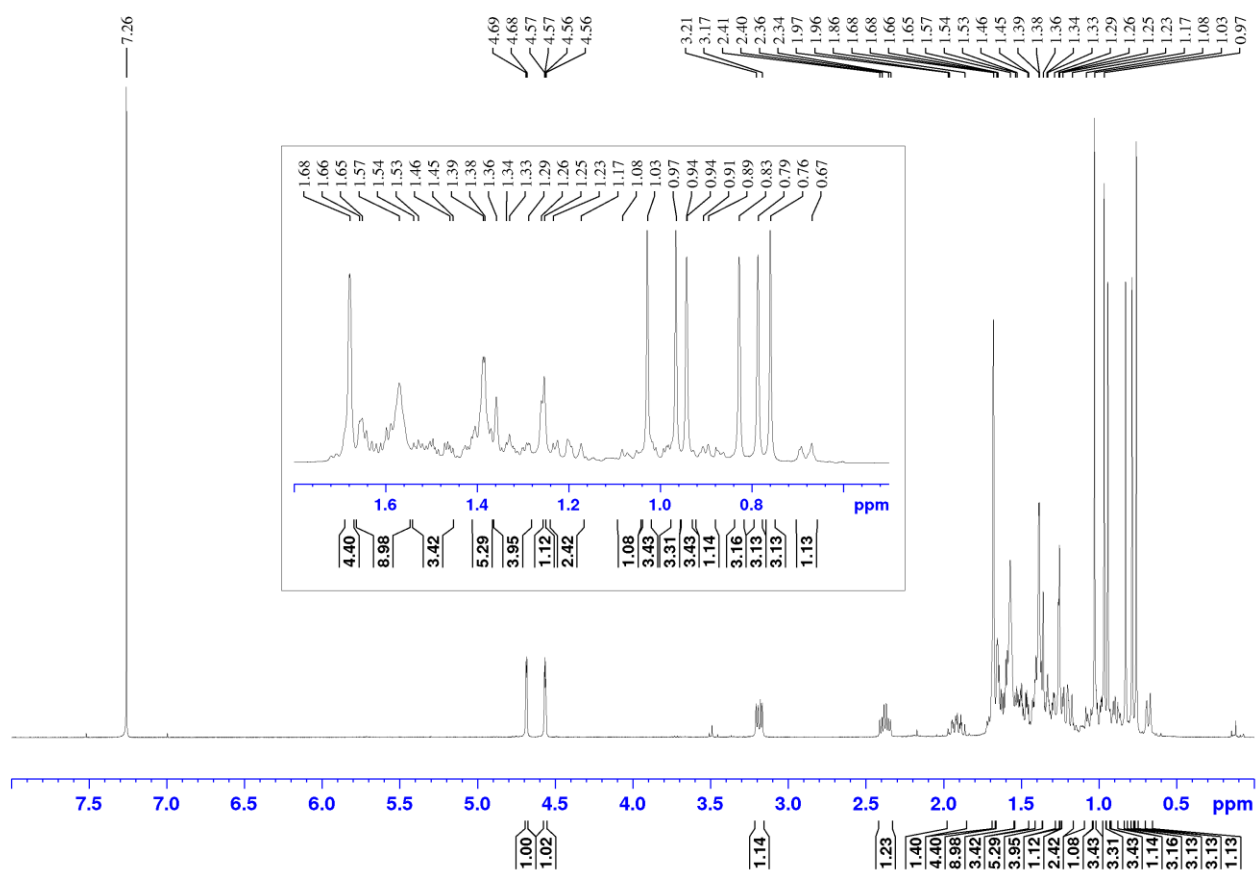
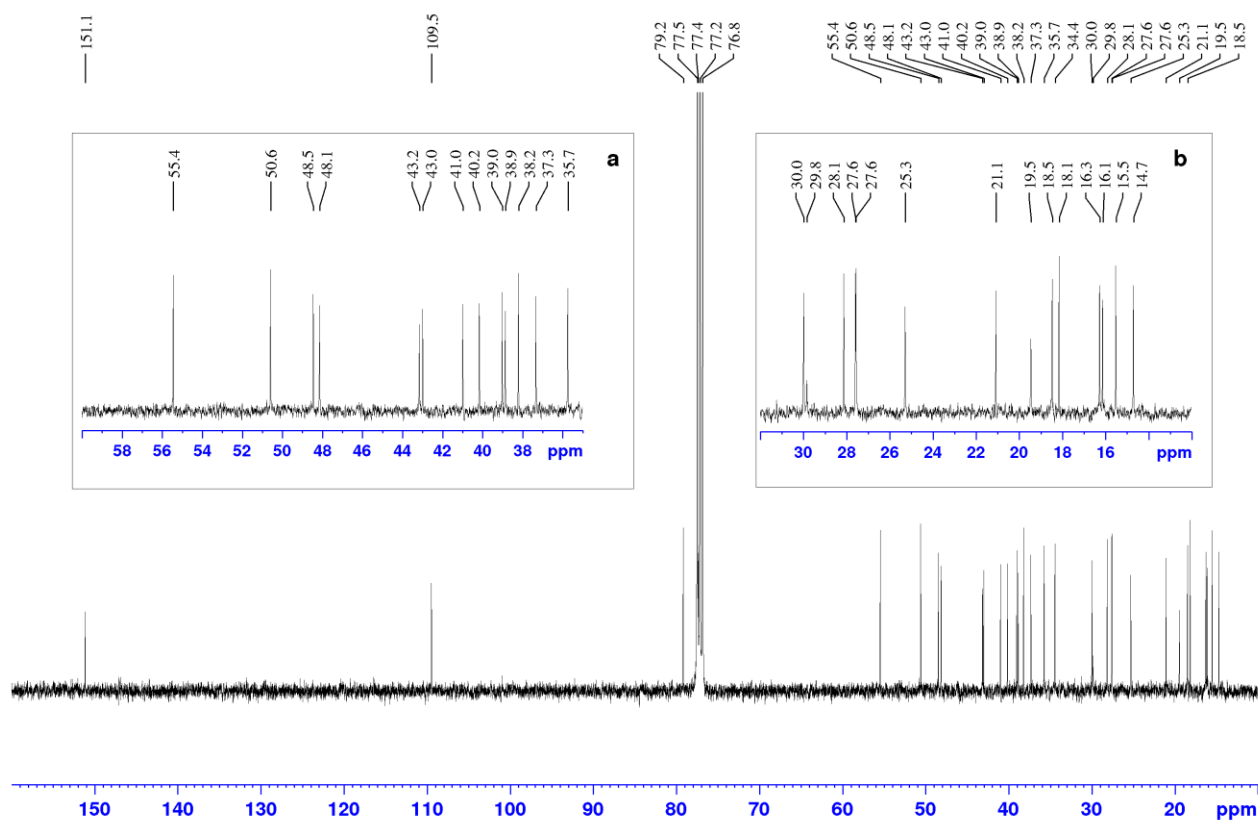


Figure S11. <sup>1</sup>H-NMR spectrum of 3 (400 MHz, CDCl<sub>3</sub>)

<sup>13</sup>C Compound3 in CDCl<sub>3</sub> 21/05/2020 (exp 5) ns-2k



**Figure S12.** <sup>13</sup>C-NMR spectrum of **3** (100 MHz, CDCl<sub>3</sub>) and an expanded region of the spectrum (a) chemical shift at 35.0-60.0 ppm and (b) chemical shift at 12.0-32.0 ppm

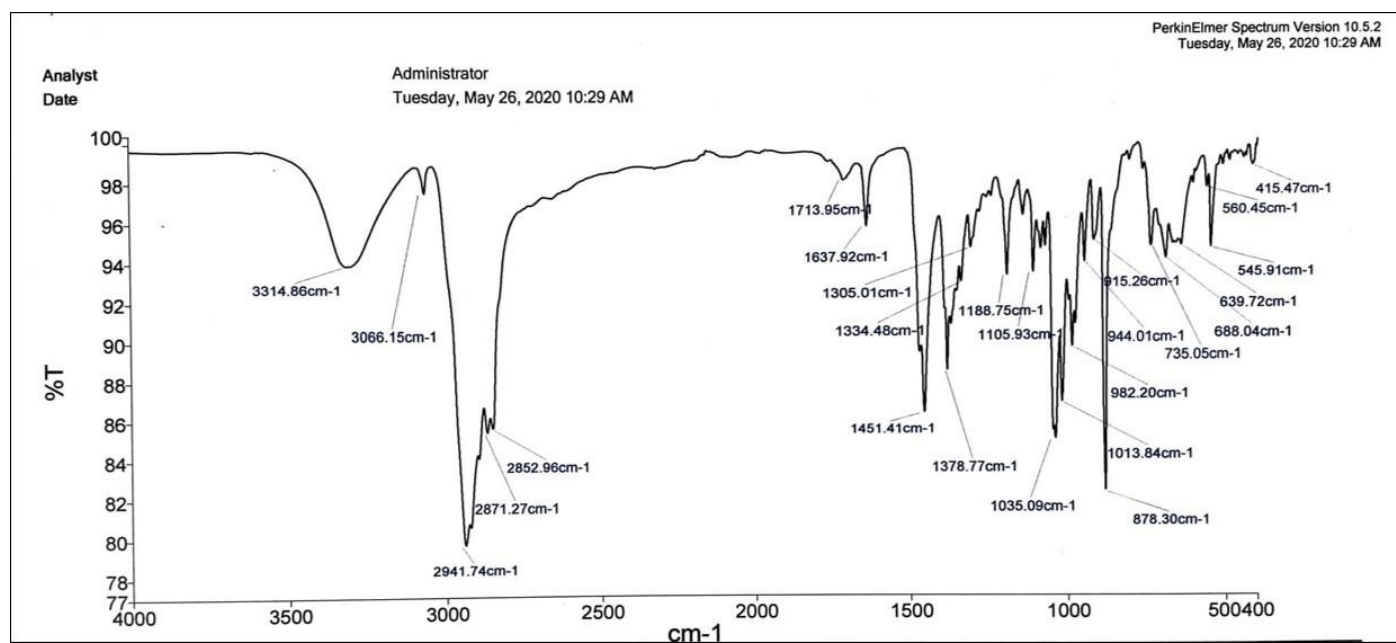


Figure S13: FT-IR (ATR mode) spectrum of 3

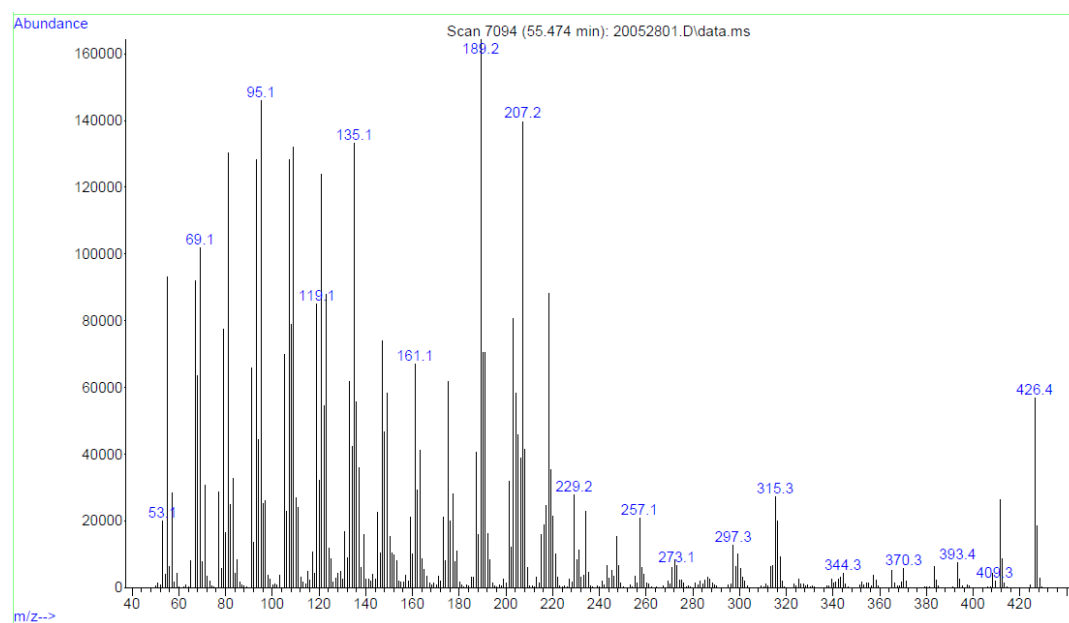


Figure S14: EI-MS spectrum of 3

**Table S1** NMR data of **1** and **2** (in CDCl<sub>3</sub>) measured at 100 (<sup>13</sup>C) and 400 (<sup>1</sup>H) MHz

Position	<b>1</b>		<b>2</b>	
	<sup>13</sup> C (ppm) <sup>a</sup> , mult. <sup>b</sup>	<sup>1</sup> H (ppm) <sup>a</sup> , mult. (J in Hz)	<sup>13</sup> C (ppm) <sup>a</sup> , mult. <sup>b</sup>	<sup>1</sup> H (ppm) <sup>a</sup> , mult. (J in Hz)
1	37.2, CH <sub>2</sub>	1.14, <i>m</i> 1.81, <i>m</i>	39.3, CH <sub>2</sub>	0.94, <i>m</i> 1.83, <i>m</i>
2	19.2, CH <sub>2</sub>	1.48, <i>m</i>	18.9, CH <sub>2</sub>	1.42, <i>m</i>
3	41.9, CH <sub>2</sub>	1.14, <i>m</i> 1.40, <i>m</i>	42.4, CH <sub>2</sub>	1.17, <i>m</i> 1.42, <i>m</i>
4	33.5, C	—	33.1, C	—
5	52.1, CH	1.14, <i>m</i>	50.2, CH	1.17, <i>m</i>
6	19.2, CH <sub>2</sub>	1.64, <i>m</i>	23.9, CH <sub>2</sub>	1.95, <i>m</i>
7	33.8, CH <sub>2</sub>	1.97, <i>m</i> 2.03, <i>m</i>	123.0, CH	5.41, <i>br s</i>
8	126.9, C	—	134.8, C	—
9	139.5, C	—	54.6, CH	1.62, <i>m</i>
10	39.3, C	—	37.0, C	—
11	26.5 CH <sub>2</sub>	2.04, <i>m</i> 2.20, <i>m</i>	25.4, CH <sub>2</sub>	1.42, <i>m</i>
12	42.0, CH <sub>2</sub>	2.20, <i>m</i>	43.8, CH <sub>2</sub>	2.12, <i>m</i> 2.37, <i>m</i>
13	163.8, C	—	163.6, C	—
14	114.5, CH	5.71, <i>br s</i>	115.1, CH	5.70, <i>br s</i>
15	171.5, C	—	171.4, C	—
16	19.4, CH <sub>3</sub>	2.20, <i>d</i> (1.1)	19.4, CH <sub>3</sub>	2.18, <i>d</i> (1.1)
17	19.7, CH <sub>3</sub>	1.57, <i>s</i>	22.3, CH <sub>3</sub>	1.69, <i>br s</i>
18	33.5, CH <sub>3</sub>	0.88, <i>s</i>	33.3, CH <sub>3</sub>	0.87, <i>s</i>
19	21.8, CH <sub>3</sub>	0.83, <i>s</i>	22.0, CH <sub>3</sub>	0.85, <i>s</i>
20	20.3, CH <sub>3</sub>	0.94, <i>s</i>	13.7, CH <sub>3</sub>	0.75, <i>s</i>

<sup>a</sup> Assignments were based on <sup>1</sup>H, <sup>13</sup>C, and HMQC experiments; <sup>b</sup> Multiplicities were established by DEPT experiment.

**Table S2** NMR data of **3** (in CDCl<sub>3</sub>) measured at 100 (<sup>13</sup>C) and 400 (<sup>1</sup>H) MHz

Position	<sup>13</sup> C (ppm)	<sup>1</sup> H (ppm), mult. (J in Hz)
1	38.9	0.89, 1.65, <i>m</i>
2	27.6	1.62, <i>m</i>
3	79.2	3.18, <i>dd</i> (11.2, 5.0)
4	39.0	-
5	55.4	0.67, <i>m</i>
6	18.5	1.36, 1.53, <i>m</i>
7	34.4	1.39, <i>m</i>
8	41.0	-
9	50.6	1.33, <i>m</i>
10	37.3	-
11	21.1	1.31, 1.40, <i>m</i>
12	25.3	1.08, 1.62, <i>m</i>
13	38.2	1.65, <i>m</i>
14	43.0	-
15	27.6	1.05, 1.60, <i>m</i>
16	35.7	1.50, <i>m</i>
17	43.2	-
18	48.5	1.38, <i>m</i>
19	48.1	2.38, <i>dt</i> (11.0, 5.8)
20	151.1	-
21	30.0	1.29, <i>m</i>
22	40.2	1.17, <i>m</i>
23	28.1	0.96, <i>s</i>
24	15.5	0.76, <i>s</i>
25	16.1	0.83, <i>s</i>
26	16.3	1.03, <i>s</i>
27	14.7	0.94, <i>s</i>
28	18.1	0.79, <i>s</i>
29	109.5	4.56, <i>br d</i> 4.69, <i>br d</i>
30	19.5	1.68, <i>s</i>