

## Supplementary Material

### 1-(+)-Dehydroabietylimidazolium Salts as Enantiomer Discriminators for NMR Spectroscopy

H. Q. Nimal Gunaratne<sup>a</sup>, Tiina Laaksonen,<sup>b</sup> Kenneth R. Seddon<sup>a</sup> and Kristiina Wähälä<sup>\*b,c</sup>

<sup>a</sup>QUILL Research Centre, School of Chemistry and Chemical Engineering, The Queen's University of Belfast, Belfast BT9 5AG, Northern Ireland, UK

<sup>b</sup>Department of Chemistry, University of Helsinki, A.I. Virtasen aukio 1, P.O. Box 55, FI-00014 University of Helsinki, Finland

<sup>c</sup>Medicum, P.O. Box 21, FI-000140 University of Helsinki, Finland

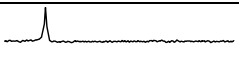
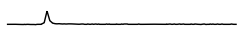
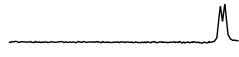







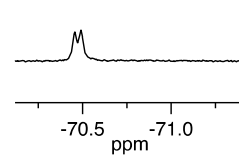
\*Corresponding author  
e-mail: [kristiina.wahala@helsinki.fi](mailto:kristiina.wahala@helsinki.fi)

#### Table of Contents

Testing of the developed cationic CSAs with Mosher's acid .....	2
Resolution of racemic carboxylic acids.....	6
Detection of enantiomeric resolution in <sup>13</sup> C NMR.....	8
NMR spectra of CSAs synthesised.....	9
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 1a.....	9
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 1b.....	11
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 2a.....	13
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 2b.....	15
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 2c.....	17
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 3a.....	19
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 3b.....	21
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 4a.....	23
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 4b.....	25
<sup>1</sup> H, <sup>13</sup> C and HSQC spectra of compound 4c.....	27

## Testing of the developed cationic CSAs with Mosher's acid

**Table 1.** Change of chemical shift and chemical shift difference ( $\Delta\delta$ ) between the enantiomers of **6** in the presence of chiral solvating agents **1a-4c** in  $^{19}\text{F}$  NMR (470 MHz).

Host	$\text{CF}_3$ peak	<b>6</b> $\Delta\delta$ /ppm; (J/Hz)
None		0.000
<b>1a</b>		0.000
<b>1b</b>		0.024 (11.4)
<b>2a</b>		0.074 (35.0)
<b>2b</b>		0.092 (43.5)
<b>2c</b>		0.110 (49.8)
<b>3a</b>		0.060 (28.1)
<b>3b</b>		0.065 (30.6)
<b>4a</b>		0.028 (13.4)
<b>4b</b>		0.034 (15.7)
<b>4c</b>		0.034 (15.8)

**Table 2.** The chemical shift differences between the *R* and *S* enantiomers of racemic Mosher's acid (**5**) and its tetrabutylammonium salt (**6**) and corresponding spectra (both <sup>1</sup>H and <sup>19</sup>F NMR) in the presence of chiral solvating agents **1a-4c** in CDCl<sub>3</sub> at 27 °C.

Host	Host: Guest	<b>5: <math>\Delta\delta</math>/ppm; (J/Hz)</b>		<b>6: <math>\Delta\delta</math>/ppm; (J/Hz)</b>					
		OCH <sub>3</sub> peak	$\Delta\delta$	CF <sub>3</sub> peak	$\Delta\delta$	OCH <sub>3</sub> peak	$\Delta\delta$	CF <sub>3</sub> peak	$\Delta\delta$
None									
<b>1a</b>	1:1		0.0092 (4.6)		0.031 (14.8)		0.000		0.000
	2:1		0.011 (5.7)		0.041 (19.3)		0.000		0.000
<b>1b</b>	1:1		0.002 (0.99)		0.000		0.0044 (2.2)		0.024 (11.4)
	2:1		0.000		0.000		0.0042 (2.1)		0.026 (12.2)
<b>2a</b>	1:1		0.0056 (2.8)		0.000		0.000		0.074 (35.0)
	2:1		0.0091 (4.5)		0.000		0.000		0.080 (37.7)
<b>2b</b>	1:1		0.0071 (3.5)		0.000		0.000		0.092 (43.5)
	2:1		0.0099 (5.0)		0.000		0.000		0.102 (47.9)
<b>2c</b>	1:1		0.002 (1.0)		0.000		0.0029 (1.5)		0.110 (49.8)
	2:1		0.000		0.000		0.0061 (3.0)		0.110 (49.9)
<b>3a</b>	1:1		0.000		0.007 (3.3)		0.000		0.060 (28.1)
	2:1		0.000		0.015 (7.0)		0.000		0.077 (36.4)
<b>3b</b>	1:1		0.0019 (1.0)		0.000		0.000		0.065 (30.6)
	2:1		0.000		0.000		0.000		0.074 (34.7)
<b>4a</b>	1:1		0.000		0.000		0.000		0.028 (13.4)
	2:1		0.000		0.000		0.000		0.033 (15.7)
<b>4b</b>	1:1		0.000		0.000		0.000		0.034 (15.7)
	2:1		0.000		0.000		0.000		0.036 (17.0)
<b>4c</b>	1:1		0.0017 (0.8)		0.000		0.000		0.034 (15.8)
	2:1		0.000		0.000		0.000		0.032 (15.3)

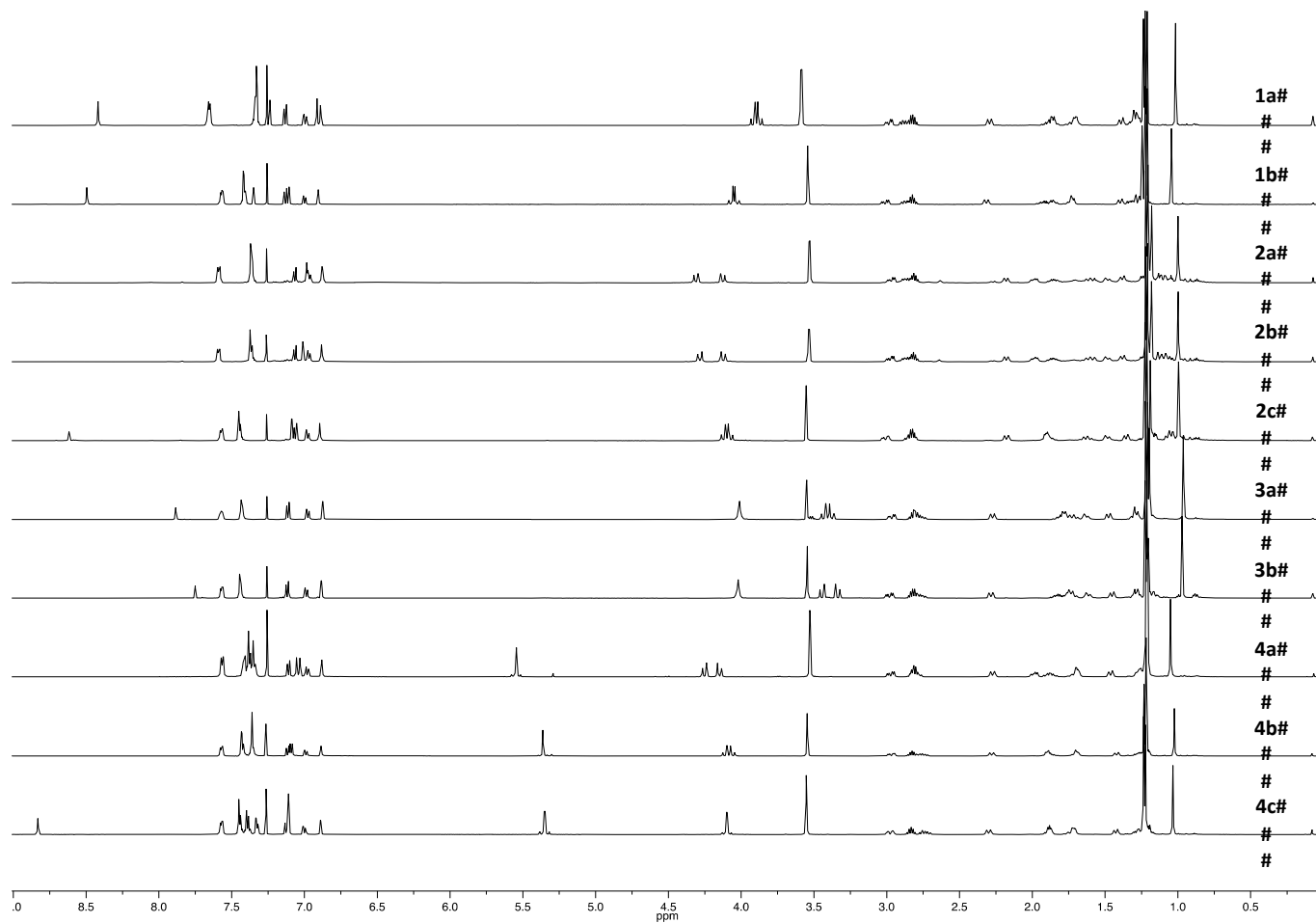


Figure 1. Full <sup>1</sup>H NMR spectra of Mosher's acid and CSAs **1-4abc** in 1:1 ratio.

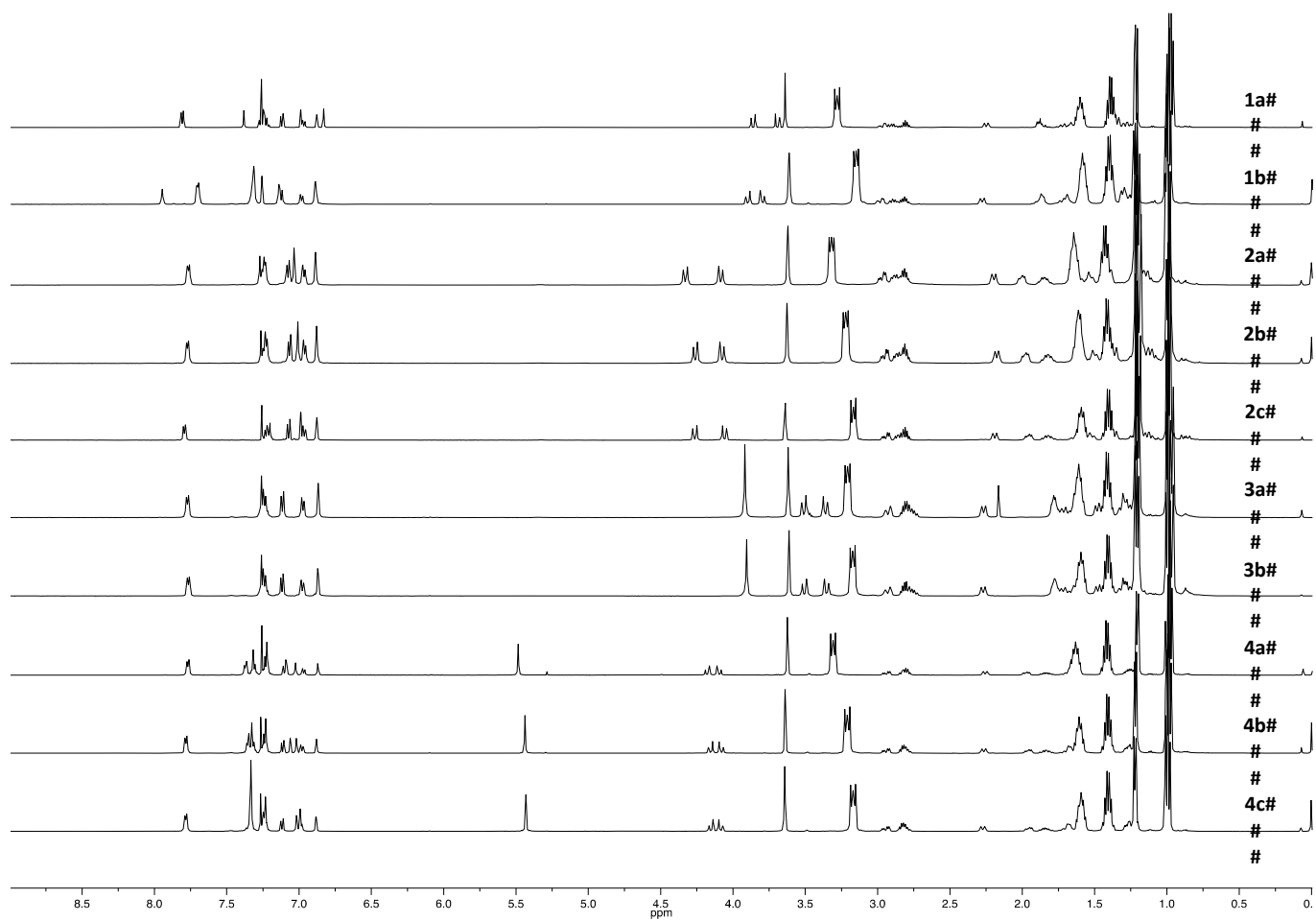

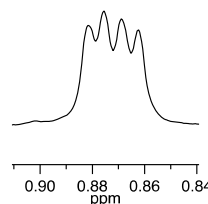
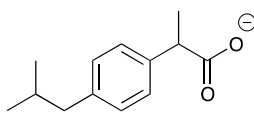
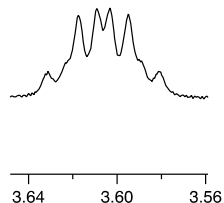
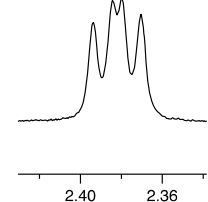
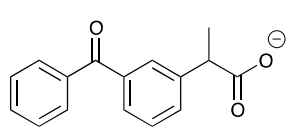
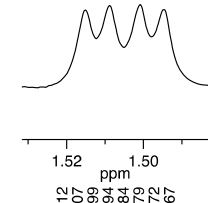
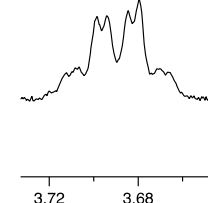


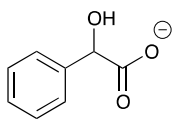
Figure 2. Full <sup>1</sup>H NMR spectra of Mosher's acid tetrabutylammonium salt and CSAs **1-4abc** in 1:1 ratio

## Resolution of racemic carboxylic acids

Table 3. Determination of magnitude of non-equivalence ( $\Delta\delta$ ) of five carboxylic acids  $[\text{NBu}_4]^+$  salts in the presence of **2c**, using  $^1\text{H}$  NMR (500 MHz) in  $\text{CDCl}_3$  at 27 °C.

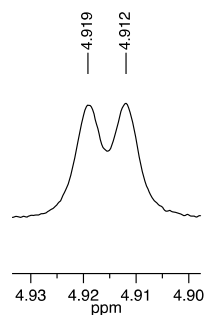
Compound	$[\text{NBu}_4]^+$ salt of racemic carboxylic acid	Multiplet	Spectrum	Split $\Delta\delta$		
				ppm	Hz	
		Me	d	-	-a	
					-a	
		<i>i</i> -Pr	d		0.0096	4.79298
7		H	q		0.0087	4.36566
				0.006	2.99449	
8		Me	d		0.0065	3.22805
		H	q		0.0047	2.33297

9



H

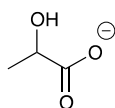
s



0.0077

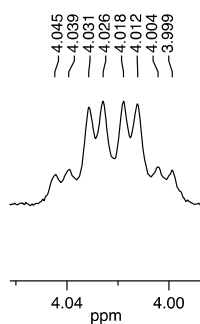
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10



H

q



0.0055

2.77185

Me

d

-

-a

-a

H

q

-

0.0

0.0



NH

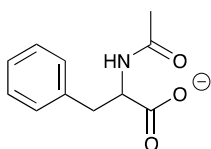
d



0.019

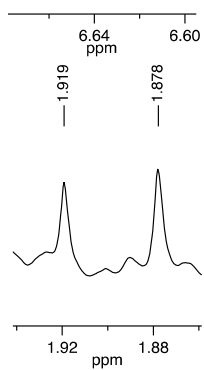
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11



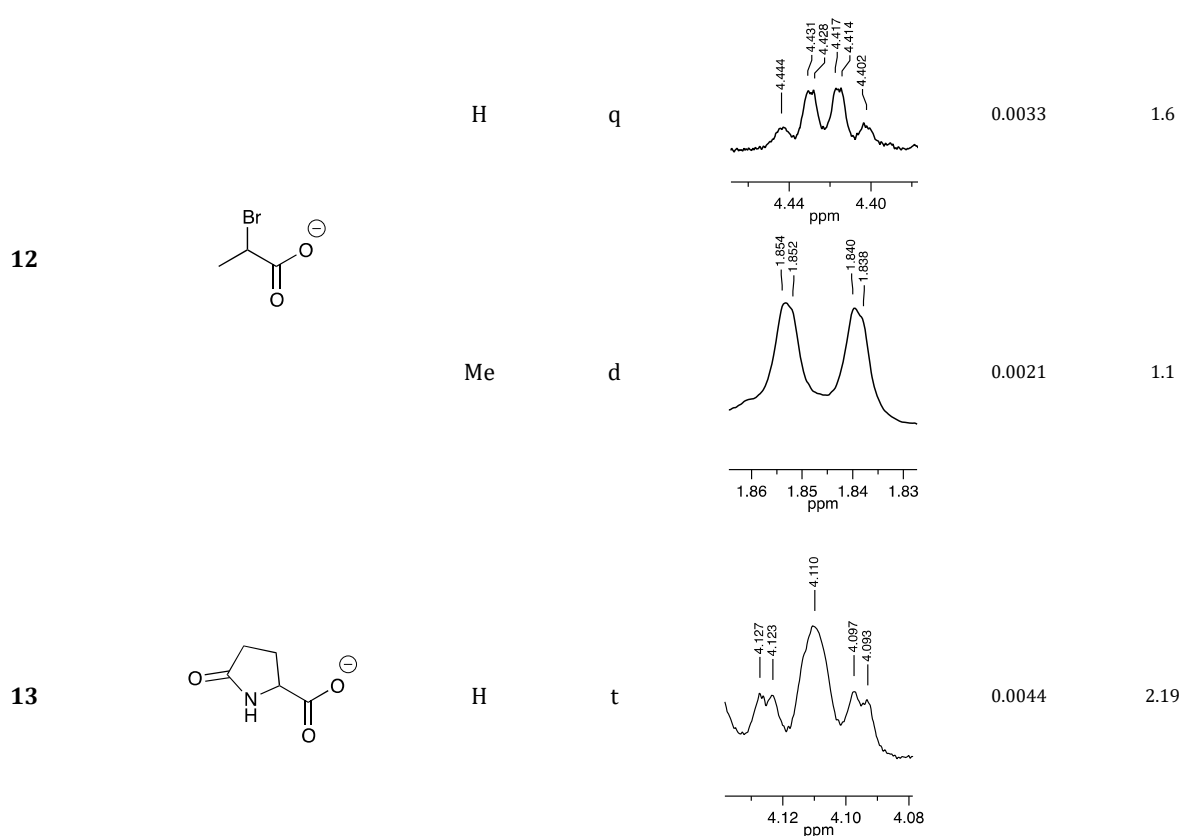
Me

s



0.041

20.5579



## Detection of enantiomeric resolution in $^{13}\text{C}$ NMR

Enantiomeric resolution may also be seen in  $^{13}\text{C}$  NMR. However, the detection of enantiomeric resolution by  $^{13}\text{C}$  NMR compared to  $^1\text{H}$  NMR is more time consuming due to sensitivity issues in the former. As we used 2.0 mmol solutions and therefore ordinary  $^{13}\text{C}$  NMR and DEPT analyses are too time consuming we inspected the possible enantiomeric resolution of three carboxylic acids (**7**, **11**, **12**) by using HSQC. Only in the case of **11** could resolution (6.15 Hz, 0.049 ppm) be detected (Fig. 5).

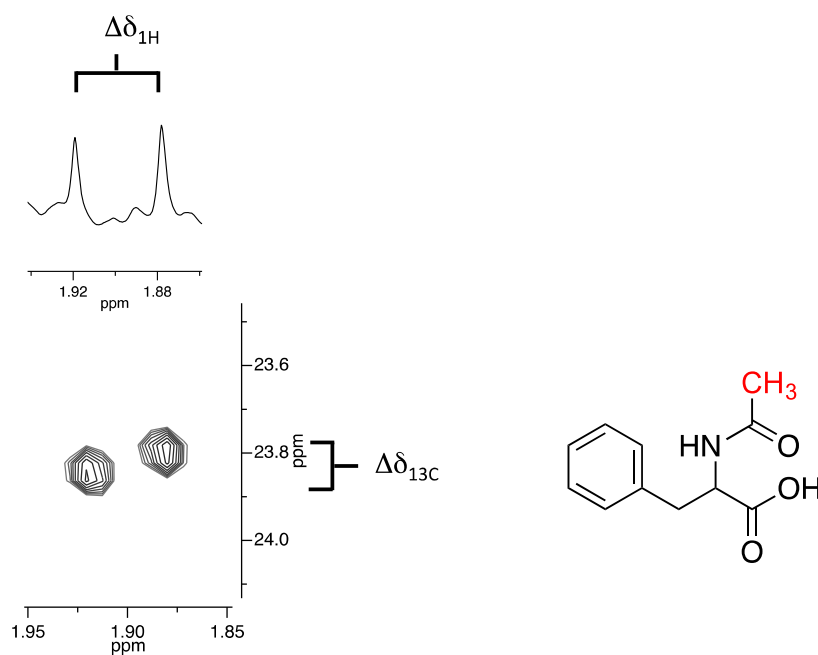
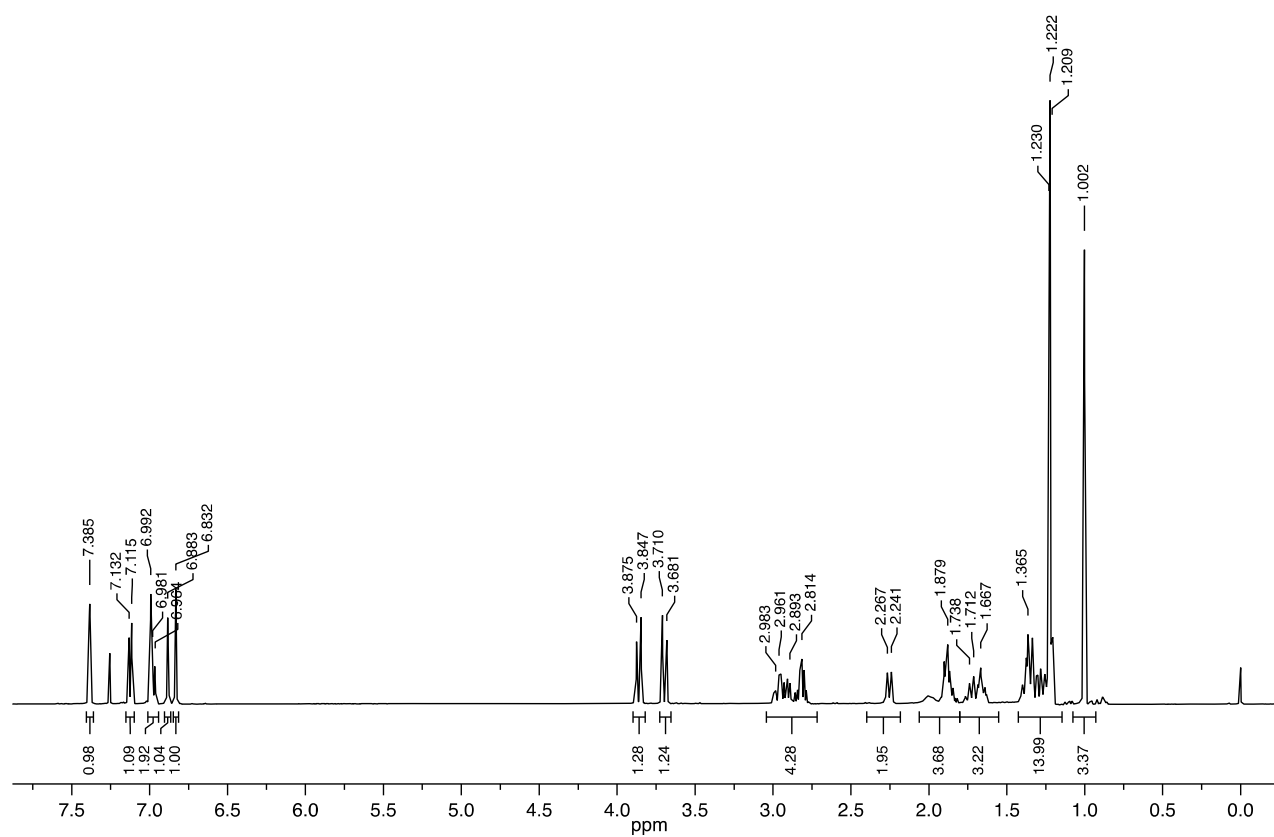
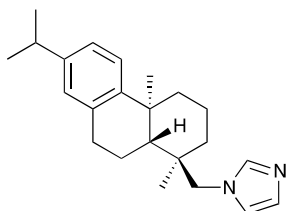


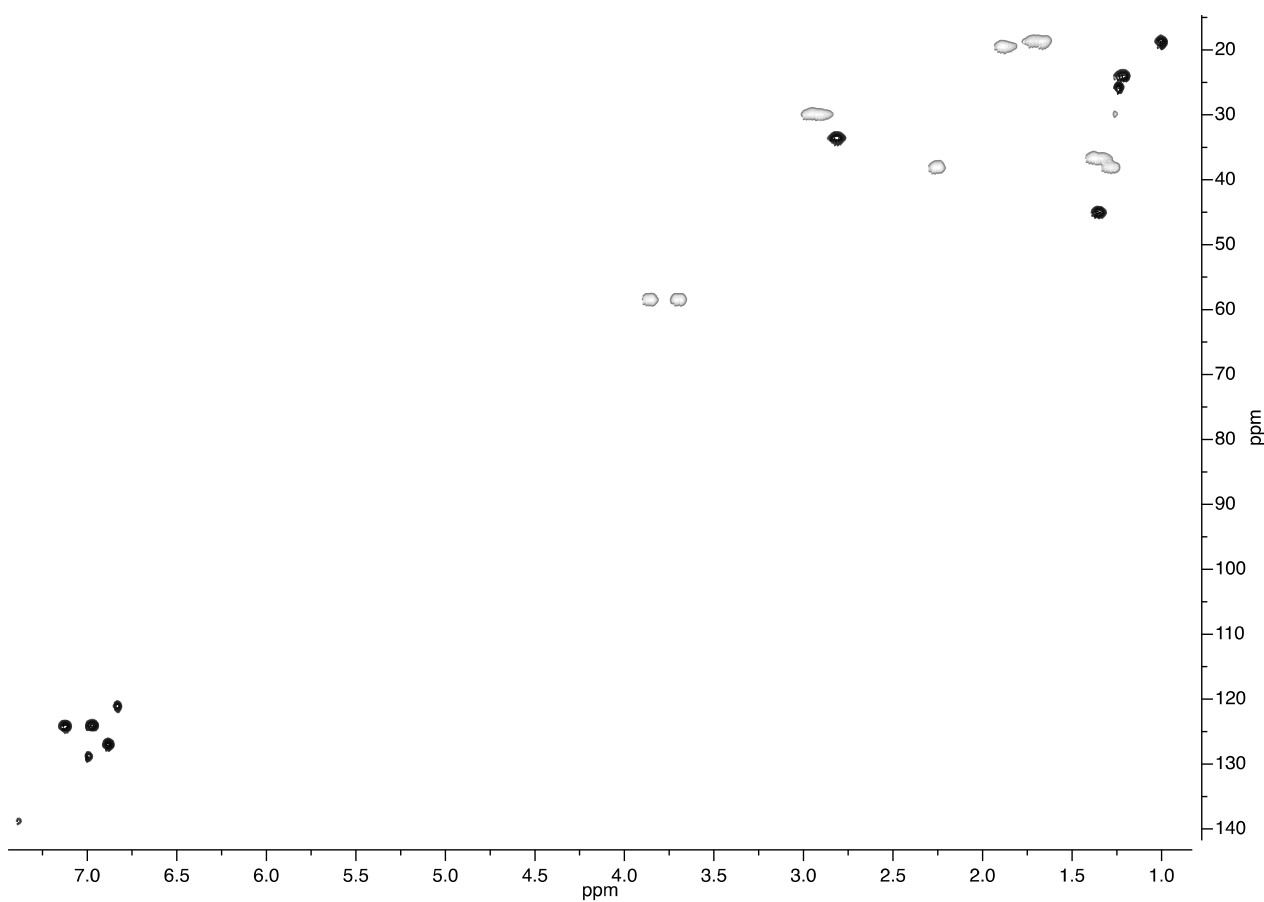
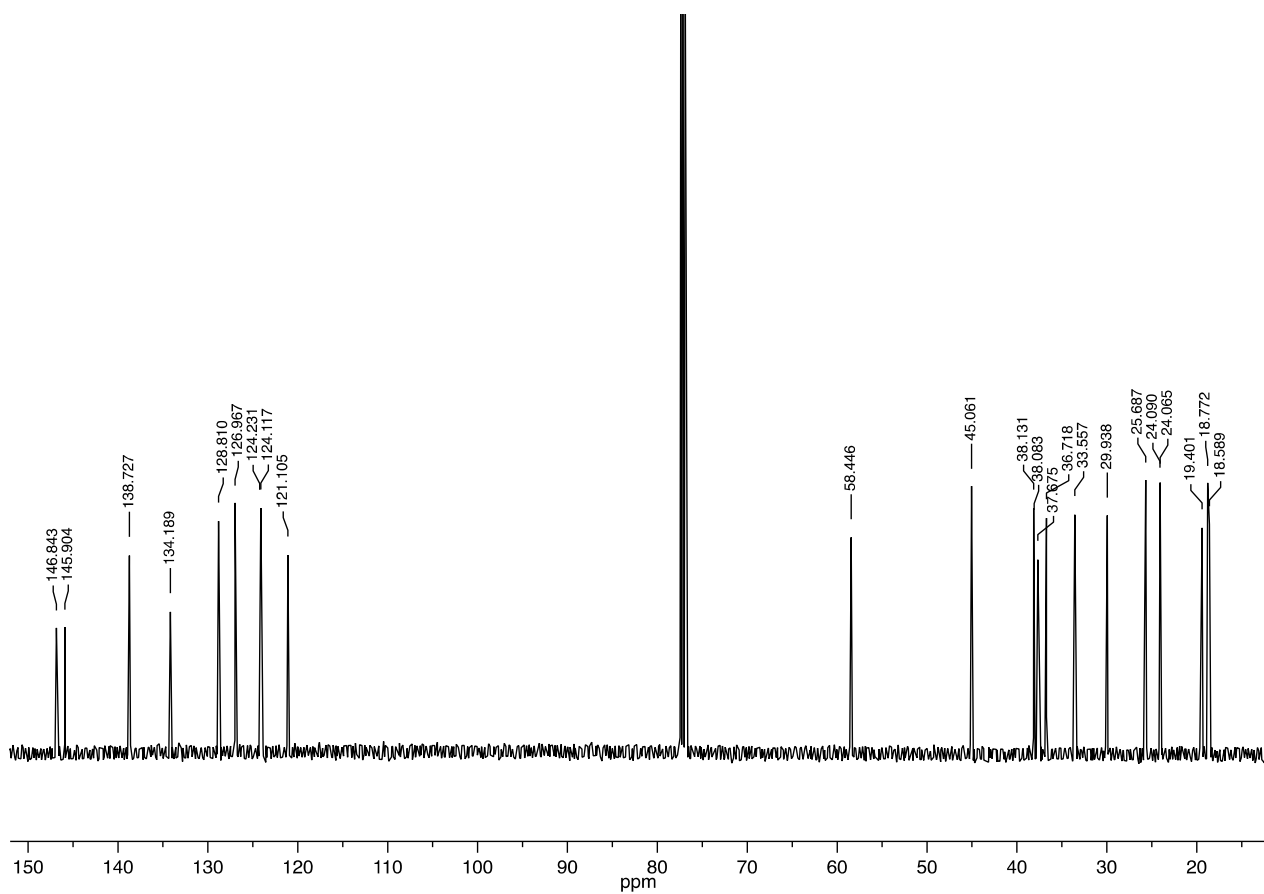
Figure 5. Enantiomeric resolution of methyl group ( $\text{CH}_3$ ) in **11** with CSA **2c** in proton ( $^1\text{H}$  NMR) and carbon (HSQC).



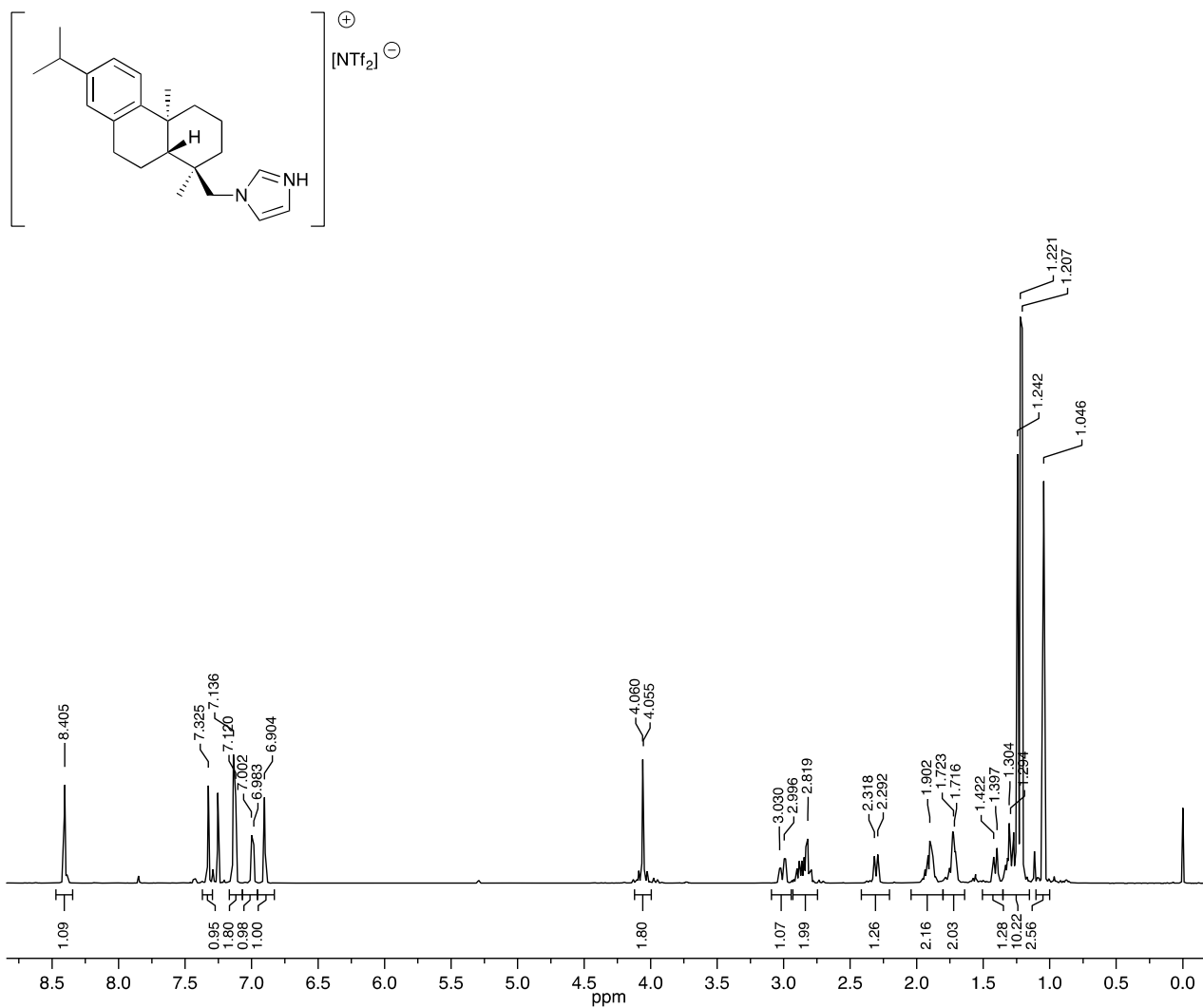
## NMR spectra of CSAs synthesised

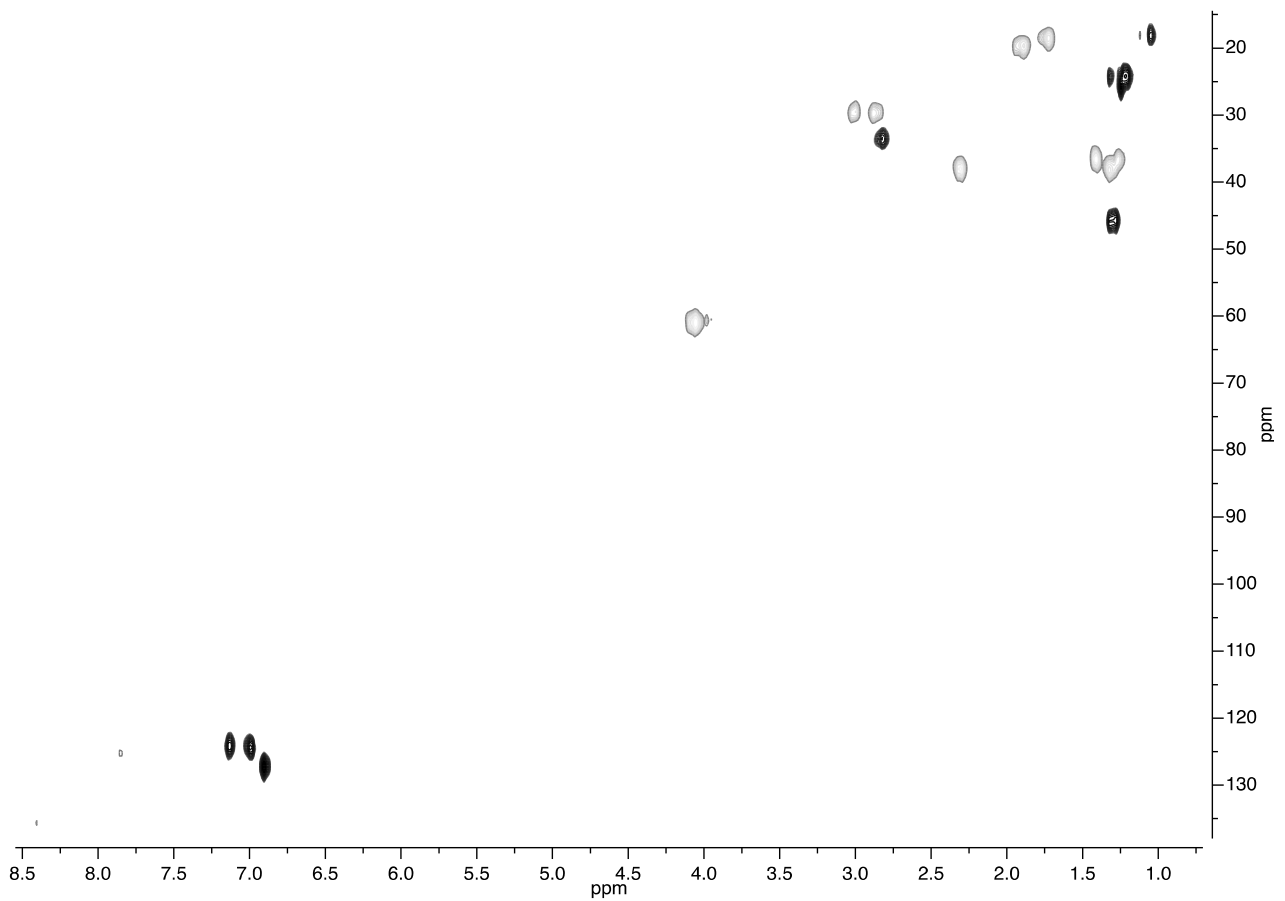
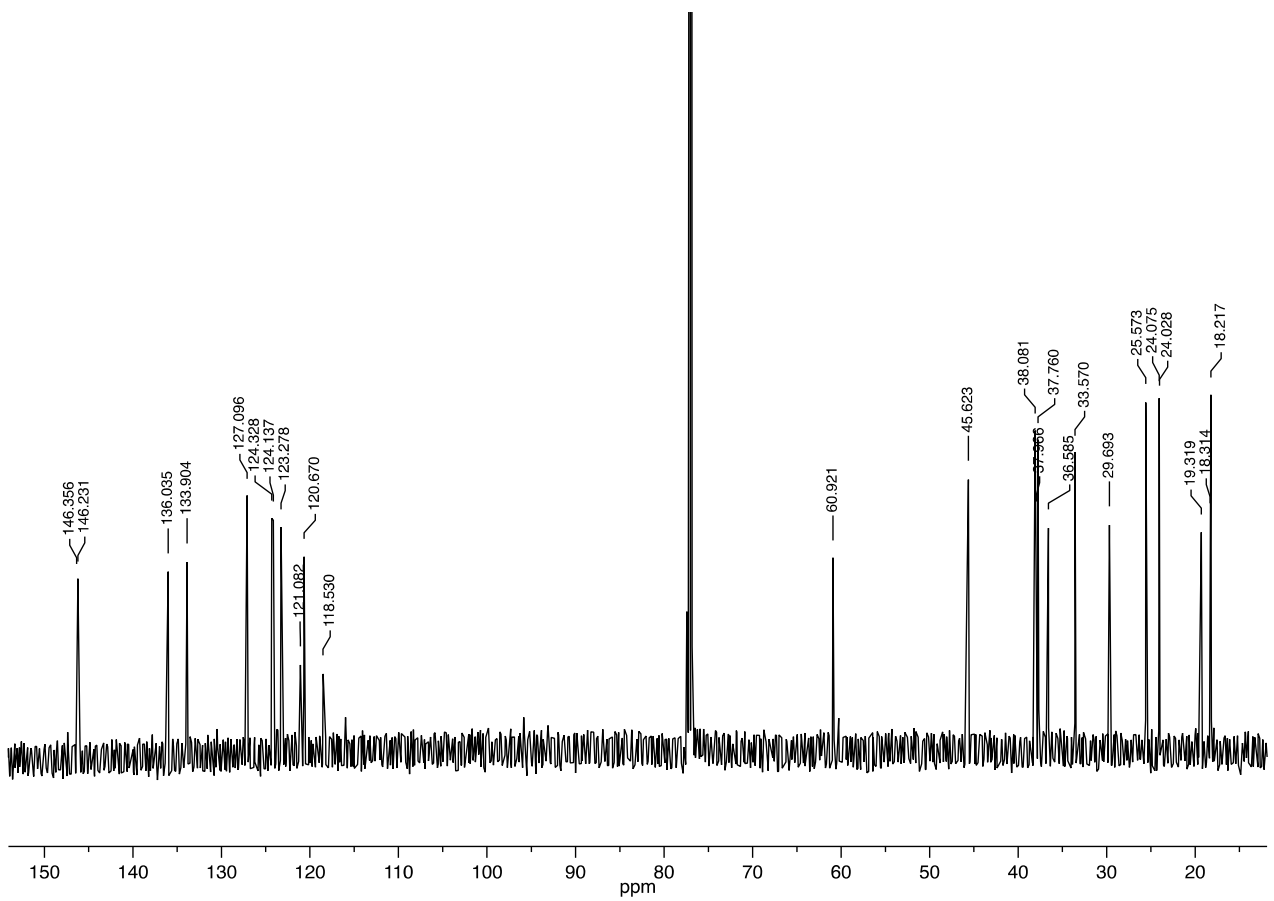
### $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 1a



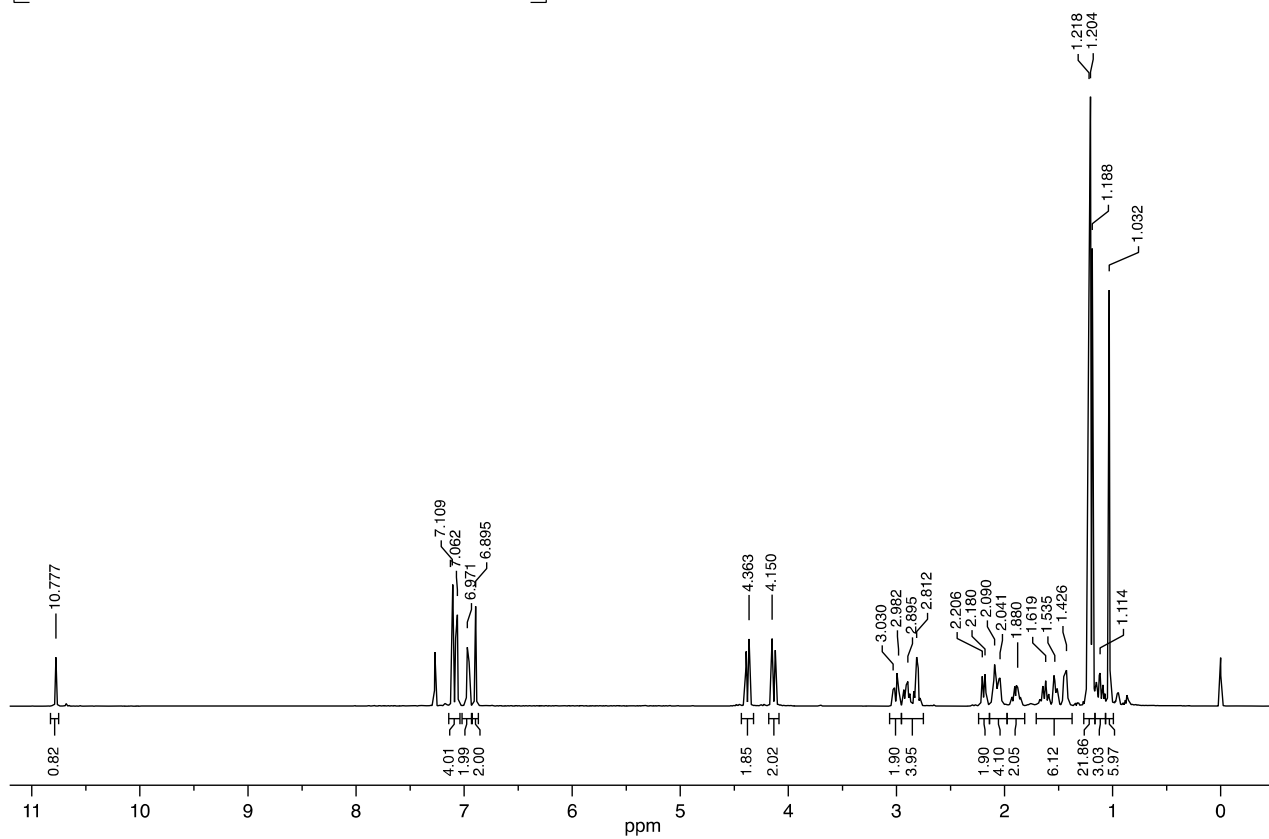
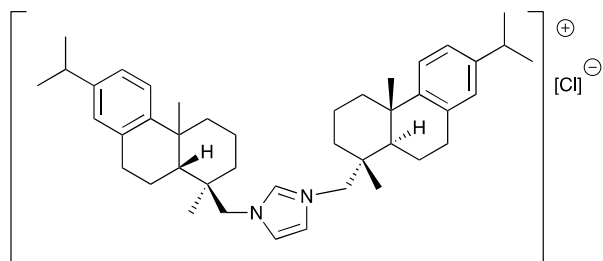


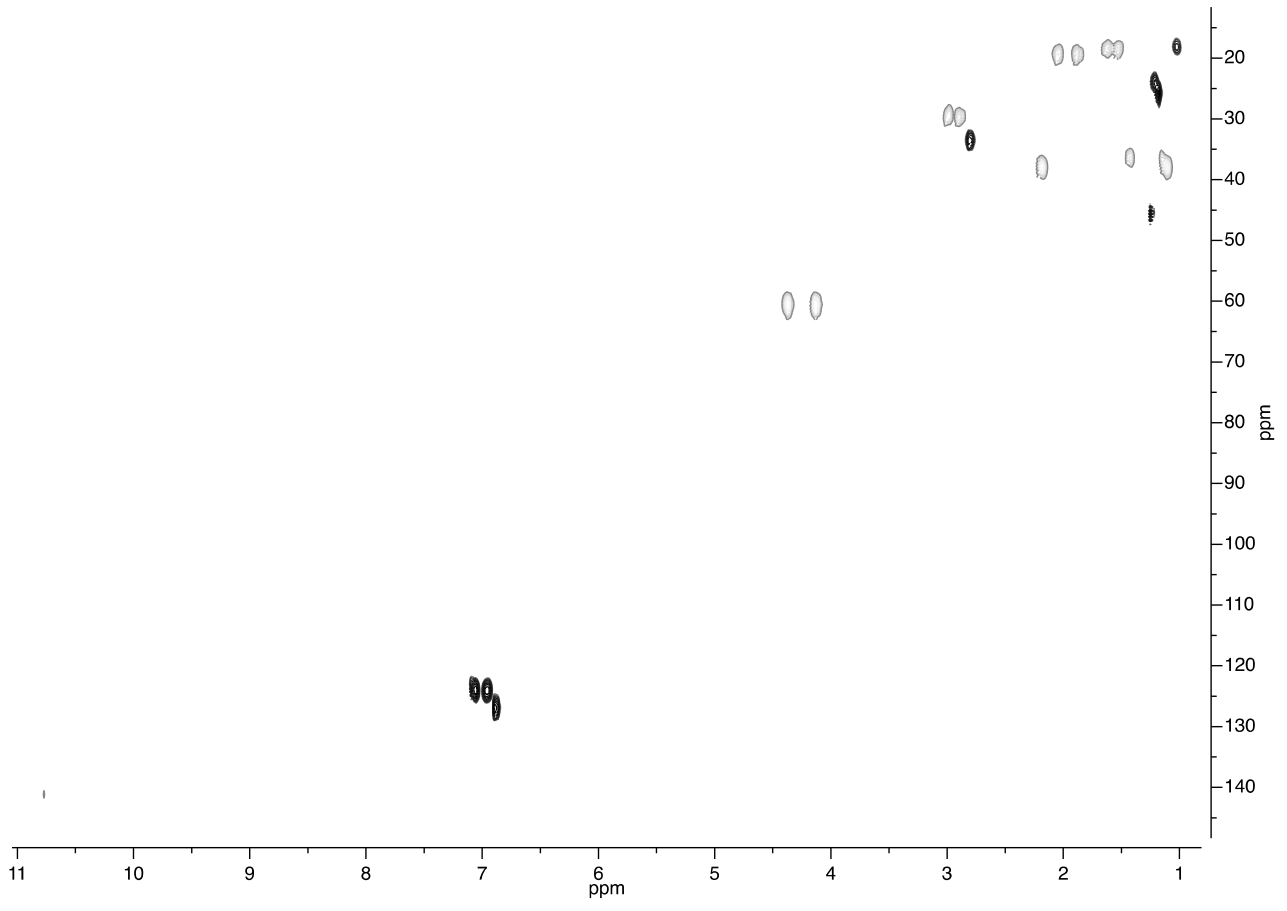
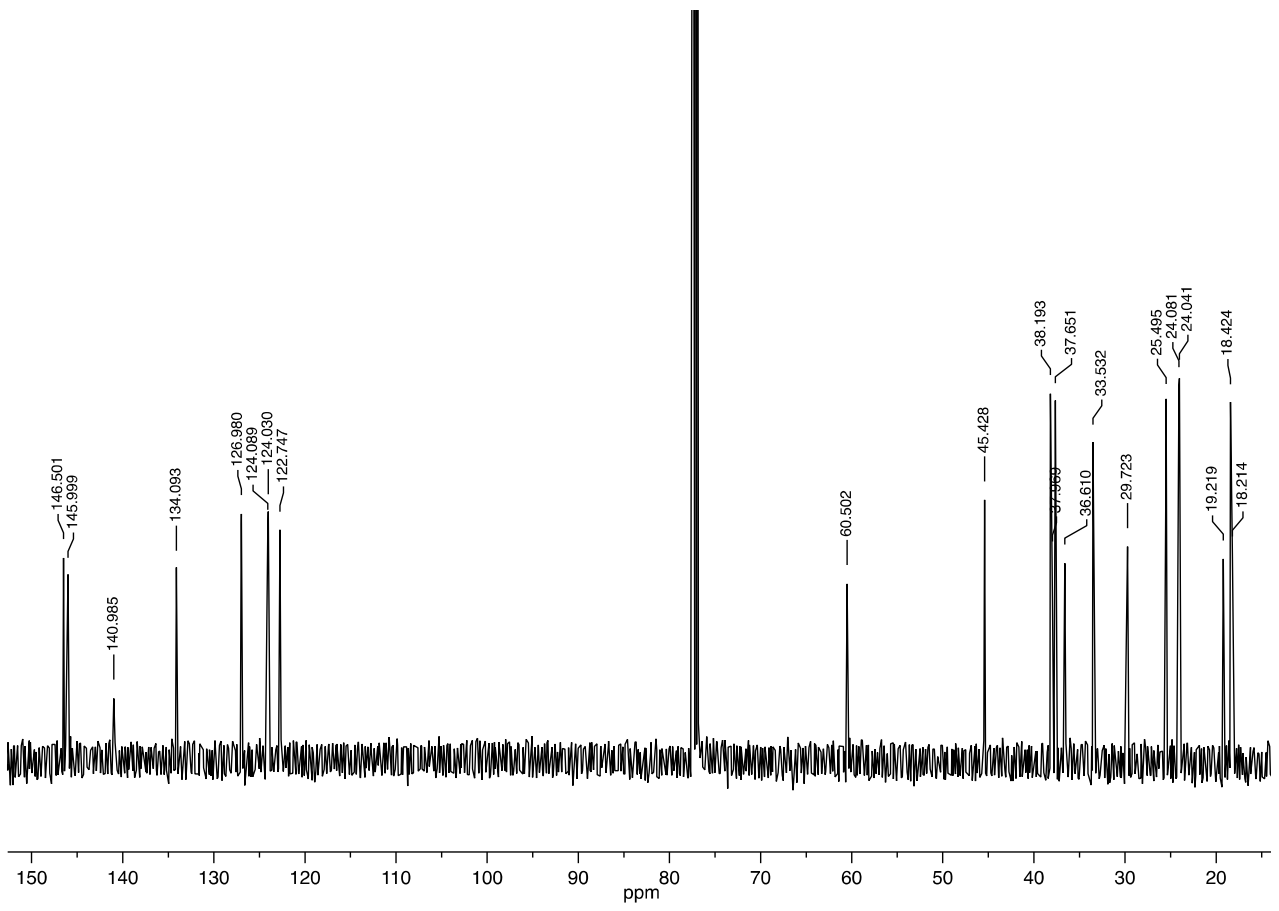
# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 1b



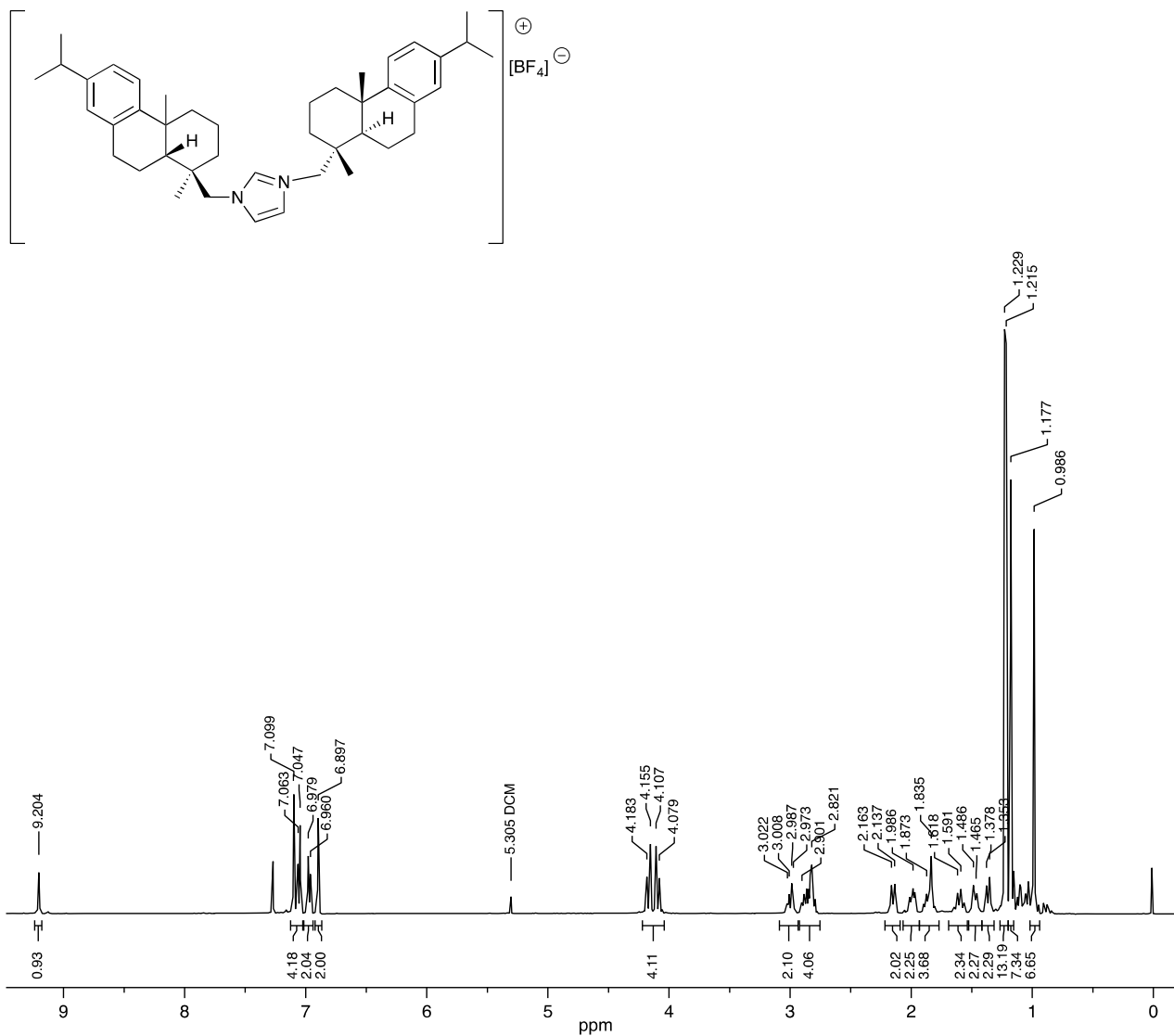


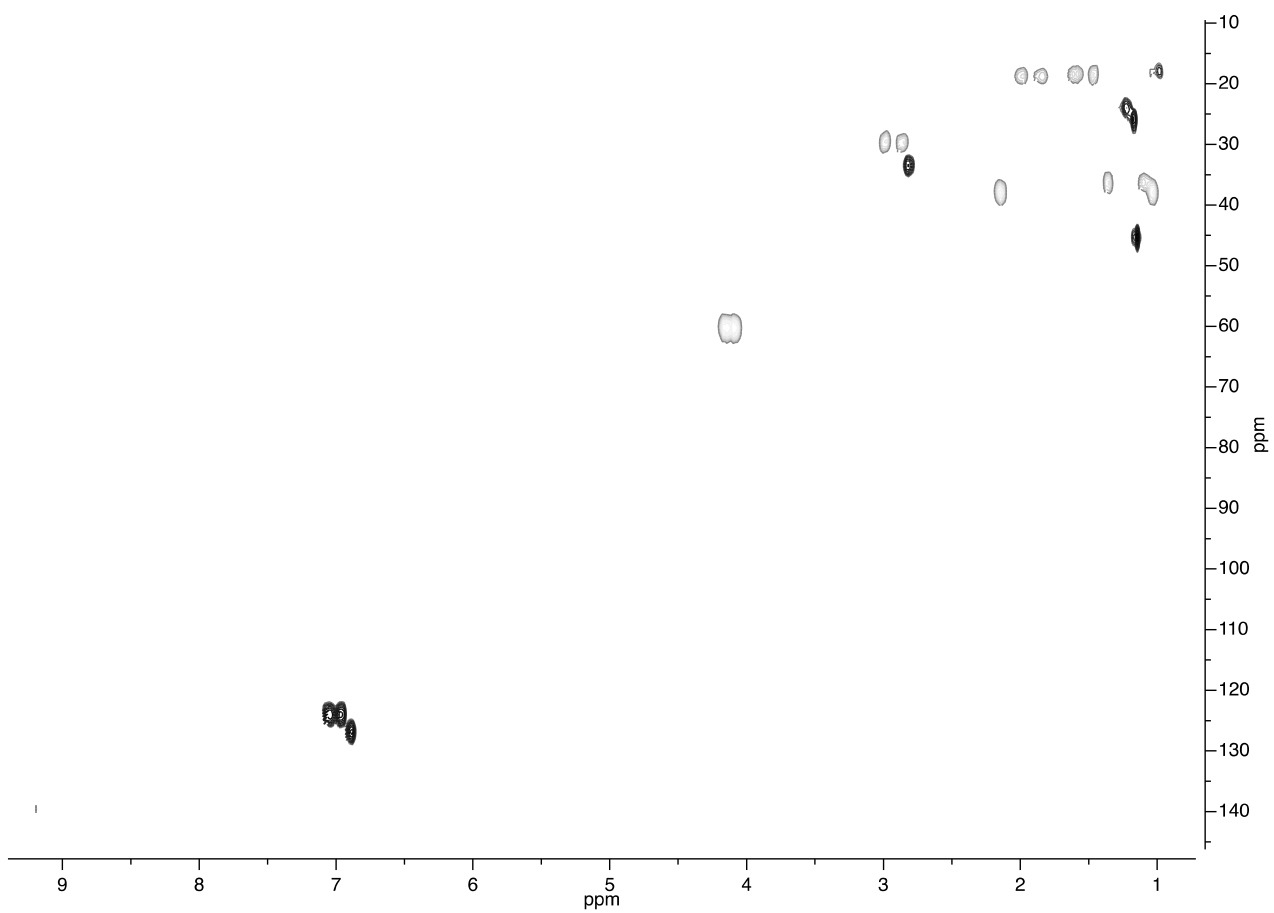
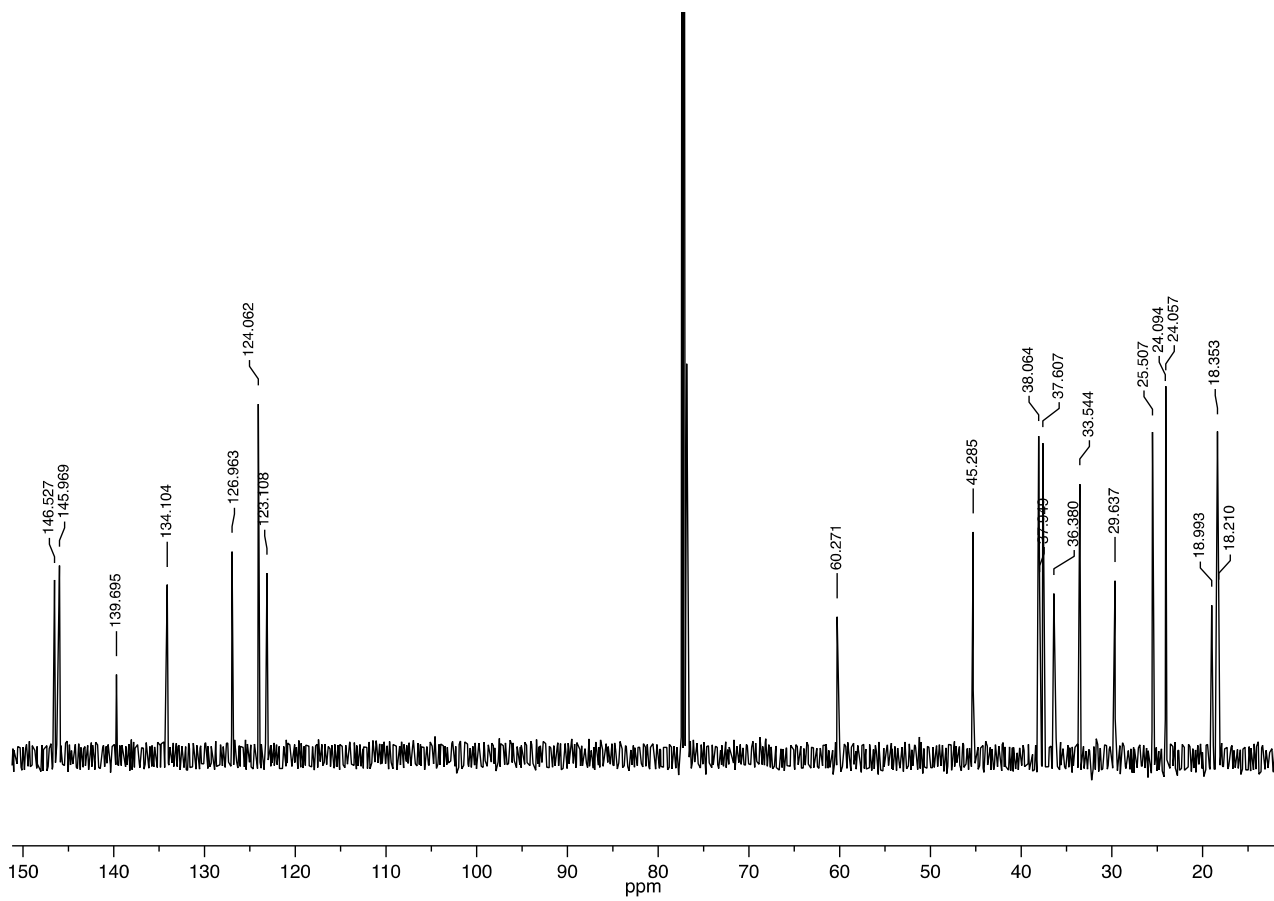
# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 2a





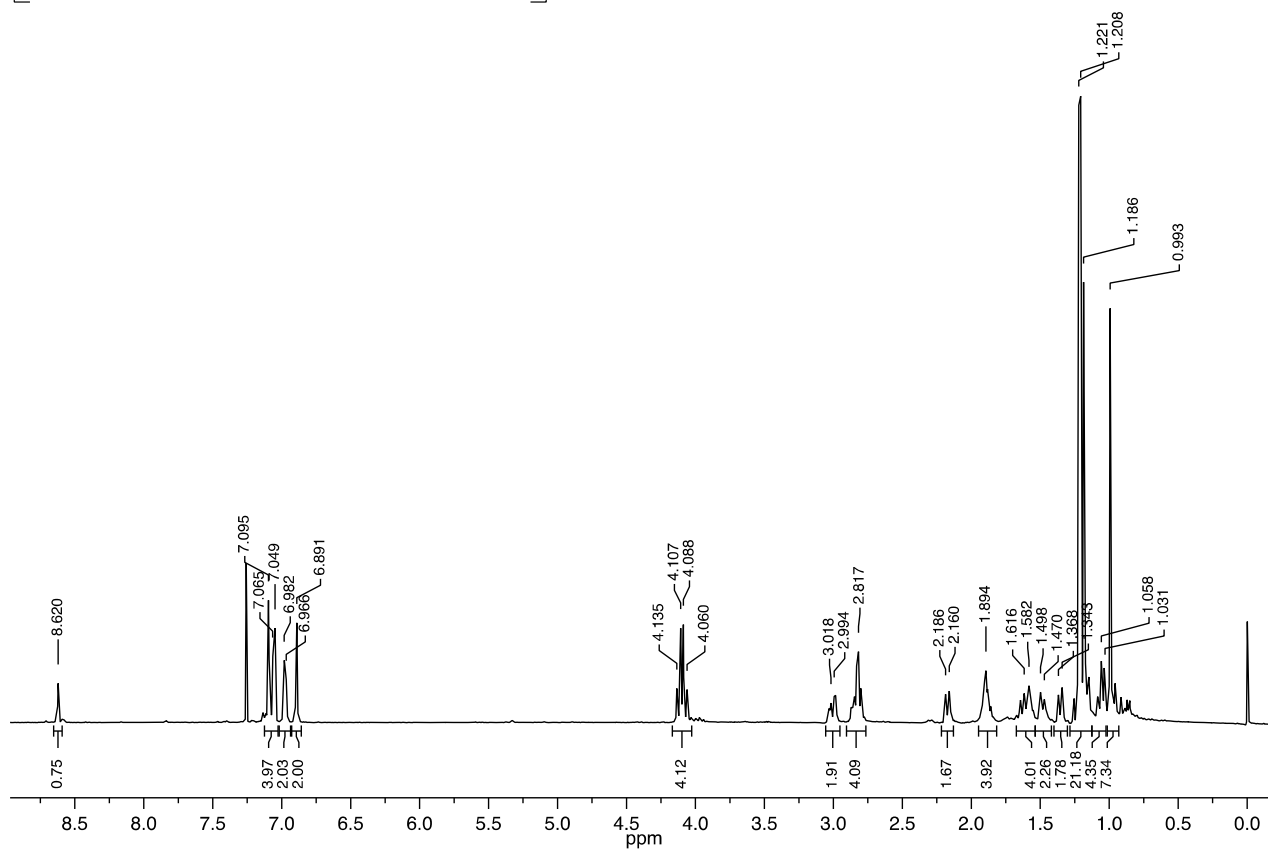
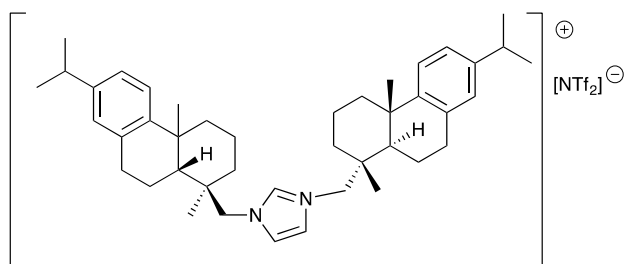
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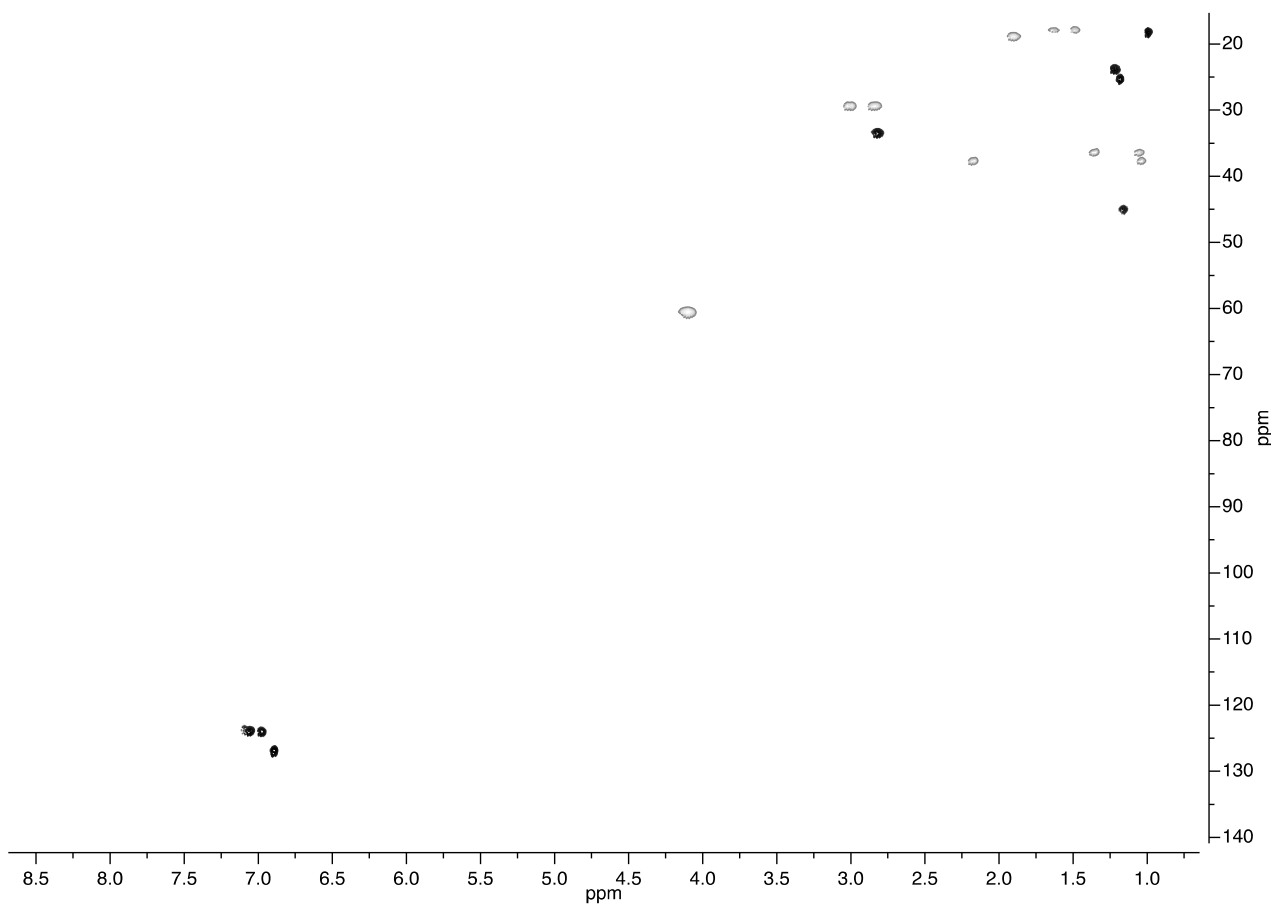
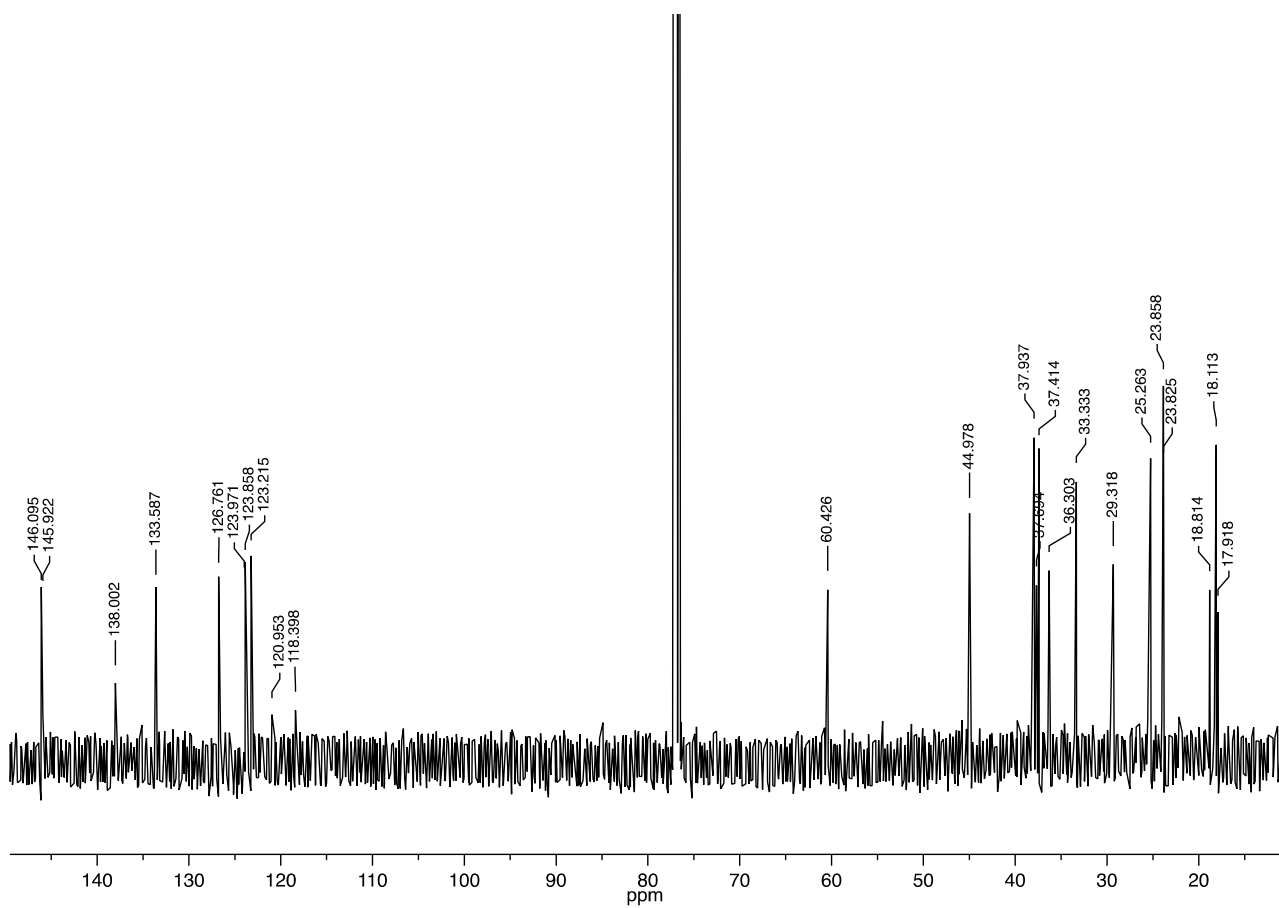




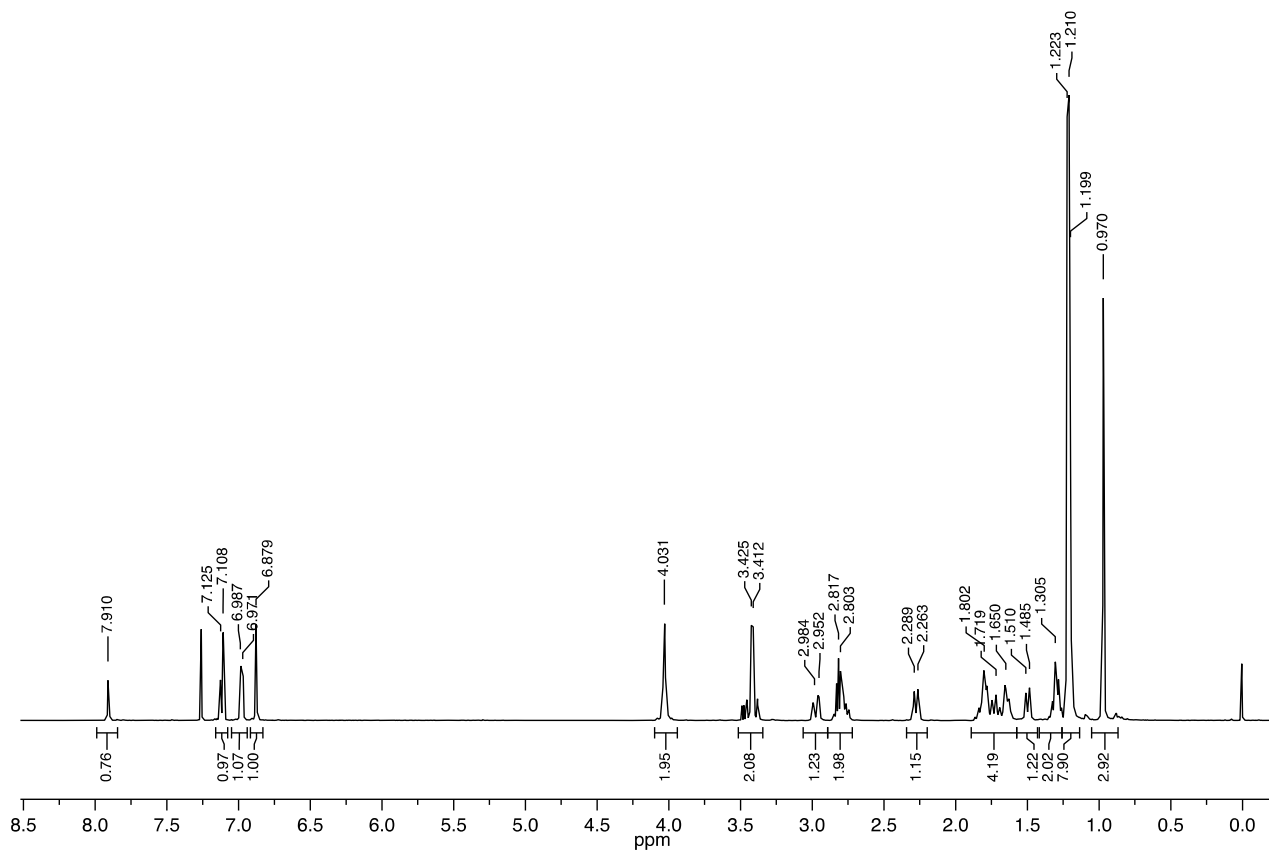
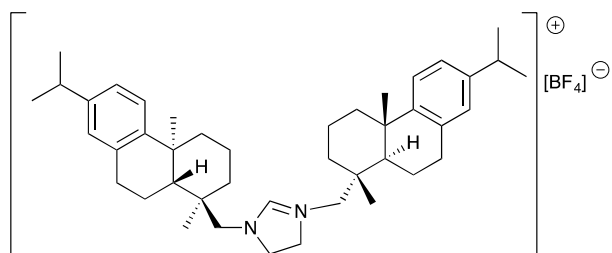


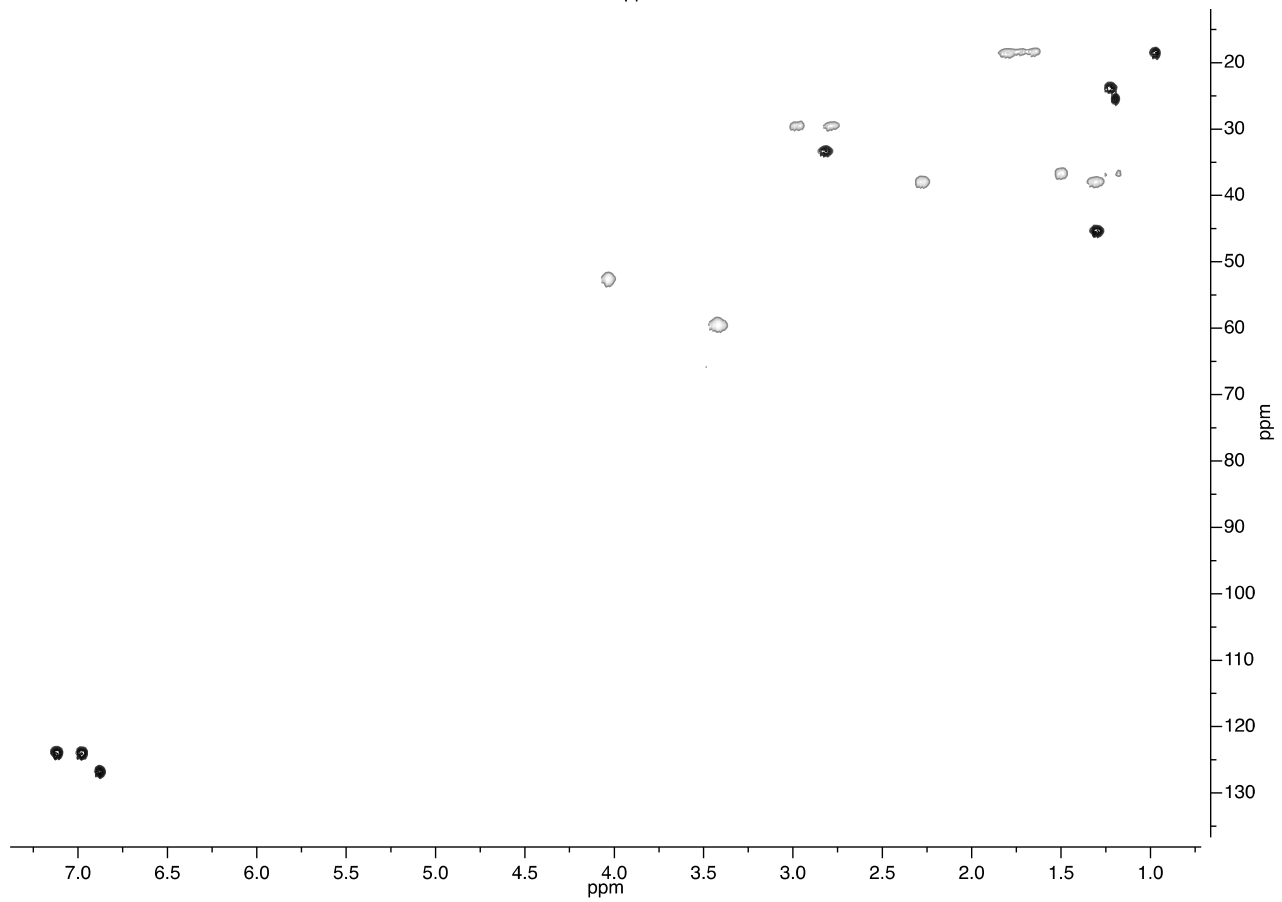
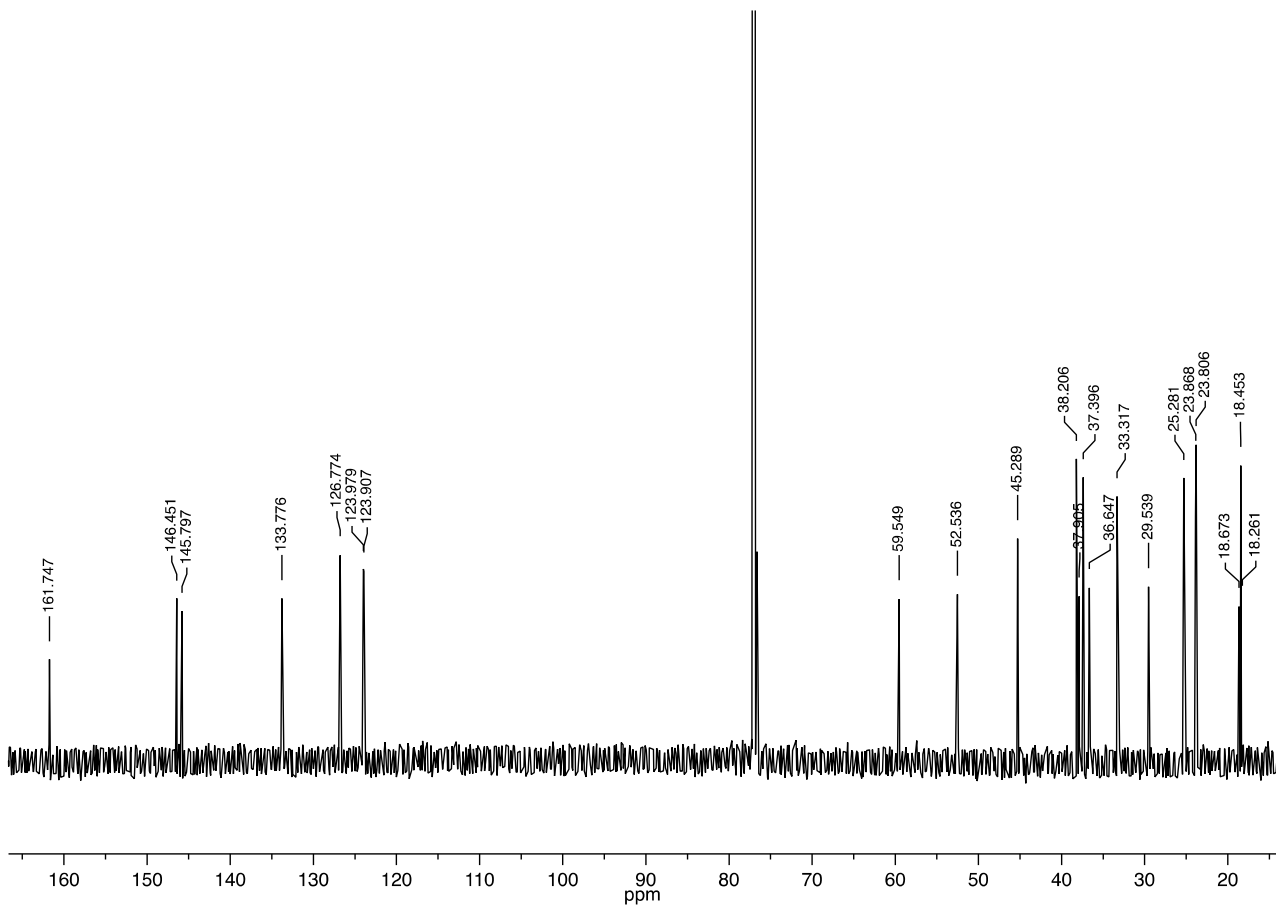
# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 2c



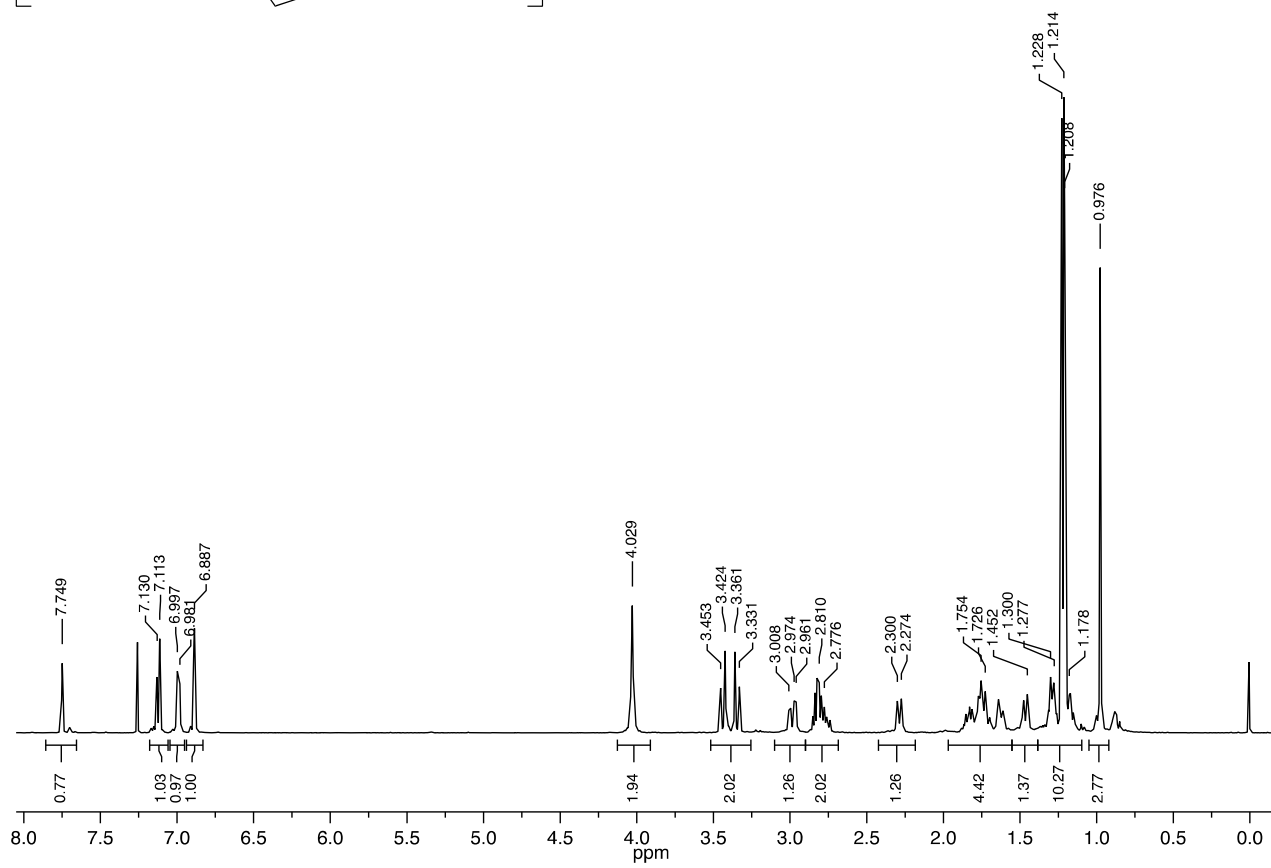
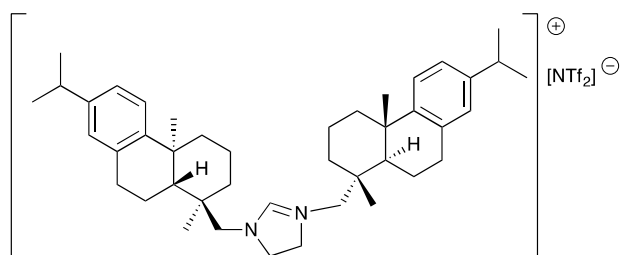


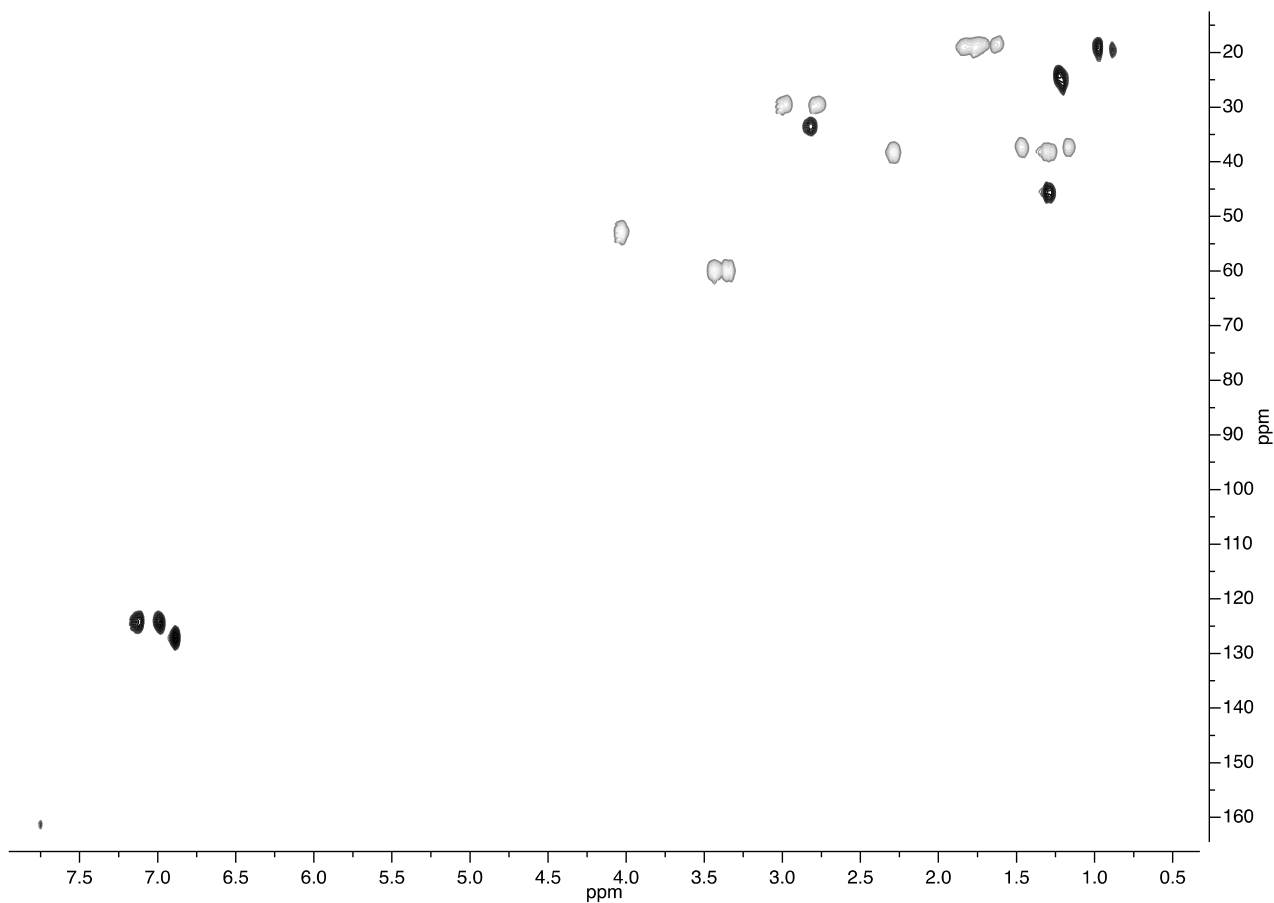
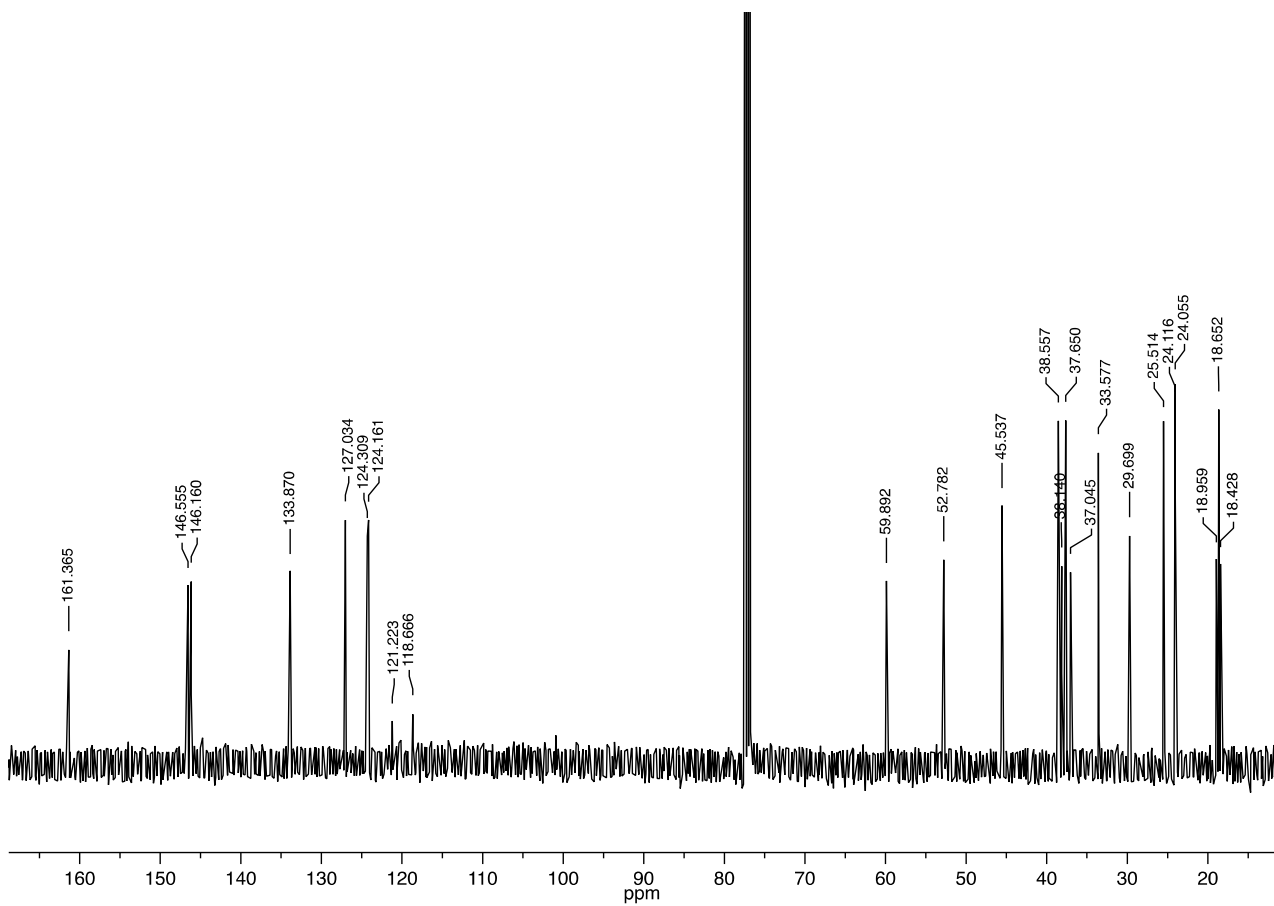
# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 3a



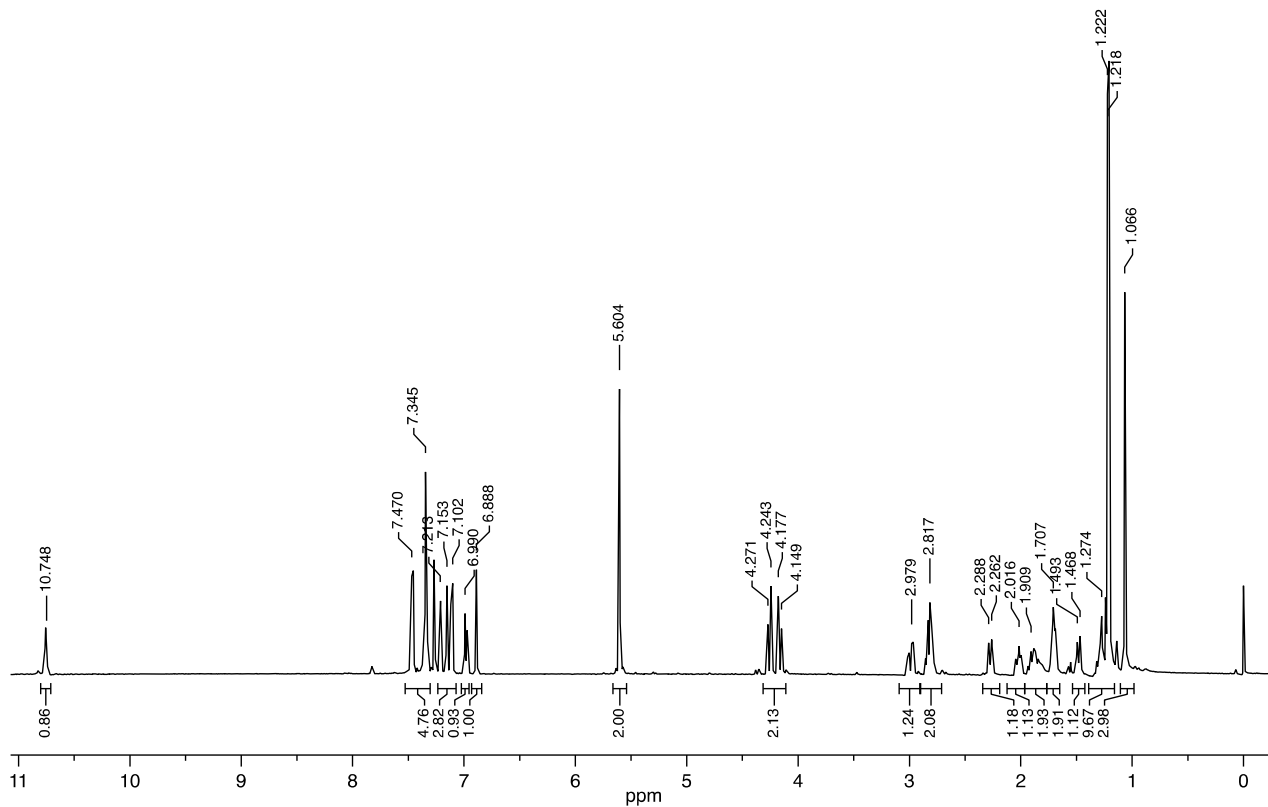
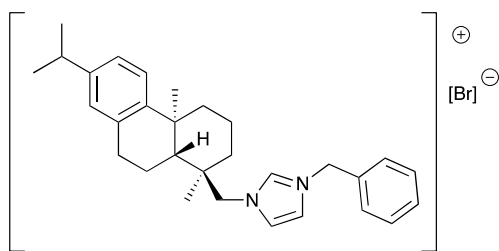


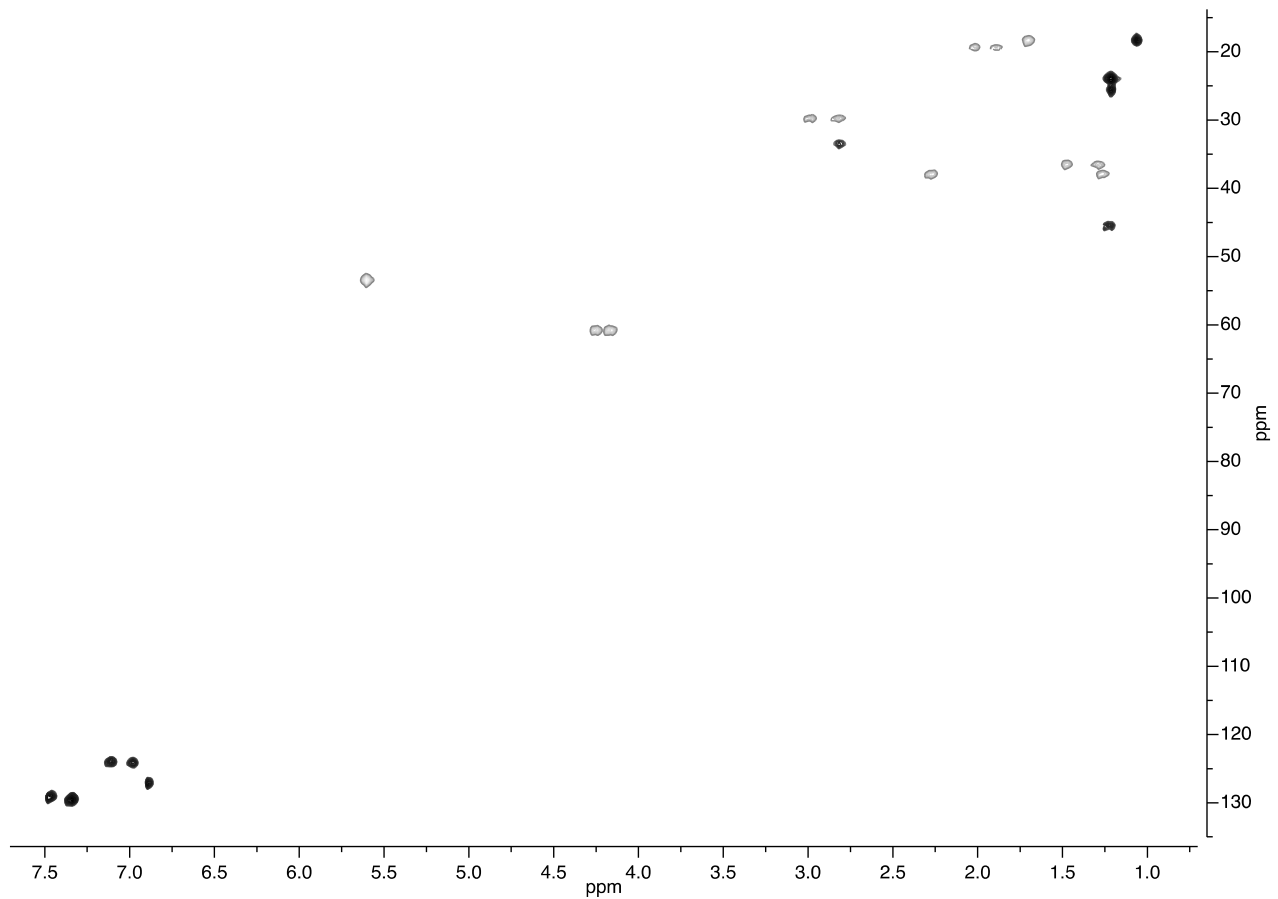
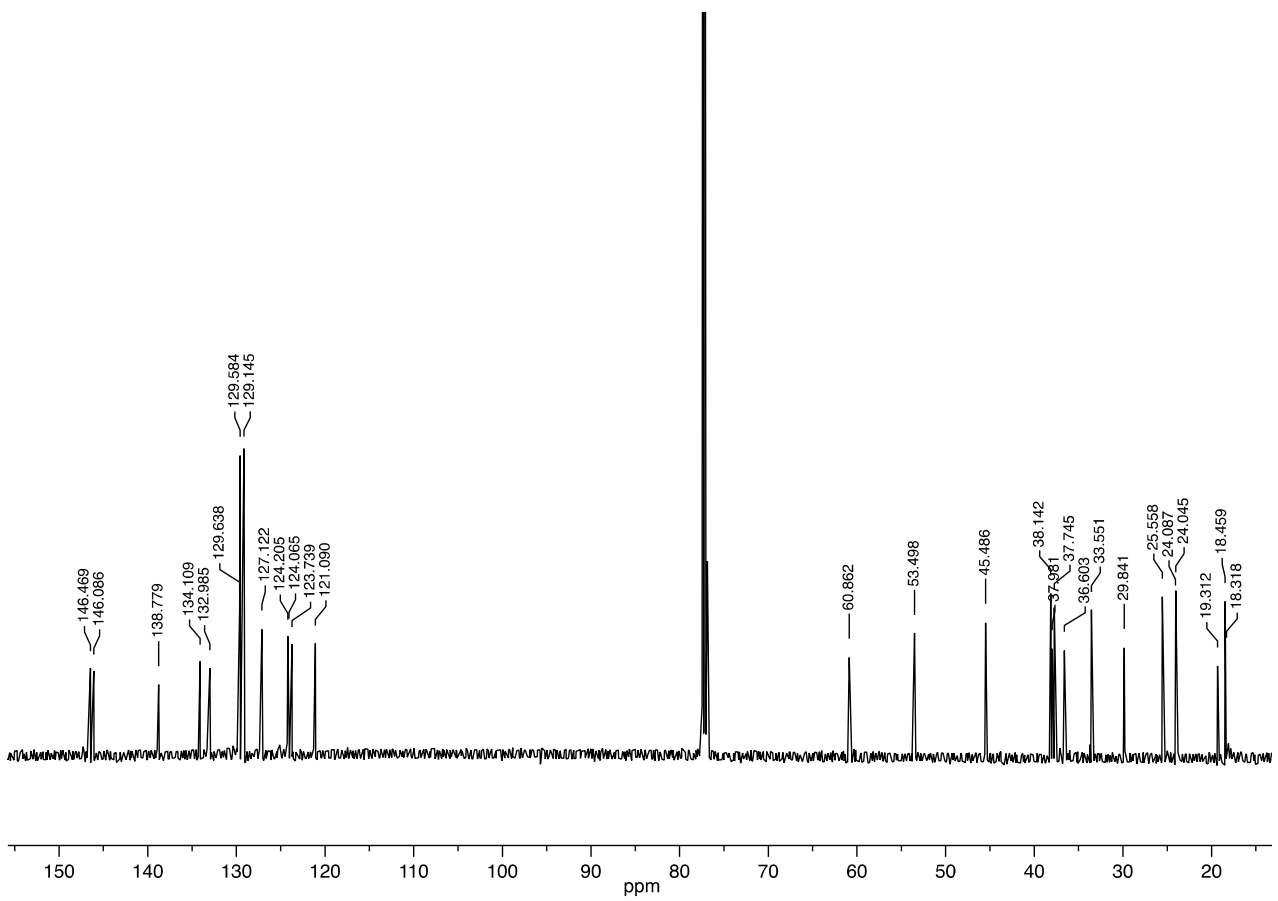
# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 3b





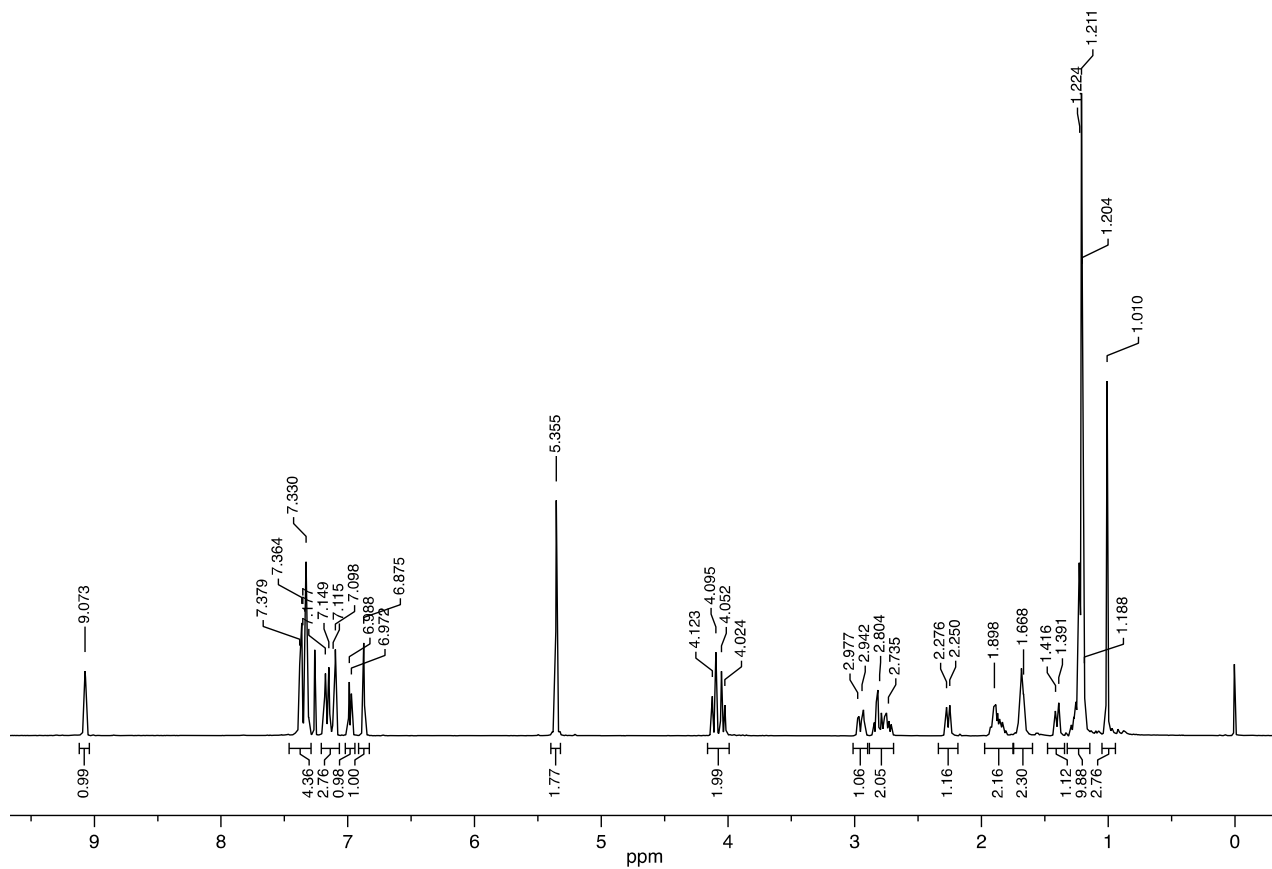
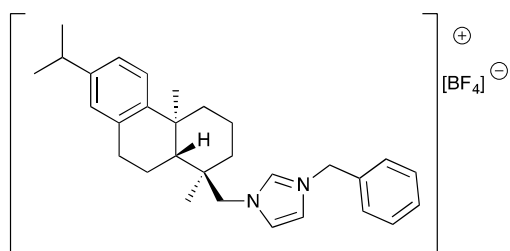
# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 4a

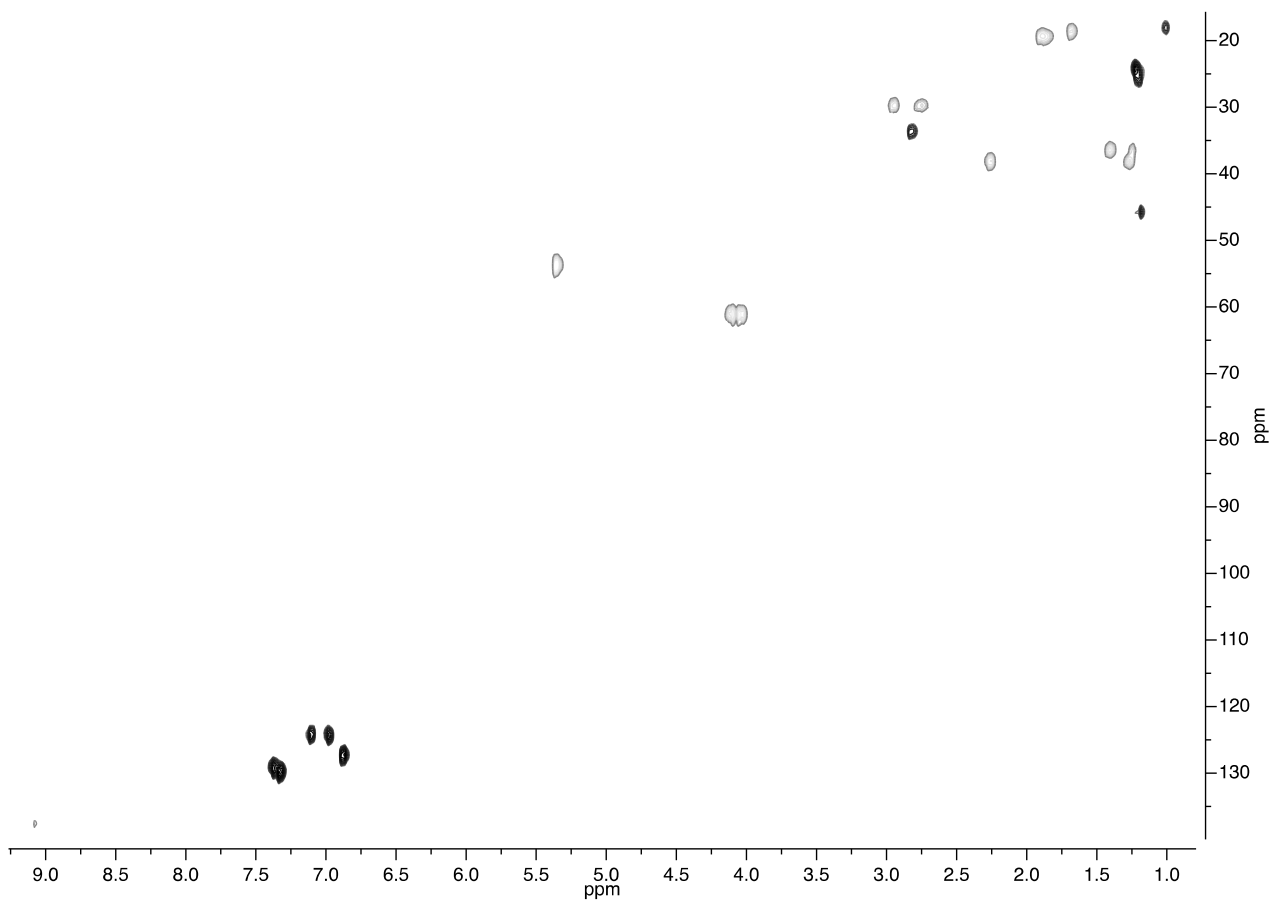
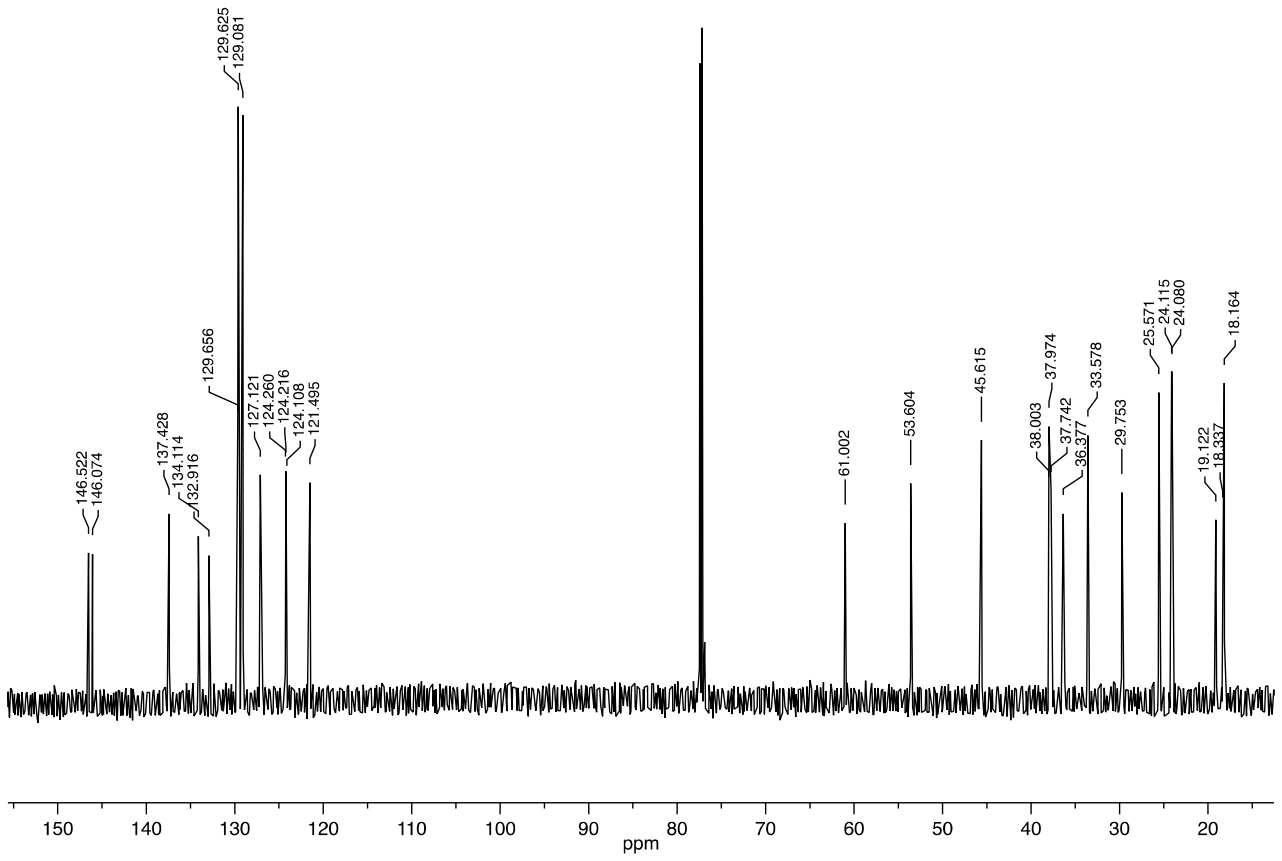






# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 4b





# $^1\text{H}$ , $^{13}\text{C}$ and HSQC spectra of compound 4c

