

## Supplementary Information

### Isolation of C3-Metalated Indolizine Complex and Phosphonium Ring-Fused Bicyclic Metallafuran from Osmium-Induced Transformation of Pyridine-Tethered Alkynes

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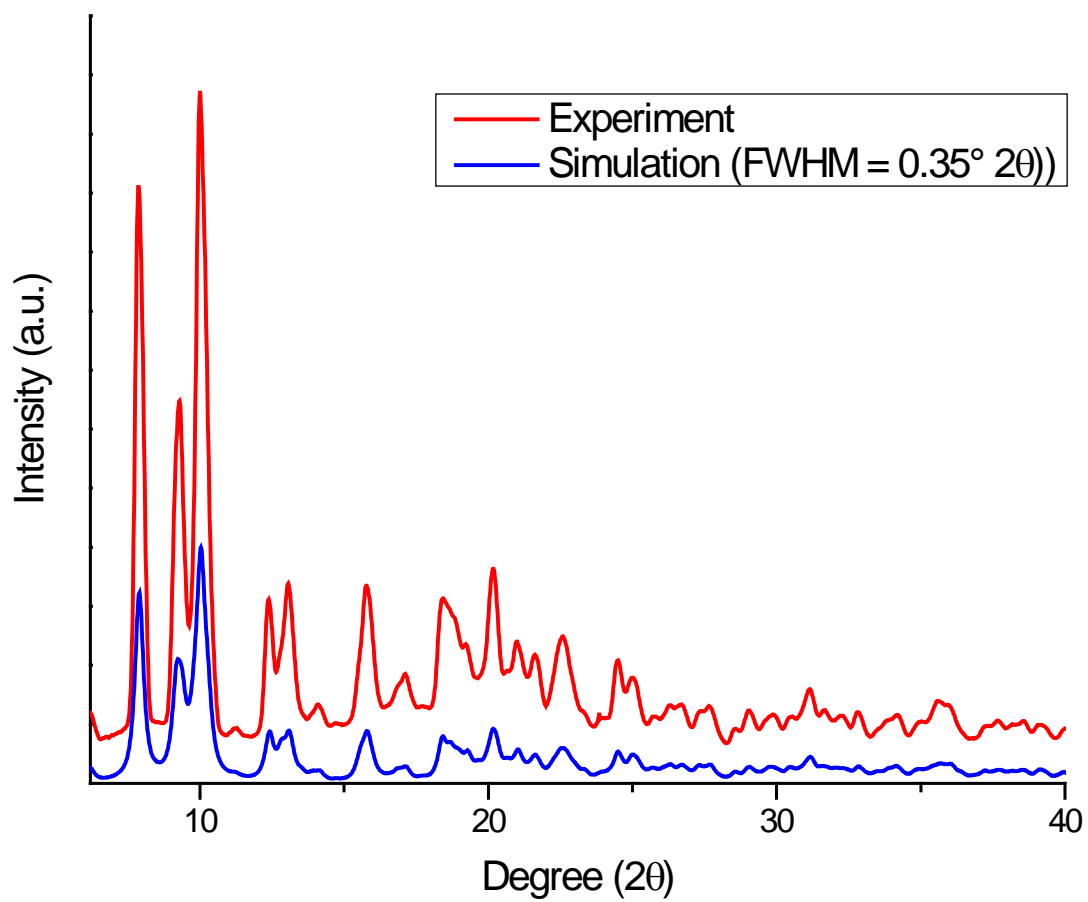
E-mail: acywong@cityu.edu.hk

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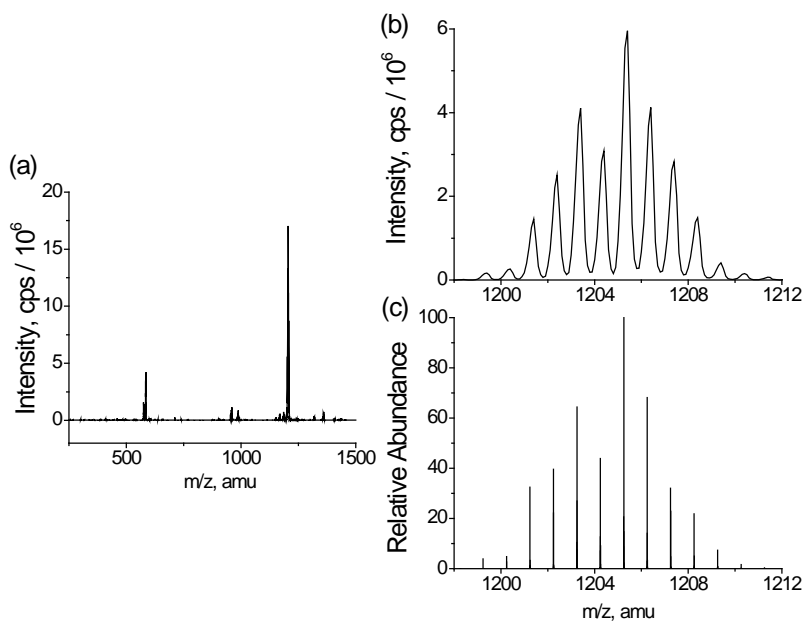
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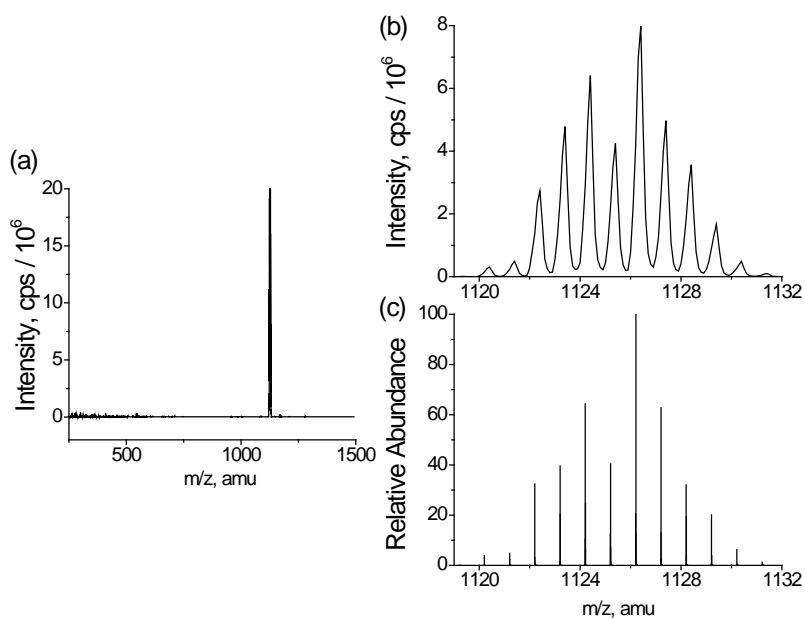
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**Fig. S1** Experimental and Simulated Powder X-ray Diffraction Patterns for the Bulk Sample of **3(OTf)**.



**Fig. S2** (a, b) ESI mass spectrum (positive mode) and experimental isotopic distribution pattern of **3(OTf)** in MeOH. (c) Simulated isotopic distribution pattern of **3(OTf)**.

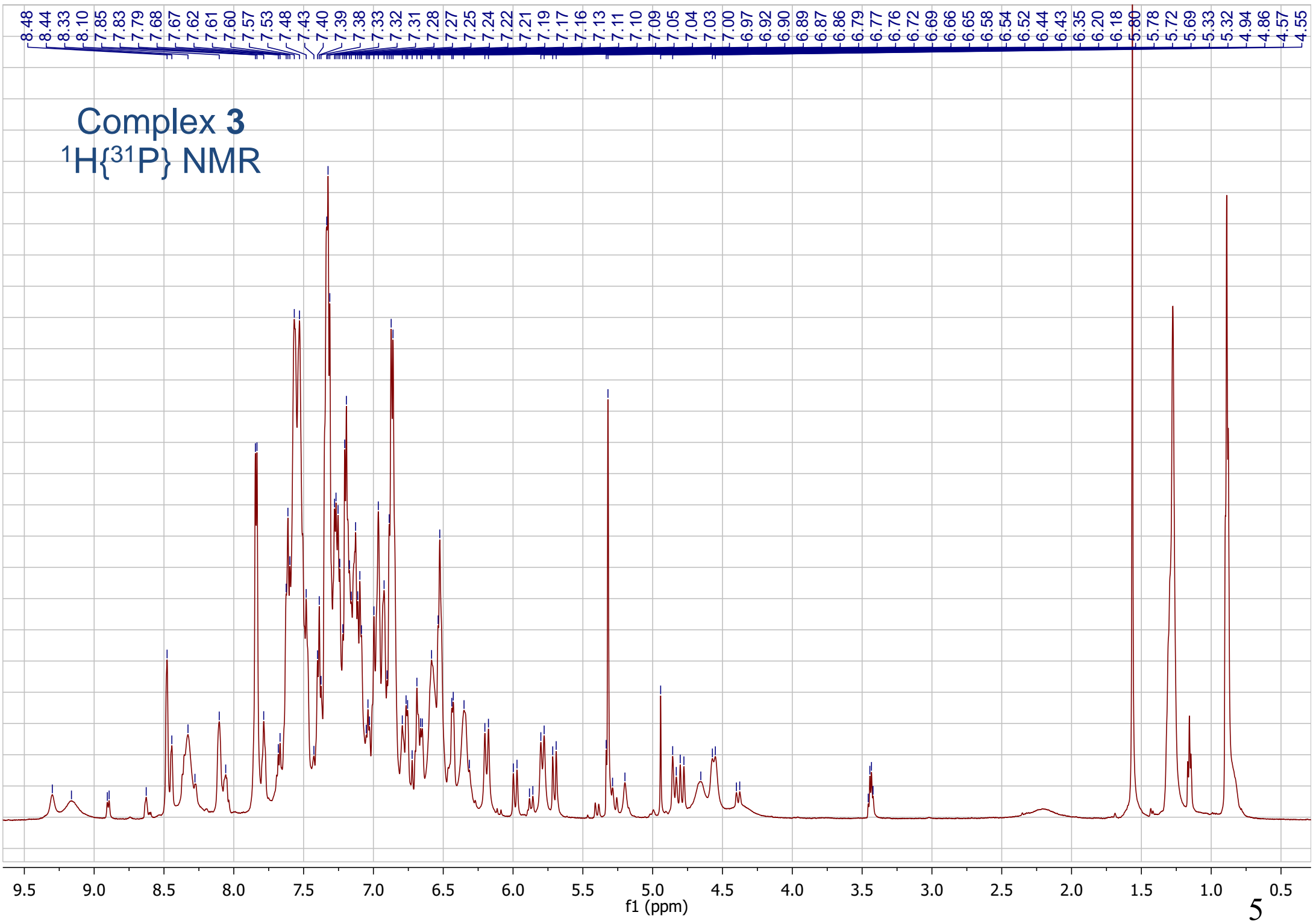


**Fig. S3** (a, b) ESI mass spectrum (positive mode) and experimental isotopic distribution pattern of **4(OTf)** in MeOH. (c) Simulated isotopic distribution pattern of **4(OTf)**.

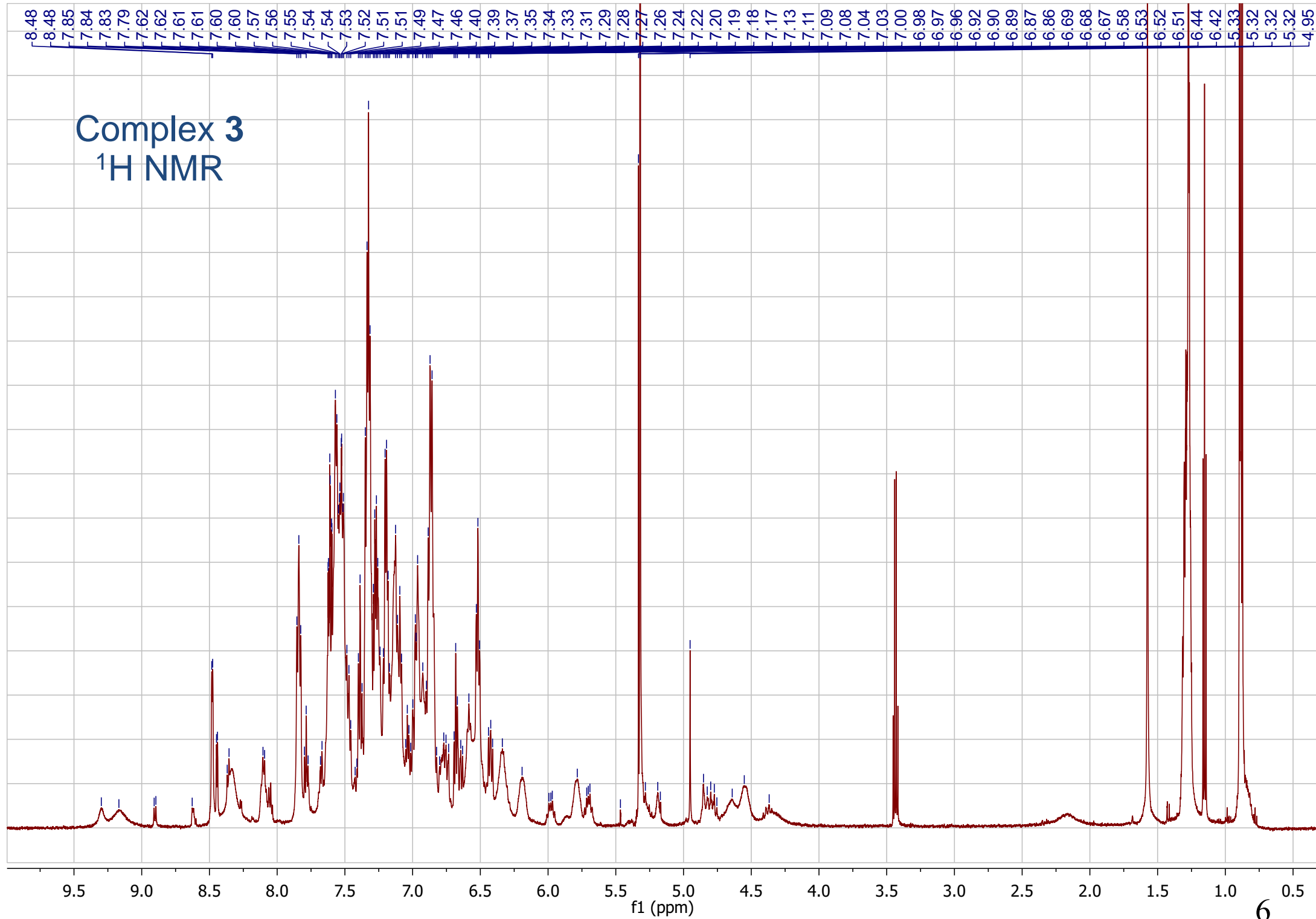
### **NMR Spectra for complexes 3 and 4**

Remark: Solution ( $\text{CD}_2\text{Cl}_2$ ) NMR spectra of **3**(OTf) displayed a combination of at least three species (determined from  $^{31}\text{P}$ – $^{31}\text{P}$  COSY experiment, see pages 8 and 14); similar NMR spectra were still obtained after multiple recrystallization (two to four times). Investigation on the solution behavior by low-temperature NMR experiments was hampered by the poor solubility of the sample in  $\text{CD}_2\text{Cl}_2$ . The NMR spectral data for **3**(OTf) were therefore not assigned, but all NMR spectra recorded are included in the Supplementary Information for reference.

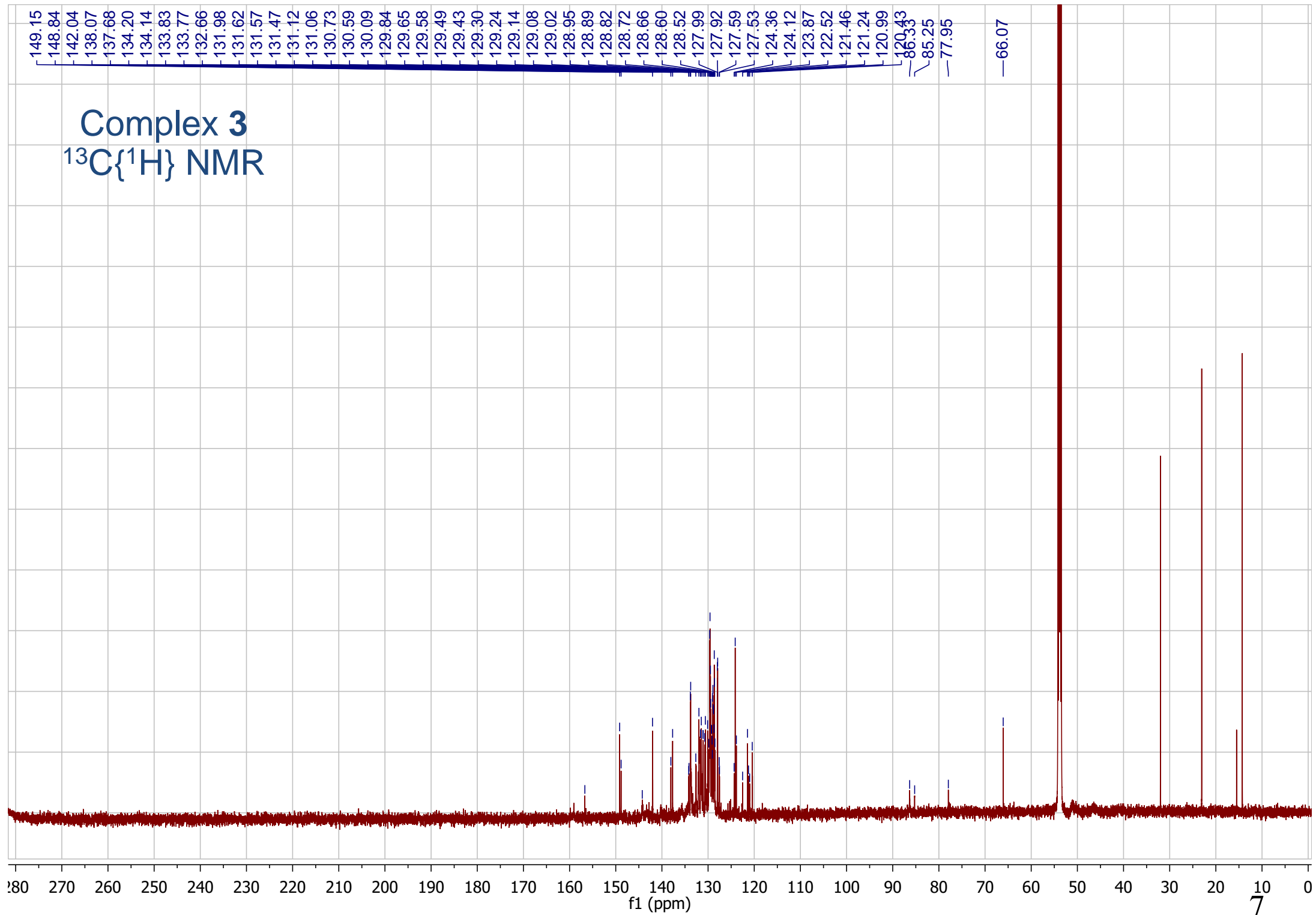
# Complex 3 $^1\text{H}\{^{31}\text{P}\}$ NMR



Complex 3  
<sup>1</sup>H NMR



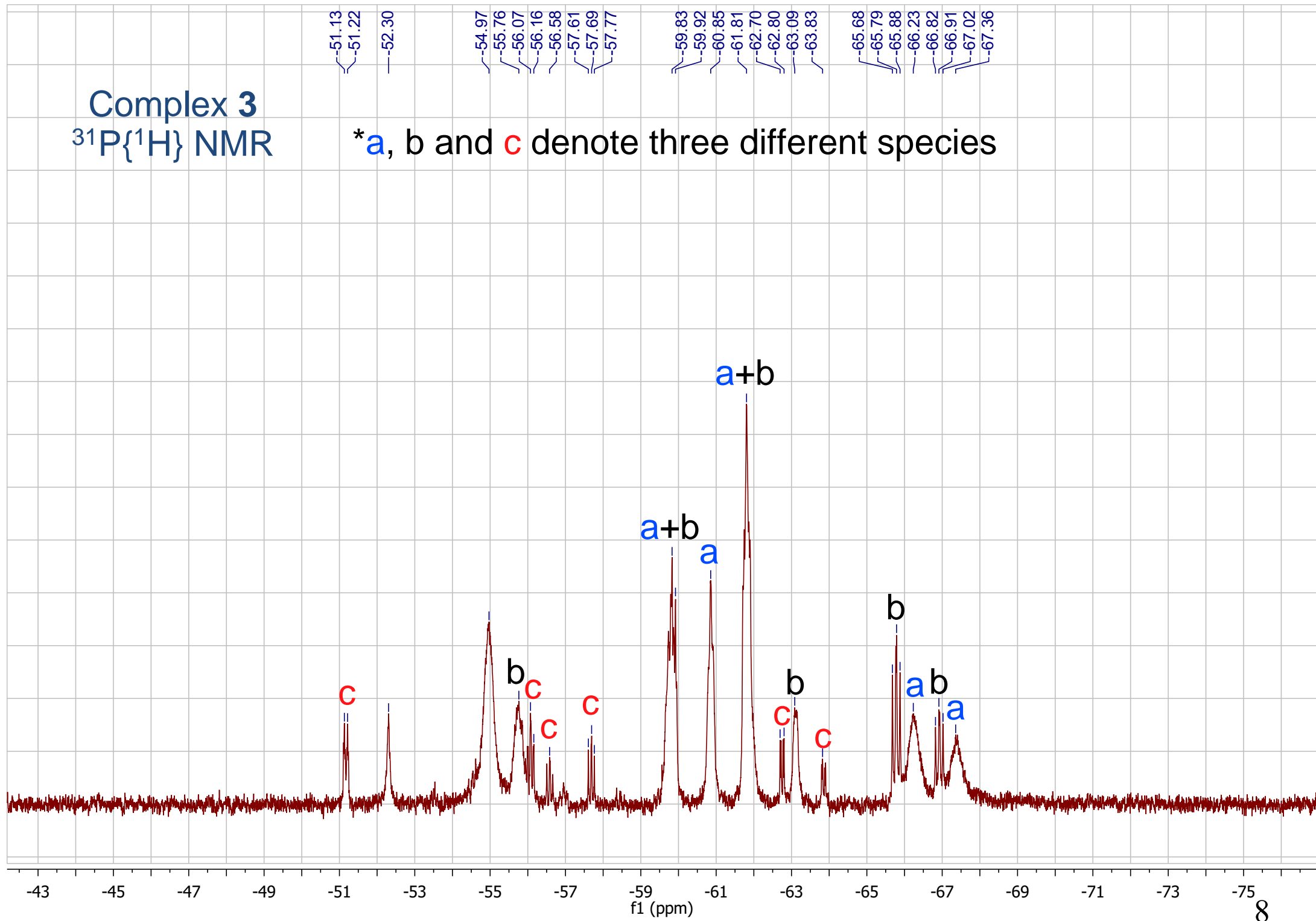
Complex 3  
 $^{13}\text{C}\{^1\text{H}\}$  NMR



# Complex 3 <sup>31</sup>P{<sup>1</sup>H} NMR

\*a, b and c denote three different species

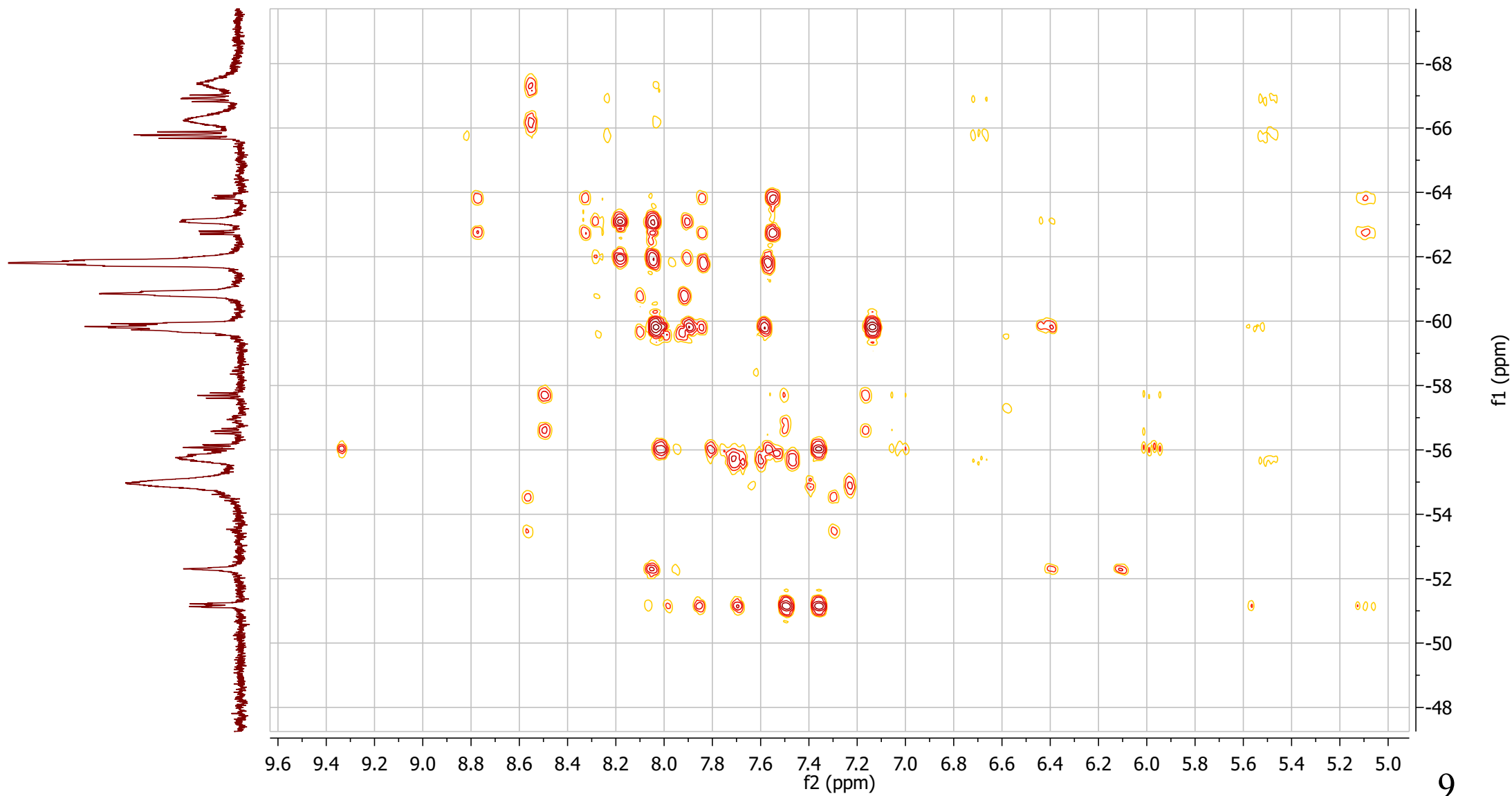
- 51.13
- 51.22
- 52.30
- 54.97
- 55.76
- 56.07
- 56.16
- 56.58
- 57.61
- 57.69
- 57.77
- 59.83
- 59.92
- 60.85
- 61.81
- 62.70
- 62.80
- 63.09
- 63.83
- 65.68
- 65.79
- 65.88
- 66.23
- 66.82
- 66.91
- 67.02
- 67.36



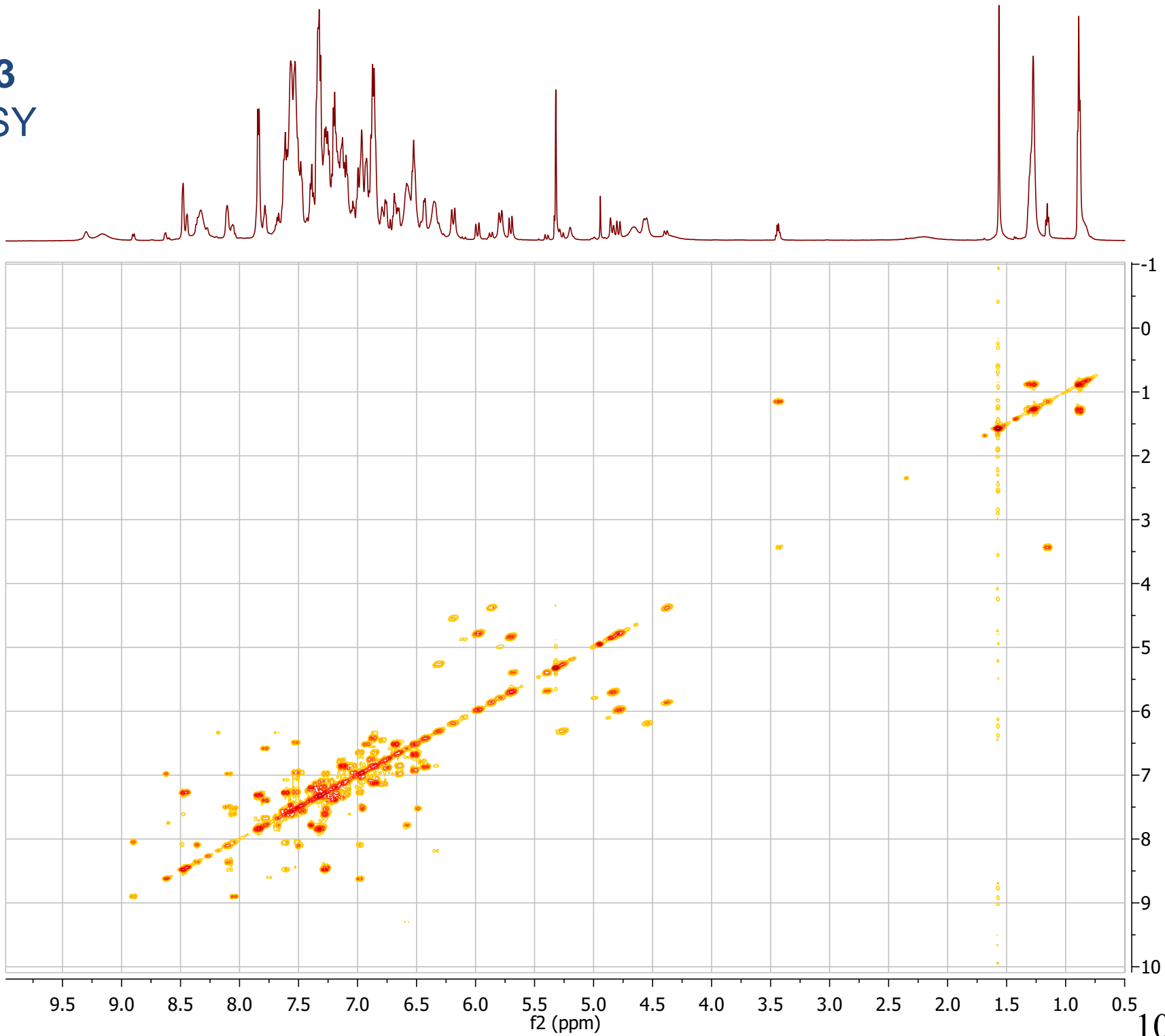


# Complex 3

## $^1\text{H}$ - $^{31}\text{P}$ HMBC

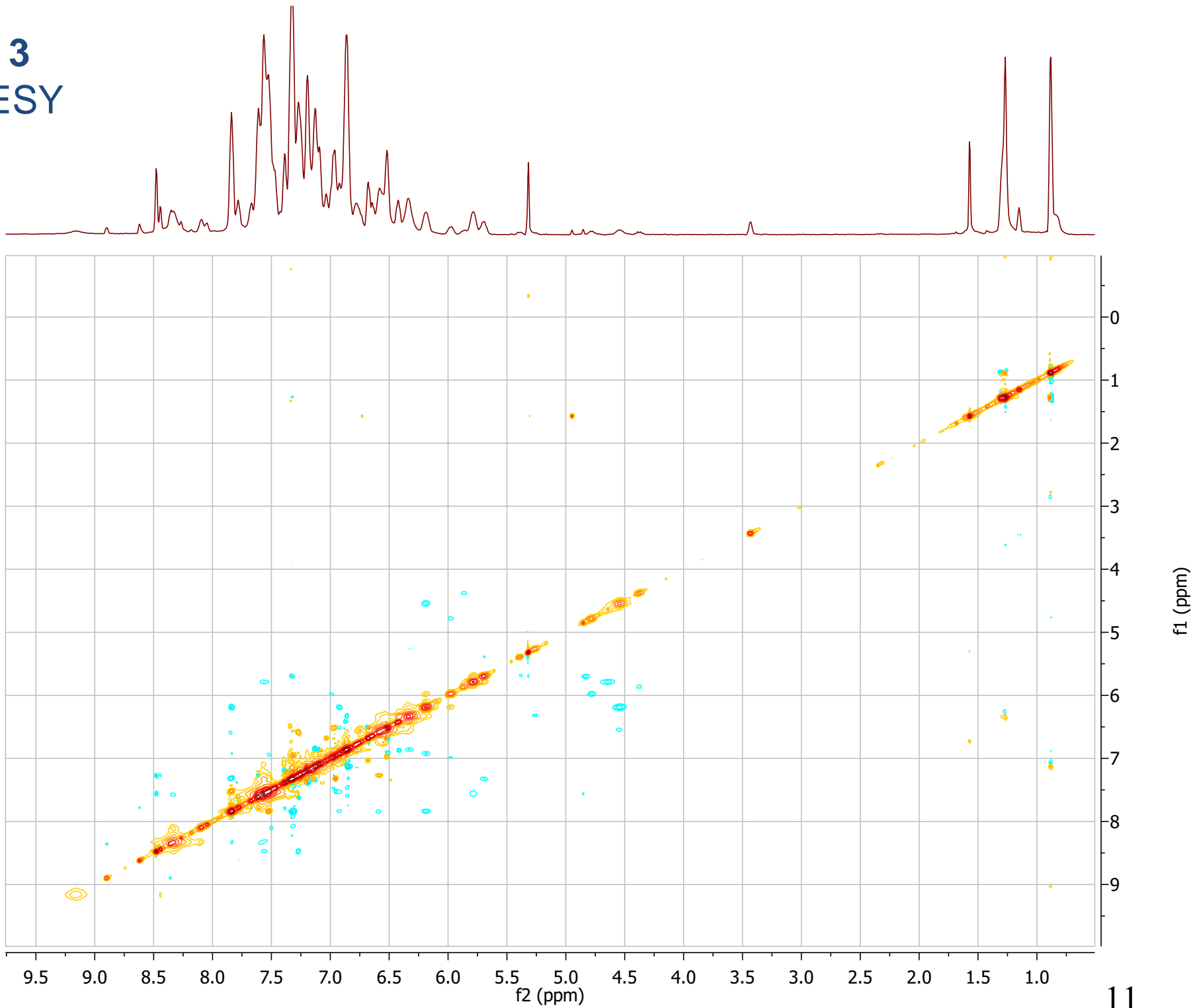


Complex 3  
 $^1\text{H}-^1\text{H}$  COSY



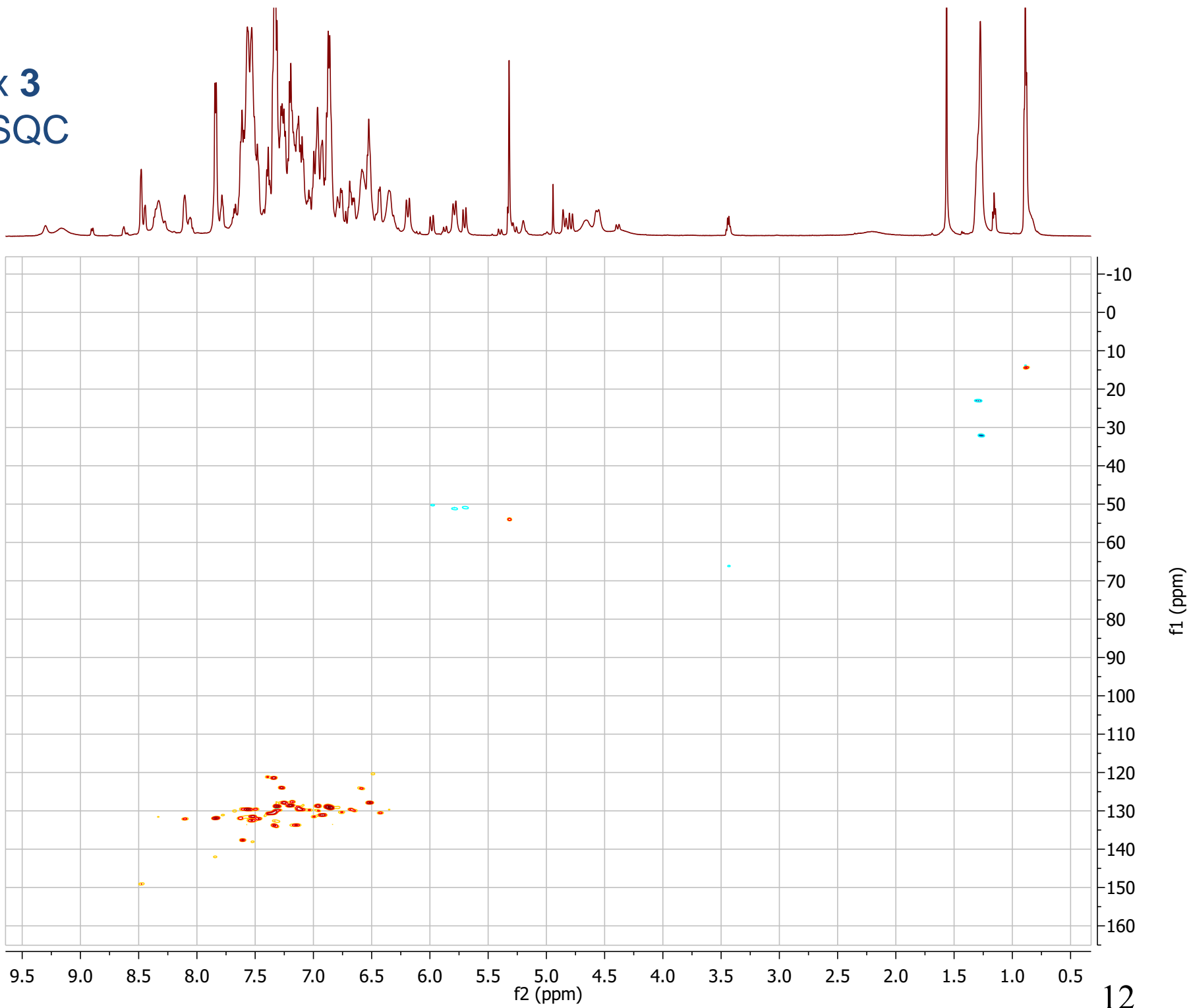
# Complex 3

## $^1\text{H}$ - $^1\text{H}$ ROESY

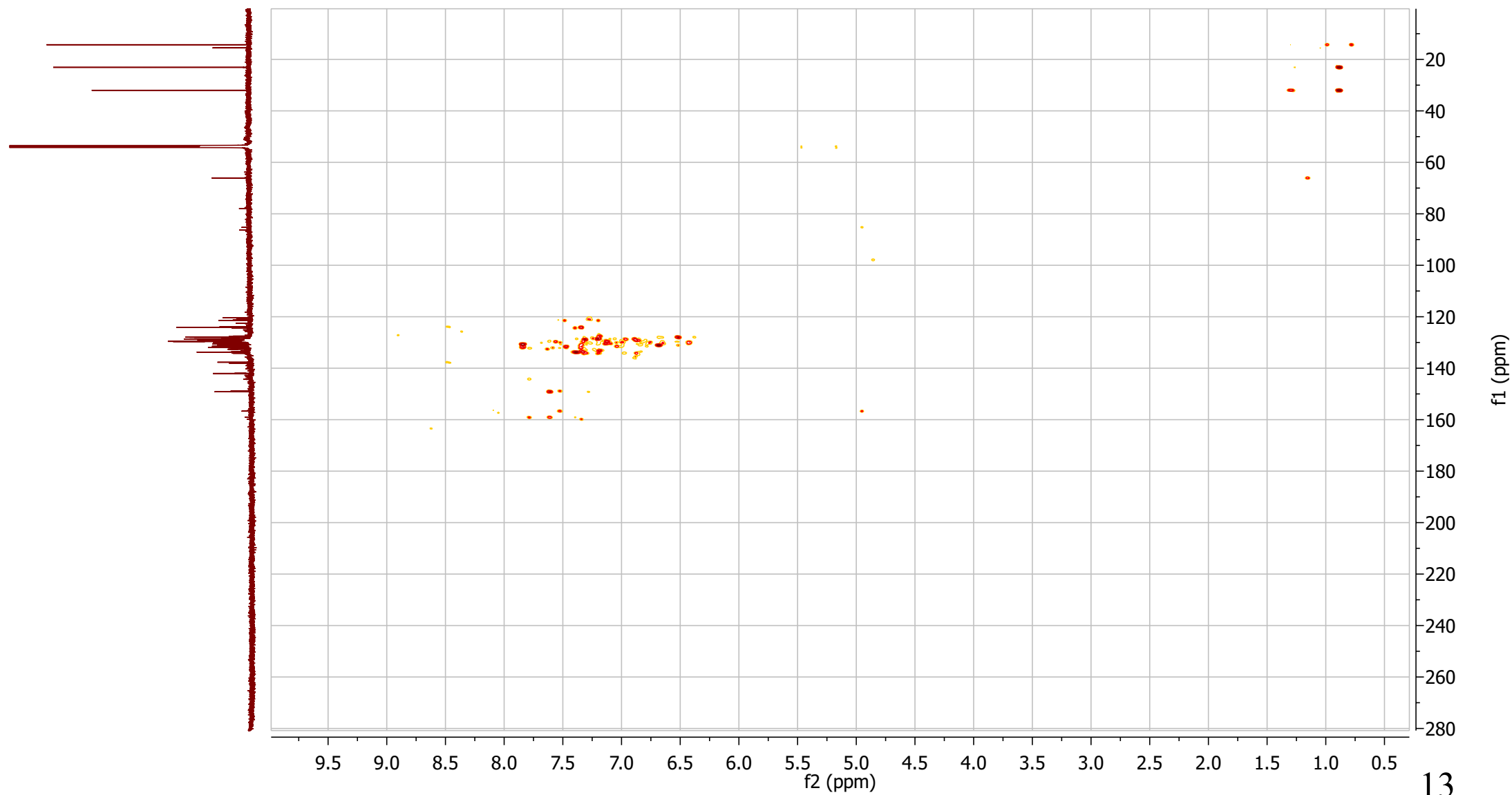
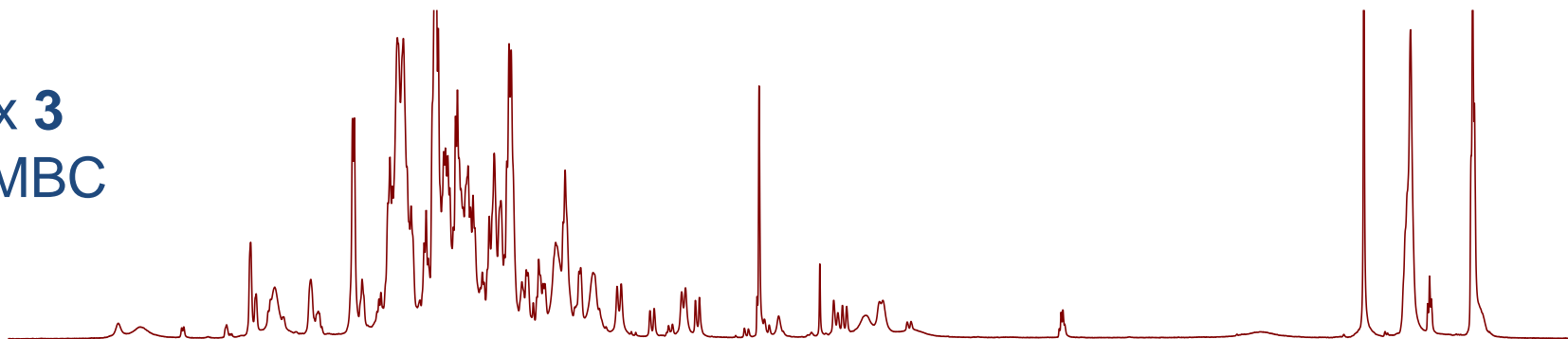


# Complex 3

## $^1\text{H}$ - $^{13}\text{C}$ HSQC

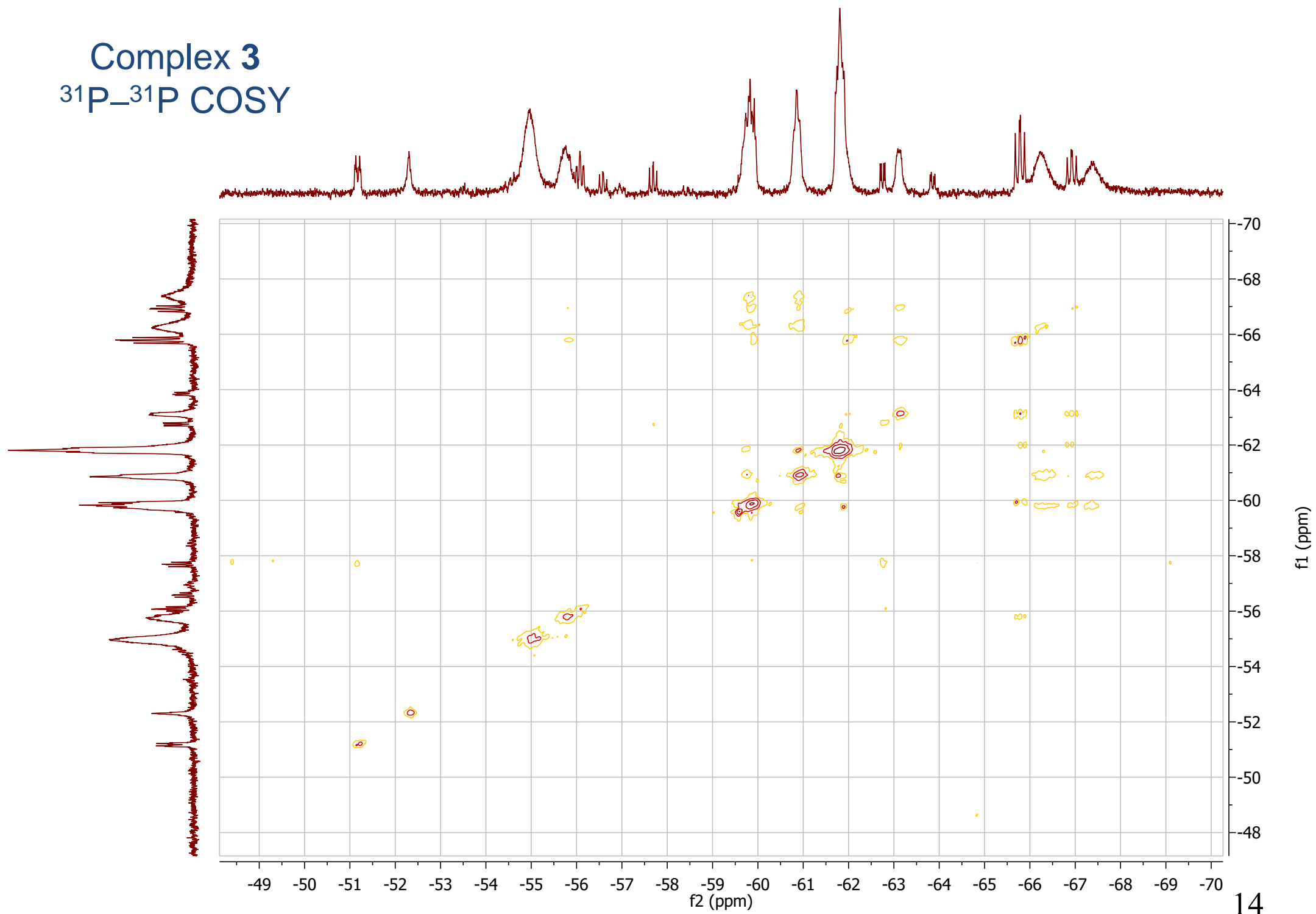


Complex 3  
 $^1\text{H}-^{13}\text{C}$  HMBC

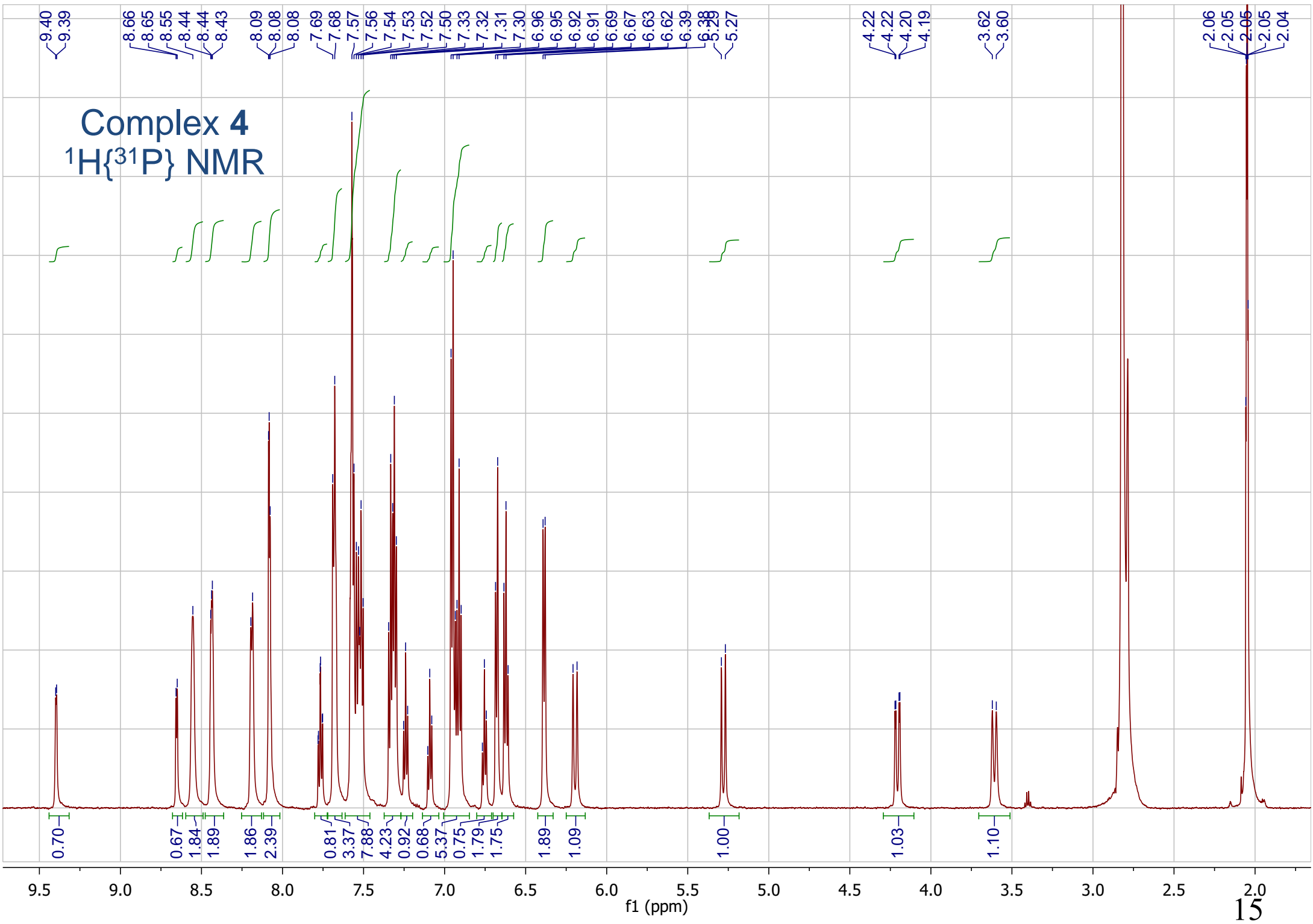


# Complex 3

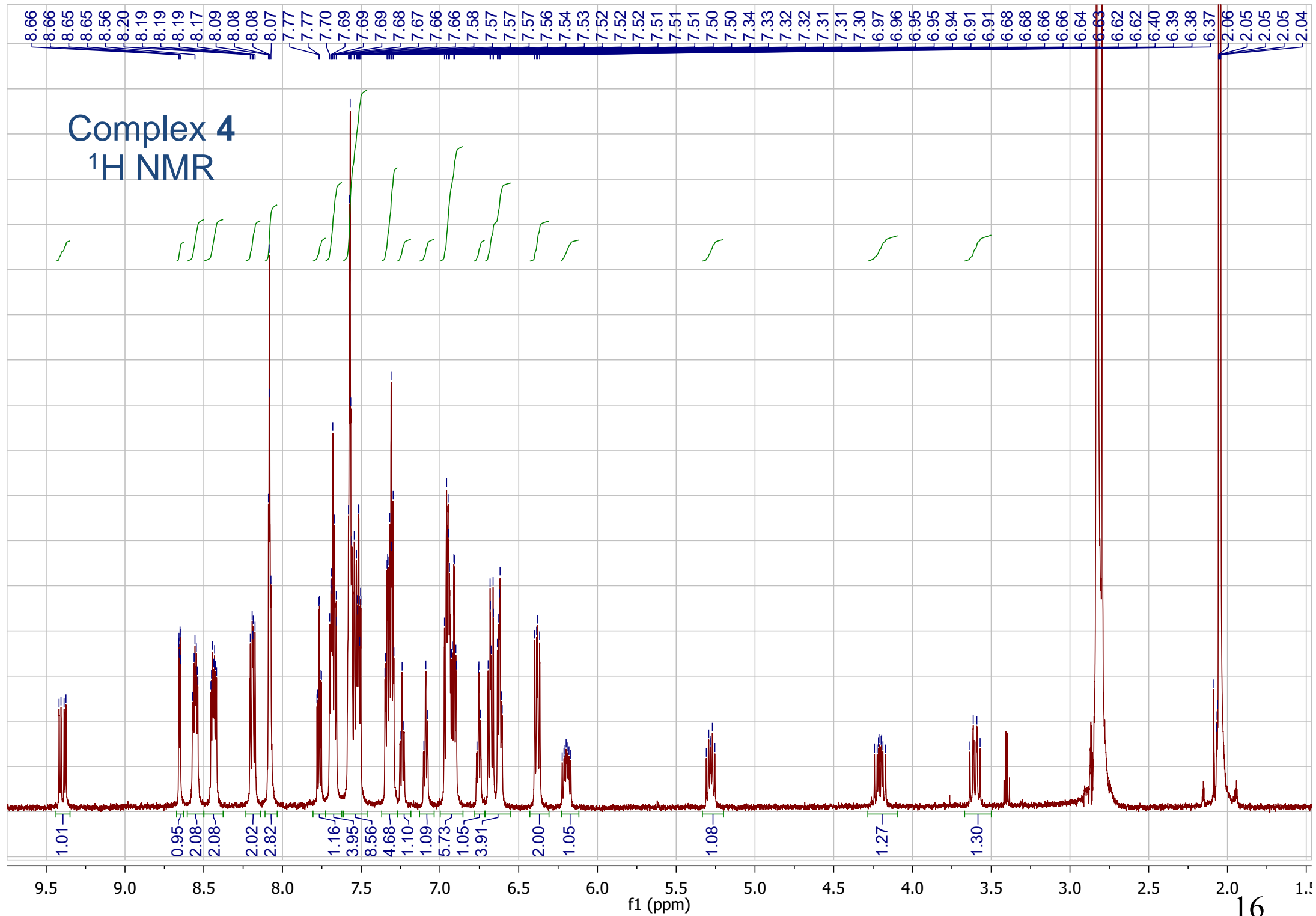
## $^{31}\text{P}$ - $^{31}\text{P}$ COSY



Complex 4  
 $^1\text{H}\{^{31}\text{P}\}$  NMR

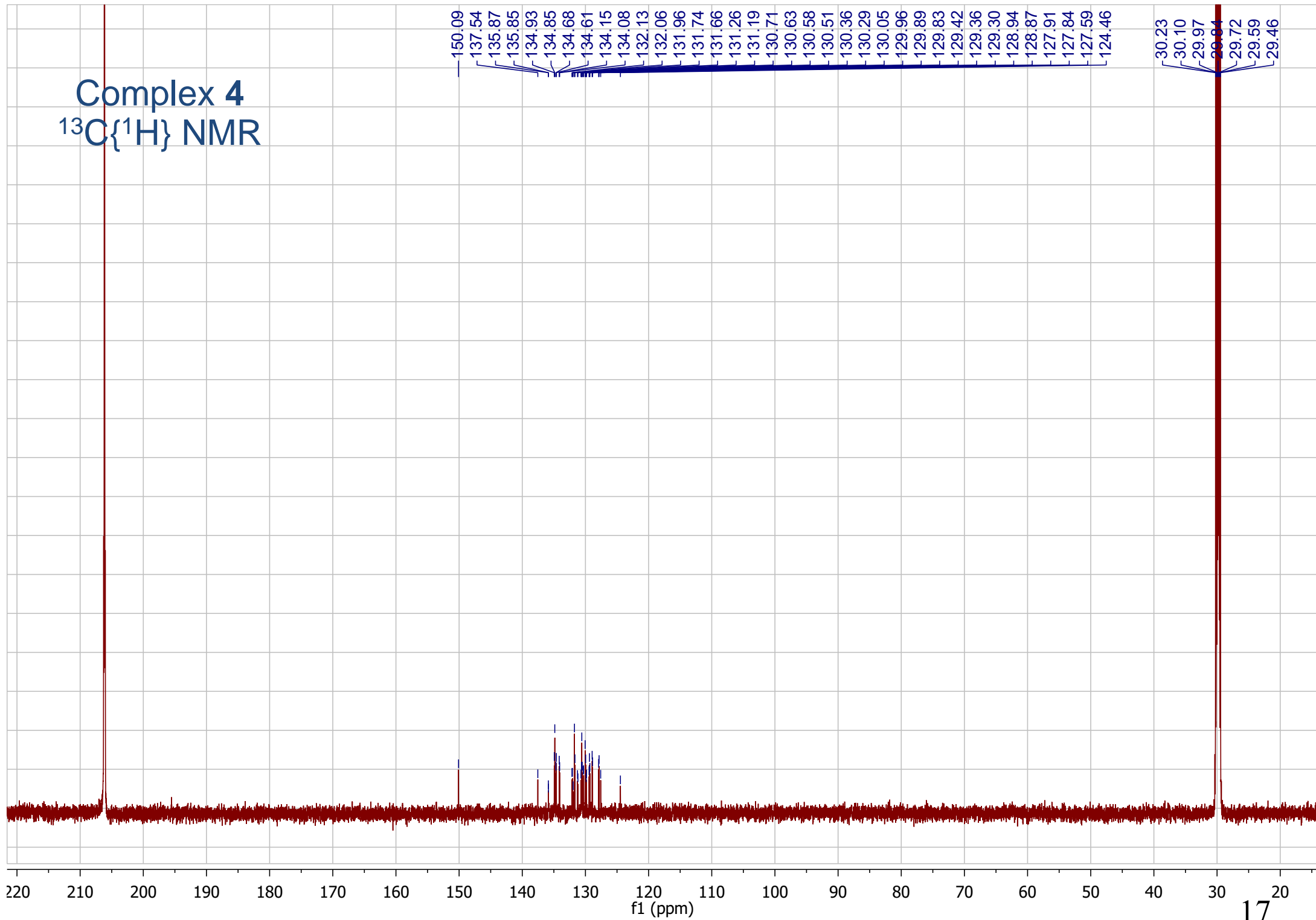


# Complex 4 <sup>1</sup>H NMR





Complex 4  
 $^{13}\text{C}\{^1\text{H}\}$  NMR



Complex 4  
 $^{31}\text{P}\{^1\text{H}\}$  NMR

27.02  
26.93  
26.88  
26.80

-5.94  
-5.99  
-6.09  
-6.15

-42.02  
-42.08  
-42.17

-63.65  
-63.73  
-63.82

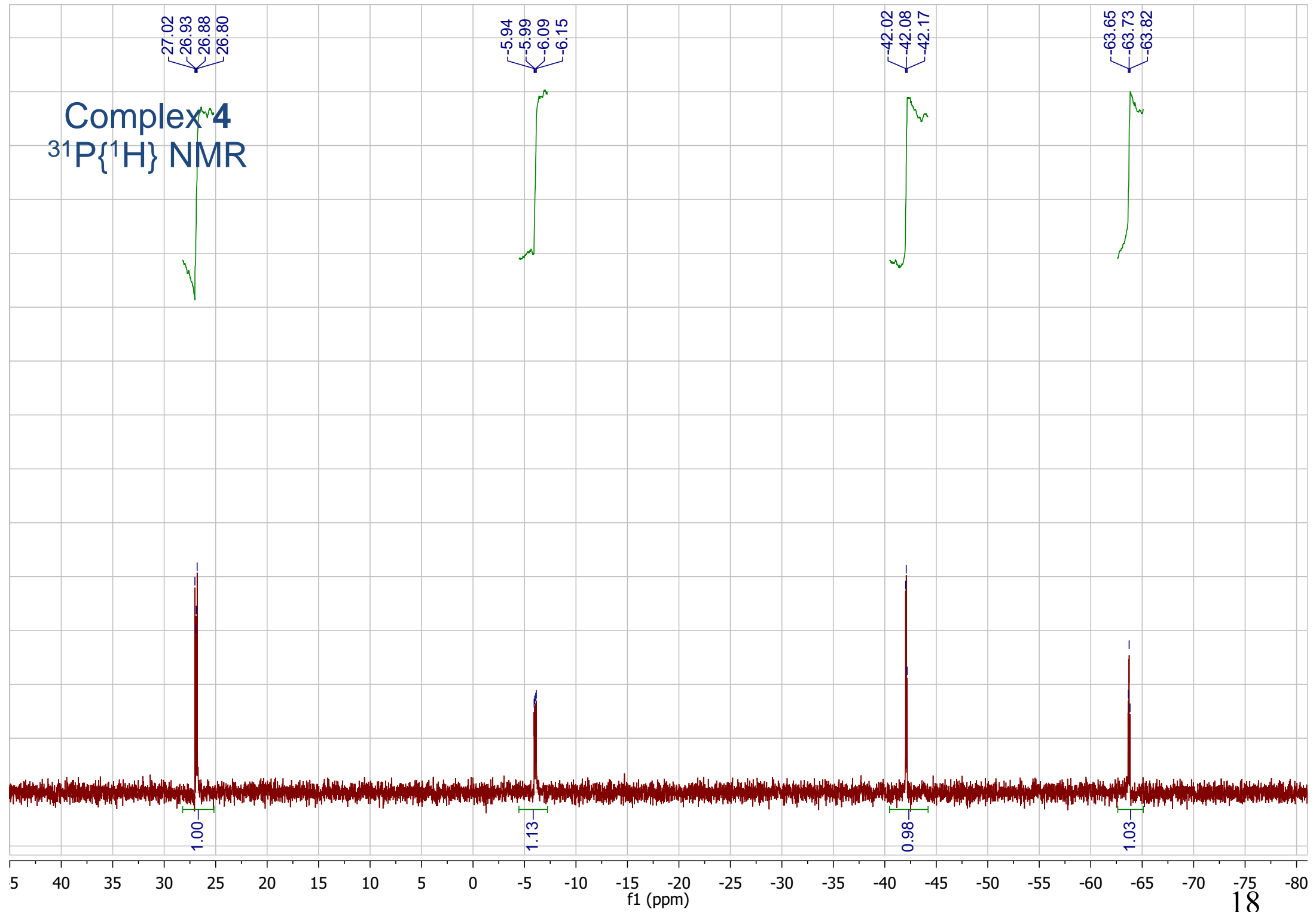
1.00

1.13

0.98

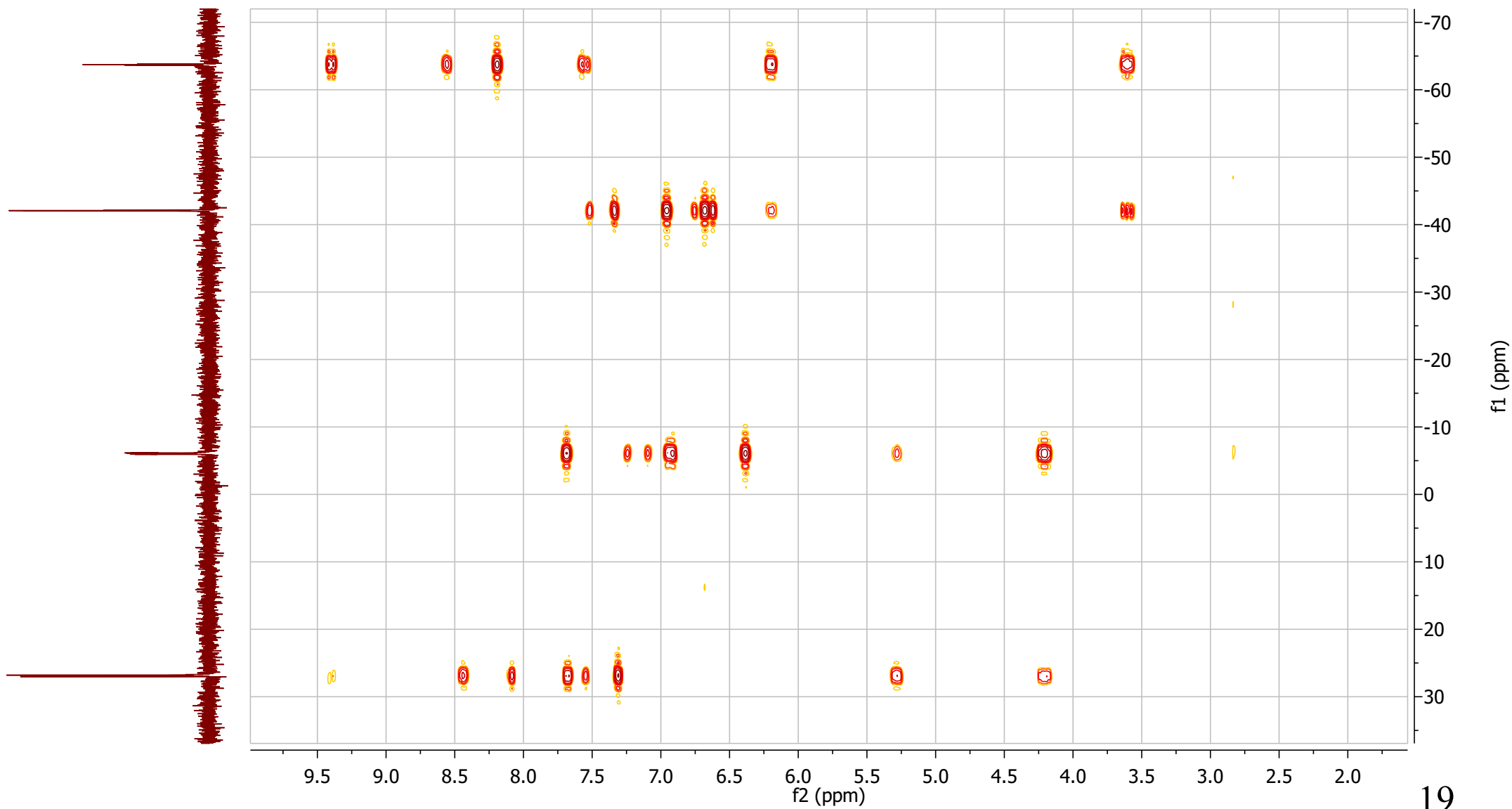
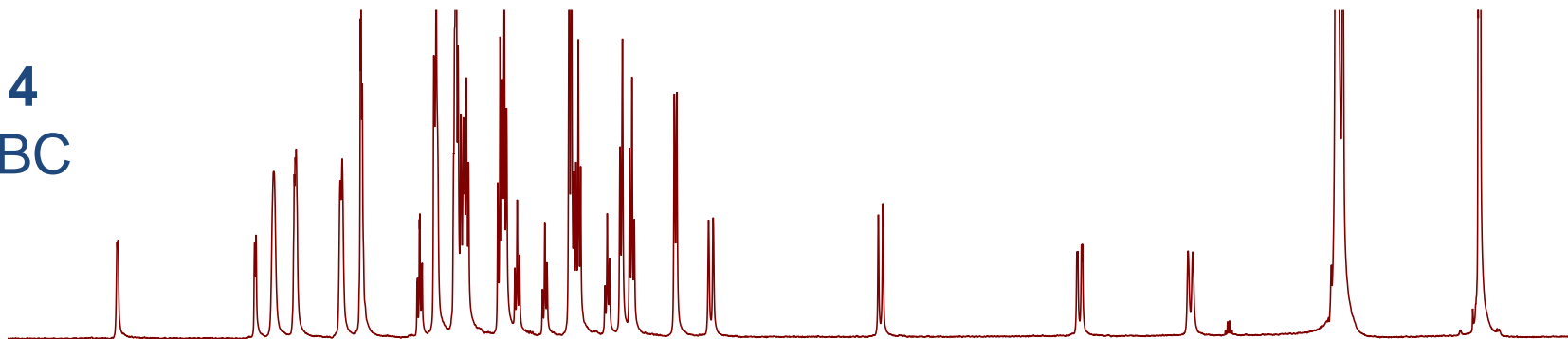
1.03

f1 (ppm)

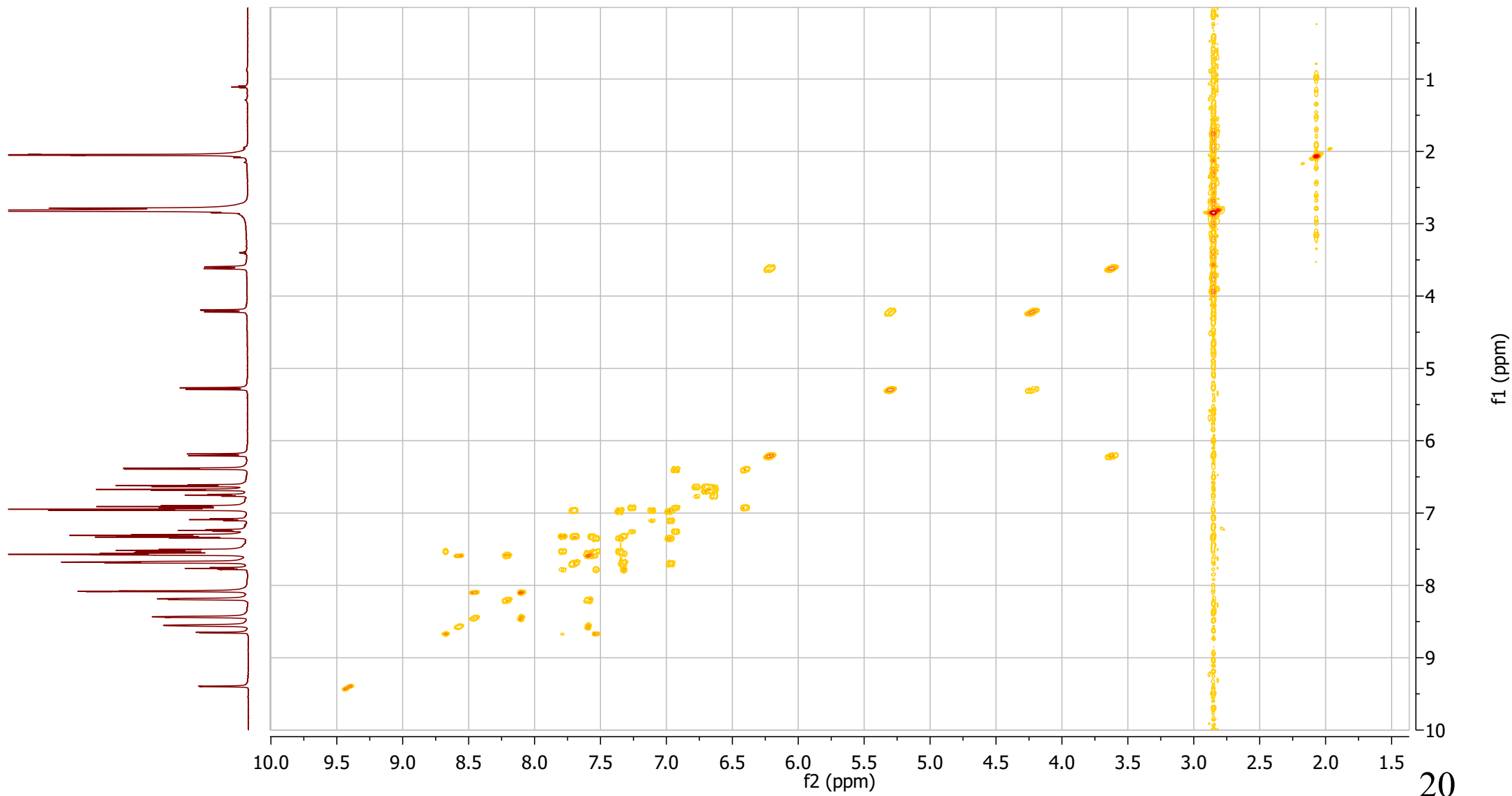
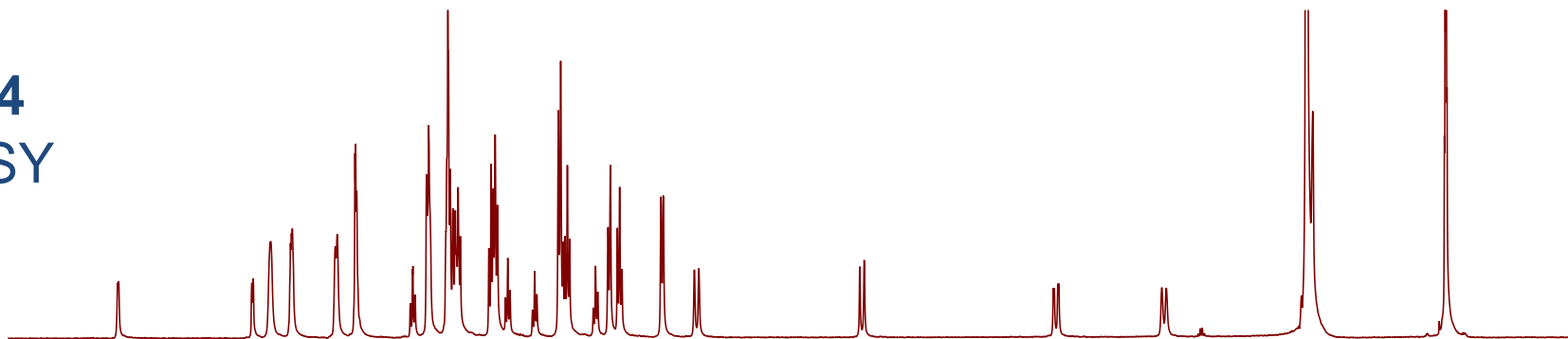


# Complex 4

## $^1\text{H}$ - $^{31}\text{P}$ HMBC

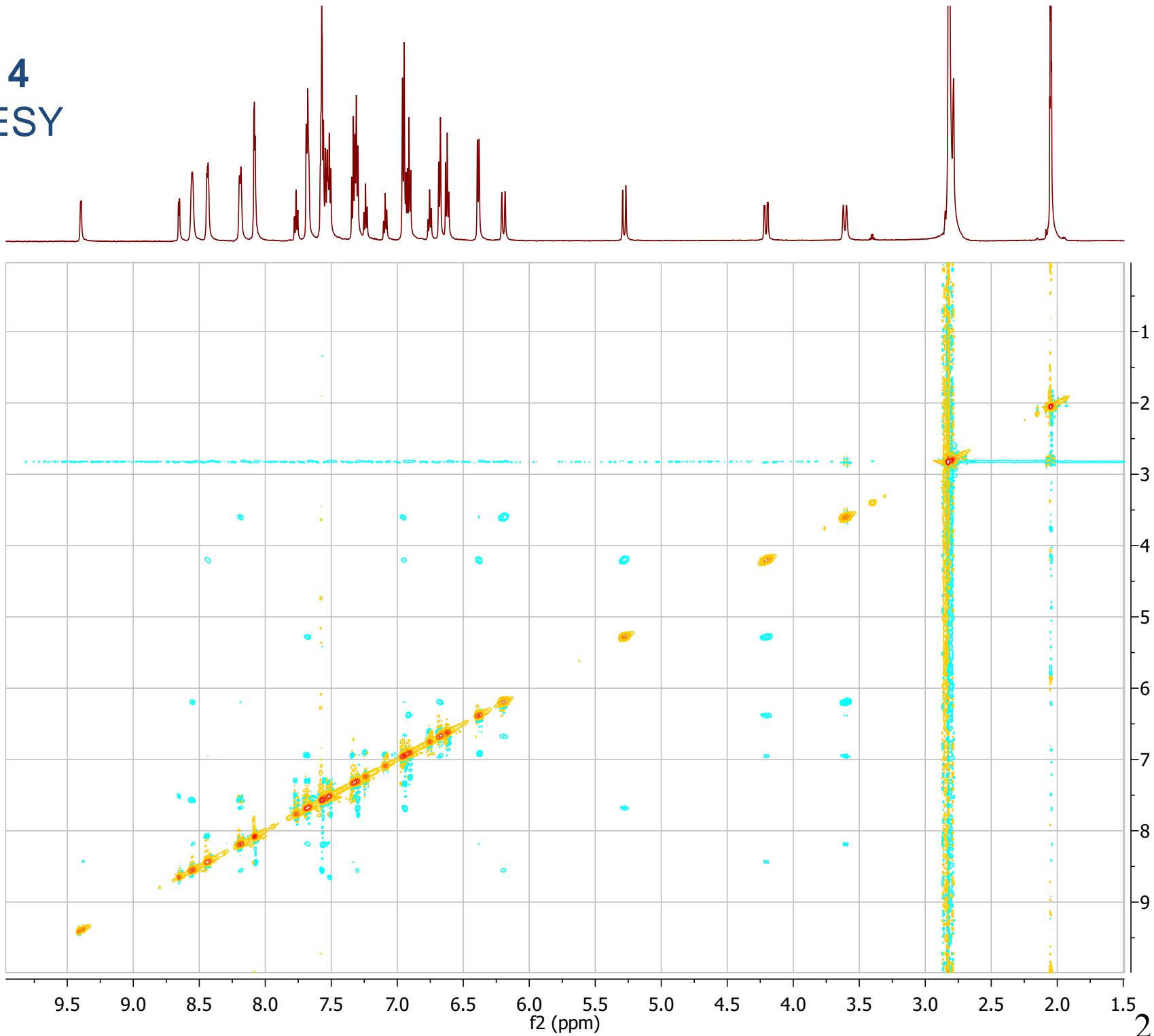


Complex 4  
 $^1\text{H}-^1\text{H}$  COSY

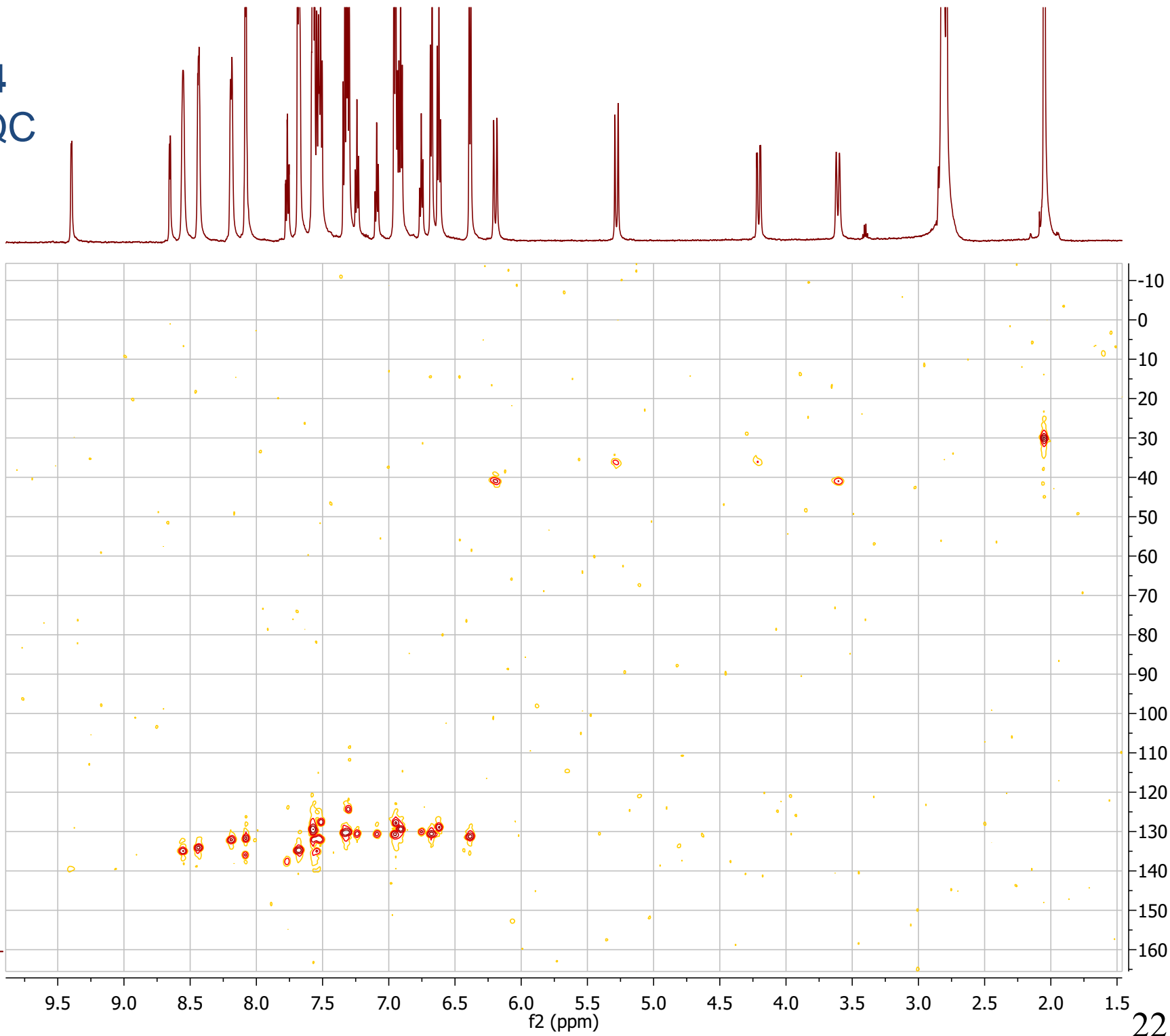


# Complex 4

## $^1\text{H}-^1\text{H}$ ROESY



Complex 4  
 $^1\text{H}-^{13}\text{C}$  HSQC



Complex 4  
 $^1\text{H}-^{13}\text{C}$  HMBC

