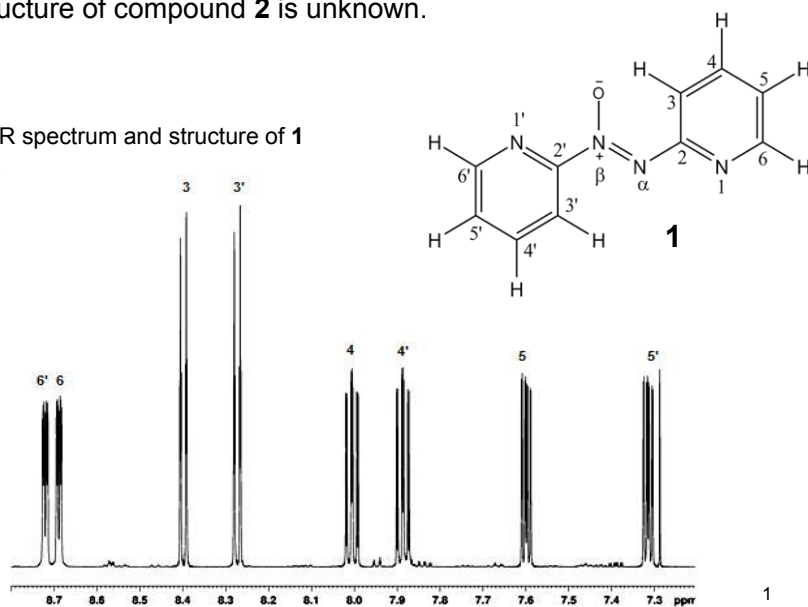
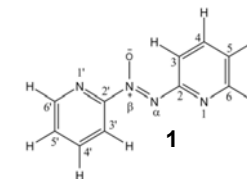


A reaction has produced two compounds, **1** and **2**,
the structure of compound **2** is unknown.

¹H NMR spectrum and structure of **1**



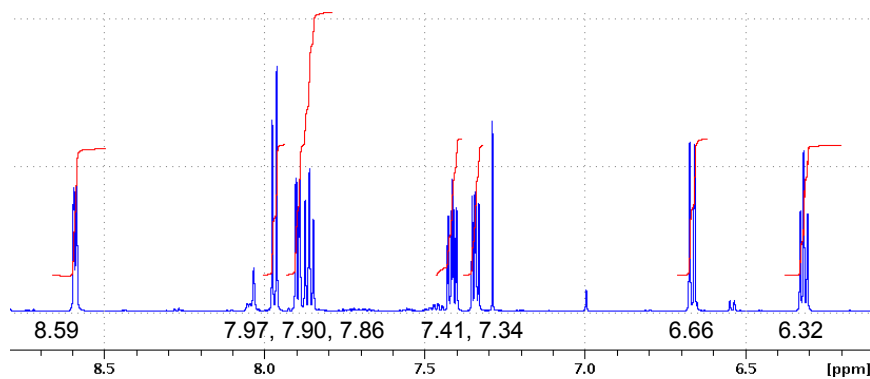
NMR data of **1**



Number	¹ H chemical shift (ppm)	Multiplicity/Coupling Constant (Hz)	¹³ C chemical shift (ppm) ^a	¹⁵ N chemical shift (ppm)
1				292
2			157.12, s	
3	8.40	dt: 8.2, 0.9, 0.9	117.67, d	
4	8.01	ddd: 8.2, 7.4, 1.9	139.21, d	
5	7.60	ddd: 7.4, 4.7, 1.0	127.24, d	
6	8.69	ddd: 4.7, 1.8, 0.9	148.68, d	
1'				304
2'			156.27, s	
3'	8.27	dt: 8.2, 1.0, 1.0	118.55, d	
4'	7.89	ddd: 8.2, 7.5, 1.9	137.86, d	
5'	7.32	ddd: 7.5, 4.7, 1.0	123.78, d	
6'	8.72	ddd: 4.7, 1.9, 0.9	149.61, d	
α				330
β				331

¹H NMR spectrum of **2**

C₁₀H₈N₂O (M_w = 172.18 g/mol)

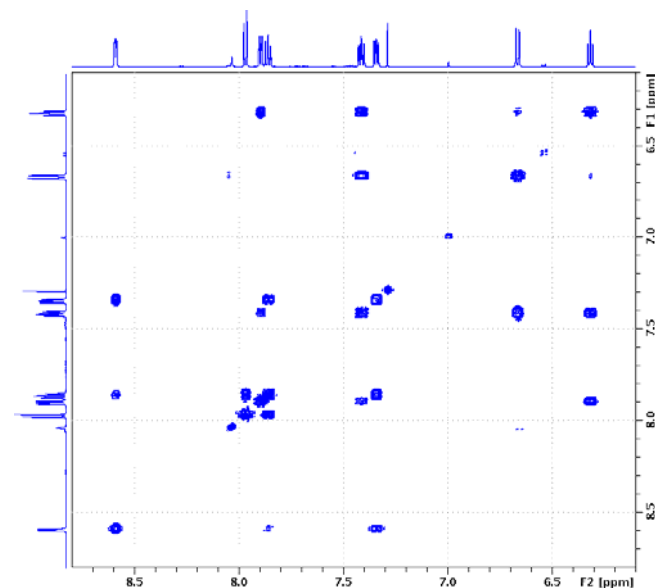


Tasks.

- Label the ¹H signals a-h starting from the most deshielded proton.

3

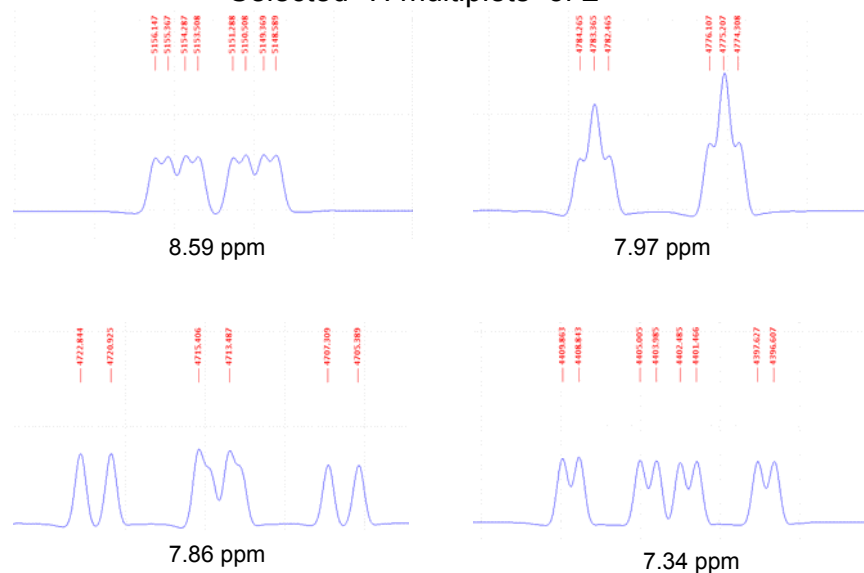
2D COSY spectrum of **2**



Analyse the COSY spectrum and identify two spin systems in compound **2**. Hint: the intensity of cross peaks increases with the size of the J coupling. Follow the strongest cross peaks.

4

Selected ¹H multiplets of 2



1. Determine the coupling constants from these multiplets and fill in the table provided in the following sheet.

5

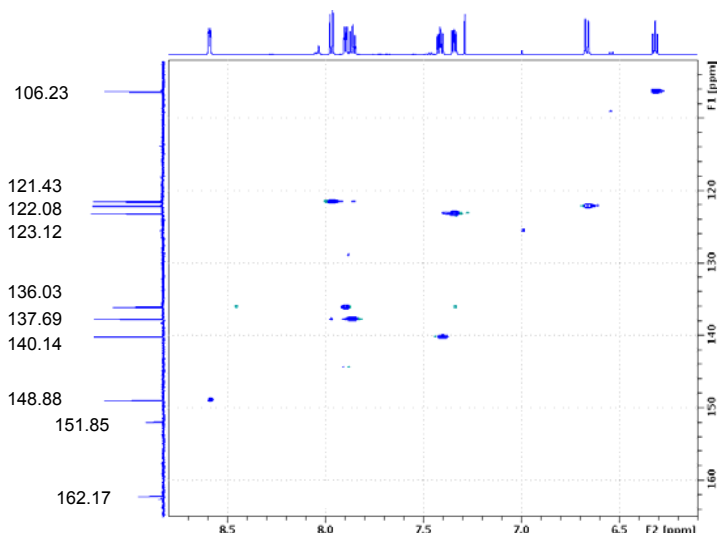
NMR data of 2

Number	¹ H chemical shift (ppm)	Multiplicity/Coupling Constant (Hz)	¹³ C chemical shift (ppm) ^a	¹⁵ N chemical shift (ppm)
a				
c	7.90	ddd: 7.1, 2.1, 0.7		
h	6.32	ddd: 6.8, 6.8, 1.3		
e	7.41	ddd: 9.2, 6.5, 2.1		
g	6.66	ddd: 9.2, 1.3, 0.7		

- Does any of the spin systems resemble the spin systems identified in compound 1?
- If yes, assign the protons of the pyridine ring.

6

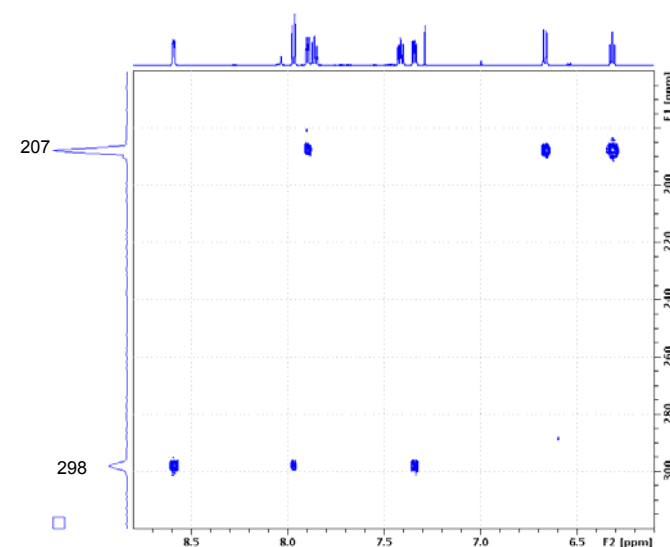
2D ¹H, ¹³C HSQC spectrum of 2



- Using Arabic numbers and starting from the most deshielded carbon, label the carbon resonances of 2.
- Assign the carbon resonances of 2 and write the carbon chemical shifts into the Table for compound 2.
- Calculate the molecular formulae of the non pyridine part of the molecule.
- Calculate the double-bond equivalent for $C_{10}H_8ON_2$: $C_aH_bO_cN_d$; $Dbe = [(2a+2) - (b-d)]/2$, for the whole molecule and also for the fragments.
- Draw possible molecular fragments for the non-pyridine part of the molecule.

7

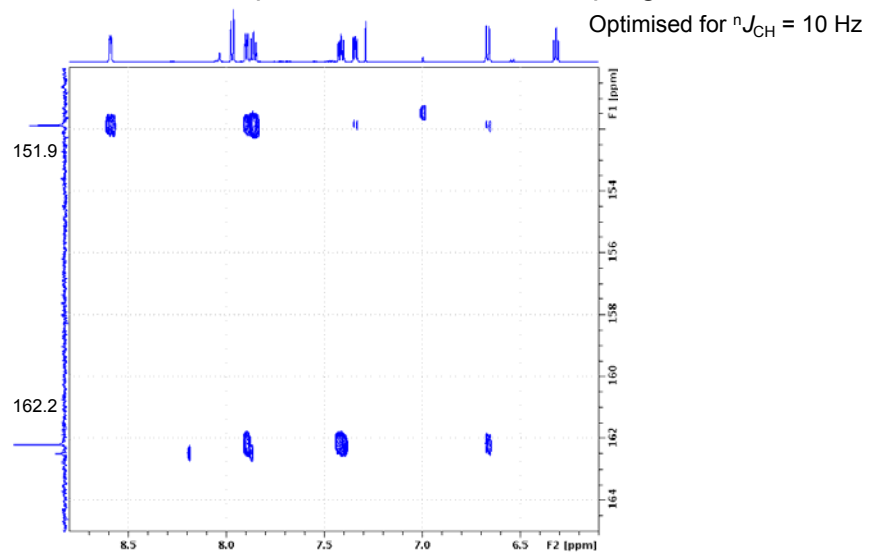
2D ¹H, ¹⁵N HMBC spectrum of 2



- Assign the cross peaks in this spectrum and in the process identify which nitrogen belongs to which ring.
- Identify the missing cross peak that you would have expected to see.

8

Partial 2D ^1H , ^{13}C HMBC spectra of **2** (quaternary carbons only)
optimised for different couplings



1. Assign cross peaks in these HMBC spectrum and highlight cross peaks that confirm the linkage between the two rings. Assign the two quaternary carbons of **2**. Suggest a structure of compound **2**.

9