Interpretation of NMR-Spectra

Strategy and Conclusions

Mass: 298 g/mol

Sum formula: Calculate the double-bond equivalents from the sum formula $C_{18}H_{22}N_2O_2$.

$$DBE = \frac{(2*20+2)-24}{2} = 9$$

$^{13}C\{^1H\}$: Number the ^{13}C signals sequentially, starting with No. 1 for the most high field signal.

The signals of C(9) and C(10) are twice as high as the others.
$\rightarrow 2 * {}^{13}C$
<u>Attention:</u> Relaxation

16 Carbons: does not agree with the sum formula $C_{18}H_{22}N_2O_2$. That means 2 ^{13}C-signals correspond for 2 carbons each.

HSQC : Assign the ^1H signals to the corresponding ^{13}C signals.

^1H : Copy the numeration of the ^1H signals in the HSQC to the ^1H spectra.

Use the integrals to define the signals or some integral regions.

1	CH_3	
2	CH_2	
3	CH_2	
4	vermutlich CH_2 +Lsgm.	
5	CH_2	
6	CH_2	
7	CH	
8	CH	
9	2* CH	
10	2* CH	∓ - Bereich
11	CH	
12	C	
13	CH	
14	C	
15	C O=C-N	
16	C O=C-O	

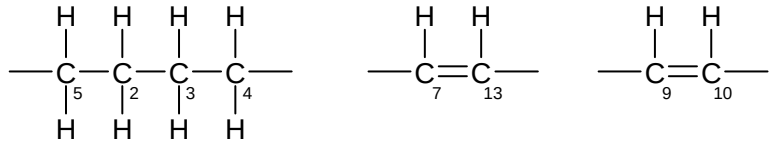
Sum: $C_{18}H_{21}$
Δ Mass: 61 g/mol
No C

2x O=C-O: 64
2x O=C-N: 60
O=C-N and O=C-O: 62

Solvent signals (methanol-d4: δ=3.31ppm und water: δ=4.8ppm)
In methanol no exchangeable protons are visible.

Summenformel : The difference to the sum formula is one Proton, an –OH or –NH.

DQF-COSY : Label the ^{1}H signals on the F1- and F2-axis.
Put several structure fragments together.



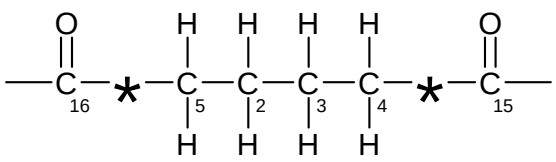
HMBC : Label the ^{1}H signals on the F2-axis and the ^{13}C signals on the F1-axis.
We recommend to create a list of the C→H and H→C correlations.

^{13}C	^{1}H			
1	(6)			
2	5	4	3	
3	5	4	2	
4	2	3		
5	2	3		
6	(8)	5	1	
7	13			
8	6	1		
9	9	13	11	
10	10			
11	9			
12	10	7		
13	9			
14	6	1		
15	13	7	4	
16	(8)	6	5	(1)

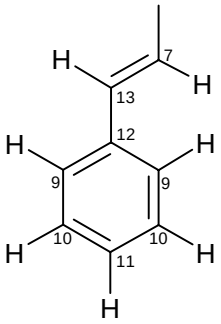
^{1}H	^{13}C			
1	6	8	14	(16)
2	3	4	5	
3	2	4	5	
4	2	3	15	
5	2	3	6	16
6	(1)	8	14	16
7	12	15		
8	6	16		
9	9	11	13	
10	10	12		
11	9			
13	7	9	15	

Put several structure fragments together:

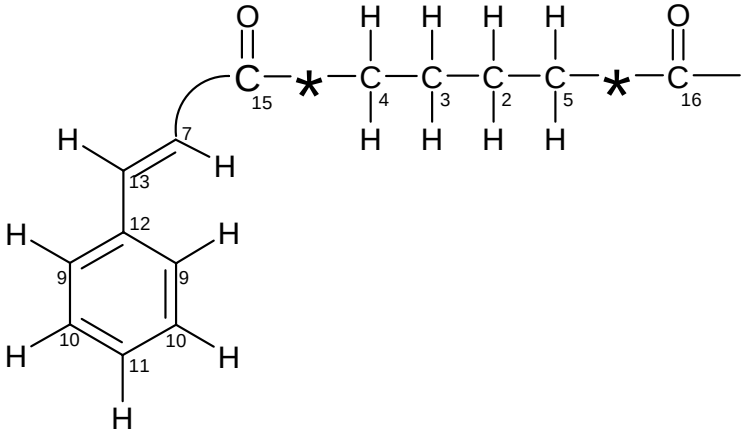
H(5) = C(16) H(4) = C(15)
H(2) ≠ C(16) H(3) ≠ C(15)



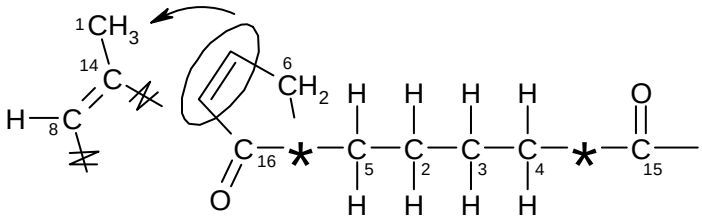
In the aromatic region:
H(9) = C(9) H(10) = C(10)
H(9) = C(11), C(13) H(10) = C(12)



C(15) = H(13), H(7), H(4)



H(5) = C(6), C(16), C(2), C(3)
H(6) = C(8), C(16), C(14),
H(1) = C(6), C(8), C(24), C(16)

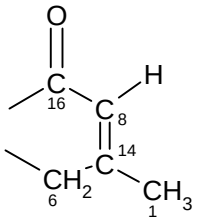


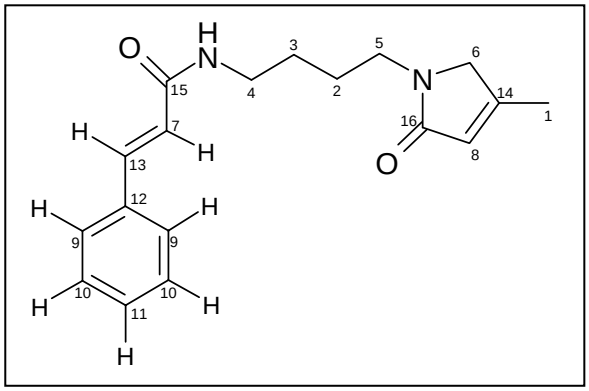
In the peak list of the protonen spectrum determine the coupling $^{3}J_{H7,H13}$=15.7Hz .
→ trans

1 CH_3
~~2 CH_2~~
~~3 CH_2~~
~~4 CH_2~~
~~5 CH_2~~
6 CH_2
~~7 CH~~
8 CH
~~9 2* CH~~
~~10 2* CH~~
~~11 CH~~
~~12 C~~
~~13 C~~
14 C
~~15 C~~
~~16 C~~
N
N
~~O~~
~~O~~

It is not defined yet where the double bond is located in the ring. The $J_{H6,C14}$ and $J_{H6,C8}$ can be ^{2}J- or ^{3}J-couplings.

INADEQUATE: With help of the INADEQUATE-Experiment determine the location of the double bond and verify the other fragments.





If the chemical shift of two neighboured carbons are very similar, the cross peaks can not be distinguished.