NMR Spectroscopy of Organic Compounds

Lesson 5:
2D experiments - continuation

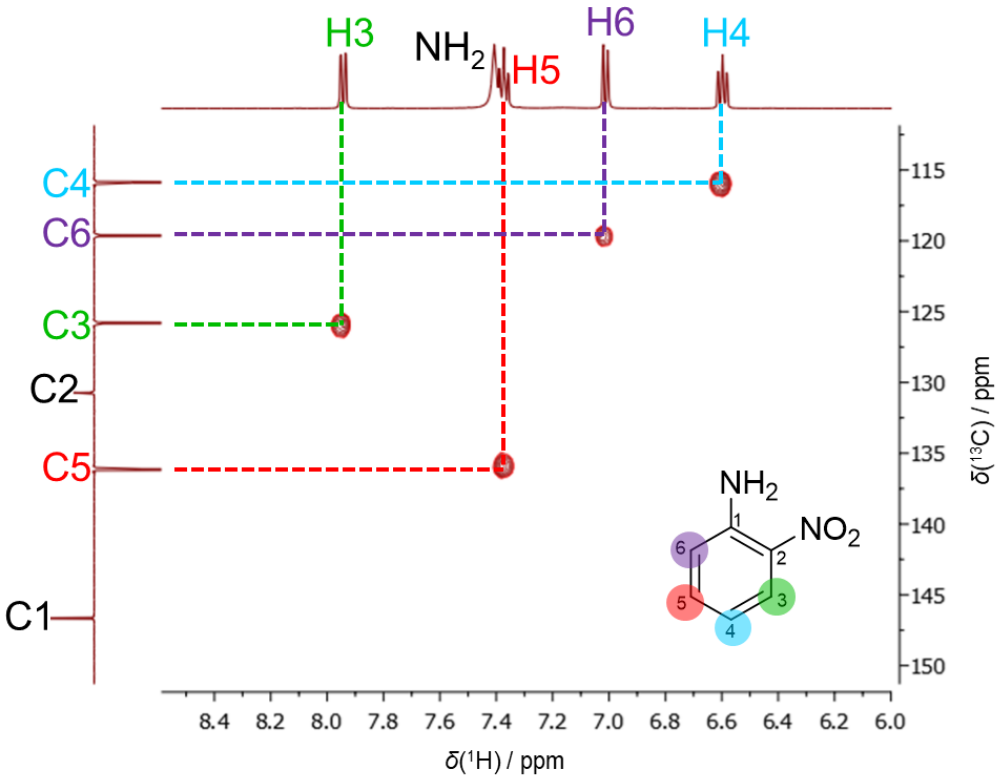
Martin Dračínský

Overview

- 2D NMR experiments - continuation
- Gradients, DOSY
- MR Imaging

2D NMR experiments

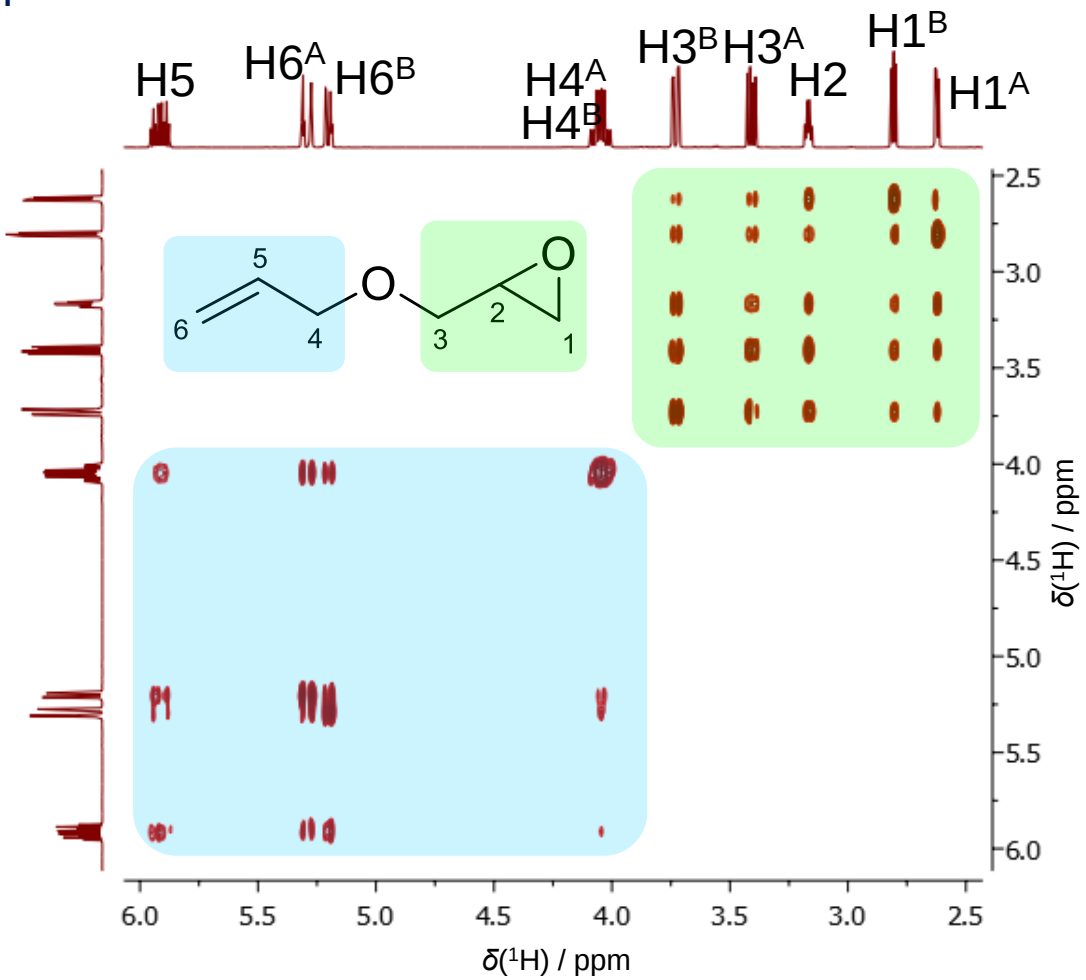
- Direct detection / indirect detection
- Resolution in direct/indirect dimension
- Inverse detection, inverse experiments, inverse probes



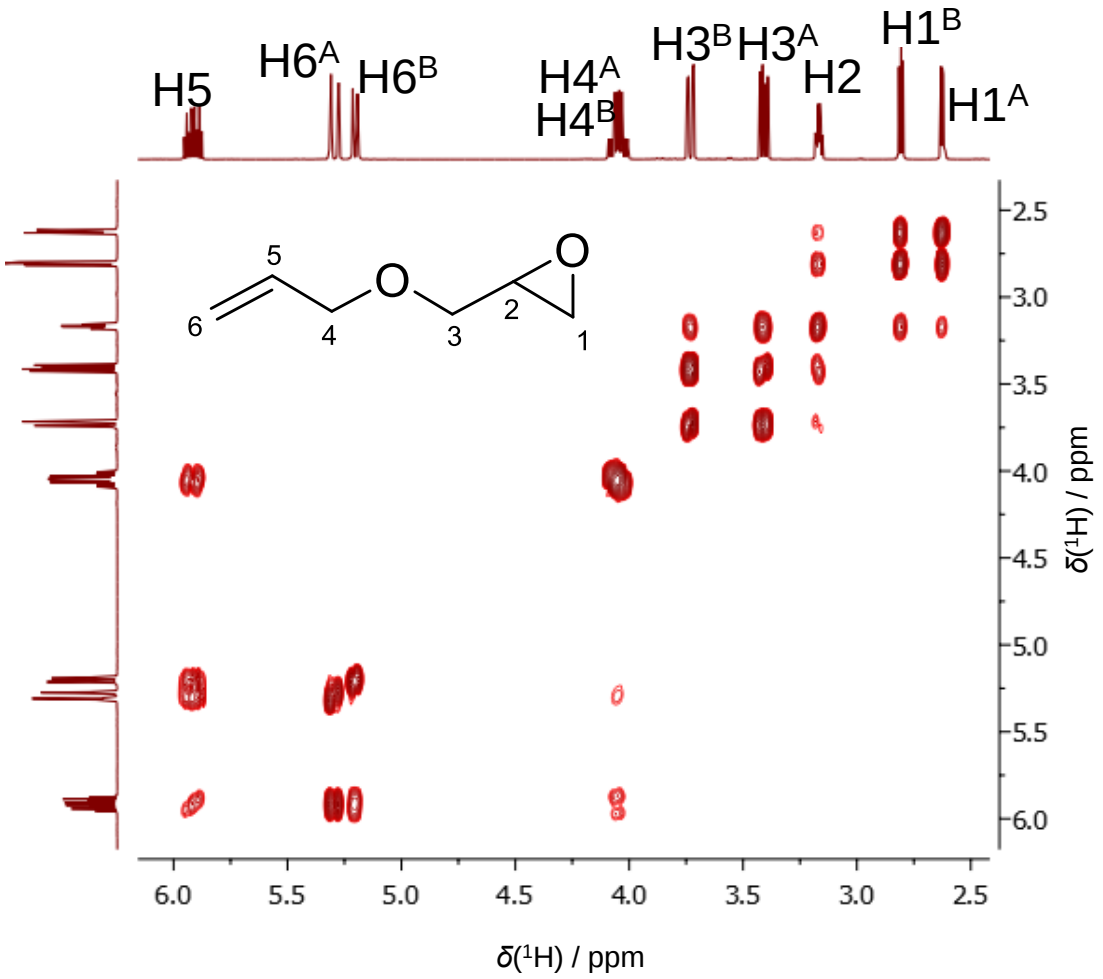
Total Correlation SpectroscopY (TOCSY)

Correlation between all nuclei

in one spin system

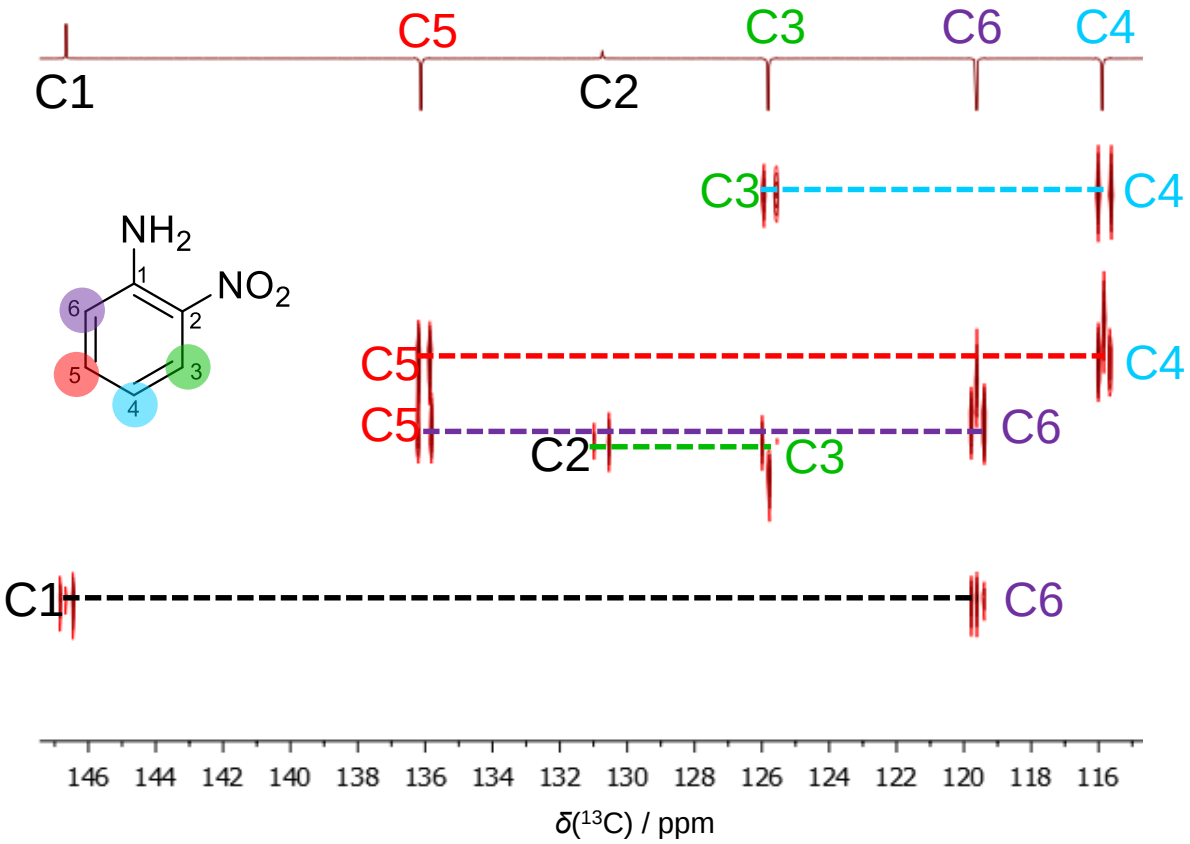


COrrelation SpectroscopY (COSY)

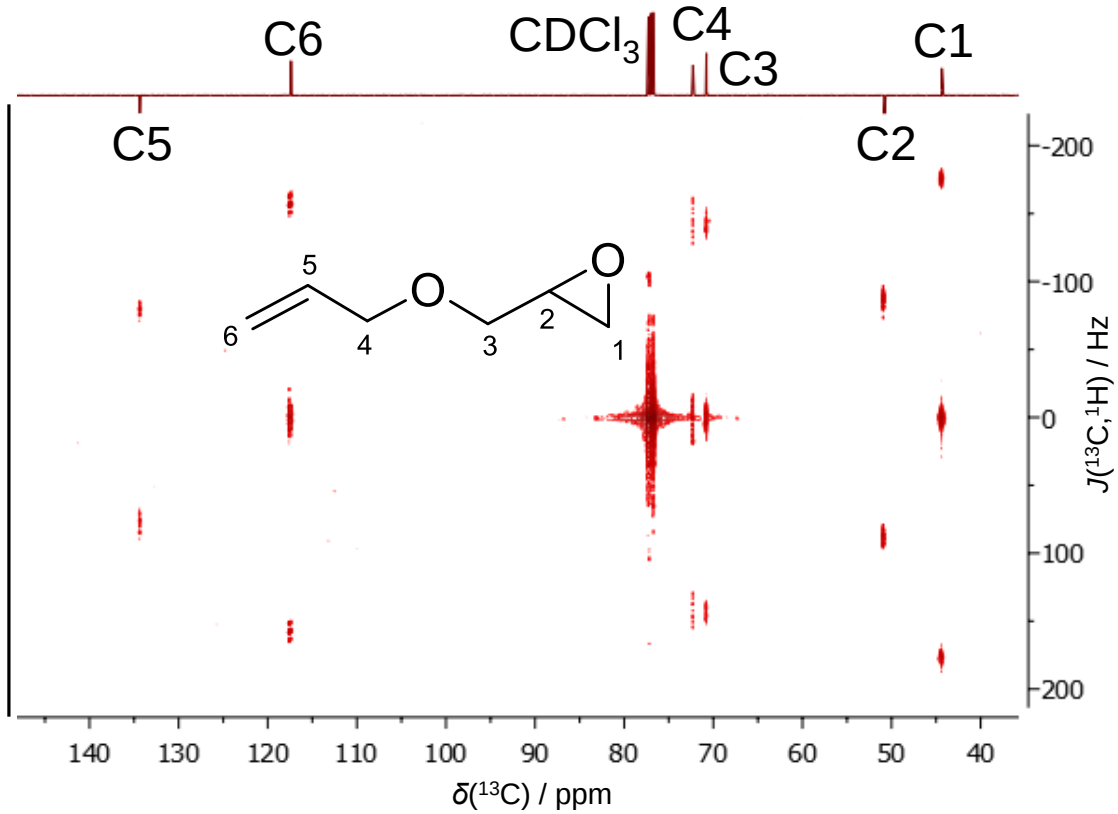


INADEQUATE

C–C correlations

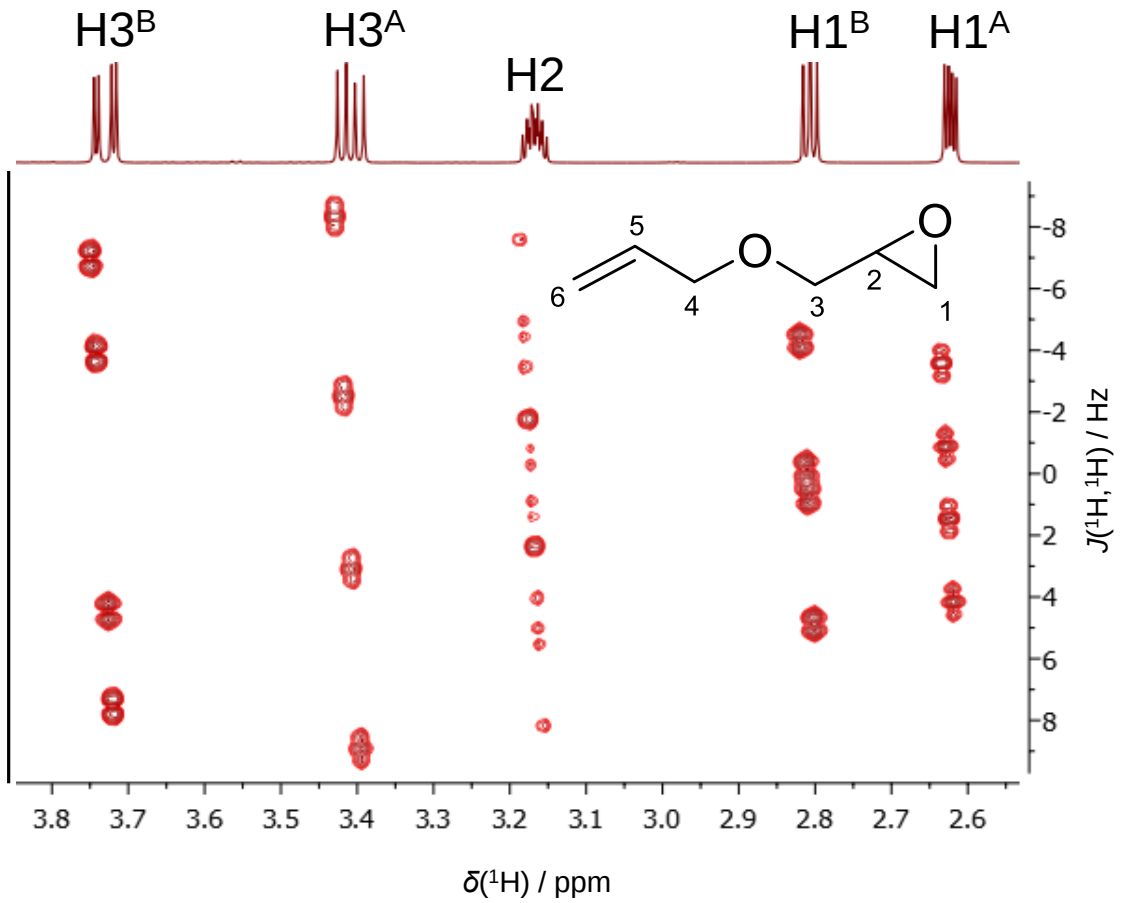


Heteronuclear *J*-resolved experiment

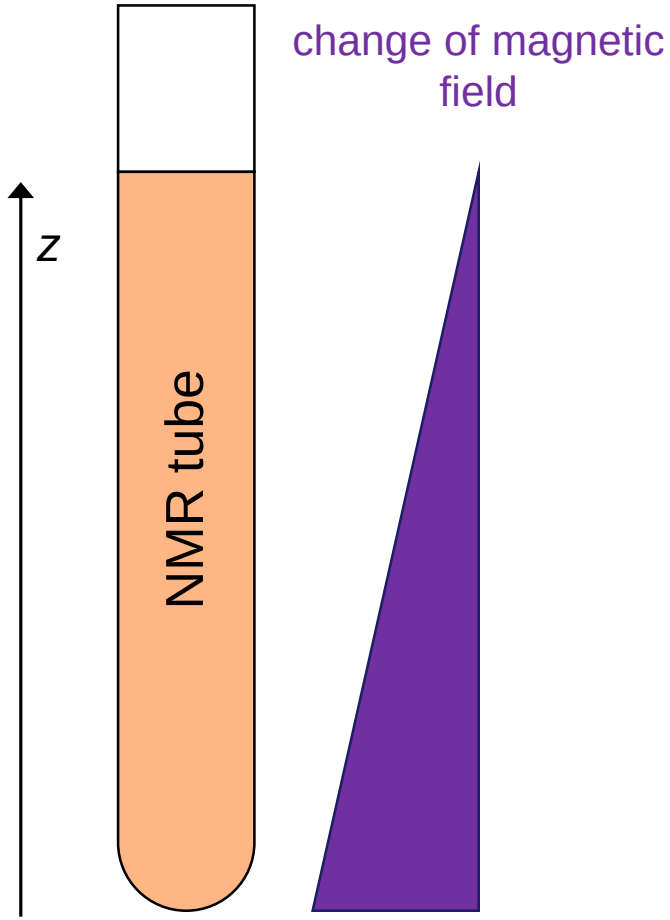


Homonuclear *J*-resolved experiment

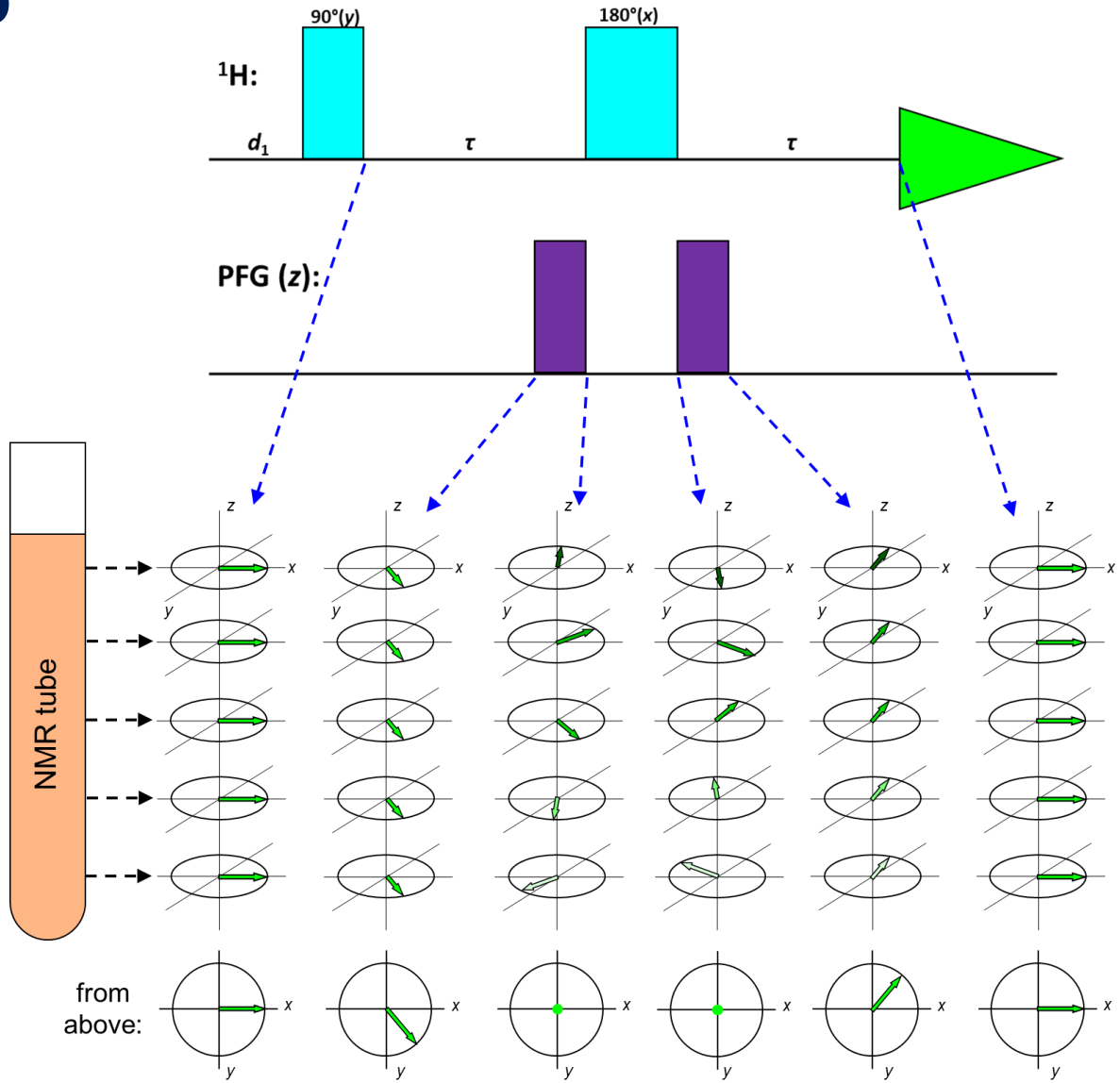
Resolution can be better than in 1D proton experiment



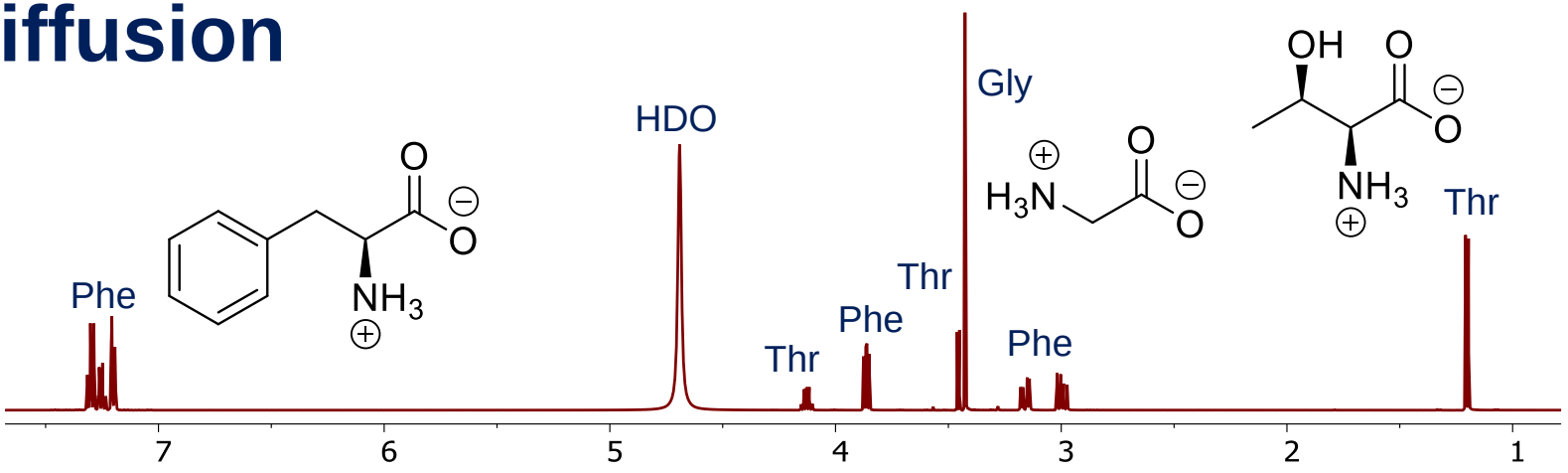
Gradients



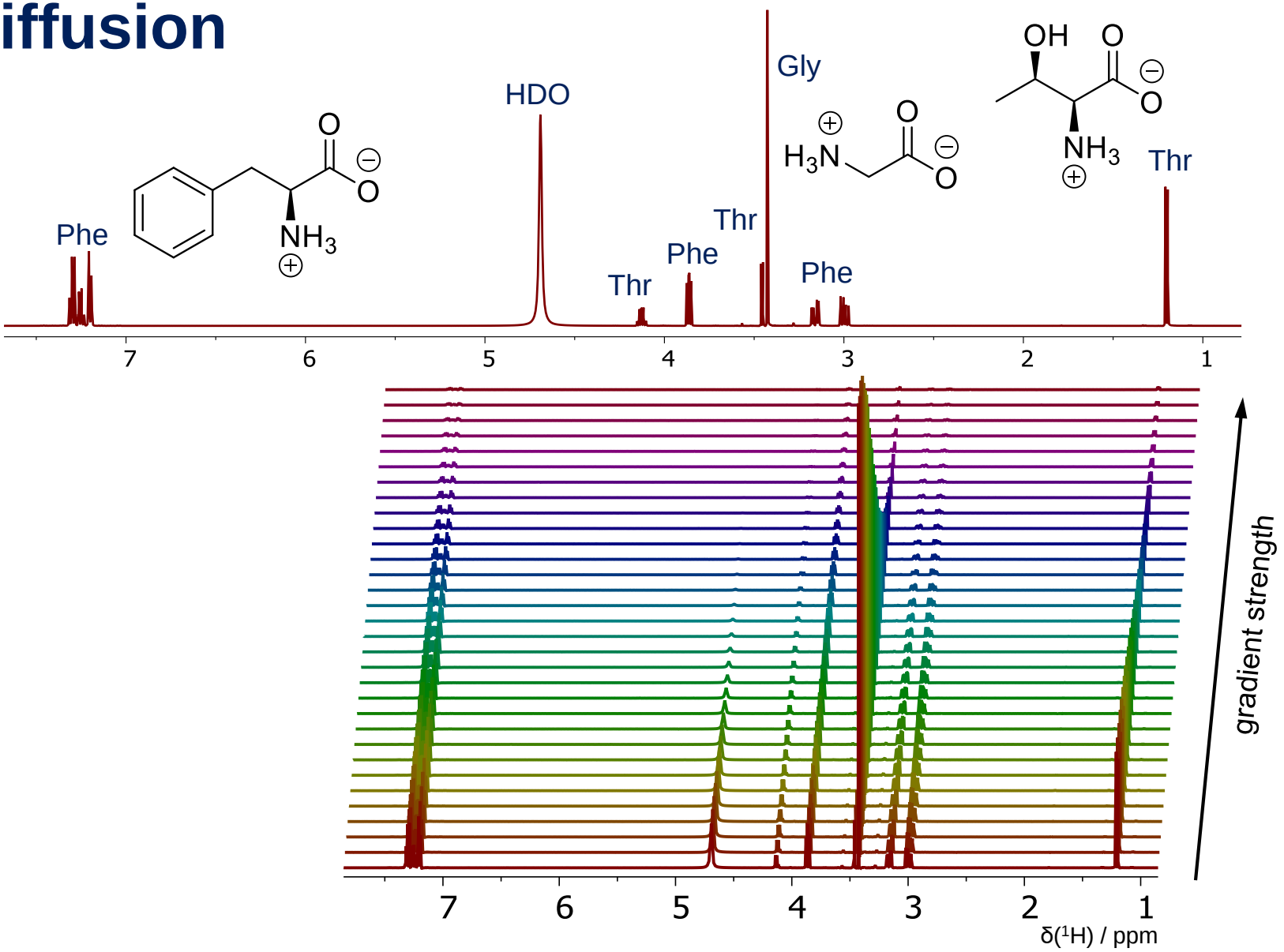
Gradient echo



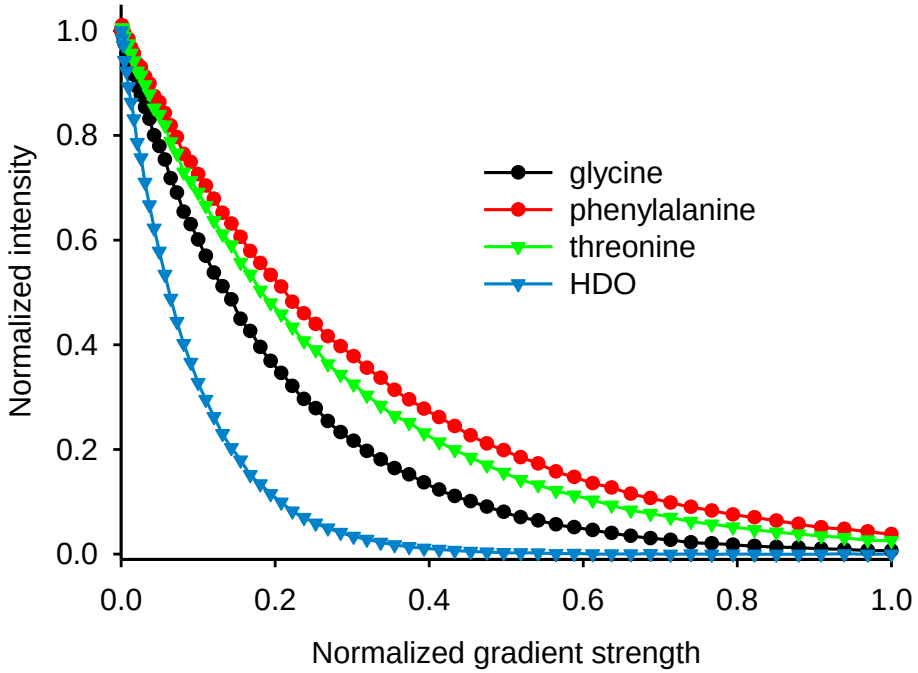
Diffusion



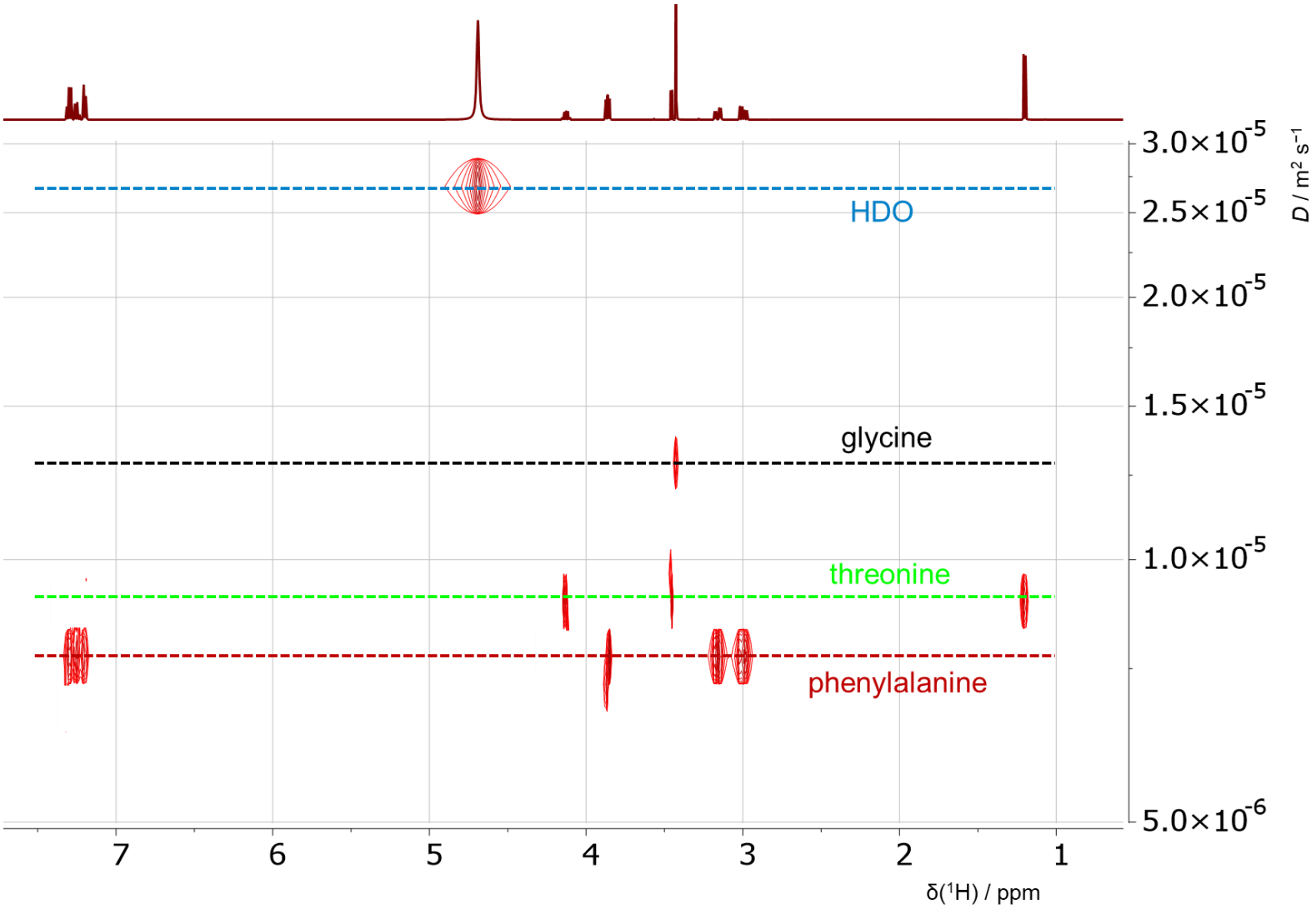
Diffusion


Phe
HDO
Thr
Phe
Thr
Gly
Phe
Thr
7
6
5
4
3
2
1
gradient strength
δ(¹H) / ppm

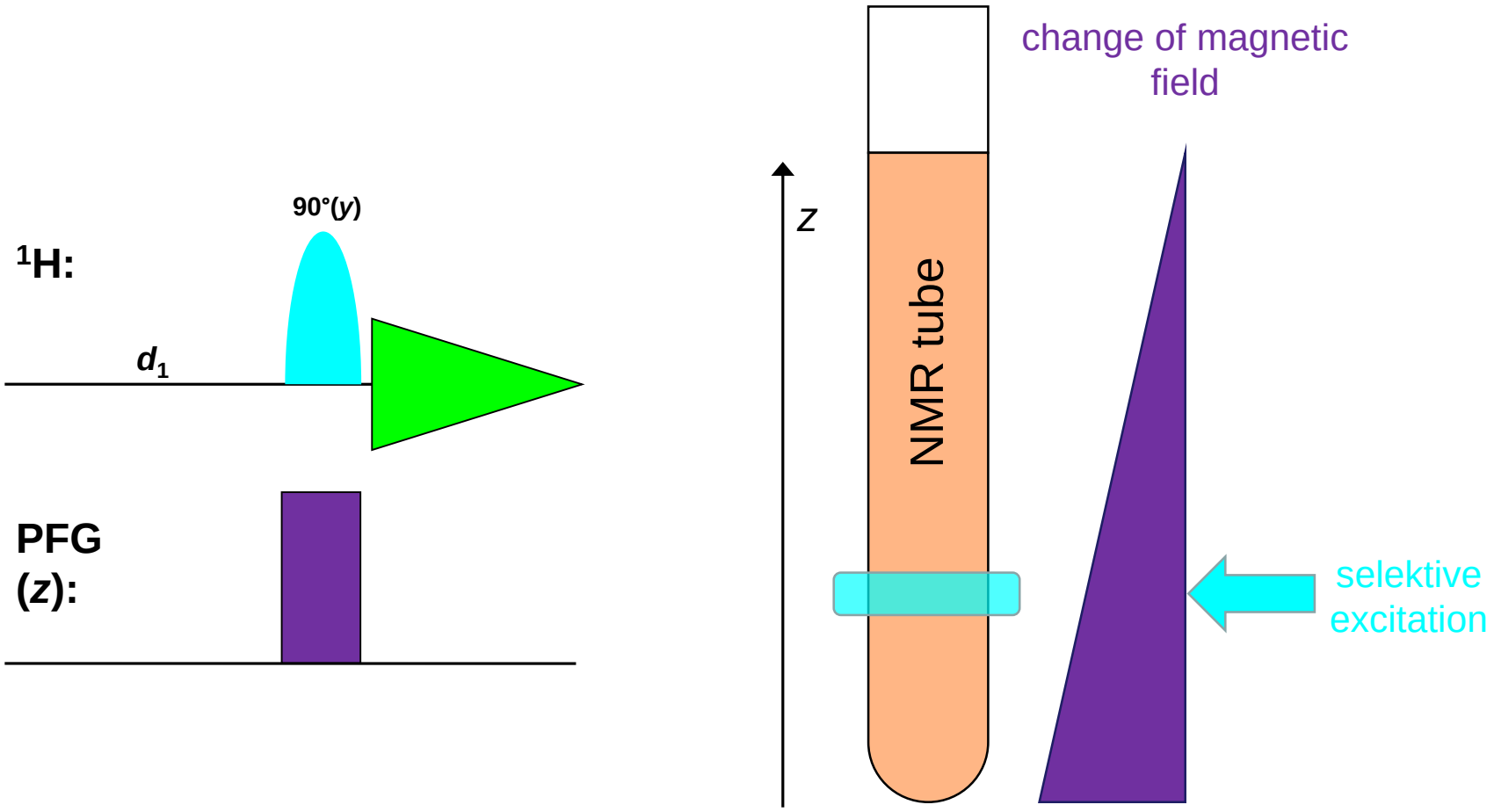
Diffusion


1.0
0.8
0.6
0.4
0.2
0.0
Normalized intensity
0.0
0.2
0.4
0.6
0.8
1.0
Normalized gradient strength
glycine
phenylalanine
threonine
HDO

Diffusion Ordered SpectroscopY (DOSY)



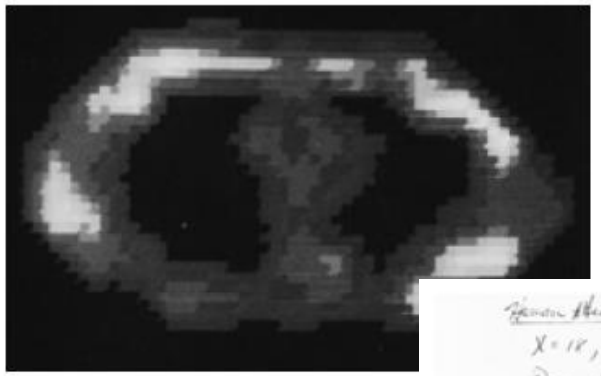
MR imaging


90°(y)
1H:
d1
PFG
(z):
z
NMR tube
change of magnetic
field
selektive
excitation

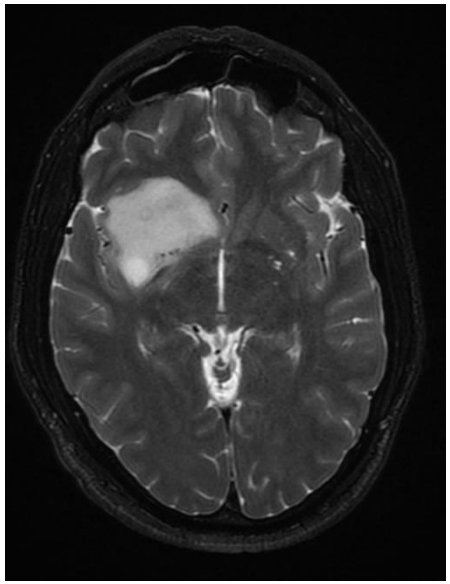
MR imaging

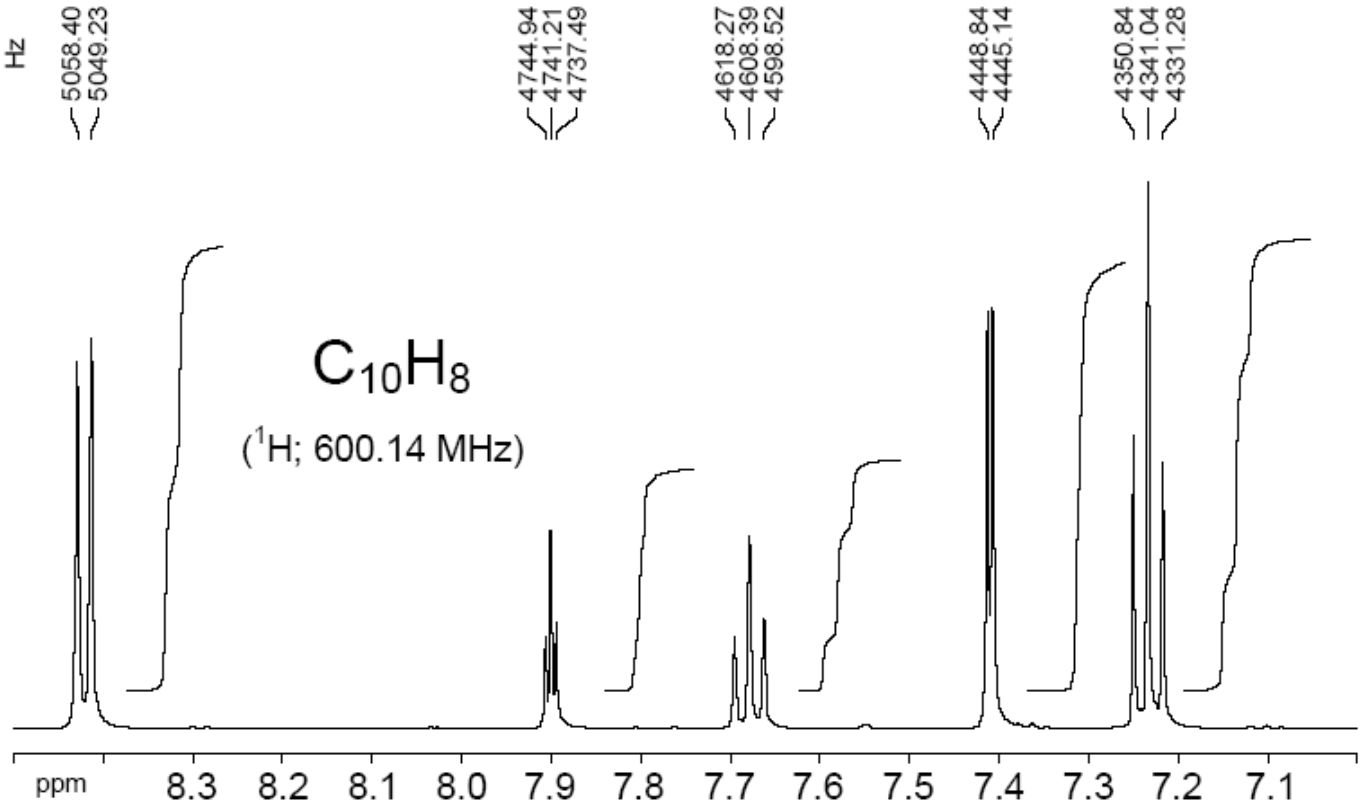
Peter Mansfield Paul Lauterbur

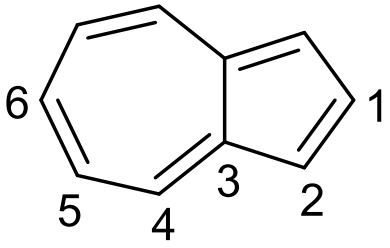
2003 Nobel Prize

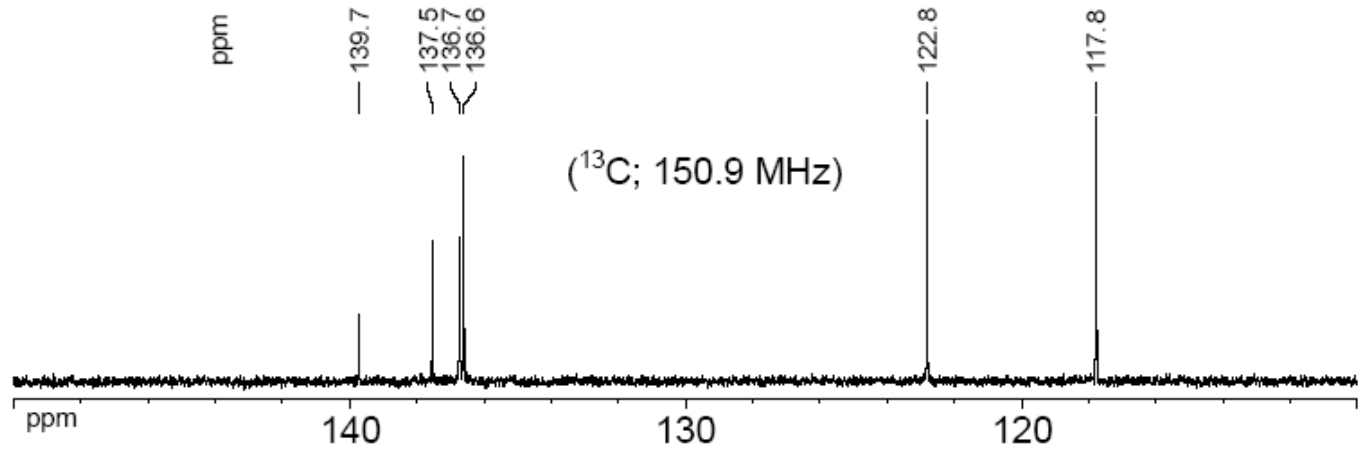


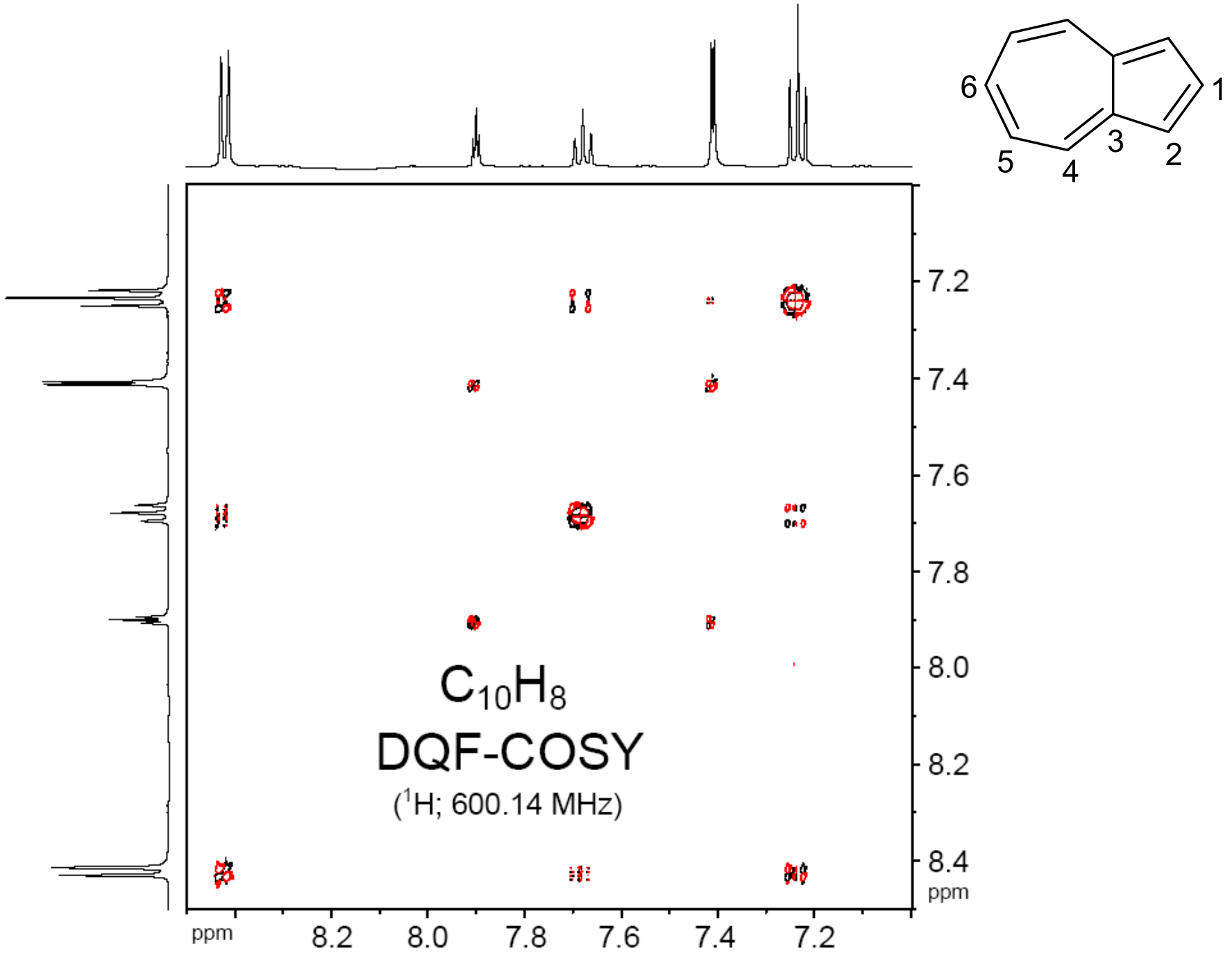
MR imaging

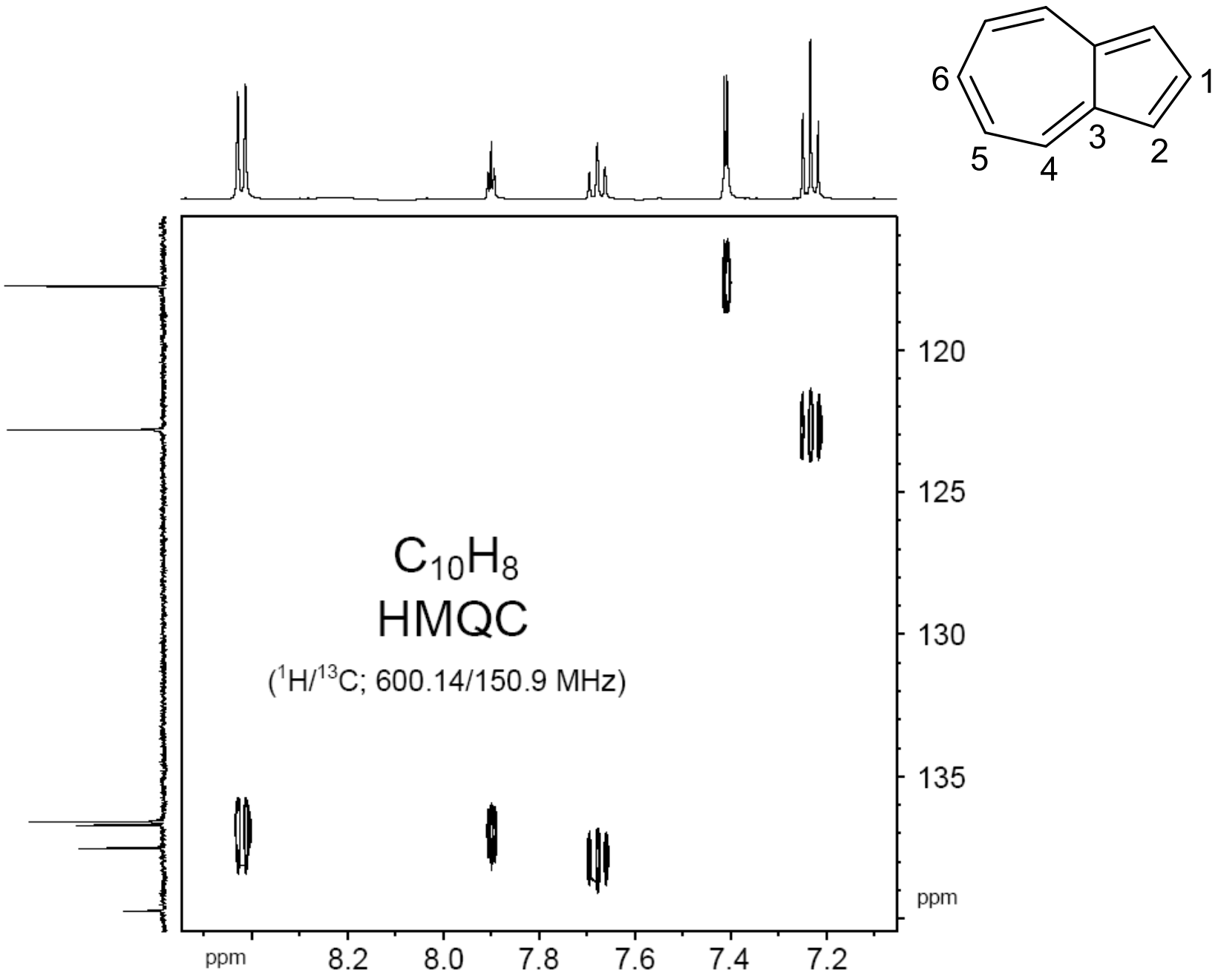


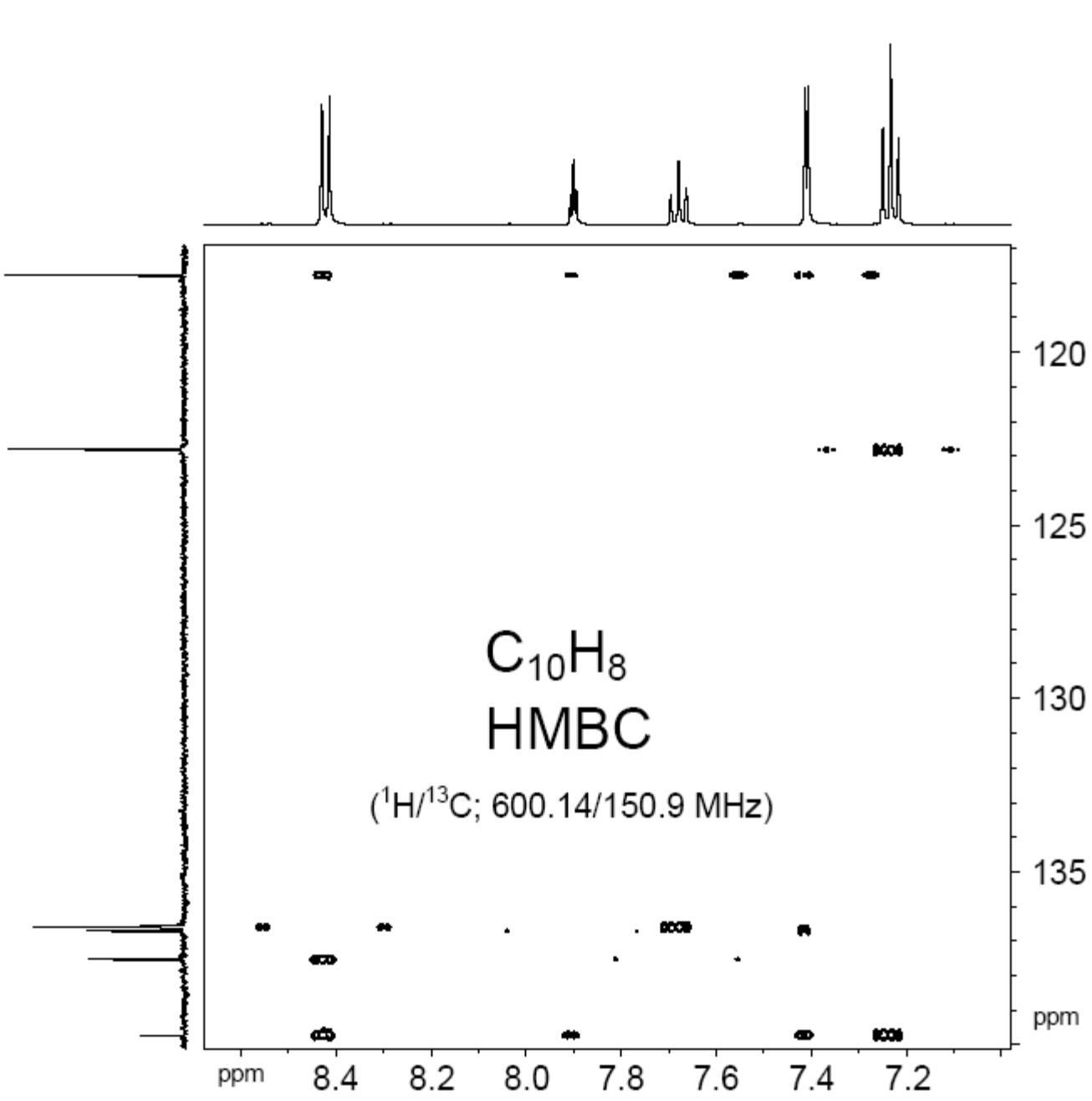

Hz
5058.40
5049.23
4744.94
4741.21
4737.49
4618.27
4608.39
4598.52
4448.84
4445.14
4350.84
4341.04
4331.28
$C_{10}H_8$
(^{1}H; 600.14 MHz)
ppm
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1

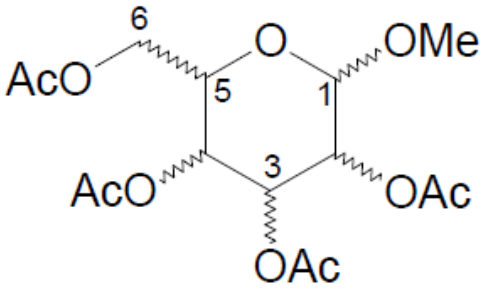

6
1
5
4
3
2

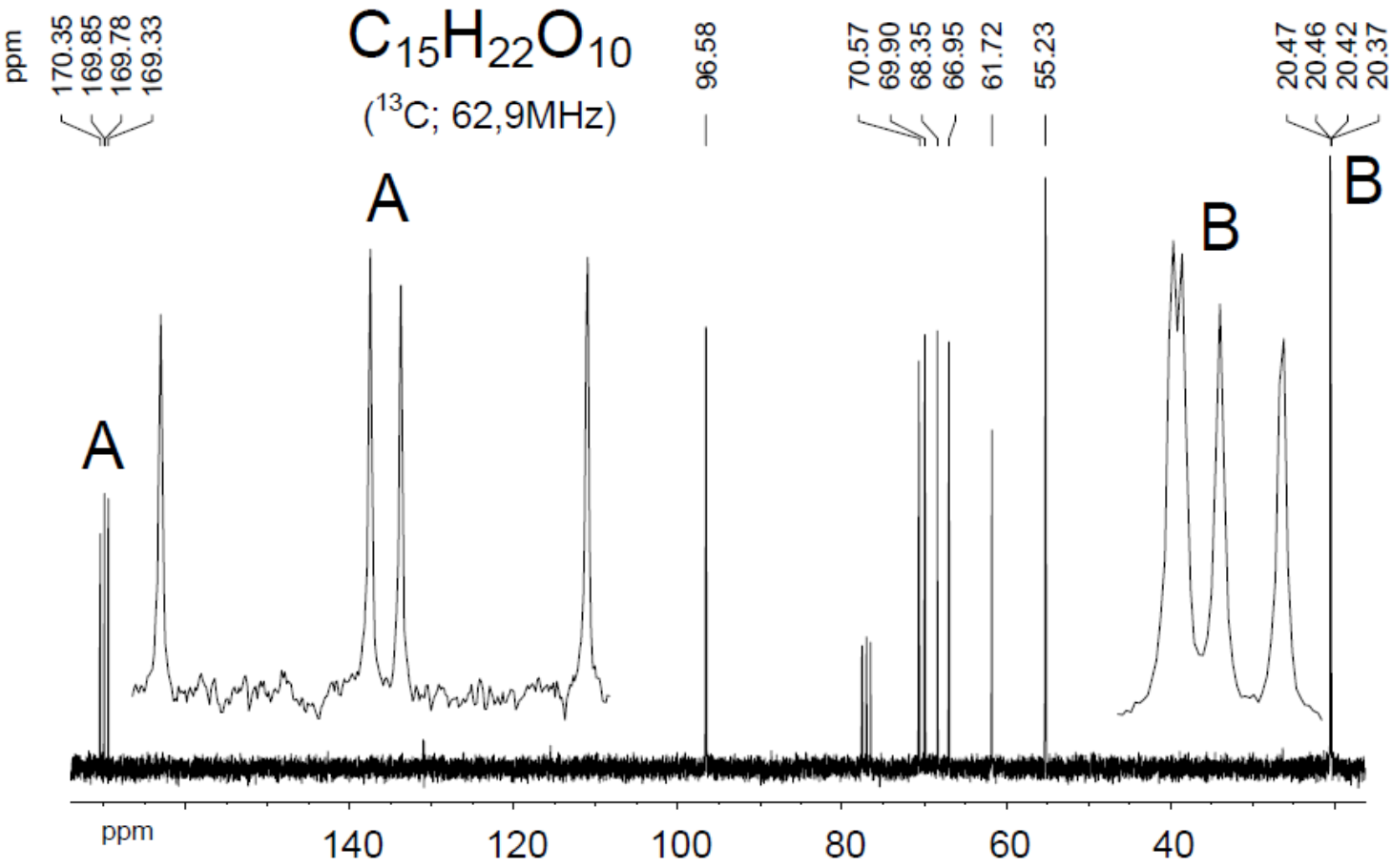

ppm
139.7
137.5
136.7
136.6
122.8
117.8
(^{13}C; 150.9 MHz)
ppm
140
130
120

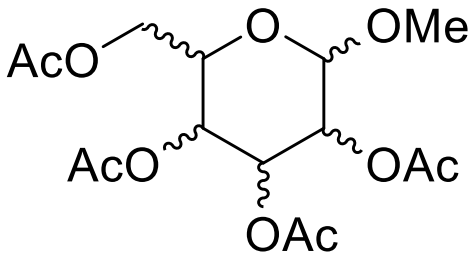

6
1
5
4
3
2
$C_{10}H_8$
DQF-COSY
(^{1}H; 600.14 MHz)
7.2
7.4
7.6
7.8
8.0
8.2
8.4
ppm
ppm
8.2
8.0
7.8
7.6
7.4
7.2


6
1
5
4
3
2
$C_{10}H_8$
HMQC
($^1H/^{13}C$; 600.14/150.9 MHz)
120
125
130
135
ppm
ppm
8.2
8.0
7.8
7.6
7.4
7.2


6
1
5
4
3
2
$C_{10}H_8$
HMBC
($^1H/^{13}C$; 600.14/150.9 MHz)
120
125
130
135
ppm
ppm
8.4
8.2
8.0
7.8
7.6
7.4
7.2

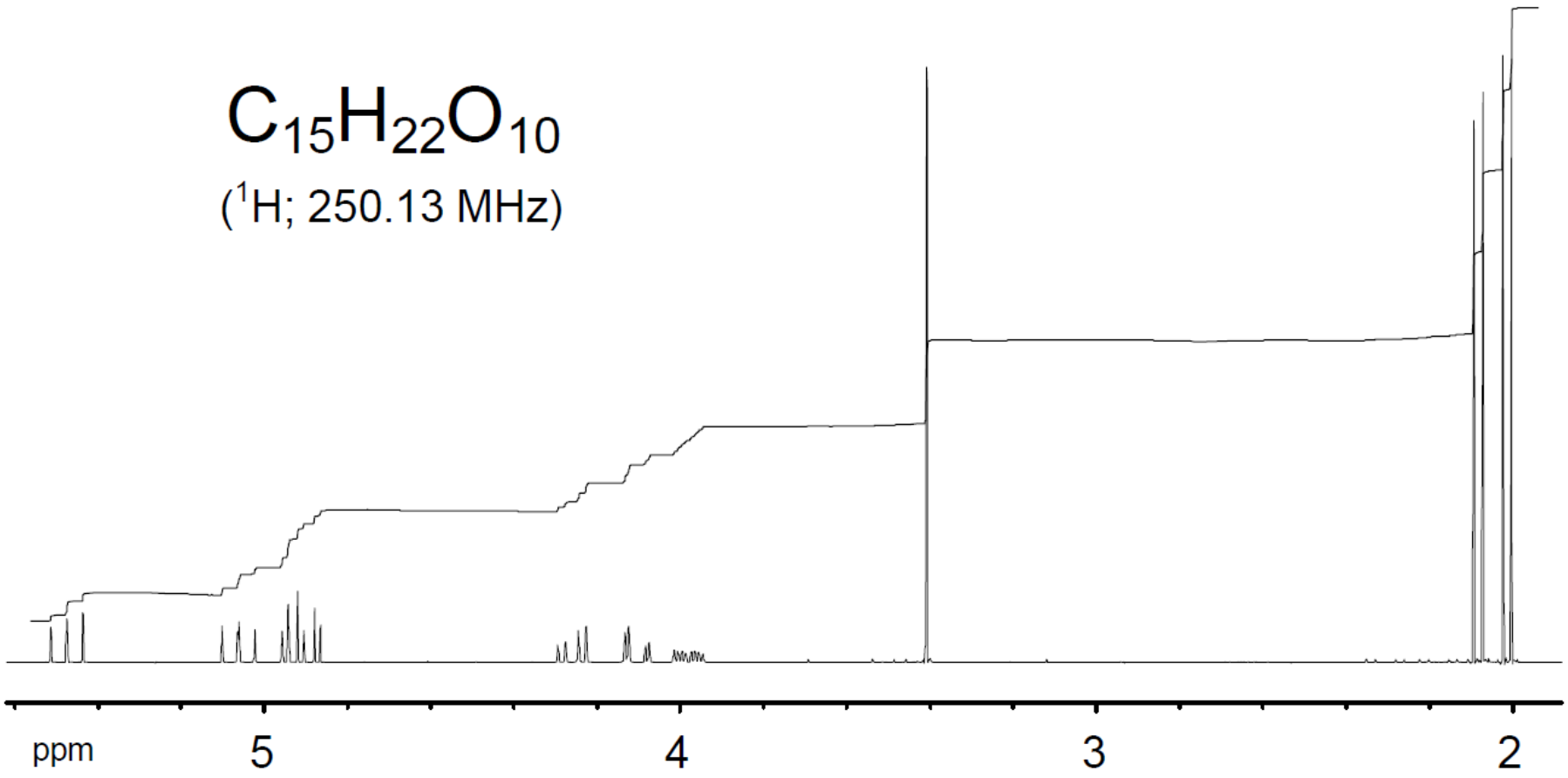

6
O
OMe
AcO
5
1
3
AcO
OAc
OAc

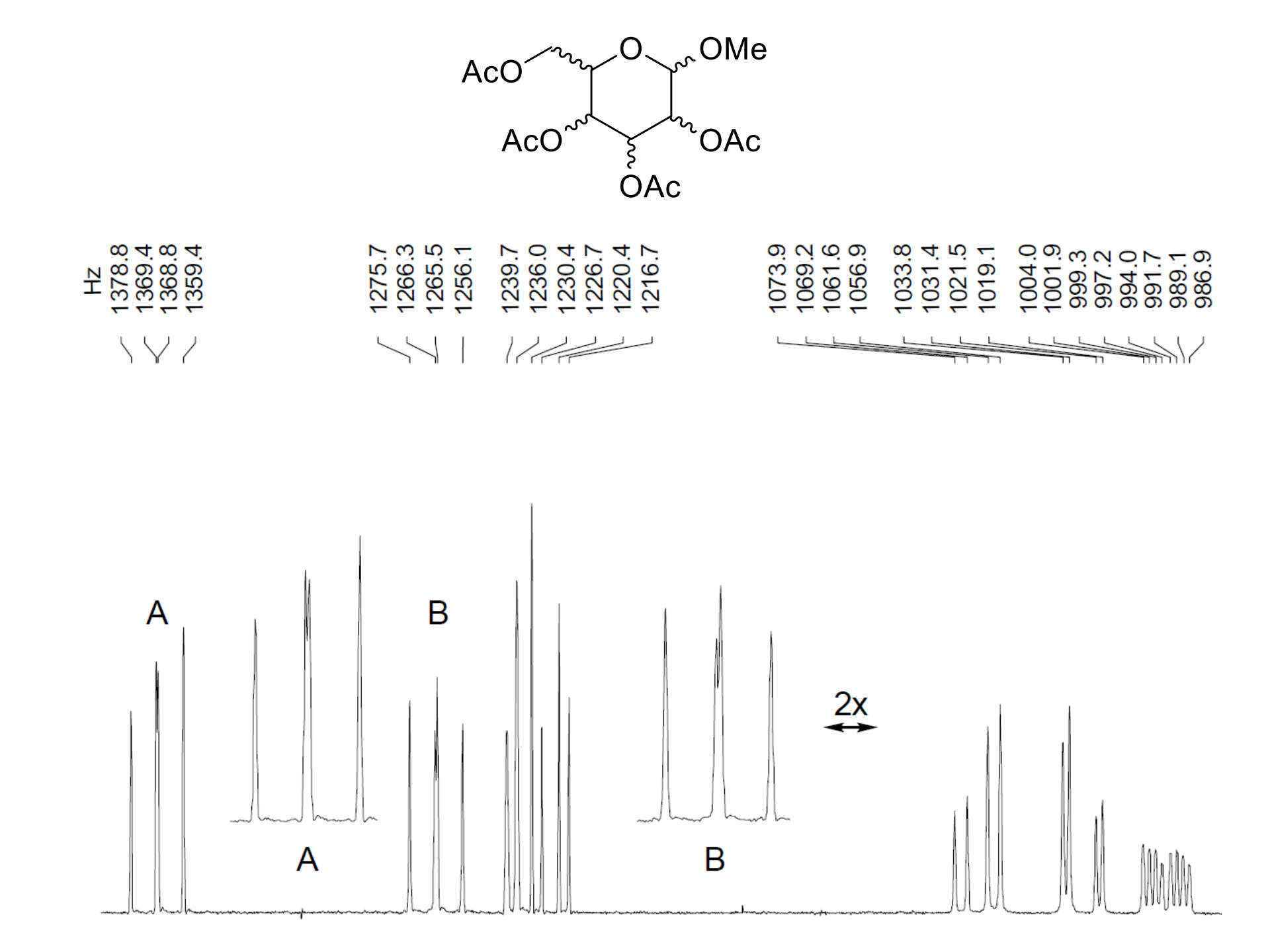

ppm
170.35
169.85
169.78
169.33
$C_{15}H_{22}O_{10}$
(^{13}C; 62,9MHz)
96.58
70.57
69.90
68.35
66.95
61.72
55.23
20.47
20.46
20.42
20.37
A
A
B
B
ppm
140
120
100
80
60
40

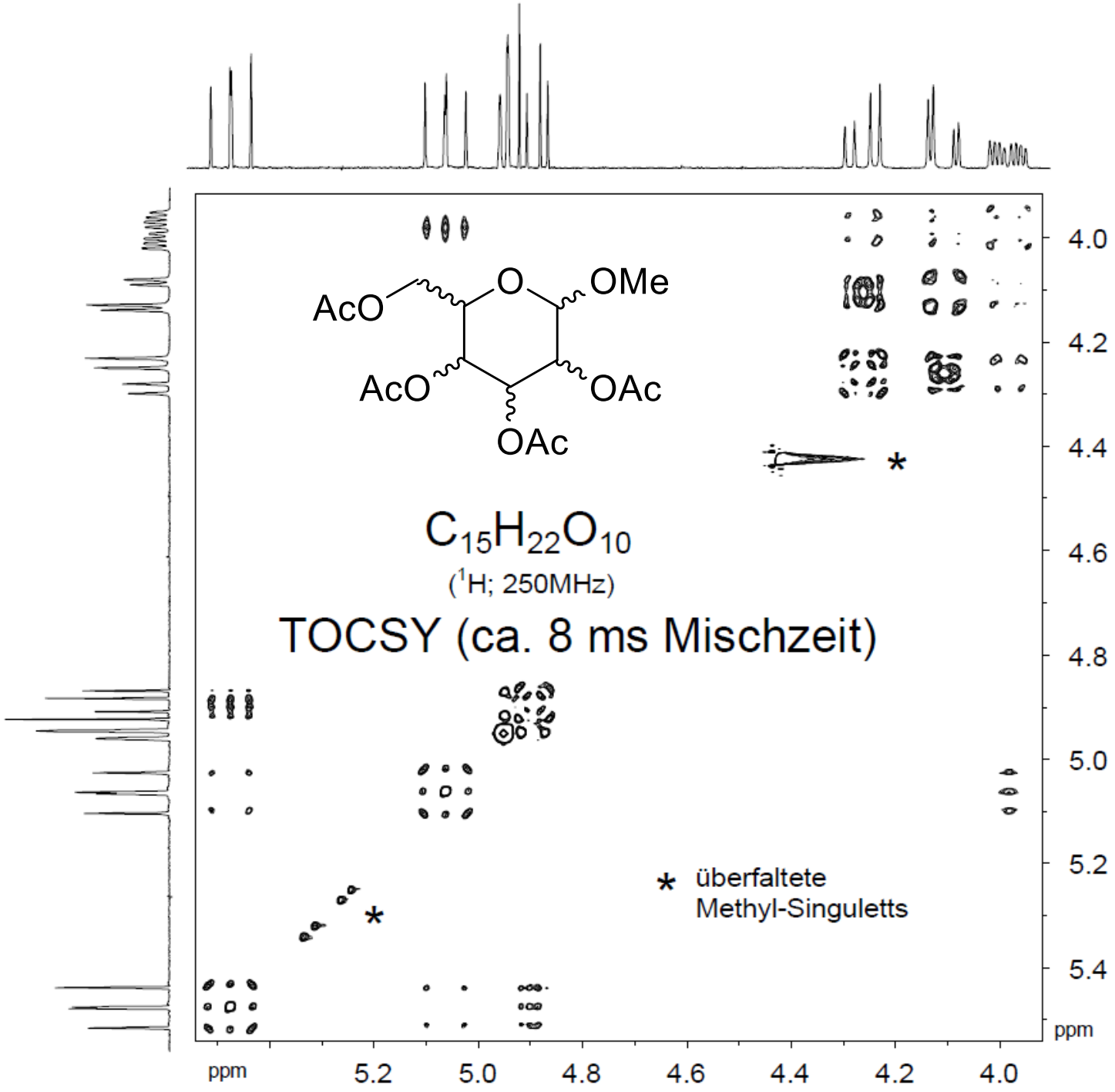


$C_{15}H_{22}O_{10}$

(^{1}H; 250.13 MHz)




AcO
O
OMe
AcO
OAc
OAc
Hz
1378.8
1369.4
1368.8
1359.4
1275.7
1266.3
1265.5
1256.1
1239.7
1236.0
1230.4
1226.7
1220.4
1216.7
1073.9
1069.2
1061.6
1056.9
1033.8
1031.4
1021.5
1019.1
1004.0
1001.9
999.3
997.2
994.0
991.7
989.1
986.9
A
B
A
B
2x


AcO
O
OMe
AcO
OAc
OAc
$C_{15}H_{22}O_{10}$
(^{1}H; 250MHz)
TOCSY (ca. 8 ms Mischzeit)
* überfaltete Methyl-Singuletts
ppm
5.2
5.0
4.8
4.6
4.4
4.2
4.0
ppm