Spinsolve Benchtop NMR -

High-resolution NMR spectroscopy on your lab bench

Dr. Klas Meyer
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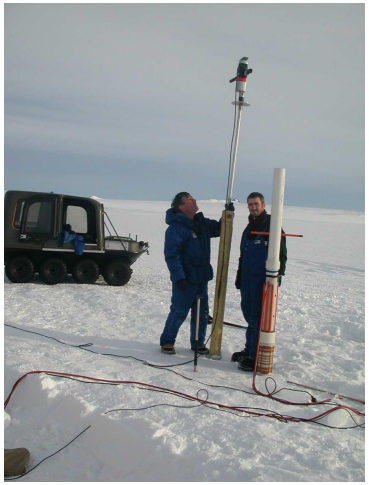
Spinsolve™
The fastest compact NMR spectrometer

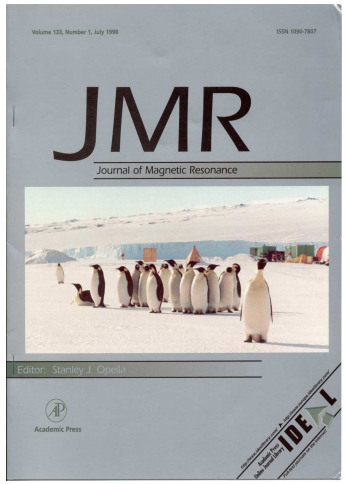
Magritek

Magritek was founded in 2004 to develop mobile and benchtop NMR and MRI instruments to enable new applications in Industry, Research and Education.

The original IP was developed during two decades of research by teams at RWTH Aachen in Germany and Massey and Victoria Universities in New Zealand

Prof Bernhard Blümich, Ampere Prize Mobile NMR, Germany





Prof Paul Callaghan, Günther Laukien Prize, NMR in Antarctica, New Zealand

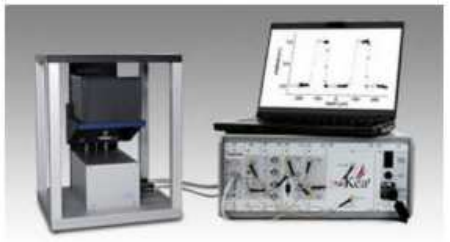
Magritek

Magritek today has manufacturing and research facilities in Germany and New Zealand, sales and support offices in the USA and UK, and a network of distributors and agents around the world

NMR Spectroscopy

MRI

TD-NMR T1, T2, Diffusion



Why Benchtop NMR?

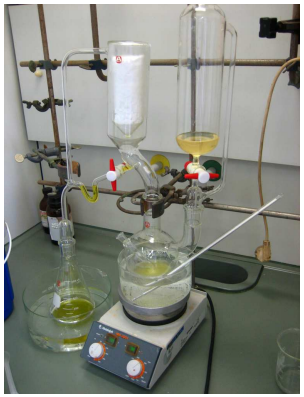
High-field NMR systems

- **Expensive** (capital, cryogen fills, operation, requires special facilities)
- **Complicated** (limited to experts)
- **Fragile** (environment)

Take the sample to the spectrometer

Benchtop NMR technology

- **Affordable** (cryogen-free, no special requirements, minimum running costs)
- **Easy to use** (more people can access the technology)
- **Robust** (operates in wide range of environments)



Take the spectrometer to the sample

Spinsolve Benchtop NMR

Spinsolve™ 43 Spinsolve™ 60 Spinsolve™ 80 NEW

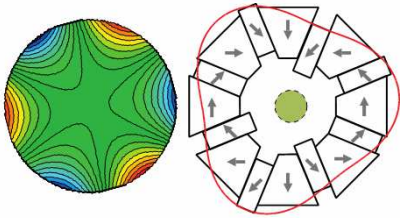
43, 60 and 80 MHz Permanent Magnets
with ^{1}H, ^{19}F
and
^{13}C, ^{31}P, ^{15}N, ^{29}S, ^{11}B, ^{2}H Capabilities
(one of those / X nuclei)

[1] Each Spinsolve System features patented magnet technology.

The following patents apply: US20100013473A1, US8148988, EP2144076A1, EP2144076B1

Spinsolve System Description

1 Tesla Halbach, SmCo permanent magnet
43 MHz proton, 11 MHz carbon, 5 mm sample
Weight 55 kg, Footprint 58 cm x 43 cm

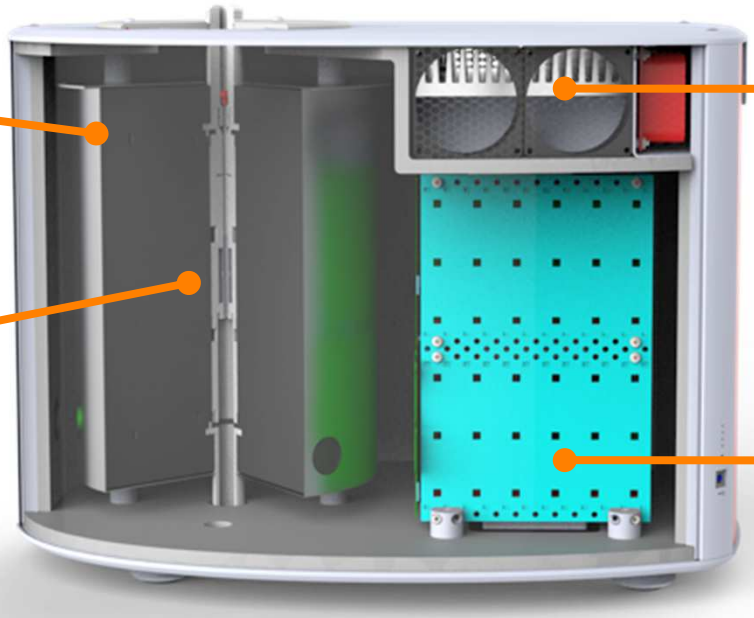


Magnet and environmental stabilisation

Dual channel RF probe

External Lock
(no deuterated solvents)

Sample bore open from the top to bottom



240 V or 110 V power

Temperature Stabilisation

Dual channel Spectrometer

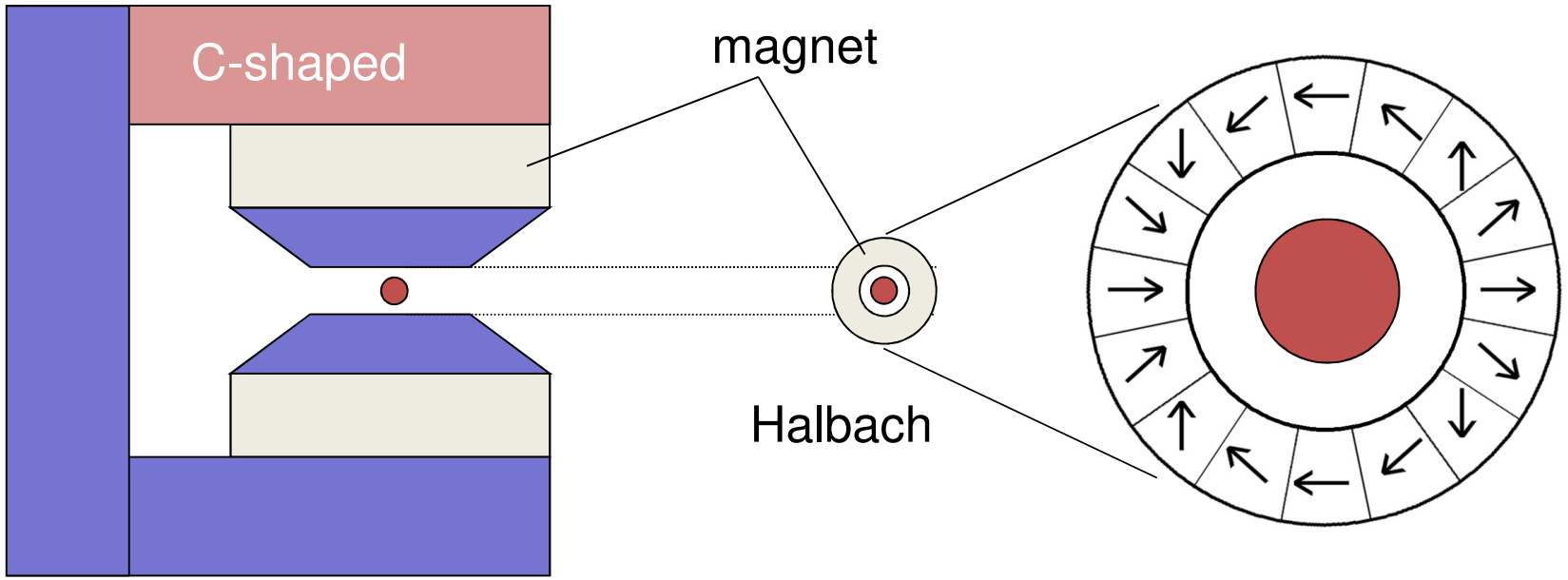
Lock system

Integrated magnet temperature control

USB connection

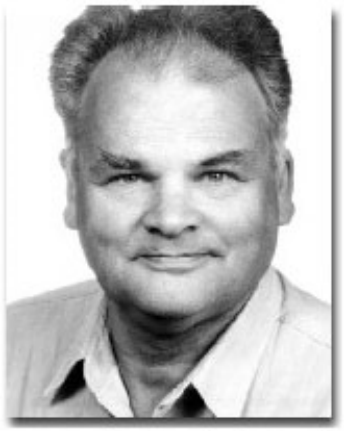
Very similar situation for the 60/80 MHz Spinsolve Systems (60/73 kg)

Halbach Magnet vs. C-Shaped Magnet



$$B_0(\vec{r}) = K \ln(r_{out} / r_{in})$$

K. Halbach, Nucl. Instr. Meth. 169 (**1980**)



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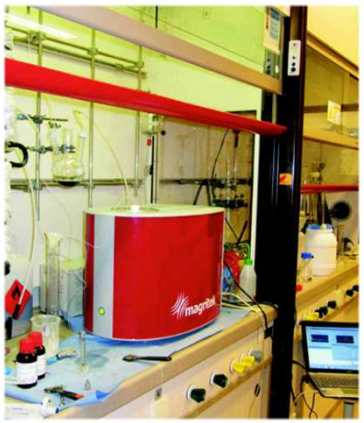
Applications of Benchtop NMR

Applications:

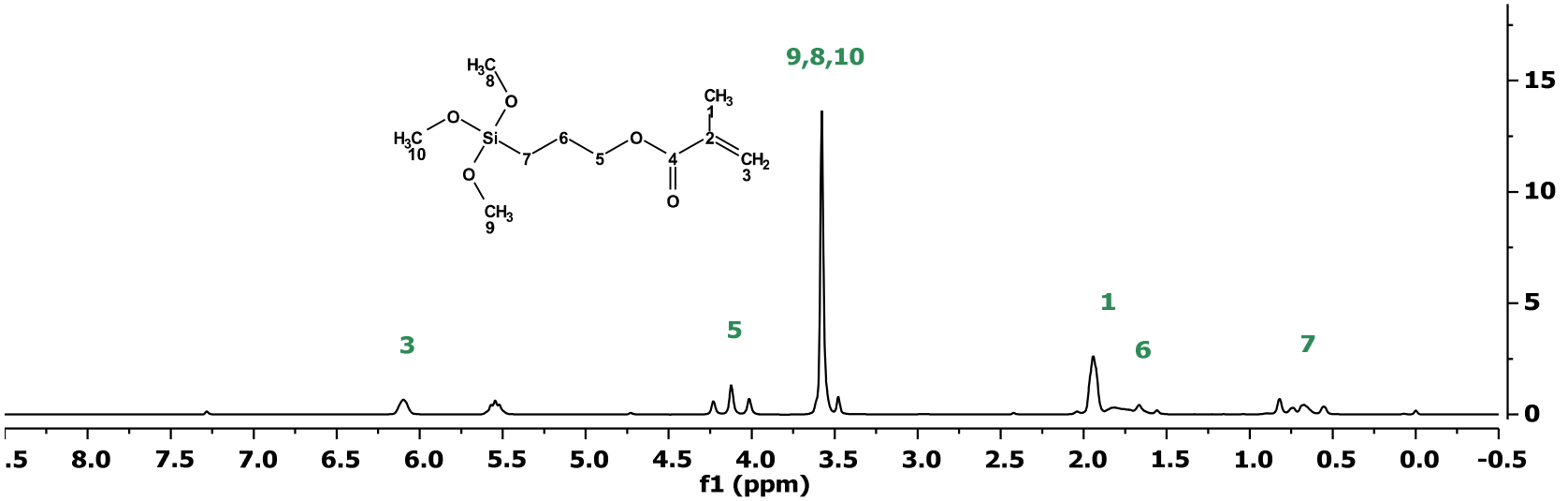
- Education
- Organic Synthesis
- Process Control
- QA/QC
- Chemical Analysis
- Quantification (qNMR)
- Reaction Monitoring



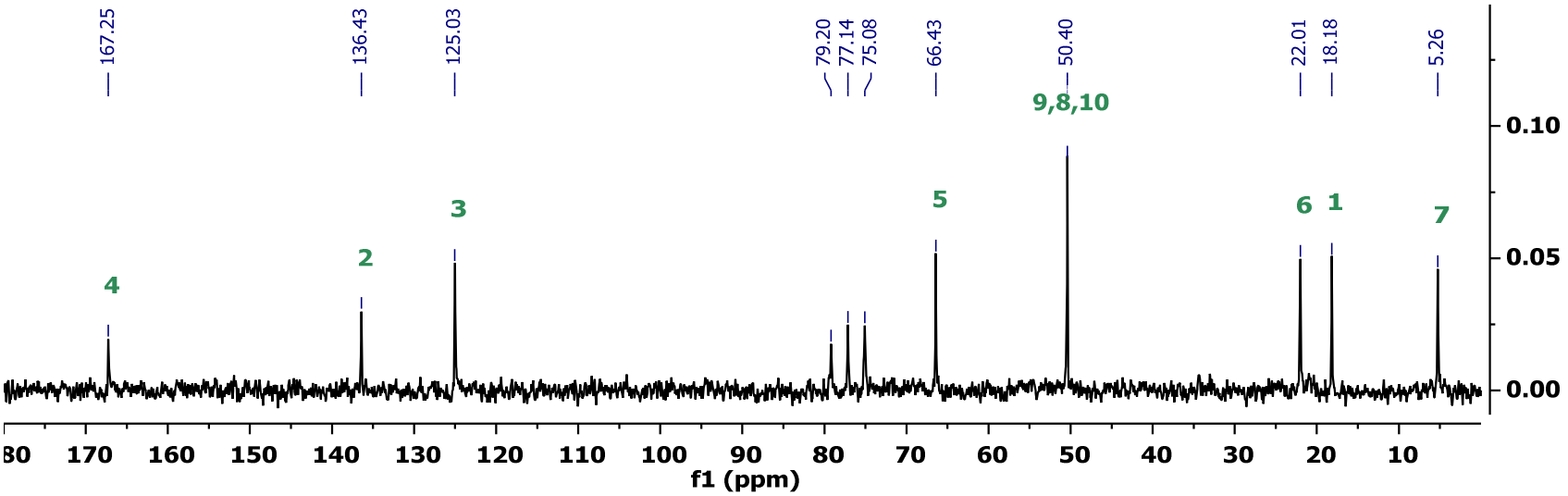
Structure confirmation of monomers

3-(Trimethoxysilyl)propyl methacrylate (C = 4.8 wt% - 0.45 mol/L – 30 mg in 400 µL $CDCL_3$)

1D 1H



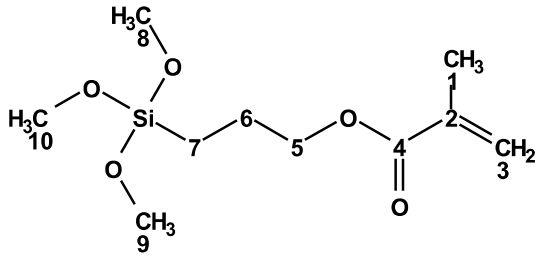
1D 13C



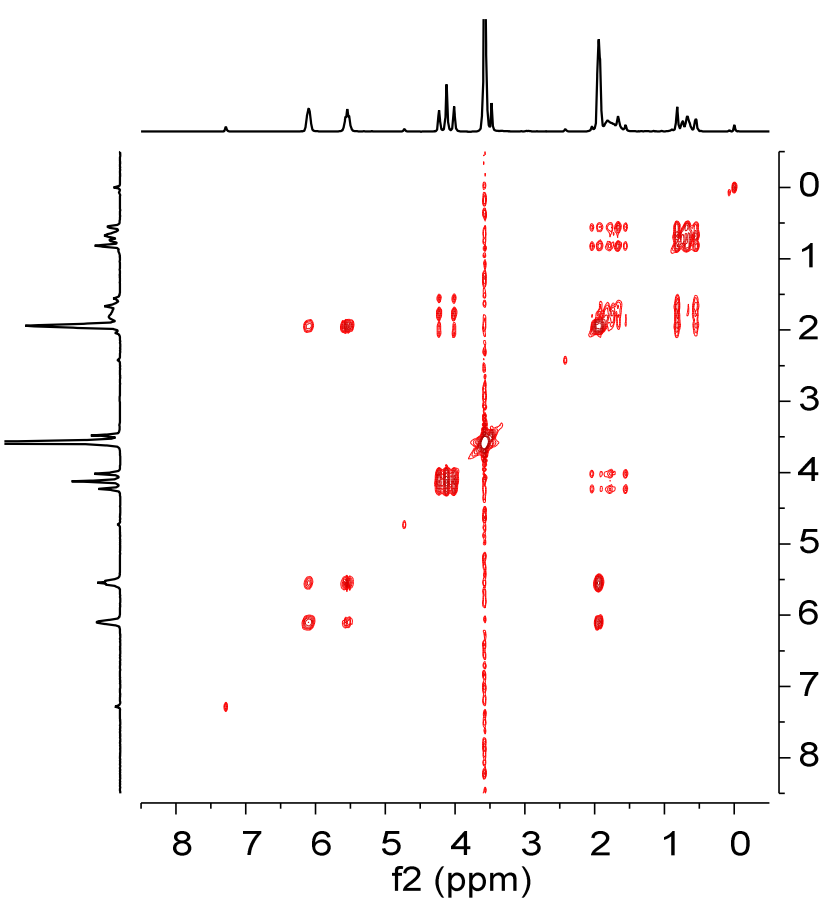
Structure confirmation of monomers

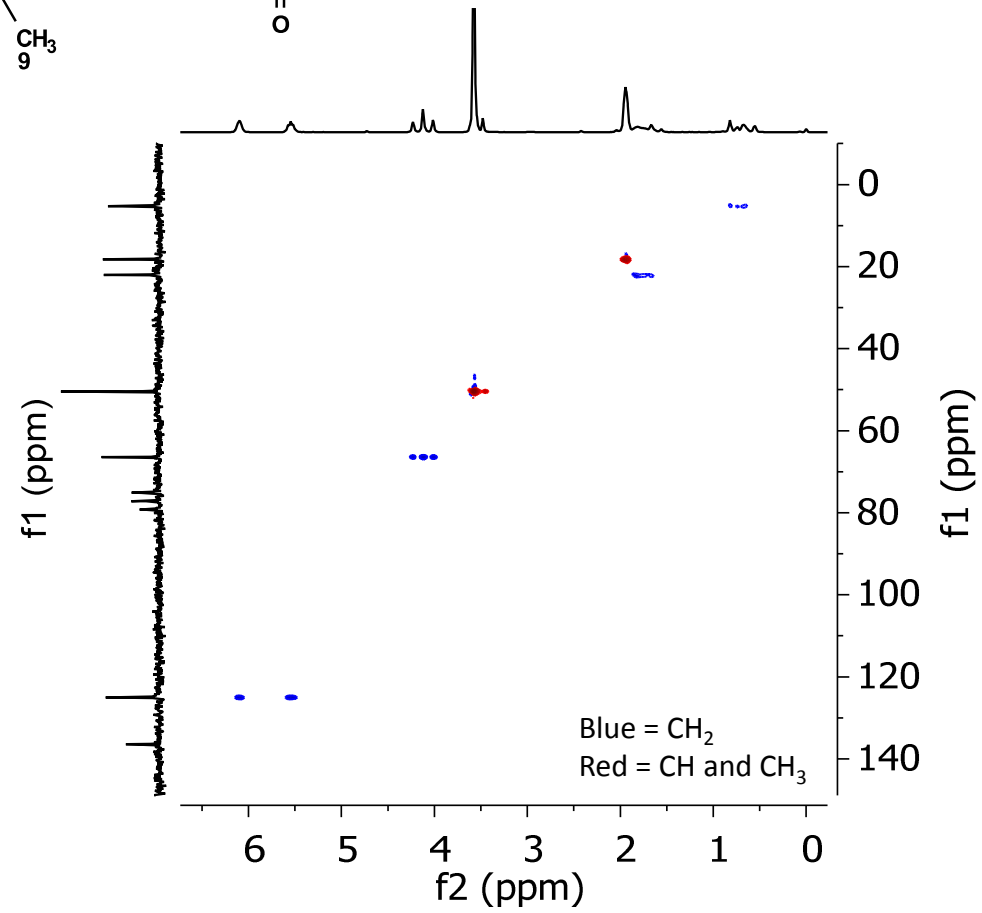
3-(Trimethoxysilyl)propyl methacrylate (C = 4.8 wt% - 0.45 mol/L – 30 mg in 400 µL $CDCL_3$)

2D H-H COSY



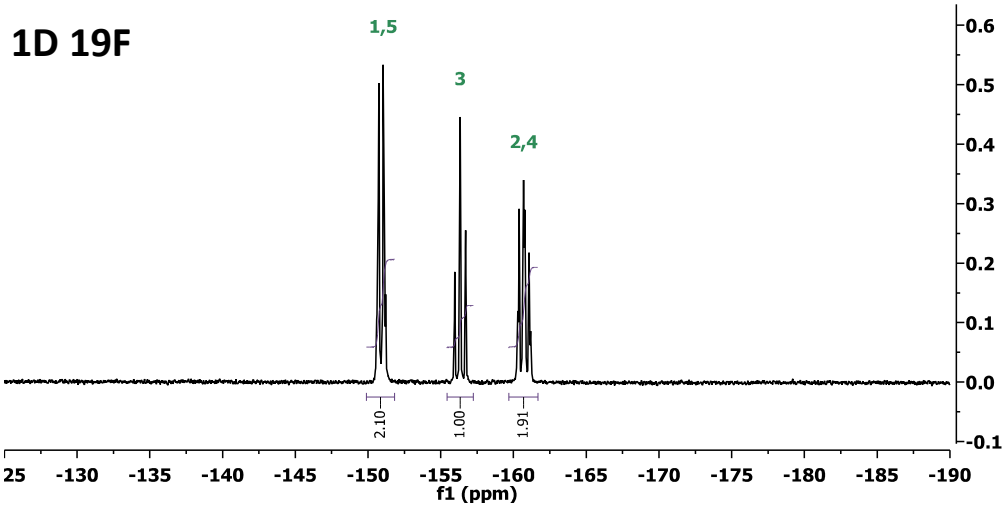
2D HSQC-ME

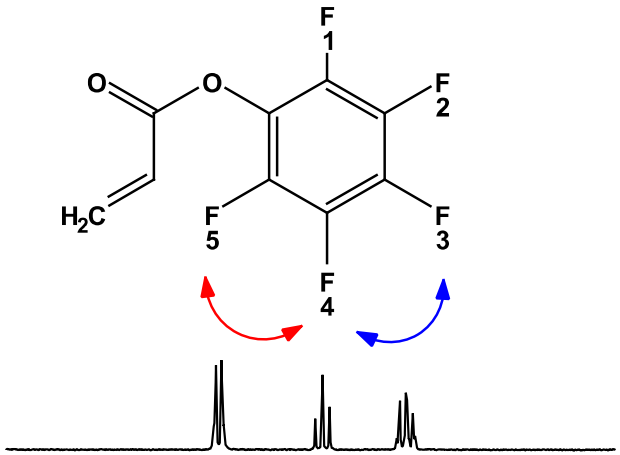


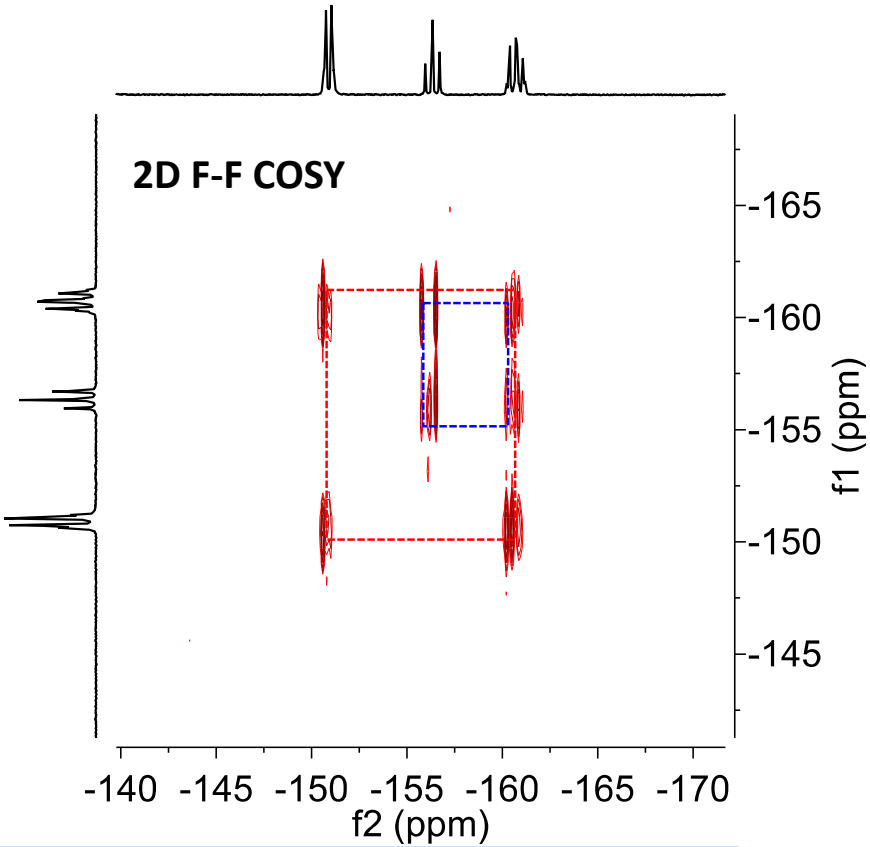


Structure confirmation of monomers

Pentafluorophenyl methacrylate (PFMA)



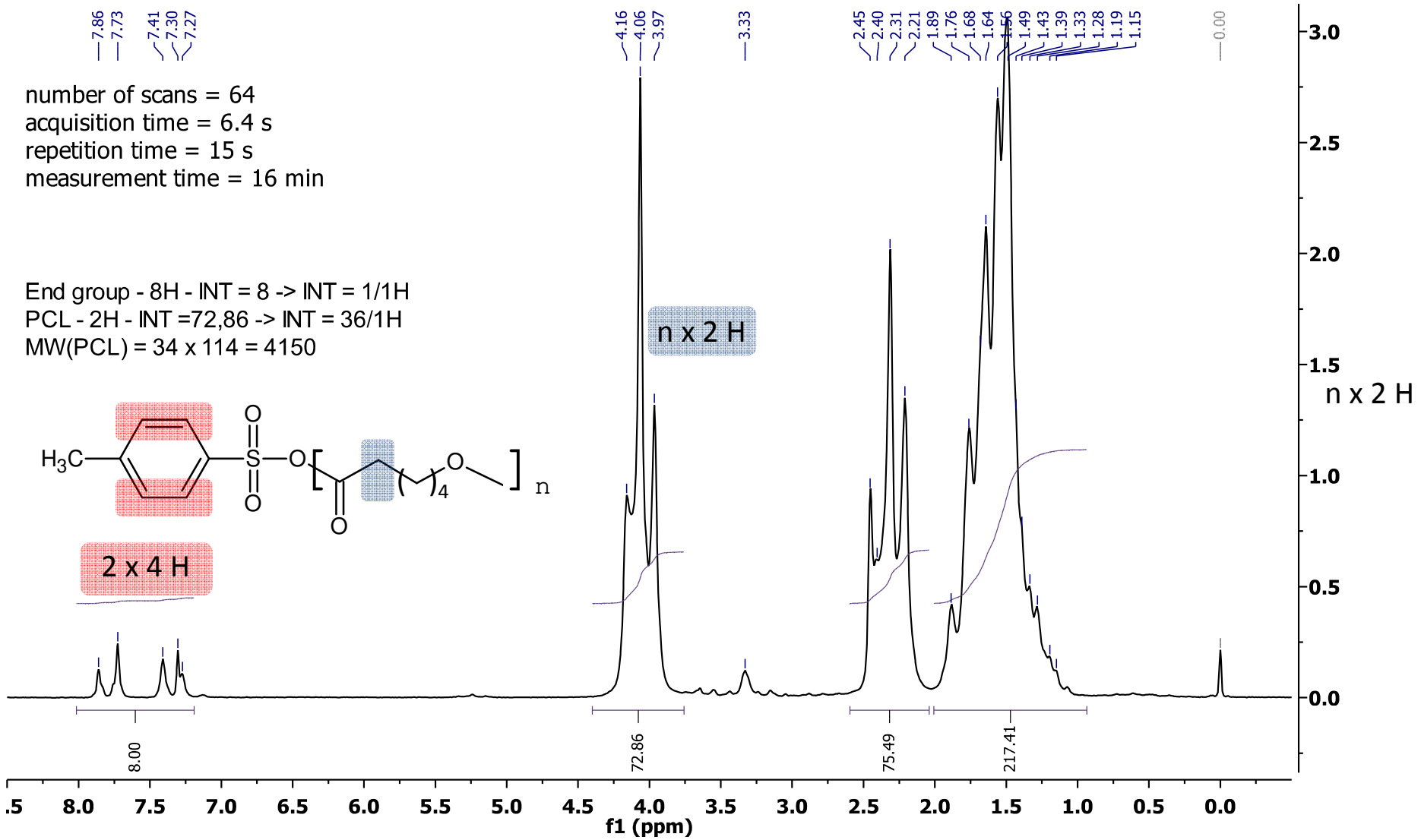




- 19F spectra can easily interpreted
- No retuning is required between measuring different nuclei

End group analysis

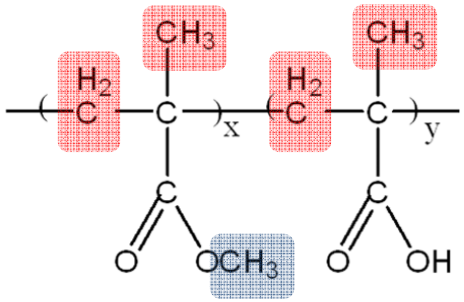
PCL-(Ots)2; C = 89 g/L; 1D 1H



Composition of copolymers

Poly(methacrylic acid, methyl methacrylate) 1:2; 20 mg/mL; 1D 1H; 80 MHz

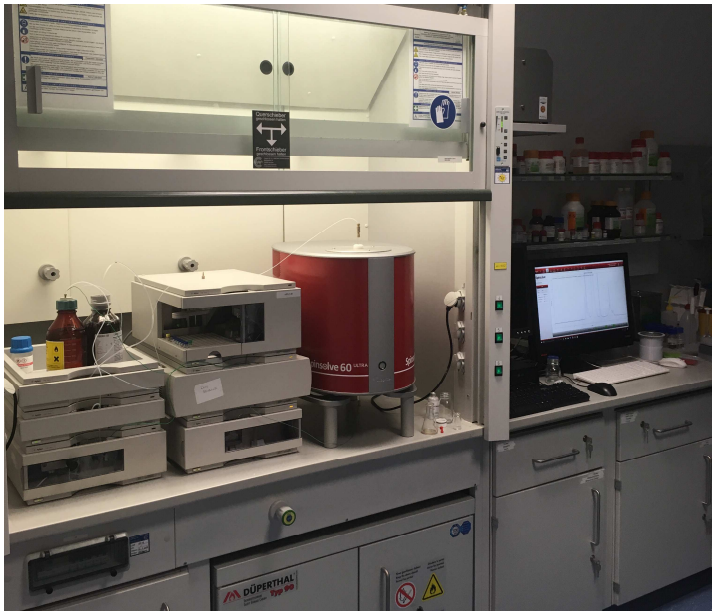
number of scans = 64
acquisition time = 6.4 s
repetition time = 15 s
measurement time = 16 min

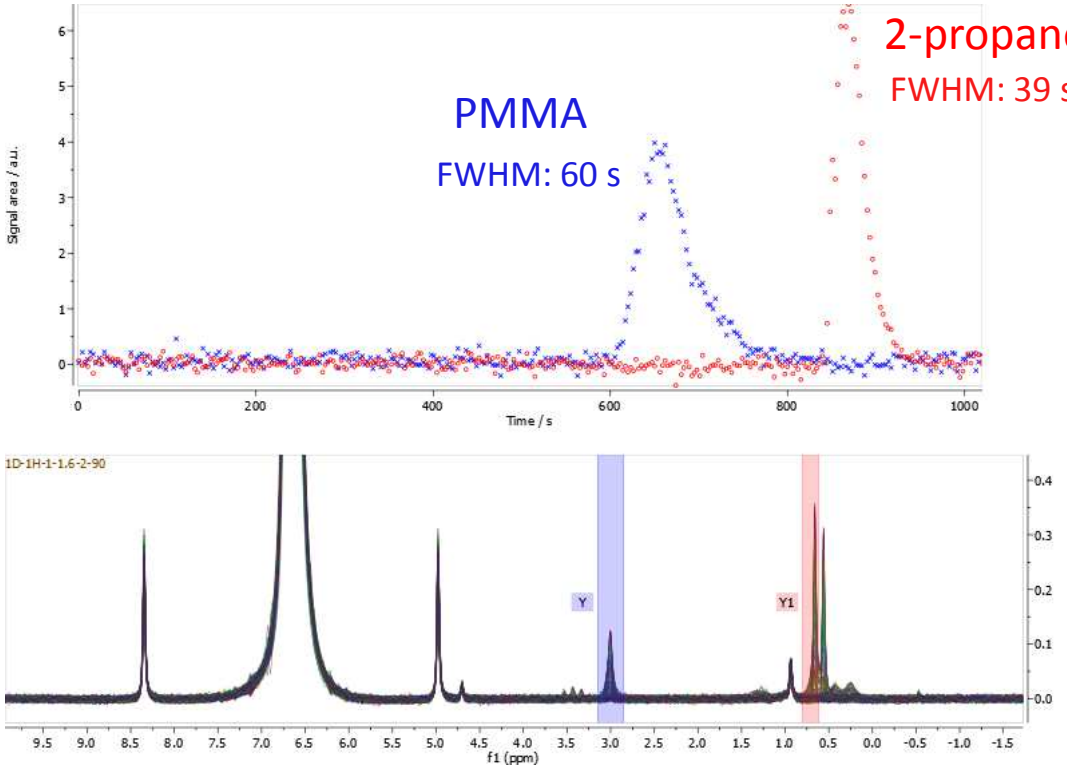


For every 10 monomer units,
there is 7 ester units
-> ester/acid ratio = 0,7

SEC-NMR coupling

Setup of SEC and NMR
in the fume hood





- Direct coupling of NMR with SEC using Magritek Glass Flow Cell
- Extra-column band broadening low regarding extra volume of the flow cell
- Studies in non-deuterated solvents (Chloroform, Dichloromethane)

SEC-NMR coupling

- Composition analysis of polymer blend with SEC-NMR coupling

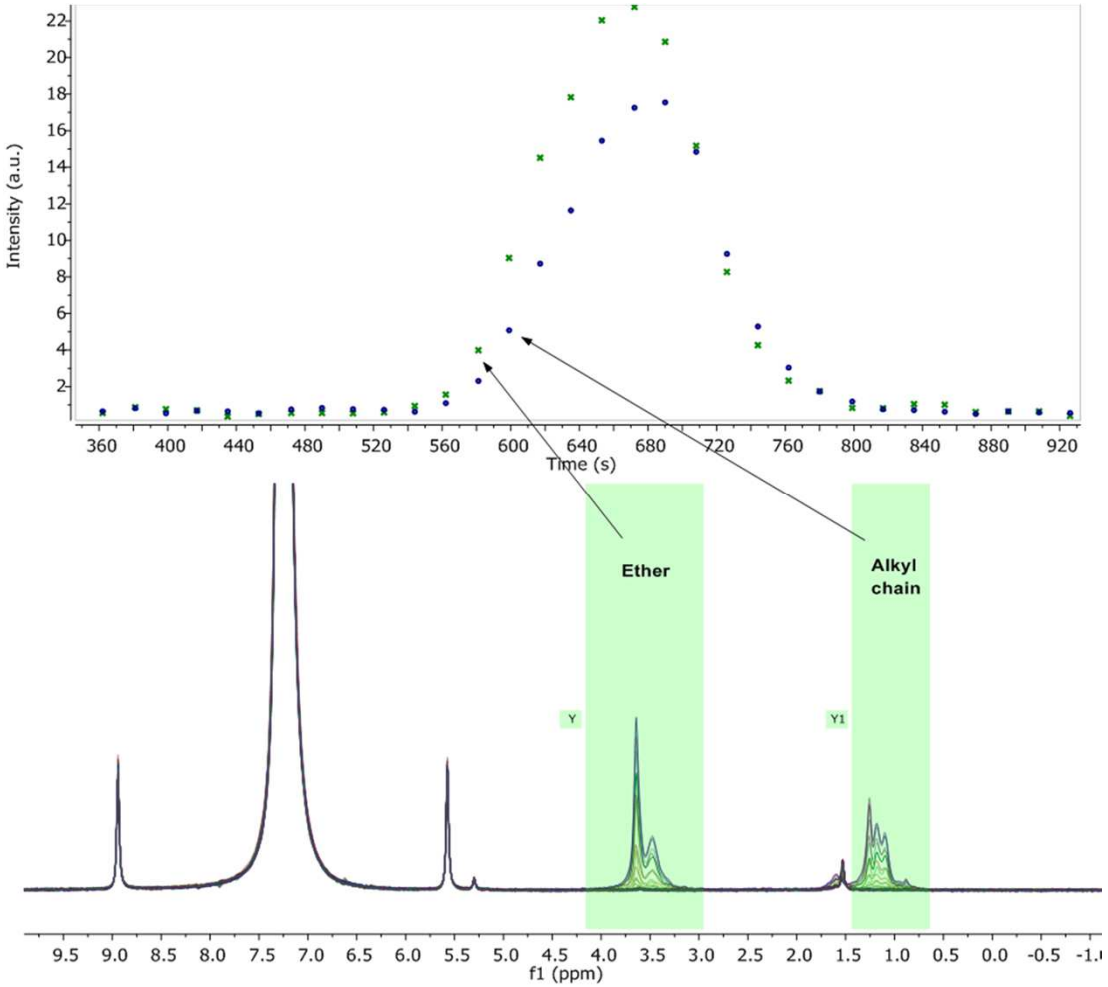
Blend of ethoxylated / propoxylated alcohols

$C_{12}O\ EO_{4.5}\ PO_{5.5} \rightarrow H_E/H_A = 0.92$

$C_{10}O\ EO_{9}\ PO_{12} \rightarrow H_E/H_A = 1.35$

6.3 mg of each substance injected

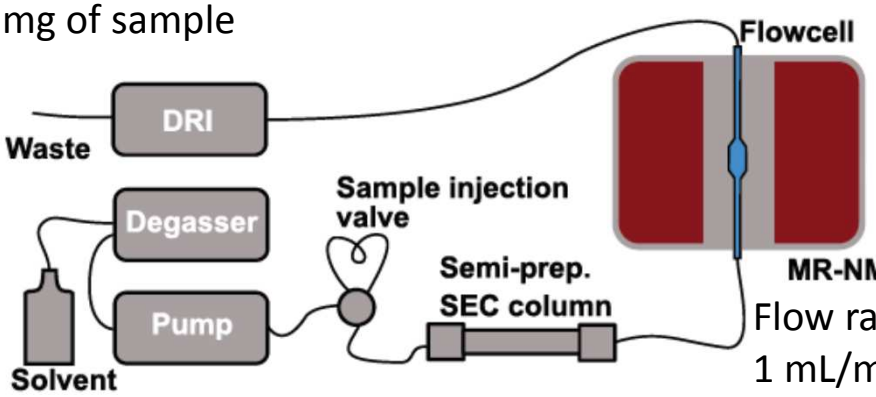
He/Ha	Retention time (s)
1.32	672
0.89	726



- Changes in composition during elution of the mixture

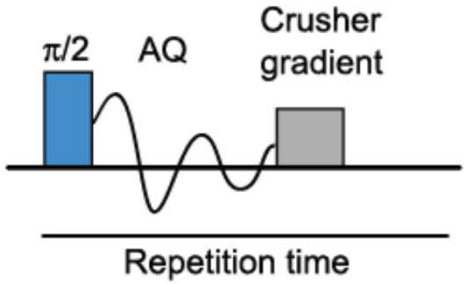
SEC-NMR coupling

Only 1 mg of sample

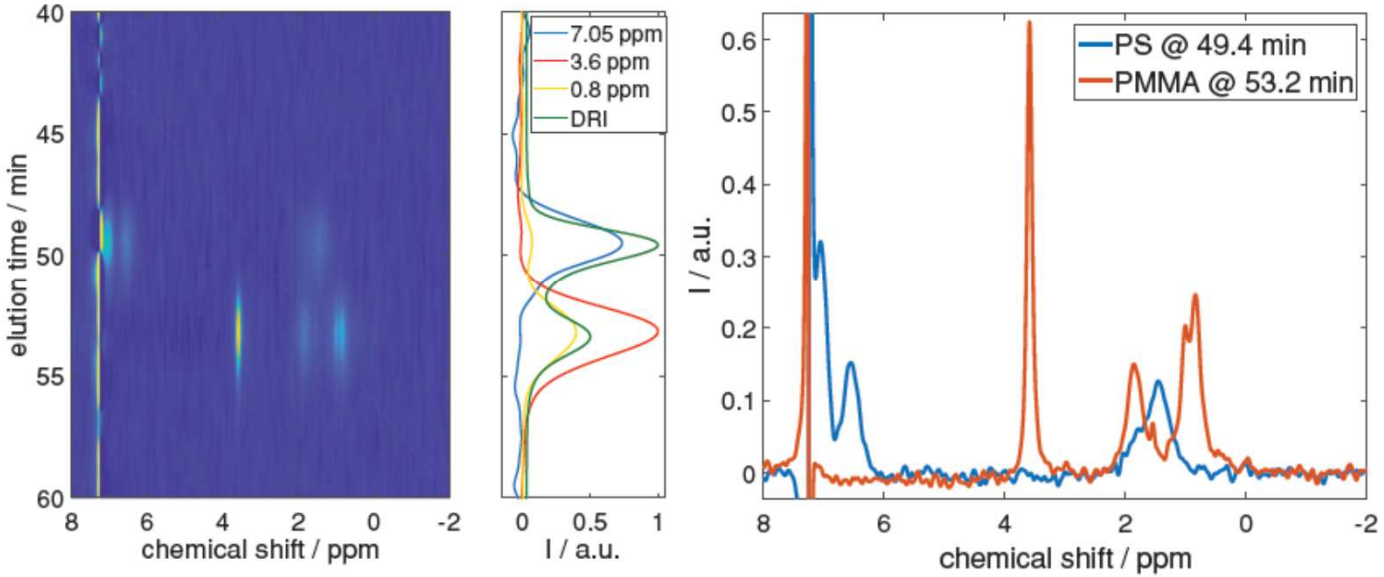


Flow rate of 1 mL/min

Pulse sequence

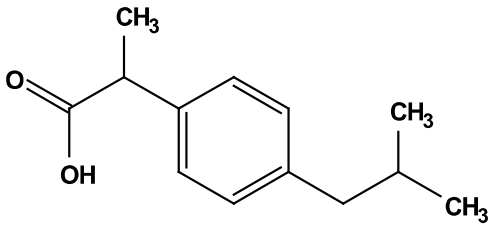


Example with a blend:
PS54k and PMMA23k

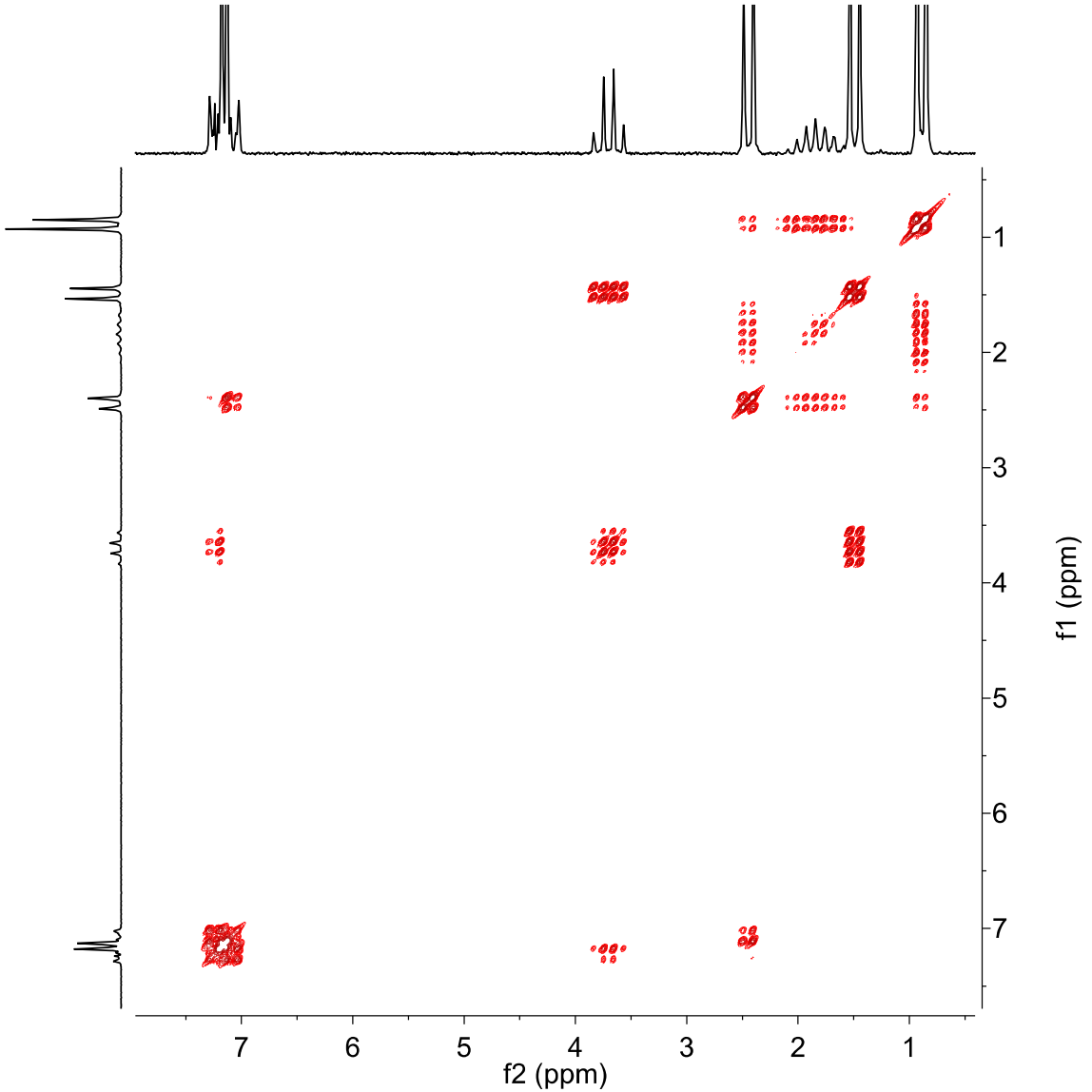


Medium Resolution 1H-NMR at 62 MHz as a New Chemically Sensitive Online Detector for Size-Exclusion Chromatography (SEC–NMR) *J. Höpfner; K.-F. Ratzsch; C. Botha; M. Wilhelm; Macromol. Rapid Commun.* **2018**, 1700766

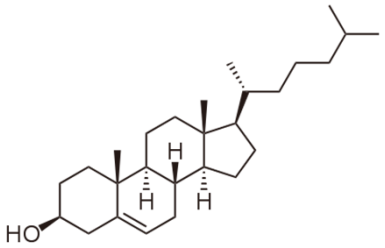
Structure verification at 80 MHz



Ibuprofene



Comparison with HF-NMR ^{13}C



Cholesterol

(75 MHz – $CDCl_3$)

300 MHz ^1H

(15 MHz – $CDCl_3$)

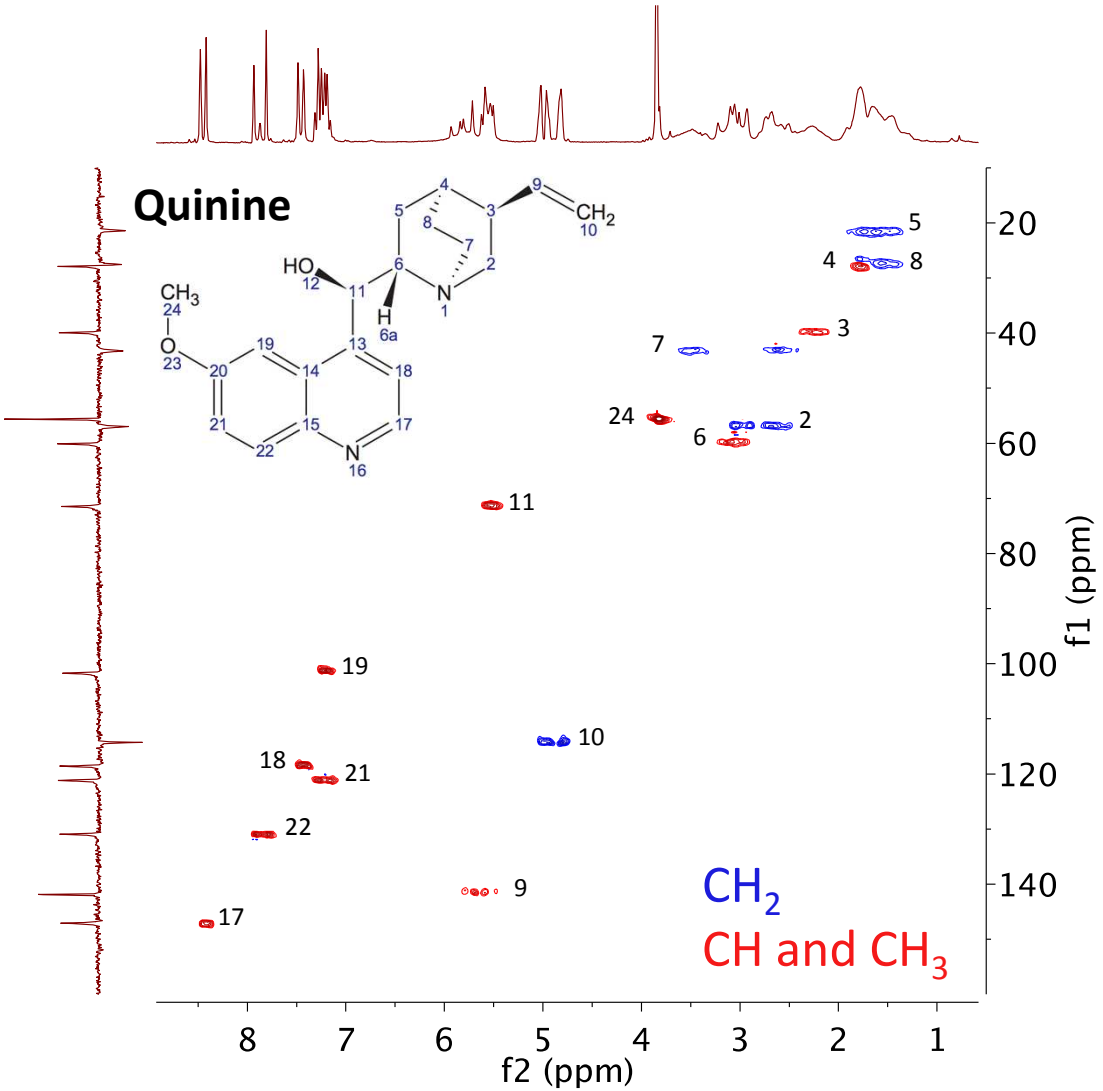
60 MHz ^1H

Structure verification at 80 MHz

HSQC-ME (Heteronuclear Single Quantum Coherence – Multiplicity Edited)

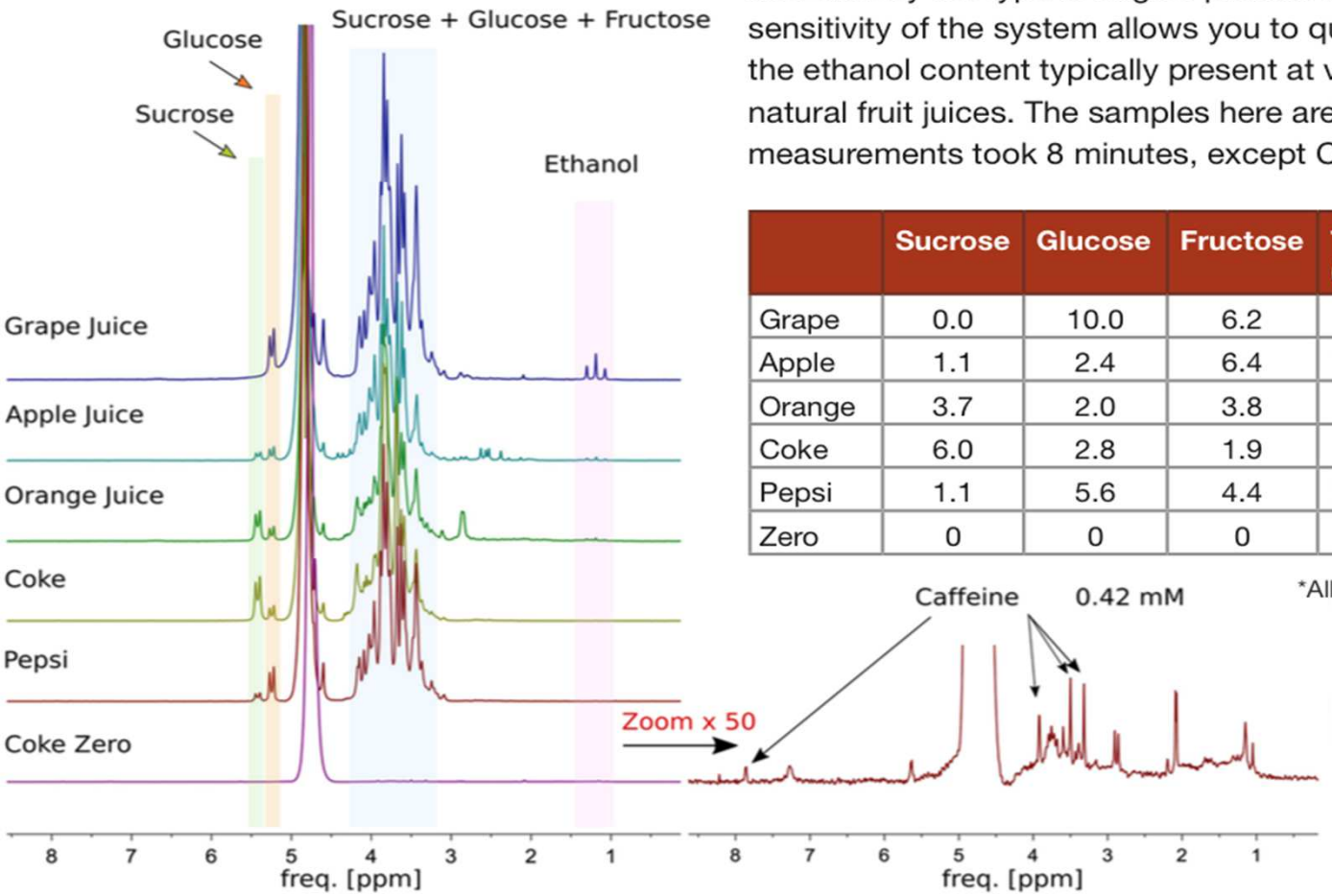
Information of HSQC and DEPT-135 in a single experiment

Quinine is used for treatment of malaria as well as flavor in beverages like tonic water.



Sugar and EtOH in Beverages with ULTRA

Measuring sugar content in soft drinks



Spinsolve ULTRA measures not only the sugar content but can also identify the type of sugars present in the sample. The high sensitivity of the system allows you to quantify with high accuracy the ethanol content typically present at very low concentrations in natural fruit juices. The samples here are all neat. All measurements took 8 minutes, except Coke Zero was 1 hour.

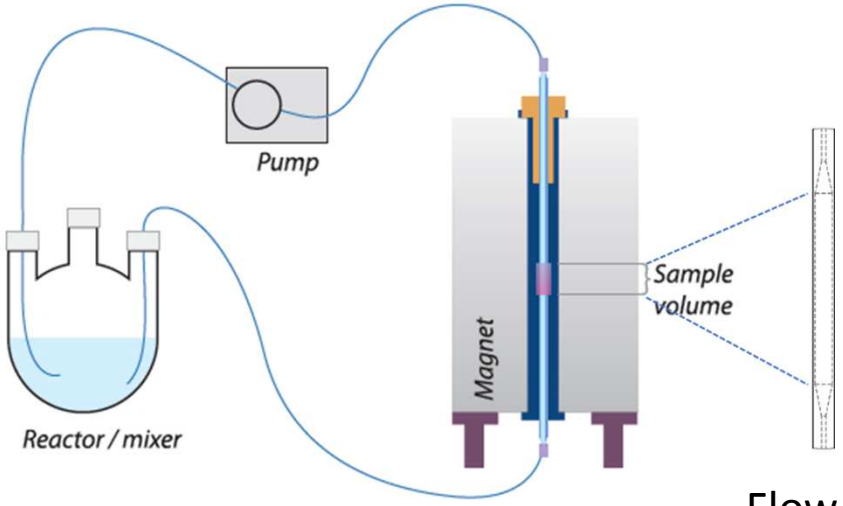
	Sucrose	Glucose	Fructose	Total (NMR)	Total (Label)	Ethanol
Grape	0.0	10.0	6.2	16.2	16.0	0.16
Apple	1.1	2.4	6.4	9.8	9.8	0.03
Orange	3.7	2.0	3.8	9.5	9.0	0.02
Coke	6.0	2.8	1.9	10.7	10.7	0
Pepsi	1.1	5.6	4.4	11.2	10.7	0
Zero	0	0	0	0	0	0

*All units g/100ml. Ethanol is % vol.

Online Reaction Monitoring

Following the progress of a reaction from starting material to end-point

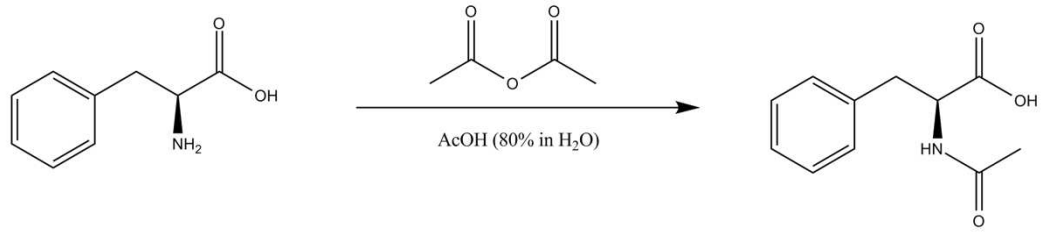
Flow reaction monitoring setup



Flow cell

- Hyphenation to chemical reactions using a flow setup
- NMR can be placed directly next to the reactor, e.g. in a fumehood
- Very low restrictions regarding interferences with other lab equipment

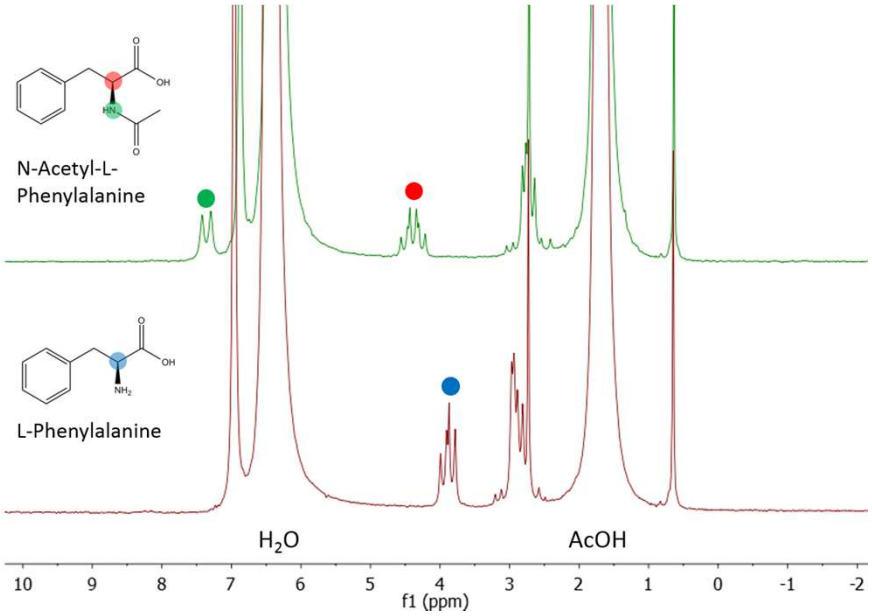
Monitoring Protection of L-Phenylalanine



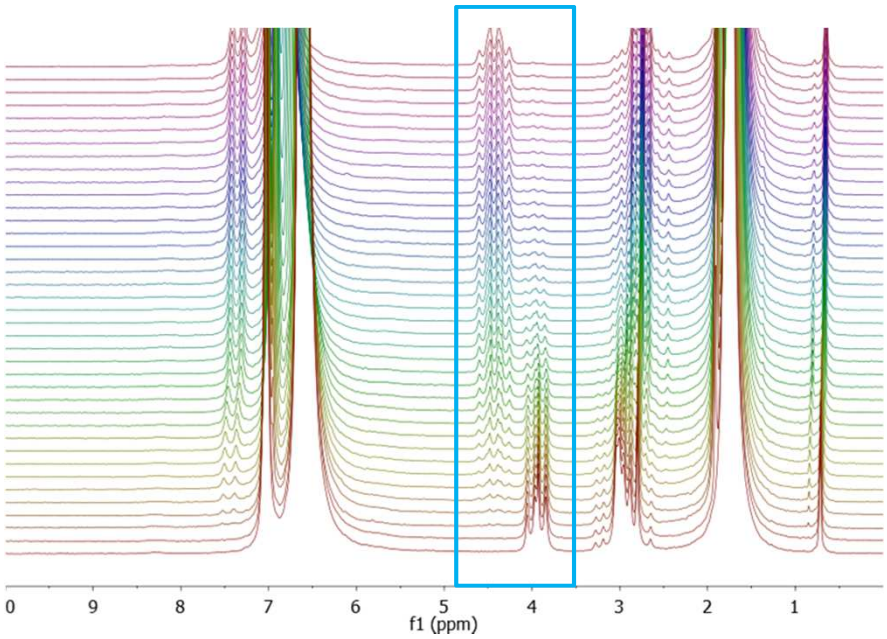
(*S*)-2-amino-3-phenylpropanoic acid

(*S*)-2-acetamido-3-phenylpropanoic acid

Regions of Interest (5 mm tube)

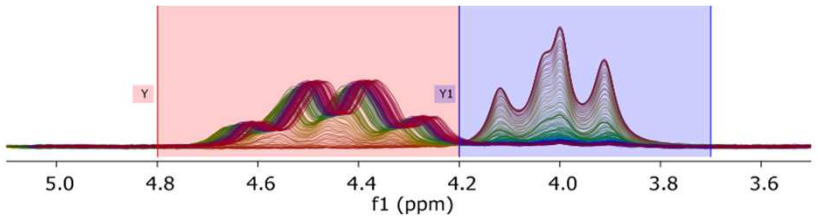


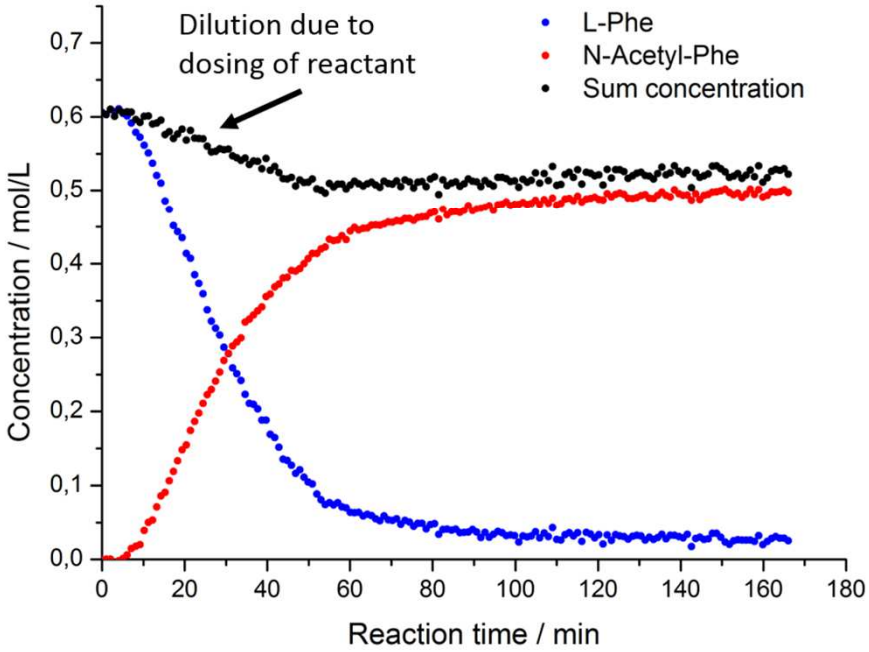
Stacked spectra (flow setup)



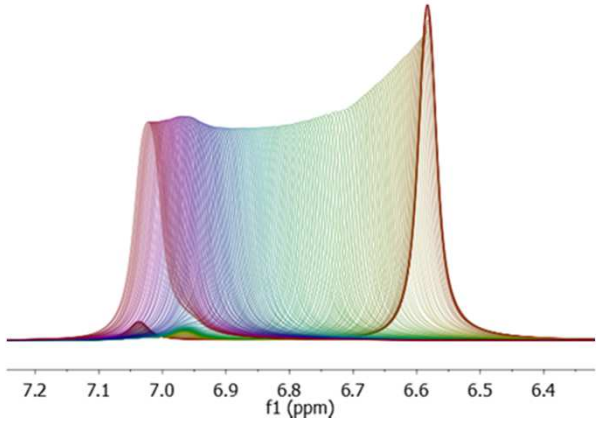
Monitoring Protection of L-Phenylalanine

- Concentration plot of acetylation reaction
- Additional information from NMR spectra

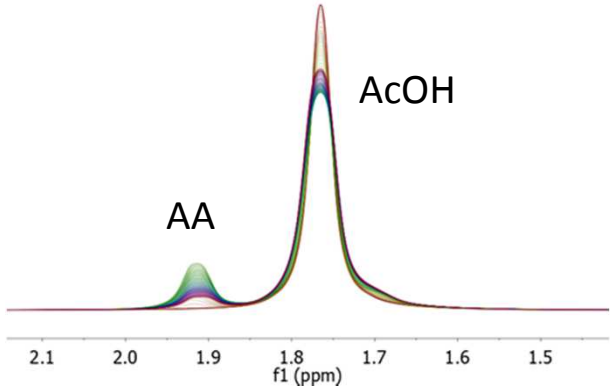




pH induced shift of water signal



Hydrolysis of excess acetic anhydride



Thank you

- For further Information, please contact sales@magritek.com
- Further information is also located on the Web page www.magritek.com
- Typical Spinsolve Example Spectra can be found via http://www.magritek.com/products/spinsolve/nmr-spectra-examples/

Standard Laboratory Setup

Reaction Monitoring Setup