

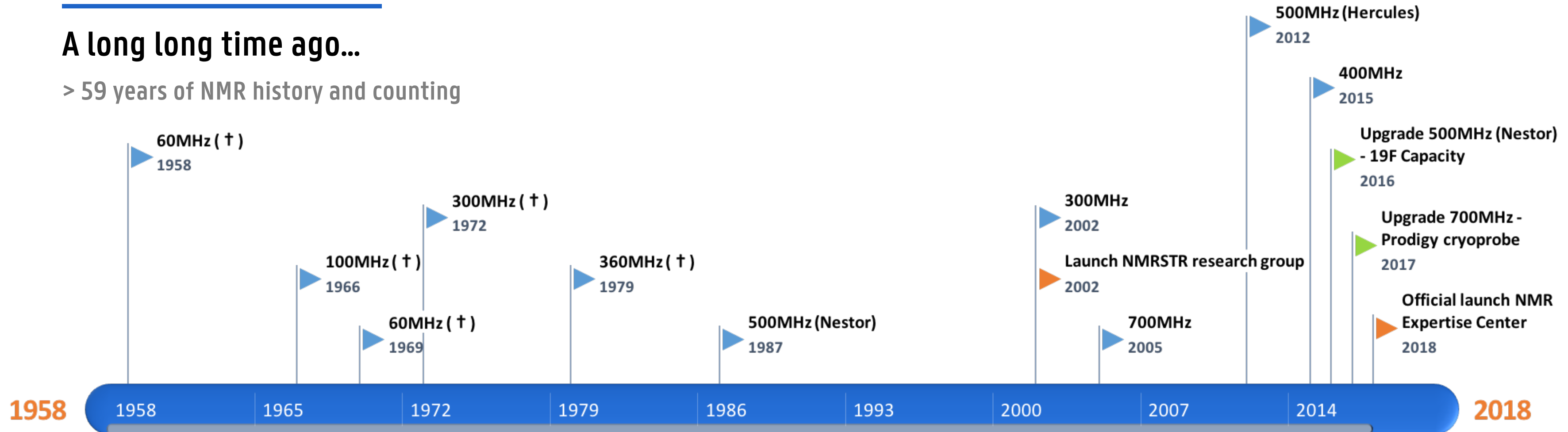
# NMR EXPERTISE CENTER

Est. August 2018

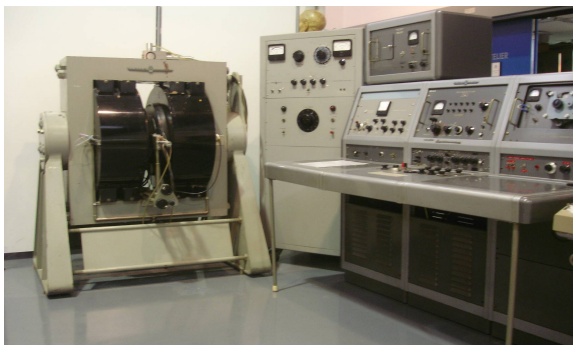
# SOME HISTORY

## A long long time ago...

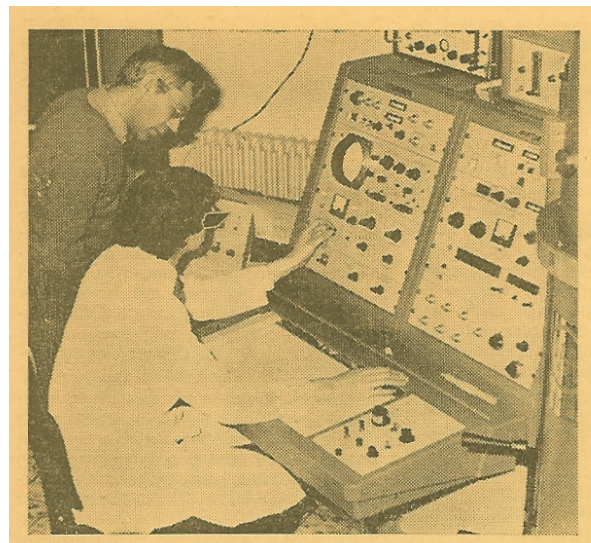
> 59 years of NMR history and counting



Expo '58 – Varian introduces the first commercial NMR spectrometer. UGent acquires the first NMR in Belgium



1<sup>st</sup> 300MHz NMR spectrometer in Europe is installed at UGent.



Interuniversity collaboration to fund the investment of the 700MHz high-field equipment for a total value of 2.019.502 €



# NMR SPECTROSCOPY

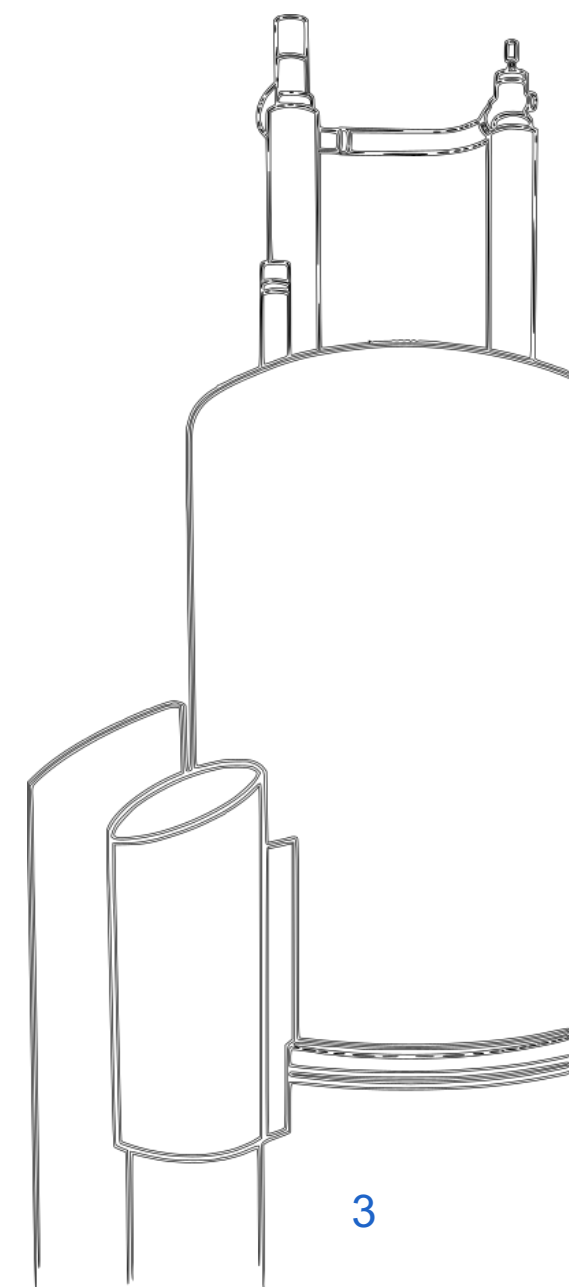
NMR is a spectroscopic technique can provide detailed information on the **structure, dynamics, reaction state** and **chemical environment** of molecules

## Advantages

- Multi-element capabilities  
In principle any nuclei possessing spin can be studied
- Provide unique, well-resolved, analytically tractable and often highly predictable spectra for small molecules
- High-throughput method with high degree of automatization
- Non-destructive method  
Samples can be recuperated afterwards

## Disadvantages

- Lower sensitivity  
Counteracted by the use of high-field & specialized probes  
Higher sample amounts can be used and recuperated afterwards

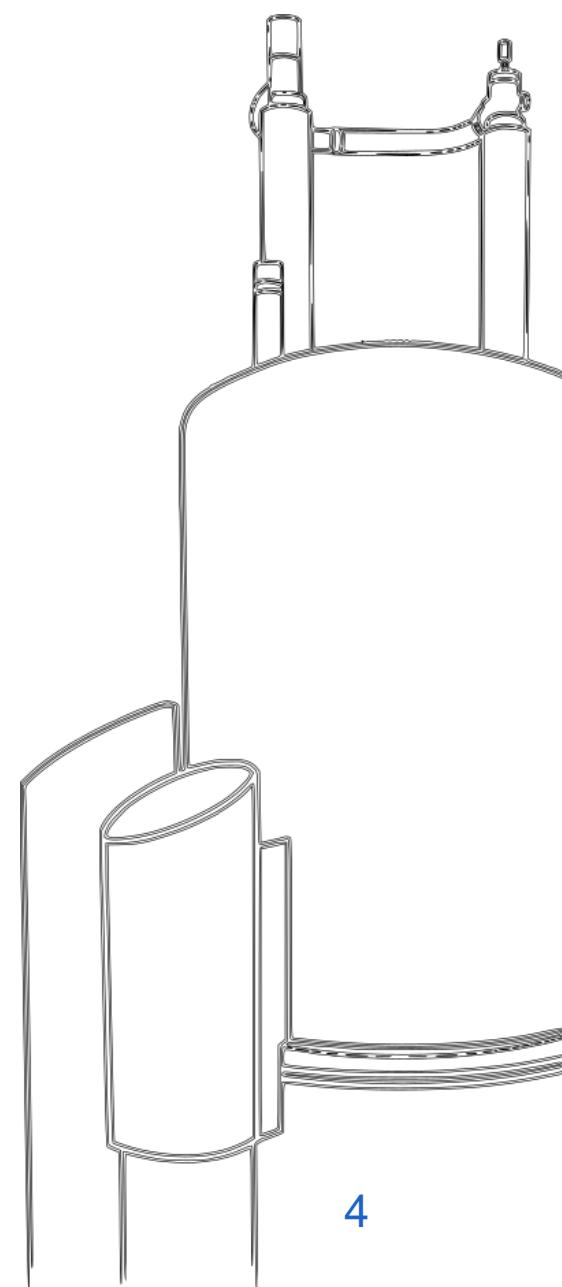


# NMR SPECTROSCOPY

NMR is a spectroscopic technique can provide detailed information on the **structure, dynamics, reaction state** and **chemical environment** of molecules

## Everyday NMR applications

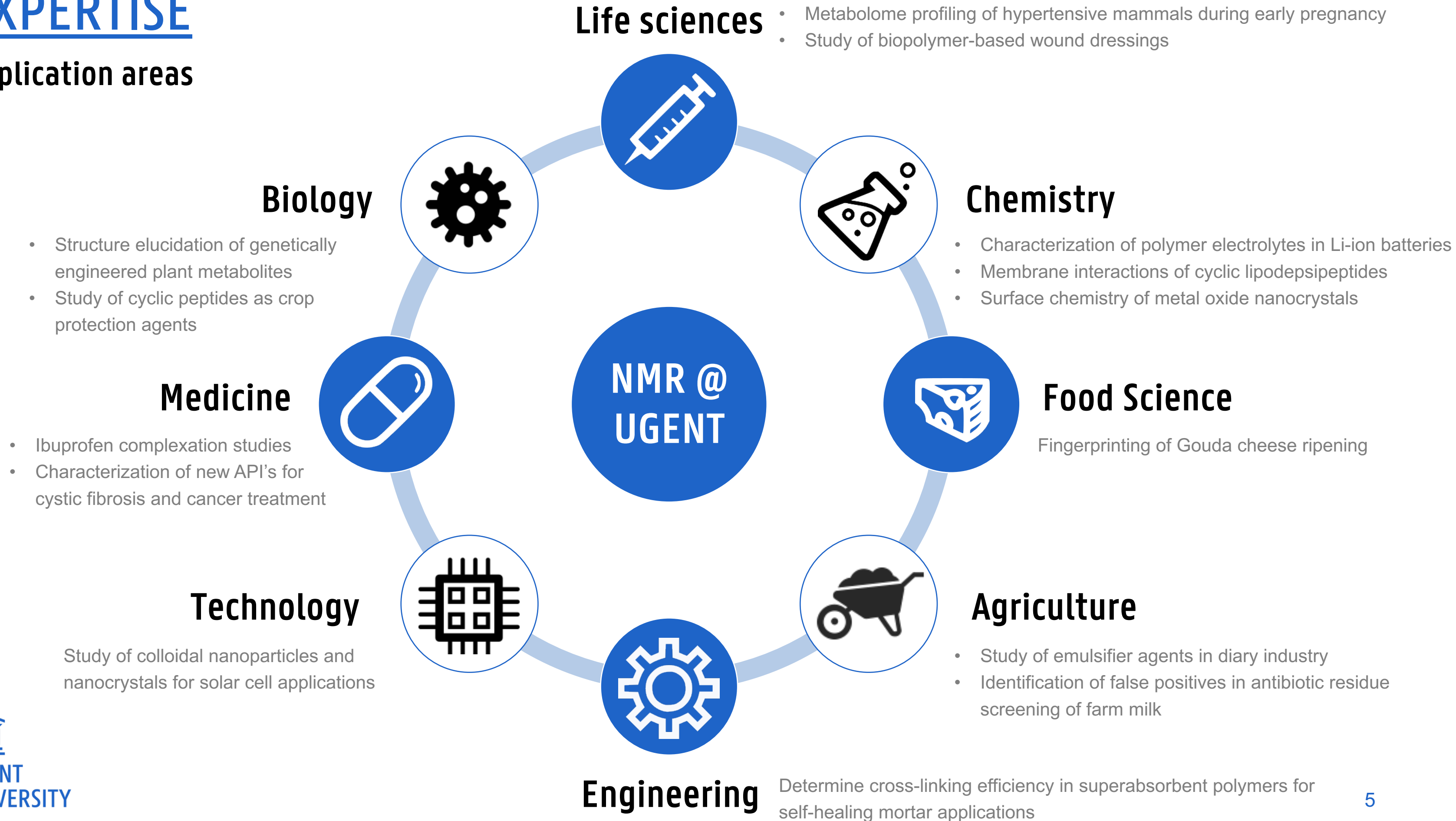
- Small molecule structure elucidation and quantification
  - Determine or confirm known and unknown structure(s)
  - Determine how much of a specific material is effectively present
  - Quality control or reaction monitoring measurements
- Macromolecular structure
  - Determine 3D conformations of biomacromolecules such as peptides and nucleic acids
- Mixture analysis
  - Digital separation of mixtures using diffusion NMR
  - Analysis of complex mixtures from metabolomic or foodstuff origins
- Study of materials
  - Study polymer structure, polymorphism and physical properties





# EXPERTISE

## Application areas



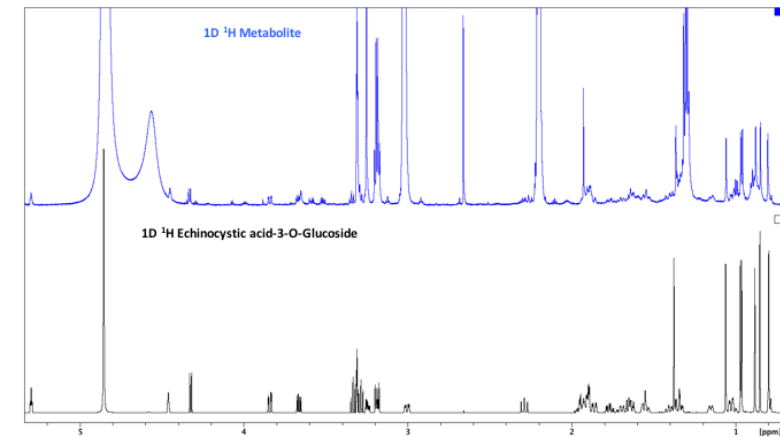
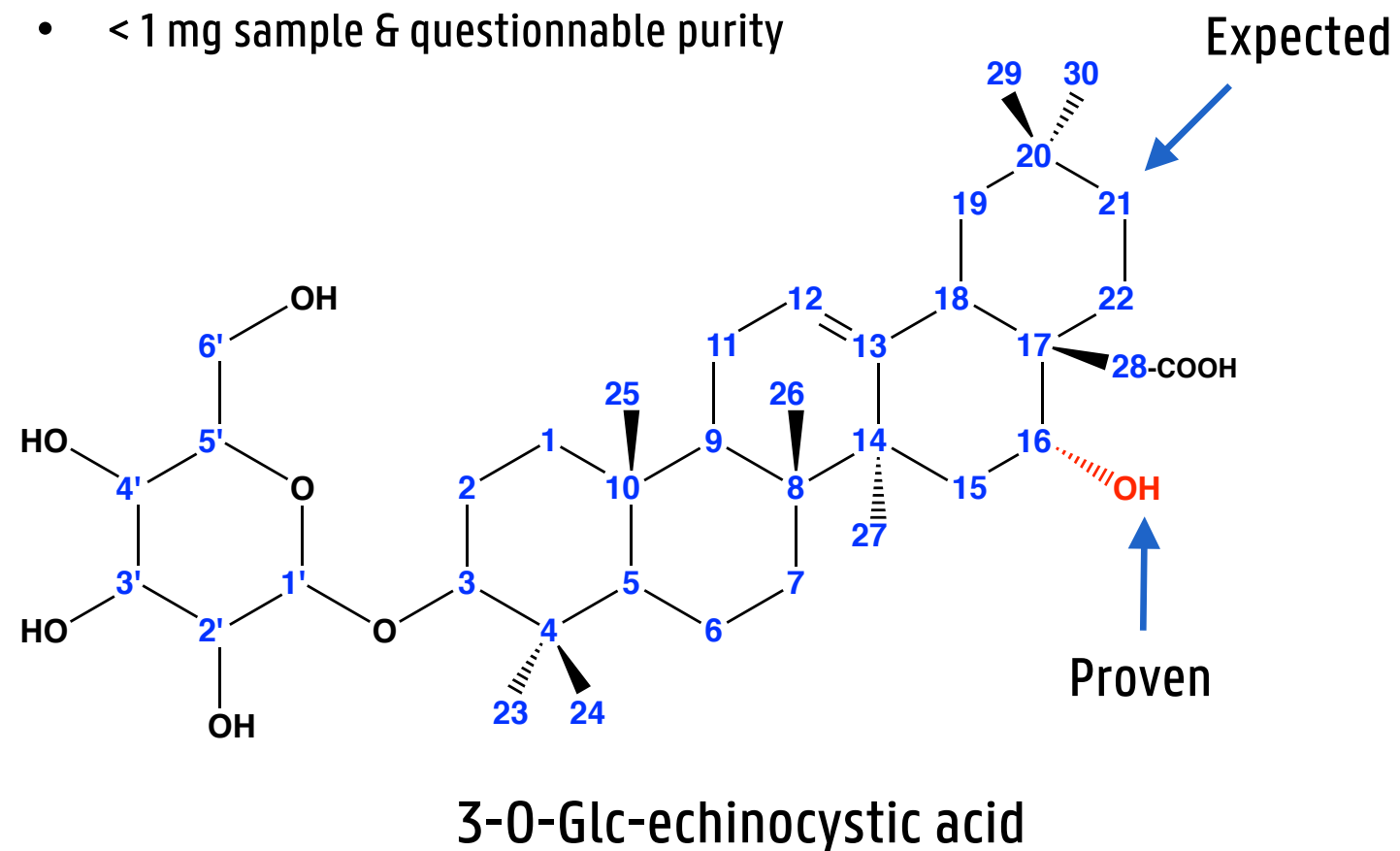
# EXPERTISE

## Structure elucidation and confirmation of small to medium organic compounds

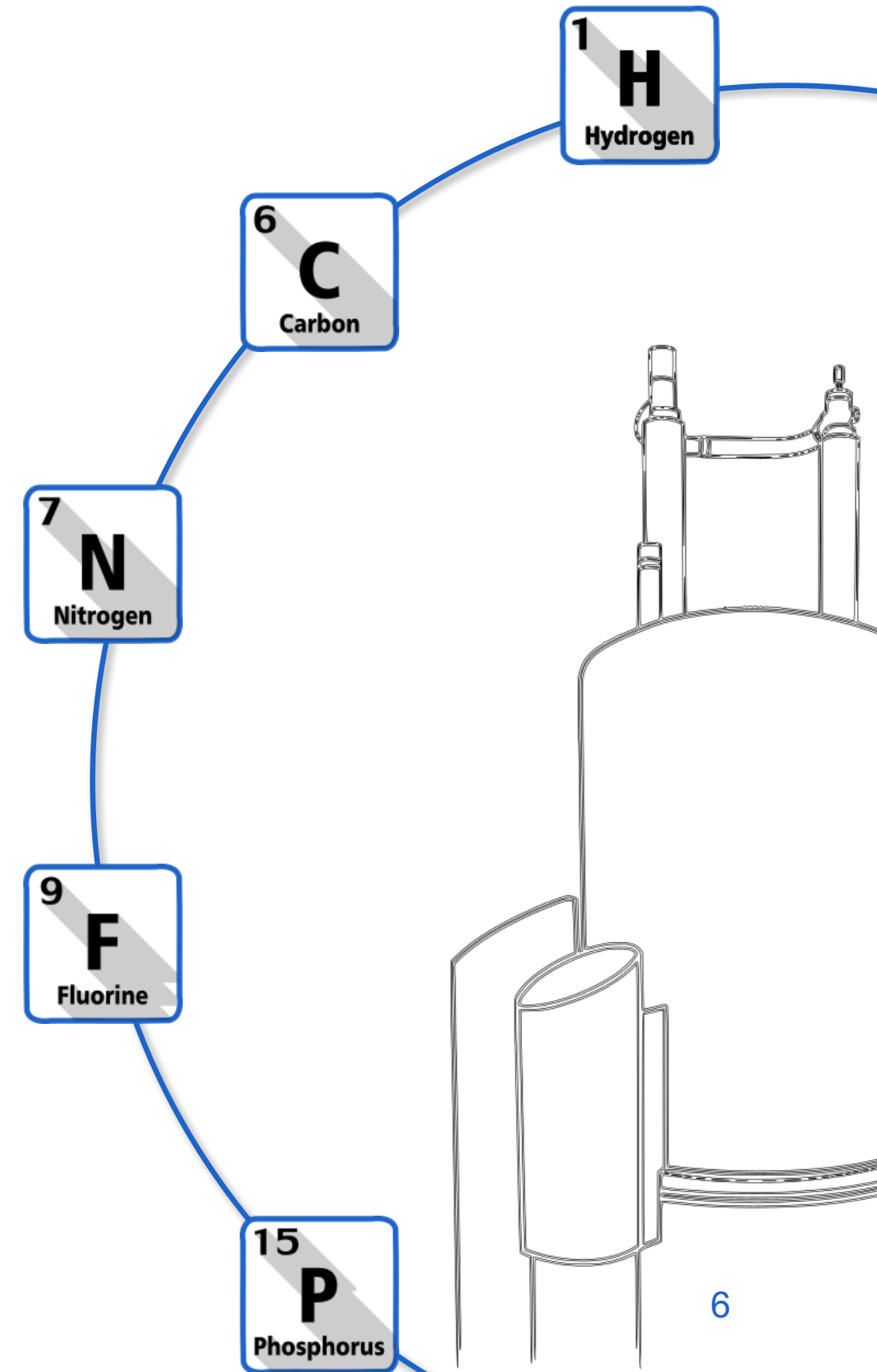
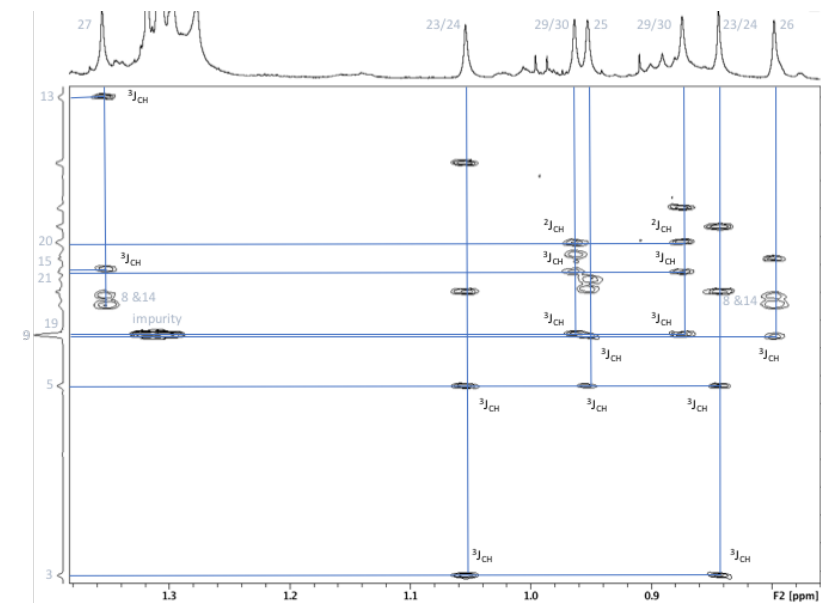
### A. Goossens

Towards synthetic biology platforms for the production of bioactive triterpene sapo(ge)nins.

- Position of hydroxylation in 3-O-Glc-echinocystic acid?  
-OH @ C<sub>16</sub>, C<sub>21</sub>, C<sub>28</sub> or C<sub>30</sub> position?  
Relative stereochemistry?
- Structure intact?
- < 1 mg sample & questionable purity



1D <sup>1</sup>H & <sup>13</sup>C spectra for comparison with know standard



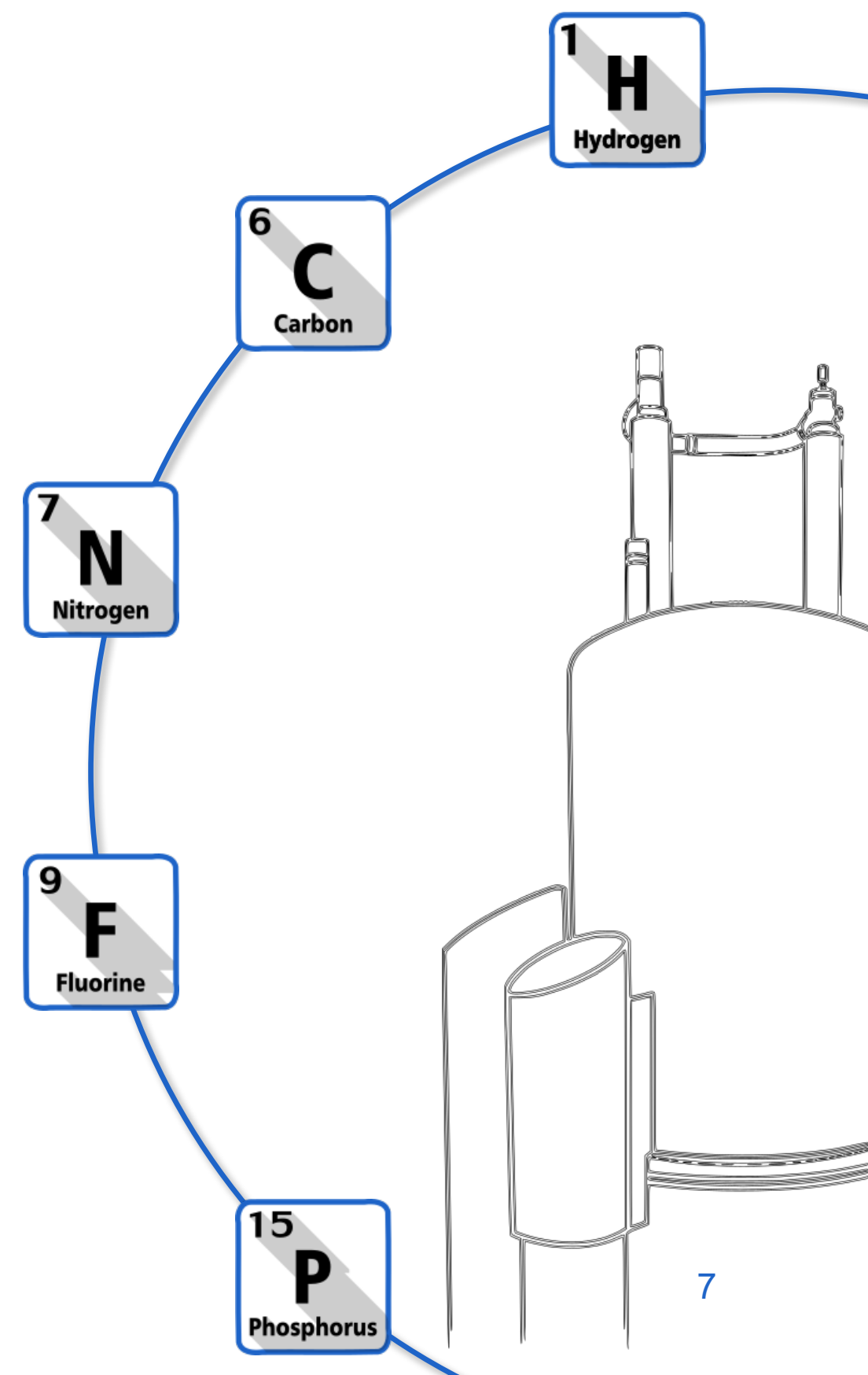
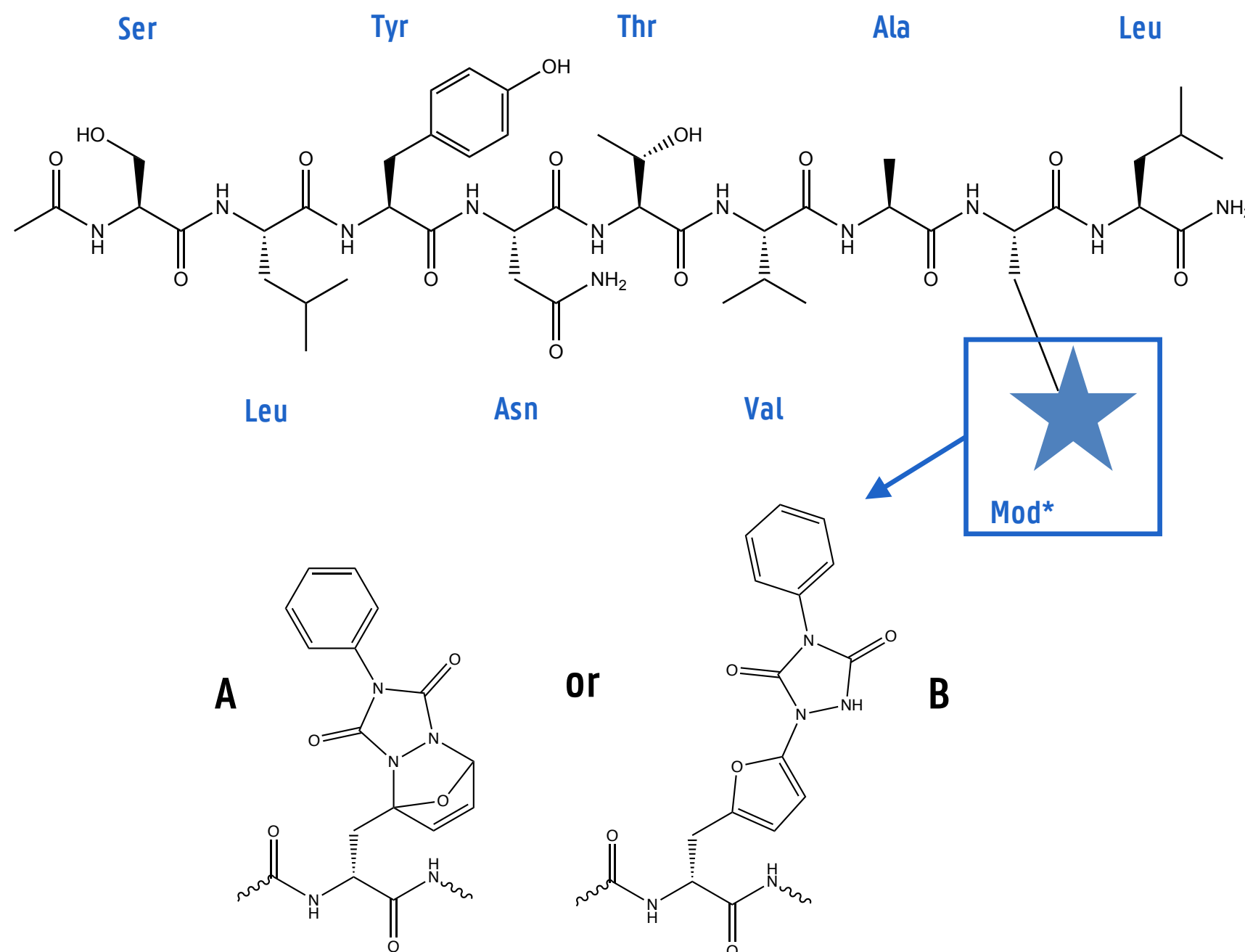
# EXPERTISE

## Structure elucidation and confirmation of peptides

K. Hoogewijs and A. Madder

Furan chemistry in reversible and irreversible orthogonal peptide labeling

- Amino acids and overall peptide sequence intact?
- Structure of the furan labeled peptide (Mod\*)?



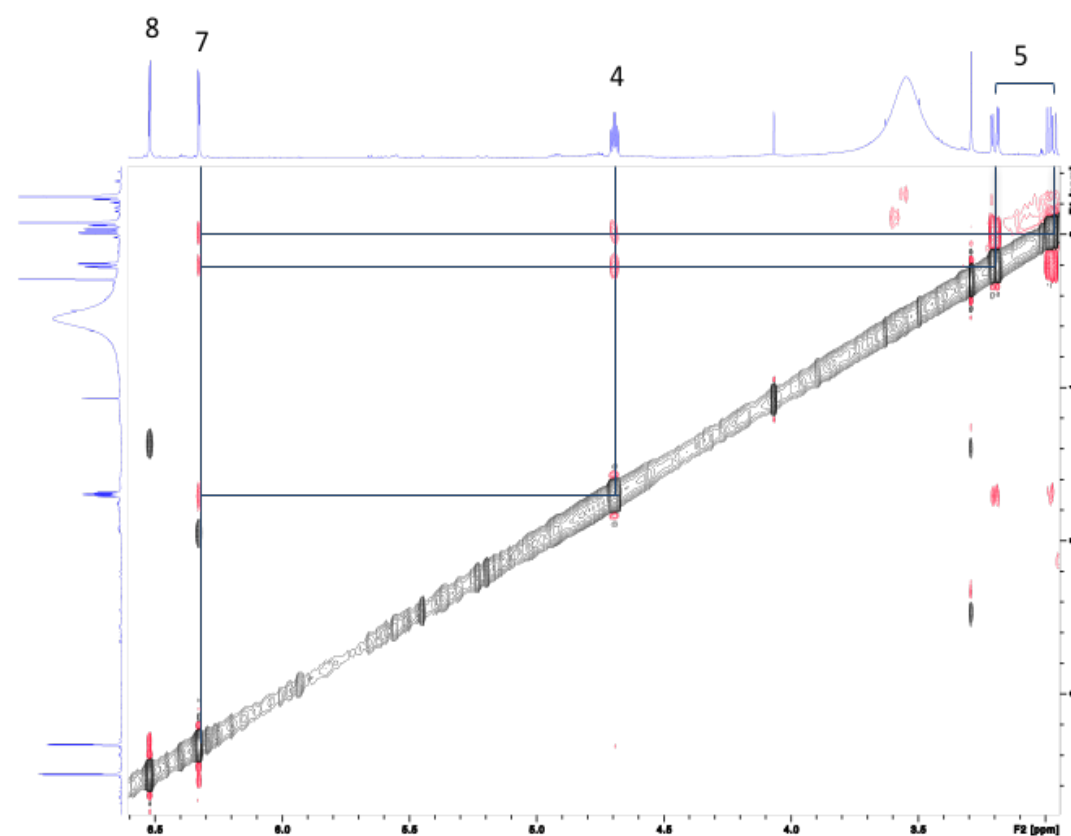
# EXPERTISE

## Structure elucidation and confirmation of peptides

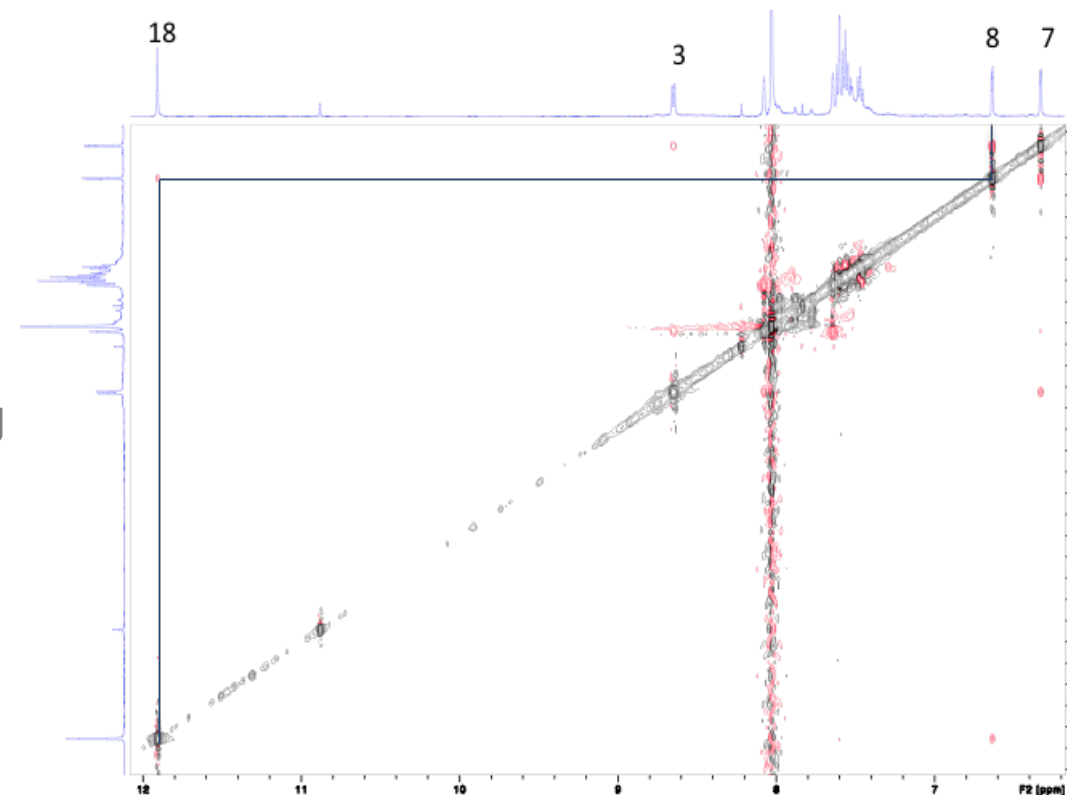
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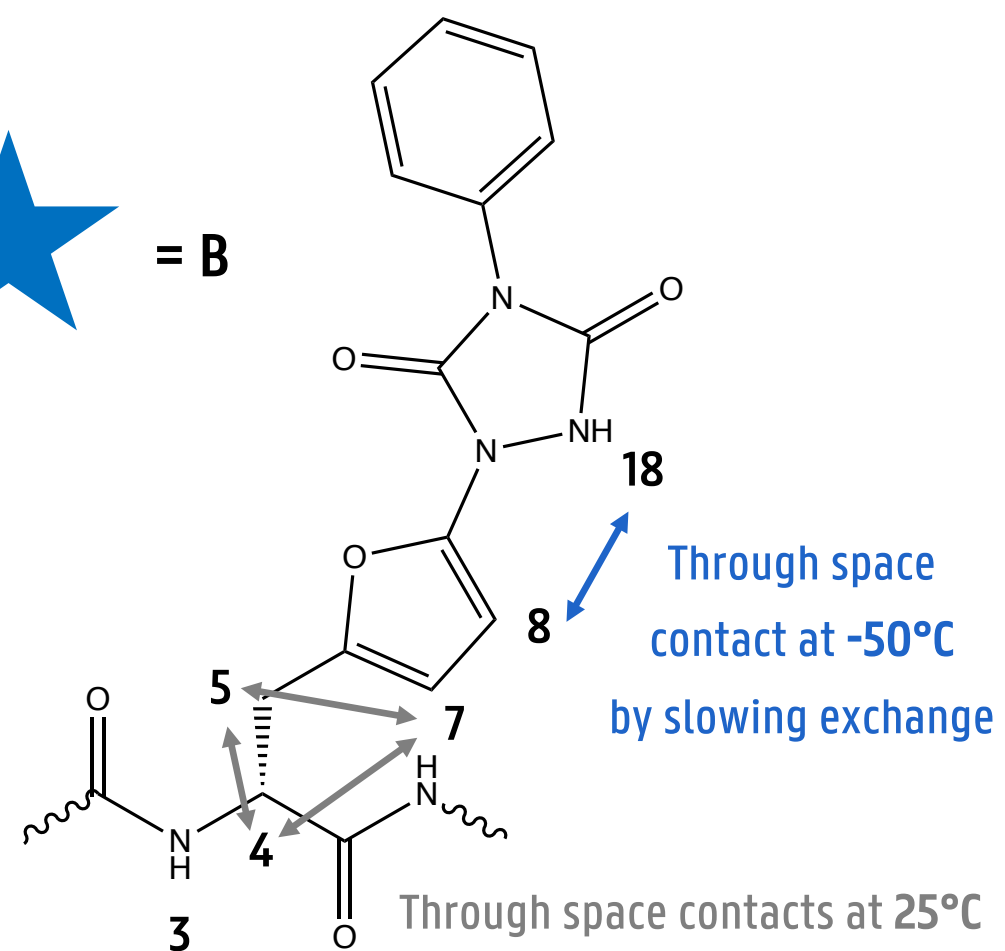
2D ROESY 500MHz, 150ms, 25°C



2D ROESY 500MHz, 150ms, -50°C



= B



<sup>1</sup>H  
Hydrogen

<sup>6</sup>C  
Carbon

<sup>7</sup>N  
Nitrogen

<sup>9</sup>F  
Fluorine

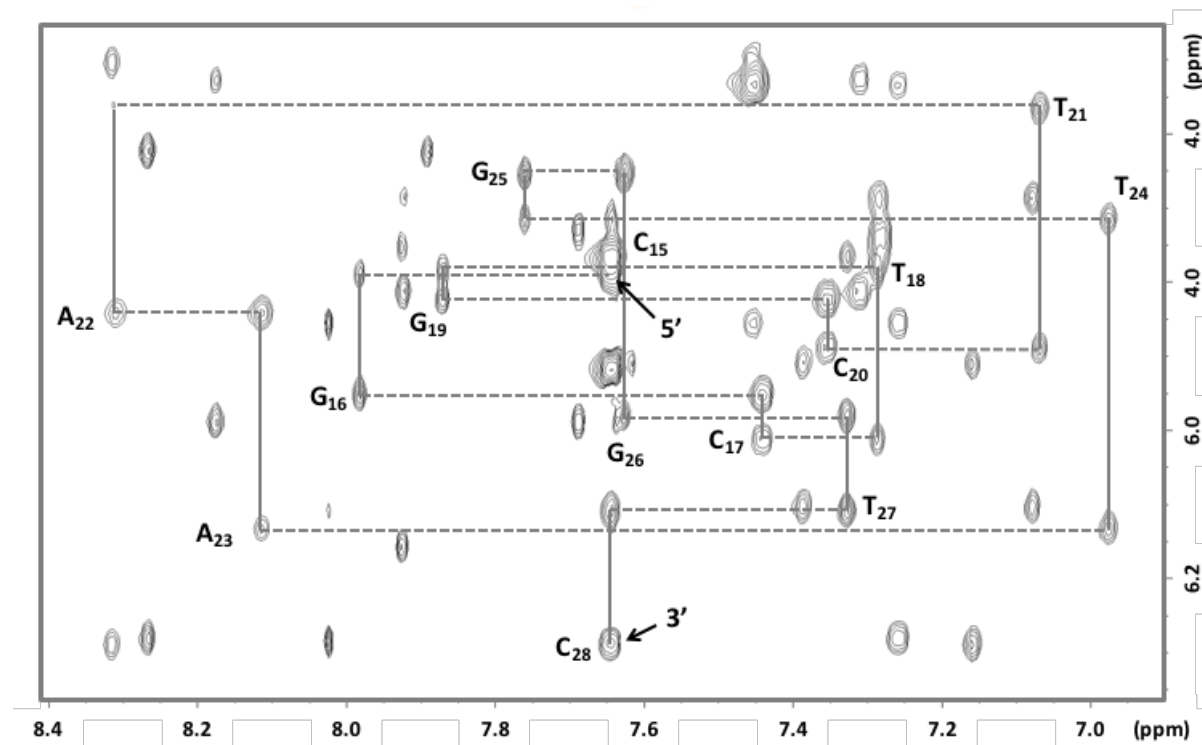
<sup>15</sup>P  
Phosphorus

# EXPERTISE

## Structure elucidation and 3D structure determination of biomacromolecules

A. Madder and J.C. Martins

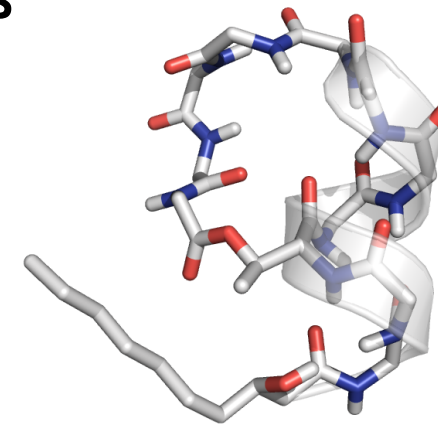
Cyclic lipodepsipeptide & chemically modified aptamer research topics



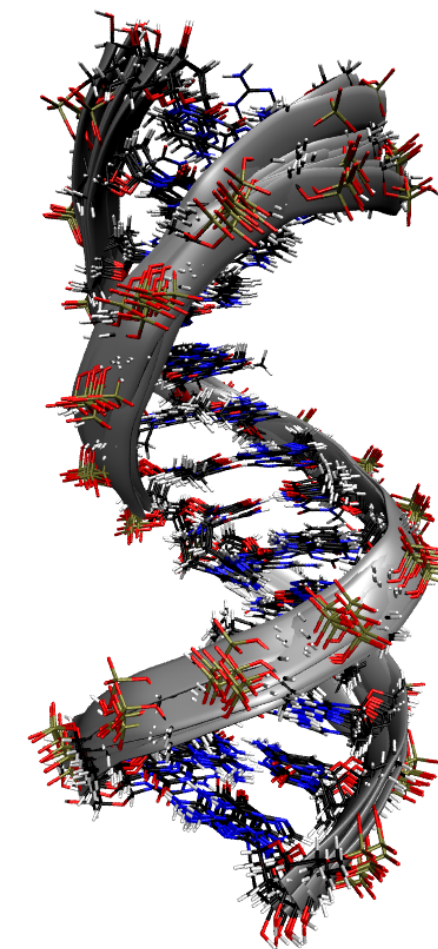
2D  $^1\text{H} - ^1\text{H}$ ,  $^1\text{H} - ^{13}\text{C}$  spectra

&

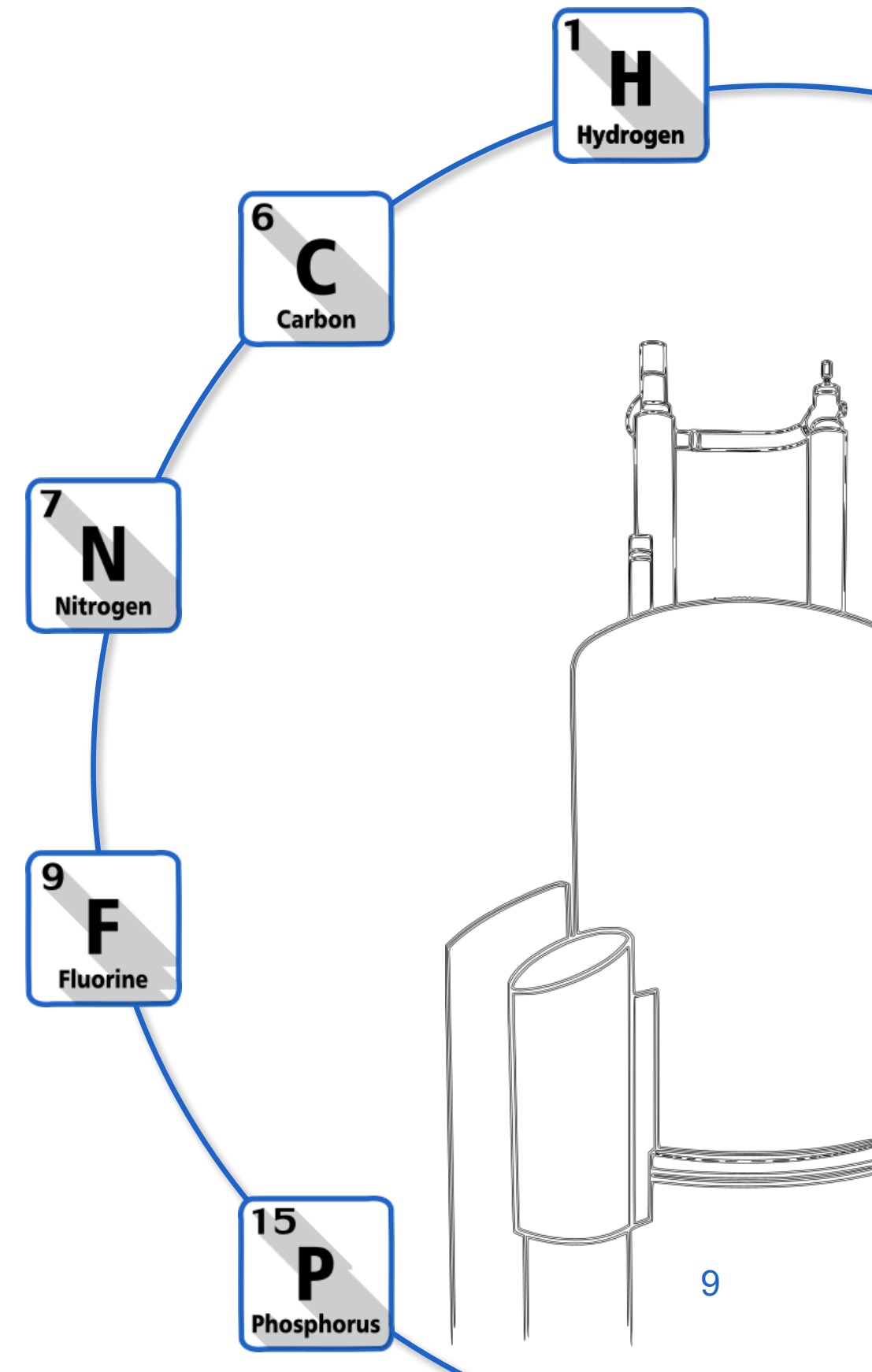
Computer assisted  
model building



Peptides



Nucleic acids





# EXPERTISE

## hr-MAS as a tool to study polymer networks

F. Du Prez & B. Fritzing

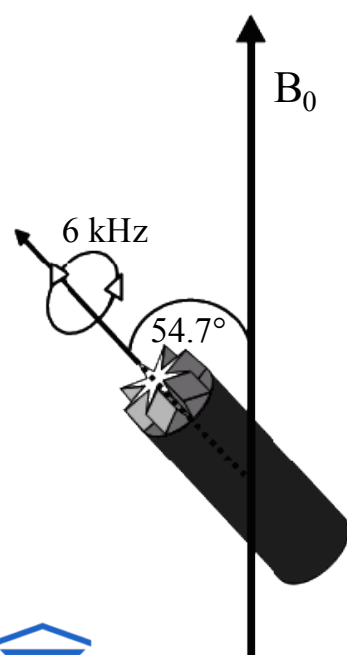
hr-MAS NMR to monitor and quantify chemical modifications of cryogels

High resolution NMR  
> Samples in solution

Magic Angle Spinning (MAS) NMR  
> Solid materials as powder

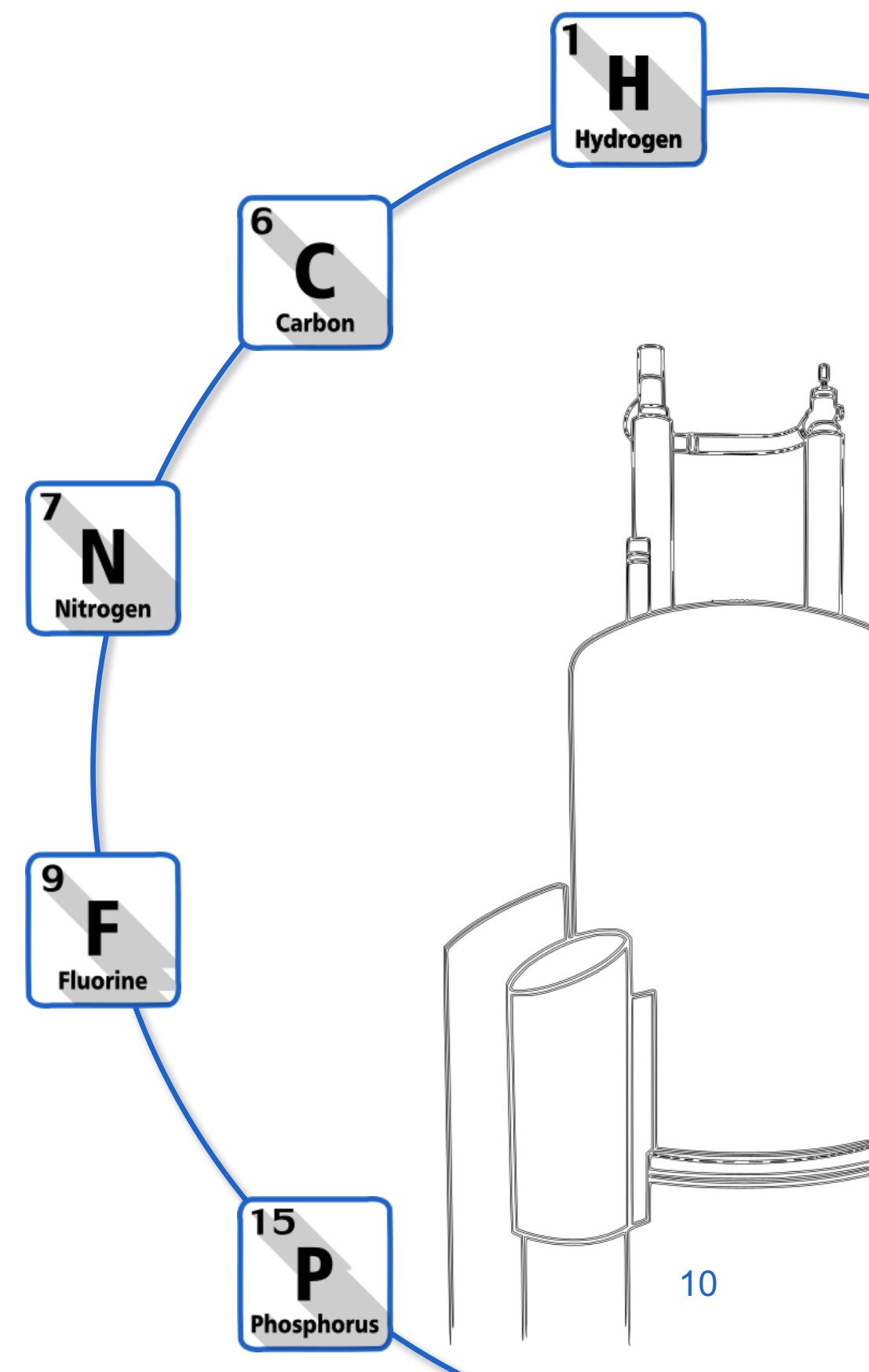
### High resolution MAS (hr-MAS) NMR

> Materials at the interface of solution and solid state



Sample in rotor is spun at high frequency under the magic angle

This allows the application of most high-resolution techniques to  
characterize molecules grafted onto insoluble supports  
= interfaces, networks, matrices

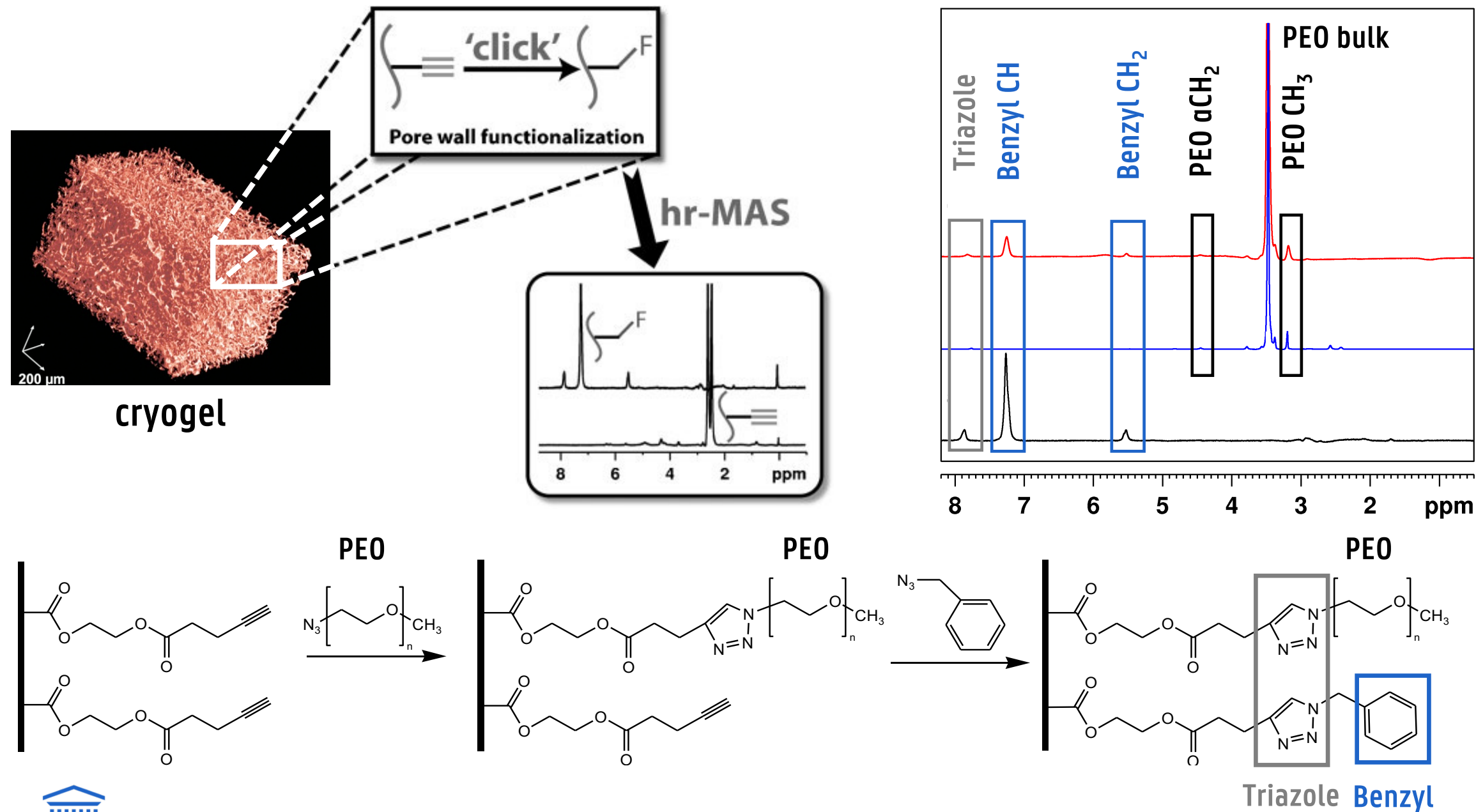


# EXPERTISE

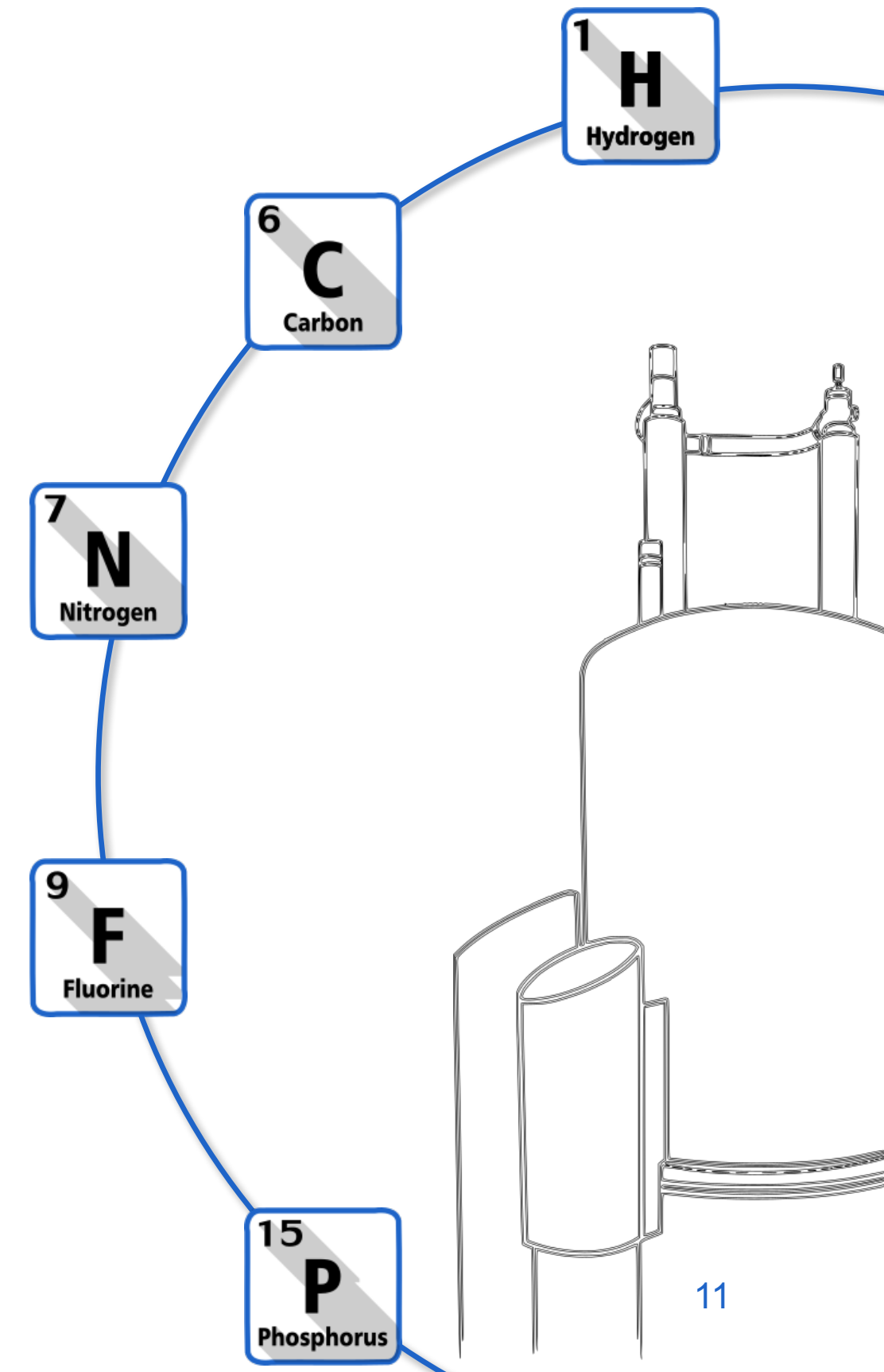
## hr-MAS as a tool to study polymer networks

F. Du Prez & B. Fritzing

hr-MAS NMR to monitor and quantify chemical modifications of cryogels



Quantifying multistep reactions in 'solid-like' materials



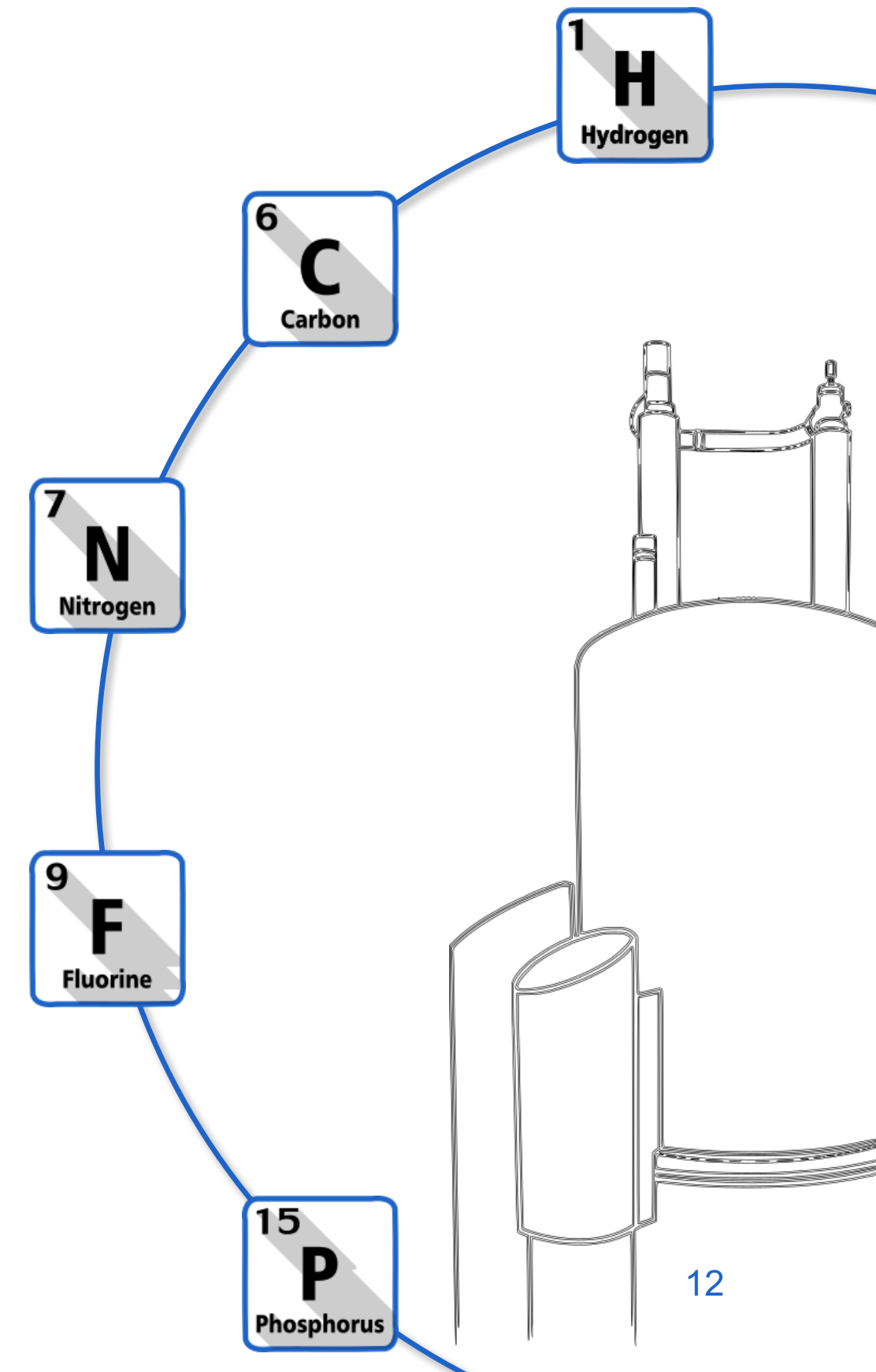
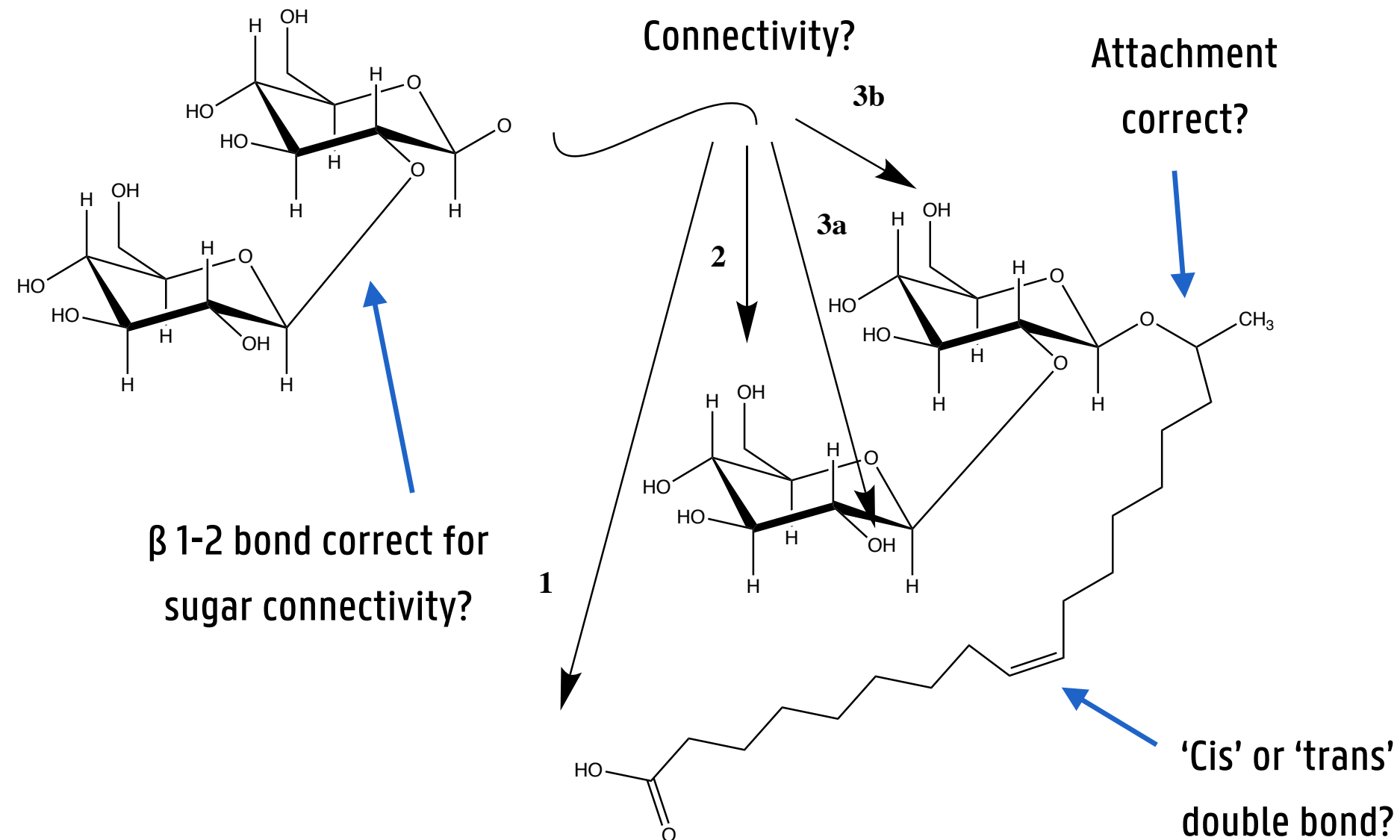
# EXPERTISE

## Structure elucidation of surfactants

I. Van Bogaert

Synthesis of bolaform surfactants by an engineered *Starmerella bobicola* yeast

- How is the aliphatic tail attached to the sugar entity 'A,B'?
- How is sugar entity 'C,D' attached to the main part of the molecule and what is their mutual connectivity?
- What is the configuration of the aliphatic tail double bond?
- What are the anomeric configurations of the four sugar rings?



# EXPERTISE

## Structure elucidation of surfactants

I. Van Bogaert

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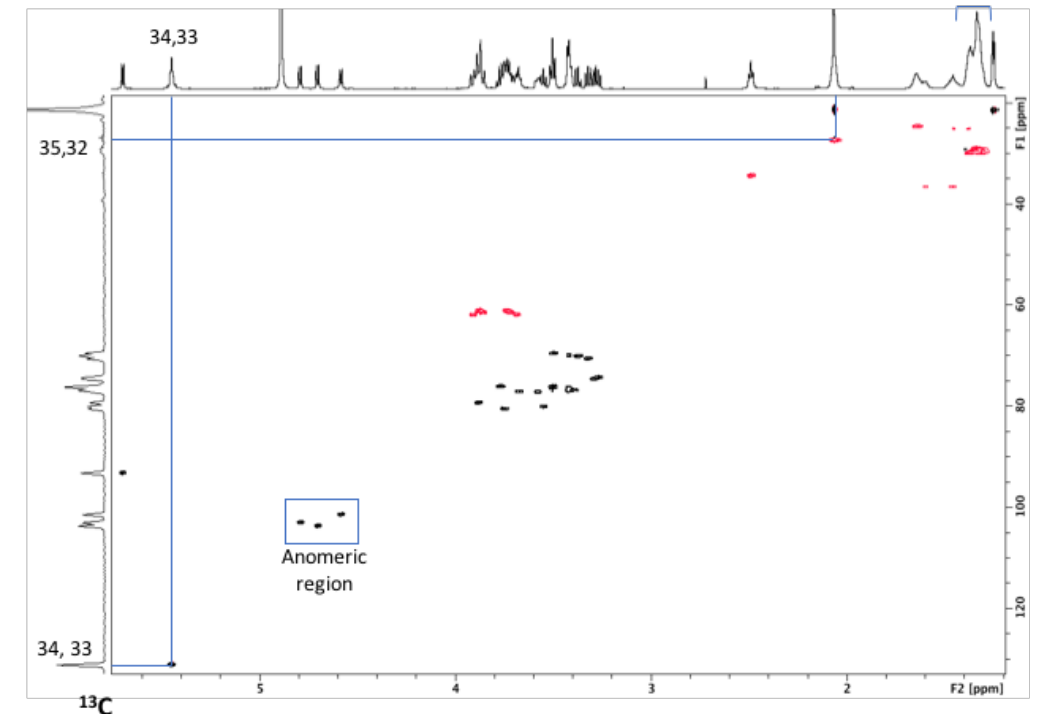
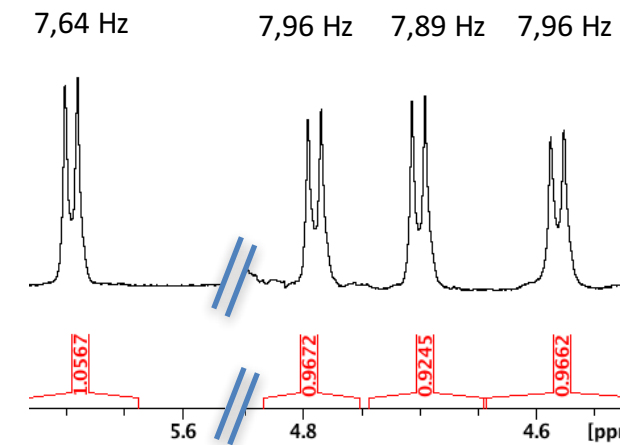
Correct!

Anomeric  $^1\text{H}$  scalar couplings prove overall  $\beta$  configuration of all sugars

Connectivity proven!

'Cis' !

$\beta$  1-2 bond correct!



$^1\text{H}$ - $^{13}\text{C}$  assignment shows connectivities of different sugars &  $^{13}\text{C}$  chemical shift analysis shows orientation double bond in the aliphatic tail

$^1\text{H}$   
Hydrogen

$^{13}\text{C}$   
Carbon

$^{15}\text{N}$   
Nitrogen

$^{19}\text{F}$   
Fluorine

$^{31}\text{P}$   
Phosphorus

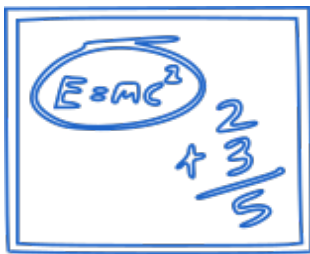
# SOME KEY NUMBERS

## Service provided so far within UGent

>250



12



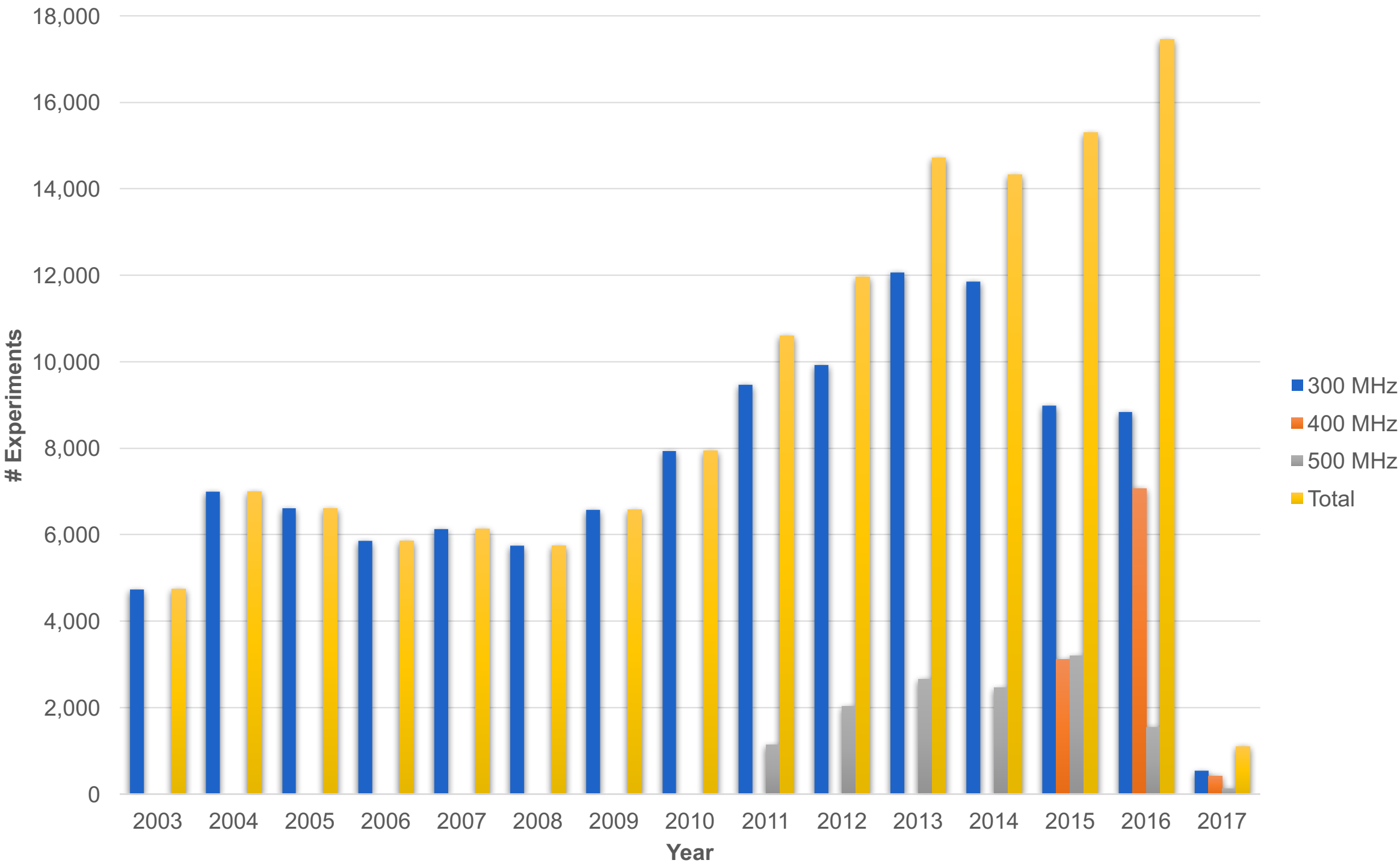
Research groups

3

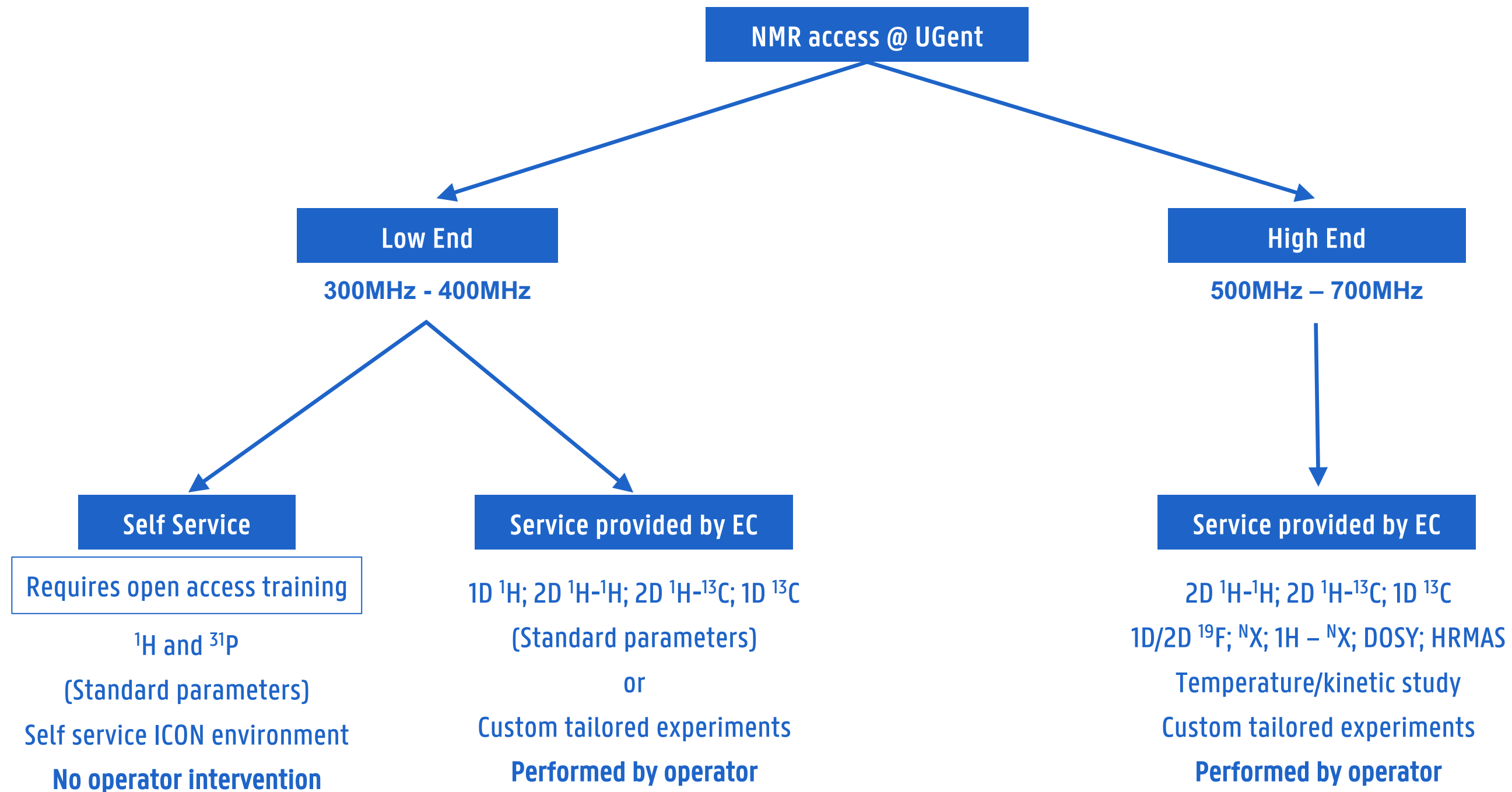


Faculties

# Experiments over the years







# SERVICES FOR INDUSTRY



## What

Any soluble material that has at least one NMR active isotope (spin  $>0$  and preferably  $= 1/2$ )



## Our offer

- Expert team with  $> 20$  years of experience
- State-of-the-art infrastructure
- High sample throughput in routine applications
- Standardized or highly customized measurements
- NMR support from low to high-end applications
- Training for basic to advanced spectral interpretations (1D – 2D techniques)



## Pricing

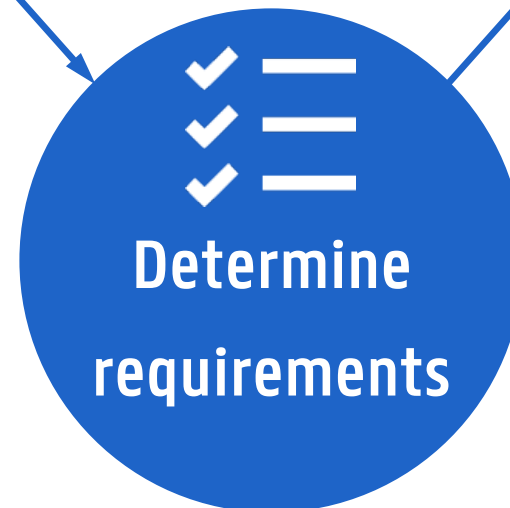
Please contact us to receive a customized quote

# ANALYSIS REQUEST FLOW

1. Client defines the problem or questions to be solved



2. During a meeting or call the requirements for the measurements or analysis are discussed



3. Based on the requirements, a custom agreement is made



4. The client formally agrees with the offer and the samples are made available



5. NMR analysis is performed and a report is drafted



6. Following the report, the results can be elucidated in detail and feedback is given



# TOOLBOX



## Bruker 700MHz Avance II

“Hera” – INMRF infrastructure

Liquid state spectrometer

Three channel probes

5mm Prodigy N<sub>2</sub> cryoprobe

5mm TXI (<sup>1</sup>H; <sup>13</sup>C; <sup>15</sup>N)

5mm TBI (<sup>1</sup>H; <sup>119</sup>Sn; BB)

4mm HR-MAS (<sup>1</sup>H; <sup>13</sup>C; <sup>119</sup>Sn)

1mm TXI (<sup>1</sup>H; <sup>13</sup>C; <sup>15</sup>N)

Topspin 3.2

Sample changer (60 samples)

## Bruker 500MHz Avance III

“Hercules”

Liquid state spectrometer

Two channel probes

5mm BBI (<sup>1</sup>H; BB)

5mm High Gradient Probe

Topspin 3.2

Sample changer (60 samples)





# TOOLBOX



## Oxford 500MHz Avance II

“Nestor”

Liquid state spectrometer

Three channel probes

5mm TXI ( $^1\text{H}$ ;  $^{13}\text{C}$ ;  $^{31}\text{P}$ )

5mm TBI ( $^1\text{H}$ ;  $^{31}\text{P}$ ; BB)

5mm TXO ( $^1\text{H}$ ;  $^{13}\text{C}$ ;  $^{19}\text{F}$ )

Topspin 3.2

Sample changer (60 samples)

## Bruker 400MHz & 300MHz

Avance II – Avance I

“Phobos” and “Deimos”

Liquid state spectrometers

Three channel probes

5mm BBO ( $^1\text{H}$ ; BB)

Topspin 2.1 in ICONNMR

Sample changers (60 samples each)





# TEAM

A triad of experts at your disposal



Dieter Buyst, PhD

Chief Scientific Officer (CSO)

- Point of contact
- Analysis & Measurements
- Management & Organization
- Training



Prof. José Martins

NMRSTR Group leader

- Scientific support

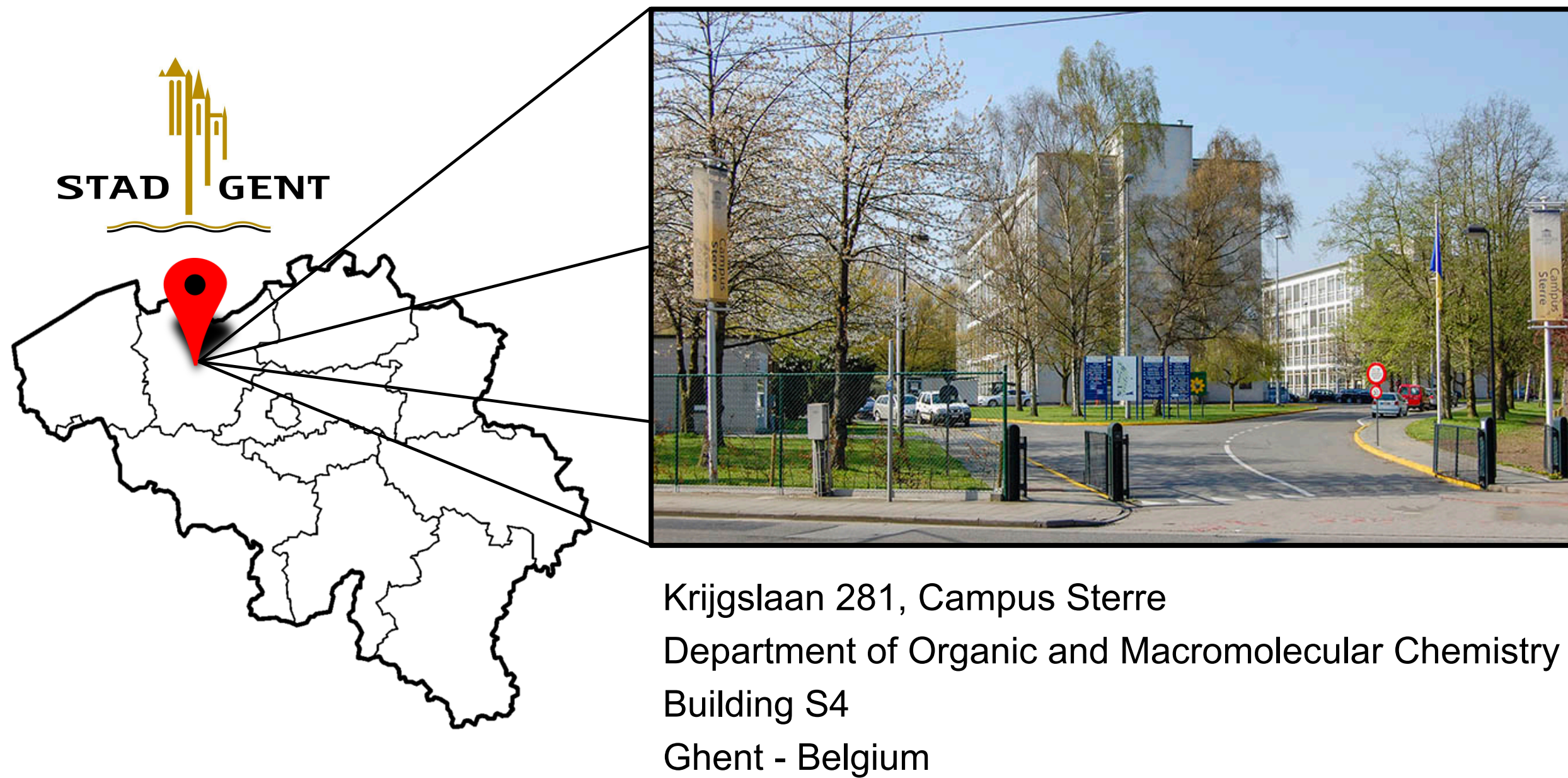


Timothee Courtin, Ing.

Chief Technical Officer (CTO)

- Technical support
- Routine Measurements
- ICT infrastructure

# LOCATION



## Dieter Buyst, PhD

Chief Scientific Officer

### NMR EXPERTISE CENTRE

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M +32 474 54 12 56

[www.ugent.be](http://www.ugent.be)



Ghent University



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