VITO Laser Polarization Setup and bio β-NMR

Lina Pallada,

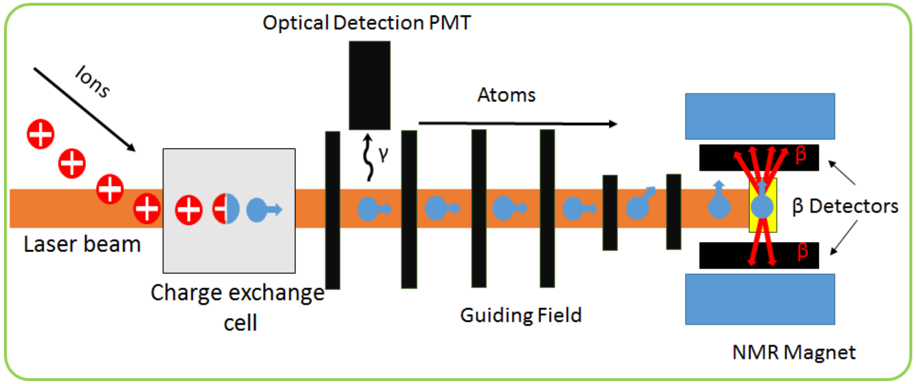
ISOLDE Workshop and Users meeting 2016,

9 December 2016

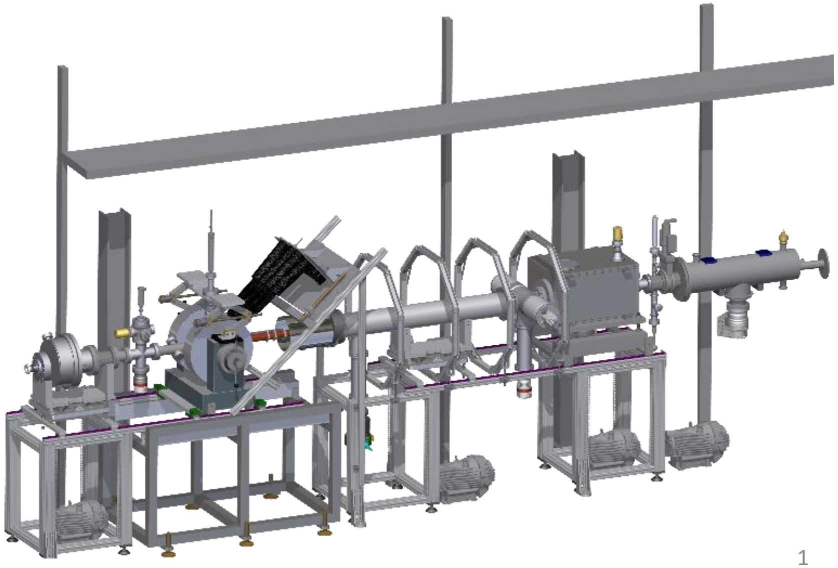
Why Spin Polarized Nuclei?

Growing interest

- ❑ Nuclear structure studies
- ❑ Fundamental Interactions
- ❑ Material science
- ❑ Life Science
- ❑ Medical imaging



Motivation for building a dedicated laser polarization setup
We are interested in using spin polarised beams for β-NMR in biology



- ➢ Polarisation setup design and simulations for the B field completed in the beginning of 2016
- ➢ Setup commissioning summer/autumn 2016

Laser Spin Polarization and β-asymmetry

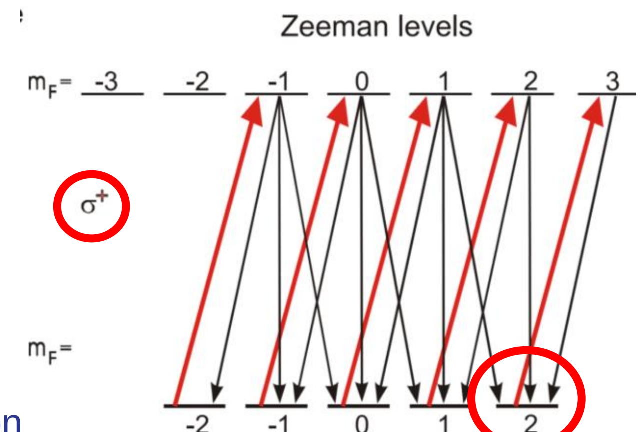
Optical Pumping

1. Polarization of atomic spins (F)

- Atoms/ions overlap with laser light
- σ+ (ΔmF=+1) and σ- (ΔmF=-1) circular polarised laser light and decay ΔmF = 0, ± 1
- Repeat pumping-decaying cycle

2. Polarization of nuclear spins (I)

- Decoupling of atomic and nuclear spins with increasing B field
- The strong B field maintains the nuclear spin polarization



Optical pumping via σ+ of F=2 → F=3

Most populated after many excitation/decay cycles for σ+

B-decay asymmetry

B-decay of spin polarized nuclei anisotropic in space due to parity violation of weak interactions

Angular distribution of β-radiation

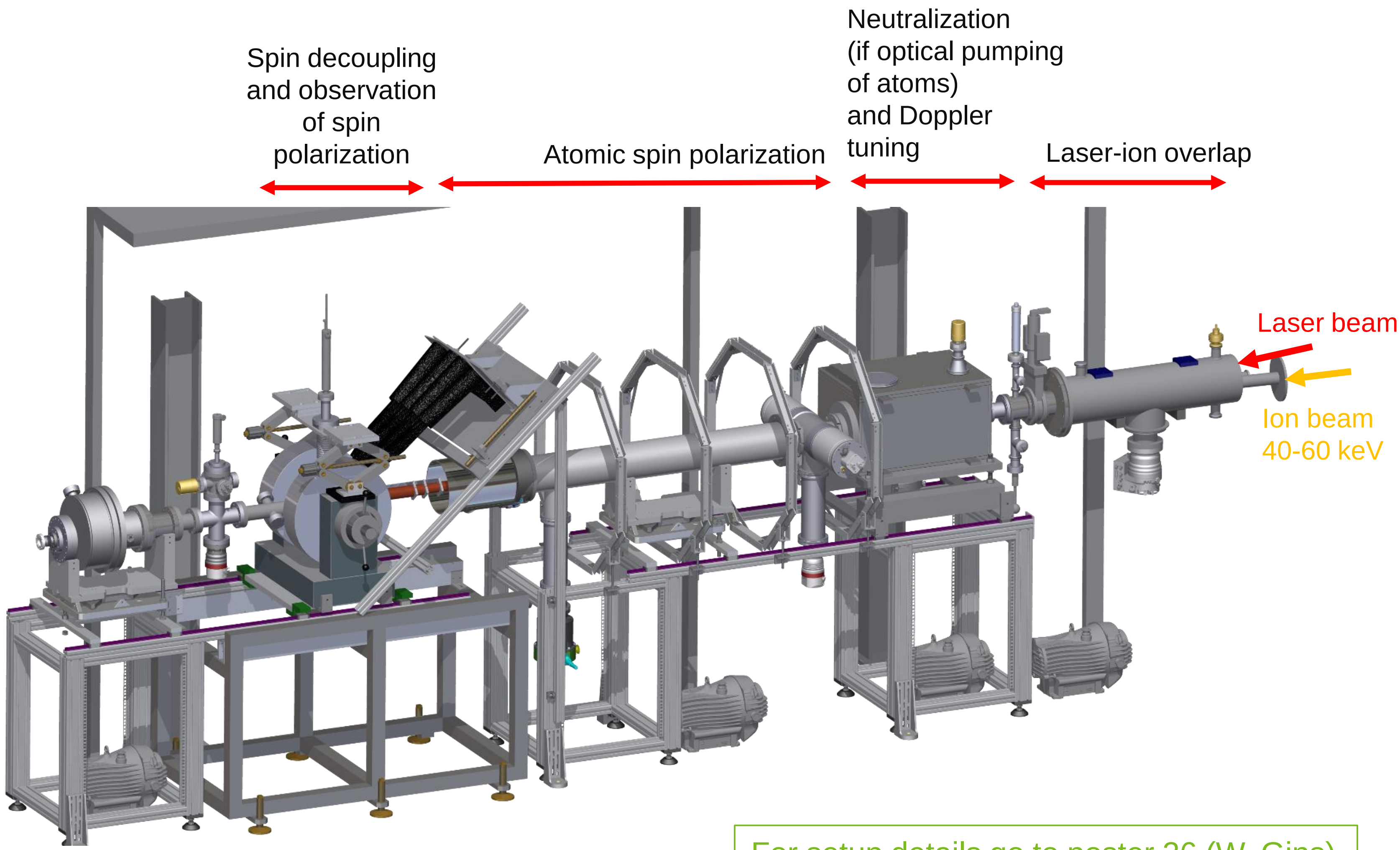
$$W(\theta) = 1 + a_\beta * \frac{v}{c} * P_I * \cos\theta$$

a_β: asymmetry parameter
v: velocity of β particle
θ: angle between I and particle emission

Measured β-decay asymmetry

$$\boxed{A_{exp} = \frac{N_0 - N_{180}}{N_0 + N_{180}}}$$

New polarisation setup at ISOLDE

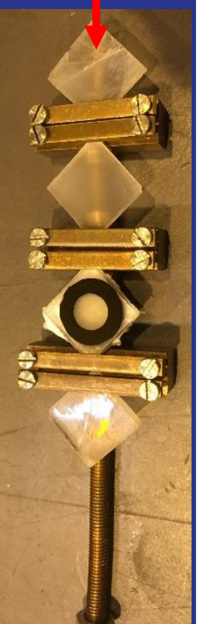


For setup details go to poster 26 (W. Gins)

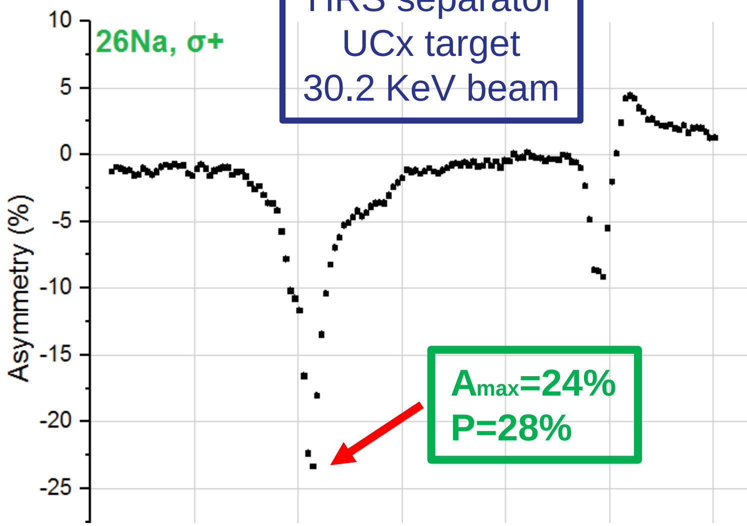
Designed by F. Wienholtz, M. Bissell, W. Gins in 2016

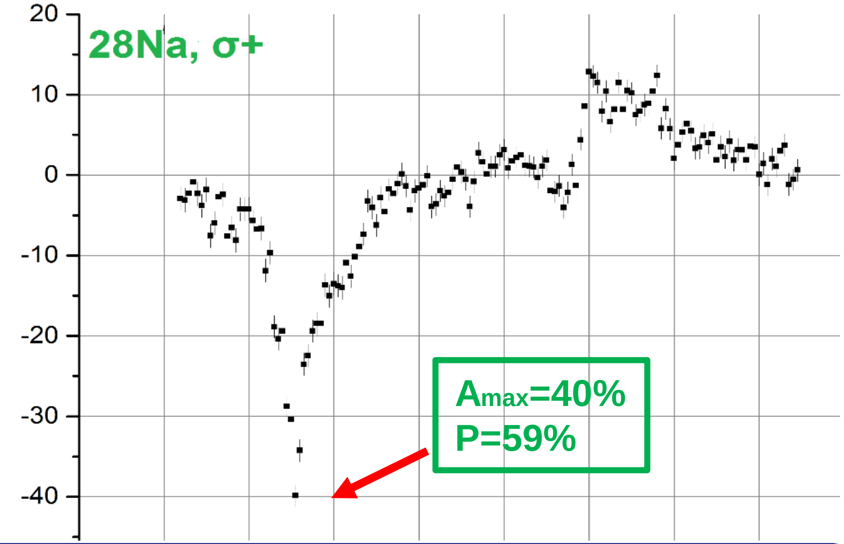
26,28 Na commissioning results
14 Nov 2016

NaF crystal



HRS separator
UCx target
30.2 KeV beam

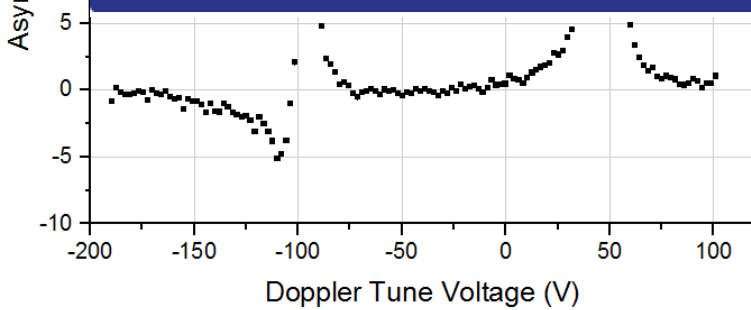


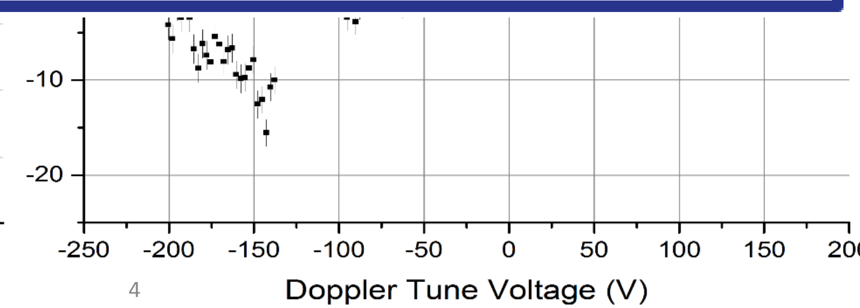


Goal of the 3 short commissioning runs:
Maximize the degree of polarization after implementing improvements to the setup achieved!

Results:
Polarization comparable to that achieved at COLLAPS

Commissioning of Laser Polarization Setup Successful



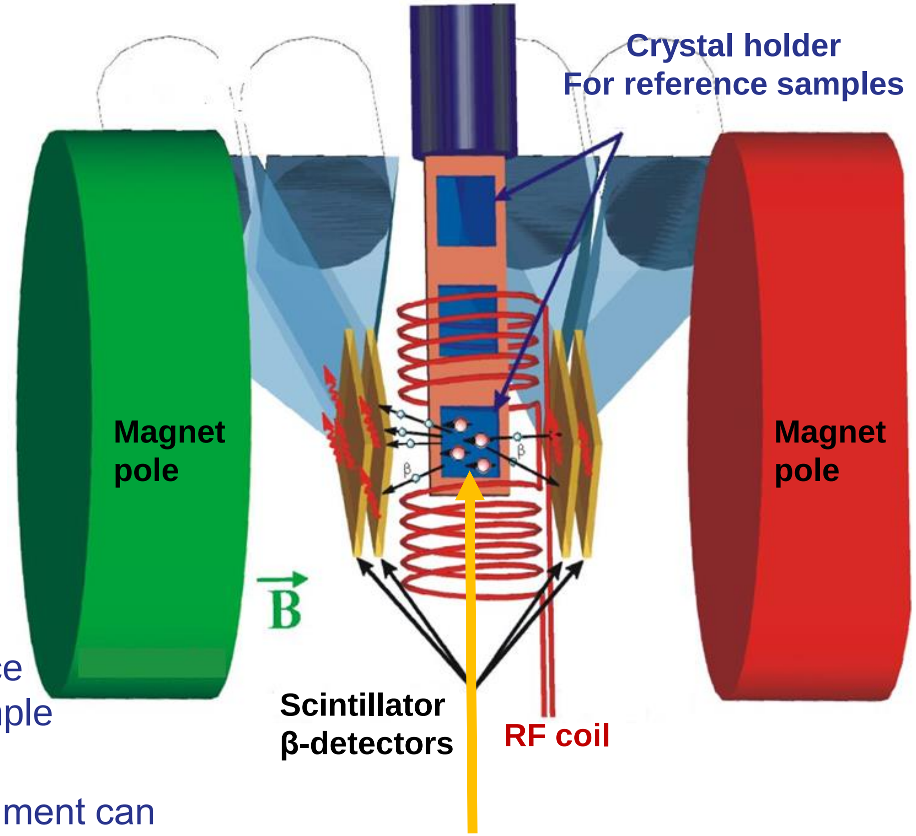


β-NMR introduction

Why β-NMR?

Main differences between β-NMR and conventional NMR

- Detection of β-particles instead of RF absorption
- <u>9 orders of magnitude more sensitive</u>, less probe nuclei needed (10E7 vs 10E16)



β-NMR setup and Observables

- β-NMR setup placed in the end of the polarization setup
- In the implantation site, RF coil destroys the nuclear polarization
- We observe the asymmetry loss
- Measure the Larmor frequency

$$\omega_L = \gamma * B$$

Chemical shift = difference in the resonance frequency of a nucleus compared to ref sample

Information about nucleus' chemical environment can be derived

$$\boxed{\delta = \frac{\nu_{sample} - \nu_{ref}}{\nu_{ref}} \times 10^6 \text{ [ppm]}}$$

β-NMR towards liquids

Radioactive beam from ISOLDE → (Ion beam) → Experimental setup for optical pumping at VITO → (Polarized beam) → NMR magnet and chamber for liquids

Challenges

Radioactive beams like high vacuum while most liquids don’t

1E-06 mbar → (Ion beam) → 1E-06 mbar → (Polarized beam) → Water: 1-10 mbar

Future plans and solutions

Radioactive beam 1E-06 mbar → (Ion beam) → optical pumping 1E-06 mbar → [Differential pumping / thin foil] → NMR magnet and chamber for liquids

Aims

Resolve the chemical shift

ionic liquid → biomolecule in ionic liquid → biomolecule in water

Sample preparation for β-NMR in liquids

University of Copenhagen-Chemistry Department

- More than 20 different samples were tested
- The sample preparation conditions were optimised

Solvents used for the sample preparation: Ionic Liquids (IL's)

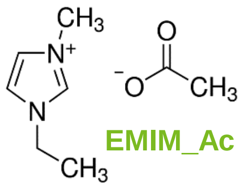
What IL's are?

IL is a salt in which the ions are poorly coordinated → these solvents are liquid below 100 oC even at **room temperature**

Why do we use IL's?

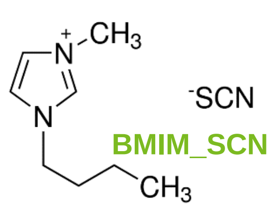
- They remain liquids at room temperature
- They have very low vapour pressure → No need for bad vacuum (no differential Pumping needed)

Which IL's?



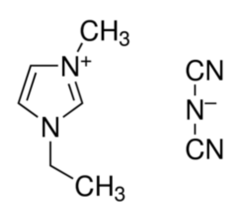
EMIM_Ac

1-ethyl-3-methylimidazolium acetate



BMIM_SCN

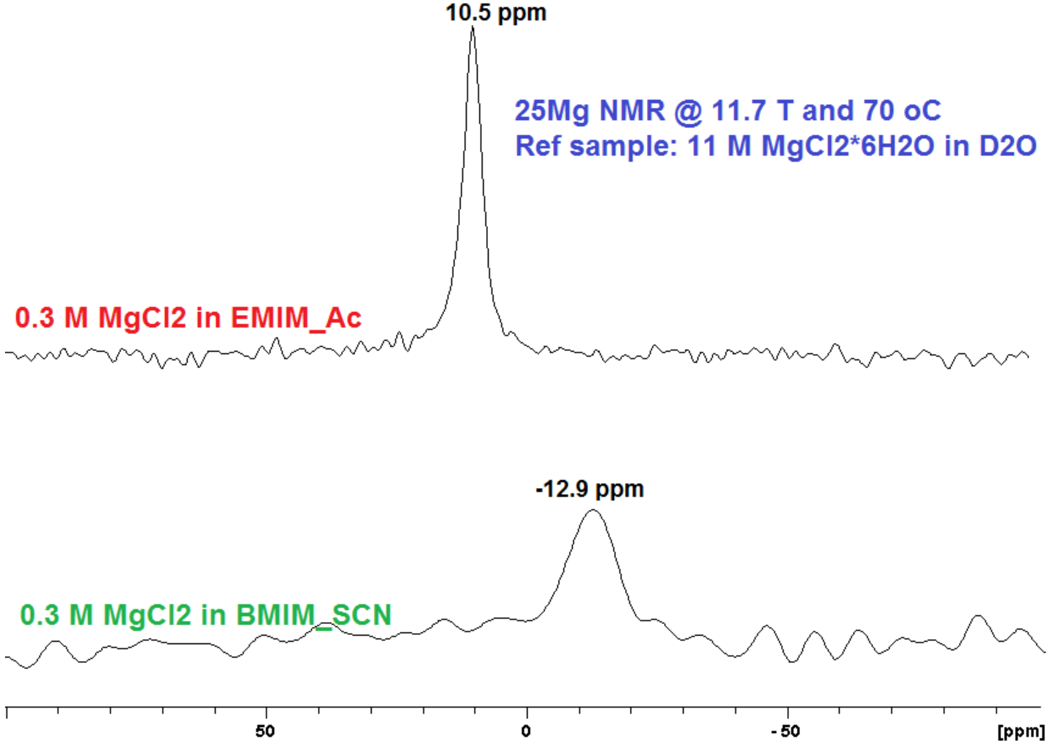
1-butyl-3-methylimidazolium thiocyanate



EMIM_DCA

1-ethyl-3-methylimidazolium dicyanamide

25Mg (I=5/2) conventional NMR

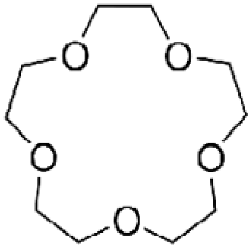


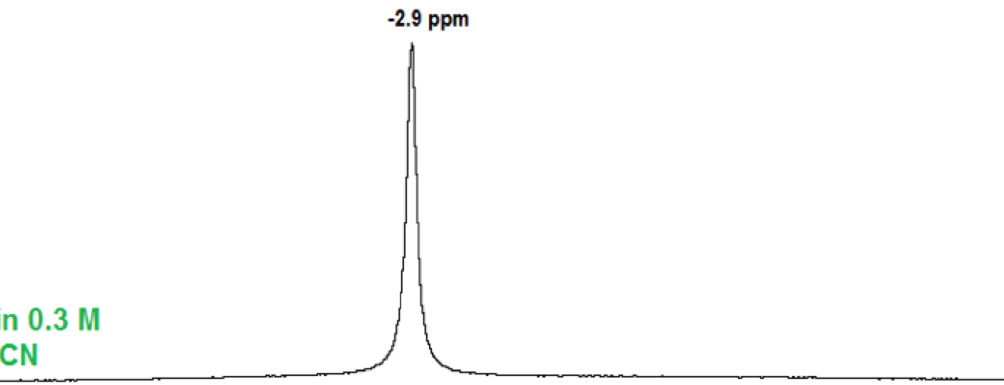
- ❑ Δδ=~20 ppm difference in the chemical shift of MgCl2 in EMIM_Ac and BMIM_SCN
- ❑ Different chemical environments of Mg nucleus.
- ❑ More asymmetric environment in BMM_SCN due to broaden of the peak

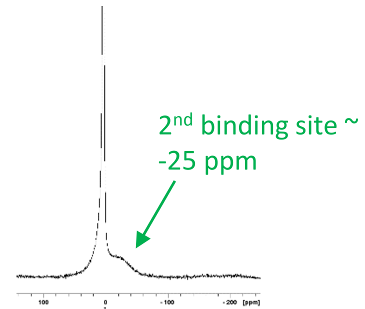
Aims for β-NMR experiments

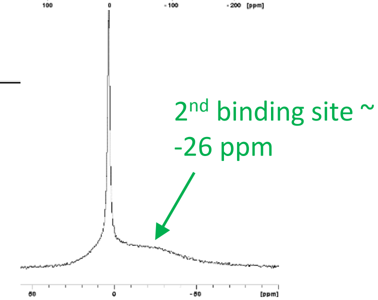
- ❑ Find the mixture ratio of the two solutions in order to observe two distinct resonances in the same NMR spectrum
- ❑ Use this mixture for β-NMR experiments to test if at 0.6 T the resolution of our system is enough to resolve the two distinct resonances
- ❑ Find an appropriate biomolecule (i.e. protein, RNA) that can be dissolved in the IL's, bind with Mg and give a different chemical shift

23Na (I=3/2) conventional NMR

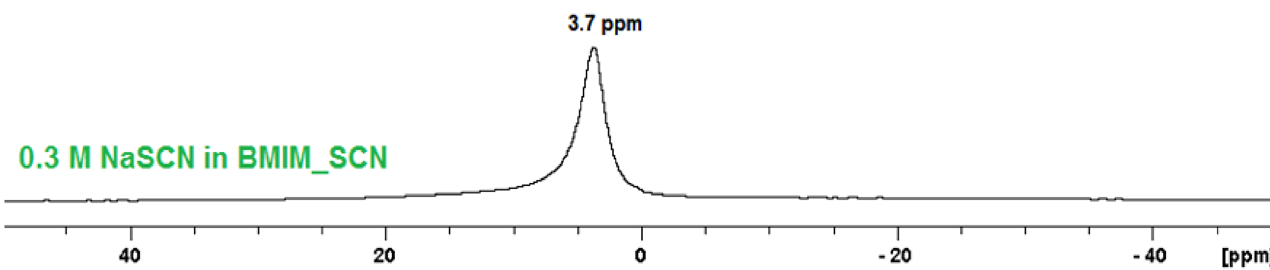


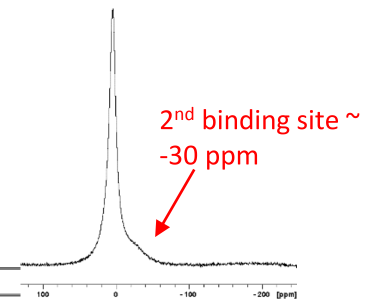






When a crown-ether was added shifted the chemical shift downfield→ better shielding of Na as expected

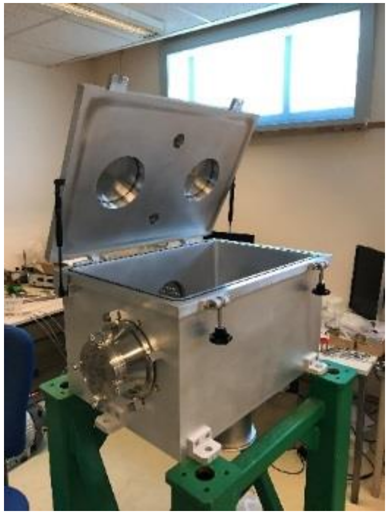




- ❑ Na seems to have two different binding sites in EMIM_Ac and BMIM_SCN, predominant site at ~3.5 ppm
- ❑ In different environments of Na we observed a range of chemical shifts from **3.3 ppm to – 2.9 ppm**

Outlook

- Work on the good and bad vacuum interface
- Preparation of the setup (chamber and RF coil) to see the chemical shift in good and bad vacuum
- Put into place the new CEC (designed by F. Wienholtz)
- Improve the B homogeneity of the magnet (shimming plates)
- Upgrade the beamline setup to work with ions
- Get 31Mg ions into resonance with laser
- Test polarization of Cu



Laser polarization setup picture provided by R. Harding

CEC pictures provided by F. Wienholtz

Acknowledgements

Laser polarization core group


M. Kowalska


F. Wienholtz


M. Bissell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Other collaborators

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NMR in biology


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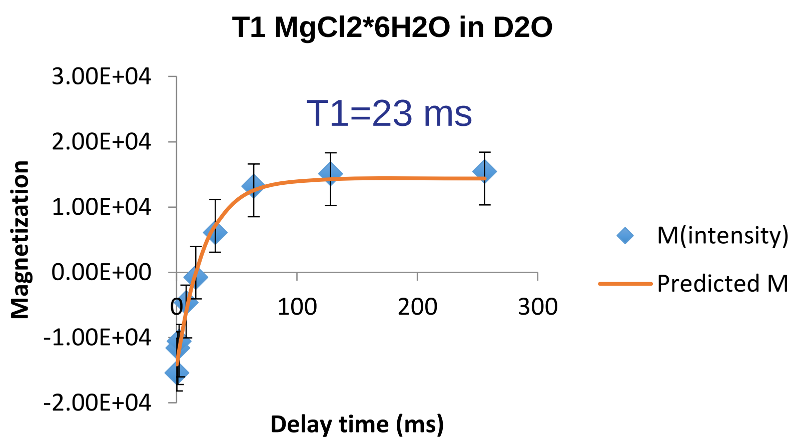
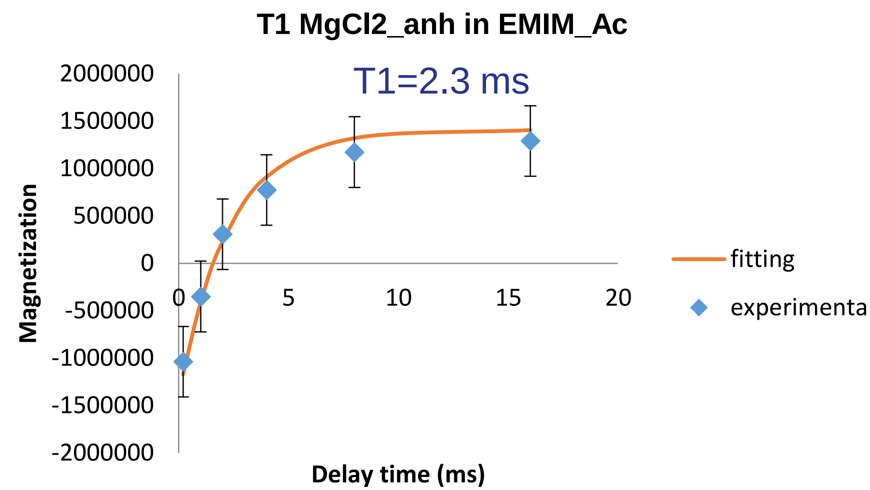
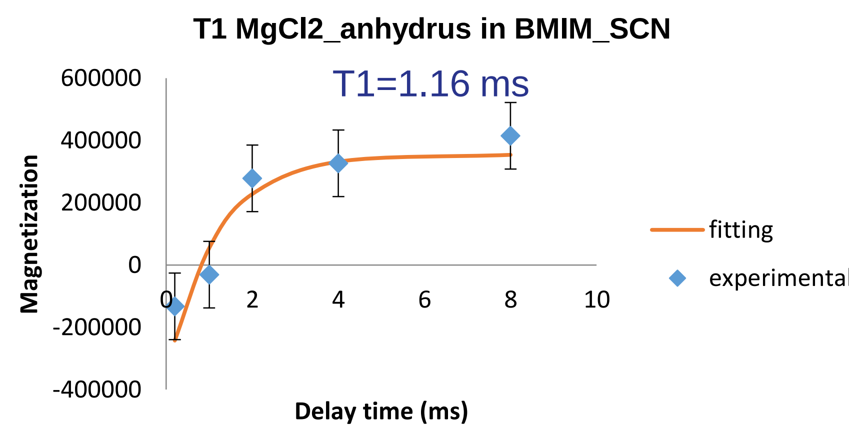
T1 relaxation times for 25Mg and 23Na

$M(\tau)=M_o[1-2\exp(-\tau/T_1)]$







23Na T1 could not be defined

Probably too short <1 ms because 23Na is quadrupolar

Why 26,28Na and 31Mg as test cases

1. 26,28Na easily polarised as atoms and 31Mg easily polarised as ion. Test different setup configurations
2. They are both tested previously at COLLAPS
3. Both Na and Mg biologically interesting elements (for β-NMR in liquids)

26,28 Na characteristics

- 26Na (3+) $T_{1/2}$=1 s, 28Na (1+) $T_{1/2}$=30.5 ms
- easily polarised as atoms in the atomic D2 transition from gs
- For neutralization need CEC
- Relatively easy laser system
- Well produced from ISOLDE targets (only hot surface ion-source required)
- Very high degree of β-asymmetry

31Mg characteristics

- 31Mg (I=1/2), $T_{1/2}$=230 ms
- polarised as ions in the atomic D2 transition from gs
- No need for CEC
- More complicated laser system (need UV light)
- Well produced from ISOLDE targets (RILIS required)
- High degree of β-asymmetry

M. Bissel et all, Letter of Intent to the INTC 2016

Information of 26Na, 28Na and 31Mg for laser polarization

	26Na	28Na	31Mg
Spin	3	1	½
Half life	1.1 s	30 ms	232 ms
Beta asymmetry parameter	-0.93(2)	-0.75	-
Yield/s [Ref]	3E07	6E05	1.5E05
Nuclear polarization at COLLAPS [Ref]	39%	59%	
Nuclear polarization achieved 2016	28%	59%	-

Lower polarization of 26Na compared to 28Na mainly due to the higher nuclear spin

M. Kein et al, European Physical Journal A 1 (200), 31

ISOLDE

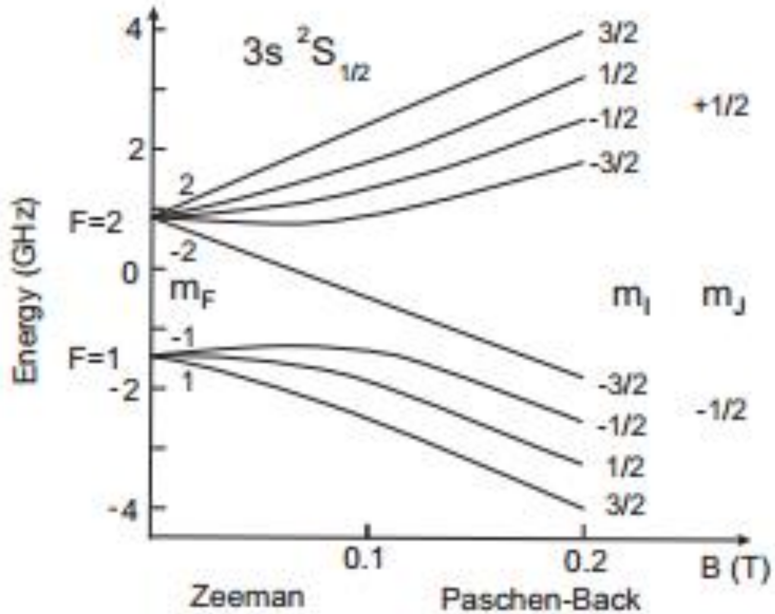


Figure 4.3: Behaviour of the ground state hyperfine structure of ^{29}Mg for weak and strong magnetic field ($I = 3/2$, $\mu_I > 0$).