

# Laboration 1

## Introduction

<sup>1</sup>H-NMR, tune and match, shimming

KR

## Teori

### Tune and match

A central component of a *probe* is a small *coil*, which surrounds the sample when injected to the magnet. The coil should send and receive signals in the best way. Electric parameters in the circuit, that the coil is a part of, must be optimized for every sample. These optimizations are called *tuning* and *matching*.

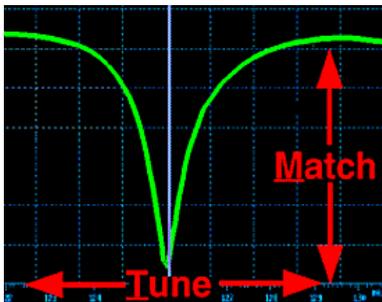
To tune is to set the probe on the right frequency for the nucleus you want to study. You can see it as scanning for a radio channel.

To even out the impedance in the coil/sample you match, so that maximal energy can be transferred to the sample.

Tuning and matching are dependent of each other and must therefore alternately be optimized. This must be done for every nucleus involved in the experiment separately.

Some of the parameters affecting tune and match are temperature, solvent and salt content.

Optimal sensitivity and a minimisation of the pulse angel are what you gain with these corrections.

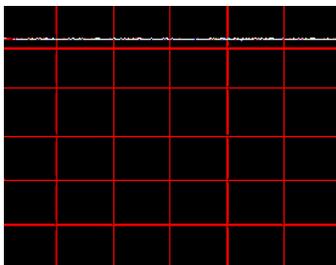


### Lock

The strong magnetic field in a NMR-spectrometer is changing over time. This affects the resolution and the sensitivity in an experiment.

In short experiments this effect is negligible but in longer you have to “lock”. You lock on a deuterium signal from the solvent.

This is done by continually follow the  $2\text{H}$  signal with  $2\text{H}$ -NMR experiments.



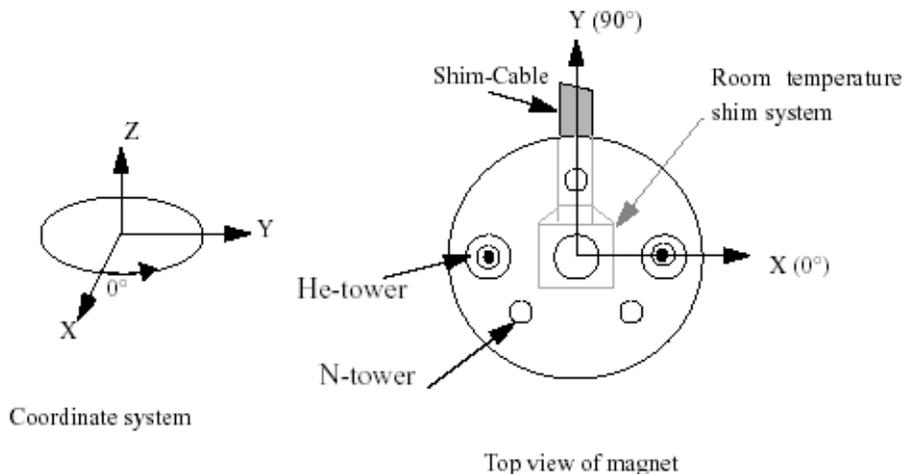
### Shimming

In NMR-spectroscopy small changes in for example the static magnet field ( $B_0$ ) cause faults that are bigger than the signals that shall be detected. One of these faults is in homogeneity of  $B_0$ , which can cause broad and distorted peaks.

The NMR-spectrometer is equipped with coils, called shims.

Voltage can be put on these shims that induce small magnetic fields to correct irregularities in  $B_0$ . A NMR-spectrometer contains a variety of shims that affect  $B_0$  in different ways.

The field axes used to designate the different shim coils are defined such that the Z direction lies along the sample tube axis. X and Y lie in the transverse plane with Y oriented along the shim cable as shown in the Figure.

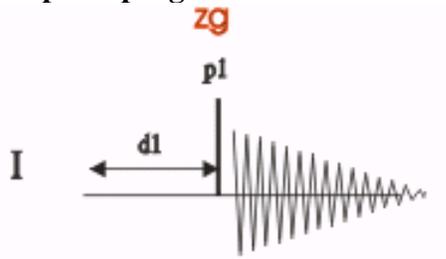


To obtain an optimal magnet field homogeneity the shims are changed until a maximal lock level is obtained or and the peaks looks good.

The lock level, then, serves as useful guide for basic shim adjustment, thou the lock level corresponds to the lock substance signal amplitude.

See, appendix "Shimming"

<sup>1</sup>H pulse programme



A standard 1-D experiment would use a 90 degree pulse to excite the NMR signal to maximize the emitted signal.

However, this also maximizes the duration that must elapse between successive pulses in order for the sample to relax. It can be shown that in the case of repeated excitation of a sample, a more efficient method is to excite the sample with a 30 degree pulse and reduce the relaxation delay accordingly. Although each individual emitted signal is weaker, the faster accumulation of data that the 30 degree pulse allows results in an overall increase in sensitivity.

The two standard programs are "zg" for a 90 degree excitation pulse and "zg30" for a 30 degree excitation pulse.

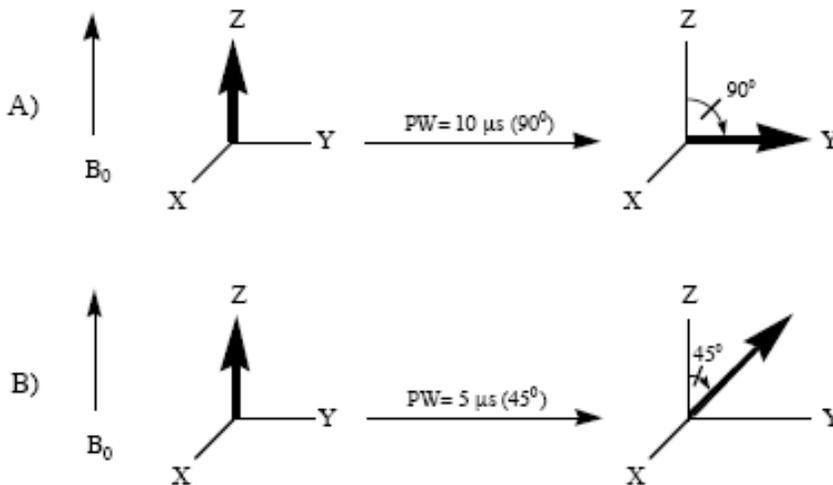


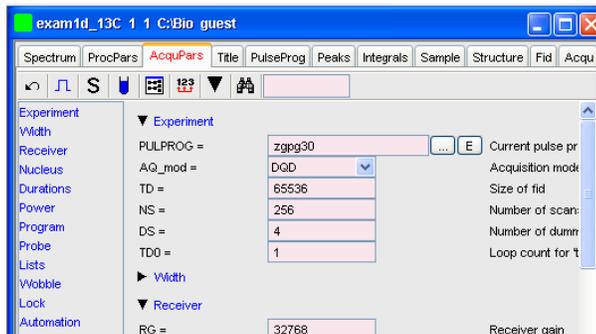
Figure 2. The average nuclear spin magnetization (bold arrow) for an NMR sample placed in a magnetic field aligned along the Z-axis before and after application of a pulse.

## Utförande

### 1H

1. Run a *PROTON* according to "Bruker run manual for 500 MHz NMR".

If required, any acquisition parameter can be modified manually or in the *AcquPars* section, you can see what is valid for the parameters in *PulseProg*.



Check the parameters for the current experiment. Look especially on the parameters named below.

- PULPROG** (the pulse-programme), in this experiment *zg* or *zg30*
- sw** (spectral frequency window). Choose a suitable value for the spectrum.
- o1p** is the center of the 1H spectrum.
- ns** (number of scans)
- d1** (delay time)

*\*Why do you set the receiver gain (rga)? How does it affect the result if the receiver gain level is too high or too low?*

- Process the spectra according to the "Bruker 1D processing NMR-manual for  $^1\text{H}$  and  $^{13}\text{C}$ "

*\*What happens and how does a zero-filling affect your spectrum?*

*\*How can you manipulate your spectra with window functions?*

*\*What affect the accuracy of your integrals?*