Simultaneous determination of water and fat by TD-NMR

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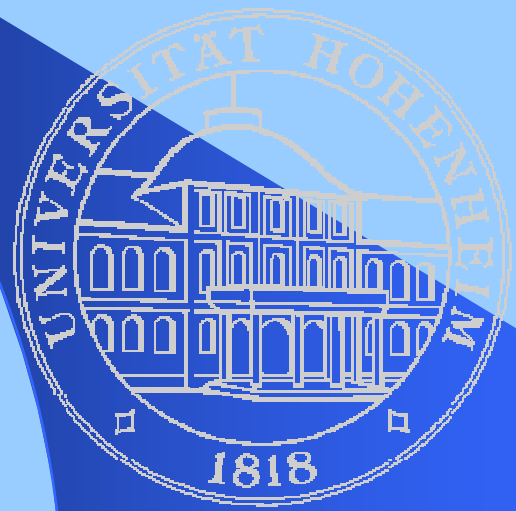
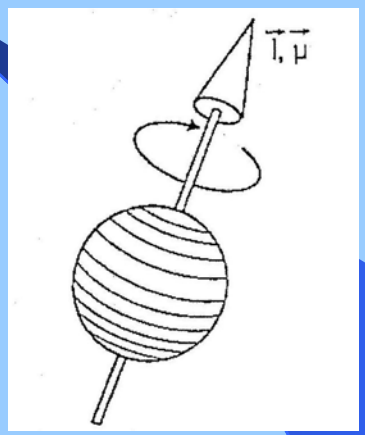


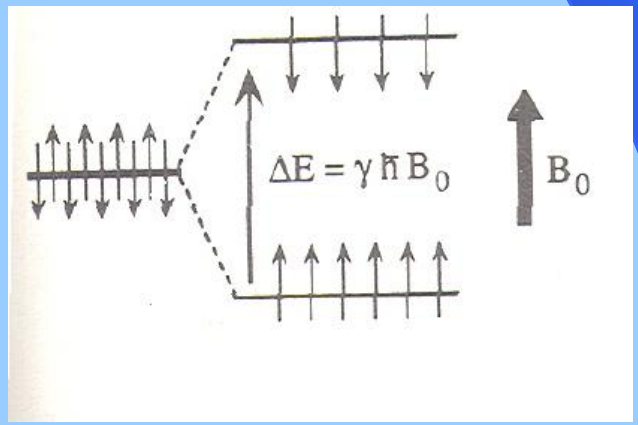
Overview

- Basic principles of NMR
- Application of NMR method
  - Classical method
  - Combined relaxations experiment
- Evaluation
- Reference methods
- Results
- Summary and expectations

Basic principles of NMR (I)

- Some isotopes have spins, I
- magnetic moment, μ, results from spins
- „random“ orientation of spins
- Inducing transition between energy levels
- Parallel and anti-parallel orientation of spins depending on B_0

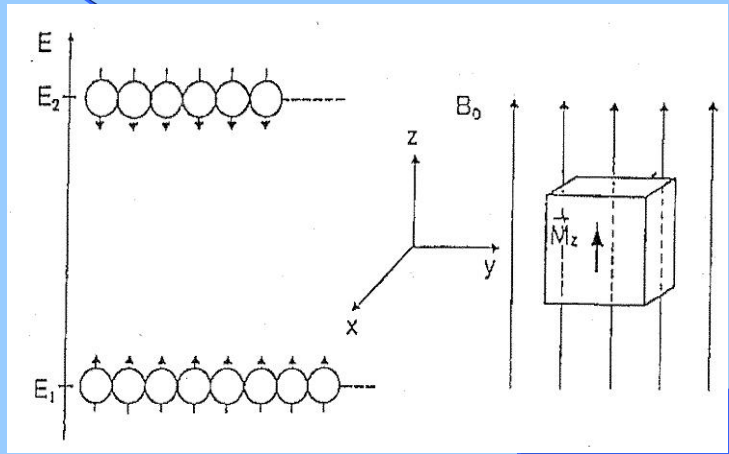


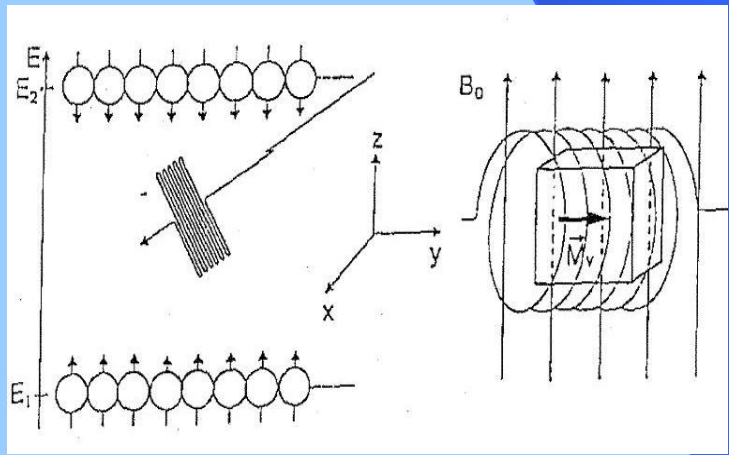


Basic principles of NMR (I)

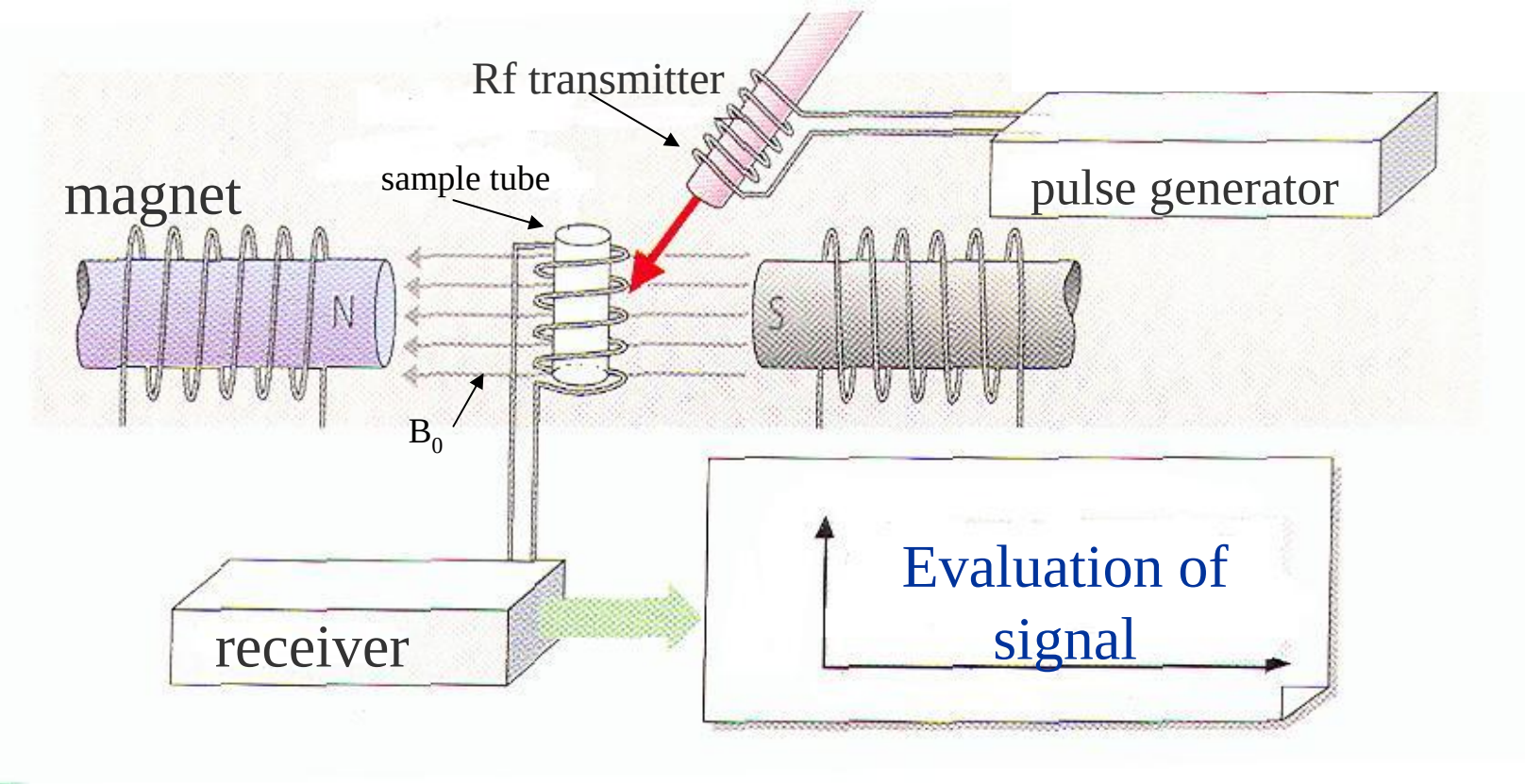
- On a lower energy level more spins
- Makroscopic magnetization, M_z
- M_z depends on N and B_0

- M_z in transverse plane
  - Fluctuating magnetic field generates a current in a coil
  - Demonstration of M_z





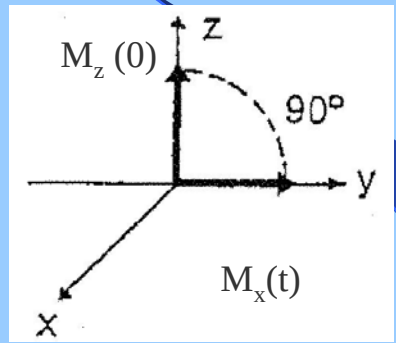
NMR apparatus

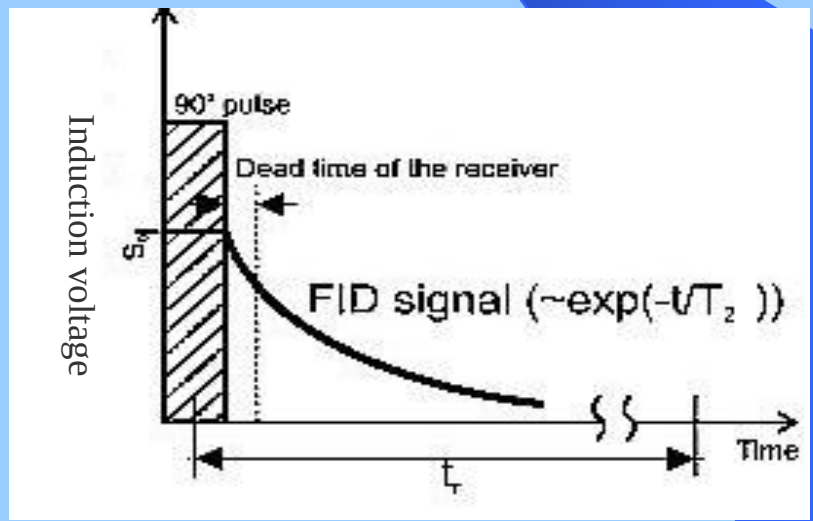


The Minispec mq20 NMR Analyzer, Bruker optics

Methods of TD-NMR (FID)

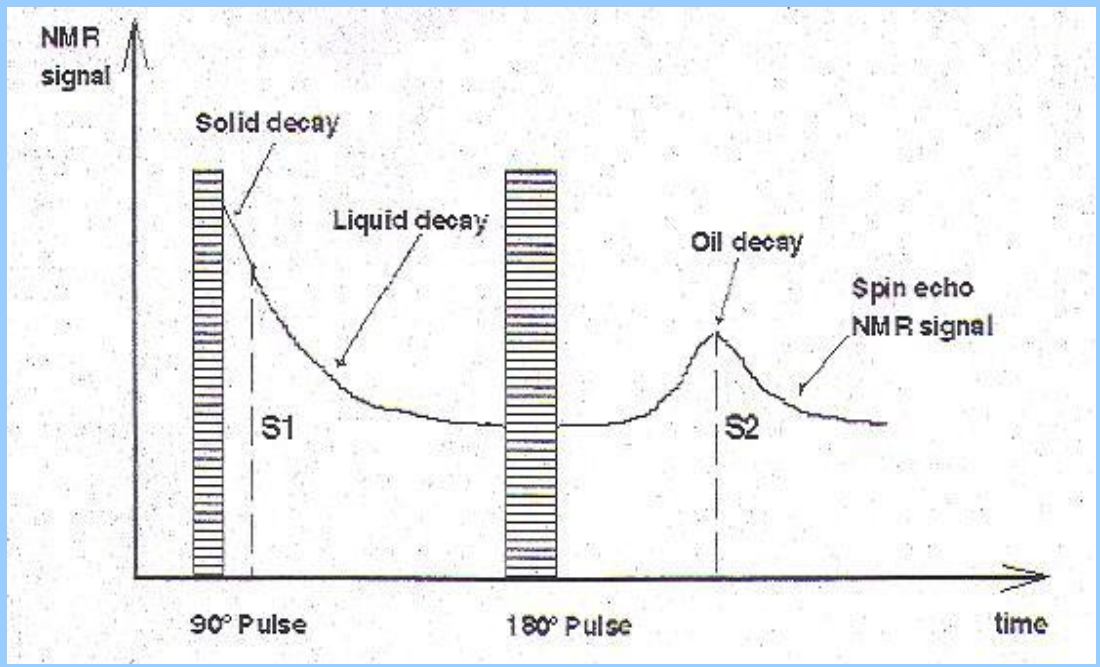
- Rf pulse of 90°
- Signal decays with characteristic time constant T_2
- Relaxations of solid and liquid components are different
- T_1 and T_2: properties of a sample





Methods of TD-NMR (spin-echo)

- Pulse sequence consists of two pulses
- FID- and echo signal are measured

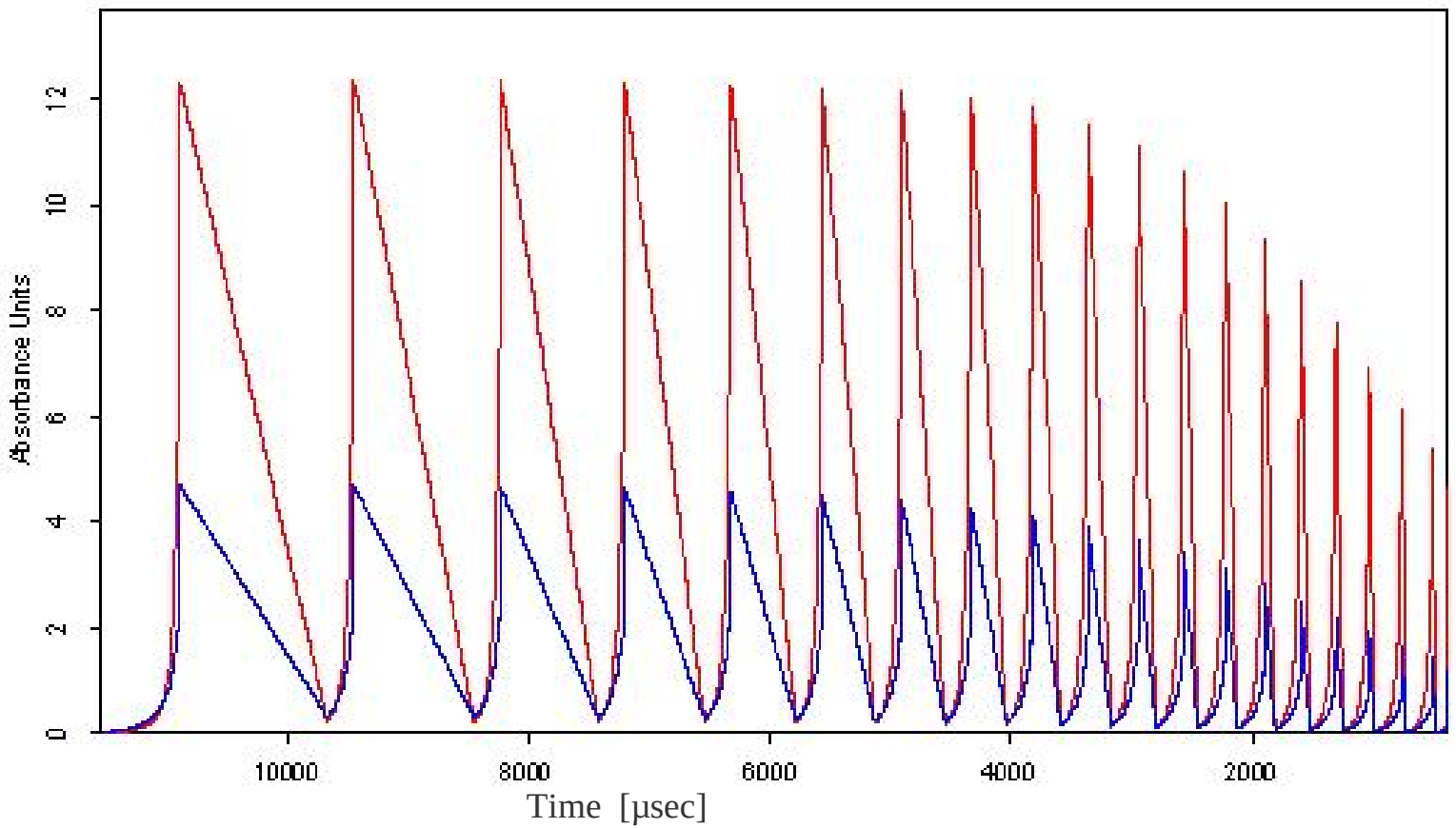


- S_1 = moisture + oil
- S_1-S_2 = moisture
- Determination of sample only with low water content

Measurement of water and fat content in samples with high water content

- T_1 and T_2 of water and fat in a sample are different
- The differences are small and depend on other parameters
- Single measurements of T_1 or T_2 are not sufficient
- Combined relaxations experiment

TD-NMR spectra of the combined relaxations experiment



Chemometric method for evaluation of NMR data

- A single measurement sequence consists of a wealth of information
- Statistical chemometric method
- Multivariate calibration model
- Partial Least Squares Analysis
  - Reduction of data information

Reference methods

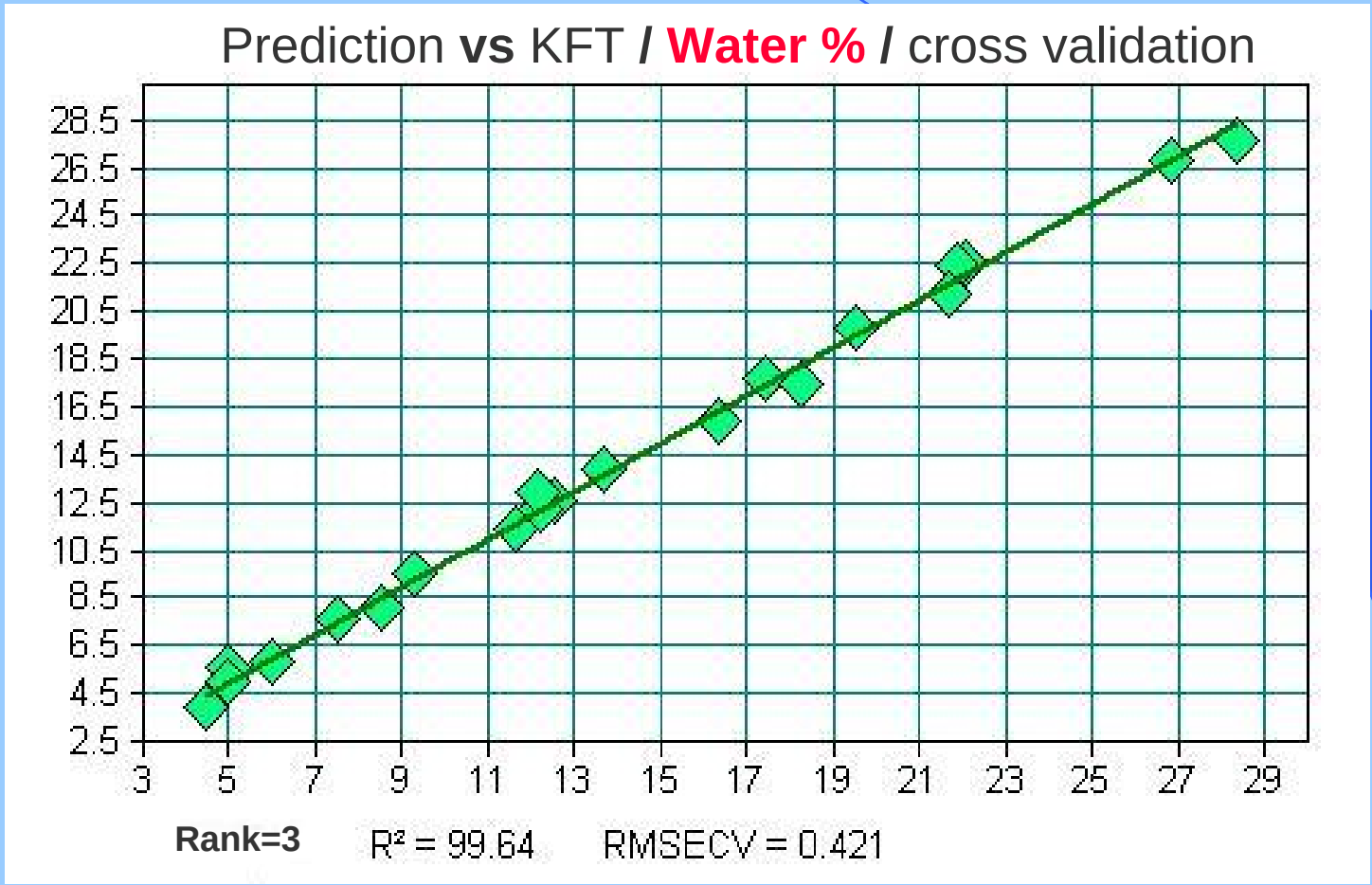
- Fat content determination
  - Soxhlet extraction
  - Time consuming, inflammable solvents
- Water content determination
  - Karl Fischer Titration
  - Addition of formamide
  - Measurement at elevated temperature

Results

- Caramel mass
  - Water content is important for caramel quality
  - Water and fat content vary
- Temperature of samples:

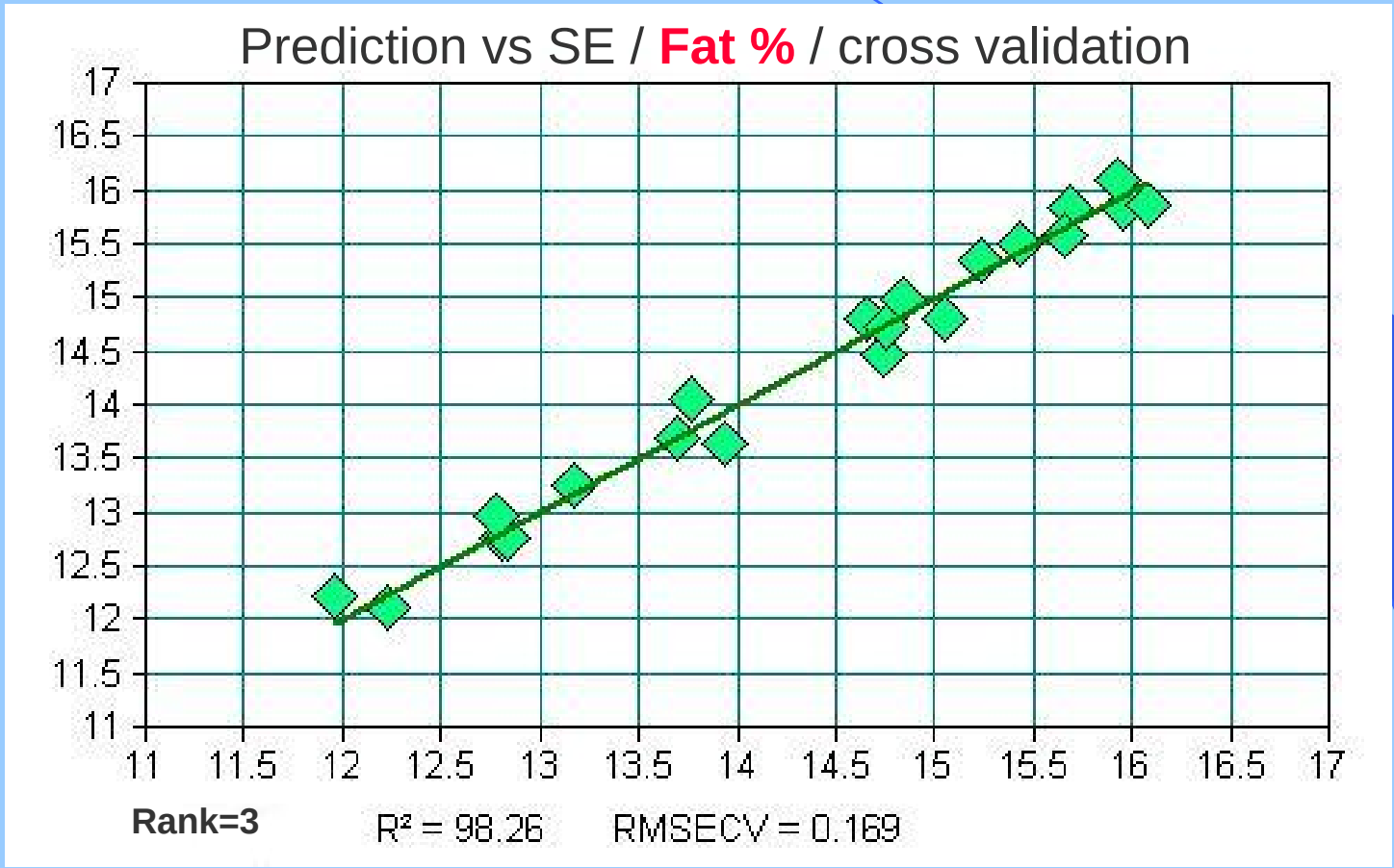
  40 or 20 °C
- Measurement of NMR and reference data simultaneously

Results of the determination
(I) Calibration 20 °C

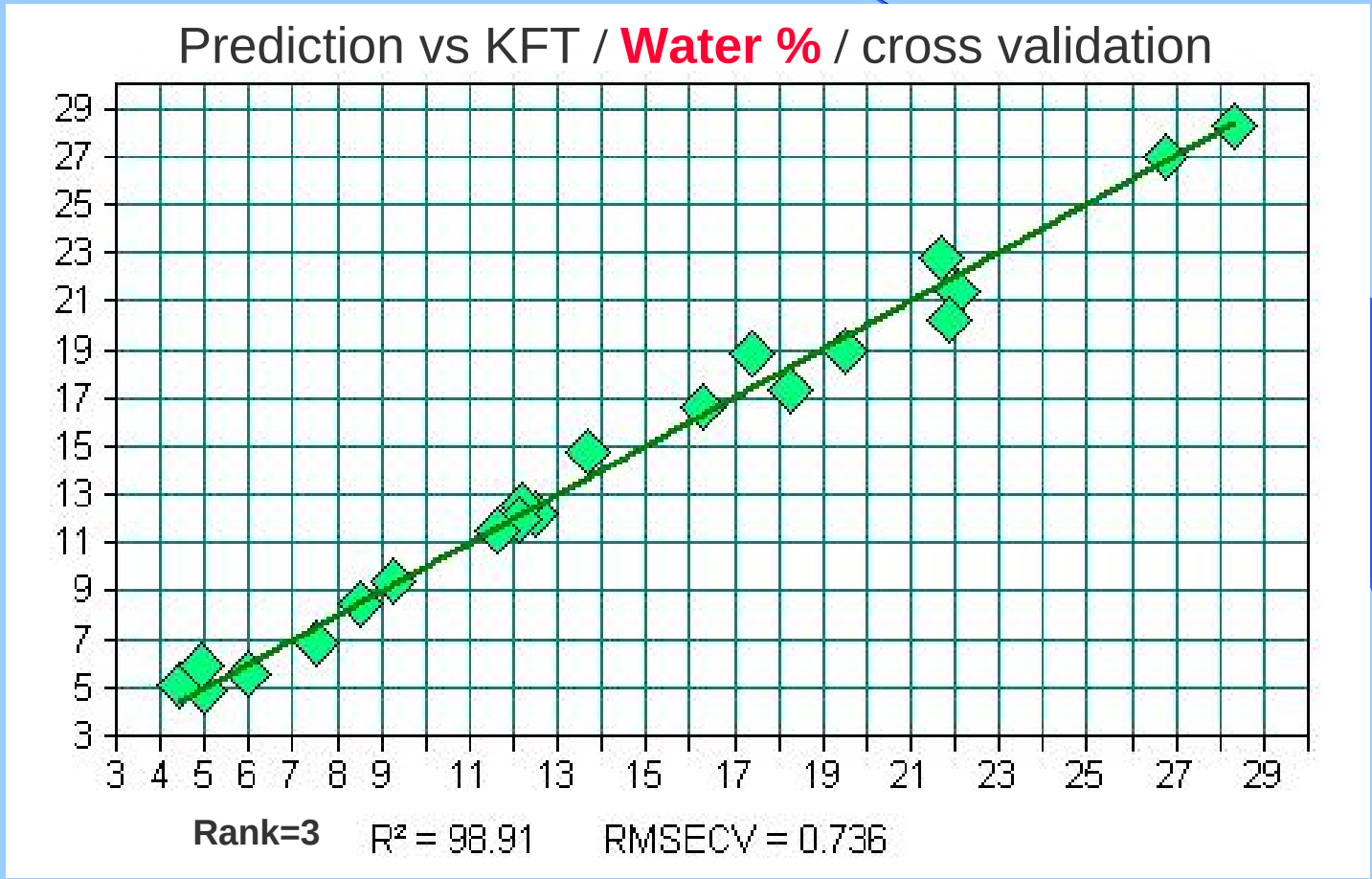


Results of the determination

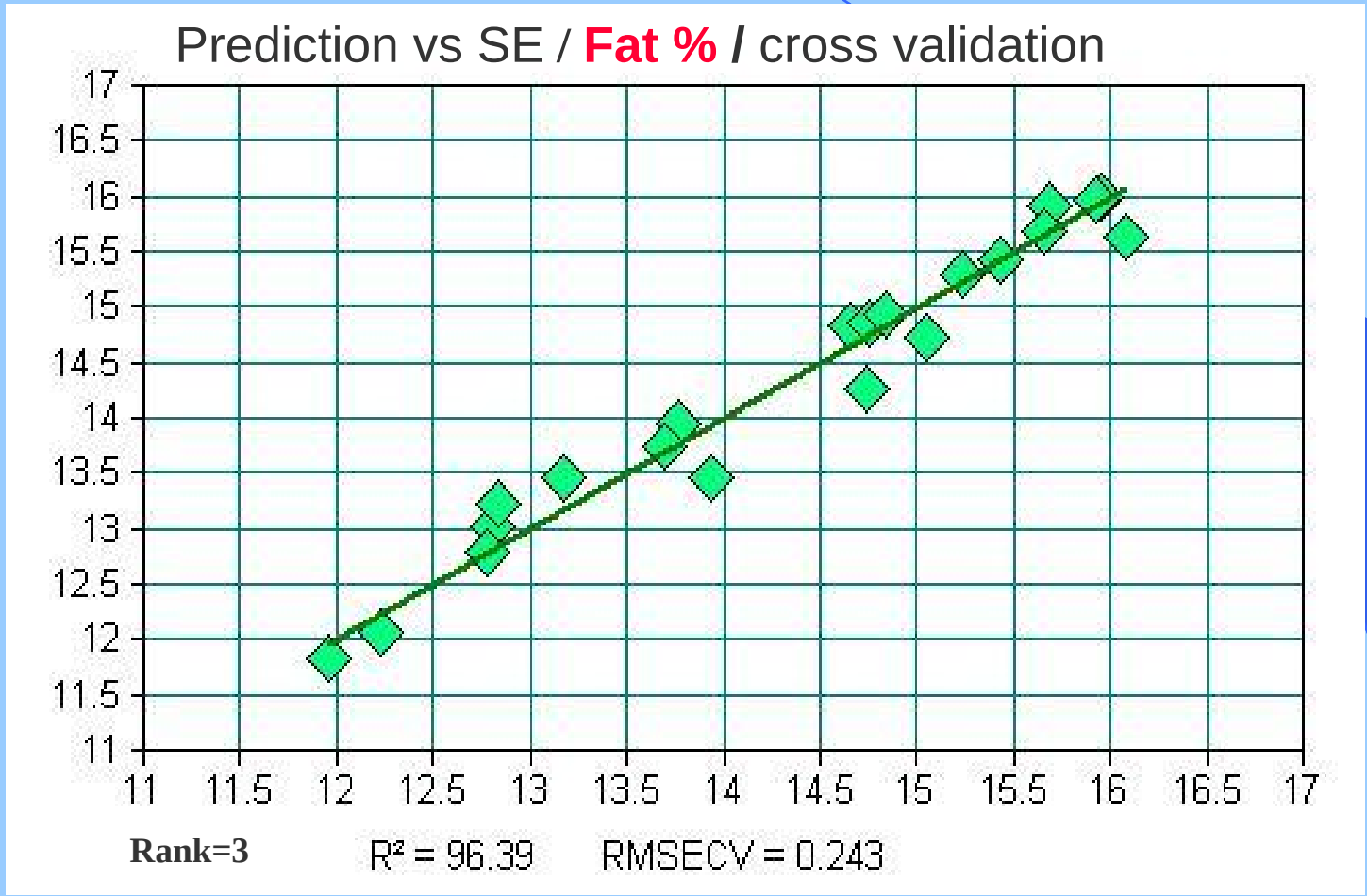
(I) Calibration 20 °C



Results of the determination
(II) Calibration 40 °C



Results of the determination (II) Calibration 40 °C



Summary and expectations

- Application of the calibration in QC is possible
- Advantage of the TD-NMR method
  - Samples with high water content can be measured
  - A good alternative for reference methods
- Next projects of this work

Thank you for your attention