The Bochum NMR box and measurements with the VNA

RUB

Verification of a DG8SAQ VNWA 3 for polarization measurement of solid targets

MSc-thesis Florens Grimm

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INSTITUT FÜR EXPERIMENTALPHYSIK I
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Content

- Motivation: Polarization experiments
- NMR-Spectroscopy
- Bochum NMR-box
- VNA fundamentals:
  - Motivation for DG8SAQ VNWA 3
  - S-Parameters
- Measurements
  - Experimental setup
  - Fit procedure
  - TE-method
  - Results
- Summary / Outlook

Motivation: Polarization experiments

- Study of resonance and spin structure of nucleons
- Structure of matter:
- Polarization information is important to know to further study the structure of nucleons like protons and neutrons:
    - Valence quark vs virtual quark/anti-quark pairs

- **COMPASS**
- Double polarized deep inelastic scattering at high energies
    - Contribution of quarks, gluons, angular momentum towards proton spin
    - Spin composition of the proton/neutron spin $\frac{1}{2}$

$$\frac{1}{2} = \frac{1}{2}(\Delta u + \Delta d + \Delta s) + \Delta \mathrm{G} + \mathrm{L_z}$$

- **ELSA (Bonn), MAMI (Mainz)**
- Scattering experiments with polarized beams and targets at medium energies
    - Study of nucleon resonance region

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NMR-spectrometer

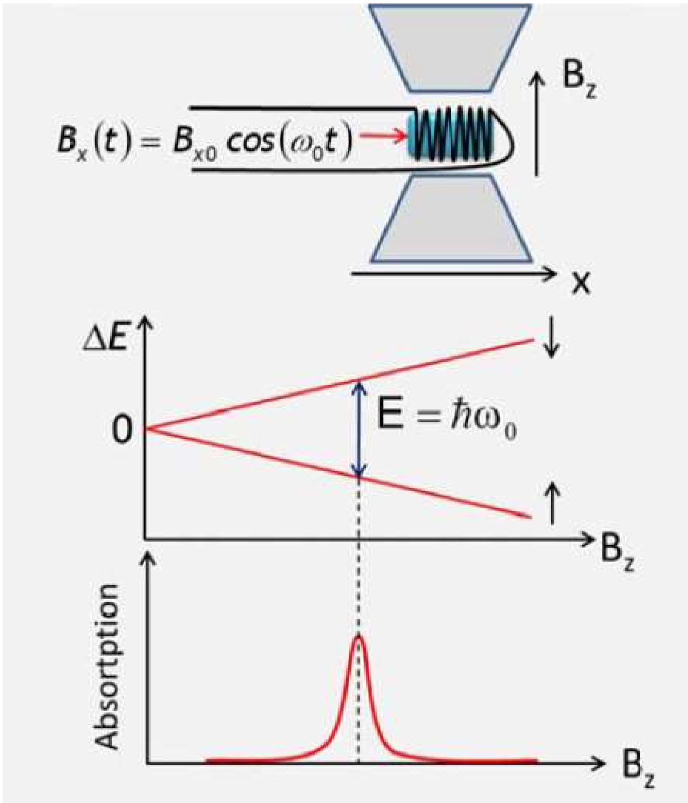
- Zeeman effect
- Magnetic field in z-direction leads to energy splitting
- Radio frequency coil provides oscillating magnetic field in x-direction
  - Realized as LC-circuit
- $B_x(t) = B_{x,0}\cos(\omega_0 t)$

- **Alternating magnetic field drives spin-flip transitions:**

  $$\boldsymbol{\Delta E = \hbar\omega_0 = \hbar\omega_L}$$

- Resonance condition can be achieved in two ways:
  - **Frequency-sweep: Constant B-field and sweep frequency**
  - **Field-sweep: Constant frequency and sweep B-field**
    (not practicable during DNP)

- Two types:
  - Continues wave NMR
  - Pulsed NMR
    (not used because one loose important information during ringing down of the pulse)



Polarization

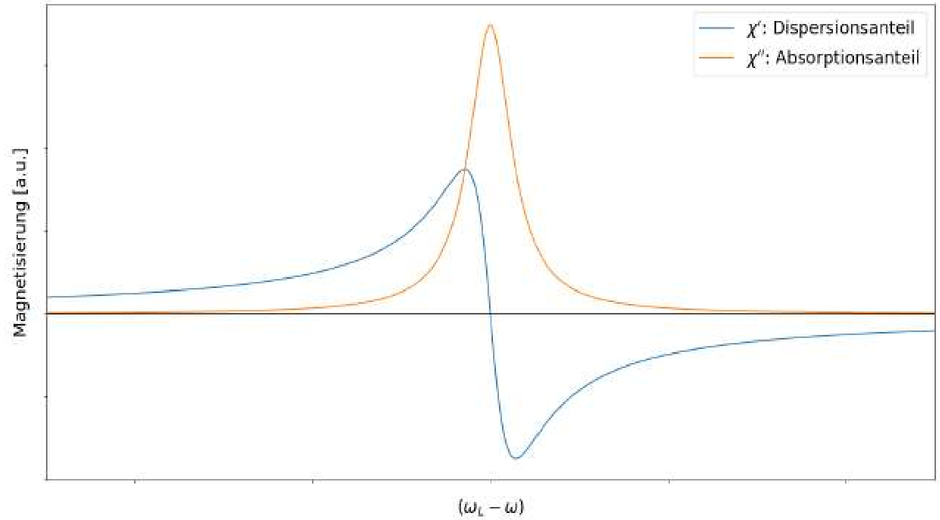
- **Polarization is connected with susceptibility**
- **complex susceptibility** of sample:
  $\chi(\omega) = \chi'(\omega) - i\,\chi''(\omega)$

- Target material in coil modifies inductance:
  $L(\omega) = L_0(1 + 4\pi\eta\boldsymbol{\chi(\omega)})$ η: effective filling factor

- Resonate coil with capacitor → LC-circuit
- Resonance frequency: $\omega_0 = 1/\sqrt{L_0 C}$

- Changes in $\chi''(\omega)$
  → changes in Q-factor of LC-circuit
  → Changes in mean power loss of coil
  → for constant current: change in voltage

→ $P = K \int_{-\infty}^{+\infty} \chi''(\omega) d\omega$



- Internal fields in probe
  → **linewidth of signal**
  → **Frequency scan required**

Polarization ∝ Area under signal

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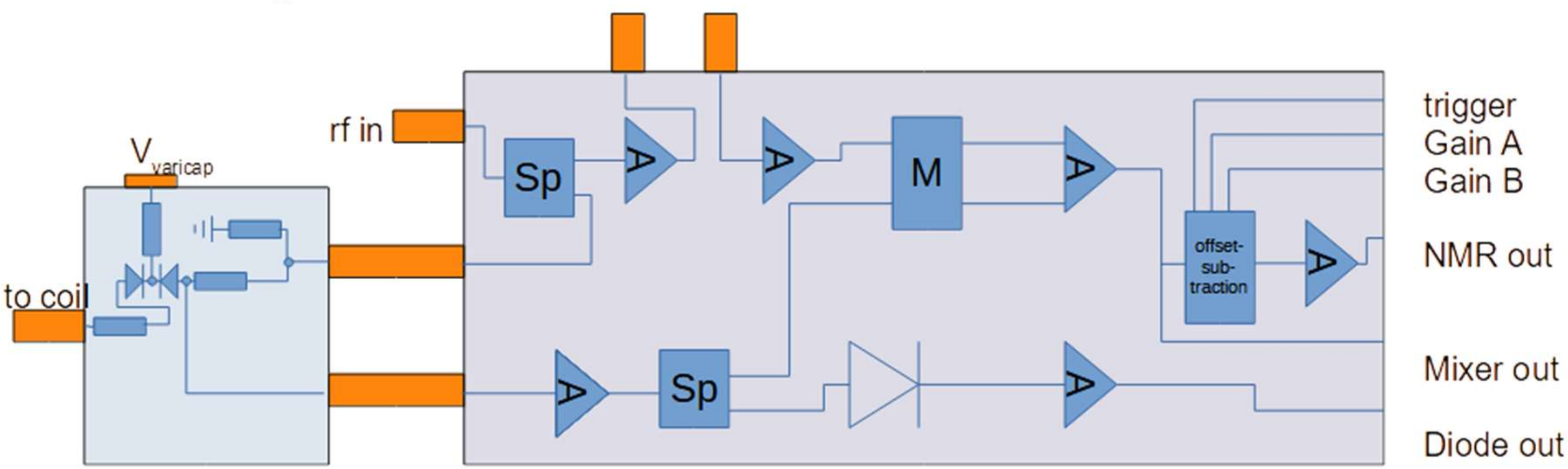
Bochum NMR - box

Consists of three parts:

- RF – board
- dc-offset board
- tuning-box



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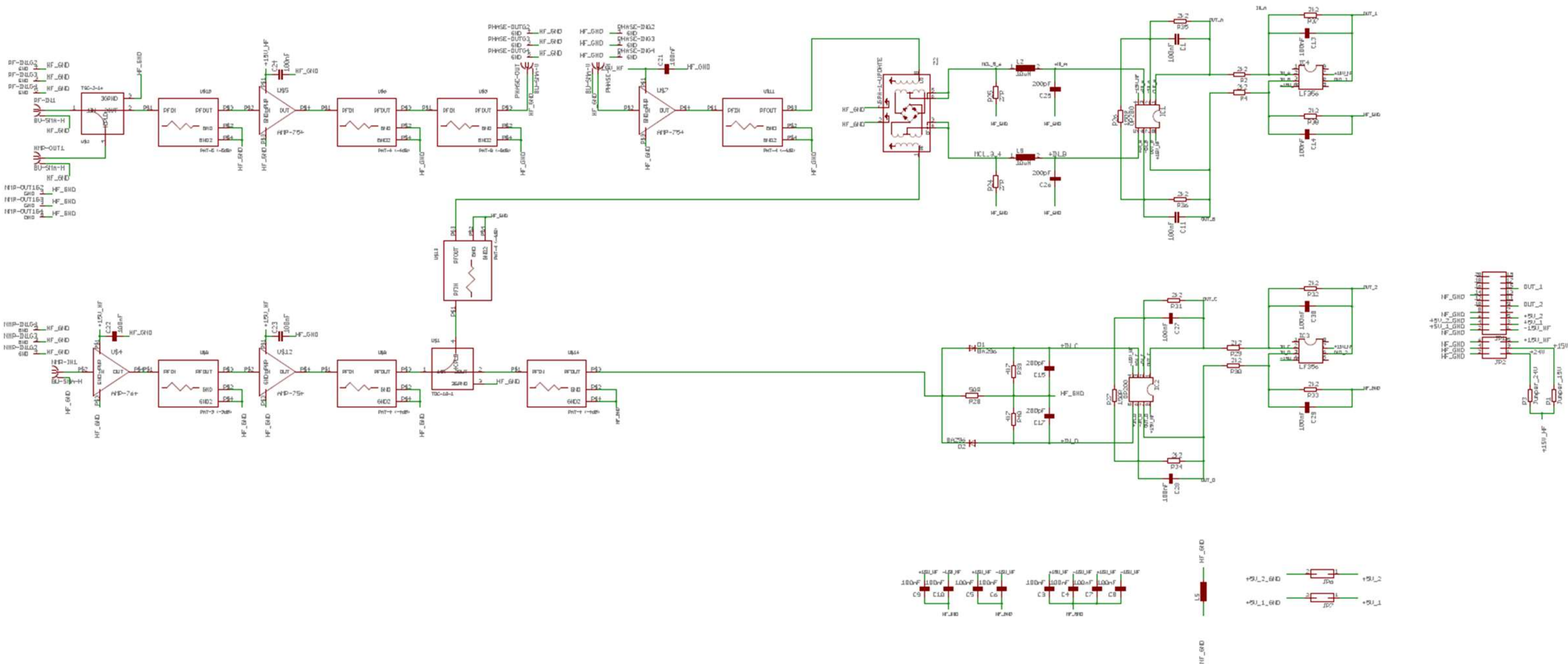
RF- schematics



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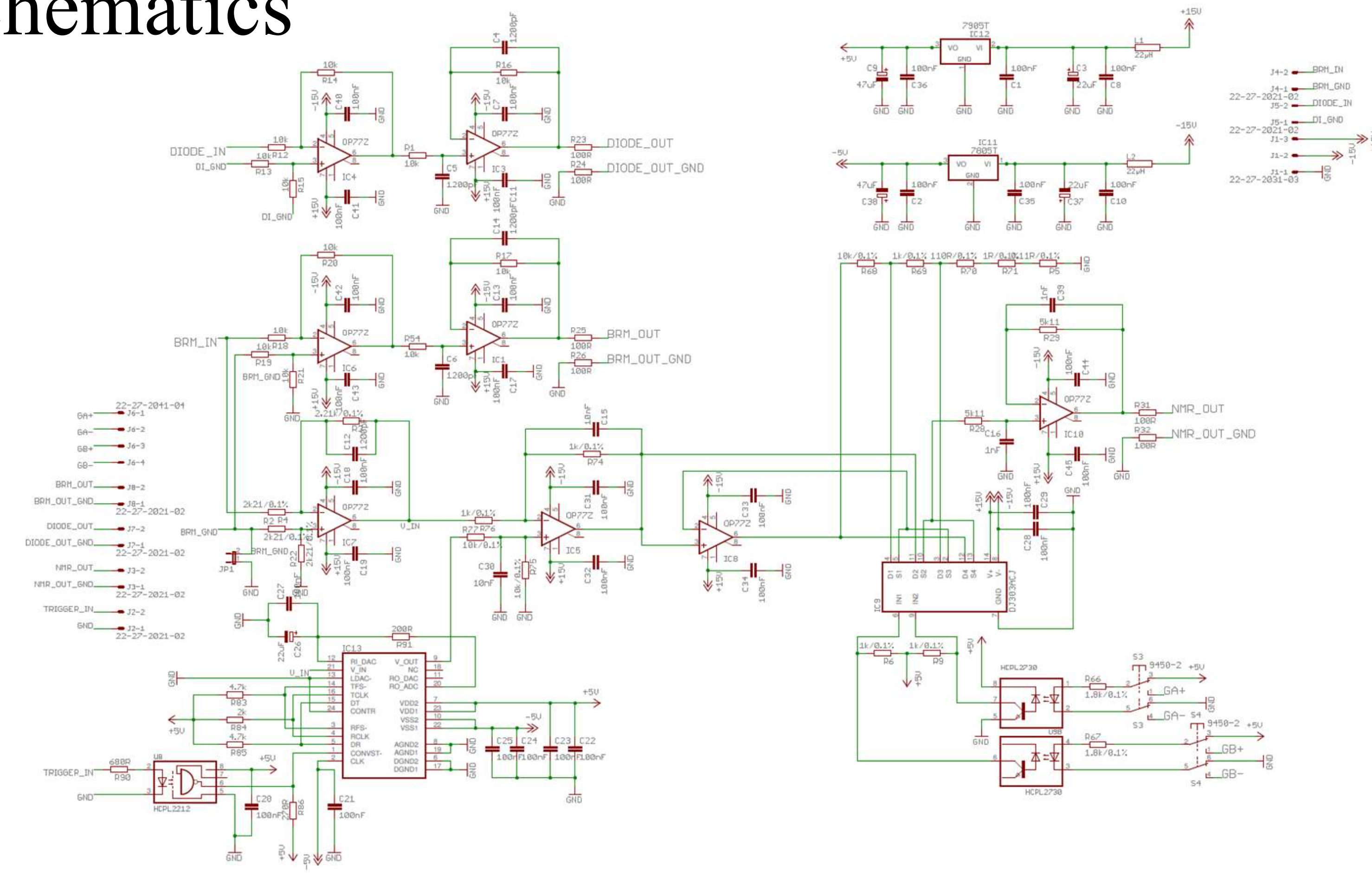
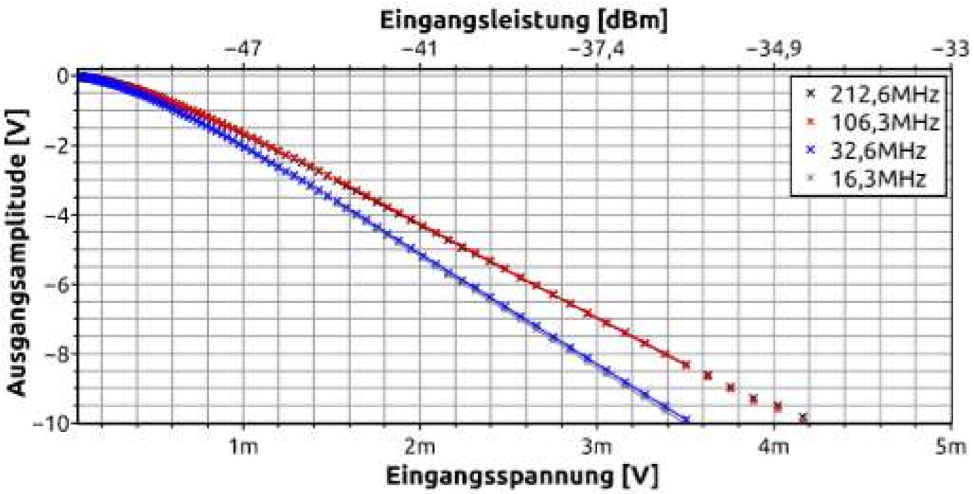
LF- schematics



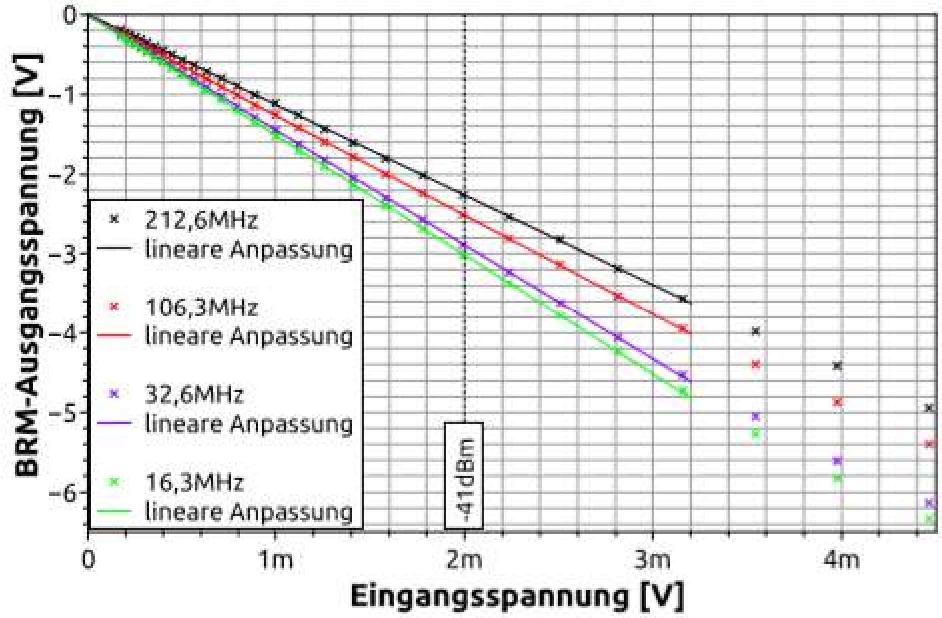
Bochum NMR – box 2

Diode output voltage vs input RF voltage



SMS7630

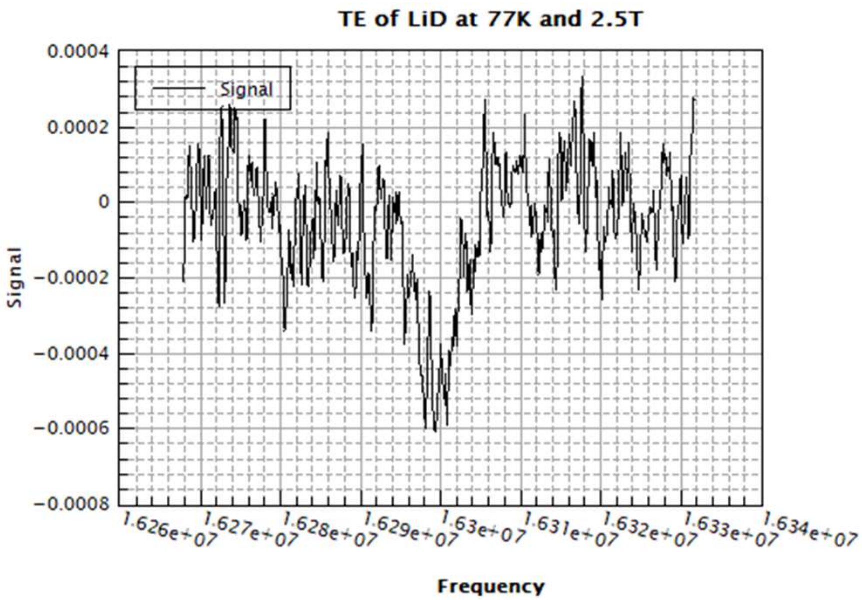
Mixer output voltage vs input RF voltage

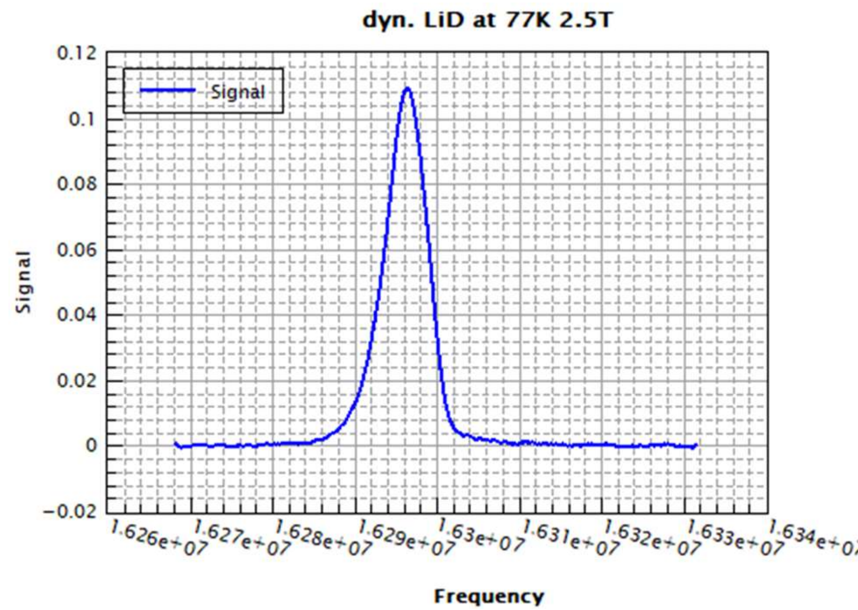


SMA1

Bochum NMR – box 3

Signals of ^{6}LiD at 77K ad 2.5 T





Result: SNR is slightly better than that of the well known Liverpool NMR Modul!

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NMR measurement with Vector Network Analyser

Idea: Elena Long, PSTP 2019 Knoxville

Thesis: MSc Florens Grimm, Bochum, 2022

Despite many problems
such as the corona pandemic and lack of cooling water (leaks),
the measurements could finally begin in February.

Motivation: DG8SAQ VNWA3

- Why the DG8SAQ VNWA3?

- Biggest Factor: **Price**
  - DG8SAQ VNWA + Low Noise Amplifier:
    - 500€ + 70€
  - Compared to:
  - RF-Generator + Q-Meter + additional components:
    - 10 000€ - 50 000€

- VNWA would contain all necessary components to realize a polarization measurement

- **Question:**
- Does the VNWA3 allow precise and linear measurements over a wide range of polarizations (0.25% at thermal eq. up to >70%)

500€

8 000€

47 000€

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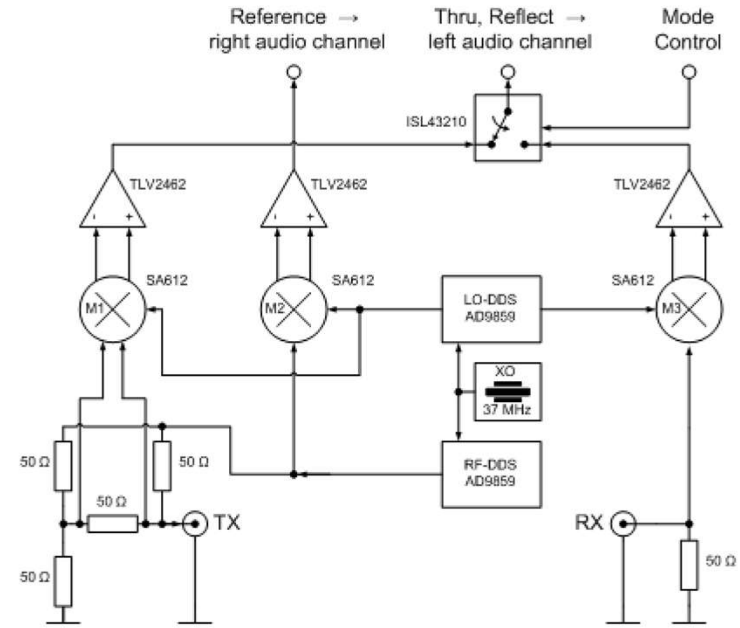
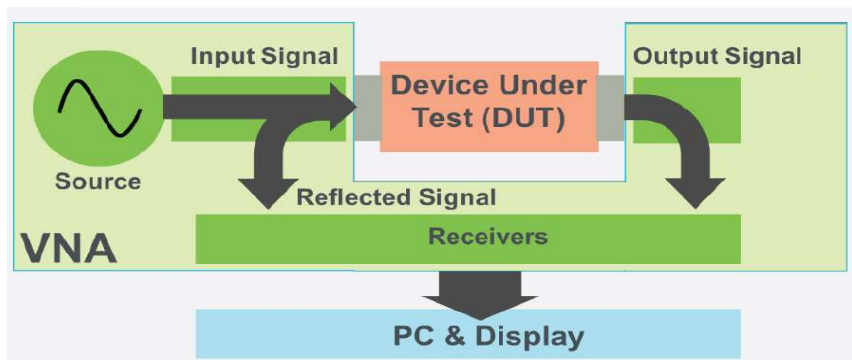
DG8SAQ VNWA3
Vector Network Analyzer

- VNA contains both a source and a receiver:
  - Source generates known stimulus signal
  - Receiver determines changes in stimulus signal caused by device-under-test (DUT)
- Stimulus Signal is injected into DUT:
  - Transmitted and reflected signal is measured
  - Compared to original signal





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S-parameters

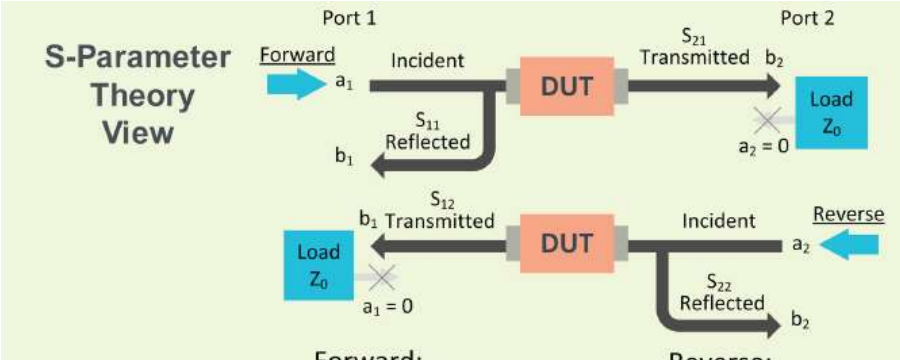
- Scattering Parameters (S-Parameters)
- high frequencies make it difficult to measure voltage or current
- related to measurements such as:
  - gain, loss, reflection coefficient

- Portion of the incident signal is taken as reference
- S-Parameters defined as ratios of signals

- **Reflected or transmitted signal is compared to reference signal**

- Stimulus Signal changes as it is reflected/transmitted at DUT
  - Transmitted signal experiences changes in magnitude and phase

- **For NMR measurements: S_{21}**



	Forward:	Reverse:
Reflection:	$S_{11} = \frac{\text{Reflected}}{\text{Incident}} = \frac{b_1}{a_1}\Big\rvert_{a_2=0}$	$S_{22} = \frac{\text{Reflected}}{\text{Incident}} = \frac{b_2}{a_2}\Big\rvert_{a_1=0}$
Transmission:	$S_{21} = \frac{\text{Transmitted}}{\text{Incident}} = \frac{b_2}{a_1}\Big\rvert_{a_2=0}$	$S_{12} = \frac{\text{Transmitted}}{\text{Incident}} = \frac{b_1}{a_2}\Big\rvert_{a_1=0}$

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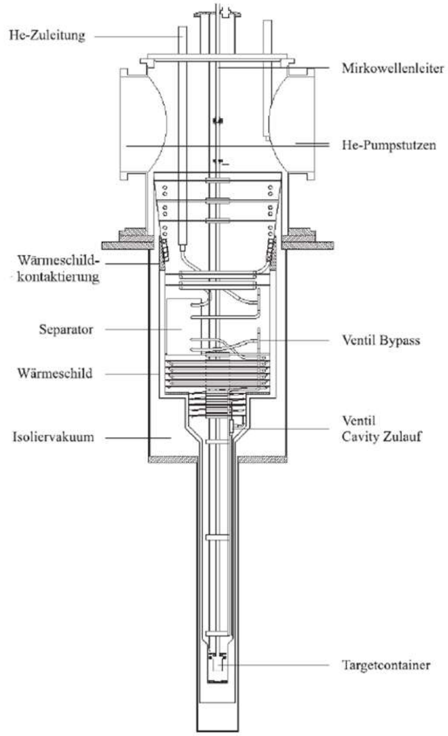
Components of a NMR-experiment



- Magnetic field: C-Magnet, B=2,5T
- Low temperature: Cryo-system, T=1K

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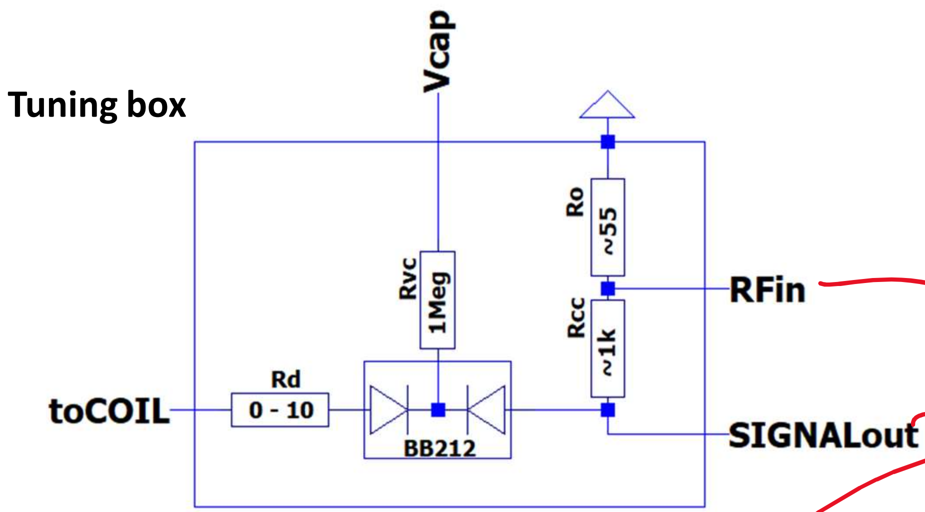
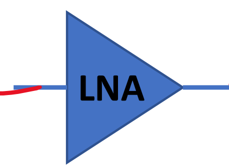
Experiment - setup

For **linearity** and comparison reasons we used a series resonance circuit with same components

Constand current in the coil is needed to reach the linearity for pos. and neg. dynamic signals.



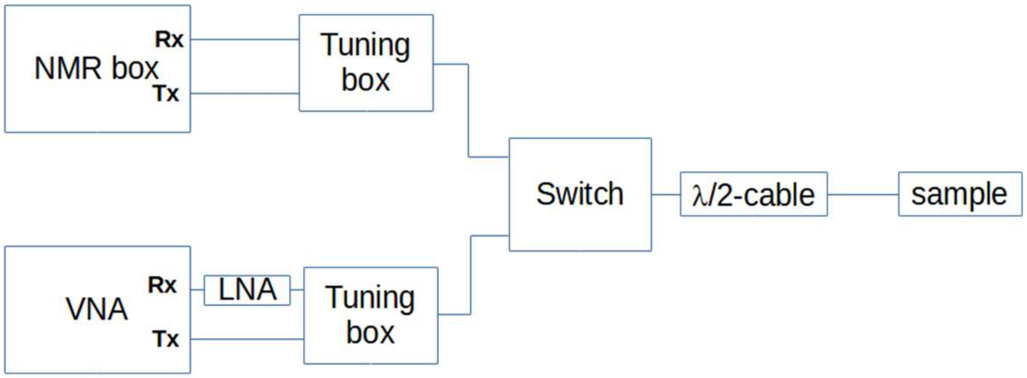


In the VNA setup we used an additional LNA between SIGNALout and RX In!

Experiment - setup

- Magnetic field: $B \approx 2.5\, Tesla$
- Temperature: $T \approx 1\, Kelvin$

- Sample materials:
  - Lithium deuteride: ^{6}LiD
    - Larmor frequency: 16.3 MHz, $\lambda = 18\, m$
  - Ammonia: NH3
    - Larmor frequency: 106.4 MHz, $\lambda = 2.8\, m$
  - TEMPO doped n-Butanol
    - Larmor frequency: 106.4 MHz, $\lambda = 2.8\, m$
  - (TEMPO=(2,2,6,6-tetramethylpiperidin-1-yl)oxidanyl)



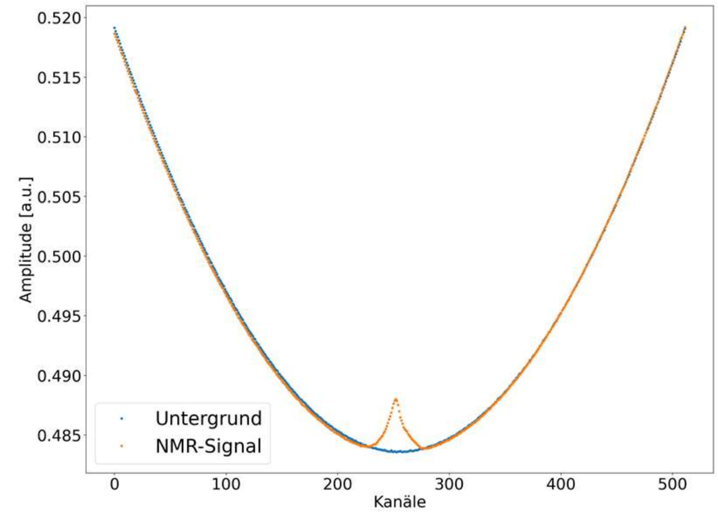
Ensures quasi parallel measurement in a minute change between NMR-box and VNA.

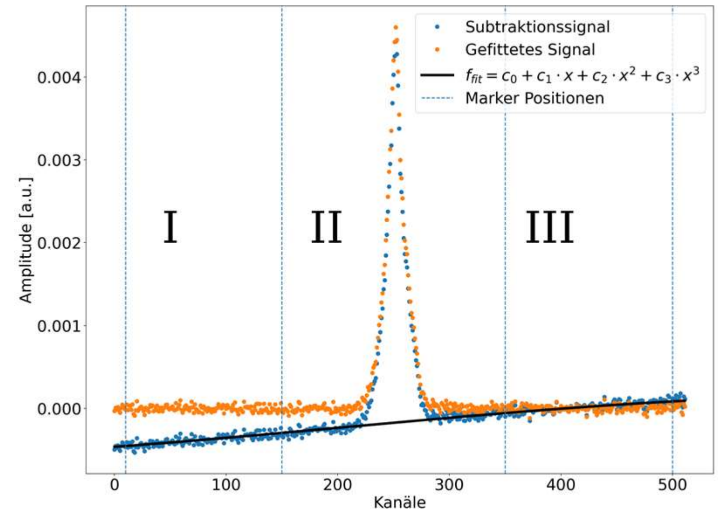
- Sample material polarized via DNP
  - Alternating signal detection with Bochum NMR-module and VNA setups
  - Connection between detection setup and nmr coil controlled by switch
    - Only one setup connected to nmr coil at a time

Signal preparation

- Resonance curve of LC-circuit: parabola
- Subtract background signal without NMR-Signal
- Baseline fit with polynomial function:
  $f_{fit} = c_0 + c_1 \cdot x + c_2 \cdot x^2 + c_3 \cdot x^3$
  - Data used in sections **I** and **III**
- Subtract polynomial fit from data
- Area under signal ⇔ Summation of fitted data (section **II**)

Calculate area under signal → Polarization





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TE-method

- Area under signal is proportional to polarization

$$P = K \int_{-\infty}^{+\infty} \chi''(\omega) d\omega$$

- Constant K needs to be determined for absolute polarization values:
  - Calibration with TE-polarization

$$P_{dyn} = \frac{P_{TE}}{F_{TE}} \cdot F_{dyn} = E \cdot F_{dyn}$$

- P_{TE}: calculated
  - Precise measurement of current magnetic field and temperature necessary
- F_{TE}: Signal measurement in thermal equilibrium (no DNP used)
- F_{dyn}: Signal area while using DNP
- E: Enhancement factor

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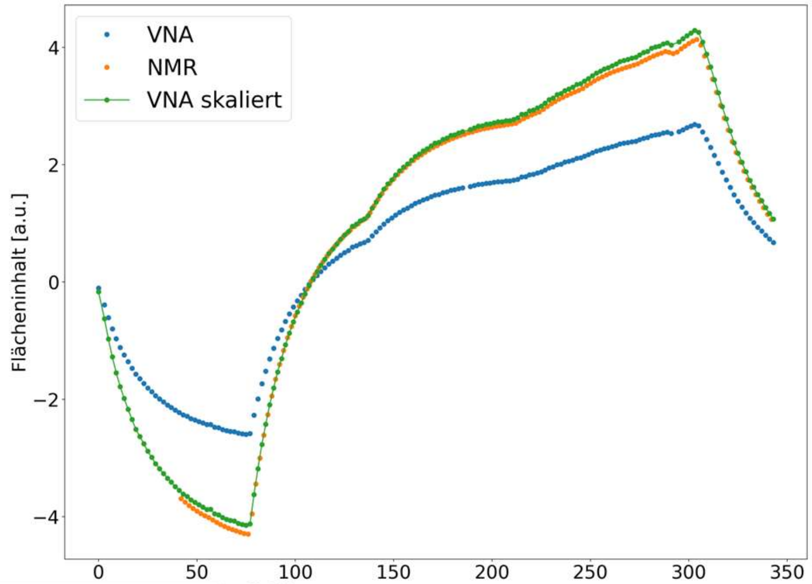
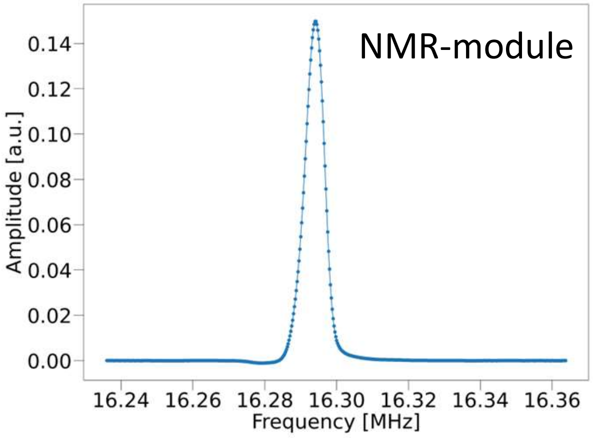
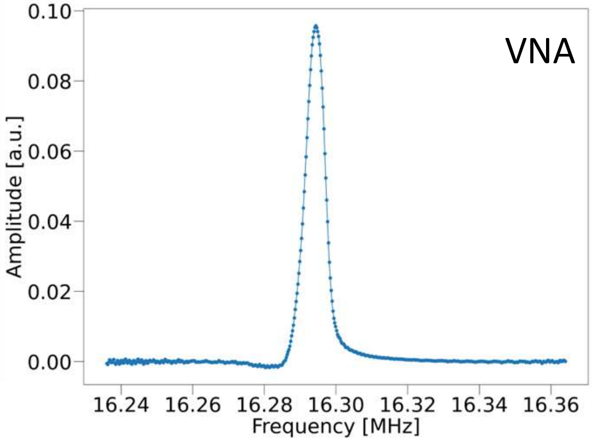
Lithiumdeuterid ^{6}LiD at 77K and 2.5T

- Lithium deuteride has a long relaxation time → no TE-signals
- Comparison by comparing signal-areas between VNA and NMR-module
- Scaling factor SK:
  - Ratio of max. signal area of NMR and VNA
  - Ratio of min. signal area of NMR and VNA
  - → Mean of both scaling factors
- Result:
  - $SK = 1{,}60 \pm 0{,}06$
- Signal-areas align for smaller values but divert for higher values (may be a offset problem)



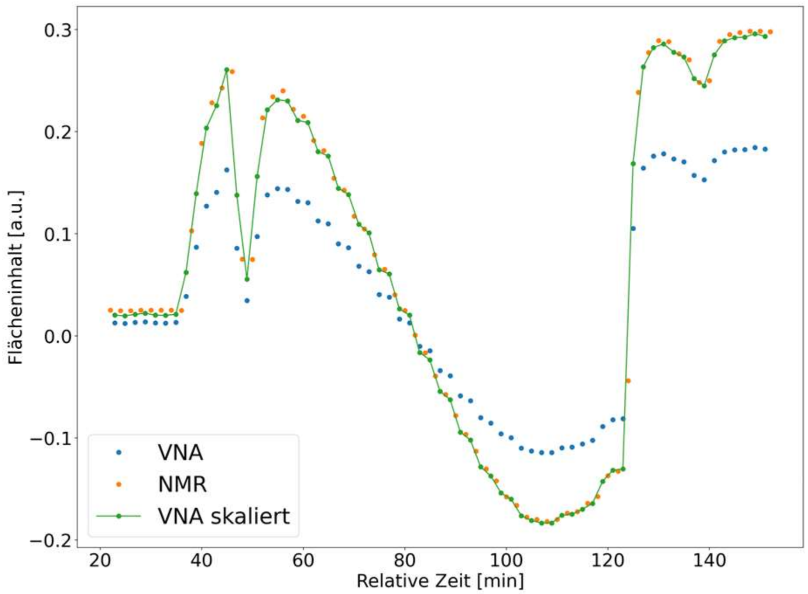


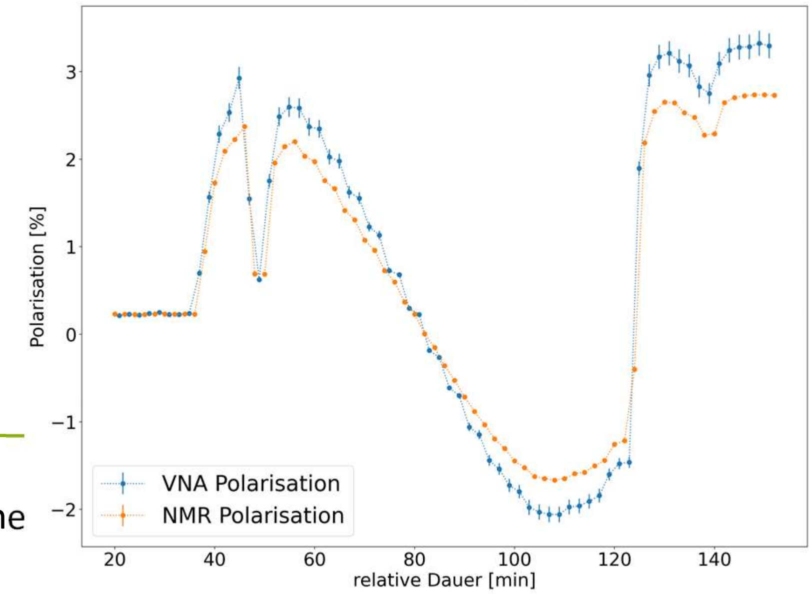


Ammonia NH_3

- TE-signals for ammonia possible
  →TE-method
- Scaled VNA areas and NMR areas align
  - Only TE-signals divert
- Maximal polarization of ~3% still to low → TEMPO n-Butanol

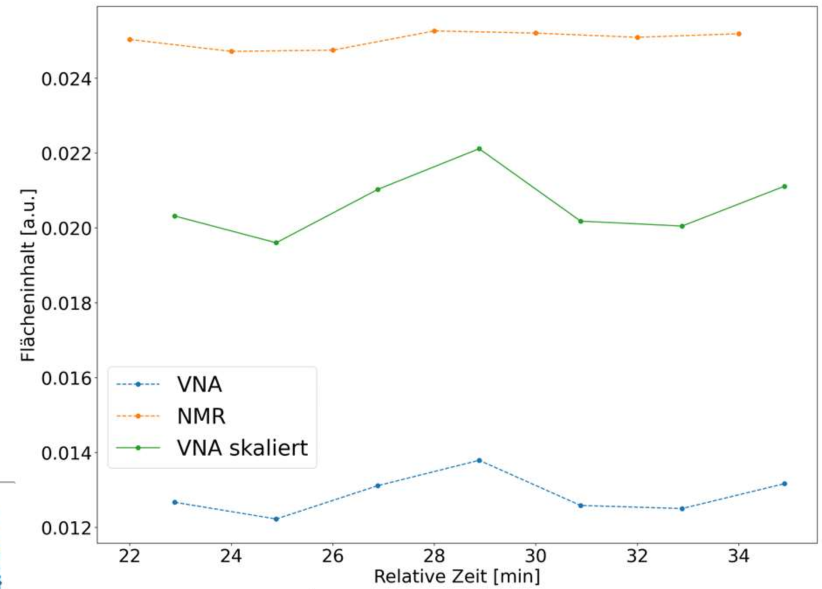
	VNA	NMR
Scaling factor	1.60 ± 0.01	
Enhancement factor	17.3 ± 1.1	9.17 ± 0.07
Polarization max.	$(3.20 \pm 0.21)\%$	$(2.74 \pm 0.02)\%$
Polarization min.	$(-1.98 \pm 0.13)\%$	$(-1.67 \pm 0.01)\%$
Rel. error $\Delta P/P$	± 0.066	± 0.007

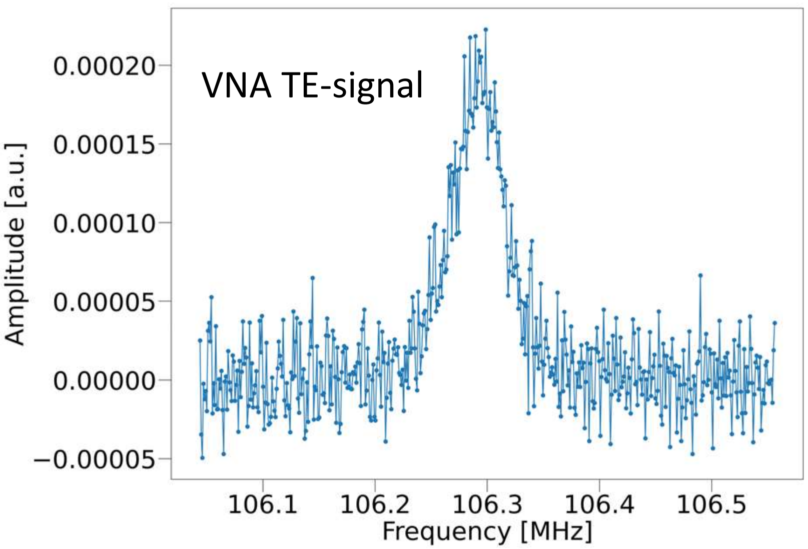


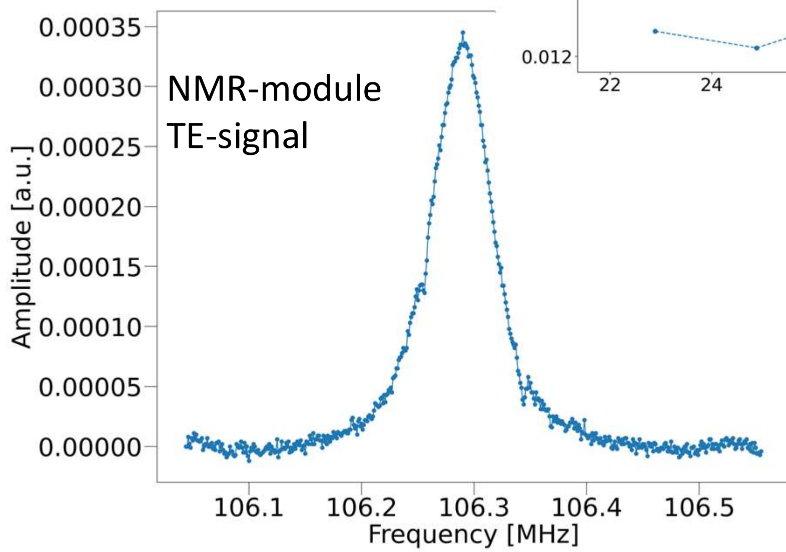


Ammonia NH_3 TE-signals

- TE-Signals of VNA show high noise compared to NMR-module
- But TE-signal area is important for polarization TE-method
- Error of TE-Signal area dominates polarization measurement







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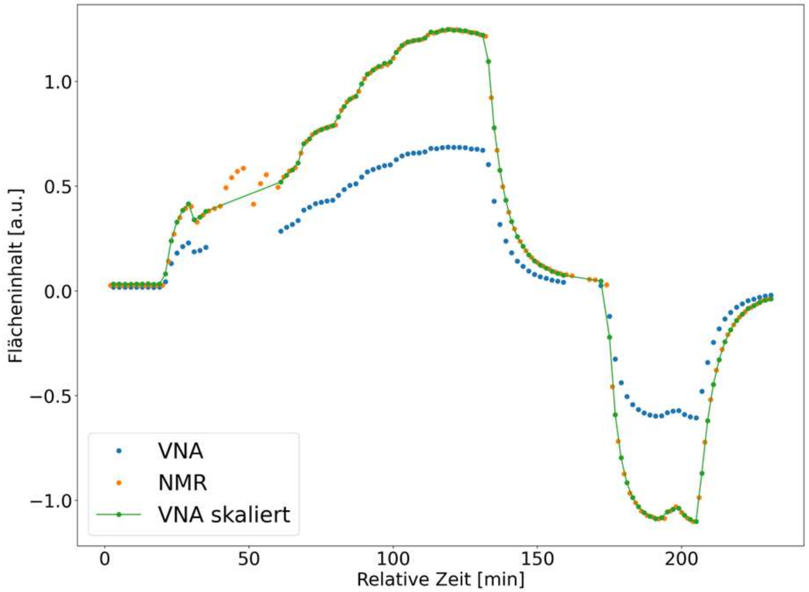
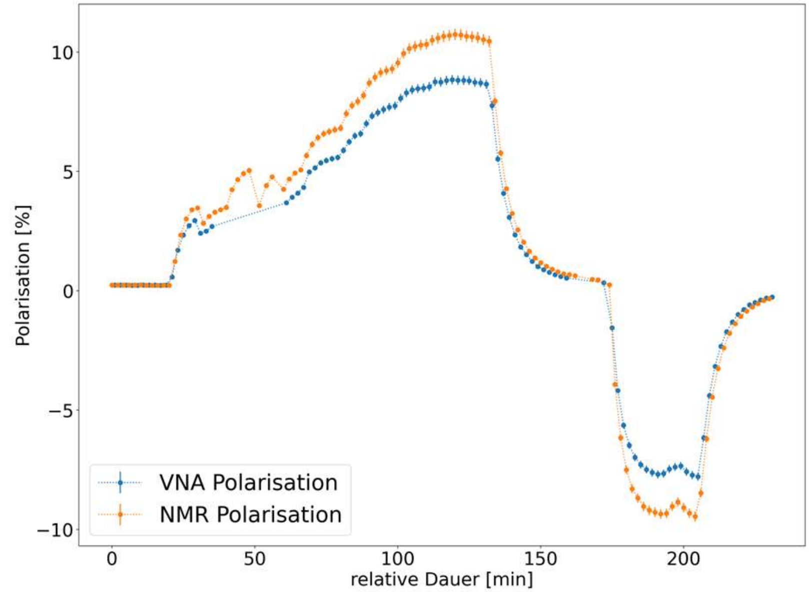
TEMPO doped n-Butanol

	VNA	NMR
Scaling factor	1.81745 ± 0.00004	
Polarization max.	$(8.84 \pm 0.20)\%$	$(10.73 \pm 0.23)\%$
Polarization min.	$(-7.79 \pm 0.17)\%$	$(-9.46 \pm 0.21)\%$
Rel. error $\Delta P/P$	± 0.023	± 0.021

- Higher max. polarization than for NH_3 expected
- TE-Signals possible → TE-method





TEMPO doped n-Butanol TE-signals

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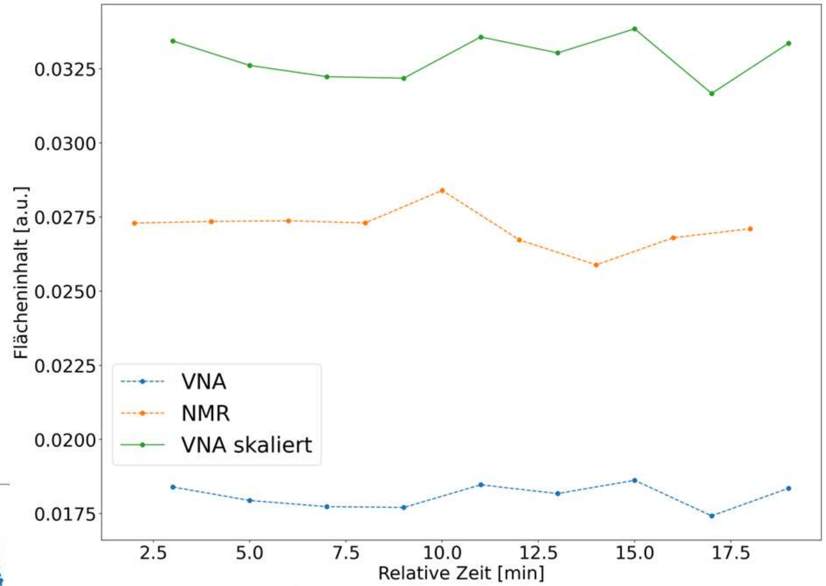

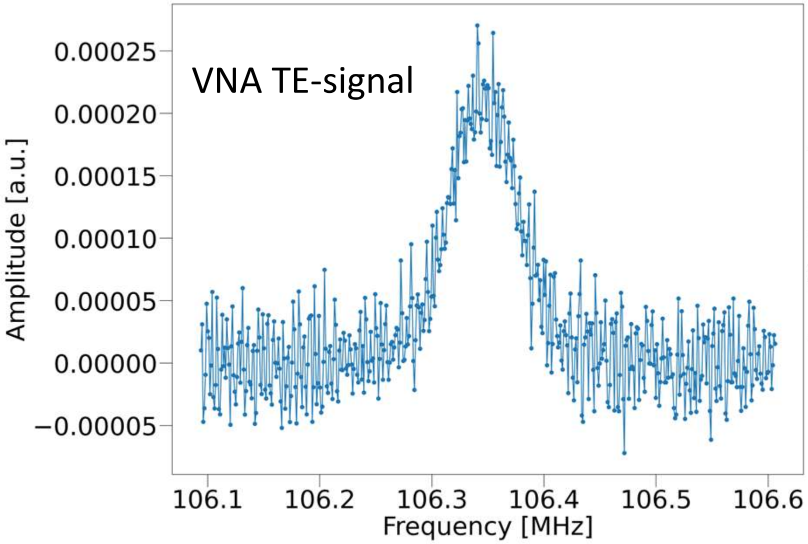

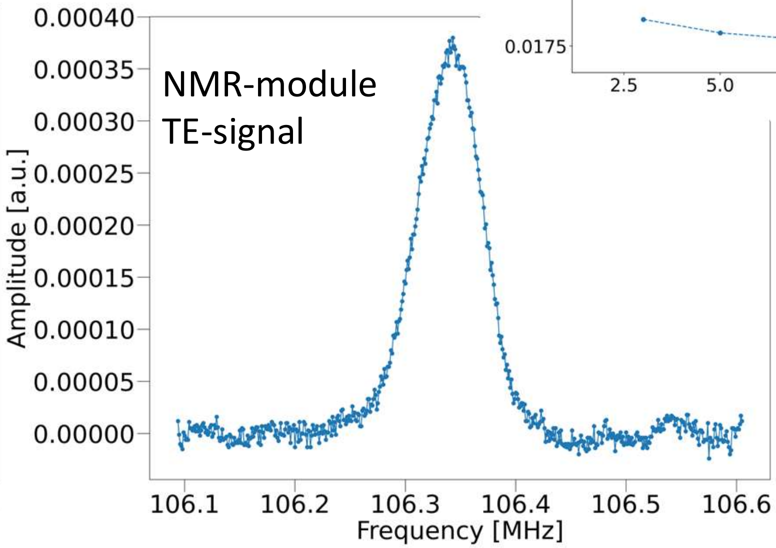

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TEMPO doped n-Butanol relaxation

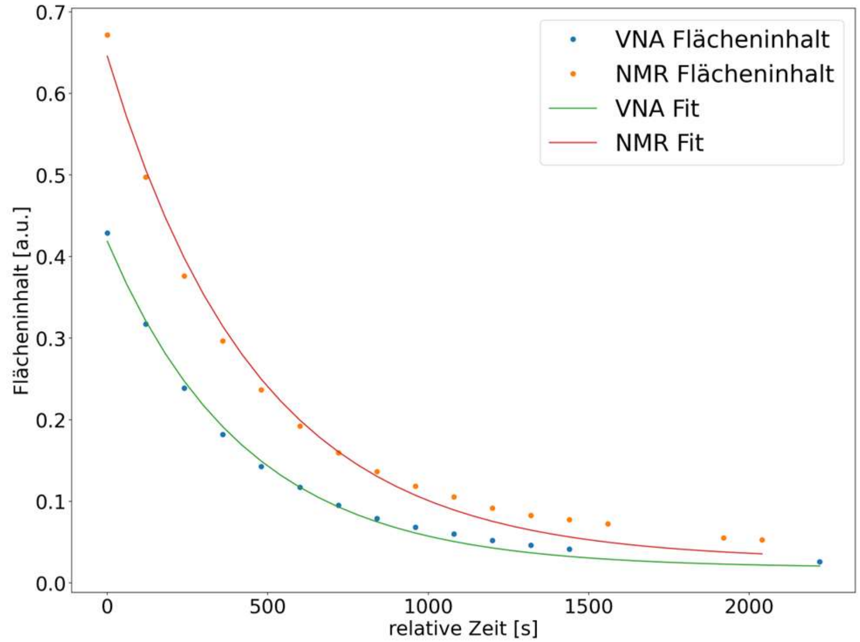
- Determination of T_1 relaxation constant through exponential fit of relaxation process
- DNP → high polarization → turn off microwaves → relaxation process

- Exponential-Fit:
  - $f_{fit} = y_0 + A \cdot e^{-\frac{x}{\tau}}$
- Problems:
  - Low number of datapoints
    - Especially due to sharing of measurement time

    → Final TE-polarization must be TE-polarization from TE-signal measurements

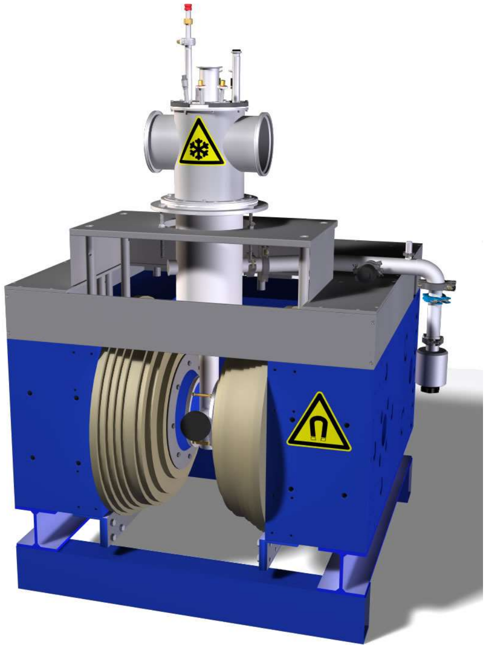
    → Fit-parameter fixed to TE-values

	A	$\boldsymbol{\tau}$	$\boldsymbol{y_0}$
VNA	$(0.4 \pm 0.008)a.u.$	$(429 \pm 21)s$	$(0.0185 \pm 0.018)a.u.$
NMR	$(0.618 \pm 0.016)a.u.$	$(469 \pm 30)s$	$(0.0277 \pm 0.027)a.u.$



Summary/Outlook

- The VNA allows the precise measurement of the signal areas of NMR-signals
- Due to high noise in TE-Signals
  → determination of absolute polarization with VNA shows deviations compared to the NMR-module of up to 2 percentage points
- Possible solution:
  - Low pass filter could be used to reduce noise
- Future:
  - Measurements with asymmetry-method allow for independent verification of VNA linearity



Bibliography

- Talk: "DNP with Solid-State mm-Waves, 3D-Printed Components, & SDR-based NMR" by Elena Long
- Talk: „Polarized Solid Targets" by Gerhard Reicherz
- „Medical Physics" by Hartmut Zabel
- https://twitter.com/j_bertolotti/status/1369962983074455552
- https://ep1.rub.de/poltarg/Forschung/webposter.jpg
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