

Development of a low-field NMR unit using a crossed coil setup for calibration of a PASADENA polarizer

Robert Borowiak¹, Elmar Fischer¹, Sebastien Bär¹, Jochen Leupold¹, Frank Huethel², Thomas Lange¹, Jeff Snyder¹, Jürgen Hennig¹, Dominik von Elverfeldt¹, and Jan-Bernd Hövener¹

¹Medical Physics, Dep. of Radiology, University Medical Center Freiburg, Freiburg, Baden-Württemberg, Germany, ²Neurozentrum, scientific workshop, University Medical Center Freiburg, Freiburg, Baden-Württemberg, Germany

Introduction

Low-field NMR has received increasing attention in the recent years as several new approaches were published [1-3]. We present an experimental setup for a variable static field, specifically designed to aid the PASADENA/PHIP and SABRE experiment [4, 5]. PASADENA and SABRE are unique liquid-state hyperpolarization techniques which have generated ¹³C NMR signal enhancement of several orders of magnitude. As PASADENA relies on a RF spin-order-transfer sequence SOT [6] at low field (1.8 mT), fine calibration of the frequency and flip angle is key for the success of the experiment. As previous polarizers had no detection device at $B_0 = 1.8$ mT, these calibrations were time consuming, and required a high-field system for pre-polarization and signal detection. SABRE requires $B_0 \approx 6$ mT for optimal polarization yield. To address these needs and facilitate the polarization experiments, we developed an apparatus specifically for the fields required by SABRE and PASADENA ($B_0 = 1 - 6$ mT), suitable for RF calibration and the direct detection of NMR signal.

Materials and Methods

The static magnetic field B_0 is generated in a solenoidal coil ($r = 5.5$ cm, length = 36 cm, 1 mm wire diameter) driven by a power supply (Agilent E3615A). Strength and stability are monitored by a Hall probe (Lakeshore 475 DSP).

The excitation pulses are generated as text files prior to an experiment and loaded into a custom PC software that controls experimental parameters such as TR, phase cycling, pulse amplitude, sampling rate and post-processing (apodization, zero-filling). The actual transmission of pulses and signal acquisition is performed by a digital I/O card (National Instruments USB 6251 BNC) controlled by LabView. An audio amplifier (Onkyo TX-8555) is used to enhance the excitation pulse ($U_{\max} \approx 50$ V), which is generated by an untuned saddle-shaped B_1 transmission coil mounted on an acrylic tube within the B_0 coil (2 loops 10 cm \times 20 cm, each with 6 turns of 0.63 mm dia. wire). Crossed diodes reduce the noise generated by the amplifier during signal detection.

For the detection of ¹H-NMR signal, a solenoidal coil is employed, tuned to $f_0 = 77.9$ kHz ($r = 3$ cm, length = 6 cm, ≈ 120 turns of 0.50 mm diam. wire, inductance $L = 135$ μ H; capacitance $C = 32$ nF). The coil contains a vessel filled with tap water and is connected by a nonmagnetic coaxial cable with SMA plug to the I/O card.

To minimize the delay between signal excitation and detection, geometric decoupling between both coils is crucial (i.e. a 90° angle between the axes of both coils). To permit fine adjustment of this angle, a device was constructed allowing the receive coil to freely rotate in the isocenter of the setup. This was performed prior to each experiment. The quality of the decoupling was quantified by dividing the voltage of a pulse measured at the transmit coil by the voltage observed in the receive coil. External noise is reduced by a copper sheet (1 mm thickness) between the B_0 and B_1 coil and end caps (Figs. 1B, C).

Results

¹H NMR signal was readily detected at $B_0 \approx 1.83$ mT. To verify the NMR origin of the signal, the experiment was repeated at $B_0 \approx 2$ mT ($f(\text{spins}) \neq f(\text{pulse})$) where no signal is observable (Fig. 2). After 100 averages, an $\text{SNR}_{N=100} = 13$ was obtained (amplitude of Voigt fit divided by peak-to-peak noise amplitude). A delay between the end of the excitation pulse and signal acquisition is required to avoid detection of the ring-down of the receive coil. At a geometric decoupling factor $U_{\text{transmit}}/U_{\text{receive}} > 300$, a delay of 1.5 ms was chosen. Further shortening of this delay would result in moderate signal enhancement only, as the transverse relaxation time constant of tap water was measured (by varying the delay between excitation and detection) to be $T_2^* = (7.6 \pm 0.5)$ ms. A longitudinal relaxation time of $T_1 = (1.7 \pm 0.1)$ s using saturation recovery and a transversal relaxation time of $T_2 = (1.0 \pm 0.2)$ s measured by a spin echo experiment were obtained.

Conclusion

In this contribution, we present a low-field NMR system working at static fields (B_0) between 1 and 6 mT. The setup achieved a $\text{SNR} = 13$ for 100 averages at $B_0 = 1.8$ mT, and allowed the measurement of relaxation parameters e.g. of tap water ($T_2^* = (7.6 \pm 0.5)$ ms, $T_2 = (1.0 \pm 0.2)$ s, $T_1 = (1.7 \pm 0.1)$ s at 1.8 mT). The design allows the incorporation of this experiment into a PASADENA or SABRE polarizer. As a result, the efficiency and overall success of the hyperpolarization experiment is expected to be facilitated considerably, as the field strength and RF flip angles (decisive factors for the success of hyperpolarization experiments) can be calibrated and monitored with greater precision and speed than previous designs.

References:

[1] Aktham Asfour, IEEE 2008 [2] I. M. Savukov et al., JMR 185 (2007) 214-220 [3] Steven R. Parnell et al., 2008 Meas. Sci. Technol. 19 045601 [4] Bowers C.R., and Weitekamp D.P. (1987) J. Am Chem. Soc. 109: 5541-5542 [5] Ralph W. Adams¹, Juan A. Aguilar, Kevin D. Atkinson, Michael J. Cowley, Paul I. P. Elliott, Simon B. Duckett et al., science 2009 [6] Goldmann M., and Johannesson H. (2005) MRI. C.R. Phys. 6: 575-581

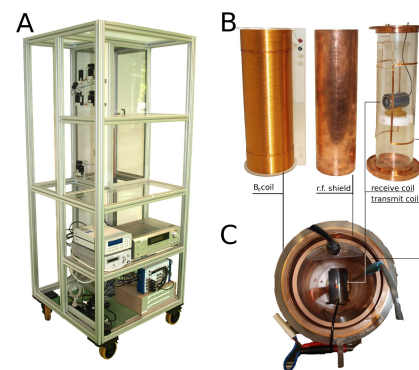


Figure 1: Low-field NMR unit: Entire setup (A), individual components (B_0 , B_1 coil and Cu-shield) (B) and assembled setup seen from above (C).

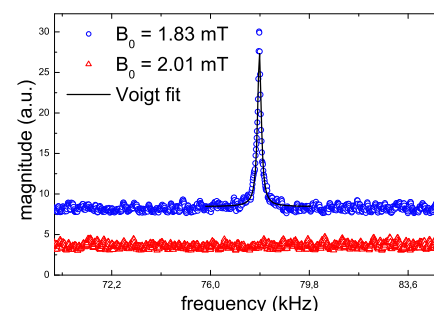


Figure 2: ¹H-NMR spectra of tap water obtained by pulse-acquire experiments ($N = 100$). An $\text{SNR} \approx 13$ was obtained after 100 averages. To verify the origin of the signal, the magnetic field was shifted to $B_0 = 2$ mT ($f_{\text{spins}} = 100$ kHz), while all other parameters were kept constant (frequency of the excitation pulse $f_{\text{pulse}} = 76$ kHz). Consequently, no signal was observed (red, bottom), thus confirming the NMR origin of the signal. (blue, top).

Experimental details: Pulse power $U_{\text{pp}} \sim 6.80$ V, pulse duration $t_p = 397$ μ s, $B_0 = 1.83$ mT, $T_R = 5$ s, acquisition time $T_a = 25$ ms, delay = 1.5 ms.