In-situ NMR – Apparatus and demonstration experiments

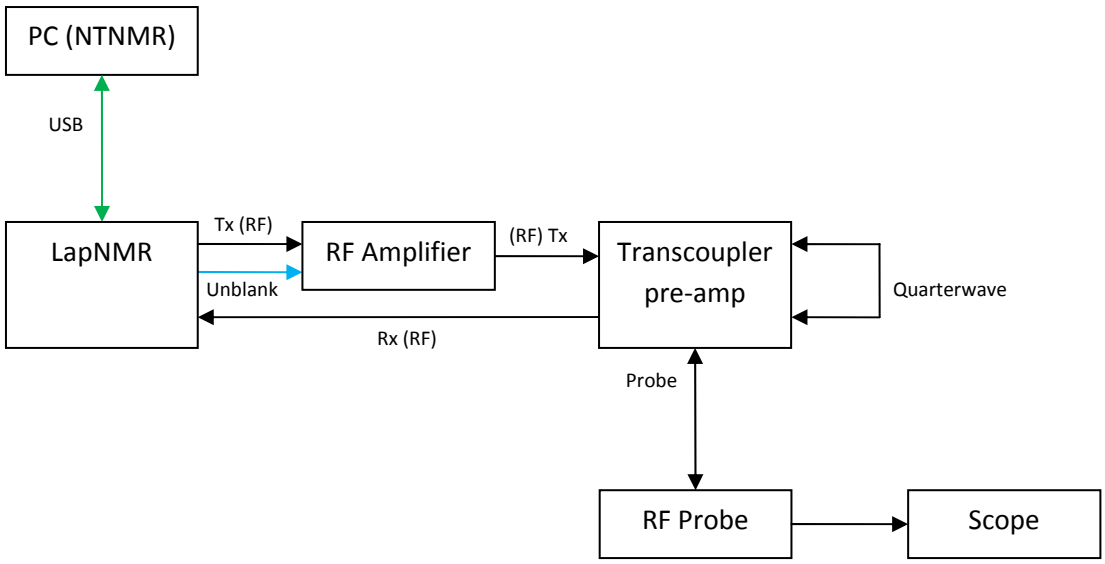
Introduction

The aim of this project is to deliver an NMR system than can measure signals from samples mounted in both NMR optimised and typical RF μSR coils. The primary objective is to provide a means of confirming coil functionality and calibrating the RF field strength prior to user experiments. However, it is hoped that the system may eventually find use as a general tool for in-situ NMR relaxation measurements.

Equipment

The spectrometer is based on Tecmag's LapNMR product (http://www.tecmag.com/lapnmr.html), which provides a complete broadband NMR spectrometer on an easily portable single board system. Portability and ease of setup are both vital for a system designed to work in tandem with a beamline experiment – the sample will typically be mounted within a muon spectrometer and the user will be asking for limited NMR data to confirm behaviour in that environment. The Tecmag system achieves this, requiring only that the unit is connected to an RF power amplifier (typically available for RF μSR experiments) and a laptop PC to make a fully functioning NMR spectrometer.

An overview of the connection of the system is shown below:



For testing, an AMT 3200 RF power amplifier capable of delivering up to 1 kW of RF power over a frequency range of 6-220 MHz was used. The RF probe was mounted in an Oxford Instruments OptistatCF cryostat (adapted for beamline measurements), although all results presented here were carried out at room temperature. The probe was built around a standard cryostat centre stick, with an appropriate RF coil mounted approximately 600mm away from a capacitance matching/tuning box – a parallel tank circuit was used for the test, with an additional capacitor placed in series to match the probe to 50Ω.

Example measurements

NMR measurements

Initial measurements were carried out using a coil optimised for NMR, consisting of a 5mm diameter solenoid of approximately 10mm length. Red rubber was used as the test sample since, for a solid material, it has a comparatively long T_2 in combination with a short T_1 to enable rapid signal averaging. The example signal (Figure 1) shows the proton free induction decay following a 4 µs RF pulse measured ~5 kHz off-resonance, the envelope is well modelled by an exponential decay and fitting suggests a T_2 relaxation rate for this sample of ~400 µs. The inset shows the early time data on an expanded scale, enabling an estimate of the recovery time for this particular configuration (~15 µs) to be made.

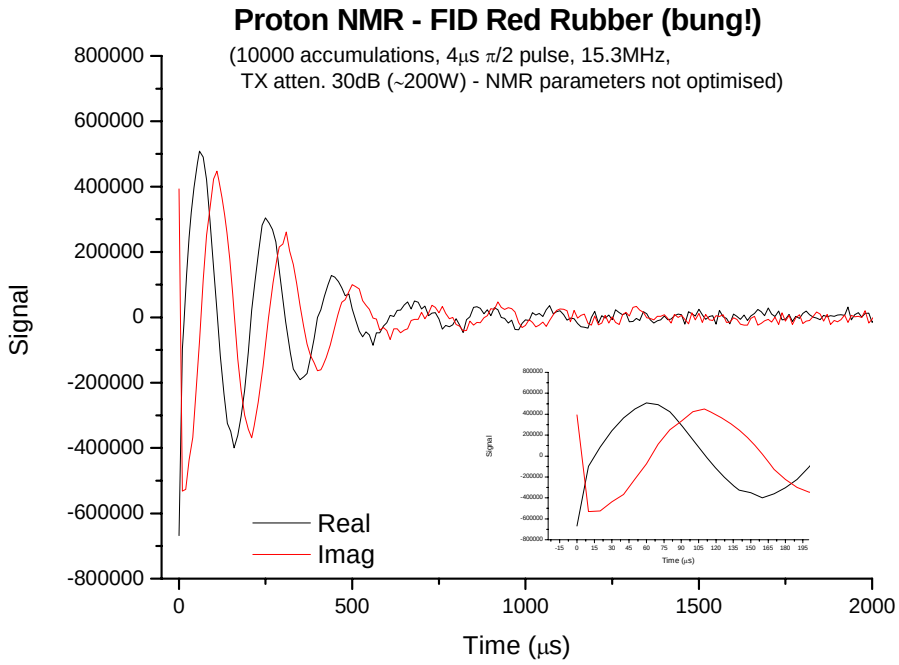


Figure 1: Proton NMR signal from red rubber with an expansion of the early time data shown (inset) to identify system recovery time.

Pulse length and RF field strength calibration using NMR

With the primary objective of the NMR apparatus being to provide a means of characterising RF coils in advance of-beam experiments, a test experiment was carried out to demonstrate useful information can be obtained. In this case the measurement was carried out using the sample and optimised NMR coil described previously.

The NTNMR software was configured to measure 128 single pulse spectra with the RF pulse width stepped from 0.5 µs to 32 µs. Display is initially as a 2D grey scale image (Figure 2) representing the variation in signal intensity through the spectra, although cross-sections can easily be defined as shown in figure. Analysis of this data suggests an RF field strength of ~321 kHz, giving an optimum $\pi/2$ pulse length of 0.8 µs.

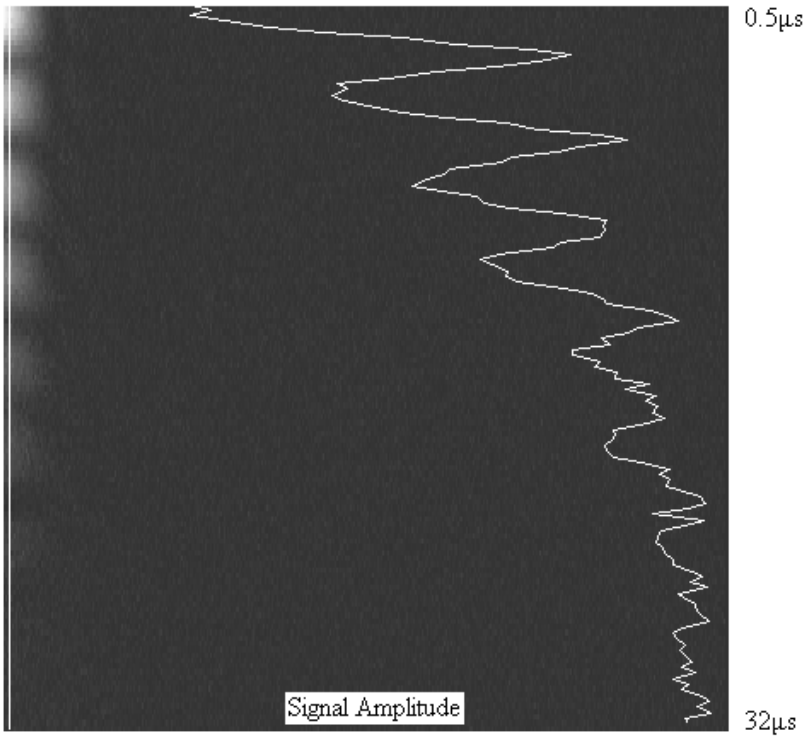


Figure 2: Scanned RF pulse length to determine optimum $\pi/2$ pulse and the corresponding RF field strength. A grey scale representation of the signal amplitude is shown (left) together with a cross-section plot (right).

NMR measurements from typical RF μSR coils

A test experiment was also carried out to demonstrate that useful NMR data could be obtained from typical RF μSR coils; these are large but poorly shaped (~ 20x20x2.5 mm), and are designed to maximise the sample area presented to the beam.

The RF coil was tuned and matched at 15.885 MHz and, as before, data was taken ~5 kHz off-resonance. The proton signal measured for red rubber following a 4 μs RF pulse (determined to provide an approximate $\pi/2$ pulse) is shown in Figure 3. As expected, the non-optimised coil geometry compromises measurement of the NMR signal: recovery time is extended to ~80 μs and the signal to noise ratio is significantly reduced. However, the FID remains well defined, and will clearly yield acceptable results as the pulse length is scanned to determine the RF field strength.

Conclusion

This work has resulted in a portable NMR apparatus suitable both for on-beam relaxation measurements and, of particularly importance, off-line coil characterisation. Despite the poor geometry of typical RF μSR coils, NMR signals can successfully be measured and the system used to quantify important coil parameters, such RF field strength, and coil reliability.

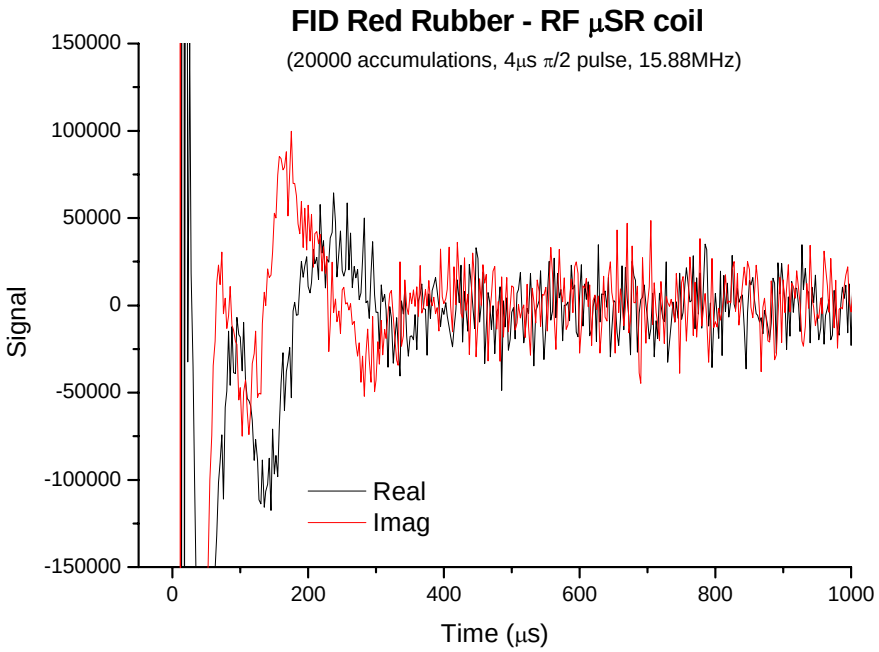


Figure 3: Proton NMR signal measured in red rubber mounted in a typical RF μSR coil.

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5th May, 2010