Low magnetic field manipulation of $^3$He spins using
digital methods

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Abstract. Simple methods for manipulating $^3$He spins at low magnetic fields using low cost
digital I/O cards are presented. A brief overview of a digital NMR spectrometer is given along
with practical information on the implementation and an assessment of the losses per pulse.
Through the use of coils with different quality factors the effects of radiation damping are
investigated and assessed in the context of the sample polarisation using neutron transmission
measurements. The hardware is further used to synthesize an adiabatic swept pulse which is
used to reverse the $^3$He spin direction, allowing the selection of a particular neutron spin.

1. Introduction
Spin-polarised or 'hyperpolarised' (HP) noble gases are now routinely used across a range of
disciplines such as neutron physics [1], polarised targets [2] materials science [3] and medical
imaging [4]. The high level of polarisation is usually created by using either metastable [5, 6]
or spin exchange [7] optical pumping. The optical pumping is typically performed in a low
magnetic field, $\sim$ 10-30G, which defines the optically pumped state and minimises polarisation
losses due to magnetic gradients. These fields correspond to Larmor frequencies of the order
$\sim$ 30-100kHz (for $^3$He), which can now be easily synthesized using low cost digital I/O cards
allowing filtering, RF synthesis and timing to be easily controlled using a minimal amount of
hardware. Due to the high nuclear polarisations which are now routinely obtainable [8, 9], it is
possible to detect the magnetisation of the gas with no additional pre-amplification.

2. Polarimetry by NMR
The standard method of measuring the relative polarisation of HP $^3$He is by pulse acquire
NMR which measures the free induction decay (FID) signal from the excited nuclear spins. For
most measurements it is important that the fraction of spins destroyed by each measurement
is small in comparison to the total magnetisation. Small surface coils on the cell walls are
used as standard. Accurate measurement of the polarisation is necessary in many different
applications of hyperpolarised gases. For example in studies into polarisation limits [9] where
the X-factor determination requires accurate knowledge of the polarisation build up or 'spin

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up’ and relaxation ($T_1$) time constants. Or neutron scattering where knowledge of the $^3\text{He}$ polarisation is used to calculate the neutron polarisation and hence correct for the polariser efficiency.

For data analysis the shape of the FID is well described by equation 1:

$$f(t) = \frac{1}{2} V_{pp} g(t) \sin(\omega t + \phi)$$

where $V_{pp}$ is the peak-to-peak voltage at $t=0$ with $\omega$ the difference between the Larmor frequency and the transmit frequency of the pulse, $g(t)$ the envelope function to describe the FID lineshape and $\phi$ an arbitrary phase angle to account for the phase shift between transmit and receive pulse.

2.1. Hardware

The hardware required for implementing a simple digital NMR spectrometer consists of a PC with a digital I/O card (e.g. NI-6251, 1.25MHz), a BNC connection box, a home made T-R switch (duplexer)[10] and a combined transmit/receive ($T_x/R_x$) coil, which is grounded to the aluminium frame of the coils. In addition a variable capacitance box (100pF to 11$\mu$F) is used to adjust the tuning of the coil such that the resonance frequency can be easily changed for use at different magnetic field strengths. In-situ polarimetry of gas in the cell typically relies on small tip angle NMR from a coil close to the cell. The transmit/receive coil is 3cm in diameter, hand wound from 0.25mm wire with $\sim 300$ turns and has a quality factor $Q$ of 10. For higher values of $Q$ the coil is spoilt using a resistor of a few ohms. Typical ring-down times for the coil are of the order 6ms. It is important to have a low $Q$ coil as distortion of the FID can occur due to the high level of magnetisation (see section 2.3).

The simplicity of the hardware is due to the passive T-R switch, which consists of 4 (high speed 1N4148) diodes and a resistor. This enables the use of the single $T_x/R_x$ coil to measure longitudinal ($T_1$) and transverse ($T_2^*$) relaxation time [11] in different regions. The spectrometer has been used to assess and optimise polarisation losses in end compensated solenoids, mu-metal boxes [12] and Helmholtz coils. Such optimisation is performed by minimising the NMR linewidth i.e. achieving a long $T_2^*$. However, care must be taken to sample enough of the volume of the cell as the small diameter coil only samples a small region. To overcome this limitation it is possible to use this system with larger coils, which encompass the whole cell volume and inevitably the whole cell $T_2^*$ is shorter due to the $B_0$ inhomogeneity. However, this introduces a problem as the larger coils will tend to destroy more polarisation per excitation pulse.

2.2. Losses

In order to accurately measure the spin up and relaxation ($T_1$) constants it is essential that the destruction per NMR pulse is negligible or well characterised. In order to check this we performed a series of 500 pulses sequentially with a total measurement time of $\sim 25$ minutes. As the $T_1$ of the cell under investigation was 90 hrs (measured via neutrons[13]) then any contribution from the normal decay is negligible and all losses are assigned to the NMR. Shown in figure 1 is the initial amplitude of each pulse ($V_{pp}$). The total destruction per pulse is $1 \times 10^{-4}$ per pulse. Figure 1 inset shows a typical pump up of a cell with a spin up constant of 849 $\pm$ 8.8 minutes.

If we correct the data for the pulse destruction losses and fit to this data we get an increase of less than one part in ten thousand, therefore these losses are accepted as negligible.

2.3. Radiation Damping

It is important to note the effects of the high magnetisation on the observed FID from the optical pumping cell in both the high and low energy states and the effect of radiation damping on the observed total magnetisation. Radiation damping [14] occurs when spins are tipped from
Figure 1. Destruction per pulse. Inset : typical spin up data uncorrected for polarisation losses.

Figure 2. Spin up curve for the high Q coil with the peak voltage determined by fitting of the FID to equation 1. Inset :FID’s taken at various levels of polarisation, indicated (i),(ii) and (iii).

a longitudinal to a transverse plane. For sufficiently high magnetisation the precessing spins create a ‘counter field’ in the receive coil which acts to re-orientate the magnetisation back into the longitudinal plane. This has two principle effects upon the measurement. The first is that the effective tip angle experienced depends upon the sample magnetisation and $T_2^*$. Secondly the measured FID $T_2^*$ or linewidth (in the Fourier domain) will change with sample magnetisation.

Radiation damping [15] changes the shape of the FID by addition of a term sech(t/$\tau_{rd}$) to the original $T_2^*$ decay where $\tau_{rd}$ is the radiation damping time constant and therefore is observed for long $T_2^*$ values. $\tau_{rd}$ is given in SI units by;

$$\tau_{rd}^{-1} = Q\gamma m_0\eta \mu_0 / 2$$  \hspace{1cm} (2)$$

where $Q$, $\gamma$, $m_0$, $\eta$ are the coil quality factor, gyromagnetic ratio, longitudinal magnetisation
density and filling factor respectively [16]. Radiation damping from $^3$He gases has been observed in a number of different studies. For example MRI studies at low field (20.6 G) were performed by Wong et al [17]. These identified radiation damping by using a constant RF pulse and different tuning of the receive coil. They noted a difference in the calculated tip angles indicative of radiation damping. At higher fields (1.5T) effects have also been observed [18] using highly polarised samples in birdcage resonators.

In order to investigate these effects the polarisation build up was monitored using the spectrometer detailed earlier with two orthogonal $T_x/R_x$ coils of different Q. For low Q and $m_0$ the radiation damping should be negligible however at higher values radiation damping effects can occur, these will be manifested in a narrowing of the FID linewidth. In order to observe this the two coils of differing Q were mounted on the same optical pumping cell, hence measuring the same $m_0$. The coils were in a direction mutually orthogonal to the $B_0$. The cell used in this investigation was a 2.0Bar cell with a saturation polarisation of 69% [13], polarised in the low energy state by spin exchange optical pumping using a 40W narrowed external cavity diode laser (ECDL) [19].

The different Q values were obtained by winding different number of turns (100 Q=35 and 300 Q=3), in addition the 300 turn coil had a 10Ω resistor in series in order to artificially dampen the Q. Q was measured using a function generator and search coil to transmit and the pickup was measured using an oscilloscope connected to the coil. All experiments were carried out in a $B_0$ field of ~10G using a Helmholtz pair (diameter 0.8m) with a uniform magnetic field over the cell volume. Pulses were 0.7V with a duration of 1.2ms and in each case the coils were pulsed sequentially with a delay of ~1s between them, therefore over the course of a spin up (~12 hours) they are measuring the same magnetisation (within error).

Shown in figure 2 is the initial FID voltage ($V_{pp}$) as a function of time whilst a 2 Bar $^3$He cell is polarised. This is measured using the high Q coil, the inset shows the recorded FID’s. clearly observable is a shortening in the decay time with increasing magnetisation (polarisation).

Figure 3 shows the change in linewidth with increasing polarisation (spin-up time) for the two coils. The low Q coil has a smaller change, during the start of the measurement at low $m_0$ both coils have a similar observed linewidth and converge to a value $\ll 200\text{ms}$ representing the background $T_2^*$, however with increasing polarisation the higher Q coil has an observed $T_2^{obs}$ which changes more significantly. Analysis of spin up time gives two different times ($Q=35$ $849 \pm 9\text{min}$ and ($Q=3$) $722 \pm 26\text{min}$). This is consistent with the high Q coil having a lower effective tip angle as expected due to the radiation damping and shows that any polarisation measurements inferred from NMR with this coil are more sensitive to error, especially at high polarisation.

Absolute measurements of the $^3$He polarisation were performed on the neutron beamlines POLTAX and CRISP[13, 20] whilst simultaneously measuring the polarisation via NMR. Typical NMR data for two different coils is shown in figure 4. As the neutron transmission measurement is an absorption measurement it is not affected by radiation damping effects and hence is an accurate measurement of the gas polarisation. The data was collected using a two coil system similar to the lab system described earlier. A polarised $^3$He cell of 2.16 Bar was placed in a ‘magic box’ [12] and allowed to decay whilst simultaneously measuring the relative gas polarisation using NMR and absolute polarisation using neutrons. The neutron data was measured as a time series and interpolated to match the NMR data. In these measurements good agreement was obtained between the NMR and neutron data for coils with Q of up 10, as indicated by the linear fit to the data. From equation 2 it can be seen that the total ‘figure of merit’ ($Q \times \text{pressure} \times \text{polarisation}$) is at least as high as $\sim 12$. However this is only valid for monitoring the $^3$He polarisation in the low energy state.

Shown in figure 5 are a series of FIDs when pumping in the high energy state, this was done by adjusting the circular polarisation of the pump beam (relative direction of the $B_0$ and $\lambda/4$
Figure 3. Linewidth $^{-1}$ from exponential fit for the two different coils during polarisation build up.

Figure 4. NMR peak voltage for two different coils as a function of $^3$He polarisation measured by neutron transmission. Line is to indicate the linearity between the neutron and NMR measurements.

polarising plate). The data is labelled (i), (ii) and (iii) in increasing polarisation. The form of the decay is non-exponential obeying a simple sech relationship and can be used to determine when pumping in the high energy state, however no reliable NMR polarimetry can be performed with these FID’s for most cases it is preferable to reverse the spin state using adiabatic fast passage (AFP) [11] methods.
Figure 5. Typical FID data obtained from $^3$He pumped into the high energy state. Polarisation (magnetisation) increasing with number.

3. $^3$He spin reversal

AFP methods have been used in conventional NMR for decades [11], however recently these techniques have been applied to neutron spin filters on neutron beams allowing the construction of a combined neutron polariser and flipper [21, 22]. These rely on the use of an effective magnetic field which the nuclear spins follow from an alignment parallel (anti-parallel) to the holding field $B_0$ to an alignment anti-parallel (parallel).

Practically the RF field waveform is a sine wave which is swept through the Larmor frequency of the $^3$He, in addition to this the function is convoluted with a Gaussian envelope. This envelope serves to avoid abrupt changes in the total magnetic field which may give rise to non adiabatic transitions and lead to depolarisation. This results in a function of the form:

$$ B_{RF}(t) \propto \exp \left( -\frac{[\omega(t) - \omega_L]^2}{\sigma^2} \right) \sin [2\pi \omega(t) \times t] $$

where $\omega_L$ is the resonant frequency and $\sigma$ is the width of the Gaussian envelope. Earlier systems [22] employed dedicated analogue electronics for the synthesis of the swept waveform, in this work the polarisation losses were $2 \times 10^{-5}$ per AFP flip. These losses were attributed to noise in the AFP electronics. In a more recent work the same digital I/O card used for NMR in the earlier section was adapted to provide digital synthesis of the RF AFP waveform[23]. With an additional analogue amplifier losses of $(1.78 \pm 0.03) \times 10^{-5}$ per AFP flip were obtained. Moreover the digital system requires minimal hardware and can be easily reproduced allowing routine spin selection of the neutron polarisation with a negligible effect on the $^3$He gas polarisation.

Combining this on a neutron beam line with the NMR allows for a compact polarimetry system which is capable of determining the polarisation and $T_1$ of the gas (via NMR), which
is necessary for polarisation corrections along with rotation of the polarisation (via AFP) and monitoring the magnetisation state of the gas (via FID lineshape).

4. Discussion
The work presented in this paper illustrates how simple instrumentation for polarisation manipulation and measurement of $^3$He can be produced. Such systems can also be used for other noble gases such as $^{129}$Xe [24], however for low $^{129}$Xe concentrations averaging is required to achieve a high signal to noise ratio. Furthermore the modularity of these digital systems allows additional outputs to be synchronised with the $T_x$ pulse allowing the addition of gradients (with suitable amplifier and coils) to the system which can be utilised for the measurement of cells pressure, temperature and polarisation distribution [25].

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