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A Microcontroller System for The Automation of Transient Effect Determination of the Spin-Lattice Relaxation Time Using Continuous Wave NMR †

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Abstract: A simple transient effect method for the determination of the spin-lattice relaxation time using continuous wave NMR (TEDSpiL) with a marginal oscillator was recently reported (doi:10.1002/mrc.4594). Such a sys。tem measures a parameter, called T_x, that is related to T_1 and allows T_1 to be determined with the aid of calibration samples. For such a system, the process of making the T_x measurement does not require variable parameters and so is ideal for implementing in microcontroller code. In this article, we demonstrate that TEDSpiL may be automated using two microcontrollers from the Teensy family to make a low power and portable system. .

Keywords: Continuous wave NMR; microcontroller; Teensy; TEDSpiL; spin lattice relaxation time; T1

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1. Introduction

Nuclear magnetic resonance (NMR) finds many applications from the chemical finger prints of NMR spectroscopy [1] to the whole body magnetic resonance imaging (MRI) scanners found in many hospitals [2]. Two of the parameters that can be used in the production of MRI images are the spin-lattice (T_1) and spin-spin (T_2) relaxation times and measurements of these have been used in a wide variety of sensor applications that may use permanent magnets and less expensive electronics[3-5]. Most applications have used pulsed NMR for these measurements however continuous wave (CW), which was the predecessor to pulsed, is conceptually simpler and with simpler electronics but often more difficult in the implementation and so fell out of favor. Recent advances in permanent magnet technology and speed of low cost electronics have given some interest in the topic and the electronics has been recently reviewed [6]. A simplified view of NMR considers the intrinsic nuclear magnetic moment of certain nuclei to be like microscopic bar magnets that tend to orientate generally aligned with or opposed to the presence of a strong magnetic field (B_0). Radio frequency (rf) radiation can be absorbed at a frequency that satisfies the Larmor condition $\omega=\gamma B_0$ (where ω is the angular frequency and γ the gyromagnetic ratio which is a constant for a given nuclei) corresponding to energy difference between the two states. In CW-NMR either the frequency of the rf can be fixed and the magnetic field swept through the resonance condition, or the magnetic field held constant and the frequency of the rf changed. Where the field is swept, an alternative strategy is to use a marginal oscillator where the oscillation amplitude dips as it passes through resonance[7,8]. Look and Locker [9] demonstrated that there is a transient effect when the sweep of the magnetic field is initiated and that this may be used to determine the value of T_1. More recently, a simple variation that used a marginal oscillator and calibration samples was presented which was referred to as the transient effect determination of the spin-lattice relaxation time using continuous wave NMR or TEDSpiL [10].

In this work we demonstrate that TEDSpiL may be automated using two microcontrollers from the Teensy family.

2. Materials and Methods

The schematic diagram of the continuous wave system is shown in Fig. 1. The sample is placed in a coil which forms part of a marginal oscillator tank circuit. For convenience, we used a commercial marginal oscillator operating from around 14MHz to 21MHz (LD Didactic GmbH, Hürth, Germany 514-602 http://www.ld-didactic.de/) however there have been many different marginal oscillator circuits published which have recently been reviewed [6]. The coil used, also produced by LD Didactic as part of their NMR probe bar (514-606), is placed in the fixed magnetic field with sweep coils that allow the field to be swept about the resonance value. The fixed field can be produced by using Neodymium Iron Boron (NdFeB) magnets as shown in Figure 1 or by electromagnet coils however the latter do draw significant current and cause elevated sample temperatures. A digital to analogue converter on a teensy 3.5 [11] microcontroller was used to generate 100 cycles of a voltage ramp which was amplified, using a power amplifier built from a 2N3055H power transistor and LM 358N buffer amplifier [10] and providing a gain of five, before driving the sweep coils (part of the 514-606). The oscillator frequency was adjusted such that the resonance occurred at the center of the ramp. The ramp was started using a push button attached to a digital input (pin 12) on the Teensy 3.5 microcontroller and a digital output (pin 11) was used to trigger a Teensy 3.6 [12] to make measurements of the marginal oscillator output after it had been amplified by a simple inverting op-amp (MCP6002) with 1kΩ input resistor and 10 kΩ feedback resistor giving a gain of -10. A LCD display was attached to pin 10 of the Teensy 3.5 to display the calculated value of T_x. Code for the Teensy 3.5 is shown in supplementary S1.

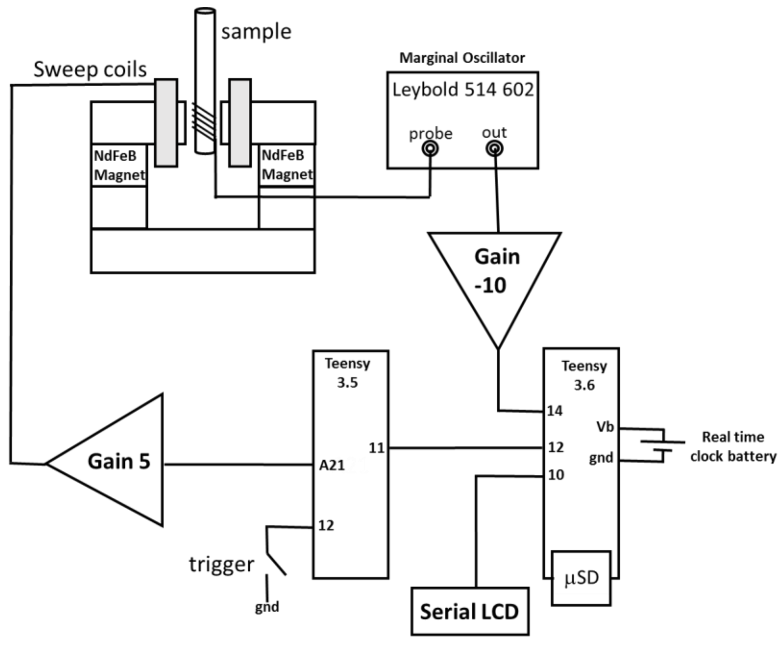

Figure 1. Schematic diagram of the experimental arrangement. Only the power (4.5V to 'Vin' pins) and ground (power ground to pin 'GND' and analogue input ground to 'Analog GND') connections have been left off the Teensy circuits and the gain -10 amplifier is an inverting op-amp.

The Teensy 3.6 is a 32 bit 180 MHz ARM Cortex-M4 processor with floating point unit, 256 Kbytes of RAM and 1024 Kbytes of flash memory including an on-board μSD card holder and under US$30. It contains two analogue to digital converters (ADC) with a usable resolution of 13 bits. Much of the material for the Teensy has been developed open source by Paul Stoffregen and his team at pjrc.com however there is an implementation library for the ADC created by Pedro Villanueva [13] which allows the full power of the ADC to be utilized

For maximum speed, data is saved into an array in memory and the maximum array size for 16bit sample data is around 64000 data points. This is not sufficient to capture the whole of the data for 60 sweeps at 40Hz and a lower sample rate may not clearly identify the peak value. To allow for this, the Teensy 3.5 provided a trigger pulse to the Teensy 3.6 a quarter of the way up the ramp sweep so that only the 1000 points around the peak were captured to an array in memory. It was then a straightforward loop to find the maximum value within that data to give the peak value which was stored in a separate array of peak values.

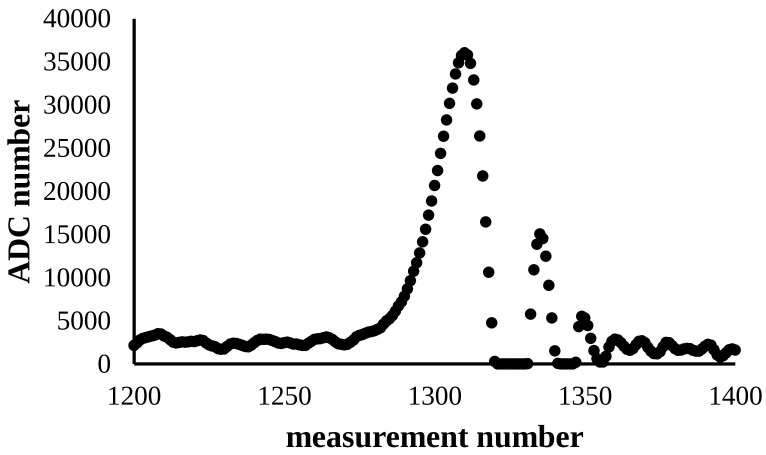

Figure 2. The number returned from the Teensy 3.6 ADC from the amplified marginal oscillator signal for a single magnetic field sweep. This demonstrates that the Teensy ADC is capable of accurately capturing the peak amplitude.

The Teensy 3.6 code then carries out a simple least squares fit on the natural log of the peak values and the gradient gives a value for T_x which is displayed on the serial LCD display. The time from pressing the trigger to the display of T_x is less than five seconds and the system then waits a further ten seconds before the trigger can be pressed again to allow the sample to equilibrate away from the resonant value of magnetic field. All the data from the arrays and the peak values were stored to the onboard μSD card with the real time clock being used to generate a file name which is the last 8 digits of the UNIX time stamp of when the measurement was made. It should be noted that the Teensy 3.6, unlike to 3.5, is not 5V tolerant so the amplifier is supplied with only 3V from two 1.5V batteries and both the Teensy 3.5 and 3.6 are supplied with 4.5V from three 1.5 volt batteries. In our circuit, both amplifier and Teensy shared batteries to save weight and the amplifier supply was 'tapped' off.

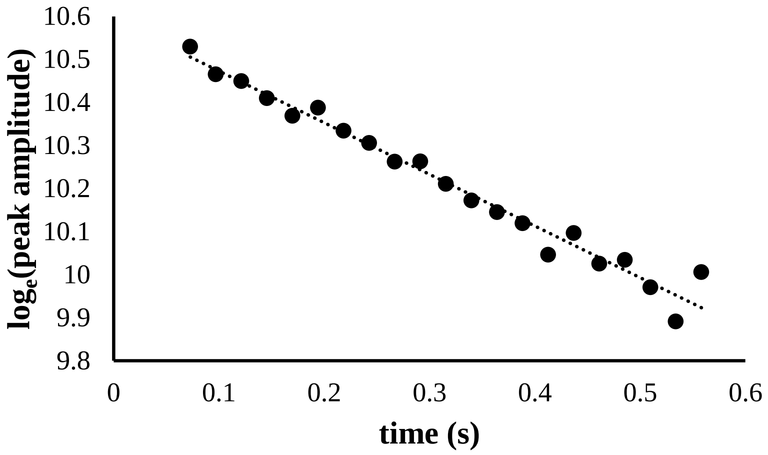

Figure 3. Typical example for water of natural log of the peak amplitude from the amplified marginal oscillator signal as a function of time for peaks 5 to 25. The gradient of the linear fit, shown as the dotted line, gives a value for T_x.

3. Results

Figure 2 shows the number returned from the Teensy 3.6 ADC from the amplified marginal oscillator signal for a single magnetic field sweep. With a sampling speed of around 160kSample/second, this demonstrates that the Teensy ADC is capable of accurately capturing the peak amplitude. Note that the positive going 'wiggles' that are common in this type of system [6] are also visible but as we are only interested in the peak value, no offset is provided to the amplifier to capture the negative going part of the 'wiggles'. By triggering the 3.6 to capture part way through the sweep ramp it is easily possible to capture up to 60 peaks with the available RAM but only around 25 peaks are required to get a reasonable linear fit to provide a value for T_x; the first 5 values were also discarded.

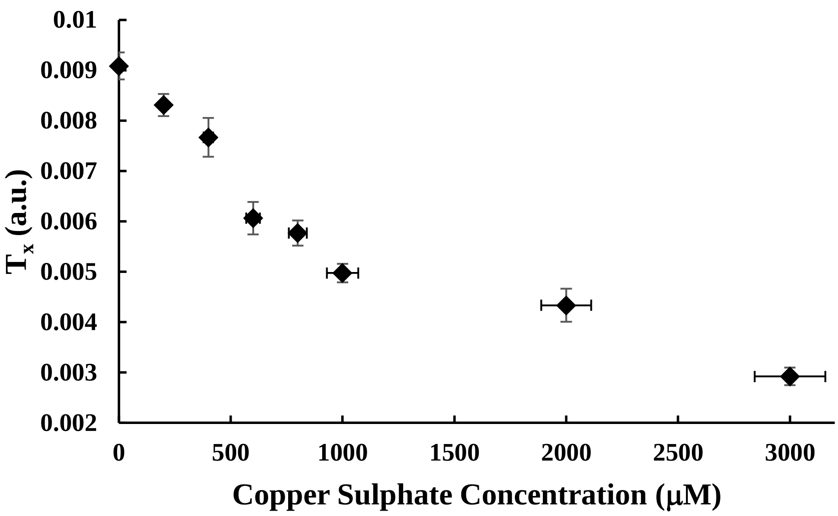

Figure 4. T_x values as a function of concentration of Copper Sulphate in water.

Figure 3 shows a typical example, for a sample of water, of natural log of the peak amplitude from the amplified marginal oscillator signal as a function of time for peaks 5 to 25. Note that the scatter in the data increases as the marginal oscillator peak amplitude tends to the equilibrium value so including peaks beyond the 25th does not improve the T_x measurement. In Figure 4 we show a set of T_x values as a function of concentration of Copper Sulphate which is often used as a standard test set of T_1 values in pulsed NMR; the change is consistent with data reported for pulsed NMR [14,15].

4. Discussion

The electronics, commonly referred to as the console, for low field pulsed NMR systems typically cost in excess of $20k as they are designed to be wide band and highly programmable spectrometers. The process of making T_1 measurements with such a pulsed system usually use recovery sequences that will typically take many minutes to give a single T_1 value. Whilst the T_x value is not a direct equivalent to T_1, there are applications where the use of T_x may provide a cost effective measurement solution. One such application may be to sample the outlet water from constructed wetland treatment bed for coal mine drainage water. Such water often contains iron oxide, which is a known T_1 contrast agent, and the presence of a significant amount of iron oxide in the water would indicate that a constructed wetland bed was no longer functioning correctly and remediation work was required. As there are environmental, and possibly financial, consequences for clogged beds discharging untreated water the measurement system reported in this article could form the basis of an appropriate monitoring system.

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