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THE TRIUMF POLARISED DEUTERON TARGET NMR SYSTEM

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ABSTRACT

An NMR system has been built for a small volume polarized deuterium target that has been recently been commissioned at TRIUMF. The target material consists of 1.3 cc of deuterated butanol beads which are contained in the coil of a tuned circuit and is cooled to 100 mK in a $^3He/^4He$ dilution refrigerator. A frequency synthesizer under the control of an 8085 based microprocessor (TRIMAC) provides the RF frequency sweep. A Starburst (J-11) and an LSI-11 are used to process the NMR signal. The RF phase angle is measured and is used to generate a correction voltage which is fed back to keep the tuned circuit at resonance and permit the measurement of the real part of the NMR signal. The signal normalization factor (TE signal area × temperature) is measured with an accuracy of 8%. The best polarization achieved so far was -.37 under target laboratory test conditions. Polarizations of -.33 were routinely achieved during a three week run in the TRIUMF M11 pion beam.

1. Target

A polarized deuteron target has been constructed for experiments to study the spin dependence in (π,D) scattering and absorption reactions. The target consists of 1.3 cc of deuterated butanol ($C_4D_{10}O$) and 5% D_2O doped with 6×10^{19} atoms per ml of EHBA. Target beads 1 mm in diameter are held in an FEP container which also serves as a support for the NMR coil. The target is cooled by a dilution refrigerator which is a reconstruction of a ^3He evaporation refrigerator system originally built at the University of Liverpool [1]. Magnetic fields up to 2.53 Tesla are obtained with a superconducting magnet that is incorporated into the refrigerator cryostat structure. A 100 mW Impatt sourve provides microwaves in the frequency range from 69 to 72 GHz. The target polarization is enhanced at power levels of 1 mW delivered to the target microwave cup. The microwave frequency is measured with an EIP model 548A microwave frequency counter and is stabilized by computer control to within 1 MHz.

2. RF Tuned Circuit

The NMR tuned circuit consists of a 48 turn coil of 0.01 cm diameter copper wire bonded to an FEP target holder in series with a voltage controlled capacitor. The NMR electronics is shown in fig. 1. The capacitor consists of 2 NE41137 FET's in parallel with the drains and sources connected to ground and the gates connected to the coil. The resonant frequency can be varied by applying a voltage to the gates of the FET's through a 1 MegOhm resistor. A tuning range from 16.3 to 17.5 MHz can be achieved by applying voltages from -1.0 to 0.5 V.

The RF is supplied to the coil at a constant current which is set by a 1K resistor. The resistive load offered by a peak in the negative enhanced

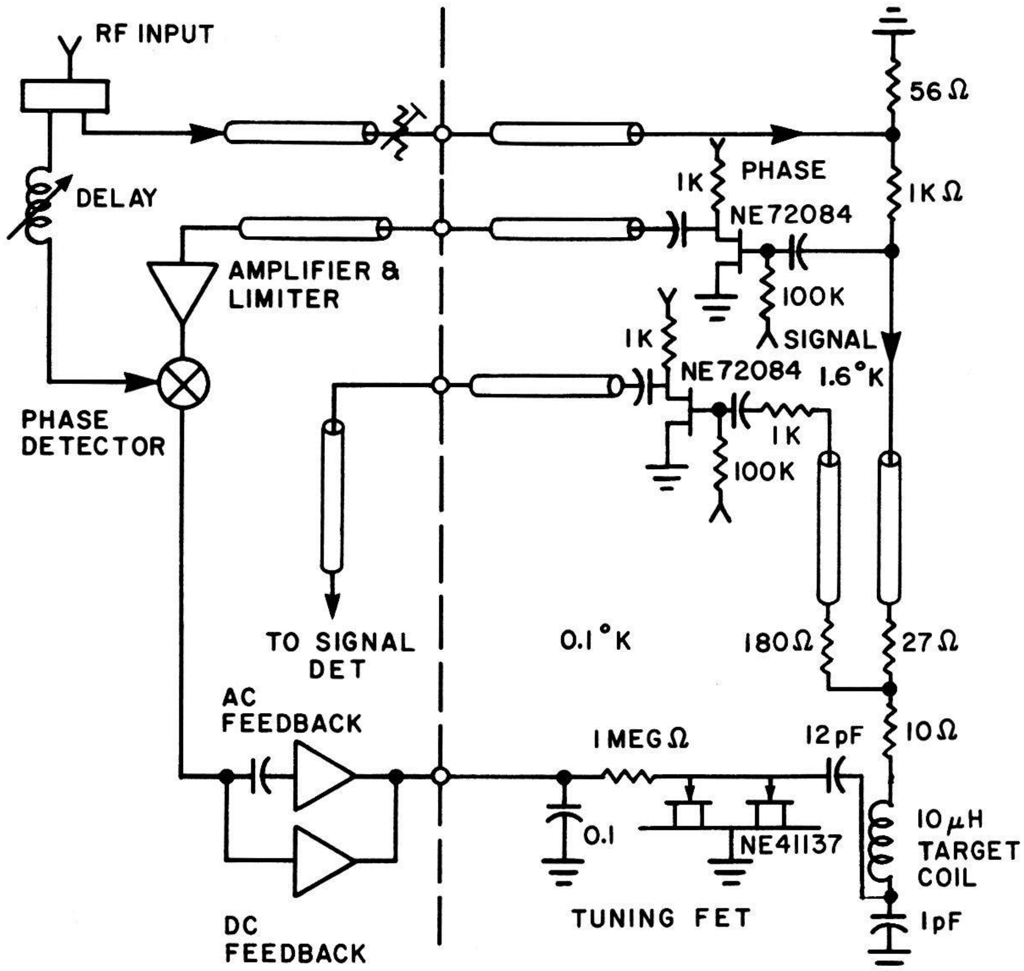


Fig. 1. Block diagram of NMR electronics. The components to the right of the dashed line are located inside the refrigerator.

deuteron signal of .37 is about 10 Ohms which introduces a 1% nonlinearty in the amplitude of the peaks relative to the pedestals of the signal. A series resistor is used to fix the Q of the tuned circuit to a value of about 60. The total dissipative load without an NMR signal is about 15 Ohms. Thus a negative enhanced polarization in excess of about .55 could be measured before the RF signal would saturate.

The signal detector amplifier is mounted on the refrigerator condenser and operates at a temperature of about 1.6°K. It consists of an NE72084 FET operated with a source to drain voltage of 2 V. The power dissipation is about 1 mW. The signal is fed from the coil to the amplifier through 40 cm of 50 Ohm BeCu semi-rigid coaxial cable. It would be preferable to mount the amplifier directly above the coil and this was attempted with limited success. However during a TE measurement this caused an uncertainty in the target temperature due to the proximity of the thermometers to the 1 mW heat source of the amplifier. In addition this heat caused the target to depolarize when it was operated in frozen spin mode.

3. RF Phase Compensation

The total susceptance of the series tuned circuit is given by:

$$\chi = \chi_o + \chi'(w) - j\chi''(w)$$

The polarization is proportional to the integral of $\chi''(w)dw$. If a series tuned circuit is phase compensated and kept at resonance the NMR signal is proportional to χ'' [2,3] which is the dissipative part of the circuit impedance. The background is essentially flat with the phase compensation circuits operative and this solves the dynamic range problems inherent in searching for a small signal in large Q curve shaped background.

The phase detector amplifier is identical with the signal detector amplifier and is mounted on the condenser beside the signal amplifier. The phase angle of the RF on the coil is measured by sampling the voltage on a 27 Ohm resistor in series with the coil. The phase detection could in principle be done with the signal detector directly on the coil but the RF amplitude is smaller and the phase is more difficult to measure.

In the first attempt to compensate for the phase angle the feedback circuit consisted of an amplifier with a time constant of 10 µs. It was found that the tuned circuit would slowly drift in and out of resonance. A second amplifier with a longer time constant of 0.5 s was placed in parallel with the fast amplifier and this solved the long term stability problem. The phase angle can be set to any arbitrary value with a dc offset which is incorporated into the feedback circuit.

Figure 2a shows a typical 16.5 MHz RF signal swept over 512 KHz with no phase compensation. The variation of phase angle is seen to be 28.8% of 360°=104°. The variation in RF amplitude due to the tuned circuit is also apparent. Figure 2b shows the effect of closing the feedback loop to set the phase angle to zero degrees. Figure 3a and b shows a Q curve observed on a spectrum analyzer before and after applying the phase compensation. The voltage variation of the Q curve is reduced by a factor of 20 or more when the phase loop is closed.

a) b)

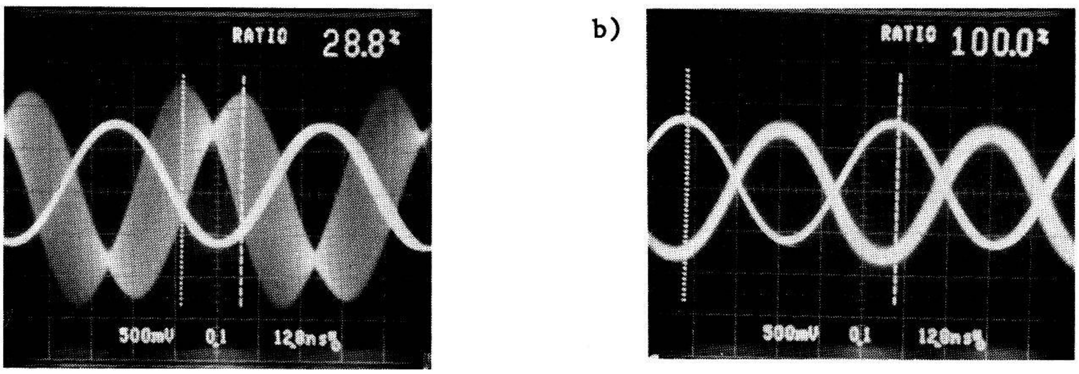
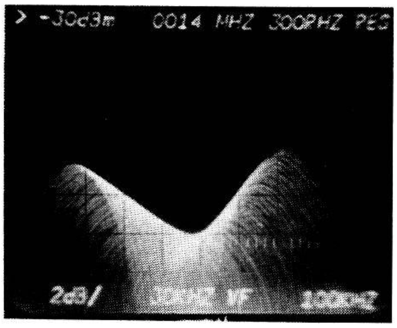


Fig. 2. a) The oscilloscope display shows the relative magnitude and phase of the RF signal from the tuned circuit relative to the source without phase compensation. b) RF signal with phase compensation.

a)



b)

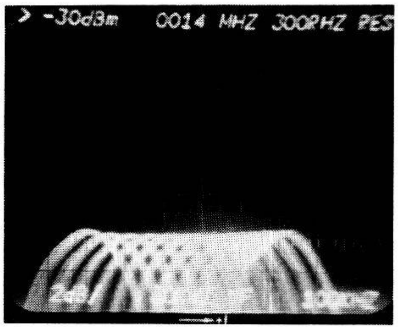


Fig. 3. a) Q curve on a spectrum analyzer without phase compensation.
b) Q curve on a spectrum analyzer with phase compensation.

4. Magnetic Field Influences

In the development phase of the NMR system it was found that the RF carrier amplitude depended on dB/dt as well as on B. These effects were minimized by aligning each resistor and FET so that the direction of current flow was parallel to the magnetic field. One would expect that the v × B forces would be minimized by aligning the channel region parallel with the B field. In a transistor with a simple structure the charge carriers move in the channel region in a direction between the source and the drain. This was verifed with the tuning FET used in this system by measuring the transconductance (gm) as a function of B from 0 to 2.5 Telsa at various orientations of the FET. Variations in gm of 10% were observed with the field perpendicular to the channel direction whereas this variation was only 0.5% with the B field parallel to the channel. After the devices were aligned properly with the magnetic field the RF carrier amplitude was much more stable although the magnetic field influence was not entirely eliminated.

5. Computer Control System

A model 5600 Rockland frequency synthesizer under the control of the TRIUMF 8085 microprocessor (TRIMAC) [4] provides the RF frequency sweep for the NMR tuned circuit. The frequency is swept at a rate of 83 times per second. This rate is limited by the cycle time of the TRIMAC. The NMR signal is recorded by an Interface Standards model ADS-120 analogue digitizer gated by the TRIMAC. The digitizer has a memory capable of storing 1024 scans of 256 steps and has a digitizing time of 2 µs. At the end of 12.5 s the memory is full and is read by a Starburst [5] J11 microprocessor. As soon as the Starburst has read the data from the memory of the digitizer it signals the TRIMAC to begin scanning again and proceeds to average the data. It takes 5 s to read and average 1024 NMR scans. When this pre-processing is completed a flag is set in a mailbox within the Starburst memory which is used for interprocessor communication. The host LSI-11 computer then reads the data from the Starburst memory via CAMAC. At this point the flag is reset by the LSI-11 to permit the Starburst to read the next 256K of data stored in the digitizer.

The LSI-11 subtracts background and applies a least square fit of a straight line or a quadratic to the wings of the difference spectrum. The wings are regions 80 KHz wide above and below a central region that is 352 KHz wide. The area under the NMR curve is determined by subtracting the fitted curved from the signal. The analysed NMR signal is usually displayed graphically in groups of 1024 scans. Various parameters relating to the NMR program can be changed easily from the keyboard. The overall rate of signal processing is about 60 scans per second.

An additional TRIMAC in conjunction with the LSI-11 is used to read, display and store on disc various refrigerator pressures, temperatures and flow rates. Alarm conditions are also annunciated.

6. Thermal Equilibrium Measurements

The area under the NMR curve for an enhanced polarization is normalized to a thermal equilibrium (TE) signal which is obtained in a magnetic field of 2.5 Tesla at a temperature of about 0.8°K. The TE polarization is determined by the Boltzman Law;

$$N_+/N_- = \exp(2\Delta E/kT) \qquad (2)$$

where k=Boltzman constant
T=Temperature
ΔE=Transition energy
N=Substate population

The TE polarization is extremely small (about 0.0005 at 1°K) and is very difficult to extract from the background noise. Background of 1024 scans is entered into the computer memory with the magnetic field adjusted so that the NMR signal is moved out of range. Measurements showed that a correction must be applied to the background which varies linearly with the magnetic field in the region of 2.5 telsa. The procedure for measuring the background during TE measurements is to measure the background with the magnetic field adjusted above the NMR signal and also to measure the background with the magnetic field adjusted below the range of the NMR signal. TE measurements are done by repeatedly measuring background above the signal, the signal area and the background below the signal. Typically 80 groups of 1024 scans are measured to check on systematic errors and statistical errors. This takes about two hours.

The day to day reproducibility of the TE signal area was measured during each of several test runs and the standard deviations observed varied from 4% to 6% over 3 or 4 days. If the uncertainty in the absolute temperature of about 5% is also included then a conservative estimate on the relative error associated with the TE calibration factor is 8%. Figure 4 shows a typical TE signal at a temperature of 1.2°K averaged over 1024 scans.

7. Polarization Measurements

The enhanced signal area is measured at an RF power level that is 20 db below the level used to determine the TE area. The relative response of the system at the two RF power levels was measured directly with the polarization at about .02 to .04. The variation in the response to the NMR signal as a function of frequency was measured by incrementing the magnetic field strength and measuring the area of the NMR signal. The observed variation is less than 2% over the region between the pedestals. The polarization was determined using the following relation.

$$P = \text{signal area} \times \text{TE polarization/TE area} \qquad (3)$$

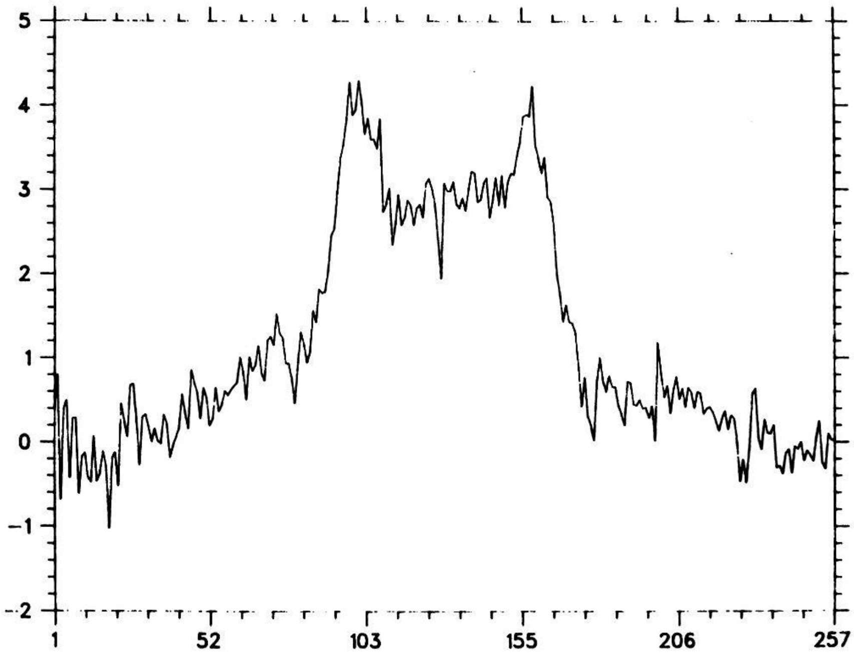


Fig. 4. A thermal equilibrium spectrum observed at 1.2°K averaged over 1024 RF sweeps.

The best polarizations achieved so far were −.37 and +.31 under target laboratory test conditions. Polarizations of −.32 to −.34 were routinely achieved during a three week run [6] in the TRIUMF M11 pion beam. The experiment requires a tensor polarized target with the magnetic field horizontal and parallel to the direction of the meson beam. Vector polarizations of .32 to .34 give rise to tensor polarizations of .078 to .089.

The polarization can also be determined from the asymmetry of the deuteron NMR curve using the relation

$$P = (R^2 - 1)/(R^2 + R + 1) \qquad (4)$$

where R is the ratio of the two transition intensities. The deuteron NMR signal shapes were analyzed using methods described in Ref. [7] by two different programs [8] to determine the ratio R. The results from the fitting programs agreed with each other and gave values that were consistent with the TE method within the 8% error. A further test of the frequency response of the RF was done by measuring the NMR signal at a fixed RF frequency and sweeping the magnetic field at a constant rate. The resulting spectrum was stored on disc and analyzed with a fitting program. This was compared to the results obtained by fitting the spectrum obtained by sweeping the RF field. The three methods (TE, fit of RF scan & fit of magnet scan) of determining polarizations gave values that agreed to within ±3%.

8. Summary

The TE signal is extracted from the noise with sufficient accuracy to allow a continuously updated display of the polarization during enhancement without the necessity of relying on off-line analysis of the line shape.

There is good agreement between the TE method and that in which the deuteron line shape is analyzed giving us confidence that the system is operating reliably.

9. References

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Rapporteur's report, Session (K), W. Wenckebach:

A C.W.-NMR Q-meter system based on a series circuit and a pre-amplifier at liquid helium temperatures was built and appears to operate satisfactorily. It appears that the choice of components for the pre-amplifier is still critical. Failures occur after three to four times cooling down to liquid helium temperatures.