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Polarization of the large COMPASS $^{14}\text{NH}_3$ target

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Abstract. The COMPASS experiment in the CERN M2 beam line is using 1508 cm$^3$ granular solid $^{14}\text{NH}_3$ as polarized proton target material to study the nuclear spin with deep inelastic scattering of polarized muons. The target consists of 2.5 T solenoid and 0.63 T dipole magnets, microwave cavity and large dilution cryostat. Continuous wave NMR is used to determine the polarization of the nuclei that are polarized with dynamic nuclear polarization method using 4 mm microwaves. Determination of the target proton polarization with thermal equilibrium NMR signals at temperatures 1.0 - 1.6 K is discussed.

1. Introduction
The COMPASS experiment [1] in the CERN M2 beam line is using 1508 cm$^3$ solid state $^{14}\text{NH}_3$ target in study of the origin of the nuclear spin in deep inelastic scattering with -80 % polarized muons. The beam momentum is 160 GeV/c and flux $2 \cdot 10^8 \mu/16.8$ s with 4.8 s long spills. The target material beads with typical packing factor of about 0.6 are inside 4 cm diameter cells in the mixing chamber of a large dilution cryostat. The target is at the upstream end of the COMPASS forward spectrometer with opening angle of 180 mrad, see Fig. 1. The 30, 60 and 30 cm long target cells are each in different microwave cavity volume, which allows building opposite spin directions for the upstream and downstream cells compared to the middle cell. The use of three cells allows to reduce possible false asymmetries [1] with the spin reversals using opposite current polarities in the solenoid magnet. The nuclei in the 2.5 T field are negatively (positively) polarized through paramagnetic centers using microwave frequencies that are slightly higher (lower) than the electron paramagnetic resonance (EPR) frequency 70105 MHz plus (minus) the proton nuclear magnetic resonance (NMR) frequency 106 MHz.

2. Target system
The target system consists of large diameter superconducting solenoid and dipole magnets with 16 trim coils to give 2.5 T longitudinal field with homogeneity about 80 ppm in the target material volume. A 0.63 T transverse dipole field is used when the solenoid field crosses zero during field rotations and in transverse physics data taking. The large dilution cryostat [2] can achieve high cooling power during the polarization build up and maintain temperatures between 100 - 300 mK when the electron spins absorb microwave powers of 40 - 400 mW [3]. The microwave power for the opposite polarizations is generated in two separate EIO microwave tubes (Varian VKE 2401). In frozen spin mode the dilution cryostat can keep the target material
at temperatures below 60 mK for long physics data taking periods. The large diameter of the microwave cavity is needed to reduce the amount of extra material inside the 180 mrad acceptance.

Typical density of paramagnetic centers in solid polarized target materials is about $10^{-4} - 10^{-3}$ free electrons/nucleus. In case of ammonia these are NH$_2$ radicals [4]. They have been created in 20 MeV electron beam at the Bonn linac at 80 - 90 K irradiation temperatures [2]. The hyperfine interaction of the free electron works only to the neighboring nuclei and rest of the nuclei get polarized through spin diffusion process [5, 6]. The target proton nuclear polarization is determined with continuous wave NMR. Ten single loop NMR coils 50 mm long and 13 mm wide made from 1.0 mm stainless steel tube were used. Inductance was estimated to be 60 - 80 nH. The coils were mounted on the outer surface of the target cell and connected with 0.9 mm diameter coaxial cable (Precision tube KA50034) inside the mixing chamber to the Q-meters [7].

![Figure 1. Side view of the polarized target in the COMPASS forward spectrometer: (1) upstream target cell, (2) middle target cell, (3) downstream target cell, (4) microwave cavity, (5) upstream microwave stopper, (6) downstream microwave stopper, (7) target holder, (8) still or $^3$He evaporator, (9) $^4$He evaporator, (10) $^4$He liquid/gas separator, (11) $^3$He pumping port, (12) solenoid magnet, (13) correction coils, (14) dipole coil, (15) solenoid end compensation coil and (16) magnet current leads.](image)

3. Nuclear spin system
The proton nuclear spin Hamiltonian is dominated by the Zeeman term of about 106 MHz in frequency units in the 2.5 T field. The dipole term between protons is about 25 kHz. The $^{14}$N nuclei with spin 1 have also quadrupole moment, which gives about 2.4 MHz wide NMR
signal around 7.7 MHz [2]. Low RF power is used in the cw NMR and thus its influence to the spin Hamiltonian can be neglected. The density of the paramagnetic centers is small making it difficult to observe the hyperfine interaction term in NMR. For the thermodynamics of the frozen proton spins only the Zeeman term is needed, while the $^{14}$N atoms give rise to quadrupole heat capacity too [8].

Unfortunately the crystal structure of the ammonia beads is unknown. One possibility is the cubic symmetry with $P2_13$ space group and with $a = 5.1305$ Å, $r_{\text{NH}} = 1.0099$ Å, $r_{\text{NN}} = 3.3769$ Å and H-N-H bond angle of 107.5 - 109.0° [9]. The calculated density 0.838 g/cm$^3$ is in agreement with the measured density of the crystals [2]. The second moment [10] of the proton NMR line was calculated in center of a spherical sample of a static crystal with radius of 4 unit cells. The calculated average $M_2$ for many crystal directions was about 890 kHz$^2$. Gaussian fit to the measured signals at 1.0 K gives more narrow line width of 450 - 520 kHz$^2$, see Fig. 2. Memory function [11] fit gives a little higher second moment of 520 - 680 kHz$^2$. Hydrogen bond scheme results in residual entropy similar that is known from the low temperature water ice. The hydrogen atoms can act as donors or acceptors between the NH$_3$ molecules. Tunneling between these two different positions could provide enough motion for the hydrogen atoms to explain the more narrow line width than calculated. In the low temperature water these excitations seem to be absent however [12].

The line width of protons in H$_2$O water molecules can be expected to be similar to those in NH$_3$. This can be understood since $M_2 \sim 1/r^6$ [10] and $(1.710 \text{ Å}/1.515 \text{ Å})^6 = 2.07$, so the the extra proton in NH$_3$ compensates the longer atomic distance of 1.710 Å. Thus it is difficult to distinguish water NMR signal from ammonia. The background proton signal of the empty target cell in Fig. 2 did not change much after heating up to 30 - 40 °C, so it is probably due to water absorbed into target cell construction materials or hydrogen in plastics. The second moments from a Gaussian fit were 600 - 1400 kHz$^2$ for the background protons.

4. Proton polarization

The proton relaxation times of about 1 - 3 hours were observed at the calibration temperatures 1.0 - 1.5 K in the 2.5 T field. For the empty target cell the relaxation times were slightly longer 1.5 - 4 hours. $^3$He vapor pressure was used as primary thermometer [13]. In addition two calibrated resistive RuO thermometers were mounted on the middle and downstream target cells. When the nuclear spin system is in thermal equilibrium with superfluid $^4$He the polarization can be calculated from the Brillouin function

$$P_J(x) = \frac{2J + 1}{2J} \coth\left(\frac{2J + 1}{2J} x\right) - \frac{1}{2J} \coth\left(\frac{x}{2J}\right),$$

where the spin number $J = 1/2$ for protons and $J = 1$ for $^{14}$N. Here $x = Jhf/k_BT_s$ with $h$ Planck constant, $f$ NMR resonance frequency in 2.5 T field, $k_B$ the Boltzmann constant and $T_s$ the spin temperature. The polarization of the protons at 1.0 K is then about 0.25 %.

Borghini’s equations [6, 14] predict the lowest achievable spin temperature $\beta = 1/k_BT_s$ in the dynamic nuclear polarization process

$$\beta D \simeq \frac{1}{2} \beta L\omega_S,$$

where $\beta_L = 1/k_BT_L$ is the inverse lattice temperature, $\omega_S$ is the EPR frequency and $D$ the EPR line width. For $^{14}$NH$_3$ EPR line width of 120 MHz in 10 GHz X-band spectrometer with 350 mT field has been observed [4]. Lower lattice temperature $T_L$ helps but is not always easy to achieve due to the heat coming from pumping of the electron spins with microwaves. Thus the practical way to cool the solid materials efficiently has been to use small 2 - 5 mm diameter material beads inside mixture of liquid $^3$He/$^4$He.
Figure 2. Left: NMR signal from thermal equilibrium calibration at 1.3 K. The small signal is the background from empty target holder at the same temperature. Both protons from NH$_3$ and background protons from two consecutive empty holder measurements fit well in a Gaussian. Right: NMR signal line shape for approximate proton polarizations -20 %, -40 %, -60 %, -80 % and -90 %.

The polarization is started by first increasing the flow of $^3$He in the dilution cryostat with higher heating power to the $^3$He evaporator. Typically $^3$He flow of 100 - 120 mmol/s is used. The Speer resistors fixed on each of the three target cells are used as bolometers to detect the microwave power. The polarization of irradiated ammonia is different from $^6$LiD [15] in that the microwave frequency does not need to be tuned during polarization build up although the power is gradually decreased to reach lower lattice temperature. The frequency modulation that clearly helps in building polarization in $^6$LiD has only small about 2 % effect to the final polarization value. 90 % nuclear polarizations were reached in 36 hours with continuous microwave pumping.

The NMR line shape during polarization build up is shown in Fig. 2. The NMR line becomes asymmetric with shift of the peak +22 – +27 kHz (-22 – -27 kHz) for positive (negative) polarization. This value is close to the proton-proton dipole term in the spin Hamiltonian. In the frozen spin mode the 1.0 T longitudinal and 0.63 T dipole fields were used. The loss of proton polarization was 0.2 - 0.9 %/day in frozen mode. This corresponds to an effective heat flow of 100 - 300 pW/mol to the proton spin system at helium temperatures 40 - 70 mK during physics data taking. Maximum average cell polarizations of about ±90 % were reached corresponding to spin temperatures around ±1.7 mK.

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