

# Spin polarization of xenon films at low temperature induced by $^3\text{He}$

N. Biškup<sup>a,b</sup>, N. Kalechofsky<sup>b</sup>, D. Candela<sup>a,1</sup>

<sup>a</sup> *Physics Department, University of Massachusetts, Amherst, MA 01003, USA*

<sup>b</sup> *Oxford Instruments America, Concord, MA 01742, USA*

---

## Abstract

We have measured the  $^{129}\text{Xe}$  spin-lattice relaxation time  $T_1$  for xenon films adsorbed on silica gel in an 8 T magnetic field at dilution refrigerator temperatures, both with and without  $^3\text{He}$  filling the sample cell. Without  $^3\text{He}$ ,  $T_1$  increases rapidly as the temperature is lowered. With  $^3\text{He}$ ,  $T_1$  has a temperature-independent value of about 1000 s. Using this technique, it is possible to brute-force polarize large quantities of xenon in high  $B/T$  conditions.

*Key words:* helium-3; NMR; films and interfaces; hyperpolarized

---

## 1. Introduction

Recently, important applications have arisen for matter prepared with hyperpolarized nuclear spins such as  $^3\text{He}$  and  $^{129}\text{Xe}$  [1]. Usually hyperpolarization has been produced by optical pumping at room temperature, but brute force polarization at high magnetic fields and dilution-refrigerator temperatures is another possibility. For example, the equilibrium polarization of  $^{129}\text{Xe}$  in a 16 T field at  $T = 15$  mK is 29%.

A major obstacle to brute-force polarization of  $^{129}\text{Xe}$  is the long spin-lattice relaxation time  $T_1$ , which prevents bulk solid xenon from reaching equilibrium polarization in a reasonable period of time. It has been known since the 1980's that the surface nuclear spins in substances immersed in liquid  $^3\text{He}$  are rapidly relaxed by the  $^3\text{He}$  [2]. The mechanism is quantum tunneling of  $^3\text{He}$  atoms in the localized (solid-like) layer that forms near solid surfaces, a process that persists to arbitrarily low temperatures [3,4].

The difficulty with applying this method to hyperpolarizing  $^{129}\text{Xe}$  is achieving sufficiently large surface area to polarize large quantities of xenon. In this paper we describe preliminary experiments that use a new method to overcome this difficulty: the xenon is plated

onto a substrate with very high specific surface area (silica gel is used here), which is then immersed in liquid  $^3\text{He}$  in high  $B/T$  conditions.

The relaxation time for *surface*  $^{129}\text{Xe}$  spins  $T_{1s}$  depends upon the distance between the  $^{129}\text{Xe}$  and  $^3\text{He}$  nuclei, as well as the spectral density of tunneling in the localized  $^3\text{He}$  layer at the  $^{129}\text{Xe}$  Larmor frequency [3,4]. Unfortunately neither quantity is accurately known for  $^3\text{He}$ - $^{129}\text{Xe}$  interfaces, which to our knowledge have not been explored before this work. Nevertheless, by extrapolating the measured  $T_{1s}$  for other substances in contact with  $^3\text{He}$  [4] we make the following rough estimate:  $T_{1s} \approx (4100 \text{ s})(B/16 \text{ T})$  where  $B$  is the applied field. Furthermore, we estimate that for  $^{129}\text{Xe}$  films thinner than 100 atomic layers spin diffusion between surface and interior  $^{129}\text{Xe}$  spins does not present a significant bottleneck for the overall  $T_1$  value.

## 2. Experimental methods and results

We have constructed a cell containing powdered silica gel substrate [5] along with a sintered-silver heat exchanger, which was cooled by a dilution refrigerator in an 8 T NMR magnet. The silica gel was contained in an epoxy lower portion of the cell, which extended into a small birdcage NMR resonator [6] tuned to 92 MHz,

---

<sup>1</sup> Corresponding author. E-mail: candela@physics.umass.edu

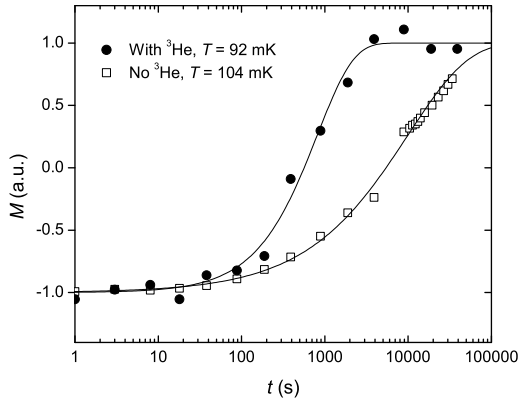


Fig. 1. Recovery of  $^{129}\text{Xe}$  nuclear magnetization following an inverting pulse or saturating comb. The magnetization data have been rescaled to an arbitrary range of  $(-1, 1)$ . The curves show fits to the data as follows. With  $^3\text{He}$ : simple exponential recovery with  $T_1 = 814$  s. No  $^3\text{He}$ : stretched exponential recovery with  $T_1 = 1.0 \times 10^4$  s and stretching exponent  $\alpha = 0.6$ .

the  $^{129}\text{Xe}$  Larmor frequency at 8 T. The magnetic field could also be lowered, to measure the  $^3\text{He}$  NMR signal at the same frequency. A vibrating-wire viscometer at the top of the cell permitted the  $^3\text{He}$  liquid level to be monitored. A heated fill line was used to admit xenon to the cell. The cell was maintained at approximately 90 K during the xenon condensation process. The volume of xenon condensed was typically 20% of the available pore space, corresponding to approximately three atomic layers on the substrate.

The spin-lattice relaxation of  $^{129}\text{Xe}$  was measured by small-angle tipping pulses after either a magnetization-inverting  $\pi$  pulse or a magnetization-destroying comb of large-angle pulses. The magnetization was sampled over a very large range of times (0.1 - 40,000 s) to ensure that very rapidly or slowly relaxing spin populations were not missed.

As shown in Fig. 1, addition of  $^3\text{He}$  to the cell significantly shortens  $T_1$ , and also changes the recovery curve from a stretched exponential ( $M(t) - M(\infty) \propto \exp[-(t/T_1)^\alpha]$ ) to a simple exponential. The stretched-exponential form is typically due to a wide distribution of  $T_1$  values for individual  $^{129}\text{Xe}$  spins.

Figure 2 shows the temperature dependence of  $T_1$  measured with and without  $^3\text{He}$  in the cell. In the absence of  $^3\text{He}$ ,  $T_1$  is strongly temperature dependent. The mechanism of relaxation without  $^3\text{He}$  is not known, although it presumably reflects interactions with the silica surface and/or adsorbed impurities such as  $\text{H}_2\text{O}$  and  $\text{O}_2$ . This relaxation rate is well fit by a power law (Fig. 2). When  $^3\text{He}$  is added to the cell, the relaxation time reduces to a temperature-independent value  $T_1 \approx 1000$  s. This compares favorably to the estimate above.

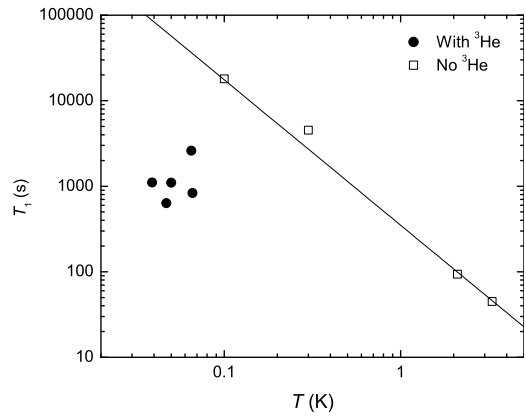


Fig. 2. Spin lattice relaxation time  $T_1$  measured for  $^{129}\text{Xe}$  as a function of temperature and  $^3\text{He}$  coverage. With  $^3\text{He}$  present in the cell,  $T_1$  is temperature independent with a value of approximately 1000 s. With no  $^3\text{He}$  present,  $T_1$  is longer and strongly temperature-dependent. The line shows a power-law fit,  $T_1 \propto T^{-1.7}$ .

### 3. Conclusions

We have demonstrated that macroscopic quantities of solid xenon can be brute-force polarized at dilution refrigerator temperatures, using the  $^3\text{He}$ -porous substrate method. A number of obstacles remain to developing this method into a practical method for producing hyperpolarized  $^{129}\text{Xe}$  gas. In particular, it may be necessary to switch off the relaxation process before attempting to remove the xenon sample to low  $B/T$  conditions. Addition of  $^4\text{He}$  to the cell could provide such a switch, as  $^4\text{He}$  preferentially occupies sites adjacent to solid surfaces. Significantly, spreading of  $^4\text{He}$  over the xenon surface will occur by superfluid film flow. Like the tunneling process that is used to induce relaxation, this is a quantum process that can proceed at arbitrarily low temperatures.

### References

- [1] T. Pietrass, *Magn. Reson. Rev.* **17** (2000) 263.
- [2] P. C. Hammel, et al., *Phys. Rev. Lett.* **51** (1983), 2124.
- [3] S. Maegawa, A. Schuhl, M. W. Meisel, M. Chapellier, *Europhys. Lett.* **1** (1986), 83.
- [4] O. Gonen, P. L. Kuhns, C. Zuo, J. S. Waugh, *J. Magn. Reson.* **81** (1989), 491.
- [5] Alfa Aesar #42725, nominal surface area 500  $\text{m}^2/\text{g}$ .
- [6] H. Akimoto, D. Candela, *J. Low Temp. Phys.* **121** (2000), 791.