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Imaging of laser-polarized solid xenon

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Abstract

The enhanced spin polarization produced by optical pumping of gaseous rubidium/xenon samples has made possible a number of recent experiments in nuclear magnetic resonance (NMR) spectroscopy and magnetic resonance imaging (MRI). Here we report MRI of laser-polarized xenon in the solid phase at low temperature. Due to the high xenon density in the solid phase and the enhanced spin polarization, it is possible to achieve high intensity and spatial resolution of the image. Signals were observed from xenon films solidified onto the glass container walls and not from an enclosed chili pepper.

Keywords: Laser-polarized xenon; Magnetic resonance imaging; Nuclear magnetic resonance spectroscopy

A well-known problem that leads to poor sensitivity in nuclear magnetic resonance (NMR) and magnetic resonance imaging (MRI) is the low thermal equilibrium spin polarization, typically less than 1×10^{-5} at room temperature and a magnetic field of a few Tesla. One approach to ameliorating the limitations of low sensitivity is the use of optically polarized gases [1–4], xenon or helium, which can reach spin polarizations 4–5 orders of magnitude greater than those in thermal equilibrium. This method has been used to obtain MRI images of the gas space in lungs [5–9], in materials [10], and most recently, in animal and human tissue [11,12].

Due to the low density of the gas phase, typically three orders of magnitude lower than that of liquids or solids, the total magnetization density of the laser-polarized xenon gas is similar to the thermal equilibrium proton magnetization density in water. In fact, the faster diffusion of xenon in the gas phase (the diffusion constant is 5 orders of magnitude higher than that in a liquid), limits the resolution to about 0.1 mm [10]. In the solid phase, the xenon density is similar to that of water, so the magnetization density of the polarized xenon solid can be 3–4 orders of magnitude higher than for protons in water. In this communication, we report the first MRI images of solid laser-polarized ^{129}Xe obtained at 77 K, images that benefit in intensity and resolution from the enhanced spin polarization.

Polarized xenon was obtained by laser optical pumping (OP) of rubidium followed by spin exchange between the rubidium and ^{129}Xe [1–4]. The details of our optical pumping apparatus have been published previously and we repeat [13] a brief description herein. The central part of the optical

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pumping apparatus is the pumping cell, a 35-cm^3 glass cylinder connected to the NMR sample cell and the vacuum pumps via glass tubing and detachable joints. Several stopcocks facilitate convenient gas handling. All parts of the glassware are carefully treated with a siliconizing agent (SurfaSil, Pierce Scientific). In the OP stage of the experiment, a magnetic field of about 30 G at the pumping cell is provided by a pair of Helmholtz coils.

The pumping cell is illuminated at the D1 transition of Rb (794.8 nm) along the direction of the magnetic field for typically 30 min using a Ti–Sapphire laser (Schwartz Electro Optics), which is pumped by a 10-W Ar^+ laser (Coherent). The helicity of the light is adjusted by a quarter-wave plate to be either of left or of right circular polarization. The pumping cell is maintained at 60–80°C using a heated nitrogen gas stream. The elevated temperature determines the density of Rb atoms in the vapor phase, allowing adjustment for optimal absorption of the laser light. Before NMR experiments, the xenon is separated from the Rb by allowing the pumping cell to cool to ambient temperature, thus condensing the Rb. Xenon gas is then allowed to expand into the sample region. Enriched ^{129}Xe gas (80% ^{129}Xe, EG &G Mound) was used in all experiments described below.

The imaging experiments were performed on a commercial micro-imaging spectrometer (Quest 4300, Nalorac Cryogenics). At the operating magnetic field of 4.3 T, the ^{129}Xe NMR frequency is 51.4 MHz. A FLASH (Fast Low Angle SHot) imaging sequence [14], shown in Fig. 1, was used for the two-dimensional imaging experiments. The tipping angle used was 3°. Sixty four phase-encoding steps were used to produce a 64×128 pixel time domain data set and one scan was acquired for each phase-encoding step. The data sets were zero-filled to 256×256 during data processing.

The xenon film was formed by freezing the hyperpolarized ^{129}Xe onto the inside of the cold sample tube. First, the sample tube was attached to the OP apparatus and evacuated. The end section of the sample tube was immersed in liquid nitrogen for a few minutes. After optical polarization, the xenon was allowed to enter the sample tube and to solidify on the inside of the cold tube. The freezing process was performed in the fringe field (about 500 G) of a small permanent magnet in order to maintain the ^{129}Xe polarization in the solid phase. The sample cell was then closed, disconnected from the pumping apparatus, and moved to the imaging spectrometer for experiments. The sample region of the NMR probe was precooled to liquid nitrogen temperature, and the ^{129}Xe MRI was performed at the same temperature.

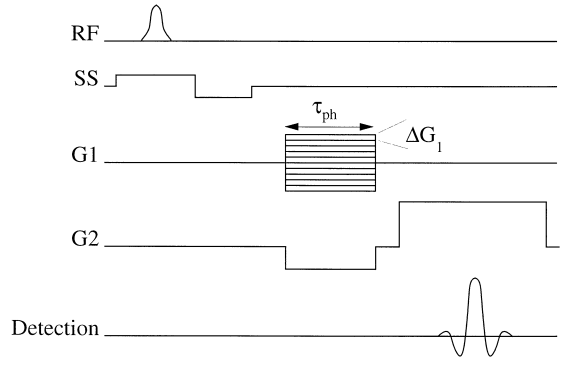

Fig. 1. A FLASH (Fast Low Angle SHot) imaging sequence was used to obtain two-dimensional image of ^{129}Xe solid and gas. The r.f. pulses were Gaussian-shaped and 500 μs long, and the tipping angle was 3°. The frequency-encoding gradient γG_2 (γ is the gyromagnetic ratio of ^{129}Xe) was 6.5 kHz/mm, and the phase-encoding gradient were 500 μs in duration, and the gradient step $\gamma \Delta G_1$ was 0.195 kHz/mm.

In Fig. 2, we show the intense cross-section image of a solid xenon film on the inside of the sample tube (5.8 mm in diameter) containing a red chili pepper. The slice-selection length along the sample tube is about 2 mm. The thickness of the xenon film determined from the image, approximately 0.3 mm, varies around the circle; in part, the variation may arise from a tilting of the sample. The thickness of ~ 0.3 mm compares well with an estimate considering the amount of xenon used in the experiment. The signal-to-noise ratio is about 300, with an intensity variation at different parts of the circle due in part to the inhomogeneous r.f. field within the coil. No xenon signal was observed from xenon on the chili pepper surface.

The NMR linewidth of the solid ^{129}Xe spectrum is ~ 300 Hz at 77 K. Since one of the dimensions of the data is acquired in the time domain during the frequency encoding sequence, the decay of the magnetization limits the resolution obtained in that dimension. This limit is estimated to be 0.05 mm,

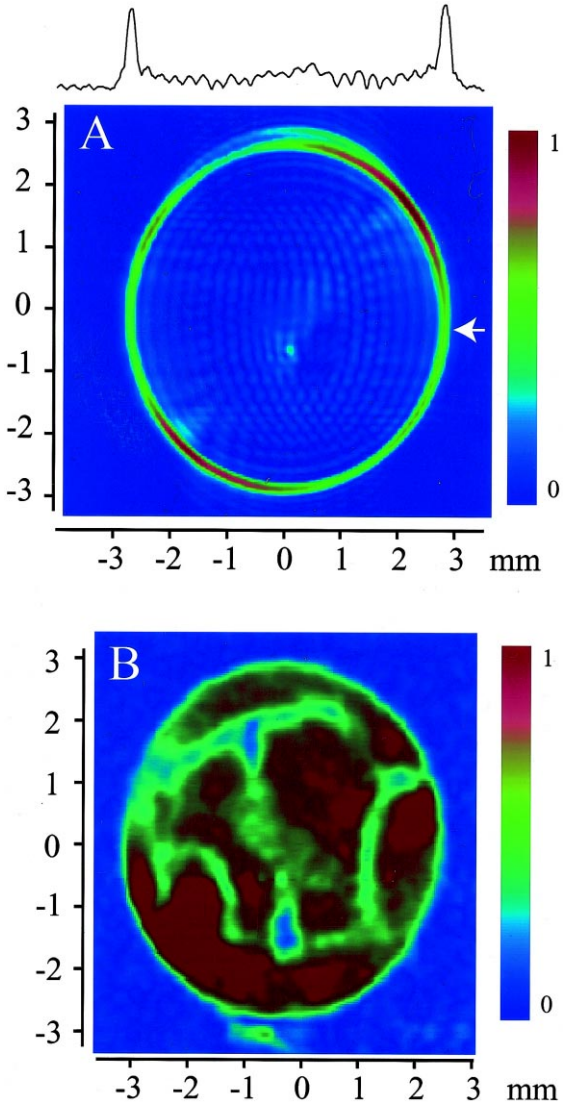

Fig. 2. (A) Two-dimensional cross-section image of solid xenon forming a film on the inside of a glass tube of 5.8 mm inside diameter, at a temperature of 77 K. The ripples in the middle of the image, at less ~20 signal intensity, arises from truncation in the phase-encoding dimension. The full linewidth at half maximum of the solid ^{129}Xe signal is about 300 Hz and may have caused broadening in the image along the frequency-encoding dimension. The inset above the image shows the signal intensity across the middle of the sample indicated by the arrow in the main figure. The signal-to-noise ratio is ~300. (B) Two-dimensional image of gas ^{129}Xe obtained after the solid image was taken and the sample used in (A) was warmed up. The pattern in the image shows a cross-section of a dried (somewhat crumpled) red chili pepper contained in the glass tube. The image from experiment (A) indicates that xenon solidifies or maintains polarization preferentially on the glass tube and not on the pepper.

similar to the digital resolution of the image. This limitation could be overcome by using higher magnetic field gradients; for example, with a gradient γG of 260 kHz/mm (where γ is the gyromagnetic ratio of ^{129}Xe and G is the magnetic field gradient), we could approach resolution of order micrometers. Line narrowing pulse sequences [15,16] and sample reorientation could also be used to improve resolution without the need for higher gradients. Additionally, a two-dimensional image of gaseous ^{129}Xe was obtained after the sample was warmed up (Fig. 2B). The pattern in the image indicates the cross-section of a dried (somewhat crumpled) red chili pepper contained in the glass tube. By comparing this image with that in Fig. 2A, one can see that the xenon solidifies or maintains polarization preferentially on the glass tube and not on the pepper.

In conclusion, we have shown an image of solid polarized xenon at low temperature. The high density of xenon in the solid phase and the high spin polarization makes it possible to achieve spatial resolution better than 0.05 mm without signal averaging.

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