LETTER

Long-distance delivery of laser-polarized $^{129}$Xe using a capillary tube

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Abstract

A long-distance delivery system for laser-polarized $^{129}$Xe that uses a capillary tube is described. To our knowledge, this is a first nuclear magnetic resonance (NMR) study of laser-polarized $^{129}$Xe fluid behavior that focuses on the effects of the length and inner diameter of the delivery tube of the system. It is found that the maximum signal intensity of laser-polarized $^{129}$Xe in a laminar flow is hardly affected by depolarization, even when a tube with a length of 10 m is used under continuous flow conditions. The sensitivity of Xe to its local magnetic environment was successfully applied to demonstrate the usefulness of fused silica capillary tubes with inner diameters $\leq 0.53$ mm. Extensions of this technique should enable the more efficient delivery of $^{129}$Xe gas with the sustainable polarization of laser-polarized $^{129}$Xe.

Since optical pumping was first used to enhance the signals of noble gases, nuclear magnetic resonance (NMR) using laser-polarized $^{129}$Xe has been well known as a method with extremely high sensitivity [1–15]. A batch-type production technique has already been established for laser-polarized $^{129}$Xe gas, which enables continuous polarized gas generation [16, 17]. However, the nonrenewable character of the nonequilibrium polarization is a serious problem that limits the laser-polarized $^{129}$Xe study of materials to a narrow set of carefully selected systems. The spin-lattice relaxation ($T_1$) of gaseous $^{129}$Xe is mainly governed by the spin–rotation mechanism in short-lifetime Xe–Xe van der Waals molecules [18]. However, other relaxation mechanisms move the polarization toward thermal equilibrium during gas delivery. The most important relaxation mechanism results from the interaction of $^{129}$Xe gas nuclei with the inner surface of the gas chamber (wall relaxation). This relaxation has been interpreted as the result of the dipolar interaction between $^{129}$Xe gas nuclei and the neighboring surface nuclear or electronic spins. Many materials of interest possess relaxation sources such as paramagnetic centers with a strong depolarizing effect [19–22]. Once laser-polarized $^{129}$Xe is brought into contact with a material, all the polarization may be lost in a very short time. Therefore, a continuous flow of $^{129}$Xe is required to extend the use of laser–polarized $^{129}$Xe NMR to new fields of measurement.

In general, surface relaxation in the delivery path has prevented the continuous flow of laser-polarized $^{129}$Xe gas with little or no loss of polarization [17, 23–25]. As a result, the production rate is substantially lowered until most of the laser-polarized $^{129}$Xe have been transported to a detection cell, even though Xe is chemically inert to functional groups of materials. Irrespective of whether a batch or continuous flow is employed, the development of a technique that delivers laser-polarized Xe gas efficiently to an NMR apparatus still remains an urgent problem. Particular interest in the fluid properties of a laser-polarized $^{129}$Xe flow has also been stimulated by the attempt to establish real-time, high-resolution NMR/MRI. In this context, a number of researchers have exploited the high rate of $^{129}$Xe polarization for solution, solid, and imaging studies.

Saito et al have investigated the effect of pipeline surfaces passivated with different metallic materials on the laser-polarized $^{129}$Xe signal intensity $M_0$, and found that a film passivated with iron fluoride maintained the highest level of hyperpolarization, whereas a film passivated with chromium oxide maintained the lowest level [26]. They suggested that the wall relaxation strongly depends on the chemical structure of the pipeline surface. Some researchers have used perfluoroalkoxyalkane (PFA) plastic tubes (i.d. $\sim 1.6$ mm) as the delivery pipeline for laser-polarized $^{129}$Xe because PFA was considered to ensure a tight connection to the probe to minimize the
loss of polarization via Xe-wall relaxation [23, 27–29]. Song et al reported a two-dimensional image of $^{129}$Xe in a Teflon tube with an i.d. of ca. 0.3 mm in the form of an NMR image [30]. Moulé et al discussed $^{129}$Xe signal amplitude versus travel time plots as a function of the flow time [31]. They found that laser-polarized $^{129}$Xe gas traveled through a 1/8-inch (ca. 3.2 mm) Teflon tube between two magnets separated by a distance of ca. 5 m in 7–10 s. When it comes to polarization loss in the case of polarized particles traveling from one region to another, one also needs to take a look to see whether the condition for adiabatic fast passage is met. If it is met, then spins is expected to follow the magnetic field, and result in no loss or minimum loss of the polarization. Seydoux et al suggested the field sweep through the adiabatic condition was provided by the gas transport itself [24].

Even with the use of a Pyrex glass pipe having an inside diameter of 4 mm, the distance for which the laser-polarized noble gas can be supplied without allowing spin relaxation to arise is at best somewhat under 1 m. Hence, it was not possible to supply the laser-polarized noble gas over a long distance. In particular, in the case in which a superconducting magnet having a large magnetic field, for example, is used in the NMR spectrometer or the magnetic resonance imager, there is a large leakage magnetic field from the superconducting magnet. This leakage magnetic field apparently has an adverse effect on the laser-polarized noble gas generating cell located upstream, which lowers the amount of laser-polarized noble gas generated. Accordingly, it is necessary to have the distance between the laser-polarized noble gas generating cell and the NMR spectrometer or the magnetic resonance imager be at least 1 m, and preferably 2 to 3 m or more. Moreover, a glass pipe having an inside diameter of about 4 to 7 mm is more fragile to impacts than a stainless steel pipe, and thus more subject to failure. This, combined with the fact that it cannot be bent, makes it inconvenient to handle for a long-distance supply of the laser-polarized noble gas. Capillary delivery of the laser-polarized noble gas avoids a decline in the NMR signal intensity even when the gas is supplied over a long distance.

Although the sensitivity of laser-polarized $^{129}$Xe to surface relaxation makes this technique an important diagnostic probe of the nature of a fluid boundary layer as well as the chemical state of a surface, the effects of the length and inner diameter of the delivery tube on the signal loss of laser-polarized $^{129}$Xe are not clear. Details of the physical mechanisms of surface relaxation under a continuous flow are poorly understood at present. In this study, using capillary tubes with i.d. $\leq$ 0.53 mm, the signal intensity of laser-polarized $^{129}$Xe was quantitatively measured as a function of the Xe flow rate $Q$. We found that the laser-polarized $^{129}$Xe gas was successfully delivered to the detection cell via tubes with a length of more than 10 m. This suggests that the signal loss can be markedly reduced when delivering Xe gas in a laminar flow. The first ever observation of the long-distance delivery of laser-polarized $^{129}$Xe under a continuous flow is described in this paper.

The gas flow system mainly consists of a laboratory-built apparatus similar to that reported in a previous publication [25, 26], which provides a continuously flowing gas stream carrying laser-polarized $^{129}$Xe gas with up to ~20% polarization at a flow rate of 50 cm$^3$ min$^{-1}$ [32]. The laboratory-built probe was connected to the apparatus using a fused silica capillary tube (GL Sciences, Inc.), and then was inserted into a permanent magnet (Sumitomo Special Metals Co., Ltd) with a field $B_0$ of 0.3 ± 0.01 T. Two silica tubes with i.d. of 0.25 and 0.53 mm, denoted as N025 and N053, respectively, were used as delivery paths without any pretreatment of their interior surfaces with a chemical reagent. The tube length $L$ was varied from 1 to 22 m.

Naturally abundant Xe gas (99.9995%, Japan Fine Products Co.) was used. Rb metal (99.99%, Furuuchi Chemical Co.) was deposited on one side of a quartz cell, which was set inside a Helmholtz coil (1.25 $\times$ 10$^{-2}$ T). Low-field regions existed between the Helmholtz and the permanent magnet. The Xe gas and Rb vapor were mixed at 145 ± 3°C and optically pumped at the wavelength of the Rb D$^1$ transition (794.7 nm) with circularly polarized light from an air-cooled laser diode array (B1-79-40C-19-30-A, Coherent Inc.). A total light power of 40 W at wavelengths of 794.7 ± 1 nm was delivered using two combined lasers. The absorbed photon intensity depends on the Rb vapor density, which was determined by the cell temperature and its ‘conditioning’ [29].

The signals of laser-polarized $^{129}$Xe were detected using a laboratory-built NMR spectrometer (Thamway Co., Ltd) [25, 26] at room temperature, after the gas flow in the laboratory-built NMR cell region had become stable. The resonant frequency of $^{129}$Xe in a solenoid coil (2.0 cm i.d. and 3.3 cm length) was 3.58 MHz at a 0.3 T permanent magnet, which also provided a fringe field of ca. 2 $\times$ 10$^{-4}$ T.

The data were acquired in the direction of increasing flow rate $Q$, where $Q$ can be expressed as

$$ Q = \left( \pi / (8\eta L) \right) a^2 (p_1 + p_2)(p_1 - p_2) / 2 $$

(1)

Here, $\eta$ is the viscosity coefficient, $L$ the length of the delivery tube, $a$ the inside radius of the tube, and $p_1$ and $p_2$ the pressure at both ends of the tube (Hagen-Poiseuille’s law). The higher $p_1$ in the optical cell was monitored by a pressure gauge, and the lower $p_2$ in the NMR cell is assumed to be 760 Torr. Xe gas was allowed to flow for 5 min to ensure that it was homogeneously distributed in the NMR cell. As a result, ($p_1 + p_2$($p_1 - p_2$) reached at equilibrium.

Figure 1 shows a schematic of the laboratory-built low-field NMR system in which a capillary tube is connected to the originally designed optical cell [32].
The laser polarization conditions at the cell temperature of 145 ± 3 °C were optimized to obtain ideal conditions for optical pumping. Typical NMR signals of laser-polarized $^{129}$Xe in the NMR cell are shown in figure 2, where the mass flow controller was set at 10 cm$^3$ min$^{-1}$.

Then the flow rates of $Q$ were calculated to be $6.0 \times 10^{-3}$ and $3.7 \times 10^{-3}$ Pa m$^3$ s$^{-1}$, where the maximum signal intensity $M_{\text{max}}$ appeared for the N053 tubes with (a) $L = 1$ and (b) $L = 10$ m, respectively. The observed $M_{\text{max}}$ in this study was normalized by that for the N053 tube with $L = 1$ m, which enabled us to analyze the data observed under different conditions. In the case of the tube with $L = 10$ m, $M_{\text{max}}$ was ca. 80% of that for the tube with $L = 1$ m. Interestingly, however, the signal of the laser-polarized $^{129}$Xe could be detected despite passing through the low-field regions between the Helmholtz coil and the permanent magnet. The reason that we were able to keep the Xe polarized over long distances is that because of laminar flow, only the Xe atoms at the tube wall are relaxed by it, and they don’t intermix much with the bulk Xe flowing in the tube. Moreover, the Xe doesn’t get the chance to move back and forth through the field gradients, given the laminar flow.

The dependence of $M_0$ on $Q$ for laser-polarized $^{129}$Xe for the 1 m tube was observed using the N053 tube. $M_0$ increased with increasing $Q$ from $3.7 \times 10^{-3}$ to $6.0 \times 10^{-3}$ Pa m$^3$ s$^{-1}$ and decreased above $8.3 \times 10^{-3}$ Pa m$^3$ s$^{-1}$. The initial increase in $M_0$ suggests that the number of Xe atoms that can undergo spin exchange initially
increases in the optical cell. In contrast, the decrease in $M_0$ above $8.3 \times 10^{-3}$ Pa m$^3$ s$^{-1}$ may indicate that the polarization begins to level off owing to the high Xe density, where the Xe itself is the dominant cause of Rb depolarization [33].

Brunner et al found that in their Fourier image of laser-polarized $^{129}$Xe flowing through a sample tube containing nine glass capillaries, the capillary walls appeared as dark circles. They considered that this feature was due to the broad velocity distribution in the central region and was unrelated to the difference in the level of spin-lattice relaxation [34]. However, in the vicinity of the tube surface, the laser-polarized $^{129}$Xe gas flows slowly, as expected from the calculated velocity profiles (not shown) and may be relaxed by coming in contact with the paramagnetic centers in the glass wall on the tube surface [22]. Accordingly, the net amount of laser-polarized $^{129}$Xe detected by NMR should correspond to the components that were not relaxed in the silica tube under a continuous flow. This means that the $Q$ dependence of $M_0$ contains essential information about the fluid dynamics in a capillary tube.

The fluid behavior for a gas in a tube may be explained by the Reynolds number $Re$ in a cylindrical channel, which is expressed as

$$Re = \frac{D \nu \rho}{\eta},$$

where $D$ (=2$a$) is the inside diameter of the tube, $\nu$ the linear fluid velocity, and $\rho$ the fluid density. $Re$ is a nondimensional parameter that ensures hydrodynamic similarity between different flows. Any two flows with the same $Re$ will produce the same fluid mechanical behavior. Generally, $Re < 1200$ is considered to represent a laminar flow and $2200 < Re$ is considered to represent a turbulent flow. The specific relaxation behavior of laser-polarized $^{129}$Xe in the tube under a continuous flow can mainly be attributed to the interaction between the $^{129}$Xe gas and the tube surface. Therefore, $^{129}$Xe in a laminar flow is expected to be less relaxed than that in a turbulent flow characterized by unsteady and irregular fluid motion.

Figure 3 shows the dependence of the magnetization $M_0$ on the Reynolds number $Re$ for the laser-polarized $^{129}$Xe gas flowing through the N053 tube. The tube length $L$ was varied from 1 to 10 m. Here, equation (2) can also be expressed as

$$Re = \frac{4M}{(\pi \eta RT)} \left(\frac{Q}{D}\right),$$

where $M$ is the molecular weight, $R$ the gas constant, and $T$ the absolute temperature. The calculation of $Re$ using equation (3) assumes that the laser-polarized gas flows through the silica tube at 25 °C.

The maxima of the $M_0$ versus $Re$ curves in figure 3 were distributed over a wide $Re$ range between 33 and 203. This observation is not in agreement with the prediction from a hydrodynamic viewpoint, because the values of $Re$ where the maximum intensities appeared were much lower than the transition point ($Re \approx 2300$) from a laminar to turbulent flow. The similarity of the fluid behavior observed for different tubes means that the $^{129}$Xe gas flows through the tubes in a laminar flow. Although the reason for the single maximum appearing at

![Figure 3. Reynolds number $Re$ dependence of the signal intensity $M_0$ for laser-polarized $^{129}$Xe. The tube length $L$ was varied between 1 and 10 m. The curves are shown as eye-guidelines for the experimental data.](image-url)
Re < 2300 for the Xe fluid is unknown, this phenomenon may be responsible for the onset of Rb spin destruction. As mentioned above, the polarization may begin to level off at a high Xe density where the dominant source of Rb depolarization is the Xe itself [33].

Laser-polarized $^{129}$Xe, which is sensitive to the inhomogeneity in a flow field, can be used to monitor the deactivation at a boundary layer, i.e., in the vicinity of a tube wall. Figure 4 shows the dependence of the maximum signal intensity $M_{\text{max}}$ and flow rate $Q_{\text{max}}$ on the length $L$ of N053 and N025 tubes. Here, the $Q_{\text{max}}$ means the flow rate where the $M_{\text{max}}$ for N053 tube appeared.

The values of $M_{\text{max}}$ and $Q_{\text{max}}$ are normalized by those for the N053 tube with $L = 1$ m. To our knowledge, this is a first NMR study of laser-polarized $^{129}$Xe fluid behavior that focuses on the effects of the length and inside diameter of the delivery tube. The $M_{\text{max}}$ decreased almost linearly with increasing $L$, which is proportional to surface area $S$ for the tube shorter than 10 m. As mentioned above, this observation may suggest that because of laminar flow, only the Xe atoms at the tube wall are relaxed by it, and they don’t intermix much with the bulk Xe flowing in the tube. However, the other mechanism like the spin-rotation relaxation might contribute to non-linear decrease of $M_{\text{max}}$ for the tubes longer than 10 m, because of lower delivery of laser-polarized $^{129}$Xe [22]. Moreover, the amount of $M_{\text{max}}/Q_{\text{max}}$ is proportional to the density (mol/cm$^3$) of laser-polarized $^{129}$Xe gas in the detection cell. Therefore, figure 4 also suggests that the depolarization is apparently suppressed with increasing $L$ for the tubes longer than 3 m due to lower $Q_{\text{max}}$.

The difference in $M_{\text{max}}$ observed for the N025 and N053 tubes with $L = 1$ m may be related to contraction and/or expansion at both ends of the tube, because in such a case the depolarization of the $^{129}$Xe spin due to flow fluctuation can be induced by the onset of a turbulent flow.

The sensitivity of Xe to its local magnetic environment was successfully applied to demonstrate the usefulness of fused silica capillary tubes with i.d. $\leq 0.53$ mm. On the basis of the above discussion, it is considered that the amplitude of $M_{\text{max}}$ is governed by the Rb-Xe spin exchange rate and that the level of surface relaxation can be reduced by delivering laser-polarized $^{129}$Xe gas in a laminar flow.

In summary, we have reported the long-distance delivery of laser-polarized $^{129}$Xe under a continuous flow. This is the first NMR study of laser-polarized $^{129}$Xe fluid behavior that focused on the effects of the length and inside diameter of the delivery tube. It was demonstrated that laser-polarized $^{129}$Xe gas in a laminar flow can be delivered above 10 m by flowing it through a capillary tube with i.d. $\leq 0.53$ mm. Moreover, the high sensitivity of laser-polarized Xe can provide indirect insights into the specific fluid behavior of $^{129}$Xe gas flowing through silica capillary tubes. This potentially low-cost delivery system suggests the possibility of real-time high-resolution NMR/MRI.

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\begin{figure}[h]
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\includegraphics[width=\textwidth]{fig4.png}
\caption{Dependence of the maximum signal intensity $M_{\text{max}}$ and flow rate $Q_{\text{max}}$ for laser-polarized $^{129}$Xe on tube lengths $L$. Two silica tubes N025 and N053 with inside diameters of 0.25 and 0.53 mm, respectively, were used as delivery paths without pre-treatments. Both $M_{\text{max}}$ and $Q_{\text{max}}$ are normalized by those of N053 with $L$ of 1 m. The error bars were estimated by $M_0$ versus Re curve fittings for each tubes.}
\end{figure}
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