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Analysis of the effect of foreign gases in the production of hyperpolarized ^{129}Xe gas on a simple system working under atmospheric pressure

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Abstract

Experimental conditions that affect the degree of polarization of ^{129}Xe gas were tested for a higher degree of polarization to facilitate a laboratory use of ^{129}Xe NMR, primarily on the effect of addition of foreign gases. When He, N_2, or D_2 gas was added separately to pure Xe gas with natural isotope abundance, D_2 gas gave better results than the others in enhancing the degree of polarization in ^{129}Xe atom. When these gases were added in mixture, however, N_2 plus He was proved to be more efficient than D_2 or He in enhancing the degree of polarization. As a result, the degree of polarization was found to be increased by more than an order, when diluent gases were properly mixed; polarization as high as 35% was reached at gas composition of 5% Xe, 10% N_2, and 85% He, whereas only a few percent was attainable when Xe gas was polarized without mixing any foreign gases [J. Magn. Reson. 150 (2), 156–160 (2001)]. These results were discussed on a basis of quenching and buffer effects of foreign gases. Polarization was also measured after separating the pure Xe gas from the mixture; value of 22% was obtained for the Xe gas isolated after solidification in liquid nitrogen trap. Build-up time of the polarization was also tested, which did not change remarkably depending on the gas composition.

Keywords: ^{129}Xe NMR; Hyperpolarization; Degree of polarization; Effect of foreign gas; Build-up time

1. Introduction

Recently, hyperpolarized noble gases, such as ^{3}He and ^{129}Xe nuclei, have been developed as a high-sensitivity source of magnetic resonance, and their achievements have become center of attraction in magnetic resonance imaging of void spaces and related parts in living systems [1,2]. Usually, ^{3}He is better suited for the alveolar and bronchial space imaging because of its higher sensitivity, and ^{129}Xe can be used in brain and vascular imaging because of its higher solubility in blood and lipid-rich tissue [3,4]. The hyperpolarized noble gases are also providing great stimulus to advance the NMR studies of porous materials [5,6].

There are three major considerations in these hyperpolarized noble gas studies. First, higher polarization is primarily demanded, since its inherent sensitivity is very low for gases. This is the case especially with ^{129}Xe, since the inherent NMR sensitivity for this nucleus is as low as 5% of that of ^{3}He. Usually, lower gas pressure in a polarizing cell has an advantage in obtaining higher polarization because of a reduction in spin relaxation induced from molecular collisions in gas state and probably of the increased lifetime of van der Waals molecules under reduced pressure [7]. Also an addition of foreign gases, such as nitrogen or helium, is known to be useful in enhancing the polarization of noble gases [8]. Here nitrogen gas works as a quenching gas that serves to deexcite rubidium atoms in a non-radiative way, inducing an enhanced net polarization in rubidium atoms after repeated processes of excitation and deexcitation. The high-pressure helium molecules

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collisionally broaden the Rb D_1 absorption profile enabling up to 60% of a broad spectral output of 120 W fiber-coupled diode-laser array to be absorbed [9]. Secondly, a certain amount of the hyperpolarized gas needs to be taken out of the polarizing cell and be provided in the pure noble gas state. A higher-pressure type is preferred for such production of a large amount of gases, even if the polarization reached is more or less reduced [7]. Finally, high throughput and easy manipulation of gas is important for the successful works in NMR and MRI experiments in laboratory. Respecting these points, we have been developing a new system in which gases can be handled and optically-pumped sequentially under atmospheric pressure. Previously we reported the results of 1–2% of polarization without mixing foreign gases [10]. In order to enhance this polarization by more than an order, addition of foreign gases such as nitrogen, helium, and deuterium was tested in this study, and optimized conditions were sought for the production of hyperpolarized ^{129}Xe gas on our home-built system. After obtaining the optimum conditions for higher polarization, a polarization as high as 35% was realized for ^{129}Xe atom, and separation of the foreign gases was attempted by cooling the gases with liquid nitrogen and isolating as Xe ice. Evaporation of the Xe ice thus separated was confirmed to give pure Xe gas with polarization as high as 22%.

2. Experimental

2.1. Atmospheric pressure mode hyperpolarizing system

The experimental apparatus built for the present study is shown schematically in Fig. 1. A cylindrical glass cell (Pyrex polarizing cell, 6 cm diameter and 10 cm length) was placed in a fringe field (about 12 mT) of a super-conducting NMR magnet (9.4 T). The cell surface was not treated with chemical reagent for our simplicity in handling the cell. A droplet of rubidium (about 0.2 g) was deposited into the polarizing cell whose temperature was maintained constant at about 110 °C in an oven equipped with a heated-air blower LEISTER CH-6056. This temperature was selected from the preliminary monitoring of the polarization using pure Xe gas, which showed a plateau near 110 °C and decreased above 150 °C. A laser diode array (COHERENT Japan, FAP system, 795 nm) was used under 20 W output power and the laser light was transferred to the circularly polarizing unit through a glass fiber, and finally emitted into the polarizing cell. Here the linearly and randomly polarized light from the fiber cable was converted to circularly polarized light through a polarizing unit supplied from COHERENT Japan. A vessel containing K–Na alloy was used to dry the mixed gas at the entrance of the polarizing cell. The alloy is very effective in removing

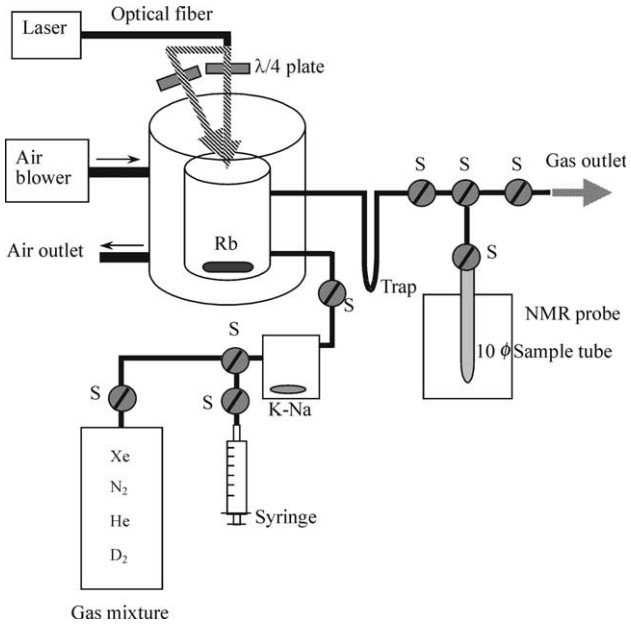

Fig. 1. The hyperpolarizing system operating under atmospheric pressure. Trap: cold trap of liquid nitrogen. S: Stopcock.

water, oxygen, and carbon dioxide gases that are possibly contained in Xe, He, N_2, and D_2 gases. The hyperpolarized gas was introduced into a 10 ϕ NMR tube in the NMR probe directly through Pyrex tubes (id. 2 mm).

In order to separate Xe gas from the foreign gases after polarization, a liquid nitrogen cold trap was attached to the polarizing cell. Once the hyperpolarized Xe gas was solidified in this trap, it was separated from the foreign gases and then evaporated before use when mixed gases were used in the hyperpolarizing experiment.

2.2. Materials

High purity Xe gas (over 99.995%) supplied from Himeji Daisan, deuterium gas (over 99AT%) from Showa Denko and the standard gases of N_2 and He from Osaka Sanso Kogyo were used after drying over K-Na alloy as shown in Fig. 1. Potassium (EP Grade, over 98%), Sodium (Reagent Grade, over 99%), and Rubidium (EP Grade, over 99.5%) metals supplied from Nacalai Tesque were used without further purification.

2.3. Methods

Optimization of the gas composition was attained by monitoring the degree of polarization while changing the types and contents of the gases mixed. The degree of polarization was estimated from the S/N ratio in ^{129}Xe NMR spectrum. Details of this method are described in Appendix. The gas mixture tested were: (a) Xe gas diluted with He, N_2, or D_2 gas so that Xe composition

changed from 5 to 100%, (b) Xe, N_2, and He mixtures, in which Xe composition was changed from 5 to 80% and N_2 composition from 1 to 30%, (c) Xe, N_2, and D_2 mixtures, in which N_2 composition was changed from 5 to 20%, while Xe composition was set equal to 5 or 10%, and (d) Xe, D_2, and He mixtures, in which D_2 composition was changed from 0 to 90%, while Xe composition was kept constant at 10%. Here, D_2 gas was used instead of H_2 as a quenching gas similar to N_2, preventing the possible effect of dipolar relaxation from H_2 [11]. In these experiments, all the gases used were from commercial sources with natural abundance, and 26.24% ^{129}Xe was included in the Xe gas. The mixed gases were hyperpolarized in the cell about 30 min before the NMR S/N ratio measurement unless otherwise specified. The mixed gas was once deposited in a syringe, which worked under atmospheric pressure and transferred to polarizing cell. Although our polarizing system can be operated under continuous flow mode, the present experiment was done under a batch mode.

In order to investigate the build-up and build-down profiles of polarization, S/N ratio was measured at a definite interval from just before the switching on of the laser light until after the switching off of it.

All NMR measurements were performed on a high-resolution NMR spectrometer Varian INOVA 400WB (9.4 T). The sample gas was introduced into a 10 ϕ NMR tube in the probe through a Pyrex glass line connected to the polarizing cell. The NMR spectrum of hyperpolarized ^{129}Xe was measured at 110.6 MHz, using 1 μs pulse width (flip angle = 8°) with the spectral width of 49321.8 Hz and the data points of 16,704, in which only one transient was accumulated. To calculate the degree of polarization, the S/N ratio measurement was made for a standard sample of Xe gas that included 50% of O_2 to promote the efficiency of repeated accumulation by reducing the relaxation time T_1. For this sample 40000 transients were accumulated using the same pulse width of 1 μs with the repetition interval of 1.0 s. All of these S/N ratio measurements were repeated three times and the data were averaged out to give final values.

2.4. Separation of foreign gases

Xe has a vapor pressure of 10^{-2} to 10^{-3} hPa at the liquid-nitrogen temperature. Therefore, it is possible to separate Xe from nitrogen, helium, and hydrogen by cooling the mixed gas down to 77 K and slowly pumping it out. At 77 K, only the Xe gas is frozen and other gases are easily pumped out from the system. In the present study the mixed gas was frozen in repeated times to obtain the desired amount of Xe ice, and then Xe was thawed and passed to NMR sampling tube to measure the S/N ratio. In this case, the optical-pumping time was set to 5 min in each run, which was determined from the results of the build-up time experiment.

3. Results

3.1. Enhancement of polarization by diluting with foreign gases

When Xe gas was mixed with various foreign gases and polarized, the S/N ratio in ^{129}Xe spectrum was observed to change as listed in Table 1. In this Table,

Table 1
The standardized S/N ratios measured for the Xe gas mixed with a few foreign gases[a]

Foreign gas[b]	Xe% in the mixture[b]								
	5	10	20	30	40	60	70	80	100
He	510	489		259			201		100
N_2	1438	695	496	346	296		160		100
D_2	2304	1393	563	481			191		100
N_2 1% + He	1587	1074	739	410	287	200		204	100
N_2 5% + He	2035	1532	836	509		245			100
N_2 10% + He	2903	1719	934	605	262	238			100
N_2 20% + He	2318	1600	850	651					100
N_2 30% + He	1571	1068		348					100
N_2 5% + D_2	2342								100
N_2 10% + D_2	2592	1135							100
N_2 20% + D_2	2298								100
	D_2% in the mixture[b]								
	0	5	10	30	50	70	90		
Xe10% + He	489	747	821	1147	1317	1390	1546		

[a] The S/N ratios are expressed by taking the 100% Xe gas as a standard ($S/N = 100$).

[b] The percentages specified for the mixture and the foreign gas mean the value after mixing all of the gases, e.g., the standardized S/N ratio of 2903 was obtained for the mixtures of 5% Xe, N_2 10%, and He 85% gases.

Table 2
Degrees of polarization (%) of ^{129}Xe observed for the gas mixtures

Foreign gas	Xe% in the mixture[a]								
	5	10	20	30	40	60	70	80	100
He	6.1	5.8		3.1			2.4		1.2
N_2	17.1	8.3	5.9	4.1	3.5		1.9		1.2
D_2	27.4	16.6	6.7	6.0			2.3		1.2
N_2 1% + He	18.9	12.8	8.8	4.9	3.4	2.4		2.4	1.2
N_2 5% + He	24.2	18.2	10.0	6.1		2.9			1.2
$N_2$10% + He	34.8	20.4	11.1	7.2	3.1	2.8			1.2
N_2 20% + He	27.6	19.0	10.1	7.8					1.2
N_2 30% + He	18.7	12.7		4.1					1.2
N_2 5% + D_2	27.9								1.2
N_2 10% + D_2	30.8	13.5							1.2
N_2 20% + D_2	27.3								1.2
	D_2% in the mixture[a]								
	0	5	10	30	50	70	90		
Xe10% + He	5.8	8.9	9.8	12.6	15.7	16.5	18.4		

[a] See footnote b in Table 1.

S/N ratios measured were standardized to the 100% Xe gas so as to express the polarization in each ^{129}Xe nucleus, i.e., the raw data of the ratios were divided by the fraction of Xe in the mixture gas. From this Table, the degree of polarization in the ^{129}Xe nucleus can be calculated by multiplying the listed S/N ratio by 0.012 (Table 2), since the degree of polarization for 100% Xe gas with natural abundance was observed to be 1.2%, which was obtained by comparing the S/N ratio observed for the hyperpolarized 100% Xe gas with that under the thermal equilibrium. It was observed that the polarization was increased with decreasing the percentage of Xe gas (Fig. 2) and that N_2 content of 10% gave the maximum polarization, although foreign gas mixtures were changed (Table 2). The mixed gas of N_2 plus D_2 was also tested as diluent gas, in which Xe content was maintained constant at 5% (Table 2). The results showed a similar effect as the N_2 plus He mixture in enhancing the polarization (Fig. 3). The maximum ^{129}Xe polarization reached in the present study was 35%,

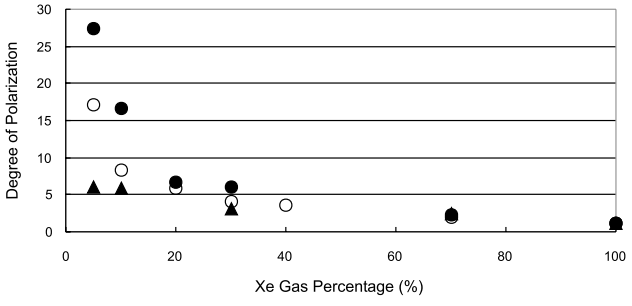

Fig. 2. The degree of polarization (%) of ^{129}Xe atom in the mixtures with He (▲), N_2 (○), and D_2 (●). The diluent gas is: He (▲), N_2 (○), and D_2 (●).

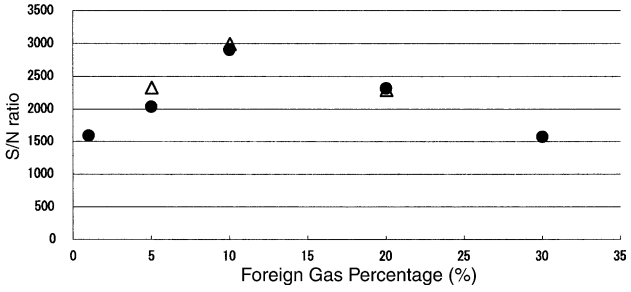

Fig. 3. The S/N ratio as a function of foreign gas composition of N_2 (●) or D_2 (△). Total gas pressure is 1 atm. Xe gas composition is fixed at 5%, N_2 or D_2 composition is changed between 1 and 30%, and the total gas composition is balanced by He gas.

which was observed at the composition of 5% Xe + 10% N_2 + 85% He, and a similar value of 31% was observed for the mixture of 5% Xe + 10% N_2 + 85% D_2. Since lower Xe percentage gave higher Xe polarization in Table 2, Xe content of lower than 5% was also tested for higher polarization. However, the mixture of 3% Xe + 12% N_2+85% He gave lower polarization (27.0%) than the maximum value in Table 2.

As for the decrease in polarization when Xe gas was separated from the mixture gases, a decrease to about 2/3 from the original value was observed in the separation from the N_2 plus He diluent gases; the polarization was decreased from 35% to 22% after solidification, separation and evaporation of the 5% Xe + 10% N_2 + 85% He mixture.

The build-up time of polarization was tested for repeated runs, and the results (Fig. 4) showed approximately 3 min for the build-up time. This time did not change significantly for the different composition of gas mixtures.

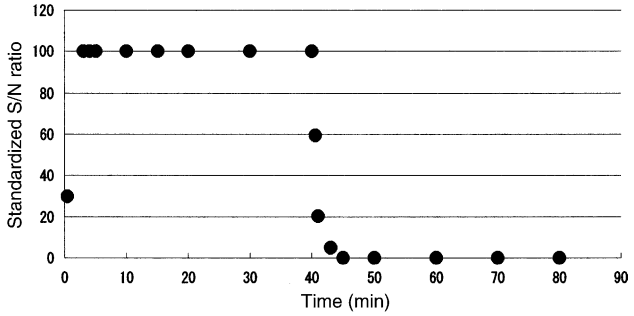

Fig. 4. The build-up and build-down profile of polarization. Laser light is turned on at time 0 and turned off at 40 min. The gas used is pure xenon with natural isotopic abundance.

4. Discussion

4.1. Comparison with other types of hyperpolarizing system

The degree of polarization observed under atmospheric pressure on our home-built system was compared with the results already reported on high-pressure and low-pressure type apparatus. The low-pressure type was reported to have a merit in obtaining higher polarization [12,13]. However, available total volume of the hyperpolarized Xe gas is small under this type of system, and other types that enable higher throughput of the polarized gas are desirable for practical (especially clinical) purpose. On the contrary, the high-pressure type is suitable for high throughput. In this case, however, special design is necessary to obtain the polarization comparable with the low-pressure type: polarization of 3–30% are reported at 6.5–9.8 atm [14,15]. The atmospheric pressure type may be welcome especially for handling in laboratory use for the reasons of simple design, easy handling, the reasonable throughput and safety in operation, but the practical data are lacking.

As for a commercial prototype released quite recently, the nominal polarization is 7% for the system supplied from Magnetic Imaging Technologies and 30% using isotopically enriched Xe (82% ^{129}Xe) for that from Amersham Health [16,17].

4.2. Effects of foreign gases

Foreign gases added as buffer gases in a polarizing cell can affect the behavior of Rb polarization through different pathways. First, foreign gases can sometimes work as quenching gases, to which excitation energies are transferred without the observation of fluorescent light. This effectiveness is evident in the quenching cross-sections, which indicates that the inert gas atoms such as He, Ne, Ar, and Kr possess no quenching effect [11]. However, molecular buffer gases possess quenching effect, and most notable one is N_2 gas. Secondly, foreign gases are known to affect the relaxation of polarized alkali atoms through collisional phenomena, which include sudden binary collisions that last for about 10^{-12} s and sticking collisions that lead to bound molecules (collision complexes) with the lifetimes of the order of 10^{-8} s [18]. Thirdly, foreign gases prevent alkali atoms to collide with wall freely by reducing the diffusion constant D_0 of alkali atoms. Such an effect may be estimated from the experimental values of D_0 that differ from one foreign gas to another [19]. When foreign gases are added excessively frequent collisions occur between alkali atoms and foreign gas molecules, and collisional disorientation becomes predominant, which can be estimated from the cross-section for spin disorientation σ [19].

Besides the mechanisms mentioned above, foreign gases can affect the polarization of alkali atoms through spin-exchange phenomena, but the alkali-depolarization cross-sections are reported to depend very little on the nuclear spins of the buffer gas molecules [11], and this pathway is not discussed further below. The results of our experiments (Table 2) may be interpreted in the following way. He gas possesses no quenching effect, and hence dilution with He gas only increases the Xe polarization in a limited magnitude (about 5 times increase on 10–20 times dilution, Table 2). This effect may come from preventing the wall relaxation in Rb atoms as well as Xe atoms, which is induced by buffer gases through the reduction of collisions with wall. When small amount of N_2 gas is added to Xe + He mixture, 10% N_2 gives the highest polarization under an excess amount of He buffer gas. This effect comes from quenching effect of N_2, and it can be said that approximately 10% N_2 is sufficient to induce quenching effectively. This is also the case for the $Xe + N_2 + D_2$ mixture. This experimental result agrees well with that reported by Franzen [20]. In the $Xe + D_2 + He$ mixture, D_2 90% gave maximum polarization. Also in the $Xe + D_2$ mixture, the highest polarization was observed for D_2 95% in Table 2. This is in clear contrast with the Xe10%+ N_2+He mixture, in which maximum polarization (20.4%) was observed for $N_2$10%. This difference in efficiency between D_2 and N_2 gases may come from the difference in quenching and transfer cross-sections of $N_2(58 \times 10^{-20}m^2)$ and $D_2(3 \times 10^{-20}m^2)$ [11].

It is unexpected in Table 2 that optimum values appear in N_2 content to obtain higher Xe polarization when light foreign gases such as He or D_2 coexist in a large amount, although the polarization changes monotonously with the N_2 content when light gases are not present ($Xe + N_2$ mixture). To explain such phenomena, a more detailed discussion on the polarization of Rb and its transfer to Xe atom, such as the formation of ternary collision complexes of Rb–Xe–N_2, seems necessary, and that is a subject of a future study.

4.3. Isolation of the hyperpolarized Xe gas

The freeze-pump-thaw cycle applied to separate Xe from the mixed gases usually results in a more or less reduced polarization. For example, the polarization was reduced to 1/4 of the initial value in one case [7], while in another case the initial polarization of 25% was reported to have been reduced by only 1–2% [21]. In the present study, this reduction was observed typically from 35% to 22%. Such a loss in polarization will be induced by some factors that affect the relaxation behavior of ^{129}Xe. One such factor is magnetic-field inhomogeneity met under the freezing process [22], and another is the polarization transfer occurring from ^{129}Xe to ^{131}Xe, which grows seriously at a lower magnetic field. In the present experiment, freezing was operated under a fringe field of 90 mT, and homogeneity was not regulated. Therefore, higher polarization reached after isolation of Xe gas is expected by improving the homogeneity of the magnetic field for freezing process and by increasing strength of the fringe field.

5. Conclusion

We have experimentally examined the effect of gas composition on the polarization of ^{129}Xe atom and on the build-up time of polarization, using the home-built hyperpolarizing system operating under atmospheric pressure. These results are utilized for the optimization of experimental conditions of the hyperpolarizing system operating under atmospheric pressure, for which detailed data have not previously been reported.

The highest nuclear polarization of 35% was obtained for the mixed gas of 5% Xe + 10% N_2 + 85% He, and the final polarization of 22% was obtained after isolating Xe gas from the mixed gas. The effect of foreign gases was explained through the quenching and buffer gas effects. By the quenching effect, rubidium atoms are deexcited in a non-radiative way, and the polarization is enhanced. This effect was marked in N_2 gas, as evidenced in the reported value of quenching and transfer cross-sections, and 10% N_2 gas was sufficient in inducing the quenching effect, whereas less effective quenching gas of D_2 was needed in as much as high content. The buffer gas effect works to suppress the molecular collisions of Rb as well as ^{129}Xe atoms with the wall, reducing the wall relaxation and works to enhance the polarization. Optimization of all theses effects is therefore, important in practical laboratory use of the hyperpolarized noble gas.

Acknowledgments

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Appendix
Estimation of the degree of polarization

The degree of polarization of the Xe atom was estimated from the enhancement in S/N ratio in the ^{129}Xe spectrum. Distribution of Xe spins at the thermal equilibrium is given by the Boltzmann distribution law,

$$\frac{N_\beta}{N_\alpha} = \mathrm{e}^{-\frac{\Delta E}{\kappa T}}, \tag{A.1}$$

where N_α and N_β are the number of spins at α and β spin levels, respectively, ΔE is the energy gap of the two spin states, and κ is the Boltzmann constant. The degree of polarization (P_{xe}), then, is the fraction of excess spins in the lower state and defined as in Eq. (A.2).

$$P_{\mathrm{Xe}} = \frac{N_\alpha - N_\beta}{N_\alpha + N_\beta} = \frac{1 - A}{1 + A} \times 10^2(\%), \tag{A.2}$$

where A means the Boltzmann factor N_β/N_α expressed as in Eq. (A.1).

The degree of polarization of a system that deviates from the thermal equilibrium condition can be estimated from the measurement of S/N ratio in the ^{129}Xe spectrum. This is based on the fact that signal intensity is proportional to the excess number of spins in the two spin states and that the noise level is constant in the ^{129}Xe spectrum, if the operating conditions of the spectrometer are all maintained constant. In fact, the noise level is very stable on the advanced high resolution NMR spectrometer, such as the one used in the present study, and hence the polarization (P_{xe}) can be estimated according to the following equation:

$$P_{\mathrm{Xe}} = P_{\mathrm{e}} \times \frac{S/N_{\mathrm{e}}}{S/N_{\mathrm{p}}}, \tag{A.3}$$

where P_{e} is the polarization at thermal equilibrium, and $\mathrm{S/N_e}$ and $\mathrm{S/N_p}$ are the S/N values measured at thermal equilibrium and hyperpolarized states, respectively. The measurement of $\mathrm{S/N_e}$ value was made using a standard (10 ϕ) sample tube after mixing 50% Xe and 50% O_2 under atmospheric pressure, in which oxygen was mixed to shorten the T_1 value of ^{129}Xe enhancing the efficiency in signal averaging.

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