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Production of Hyperpolarized ^{129}Xe Gas Without Nitrogen by Optical Pumping at ^{133}Cs D_2 Line in Flow System *

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We report production of hyperpolarized ^{129}Xe gas via spin-exchange with optically pumped Cs atoms at the D_2 line, achieved at low magnetic field in a flow system and in the absence of nitrogen gas. The nuclear spin polarization of hyperpolarized ^{129}Xe gas is enhanced by a factor of 10000 compared to that without optical pumping under the same condition, which corresponds to polarization of about 2.66%. Due to the high spin polarization, the radiation damping of hyperpolarized ^{129}Xe gas has also been observed in the flow system.

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Nuclear magnetic resonance (NMR) has been widely used in most fields of natural sciences, such as physics, chemistry, biology, and medicine. Because of the intrinsic low nuclear spin polarization at the thermal equilibrium, NMR is relatively insensitive. The polarization can be moderately increased by using lower temperature or higher magnetic fields,[1] whereas the nuclear spin polarizations of noble gases can be increased by four or five orders of magnitude via spin-exchange with optically pumped alkali-metal atoms.[2] The hyperpolarized noble gases, such as ^{129}Xe and ^{3}He, are rapidly being used in a wide variety of disciplines, including polarization targets,[3] magnetic resonance imaging (MRI),[4] neutron polarization,[5] fundamental symmetry studies,[6] high resolution NMR spectrometer,[7] surface science,[8] precision measurements,[9] biological systems,[10] and quantum computation,[11] etc.

Generally, Rb atoms and nitrogen gas are employed for optical pumping,[12] since high-power diode laser arrays at the wavelength of the Rb D_1 line (794.7 nm) are available.[13] However, Cs may be proposed as a better candidate for spin exchange with ^{129}Xe due to several advantages: the natural abundance of ^{133}Cs is 100% while Rb has two isotopes (^{85}Rb and ^{87}Rb), so that Cs is more convenient than Rb for wide applications of hyperpolarized ^{129}Xe, particularly in the clinic;[4] optical pumping cells for Cs are operated at lower temperatures with correspondingly fewer chemical corrosion problems;[14] according to the experimental results of one of authors, the spin-exchange rate of ^{133}Cs–^{129}Xe is about 10% higher than the Rb–^{129}Xe rate.[2,15] Although the experiment of spin-exchange with optically pumped (SEOP) of Cs has been reported, Cs was optically pumped at its D_1 line.[14]

As for nitrogen gas, it is usually used to quench the fluorescence light of alkali-metal atoms for production of the highly polarized noble gas,[12] but it is unwanted in many applications of hyperpolarized noble gases. Particularly in the study of single-crystal surfaces or other samples with a low number of sites, the competition between nitrogen gas and hyperpolarized noble gas for adsorption sites would reduce the utilization of polarization.[16] Therefore the production of nitrogen-free, hyperpolarized ^{129}Xe gas is necessary for this kind of experiment. Although the method of freeze–pump–thaw can separate the hyperpolarized ^{129}Xe gas from nitrogen gas,[17] this procedure would also lose a bit of polarization[18] and would be troublesome for some applications.

In this Letter, we report the production of hyperpolarized ^{129}Xe gas, by spin-exchange with optically pumped Cs atoms at the D_2 line without quenching gas nitrogen, at low magnetic field in a flow system, and the radiation damping of hyperpolarized ^{129}Xe gas has also been observed in this system due to the high spin polarization of ^{129}Xe.

In our previous experiment,[19] we obtained the solid and liquid hyperpolarized ^{129}Xe NMR signals; however, we could not observe the gaseous one by a single scan. The main reason was that the polarization was not high enough or the depolarization by various relaxation mechanisms was serious. In this experiment, in order to slow down the relaxation of ^{129}Xe upon collision with the tube wall, the improvement is to use a Teflon tube instead of a cylindrical Pyrex tube coated with silicone, which was usually used in

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most SEOP experiments,[12] but the other parts of the current experiment setup are similar to that in Refs. [19,20]. On the other hand, because of the flexibility of Teflon tube, it is convenient for applications, such as MRI, etc. Although our experimental results indicate that the improvement is effective, unfortunately, the detailed physical mechanisms of wall relaxation are poorly understood at present,[12] and this remains to be further investigated.

The pumping cell only containing several milligram of alkali-metal ^{133}Cs and 760 Torr natural isotopic xenon was placed in a 25-G magnetic field, which was generated by Helmholtz coils. Alkali-metal ^{133}Cs was evaporated at a temperature of about 80°C by a resistance heater. A 15-W semiconductor diode array (Opto Power Co. Model OPC-D015-850-FCPS) was employed to pump the ^{133}Cs vapour. After passing through a beam expander, Glan prism, quarter wavelength glass and convex lens, the laser light (wavelength of 852.1 nm) becomes a circularly polarized laser, and it resonates with the Cs D_2 absorption line (^{133}Cs $6^2S_{1/2} \rightarrow 6^2P_{3/2}$). This process produces the electron-spin polarization of ^{133}Cs vapour. Subsequent spin-exchange collisions between the polarized ^{133}Cs atoms and ^{129}Xe atoms cause the transfer of the angular momentum from a part of the polarized ^{133}Cs atoms to the unpolarized ^{129}Xe nuclei, and the hyperpolarized ^{129}Xe gas was obtained in a flow system after 25 min.

Our experimental result of spin-exchange optical pumping is shown in Fig. 1(a). The hyperpolarized ^{129}Xe NMR signal was measured at 22.16 MHz on a Bruker AC-80 spectrometer (1.879 Tesla) with a home-built probe and a tipping angle of 90° for a single acquisition at 200 K. To determine the enhancement factor of the hyperpolarized ^{129}Xe, we measured the ^{129}Xe NMR signal without optical pumping of Cs atoms, i.e. at thermal equilibrium, under otherwise identical conditions. Figure 2(b) shows the thermal ^{129}Xe NMR signal at 200 K, but with 5000-scan accumulation.

In terms of the relation of the magnetization versus the nuclear spin polarization given by Abragam,[21] and by comparison of the nuclear spin polarization P_L of ^{129}Xe, produced via spin-exchange with optically pumped Cs atoms, with the Boltzmann polarization P_B of ^{129}Xe at thermal equilibrium, the enhancement factor of the ^{129}Xe nuclear spin polarization can be calculated using the formula

$$f = P_L/P_B = I_L/I_B, \qquad (1)$$

where I_L and I_B are the integral intensities of the measured NMR signals of the hyperpolarized ^{129}Xe gas under the conditions of the laser optical pumping and the thermal equilibrium, respectively. Therefore, the enhancement factor of the hyperpolarized gas ^{129}Xe is about 10000 on the basis of our measurements. The Boltzmann polarization P_B of ^{129}Xe at 200 K and in 1.879 Tesla is about 2.66×10^{-6}; hence the nuclear spin polarization P_L of hyperpolarized ^{129}Xe gas is about 2.66%. This polarization also corresponds to the conventional ^{129}Xe gas NMR signal at 200 K and in a magnetic field of 18000 Tesla. However, until now the highest magnetic field used for NMR spectrometer is 21 Tesla in the world, i.e. the proton NMR frequency is about at 900 MHz.

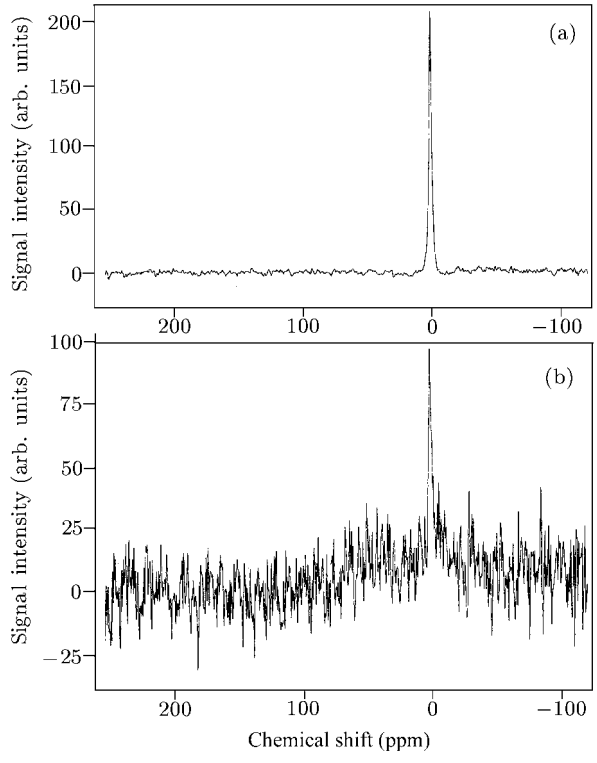

Fig. 1. (a) NMR signal of the hyperpolarized ^{129}Xe produced by spin-exchange with optical pumped ^{133}Cs in a flow system at 200 K with one scan. (b) NMR signal of the thermally polarized ^{129}Xe without optical pumping in a flow system at 200 K with 5000 scans.

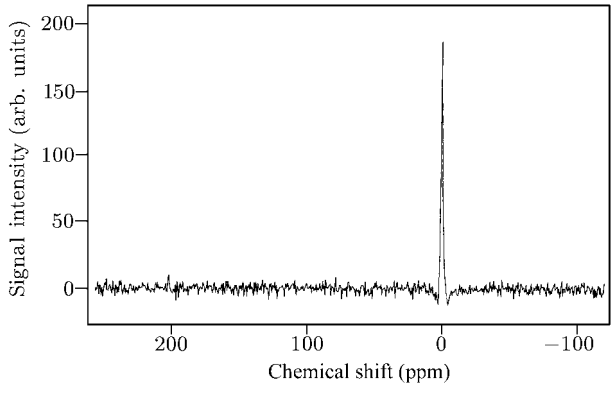

Fig. 2. Hyperpolarized ^{129}Xe gaseous phase spectrum with radiation damping when the 150° pulse was applied.

Nitrogen gas has been generally used as both buffer gas and quenching gas in order to elevate the

polarization in many former SEOP experiments. As a buffer gas, nitrogen decreases the relaxation rate due to the cell wall. As a quenching gas, during the optical pumping of alkali-metal, nitrogen suppresses re-radiation of light and eliminates radiation trapping as a source of relaxation by quenching the excited atoms. During the spin-exchange at low magnetic fields (< 50 G), nitrogen participates in three-body collisions with alkali-metal-^{129}Xe van der Waals molecules when the total pumping cell pressure is a few tens of Torr, and spin-exchange rate from van der Waals molecules is much larger than that from binary collisions.[12] However, either a magnetic field of a few hundred Gauss or a multi-atmosphere pressure would greatly suppress the relaxation due to the van der Waals molecules,[22] and binary collisions would play a primary role. In our experiments, since nitrogen gas has not been used, binary collisions would mainly transfer the spin polarization from the ^{133}Cs atoms to the ^{129}Xe at atmosphere pressure, and also the ^{129}Xe gas itself acts as buffer gas, which not only preserves the orientation of the ground state of ^{133}Cs atoms but also prevents the atoms from colliding with the walls.[23] In addition, the efficiency of optical pumping of Cs atoms at the D_2 line is generally lower than that at the D_1 line.[23] Even under the condition of optical pumping at the Cs D_2 line and without nitrogen gas, we have still obtained as high a polarization as that in Ref. [14]. Furthermore, our experiments have been implemented in a flow system, while Ref. [14] was carried out in a sealed cell.

Usually, radiation damping is observed at high magnetic fields using a high-resolution spectrometer only when the sample has high spin concentration, such as water protons.[24–26] It was at high magnetic field (14.1 Tesla) and in a sealed NMR tube that radiation damping of hyperpolarized gaseous ^{129}Xe was obtained.[27] However, after the polarization of ^{129}Xe has been enhanced by four orders of magnitude, the effect of radiation damping of hyperpolarized gas ^{129}Xe can also been observed even though at low magnetic field in a flow system. Figure 2 shows the ^{129}Xe NMR spectrum with effect of radiation damping when 150° pulse was applied. The spectrum is not the standard Lorentzian line shape of NMR but phase distortion, and such distortion is the typical result of the radiation damping.[20,28] This also indicates that the large polarization enhancement could compensate for the low spin concentration of ^{129}Xe so much that radiation damping occurs in our present experimental system.

The polarization in a flow system is not so high as that in Ref. [29]. The main cause is that in our experiment the bandwidth of the diode laser array is 30 Å, whereas the absorbed bandwidth of ^{133}Cs atom in the 760 Torr natural xenon is only 30 GHz. Therefore, the efficient pump power is only about 0.125 W, while a 140 W laser array have been employed in Ref. [29]. Also, the polarization can be lost due to the magnetic field inhomogeneity during the transfer of hyperpolarized ^{129}Xe gases in a flow system, whose loss is larger than that in a sealed NMR tube. Moreover, the relaxation of ^{129}Xe atoms at the wall also decreases the polarization. Thus further possible improvements are to use a larger power laser array and to increase the gas pressure of the pumping cell in order to enhance the effective optical-pumped absorbed power.

In conclusion, at low magnetic field in a flow system, we have obtained hyperpolarized ^{129}Xe gas via spin-exchange with optically pumped Cs atoms at the D_2 line under nitrogen-free condition. The radiation damping of hyperpolarized ^{129}Xe gas has also been observed. An important aspect of the hyperpolarized ^{129}Xe polarization method in a flow system without nitrogen described here is that it can be readily extended to produce larger quantities of hyperpolarized ^{129}Xe for other applied research, especially in material sciences, or directly in clinic nuclear magnetic imaging or biology NMR.

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