Solubility study of hyperpolarised xenon: toluene as a model system

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Introduction

Hyperpolarization has become an important technique for generating highly non-equilibrium spin-systems and thereby dramatically enhancing the MR signal by up to five orders of magnitude [1].

The possibility of using injectable solutions of hyperpolarised xenon in a suitable carrier agent has enabled *in vivo* MRI and been shown to be applicable for carrying out magnetic resonance angiography[2].

Furthermore techniques for transferring the huge non-thermal polarisation from laser-polarised ^{129}Xe to biologically relevant nuclei such as ^{13}C and ^{15}N have opened a variety of molecules that may be enhanced. These techniques rely on the one hand on dissolving the xenon into solution and on the other hand creating through space contact between nuclei.

We present a ^{129}Xe NMR study of the solubility and dissolution of liquid xenon in toluene as a model for an organic solvent. Phase change over time of xenon from solid to liquid and gaseous has been monitored with ^{129}Xe NMR spectroscopy.

Our results are in agreement with literature values.

Methods

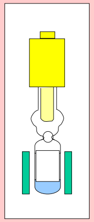
Hyperpolarized ^{129}Xe was produced by spin exchange optical pumping in both batch and flow mode, using an in-house polarizer[5]. NMR experiments were conducted on a 1.5 T Siemens Vision clinical scanner (Erlangen, Germany).

^{129}Xe NMR spectra were acquired using a home-built solenoid coil tuned at 17.60 MHz.

Hyperpolarized xenon was condensed within a glass distillator and then transferred into a glass tube containing toluene, submerged in liquid nitrogen, creating a frozen layer of xenon above the frozen toluene. Toluene was chosen for its relatively high solubility of xenon.

This process was completed inside the bore of the magnet.

Melting of frozen xenon on top of frozen toluene up to room temperature was monitored with small flip-angle pulse-and-acquire ^{129}Xe NMR spectra every 10s.



Ostwald solubility

Gas solubility can be expressed as the *Ostwald solubility*, defined as the ratio of the concentration of dissolved gas to the concentration of free gas.

In the table on the right, some literature values for xenon Ostwald solubility in water and organic compounds are reported.

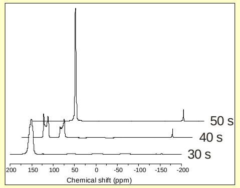
Compound	Ostwald solubility at T=25 ^{0}C
Water [3]	0.11
Hexane[3] C_6H_{14}	4.8
Benzene[3] C_6H_6	3.1
Toluene[4] C_7H_8	5.0
Carbon disulphide[3] CS_2	4.2

Many factors such as temperature, pressure, molecular size, concordance of polarity of solute and solvent, affect the proportion of gas dissolved in the liquid.

In a NMR spectrum, the ratio of the liquid/gas peak integrals divided by the respective volumes gives the Ostwald solubility at a given temperature and pressure.

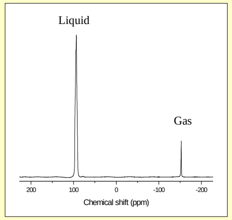
Results: ^{129}Xe NMR spectra

The first figure on the right shows the first three ^{129}Xe spectra of the melting of xenon over time, acquired after freezing xenon on top of frozen toluene.



At 30s only the solid xenon peak is observed. 10s later two complex peaks are present: the solid one, with a component due to the interaction of solid xenon with toluene, and a liquid one, with a component assigned to an interaction with the frozen xenon surface. The gas peak at –150 ppm is observed. In the next spectrum the peaks of xenon dissolved in solution and the xenon gas are present.

In the second figure, both liquid and gas phase signal are observed in the ^{129}Xe NMR spectrum. An example, acquired after 90s from the beginning of the experiment, is reported in the second figure on the right.



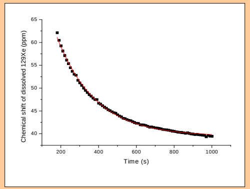
^{129}Xe T1 in both dissolved and gas phase can be calculated from the decay of the intensity of the liquid and gas peaks.

^{129}Xe T1(dissolved)= (156 ± 2) s

^{129}Xe T1 (gas) = (226 ± 3) s

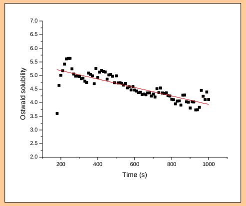
Xenon solubility

The first figure shows the change over time of the chemical shift of ^{129}Xe dissolved in toluene, during melting from frozen (liquid nitrogen bath) up to room temperature, as a consequence of the temperature change.



Gas chemical shift is temperature invariant.

The liquid to gas peak ratio, corrected for the decay due to the respective ^{129}Xe T_1s, and for the liquid and gas volumes, gives the Ostwald solubility as a function of the time, and therefore as a function of temperature of the system.



As expected from previous literature work[6], xenon solubility in organic solvents decreases as temperature increases.

Conclusion

We have presented a solubility study of hyperpolarized ^{129}Xe in toluene as a model system to investigate polarization transfer by SPINOE.

Polarization transfer to molecules of biological interest could provide invaluable information about metabolic function and thereby observe processes that have hitherto eluded the sensitivity of MR techniques.

Acknowledgements
This work was supported by the Council for Research Councils Basic Technology Programme grant number GR/S23612/01 and by Cancer Research UK [CUK] grant number C1060/A808.

References

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