Ultrafast Z-spectroscopy for ^{129}Xe NMR-based sensors

Céline Boutin,[‡] Estelle Léonce,[‡] Thierry Brotin,[†] Alexej Jerschow,[§] and Patrick Berthault[‡]

[‡] CEA Saclay, IRAMIS, SIS2M, UMR CEA/CNRS 3299, Laboratoire Structure et Dynamique par Résonance Magnétique, 91191 Gif sur Yvette, France

[†] Laboratoire de Chimie, CNRS, Ecole Normale Supérieure de Lyon, 46 Allée d'Italie, 69364 Lyon Cedex 07, France

[§] Chemistry Department, New York University, 100 Washington Square East, New York, NY 10003, USA

SUPPORTING INFORMATION

Content :

Experimental procedures

Production of polarized xenon. 83%-enriched ^{129}Xe from EurisoTop was polarized by spin-exchange optical pumping with rubidium[1] using a recently installed home-built setup based on laser diodes. The photons exiting from a duo-FAP system (2x30W) and circular polarizer from Coherent illuminated a Pyrex cell placed at the center of a 100G magnetic field. The bandwidth of the laser diodes being about 2 nm, pressure-broadening was necessary. Therefore, the pressure in the cell rose to 3 atm (measured at room temperature) with a mixture of 2% Xe – 10% N_2 – 88% He. The pumping cell was heated for 2 min to 410K via a flow of hot N_2 in an external envelope, in a fashion similar to what was developed for our previous experimental setup.[2] Then xenon was condensed in a cold finger inside a 3kG solenoid immersed in liquid nitrogen, and thus separated from helium and nitrogen. The average polarization value with this experimental setup was 15%, measured for the gaseous phase in the NMR spectrometer.

NMR. Hyperpolarized xenon was introduced above the solution in the upper part of the screwed NMR tube via a vacuum line in the fringe field of the NMR magnet. A xenon pressure of ca. 1 atm was present above the solution. Vigorous shaking of the tube followed by a 10 sec delay ensured efficient dissolution and equilibration of both gaseous and dissolved phases. A narrow-bore 11.7T Avance II Bruker spectrometer equipped with a micro-5 probehead and a dual $^{129}Xe/^{1}H$ insert were used. The length of the xenon (inner) detection coil was 1.3 cm, as confirmed by the Z-profiles. A GREAT40 gradient unit delivered a gradient of up to 200 $G.cm^{-1}$ per axis. The experiment time required to record each spectrum was on the order of several seconds. Before Fourier transformation the FIDs were apodized by a 200 Hz exponential function. The data points in Figure 3 were obtained by integration of the dip in the difference spectrum, and normalization of the measured area by the area of the Z-profile on both sides of the dip on the UFZ spectrum. With this procedure, the effect of decaying polarization is taken into account. All the NMR experiments were performed at 298K.

Figures

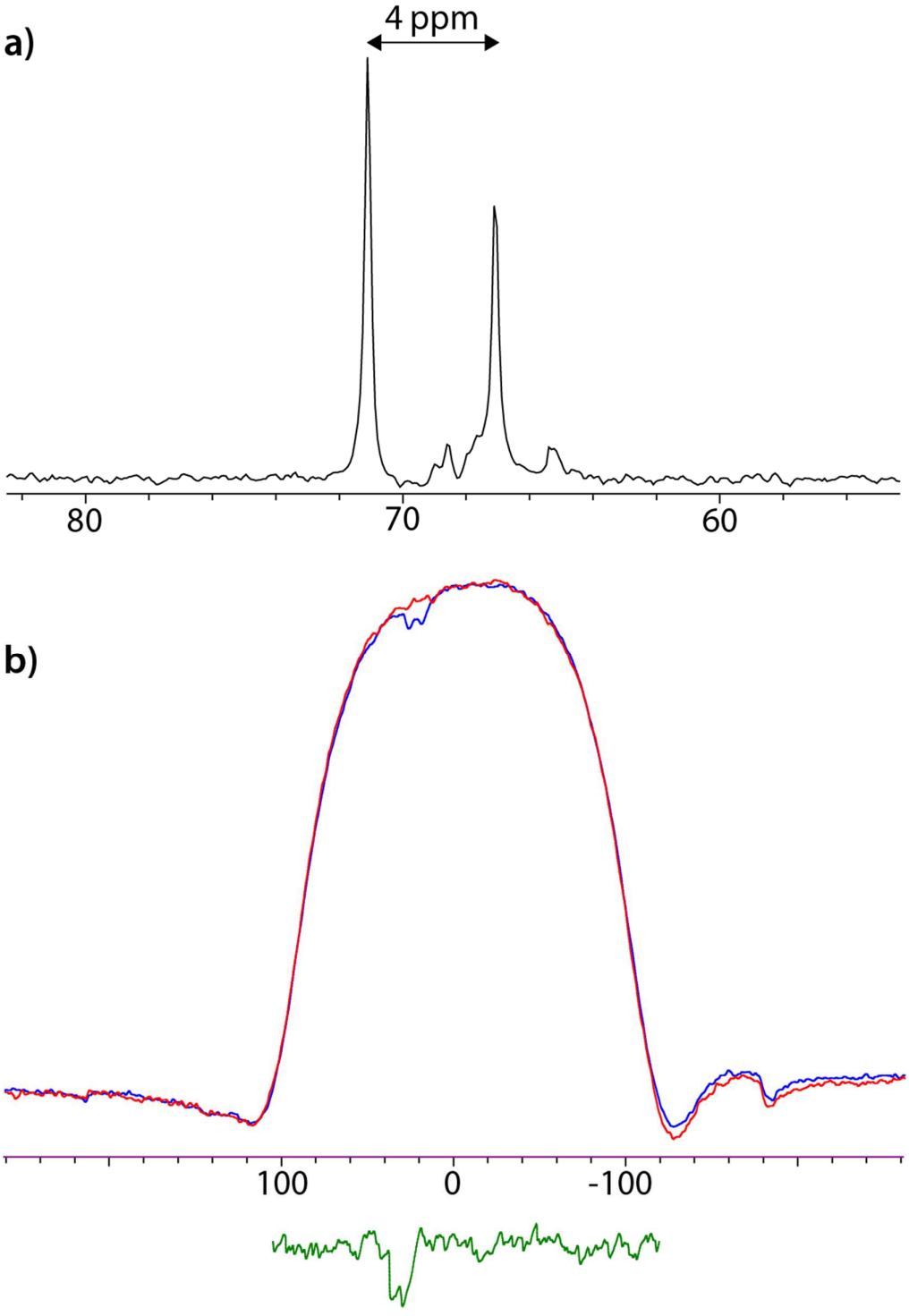

Figure S1. Test for the spectral separation of the ^{129}Xe signals in UFZ-spectroscopy. The sample is a mixture of two cryptophanes (29µM) in which the xenon resonances are separated by 4 ppm (550 Hz). a) ^{129}Xe NMR sub-spectrum obtained by the sequence (90°soft – acquire - short delay) repeated 64 times; b) ^{129}Xe UFZ spectrum obtained with the sequence of Figure 3a. Saturation duration: 8 s; saturation strength: 2.4 µT.

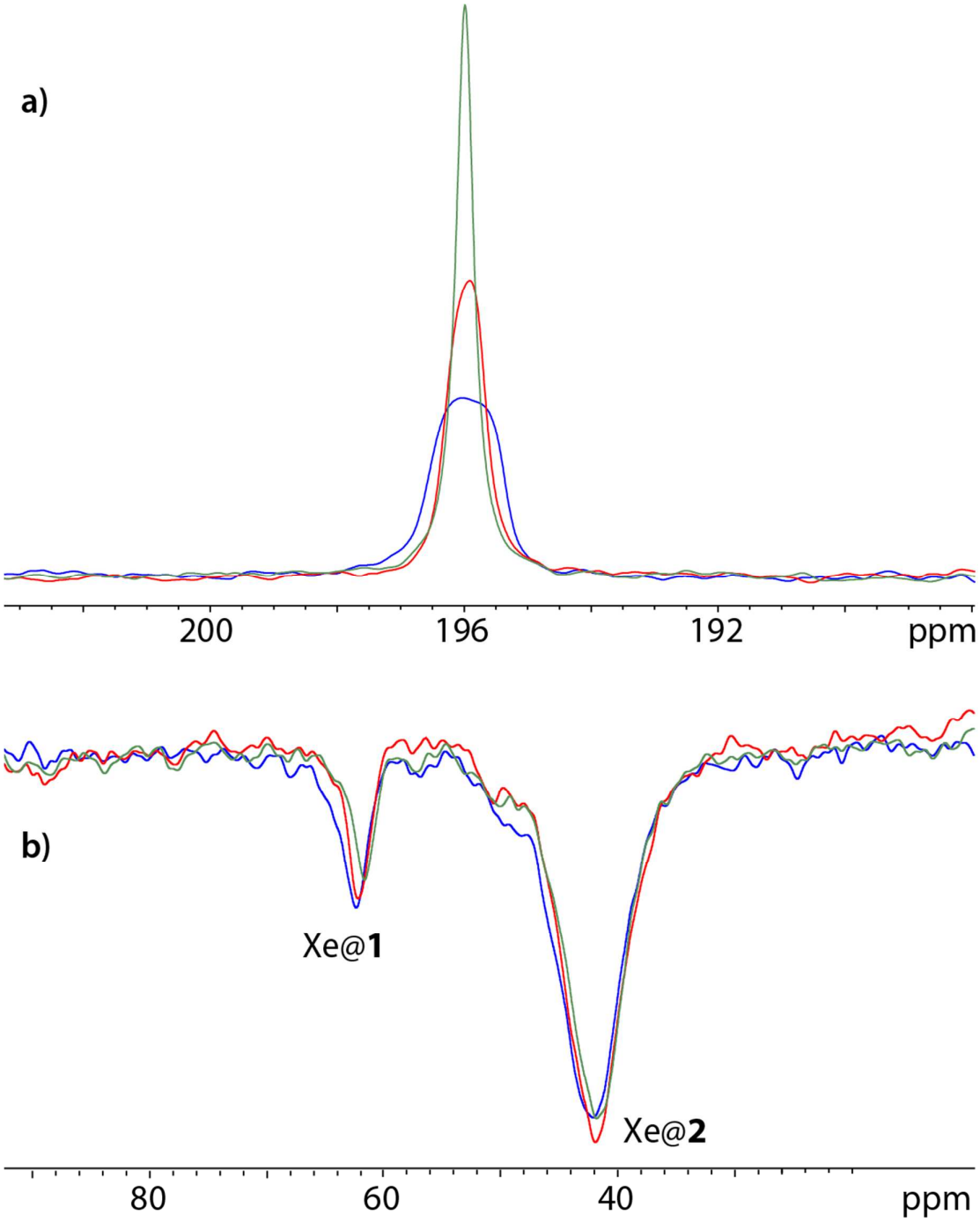

Figure S2. Effect of the static magnetic field inhomogeneity on the ^{129}Xe UFZ spectrum. The sample was the mixture of cryptophanes of Figure 1. For this experiment, the shims sample were firstly adjusted (green), then the Z shim was on purpose misadjusted (red, blue). a) ^{129}Xe NMR spectra; b) Corresponding ^{129}Xe UFZ spectra, obtained with a 8 s saturation with a B_1 strength of 2.4 µT.

[1] Walker, T. G.; Happer, W. *Rev. Mod. Phys.* **1997**, *69*, 629-641.
[2] Desvaux, H.; Gautier, T.; Le Goff, G.; Pétro, M.; Berthault, P. *Eur. Phys. J. D* **2000**, *12*, 289-296.