Determination of a paramagnetic concentration inside a diamagnetic matrix by Solid-State NMR

Supplementary Information

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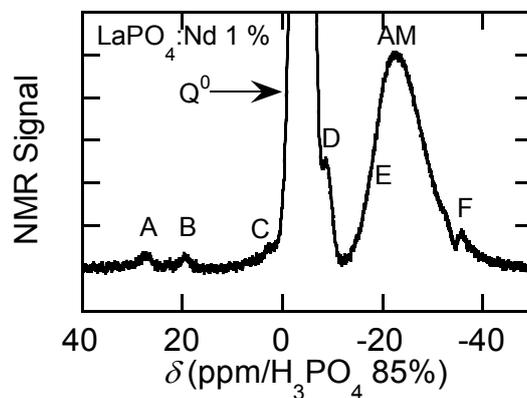
Figure SI-1: ^{31}P SSNMR of $La_{0.99}Nd_{0.01}PO_4$ with a very short repetition time (50 ms) for exalting paramagnetic peaks noted A to F. Q^0 is the peak corresponding to phosphorous without Nd in the first coordination shell and AM an amorphous phase. Figure previously published in 2014 by Maron et al.[1]
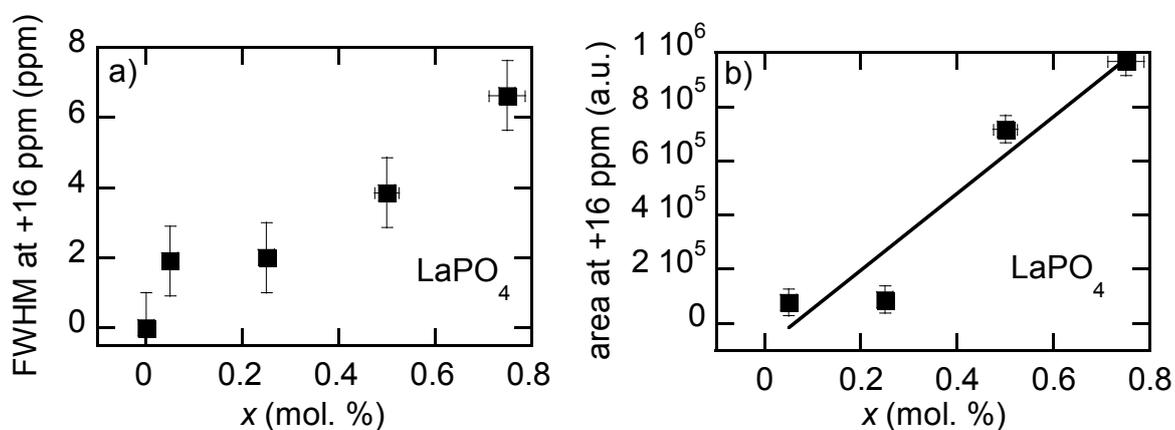
Figure SI-2: evolution of FWHM (a) and the area (b) of a paramagnetic peak of $La_{1-x}Nd_xPO_4$ (peak B at +16 ppm).
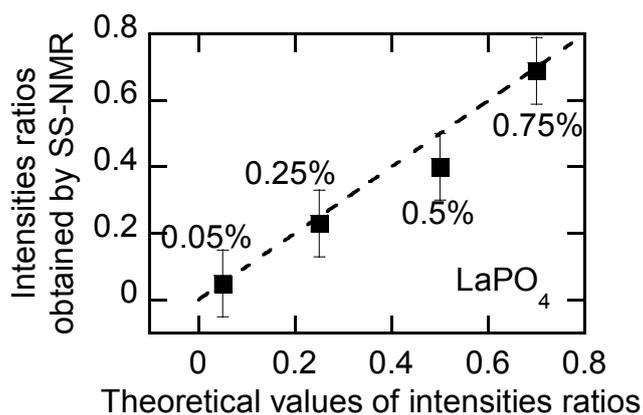
Figure SI-3: ratios between the intensities of one paramagnetic peak and the unshifted peak in function of the theoretical values of these ratios for $LaPO_4$ (paramagnetic peak at +16 ppm) .[2] The dotted line corresponds to $y = x$, indicating that the real Nd concentration matches the nominal one; numbers indicatedthe nominal concentrations.
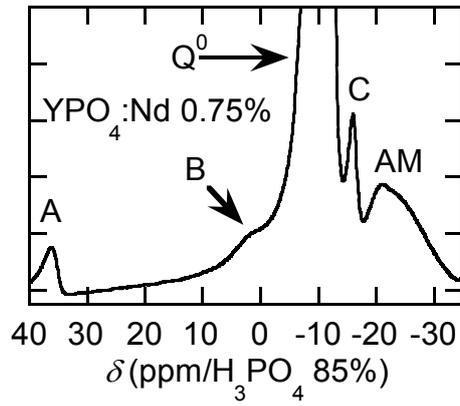
Figure SI-4: NMR spectra of YPO$_4$:Nd 0.75 %. The attribution is made according to Palke and Stebbins.[3] This spectrum is obtained with a short repetition time (50 ms). A to C are paramagnetic peaks, corresponding to ^{31}P with Nd^{3+} in their first shell coordination; Q^0 is the unshifted peak corresponding to ^{31}P without Nd^{3+} and AM is an amorphous phase due to an excess of P. Both signals are enhanced by the chosen conditions, i.e. a short relaxation time.
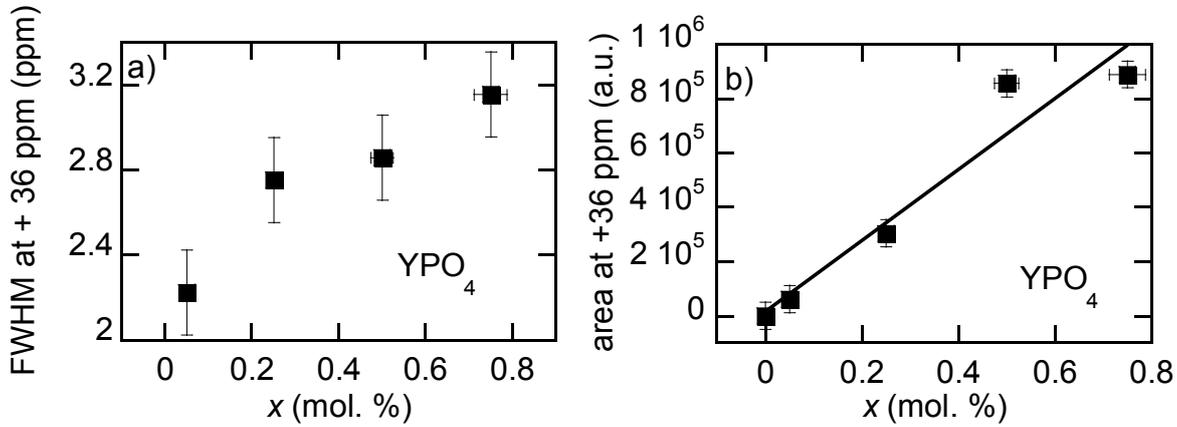
Figure SI-5: evolution of FWHM (a) and the area (b) of a paramagnetic peak of Y$_{1-x}$Nd$_x$PO$_4$ (peak A at +36 ppm).
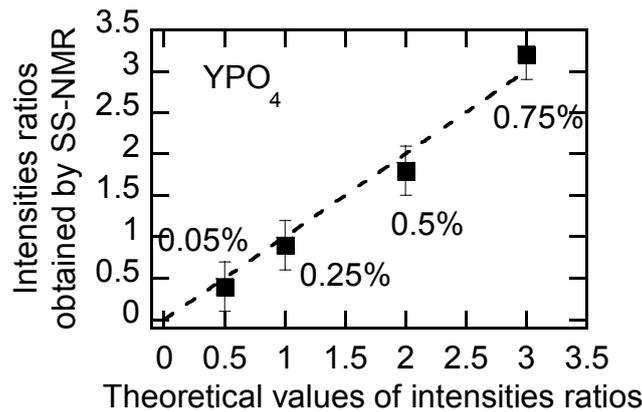
Figure SI-6: ratios between the intensities of one paramagnetic peak and the unshifted peak in function of the theoretical values of these ratios for YPO$_4$ (paramagnetic peak at +36 ppm) .[2] The dotted line corresponds to $y = x$, indicating that the real Nd concentration matches the nominal one; numbers indicated the nominal concentrations.
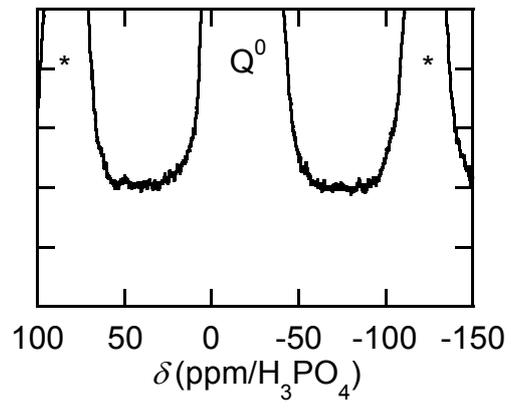
Figure SI-7: SSNMR spectra of 499P$_2$O$_5$:167MgO:333Na$_2$O:1Nd$_2$O$_3$ glasses with a short repetition time (50 ms) to try to enhance paramagnetic peaks. Q^0 corresponds to the ^{31}P with no Nd^{3+} in its vicinity and stars design spinning bands (ω_{rot} = 15 kHz).
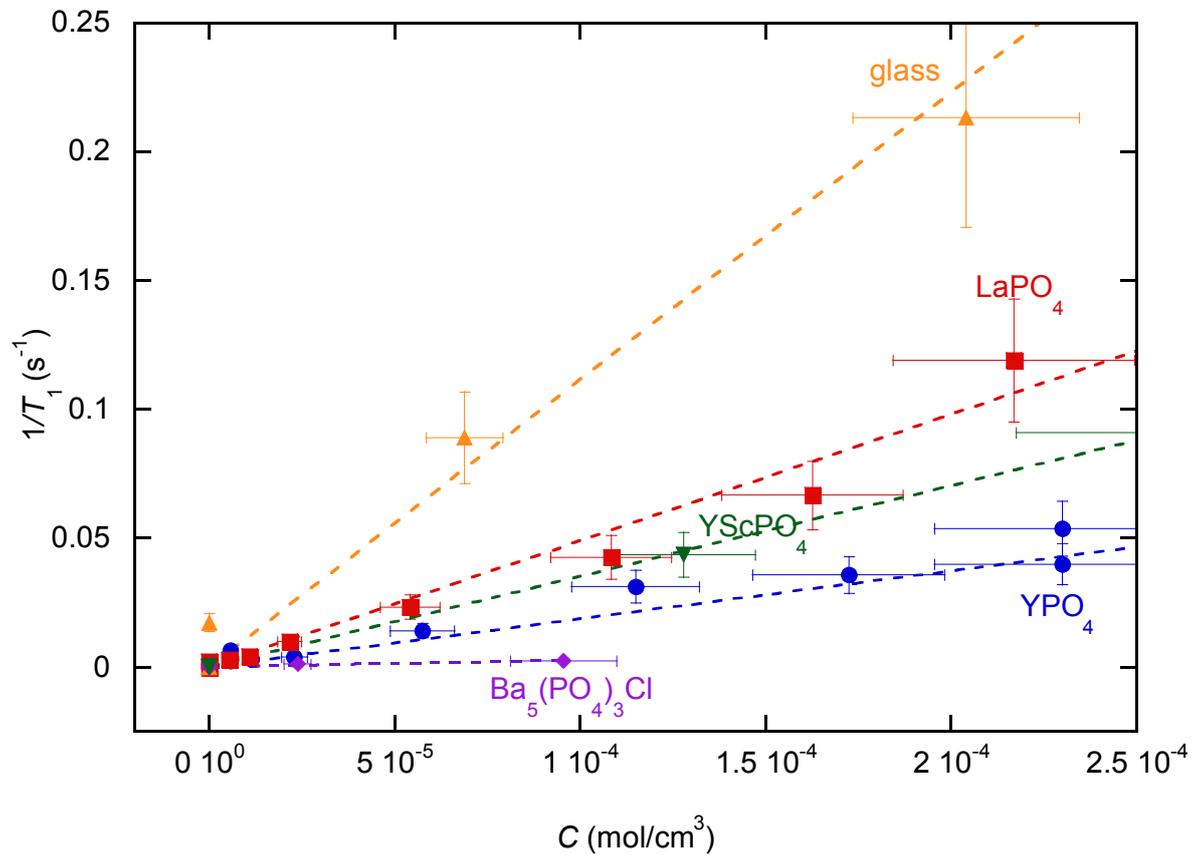
Figure SI-8: variation of $1/T_1$ in function of the concentration C (expressed in mol/cm³) for very low level doping.

1. S. Maron, G. Dantelle, T. Gacoin and F. Devreux, *Physical Chemistry Chemical Physics*, 2014, **16**, 18788-18798.
2. A. C. Palke, J. F. Stebbins and L. A. Boatner, *Inorganic Chemistry*, 2013, **52**, 12605-12615.
3. A. C. Palke and J. F. Stebbins, *American Mineralogist*, 2011, **96**, 1343-1353.