Electronic Supplementary Information

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The duality of UiO-67-Pt MOFs: connecting treatment conditions and encapsulated Pt species by *operando* XAS

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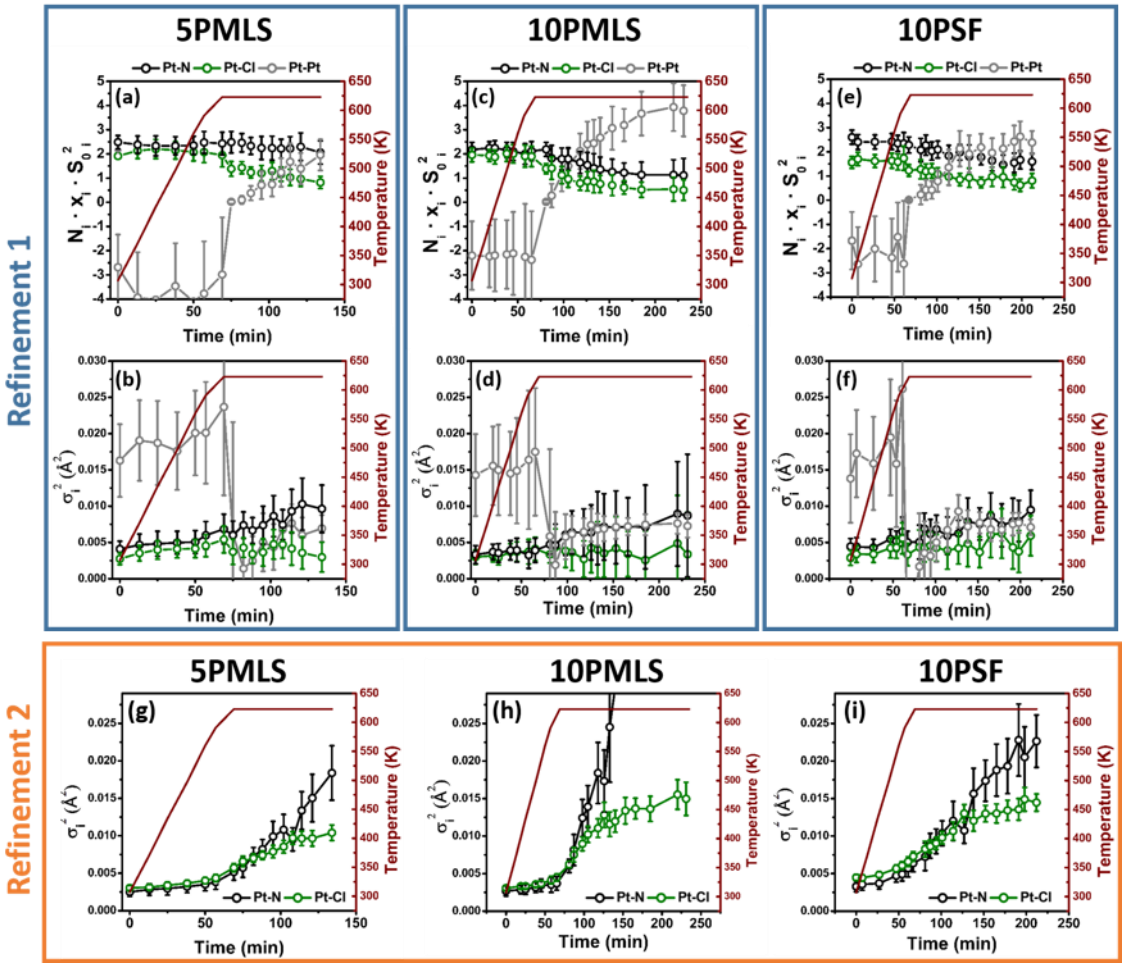

Figure S1. Results of the first and second refinement of simultaneous parametric analysis of *operando* EXAFS collected during TPR are reported in the blue and orange box respectively. Panel **(a)** and **(b)** reports the amplitude factor and the Debye-Waller factor, σ^2_i, as a function of time for the 5PMLS sample, where the amplitude factor is defined by the product between the coordination number N_i, the relative fraction x_i and by the scattering amplitude factor ${S_0}^2_i$ and where *i* denote the three kind of SS considered, i.e. Pt-N and Pt-Cl for the Pt-linker component, and Pt-Pt for the Pt-NPs phase (N in black, Cl in green and Pt in grey). Parts **(c)**, **(d)** and **(e), (f)** as part (a), (b) for the 10PMLS and 10PSF respectively. Parts **(g), (h)** and **(i)** shows the behavior, in the second refinement, of the Debye-Waller factor, σ^2_i, (where *i* indicates the SS of the species considered before) in function of time for the 5PMLS, 10PMLS and 10PSF, respectively.

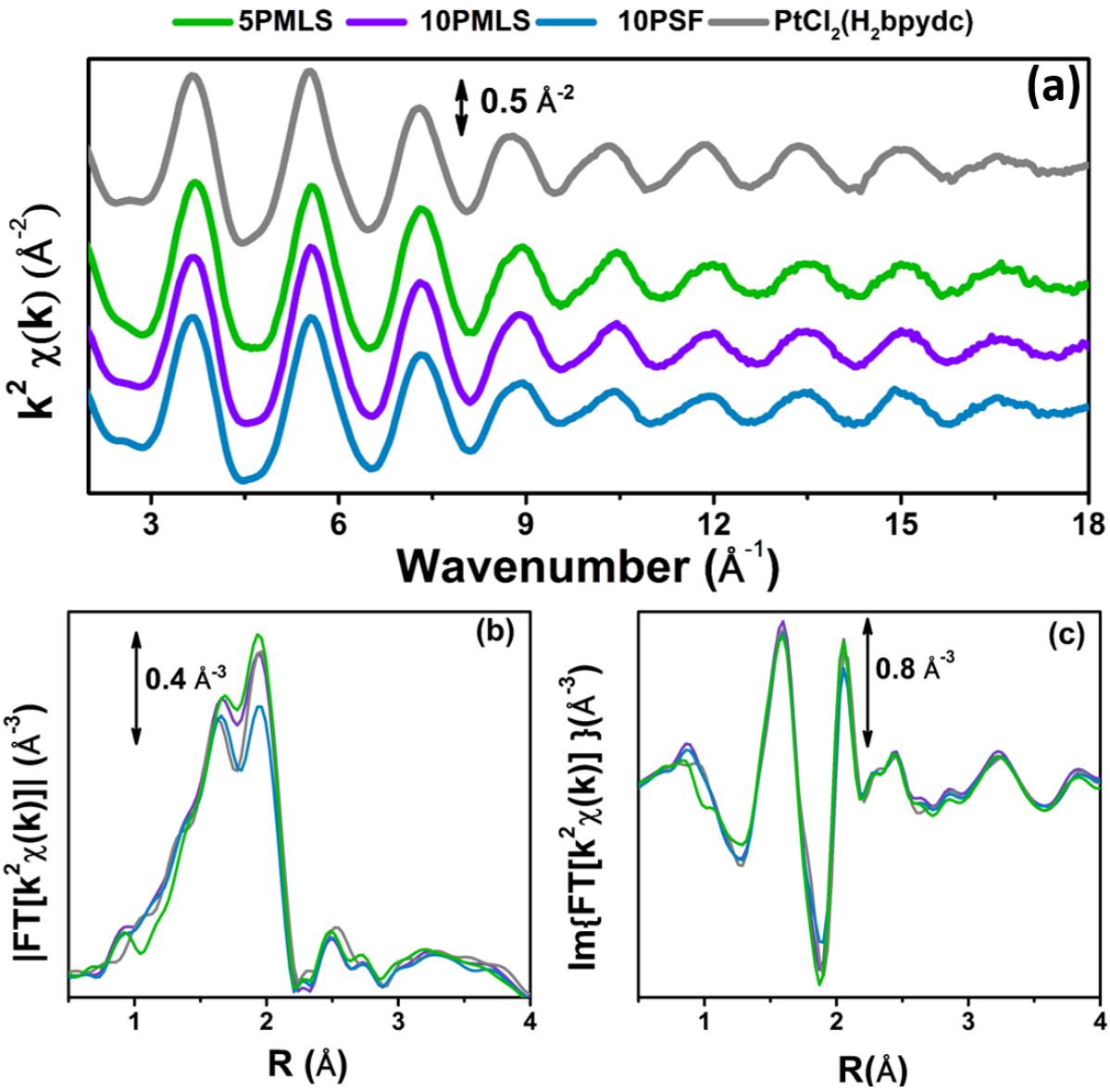

Figure S2. (a) Experimental Pt L_{III} edge $k^2\chi(k)$ phase uncorrected, modulus **(b)** and imaginary part **(c)** of the experimental FT EXAFS spectra for 5PMLS (green), 10PMLS (violet) and 10PSF (cyan) in the as-prepared state at RT in air and the $PtCl_2(H_2$ bpydc) linker (grey) as reference.

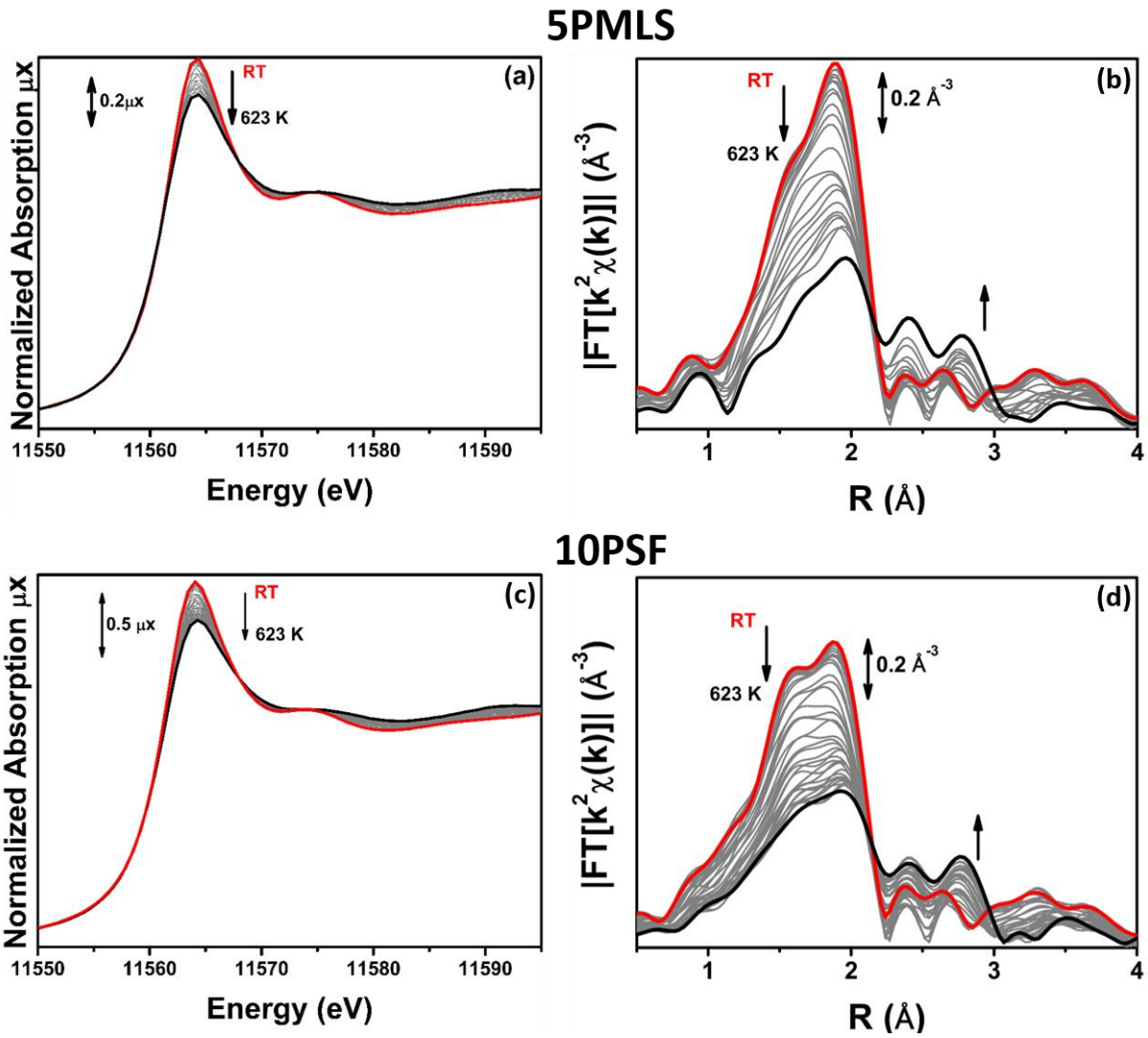

Figure S3. Part (a) and (c): Sequence of Pt L_{III} edge operando XANES spectra during TPR, collected at the starting point at RT (red) and upon heating to 623 K in 10%H_2/He (grey) until the steady state at 623 K is reached (black) of 5PMLS and 10PSF, respectively. Part (b): as part (d) for the k^2-weighted, phase uncorrected FT of the corresponding EXAFS curves.

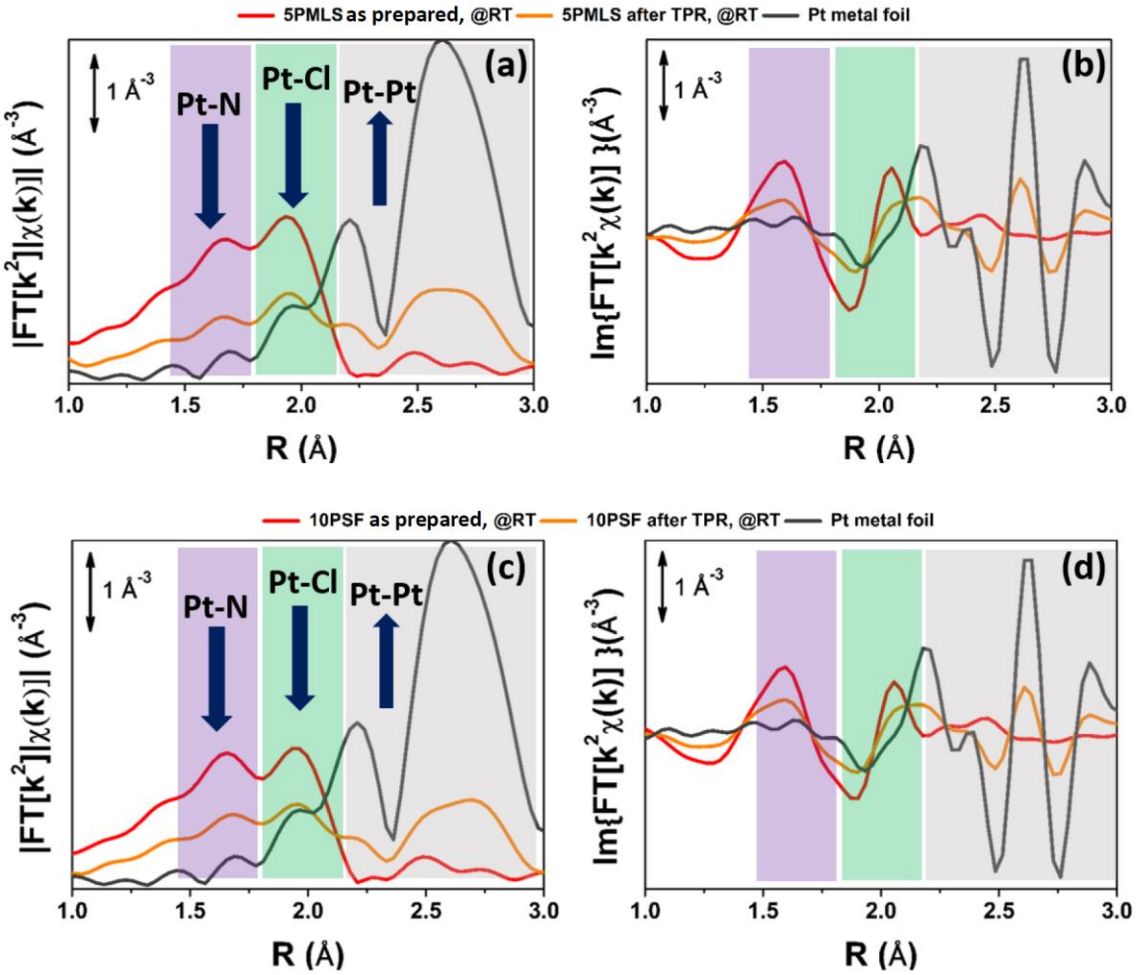

Figure S4. k^2-weighted, phase uncorrected, modulus (a), (c) and imaginary part (b), (d) of the experimental FT EXAFS spectra for 5PMLS and 10PSF in the as-prepared state at RT in air (red) and after TPR up to 623 K in 10% H_2/He and subsequent cooling to RT (orange). The FT EXAFS reference spectra of the Pt-metal foil are also reported (grey) for comparison.

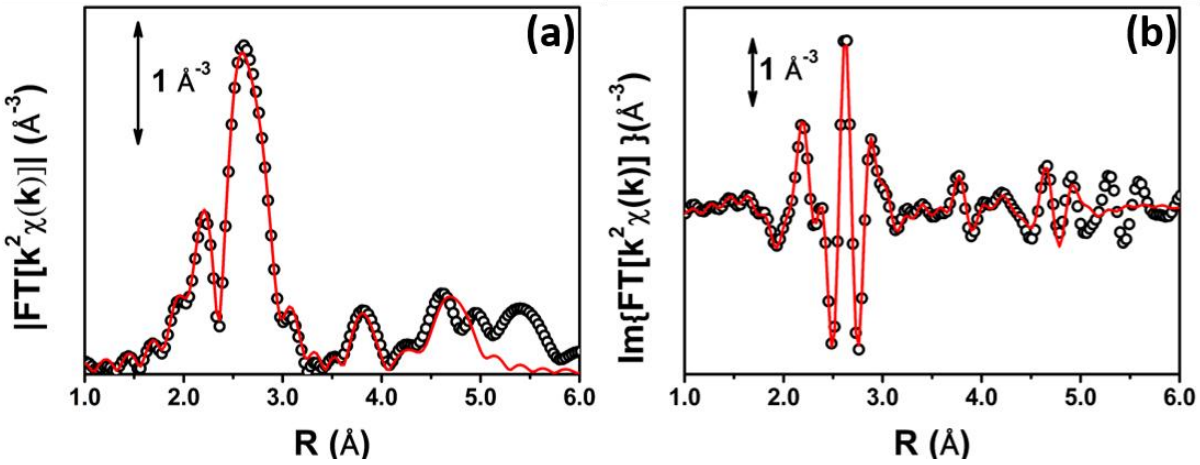

Figure S5. k^2-weighted, phase uncorrected, modulus (a) and imaginary part (b) of the experimental FT EXAFS spectra for Pt metal foil at RT. The experimental data are shown as black dots and the best fits with red solid lines.

Table S1. Best-fit values of the parameters optimized in the EXAFS fits for the Pt metal foil. The fit were performed in the k-space interval Δk = (3.2–17.0) $Å^{-1}$and R-space interval ΔR = (1.0–4.8) Å.

Sample	$R_{Pt\text{-}Pt}$ (Å)	$\sigma^2_{Pt\text{-}Pt}$ ($Å^2$)	S_0^2	ΔE (eV)	σ^2_{iso}($Å^2$)	α_{Pt}
Metal Pt foil	2.765±0.001	0.0047±0.0001	0.83±0.03	–2.7±0.4	0.0029±0.0002	0.001

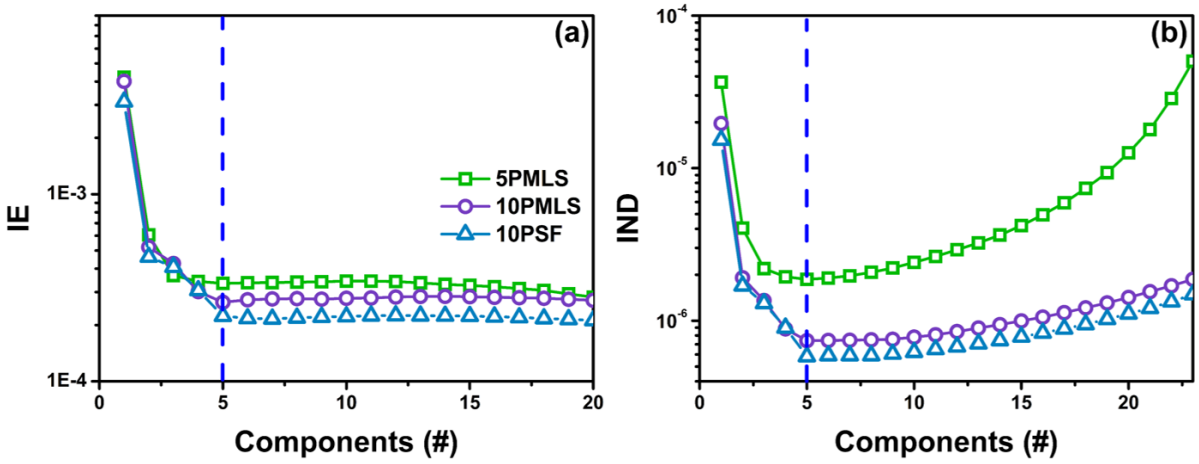

Figure S6. Imbedded error **(a)** and Malinowski indicator factor **(b)** in function of the number of components for the 5PMLS (green), 10PMLS (violet) and 10PSF (cyan) samples.

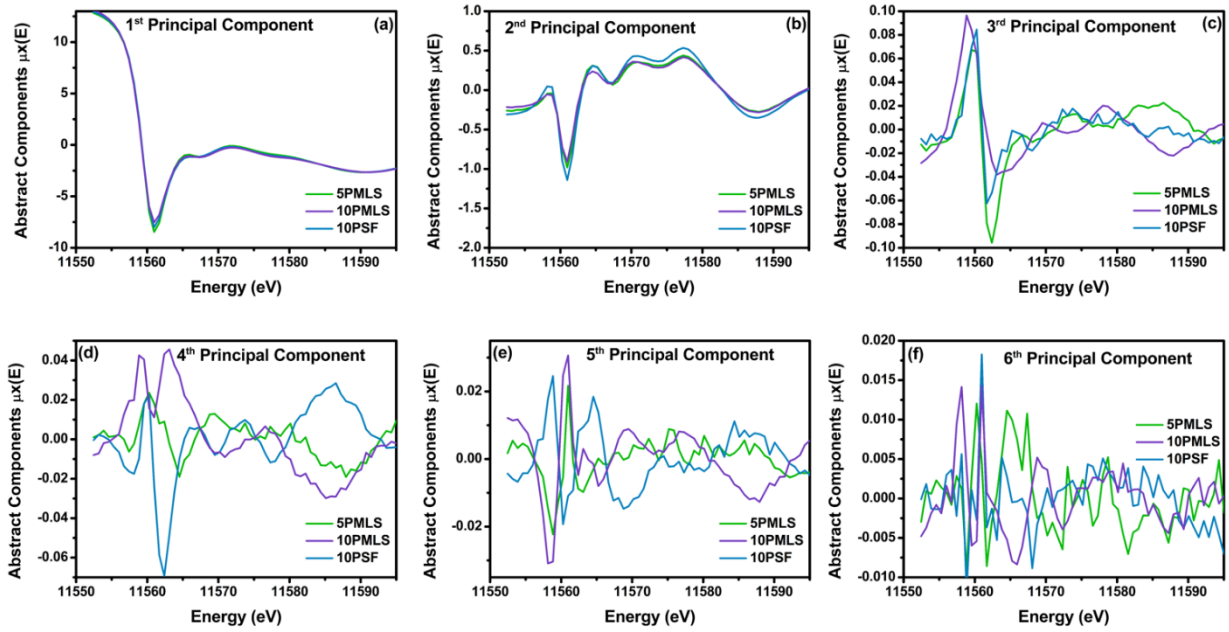

Figure S7. From part (a) to (f) follows the numerical order of the first six principal abstract components in function of the energy for the 5PMLS (green), 10PMLS (violet) and 10PSF (cyan) samples.

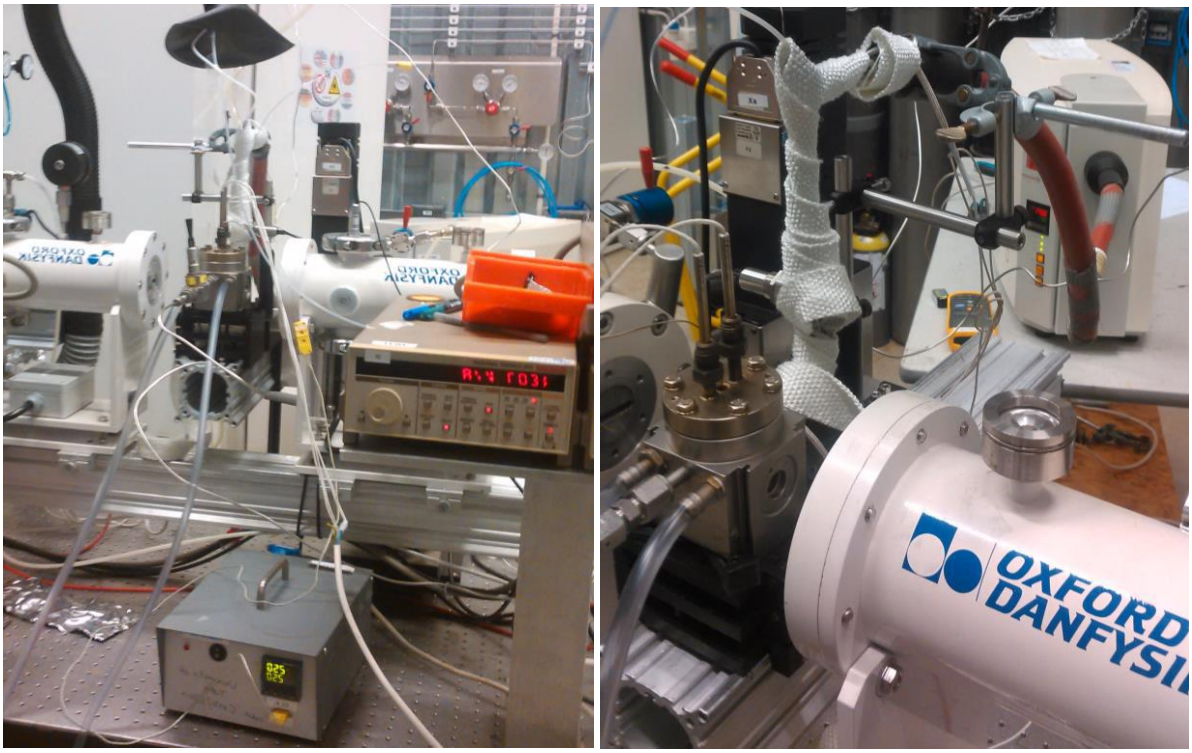

Figure S8. Images of the set up mounted at I811 beamline in Max Lab II (Lund, Sweden) to perform *operando* XAS experiments in transmission mode.