ALBA SYNCHROTRON

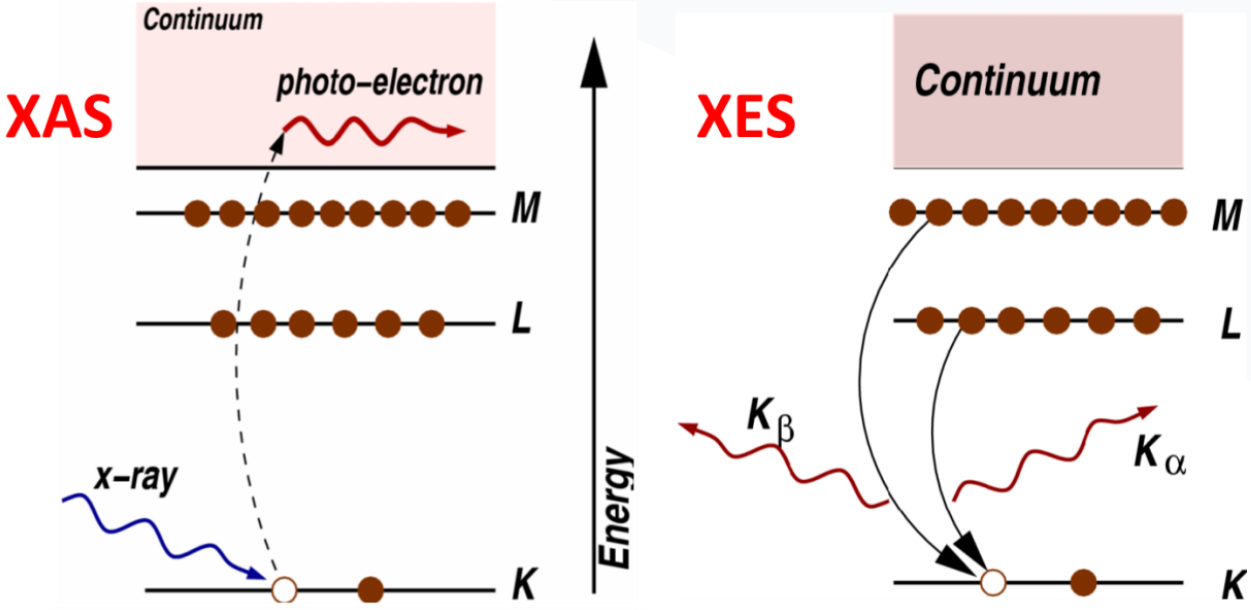
ALBA-ICMAB workshop, Alba synchrotron February 24th 2020

Structure and speciation with Hard X-ray spectroscopy in heterogeneous materials (including operando and in-situ)

Laura Simonelli
CLÆSS beamline responsible

BL22 – CLÆSS

Core **L**evel **A**bsorption & **E**mission **S**pectroscopie**s**



COMPLEMENTARY INFORMATION

BL22 – CLÆSS

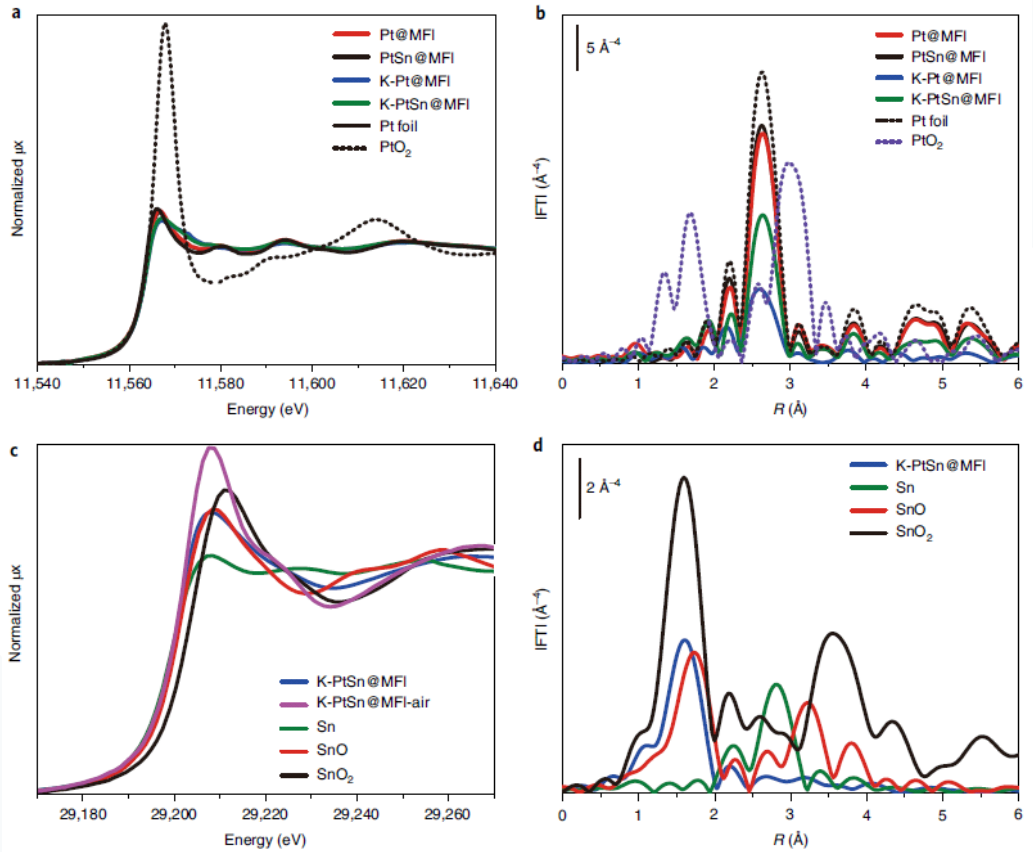
Core **L**evel **A**bsorption & **E**mission **S**pectroscopie**s**

XAS and XES are element sensitive local probe that can be applied to any kind of materials

- Speciation of individual or coexisting phases
- Local structural properties
- Local electronic properties

COMPLEMENTARY INFORMATION

Hard X-rays → in-situ / operando



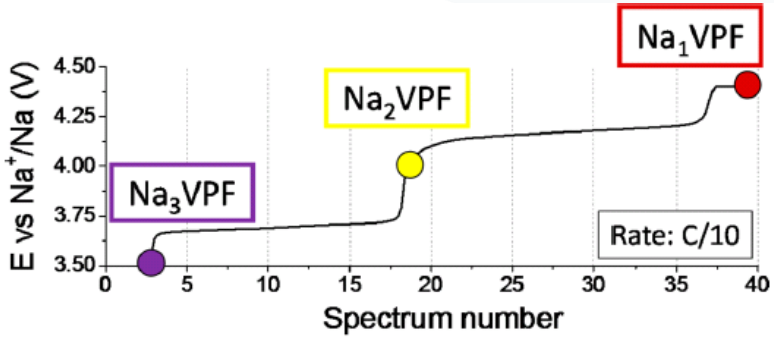
"Regioselective generation and reactivity control of subnanometric platinum clusters in zeolites for high-temperature catalysis"

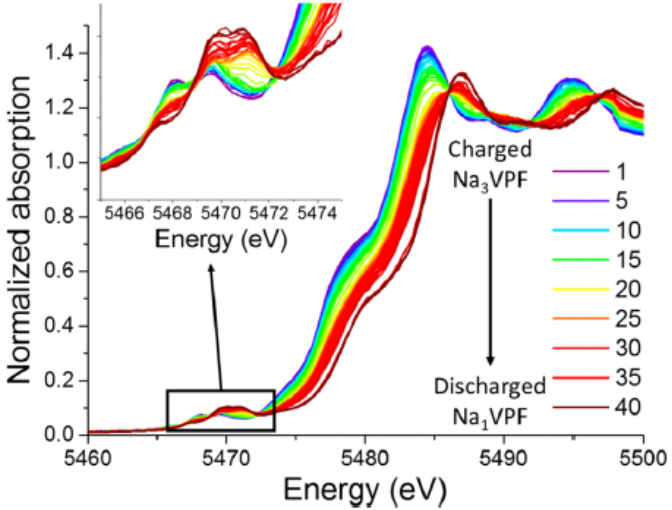
Lichen Liu, Miguel Lopez-Haro, Christian W. Lopes, Chengeng Li, Patricia Concepcion, Laura Simonelli, Jose J. Calvino and Avelino Corma

Nature Materials 18, 866–873 (2019)

Subnanometric clusters size and speciation by XAS

Hard X-rays → in-situ / operando

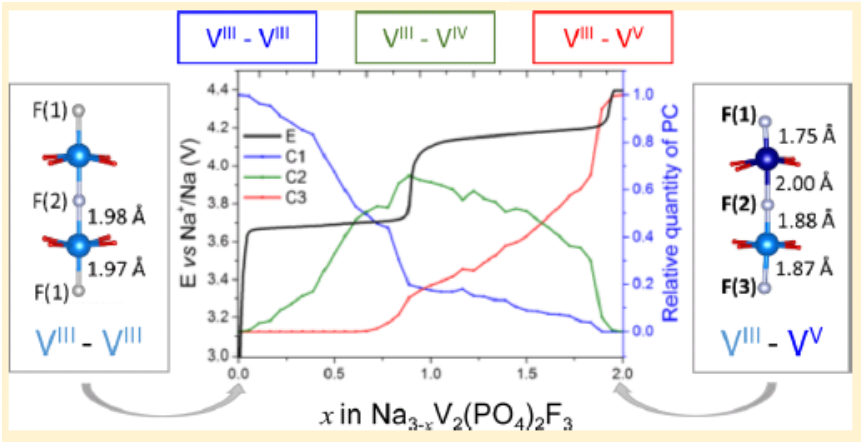




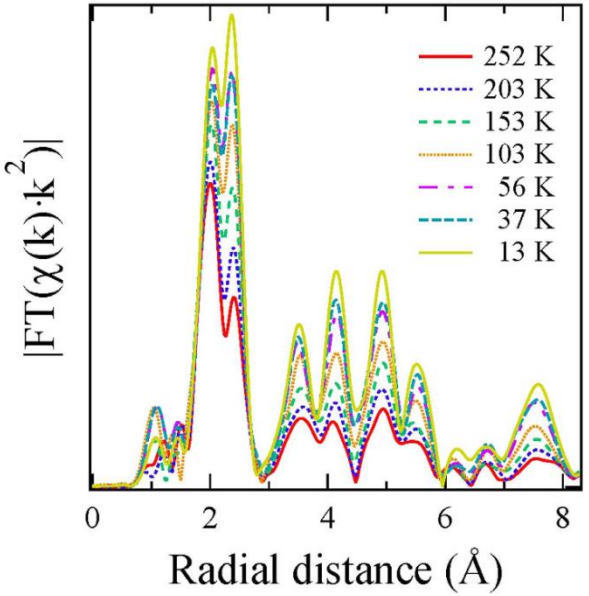
"VIV Disproportionation Upon Sodium Extraction From Na3V2(PO4)2F3 Observed by Operando X-ray Absorption Spectroscopy and Solid-State NMR"

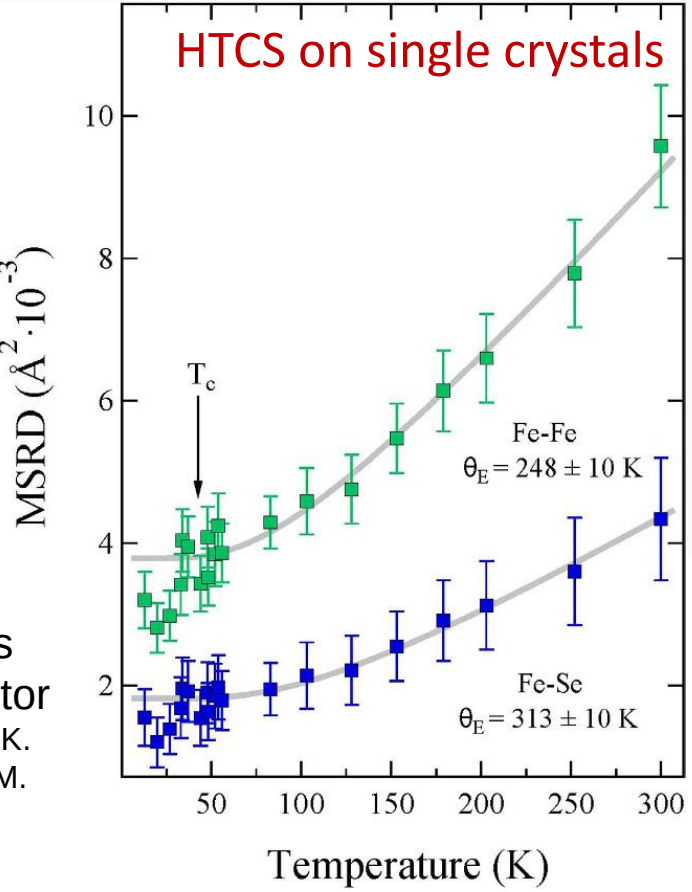
Thibault Broux, Tahya Bamine, Laura Simonelli, Lorenzo Stievano, François Fauth, Michel Ménétrier, Dany Carlier, Christian Masquelier and Laurence Croguennec, J. Phys. Chem. C 2017, 121, 4103−4111

Speciation along charge by PCA



EXAFS(T) → dynamics

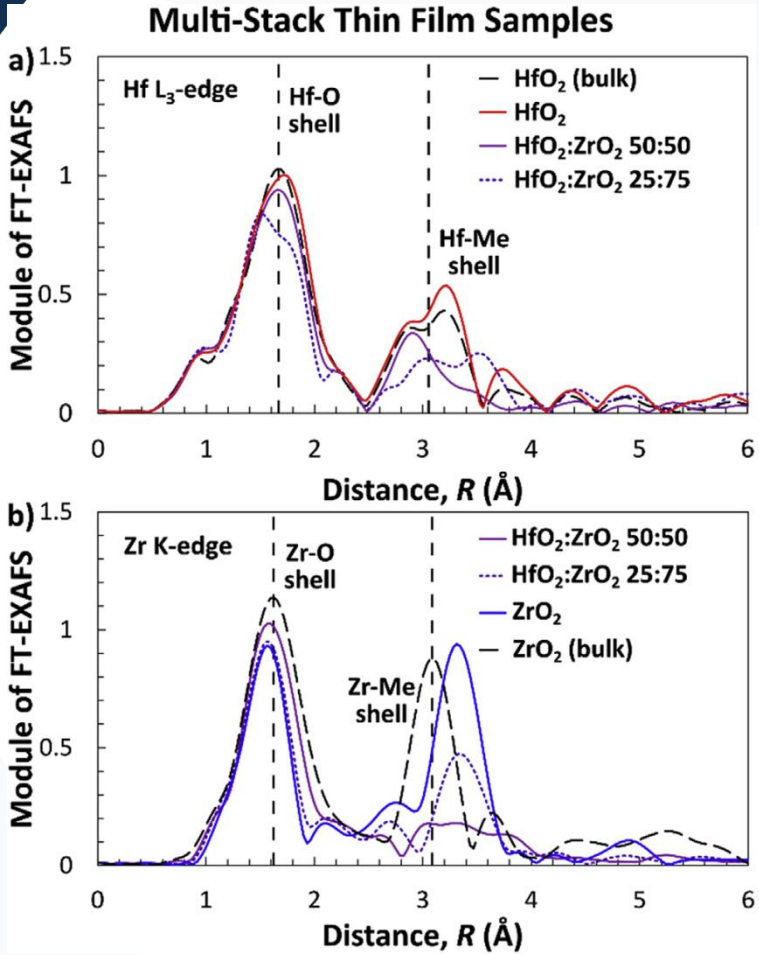




Temperature dependent local atomic displacements in ammonia intercalated iron selenide superconductor

E. Paris, L. Simonelli, T. Wakita, C. Marini, J.-H. Lee, W. Olszewski, K. Terashima, T. Kakuto, N. Nishimoto, T. Kimura, K. Kudo, T. Kambe, M. Nohara, T. Yokoya & N. L. Saini, Scientific Reports 6, 27646 (2016)

Ferro- and antiferroelectric properties on 20nm thin films



EXAFS → second coordination shell to discriminate different polymorphs.

Local structural investigation of hafnia-zirconia polymorphs in powders and thin films by X-ray absorption spectroscopy

Tony Schenk, Andris Anspoks, Inga Jonane, Reinis Ignatans, Brienne S. Johnson, Jacob L. Jones, Massimo Tallarida, Carlo Marini, Laura Simonelli, Philipp H€onicke, Claudia Richter, Thomas Mikolajick, Uwe Schroeder

Acta Materialia 180 (2019) 158e169

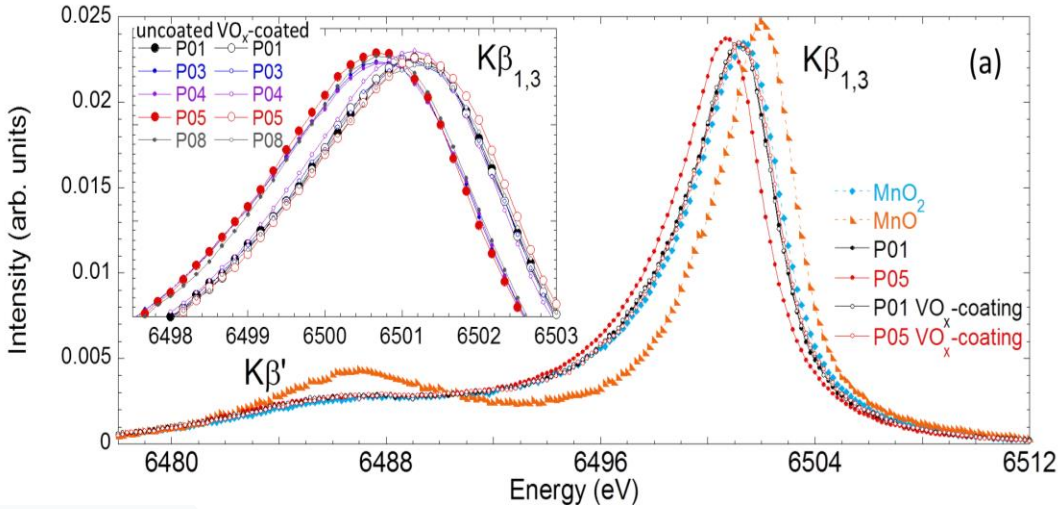
XES complementary to XAS

Role of Manganese in Lithium- and Manganese-Rich Layered Oxides Cathodes

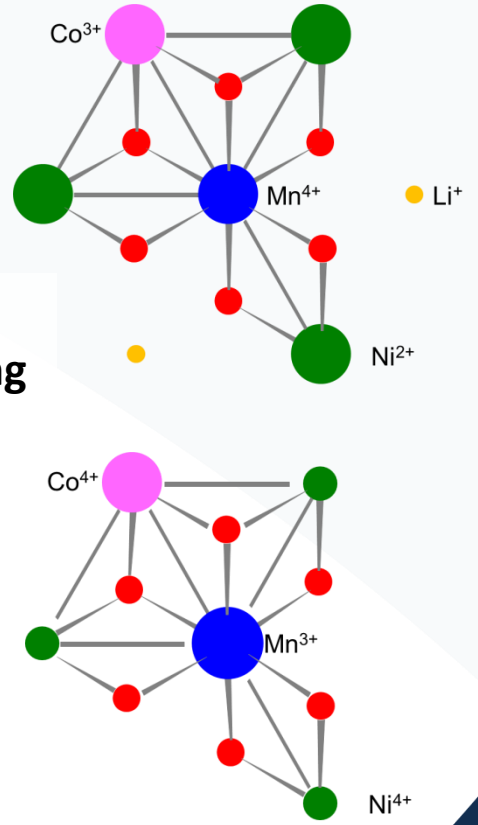
L. Simonelli, A. Sorrentino, C. Marini, N. Ramanan, D. Heinis, W. Olszewski, A. Mullaliu, A. Birrozzi, N. Laszczynski, M. Giorgetti, S. Passerini, D. Tonti

J. Phys. Chem. Lett. 2019, 10, 12, 3359-3368

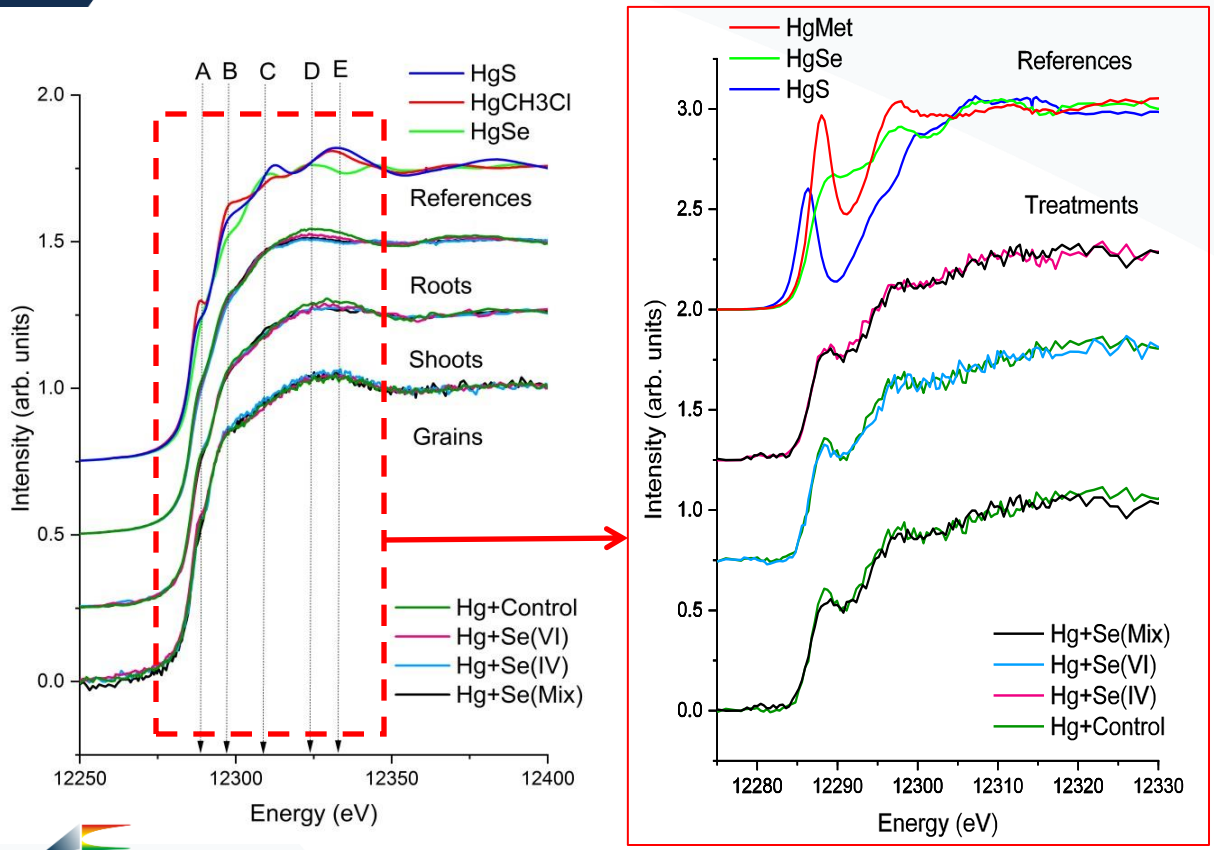
TM size depending on both oxidation and spin state



By charging



HR XANES



Mercury speciation in Selenium enriched wheat plants grown in Hg contaminated environment

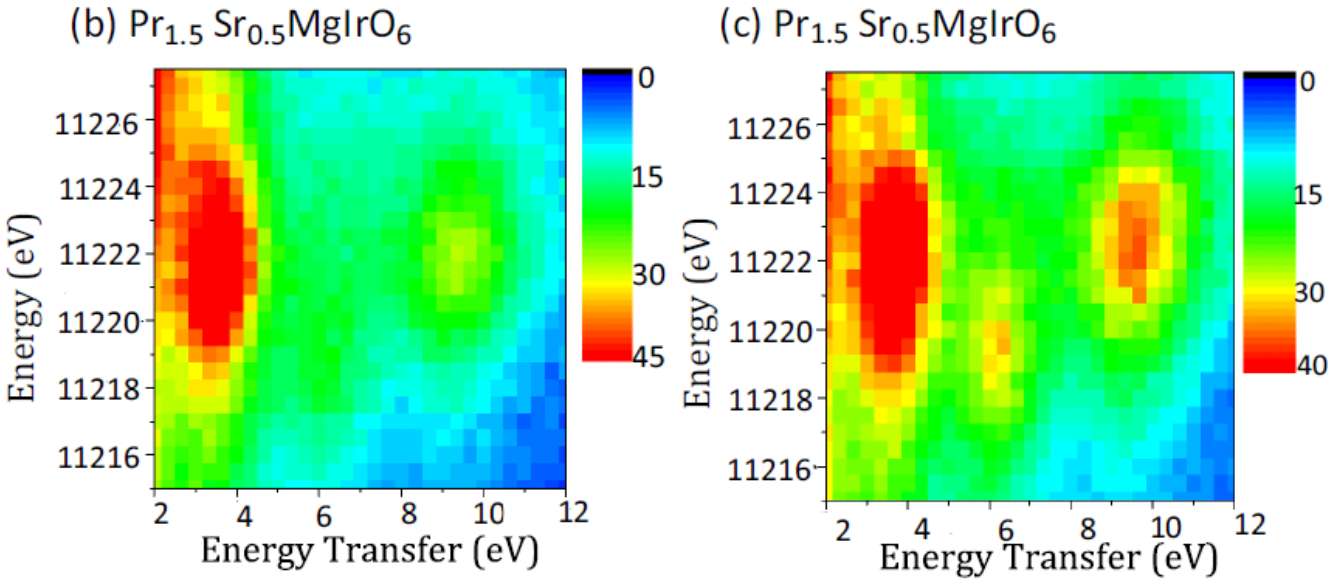
N. Manivannan, R. Boada, M. A. Subirana, C. Marini, M. Llugany, M. Valiente, L. Simonelli

In preparation

HR XANES accessible also in diluted systems (few hundreds of ppm)

Local electronic structure

L_3-edge RIXS easily accessible



Evolution of electronic and magnetic properties in a series of iridate double perovskites $Pr_{2-x}Sr_xMgIrO_6$ (x=0, 0.5, 1.0)

A. Bandyopadhyay, I. Carlomagno, L. Simonelli, M. Moretti Sala, A. Efimenko, C. Meneghini, and S. Ray

PRB 100, 064416 (2019)

Absorption energy range

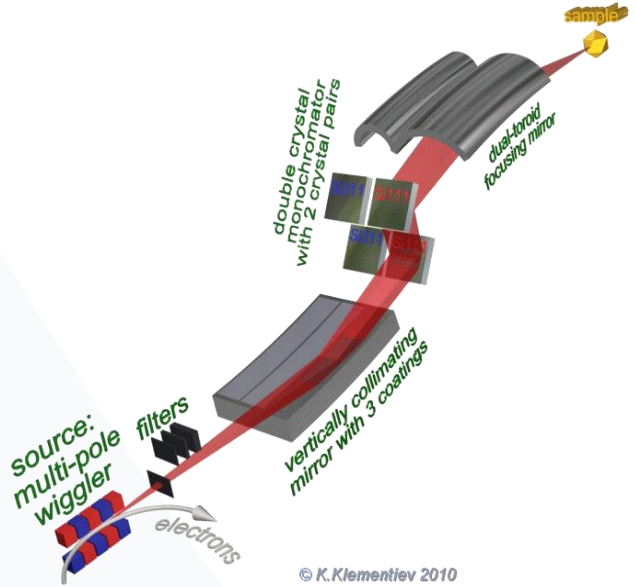
(2.4 – 63 KeV)

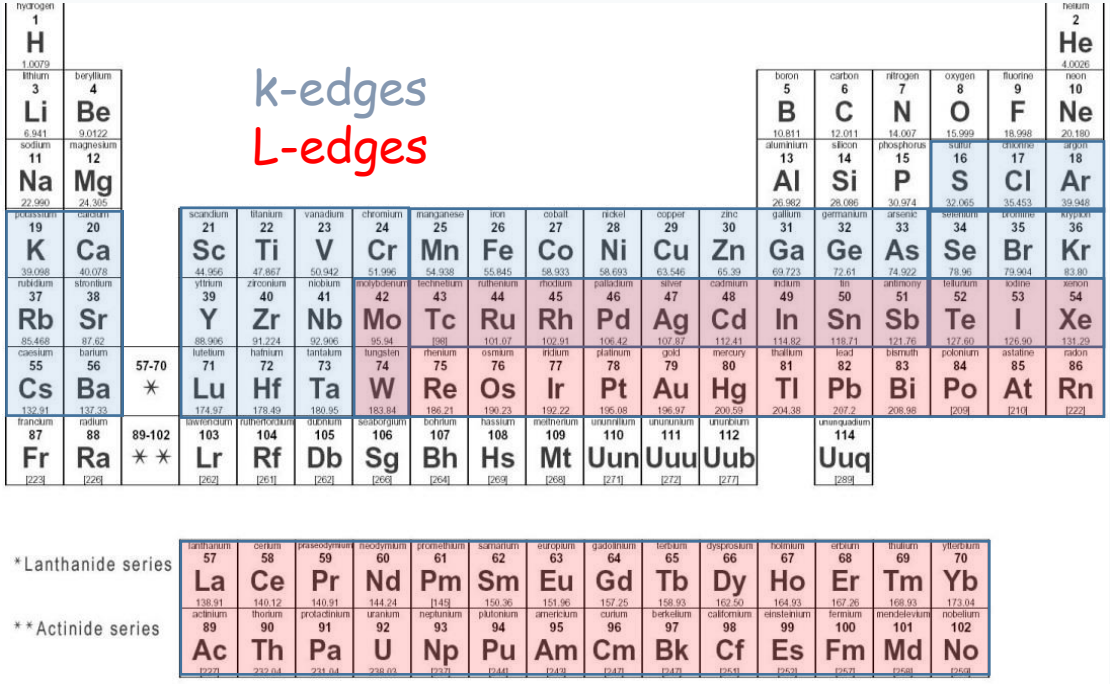
Emission energy range

(6.2 – 22 KeV)

Beam size

From 200x100 mm^2 (HxV) to 8x1 mm (HxV)





Scan speed

XAS : 1 (15) min per EXAFS and 30 s (2 min) per XANES scan in transmission (fluorescence) mode.

XES: 15 (45) min per emission line or high resolution XANES. 4-6h per RIXS map.

Sample setups

(XAS in fluorescence and transmition modes and XES)

Dynamic liquid cells (RT)

Solid-gas reactor

20-700˚C

up to 10bar

GAS mix unit with 6 lines gases at 5-50 (10-1000) ml/min

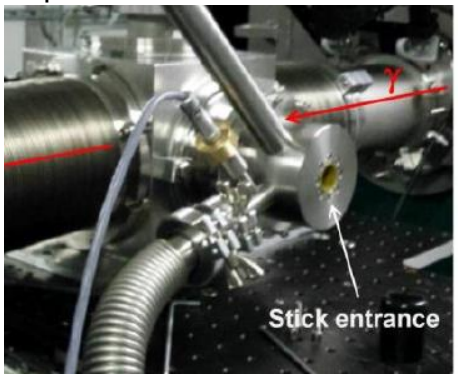
Mass spectrometer

LHe-cryo

5 K – 320 K

Low temperature sample exchange

Liquid cells



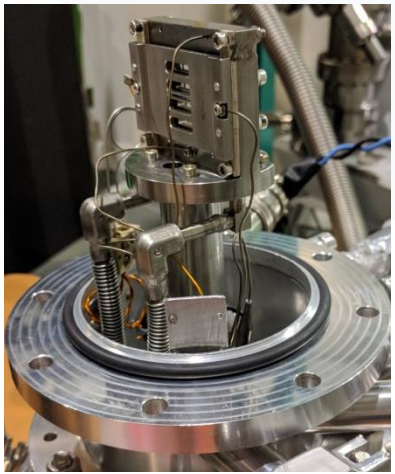
LN_2-cryo/furnace

80-830K

TEY

RT sample exchange

Static liquid cells



THANK YOU FOR YOUR ATTENTION