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Combined XRR-GIXRF analysis at LNHB: technical and analytical developments

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- The **XRR** ("X-Ray Reflectivity") and **GIXRF** ("Grazing Incidence X-Ray Fluorescence") techniques make it possible to characterize the properties at the interfaces and in depth of materials deposited in very thin layers (thickness, roughness, composition, densities), CASTOR is a goniometer dedicated to these techniques.
- At LNE-LNHB we are developing the combined XRR-GIXRF analysis in a **reference-free** mode using calibrated instrumentation (SDD, photodiodes) and a user-friendly software dedicated to the complete analysis.

CASTOR goniometer

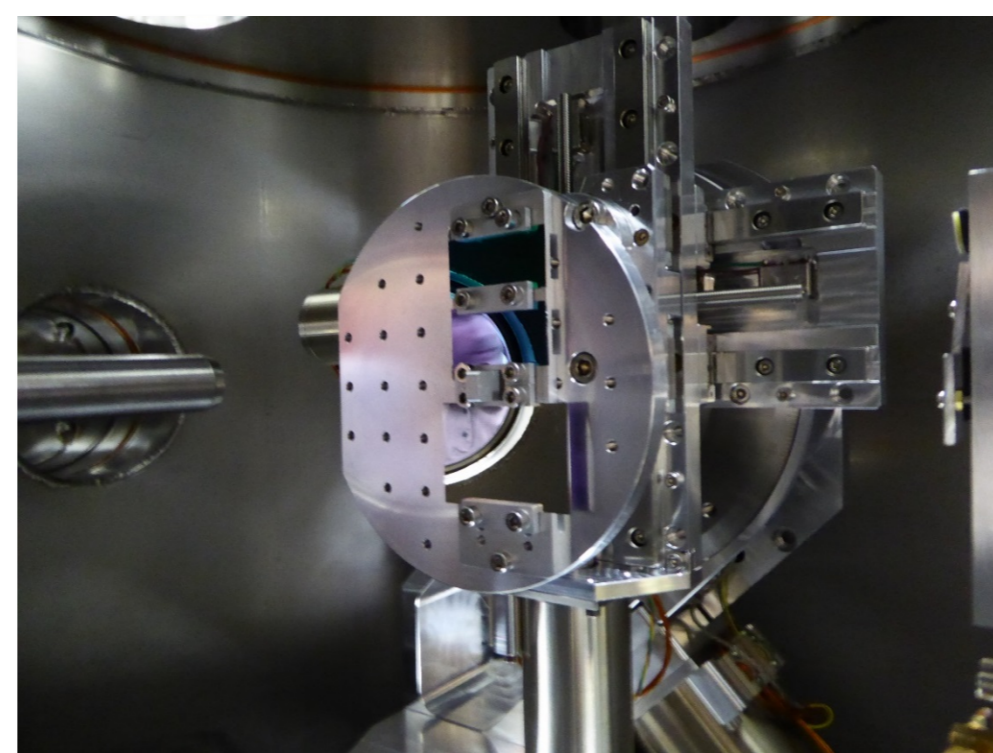
The **METROLOGIE** beamline (bending magnet) at SOLEIL has 2 branches equipped with monochromators:

- from 45 eV to 1,9 keV → "XUV", 3 VLS gratings
- from 3 keV to 35 keV → "XDURS", 2 Si(111) crystals
- Several filters and adjustable slits

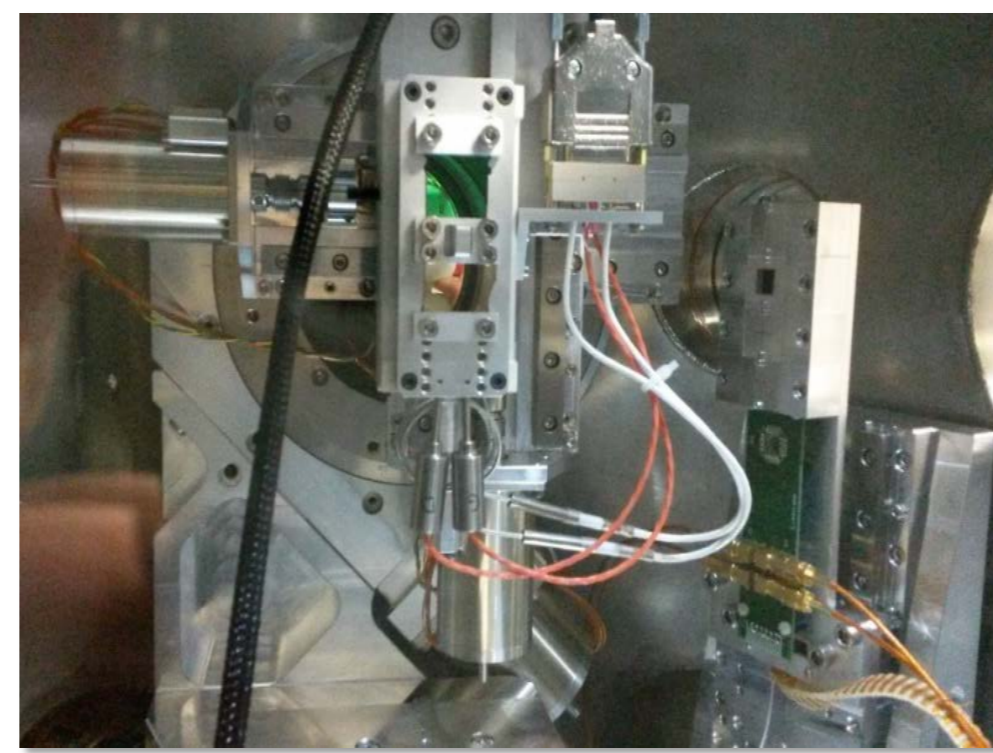
CASTOR ("Chambre d'Analyse Spectrométrique en Transmission Ou en Réflexion") :

- HV chamber (10⁻⁶ hPa)
- 7 axes manipulator (2 rotations and 3 translations for the sample & 1 rotation and 1 translation for the photodiode)
- 3 axes frame
- Up to 4 photodiodes
- Spectrometer (SDD) on a translation stage
- Heating sample holder (up to 300 °C)
- dedicated LabVIEW® acquisition program (variable scanning steps, results in ASCII format)

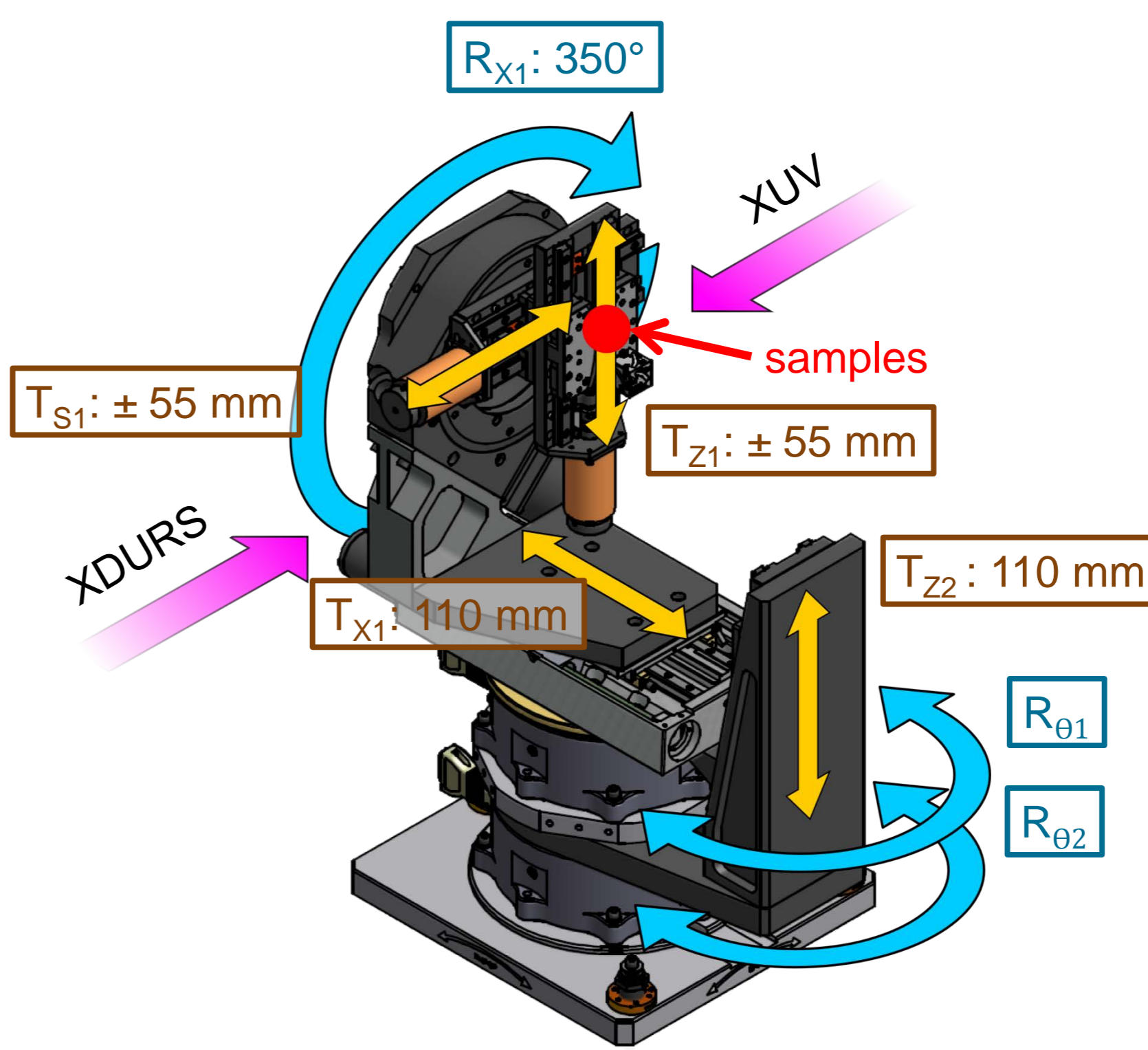
→ Ménesguen, Y. *et al.*, X-Ray Spectrometry, 46, 303-308 (2017)



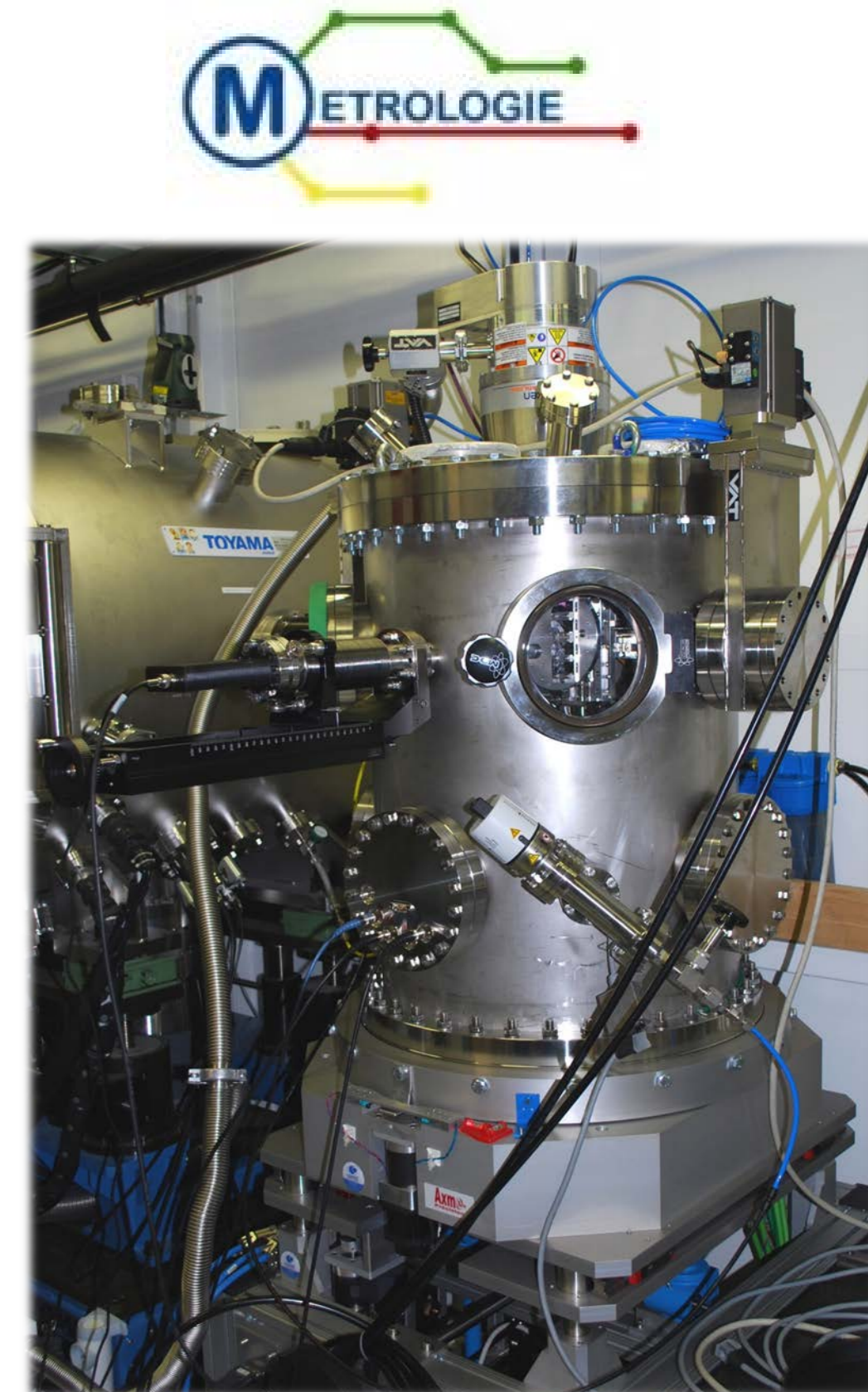
Sample holder



Heating sample holder

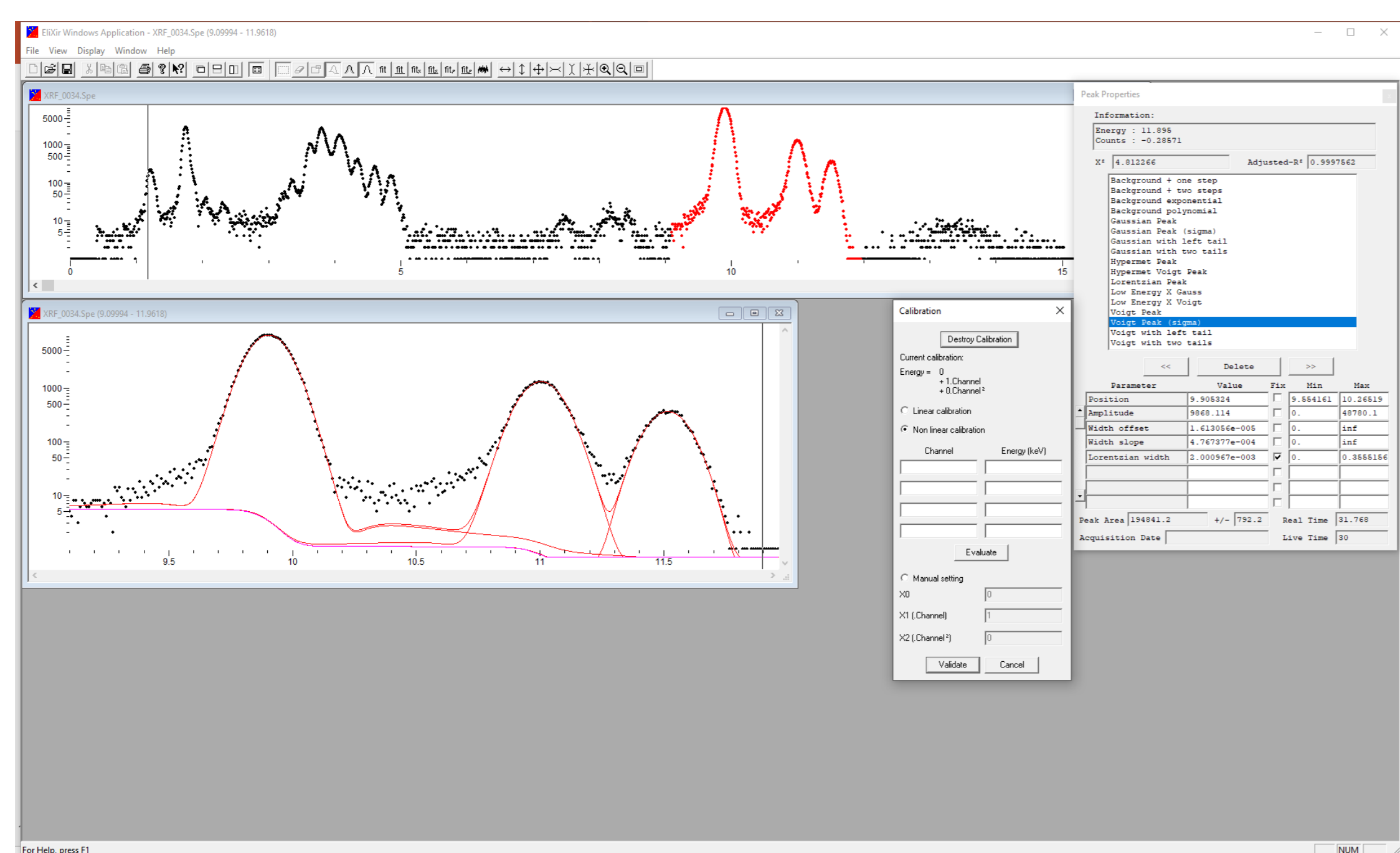


7 axes manipulator



CASTOR on XDURS

Reference-free combined XRR-GIXRF analysis with EliXir



Solid angle calculation for the XRF and the XRR

Layer definition: (thickness, roughness, density, composition)

Excitation energy (XRR, XRF), acquisition time and flux (XRF)

Optimization Parameters

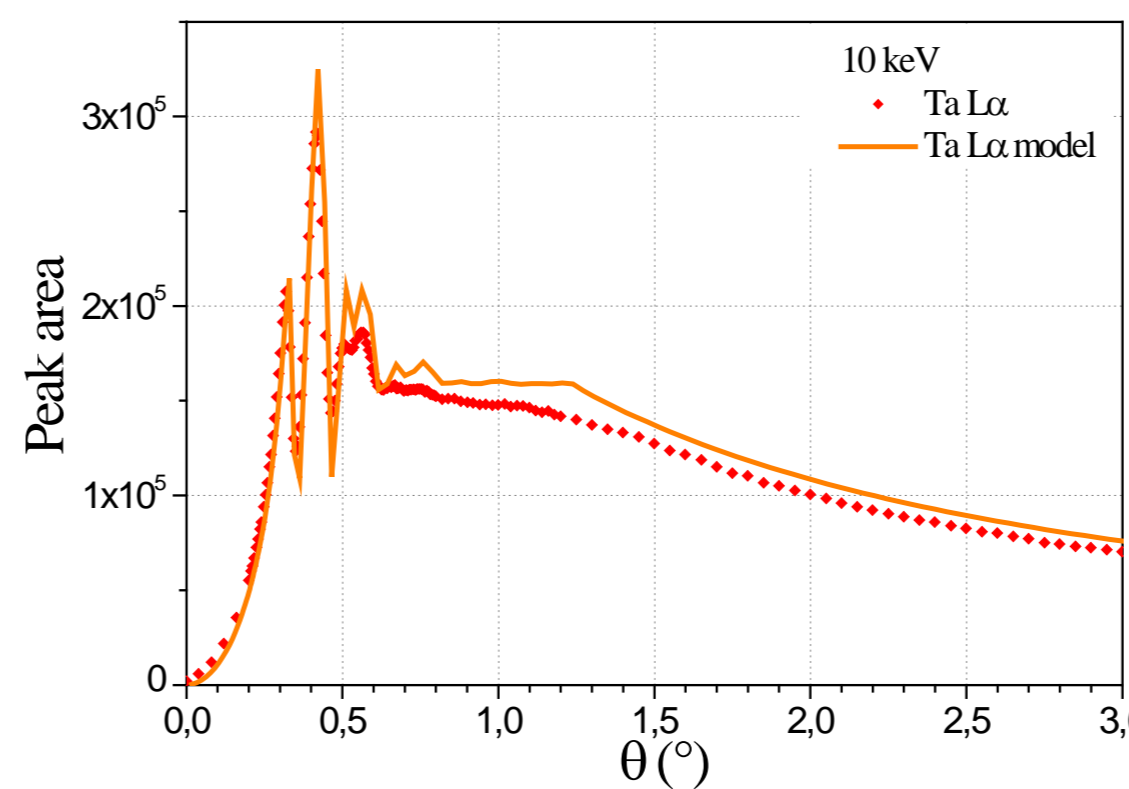
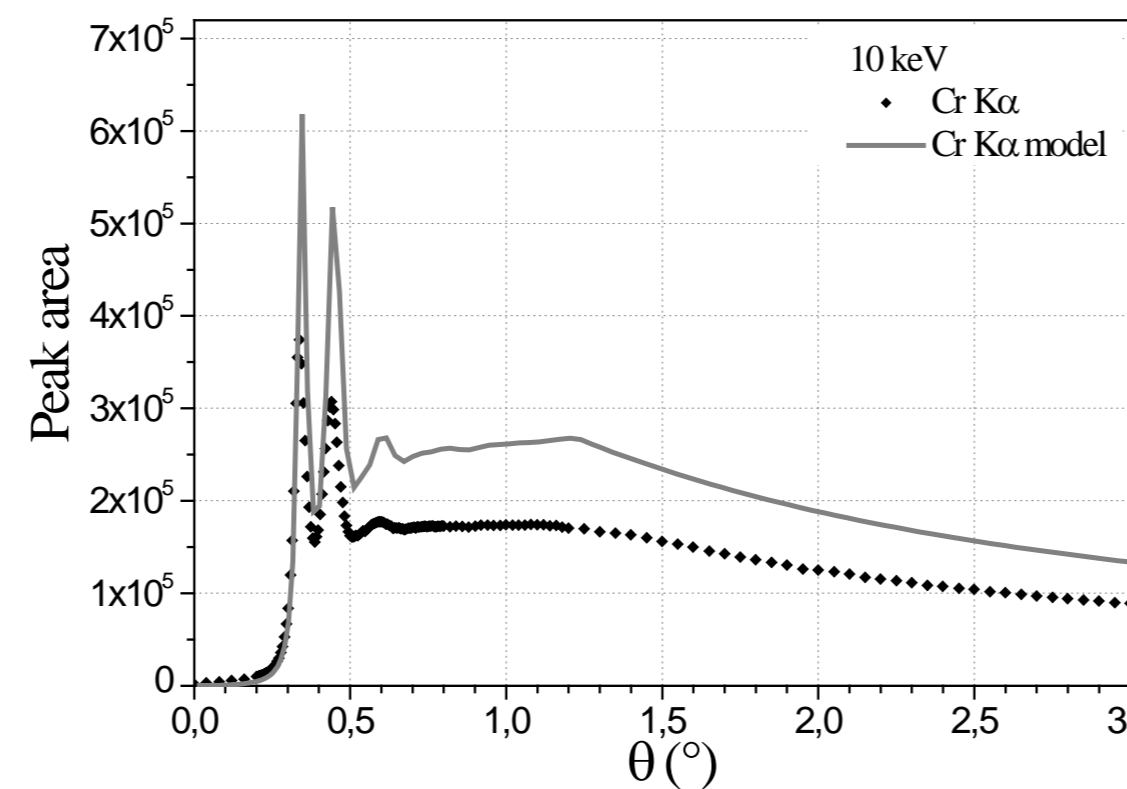
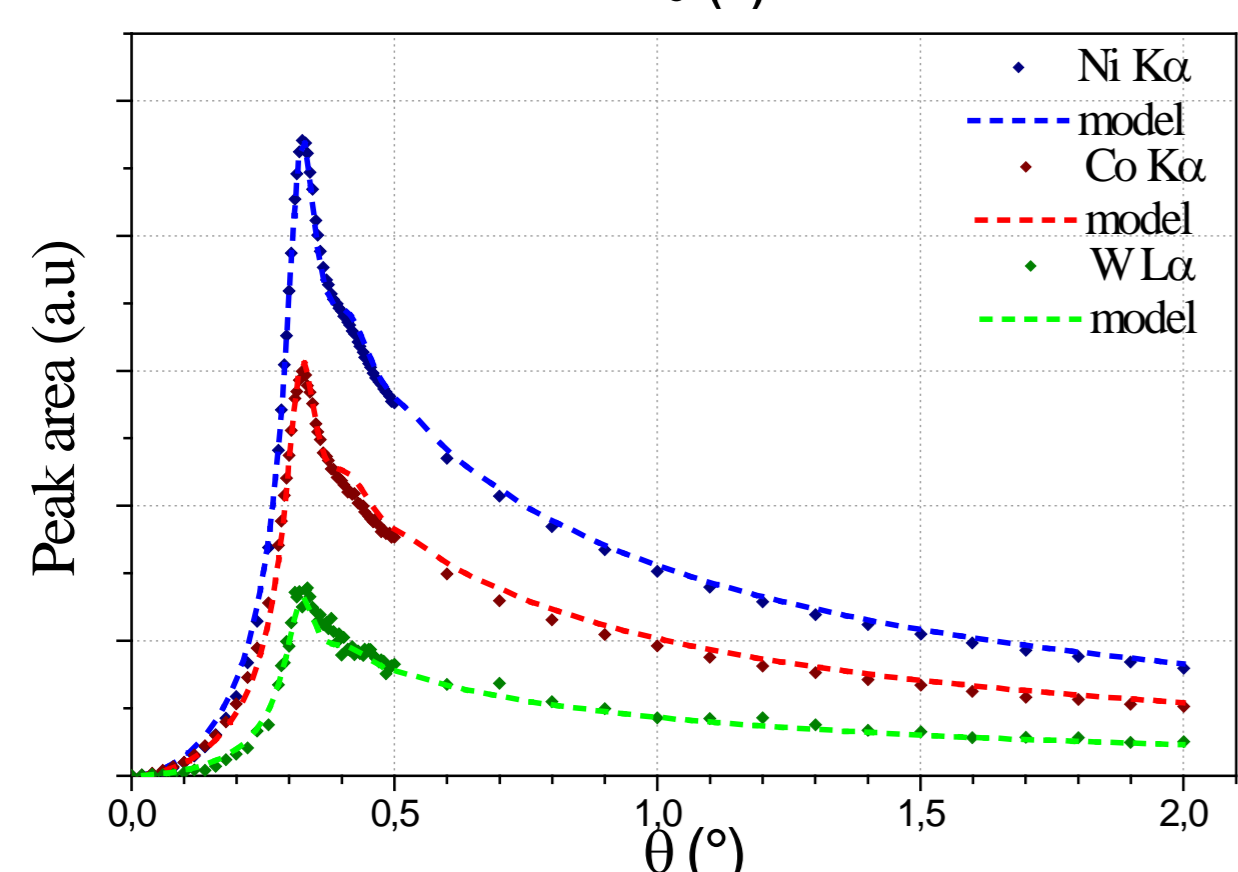
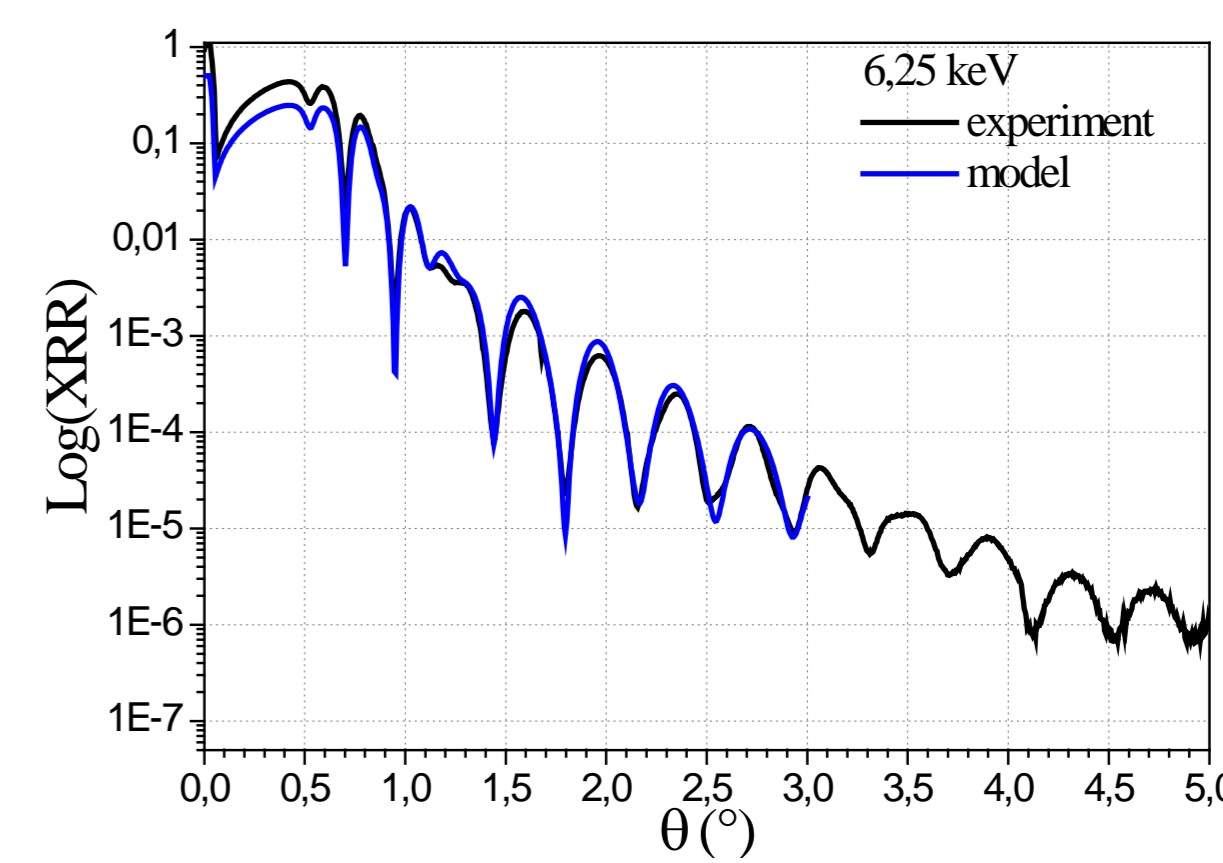
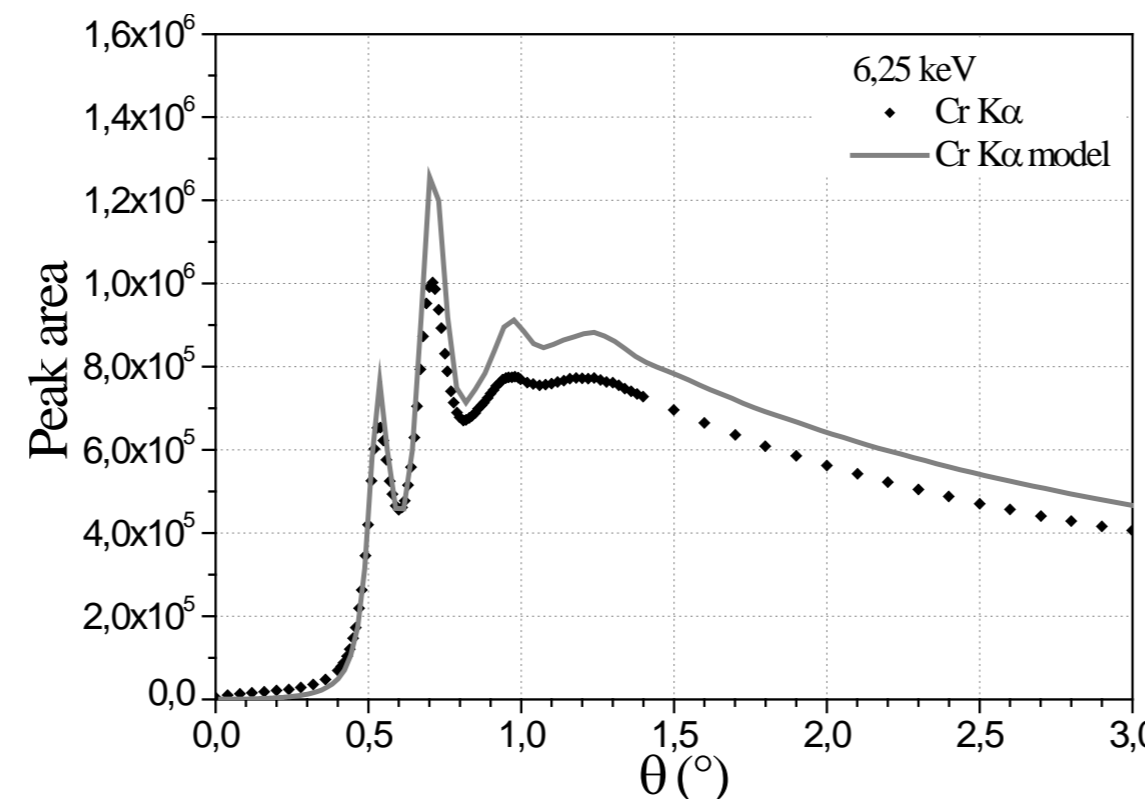
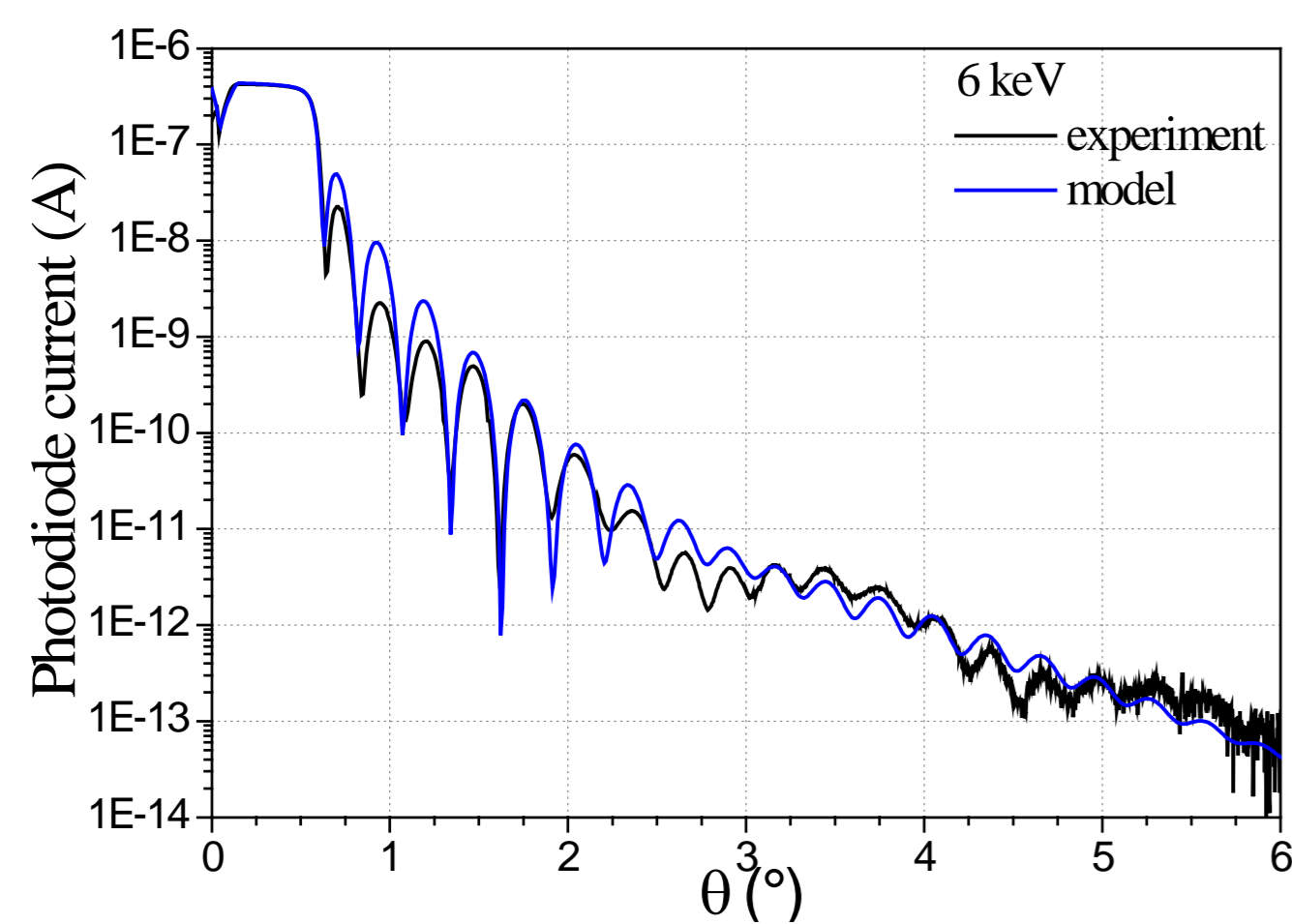
Optimization: XRR only, XRF only, both or none

Parameters constraints

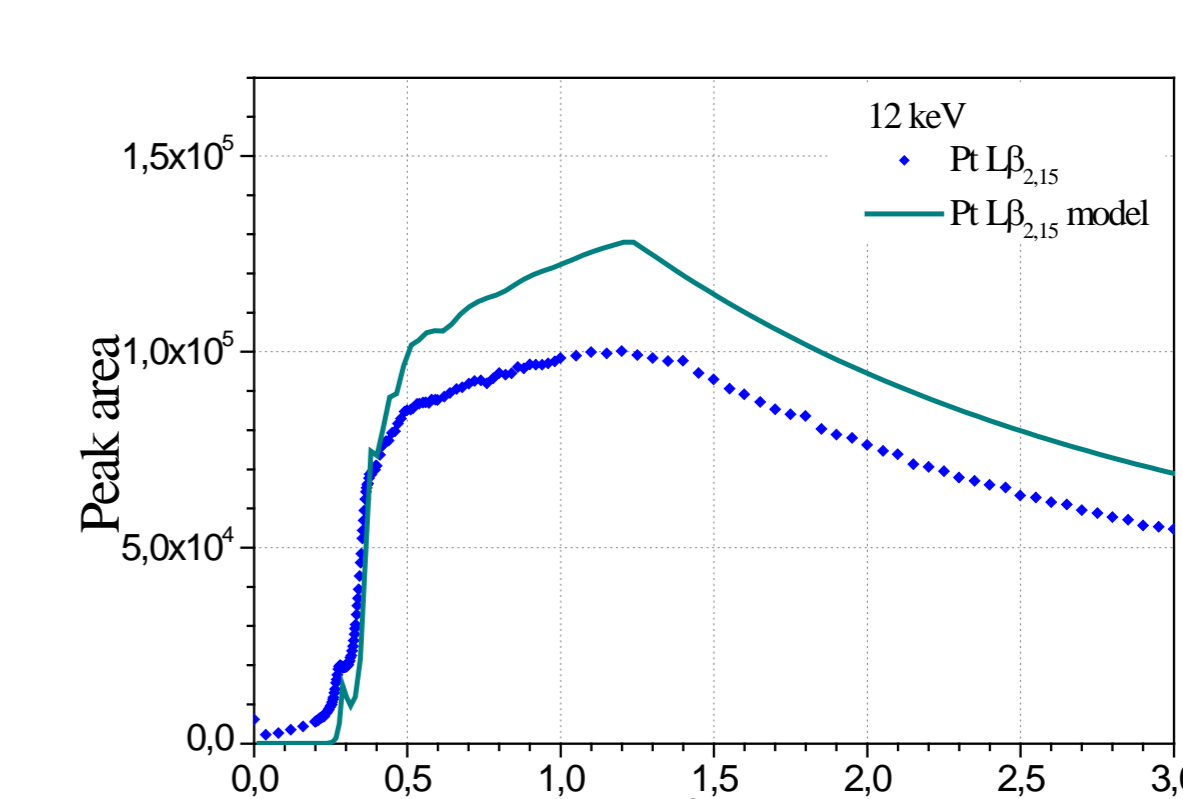
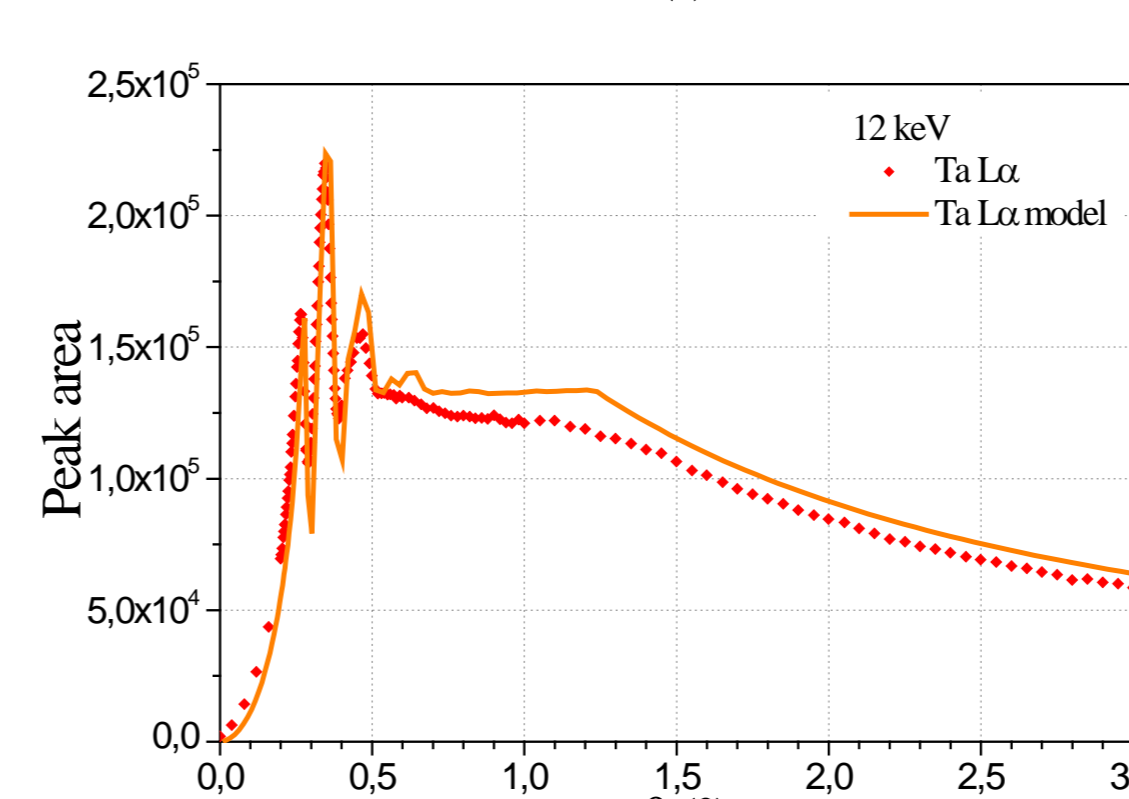
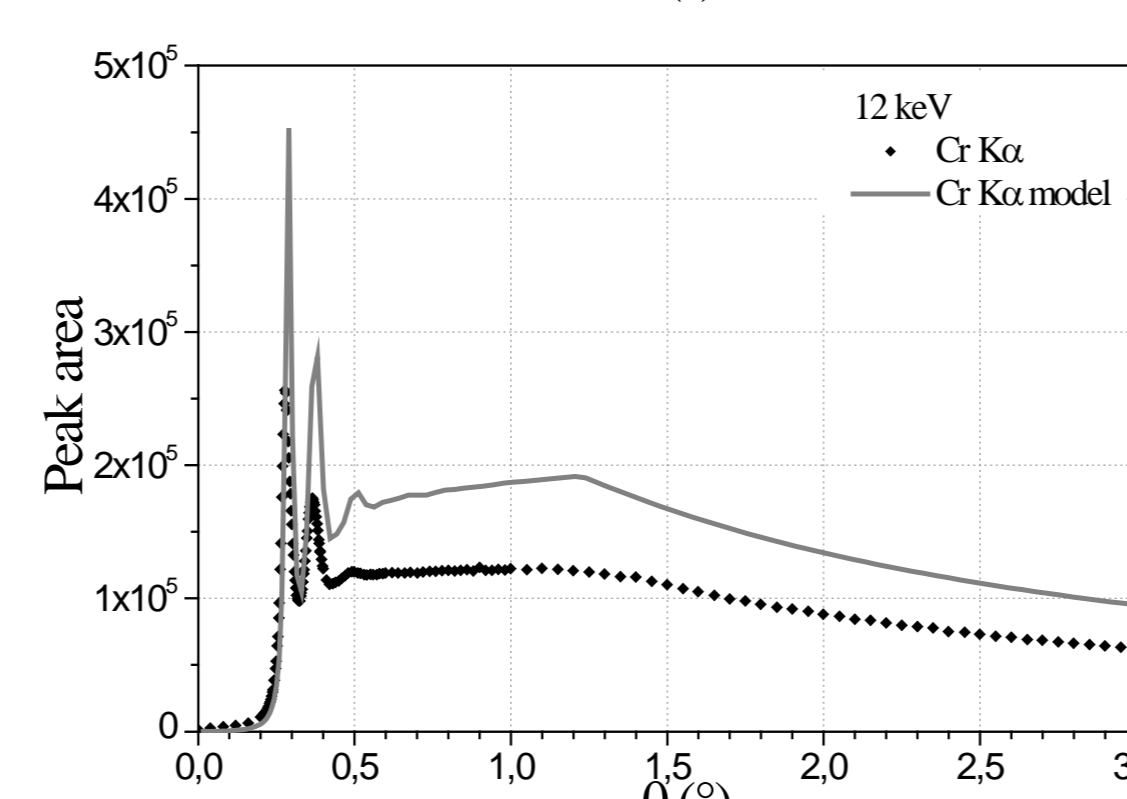
Elements to compute the GIXRF

- Same XRF spectra analysis as **COLEGRAM** (Ménesguen, Y. *et al.*, Nuclear Instruments & Methods In Physics Research A, 1003, 165341 (2021))
- Atomic fundamental parameters (μ , τ , ω) (Ménesguen, Y. *et al.*, Metrologia, 53, 7-17 (2016))
- Beam geometry (Ménesguen, Y. *et al.*, Physica Status Solidi A, 2100423 (2021))

Examples of results



Ta ₂ O ₅	d = 1,88 nm	$\rho = 6,16 \text{ g.cm}^{-3}$	$\sigma = 1,11 \text{ nm}$
Ta	d = 3,97 nm	$\rho = 16,14 \text{ g.cm}^{-3}$	$\sigma = 1,12 \text{ nm}$
Cr	d = 13,65 nm	$\rho = 7,00 \text{ g.cm}^{-3}$	$\sigma = 0,94 \text{ nm}$
Pt	d = 13,9 nm	$\rho = 21,5 \text{ g.cm}^{-3}$	$\sigma = 0,53 \text{ nm}$
PtSi	d = 0,19 nm	$\rho = 18,95 \text{ g.cm}^{-3}$	$\sigma = 0,75 \text{ nm}$
Si Substrate			



NiCo _{0,11} W _{0,01}	d = 1,02 nm	$\rho = 5,76 \text{ g.cm}^{-3}$	$\sigma = 0,53 \text{ nm}$
NiCo _{0,11} W _{0,01}	d = 17,52 nm	$\rho = 8,74 \text{ g.cm}^{-3}$	$\sigma = 0,47 \text{ nm}$
NiCo _{0,11} W _{0,01}	d = 1,15 nm	$\rho = 8,12 \text{ g.cm}^{-3}$	$\sigma = 0,47 \text{ nm}$
SiO ₂	d = 1,05 nm	$\rho = 2,65 \text{ g.cm}^{-3}$	$\sigma = 0,26 \text{ nm}$
Si Substrate			