

La odisea de medir PDF con un difractómetro “convencional”

TÉCNICOS DE LA UNIDAD: LDA. ESTEFANÍA QUINTERO MARTOS, DR. INÉS RUIZ SALCEDO Y DR. LAURA LEÓN REINA

RESPONSABLE CIENTÍFICO: DR. AURELIO CABEZA DÍAZ



UNIVERSIDAD
DE MÁLAGA

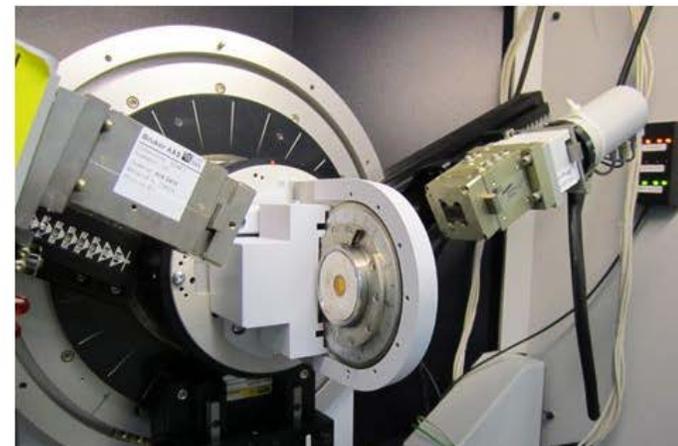


Un laboratorio para todos y para todo



ANÁLISIS CUANTITATIVO DE FASES

Con ANODO DE Cu.....poca muestra



Con EQUIPOS THETA-2 THETA.....muestra volcada

research papers



Appl Cryst
JAC
JOURNAL OF
APPLIED
CRYSTALLOGRAPHY

ISSN 1600-5767

Accuracy in Rietveld quantitative phase analysis: a comparative study of strictly monochromatic Mo and Cu radiations

L. León-Reina,^a M. García-Maté,^{b,c} G. Álvarez-Pinazo,^{b,c} I. Santacruz,^b O. Vallcorba,^d A. G. De la Torre^b and M. A. G. Aranda^{b,d*}



UNIVERSIDAD
DE MÁLAGA



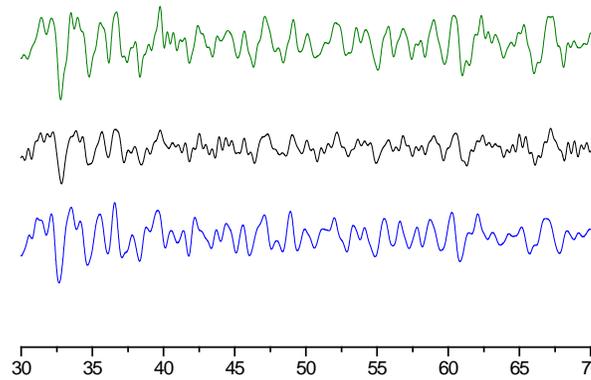
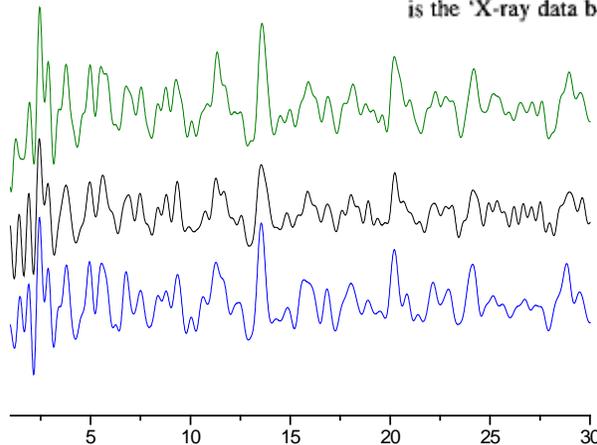
Aprovecho mi difractor de Mo para medir PDF



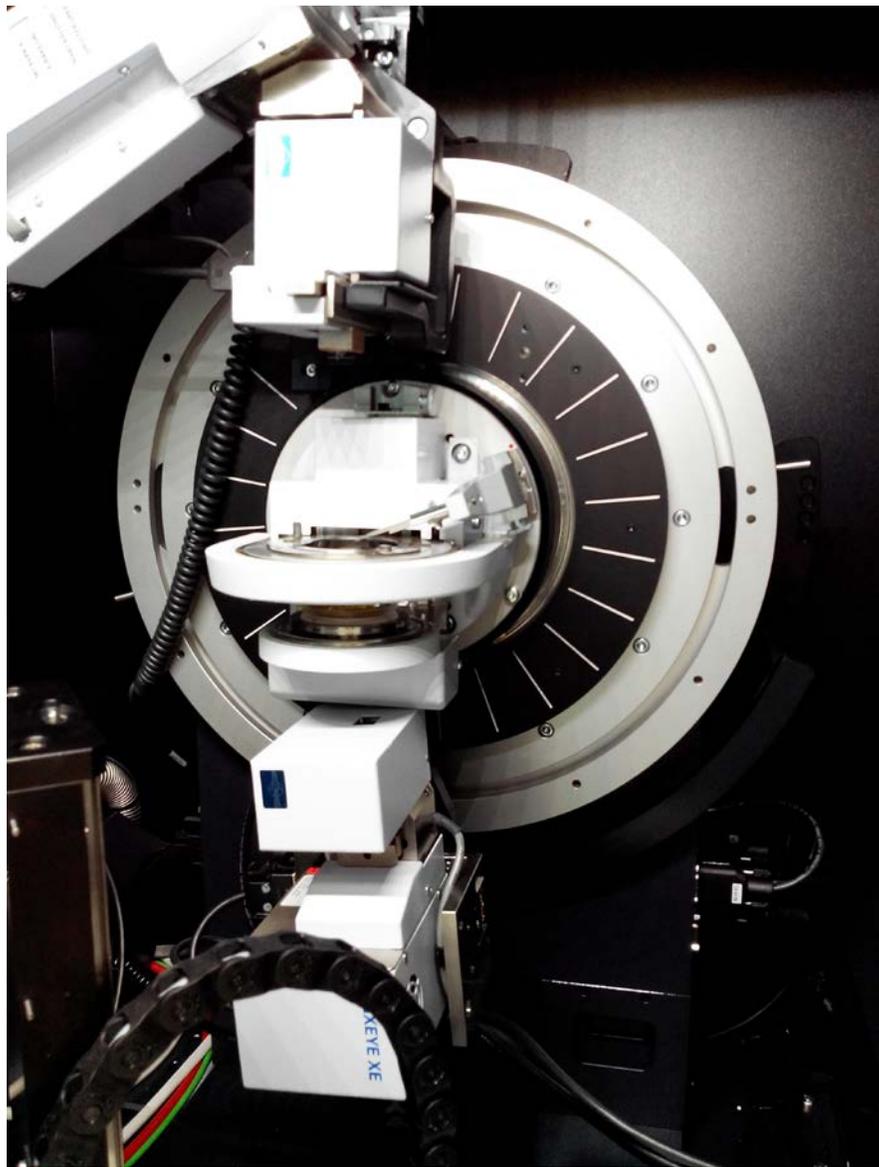
Table 4.2. X-ray energies, wavelengths and approximate Q_{\max} values for common laboratory X-ray sources.

Source	E_0 (keV)	λ (Å)	Q_{\max} (Å ⁻¹)
Cu	8.05	1.538	8.0
Mo	17.48	0.708	17.5
Ag	22.16	0.559	22.0
W	59.32	0.209	59.0

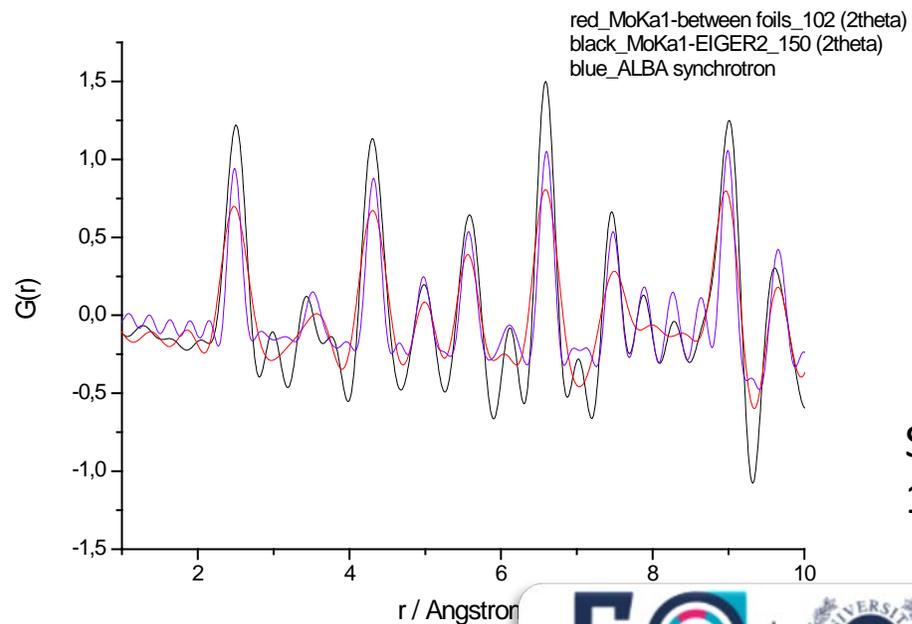
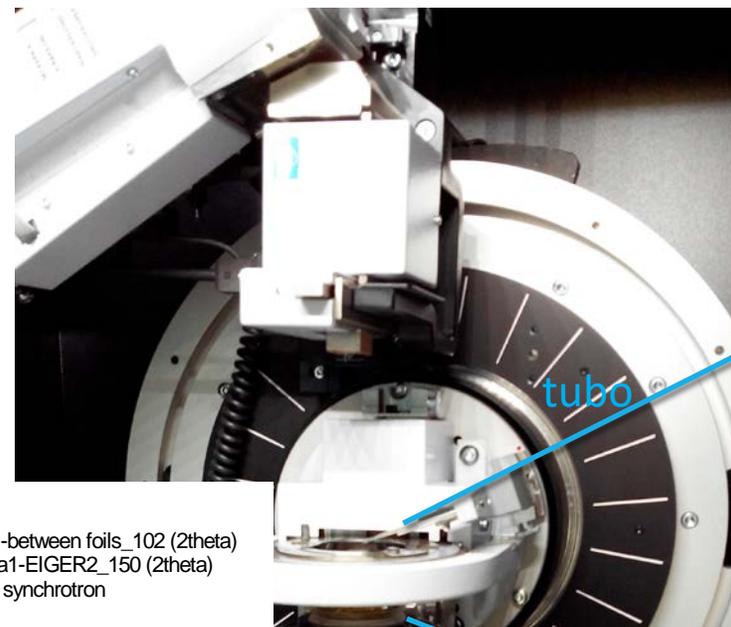
Energies shown are the $k_{\alpha 1}$ emission energies. The Q_{\max} values are calculated assuming a maximum accessible scattering angle of 160°. Note the convenient property that the Q_{\max} value in Å⁻¹ is almost the same as the X-ray energy in keV. A useful resource for finding X-ray properties of elements is the 'X-ray data book' published by Lawrence Berkeley National Laboratory and available online at <http://xdb.lbl.gov/>.



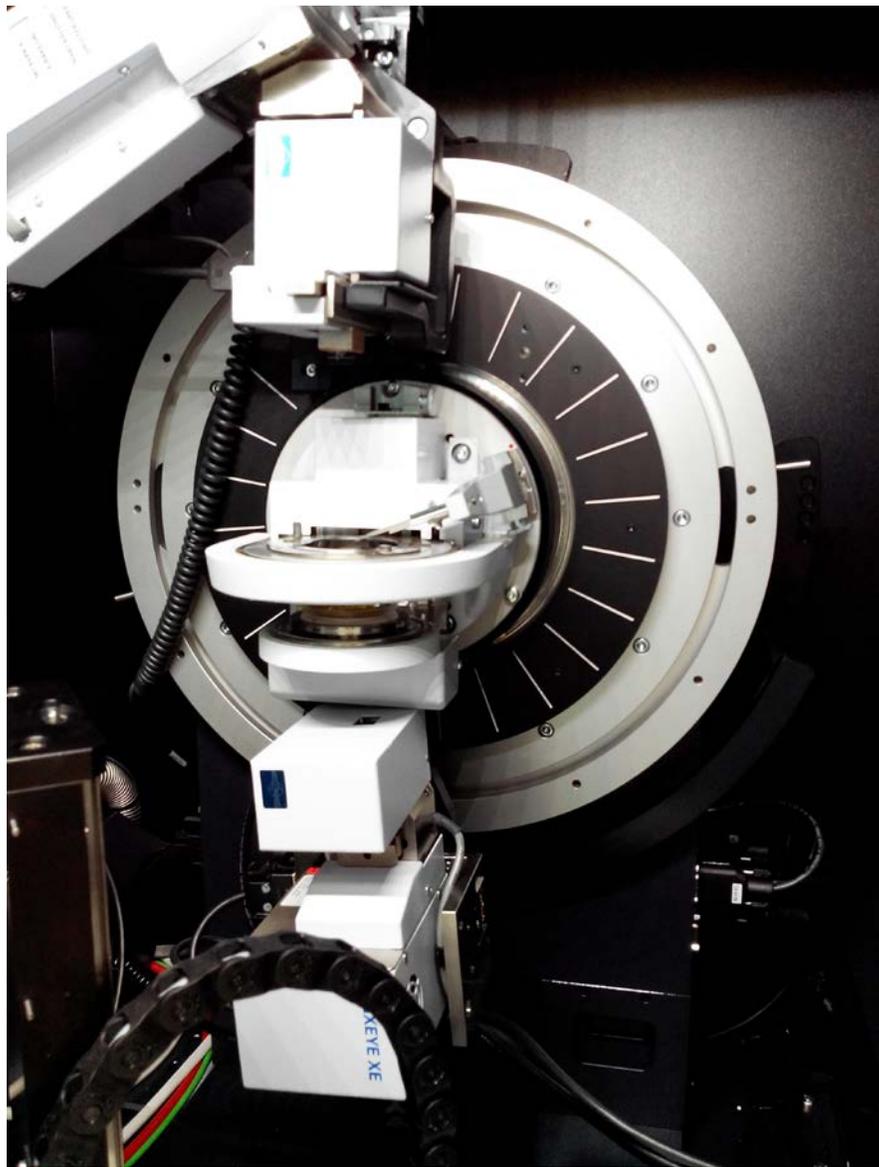
— C4A3S SRF
— C4A3S Mo
— C4A3S Ag



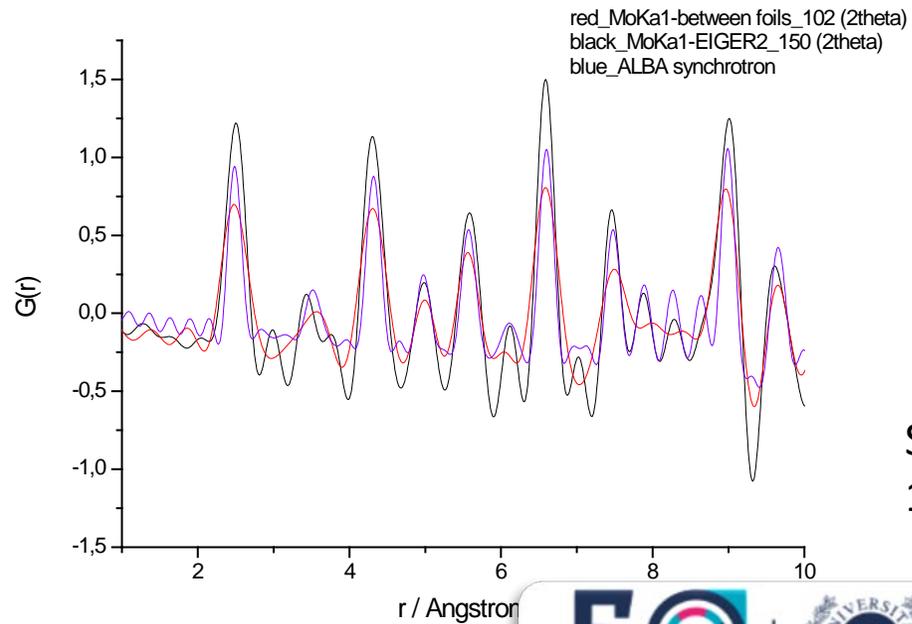
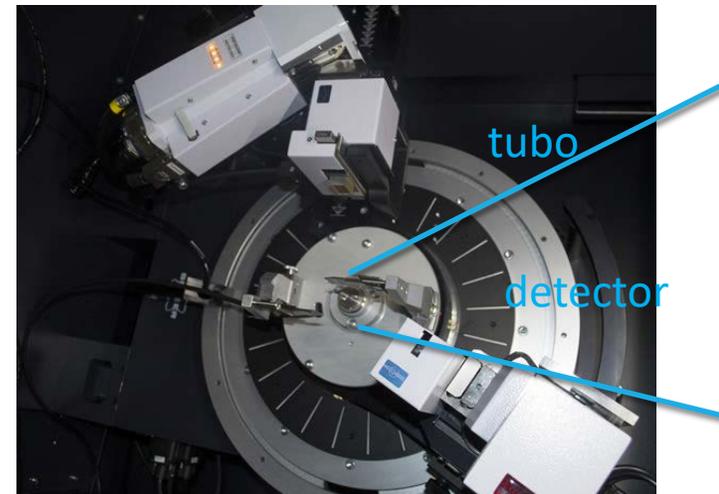
¿Puedo hacer PDF entre foils?



Sólo 102 grados 2 theta,
13,7 en Q, INSUFICIENTE



¿Puedo hacer PDF en capilar?



Sólo 130 grados 2 theta,
16 en Q, INSUFICIENTE

Cambio de detector:

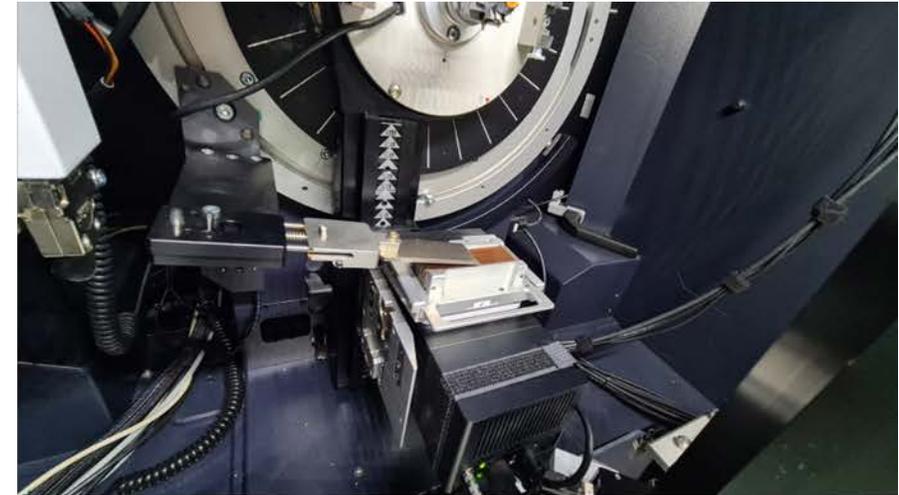
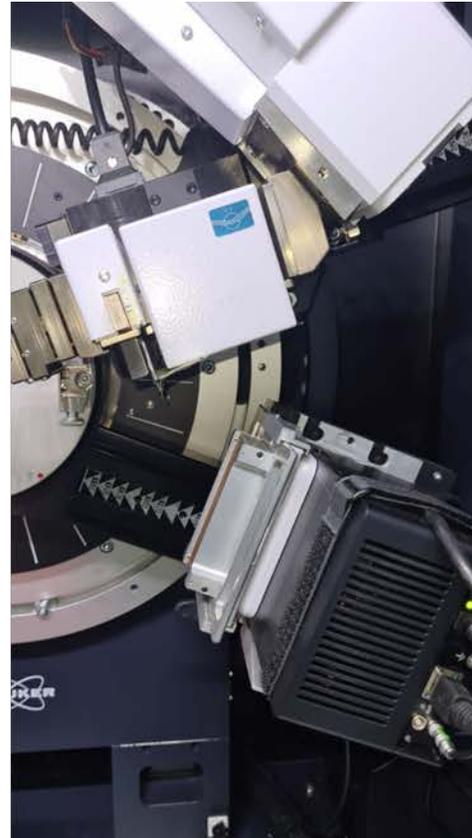
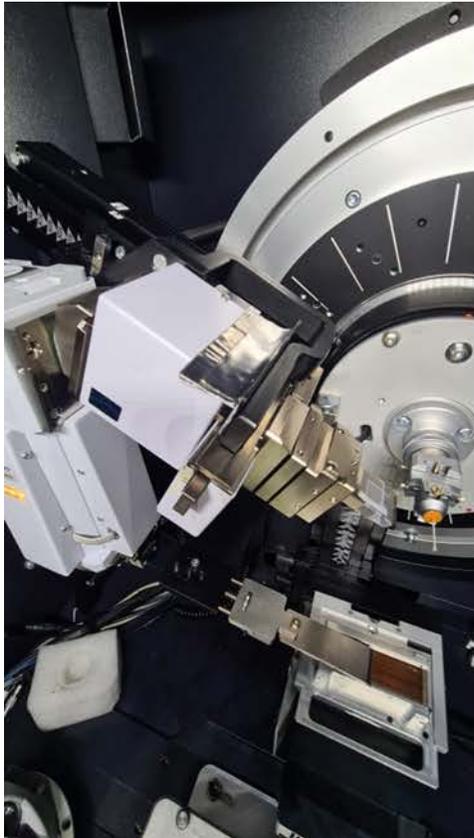


Active Window: 14.4 x 16 mm²; 3° coverage at 250 mm radius



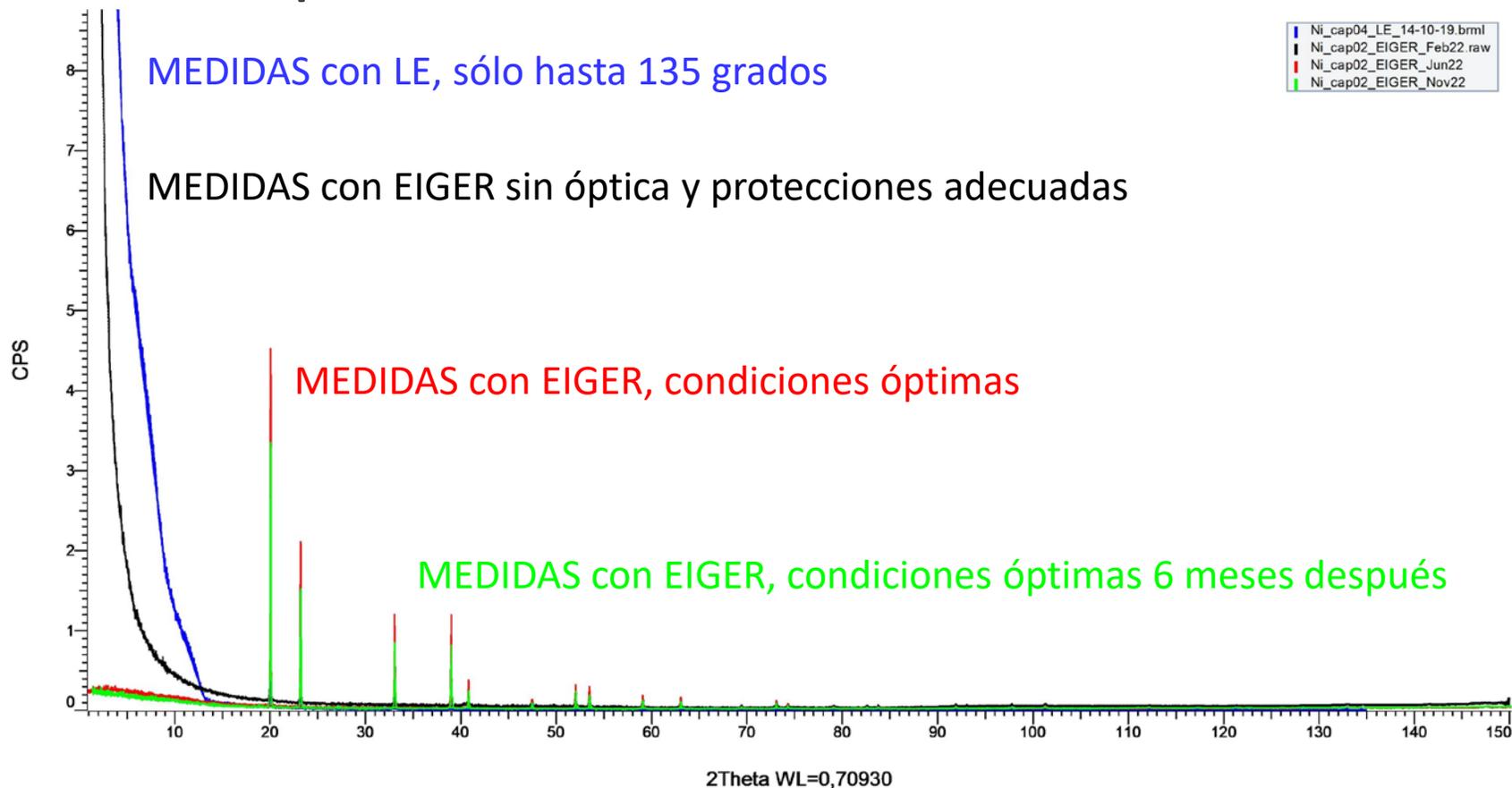
Active Area	250K: 38.4 x 38.4 = 1,475 mm ²
	500K: 77.1 x 38.4 = 2,961 mm ²

El detector que todo lo ve:



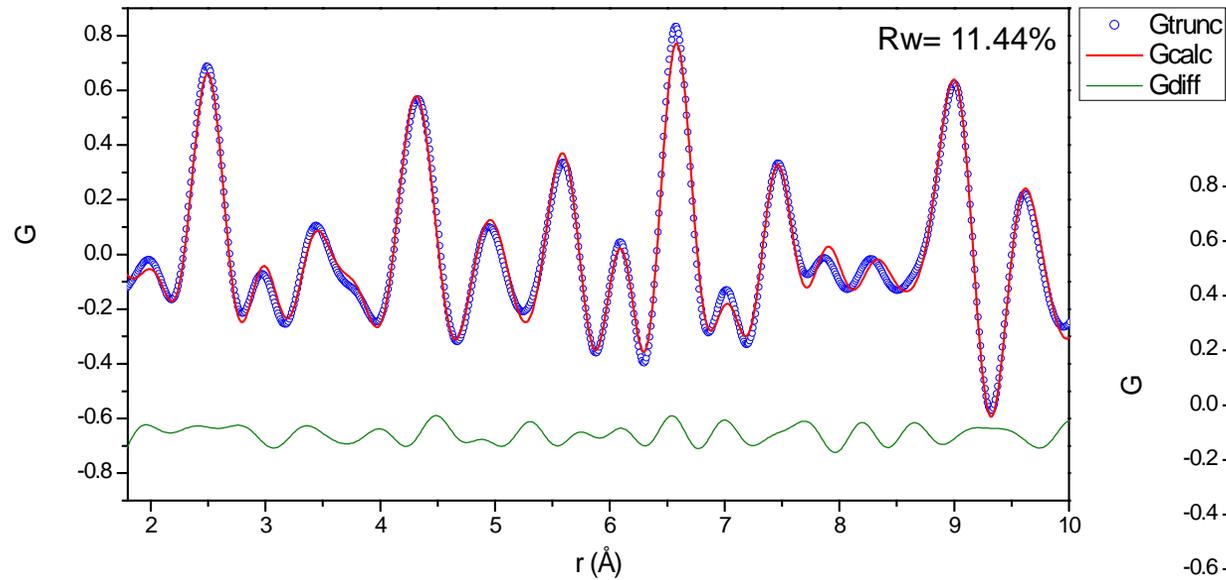
-  Snout
-  Beam knife
-  Recubrimientos de Pb

Comparativa datos de Ni

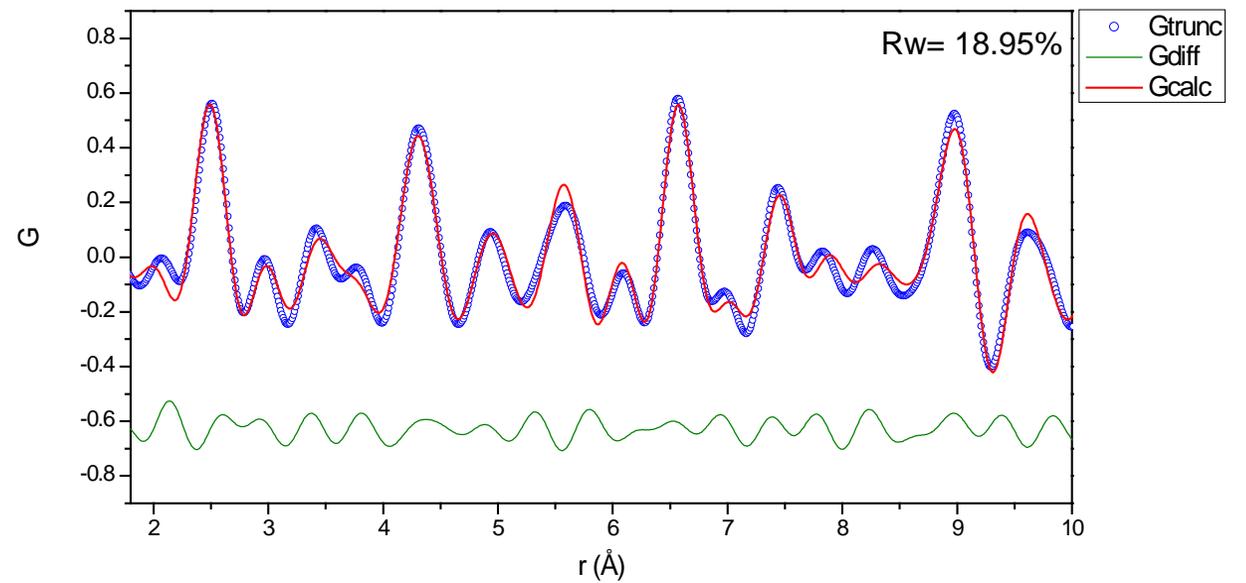


Comparativa datos de Ni

Junio 22

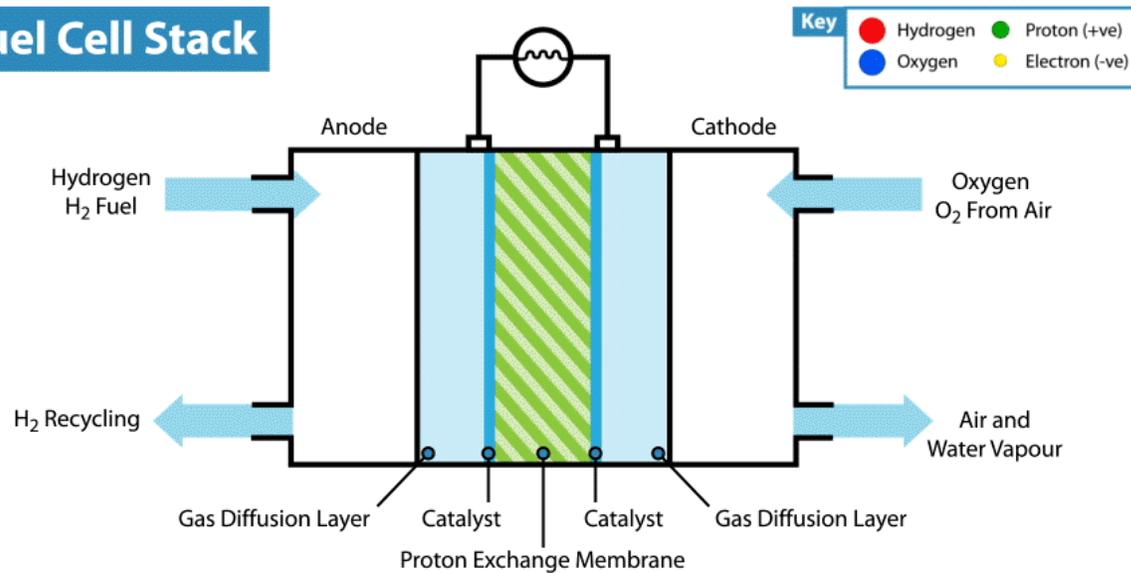


Noviembre 22



Electrocatalizador cristalino para Water Splitting

Fuel Cell Stack



Objetivo general: preparación de fosfuros metálicos como electrocatalizadores de pilas de combustible o electrolisis del agua (water splitting)

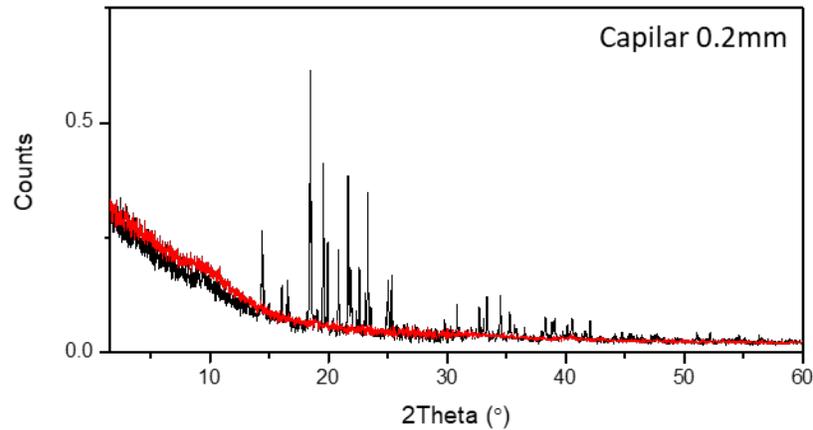
La muestra en estudio es una mezcla de fosfuros de cobalto (Co_2P/CoP) cristalinos obtenidos por pirólisis de un fosfonato de Co en atmósfera de 5%- H_2/Ar

¿Por qué PDF?: búsqueda de fases amorfas, a parte de las fases cristalinas previas, que puedan afectar al rendimiento electroquímico.

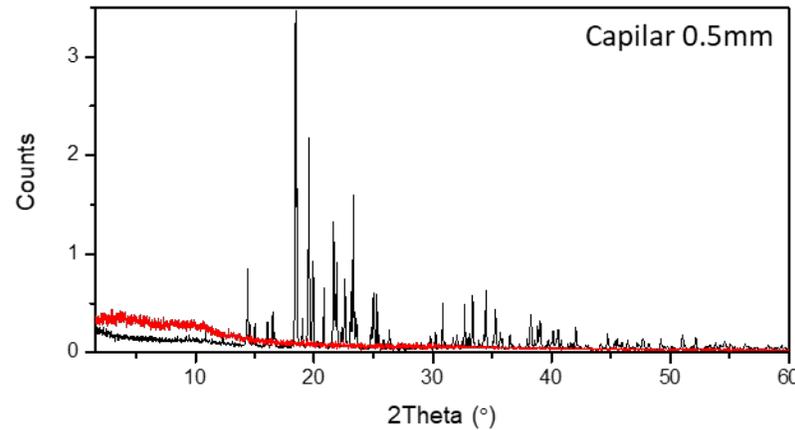
Elección capilar adecuado

MAC aprox. 280cm²/g muestra muy absorbente, preferiblemente capilar pequeño

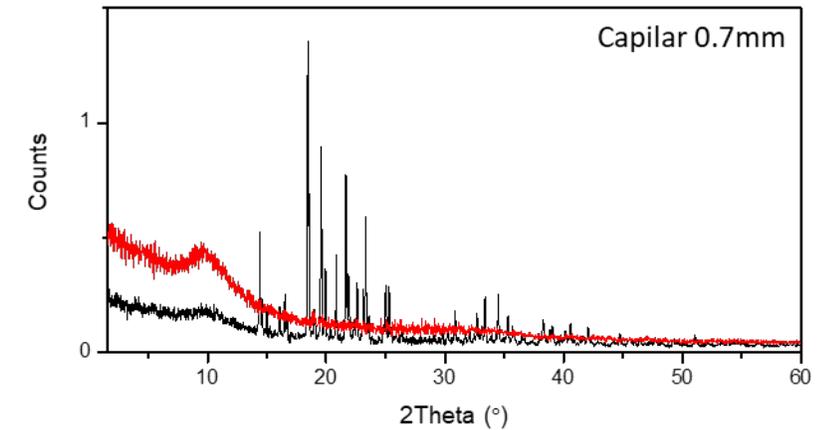
Co800
Capilar vacío



Medido Nov22



Medido Jun22

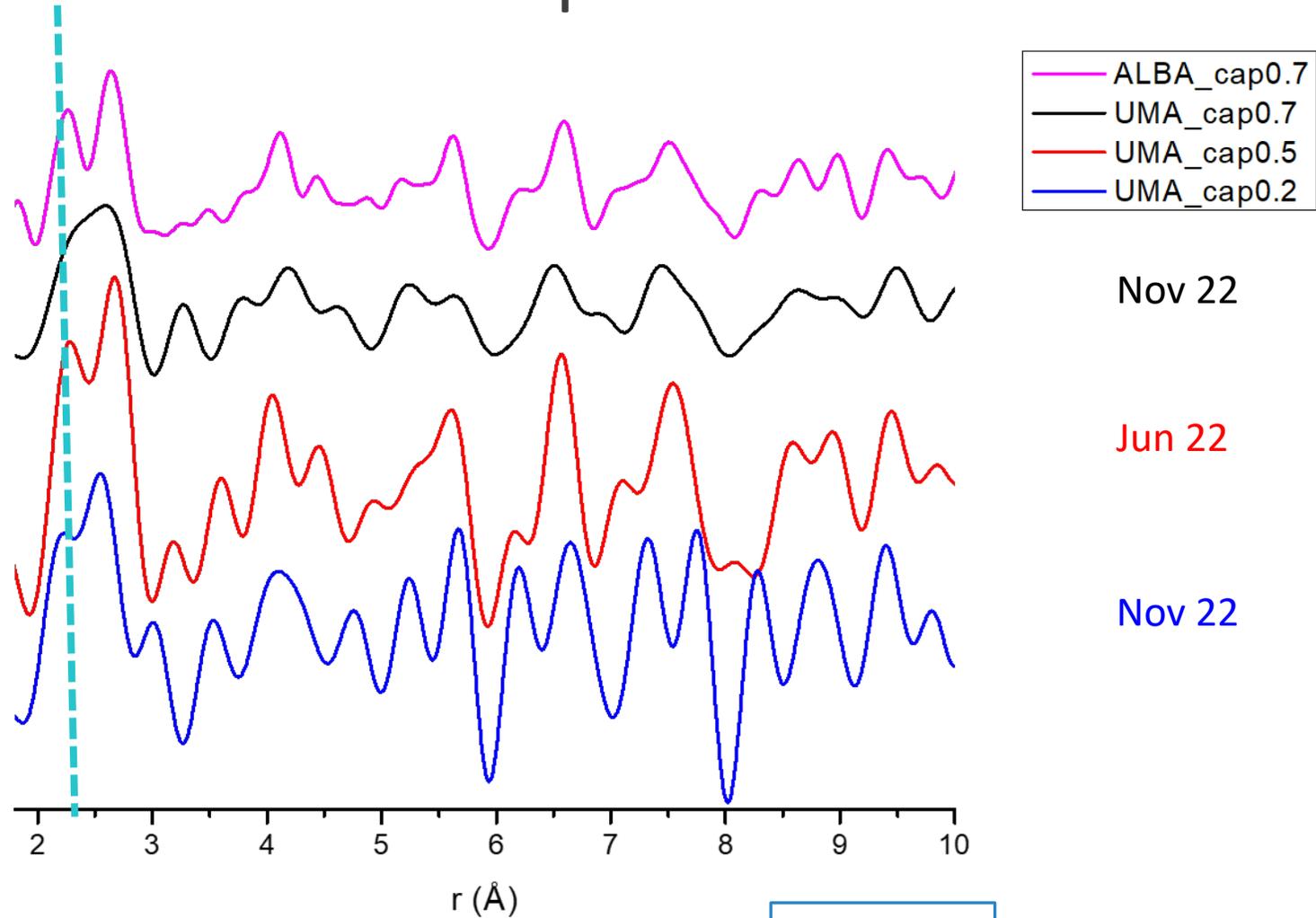


Medido Nov22

Horas de uso del tubo de rayos-X



Elección capilar adecuado



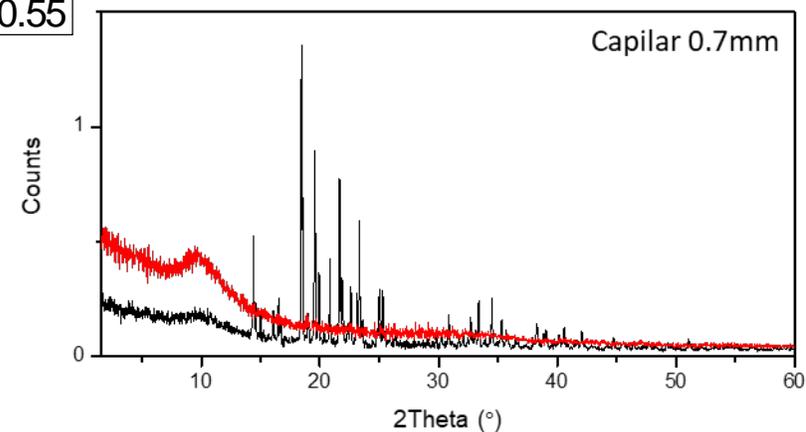
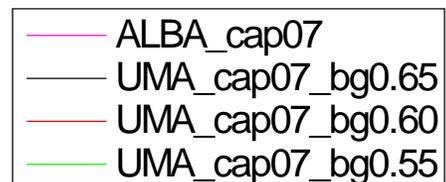
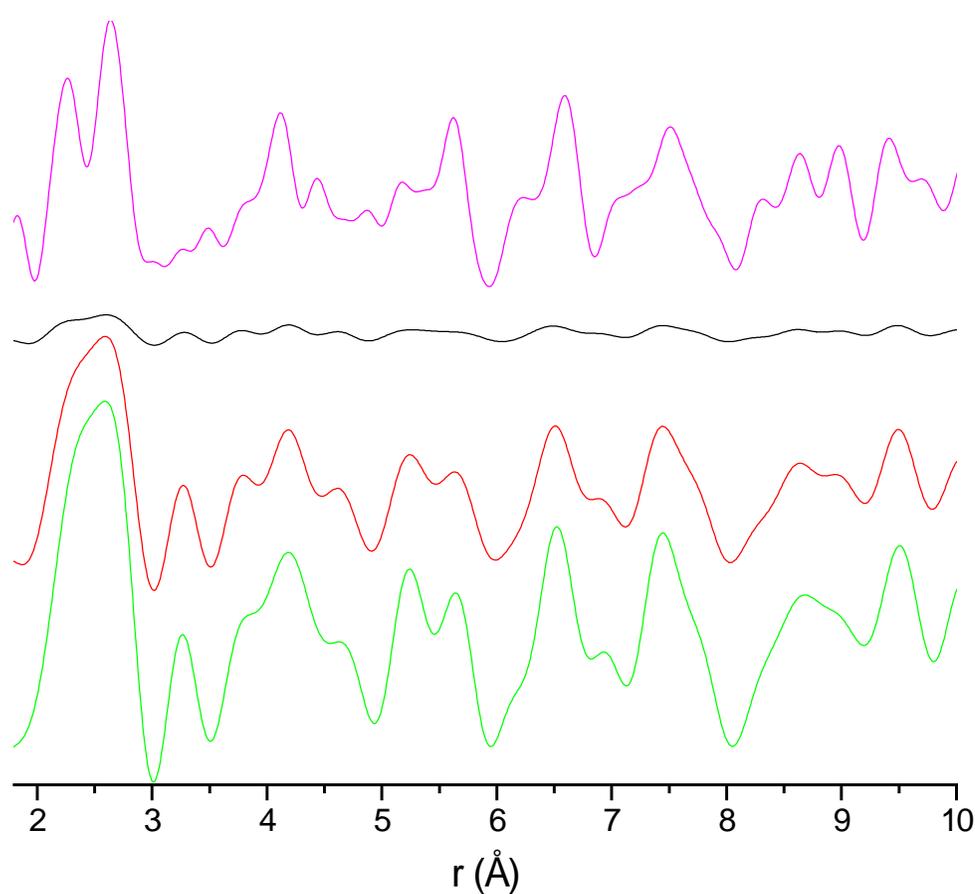
Nov 22

Jun 22

Nov 22

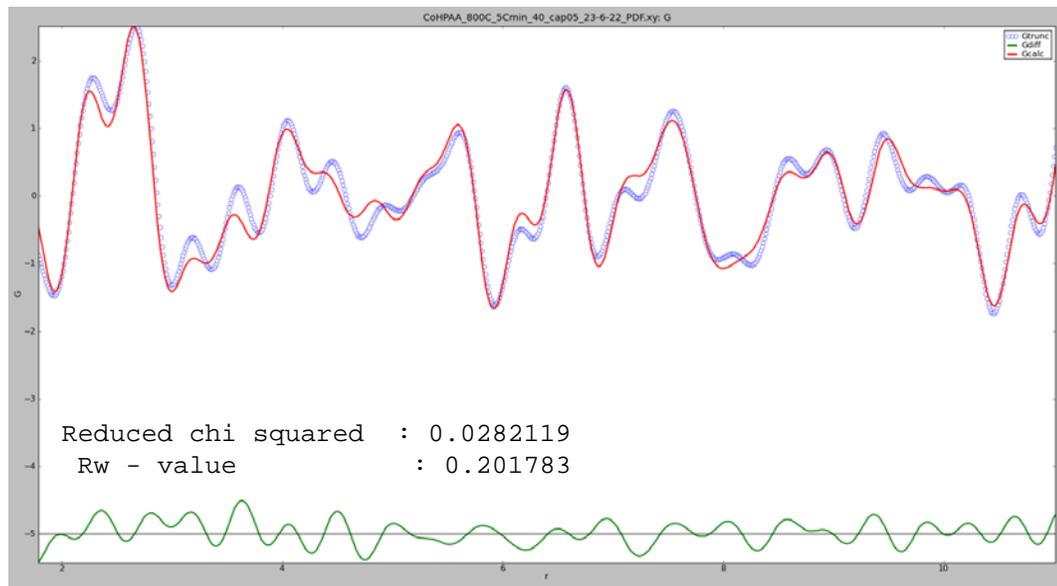


Importancia del capilar vacío

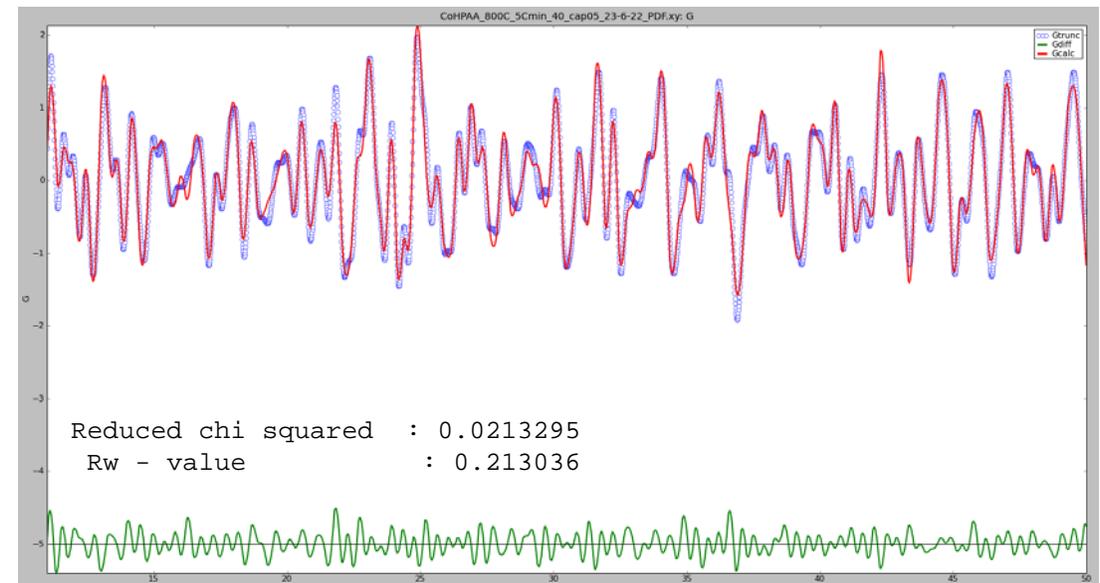


- ✓ Medir capilar vacío en iguales condiciones
- ✓ Elección del factor adecuado en la resta del fondo

Resultados del estudio de electrocatalizador cristalino (800C) por PDF



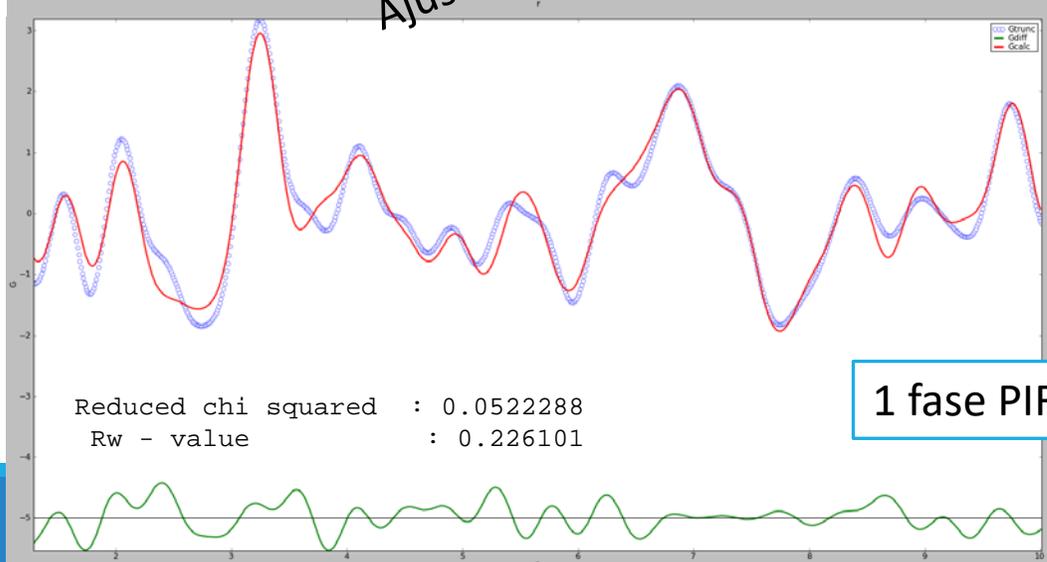
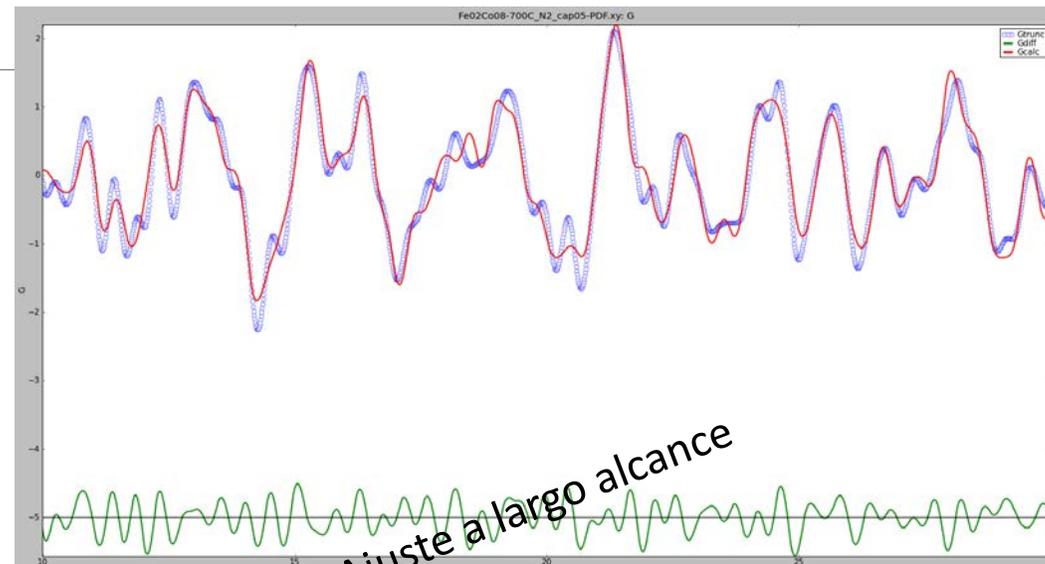
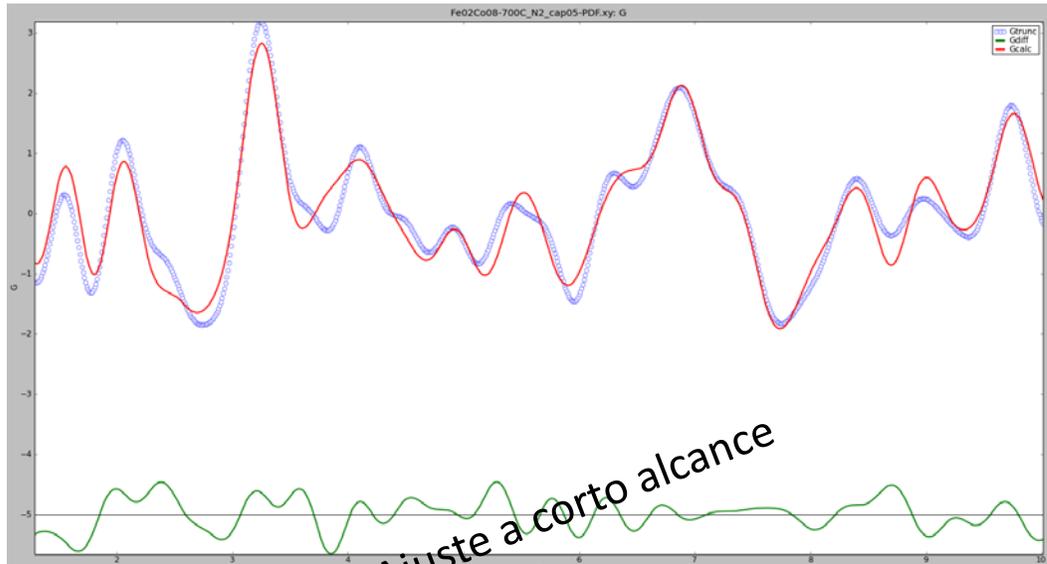
Ajuste a corto alcance



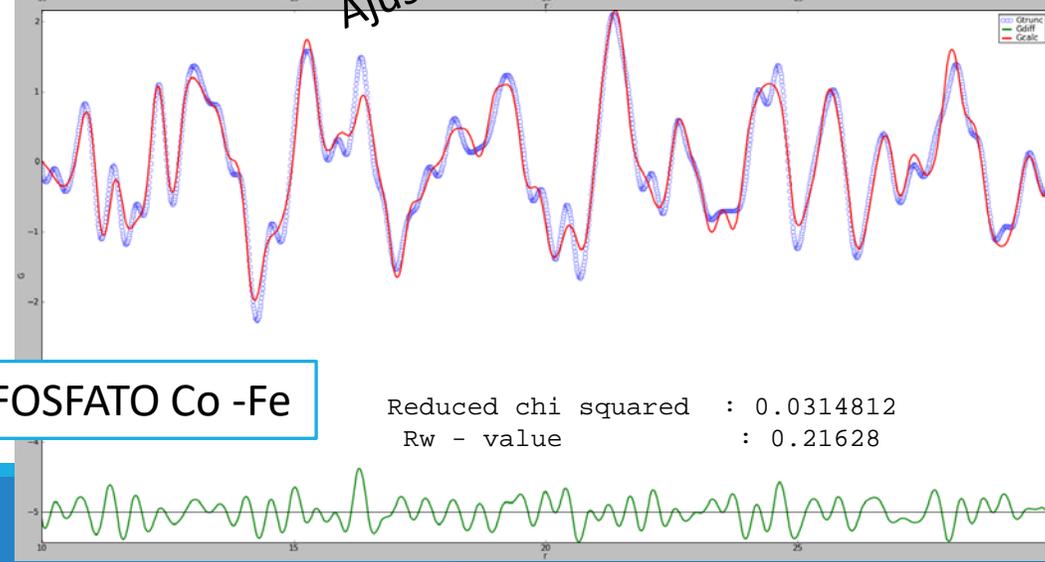
Ajuste a largo alcance

Resultados cuantitativo:
74% Co₂P
26% CoP

Resultados del estudio de electrocatalizador semi-cristalino (700C) por PDF



1 fase PIROFOSFATO Co -Fe

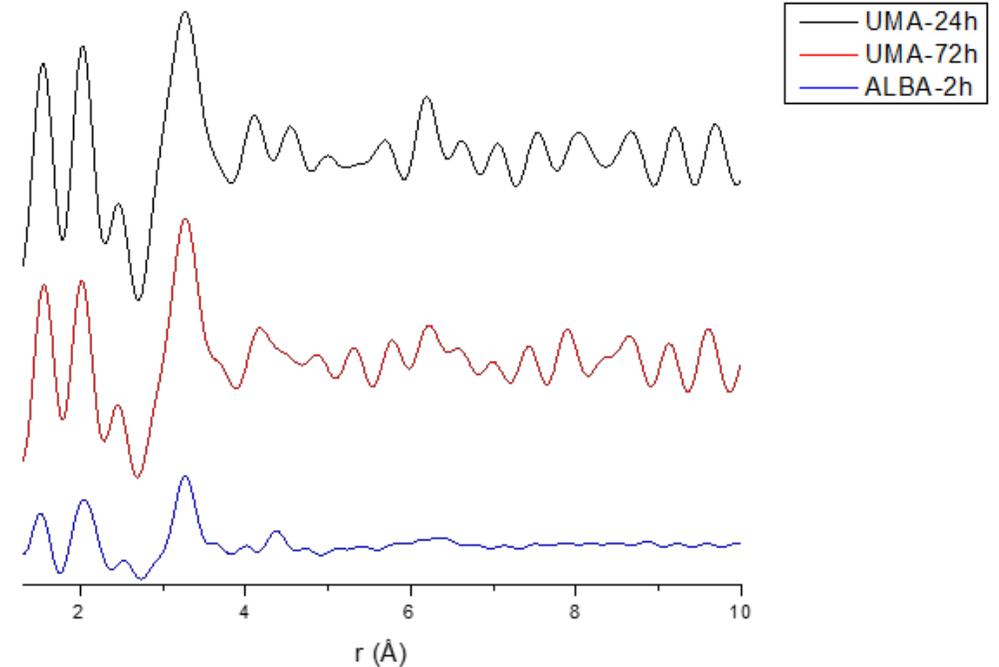
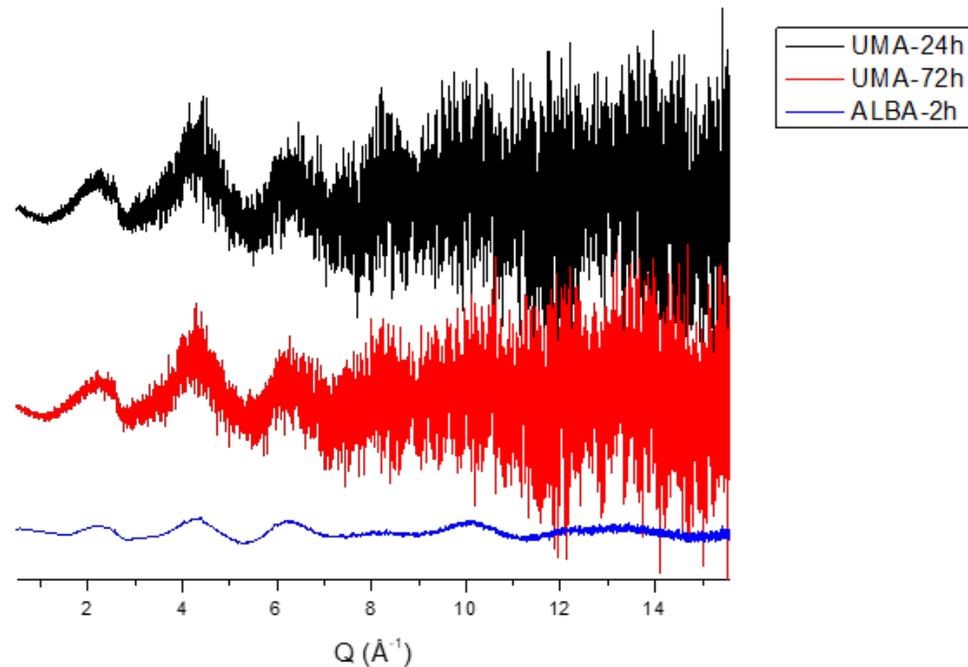


Sin afinar posiciones atómicas

Afinando posiciones atómicas

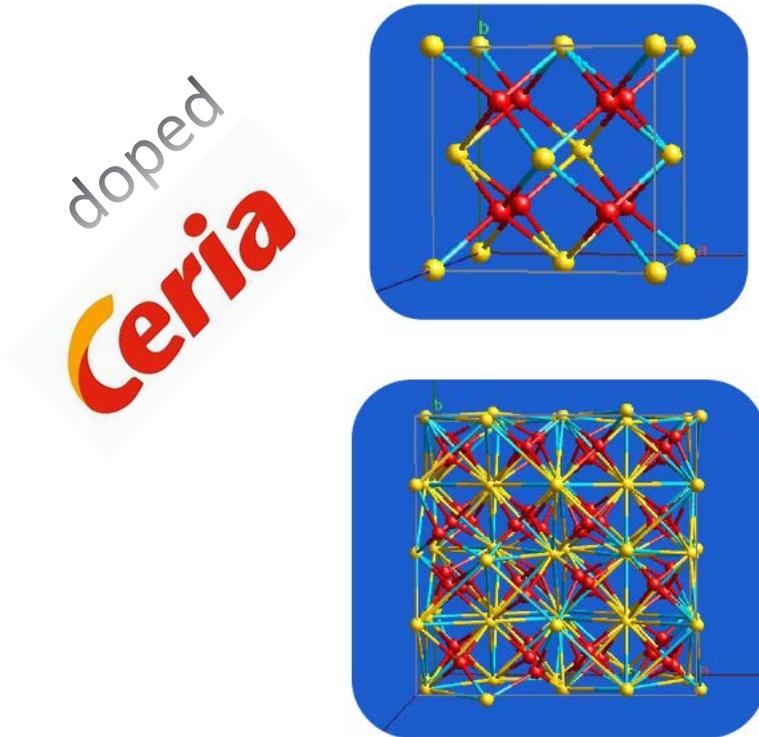
Constrains!!!

Estudio de electrocatalizador amorfo (500C) por PDF ¿hasta dónde puedo llegar?



¿1 fase PIROFOSFATO Co –Fe?????

ESTUDIO DEL DESORDEN LOCAL Y CUANTIFICACIÓN DE FASES POR PDF DE LABORATORIO



Tipo FLUORITA F

Tipo C

¿Por qué no con Rietveld?



Article

Local and Average Structure of Yb-Doped Ceria through Synchrotron and Neutron Pair Distribution Function

Mauro Coduri ^{1,2}, Dario Bozzetti ³, Stefano Checchia ^{2,4}, Michela Brunelli ⁵ and Marco Scavini ^{3,*}

4. Conclusions

The average and local structure evolution of ceria specimens with different Yb loadings was investigated, coupling synchrotron and neutron diffraction. Up to $\mu = 0.250$, long-range fluorite structure is maintained, even though evidence of static disorder is clear. C-type local ordering is suggested by the observation of further M-M distances in X-ray PDF, and longer O-O pairs by neutron.

ESTUDIO DEL ORDEN LOCAL, r de 2 a 6 Å

Ceria pura (sin dopar)

Rwp 17,3
Tipo F

Ceria dopada

Rwp 42,1
Tipo F

Fuente F type 100.00 %

Ceria dopada

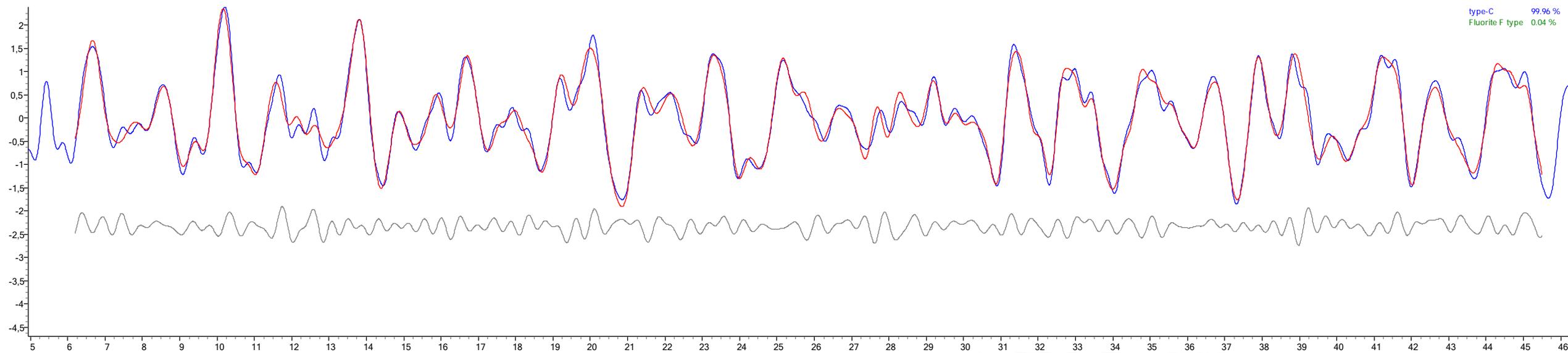
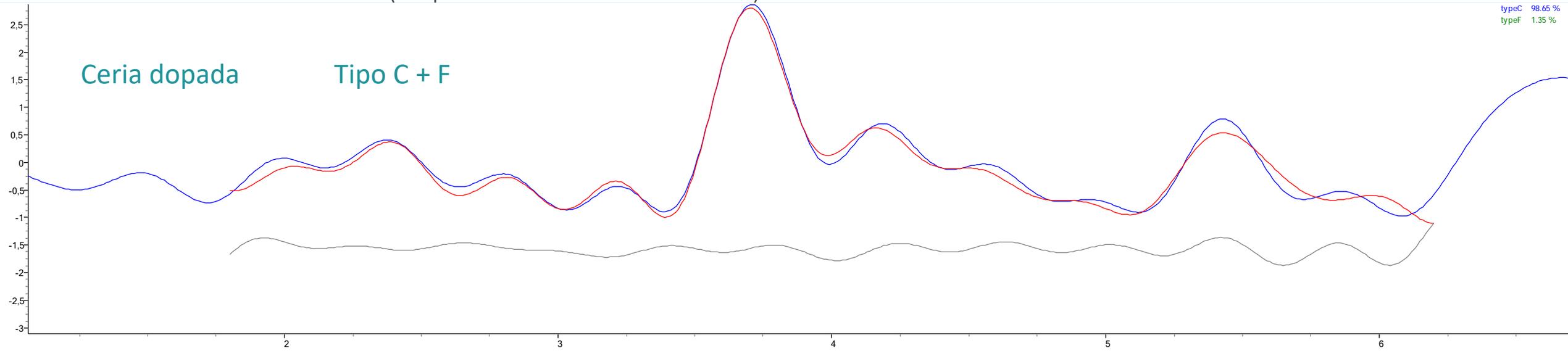
Rwp 49,7
Tipo C

Ceria dopada

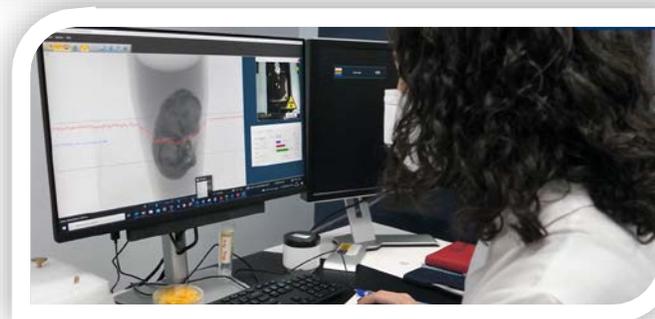
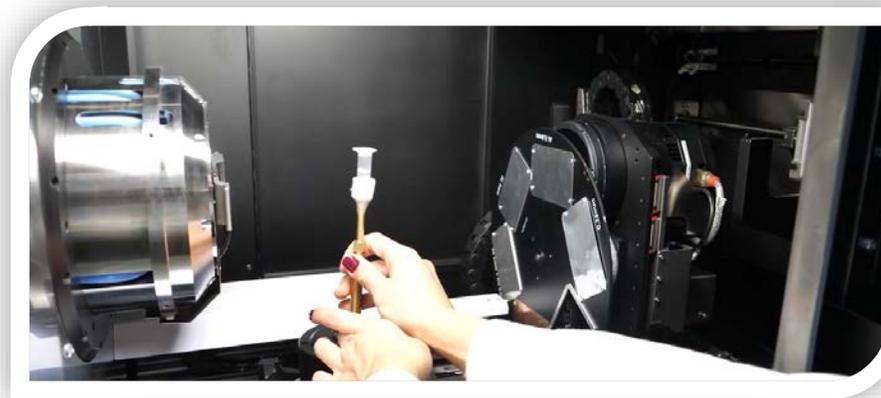
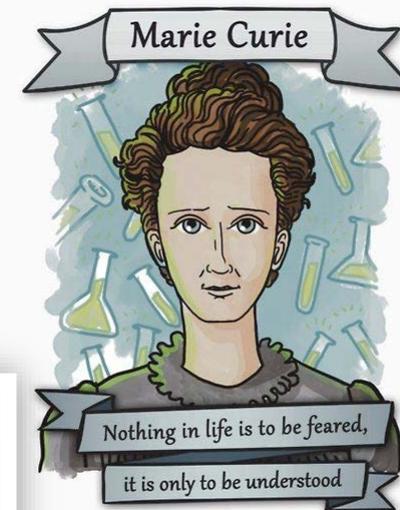
Rwp 15,2
Tipo C + F

typeC 98.65 %
typeF 1.35 %

CUANTIFICACIÓN DE FASES (empleando datos de laboratorio)



Muchas gracias por su atención



SCAI UMA
@scaiuma

El SCAI de la UMA incorpora un nuevo equipo de nano tomografía computarizada



youtube.com

El SCAI de la UMA incorpora un nuevo equipo de nano tom...
El SCAI de la UMA incorpora un nuevo equipo de nano tomografía computarizada, que se ha incorporado ...

1:36 p. m. · 23 dic. 2021 · Twitter Web App



UNIVERSIDAD
DE MÁLAGA

