

La odisea de medir PDF con un difractómetro “convencional”

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RESPONSABLE CIENTÍFICO: DR. AURELIO CABEZA DÍAZ



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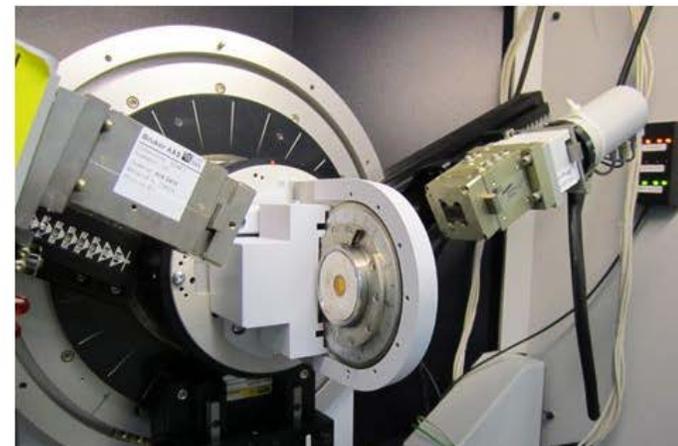


Un laboratorio para todos y para todo



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

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

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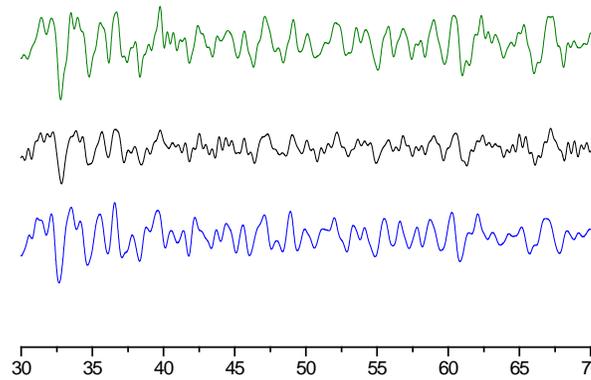
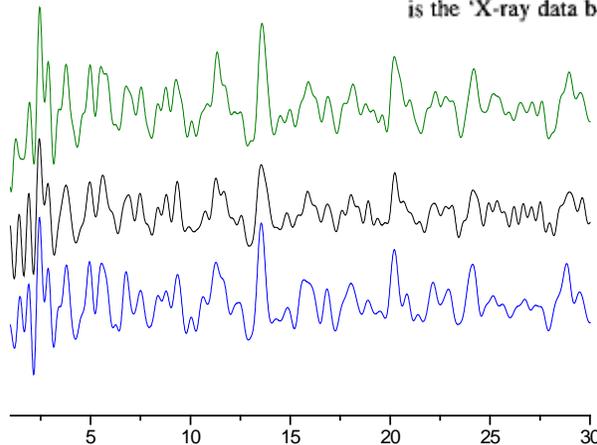
Aprovecho mi difractor de Mo para medir PDF



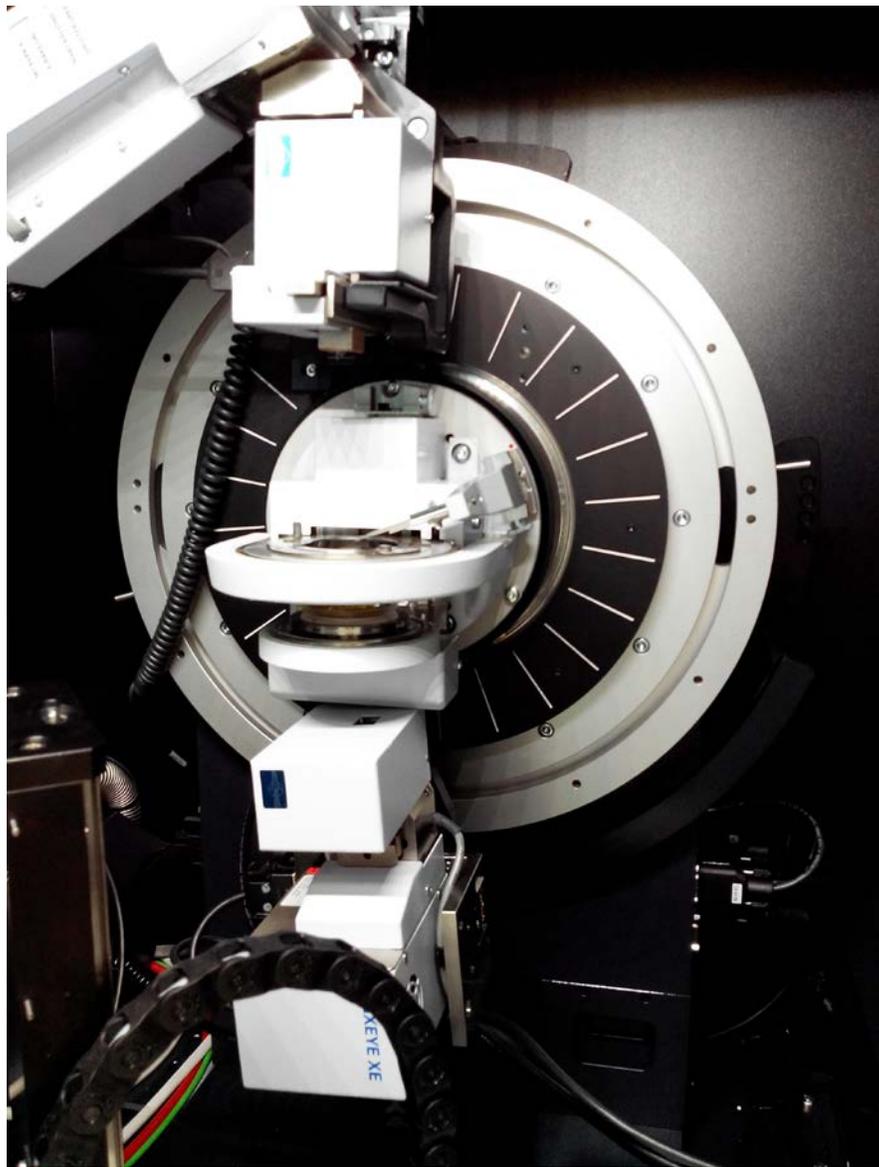
Table 4.2. X-ray energies, wavelengths and approximate Q_{\max} values for common laboratory X-ray sources.

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

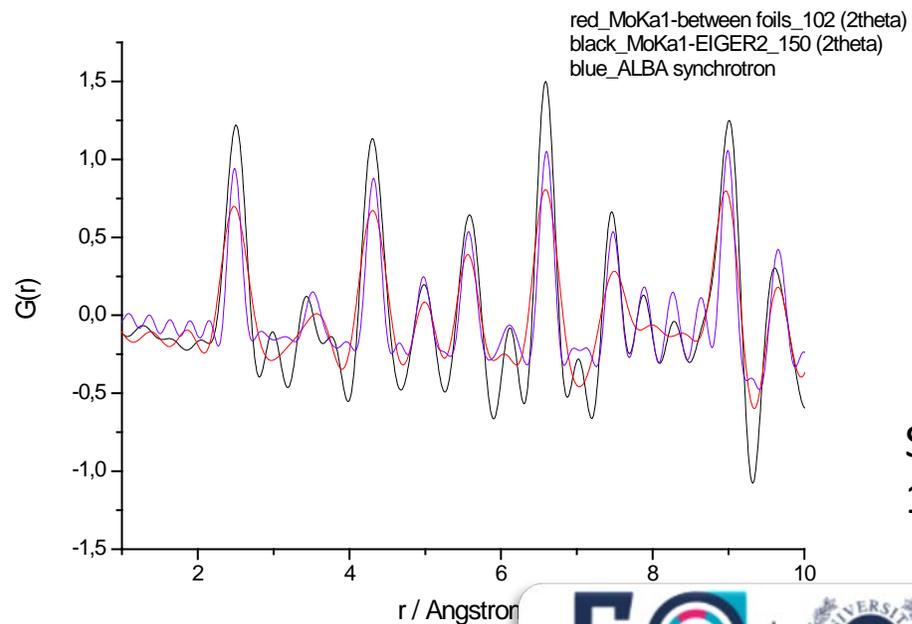
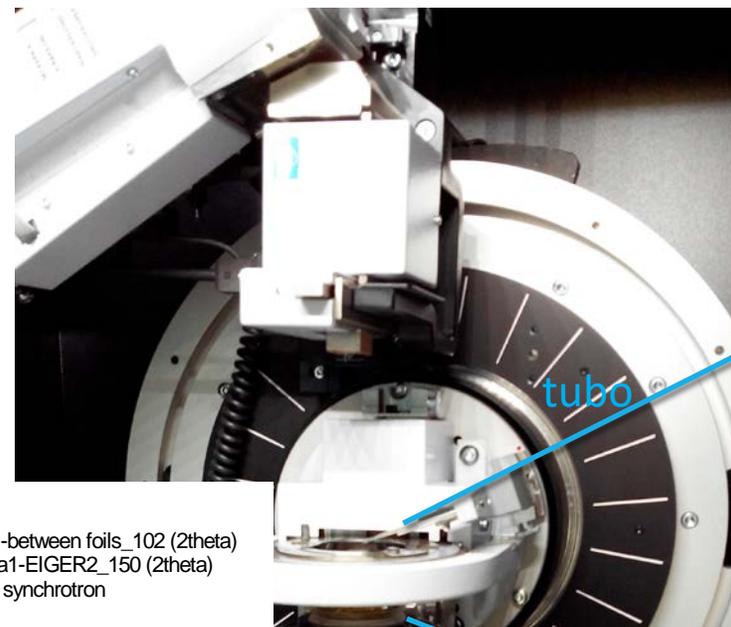
Energies shown are the $k_{\alpha 1}$ emission energies. The Q_{\max} values are calculated assuming a maximum accessible scattering angle of 160°. Note the convenient property that the Q_{\max} value in Å⁻¹ is almost the same as the X-ray energy in keV. A useful resource for finding X-ray properties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



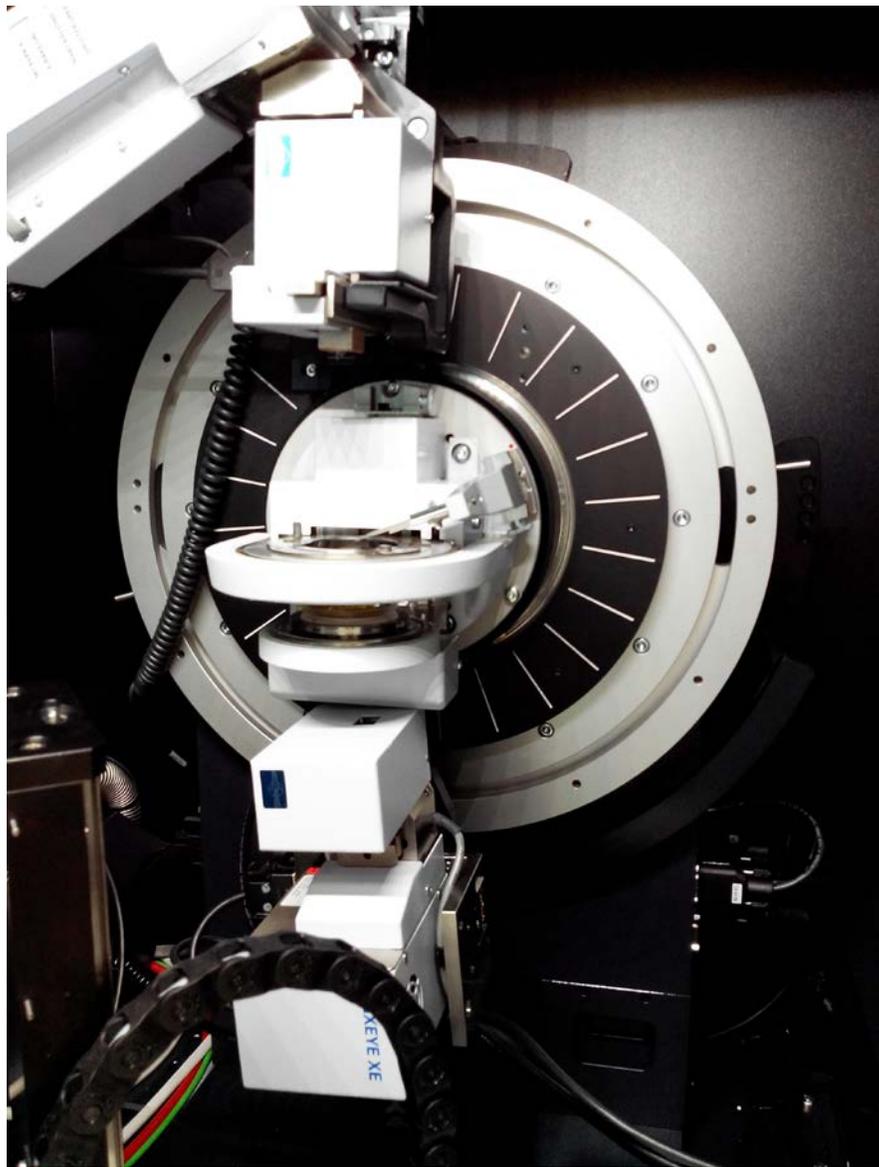
— C4A3S SRF
— C4A3S Mo
— C4A3S Ag



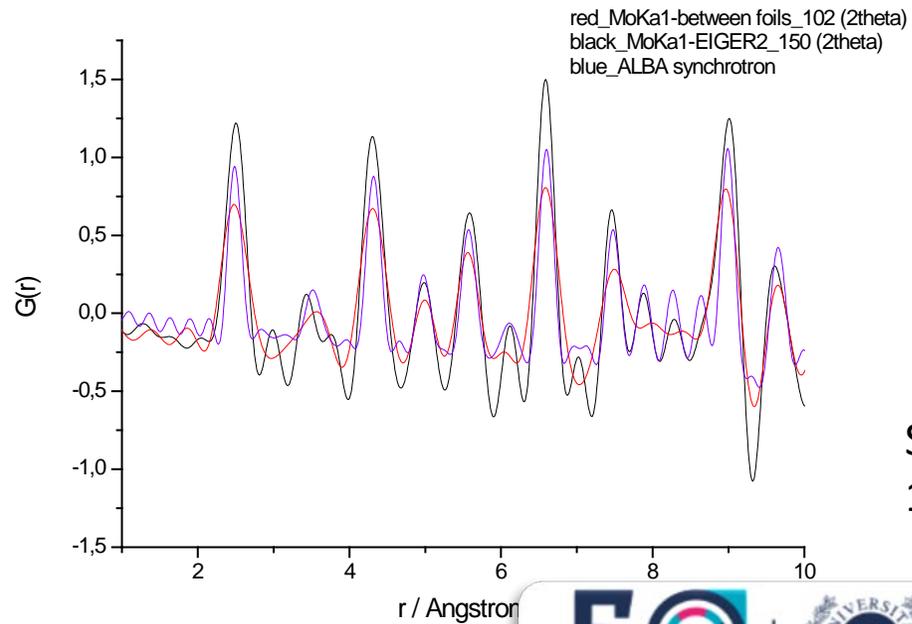
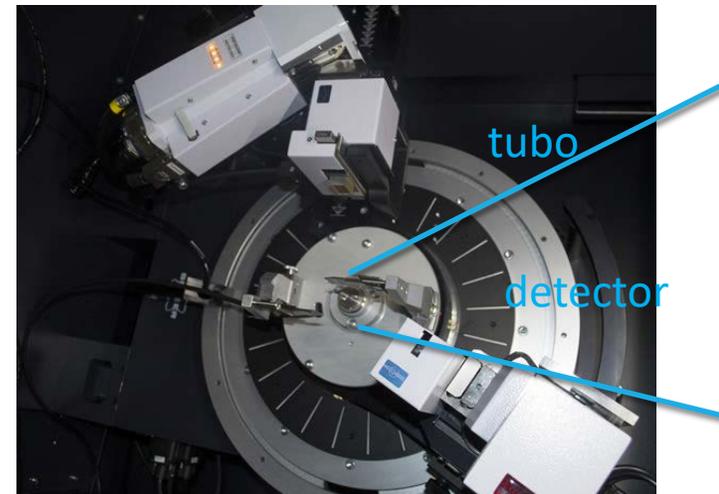
¿Puedo hacer PDF entre foils?



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



¿Puedo hacer PDF en capilar?

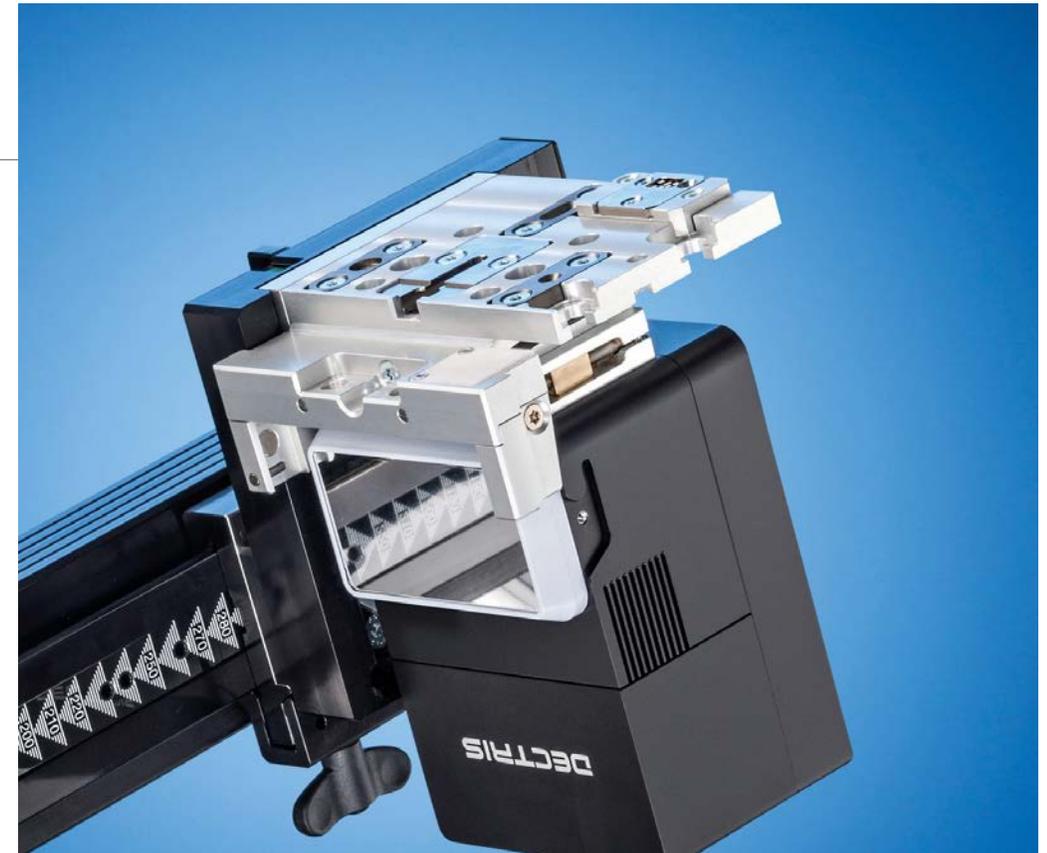


Sólo 130 grados 2 theta,
16 en Q, INSUFICIENTE

Cambio de detector:

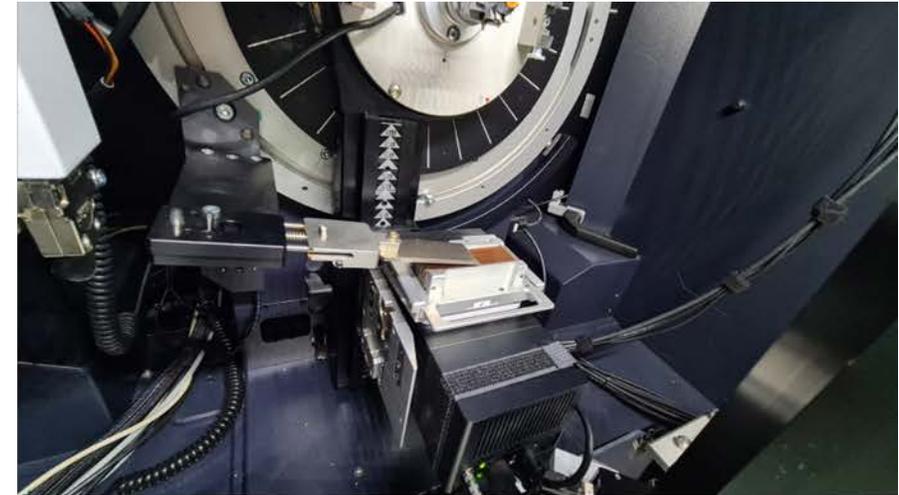
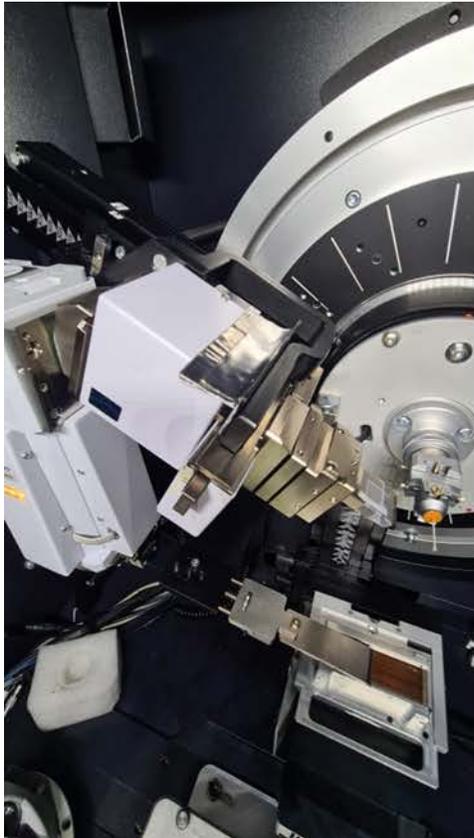


Active Window: 14.4 x 16 mm²; 3° coverage at 250 mm radius



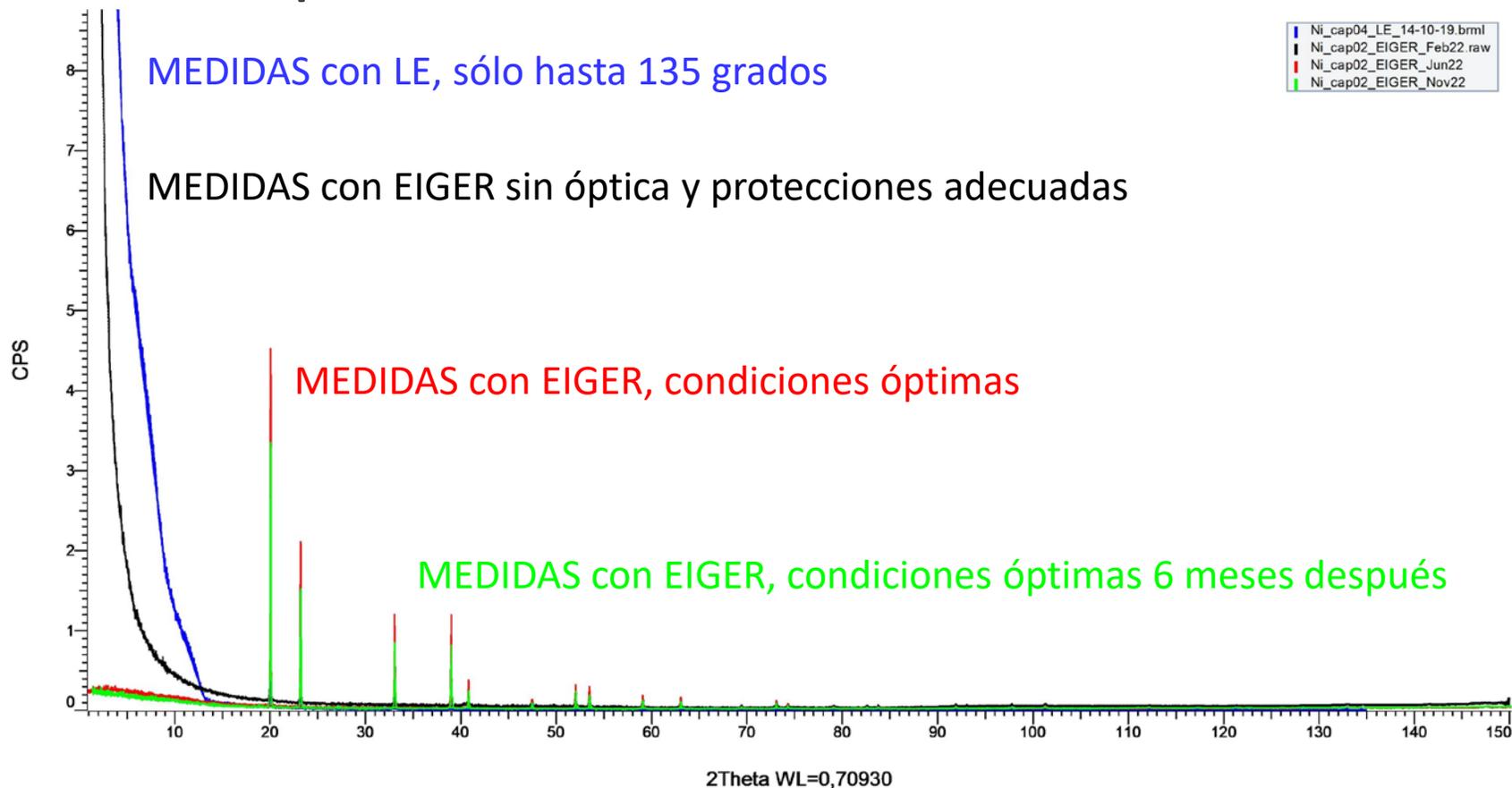
Active Area	250K: 38.4 x 38.4 = 1,475 mm ²
	500K: 77.1 x 38.4 = 2,961 mm ²

El detector que todo lo ve:



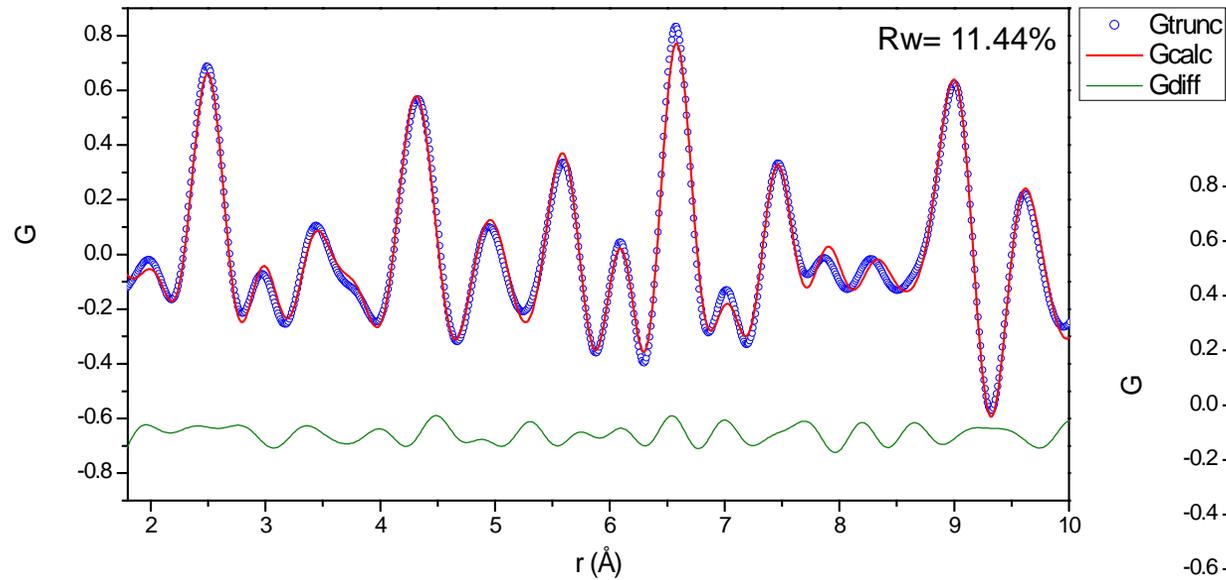
-  Snout
-  Beam knife
-  Recubrimientos de Pb

Comparativa datos de Ni

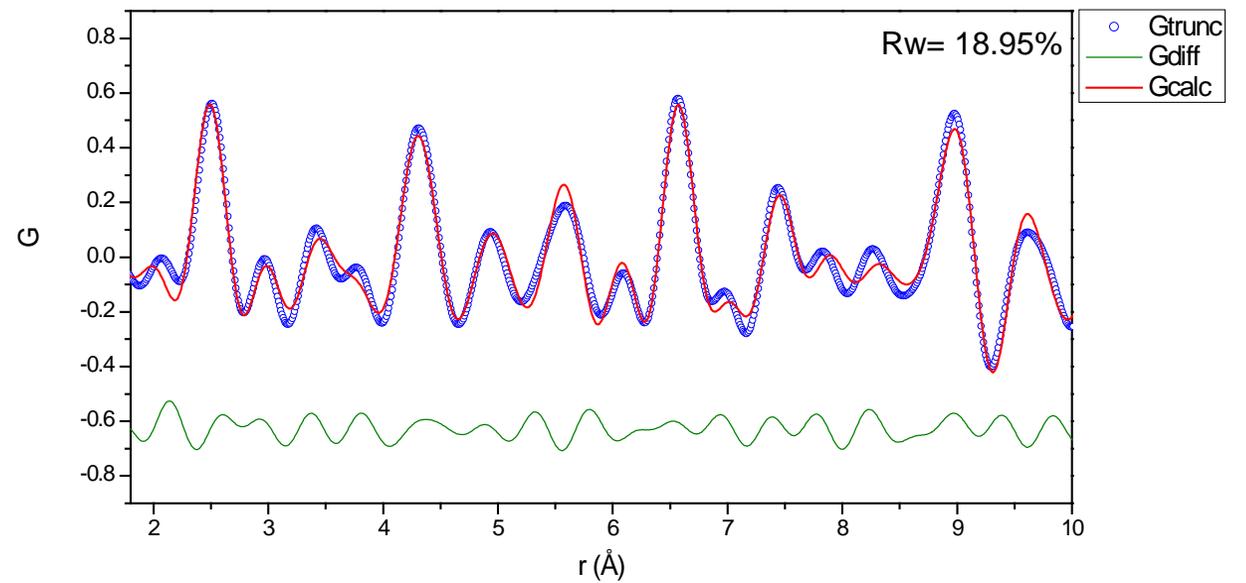


Comparativa datos de Ni

Junio 22

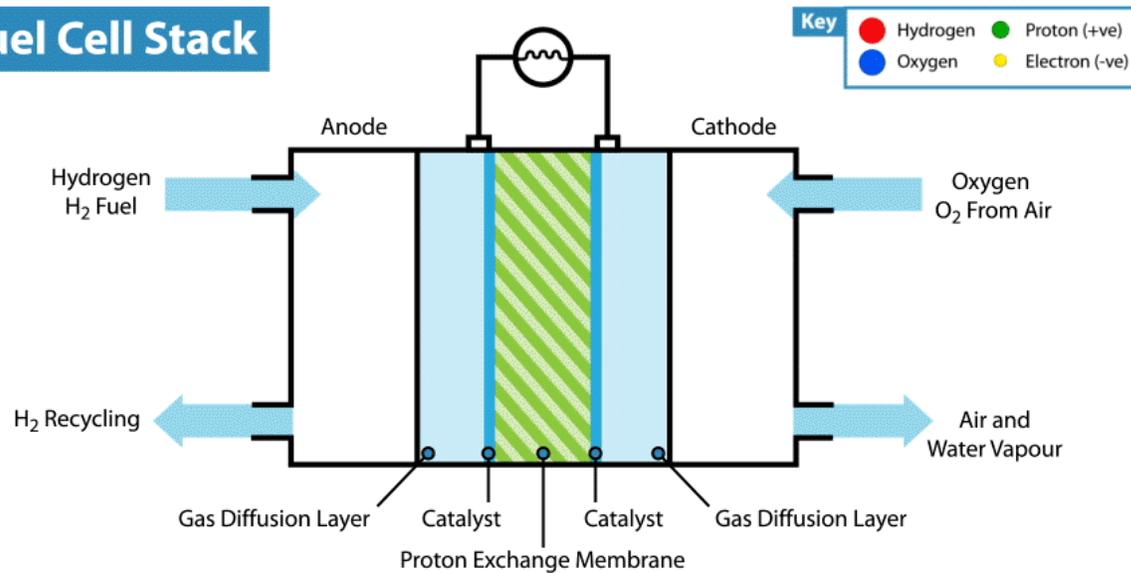


Noviembre 22



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)

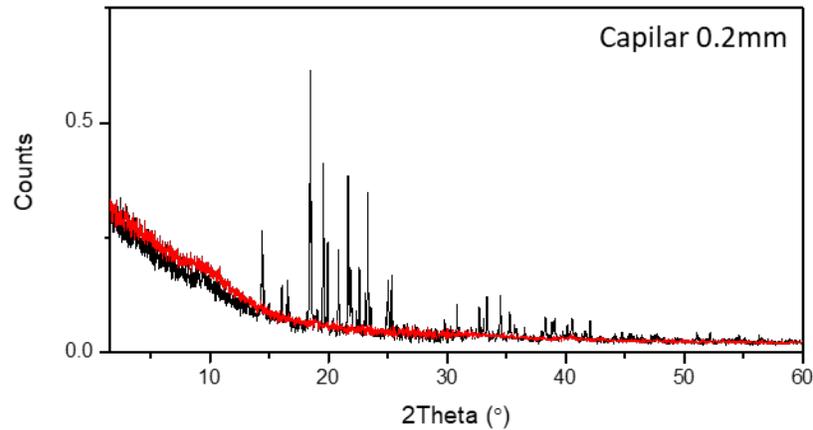
La muestra en estudio es una mezcla de fosfuros de cobalto (Co_2P/CoP) cristalinos obtenidos por pirólisis de un fosfonato de Co en atmósfera de 5%- H_2/Ar

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

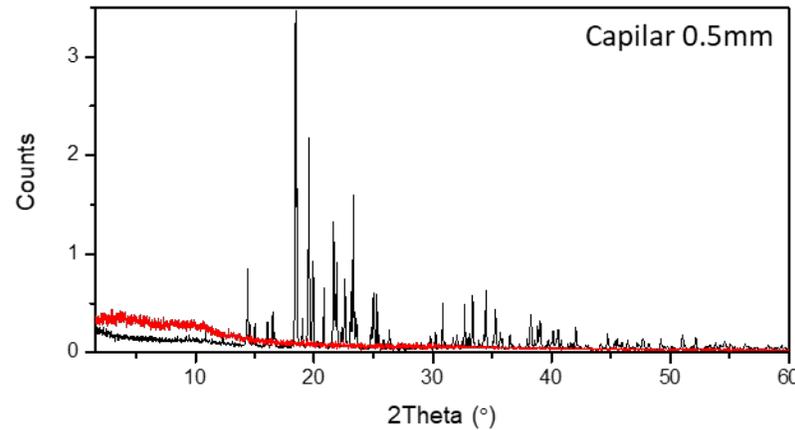
Elección capilar adecuado

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

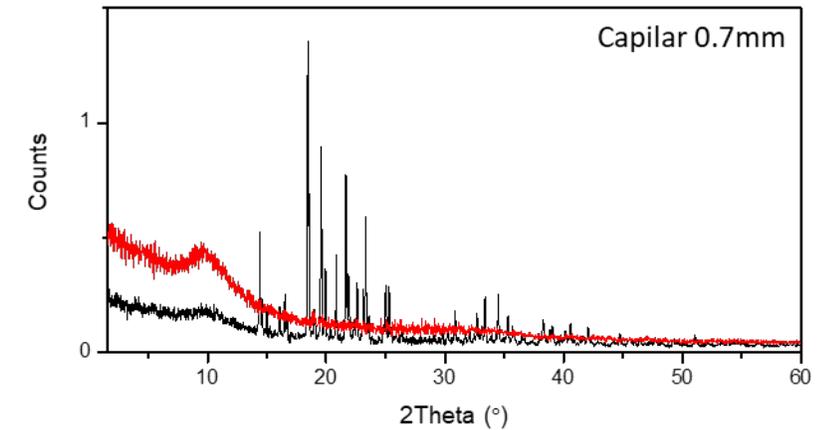
Co800
Capilar vacío



Medido Nov22



Medido Jun22

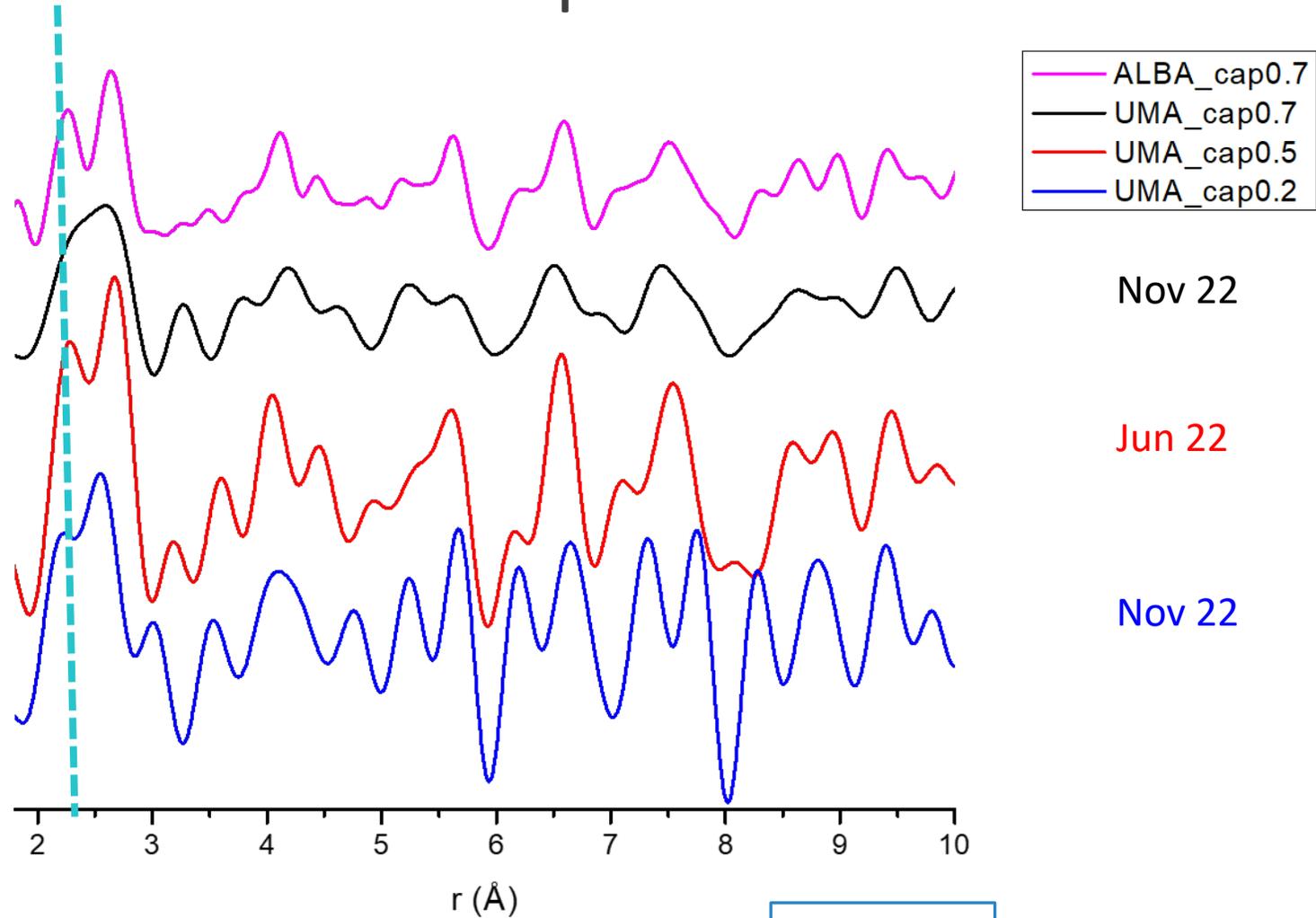


Medido Nov22

Horas de uso del tubo de rayos-X



Elección capilar adecuado



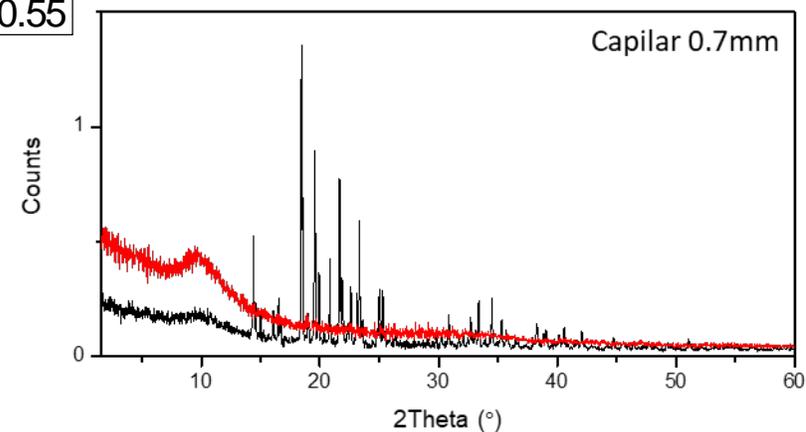
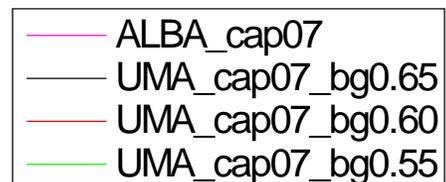
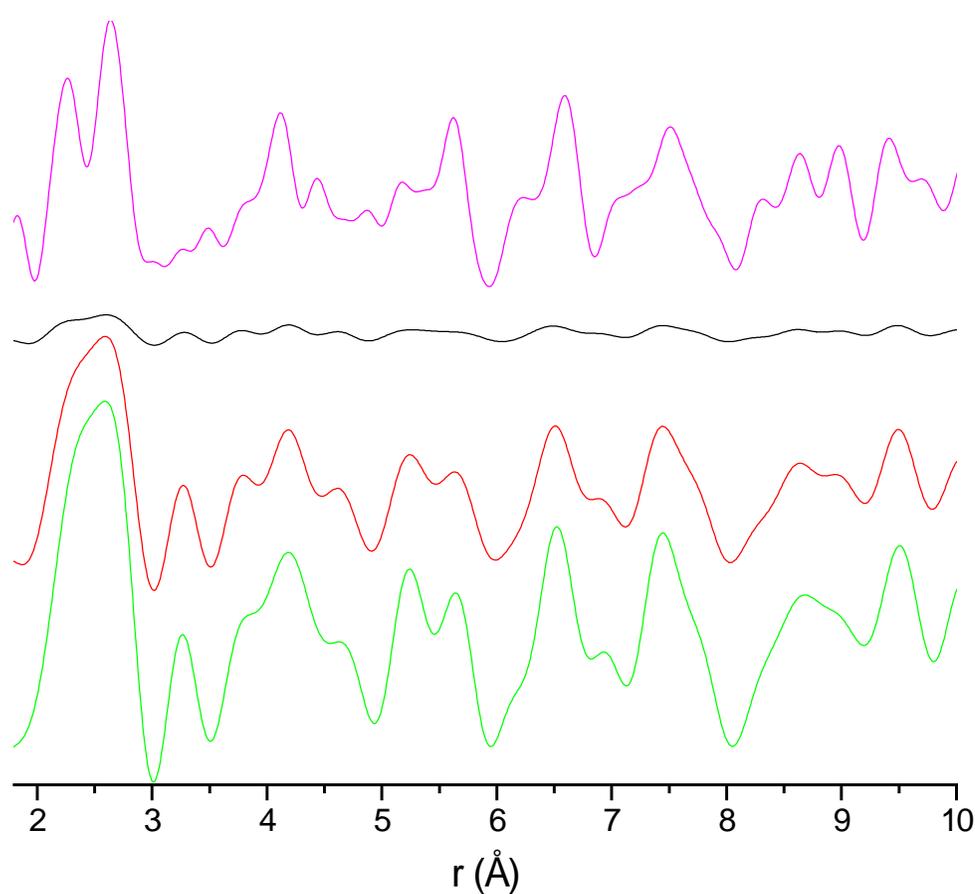
Nov 22

Jun 22

Nov 22

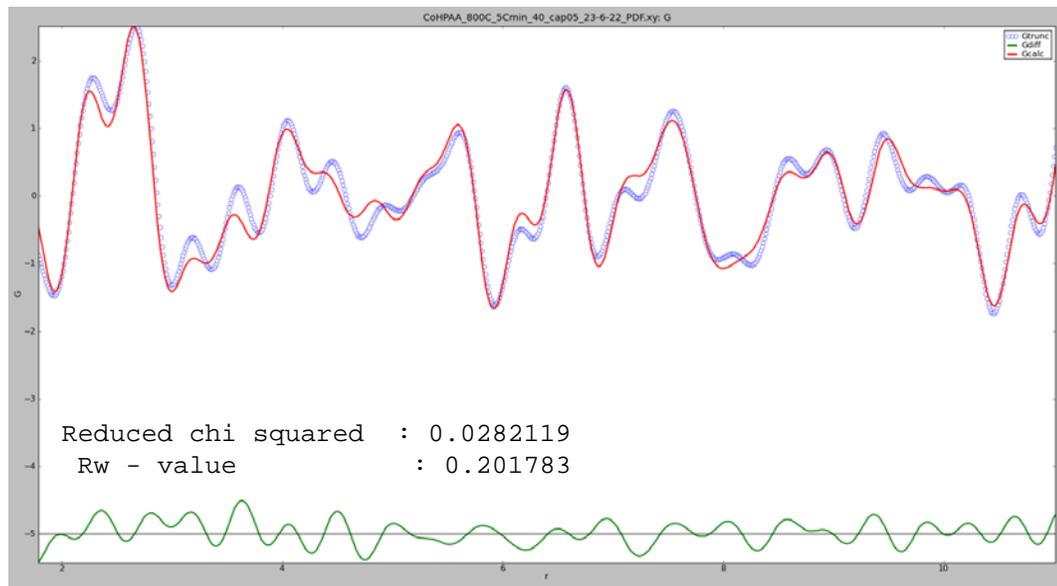


Importancia del capilar vacío

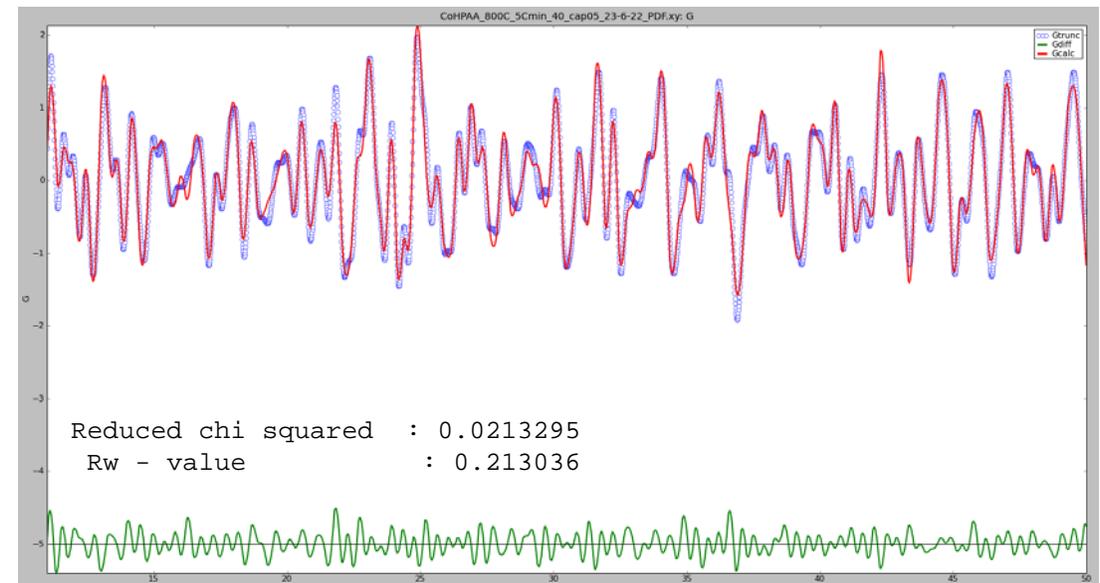


- ✓ Medir capilar vacío en iguales condiciones
- ✓ Elección del factor adecuado en la resta del fondo

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



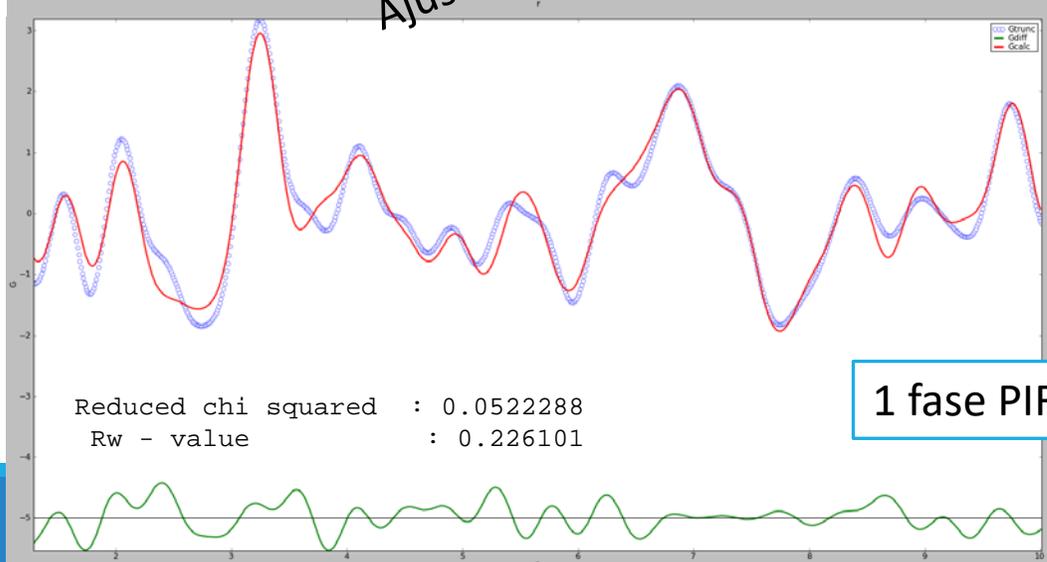
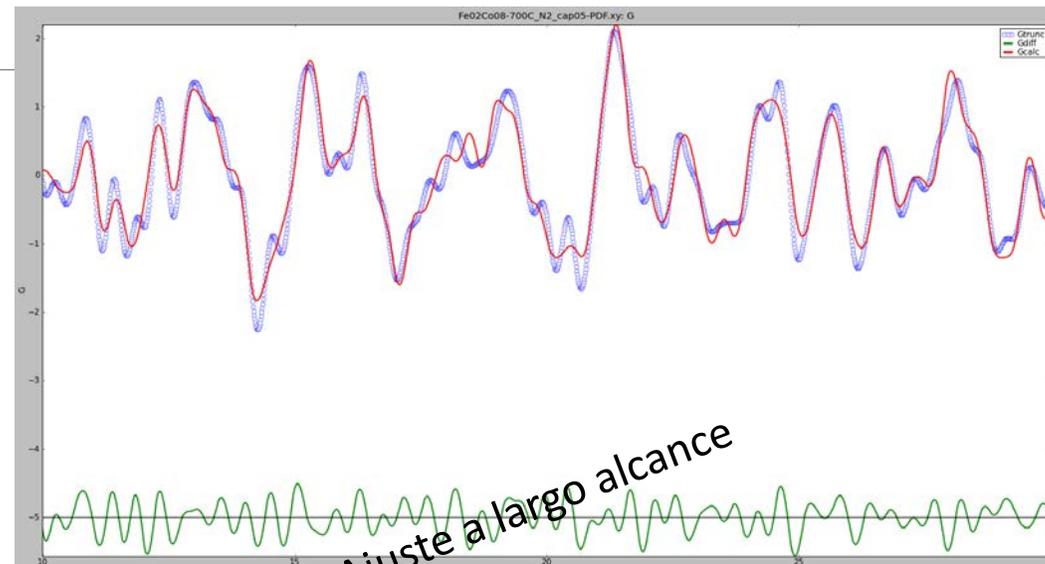
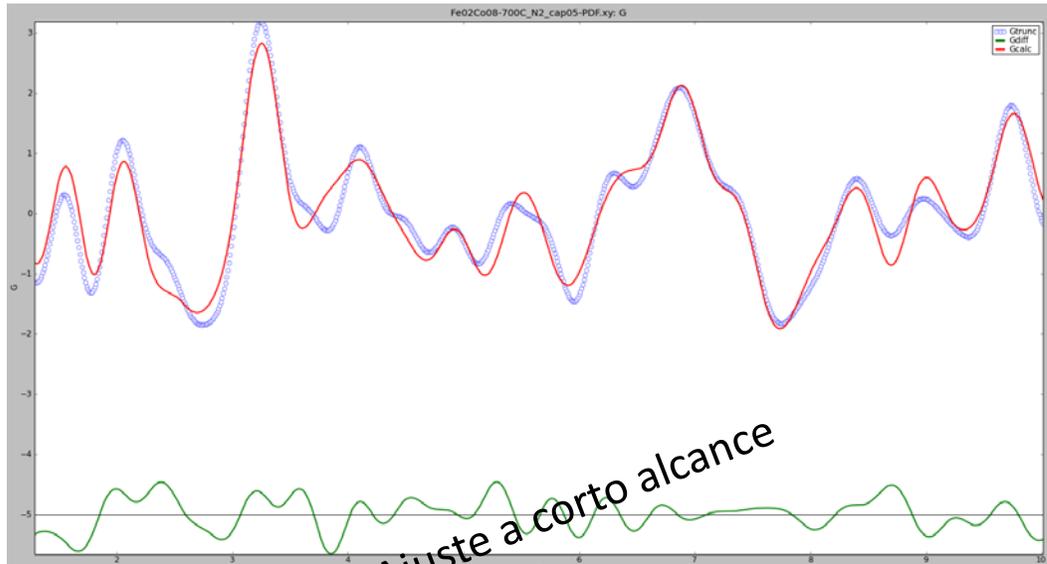
Ajuste a corto alcance



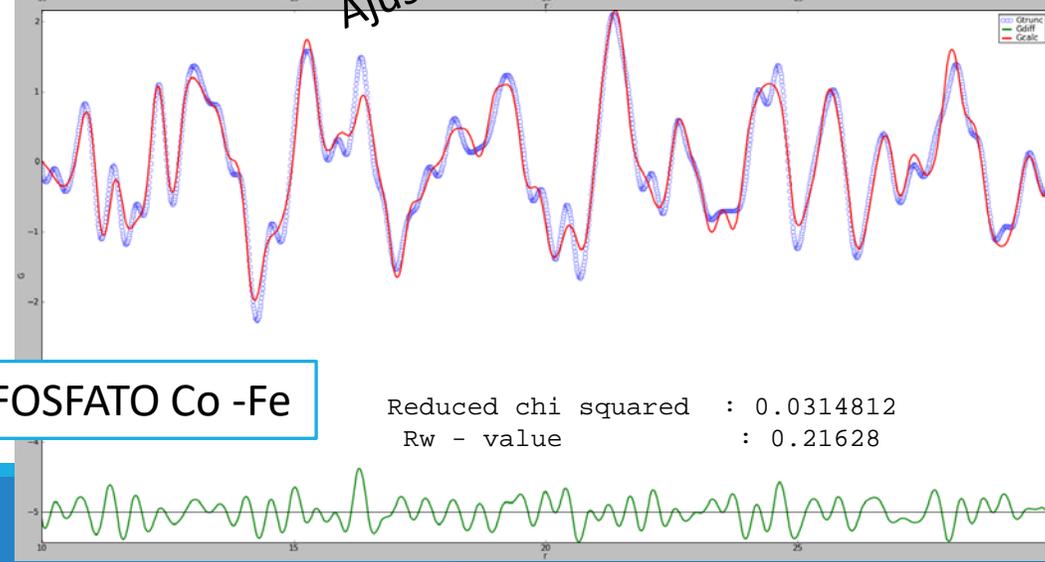
Ajuste a largo alcance

Resultados cuantitativo:
74% Co₂P
26% CoP

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



1 fase PIROFOSFATO Co -Fe

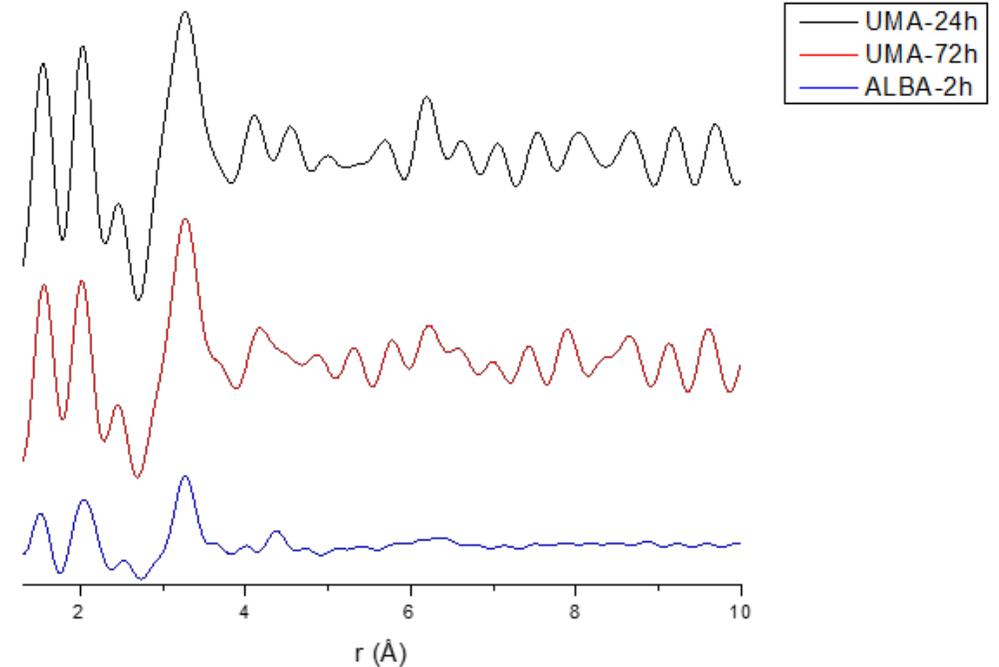
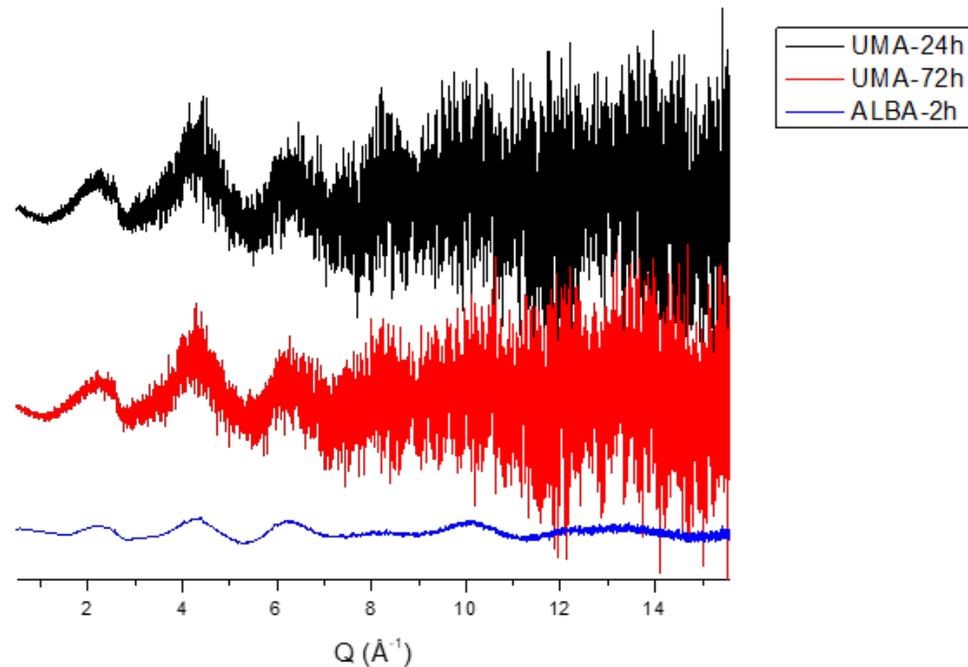


Sin afinar posiciones atómicas

Afinando posiciones atómicas

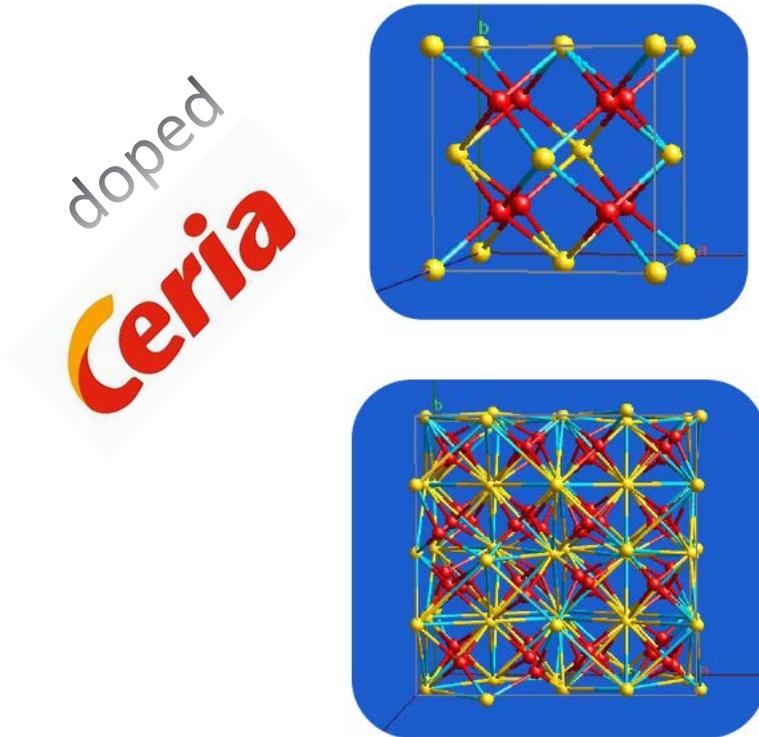
Constrains!!!

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

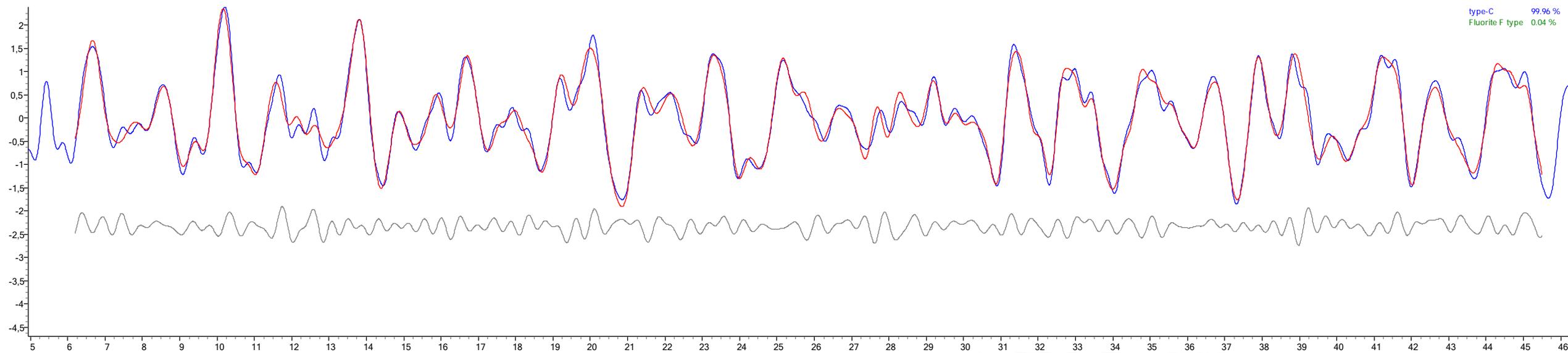
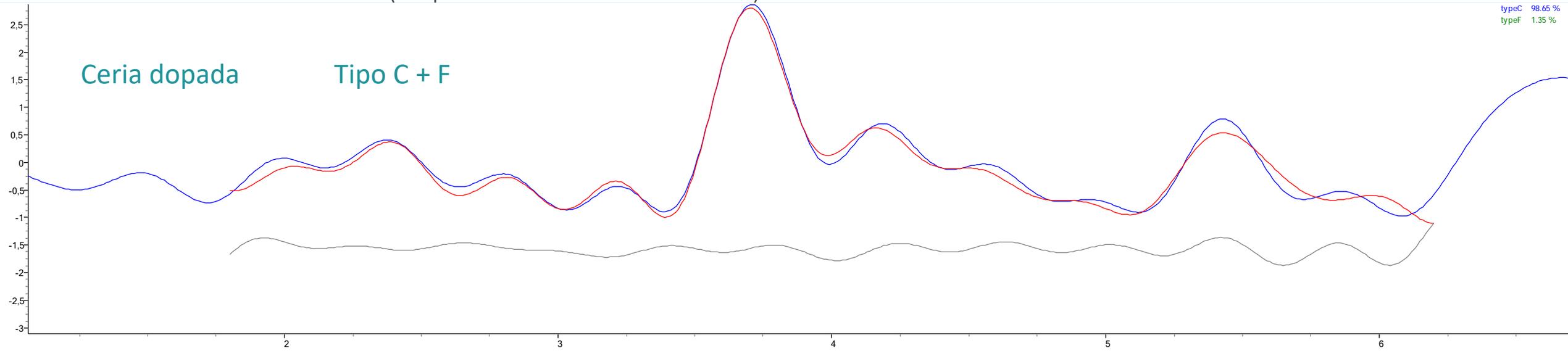
Rwp 49,7
Tipo C

Ceria dopada

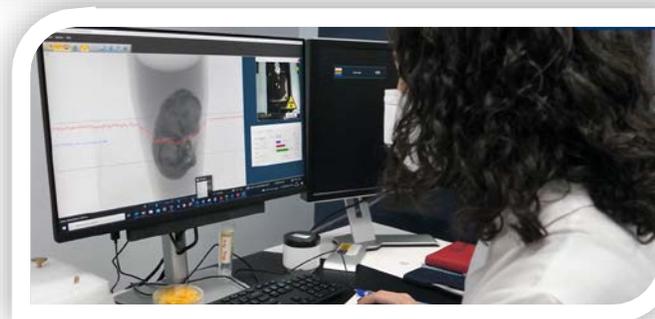
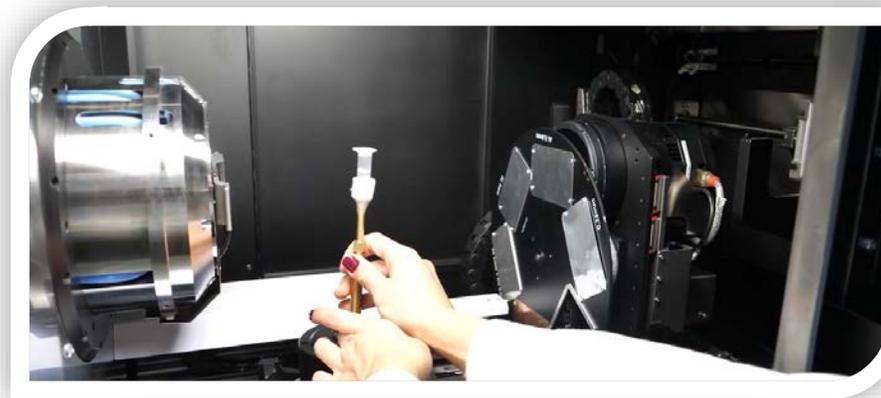
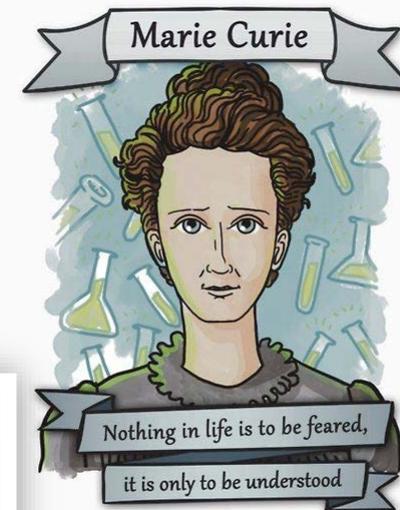
Rwp 15,2
Tipo C + F

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

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

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