



Transferencia del conocimiento: CURSOS DE FORMACIÓN EN DIFRAC.EVA y TOPAS

Experiencia desde 2001 impartiendo más de 60 cursos intensivos para empresas y universidades (modalidad on-line o presencial) en los siguientes temas:

- Introducción a la difracción de rayos-X de polvo
- Preparación de muestras por difracción de rayos-X de polvo
- Interpretación de difractogramas con Diffrac.EVA
- Bases de datos disponibles
- El método de Rietveld
- TOPAS. Introducción
- TOPAS. Análisis cuantitativo (modo GUI)
- TOPAS. Análisis cuantitativo avanzado (modo LAUNCH)
- TOPAS. Modo LAUNCH
- TOPAS. Análisis cuantitativo automatizado (modo LAUNCH)
- TOPAS. Análisis de la microestructura (modo GUI)
- TOPAS. Análisis de la microestructura avanzado (modo LAUNCH)

The collage illustrates the workflow of X-ray diffraction analysis using Diffrac.EVA and TOPAS. Key components include:

- 1. X-ray diffraction fundamentals:** Explains the diffraction phenomena, including Bragg's law and extinction conditions. It features a diagram of a crystal lattice and a table of extinction conditions.
- 4. Diffractogram interpretation with DIFFRAC.EVA (1):** Shows the process of indexing an observed diffractogram to identify a phase. It includes a diagram of a crystal structure (Cu, FCC, Fm3m, a = 3.6152 Å) and a table of indexed peaks.
- 8. Quantification of diffractograms:** Displays Rietveld refinement plots with observed data (black), fit (red), and difference (blue) curves. It includes a table of refined parameters and a list of phases.
- 3. Sample preparation:** Shows a diagram of a zero background sample holder.
- Other screenshots:** Show various diffractograms for different samples (e.g., Co 4.78897 Å, Mo 0.70932 Å, Cr 4.2897 Å) and detailed views of peak fitting and phase identification.



Características de los cursos:

- Presupuestos a medida sin compromiso
- Notas en PDF del curso y ejemplos trabajados
- Soporte bibliográfico
- Personalizados a las necesidades del cliente
- Ejemplos de casos reales
- Trabajo intensivo con difractogramas del cliente
- Apoyo científico después del curso

The collage includes several key documents:

- 1. TOPAS. Introduction 1.10 Sample preparation and analysis:** Shows images of a sample being pressed into a holder. Text: "We fill the cavity of the sample holder homogeneously." and "We lightly press the sample so that it is evenly distributed. It is necessary to hold the sample holder to the glass." Another image shows the removal of the adapter: "We remove the adapter from the sample holder."
- 7. TOPAS. Basic quantitative analysis 7.5 Microabsorption effect and corrections:** Discusses the effect of microabsorption in a two-phase system. Text: "In a two-phase system, the intensity ratio between two phases I_1/I_2 depends on the size of the particles in each phase and the difference between their absorption coefficients ($\mu_1 - \mu_2$). $\mu_1 > \mu_2$ decrease I_1/I_2 , $\mu_1 < \mu_2$ increase I_1/I_2 ." Includes a diagram of a two-phase system.
- 8. Quantification of diffractograms 8.3 Proposed problems:** Shows a table of data and a diffractogram.
- Geometrical description of the diffractometer:** A diagram showing the primary and secondary radii, receiving slit width, and receiving slit length.
- 1. TOPAS. Introduction 1.4 Working with hkl_phases:** A flowchart for diffraction profile fitting, starting with "Input data files" and leading to "Emission profile", "Background", "Add hkl phase", and "G.E.S. a.b.c.a.r.y".
- 7. TOPAS. Basic quantitative analysis 7.2 Introduction of structures:** Discusses Mullite structure: $Al_2[Al_{2-x}Si_{2+x}]O_{10-x}$, $0.25 \leq x \leq 0.4$. It is a solid solution where the end members have the composition: $2Al_2O_3 \cdot SiO_2$ Mullite 2:1 or primary $3Al_2O_3 \cdot 2SiO_2$ Mullite 3:2 or secondary. Chains of AlO_6 octahedra parallel to the c axis joined by tetrahedra (Si, Al)O₄. Includes a 3D ball-and-stick model of the structure.
- PROPERTY window of each diffractogram:** Shows a software window with a list of peaks and their parameters.
- Original diffractogram vs. Diffractogram with X-offset:** Shows two diffractograms, one with an X-offset of 0.1° and another with a displacement of -0.485 mm.
- Mo 0.70932 Å and Cr 4.2897 Å:** Labels for specific diffraction peaks.
- Chemical formula:** $(Ti_{0.4}Ni_{0.6})(Sb_{1.2}Ti_{0.8})O_6$



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2. Powder X-ray diffractometer
 2.3 Sampleholder

Effect of sample parafocality on Bragg-Brentano geometry for different 2θ angles

35.156° 2θ
 FWHM: 0.071°

Focalization circle
 Illuminated area = tangential area

sample
 sampleholder

4. Quantification of diffractograms with posed problems
 8.1 Summary of the steps to follow

2. TOPAS. Diffraction profile
 2.4 Anisotropic broadening

Sample of nanocrystalline uric acid
 Whole powder pattern fitting

1. File / Close All
 2. File / Load Files / MRI32085.RAW or MCI011070.raw / Add hkl file
 3. MRI32085.RAW or MCI011070.raw / P-3m
 4. Space Group / P-3m
 5. a (Å) / 3.14, b (Å) / 4.78 Refinings
 6. LVoI=1B
 7. LVoI=1B
 8. LVoI=1B
 9. LVoI=1B
 10. LVoI=1B

7. TOPAS. Basic quantitative analysis
 7.8 Quantification of clays

The TOPAS program allows these stacking defects to be modeled using the LAUNCH programming mode.

The initial cell parameters are the same as the regular structure except for the parameter c which is defined as the product of the parameter c and Nc which is the number of layers that are defined.

1. X-ray diffraction fundamentals
 1.4 The diffraction phenomena

Depending on the symmetry of the crystal structure, there are some reflections (hkl) that are systematically absent (extinction conditions). In addition, some reflections in certain parts of a structure can have some reflections to almost zero intensity.

these extinctions or extinction conditions are tabulated in the International Tables of Crystallography.

2. TOPAS. Diffraction profile analysis
 2.1 Basic concepts

Graphically, what we measure by X-ray diffraction does not correspond to the real structure.

Secondary particle or grain: solid material formed by one or more crystals surrounded by grain boundaries, crystalline or amorphous interface.

Scanning Electron Microscopy

Crystal: homogeneous solid material formed by atoms. The surface of the crystal is considered a 2D object.

Phase A: amorphous
 Phase B: crystalline, large crystallite size

2. TOPAS. Diffraction profile analysis
 2.5 Proposed examples

The Williamson-Hall plot for CeO₂ presents almost no slope as one should expect for this sample without microstrain.

The same plot for sample α-Fe with a mechanical treatment shows an slope as a consequence of the microstrain induced.

Diffractogram interpretation with DIFFRAC.EVA (1)
 Working with patterns

In this case all reflections are generated for a cubic cell with a: 5.4308Å, regardless of the extinctions.

Diffractogram interpretation with DIFFRAC.EVA (1)
 Sample stability: if a sample is scheduled so that a diffractogram is measured throughout the analysis, the diffractograms can be summed with a long acquisition time.