



# The transfer of knowledge: DIFRAC.EVA and TOPAS training courses

Experience since 2001 teaching more than 60 intensive courses for companies and universities (online or in-person) on the following topics:

- Introduction to powder X-ray diffraction
- Sample preparation for powder X-ray diffraction
- Interpretation of diffractograms with Diffrac.EVA
- Available databases
- The Rietveld method
- TOPAS. Introduction
- TOPAS. Quantitative analysis (GUI mode)
- TOPAS. Advanced quantitative analysis (LAUNCH mode)
- TOPAS. LAUNCH mode
- TOPAS. Automated quantitative analysis (LAUNCH mode)
- TOPAS. Microstructure Analysis (GUI mode)
- TOPAS. Advanced Microstructure Analysis (LAUNCH mode)

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 (zero intensity). In addition, positions of a structure can cause some reflections to have almost zero intensity.

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

4. Diffractogram interpretation with DIFRAC.EVA (1)  
 4.6 Working with patterns

Observed diffractogram

Calculated diffractogram

INDEXING

Cell parameters: a, b, c, α, β, γ

1. X-ray diffraction fundamentals  
 1.4 The diffraction phenomena

The position of the diffraction peak, or interplanar spacing, of a crystal can be determined by the crystal parameters:

Cubic  $\frac{1}{d^2} = \frac{1}{a^2} \left( h^2 + k^2 + l^2 \right)$   
 Tetragonal  $\frac{1}{d^2} = \frac{1}{a^2} \left( h^2 + k^2 \right) + \frac{l^2}{c^2}$   
 Hexagonal  $\frac{1}{d^2} = \frac{4}{3} \frac{h^2 + hk + k^2}{a^2} + \frac{l^2}{c^2}$   
 Trigonal  $\frac{1}{d^2} = \frac{1}{a^2} \left( h^2 + k^2 + l^2 \right)$   
 Rhombohedral  $\frac{1}{d^2} = \frac{1}{a^2} \left( h^2 + k^2 + l^2 \right) + \frac{2hk}{a^2} \cos \alpha + \frac{2hl}{a^2} \cos \beta + \frac{2kl}{a^2} \cos \gamma$   
 Monoclinic  $\frac{1}{d^2} = \frac{1}{a^2} \left( h^2 + k^2 + l^2 \right) + \frac{2hk}{a^2} \cos \alpha + \frac{2hl}{a^2} \cos \beta + \frac{2kl}{a^2} \cos \gamma$

8. Quantification of diffractograms with customer problems  
 8.3 Proposed problems

8. Quantification of diffractograms  
 8.3 Proposed problems

3. Sample preparation  
 3.4 Zero background sampleholder

Effect of X-offset and Displacement corrections

Low angles

High angles

Original diffractogram

Diffractogram with X-offset: 0.1°

Displacement: -0.485 mm

Co 4.78897 Å

Mo 0.70932 Å

Cr 4.2897 Å

Cu (FCC) Fm3m  
 a = 3.6152 Å



**Course features:**

- Customized quotes without obligation
- Course notes in PDF format and worked examples
- Bibliographic support
- Customized to the client's needs
- Real case examples
- Intensive work with client's diffractograms
- Scientific support after the course

**1. TOPAS. Introduction**  
 1.10 Sample preparation and analysis

We fill the cavity of the sample holder homogeneously.

We lightly press the sample so that it is evenly distributed. It is necessary to hold the sample holder to the glass.

We remove the adapter from the sample holder.

We remove the compression accessory.

**Geometrical description of the diffractometer**

Primary Radius, Secondary Radius, Receiving Slit width, Receiving Slit length

**7. TOPAS. Basic quantitative analysis**  
 7.5 Microabsorption effect and corrections

Effect of microabsorption

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  $I_1/I_2$

**8. Quantification of diffractograms**  
 8.3 Proposed problems

**TOPAS. Introduction**  
 1.4 Working with hkl phases

Diffraction profile fitting

Input data files → Emission profile → Background → Add hkl phase → G.E.S. a.b.c.a.r.y → Environmental Parameters? → YES → Select Peak Type (P, G, D, P, S) → Error standard parameters and fit graph → NO → Select Peak Type (P, G, D, P, S) → Error standard parameters and fit graph → Don't use Crystallite Size → Run (F5) → Zero error → U.V.W.X

**Basic quantitative analysis**  
 Introduction of structures

Parameters can be referred to mathematical case of Goethite,  $FeO(OH)$ . Fe can be substituted by Al, leaving the formula as  $Fe_0.7Al_0.3(OH)$  and is a linear function (Vegard's Law) parameter b:

**Mullite structure:**  
 $Al_4Al_{2x}Si_{2-2x}O_{10+x}$   
 $0.25 \leq x \leq 0.4$

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)O<sub>4</sub>.

**PROPERTY window of each diffractogram.** It is located under the diffractogram and the area under the curve on the relationship between these two areas.

**Crystallographic data for  $(Ti_{0.4}Ni_{0.6})(Sb_{1.2}Ti_{0.8})O_6$**

Formula	Subfile	Asymmetric Unit
Crystal Data		
Molecular weight	334.8	
Volume (Å <sup>3</sup> )	197.45	
Dn	5.63	
Dm	5.63	
ρ	5.63	
1/ρ	0.1775	
1/ρ <sub>air</sub>	6.140	

**TOPAS Parameters**

Parameter	Value	Units	Min	Max	Step	Fixed
Scale	1.00000					
Offset	0.00000					
Zero	0.00000					
Gamma	0.00000	deg				
Gamma2	0.00000	deg				
Gamma3	0.00000	deg				
Gamma4	0.00000	deg				
Gamma5	0.00000	deg				
Gamma6	0.00000	deg				
Gamma7	0.00000	deg				
Gamma8	0.00000	deg				
Gamma9	0.00000	deg				
Gamma10	0.00000	deg				
Gamma11	0.00000	deg				
Gamma12	0.00000	deg				
Gamma13	0.00000	deg				
Gamma14	0.00000	deg				
Gamma15	0.00000	deg				
Gamma16	0.00000	deg				
Gamma17	0.00000	deg				
Gamma18	0.00000	deg				
Gamma19	0.00000	deg				
Gamma20	0.00000	deg				
Gamma21	0.00000	deg				
Gamma22	0.00000	deg				
Gamma23	0.00000	deg				
Gamma24	0.00000	deg				
Gamma25	0.00000	deg				
Gamma26	0.00000	deg				
Gamma27	0.00000	deg				
Gamma28	0.00000	deg				
Gamma29	0.00000	deg				
Gamma30	0.00000	deg				
Gamma31	0.00000	deg				
Gamma32	0.00000	deg				
Gamma33	0.00000	deg				
Gamma34	0.00000	deg				
Gamma35	0.00000	deg				
Gamma36	0.00000	deg				
Gamma37	0.00000	deg				
Gamma38	0.00000	deg				
Gamma39	0.00000	deg				
Gamma40	0.00000	deg				
Gamma41	0.00000	deg				
Gamma42	0.00000	deg				
Gamma43	0.00000	deg				
Gamma44	0.00000	deg				
Gamma45	0.00000	deg				
Gamma46	0.00000	deg				
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Gamma50	0.00000	deg				
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Gamma79	0.00000	deg				
Gamma80	0.00000	deg				
Gamma81	0.00000	deg				
Gamma82	0.00000	deg				
Gamma83	0.00000	deg				
Gamma84	0.00000	deg				
Gamma85	0.00000	deg				
Gamma86	0.00000	deg				
Gamma87	0.00000	deg				
Gamma88	0.00000	deg				
Gamma89	0.00000	deg				
Gamma90	0.00000	deg				
Gamma91	0.00000	deg				
Gamma92	0.00000	deg				
Gamma93	0.00000	deg				
Gamma94	0.00000	deg				
Gamma95	0.00000	deg				
Gamma96	0.00000	deg				
Gamma97	0.00000	deg				
Gamma98	0.00000	deg				
Gamma99	0.00000	deg				
Gamma100	0.00000	deg				

**Original diffractogram**  
 Diffractogram with X-offset: 0.1°  
 Diffractogram with Displacement: -0.485 mm

**Mo 0.70932 Å**  
**Cr 4.2897 Å**



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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

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

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 (extinctions). In addition, some reflections in certain parts of a structure can have some reflections to almost zero intensity.

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

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P112/n  
UNIQUE AXIS C: CELL CHOICE 2

Origin of axes: x, y, z

Generators selected: (1) (0,0,1); (2) (1,0,0); (3) (0,1,0)

Positions: General, Special

Conditions: General, Special

Reflection conditions: hkl: 0 k l 2n  
hkl: 0 k + 1/2 l 2n  
hkl: 0 k + 1/2 l 2n  
hkl: 0 k + 1/2 l 2n

2. TOPAS. Diffraction profile analysis  
2.4 Anisotropic broadening

Sample of nanocrystalline brucite

Whole powder pattern fitting

- File / Close All
- File / Load Files / MRI32085.RAW or MCI011070.raw / Add hkl file
- MRI32085.RAW or MCI011070.raw / P-3m
- Space Group / P-3m
- a (Å) / 3.14, b (Å) / 4.78 Refinings
- LVoIaIB

δ<sub>001</sub>: 3.47 g/cc  
δ<sub>010</sub>: 1.89 g/cc

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.

Cell parameters:  
a, b, C, α, β, γ  
Spatial group:  
C<sub>2</sub>/c  
C<sub>2</sub>/c

INDEXING  
I obs  
2θ obs

STRUCTURE RESOLUTION  
F<sub>hkl</sub>

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.

(200) Cu K<sub>α1</sub> + K<sub>α2</sub>  
(400) Cu K<sub>α1</sub> + K<sub>α2</sub>  
(111) Cu K<sub>α1</sub> + K<sub>α2</sub>  
(400) W L<sub>α1</sub>  
(400) Cu K<sub>α1</sub>

simple stability: if a sample is scheduled so that a diffractogram is measured throughout the analysis, the diffractograms can be measured with a long acquisition time.

time line

2. TOPAS. Diffraction profile analysis  
2.5 Proposed examples

The Williamson-Hall plot for CeO<sub>2</sub> 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.

FWHM<sub>int</sub> cos(θ)

CeO<sub>2</sub>, h=0k<sub>1</sub>l=0  
α-Fe ball milling, MRI01776