Karlsruhe Institute of Technology

Convolution based Profile Fitting

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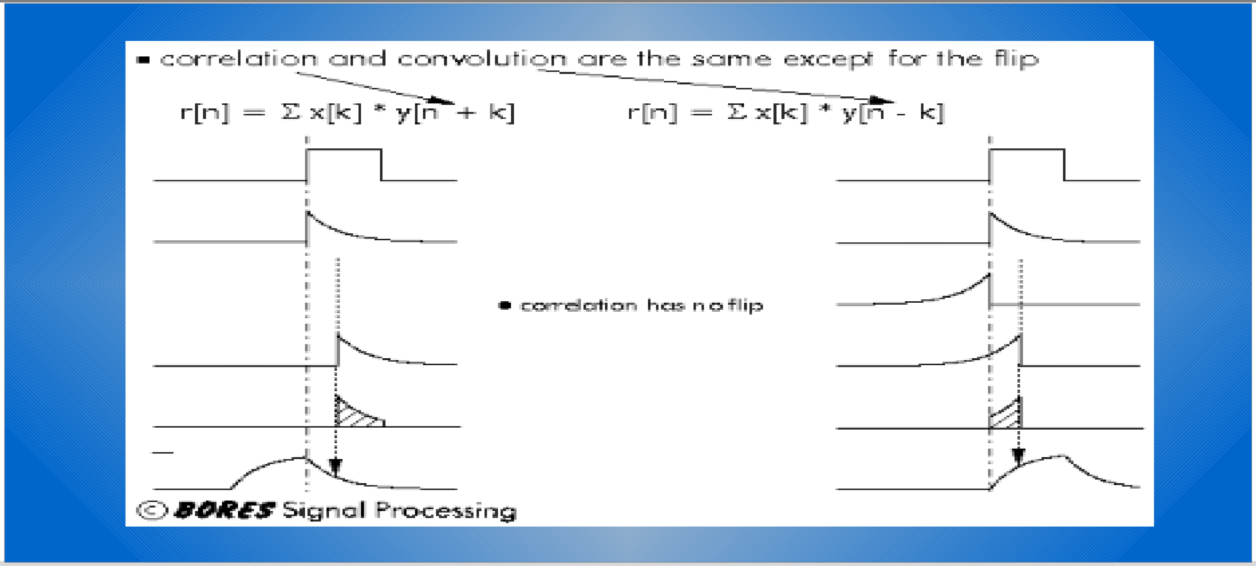
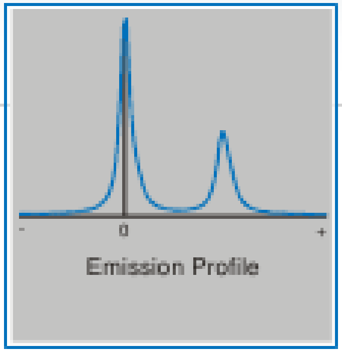


Overview

- what is convolution
- instrumental contribution

  how can we describe the influence of a slit

  on the diffractogram ?
- examples



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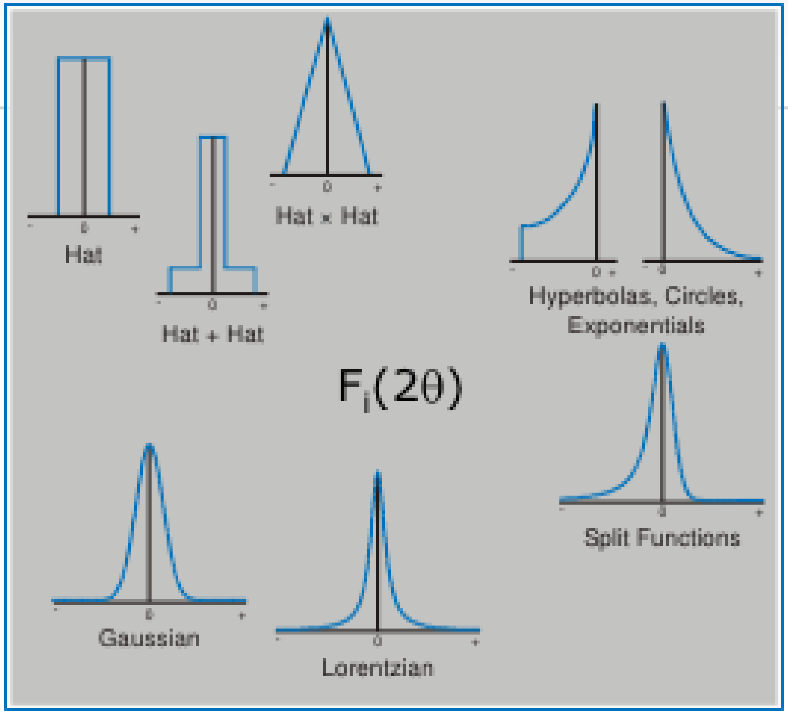
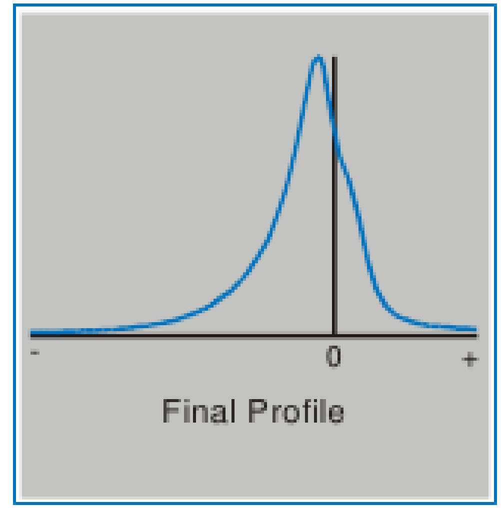


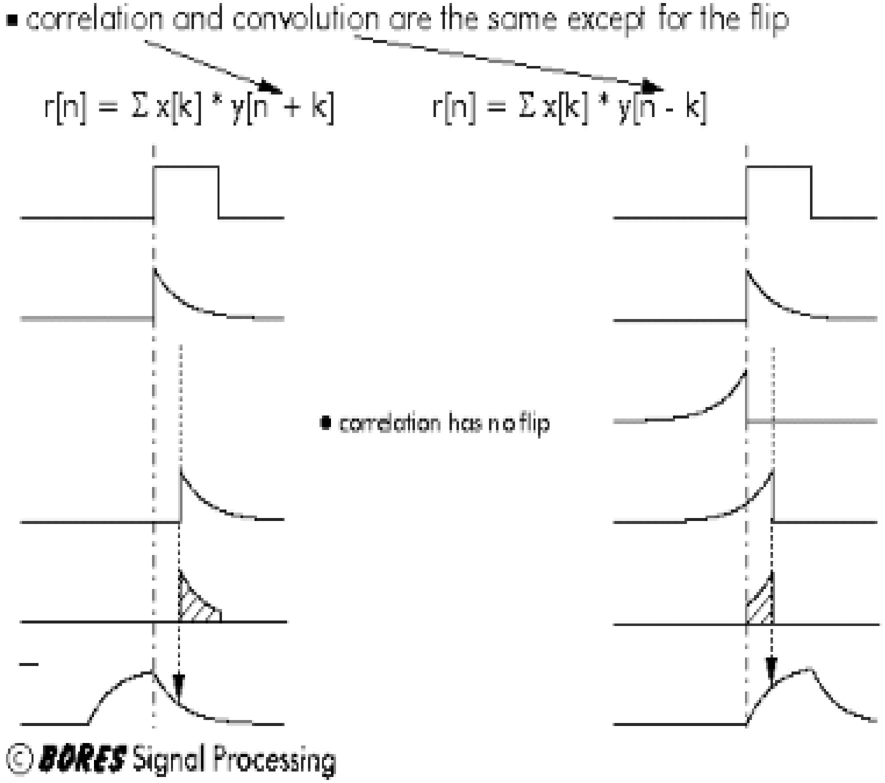


$Y(2\theta)$

$$Y(2\theta) = F_1(2\theta) \times F_2(2\theta) \times \ldots \times F_i(2\theta) \times \ldots \times F_n(2\theta)$$

Convolution

$$y(t)=\int_{-\infty}^{\infty} h(s)\,x(t-s)\,ds$$



https://en.wikipedia.org/wiki/Convolution

TOPAS features a direct convolution approach with all parameters refinable

Choice of empirical or physically meaningful profile fitting

Virtually any peak shape, angle dependence as well as hkl dependence (anisotropic line broadening) can normally be described using a minimum number of profile parameters

Represents a simplified integral breadth approach for size-strain analysis

Methodology

1. Physically meaningless parametrization of observed line profile shapes
2. Explicit discrimination of instrument and sample contributions to observed line profile shapes

$Y(2\theta) = (W \times G) \times S$, with $(W \times G) = I$

(Well known approach latest since Klug & Alexander, 1954)

a) Measured I: Based on a standard reference material

b) Calculated I: Fundamental Parameters Approach

Y(2θ): Observed line profile shape, I: Instrument function, W: Source emission profile,
G: Geometric instrument aberations

Profile shape functions (PSFs) are generated by convoluting functions together to form the observed profile shape:

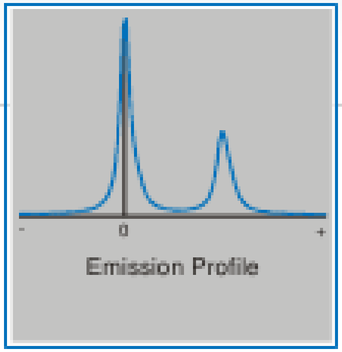
$Y(2\theta) = F_1(2\theta) \times F_2(2\theta) \times ... \times F_i(2\theta \times ... \times F_n(2\theta)$

In general any combination of appropriate functions for $F_i(2\theta)$ may be used

The functions $F_i(2\theta)$ can be interpreted as the aberration functions of the diffractometer: FPA

In simple terms, convolution can be understood as "blending" one function with another, producing a kind of very general "moving average".

The convoluted function is obtained by setting down the origin of the first function in every possible position of the second, multiplying the values of both functions in each position, and taking the sum of all operations.



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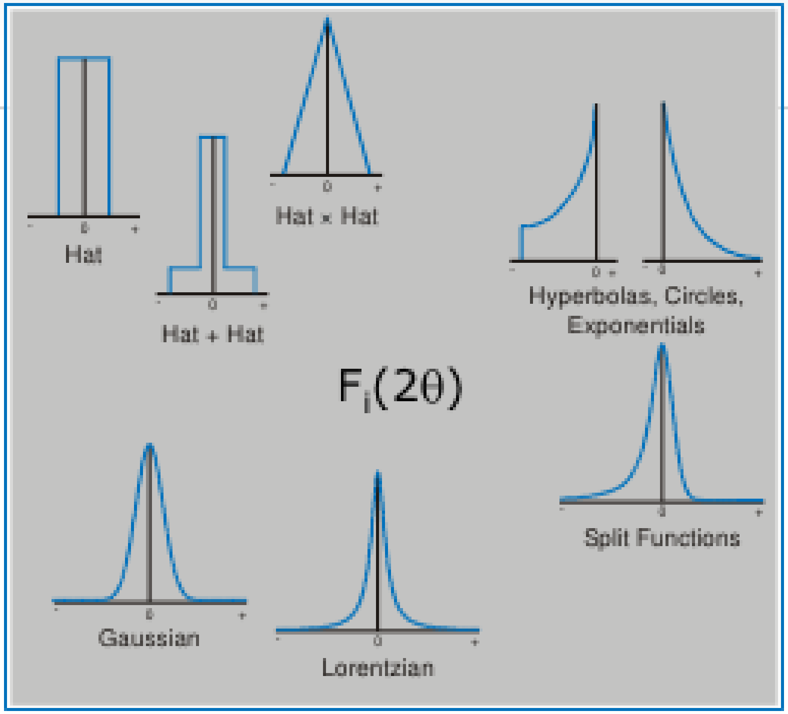

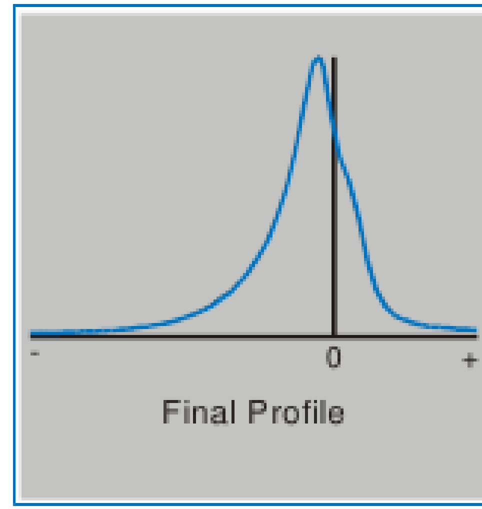

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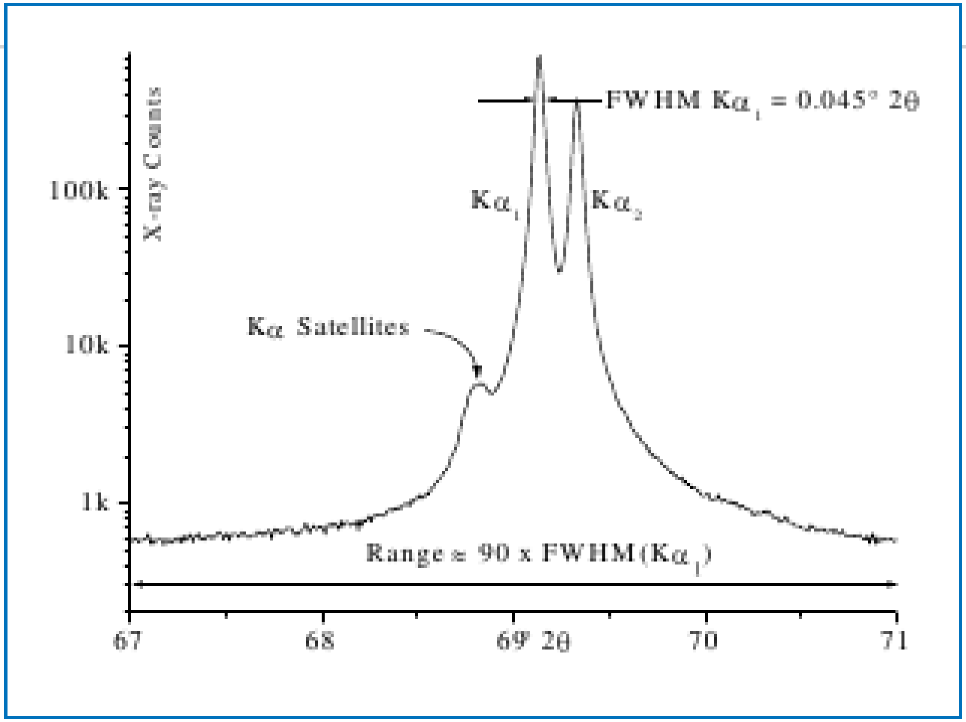
Emission Profile

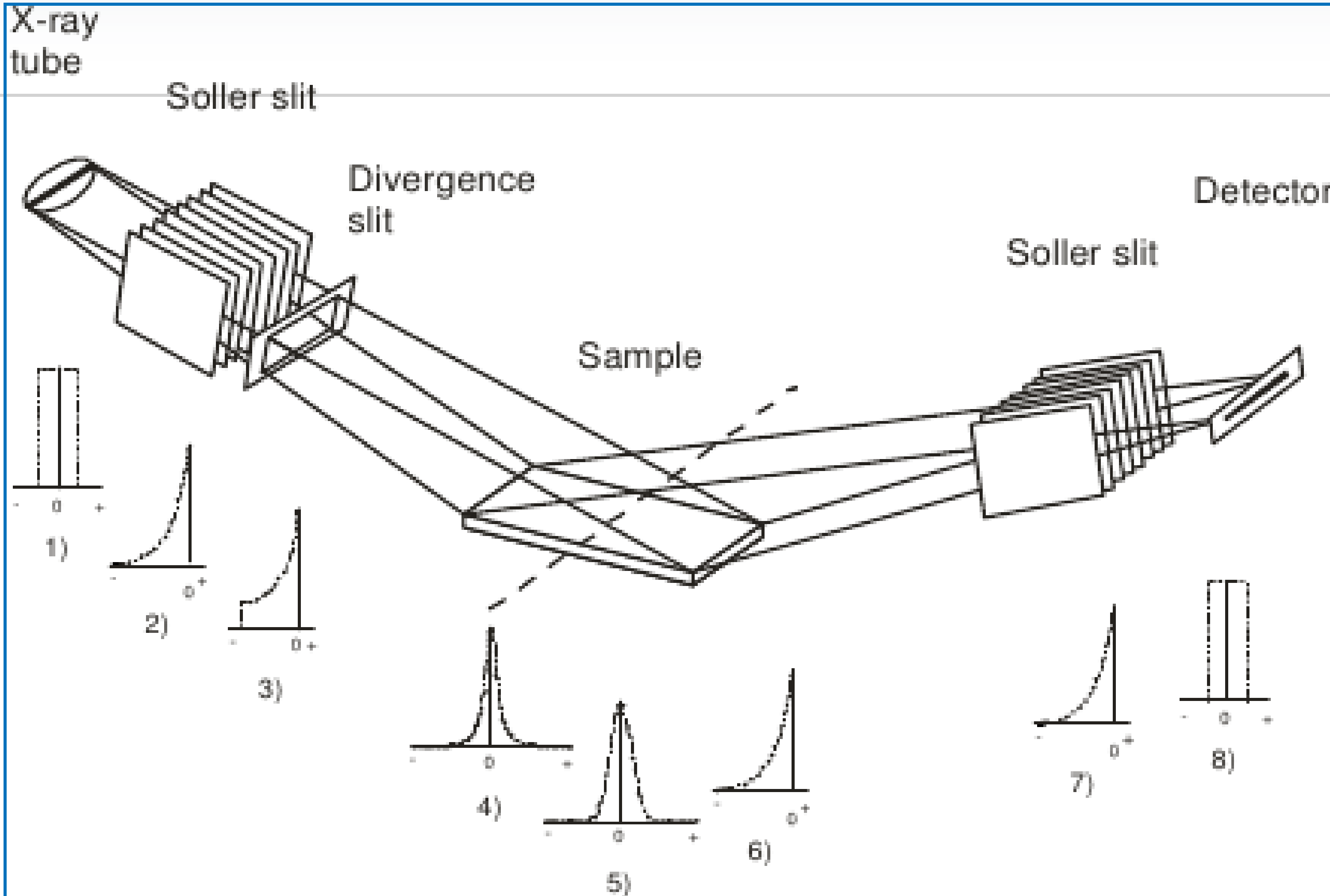
- Cu Kα spectrum is better represented using five Lorentzians

⇨ CuKa5_Berger.lam



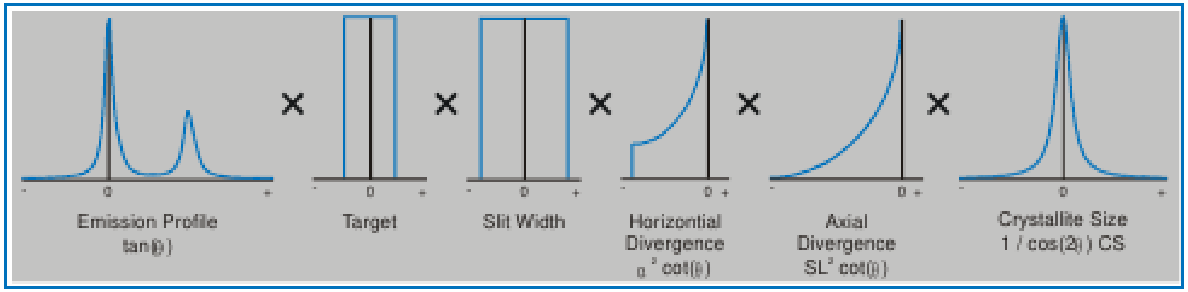
From Cheary et al. (2004)

Example:
Bragg-Brentano GeometryBeam Path and Optics



Laboratory X-ray data (D8 ADVANCE):

- Instrument function (FPA)
- 1 Lorentzian function: Crystallite size broadening
- Total number of refineable parameters: 1



- Knowledge of the most common contributions to line profile shapes and their dependence on angle helps, e.g. for laboratory instruments:

Contribution	Convolution	Angular dependence
Detector (Slit)	Hat	Constant
Crystallite size	Lorentzian	1/cos(Th)
Strain	Gaussian	Tan(Th)
Axial divergence	Circle	-1/Tan(Th)

- Do it yourself: Trial and error
  - Two back-to-back circle or exponential functions convoluted on top of a Voigt function (e.g. David & Jorgensen, 1993), each of which parametrized with appropriate dependence on angle and maybe hkl, will fit virtually anything

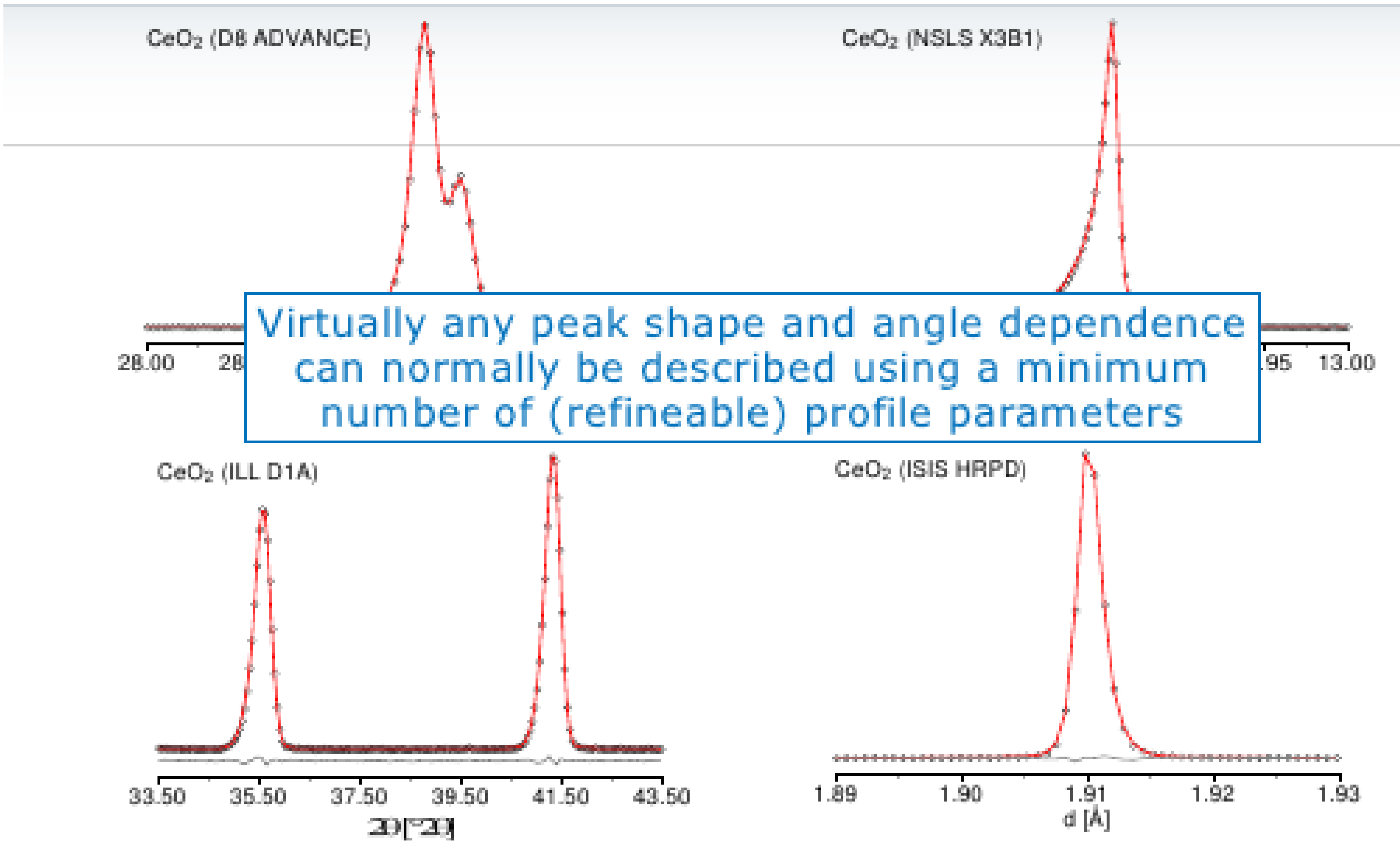
CPD Size-Strain Round Robin – CeO_2
Balzar et al. (2004)

TOPAS results submitted (Kern, A.)

	L_{Vol} [nm]	Microstrain
Measured instrument function		
D8 ADVANCE	23.43 (0.08)	0.0149 (0.0014)
NSLS X3B1	23.72 (0.08)	0.0307 (0.0014)
ESRF BM16	22.59 (0.05)	0.0143 (0.0010)
ILL D1A	23.29 (0.18)	0.0273 (0.0034)
NCNR BT1	23.88 (0.34)	0.0259 (0.0052)
ISIS HRPD	22.93 (0.06)	0.0193 (0.0021)
Calculated instrument function (FPA)		
D8 ADVANCE	22.59 (0.08)	0.0149 (0.0014)
Balzar (2001)	**22.60 (0.90)**	**"nearly strain free"**

Take home-message....

Virtually any peak shape and angle dependence can normally be described using a minimum number of (refineable) profile parameters



Size-Strain Round Robin, Balzar (2004); "sharp data".

Literature

Kern, A. (2004):
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Cheary, R.W., Coelho, A.A. & Cline, J.P. (2004): Fundamental Parameters Line Profile Fitting in Laboratory Diffractometers. J. Res. Natl. Inst. Stand. Technol., 109, 1-25.

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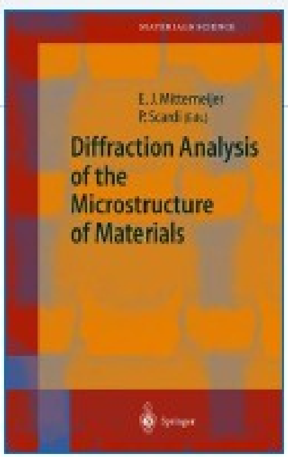

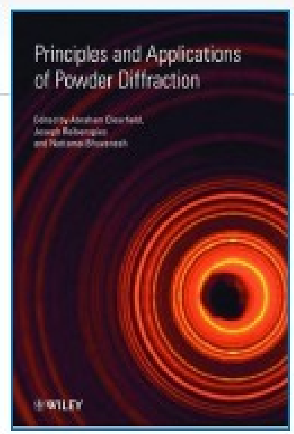

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