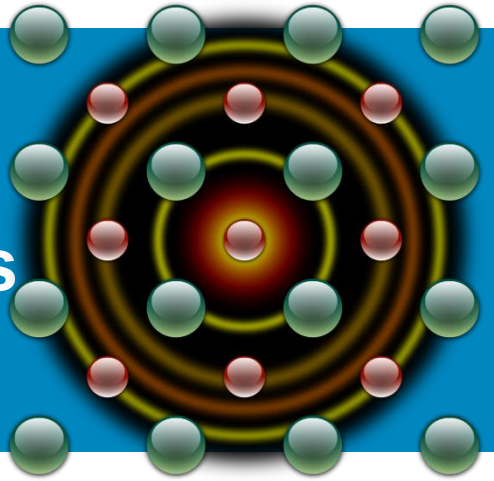
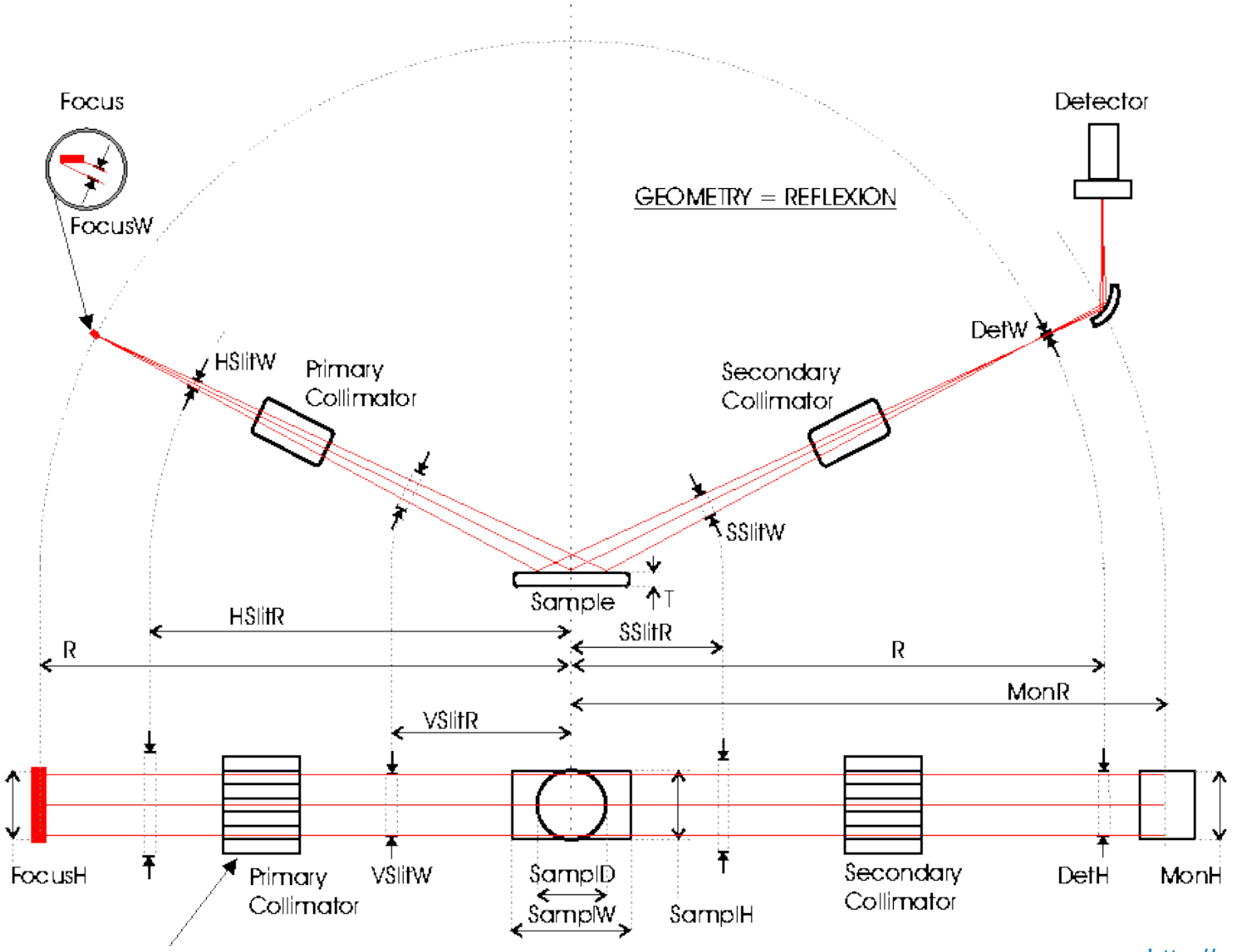
Lesson 2
Instrument Configurations
for Profex / BGMN



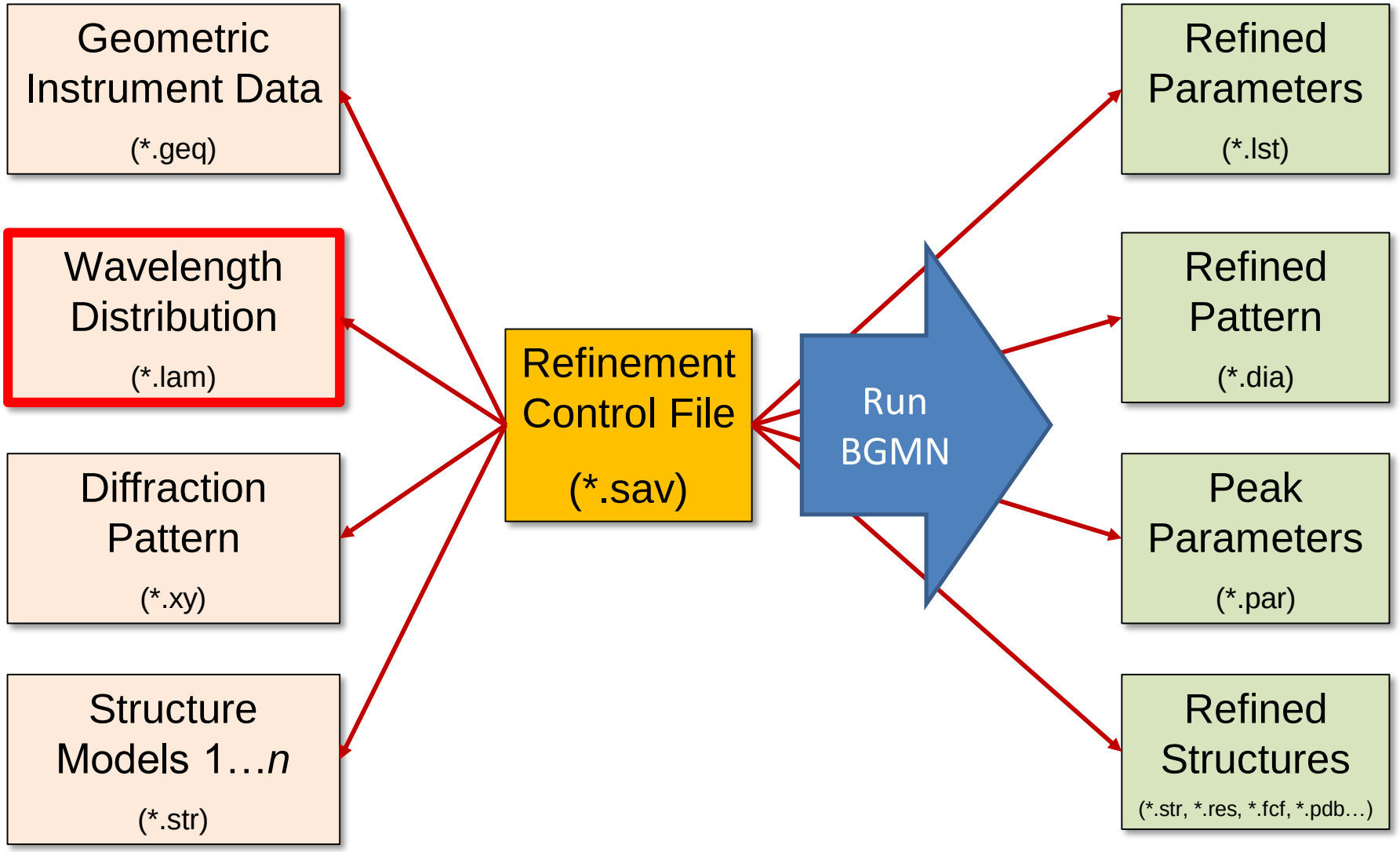
Nicola Döbelin
RMS Foundation, Bettlach, Switzerland

Fundamental Parameters Approach



http://www.bgmn.de

BGMN: Wavelength Distribution



BGMN: Wavelength Distribution

- Charakteristic X-radiation is a physical constant.
- All characteristic spectra for a certain target element look the same.
- Values can be taken from literature.

BUT:

- Filters change the spectrum recorded by the detector:
  - Kbeta filter
  - Monochromator crystal (primary or secondary beam)
  - Digital filter (energy dispersive detector)

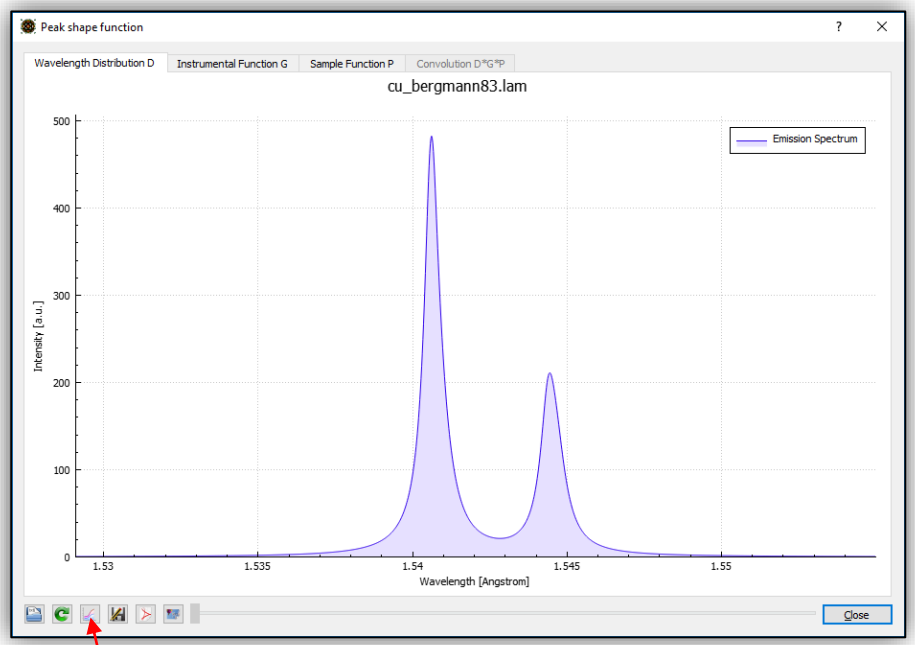
BGMN: Wavelength Distribution

cu_bergmann83.lam | CU.LAM

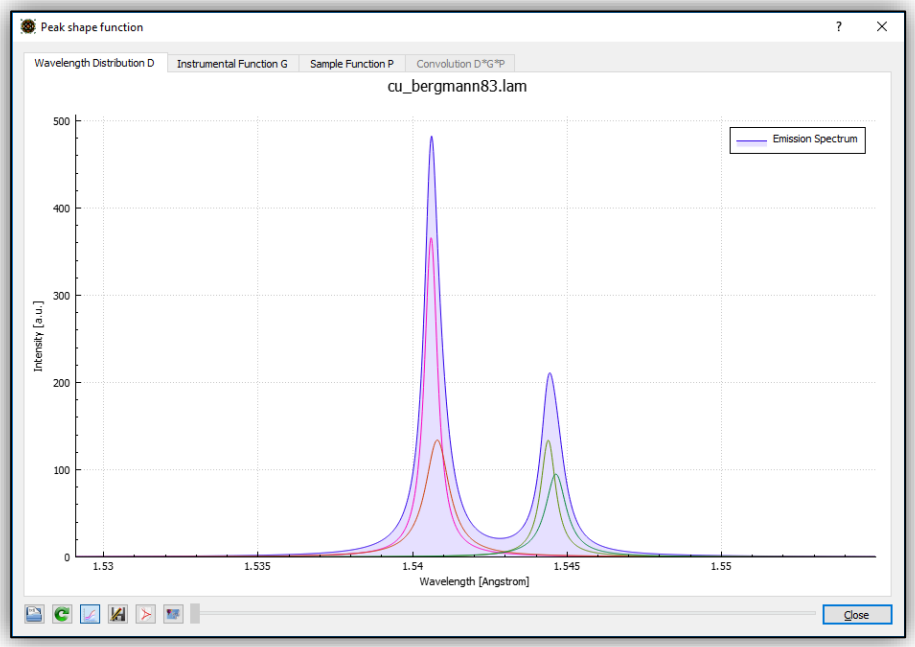
```
% Cu-K-alpha doublet as measured in october 1983 by J. Bergmann
ILAM=4 TITEL=CU_vom_Oktober_1983
0.401939 6.49098 0.0010981
0.266445 6.49013 0.0019875
0.167844 6.47504 0.0012544
0.163773 6.47400 0.0017246
```

} Lorentzian Curves

In …/Profex-BGMN-Bundle/BGMNwin/*.lam



Show sub-curves



BGMN: Wavelength Distribution

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0.266445 6.49013 0.0019875
0.167844 6.47504 0.0012544
0.163773 6.47400 0.0017246
```

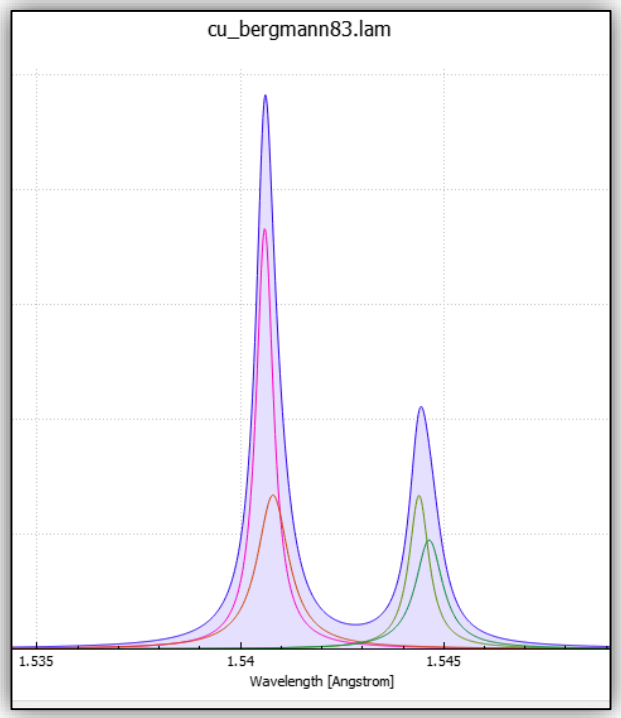
Lorentzian Curves

Half width [1/nm]

Center inverse Wavelength [1/nm]

Relative Intensity

ILAM = number of curves

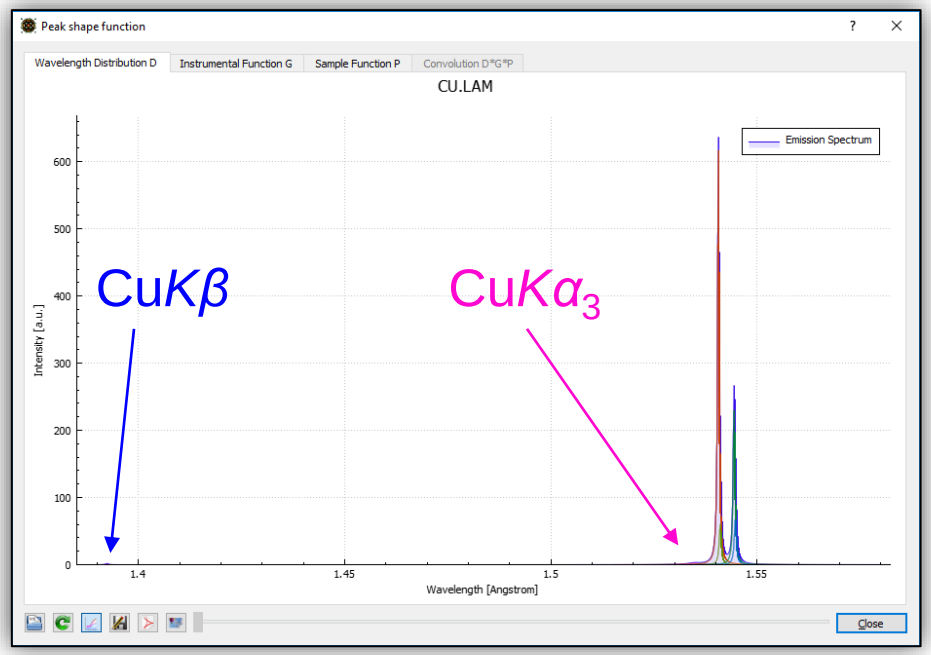


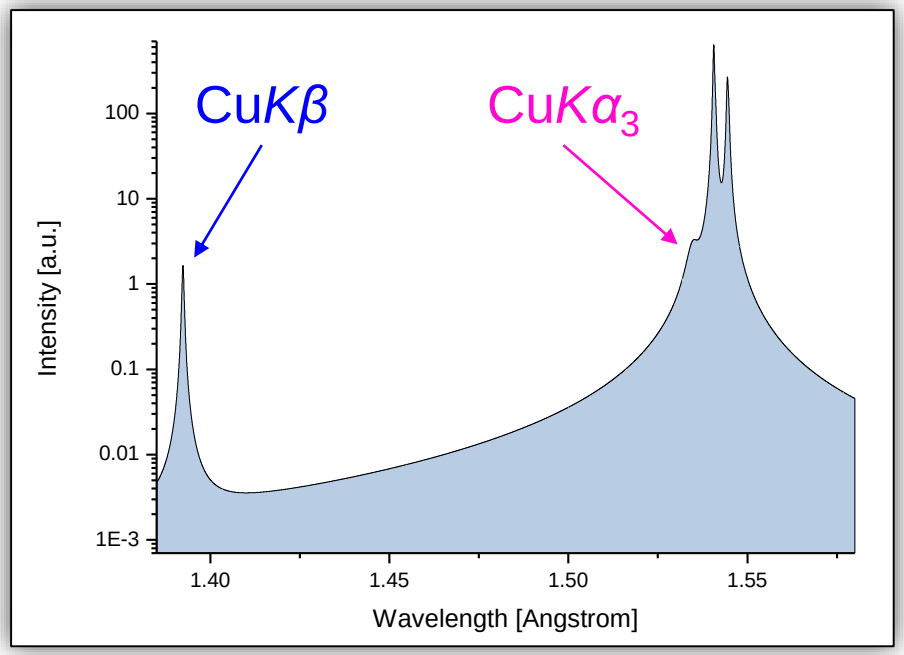
BGMN: Wavelength Distribution

cu_bergmann83.lam | CU.LAM

```
% Berger CuK1,2,3 + Kb XPERT BERN
ILAM=6
0.01586*ifthenelse(ifdef(alpha3ratio),(alpha3ratio)/0.01586,1)   6.51571   0.00783 % CaKa3
0.56768*ifthenelse(ifdef(alpha2ratio),(1-alpha2ratio)/0.64369,1)   6.49099   0.00092 % CuKa1 (1)
0.07601*ifthenelse(ifdef(alpha2ratio),(1-alpha2ratio)/0.64369,1)   6.48905   0.00126 % CuKa1 (2)
0.25107*ifthenelse(ifdef(alpha2ratio),(alpha2ratio)/0.35631,1)   6.47496   0.00109 % CuKa2 (1)
0.08688*ifthenelse(ifdef(alpha2ratio),(alpha2ratio)/0.35631,1)   6.47366   0.00130 % CuKa2 (2)
0.00249*ifthenelse(ifdef(betaratio),(betaratio)/0.00249,1)   7.18206   0.00151 % CuKb

```





BGMN: Wavelength Distribution

cu_bergmann83.lam | CU.LAM

```
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0.07601*ifthenelse(ifdef(alpha2ratio),(1-alpha2ratio)/0.
0.25107*ifthenelse(ifdef(alpha2ratio),(alpha2ratio)/0.35
0.08688*ifthenelse(ifdef(alpha2ratio),(alpha2ratio)/0.35
0.00249*ifthenelse(ifdef(betaratio),(betaratio)/0.00249,
```

If «betaratio» is defined:

$$Intensity = 0.00249 \cdot \frac{betaratio}{0.00249}$$

Else:

$$Intensity = 0.00249 \cdot 1$$

RMS_Foundation_4230_180528_082630.dia | RMS_Foundation_4230_180528_082630.sav | RMS_

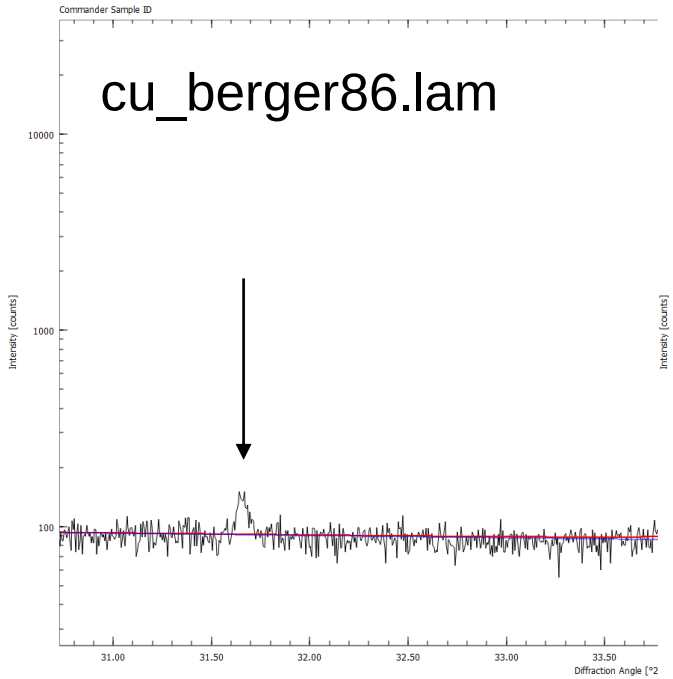
```
% SampleID: Commander Sample ID
% Theoretical instrumental function
VERZERR=RMS-D8-ADS-15-LynxEyeXE.geq
% Wavelength
LAMBDA=CU
% Phases
STRUC[1]=Al2O3-Corundum.str
% Measured background
UNT=RMS-D8-ADS-15-LynxEyeXE-bkgr.xy
RU=10
% Measured data
VAL[1]=RMS_Foundation_4230_180528_082630.xy
% Minimum Angle (2theta)
WMIN=10
% Maximum Angle (2theta)
WMAX=100
% Result list output
LIST=RMS_Foundation_4230_180528_082630.lst
% Peak list output
OUTPUT=RMS_Foundation_4230_180528_082630.par
% Diagram output
DIAGRAMM=RMS_Foundation_4230_180528_082630.dia
% Global parameters for zero point and sample displacement
EPS1=0
PARAM[1]=EPS2=0_-0.01^0.01
EPS3=0
alpha3ratio=0.020
betaratio=0.005
NTHREADS=8
PROTOKOLL=Y
SAVE=N

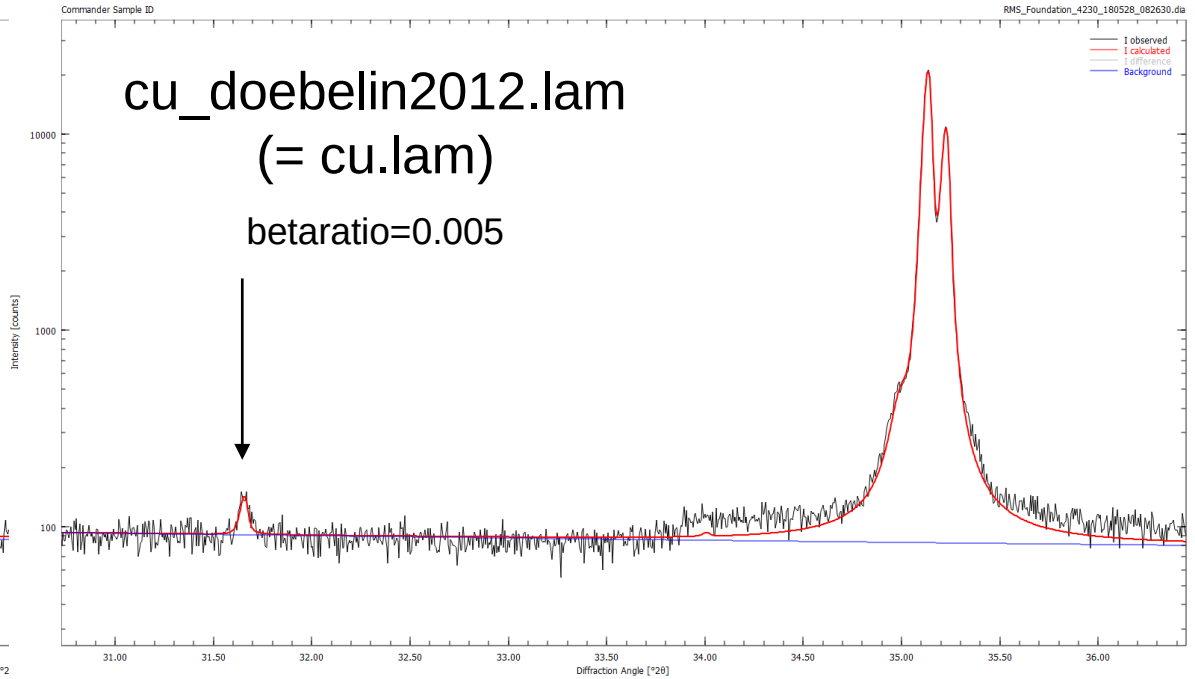
sum=Corundum
QCorundum=Corundum/sum

GOAL[1]=QCorundum
```

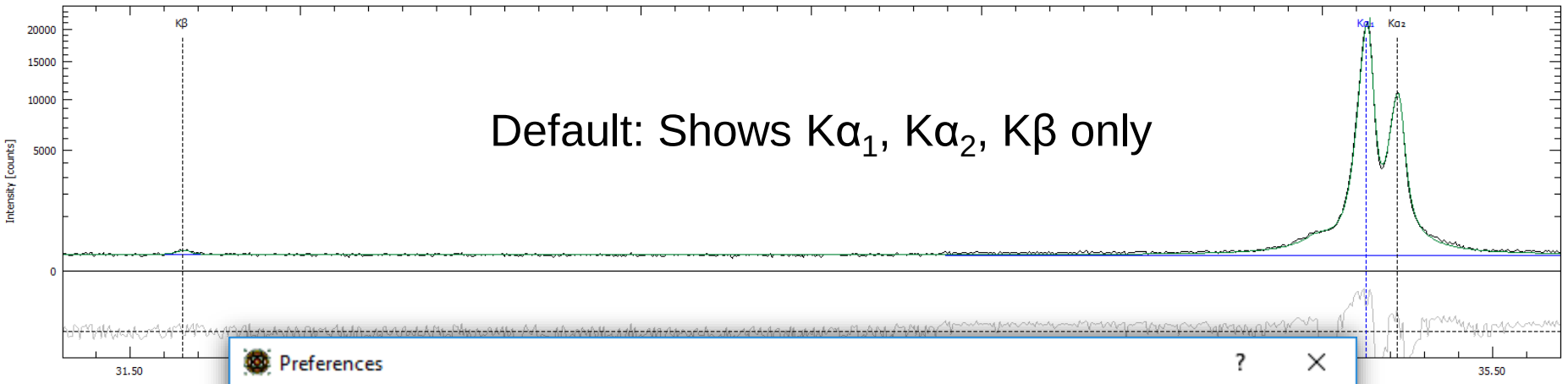
When to change the *.lam file?

- When default Kβ / $K\alpha_3$ intensity is not correct
- When using a primary-beam monochromator

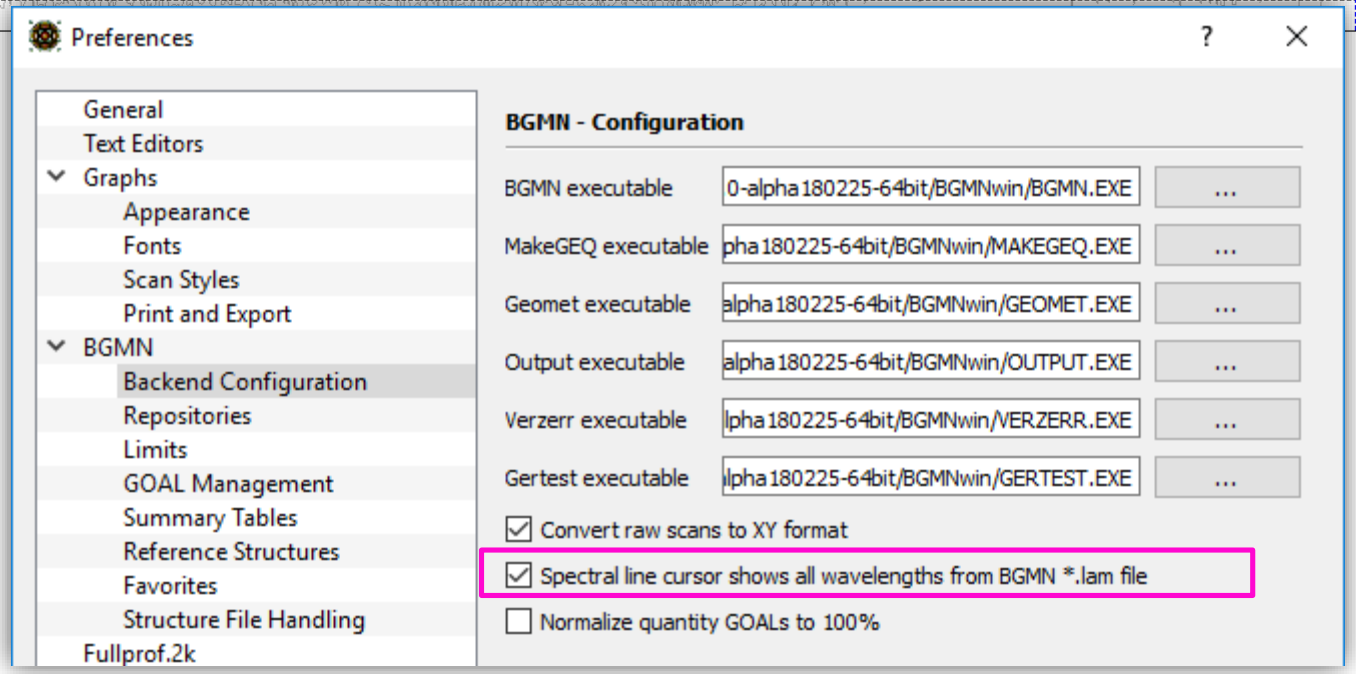




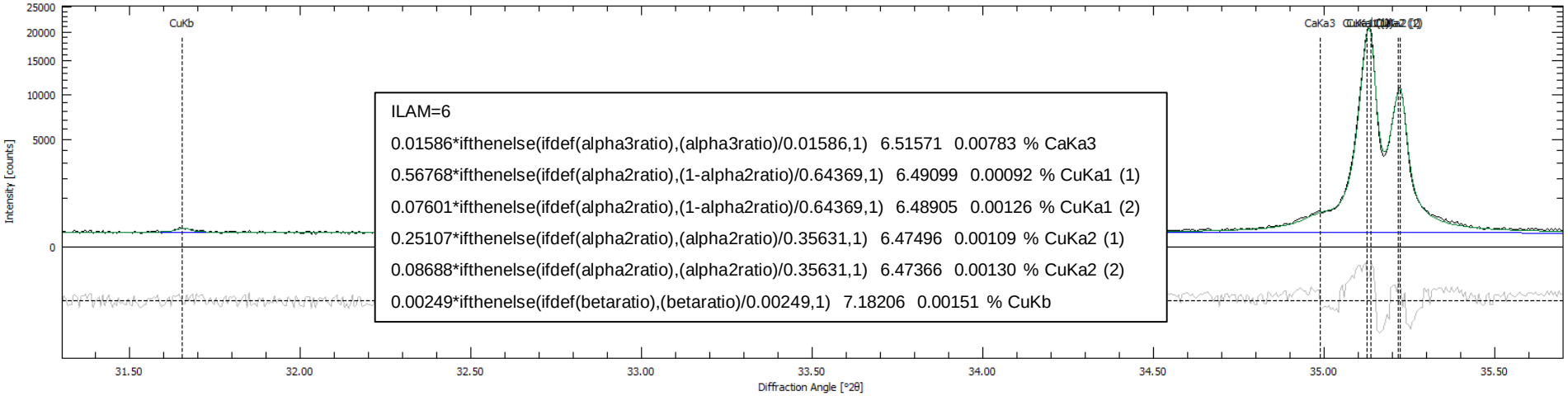
«Spectral Line Cursor» (S key)

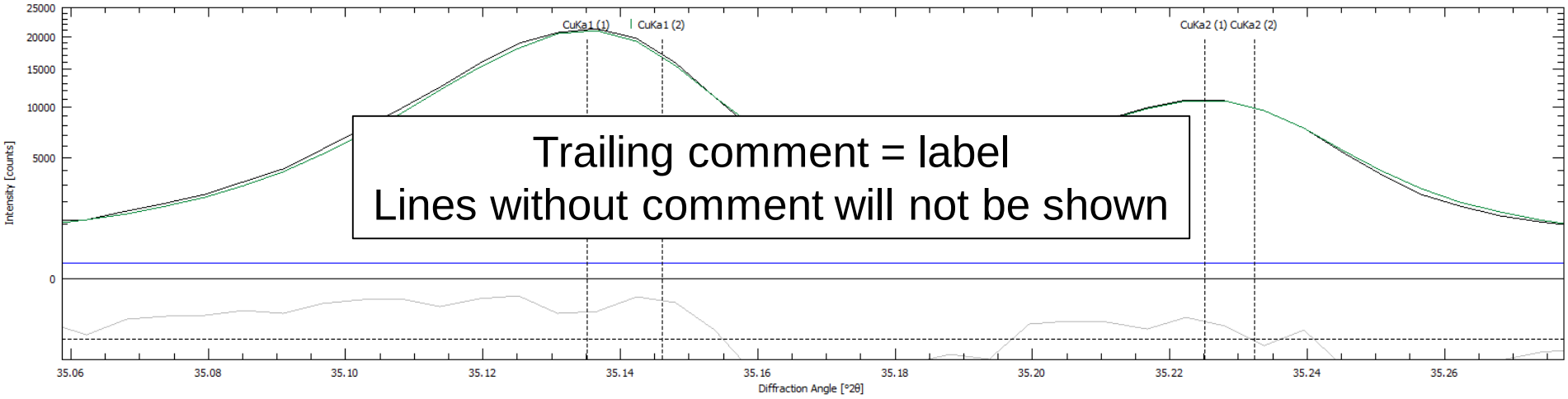


Default: Shows $K\alpha_1$, $K\alpha_2$, $K\beta$ only

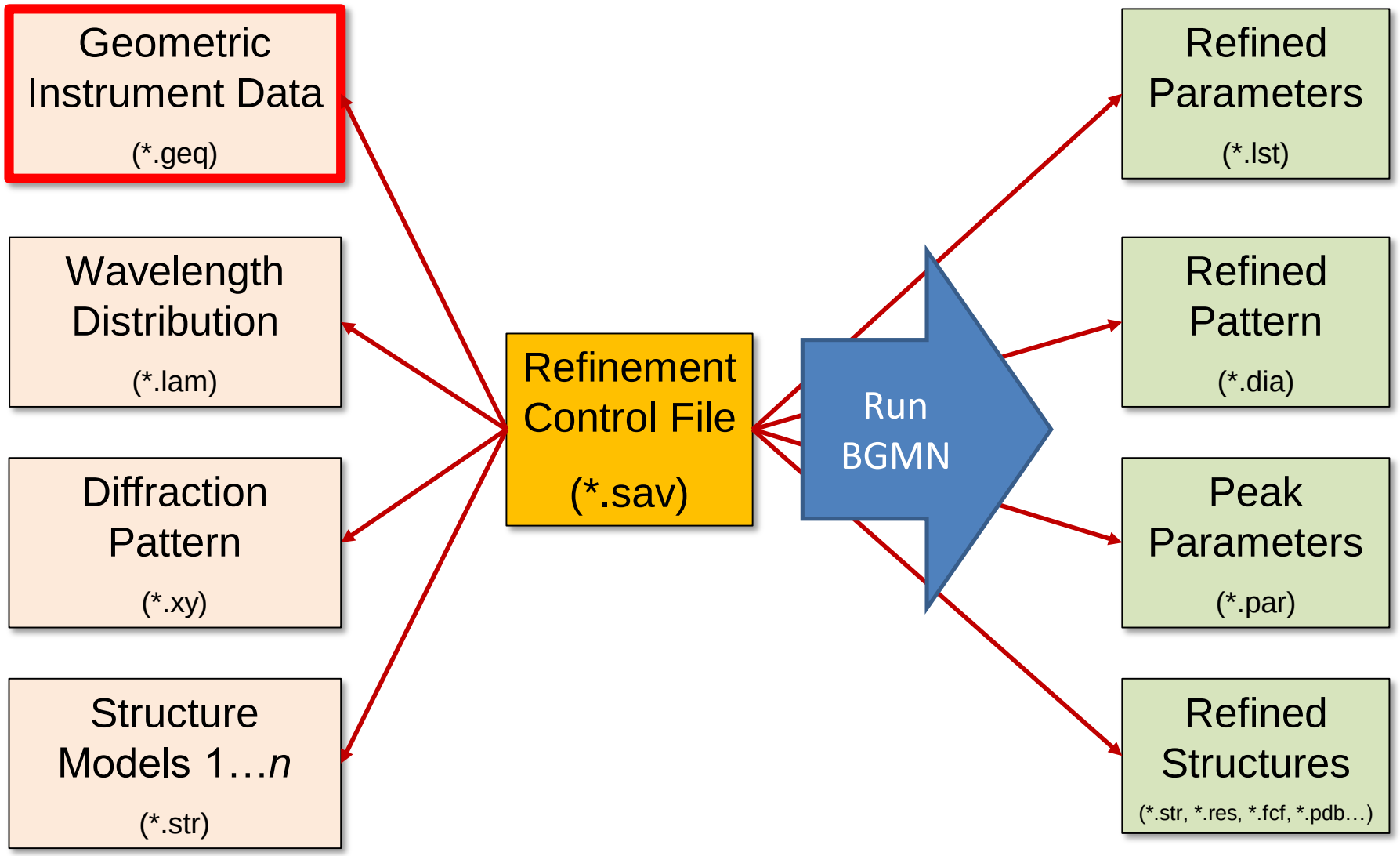


«Spectral Line Cursor» (S key)





BGMN: Geometric Instrument Data



BGMN: Geometric Instrument Data

Profile Interpolation

Raytraced Peak Profiles (*.ger)

Monte-Carlo Simulations

Instrument Description (*.sav)

Geometric Instrument Data (*.geq)

Wavelength Distribution (*.lam)

Diffraction Pattern (*.xy)

Structure Models 1…*n* (*.str)

Refinement Control File (*.sav)

BGMN: Geometric Instrument Data

Geometric Instrument Data

(*.geq)

Profile Interpolation

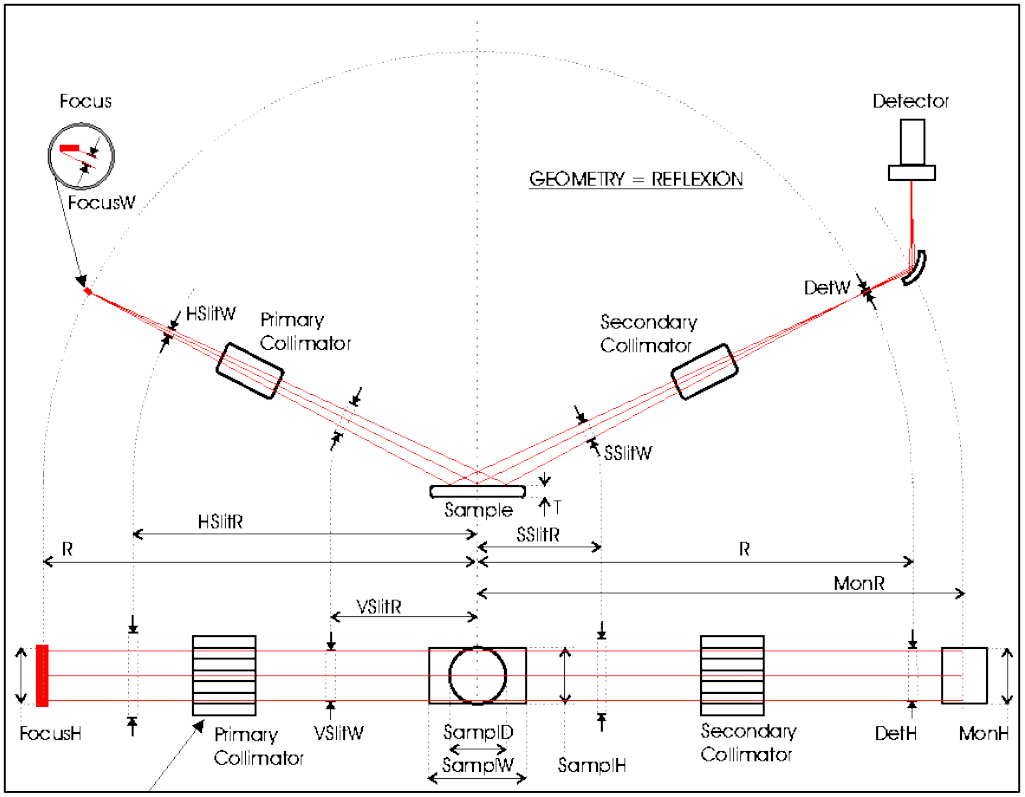
Raytraced Peak Profiles

(*.ger)

Monte-Carlo Simulations

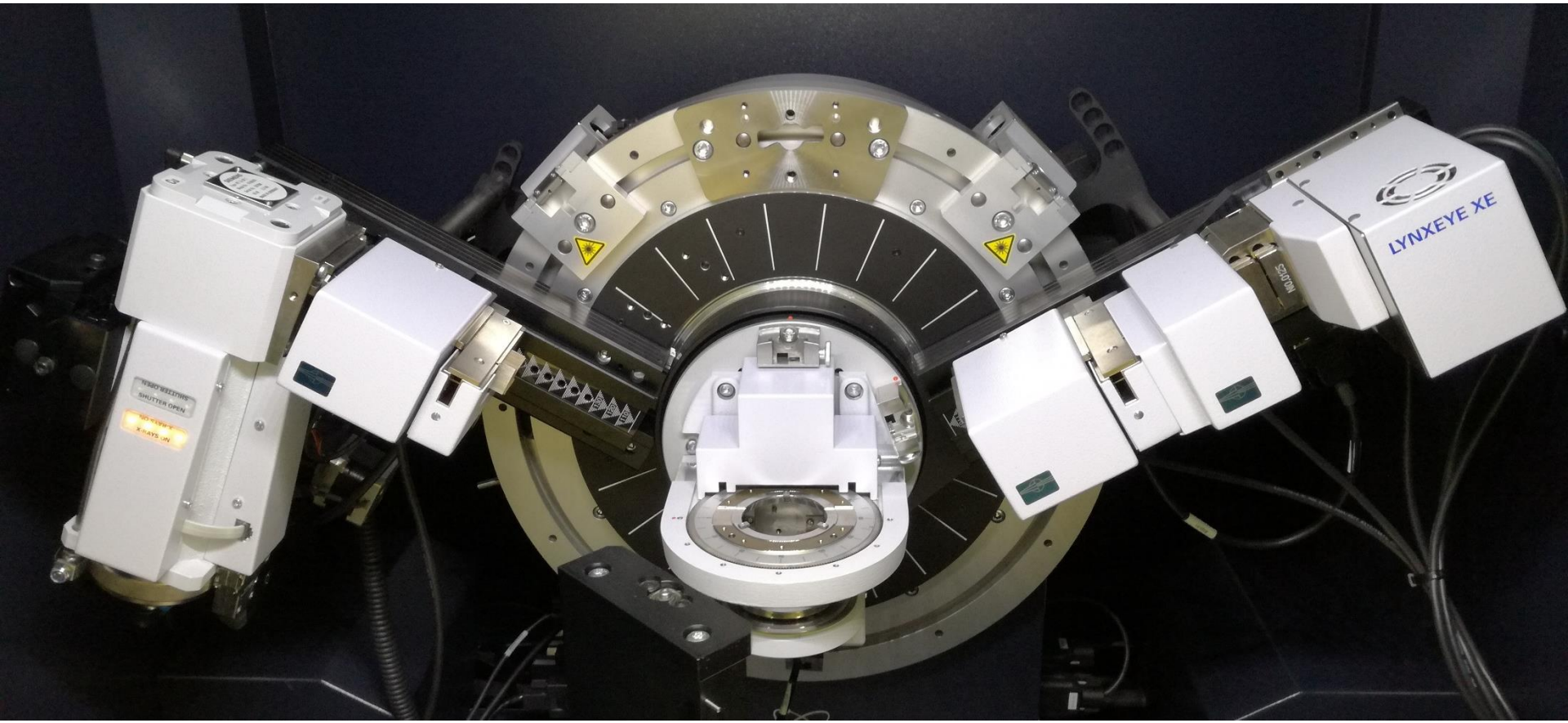
Instrument Description

(*.sav)



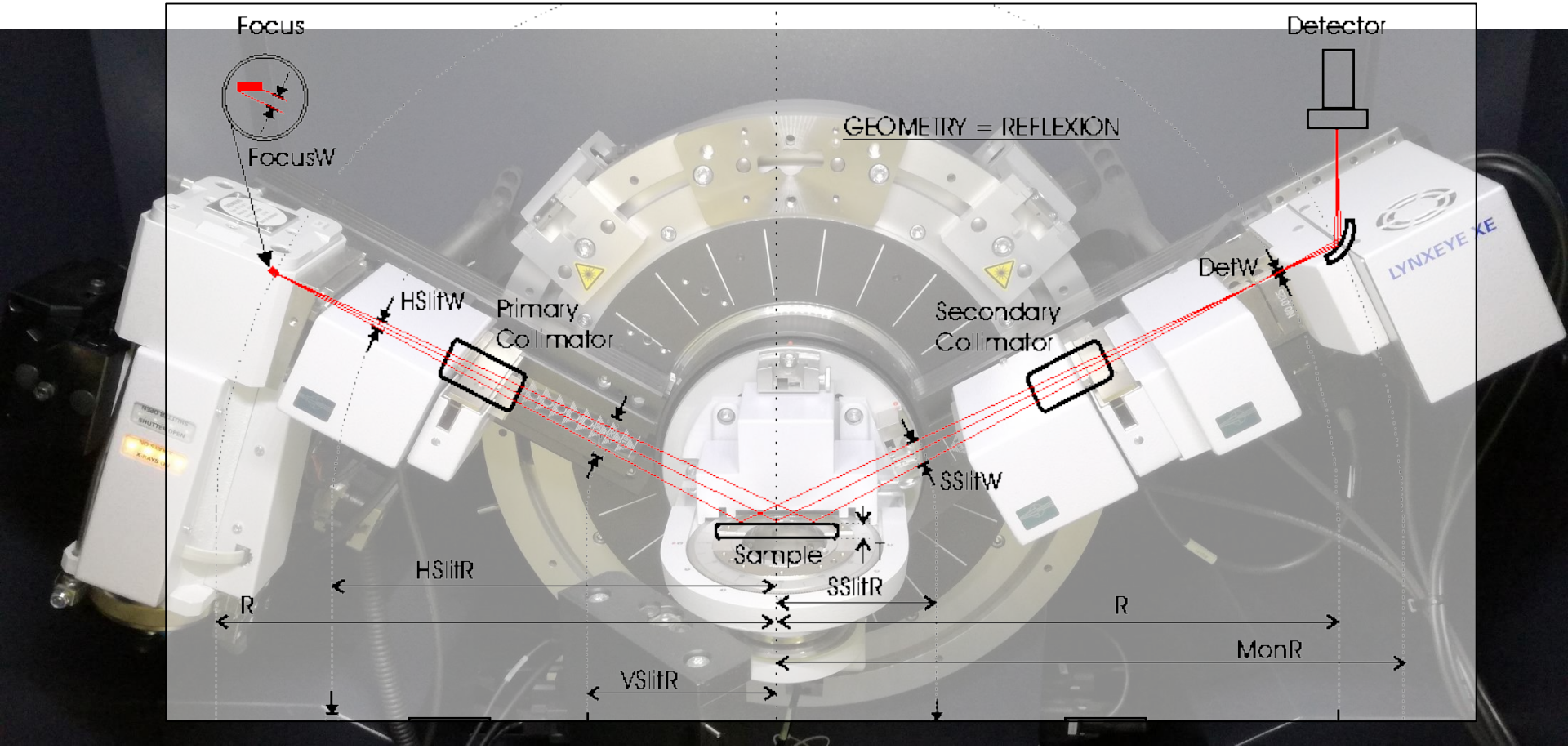
BGMN: Geometric Instrument Data

3 things required



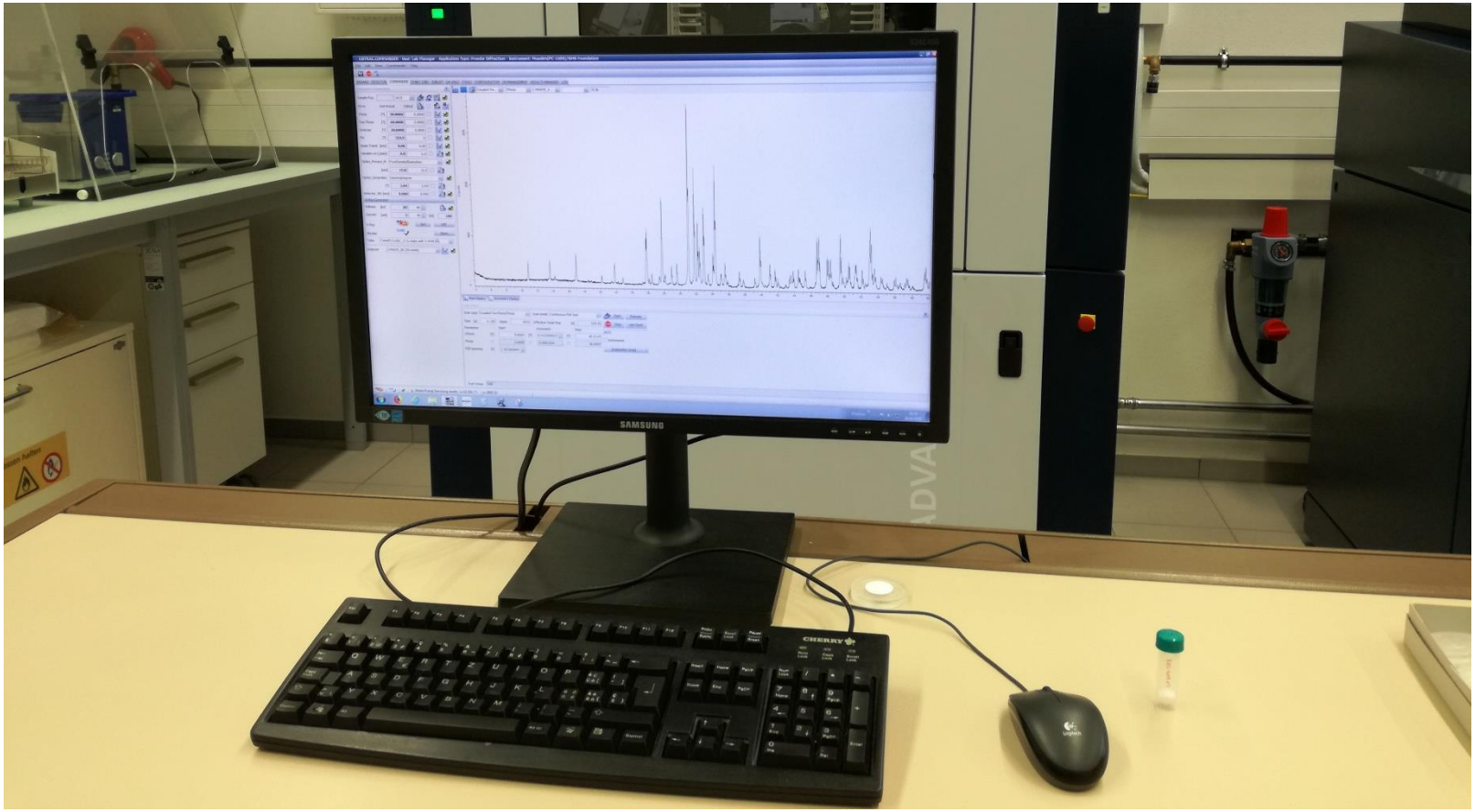
BGMN: Geometric Instrument Data

1. Physical access to the instrument and technical documentation



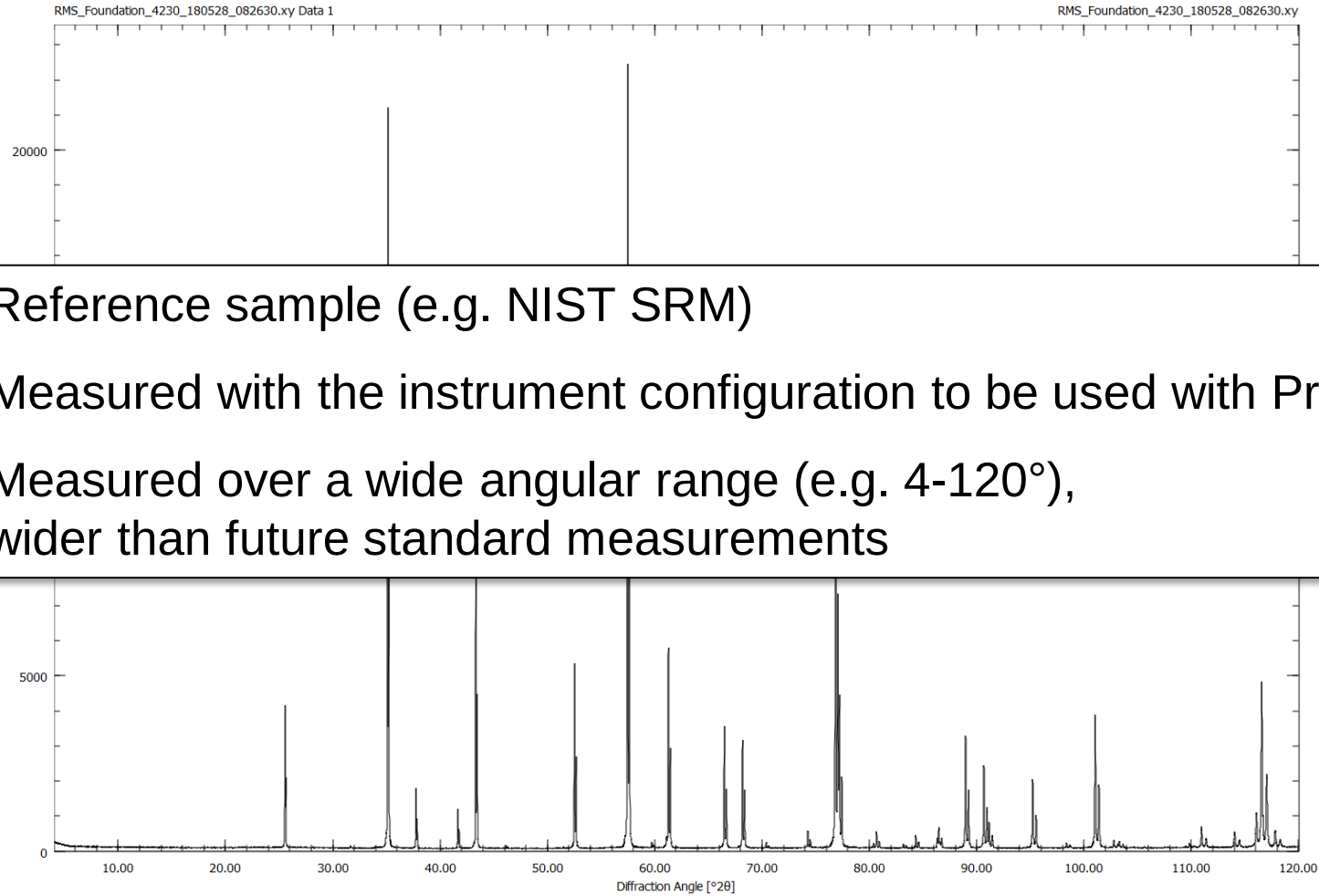
BGMN: Geometric Instrument Data

2. Access to the control software



BGMN: Geometric Instrument Data

3. A reference dataset



- Reference sample (e.g. NIST SRM)
- Measured with the instrument configuration to be used with Profex
- Measured over a wide angular range (e.g. 4-120°), wider than future standard measurements

BGMN Variables

http://www.bgmn.de/variables.html

Variables in des task description SAV file for computation of standard profiles

As no other said, all lengths are in millimeters an all angles are in degree.

NTHREADS
Number of threads as used for calculation in parallel. Should less or equal the number of CPUs in your PC. As of level 5.0.7, all three geometry programs (GEOMET, MAKEGEQ, VERZERR) are parallelized, altough with quite different efficiency:
GEOMET
will use multiple threads for increased precision: There will be additional angular po
MAKEGEQ
will even use a smaller step size, except for capillary geometry where MAKEGEQ i
VERZERR
will speed up, but parallel efficiency is even far from being 100%.
You may put assignments to NTHREADS into the geomet.cfg/makegeq.cfg/verzerr.cfg file

VERZERR
Name of the GER-file (output of GEOMET and VERZERR) and (if no variable GEQ is de

TubeTails
File with pattern data for Tube Tails correction. Implementation restriction: This file may c
VAL[i] entry.

R
Gomiometer radius.

FocusH FocusW
Axial (FocusH) and equatorial (FocusW) dimension of the X-ray tube's focus. FocusW me
X-ray tube. The optical equatorial focus dimension is reduced by the take-off angle of X-ra
dimension equals the 10th part of the thermal one.

HSlitR, HSlitW
Radius (distance from device axis) and width of the equatorial divergence slit. May depend
GSUM=Y should be used.

RoundSlitR, RoundSlitD
Radius (distance from device axis) and diameter of a round slit (plate with a circular hole)

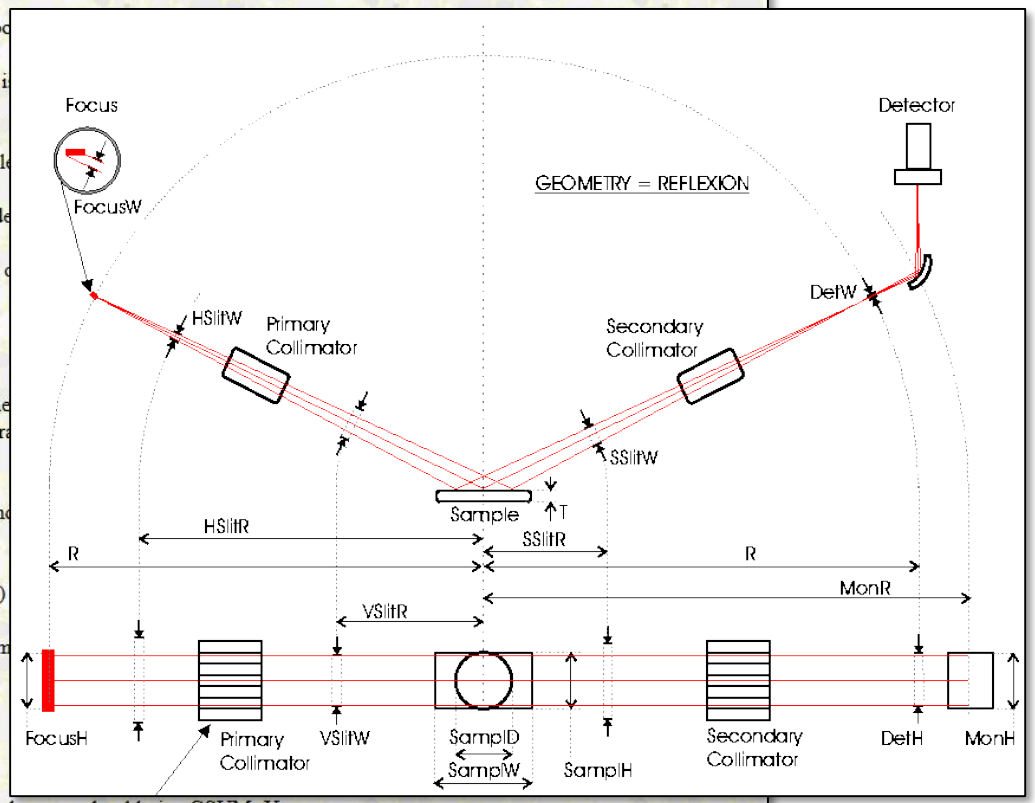
PColl
Divergency angle of the primary soller slit, e.g. 0.5/25 (unit is radian). Default is no collim

PCollA
Out-of-plane-angle of primary soller slit.

VSlitR, VSlitH
Radius (distance from device axis) and width of the axial divergence slit.

SamplD
Diameter of a round specimen. Default value is infinite (sufficient large). Using a small value, you should give GSUM=Y.

DeltaOmega
Ω twist of the specimen, valid only for `GEOMTRY=REFLEXION`. Changes profile shapes plus intensities (GEOMET) and changes effective penetration depths depending from



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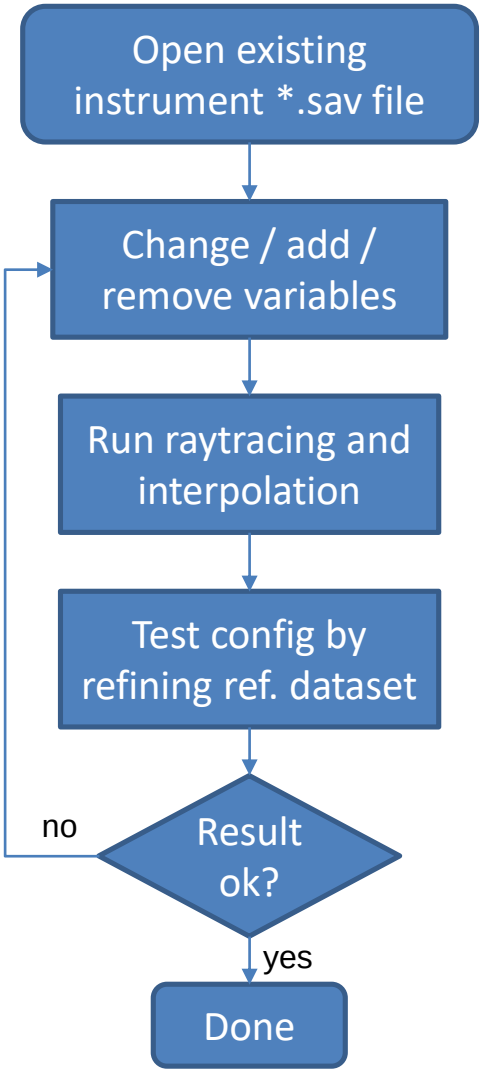


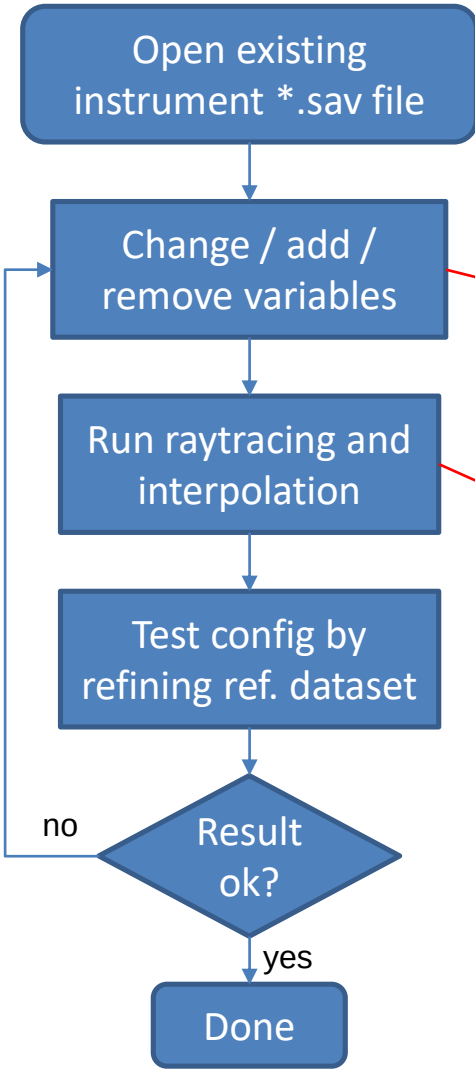
- Choose one that resembles your configuration (same manufacturer, same detector, variable or fixed divergence slit)

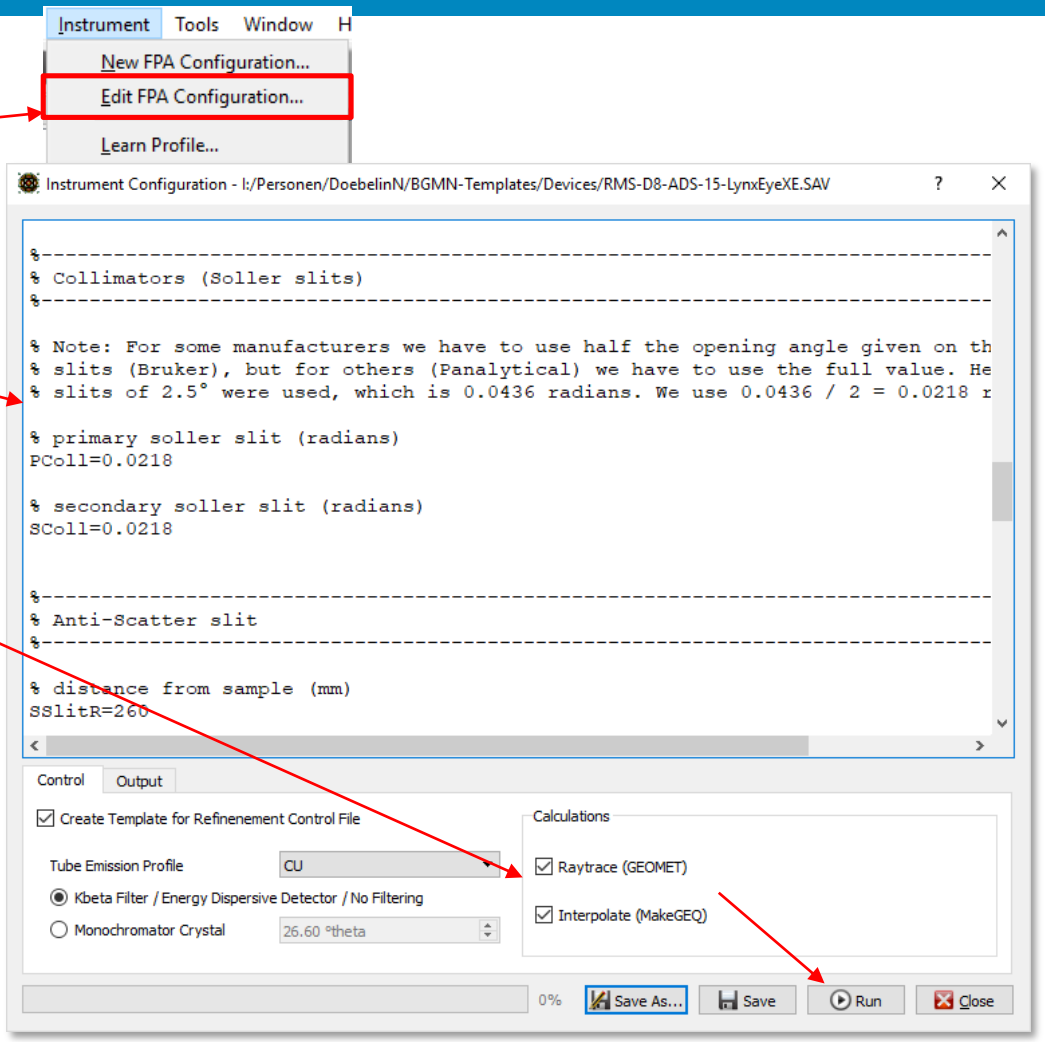
- Go through the file line by line
- If your instrument has the optical element but you don't know the setting, leave it at the default
- If your instrument doesn't have this element, remove (or comment) the line
- If your instrument has additional elements, add them according to the schematic

- Difference curve must be nearly perfect
- Crystallite sizes must be realistic (compare with SRM certificate)

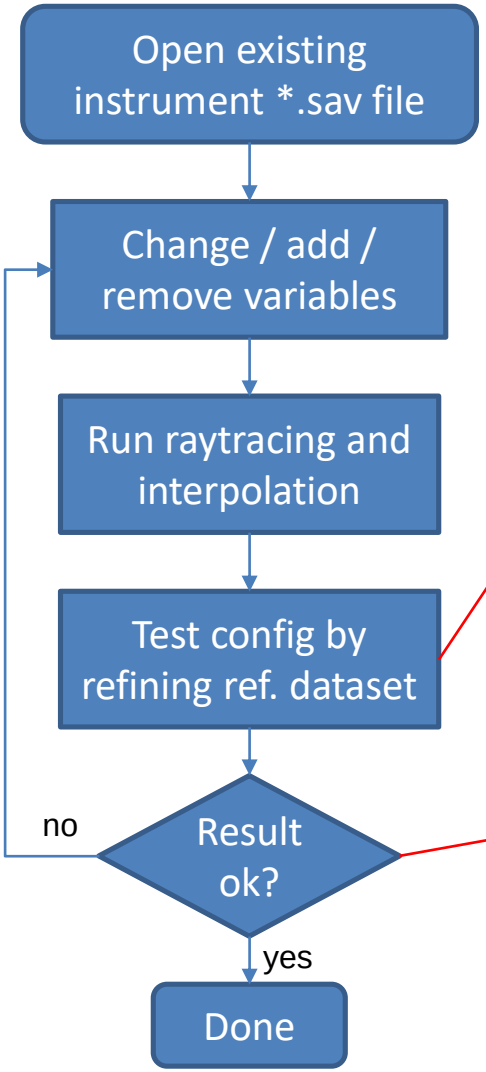
Practical examples after lunch

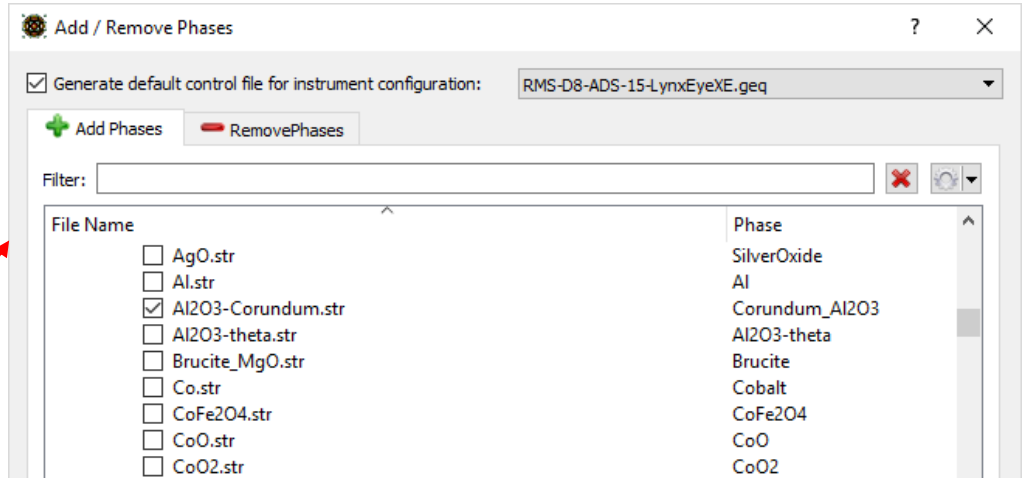
BGMN: Geometric Instrument Data

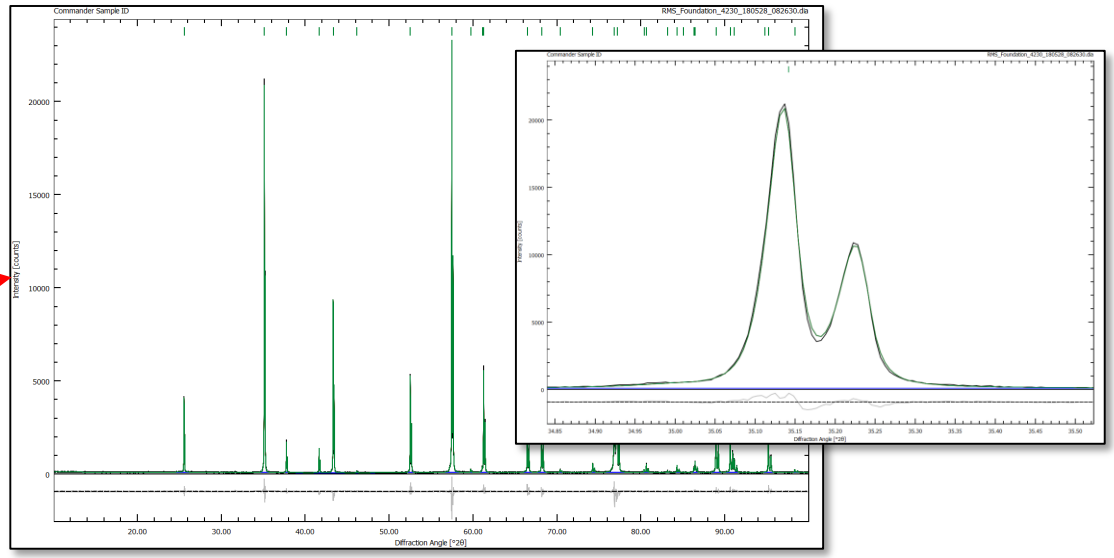




BGMN: Geometric Instrument Data

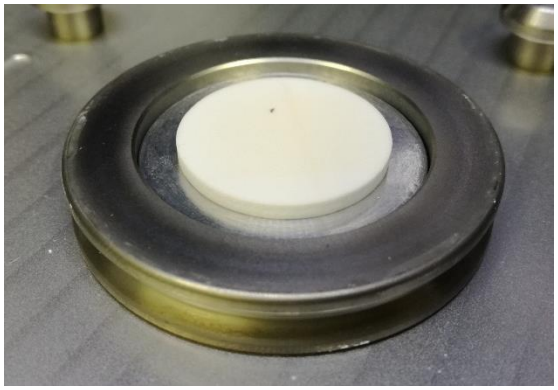






Verification with SRM

NIST SRM 1976b Al_2O_3



…The platelets were typically 5 μm to 10 μm in diameter by 2 μm to 3 μm in thickness…

…This leads to a minimal development of micro-strain and its associated line broadening…

…Given this, and the essential absence of crystallite size broadening, SRM 1976b can be used to obtain an approximation of the instrument profile function (IPF)…

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1976b

Instrument Response Standard for X-Ray Powder Diffraction

This Standard Reference Material (SRM) consists of a sintered alumina disc intended for use in calibration of X-ray powder diffraction equipment with respect to line position and intensity as a function of 2θ angle. The solid form of the SRM eliminates any variability in intensity measurements introduced by the sample loading procedure. A unit of SRM 1976b consists of a sintered alumina disc approximately 25.6 mm in diameter by 2.2 mm in thickness.

Material Description: The manufacturing process used to produce this SRM was developed for the production of substrates for electronic components. The alumina powder used as the precursor for these substrates was of high phase purity (corundum structure) with a platelet particle morphology. The platelets were typically 5 μm to 10 μm in diameter by 2 μm to 3 μm in thickness. The compaction procedure for the discs resulted in an axisymmetric texture with the basal planes tending towards parallelism with the surface of the disc. This axisymmetric character of the texture permits sample mounting in any orientation about the surface normal. The compacts were liquid-phase sintered using a small percentage of a glass phase. No crystalline impurities have been detected. The glass phase involved in the liquid-phase sintering effectively prevents inter-particle contact and relaxes during the cooling of the pieces from the sintering temperature. This leads to a minimal development of micro-strain and its associated line broadening; though it is detectable as Gaussian broadening with a tanθ dependence. Given this, and the essential absence of crystallite size broadening, SRM 1976b can be used to obtain an approximation of the instrument profile function (IPF). Use of SRM 1976b is not recommended, however, for quantitative microstructure analyses. The discs comprising the feedstock of this SRM were manufactured in a single, dedicated production run to ensure consistency of microstructure with respect to grain size, shape, micro-strain, and texture.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account. The measurands are the certified values for relative intensity (dimensionless ratios) shown in Table 1, and the lattice parameters are shown in Table 2. Metrological traceability is to the SI units for the relative intensity values are the dimension of one, and for lattice parameters the dimension is length (expressed as nanometers). The certified values and uncertainties were calculated according to the method described in the ISO/JCGM Guide [1].

Information Values: The analyses associated with certification of SRM 1976b included the computation of the diffraction line positions shown in Table 3. In order to use SRM 1976b on diffraction equipment of various optical configurations, the effects of polarization must be taken into account. The values shown in Table 4 reflect a bias applied to the certified values to account for this effect. The data of Tables 3 and 4 are presented as information values. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1976b** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Overall coordination and technical direction of the certification were performed by J.P. Cline of the NIST Materials Measurement Science Division.

John A. Small, Chief
Materials Measurement Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 04 November 2015
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

SRM 1976b

Page 1 of 7

NIST Standard Reference Materials (SRM)

	SRM	Description	Unit Size	Application
D C M	640e Now Selling	Line Position and Line Shape Standard for Powder Diffraction (Silicon Powder)	7.5 g	Line Position Line Shape
D C M	656 Now Selling	Silicon Nitride Powders (Quantitative Analysis Powder Diffraction Standard)	2 x 10 g	Quantitative Analysis
D C M	660c Now Selling	Line Position and Line Shape Standard for Powder Diffraction (Lanthanum Hexaboride Powder)	6 g	Line Position Line Shape
D C M	674b Now Selling	X-Ray Powder Diffraction Intensity Set (Quantitative Powder Diffraction Standard)	10.00 g (powder)	Quantitative Analysis
D C M	675 Now Selling	Line Position, Mica (XRD)	7.5 g	Line Position - Low 2θ
D C M	676a Out of Stock	Alumina Powder (Quantitative Analysis Powder Diffraction Standard)	20 g	Quantitative Analysis
D C M	1878b Now Selling	Respirable Alpha Quartz (Quantitative X-Ray Powder Diffraction Standard)	5 g	Quantitative Analysis
D C M	1879b Now Selling	Respirable Cristobalite (Quantitative X-Ray Powder Diffraction Standard)	5 g	Quantative Analysis
	1976b Now Selling	Instrument Response Standard for X-Ray Powder Diffraction	1 disc	
D C M	1979 Now Selling	Powder Diffraction Line Profile Standard for Crystallite Size Analysis(Nano-Crystalline ZnO Powder)	2 x 3 g	Line shape Crystalline size
D C M	1990 Now Selling	Single Crystal Diffractometer Alignment Standard - Ruby Sphere	3 spheres	Quantitative Analysis
D C M	1994 Now Selling	Standard Silicon Single Crystal Wafer for Crystalline Orientation	100-mm wafer	Crystalline Orientation
D C M	1995 Now Selling	Standard Sapphire Single Crystal Wafer for Crystalline Orientation	50-mm wafer	Crystalline Orientation
D C M	2000 Out of Stock	Calibration Standard for High-Resolution X-Ray Diffraction	1 block	Line Position
D C M	2012 Now Selling	Calibration Standard for High-Resolution X-Ray Diffraction (200 mm Wafer)	wafer	Line Position
D C M F	3600 Now Selling	Absolute Intensity Calibration Standard for Small-Angle X-ray Scattering	coupon	Small-angle scattering intensity

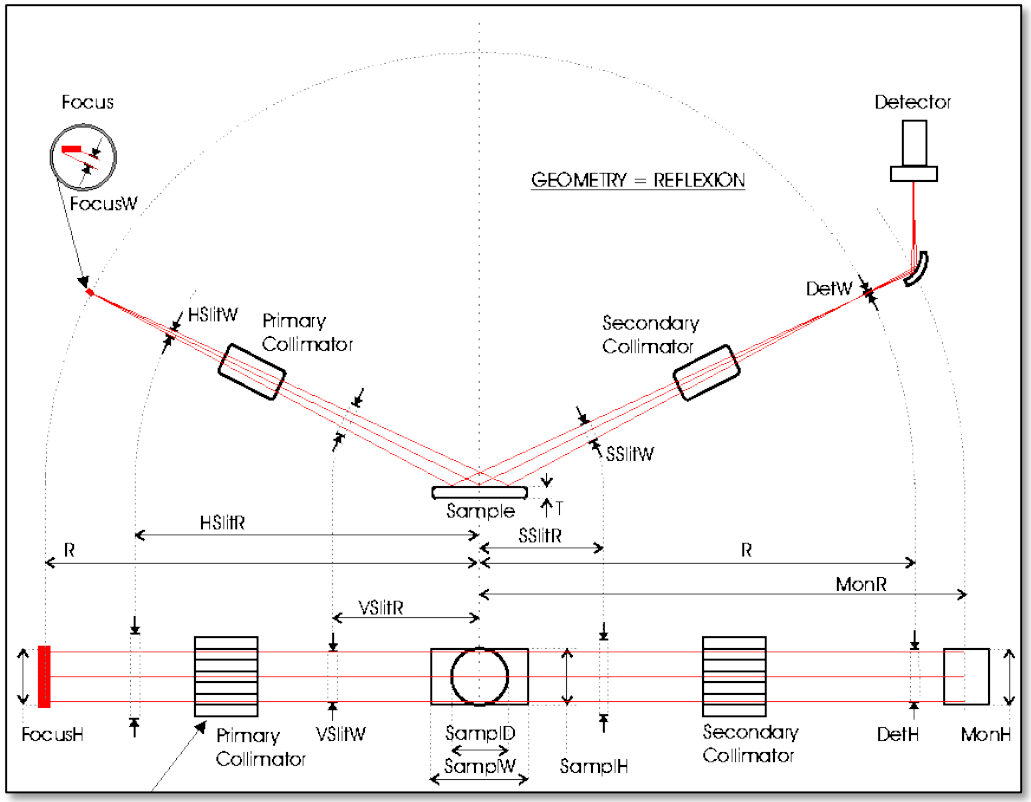
Non-standard configurations

What about:

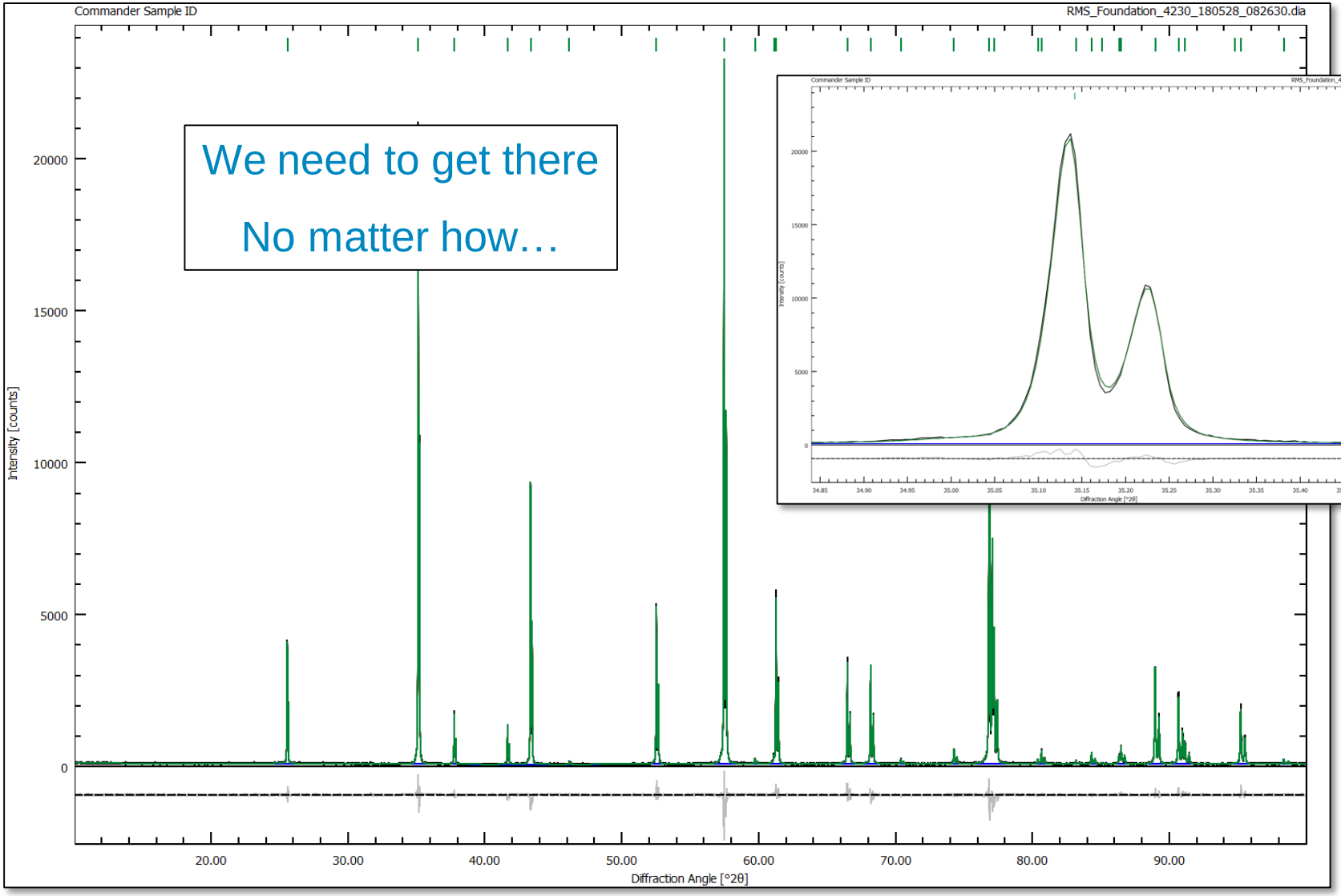
- Primary beam monochromators
- Area detectors
- Synchrotron / neutron diffractometers
- Future optics

Fake the configuration
until the peak profile matches

Or use Profex with FullProf

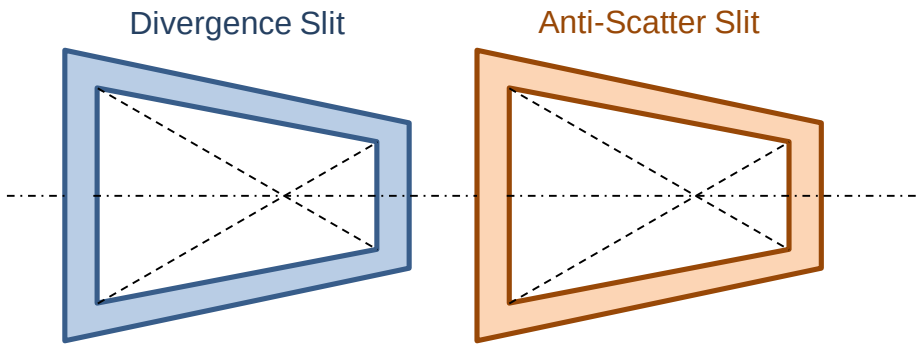


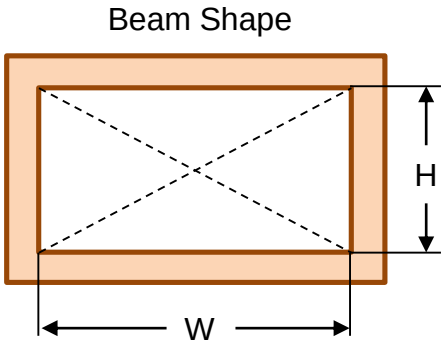
Faked Instrument Configurations



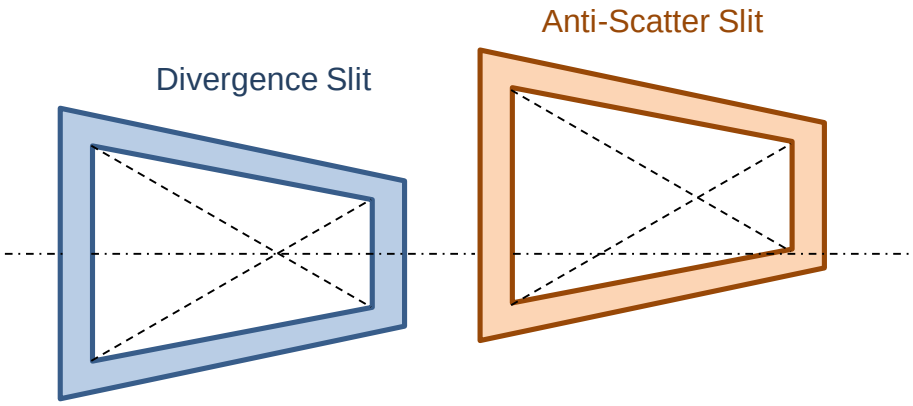
Faked Instrument Configurations

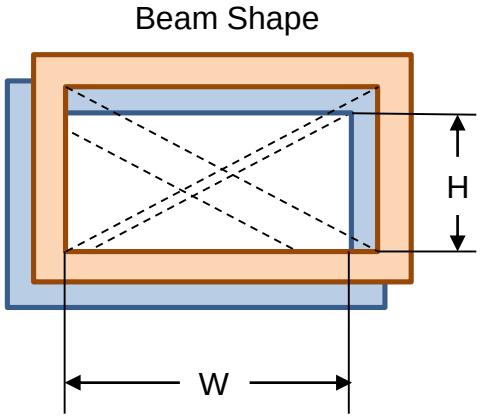
Aligned instrument





Misaligned instrument





Exercises

- Exercise 1: Configure the RMS standard instrument setup, starting from a totally wrong template.

- Exercise 2: Configure an instrument with a primary beam monochromator.
(Nobody has succeeded yet…)