

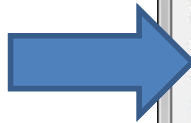
# Lesson 9

## Instrument Configuration Files

Nicola Döbelin  
RMS Foundation, Bettlach, Switzerland



# Instrument configuration file



Append Phase

Generate default control file

cubix-ads-10mm.geq

Structures

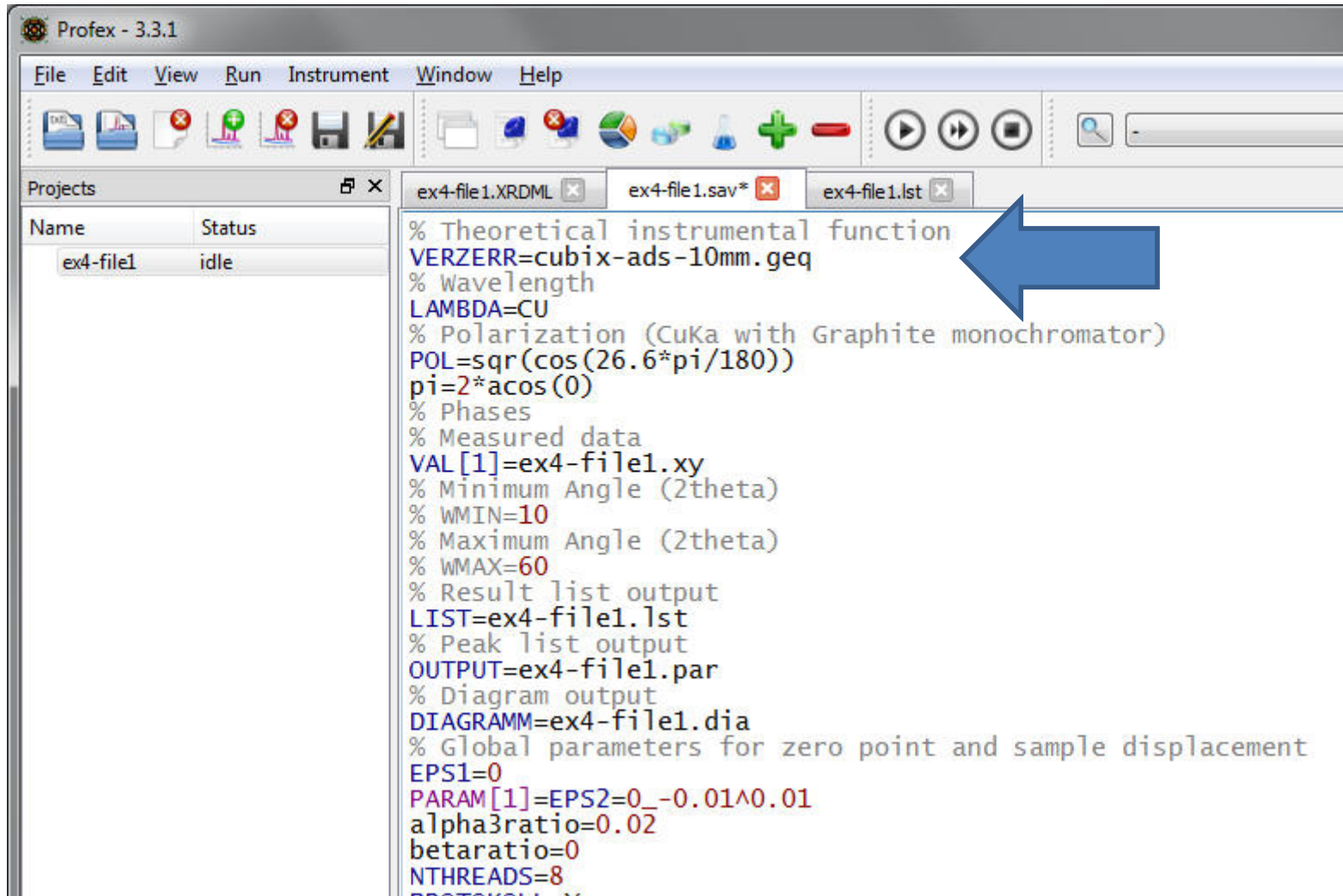
File Name	Phase	Comr
<input type="checkbox"/> Ag.str	Ag	04-001
<input type="checkbox"/> Ag2O.str	Ag2O	04-004
<input type="checkbox"/> AgNO2.str	AgNitrite	04-005
<input type="checkbox"/> Al.str	Al	04-012
<input type="checkbox"/> Al2O3-Corundum.str	Corundum_Al2O3	04-004
<input type="checkbox"/> Al2O3-theta.str	Al2O3-theta	01-086
<input type="checkbox"/> alpha_Ft.str	alpha_Ft	
<input type="checkbox"/> alphaCMP.str	alphaCaMetaphosphate	04-014
<input type="checkbox"/> alphaCPP.str	alphaCaPyrophosphate	04-005
<input type="checkbox"/> alphaTCP.str	alphaTCP	04-010
<input type="checkbox"/> AmmoniumDihydrogenPhosphate.str	AmmoniumDihydrogenPhosphate	01-085
<input type="checkbox"/> Ammoniumsulphate.str	Ammoniumsulphate	04-005
<input type="checkbox"/> Anatase.str	Anatase	04-007
<input type="checkbox"/> Aragonite.str	Aragonite	01-071
<input type="checkbox"/> Arcanite.str	K2SO4	04-006
<input type="checkbox"/> Ardealite.str	Ardealite	04-011
<input type="checkbox"/> benzoic_acid.str	Benzoic Acid	04-011

Expand/Collapse

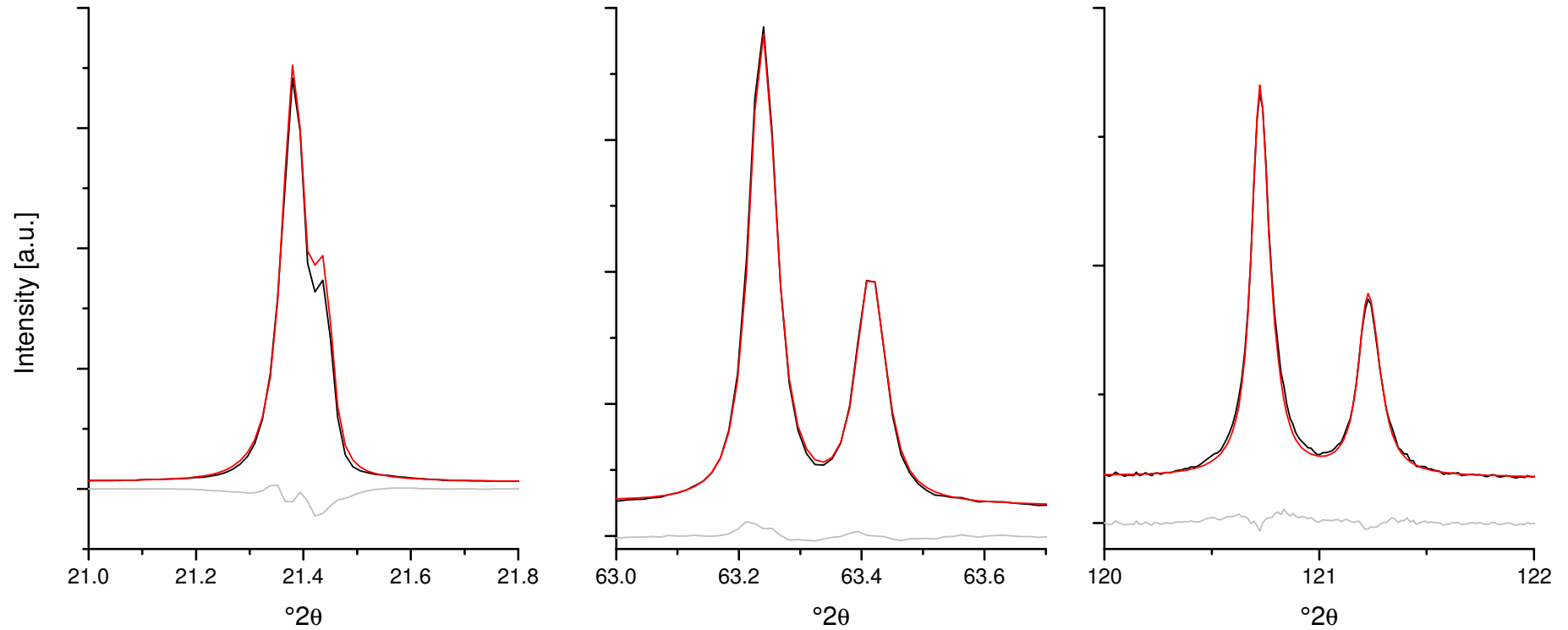
Overwrite existing files

OK Cancel

# Instrument configuration file



# Fundamental Parameters Approach



Description of the peak profile

# Instrument Configuration Files

Create / edit manually



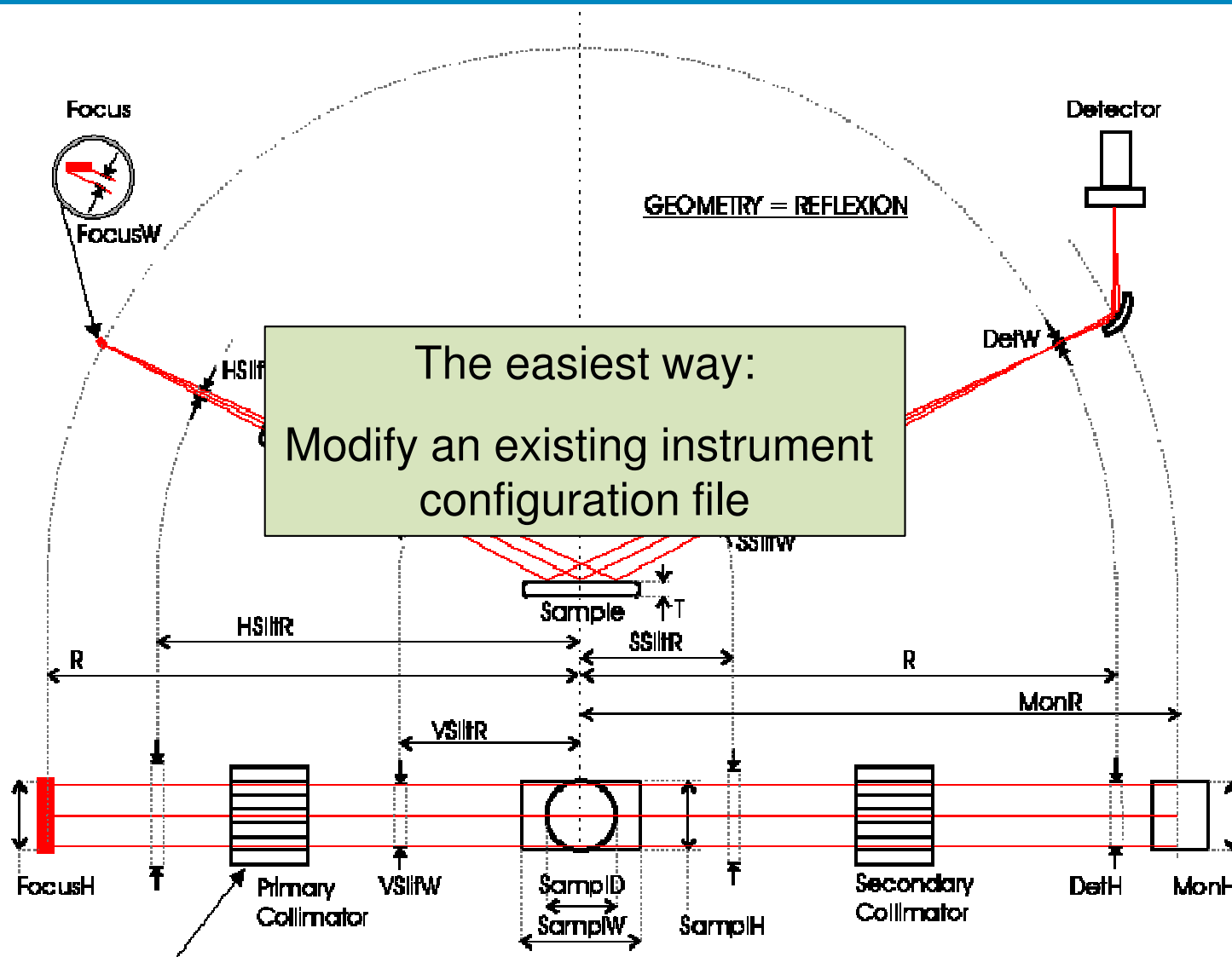
File	Description
Instrument.sav	Description of the instrument configuration
Instrument.ger	Calculated profile
Instrument.geq	Interpolated profile
Instrument.tpl	Template for *.sav control file for this configuration <sup>1)</sup> (Empty *.sav file)

All these files are stored in

...\Profex-BGMN-Bundle-3.3.1\Profex\Devices

<sup>1)</sup> Optional, only used by Profex, not required for BGMN

# Instrument Configuration Files



<http://www.bgm.de>

# Editing Instrument Files

- Try to find an instrument configuration with...
  - ... the same manufacturer
  - ... the same model
  - ... the same detector
  - ... the same divergence slit mode (FDS / ADS)

Example

Your instrument:

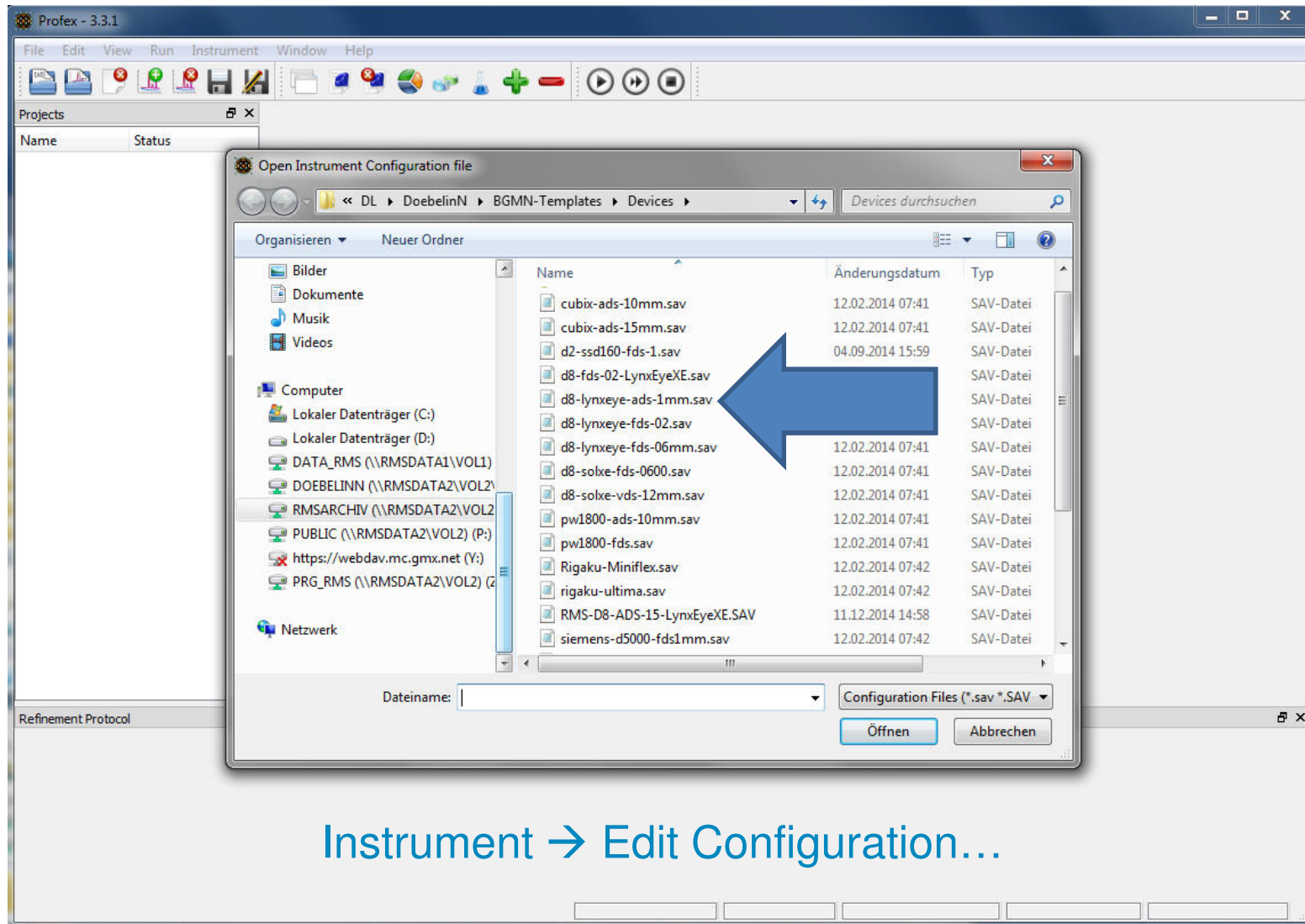
Bruker **D8** Advance

LynxEye detector

Automatic **D**ivergence **S**lit

Modify configuration «**d8**-lynxeye-**ads**-1mm.sav»

# Editing Instrument Files





# Editing Instrument Files

Instrument Configuration - I:/DL/DoebelinN/BGMN-Templates/Devices/d8-lynxeye-ads-1mm.sav

```
*****
%
%   BGMN Device Configuration File for Bruker D8
%   -----
%
%   Created by Nicola Doebelin, RMS Foundation, Switzerland
%   July 02, 2013
%
%   Device Configuration:
%   - Detector: LynxEye
%   - Radiation: CuKa, Ni-filtered
%   - Soller Slits: 2.5°
%   - Divergence Slit: automatic, 1mm irradiated length
%   - Anti-Scatter Slit: fixed, 2.38°
%   - Goniometer Radius: 280 mm
%
%*****
SAVE=N
%-----
% Output files for Geomet and MakeGeq
%-----
VERZERR=d8-lynxeye-ads-1mm.ger
GEQ=d8-lynxeye-ads-1mm.geq
%-----
% X-ray tube
%-----
% axial dimension (length, mm)
FocusH=12
<----->
```

Calculations

- Raytrace (GEOMET)
- Interpolate (MakeGEO)
- Calculate Profile (GERTEST)

Save As... Save Run Close

Comment: Description of configuration

# Editing Instrument Files

```
%-----  
% Divergence slit  
%-----  
% Note: BGMN requires the width of the slit in mm. For automatic divergence slits we must use  
% trigonometric functions to calculate the width in mm as a function of irradiated length,  
% distance of the slit from the sample, and 2theta angle.  
% If the distance of the slit is not known exactly, enter a value shorter than the goniometer  
% radius R for HSlitR. It will lead to a different slit opening, but the divergence of the beam  
% will always be correct. HSlitW will calculate the slit width resulting in a irradiated length  
% of "irr" mm.  
  
% irradiated length (mm)  
irr=1  
  
% distance from sample (mm)  
HSlitR=100  
  
% automatic slit width (mm)  
HSlitW=(2*(R-HSlitR)*irr*sin(pi*zweiTheta/360))/(2*R+irr*cos(pi*zweiTheta/360))  
  
%-----  
% Detector  
%-----  
  
% Note: The Lynxeye detector has a DetW of 0.075 mm, but this results in wrong crystallite sizes.  
% The value for DetW used here was fitted to a LaB6 pattern to obtain realistic cryst sizes.  
  
% total detector height (mm)  
DetArrayW=14.4  
  
% height of one strip (mm)  
%-----  
Calculations  
 Raytrace (GEOMET)  
 Interpolate (MakeGEQ)  
 Calculate Profile (GERTEST)  
  
Save As... Save Run Close
```

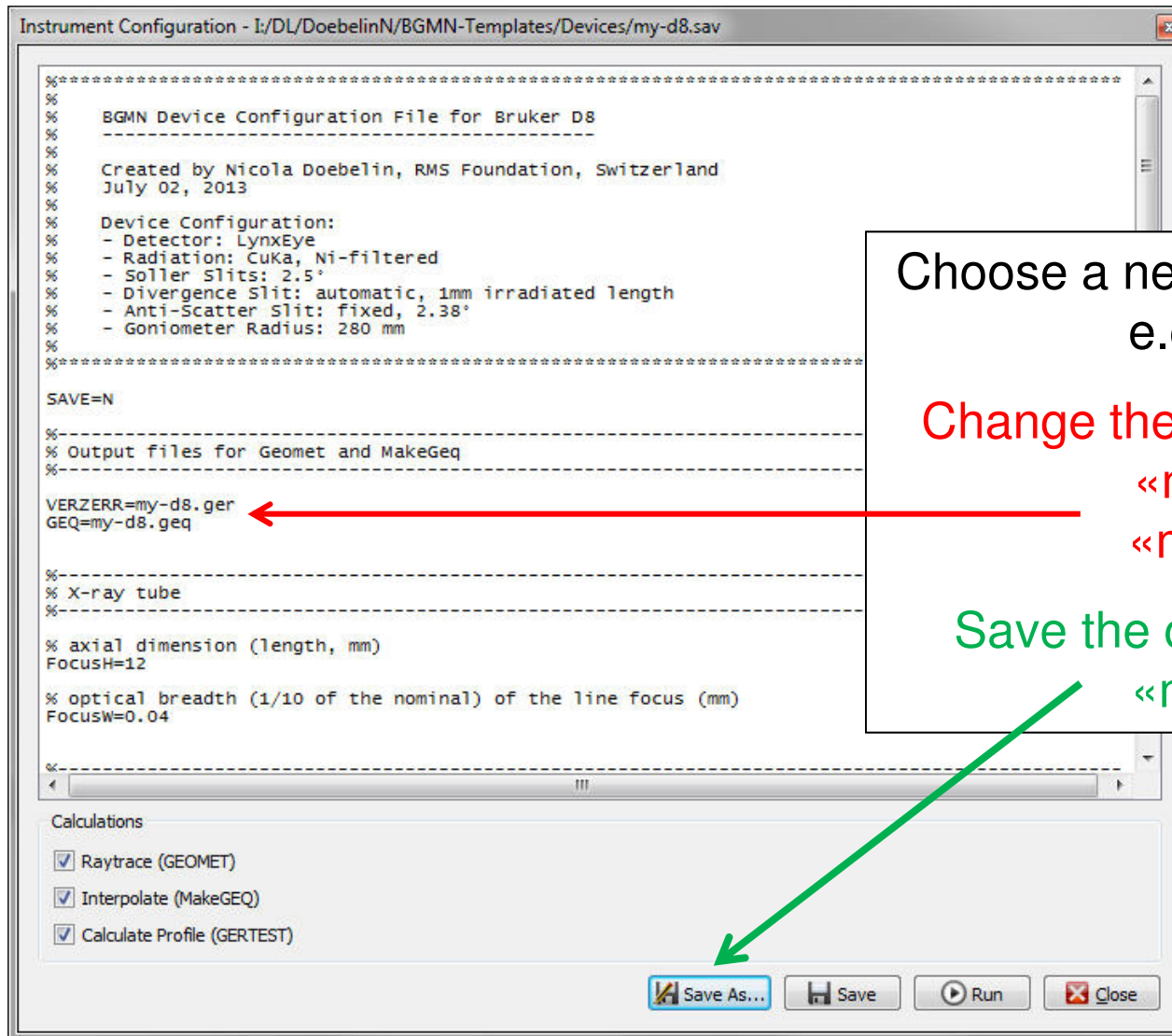
- Go through the file line by line
- Change values to match your configuration
- Verify **ALL** values

# Editing Instrument Files

```
%-----  
% Parameters for the simulation of the profile function  
%-----  
  
% angular positions for the MonteCarlo simulation (^2theta)  
zweiTheta[1]=4  
zweiTheta[2]=8  
zweiTheta[3]=13  
zweiTheta[4]=20  
zweiTheta[5]=30  
zweiTheta[6]=42  
zweiTheta[7]=56  
zweiTheta[8]=76  
zweiTheta[9]=90  
zweiTheta[10]=105  
zweiTheta[11]=120  
zweiTheta[12]=135  
zweiTheta[13]=150  
  
% angular range (^2theta)  
WMIN=4  
WMAX=150  
  
% step width for the interpolation of the geometric profiles (^2theta)  
WSTEP=3*sin(pi*zweiTheta/180)  
  
% switch for applying the intensity correction for beam overflow resp. ADS function  
GSUM=Y  
  
% Use multithreaded calculation  
NTHREADS=2  
  
% Convenience function: Calculate PI for use in other angle-dependent calculations  
pi=2*acos(0)  
  
%-----  
% End of file  
%-----
```

Leave this block unchanged  
(at the very end of the file)

# Editing Instrument Files



Choose a new configuration name  
e.g. «my-d8»

Change the output file names to  
«my-d8.ger»  
«my-d8.geq»

Save the configuration file as  
«my-d8.sav»



# Computing Peak Profile

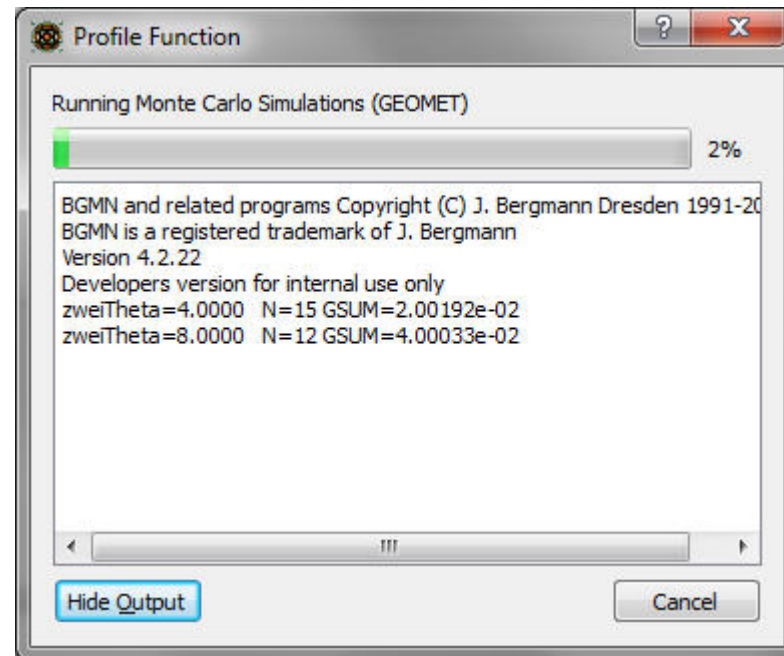
Instrument Configuration - I:/DL/DoebelinN/BGMN-Templates/Devices/my-d8.sav

```
*****  
%  
%   BGMN Device Configuration File for Bruker D8  
%-----  
%  
%   Created by Nicola Doebelin, RMS Foundation, Switzerland  
%   July 02, 2013  
%  
%   Device Configuration:  
%   - Detector: LynxEye  
%   - Radiation: CuKa, Ni-filtered  
%   - Soller slits: 2.5°  
%   - Divergence Slit: automatic, 1mm irradiated length  
%   - Anti-Scatter Slit: fixed, 2.38°  
%   - Goniometer Radius: 280 mm  
%-----  
%  
SAVE=N  
%-----  
% Output files for Geomet and MakeGeq  
%-----  
%  
VERZERR=my-d8.ger  
GEQ=my-d8.geq  
%-----  
% X-ray tube  
%-----  
% axial dimension (length, mm)  
FocusH=12  
% optical breadth (1/10 of the nominal) of the line focus (mm)  
FocusW=0.04  
%-----  
%  
Calculations  
 Raytrace (GEOMET)  
 Interpolate (MakeGEO)  
 Calculate Profile (GERTEST)  
%-----  
%  
Save As... Save Run Close
```

Click «Run»

Check all

# Computing Peak Profile



Computation may take several minutes

# Computing Peak Profile

Name	Änderungsdatum	Typ	Größe
d8-lynxeye-fds-02.ger	04.06.2012 18:06	GER-Datei	
d8-lynxeye-fds-02.sav	12.02.2014 07:41	SAV-Datei	
d8-lynxeye-fds-02.tpl	08.09.2014 10:08	TPL-Datei	
d8-lynxeye-fds-06mm.geq	12.11.2012 12:57	GEQ-Datei	
d8-lynxeye-fds-06mm.ger	12.11.2012 12:57	GER-Datei	
d8-lynxeye-fds-06mm.sav	12.02.2014 07:41	SAV-Datei	
d8-lynxeye-fds-06mm.tpl	08.09.2014 10:08	TPL-Datei	
d8-solxe-fds-0600.geq	16.01.2013 15:16	GEQ-Datei	
d8-solxe-fds-0600.ger	16.01.2013 15:15	GER-Datei	
d8-solxe-fds-0600.sav	12.02.2014 07:41	SAV-Datei	
d8-solxe-fds-0600.tpl	08.09.2014 10:08	TPL-Datei	
d8-solxe-vds-12mm.geq	16.01.2013 15:17	GEQ-Datei	
d8-solxe-vds-12mm.ger	16.01.2013 15:17	GER-Datei	
d8-solxe-vds-12mm.sav	12.02.2014 07:41	SAV-Datei	
d8-solxe-vds-12mm.tpl	08.09.2014 10:08	TPL-Datei	
my-d8.geq	11.01.2015 14:34	GEQ-Datei	
my-d8.ger	11.01.2015 14:34	GER-Datei	
my-d8.sav	11.01.2015 14:34	SAV-Datei	
my-d8.xy	11.01.2015 14:34	XY-Datei	
pw1800-ads-10mm.geq	12.06.2012 15:01	GEQ-Datei	
pw1800-ads-10mm.ger	12.06.2012 15:01	GER-Datei	

Computed peak profile

Instrument description

# Template SAV file (\*.tpl)

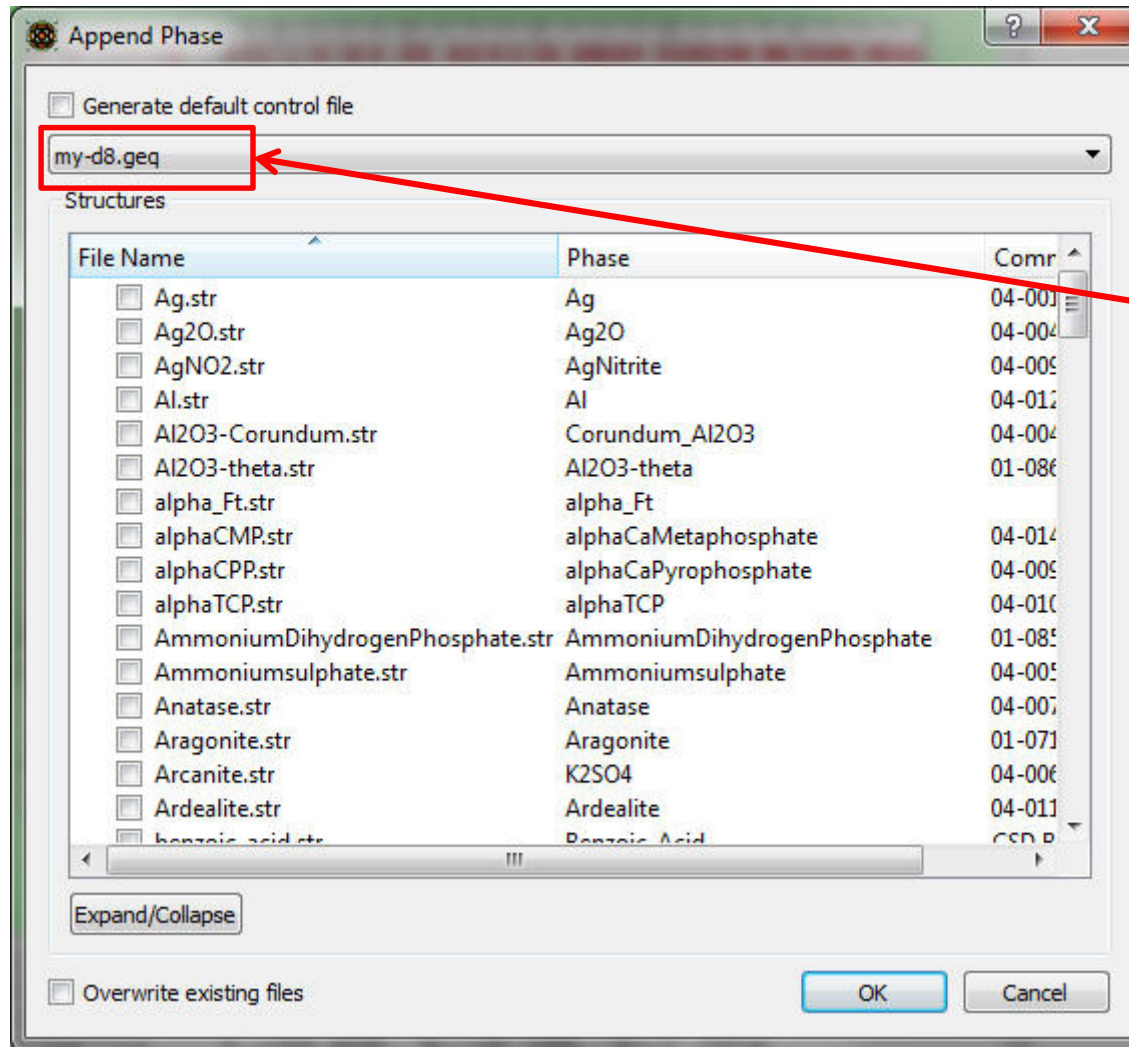
Template file for \*.sav control file:

Name	Änderungsdatum	Typ	Größe
d8-fds-02-LynxEyeXE.sav	12.02.2014 07:41	SAV-Datei	
d8-fds-02-LynxEyeXE.tpl	24.10.2013 11:33	TPL-Datei	
d8-lynxeye-ads-1mm.geq	02.07.2013 12:42	GEQ-Datei	
d8-lynxeye-ads-1mm.ger	02.07.2013 12:42	GER-Datei	
d8-lynxeye-ads-1mm.sav	12.02.2014 07:41	SAV-Datei	
d8-lynxeye-ads-1mm.tpl	08.09.2014 10:08	TPL-Datei	
d8-lynxeye-fds-02.geq	04.06.2012 18:06	GEQ-Datei	
d8-lynxeye-fds-02.ger	04.06.2012 18:06	GER-Datei	
d8-lynxeye-fds-02.sav	12.02.2014 07:41	SAV-Datei	
d8-lynxeye-fds-02.tpl	08.09.2014 10:08	TPL-Datei	
d8-lynxeye-fds-06mm.geq	12.11.2012 12:57	GEQ-Datei	
d8-lynxeye-fds-06mm.ger	12.11.2012 12:57	GER-Datei	
d8-lynxeye-fds-06mm.sav	12.02.2014 07:41	SAV-Datei	
d8-lynxeye-fds-06mm.tpl	08.09.2014 10:08	TPL-Datei	
d8-solxe-fds-0600.geq	16.01.2013 15:16	GEQ-Datei	
d8-solxe-fds-0600.ger	16.01.2013 15:15	GER-Datei	
d8-solxe-fds-0600.sav	12.02.2014 07:41	SAV-Datei	
d8-solxe-fds-0600.tpl	08.09.2014 10:08	TPL-Datei	
d8-solxe-vds-12mm.geq	16.01.2013 15:17	GEQ-Datei	
d8-solxe-vds-12mm.ger	16.01.2013 15:17	GER-Datei	
d8-solxe-vds-12mm.sav	12.02.2014 07:41	SAV-Datei	
d8-solxe-vds-12mm.tpl	08.09.2014 10:08	TPL-Datei	
my-d8.geq	11.01.2015 14:34	GEQ-Datei	
my-d8.qer	11.01.2015 14:34	GER-Datei	

Copy  
«d8-lynxeye-ads-1mm.tpl»  
to  
«my-d8.tpl»

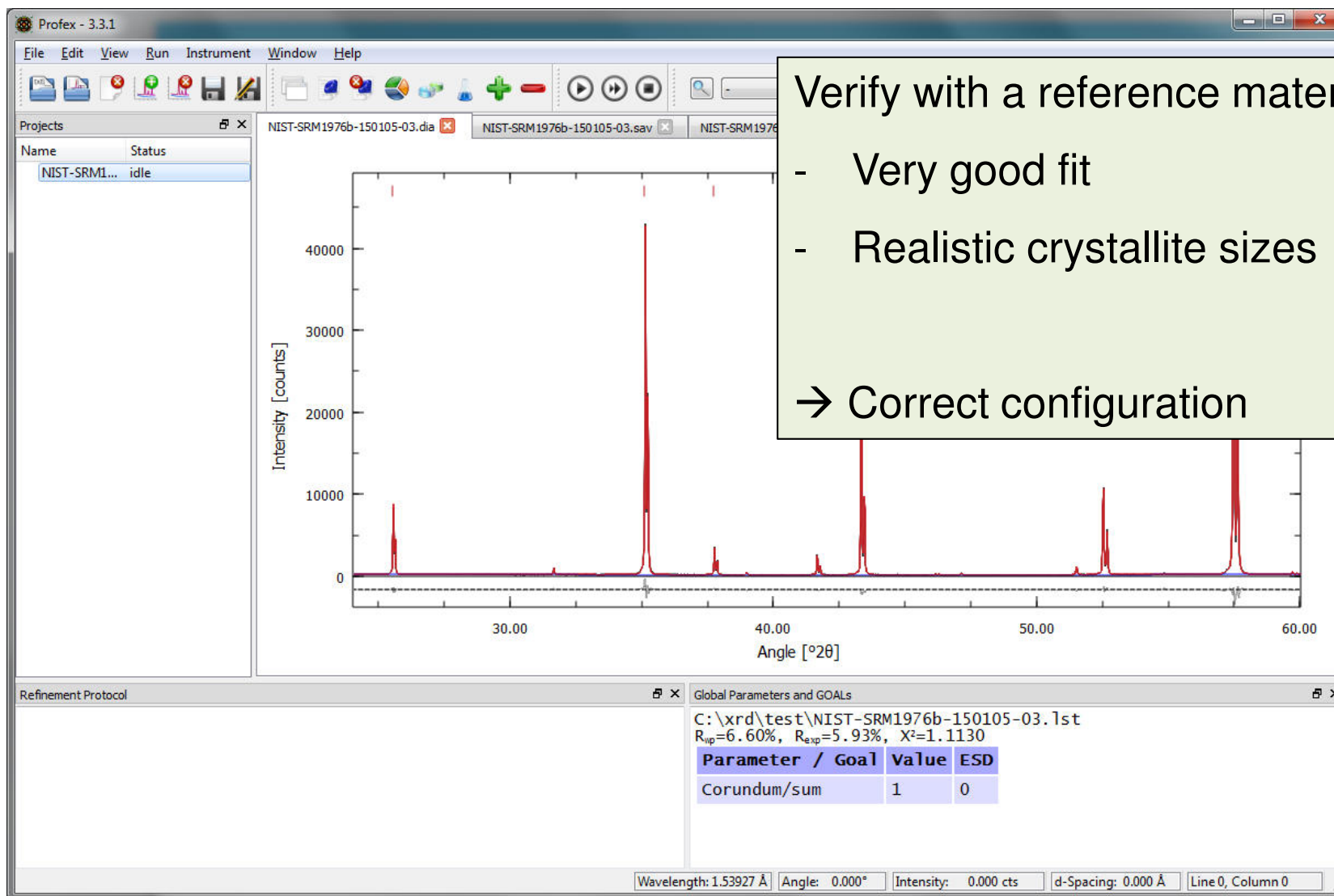


# Append Phase



In «Add Phase»  
dialog (+)

# Verification




Verify with a reference material:

- Very good fit
  - Realistic crystallite sizes
- Correct configuration

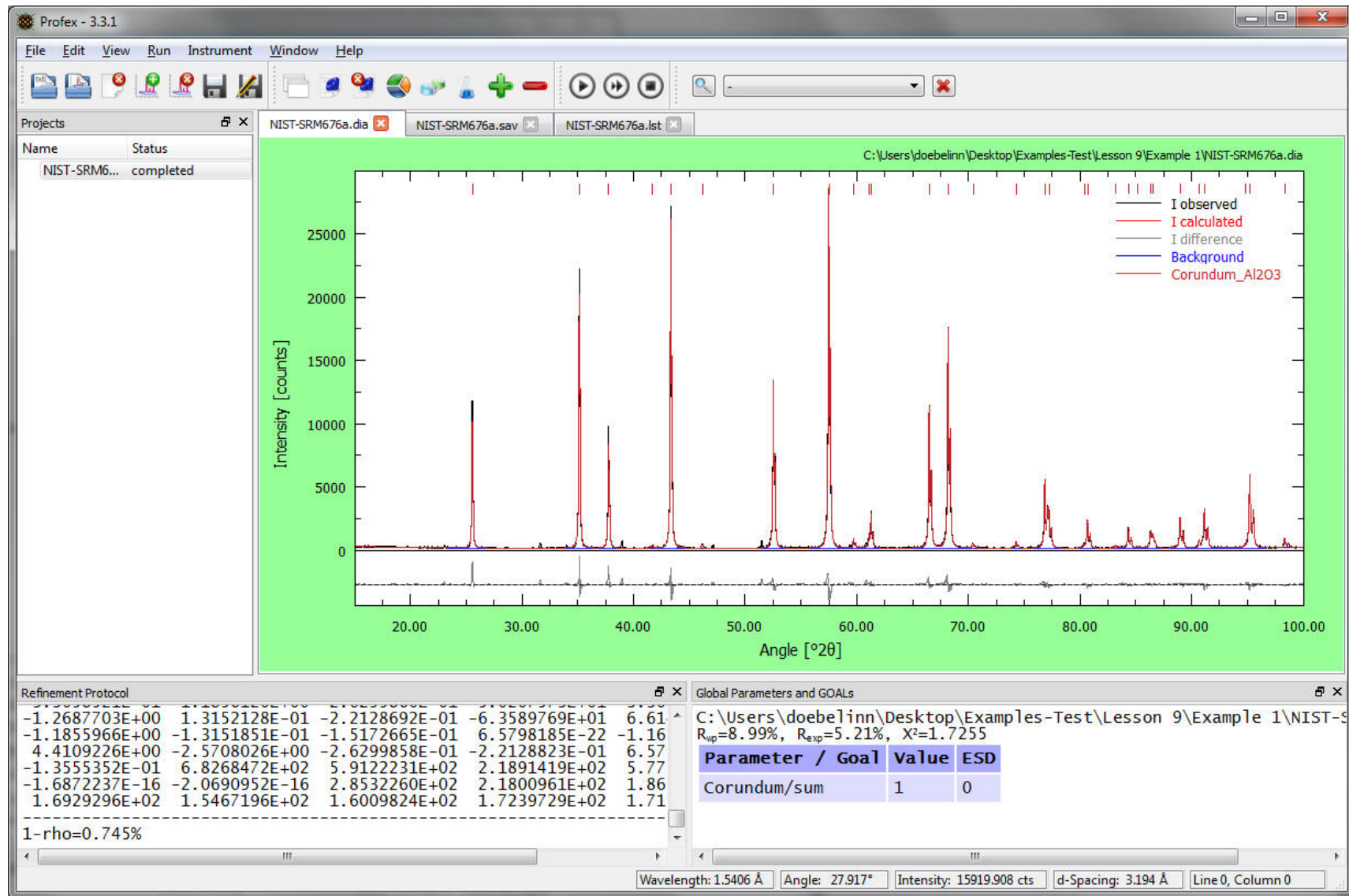


# Lesson 9: Example 1

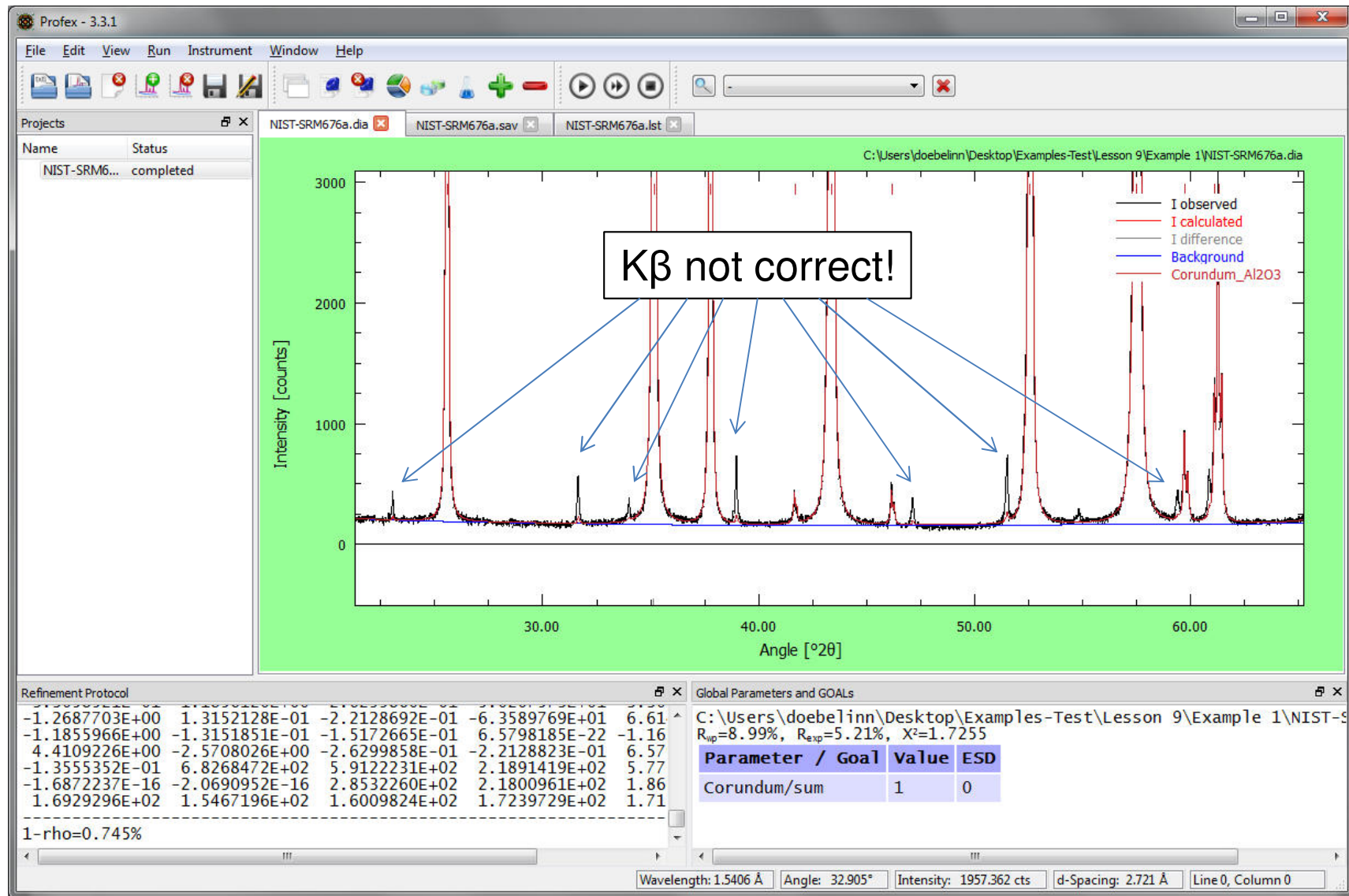
- ➔ Instrument: Bruker D8 Advance
- ➔ Geometry: Reflection
- ➔ Goniometer Radius: 350 mm
- ➔ X-ray Tube: CuKa, Target Size 12 x 0.4 mm
- ➔ Divergence Slit: Automatic, 15 mm irr. length  
Distance to sample 250 mm
- ➔ Soller Slits: 2.5° in prim. and sec. beam
- ➔ Beam Mask: Width 10.5 mm  
Distance from sample 300 mm
- ➔ Sample Diameter: 25 mm
- ➔ Anti-Scatter Slit: 9mm  
Distance to sample 260 mm
- ➔ Detector Slit: Not installed
- ➔ Detector: LynxEye XE  
Array height 14.4 mm  
Height of one strip 0.075 mm  
Array width 16.0 mm  
Number of channels 192
- ➔ Beam Knife: Not installed
- ➔ Polarization: None

1. Start from configuration  
«d8-lynxeye-ads-1mm.sav»
2. Modify according to 
3. Save under a new name  
«d8-lesson9-example1.sav»
4. «Run» to compute peak profile
5. Copy template file  
«d8-lynxeye-ads-1mm.tpl»  
to  
«d8-lesson9-example1.tpl»
6. Verify configuration with scan  
«SRM-676a.raw»

# Lesson 9: Example 1



# Lesson 9: Example 1





# Lesson 9: Example 1

The screenshot shows the Profex 3.3.1 interface. The main window displays the refinement protocol for NIST-SRM676a. The protocol includes parameters for the instrumental function, measured data, and refinement goals. A blue arrow points to the `betaratio=0.005` parameter, with the text "Increase «betaratio» manually" and "Repeat refinement" overlaid on the right side of the image.

```

% Theoretical instrumental function
VERZERR=my-d8.geq
% Wavelength
LAMBDA=CU
% Phases
STRUC[1]=A1203-Corundum.str
% Measured data
VAL[1]=NIST-SRM676a.xy
% Minimum Angle (2theta)
%WMIN=20
% Maximum Angle (2theta)
% WMAX=60
% Result list output
LIST=NIST-SRM676a.lst
% Peak list output
OUTPUT=NIST-SRM676a.par
% Diagram output
DIAGRAMM=NIST-SRM676a.dia
% Global parameters for zero point and sample displacement
EPS1=0
PARAM[1]=EPS2=0_-0.01^0.01
alpha3ratio=0.018
betaratio=0.005
NTHREADS=8
PROTOKOLL=Y

sum=Corundum
GOAL[1]=Corundum/sum
    
```

**Refinement Protocol**

-1.2687703E+00	1.3152128E-01	-2.2128692E-01	-6.3589769E+01	6.61
-1.1855966E+00	-1.3151851E-01	-1.5172665E-01	6.5798185E-22	-1.16
4.4109226E+00	-2.5708026E+00	-2.6299858E-01	-2.2128823E-01	6.57
-1.3555352E-01	6.8268472E+02	5.9122231E+02	2.1891419E+02	5.77
-1.6872237E-16	-2.0690952E-16	2.8532260E+02	2.1800961E+02	1.86
1.6929296E+02	1.5467196E+02	1.6009824E+02	1.7239729E+02	1.71

1-rho=0.745%

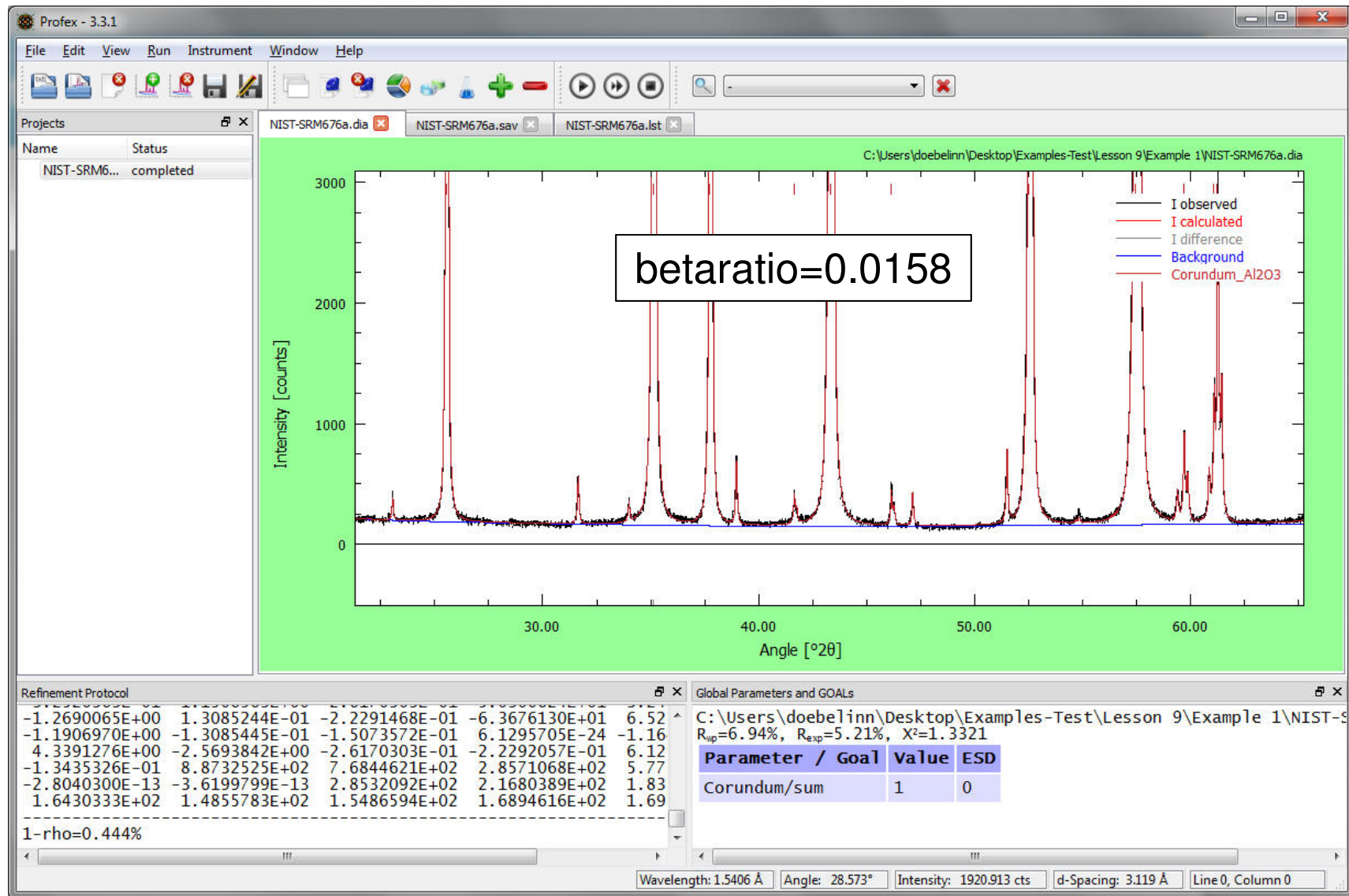
**Global Parameters and GOALS**

C:\Users\doebelinn\Desktop\Examples-Test\Lesson 9\Example 1\NIST-SRM676a.par  
 $R_{wp}=8.99\%$ ,  $R_{exp}=5.21\%$ ,  $X^2=1.7255$

Parameter / Goal	Value	ESD
Corundum/sum	1	0

Wavelength: 1.5406 Å | Angle: 0.000° | Intensity: 0.000 cts | d-Spacing: 0.000 Å | Line 0, Column 0

# Lesson 9: Example 1



# Lesson 9: Example 1

- Verify refined unit cell dimensions and crystallite size with SRM certificate

**National Institute of Standards & Technology**  
**Certificate of Analysis**  
 Standard Reference Material® 676a  
 Alumina Powder for Quantitative Analysis by X-ray Diffraction

This Standard Reference Material (SRM) consists of an alumina powder (corundum structure) intended primarily for use as an internal standard for quantitative analysis and  $I/I_c$  [1] (for a complete discussion of  $I/I_c$ , see [2]) determinations by X-ray powder diffraction. A unit of SRM 676a consists of approximately 20 g of powder bottled in an argon atmosphere.

**Material Description:** The alumina powder has been in a de-aggregated state. The alumina grains are sub-micrometer in size and the isometric form of the grains ensures high phase purity while the isometric form of the grains in this powder. The de-aggregated state of this material is maintained by conventional methods.

An analysis of the phase fractions determined from X-ray powder diffraction, SRM 640c [3], indicated that the SRM 676a is 100%  $\alpha$ -alumina.

**Certified Value:** The certified phase purity of the material is 100%  $\alpha$ -alumina.

Crystalline Alumina

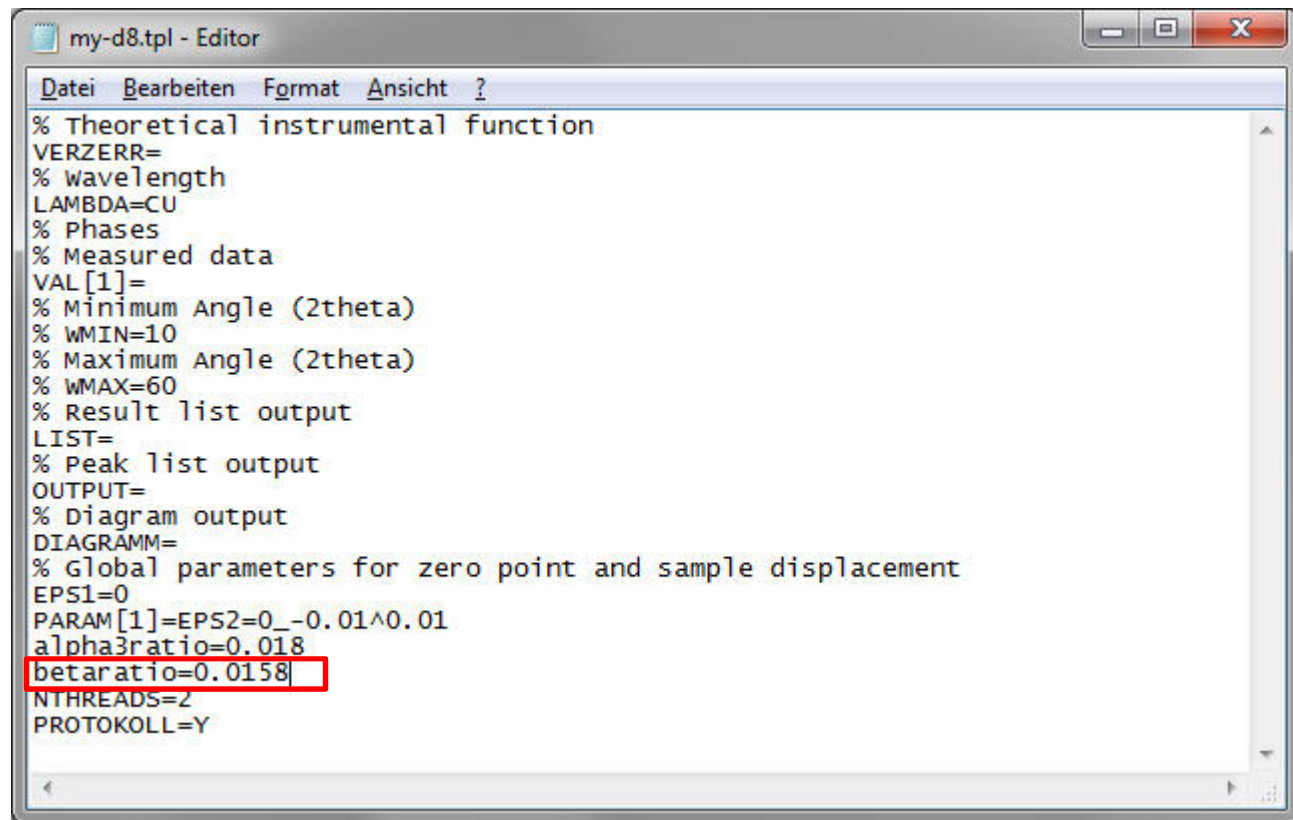
The interval defined by the certified value and its uncertainty is the range of values that is in excess of the absence of systematic error [4,5]. The error bounds define a range about the certified value that is in excess of 100% phase purity, a physical impossibility.

	Certified	Refined
Unit cell $a$	0.475936 nm	0.476021 nm
Unit cell $c$	1.29923 nm	1.299485 nm
Crystallite size	Sub-micron	131 nm



# Lesson 9: Example 1

- Write `betaratio=0.0158` to  
«`d8-lesson9-example1.tpl`»



```
my-d8.tpl - Editor
Datei Bearbeiten Format Ansicht ?
% Theoretical instrumental function
VERZERR=
% wavelength
LAMBDA=CU
% Phases
% Measured data
VAL[1]=
% Minimum Angle (2theta)
% WMIN=10
% Maximum Angle (2theta)
% WMAX=60
% Result list output
LIST=
% Peak list output
OUTPUT=
% Diagram output
DIAGRAMM=
% Global parameters for zero point and sample displacement
EPS1=0
PARAM[1]=EPS2=0_-0.01^0.01
alpha3ratio=0.018
betaratio=0.0158
NTHREADS=2
PROTOKOLL=Y
```