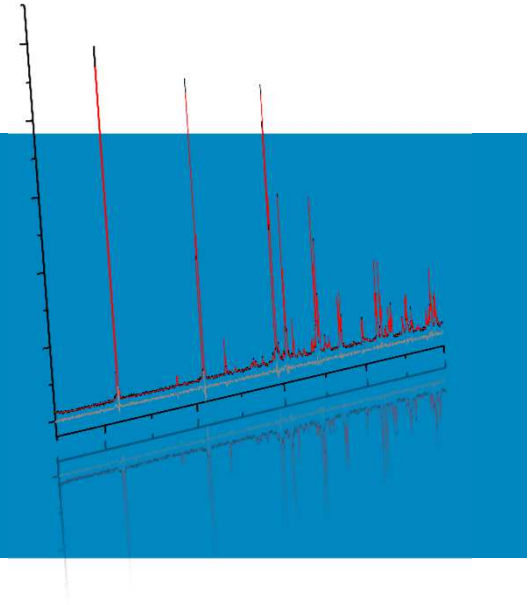
Lesson 5
BGMN & Profex



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Repetition: BGMN

BGMN:

- Fundamental Parameters Approach
- Free for academic use
- Device independent
- Very robust automatic refinement strategy
- Good usability
- Slightly less steep learning curve
- Powerful scripting language
- Multi-Platform
- Multi-threaded

Visit: http://www.bgmn.de for tutorials and documentation

BGMN Website

Program Description

Site map

Up | Program Description | BGMN FAQ | Download Page

- Instrumental Function
- Tube Tails and size/strain estimation
- Structure Description
- Download Structures
- Preferred Orientation
- Calculation Control
- Result Output
- BGMN Variables

The following pages serve as a short introduction into the BGMN Rietveld software. A description of an example (plaster) makes your test runs easier.

First, the generation of the instrumental function is described.

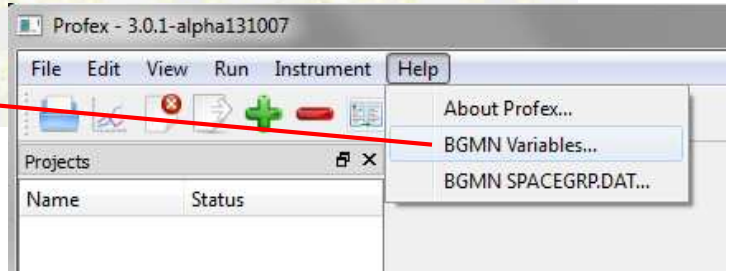
Second, the construction of structure files is explained. In an extra topic the handling of preferred orientation is mentioned in detail.

Third, the calculation control during a problem specific control file is shown.

Last, we have a look at the different result files.

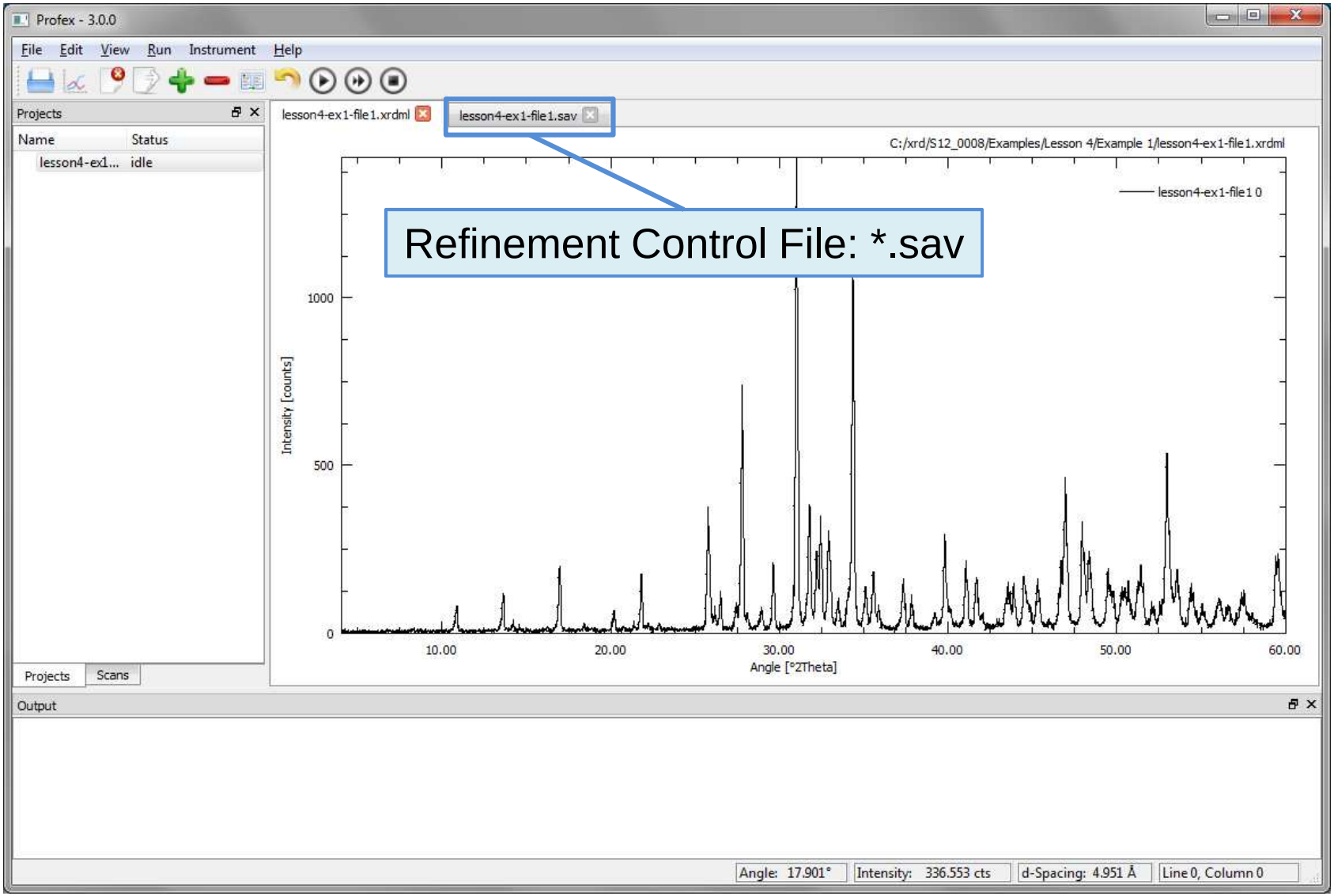
A somewhat extended explanation of the used variables is also available.

opens website

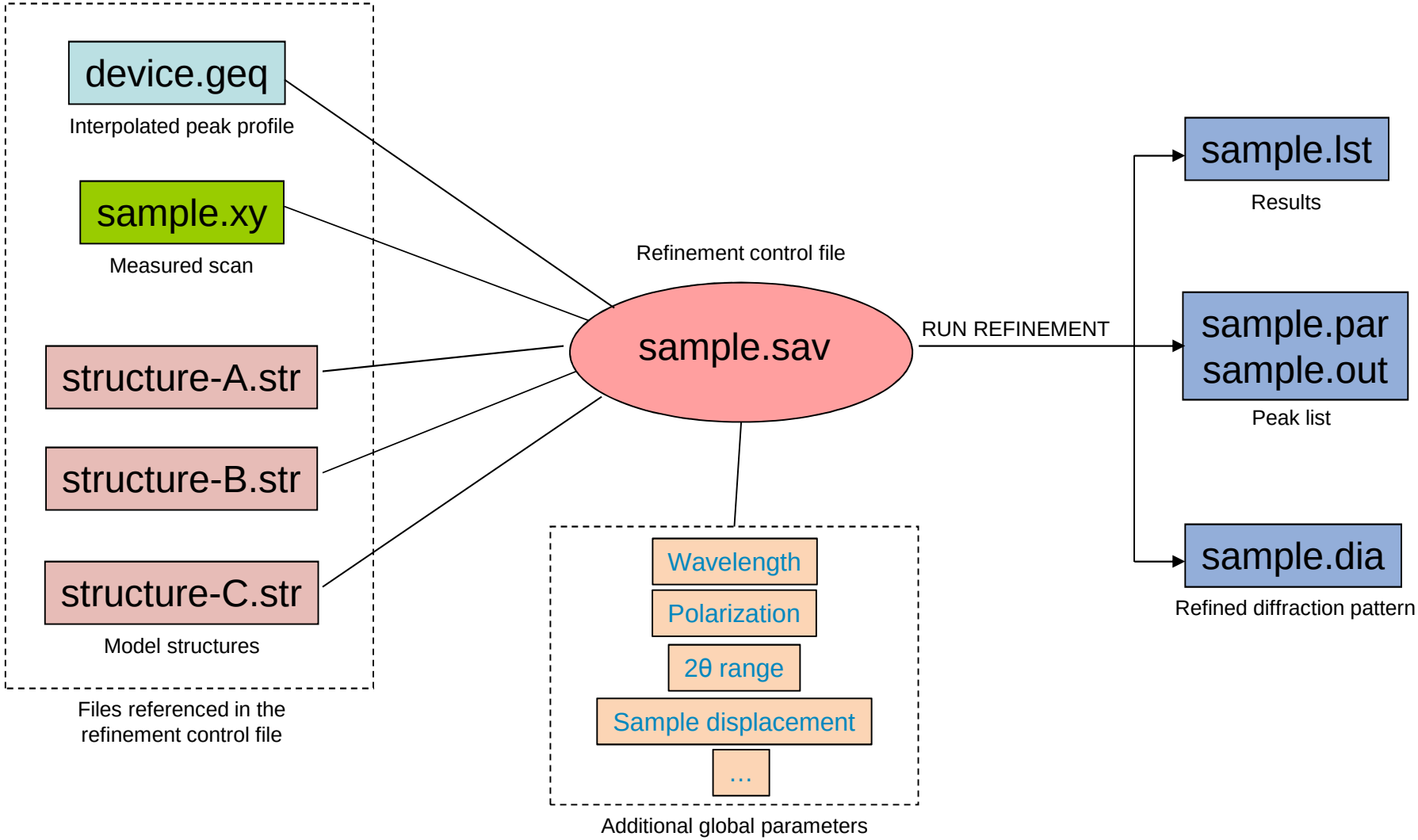


http://www.bgmn.de/program.html

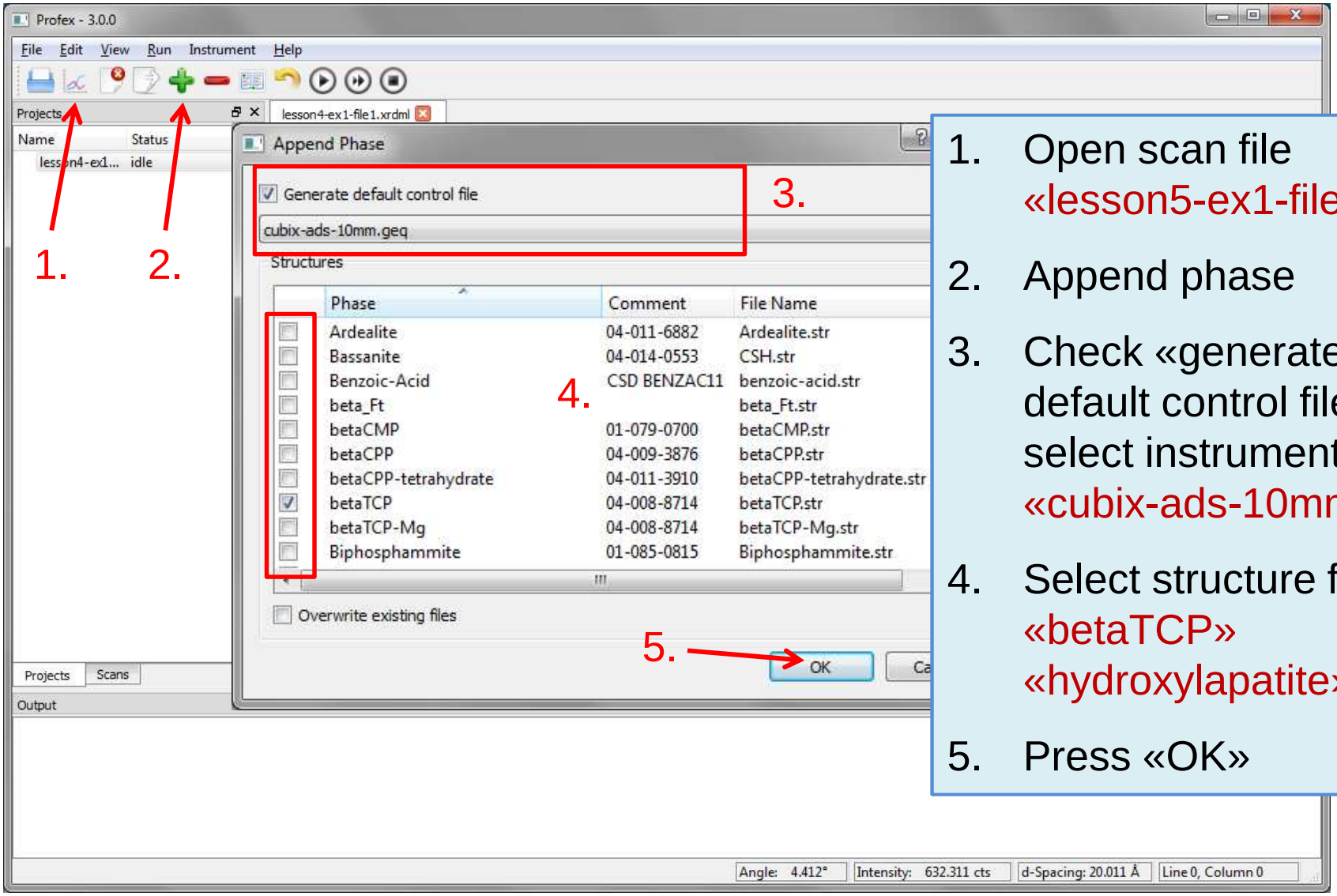
BGMN Program Structure



BGMN Control File (*.sav)

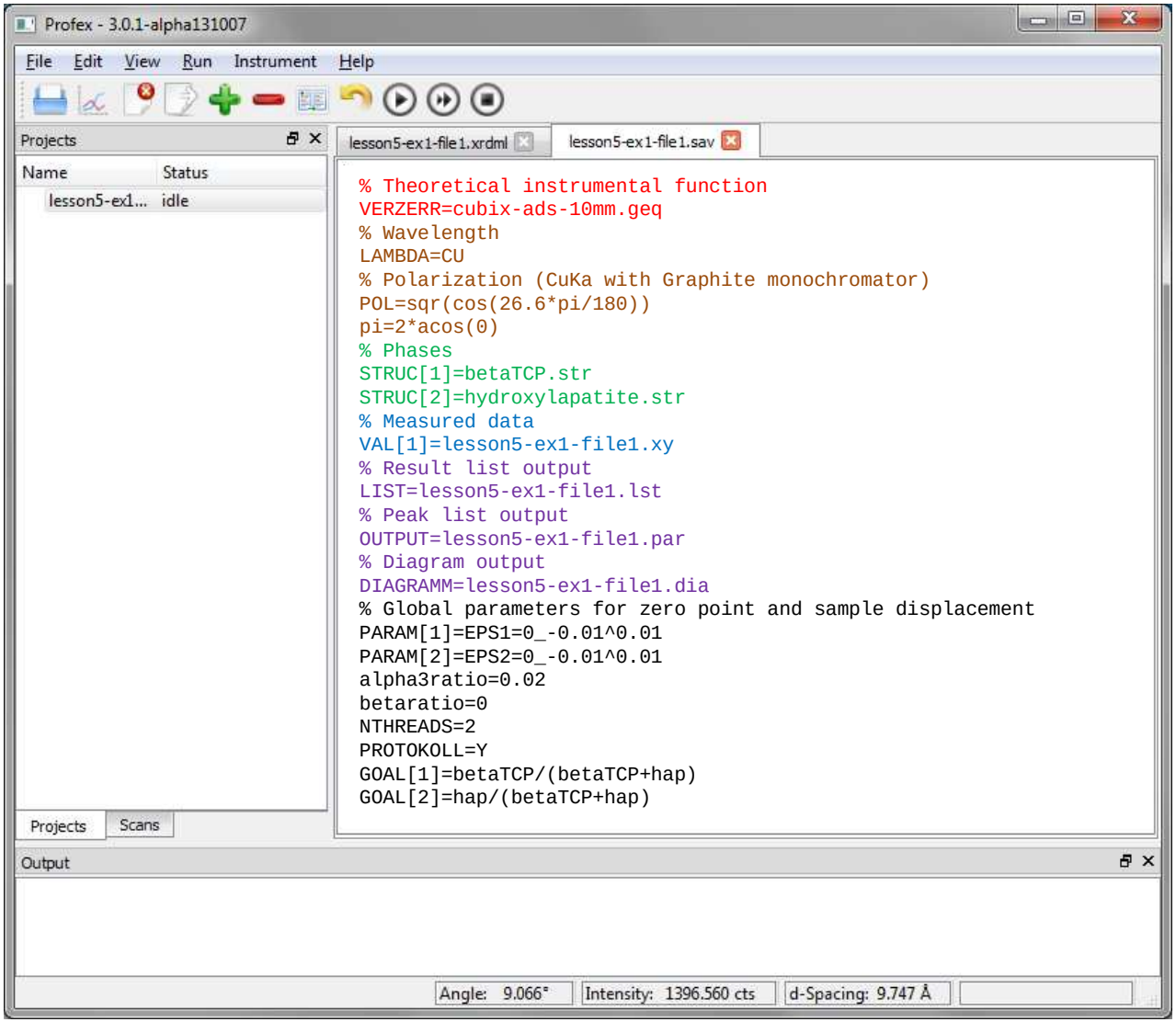


Lesson 5: Example 1



1. Open scan file «lesson5-ex1-file1»
2. Append phase
3. Check «generate default control file», select instrument «cubix-ads-10mm»
4. Select structure files «betaTCP» «hydroxylapatite»
5. Press «OK»

Lesson 5: Example 1



Instrument profile

Wavelength and polarization

Refined phases

Measured scan

Various output files
(may be empty before running BGMN)

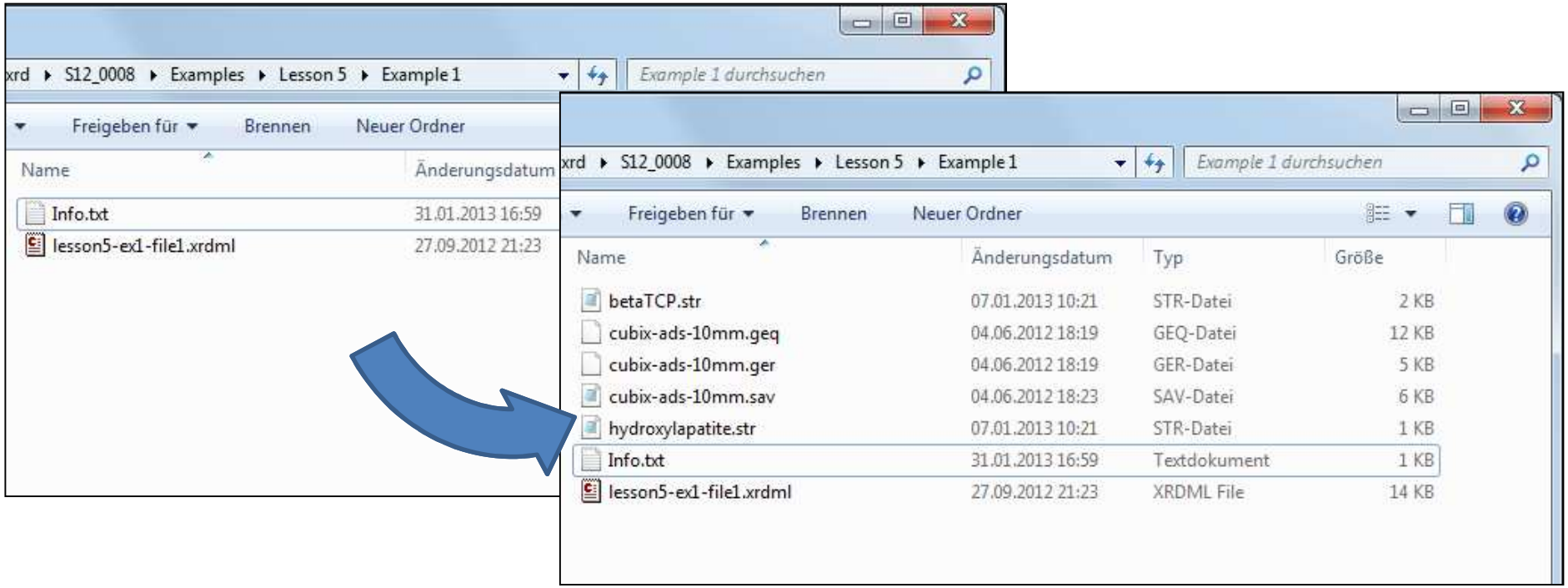
Various global parameters

- Zero-shift (EPS1)
- Sample displacement (EPS2)
- Sample transparency (EPS3)
- Calculation of phase quantities (GOAL[x])

BGMN Control File (*.sav)

What Profex does in the background:

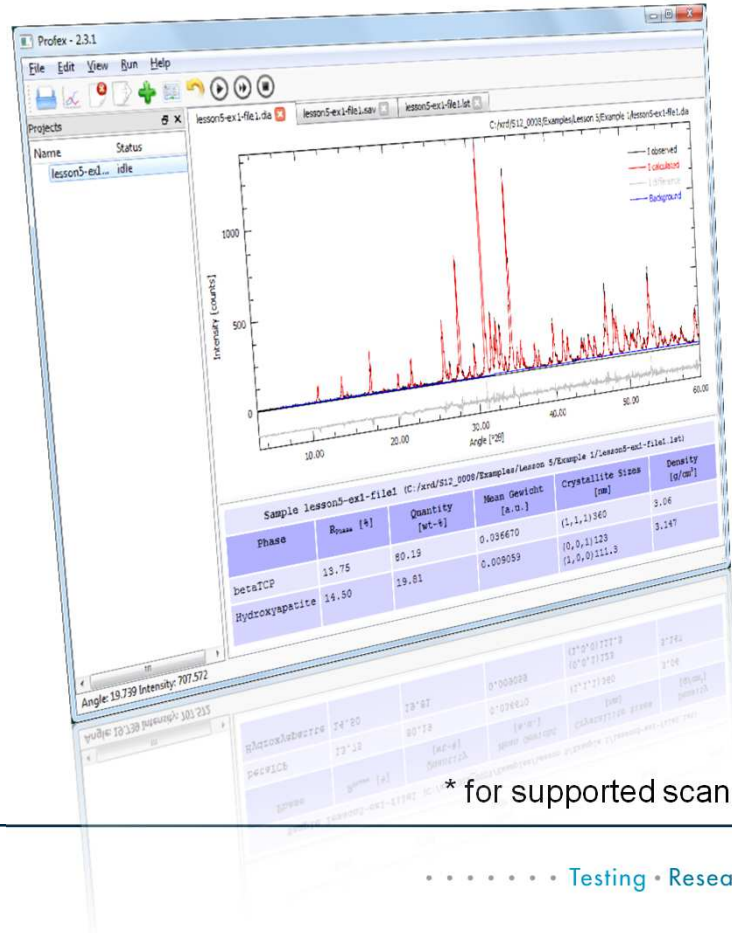
- Generate a control file
- Copy all selected structure files from local DB to location of scan file
- Copy instrument configuration file from local DB to location of scan file
- Adjust file names in control file



Profex

With Profex:

1. Load scan file
2. Use «Append phase» dialog to select phases, instrument, and generate control file
3. Run refinement

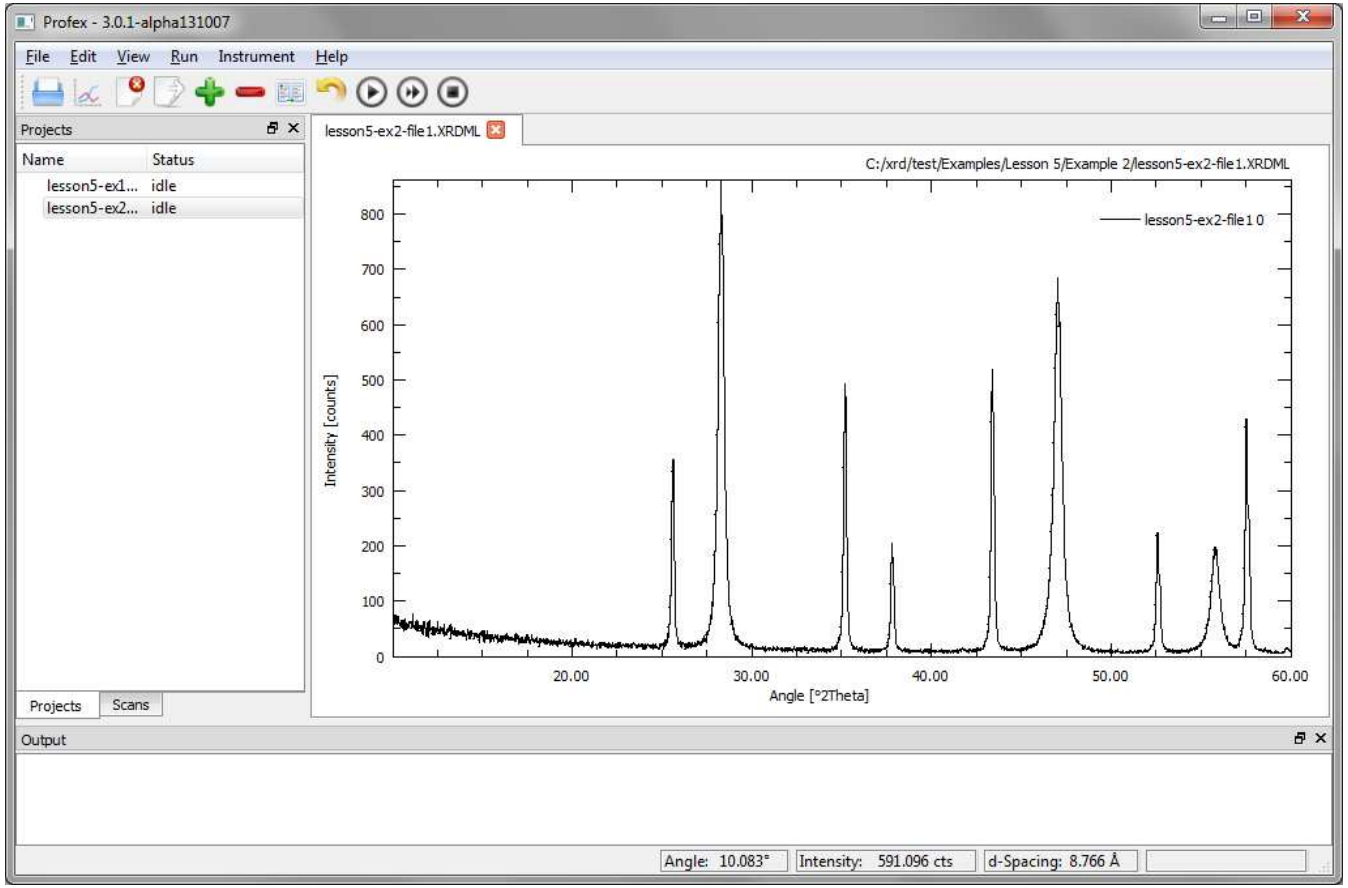


No need to:

- Copy structure / device files
- Change any file names
- Convert scan files*

* for supported scan file formats

Lesson 5: Example 2



Instrument:

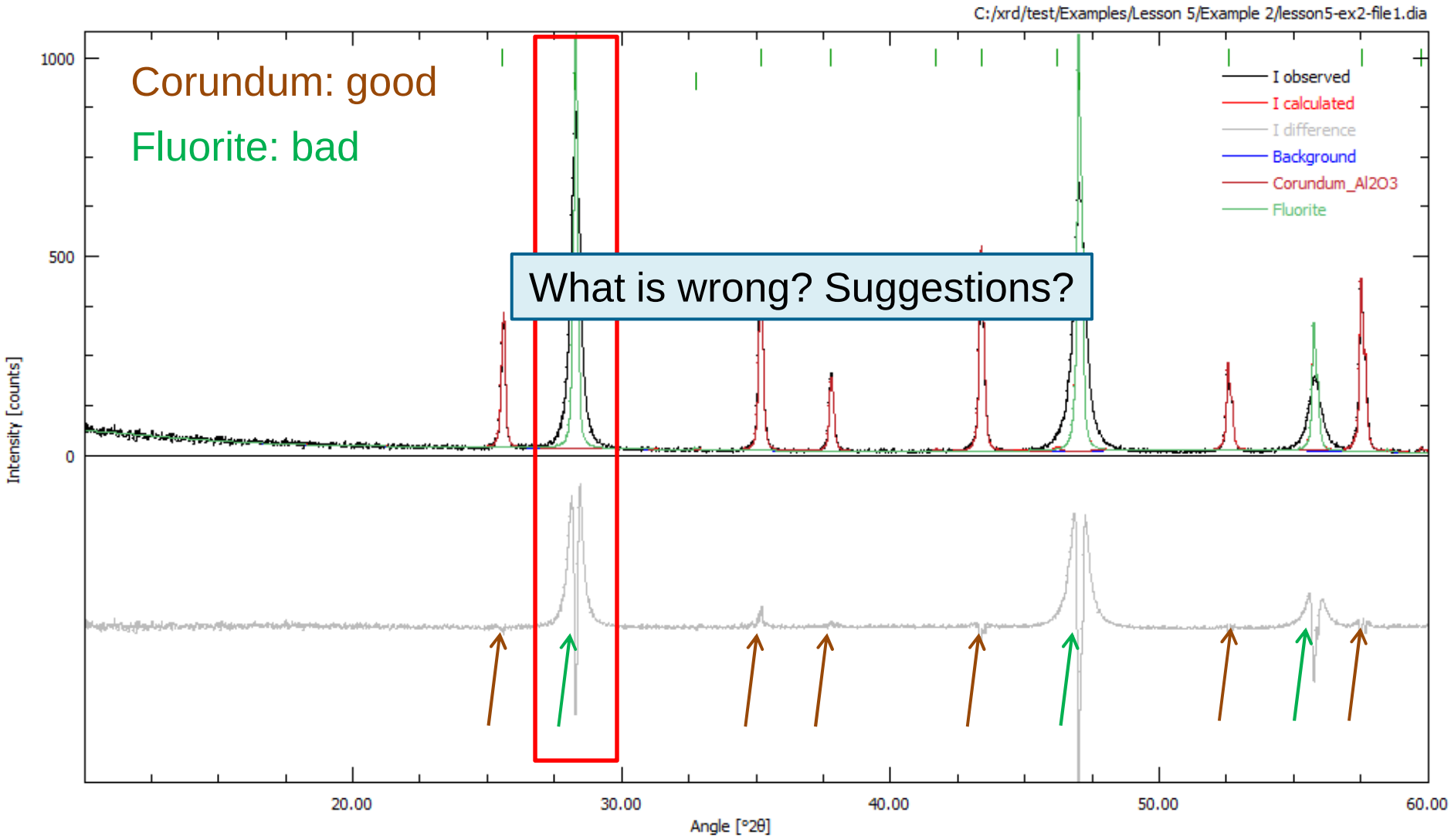
«pw1800-fds-0250»

Phases:

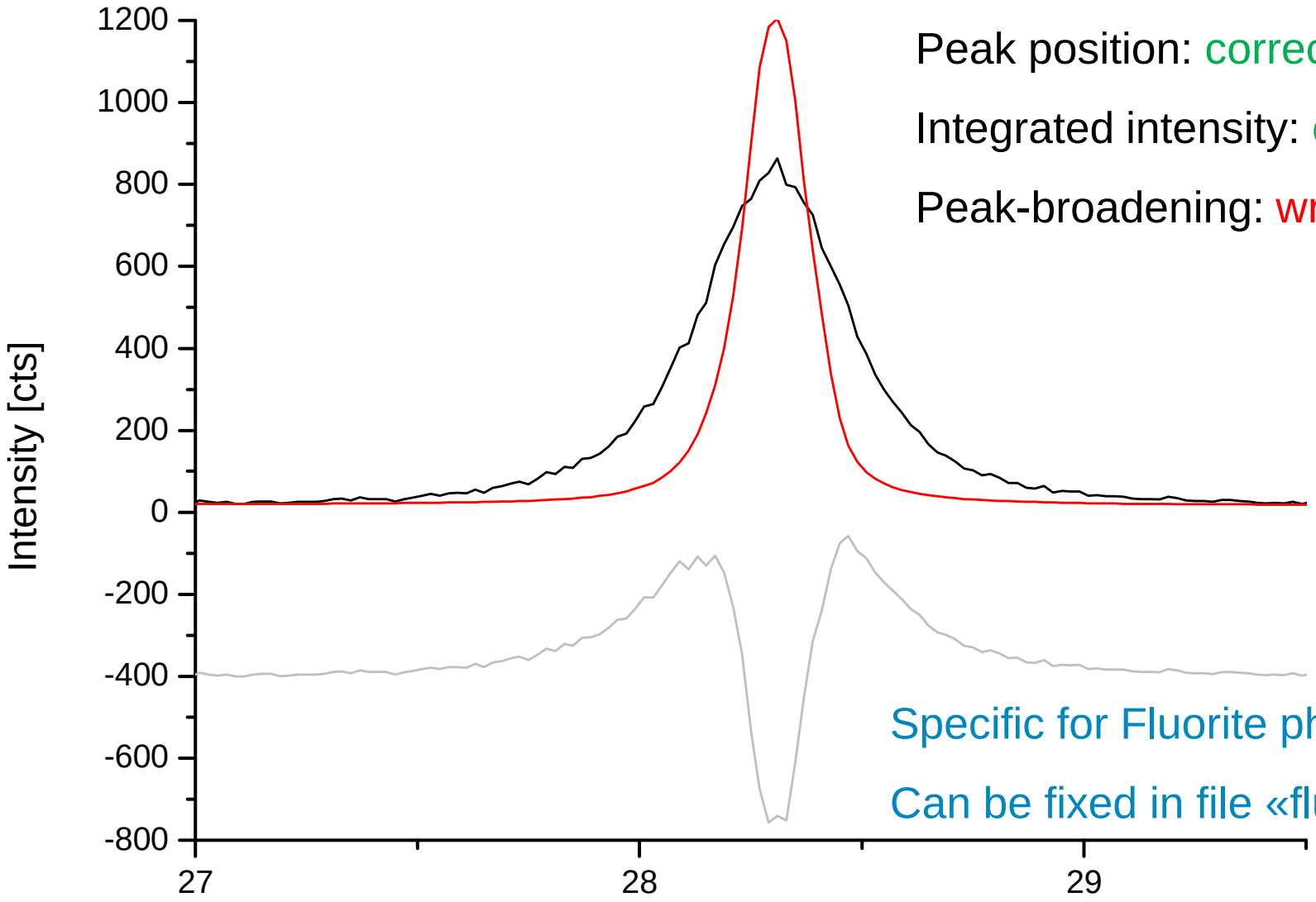
«Corundum_AL2O3»

«Fluorite»

Lesson 5: Example 2



Lesson 5: Example 2



Peak position: correct

Integrated intensity: correct (?)

Peak-broadening: wrong

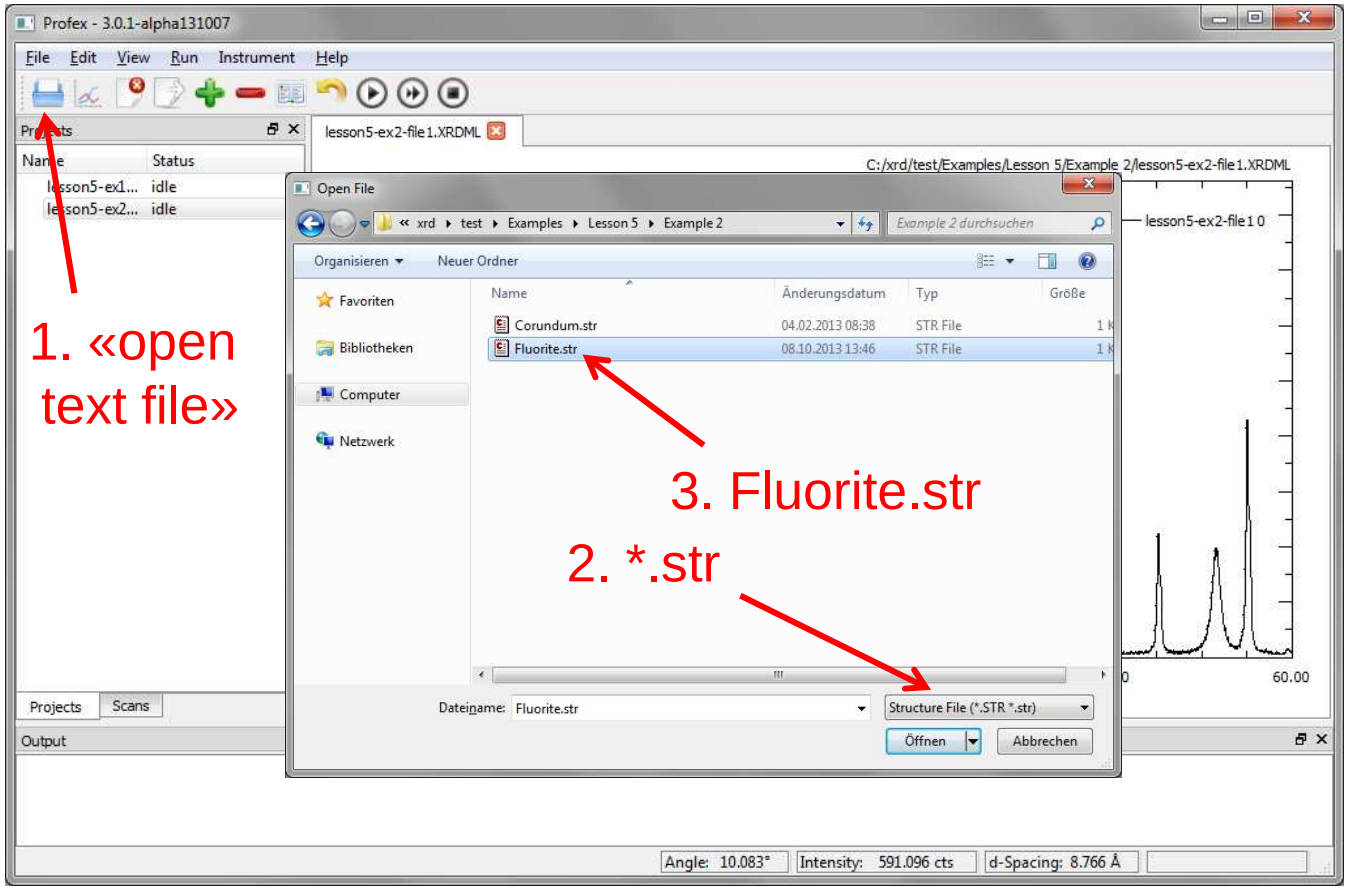
Specific for Fluorite phase.

Can be fixed in file «fluorite.str»

BGMN Structure Files (*.str)

Structure files contain a description of the crystal structure.

Similar to CIF files, but different file format.



BGMN Structure Files (*.str)

```
PHASE=Fluorite // 04-002-2191
SpacegroupNo=225 HermannMauguin=F4/m-32/m //
PARAM=A=0.5463_0.54^0.55 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.003 PARAM=GEWICHT=0.1_0 //
d=10 //
GOAL=GrainSize(1,1,1) //
GOAL=my //
GOAL=d //
GOAL:fluorite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)
E=CA+2 Wyckoff=a TDS=0.0041
E=F-1 Wyckoff=c TDS=0.0062
```

Phase name // PDF code

Space group number & H-M symbol

Unit Cell: A axis (nm)

Peak profile parameters

Mean particle size (µm), optional

Goals (optional, these values are reported in the results file)

List of atomic positions

Full notation:

E=CA+2(1) Wyckoff=a x=0.0 y=0.0 z=0.0 TDS=0.0041

Element

Wyckoff sequence

Thermal displacement parameter (B_{iso} [nm^2])

Site occupancy

Fractional coordinates

BGMN Structure Files (*.str)

```
PHASE=Fluorite // 04-002-2191
SpacegroupNo=225 HermannMauguin=F4/m-32/m //
PARAM=A=0.5463_0.54^0.55 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.003 PARAM=GEWICHT=0.1_0 //
d=10 //
GOAL=GrainSize(1,1,1) //
GOAL=my //
GOAL=d //
GOAL:fluorite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)
E=CA+2 Wyckoff=a TDS=0.0041
E=F-1 Wyckoff=c TDS=0.0062
```

Fix parameter:	Refined parameter:	Refined parameter with limits:
A=0.5463	PARAM=A=0.5463	PARAM=A=0.5463_0.54^0.55
Name Value	Release for refinement Name Starting value	Release for refinement Name Starting value Lower limit Upper limit

Lesson 5: Example 2

```
PHASE=Fluorite // 04-002-2191
SpacegroupNo=225 HermannMauguin=F4/m-32/m //
PARAM=A=0.5463_0.54^0.55 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.003 PARAM=GEWICHT=0.1_0 //
d=10 //
GOAL=GrainSize(1,1,1) //
GOAL=my //
GOAL=d //
GOAL:fluorite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)
E=CA+2 Wyckoff=a TDS=0.0041
E=F-1 Wyckoff=c TDS=0.0062
```

In Lesson 5, Example 2, peak broadening was not fitted correctly

Peak profile is controlled here

RP=4: usage of both Lorentzian and squared Lorentzian broadening

B1: lorentzian broadening caused by size effects

k1: gaussian-like part of the size effects

k2: micro strain effect

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

Lesson 5: Example 2

```
PHASE=Fluorite // 04-002-2191
SpacegroupNo=225 HermannMauguin=F4/m-32/m //
PARAM=A=0.5463_0.54^0.55 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.003 PARAM=GEWICHT=0.1_0 //
d=10 //
GOAL=GrainSize(1,1,1) //
GOAL=my //
GOAL=d //
GOAL:fluorite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)
E=CA+2 Wyckoff=a TDS=0.0041
E=F-1 Wyckoff=c TDS=0.0062
```

RP=4: uses all possible broadening contributions (good)

B1: refined, but limited to the range 0.000 – 0.003 (upper limit may be too strict)

k1: gaussian size effect: not refined (bad)

k2: micro strain effect: not refined (necessary?)

Lesson 5: Example 2

Change:

```
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.003 PARAM=GEWICHT=0.1_0 //
```

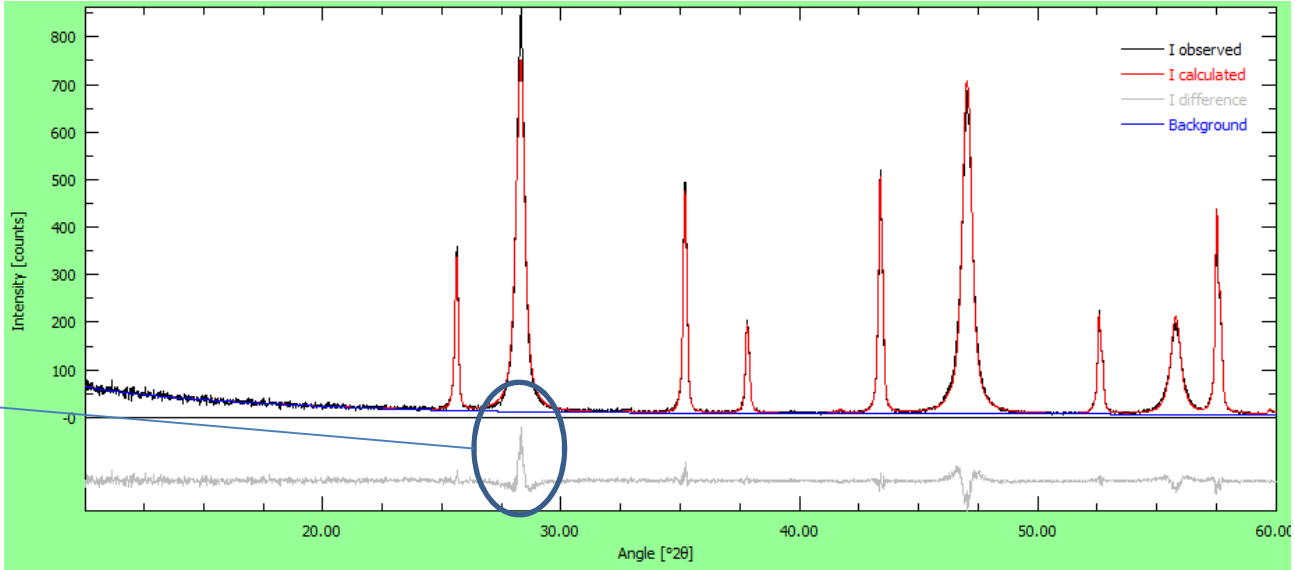
To:

```
RP=4 PARAM=k1=0_0^1 k2=0 PARAM=B1=0_0^0.03 PARAM=GEWICHT=0.1_0 //
```

Repeat Refinement

Better, but not perfect.

Is there microstrain?



Lesson 5: Example 2

Change:

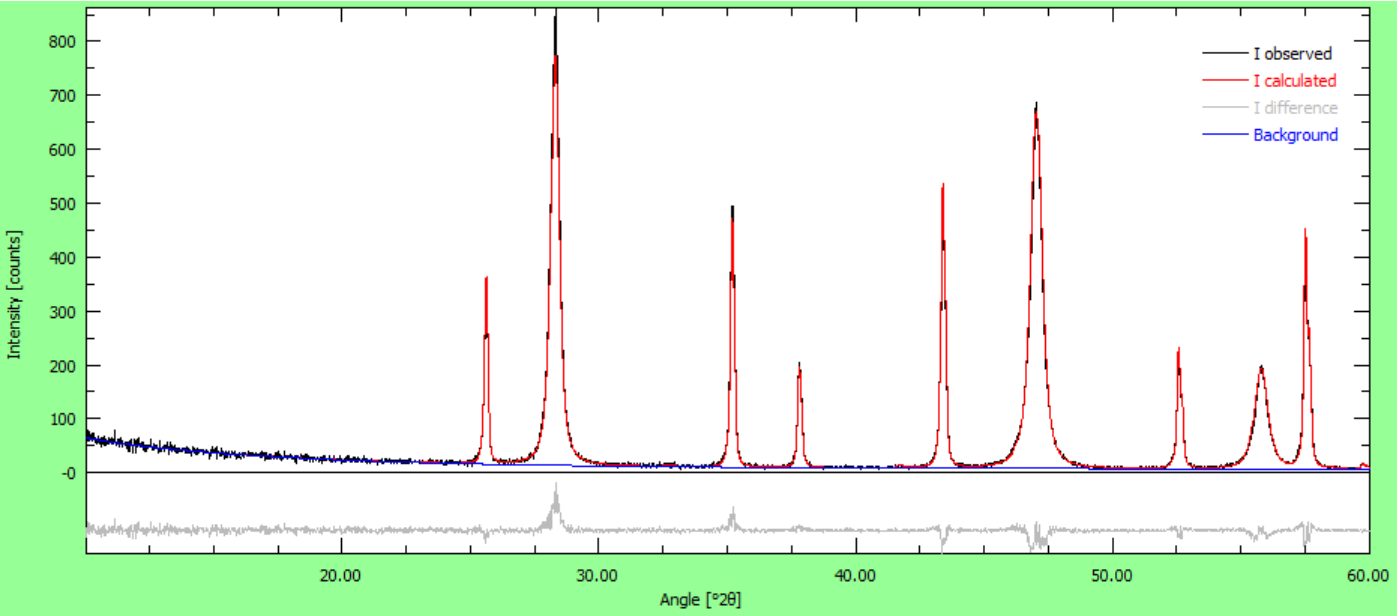
```
RP=4 PARAM=k1=0_0^1 k2=0 PARAM=B1=0_0^0.03 PARAM=GEWICHT=0.1_0 //
```

To:

```
RP=4 PARAM=k1=0_0^1 PARAM=k2=0_0 PARAM=B1=0_0^0.03 PARAM=GEWICHT=0.1_0 //
```

Repeat Refinement

Better, but still not perfect.



Lesson 5: Example 2

Preferred Orientation:

- Refine «GEWICHT» as «SPHAR6»
- Remove «PARAM=»
- Explanation follows later

Change:

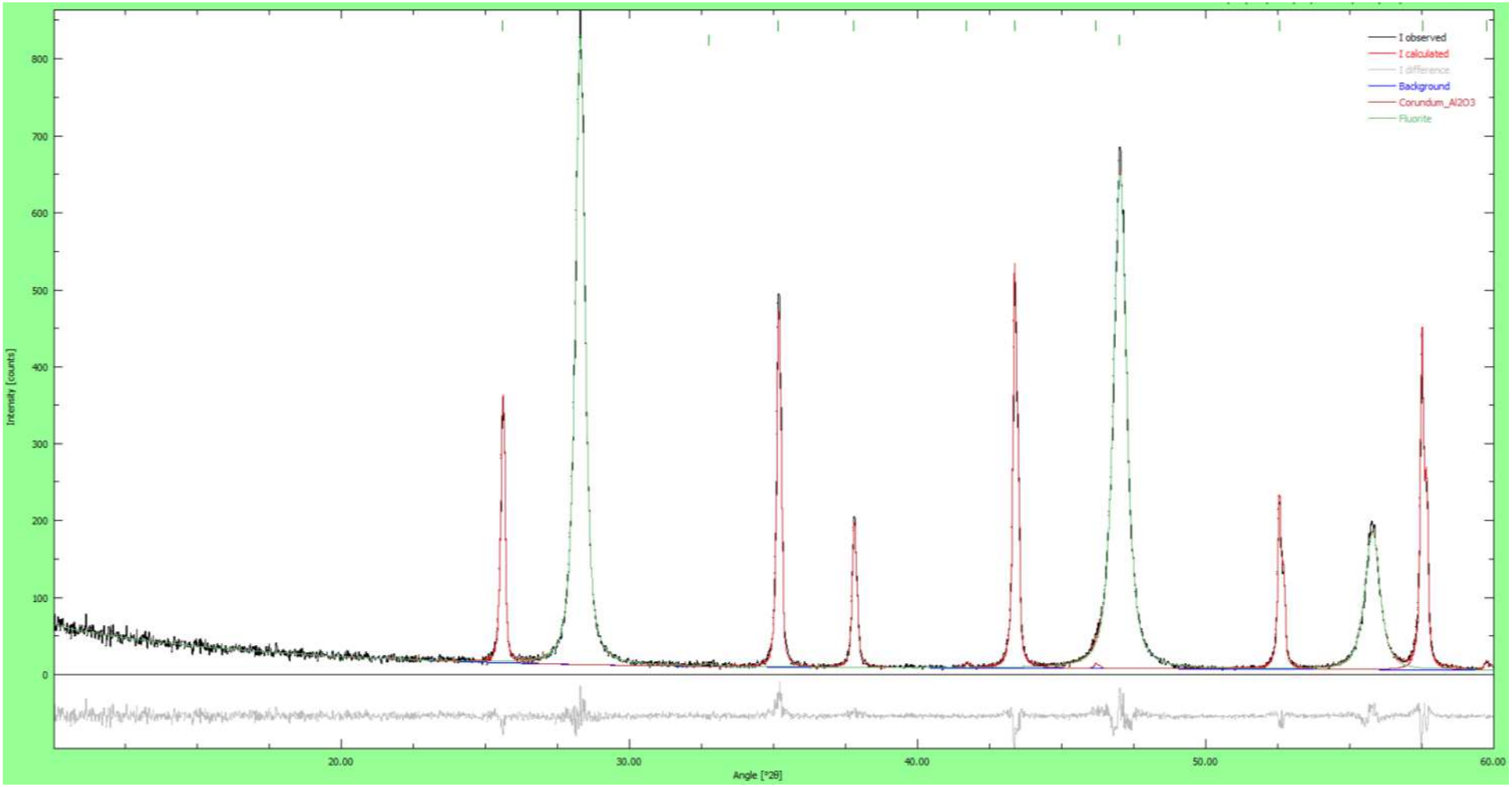
```
RP=4 PARAM=k1=0_0^1 PARAM=k2=0_0 PARAM=B1=0_0^0.03 PARAM=GEWICHT=0.1_0 //
```

To:

```
RP=4 PARAM=k1=0_0^1 PARAM=k2=0_0 PARAM=B1=0_0^0.03 GEWICHT=SPHAR6 //
```

Repeat Refinement

Lesson 5: Example 2



Summary: Structure Files (*.str)

- Structure file database (*.str) is stored in C:\Program Files\Profex-x.y.z\Structures
- Copied by Profex to the location of the scan file
- They contain:
  - Space group information
  - Unit cell dimensions
  - Profile parameters
  - Scaling (weight)
  - List of atoms (element, SOF, Wyckoff sequence, fract. coordinates, TDS)
  - Optional «Goals»: Results / values printed to the results file (*.lst)
- Release parameters for refinement:
  - «PARAM=»
  - Optionally (recommended!): «_lowerLimit» and «^upperLimit»

Summary: Structure Files (*.str)

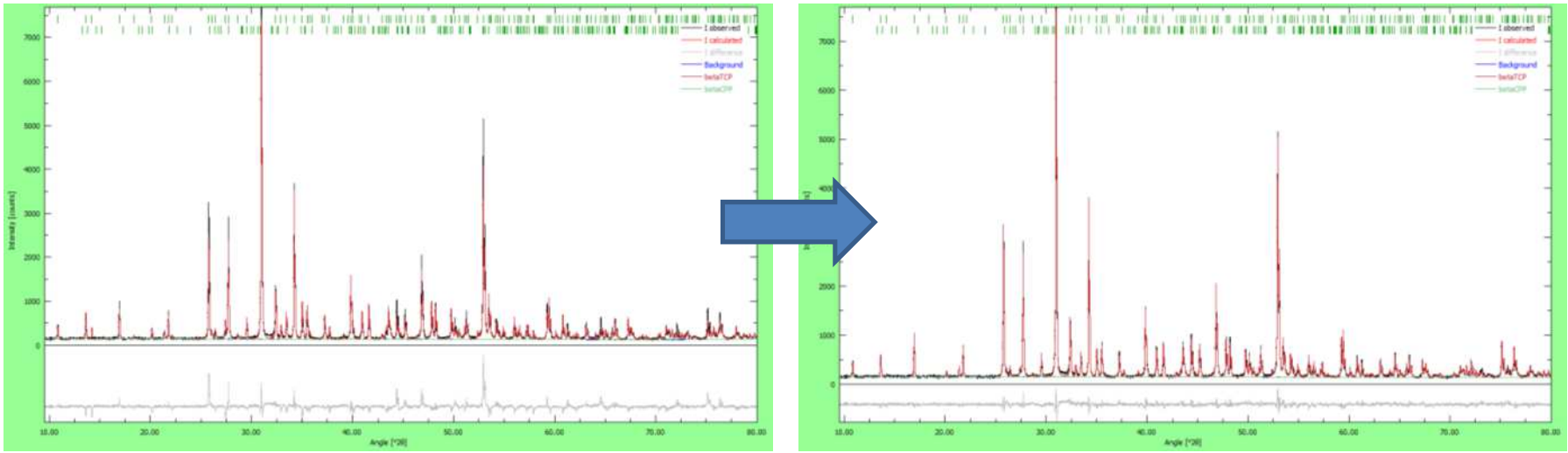
Refinements are «fine tuned» by editing Structure Files:

Commonly:

- Set reasonable upper and lower limits
- Control peak broadening
- Control texture / preferred orientation

Rarely:

- Control structural parameters:
  - atomic coordinates
  - site occupancies
  - thermal displacement parameters



Structure Databases

- STR files shipped with Profex (created manually by Nicola Doebelin)
- http://www.bgmn.de/download-structures.html

- Create manually from:
  - ICSD (http://www.fiz-karlsruhe.de/icsd.html)
  - PDF-4+ (http://www.icdd.com/)
  - American Mineralogist Structure Database (http://rruff.geo.arizona.edu/AMS/amcsd.php)
  - Crystallography Open Database COD (http://www.crystallography.net/)
  - Cambridge Crystallographic Data Centre (http://beta-www.ccdc.cam.ac.uk/pages/Home.aspx)

Creating Structure Files

Official Documentation:

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

Control File (*.sav)

Structure File (*.str)

Device Conf. File (*.sav)

Variables for use with BGMN

The expression interpreter

- standard functions
- multiple equation symbols in assignments
- user-defined string functions

Variables in the task description SAV file for BGMN

NTHREADS, VAL, VERZERR, STRUC, STRUCOUT, SimpleSTRUCOUT, PDBOUT, RESOUT, FCFOUT, OUTPUT, LIST, RU, UNT, UNTC, DDM, PARAM, LAMBDA, SYNCHROTRON, EPS1, EPS2, EPS3, EPS4, POL, PROTOKOLL, ONLYISO, ITMAX, DIAGRAMM, PLAN, STANDALONEPLAN, GOAL, WMIN, WMAX, CUT, LIMIT2, LIMIT4, LIMIT6, LIMIT8, LIMIT10, ANISOLIMIT, ANISO4LIMIT

Items in the structure description *.str-file

Definition of the lattice
SpacegroupNo, HermannMauguin, GeneralCondition, A, B, C, ALPHA, BETA, GAMMA, UNIT

Anisotropic variables
ANISO, ANISOLIN, ANISOSQR, ANISO4

Scaling factor/prefered orientation
GEWICHT, SPHAR0, SPHAR2, SPHAR4, SPHAR6, SPHAR8, SPHAR10

Real structure
B1, B2, k1, k2, k3, sk, H, h, k, l, zweiTheta, RP

Peak deselection
GeneralCondition

Atomic positions
E=, Wyckoff, x, y, z, TDS, B, betaij, U, Uij

GOAL's, quant analysis
GOAL, GEWICHT

BGMN-specific functions
PHASE, sk, B1, k2, ANISO, GEWICHT(h,k,l), GrainSize, TDS,

Advanced feature: subphases
RefMult, iref, GEWICHT[i], B1[i], B2[i], k1[i], k2[i], k3[i], DELTAsk, DELTAzweiTheta, LeBail, FPARAM

Advanced feature: Structure amplitudes etc
F, Finv, H

Advanced feature: User calculated structure amplitudes
FMult, F[i], phi[i], Finv[i], phiinv[i]

Advanced feature: molecules
set, setgitter, cross, diffvec, normvec, skalpro, distance, angle, cpXYZ, T, D, X, Y, Z, WW, WWalt, Straf, Theory, Bondings, BondLevel

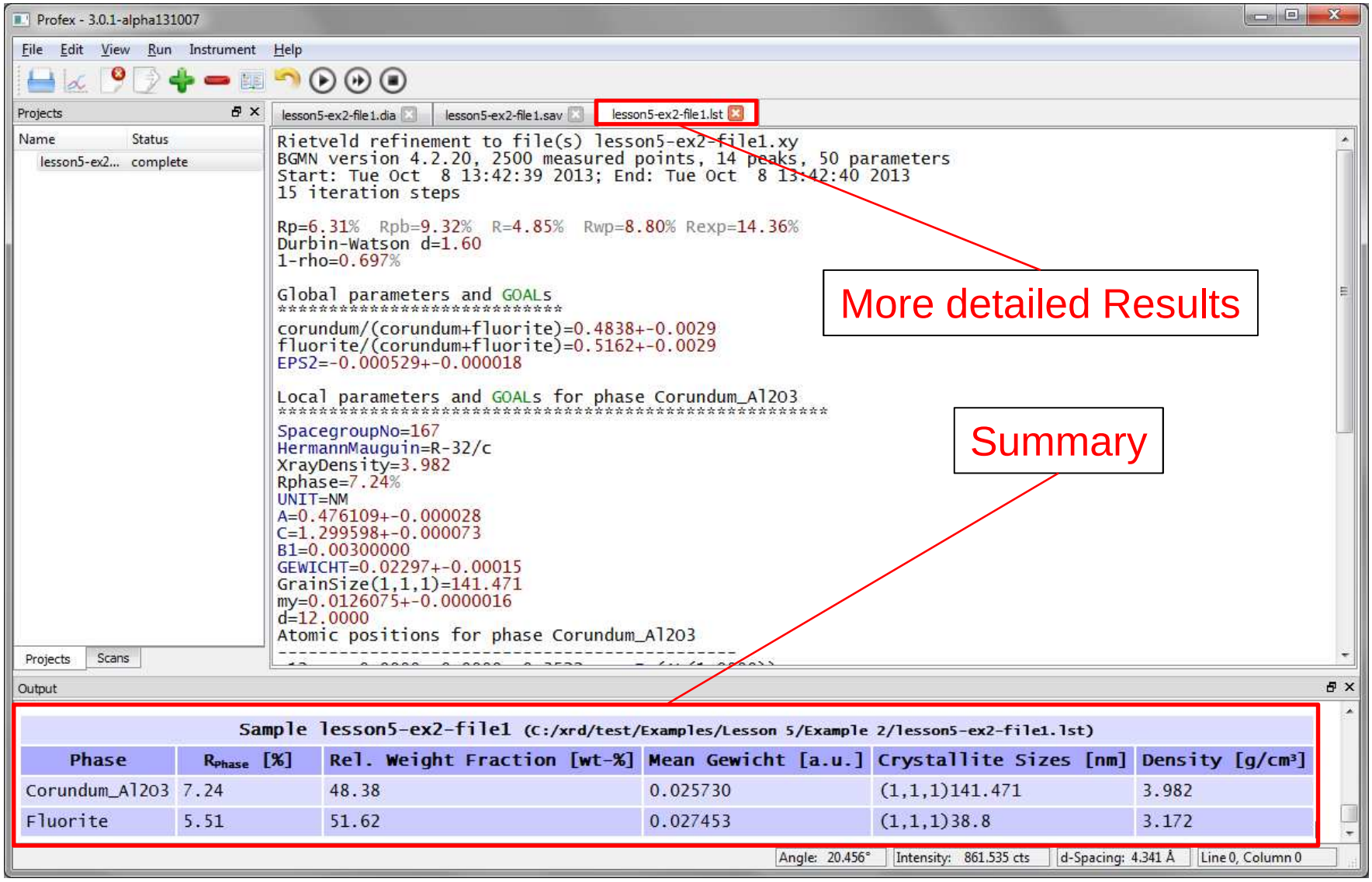
Micro absorption correction according to Brindley
my, my[i]

X ray density
density, density[i]

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

NTHREADS, VERZERR, TubeTails, R, FocusH, FocusW, HSlitR, HSlitW, RoundSlitR, RoundSlitD PColl, PCollA, VSlitR, VSlitH, SamplD, SamplW, SamplH, DeltaOmega, SCollA, SSlitW, SSlitR, SColl, SCollA, DetW, DetH, DetArrayW, MonR, MonH, EPSG, zweiTheta[i], GSUM, TSlitR, TSlitH, FocusS, FocusA, GEOMETRY, WMIN, WMAX, WSTEP, GEQ, D, T, STANDARDPAR, VAL,

Results Files (*.lst)



Results Files (*.lst)

```
Rietveld refinement to file(s) lesson5-ex2-file1.xy
BGMN version 4.2.20, 2500 measured points, 14 peaks, 50 parameters
Start: Tue Oct  8 13:42:39 2013; End: Tue Oct  8 13:42:40 2013
15 iteration steps

Rp=6.31%  Rpb=9.32%  R=4.85%  Rwp=8.80% Rexp=14.36%
Durbin-Watson d=1.60
1-rho=0.697%

Global parameters and GOALs
***************************
corundum/(corundum+fluorite)=0.4838+-0.0029
fluorite/(corundum+fluorite)=0.5162+-0.0029
EPS2=-0.000529+-0.000018

...
```

General information

Statistical Information (Goodness of fit)

Phase quantities (rel. Wt-%)

Sample displacement (EPS2)

Displacement [mm] = R * EPS2 / 2 = -0.0458 mm

R = goniometer radius [mm] = 173 mm

Results Files (*.lst)

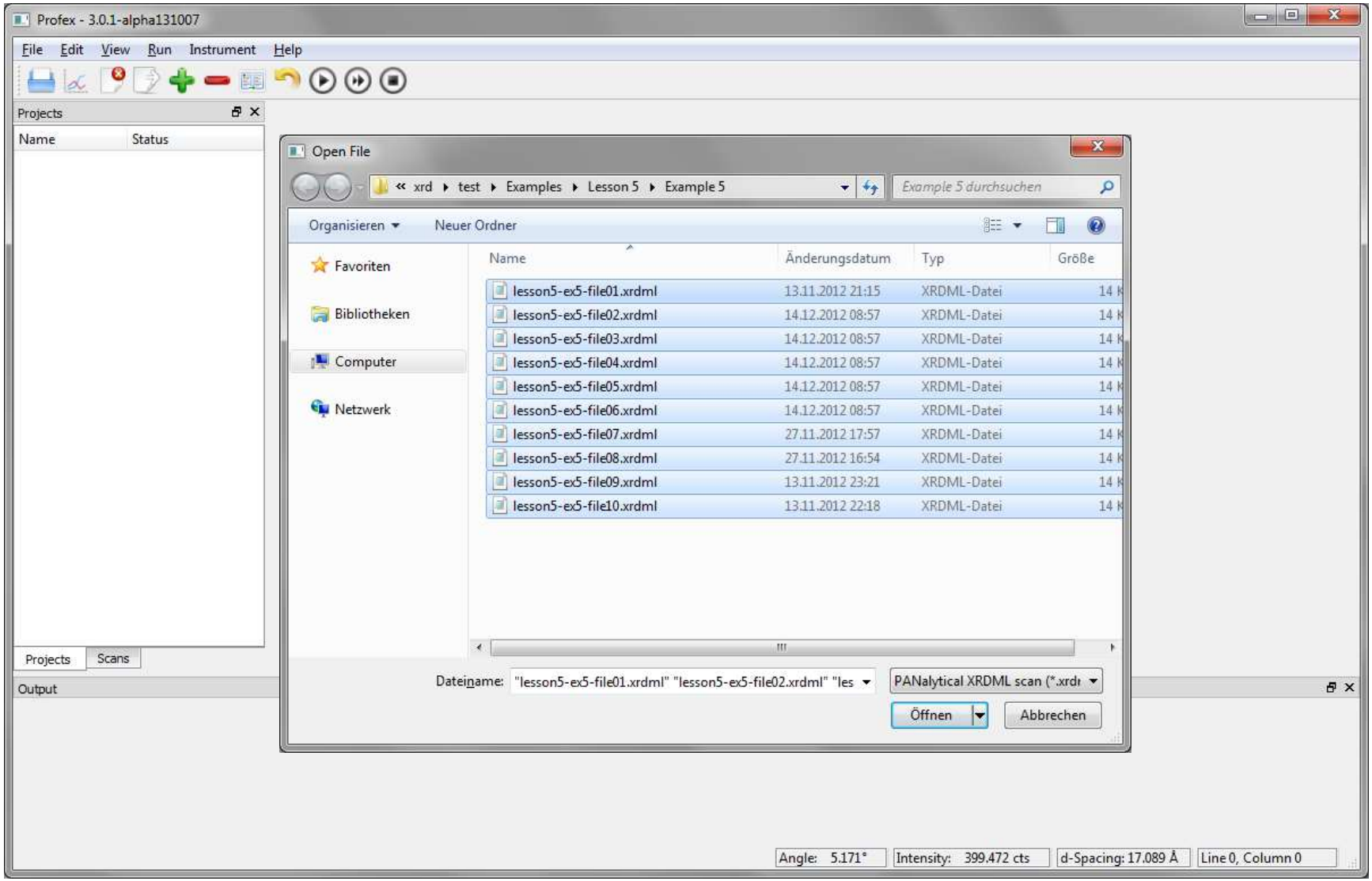
…
Local parameters and GOALs for phase Corundum_Al2O3
****
SpacegroupNo=167
HermannMauguin=R-32/c
XrayDensity=3.981
Rphase=5.76%
UNIT=NM
A=0.47615+-0.00032
C=1.29972+-0.00090
k1=0
k2=0.000000073+-0.000000071
B1=0.00344+-0.00014
GrainSize(1,1,1)=123.4+-4.9
my=0.012604+-0.000025
d=12.0000
GEWICHT=SPHAR4, MeanValue(GEWICHT)=0.0241631
Atomic positions for phase Corundum_Al2O3
---
12 0.0000 0.0000 0.3522 E=(AL(1.0000))
18 0.3062 0.0000 0.2500 E=(O-2(1.0000))
…

Refined Parameters & Goals

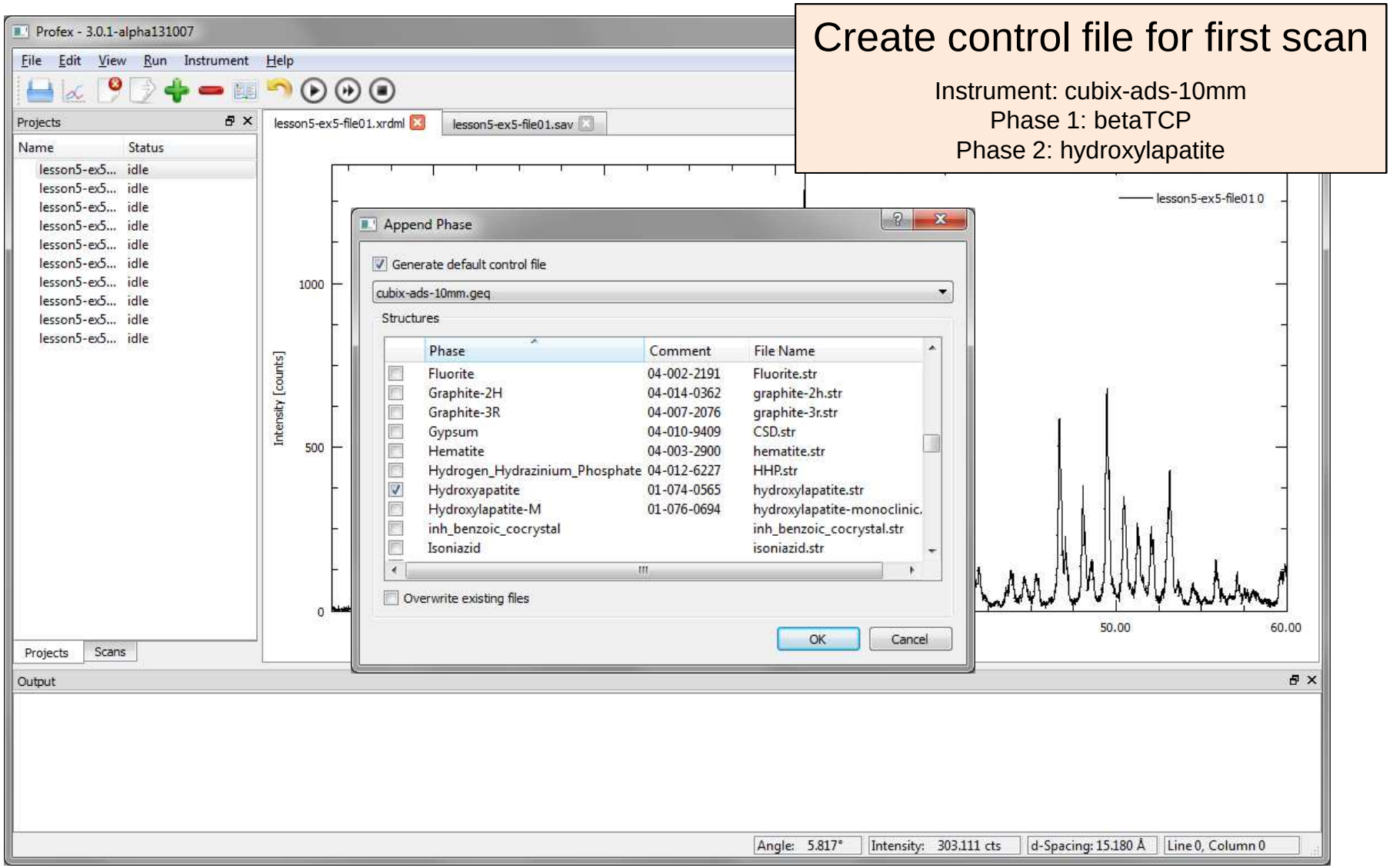
That's why the upper limit of 0.003 was too restrictive!

…
Local parameters and GOALs for phase Fluorite
****
SpacegroupNo=225
HermannMauguin=F4/m-32/m
XrayDensity=3.171
Rphase=5.44%
UNIT=NM
A=0.54682+-0.00037
k1=0
k2=0.0000223+-0.0000014
B1=0.01090+-0.00035
GrainSize(1,1,1)=38.9+-1.2
my=0.030116+-0.000061
d=10.0000
GEWICHT=SPHAR6, MeanValue(GEWICHT)=0.0220520
Atomic positions for phase Fluorite
---
4 0.0000 0.0000 0.0000 E=(CA+2(1.0000))
8 0.2500 0.2500 0.2500 E=(F-1(1.0000))

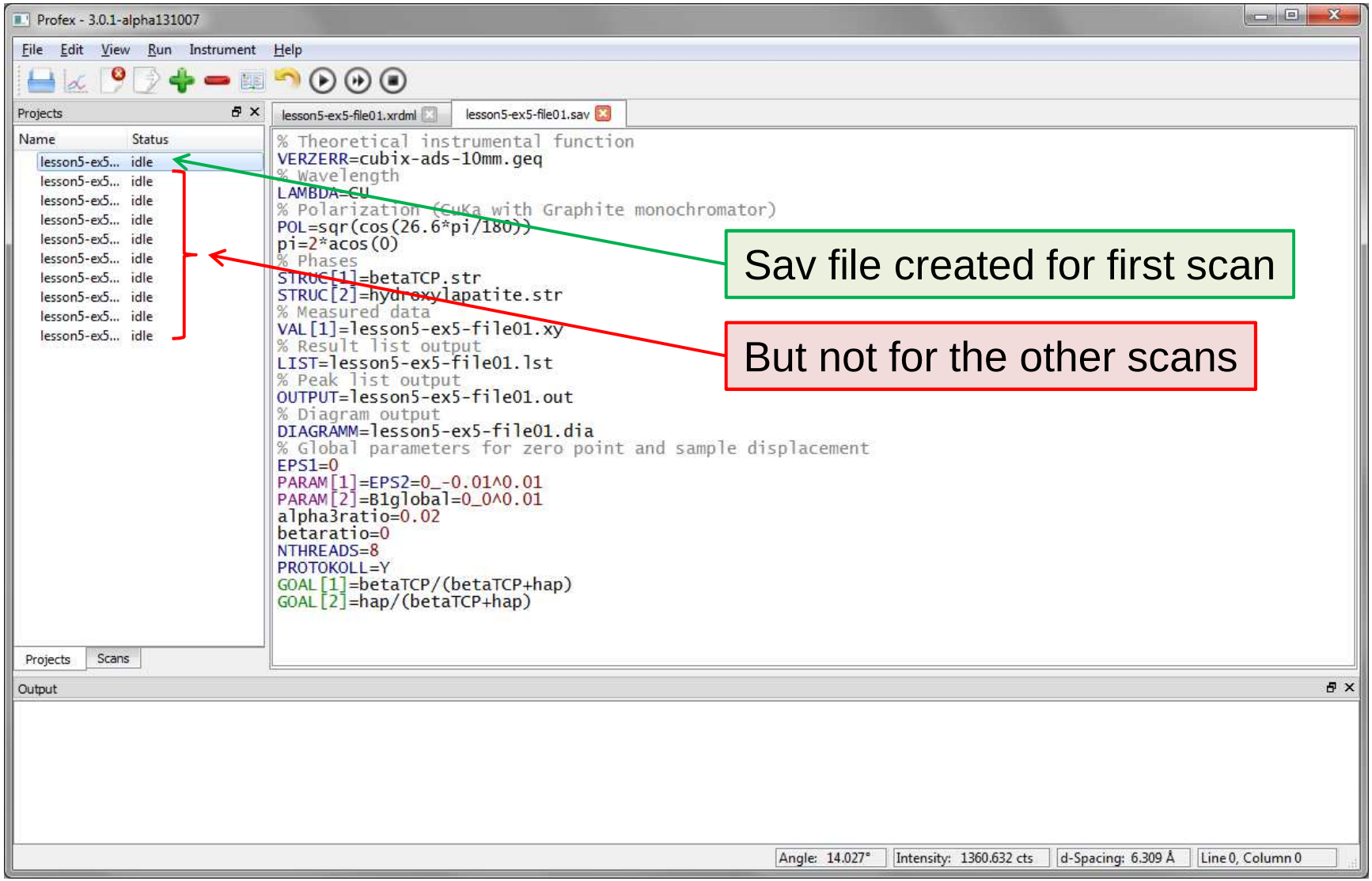
Lesson 5: Example 5 - Batch Refinement



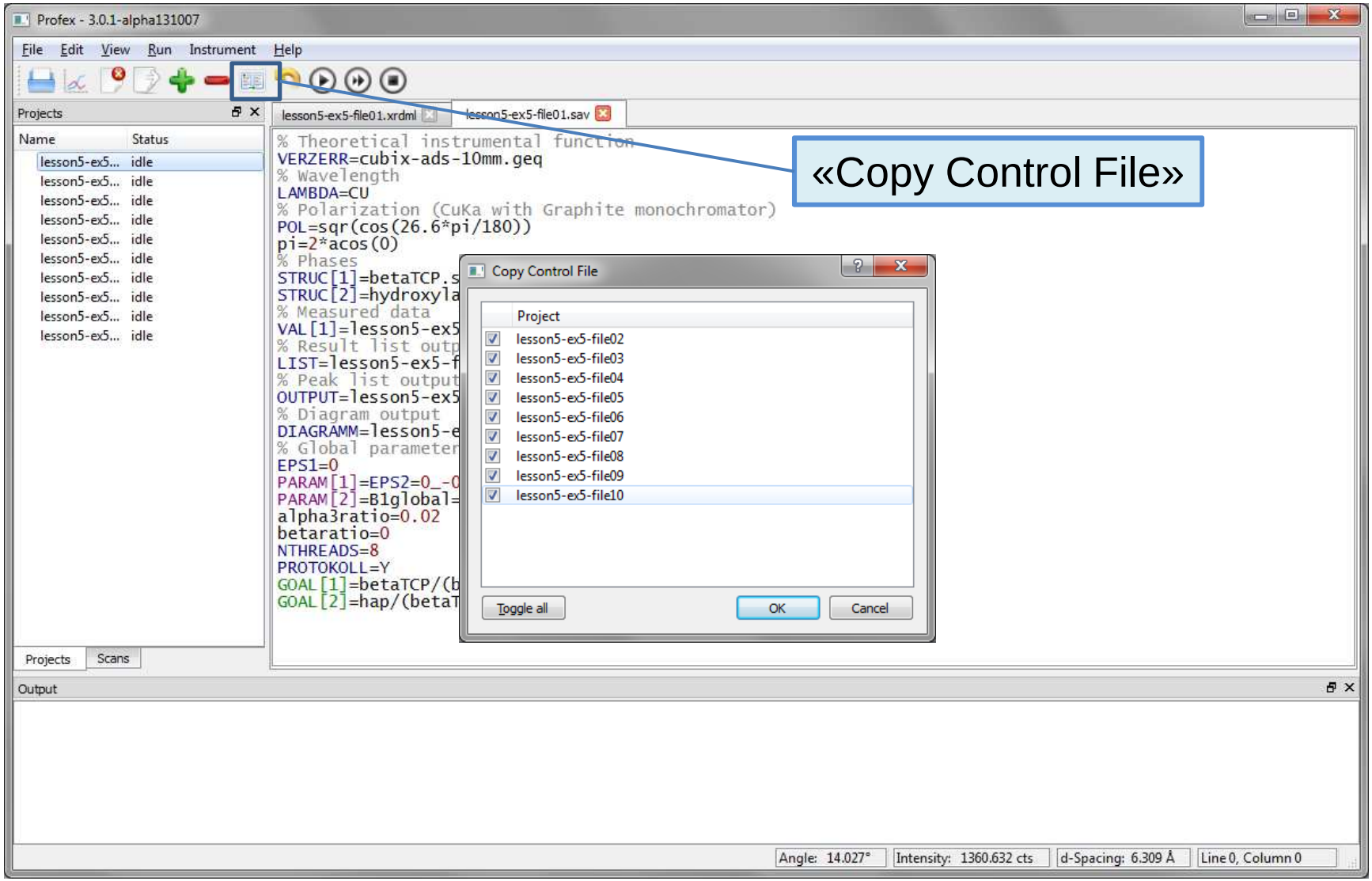
Lesson 5: Example 5 - Batch Refinement



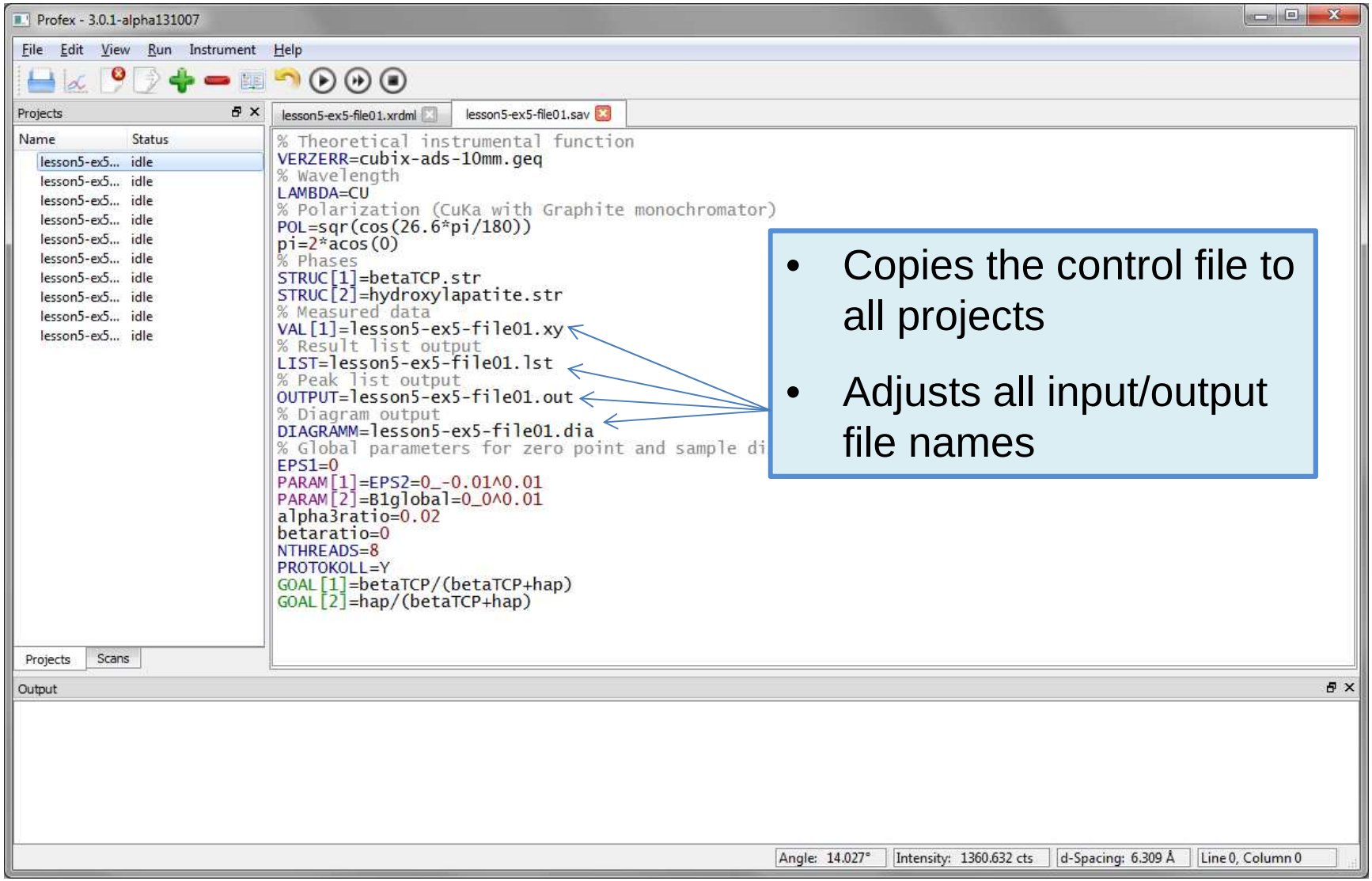
Lesson 5: Example 5 - Batch Refinement



Lesson 5: Example 5 - Batch Refinement

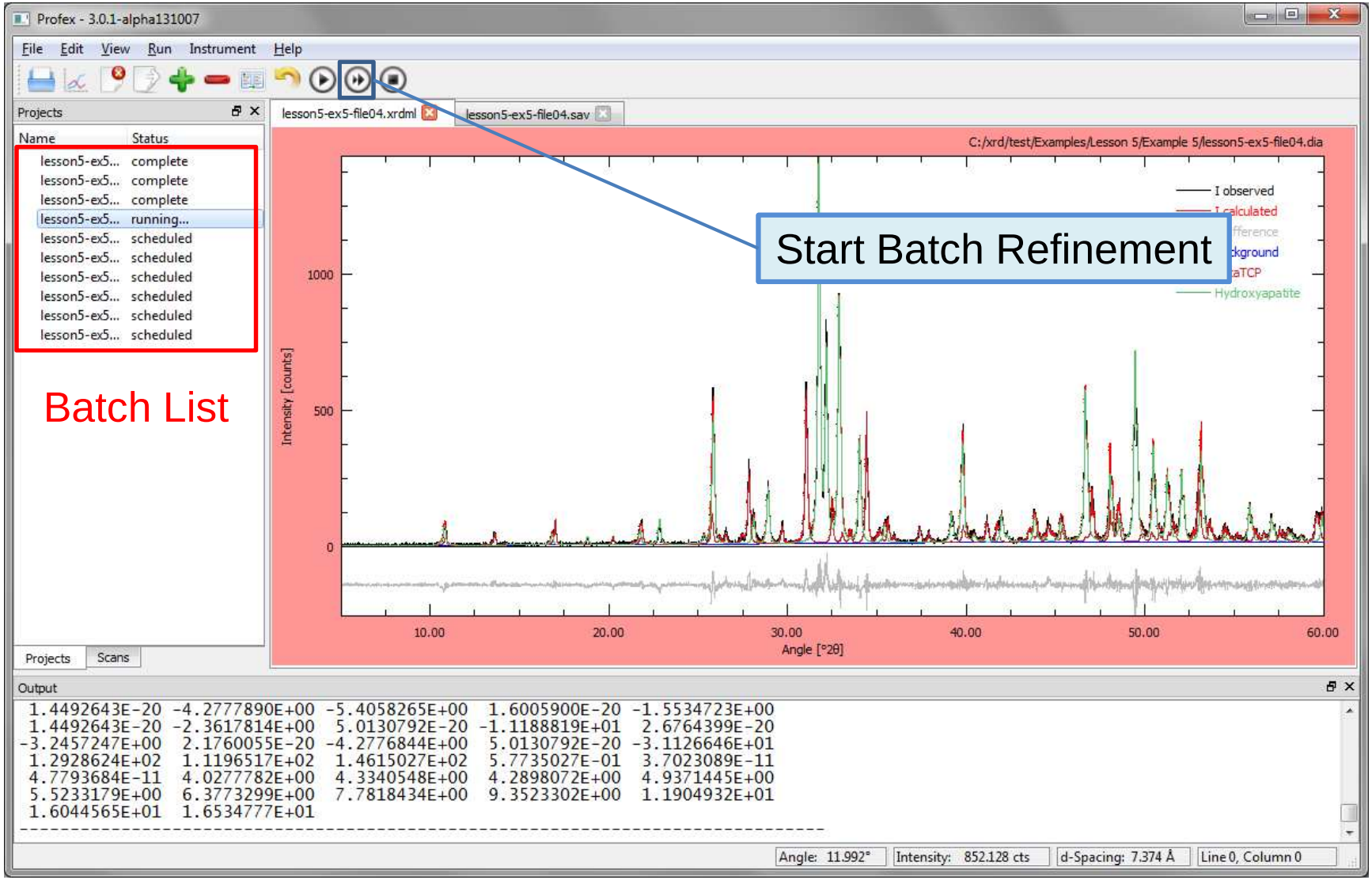


Lesson 5: Example 5 - Batch Refinement

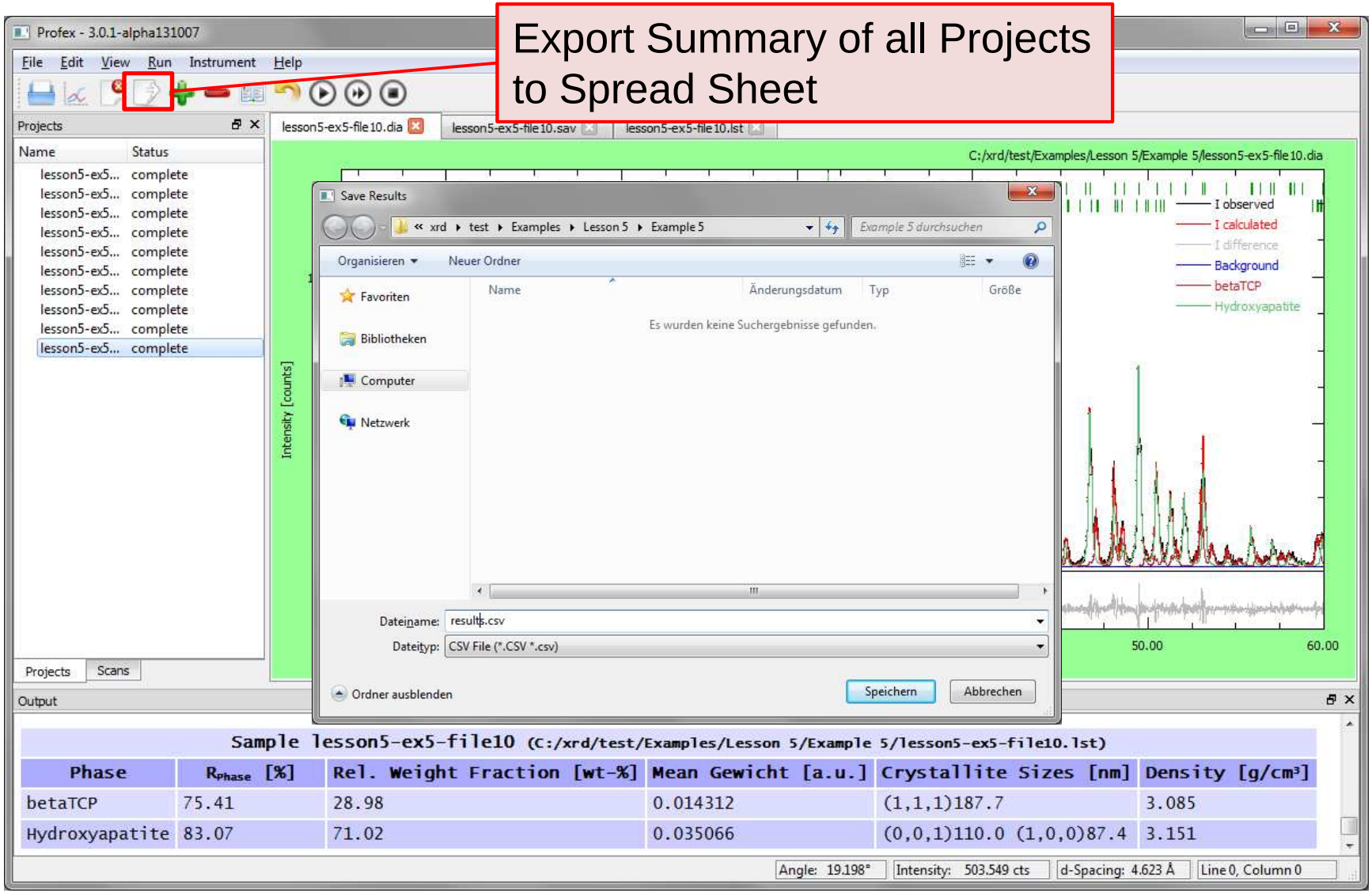


- Copies the control file to all projects
- Adjusts all input/output file names

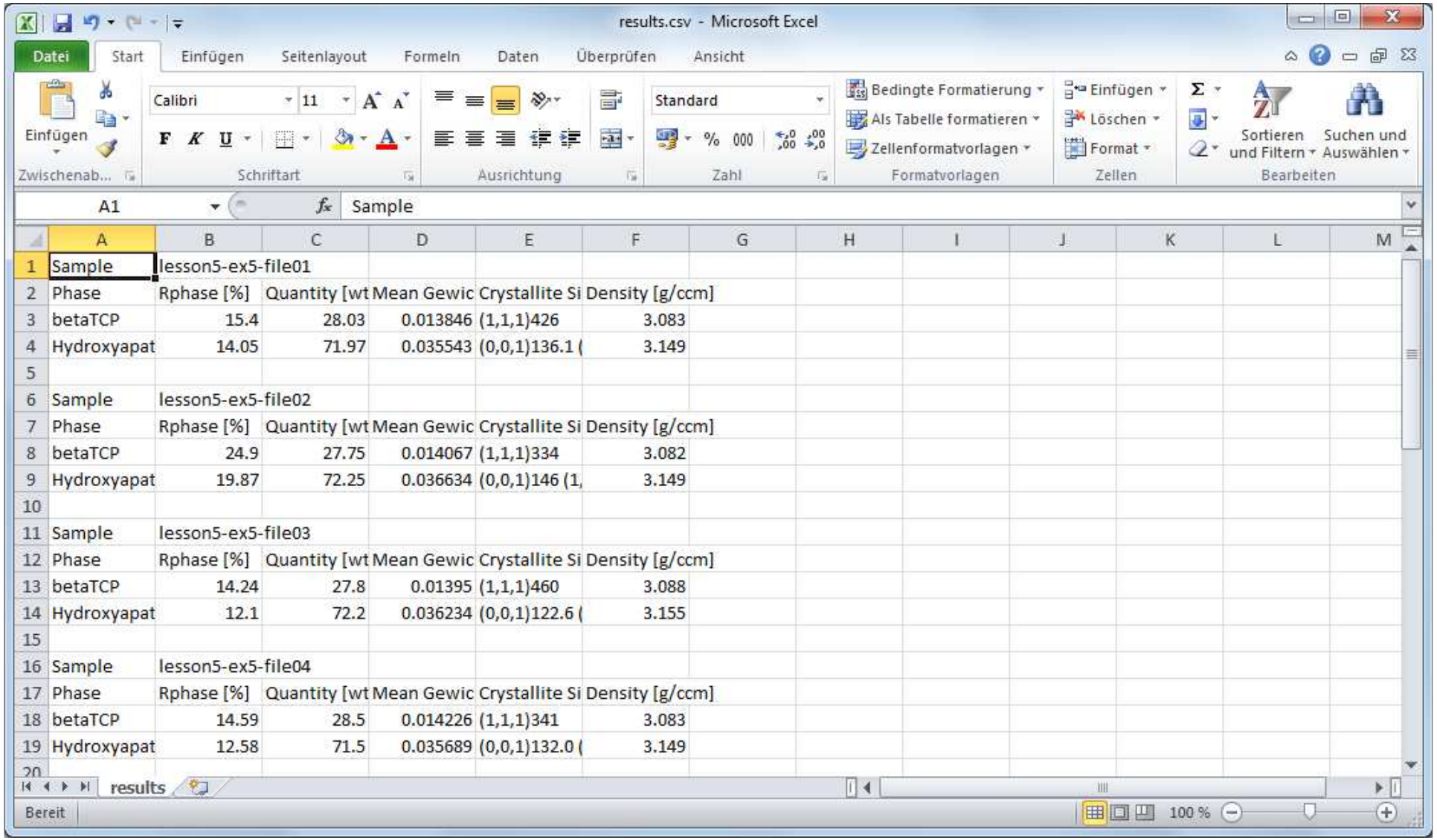
Lesson 5: Example 5 - Batch Refinement



Lesson 5: Example 5 - Batch Refinement



Lesson 5: Example 5 - Batch Refinement



	A	B	C	D	E	F
1	Sample	lesson5-ex5-file01				
2	Phase	Rphase [%]	Quantity [wt	Mean Gewic	Crystallite Si	Density [g/ccm]
3	betaTCP	15.4	28.03	0.013846	(1,1,1)426	3.083
4	Hydroxyapat	14.05	71.97	0.035543	(0,0,1)136.1 (	3.149
5						
6	Sample	lesson5-ex5-file02				
7	Phase	Rphase [%]	Quantity [wt	Mean Gewic	Crystallite Si	Density [g/ccm]
8	betaTCP	24.9	27.75	0.014067	(1,1,1)334	3.082
9	Hydroxyapat	19.87	72.25	0.036634	(0,0,1)146 (1,	3.149
10						
11	Sample	lesson5-ex5-file03				
12	Phase	Rphase [%]	Quantity [wt	Mean Gewic	Crystallite Si	Density [g/ccm]
13	betaTCP	14.24	27.8	0.01395	(1,1,1)460	3.088
14	Hydroxyapat	12.1	72.2	0.036234	(0,0,1)122.6 (	3.155
15						
16	Sample	lesson5-ex5-file04				
17	Phase	Rphase [%]	Quantity [wt	Mean Gewic	Crystallite Si	Density [g/ccm]
18	betaTCP	14.59	28.5	0.014226	(1,1,1)341	3.083
19	Hydroxyapat	12.58	71.5	0.035689	(0,0,1)132.0 (	3.149
20						