RMS Testing • Research • Consulting

Workshop „Rietveld Refinement with Profex“

Lesson 6: Structure and Device Files

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Forschungszentrum Jülich, Germany

Structure Files

GOALs = „Results“ in BGMN terminology

```
PHASE=Calcite // 04-008-0788
MineralName=Calcite
Formula=Ca_(CO3)
SpacegroupNo=167 HermannMauguin=R-32/c
PARAM=A=0.4991_0.4941^0.5041 PARAM=C=1.7062_1.6891^1.7233
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR4
GOAL=GrainSize(1,1,1)
GOAL:Calcite=GEWICHT
E=CA Wyckoff=b x=0.0000 y=0.0000 z=0.0000 TDS=0.00796938
E=C  Wyckoff=a x=0.0000 y=0.0000 z=0.2500 TDS=0.00757986
E=O  Wyckoff=e x=0.2573 y=0.0000 z=0.2500 TDS=0.01400168
```

Direct GOALs **GOAL=<value>** writes the result and its error to the list file (*.lst)

Indirect GOALs **GOAL:<name>=<value>** assigns <value> to the variable <name> and exports <name> to the control file (*.sav)

Structure Files: Direct GOALs

Apatite-OH.str

```
PHASE=Hydroxyapatite // 01-074-0565
MineralName=Hydroxylapatite
Formula=Ca5_(PO4)3_(OH)
SpacegroupNo=176 HermannMauguin=P6_3/m
PARAM=A=0.9424_0.9330^0.9518 PARAM=C=0.6879_0.6810^0.6948
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.05 GEWICHT=SPHAR4
GOAL=GrainSize(0,0,1)
GOAL=GrainSize(1,0,0)
GOAL:Hydroxyapatite=GEWICHT
E=CA Wyckoff=f x=0.3333 y=0.6667 z=0.0015 TDS
E=CA Wyckoff=h x=0.2468 y=0.9934 z=0.2500 TDS
E=P Wyckoff=h x=0.3987 y=0.3685 z=0.2500 TDS=
E=O Wyckoff=h x=0.3284 y=0.4848 z=0.2500 TDS=
E=O Wyckoff=h x=0.5873 y=0.4651 z=0.2500 TDS=
E=O Wyckoff=i x=0.3437 y=0.2579 z=0.0702 TDS=
E=O(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.1
E=H(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.0
```

240222-01.lst

```
Local parameters and GOALs for phase Hydroxyapatite
*****************************************************
SpacegroupNo=176
HermannMauguin=P6_3/m
XrayDensity=3.120
Rphase=5.46%
UNIT=NM
A=0.9425500+-0.0000033
C=0.6883876+-0.0000030
GrainSize(0,0,1)=387+-15
GrainSize(1,0,0)=379+-10
GEWICHT=SPHAR4, MeanValue(GEWICHT)=0.161184
B1=ANISOLIN, MeanValue(B1)=0.00111181, sqrt3(det(B1))=0.00111171
Atomic positions for phase Hydroxyapatite
-----------------------------------------
4  0.3333 0.6667 0.0015 E=(CA(1.000))
6  0.2468 0.9934 0.2500 E=(CA(1.0000))
6  0.3987 0.3685 0.2500 E=(P(1.0000))
6  0.3284 0.4848 0.2500 E=(O(1.0000))
6  0.5873 0.4651 0.2500 E=(O(1.0000))
12 0.3437 0.2579 0.0702 E=(O(1.0000))
4  0.0000 0.0000 0.1950 E=(O(0.5000))
4  0.0000 0.0000 0.0608 E=(H(0.5000))
```

Other examples:

```
// volume of hexagonal unit cell
GOAL=sqrt(3)*A*A*C/2

// mass absorption coefficient
GOAL=10000*my/density
```

Structure Files: Indirect GOALs

Apatite-OH.str

```
PHASE=Hydroxyapatite // 01-074-0565
MineralName=Hydroxylapatite
Formula=Ca5_(PO4)3_(OH)
SpacegroupNo=176 HermannMauguin=P6_3/m
PARAM=A=0.9424_0.9330^0.9518 PARAM=C=0.6879_0.6810^0.6948
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.05 GEWICHT=SPHAR4
GOAL=GrainSize(0,0,1)
GOAL=GrainSize(1,0,0)
GOAL:Hydroxyapatite=GEWICHT
E=CA Wyckoff=f x=0.3333 y=0.6667 z=0.0015 TDS=0.00664290
E=CA Wyckoff=h x=0.2468 y=0.9934 z=0.2500 TDS=0.00567436
E=P Wyckoff=h x=0.3987 y=0.3685 z=0.2500 TDS=0.00477426
E=O Wyckoff=h x=0.3284 y=0.4848 z=0.2500 TDS=0.00953535
E=O Wyckoff=h x=0.5873 y=0.4651 z=0.2500 TDS=0.01014069
E=O Wyckoff=i x=0.3437 y=0.2579 z=0.0702 TDS=
E=O(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.1
E=H(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.0
```

240222-01.sav

```
sum=Hydroxyapatite+Calcite
QHydroxyapatite=Hydroxyapatite/sum
QCalcite=Calcite/sum

GOAL[1]=QHydroxyapatite
GOAL[2]=QCalcite
```

Other examples:

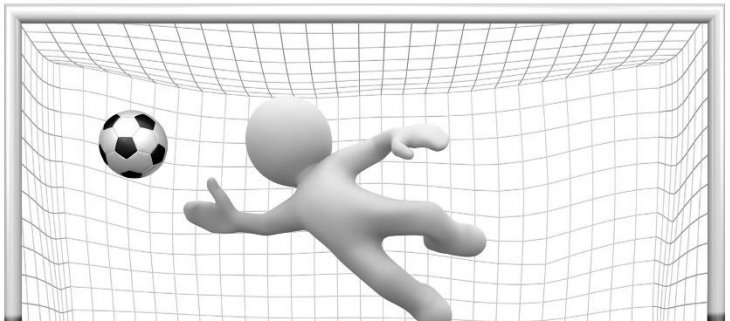
```
// volume of hexagonal unit cell
GOAL:VolApatite=sqrt(3)*A*A*C/2

// mass absorption coefficient
GOAL:MacApatite=10000*my/density
```

„VolApatite“ and „MacApatite“ are new global variables that can be accessed in the *.sav file.

Structure Files: GOALs

Summary of GOAL implementations:

Code	Accessible in STR file	Written to LST file	Accessible in SAV file
`VolApatite=sqrt(3)*A*A*C/2`	Yes	No	No
`GOAL=sqrt(3)*A*A*C/2`	No	Yes	No
`GOAL:VolApatite=sqrt(3)*A*A*C/2`	Yes	No	Yes
`GOAL:VolApatite=sqrt(3)*A*A*C/2` `GOAL=VolApatite`	Yes	Yes	Yes



Structure Files: Site Occupancy Factors and Substitutions

Apatite-OH.str

```
PHASE=Hydroxyapatite // 01-074-0565
MineralName=Hydroxylapatite
Formula=Ca5_(PO4)3_(OH)
SpacegroupNo=176 HermannMauguin=P6_3/m
PARAM=A=0.9424_0.9330^0.9518 PARAM=C=0.6879_0.6810^0.6948
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.05 GEWICHT=SPHAR4
GOAL=GrainSize(0,0,1)
GOAL=GrainSize(1,0,0)
GOAL:Hydroxyapatite=GEWICHT
E=CA Wyckoff=f x=0.3333 y=0.6667 z=0.0015 TDS=0.00664290
E=CA Wyckoff=h x=0.2468 y=0.9934 z=0.2500 TDS=0.00567436
E=P Wyckoff=h x=0.3987 y=0.3685 z=0.2500 TDS=0.00477426
E=O Wyckoff=h x=0.3284 y=0.4848 z=0.2500 TDS=0.00953535
E=O Wyckoff=h x=0.5873 y=0.4651 z=0.2500 TDS=0.01014069
E=O Wyckoff=i x=0.3437 y=0.2579 z=0.0702 TDS=0.01499127
E=O(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.1950 TDS=0.00000000
E=H(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.0608 TDS=0.02947459
```

Scheme: **E=<scattering factor>(occupancy)**

Examples:	**E=CA**	is equivalent to	**E=CA(1.0)**	(fully occupied)
	E=SI(0.5)			(partially occupied)
	E=SI+4(0.5)			(ionic scat. fact. partially occupied)
	E=(SI(0.75),AL(0.25))			(substitution)

Structure Files: Site Occupancy Factors and Substitutions

Refining Substitutions:

E=(SI(0.75),AL(0.25))  **E=(SI(p),AL(1-p)) PARAM=p=0.75_0^1**

Calcite.str

```
PHASE=Calcite // 04-008-0788
MineralName=Calcite
Formula=Ca_(CO3)
SpacegroupNo=167 HermannMauguin=R-32/c
PARAM=A=0.4991_0.4941^0.5041 PARAM=C=1.7062_1.6891^1.7233
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR4
GOAL=GrainSize(1,1,1)
GOAL:Calcite=GEWICHT
E=(CA(p),MG(1-p)) PARAM=p=1_0^1 Wyckoff=b x=0.0000 y=0.0000 z=0.0000 TDS=0.00796938
E=C  Wyckoff=a x=0.0000 y=0.0000 z=0.2500 TDS=0.00757986
E=O  Wyckoff=e x=0.2573 y=0.0000 z=0.25
```

240222-01.lst

```
Atomic positions for phase Calcite
-----------------------------------------------
6 0.0000 0.0000 0.0000 E=(CA(0.9441),MG(0.0559))
p=0.9441+-0.0055


6 0.0000 0.0000 0.2500 E=(C(1.0000))
18 0.2573 0.0000 0.2500 E=(O(1.0000))
```

Structure Files: Coupling Parameters

Assumption: Na^{+} substitutes Ca^{2+}

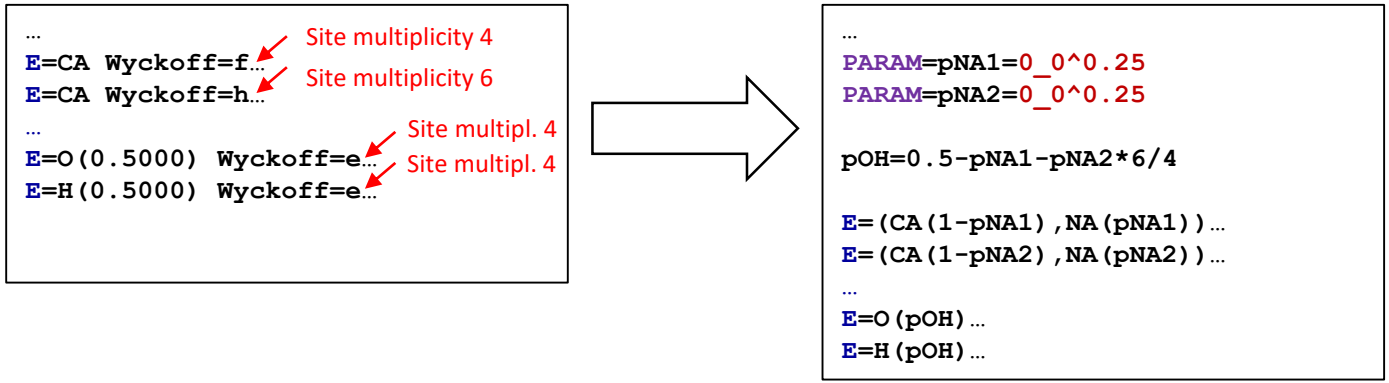
To maintain charge balance, OH^{-} is reduced accordingly

```
PHASE=Hydroxyapatite // 01-074-0565
MineralName=Hydroxylapatite
Formula=Ca5_(PO4)3_(OH)
SpacegroupNo=176 HermannMauguin=P6_3/m
PARAM=A=0.9424_0.9330^0.9518 PARAM=C=0.6879_0.6810^0.6948
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.05 GEWICHT=SPHAR4
GOAL=GrainSize(0,0,1)
GOAL=GrainSize(1,0,0)
GOAL:Hydroxyapatite=GEWICHT
E=(CA(1-p),NA(p)) PARAM=p=0_0^1 Wyckoff=f x=0.3333 y=0.6667 z=0.0015 TDS=0.00664290
E=(CA(1-p),NA(p)) PARAM=p=0_0^1 Wyckoff=h x=0.2468 y=0.9934 z=0.2500 TDS=0.00567436
E=P Wyckoff=h x=0.3987 y=0.3685 z=0.2500 TDS=0.00477426
E=O Wyckoff=h x=0.3284 y=0.4848 z=0.2500 TDS=0.00953535
E=O Wyckoff=h x=0.5873 y=0.4651 z=0.2500 TDS=0.01014069
E=O Wyckoff=i x=0.3437 y=0.2579 z=0.0702 TDS=0.01499127
E=O(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.1950 TDS=0.00000000
E=H(0.5000) Wyckoff=e x=0.0000 y=0.0000 z=0.0608 TDS=0.02947459
```

Structure Files: Coupling Parameters

Assumption: Na^+ substitutes Ca^{2+}

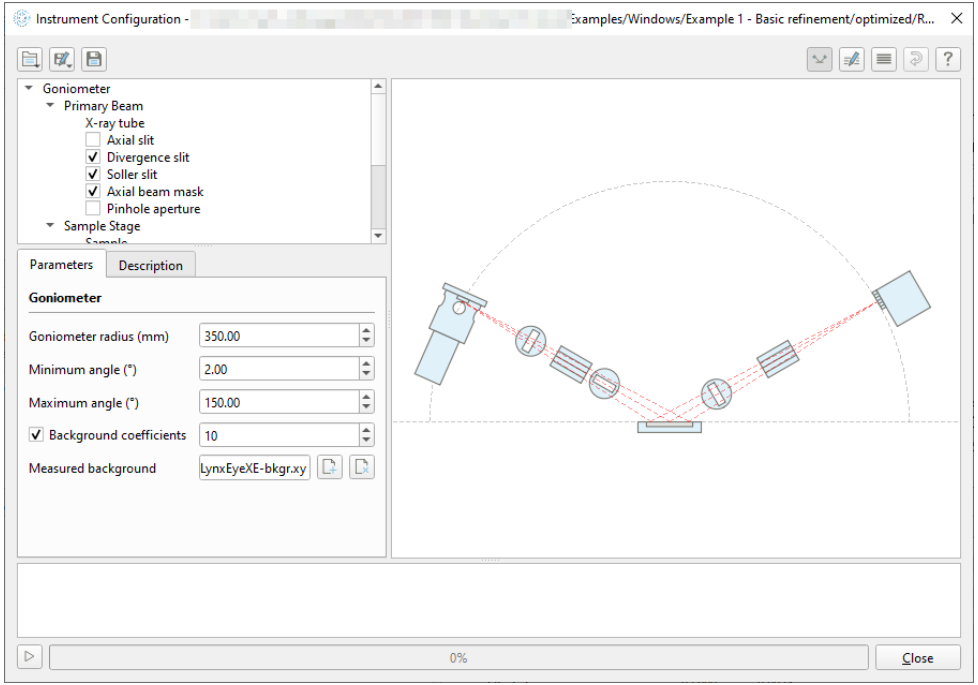
To maintain charge balance, OH^- is reduced accordingly



Parameters used for several atomic sites must be declared **above** the E= lines.

Parameters declared **within** an atomic site (E= line) can only be accessed **within** the site.

Instrument Configuration Files

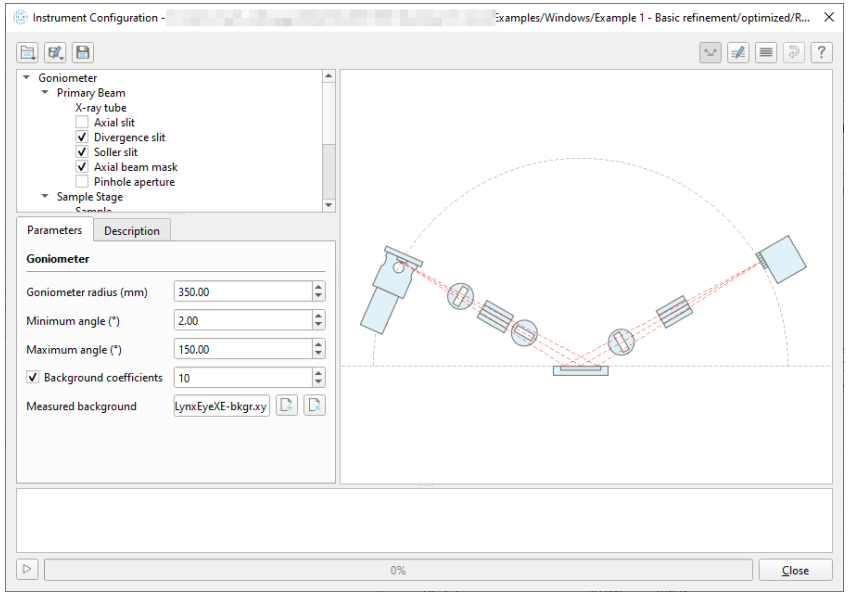


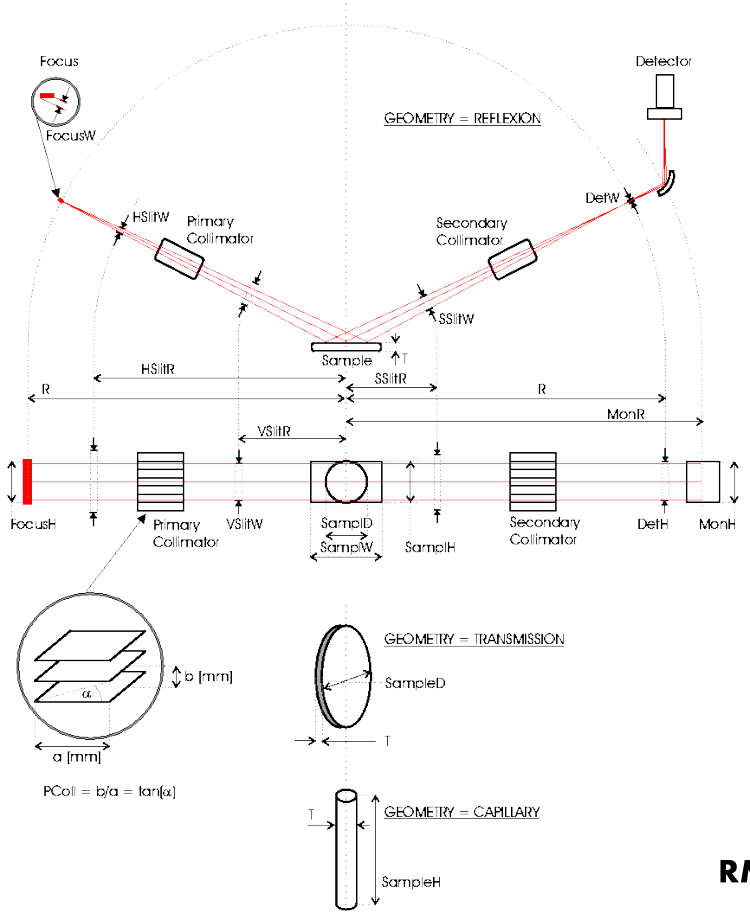
Instrument Configuration Files

- Instrument configuration files must **precisely** describe the hardware setup used to measure a dataset.
- Profex includes 70 configurations
- Often customization is necessary

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

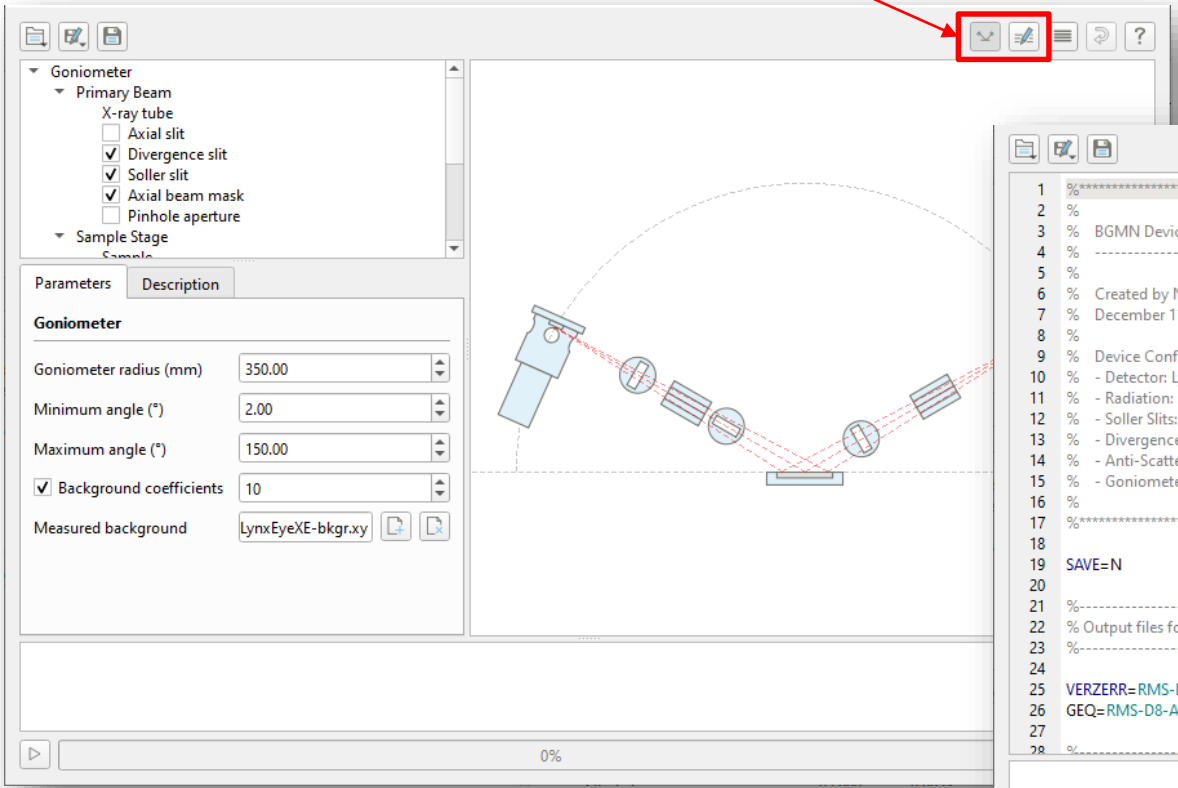
Profex: Instrument → Edit current FPA configuration



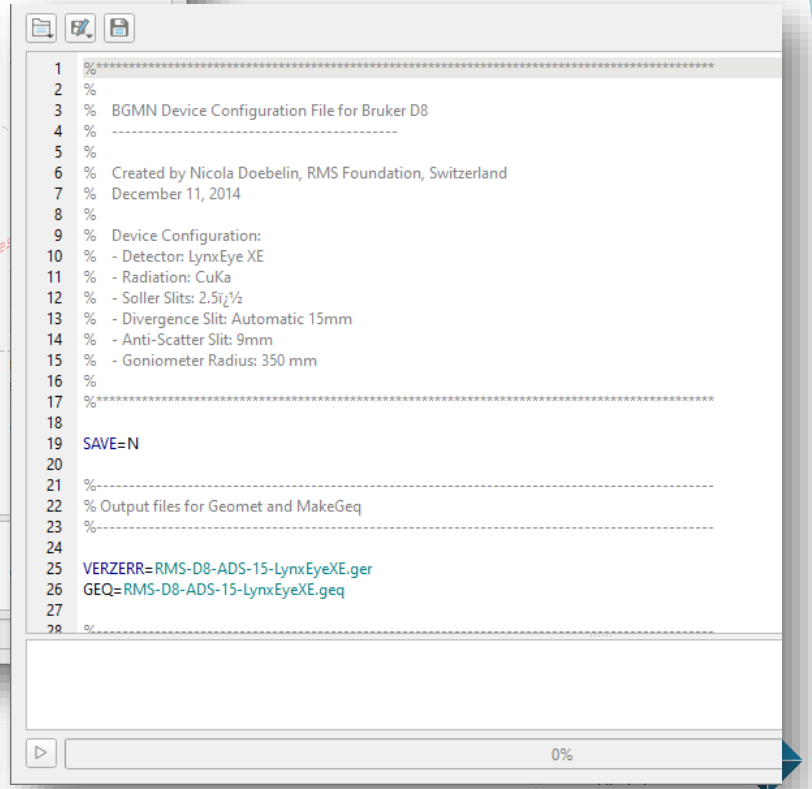


Instrument Configuration Files

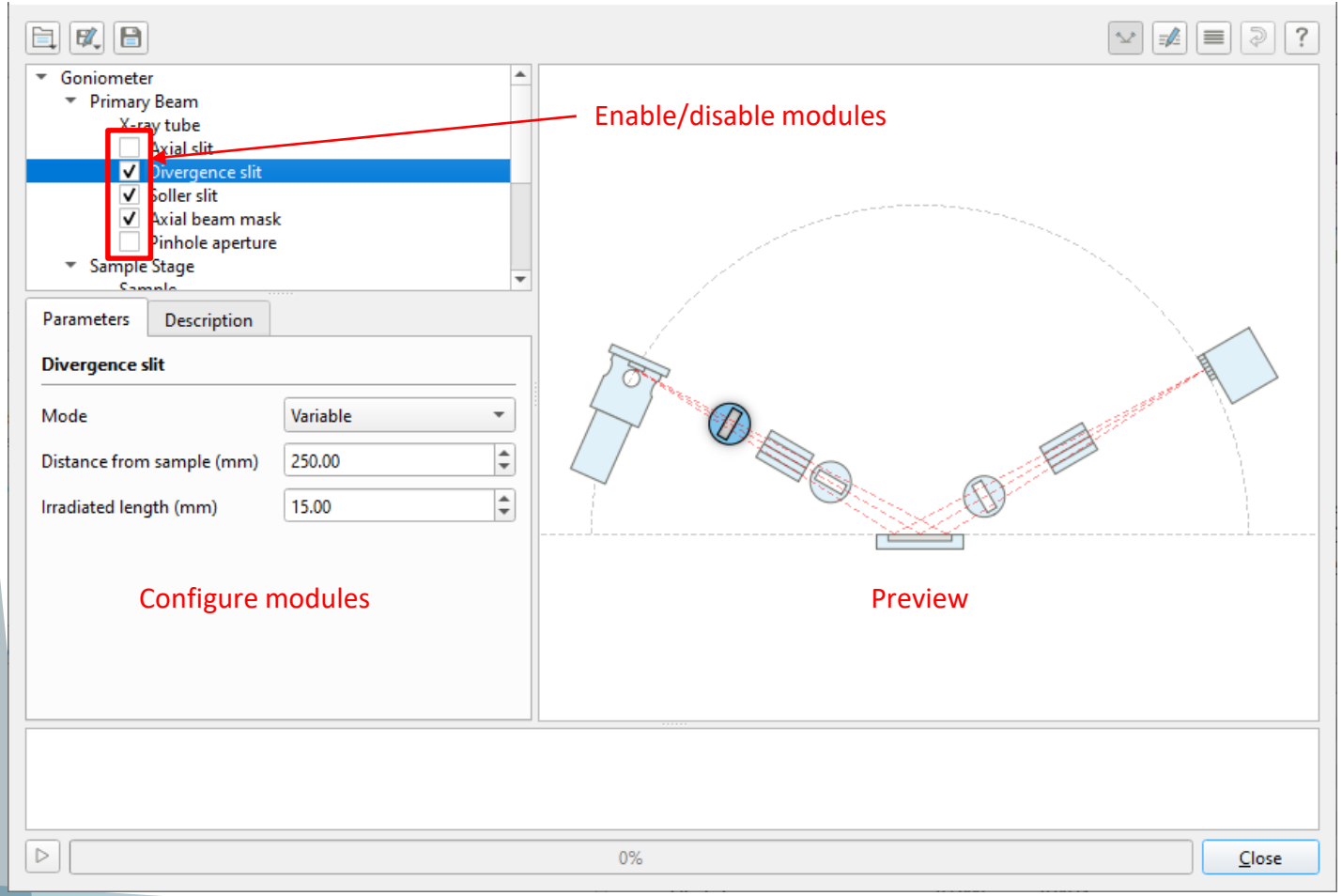
Toggle between text editor and graphical editor



Normally the text editor is not needed

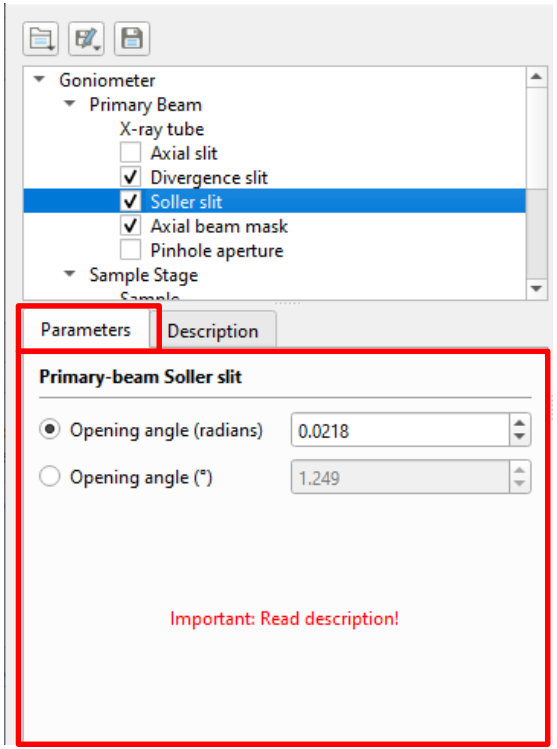


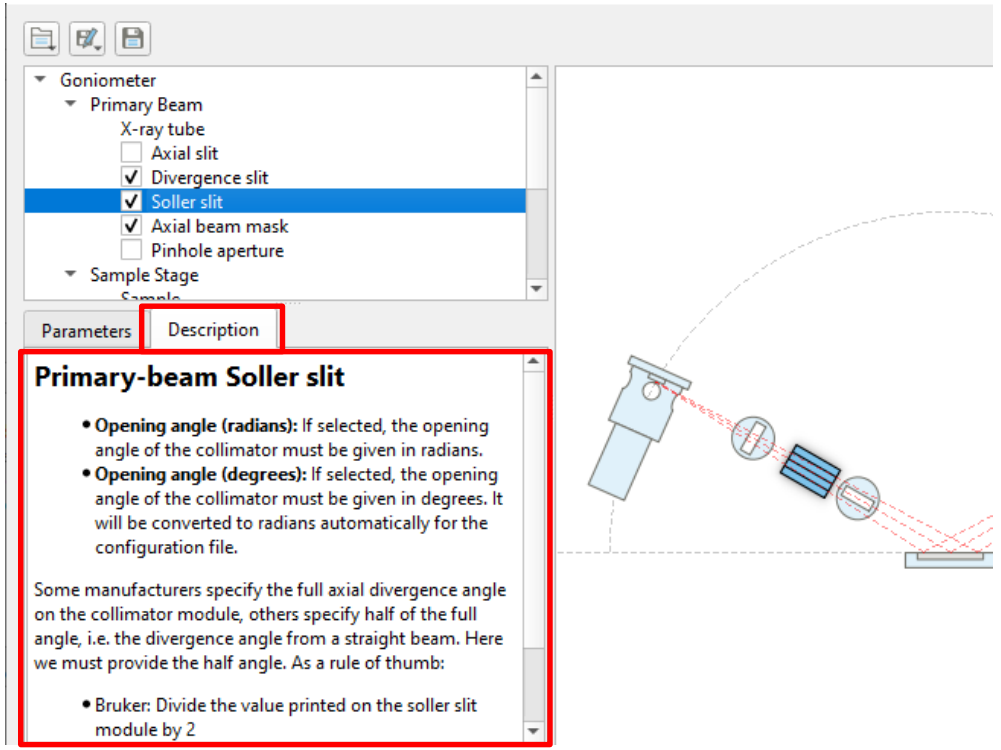
Instrument Configuration Files



Instrument Configuration Files

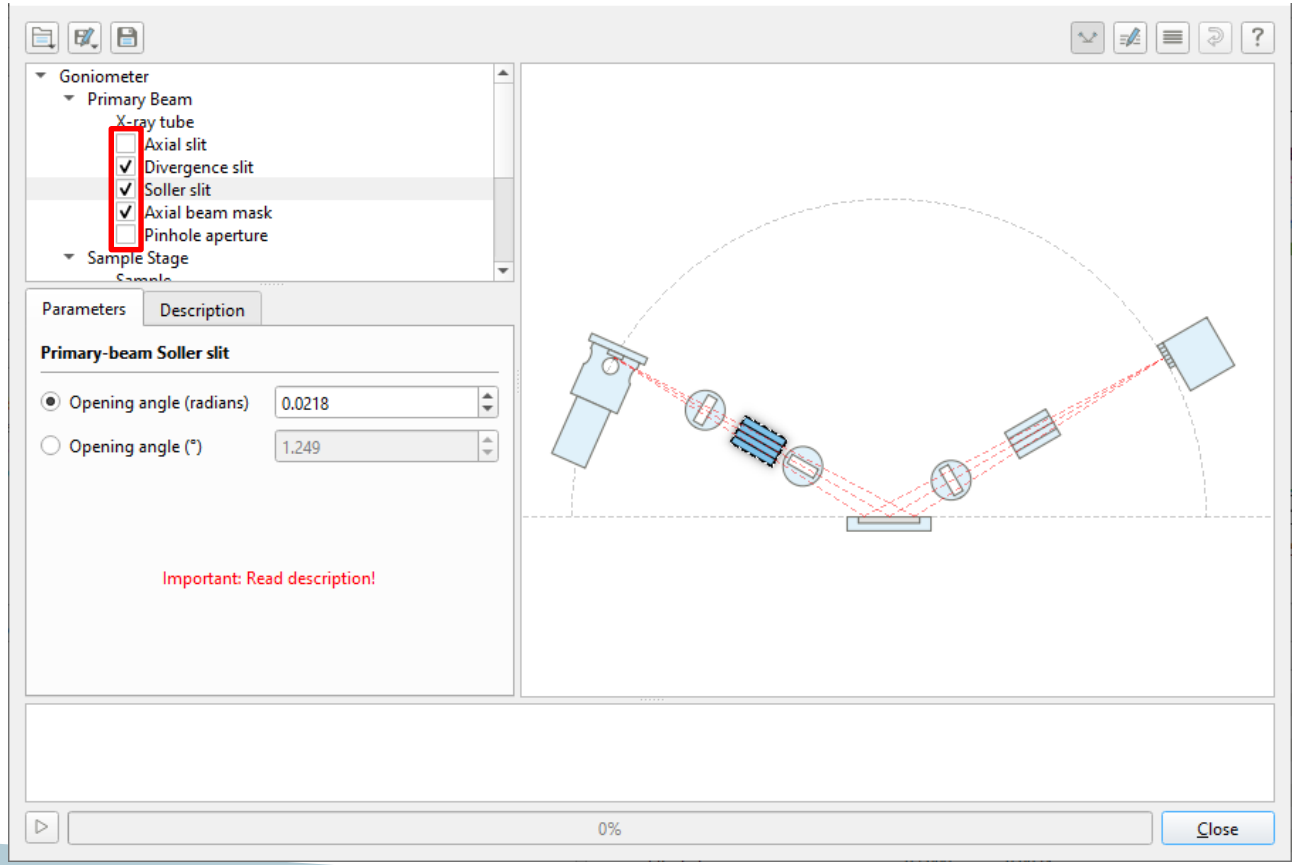
Each configuration page has a „Description“ page with detailed information





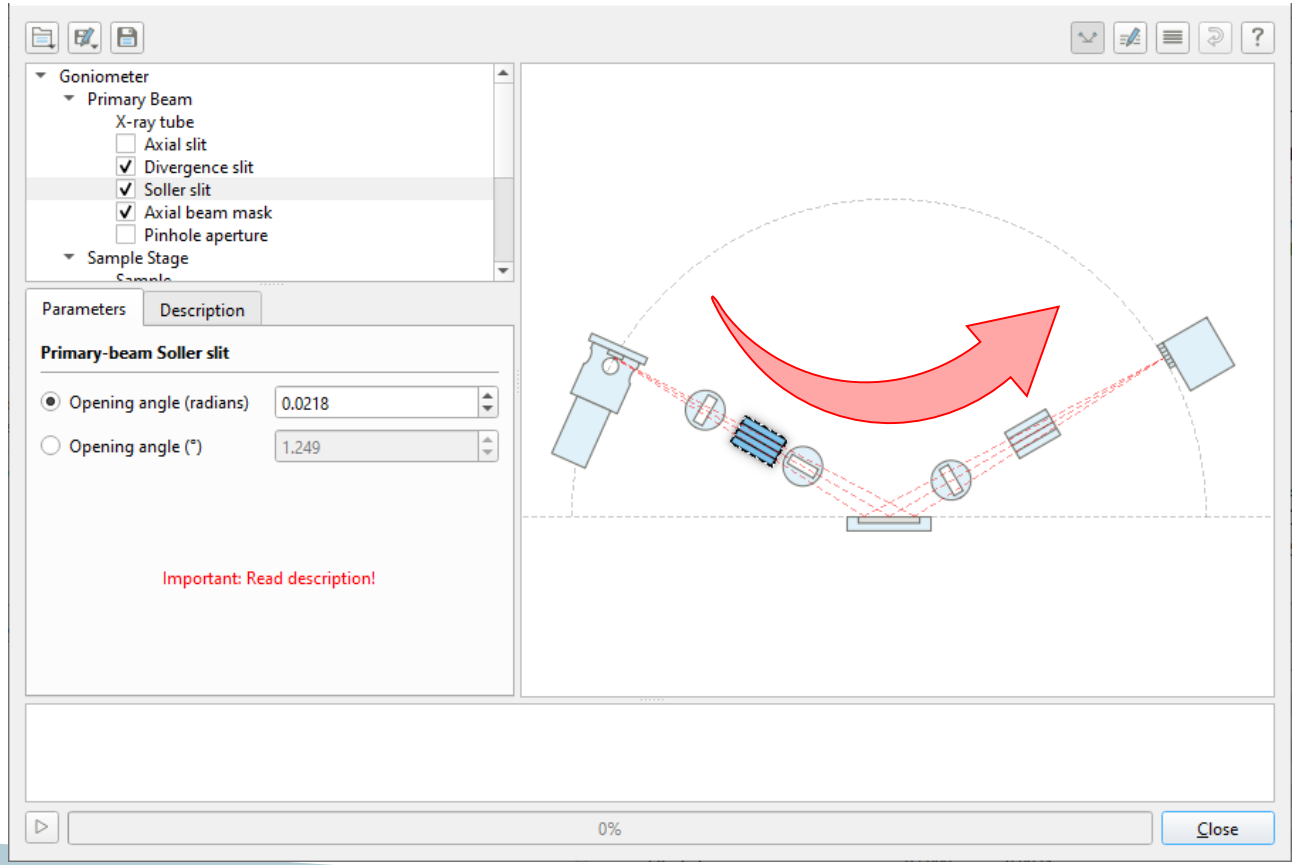
Instrument Configuration Files: Recommended Workflow

1. Enable / disable the modules according to the instrument setup



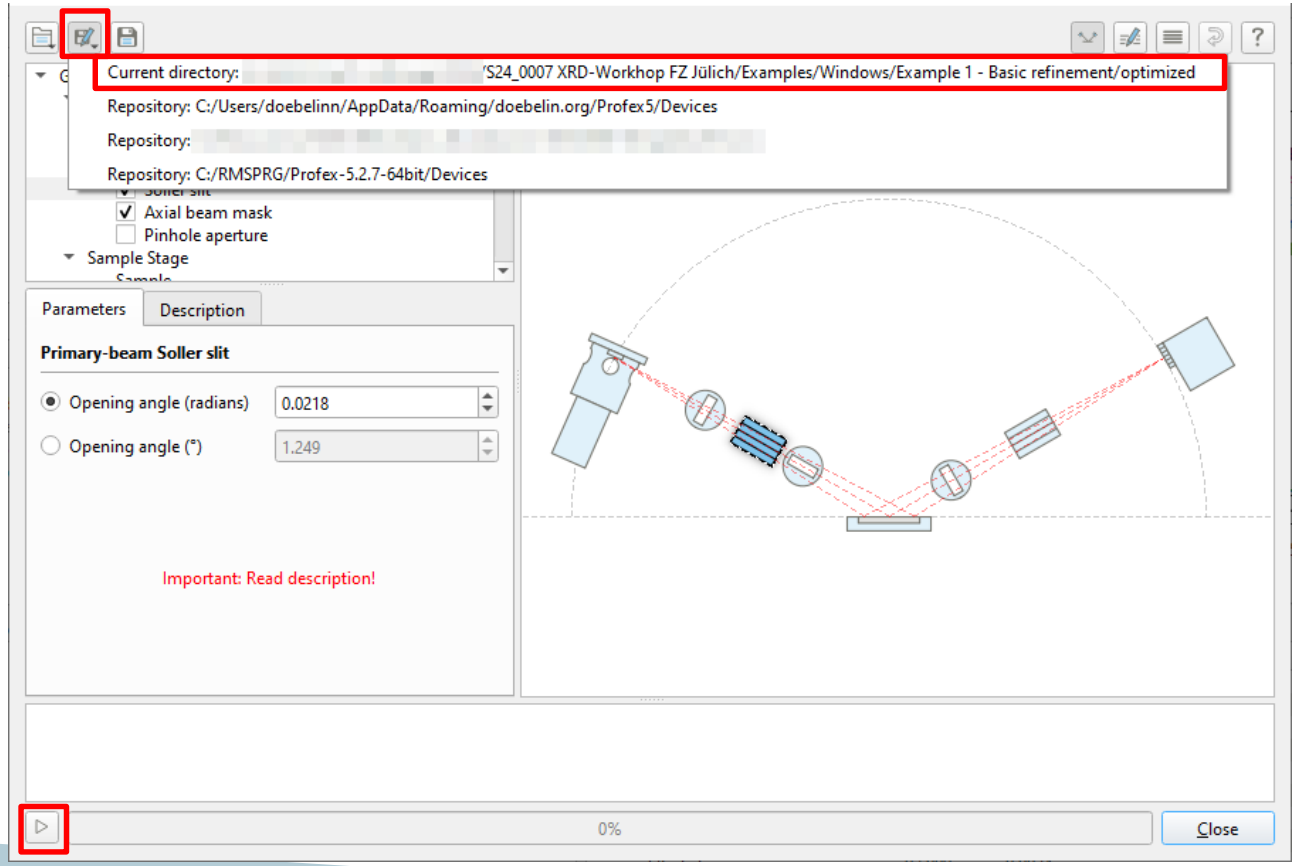
Instrument Configuration Files: Recommended Workflow

2. Go through all modules from Tube to Detector and enter the correct settings



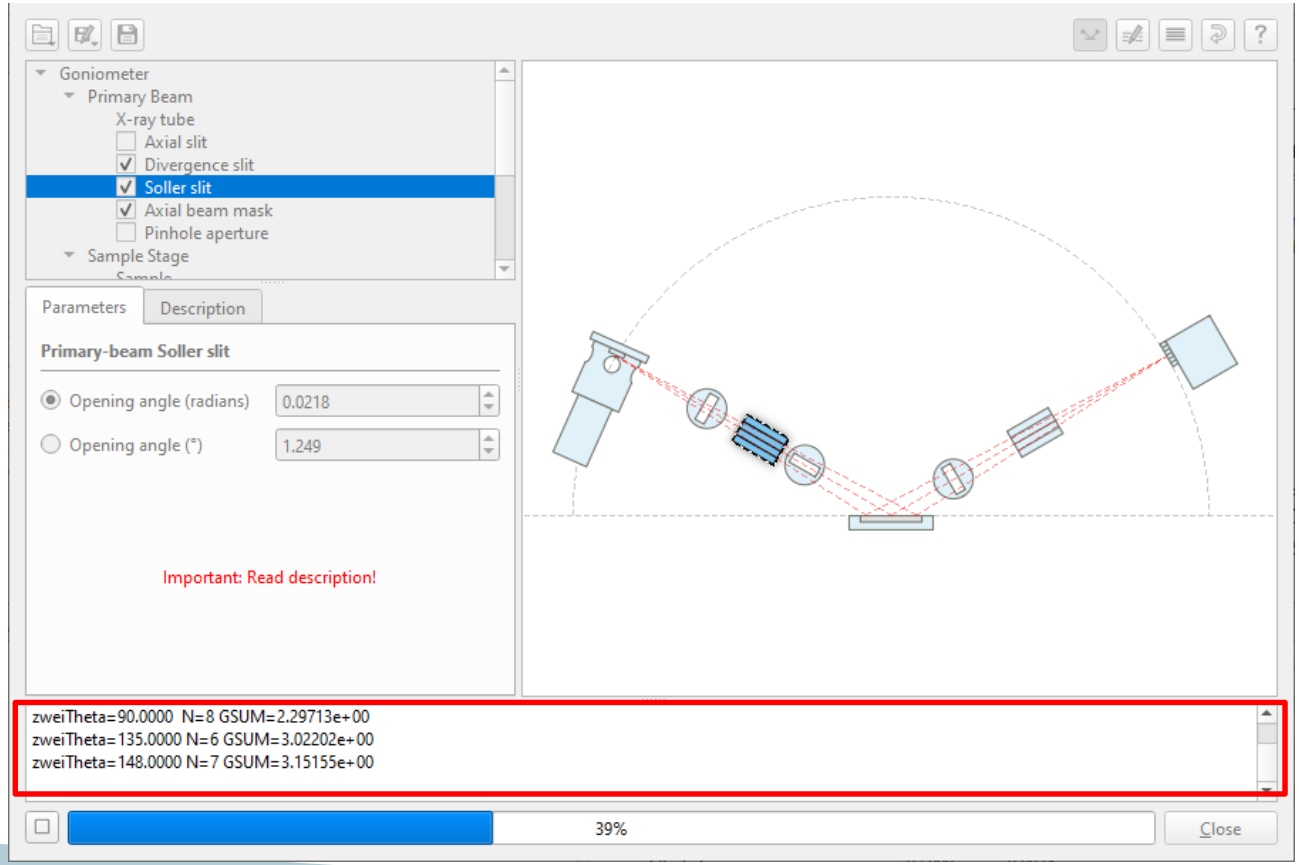
Instrument Configuration Files: Recommended Workflow

3. Save configuration under a new name in current project directory
4. Run profile calculation

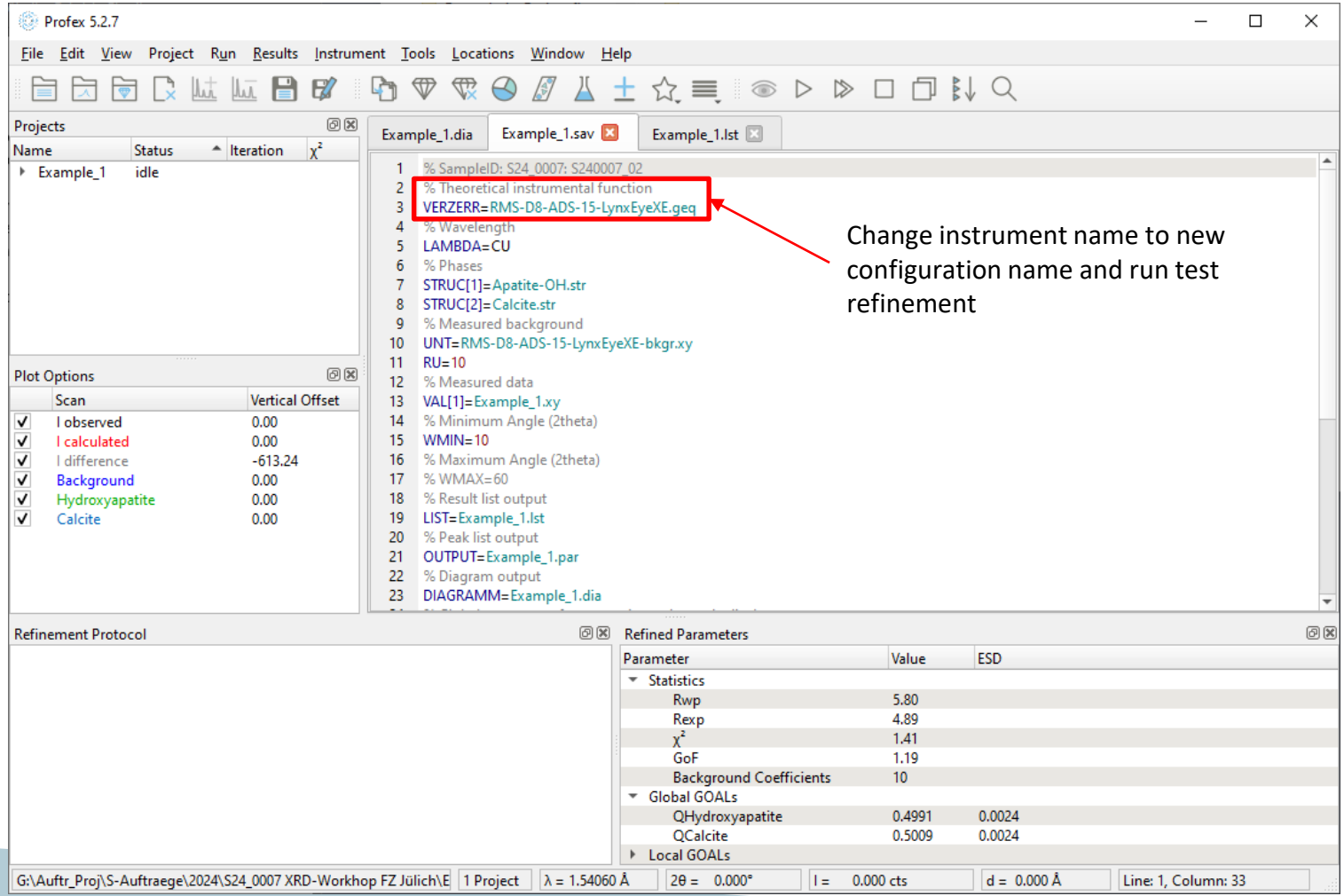


Instrument Configuration Files: Recommended Workflow

In case of errors, check messages



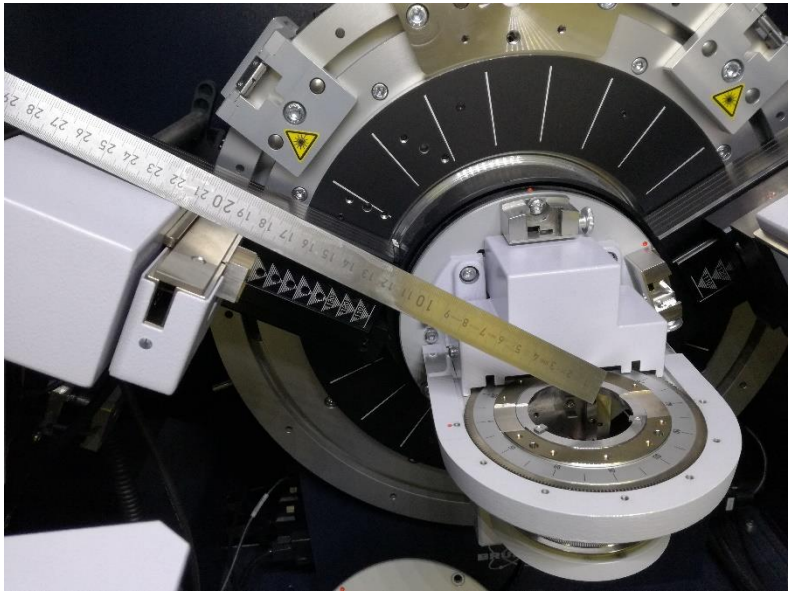
Instrument Configuration Files: Recommended Workflow



Change instrument name to new configuration name and run test refinement

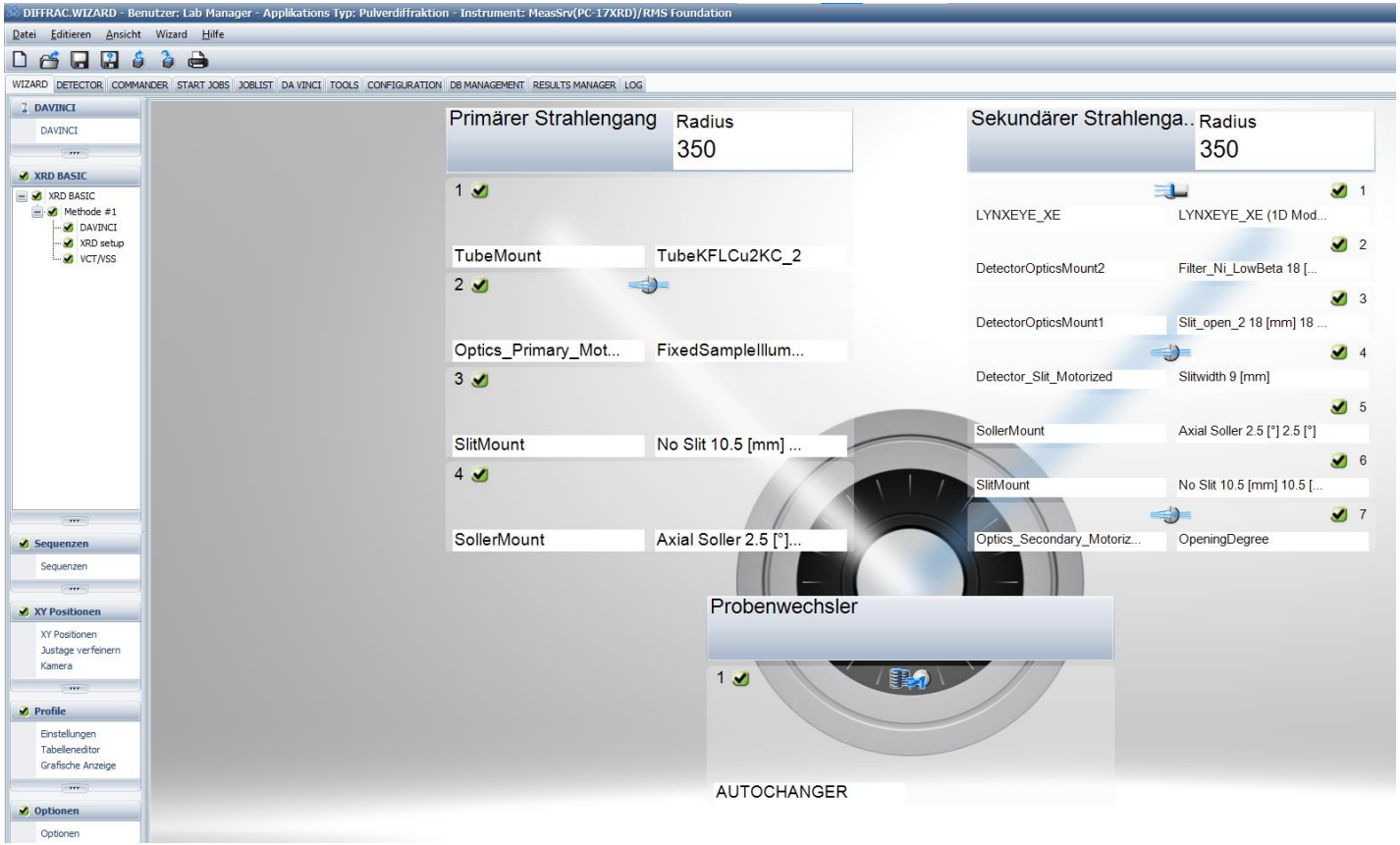
Instrument Configuration Files: Where to get Information

- Instrument control software
- Original data processing software
- XML file formats: Open in text editor or web browser
- Old school: Take measurements



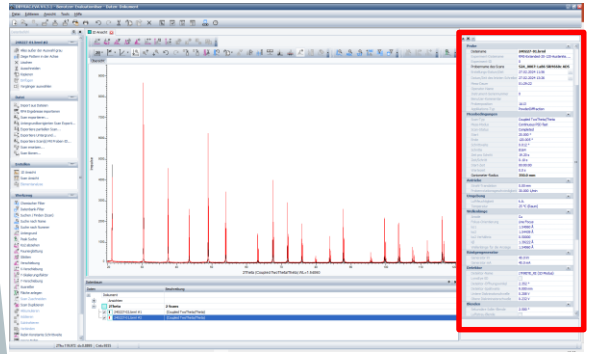
Instrument Configuration Files: Where to get Information

Instrument control software (DIFFRAC.MEASUREMENTCENTER, DataCollector, etc.)



Instrument Configuration Files: Where to get Information

Original data processing software (DIFFRAC.EVA, HighScore etc.)

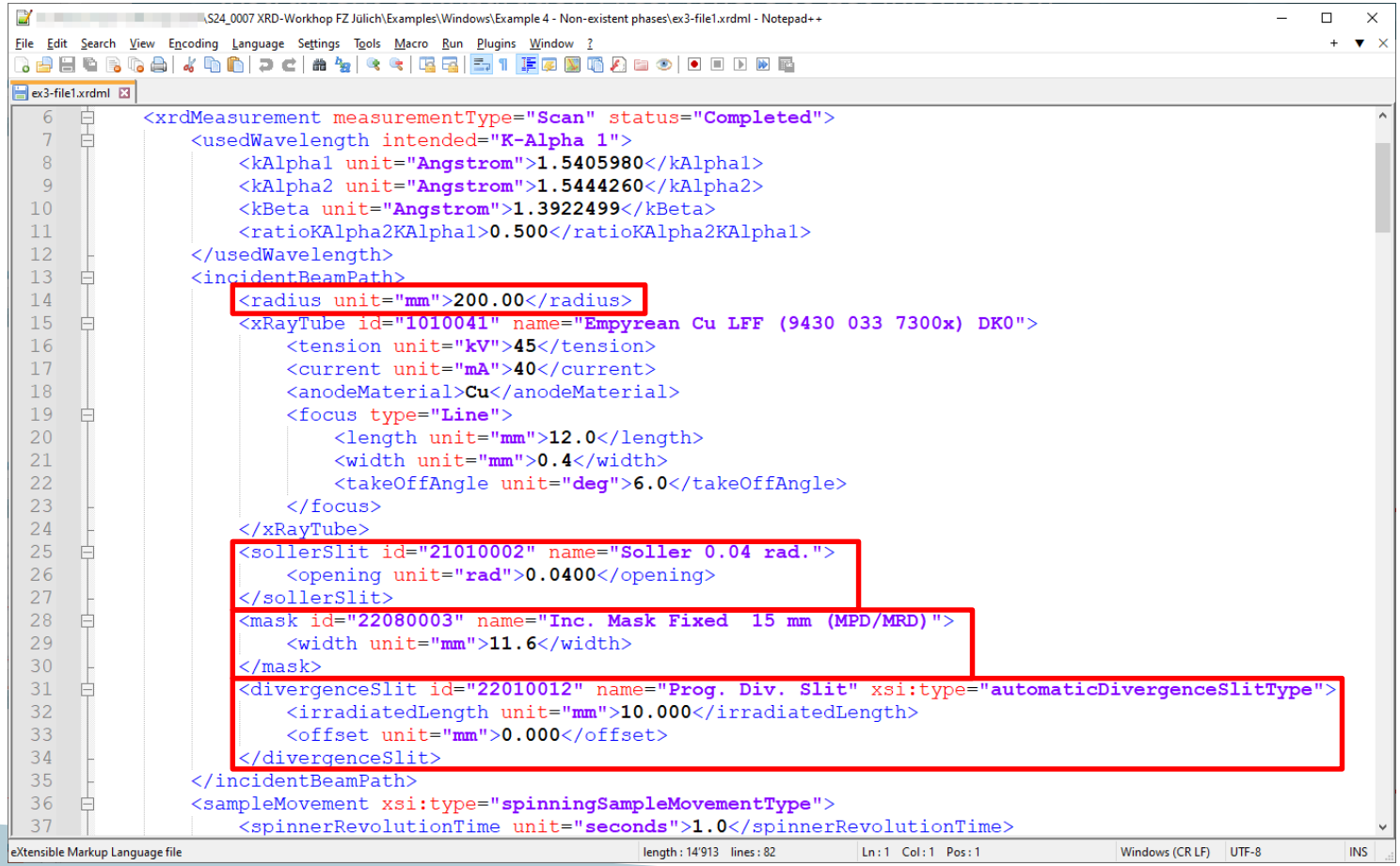


Probe	
Dateiname	**240227-01.brml**
Experiment-Dateiname	RMS-Extended-20-120-Austenite....
Experiment-ID	0
Probenname des Scans	**S24_0007: LaB6 SRM660c ADS**
Erstellungs-Datum/Zeit	27.02.2024 11:56
Datum/Zeit des letzten Schreibe	27.02.2024 13:26
Mess-Dauer	01:29:22
Operator-Name	
Instrument-Seriennummer	0
Benutzer-Kommentar	
Probenposition	1A13
Applikations-Typ	PowderDiffraction
Messbedingungen	
Scan-Typ	Coupled TwoTheta/Theta
Mess-Modus	Continuous PSD fast
Scan-Status	Completed
Start	20.000 °
Ende	120.005 °
Schrittweite	0.012 °
Schritte	8164
Zeit pro Schritt	19.20 s
Zeit/Schritt	0.10 s
Start-Zeit	00:00:00
Wartezeit	0.0 s
Goniometer-Radius	**350.0 mm**

Antriebe	
Strahl-Translation	0.00 mm
Probenrotationsgeschwindigkeit	30.000 1/min
Umgebung	
Luftfeuchtigkeit	k.A.
Temperatur	25 °C (Raum)
Wellenlänge	
Anode	Cu
Fokus-Orientierung	Line Focus
kα1	1.54060 Å
kα2	1.54439 Å
kα2 Verhältnis	0.50000
kβ	1.39222 Å
Wellenlänge für die Anzeige	1.54060 Å
Röntgengenerator	
Generator kV	40.0 kV
Generator mA	40.0 mA
Detektor	
Detektor-Name	LYNXEYE_XE (1D Modus)
LynxEye 0D	☐
Detektor-Öffnungswinkel	2.352 °
Detektor-Spaltweite	9.000 mm
Untere Diskriminatorschwelle	0.208 V
Obere Diskriminatorschwelle	0.232 V
Blenden	
Sekundäre Soller-Blende	2.500 °
Luftstreu-Blende	☐
Divergenz-Blende	15.000 mm
Streustrahl-Blende	3.030 °
Blenden-Modus	Variabel
Simul. Blenden-Modus	
Korrekturen	

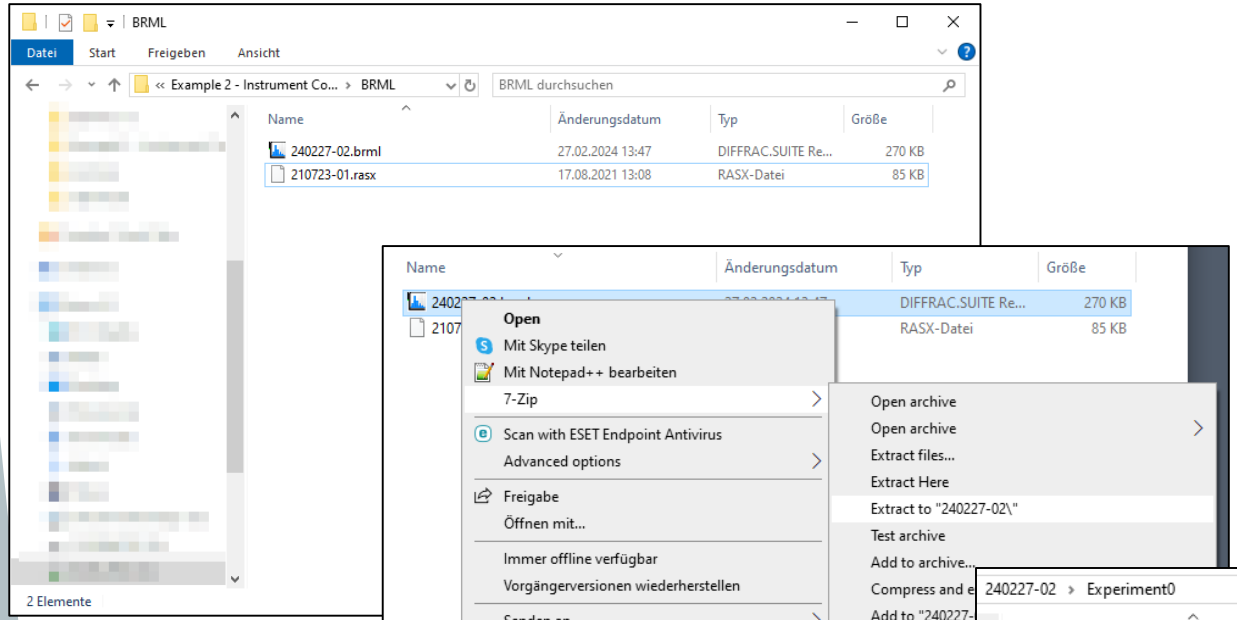
Instrument Configuration Files: Where to get Information

XML file formats: Open in text editor or web browser (XRDML)



Instrument Configuration Files: Where to get Information

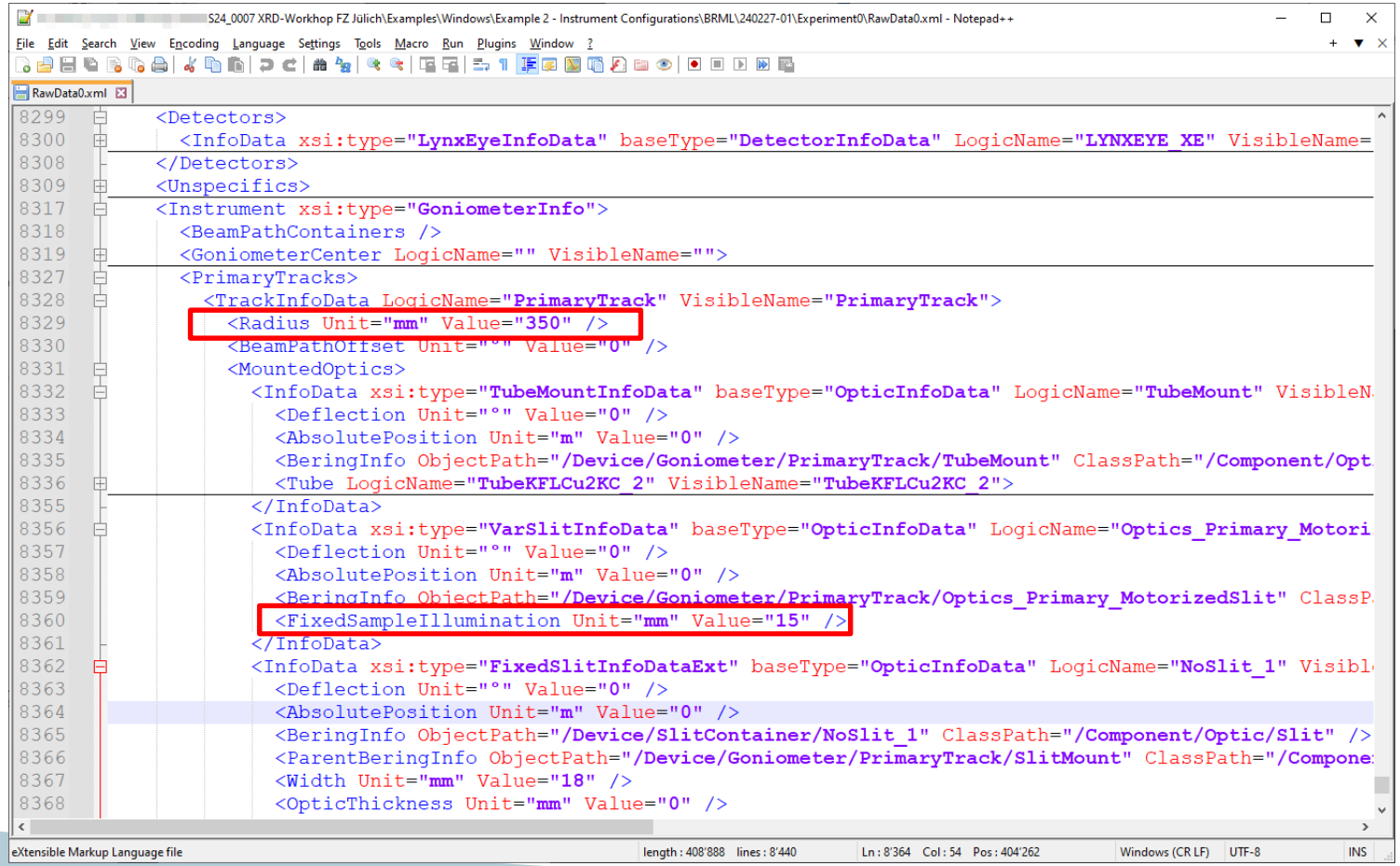
Compressed XML file formats: Extract and open in text editor or web browser (BRML, RASX)



or rename to *.zip before extracting the archive

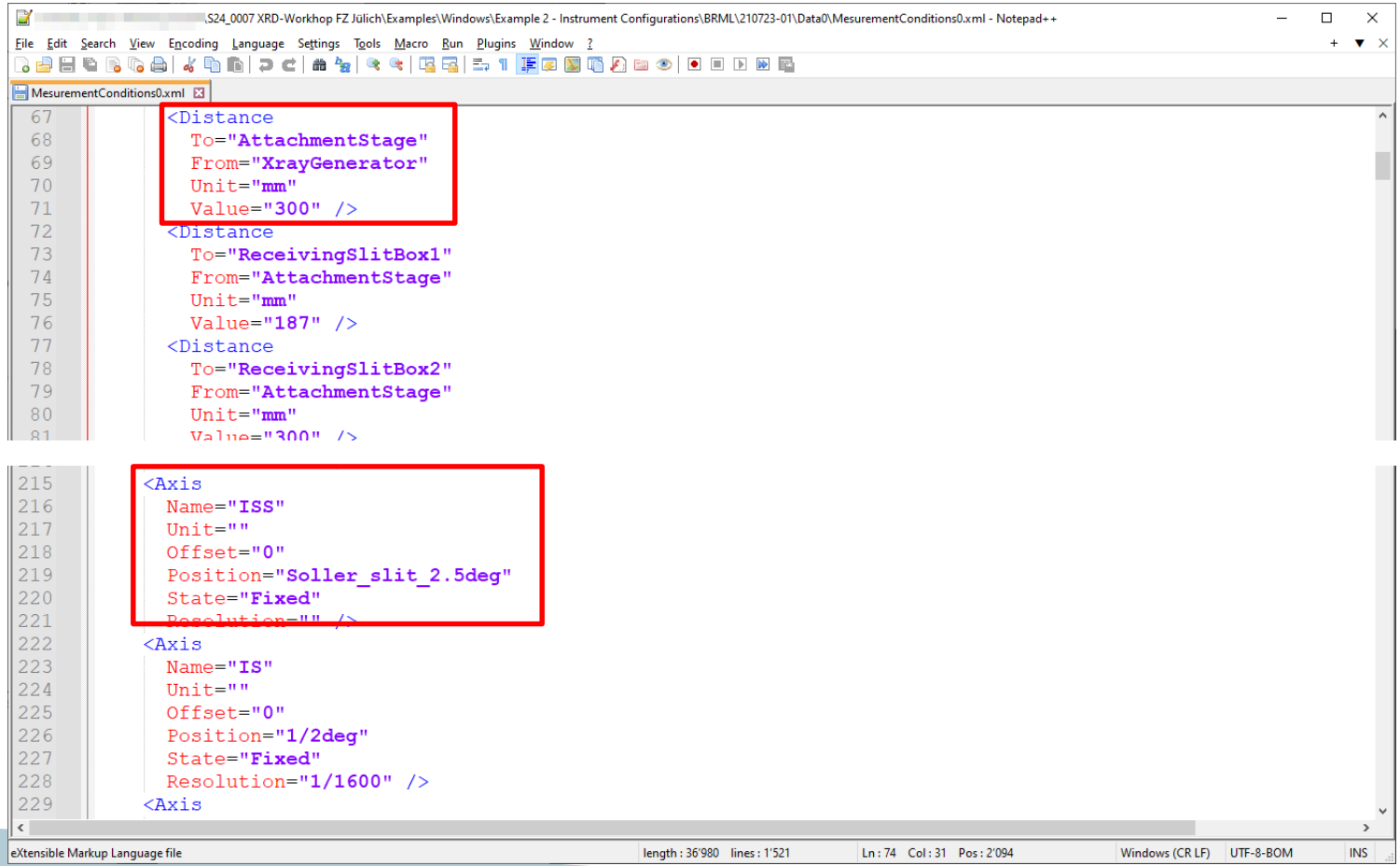
Instrument Configuration Files: Where to get Information

Bruker BRML: Open file „RawData0.xml“



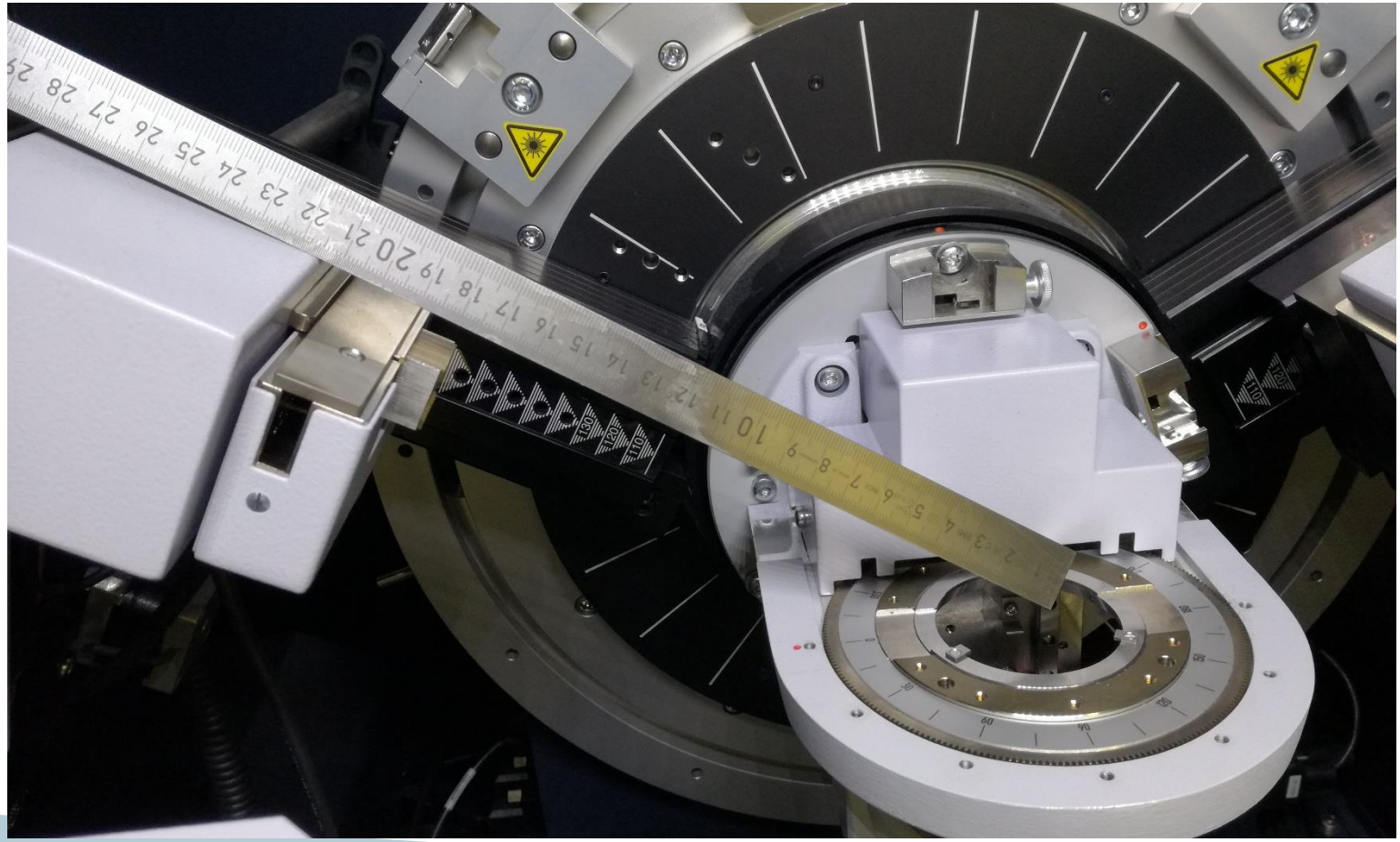
Instrument Configuration Files: Where to get Information

Rigaku RASX: Open file „MeasurementConditions0.xml“



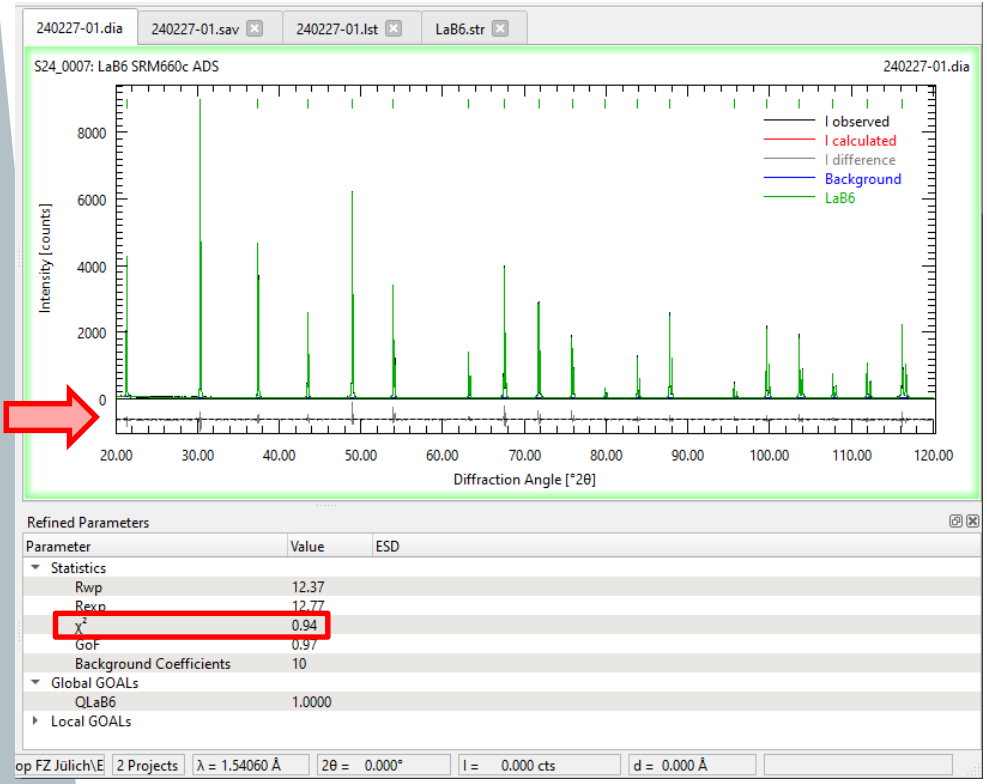
Instrument Configuration Files: Where to get Information

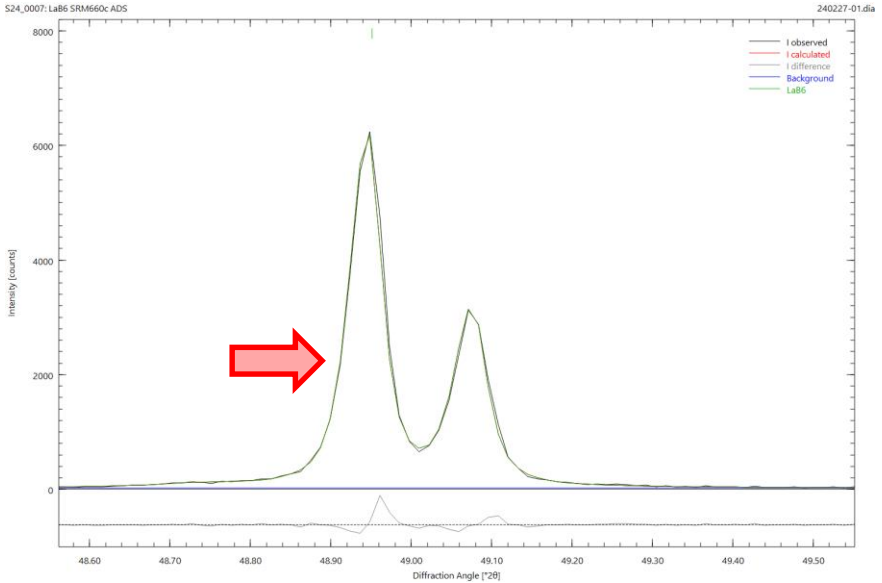
If all other options failed: Take measurements



Instrument Configuration Files

Always verify new instrument configuration files by refining a reference sample (preferrably LaB6 NIST SRM 660)





Flat difference curve
Accurate fit of the peak shape
χ^2 close to 1.0

Structure and Device Files: Summary

- Structure files:
  - Use GOAL= to write additional results to the *.LST file
  - Use GOAL: to export variables to the *.SAV file for further computations
  - BGMN expression interpreter allows to implement custom calculations (e.g. substitution models)

- Device files:
  - Must accurately represent the true instrument configuration
  - Get information from:
    - Measurement software
    - Original data processing software
    - XML scan files
    - Take measurements
  - Verify with reference measurement