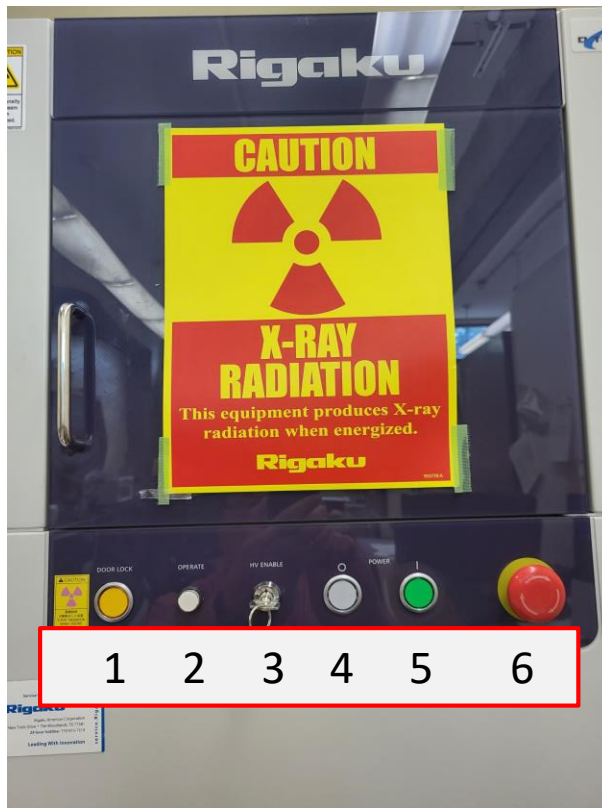


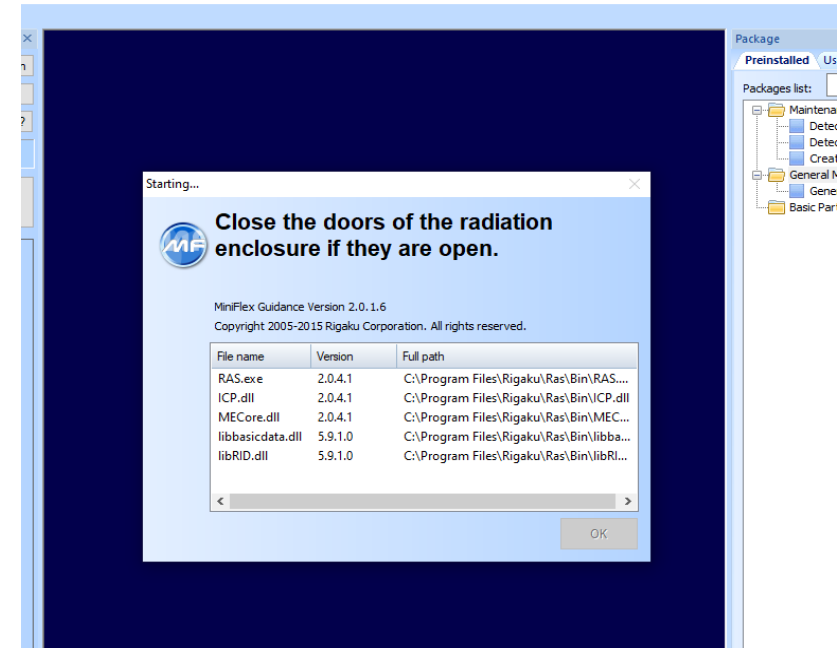
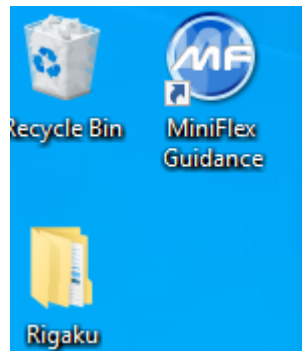
MiniFlex 600

1. Sign the log sheet. When finished be sure to enter the total time in decimal hours.
2. Log onto the control computer using your NetID /password.
3. Turn on the diffractometer. Press the **On** button then the Door access button to stop the beeping sound.

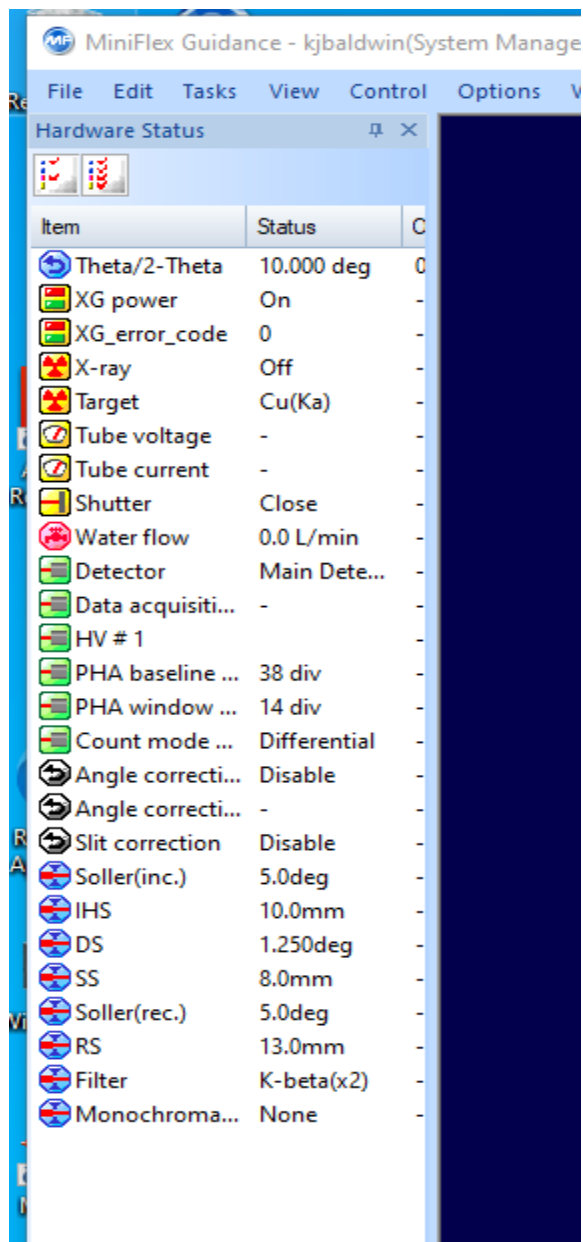


- 1 – Door access. When flashing and beeping door is unlocked.
- 2 – Indicator light
- 3 – Enable/Disable key. When key is removed diffractometer is disabled
- 4 – Off button
- 5 – On button
- 6 – Emergency Stop Button

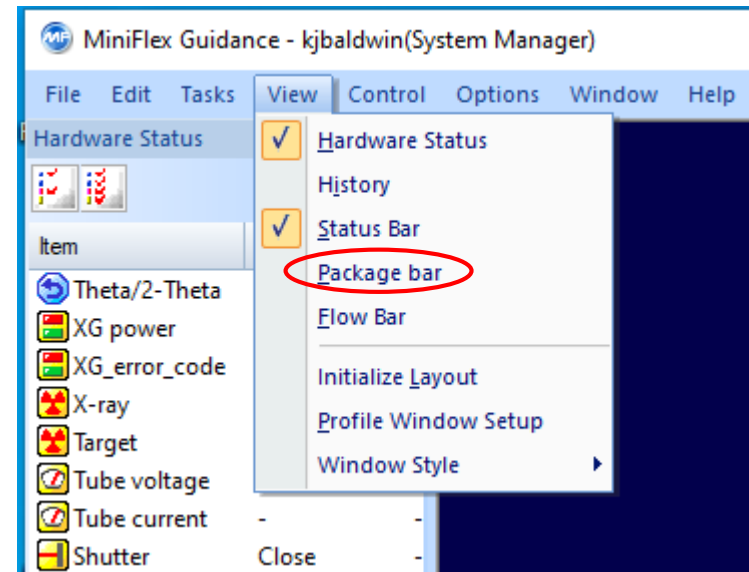
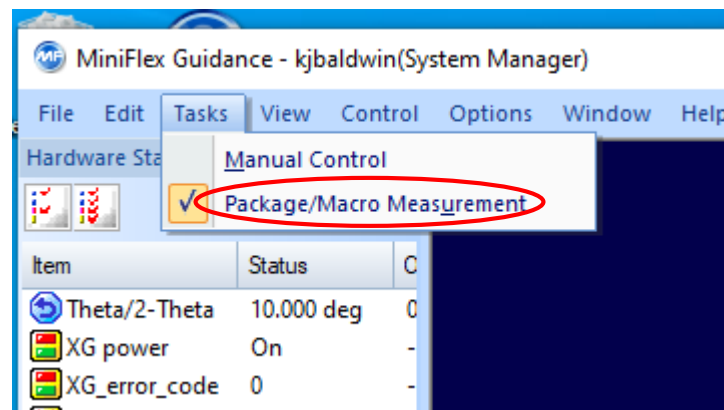
4. Start the control program. Double click MiniFlex Guidance on the desktop. The MiniFlex Guidance login screen will appear with your NetID as the default Login name. Click OK. The MiniFlex *Starting* window will appear as the system initializes.



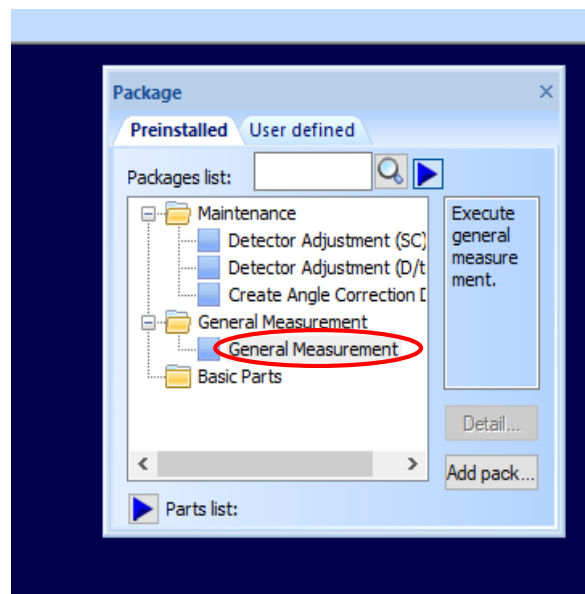
5. Once the system has initialized the main window will open.



6. Select *Tasks* → *Package/Macro Management* and *View* → *Package Bar*

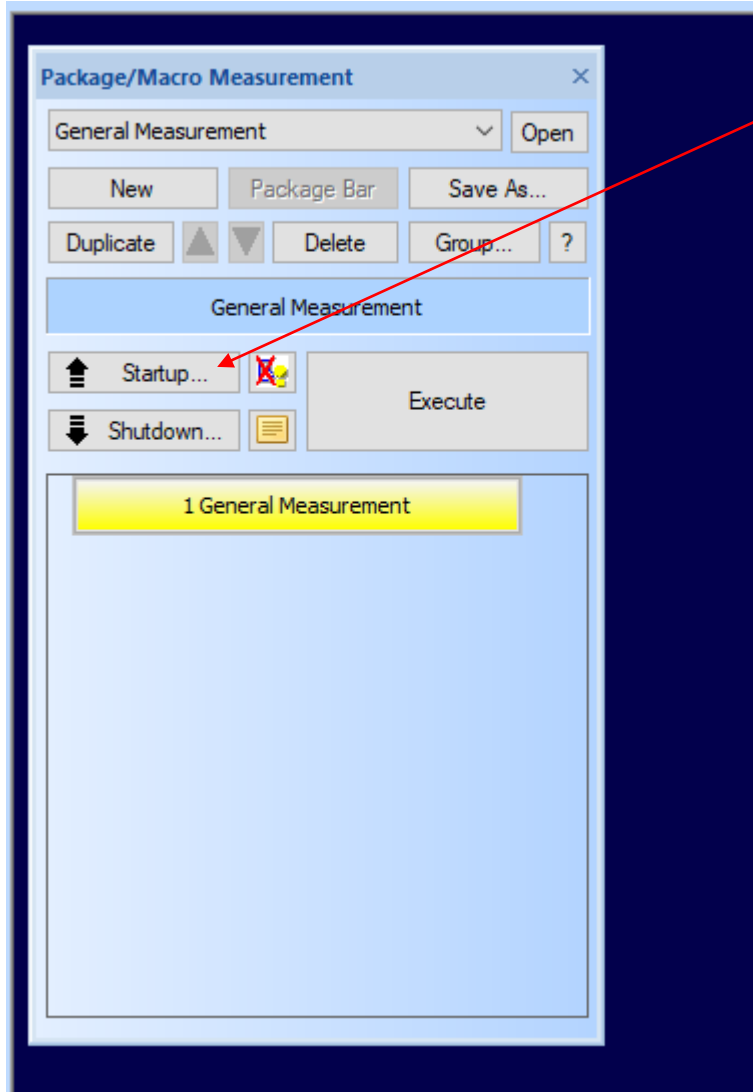


7. When the *Package* window opens double click *General Measurement*

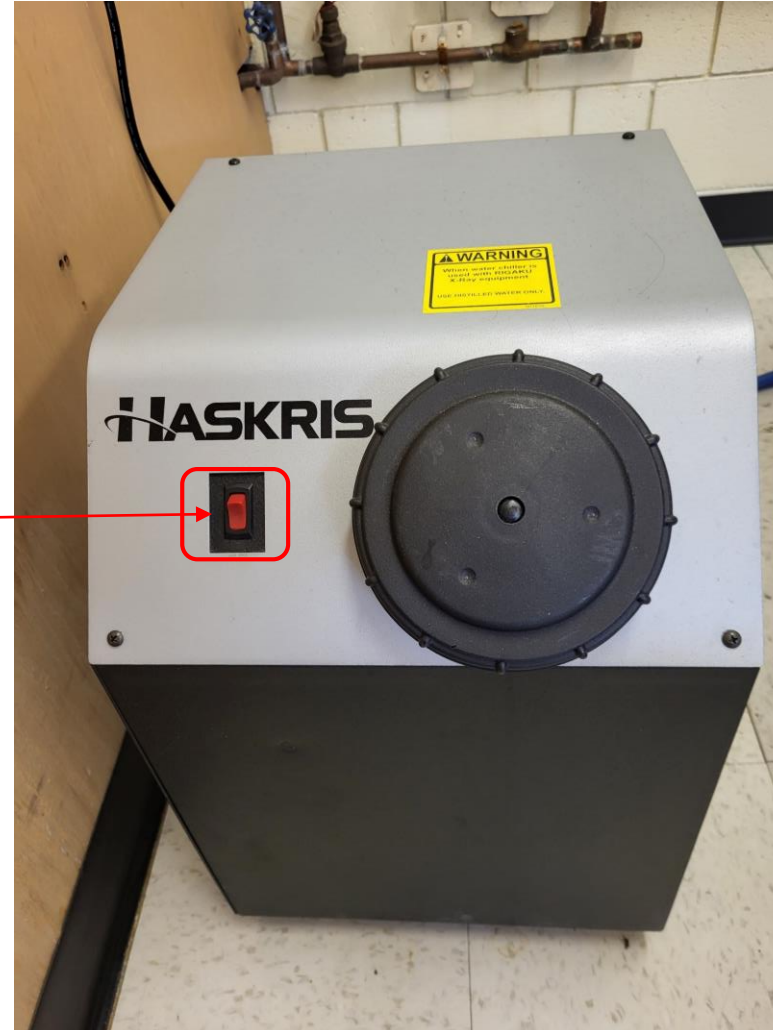


If you exit the program correctly, steps 6 and 7 will be remembered and will not have to be repeated the next time you run *MiniFlex Guidance*.

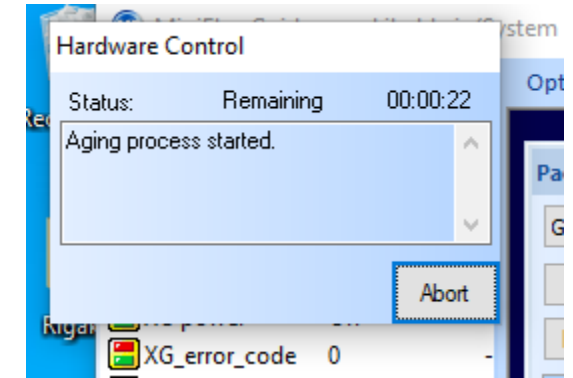
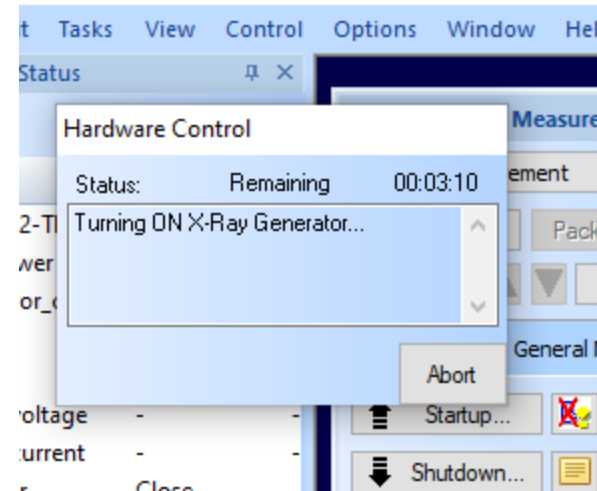
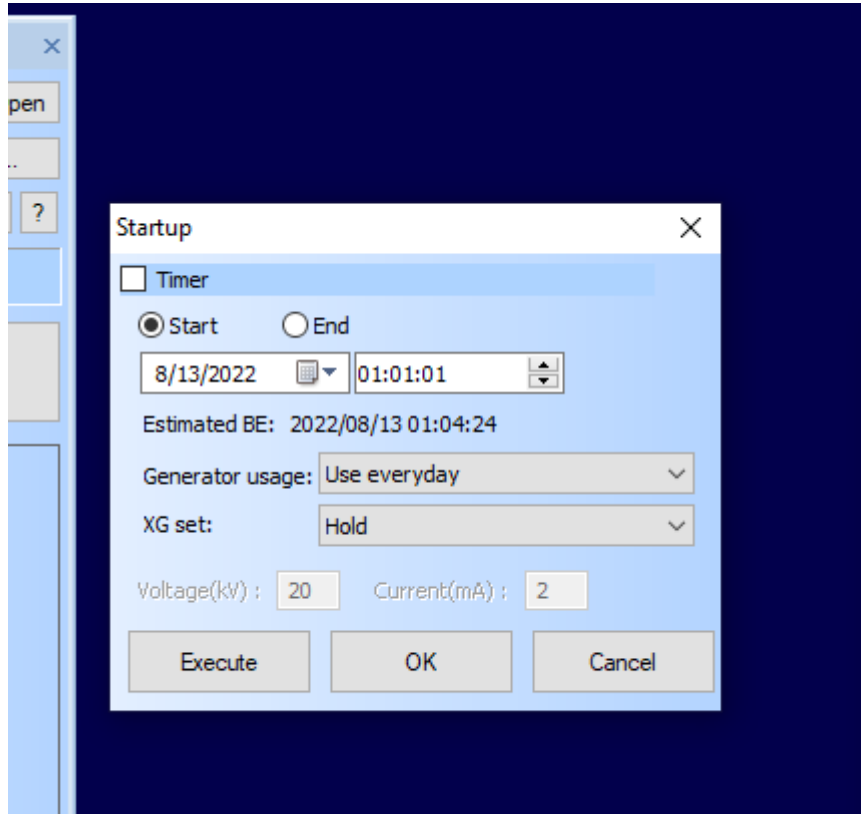
8. The *Package/Macro Measurement* window will open.



9. Turning on the x-ray generator. Turn on the Haskris heat exchanger then click Startup in the *Package/Macro Measurement* window.



10. The generator *Startup* window will open. Leave *Generator usage:* as *Use everyday* and click *Execute*. The *Turning On X-ray Generator* message will popup followed by *Aging process started*. This will take a few minutes.



11. When the generator is on and ready you can click *OK* in the *Startup* window then *1 General Measurement* in *Package/Macro Measurement* window

The screenshot displays the MiniFlex Guidance software interface. On the left, the 'Hardware Status' window shows a list of system parameters and their current states. In the center, the 'Package/Macro Measurement' window is open, showing a list of measurement packages with '1 General Measurement' highlighted. On the right, the 'Startup' window is open, showing configuration options for the measurement, with the 'OK' button highlighted.

Item	Status
Theta/2-Theta	10.000 deg
XG power	On
XG_error_code	0
X-ray	On
Target	Cu(Ka)
Tube voltage	40 kV
Tube current	15 mA
Shutter	Close
Water flow	3.5 L/min
Detector	Main Dete...
Data acquisiti...	-
HV # 1	-
PHA baseline ...	38 div
PHA window ...	14 div
Count mode ...	Differential
Angle correcti...	Disable
Angle correcti...	-
Slit correction	Disable
Soller(inc.)	5.0deg
IHS	10.0mm
DS	1.250deg
SS	8.0mm
Soller(rec.)	5.0deg
RS	13.0mm
Filter	K-beta(x2)
Monochroma...	None

Package/Macro Measurement

General Measurement

1 General Measurement

Startup

Timer

Start End

8/13/2022 01:01:01

Estimated BE: 2022/08/13 01:04:24

Generator usage: Use everyday

XG set: Hold

Voltage(kV): 20 Current(mA): 2

Execute OK Cancel

12a *General Measurement* Check the first check box.

The screenshot shows a 'General Measurement' dialog box with the following elements:

- Checkbox: Automatic generation of data file names
- Table with 10 rows and columns: No., Exec., Folder, File name, Sample name, Memo, Condition, Analysis condition, Spin, Sample.
- Buttons: Run, Set Meas. Conditions..., OK, Cancel.
- Radio buttons for XG termination condition: The status quo, Stop the X-Ray.
- Radio buttons for Show optical devices confirmation message: Show, Don't show.

No.	Exec.	Folder	File name	Sample name	Memo	Condition	Analysis condition	Spin	Sample
1	<input checked="" type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
2	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
3	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
4	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
5	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
6	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
7	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
8	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
9	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1
10	<input type="checkbox"/>					No1	Set...	<input type="checkbox"/>	1

12b *General Measurement* continued. Click the navigation button..

General Measurement

Automatic generation of data file names

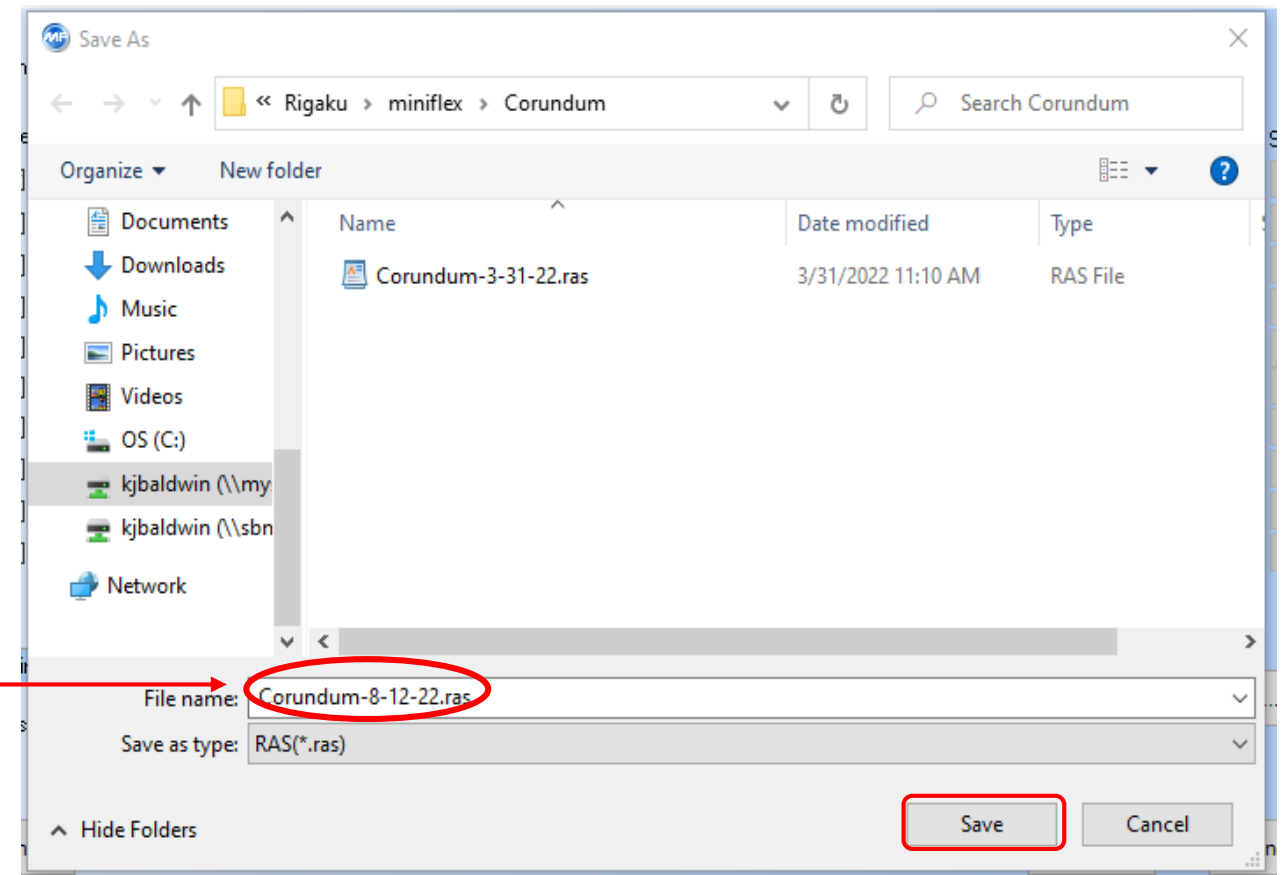
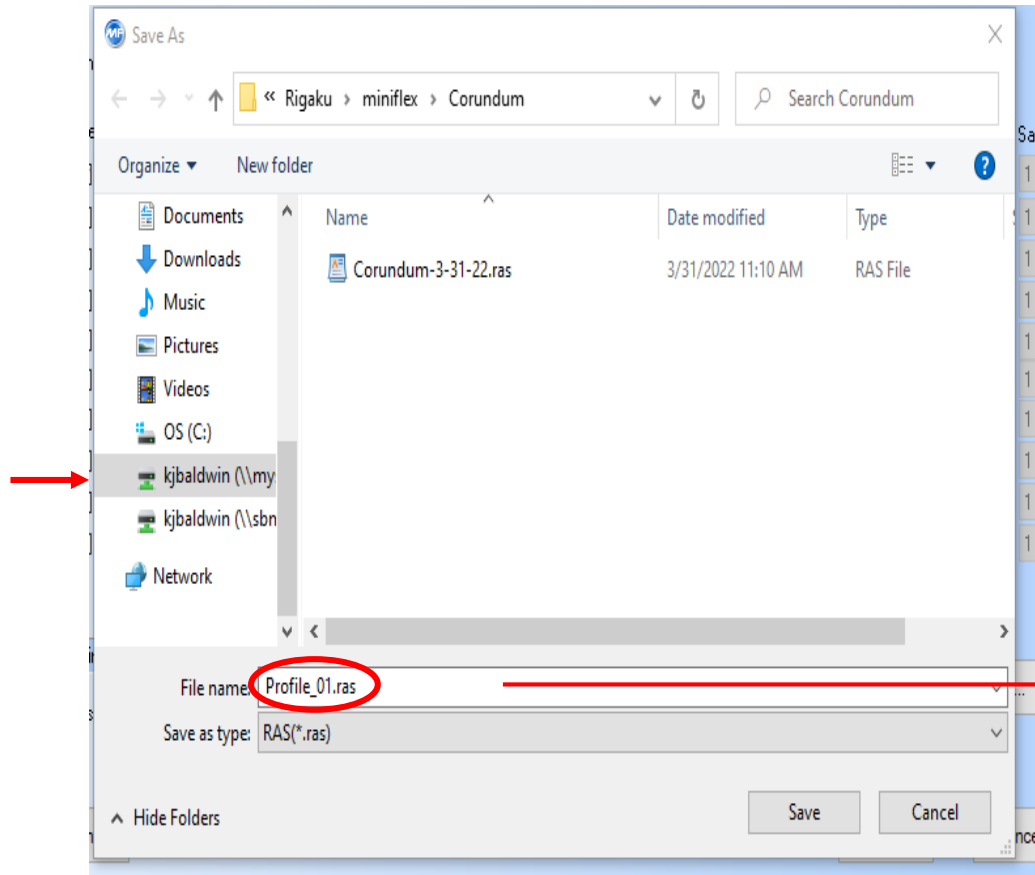
No.	Exec.	Folder	File name	Sample name	Memo	Condition	Analysis condition	Spin	Sample
1	<input checked="" type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
2	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
3	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
4	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
5	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
6	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
7	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
8	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
9	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
10	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1

XG termination condition: The status quo Stop the X-Ray

Show optical devices confirmation message: Show Don't show

Buttons: Run, Set Meas. Conditions..., OK, Cancel, ?

12c Navigate to mysbfiles and enter an appropriate filename and click Save



12d *General Measurement* continued. *Sample name* and *Memo* are text fields, enter any information you want or they may be left blank. Leave *Condition* as *No1* and click *Set Meas. Conditions...*

General Measurement

Automatic generation of data file names

No.	Exec.	Folder	File name	Sample name	Memo	Condition	Analysis condition	Spin	Sample
1	<input checked="" type="checkbox"/>	X:\Rigaku\miniflex\Corundum\	Corundum-8-12-22.ras	...		No1	Set...	<input type="checkbox"/>	1
2	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
3	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
4	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
5	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
6	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
7	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
8	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
9	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1
10	<input type="checkbox"/>			...		No1	Set...	<input type="checkbox"/>	1

XG termination condition: The status quo Stop the X-Ray

Show optical devices confirmation message: Show Don't show

Buttons: Run, Set Meas. Conditions..., OK, Cancel, ?

13a Measurement Condition

Measurement Condition

Slit condition: Variable slit system (radio), Variable+Fixed slit system (radio selected), Background condition: Background measurements (checkbox)

Optical devices:

- Soller(inc.): 5.0 deg
- IHS: 10.0 mm
- DS: 1.250 deg
- SS: 13.0 mm(Open)
- Soller(rec.): 5.0 deg
- RS: 13.0 mm(Open)
- Monochromatization: None

Measurement condition: Edit the measurement conditions, File create

Preset Condition... Copy... Reset Individual configuration files (checkbox)

Scan axis: Theta/2-Theta Mode: Continuous Unit: CPS

Exec.	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Comment	Voltage (kV)	Current (mA)	Background conditions
<input checked="" type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...

Calculated scan duration: 00:00:00

OK Cancel

Make the following changes so this information matches the physical setup of the diffractometer.

Change *Monochromatization* to *kb filter (x2)*

Change *SS* to 8.0 mm

If you exit the program correctly, these changes will be remembered and will not have to be repeated the next time you run *MiniFlex Guidance*.

Check *box 1* and enter appropriate values for *Start Stop* and *Speed*. Leave *Step* as 0.02

13b Measurement Condition continued. Click *OK* and *General Measurement* panel will open. But first load the sample.

Measurement Condition

Slit condition: Background condition:

Variable slit system Variable+Fixed slit system Background measurements

Optical devices

Soller(inc.) IHS DS
5.0 deg 10.0 mm 1.250 deg

SS Soller(rec.) RS Monochromatization
8.0 mm 5.0 deg 13.0 mm(Open) kb filter (x2)

Measurement condition Edit the measurement conditions File create

Preset Condition... Copy... Reset Individual configuration files

No1 No2 No3 No4 No5 No6 No7 No8 No9 No10

Scan axis: Theta/2-Theta Mode: Continuous Unit: CPS

	Exec.	Start	Stop	Step	Speed	Comment	Voltage	Current	Background
	(deg)	(deg)	(deg)	(deg)	(deg/min)		(kV)	(mA)	conditions
1	<input checked="" type="checkbox"/>	15.000	95.000	0.02	40.0		40	15	Set...
2	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
3	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
4	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
5	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
6	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
7	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
8	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
9	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...
10	<input type="checkbox"/>	3.0000	90.0000	0.02	10.0		40	15	Set...

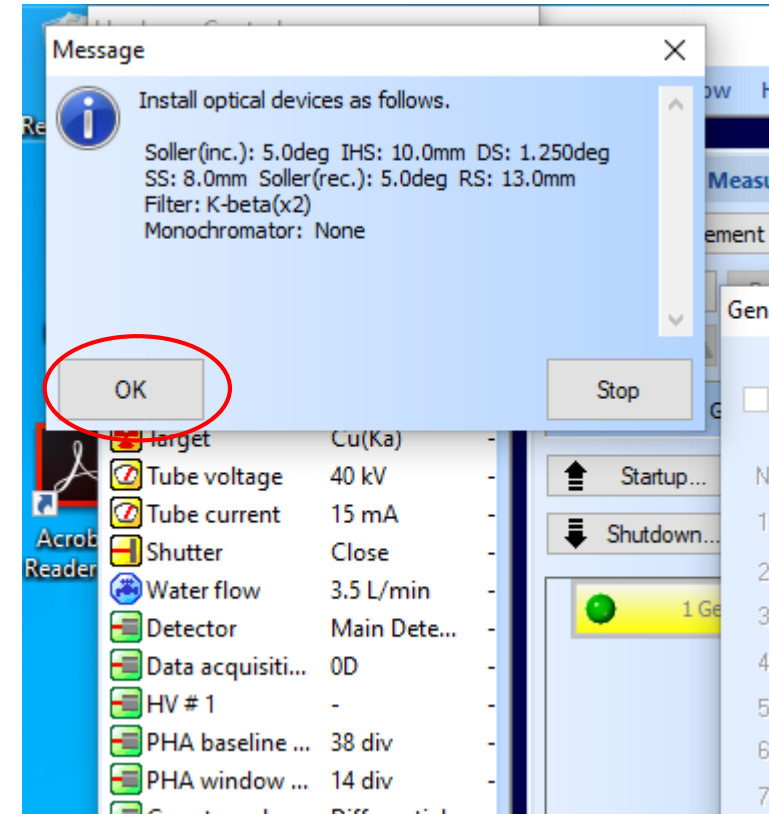
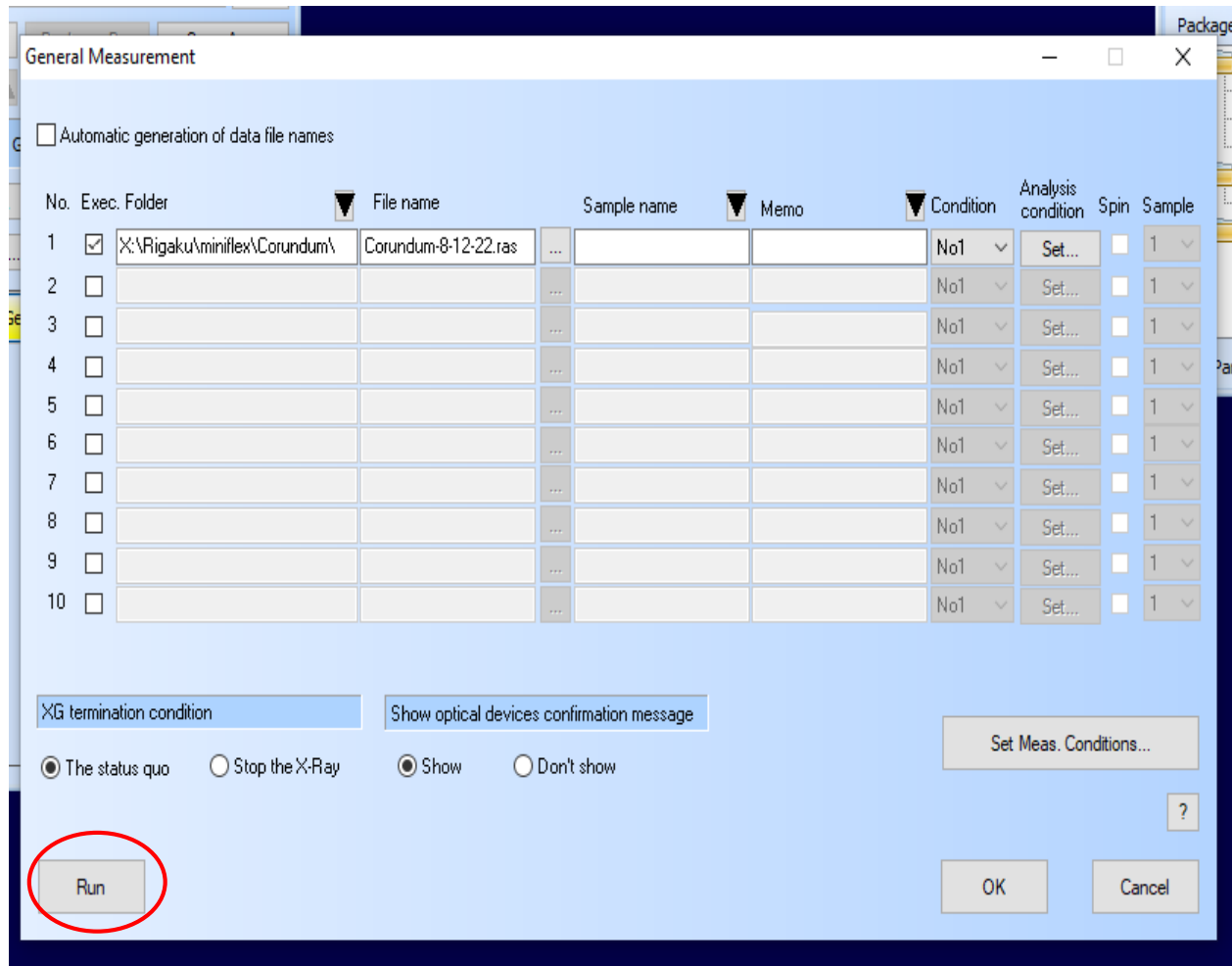
Calculated scan duration: 00:02:27

OK Cancel

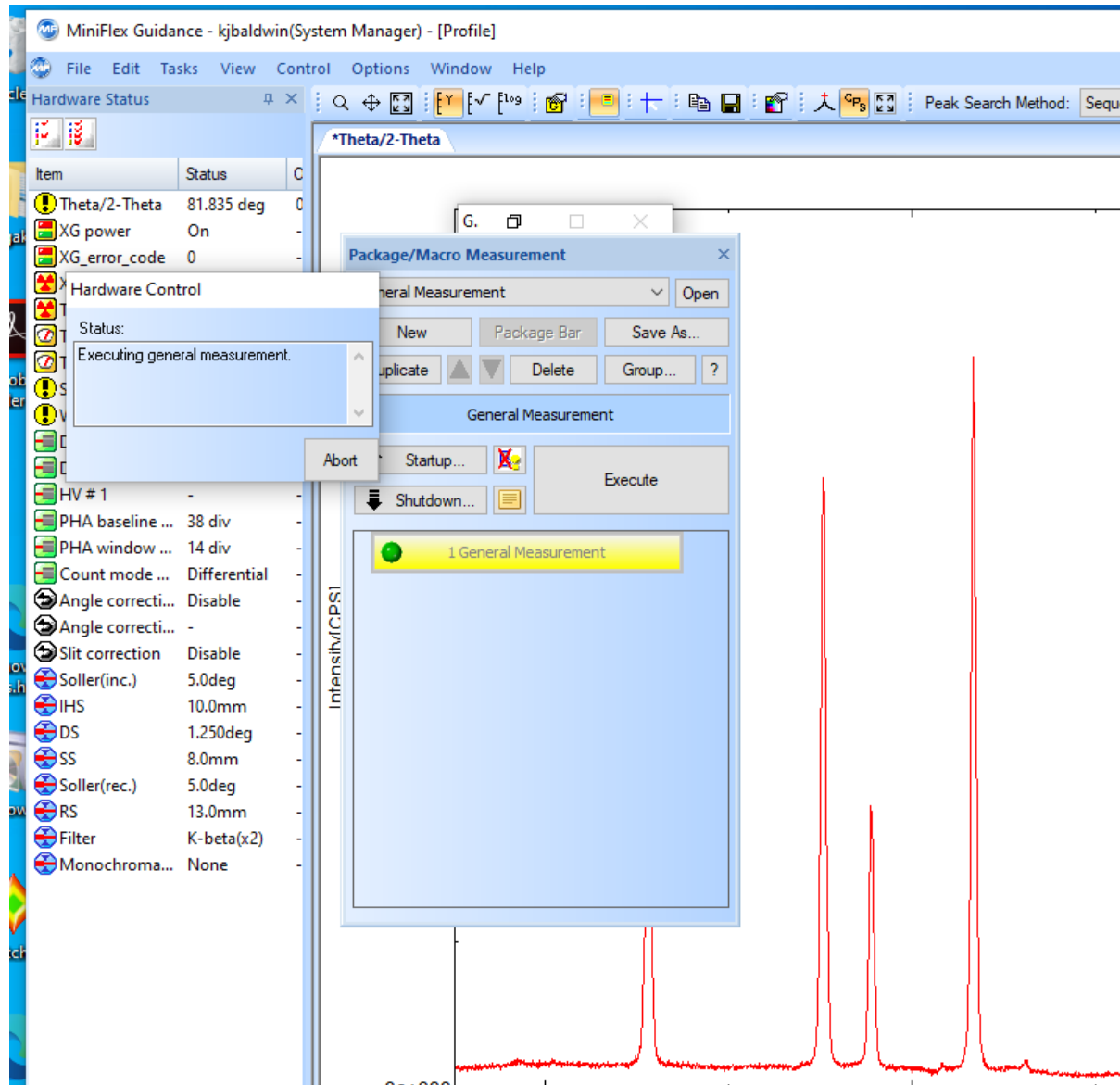
14 Load the sample.



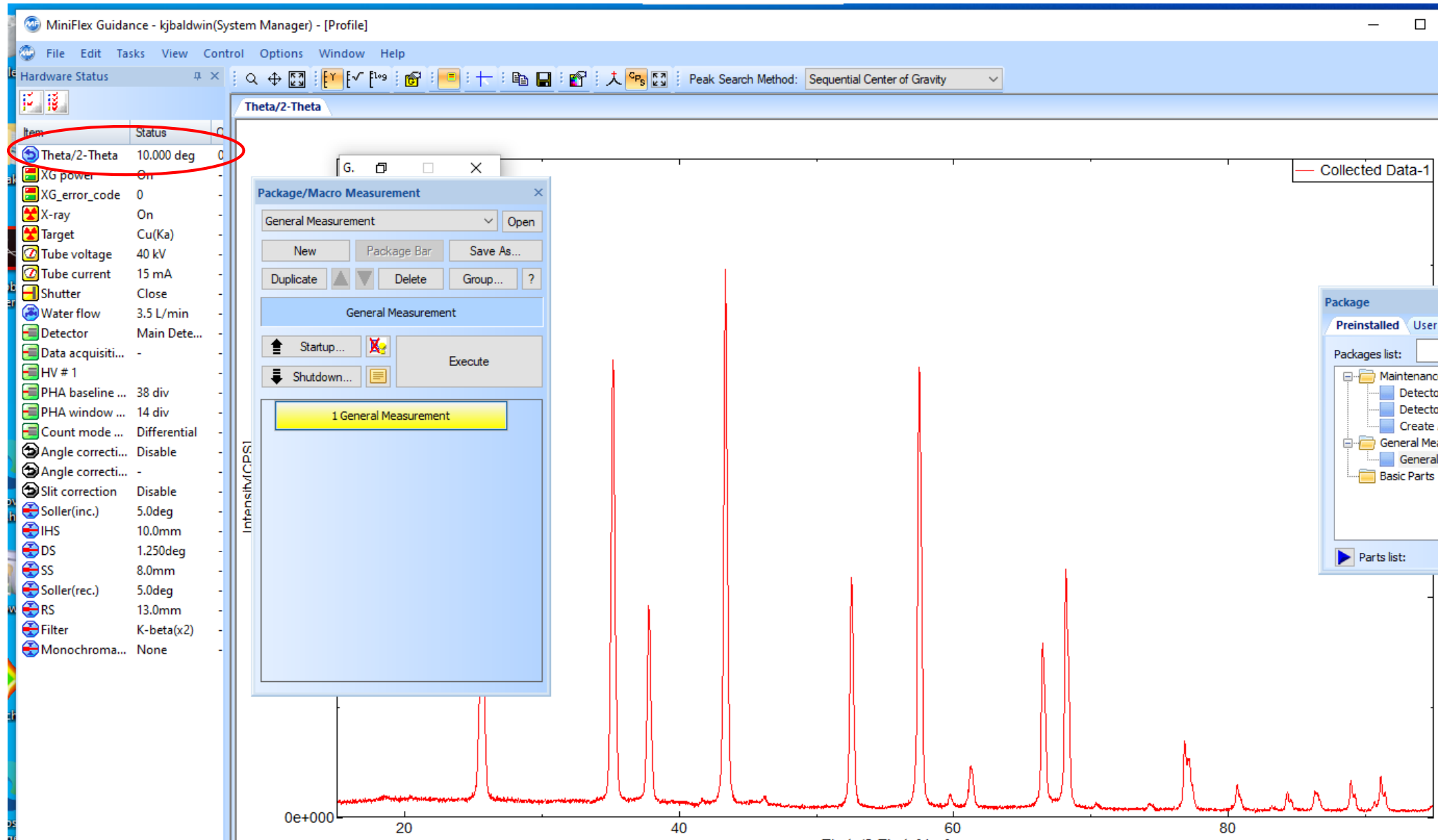
14 General Measurement Click *Run*. A confirmation *Message* will pop up. Verify the information and click *OK*



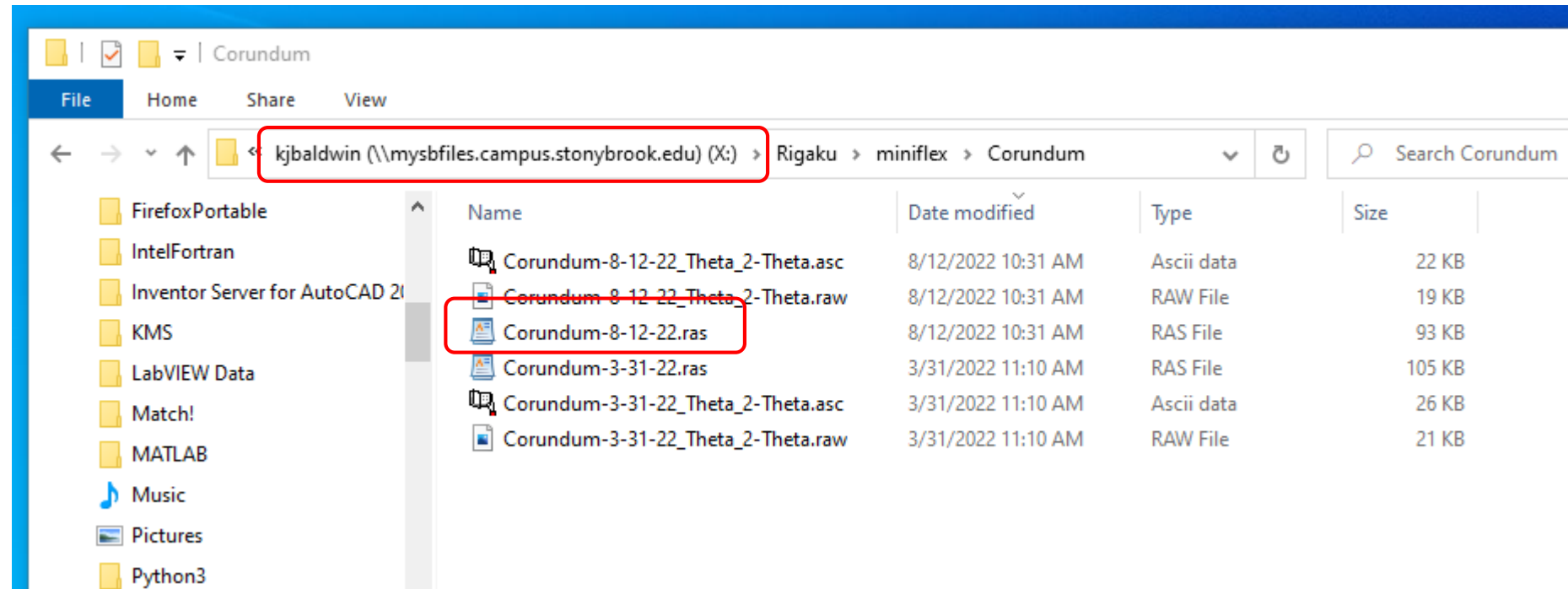
15 Data Collection in progress.



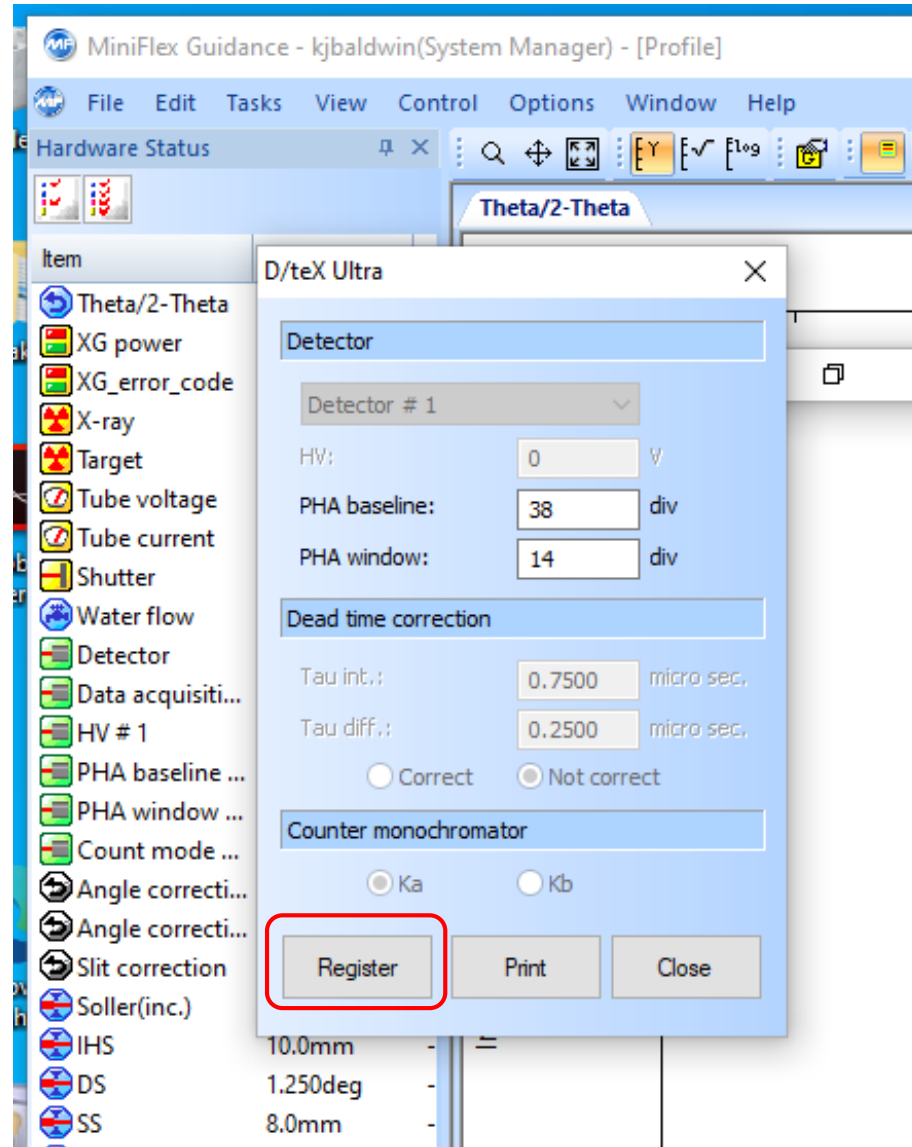
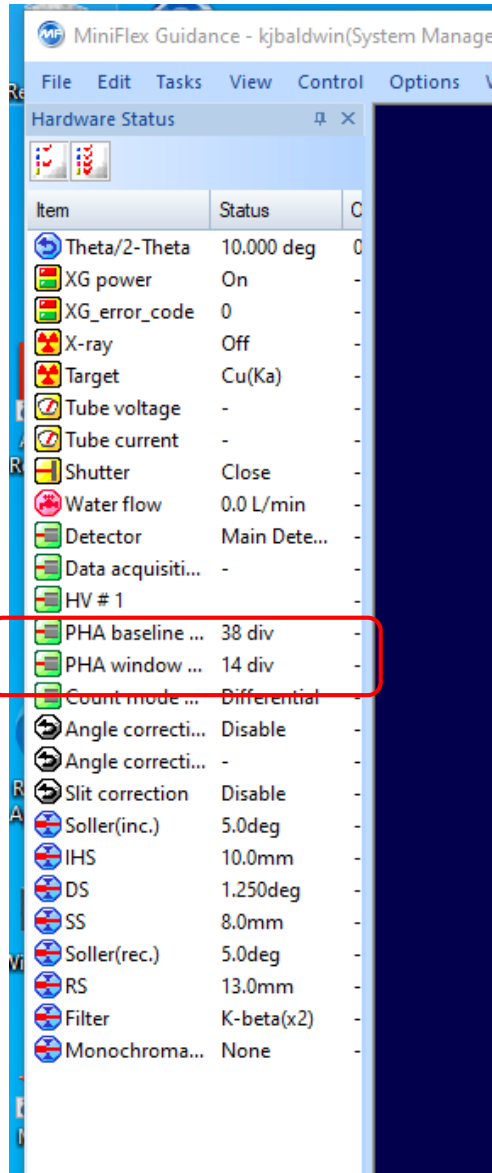
16a Data Collection finished. Notice that *Theta/2-Theta* is back to the home position, 10.000 deg.



16b Data Collection finished. Open windows explorer and navigate to your *data directory*. Notice that three files have been created. *Corundum-8-12-22.ras* is file containing the raw data.



17a Adjusting the PHA to minimize Fe fluorescence. Adjust the PHA baseline and PHA window:
 Select *Options* → *Detector Setup* → *1 D/teX Ultra*. Enter the desired values and click *Register*



Mode	Baseline	Window
Normal	38	14
Fe	45	7

18a Turning off the generator. Click *Shutdown*. In *Shutdown* window select XG Off. Click Execute

The image displays a sequence of three screenshots from the MiniFlex Guidance software, illustrating the steps to turn off the generator.

First Screenshot: The main software window is titled "MiniFlex Guidance - kjbaldwin(System Manager) - [Profile]". The "Hardware Status" panel on the left lists various components and their statuses. The "Package/Macro Measurement" window is open, showing a "General Measurement" package. The "Shutdown..." button is highlighted with a red box. Below the package list, a graph shows "Intensity(CPS)" vs. "2θ" with a peak at approximately 20 degrees.

Second Screenshot: The "Shutdown" dialog box is open. The "Execute" checkbox is unchecked. Under "Shutdown conditions", the "XG set:" dropdown menu is open, showing options: "Set", "Set to minimum", and "XG Off". The "XG Off" option is selected. The "Voltage(kV):" is set to 40 and "Current(mA):" is set to 15. The "Execute", "OK", and "Cancel" buttons are visible at the bottom.

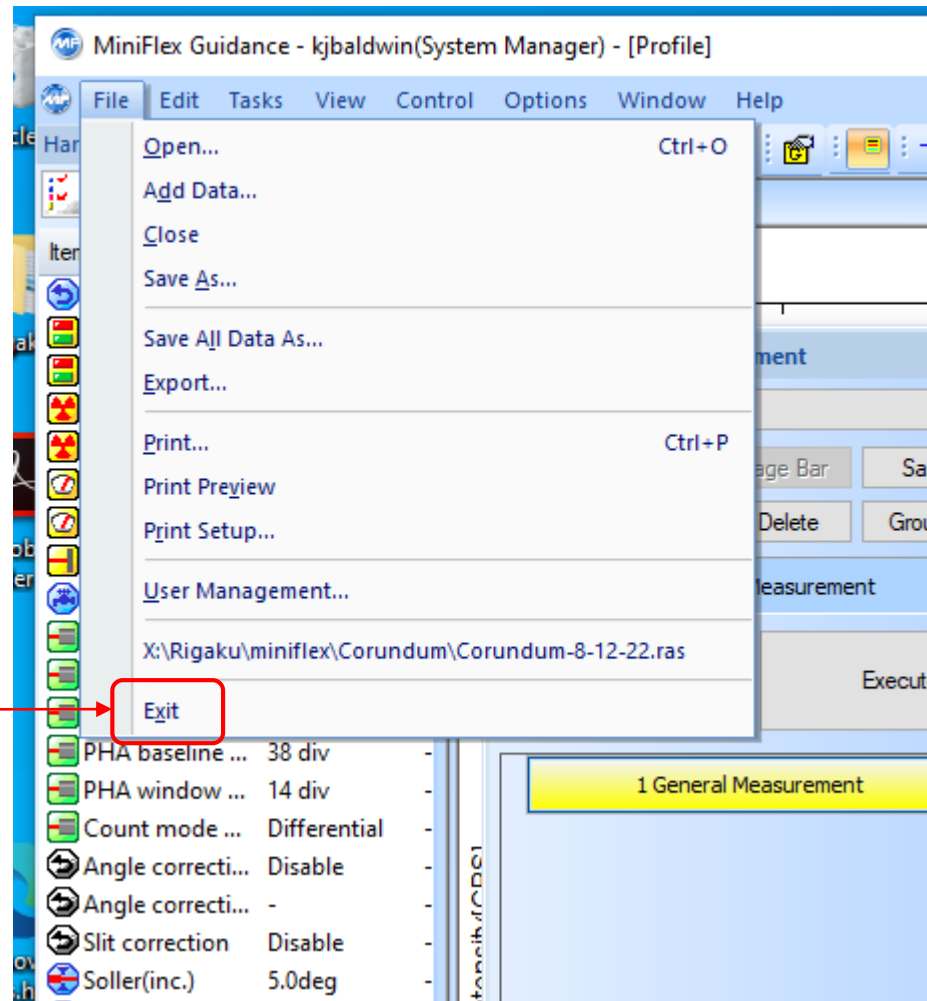
Third Screenshot: The "Shutdown" dialog box is open. The "Execute" checkbox is unchecked. Under "Shutdown conditions", the "XG set:" dropdown menu is open, showing options: "10 minutes before Power Off", "Voltage(kV):", and "Current(mA):". The "10 minutes before Power Off" option is selected. The "Voltage(kV):" is set to 40 and "Current(mA):" is set to 15. The "Execute", "OK", and "Cancel" buttons are visible at the bottom.

18b Turning off the generator. Once the generator is off the *Tube Voltage and Current* will be blank. In the *Shutdown* window click *OK*. Do not turn off the heat exchanger yet.

The screenshot displays the XRD software interface. On the left is a list of parameters with their current status. A red box highlights the 'Tube voltage' and 'Tube current' entries, which are currently blank. In the center, the 'Package/Macro Measurement' window is open, showing a 'General Measurement' package with an 'Execute' button. On the right, the 'Shutdown' dialog box is open, with the 'Execute' checkbox checked and the 'OK' button highlighted by a red box. The 'Shutdown conditions' section shows 'XG set' as 'XG Off', a 10-minute delay before power off, and 'Voltage(kV)' set to 40 and 'Current(mA)' set to 15.

Item	Status	Unit
Theta/2-Theta	10.000 deg	0
XG power	On	
XG_error_code	0	
X-ray	Off	
Target	Cu(Ka)	
Tube voltage	-	
Tube current	-	
Shutter	Close	
Water flow	3.5 L/min	
Detector	Main Dete...	
Data acquisiti...	-	
HV # 1	-	
PHA baseline ...	38 div	
PHA window ...	14 div	
Count mode ...	Differential	
Angle correcti...	Disable	
Angle correcti...	-	
Slit correction	Disable	
Soller(inc.)	5.0deg	
IHS	10.0mm	
DS	1.250deg	
SS	8.0mm	
Soller(rec.)	5.0deg	
RS	13.0mm	

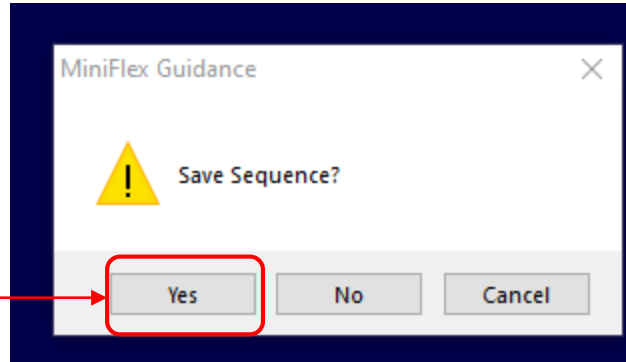
19a Exiting from MiniFlex Guidance.



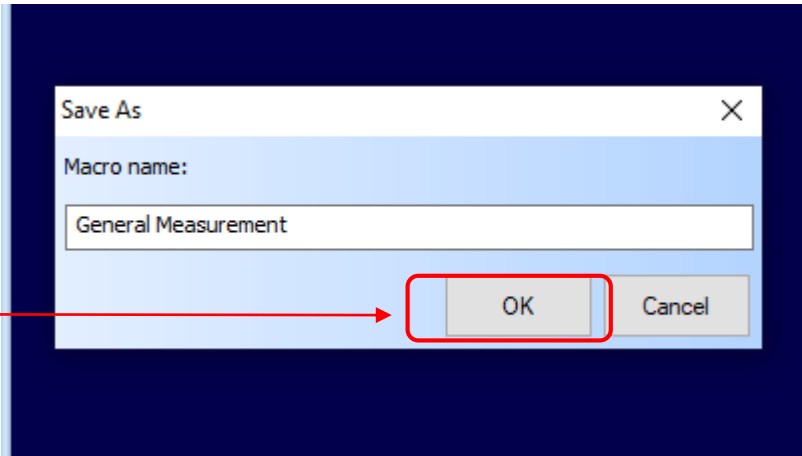
From the File menu select *Exit*

19b Exiting from MiniFlex Guidance continued. After selecting *File* → *Exit*, three message boxes will pop up .

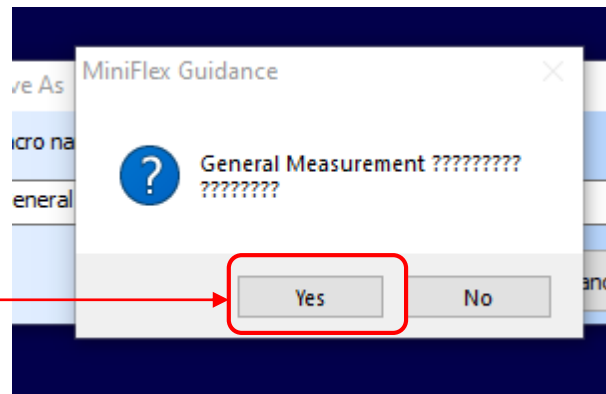
1. Click Yes



2. Click OK



3. Click Yes



20 Finishing up.

1. Remove your sample.
2. Turn off the diffractometer. Refer to the first slide.
3. Finish filling out the log sheet. Please enter total time as decimal hours.
4. Turn of the Haskris heat exchanger.
5. Log of the computer. **Do not Switch user, Sign out!**
6. And finally

- **PLEASE**
 - **No sample preparation** H
 - **No sample disposal**
 - **No sample storage**
 - **No solvents**

Using the Google Calendar to Reserve the Diffractometer

Step 1. Add the Resource Calendar to your Google Calendar.

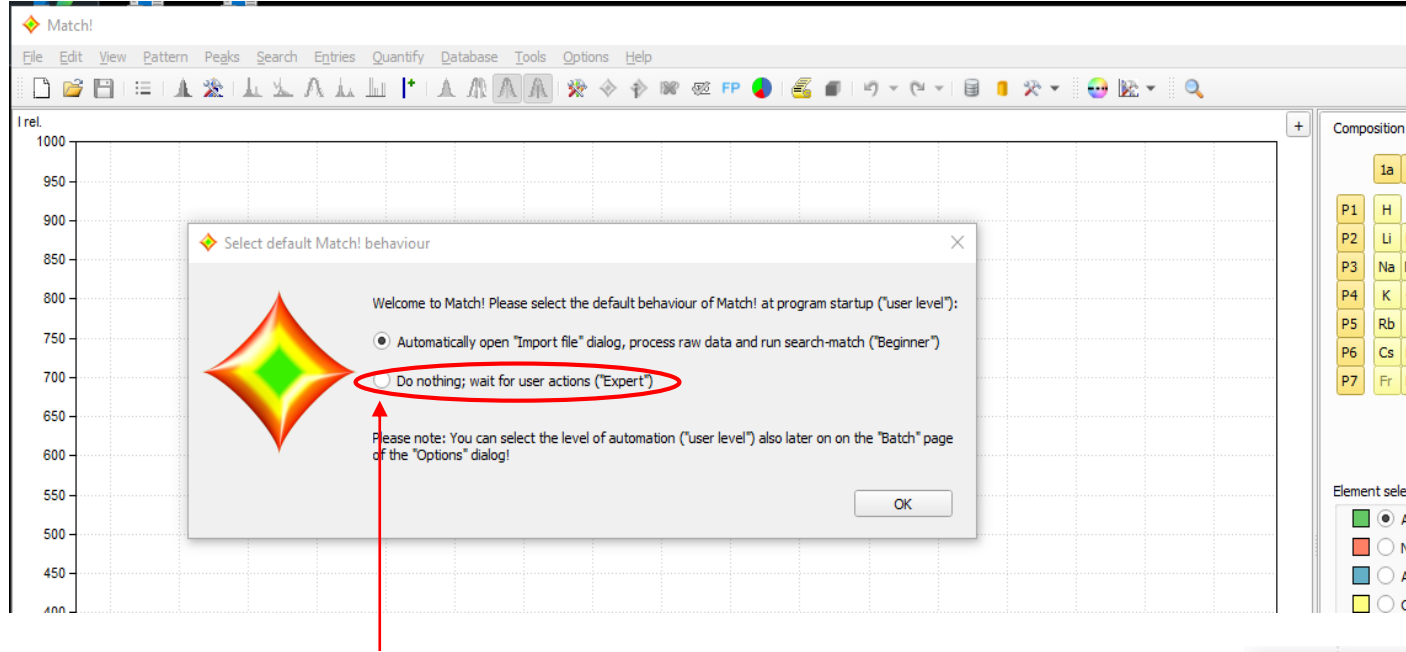
- Open your Google Calendar and scroll down the left side information until you come to "Other calendars". Click on the + symbol.
- Click on "Browse resources"
- Scroll down through the A-Z named resources until you come to "(Room)" and click on that.
- Check the box for ESS-GEO Room-245-25
- Return to your calendar
- You can make the ESS-GEO Room-245-25 calendar display on your calendar or not by giving it a distinct color and checking or unchecking the box next to it. When it is displayed, you should be able to see the details of existing reservations.

Step 2. Reserve the diffractometer

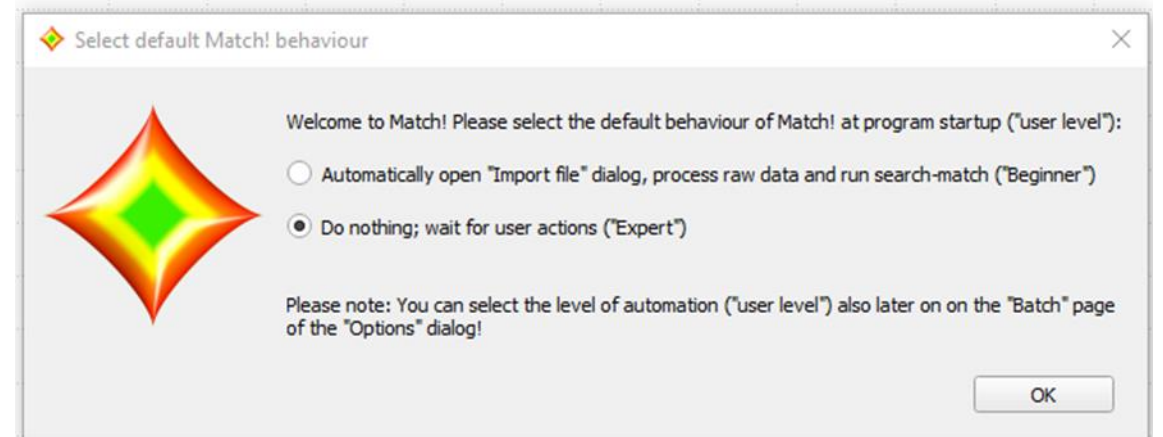
- Select a day/time in your Google Calendar giving it a title that includes 'XRD', your name and email address.
- Select the "Rooms" tab on the right hand side next to "Guests".
- In the search box below "Rooms", start to type "ESS-GEO" and you will see the resource calendar for ESS 245 show up in the results.
- Select the ESS-GEO Room-245 resource and save.

Using Match! to Reformat Diffraction Data

Step 1a. Start Match! by double clicking the desktop icon.



Step 1b. Click on *Do nothing; wait for user actions* ("Expert")



The Match! program is now loaded and ready.

The screenshot displays the Match! software interface. On the left, a plot shows relative intensity (I rel.) on the y-axis (ranging from 0 to 1000) versus 2theta on the x-axis (ranging from 10.00 to 170.00). The plot is currently empty, with a red vertical line at approximately 10.00 2theta. Below the plot, the text "Cu-Ka (1.541874 A)" is visible.

On the right side of the interface, there is a periodic table with columns labeled 1a through 8a. Below the table, there are controls for "Element selection by mouse":

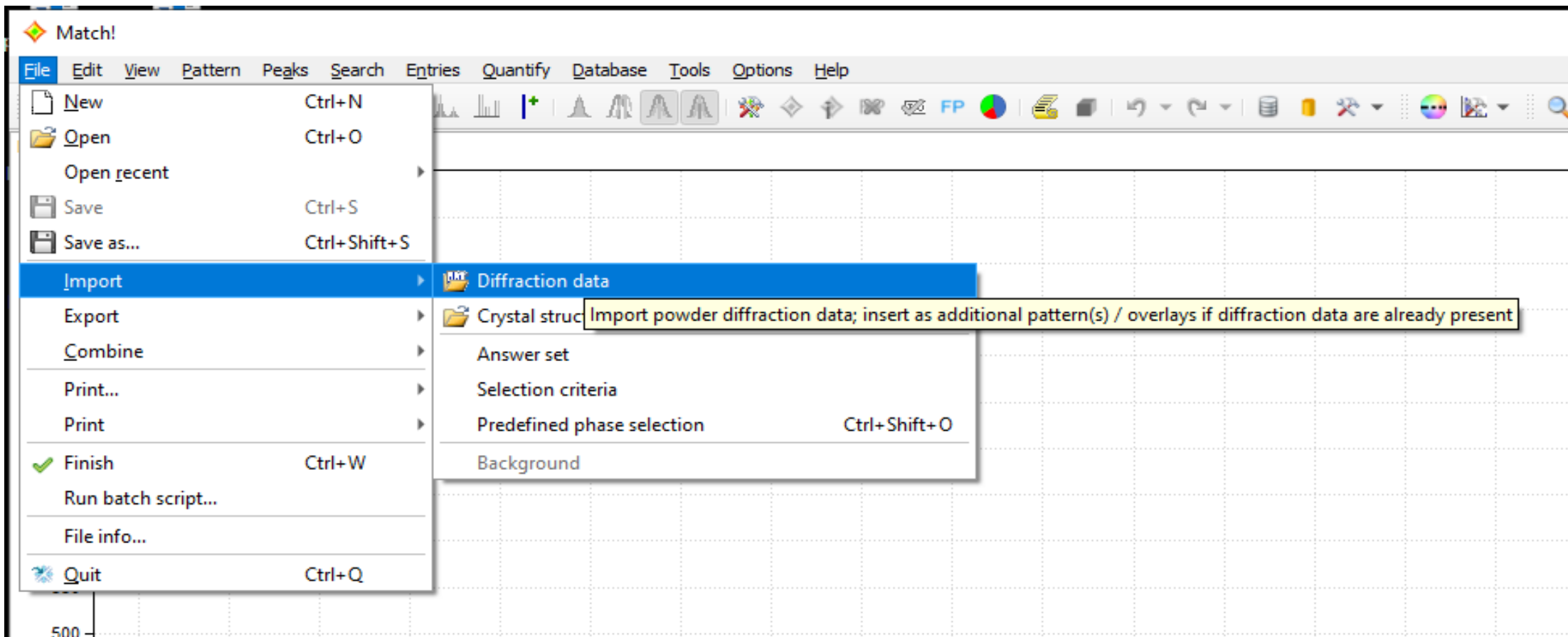
- All
- None
- Any
- Optional

There are also buttons for "Toggle" and "Reset". To the right of these controls are input fields for "Name:", "Elem. count:", and "Formula sum:", each with a small grid icon and a red 'X' icon. Below these fields is the text "More compound restraints on 'Subfiles' tab".

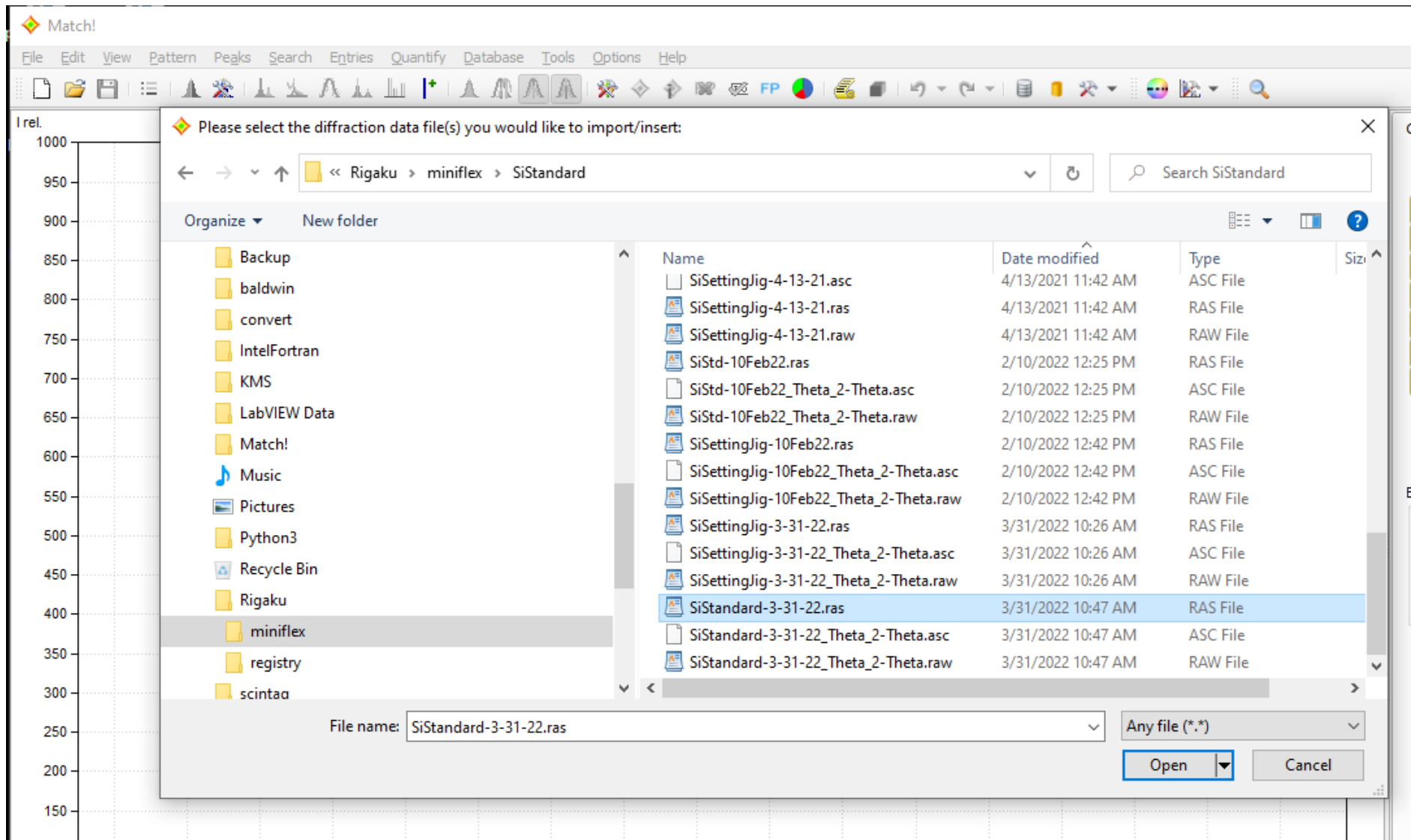
At the bottom of the right panel, there is a "Preset:" dropdown menu set to "None / new set", and buttons for "Save", "Delete", and "Reset". Below this is a "Restrains" section with buttons for "+ Add", "Peak list", "Data sheet", and "FP Refine".

The bottom of the interface features a status bar with the text: "Stony Brook University, Mineral Physics Institute, Site License" on the left, and "2th: 52.24 d: 1.7510 I rel.: 958.68 78155 entries COD-Inorg 2022.06.29" on the right.

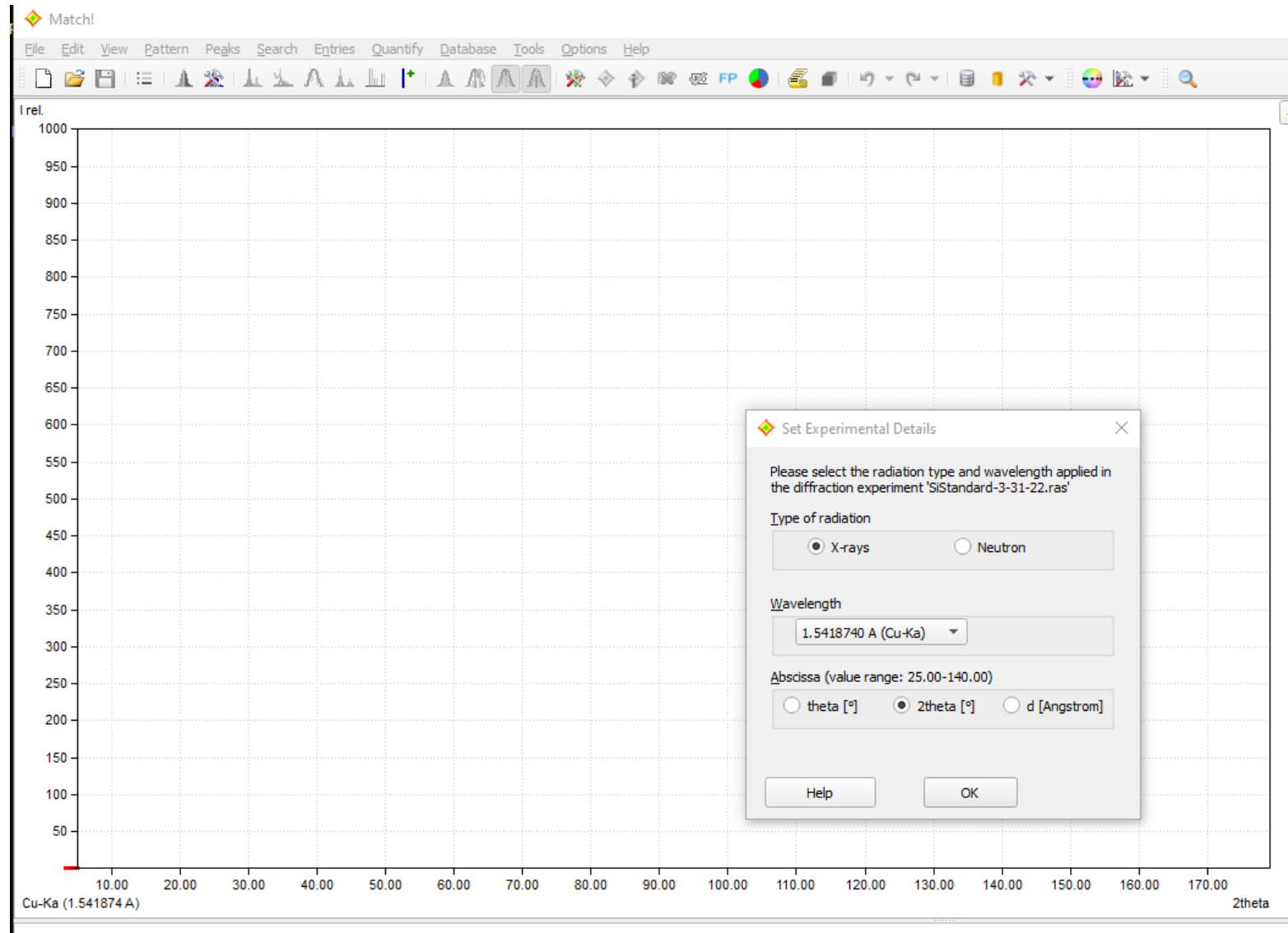
Step 2. Select *File* → *Import* → *Diffraction data*



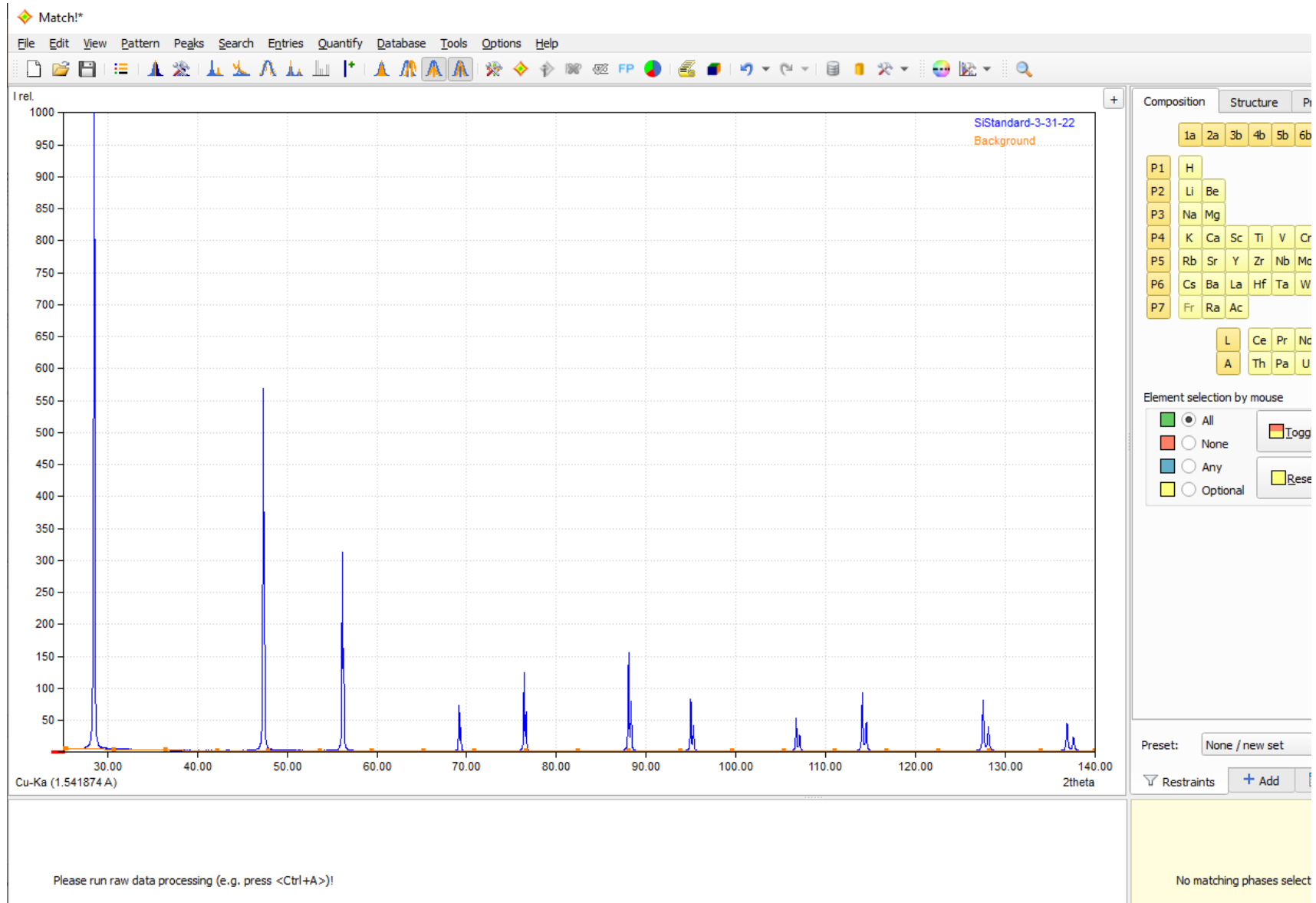
Step 2b. Select your datafile. Choose the file with the extension *ras*.



Step 2c When the window *Set Experimental Details* appears, click *OK*



Your diffraction pattern will be displayed.



Step 3a. Select File → Export → Profile data

The screenshot shows the Match! software interface. The 'File' menu is open, and the 'Export' option is selected, which has opened a sub-menu. In this sub-menu, 'Profile data' is highlighted, and a tooltip is displayed over it with the text: 'Export profile data of the current experimental pattern to a file'. The main window displays a diffraction pattern plot with intensity on the y-axis (0-500) and 2theta on the x-axis (30.00-140.00). The plot shows a blue line representing the experimental pattern and an orange line representing the background. The x-axis is labeled '2theta' and the y-axis is labeled 'Cu-Ka (1.541874 Å)'. On the right side, there is a 'Compositi' panel with buttons for P1 through P7 and a 'Elements' panel with color-coded boxes for H, L, N, K, R, C, and F.

Step 3b. For *Save as type* Select *Profile(2 columns: 2theta intensity) (*.dat)*
If need be, you can change the extension *dat* to something else.

The screenshot shows the Match! software interface. On the left, an XRD pattern is displayed with relative intensity (I rel.) on the y-axis (0 to 1000) and 2θ on the x-axis (0 to 100). A sharp peak is visible at approximately 2θ = 20°. The main window is titled 'Match!*' and contains a menu bar (File, Edit, View, Pattern, Peaks, Search, Entries, Quantify, Database, Tools, Options, Help) and a toolbar with various icons.

An 'Export current profile data as...' dialog box is open, showing the file path: $\ll Rigaku \gg \text{miniflex} \gg \text{SiStandard}$. The file name is 'SiStandard-3-31-22.dat', with the extension '.dat' circled in red. The 'Save as type' dropdown menu is open, showing the following options:

- Profile (start,step,end,intensities) (*.dat)
- Profile (start,step,end,intensities) (*.dat)
- Profile (2 columns: d-value intensity) (*.dat)
- Profile (2 columns: 2theta intensity) (*.dat)

The 'Profile (2 columns: 2theta intensity) (*.dat)' option is selected. On the right side of the software, there is a 'Composition' panel with a periodic table and 'Element selection by mouse' options.

Step 3b Continued Click Save

The screenshot shows the Match! software interface with an export dialog box open. The dialog box is titled "Export current profile data as..." and is currently displaying the contents of the "SiStandard" folder. The file name is set to "SiStandard-3-31-22.dat" and the save type is "Profile (2 columns: 2theta intensity) (*.dat)". The "Save" button is circled in red, indicating the next step in the process.

Match!*
File Edit View Pattern Peaks Search Entries Quantify Database Tools Options Help

Export current profile data as...
« Rigaku » miniflex » SiStandard
Search SiStandard

Organize New folder

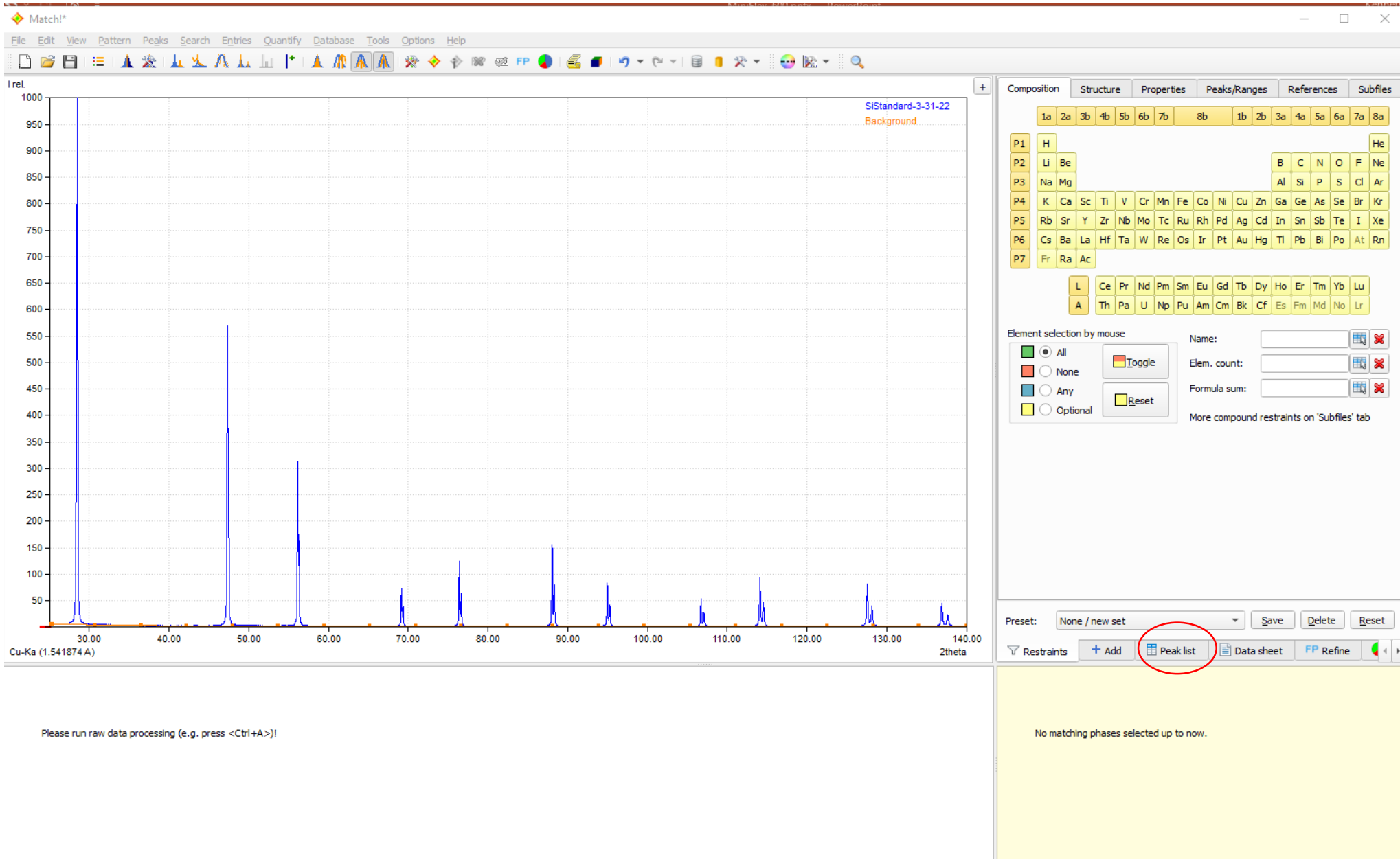
Name	Date modified	Type	Size
No items match your search.			

File name: SiStandard-3-31-22.dat
Save as type: Profile (2 columns: 2theta intensity) (*.dat)

Hide Folders

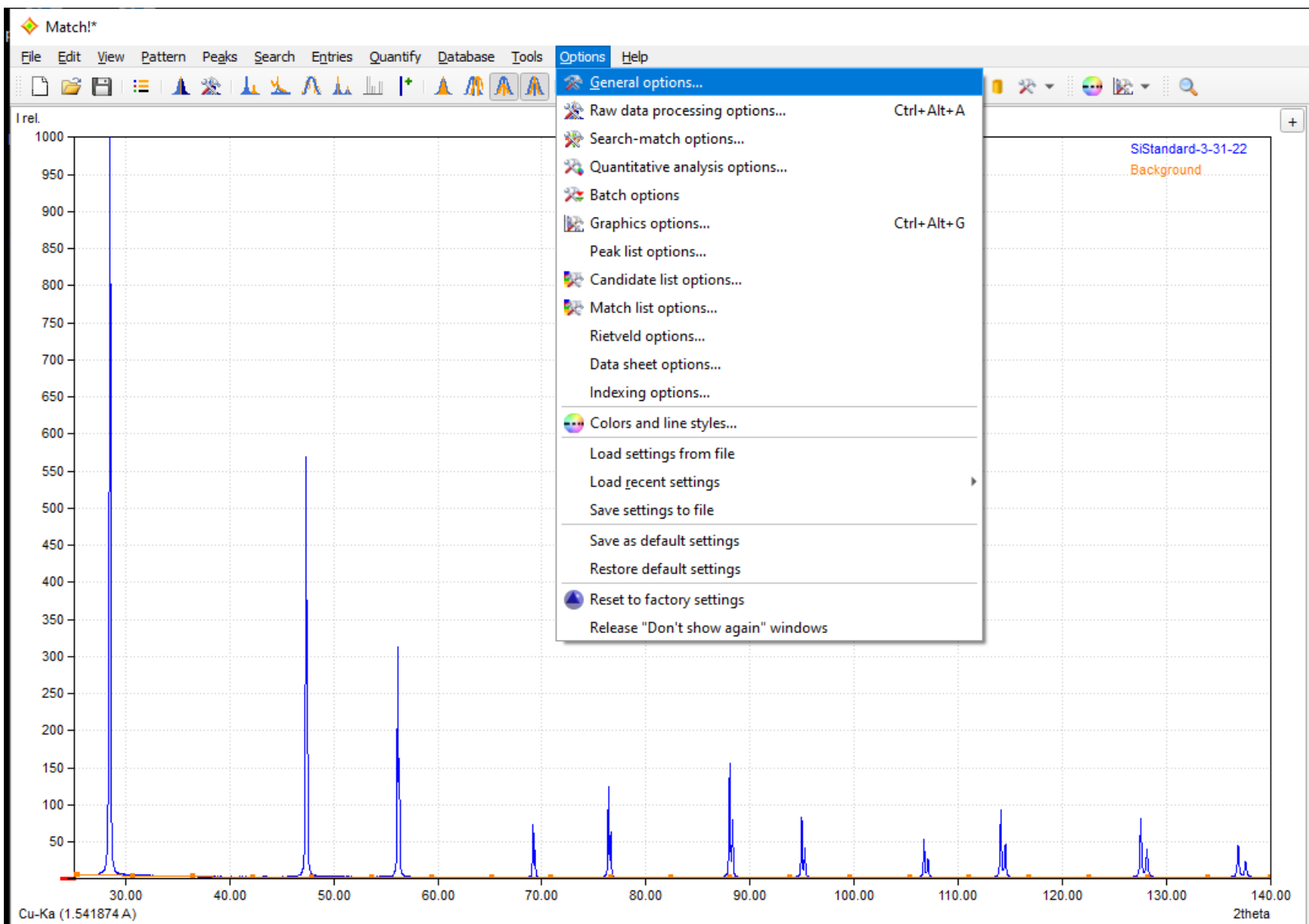
Save Cancel

Match! Continued: Preliminary data analysis

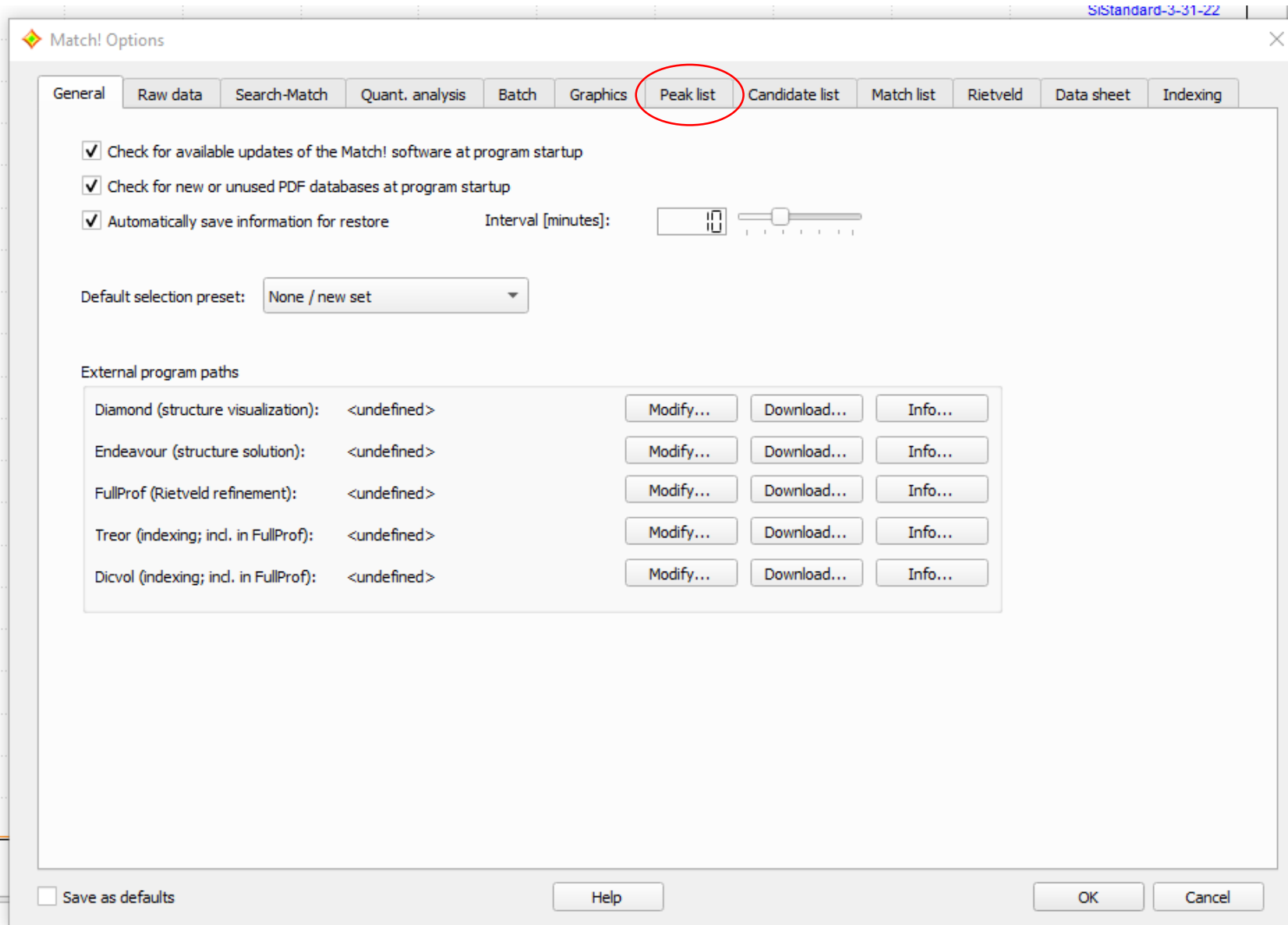


1. Click *Peak List*

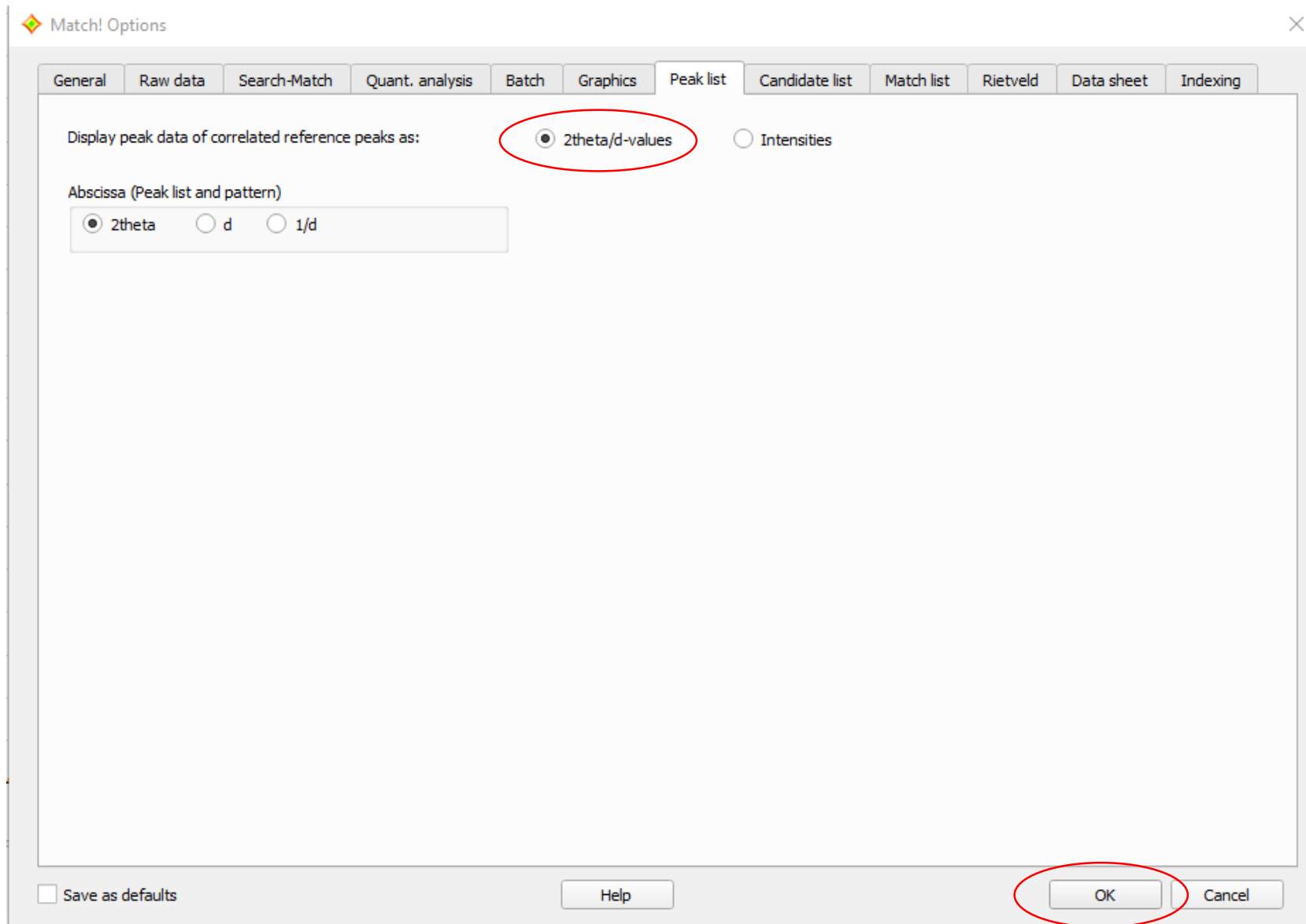
2. Click *Options* → *General options*



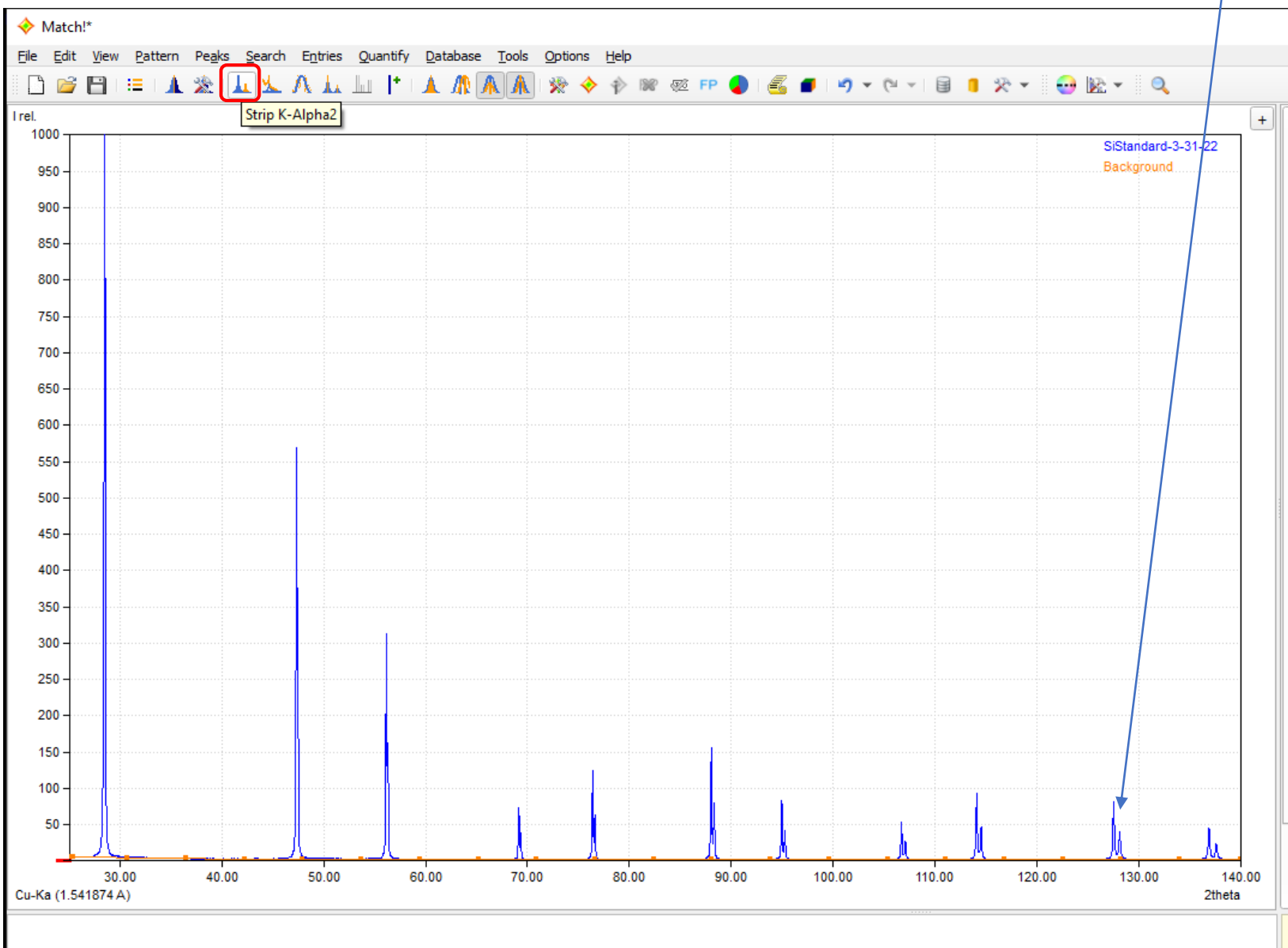
3. Click *Peak list*



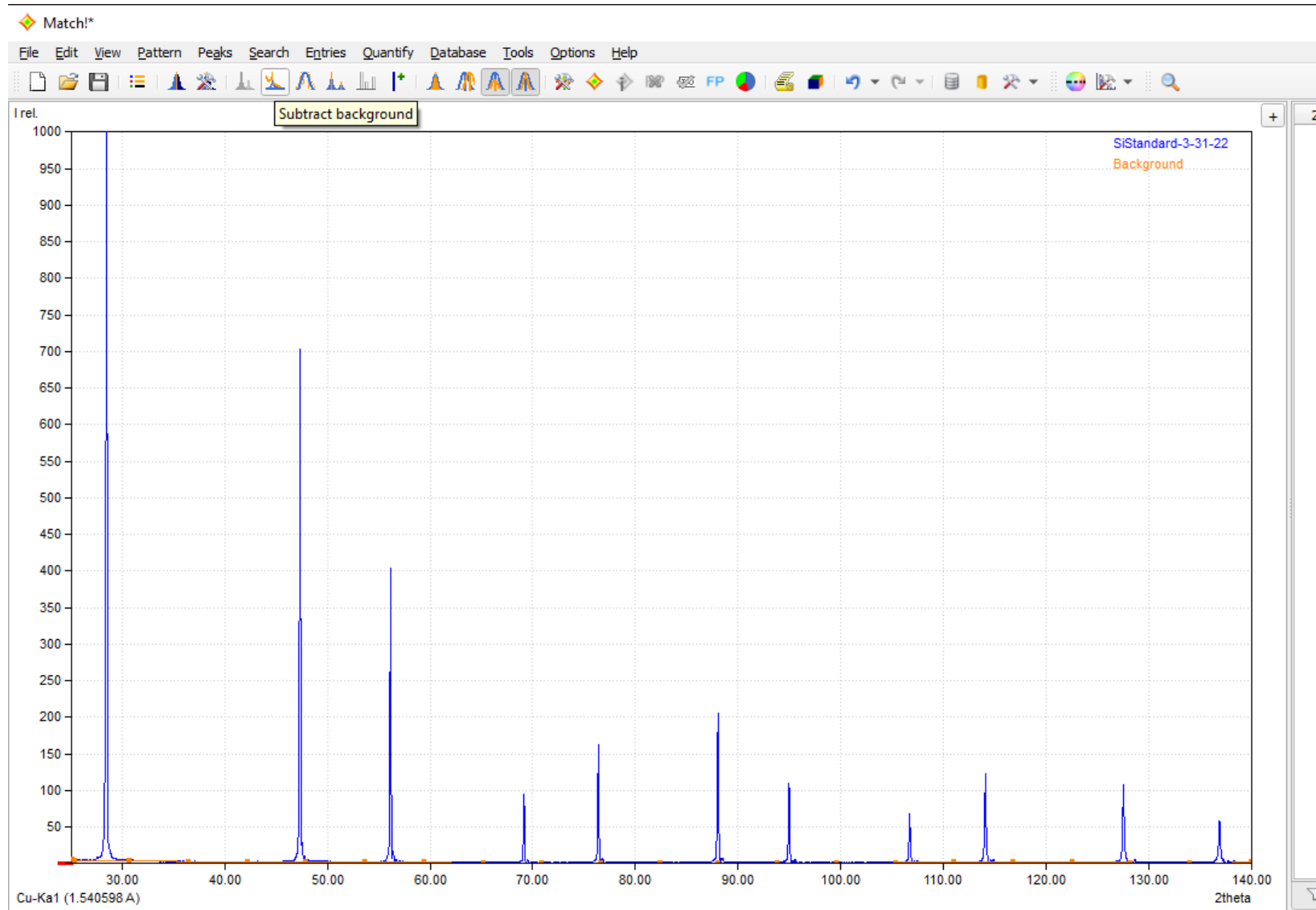
4. Select $2\theta/d$ -values then click *OK*



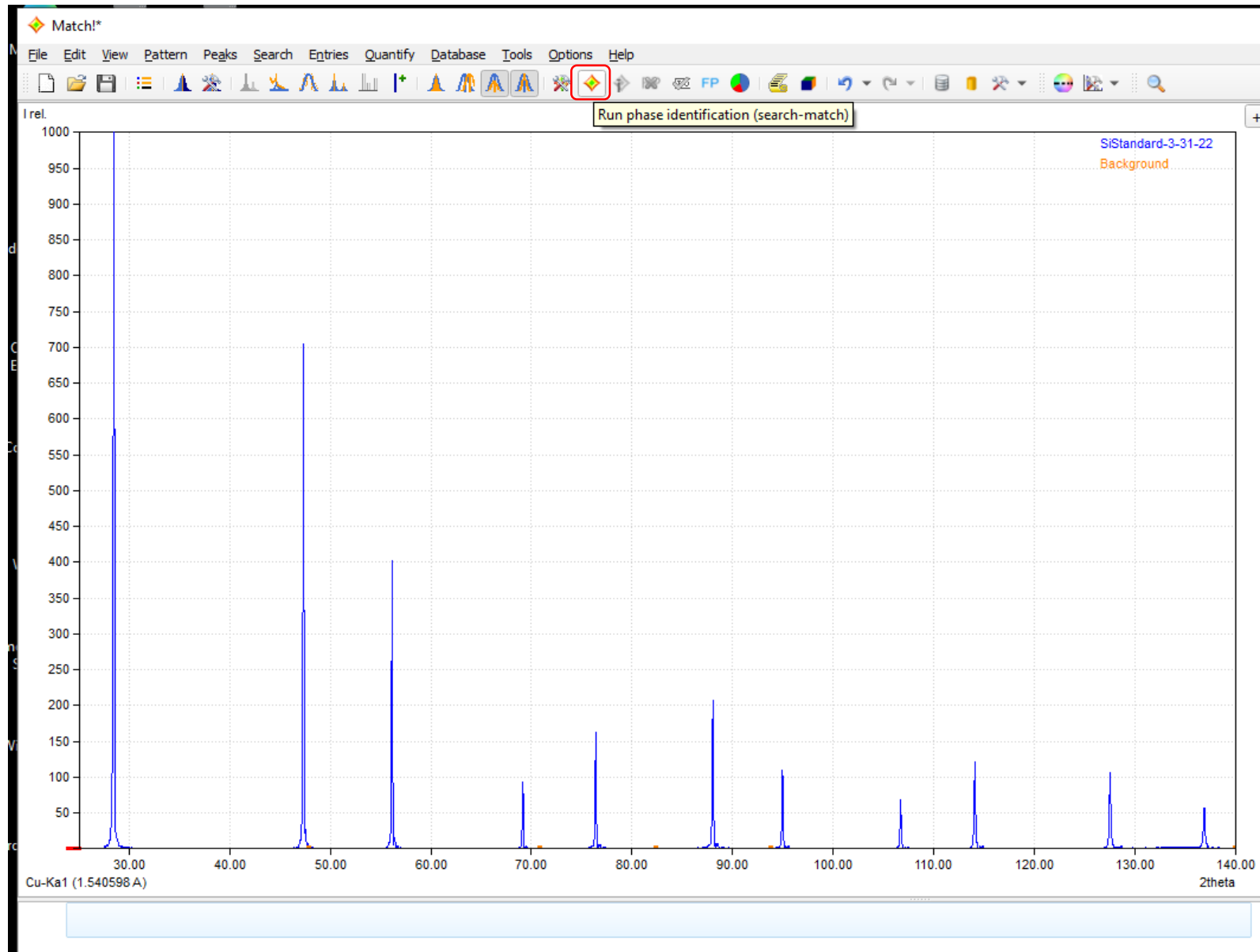
5. Click Icon for *Strip K-Alpha2*. Notice changes in the diffraction pattern. Like here.



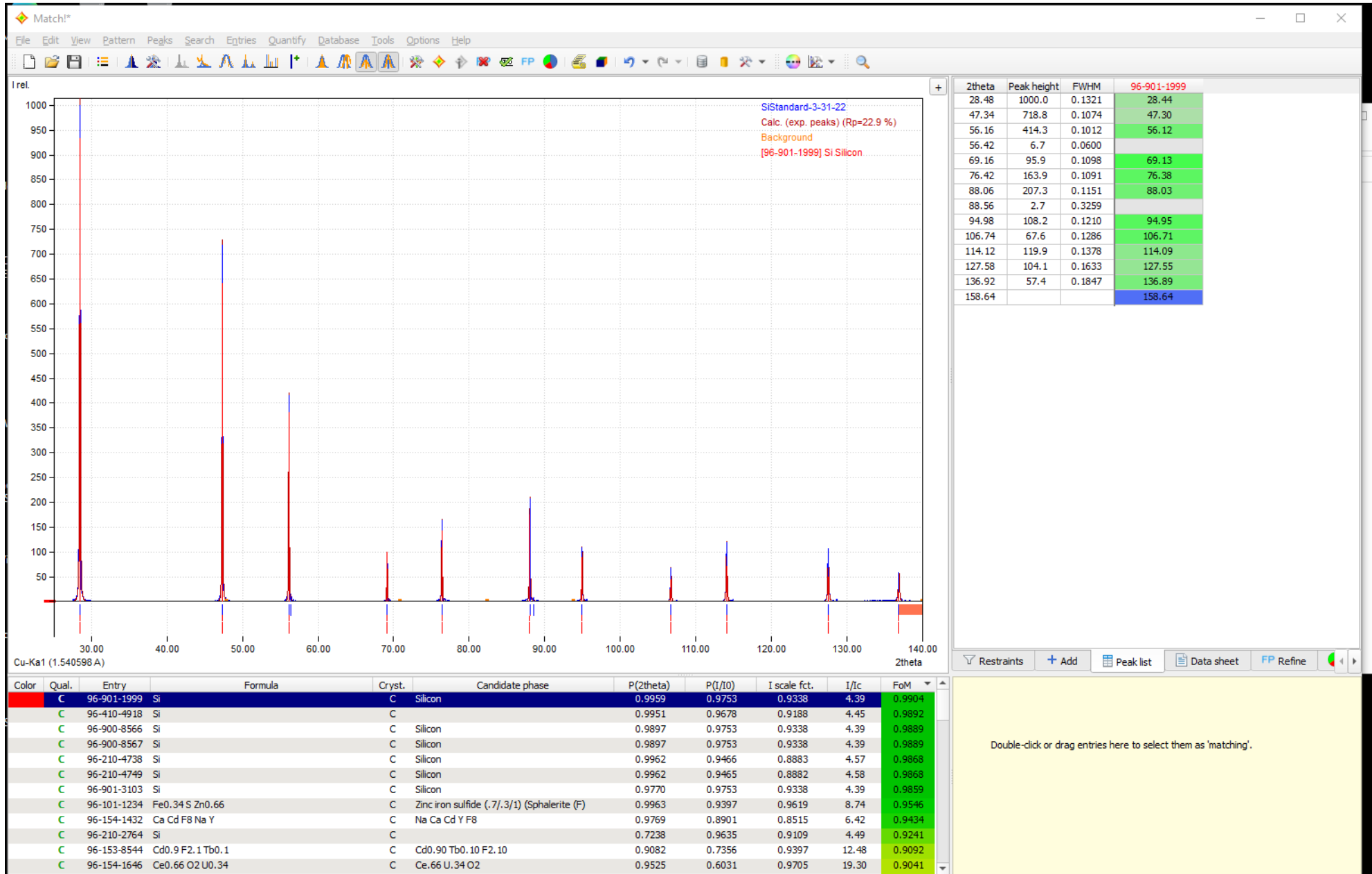
6. Click *Subtract background* and look for changes in pattern again



7. Click on Icon for *Run phase identification (search-match)*

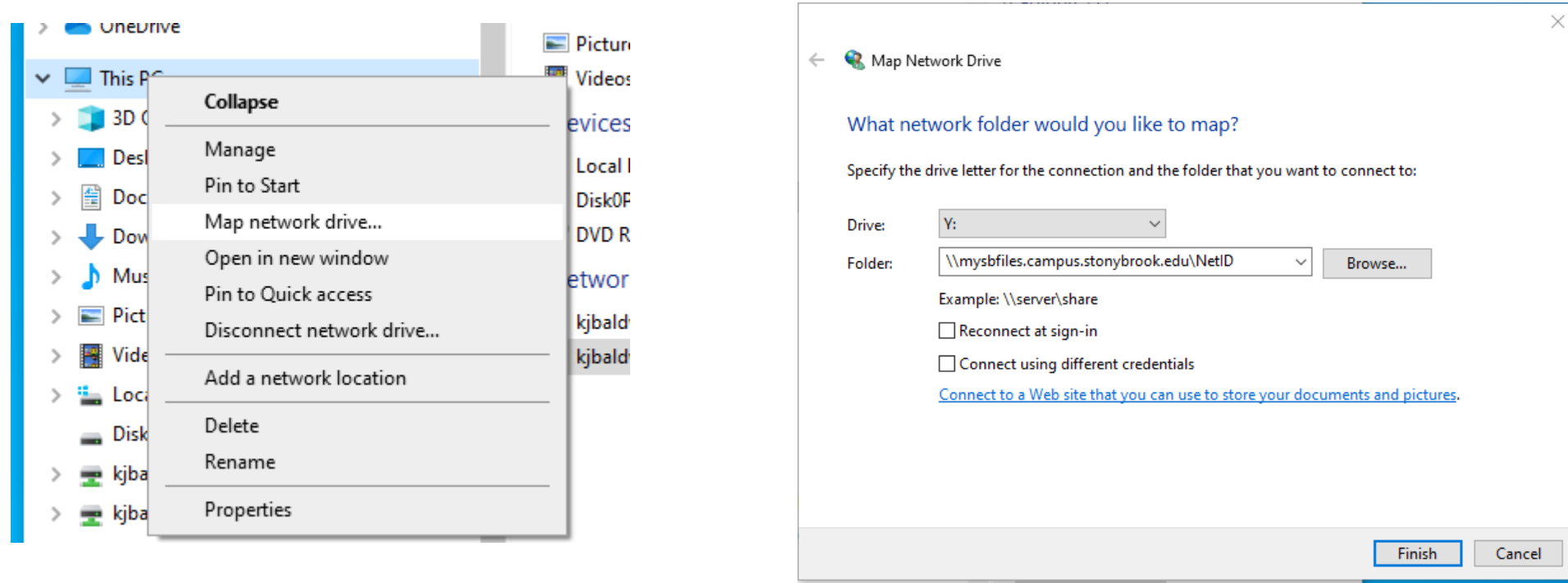


8. Observe Peak list



Manually mapping to your 'mysbfiles'

1. Open File Explorer and right click on *This PC* and select *Map network drive...*



2. Select a *Drive letter* and the *Folder*: \\mysbfiles.campus.stonybrook.edu\NetID
Be sure to replace NetID with your actual NetID.
Click *Finish*