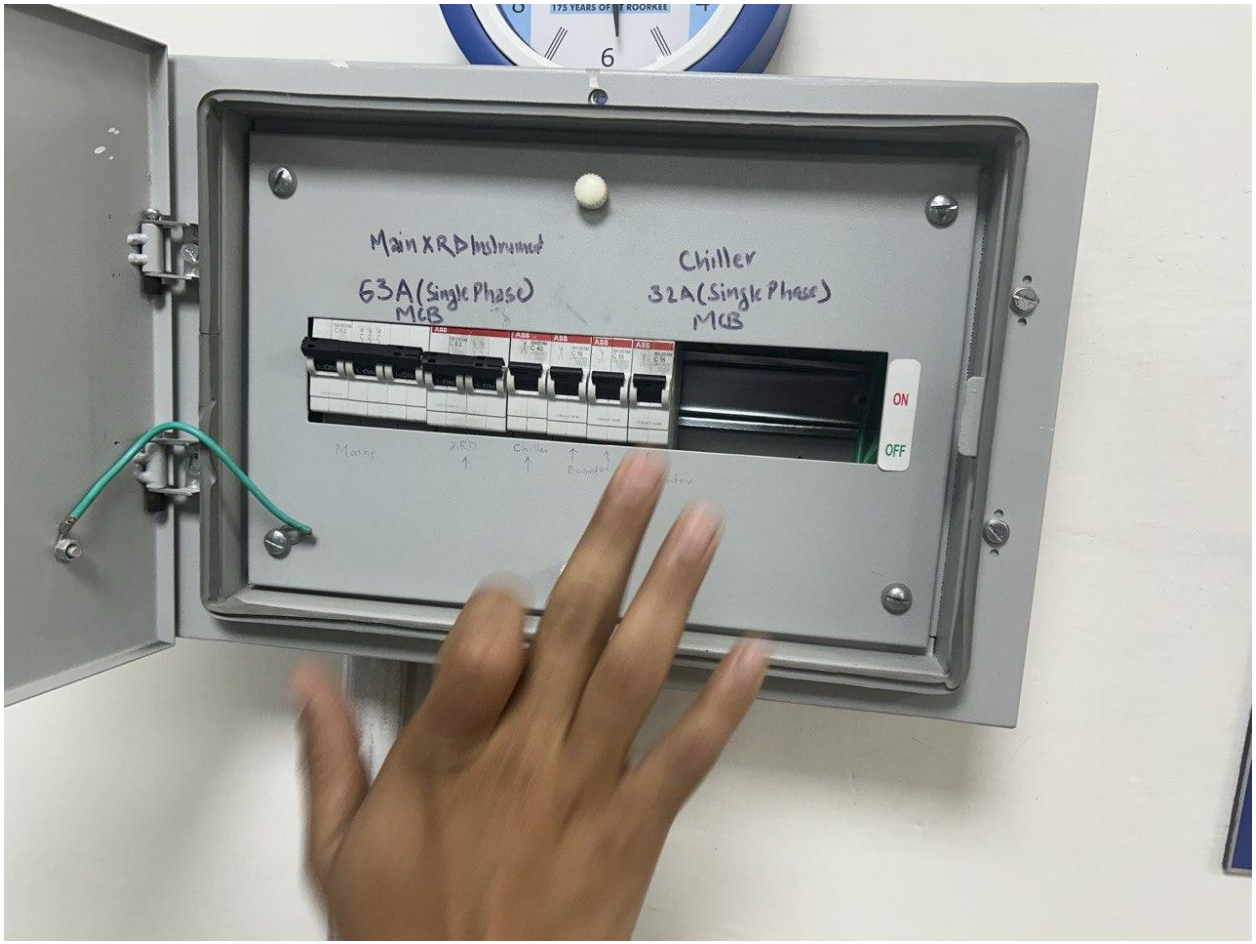
XRD machine

Turning on machine

Step 1: Switch on the AC main switch in the UPS room

Step 2: switch on the main power of the compressor from the wall and wait 4min for pressure generation

Step 3: switch on the main power in the MCB for the XRD machine and every switch



Step 4: press the power button on the machine to turn on the light press the button at the end of the row

Note: you can observe a light signifying the machine being turned on

Step 4: wait until KV and MA show 0 0 respectively, the XRD wings become horizontal

Step 5: Switch on the main switch on the MCB for the chiller by moving the nozzle clockwise.

Note: Make sure the water level is in between the range.

Step 7: Wait until the temperature reaches between 21 and 22 in the machine display (wait 1 min for stability of the value).

Step 8: Rotate the XRD machine key 90º clockwise

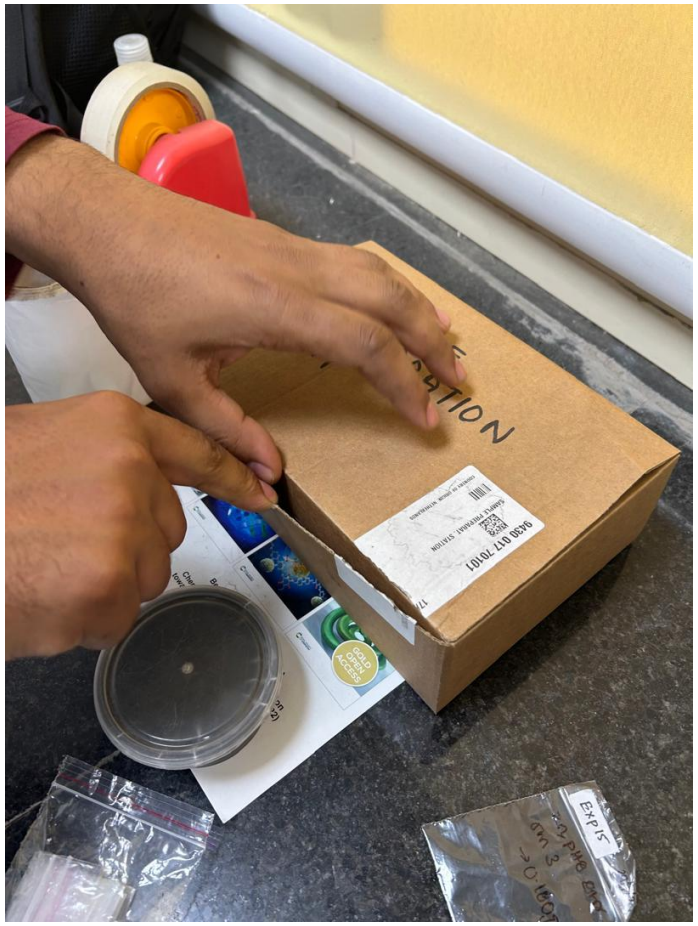
Note: Before turning on the PC and working on the software, make sure the XRD machine displays the KV and mA are 30 and 10 respectively.


WARNING
WARNING
HT ENABLED
POWER OFF
POWER ON
MEASURING
SERVICE MODE
X-RAYS ON
kV
mA
LIGHT ON
30
10
OFF
ON
UNLOCK DOORS
ON/OFF

Sample preparation

Step 7: Sample preparation and sample placement

Step one: check for all materials in the box i.e. sample holder, support board, spatula, brush, paper, and glass. And clean all using Aceton or an available cleaning agent, if you have to change the paper you can change it as well from the drawer at the back of the machine

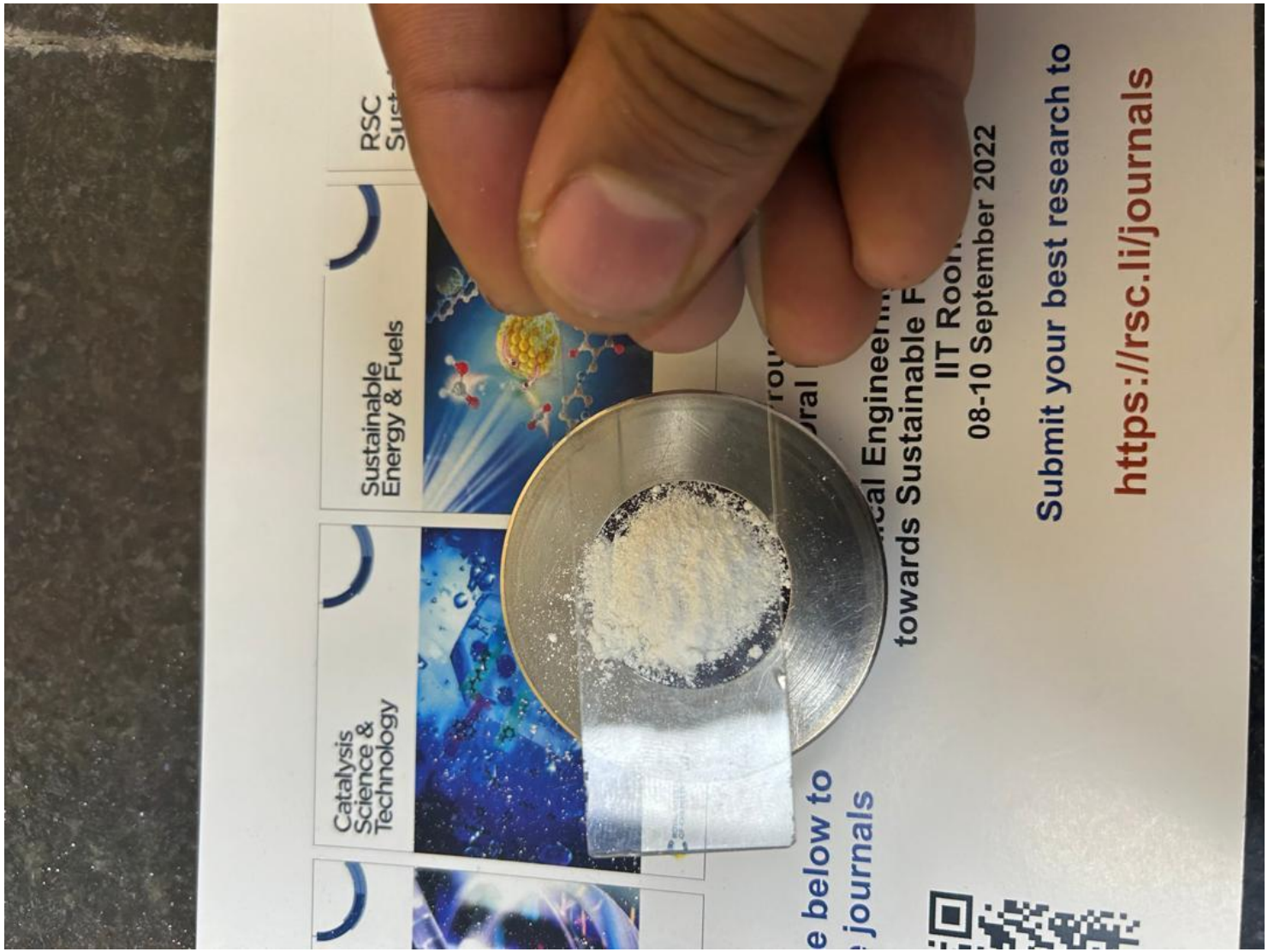


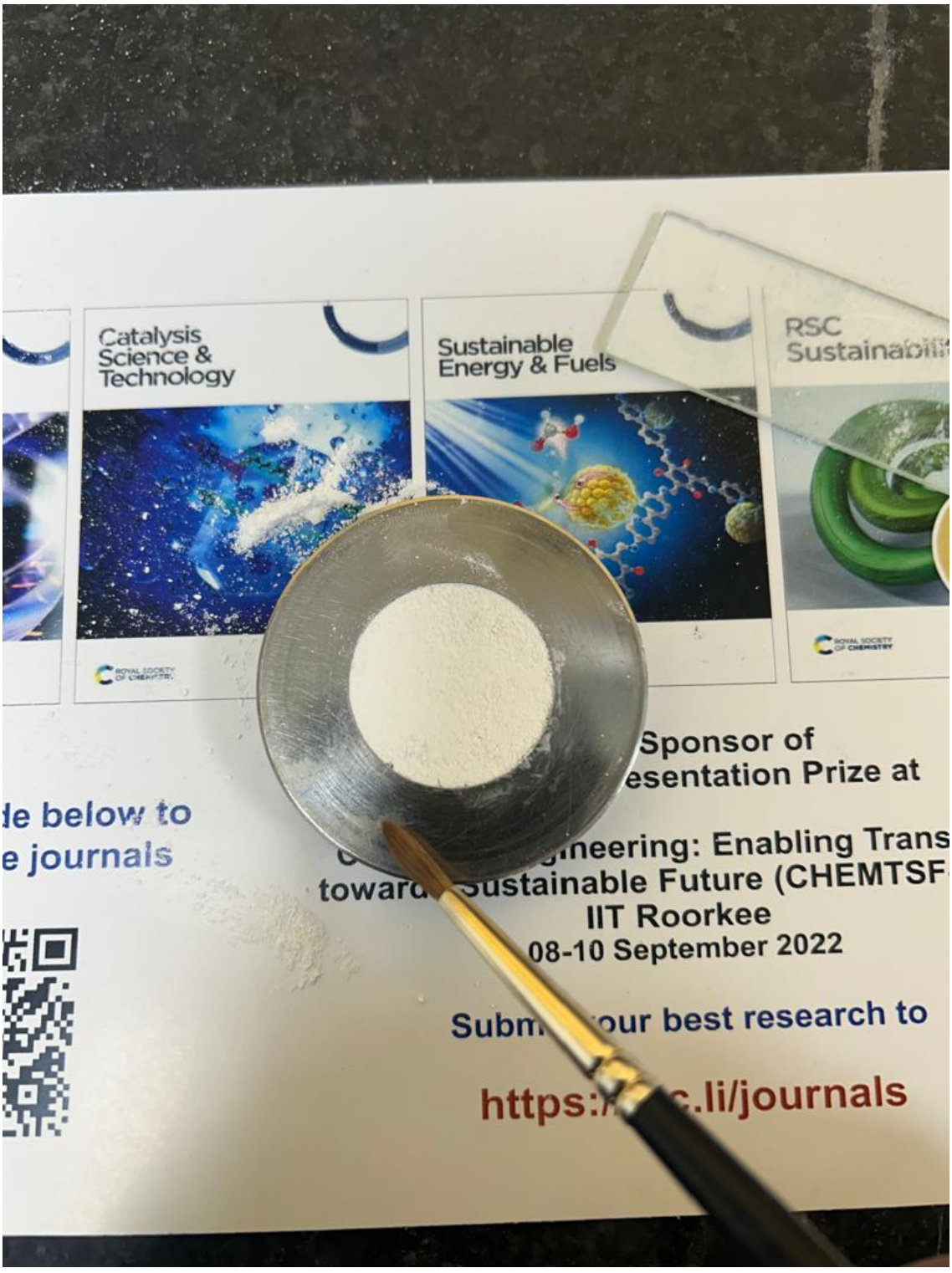
paper finding area

Step two : Put the card board on the bottom metal support and close it with the metal closure

Step three : put your fine-powered sample in the middle portion of the sampe holder

Step four: Smooth your sample using glass road in a uniform way and remove excess powder beside the sample container on the side metal using a brush.




Catalysis Science & Technology
Sustainable Energy & Fuels
RSC Sustainabili
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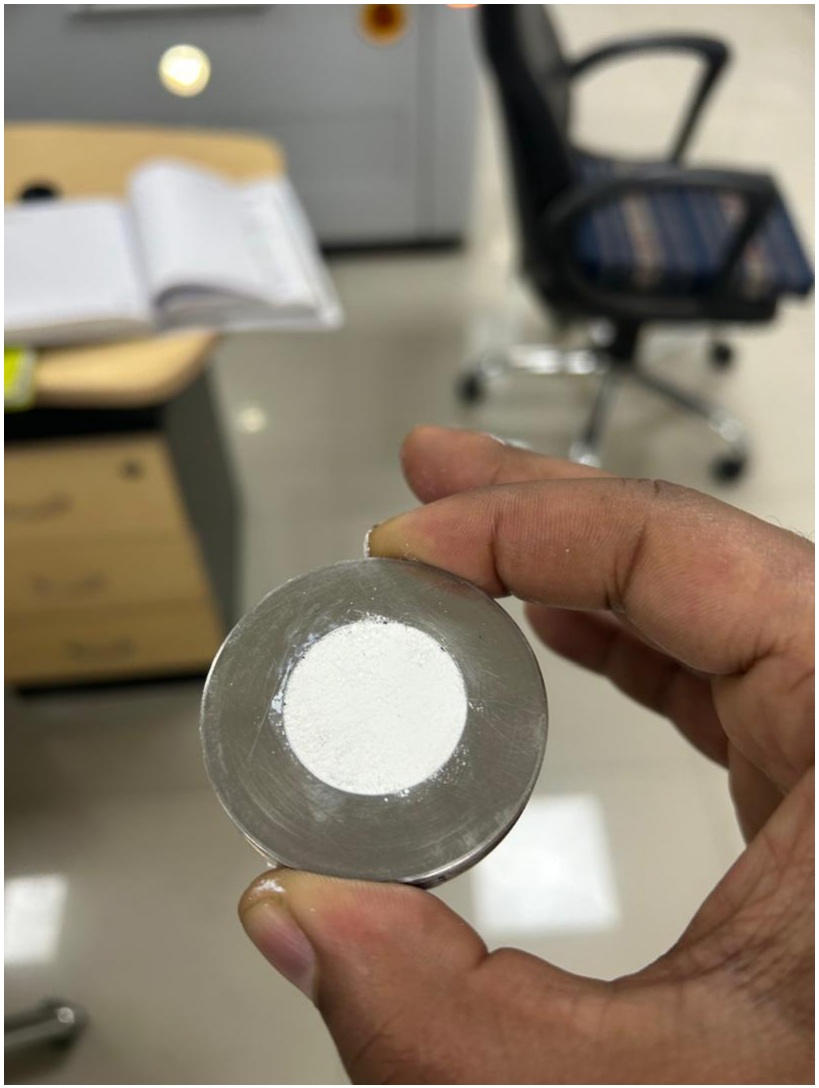
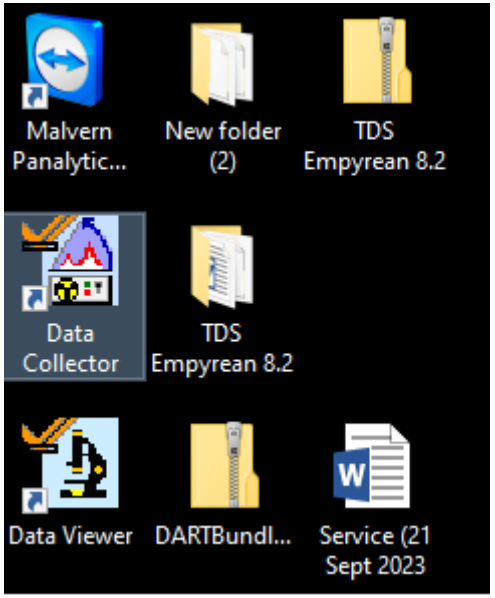
gineering: Enabling Trans
toward Sustainable Future (CHEMTSF
IIT Roorkee
08-10 September 2022
Subm our best research to
https: c.li/journals

Step five: Hold it using two fingers like shown in the picture and clean the bottom part of the sample holder for trace powder remains.

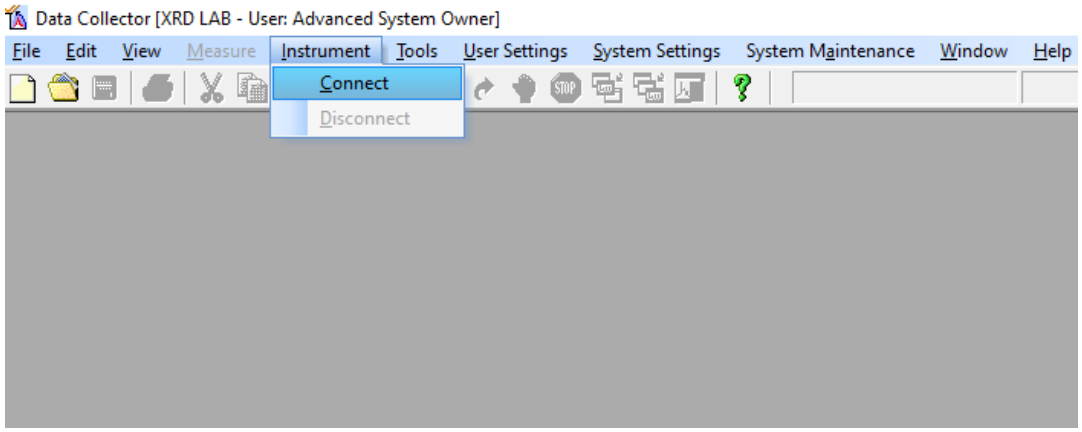


Software part

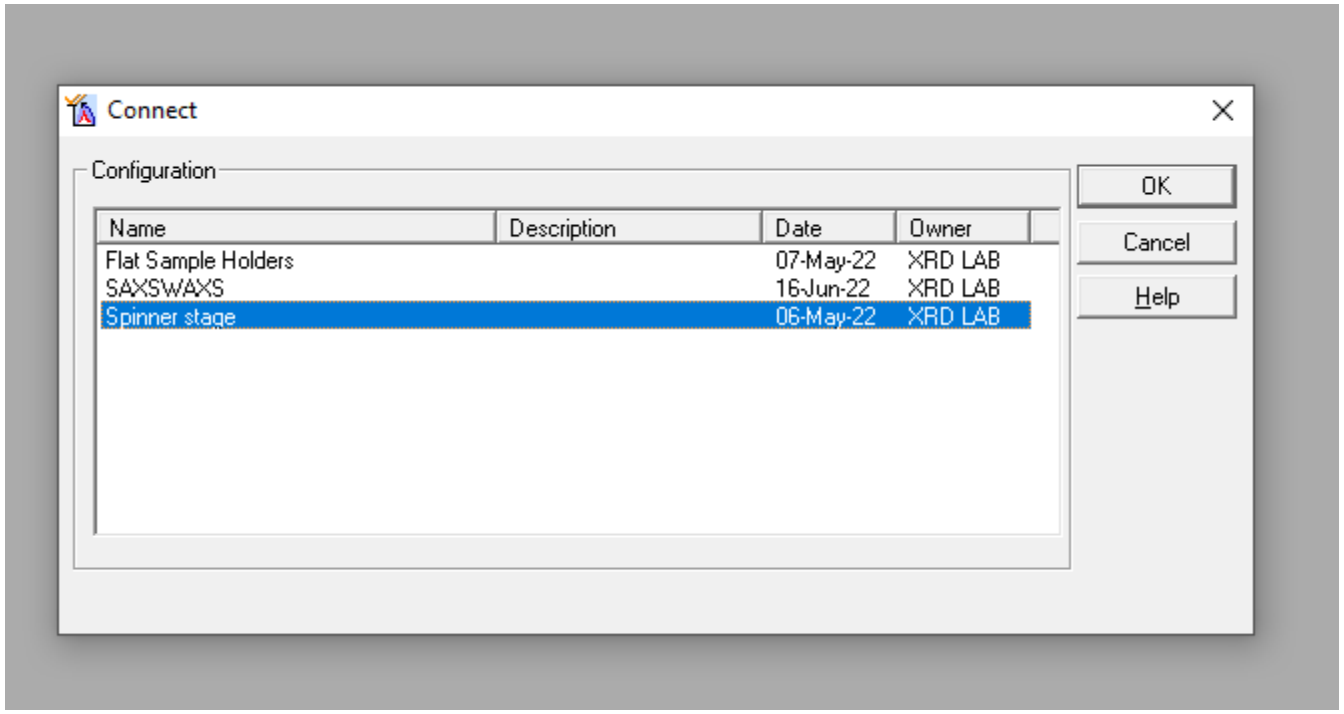
Step 1: Turn on pc then open the software by the name Data Collector PC password is : **FIST**



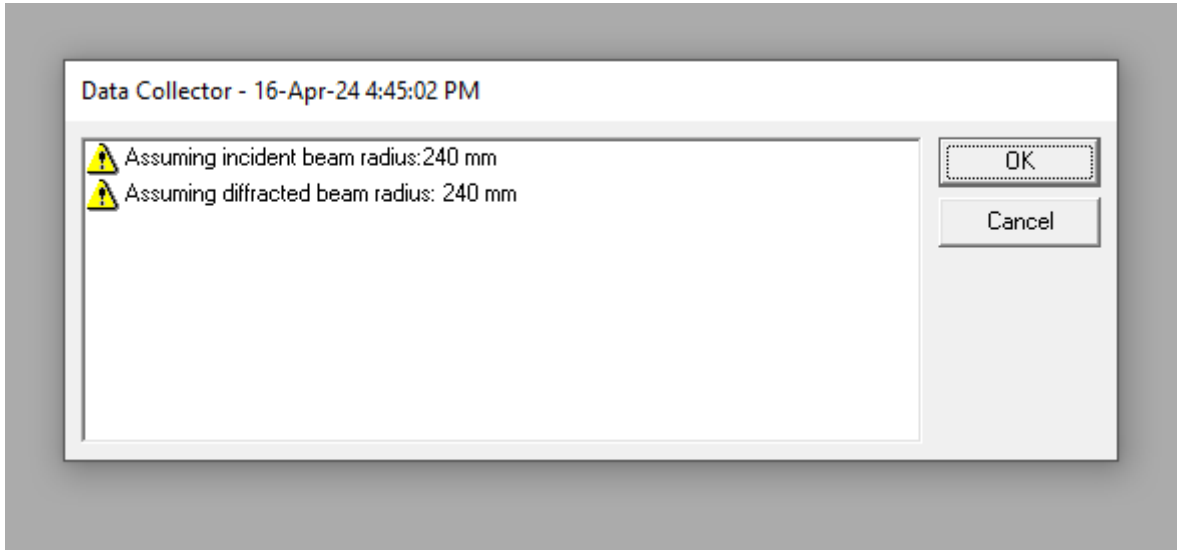
Step 2: On the opening window header part click on Instrument and click on connect to connect with the machine



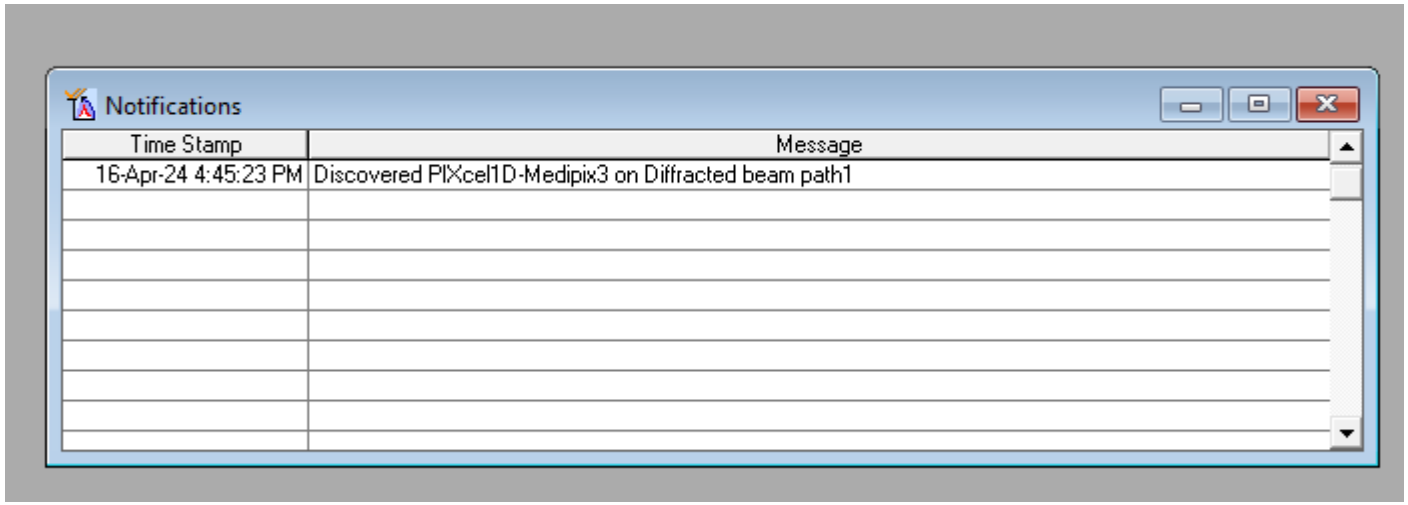
Step 3: In the stage select spinner for the showing window after clicking ok



Step 4: On the next window you have to do nothing so just click ok

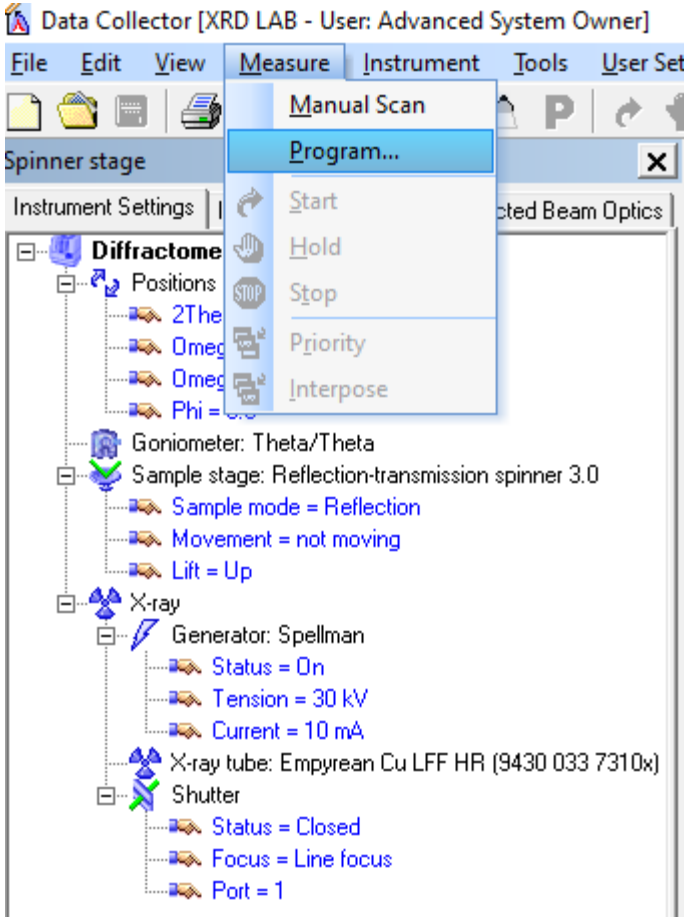


Step 5: Close the auto opened small window

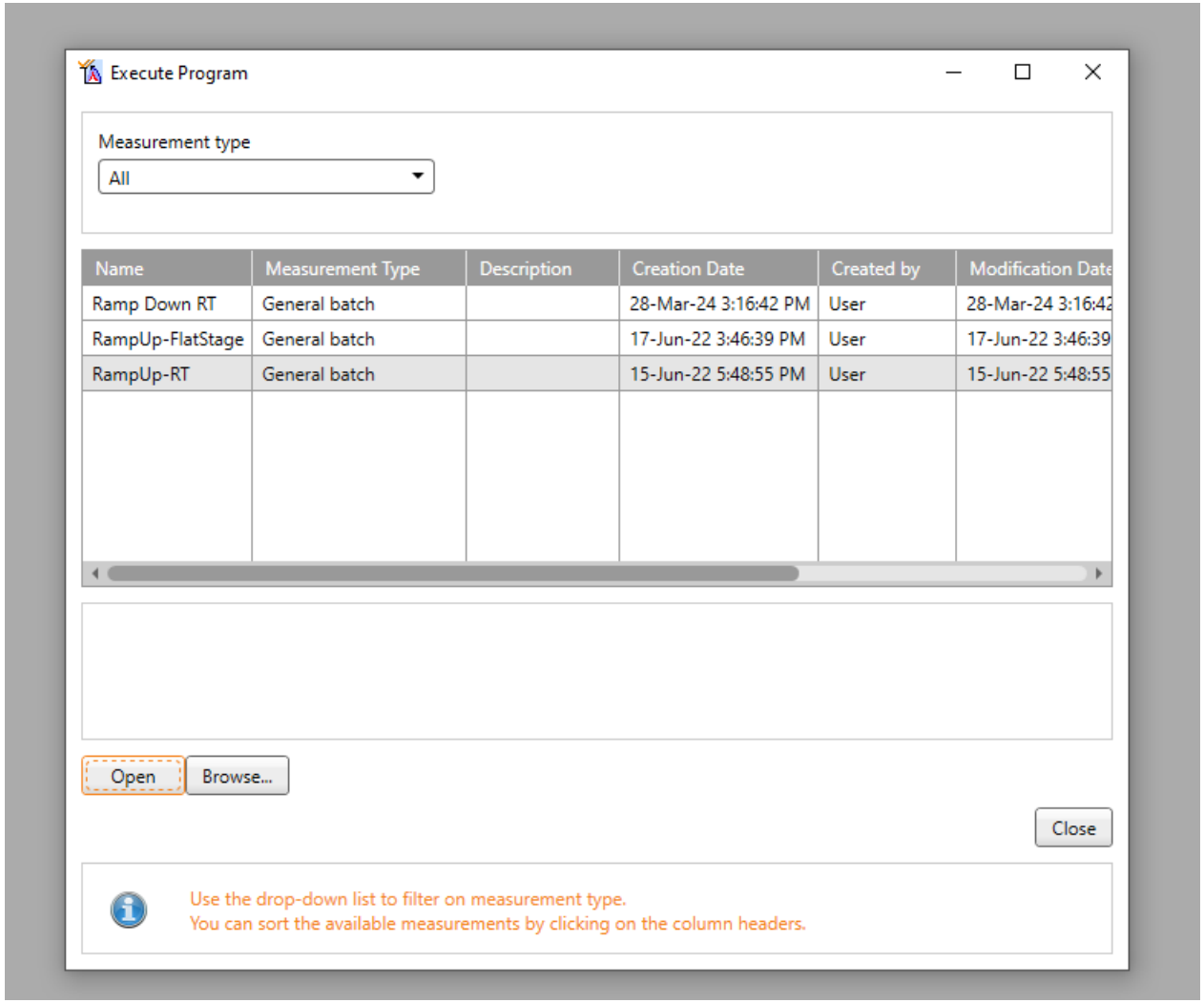


Step 6: Before the operation, we have to maintain KV and mA 45 and 40 respectively so wait till the machine displays so to do that we follow the following step.

Step one : Click on measure at header part and select program

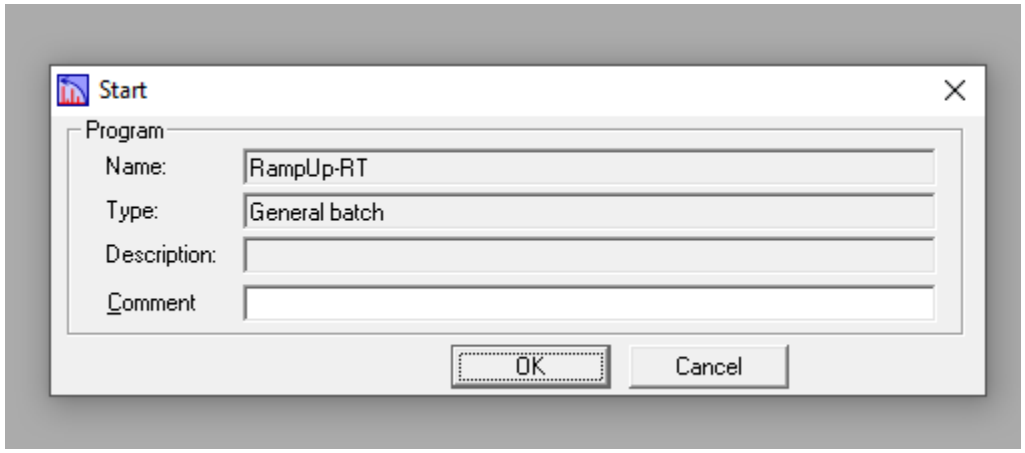


Step two: In the coming window select RampUp-RT then click on open

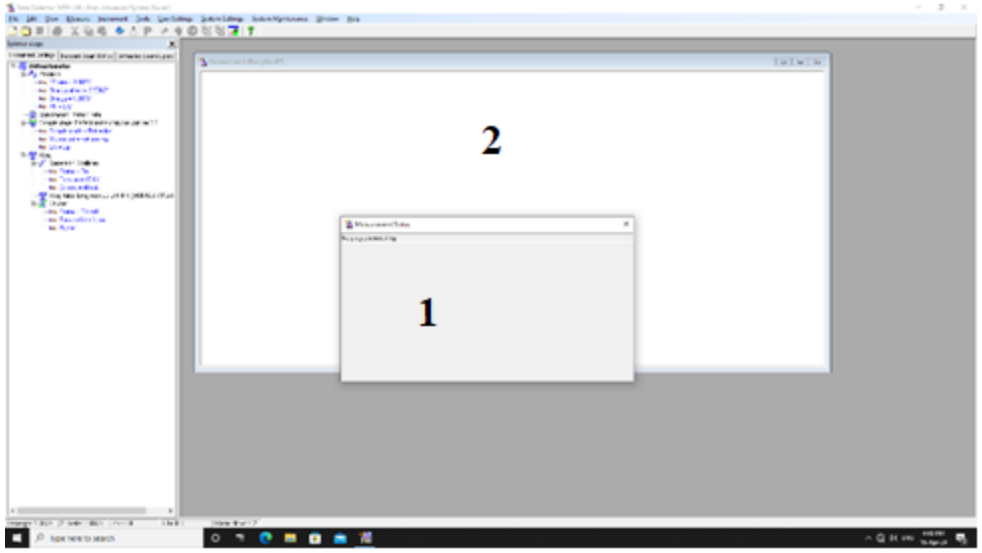


Step three : Click Ok on the coming window

Note : you will see the increase the KV and mA increasing to 45 and 40

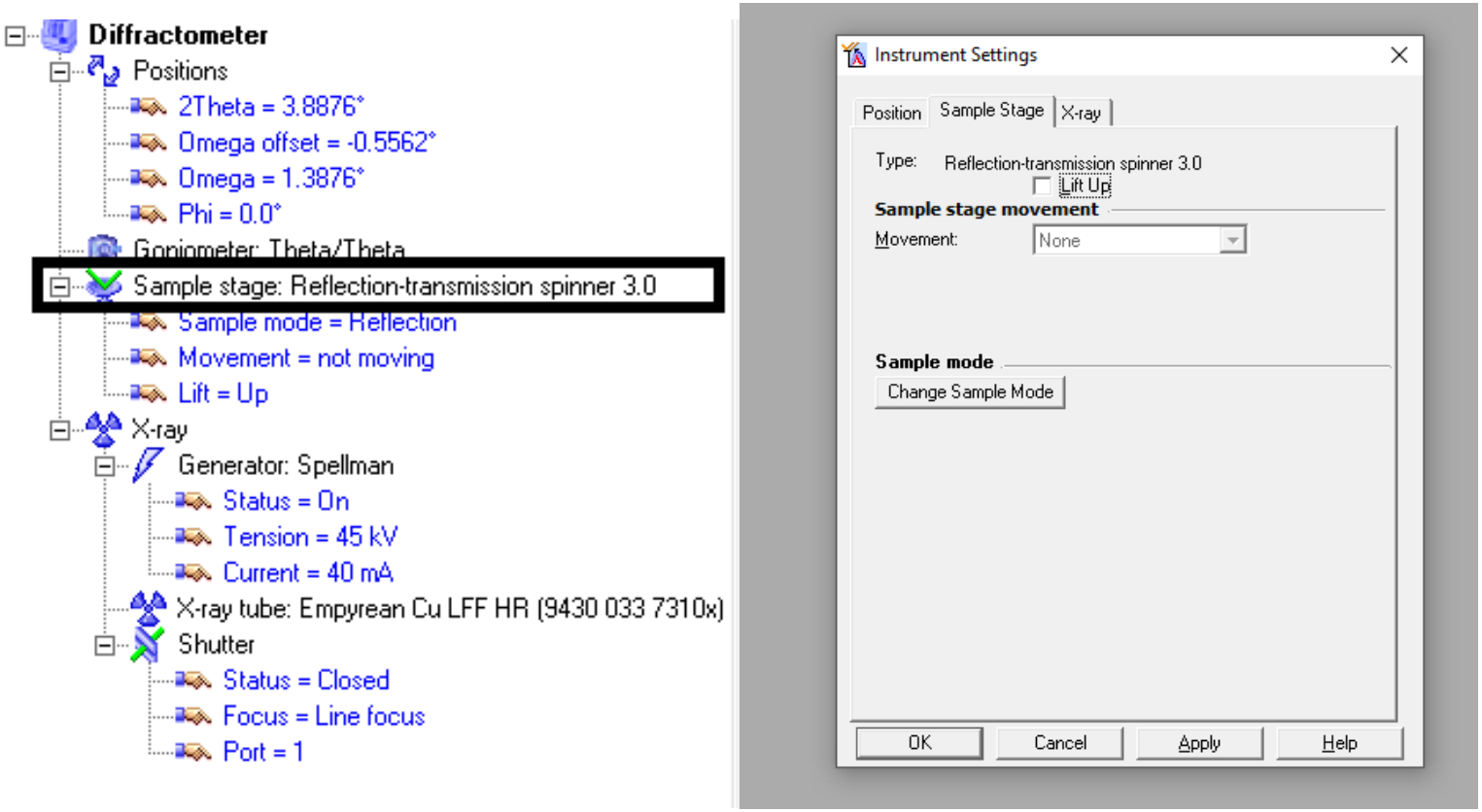


Step four : After it reaches 40 and 45 you can close both rump-up windows (which are displayed as one and two in the picture below)



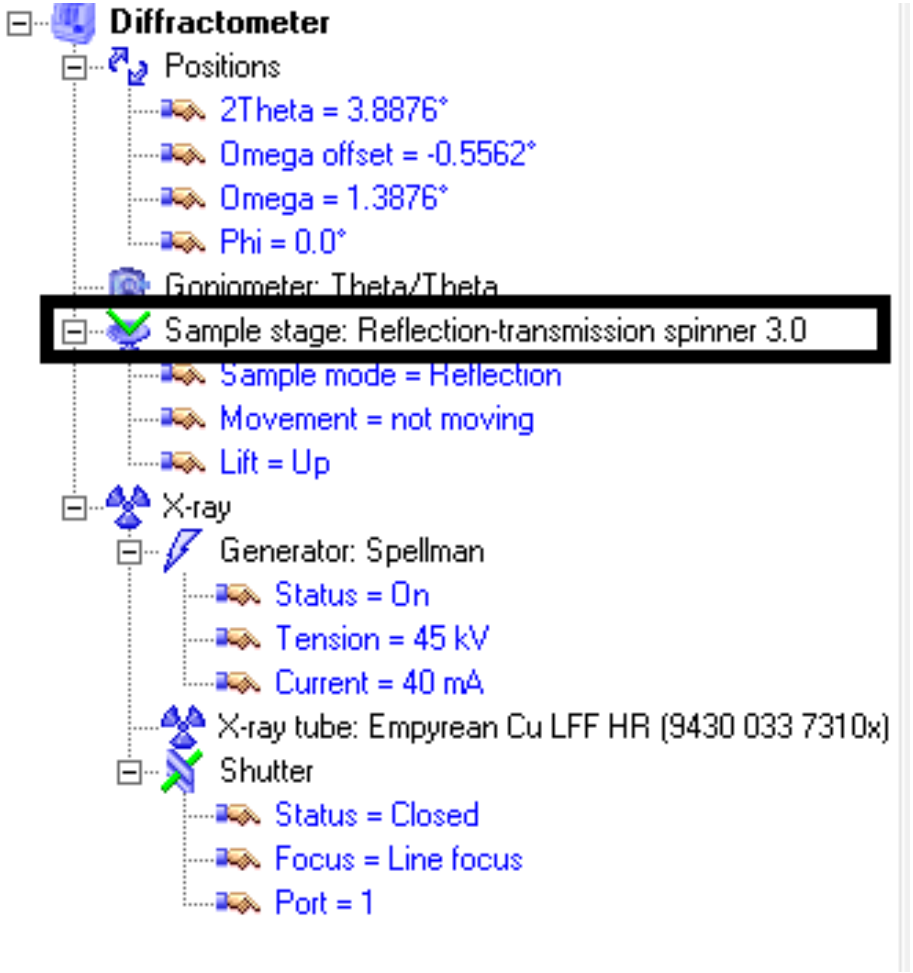
Step7: Before opening the door , click on the **unlock** doors button (and you will hear a vent release sound) and open the door by holding tight (be so gentle on the handling and don't try to open the door till you hear the vent sound), put your sample in the frame of the sample holder, and rotate your sample holder just to crosscheck if it is properly placed in the appropriate holding place of the machine and close it carefully till you hear the magnet sound for attachment.

Note: Before putting the sample make sure the stage is downward to carry your sample, if not double click on the sample stage option then as shown in the picture, **uncheck** the lift-up checkbox, and click apply then after the stage is down click ok.

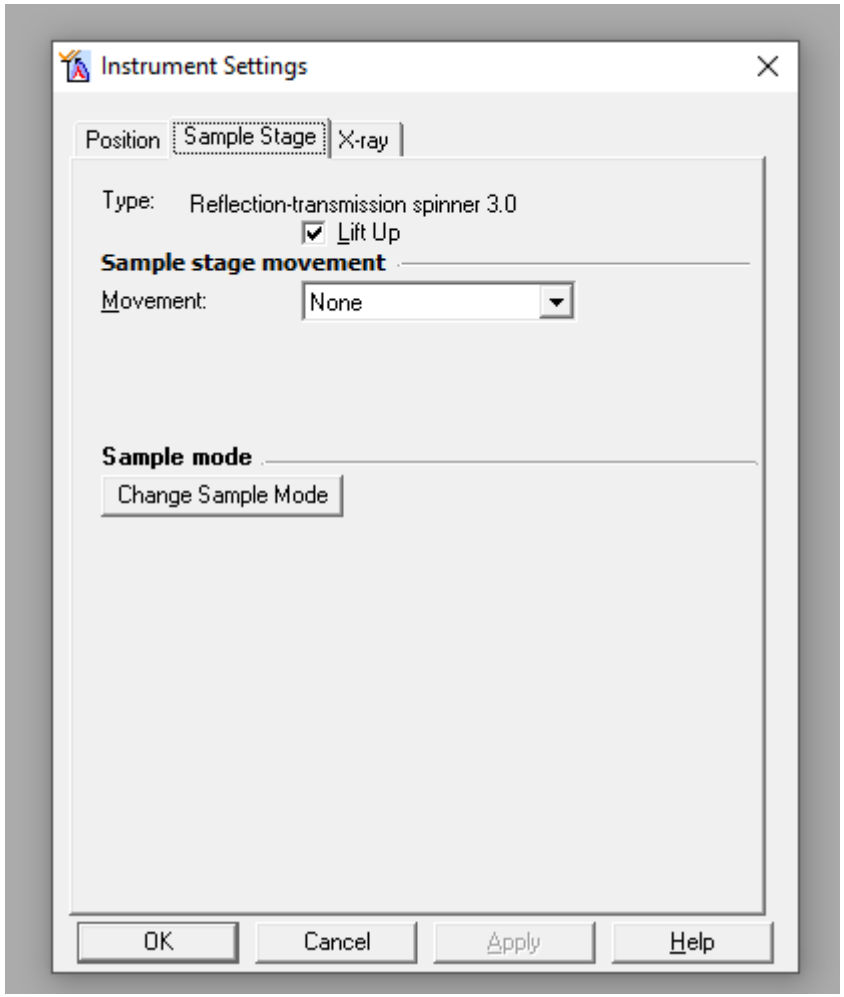

Diffractometer
Positions
2Theta = 3.8876°
Omega offset = -0.5562°
Omega = 1.3876°
Phi = 0.0°
Goniometer: Theta/Theta
Sample stage: Reflection-transmission spinner 3.0
Sample mode = Reflection
Movement = not moving
Lift = Up
X-ray
Generator: Spellman
Status = On
Tension = 45 kV
Current = 40 mA
X-ray tube: Empyrean Cu LFF HR (9430 033 7310x)
Shutter
Status = Closed
Focus = Line focus
Port = 1
Instrument Settings
Position
Sample Stage
X-ray
Type:
Reflection-transmission spinner 3.0
Lift Up
Sample stage movement
Movement:
None
Sample mode
Change Sample Mode
OK
Cancel
Apply
Help

Step 7: put your sample stage up for analysis

To do that click on the lift stage option which is shown in the picture

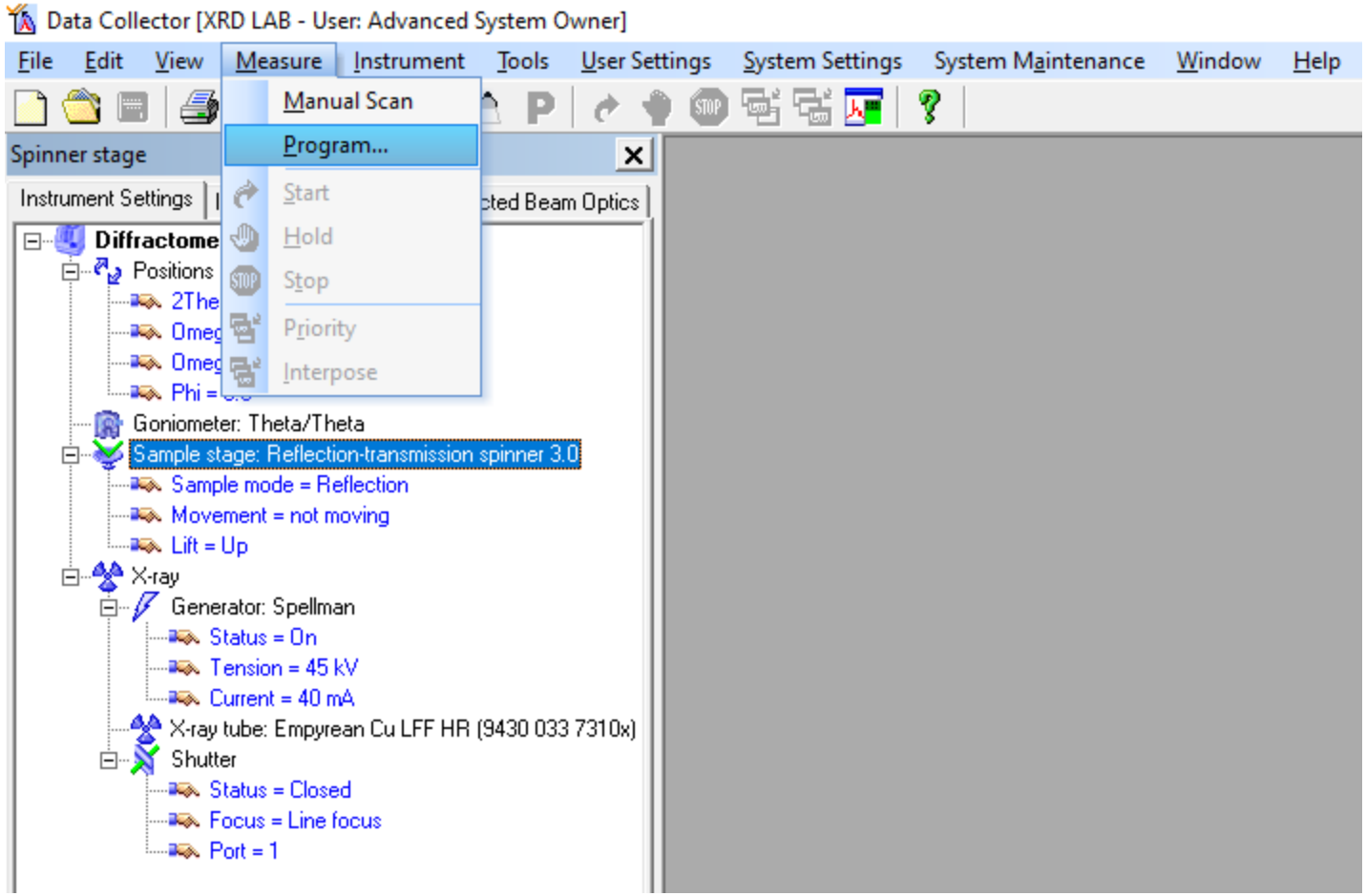


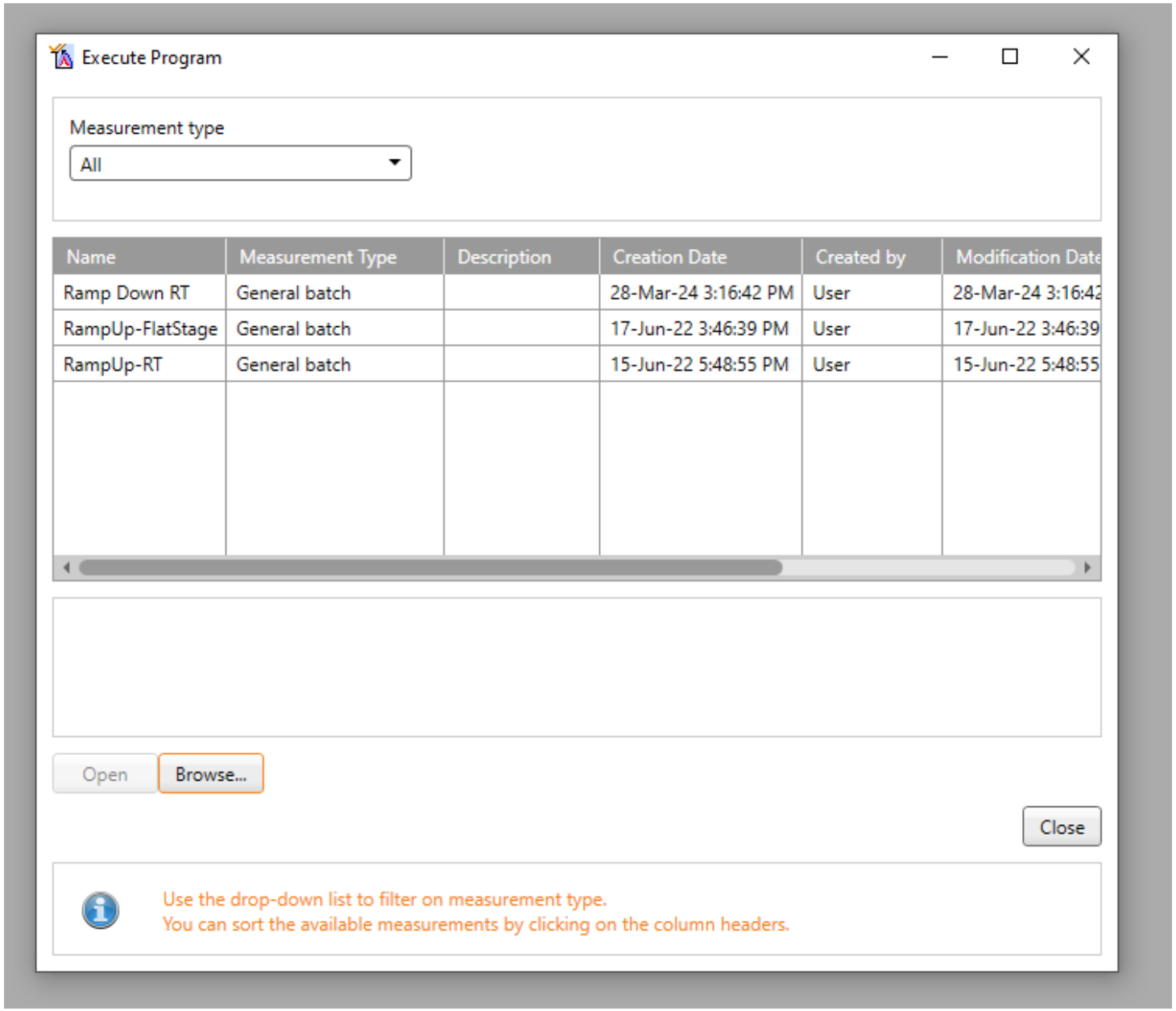
And you will get a window for lift up or down button , remember that if the lift up check box is checked it will move the lift up ward and if its unchecked and applied its for moving the lift down ward so for now we will use lift upward so check the lift up checkbox , then apply and then ok.

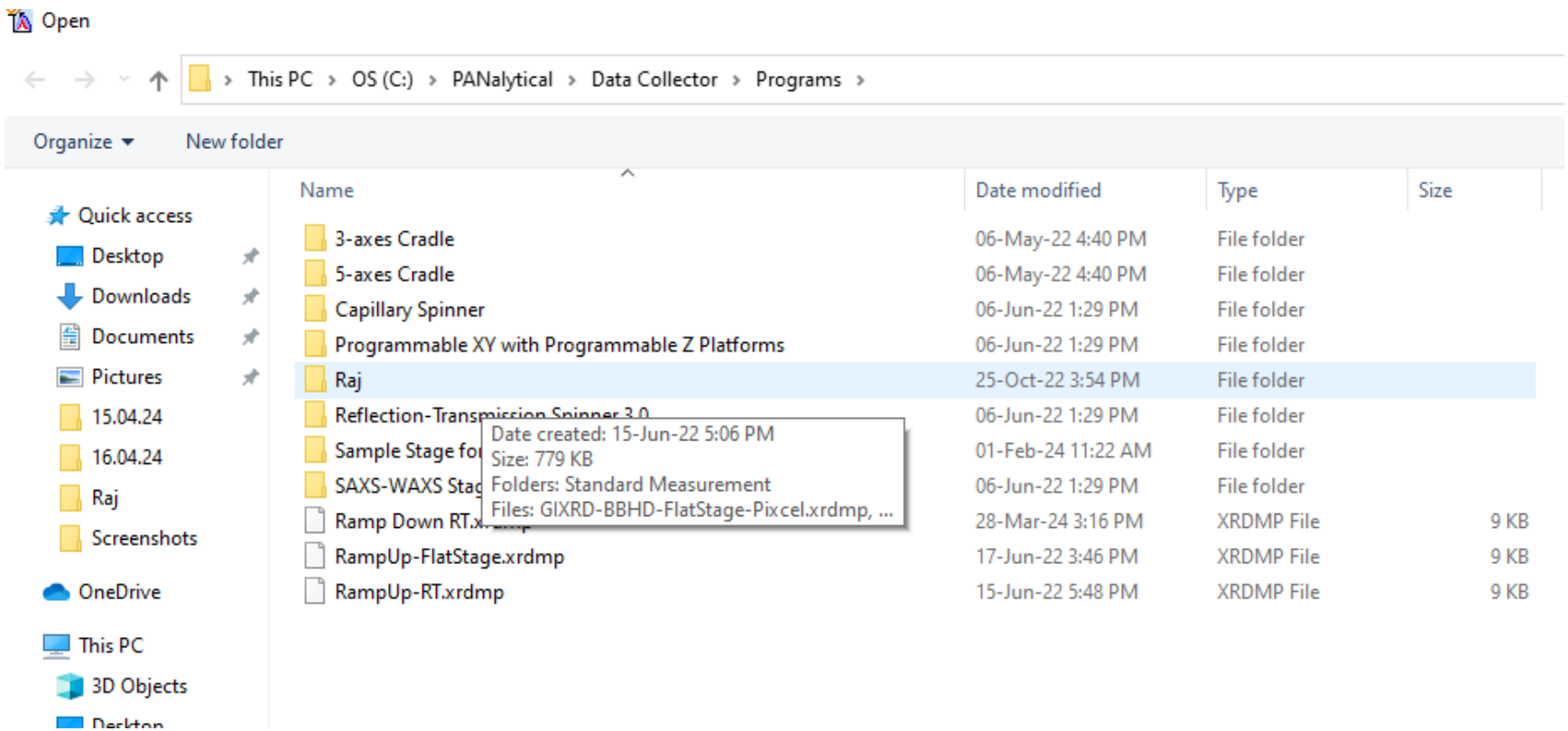


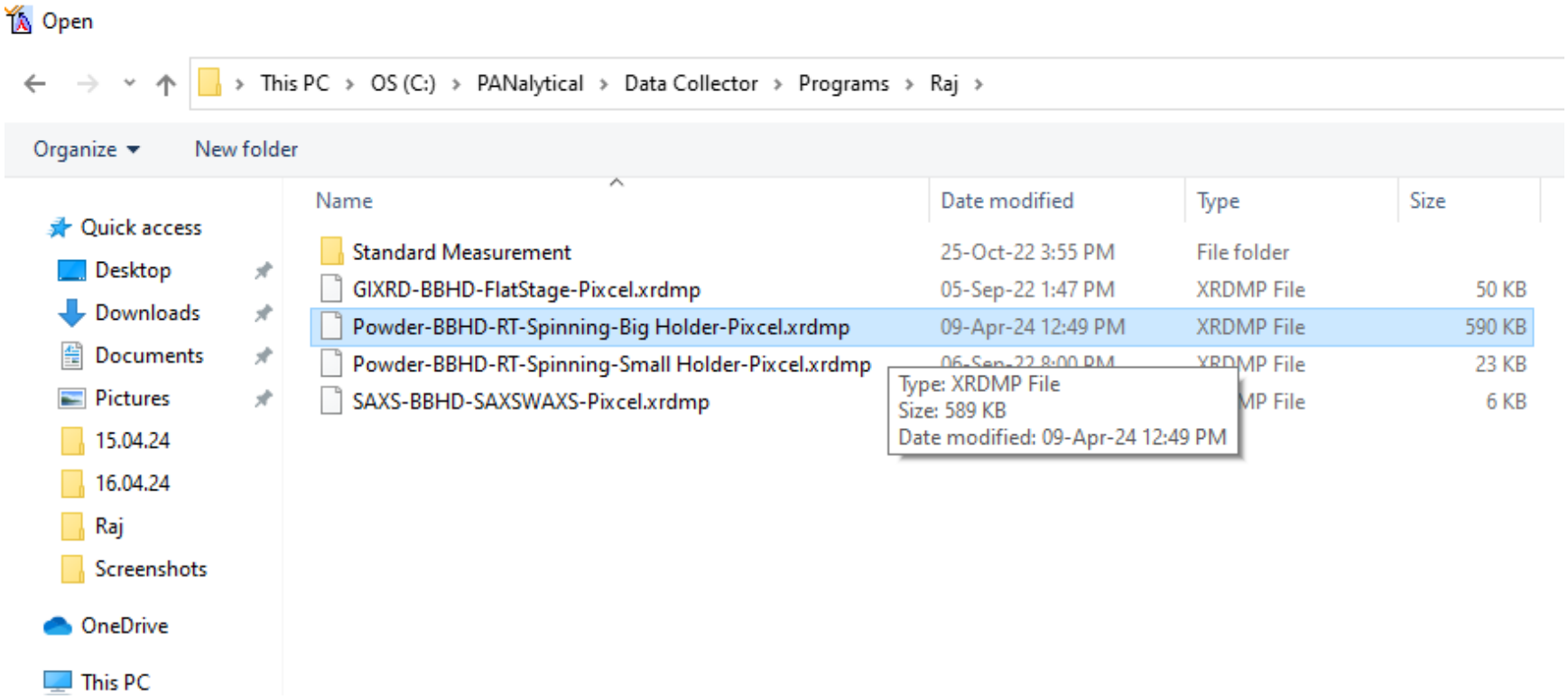
Step 8: Analysis (selecting the program)

Step one: Measure and select the program and on the next window click on Browse



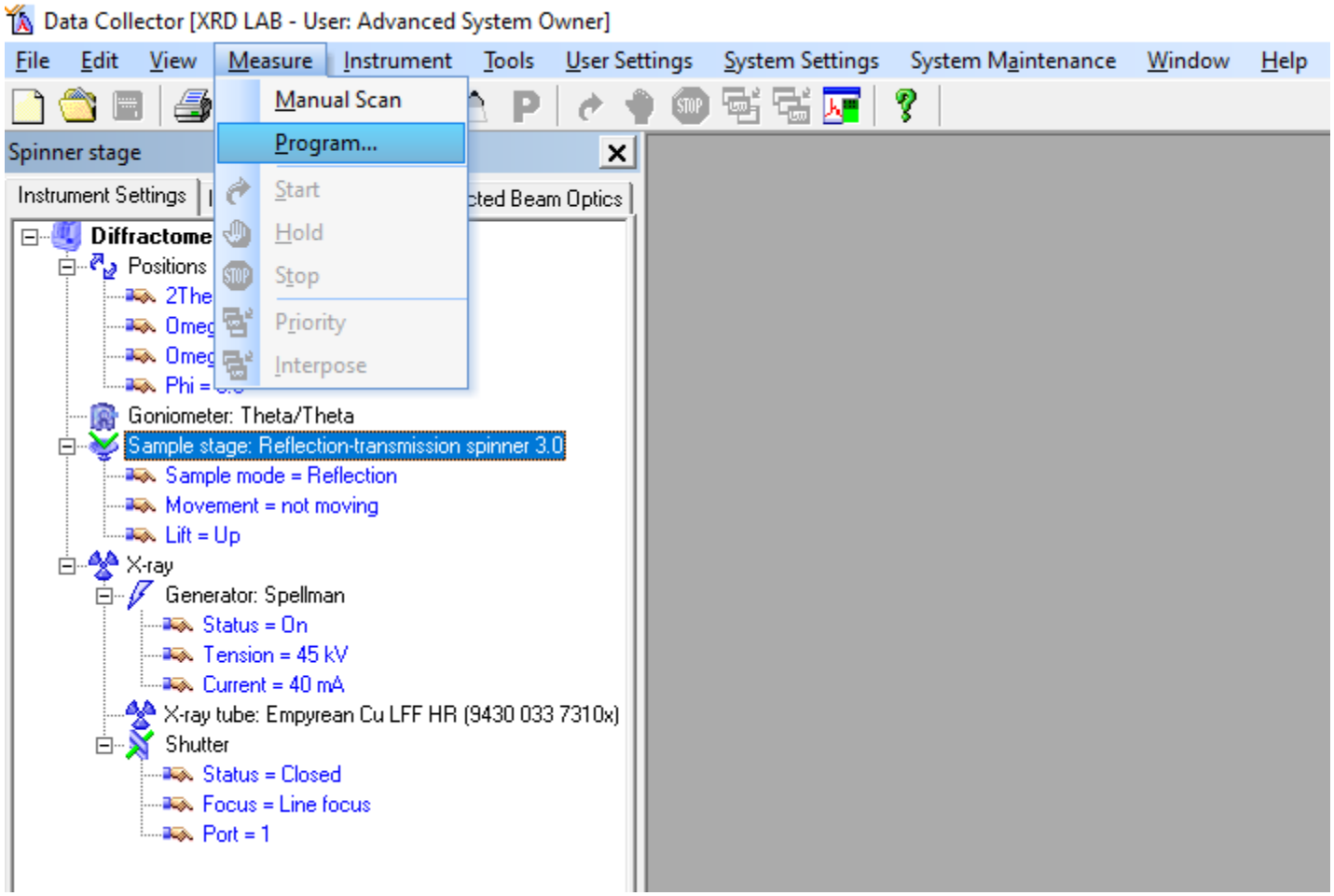

Execute Program
Measurement type
All
Name
Measurement Type
Description
Creation Date
Created by
Modification Date
Ramp Down RT
General batch
28-Mar-24 3:16:42 PM
User
28-Mar-24 3:16:42
RampUp-FlatStage
General batch
17-Jun-22 3:46:39 PM
User
17-Jun-22 3:46:39
RampUp-RT
General batch
15-Jun-22 5:48:55 PM
User
15-Jun-22 5:48:55
Open
Browse...
Close
Use the drop-down list to filter on measurement type.
You can sort the available measurements by clicking on the column headers.


Open
This PC
OS (C:)
PANalytical
Data Collector
Programs
Organize
New folder
Quick access
Desktop
Downloads
Documents
Pictures
15.04.24
16.04.24
Raj
Screenshots
OneDrive
This PC
3D Objects
Name
Date modified
Type
Size
3-axes Cradle
06-May-22 4:40 PM
File folder
5-axes Cradle
06-May-22 4:40 PM
File folder
Capillary Spinner
06-Jun-22 1:29 PM
File folder
Programmable XY with Programmable Z Platforms
06-Jun-22 1:29 PM
File folder
Raj
25-Oct-22 3:54 PM
File folder
Reflection-Transmission Spinner 3.0
06-Jun-22 1:29 PM
File folder
Sample Stage fo
01-Feb-24 11:22 AM
File folder
SAXS-WAXS Stag
06-Jun-22 1:29 PM
File folder
Ramp Down RT.x
28-Mar-24 3:16 PM
XRDMP File
9 KB
RampUp-FlatStage.xrdmp
17-Jun-22 3:46 PM
XRDMP File
9 KB
RampUp-RT.xrdmp
15-Jun-22 5:48 PM
XRDMP File
9 KB
Date created: 15-Jun-22 5:06 PM
Size: 779 KB
Folders: Standard Measurement
Files: GIXRD-BBHD-FlatStage-Pixcel.xrdmp, ...

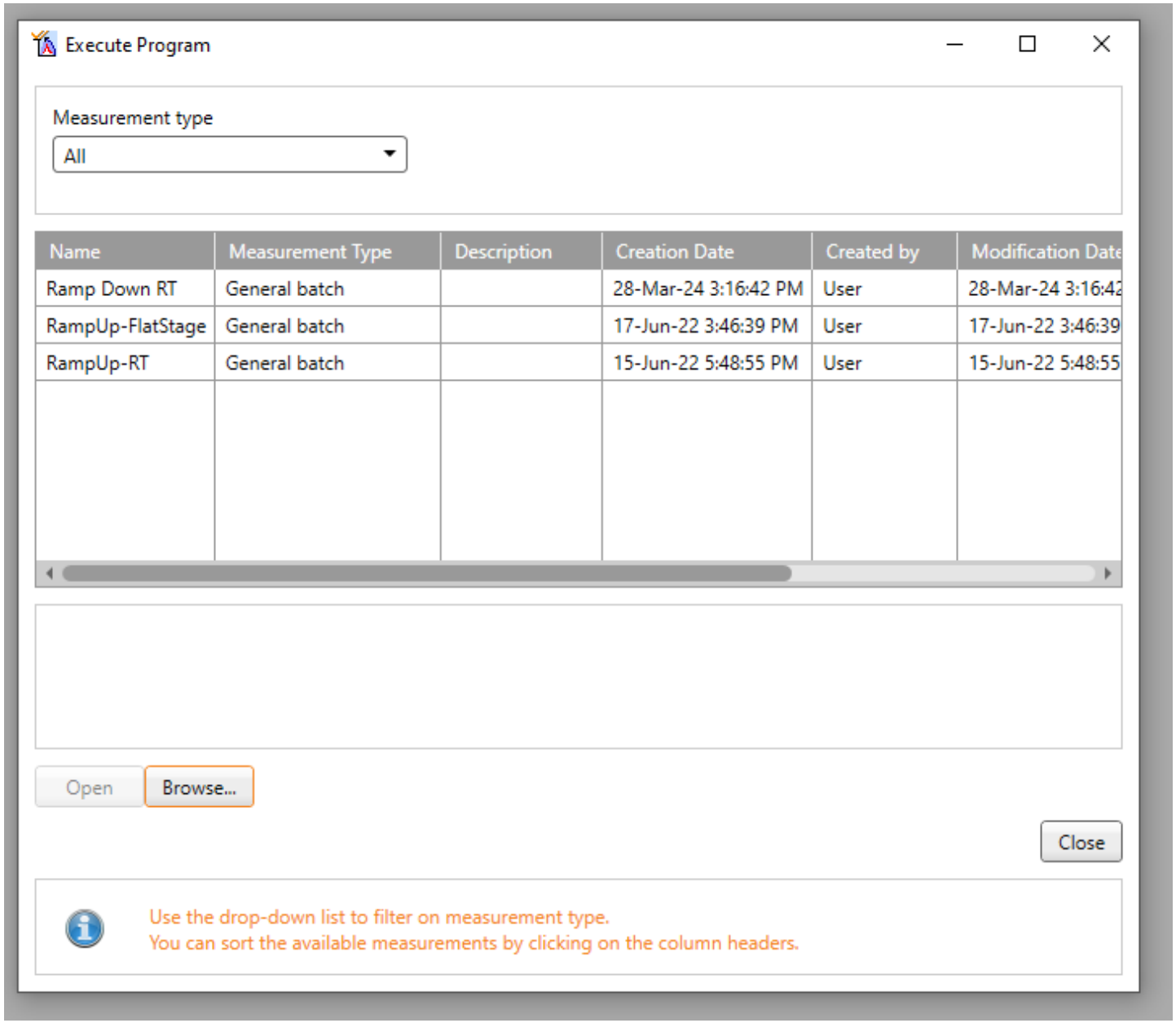


Step two : sample analysis by selecting the program and saving

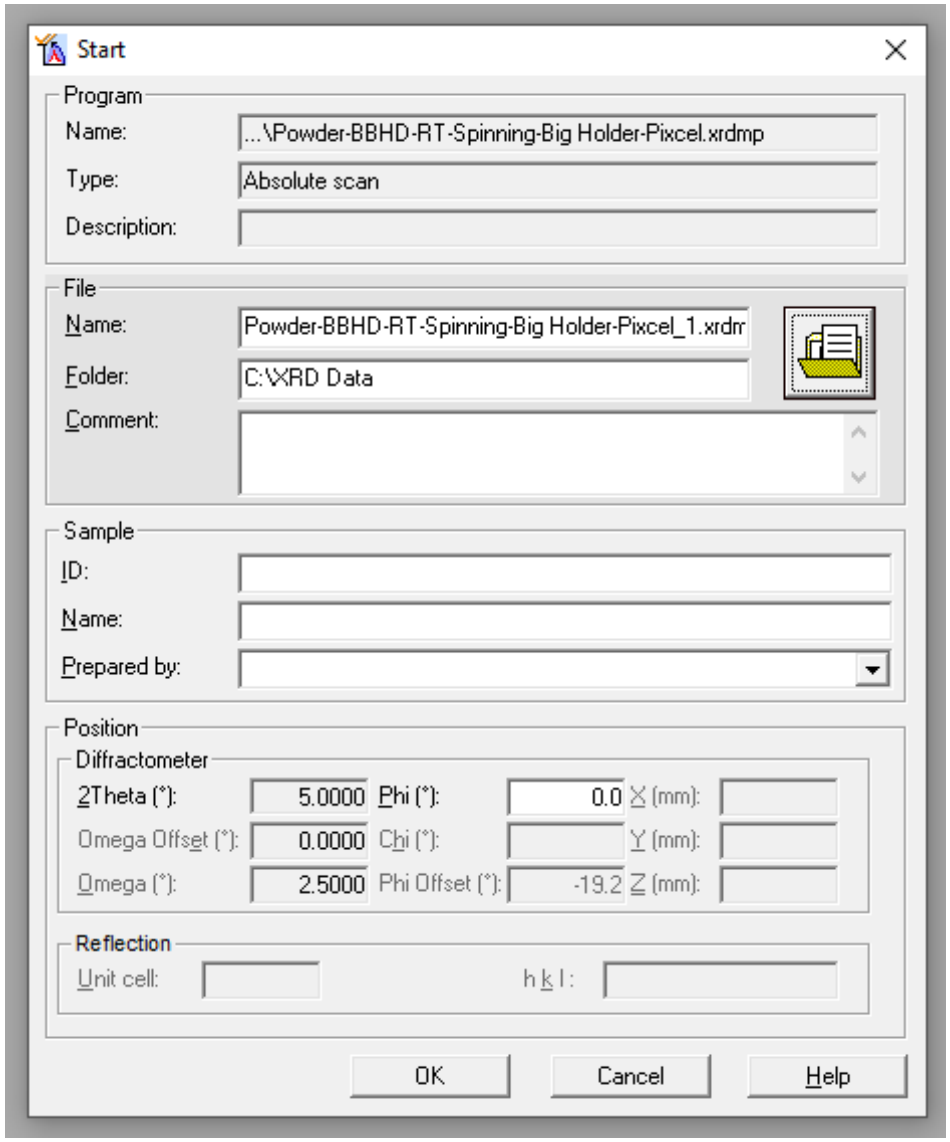
Step 1 : click on measure and select program on from the header



Step 2: Then click on Browse and you will see another window opening


Execute Program
Measurement type
All
Name
Measurement Type
Description
Creation Date
Created by
Modification Date
Ramp Down RT
General batch
28-Mar-24 3:16:42 PM
User
28-Mar-24 3:16:42
RampUp-FlatStage
General batch
17-Jun-22 3:46:39 PM
User
17-Jun-22 3:46:39
RampUp-RT
General batch
15-Jun-22 5:48:55 PM
User
15-Jun-22 5:48:55
Open
Browse...
Close
Use the drop-down list to filter on measurement type.
You can sort the available measurements by clicking on the column headers.

Step 3: Then select 'Raj' and select the third program like you selected in program setup portion, then click on the folder icon and click ok.



Step 4: Go to local disk D and select the folder name "**XRD DATA**" and create your own folder (if its first time) and select it or select if folder by your name exists and give you a sample name and save the name. after saving, It will direct you back to the previous windo , then click ok to start the analysis.

Step 5: Once the analysis is completed close both the progress and graph showing windows and , double click on" **sample stage reflection-transmission spinner 3.0** " from the left button options for stage up/down action and , uncheck the "**lift up**" checkbox then click on apply, when the ok button is active , click on ok.

Step 6 : While trying to remove the sample, first press the button to unlock the doors , then once you hear the vent sound, you can hold the door opening handler gently and open the door to remove the sample.

Data conversion using Xpert higrocore software

After completion of analysis , open expert higroscore software from the pc and open the xrd result file on the xpert software, edit necessary editings like background substraction and save it as per your interest eg. (asc,raw etc..)

Machine closing

Step one : remove sample holder

step two : make the sample holder in the machine to "lift up" position.

Step three : go to measure and select program then select "ramp down" then click on open then on the upcoming windows select ok

Step four : wait for KV and mA reading till it show 30 and 10 respectively.

Step four : disconnect the software by going to instrument tab and click on disconnect button and click ok on the upcoming window. And close the software

Step five : go to machine and rotate the key anticlockwise 90 degree to turn off X-ray you will see the x-ray light getting off from side lights of the machine

Step six : machine power off by off button by long pressing (3 to 4 seconds) .

Step seven : tun off the chiller by reversing back 90 degree anticlockwise

Step eight : turn of the compresser from main switch

Step nine : turn off computer

Step ten : tunr off power switches from MCB (xrd, chiller , and computer)

File --- > open program --- > browse -→ raj -→spinning --→ big holder --→ browse --→ go to local disk D -→ go to XRD data -→ save accordingly -→ once the program is saved the program will start automatically.