PANalytical X-Ray Diffractometer Standard Operating Procedure

Varsion 2.1 (Corrected on March 2024)

Training Rules:

First time: watch trainer and take notes

Second time: do by yourself with supervision following your notes

Third time: do by yourself with supervision without help (Dr. Bykov must approve the authorization for independent use)

After the third time, you are completely independent and responsible for the instrument.

Operating Rules:

1. Only authorized people can use XRD.
2. You must sign up for time on the XRD using the online system:

http://corelabs.ccny.cuny.edu/

3. All samples must be recorded in XRD logbook including the date, time, user, sample name, program name, and filename.
4. If you have any problems, please DO NOT troubleshoot yourself - Call:

Alexey Bykov212-650-5548 (Laboratory) or646-725-0270 (cell phone)

Outline:

1. **Turn on the instrument**
2. **Tube breeding**
3. **Make a new program**
4. **Typical operation with fixed holder**
5. **Typical operation with spinner holder**
6. **Export data**
7. **Turn off the instrument**

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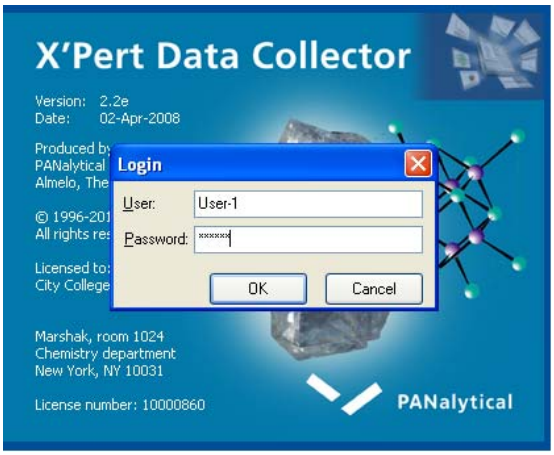
1. Turn on the Instrument:

1.1 Press power switch on the water chiller.

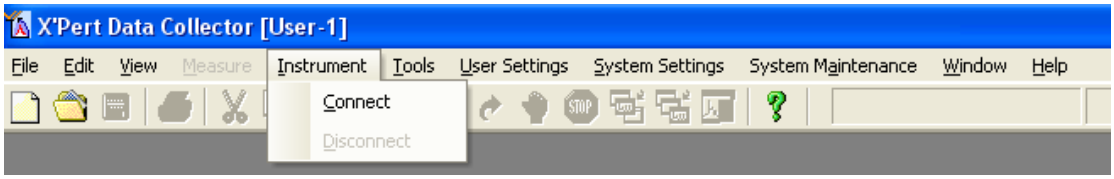
1.2 If PC is ON, double click on *X'Pert Data Collector* icon.

If softwareis already runningbut generator status is OFF (Tension = 0 kV and Current = 0 mA) skip steps1.2-1.5 and go to step 1.6. If generator status is also ON (Tension > 20 kV, Current > 5 mA) go to step 2.3.

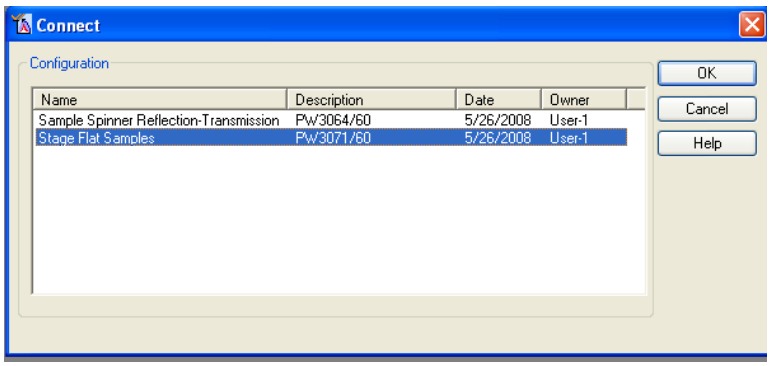
1.3 Type user name: ***User-1*** and password***:*** ***galaxy*** in Login dialogue box.



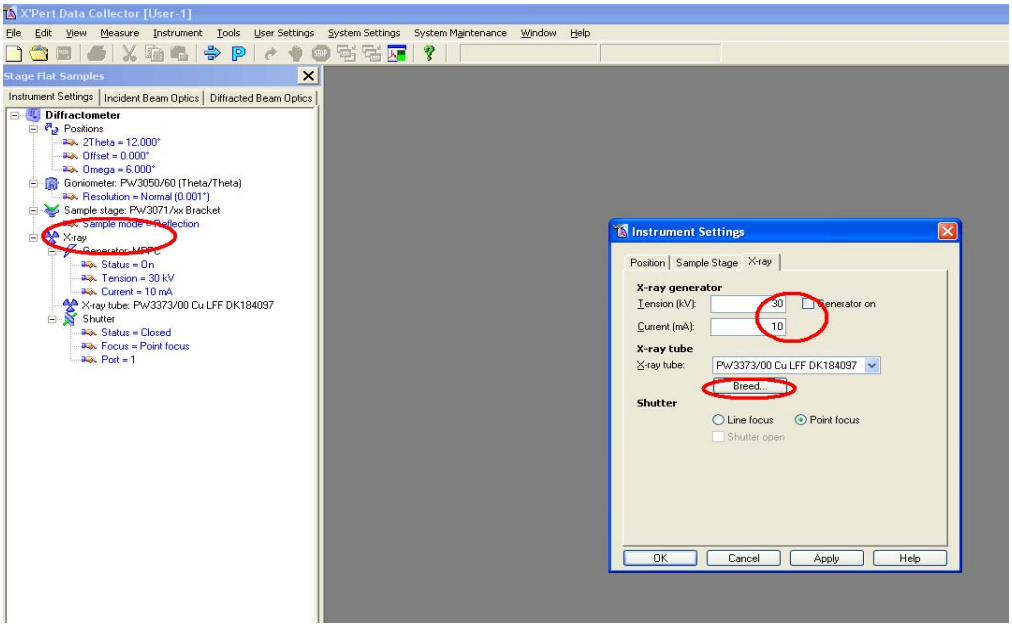
1.4 Go to *Instrument* menu and click *Connect*.



1.5 Select *Stage Flat Sample* for regular measurements or *Sample Spinner* if you need to use sample changer and/or rotate your sample during scanning procedure and press *OK*(To switch sample holders go to *Tools* menu →*Exchange Sample Stage* with following connection procedure



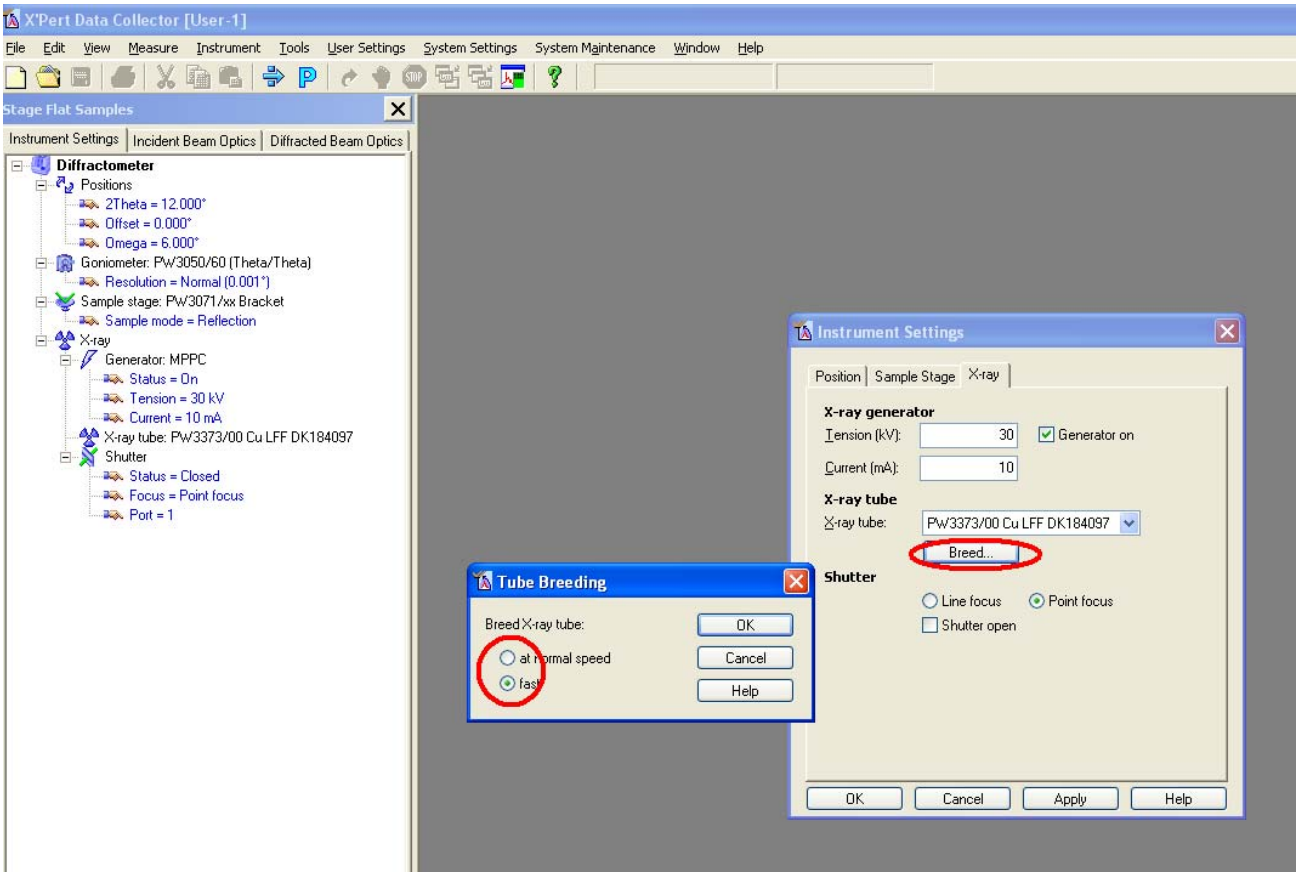
1.6 Double click on *X-ray*. Check the*Generator on* boxat the *Instrument Setting*, press *Apply*, and wait until tube powerreaches30 kV and 10 mA .



2. Tube Breeding

If X-rays have been turned off for an extended period of time, tube breeding (conditioning) isnecessary.Check the log book first to see when last time the instrument was used.Breed at normal speed if the instrument was left idle for 100 hours or more (breed takes about 30 minutes). Breed at fast speed if the instrument was used recently (within the last 100 hours). In this case, tube breeding takes about 6 min.

2.1 Click *Breed*. Choose *fast* or *normal* in *Tube Breeding* box and mark *OK*



2.2 Tension during breeding procedure goes up to 60 kV then down to 40 kV.

After breedingthe parameters of the generator will be *40 kV and 10 mA*

2.3 On the *Instrument Settings* window set measurement parameters *40 kV and 40 mA*.Click *Apply* then *OK*.

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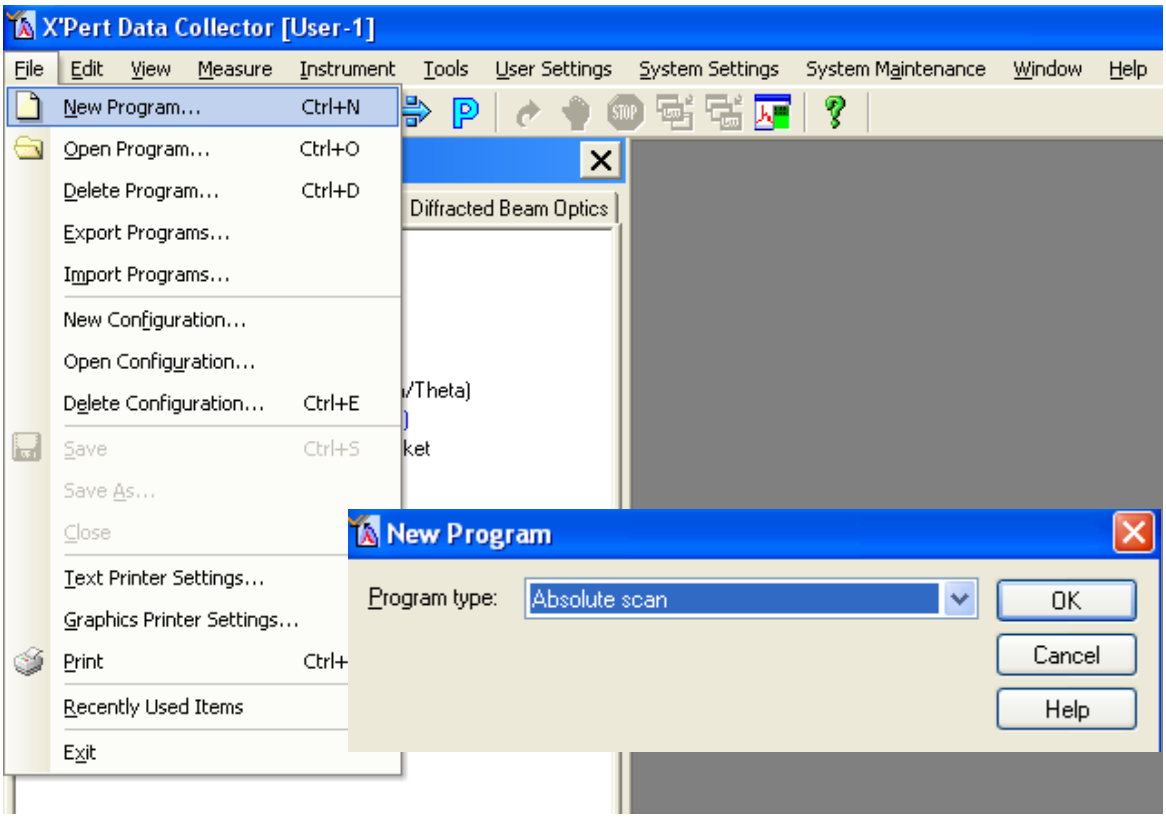
3. **To Make a New Program**:

3.1 Go to *File* menu and click *Newprogram*.



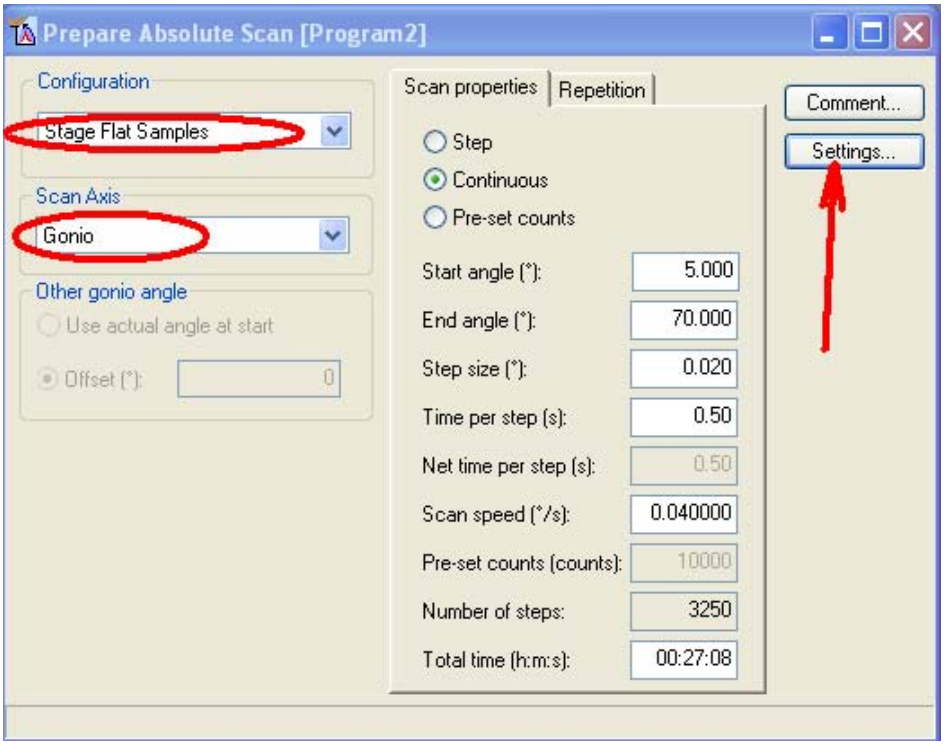
Click *OK* for *Absolute scan* program type.

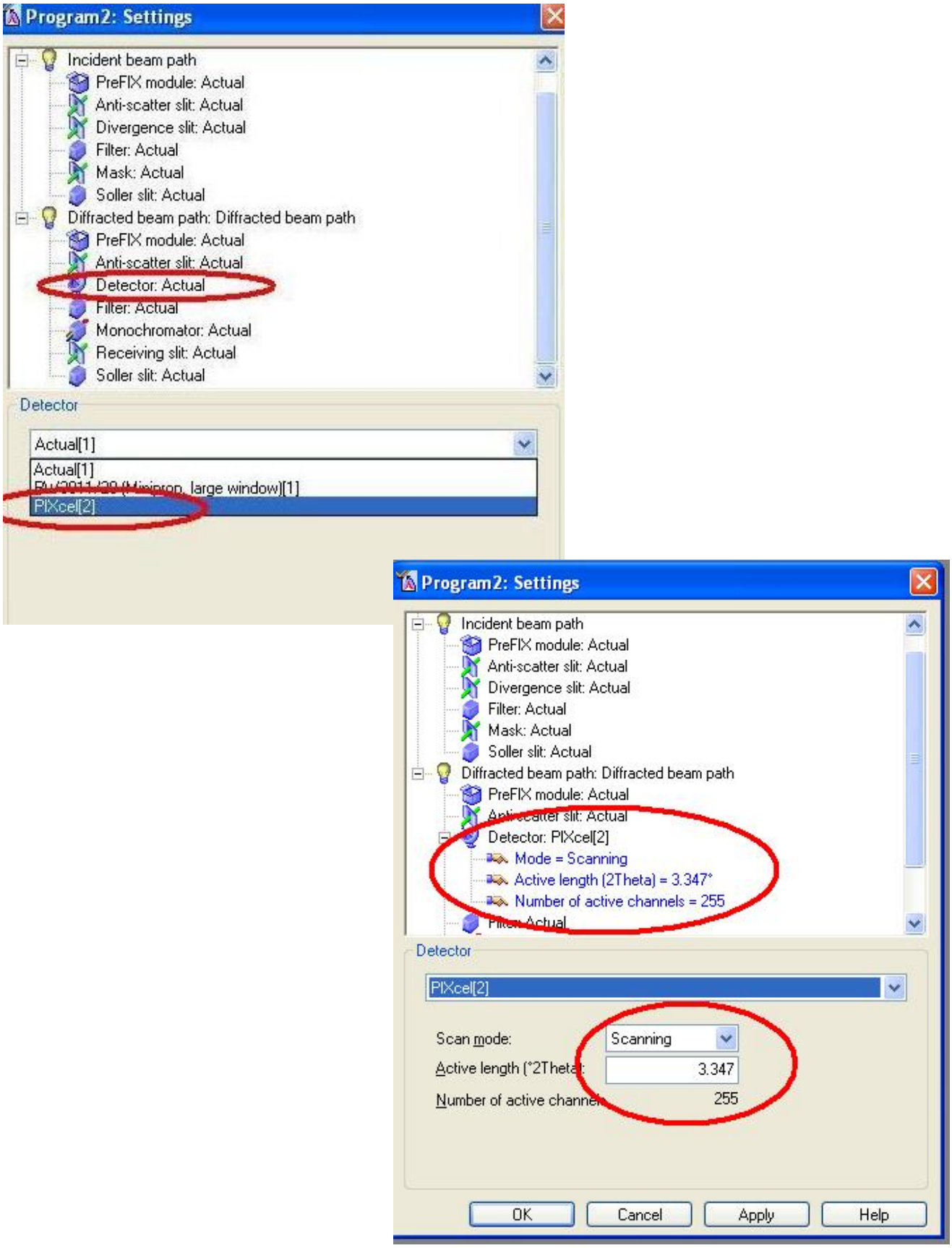
If you have already created your program in previous sessions, use *Open Program* in *File* menu and make parameters changes if you need it, then go to step 4 (skip step 3.2 -

3.3)

3.2 Choose your scanning parameters on the *Prepare absolute scan* window.

- In *Configuration*, select stageflatsamples (for fixed holder) or *Sample reflection Transmission* (for spinner holder)
- In *Scan Axis*, select the type you want, a typical scan for powders is *Gonio*.
- Click on *Repetition*, select then *Single scan* or other scan type.
- Click *Settings*
- Click *Detector: Actual*, choose *Pixcel(2)* (window shows the parameters of PIXcel detector: *Active length* (angle) and *Number of active channels*)
- Click *Apply*, then *OK*




Program2: Settings
Incident beam path
PreFIX module: Actual
Anti-scatter slit: Actual
Divergence slit: Actual
Filter: Actual
Mask: Actual
Soller slit: Actual
Diffracted beam path: Diffracted beam path
PreFIX module: Actual
Anti-scatter slit: Actual
Detector: Actual
Filter: Actual
Monochromator: Actual
Receiving slit: Actual
Soller slit: Actual
Detector
Actual[1]
Actual[1]
PIXcel[2]
Program2: Settings
Incident beam path
PreFIX module: Actual
Anti-scatter slit: Actual
Divergence slit: Actual
Filter: Actual
Mask: Actual
Soller slit: Actual
Diffracted beam path: Diffracted beam path
PreFIX module: Actual
Detector: PIXcel[2]
Mode = Scanning
Active length (2Theta) = 3.347°
Number of active channels = 255
Detector
PIXcel[2]
Scan mode:
Scanning
3.347
255
OK
Cancel
Apply
Help

PIXcel detector

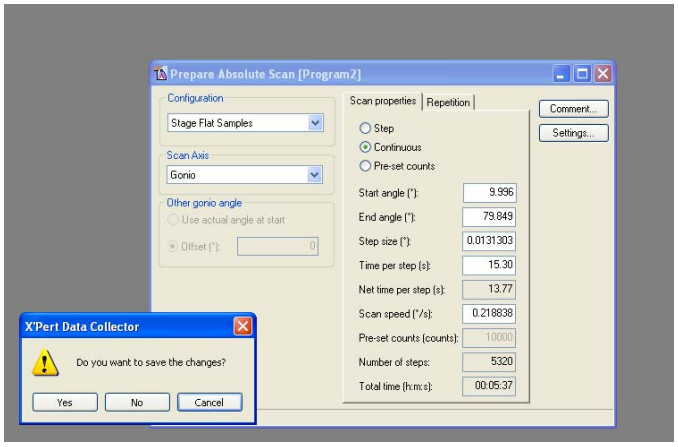
PIXcel detector has been developed by PANalyticalasan advanced two-dimensional photon counting device incorporating the latest in solid-state pixel technology. In our system, PIXcel detector works in 1D (one dimensional) mode. The detector simultaneously counts photons in 2Theta range = 3.347^{o} by 255 active channels. This is a maximum detector's active length. The active length may be decreased down to 0.013 ^{o} and only one channel will be active. At this setting the detector is converted to 0D mode and works like point detector.

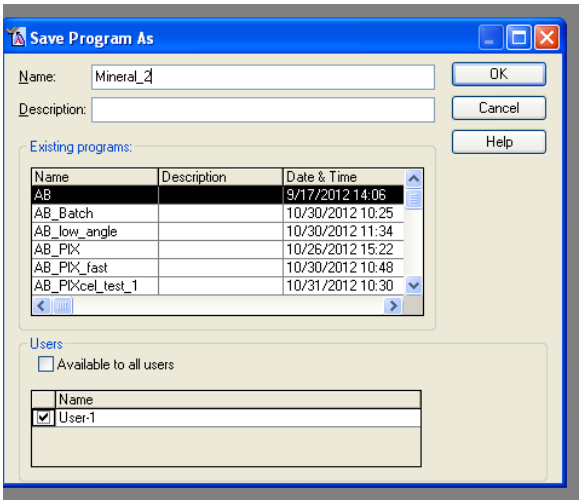
- Click *Scan properties* tab to edit the scanning parameters. Only *Continuous* scan is allowed by PIXcel detector. Set your angle range, step size, time per step (or scan speed). Based on our tests of standard samples we recommend for regular measurements step size ~0.05-0.01^{o} and scan speed ~0.1-0.2^{o}/sec. The total scan time usually should not exceed 10 min. (5 min is optimal total time for 10^{o}-60^{o} Two Theta range)

PIXcel detector at a maximum value of active length (2Theta range = 3.347^{o})allows step size range from ~ 0.002^{o} to 0.512^{o} and scan speed up to 1.27^{o}/sec

3.3 Save experiment

- Go to main menu, select *File*, click *Save as* or click close the *Prepare Absolute Scan* window and press *Yes* to save the file (use your first name or any other title(Ex: Mineral_2) and then click *OK*.





For previous saved program with running XRD you may **start here.**

4. **Typical Operation with Fixed Holder**

4.1. LoadXRD sample slides on the fixed sample holder.

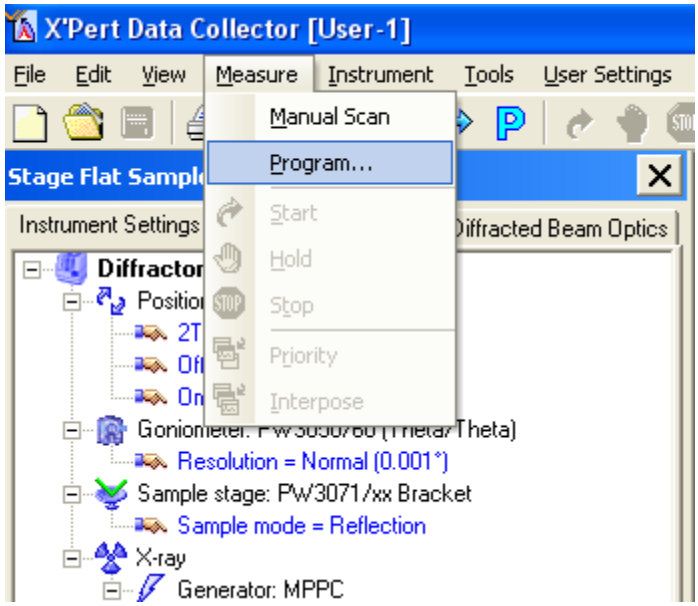
4.2. Go to main menu, click *File*, choose *Open program* and check the program settings.

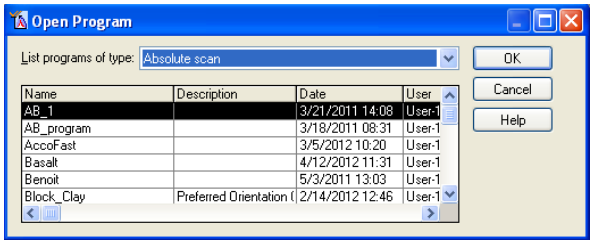
4.3. Go to main menu, Select *Measure*, click *Program*, and *OK* after you choose your program.

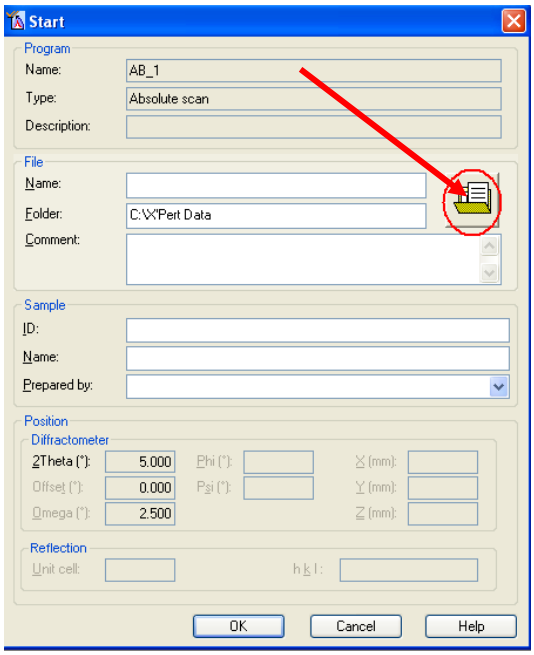
4.4. Click the folder icon on *Start* window(If you are a new user please create a folder under your group folder in *XPert Data* directory)

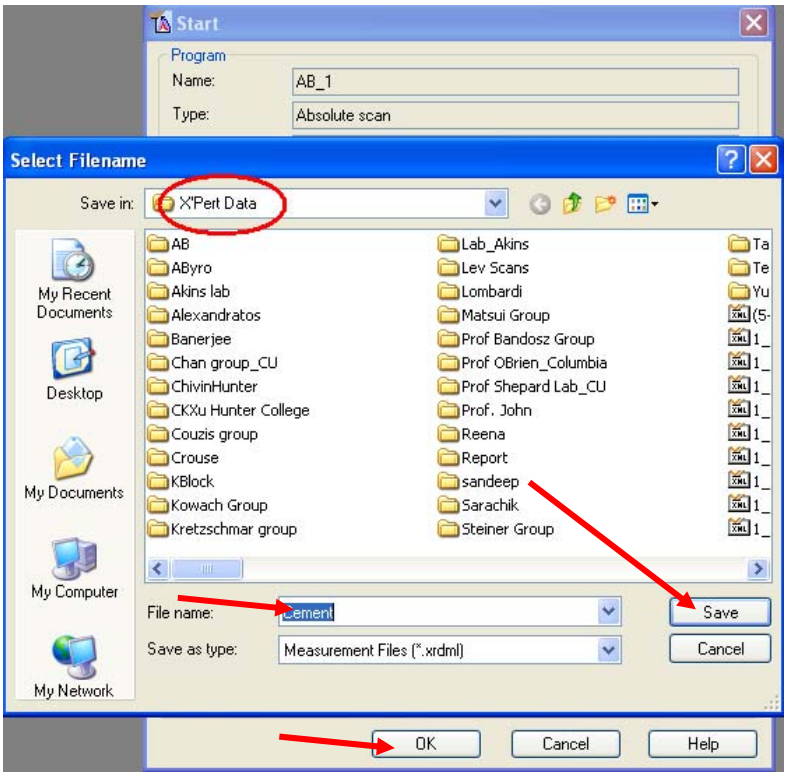
4.5. Type in the file name, then click *Save*

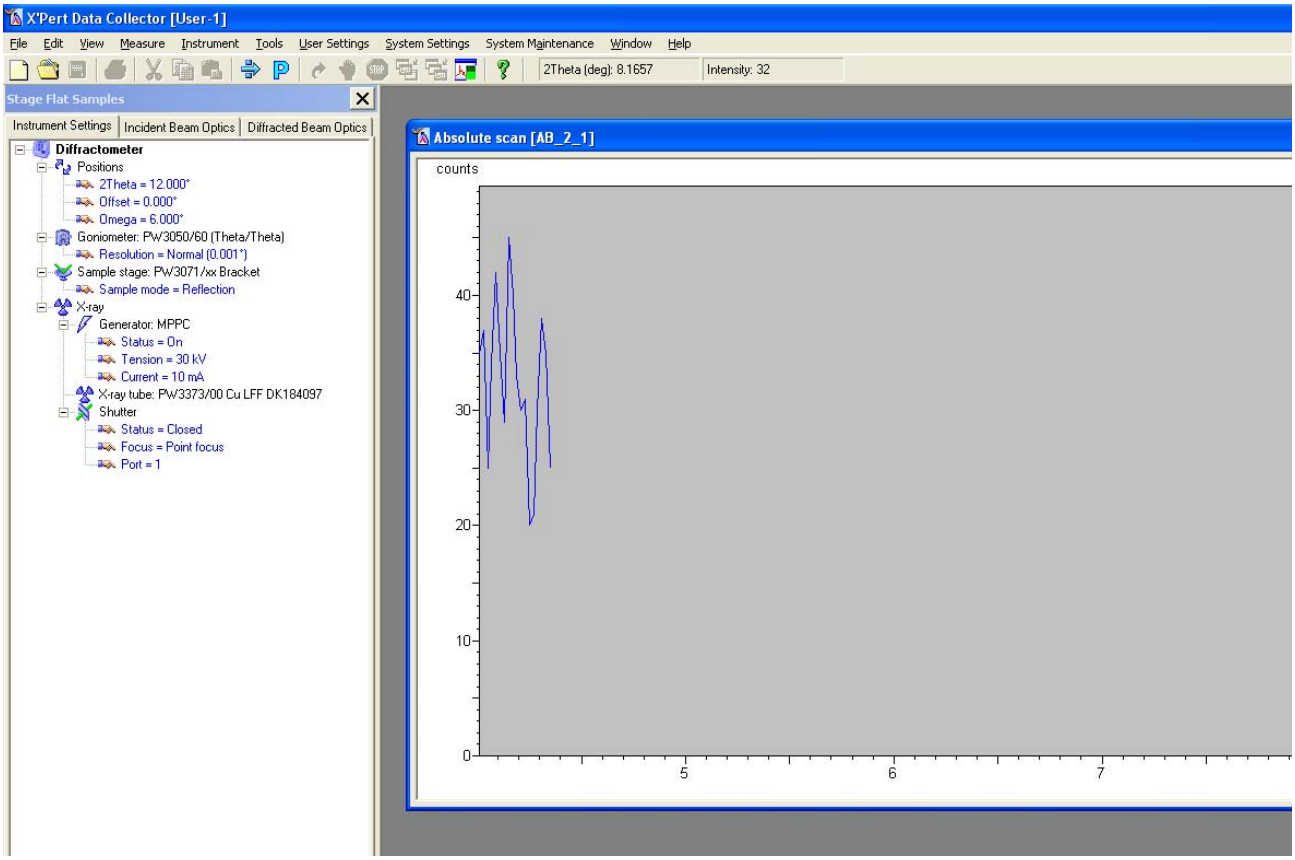
4.6. Click *OK* on *Start* window. The XRD instrument starts scanning.





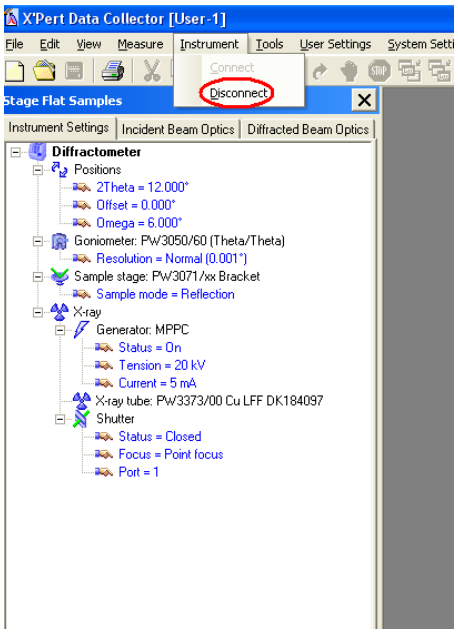

Start
Program
Name: AB_1
Type: Absolute scan
Description:
File
Name:
Folder: C:\X'Pert Data
Comment:
Sample
ID:
Name:
Prepared by:
Position
Diffractometer
2Theta (°): 5.000
Offset (°): 0.000
Omega (°): 2.500
Phi (°):
Psi (°):
X (mm):
Y (mm):
Z (mm):
Reflection
Unit cell:
h k l:
OK
Cancel
Help

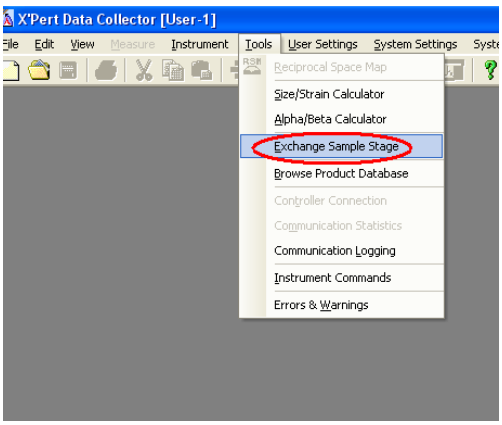

Start
Program
Name: AB_1
Type: Absolute scan
Select Filename
Save in: X'Pert Data
My Recent Documents
Desktop
My Documents
My Computer
My Network
AB
AByro
Akins lab
Alexandratos
Banerjee
Chan group_CU
ChivinHunter
CKXu Hunter College
Couzis group
Crouse
KBlock
Kowach Group
Kretzschmar group
Lab_Akins
Lev Scans
Lombardi
Matsui Group
Prof Bandosz Group
Prof OBrien_Columbia
Prof Shepard Lab_CU
Prof. John
Reena
Report
sandeep
Sarachik
Steiner Group
File name: Cement
Save as type: Measurement Files (*.xrdml)
Save
Cancel
OK
Cancel
Help

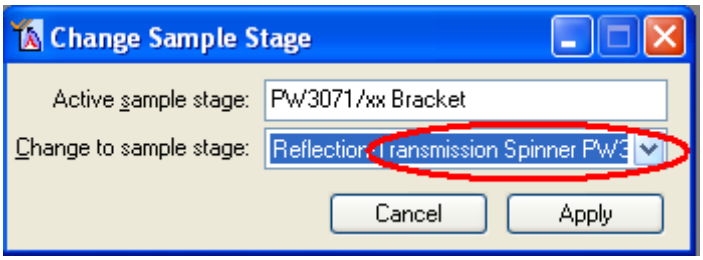

X'Pert Data Collector [User-1]
File Edit View Measure Instrument Tools User Settings System Settings System Maintenance Window Help
2Theta (deg): 8.1657
Intensity: 32
Stage Flat Samples
Instrument Settings
Incident Beam Optics
Diffracted Beam Optics
Diffractometer
Positions
2Theta = 12.000°
Offset = 0.000°
Omega = 6.000°
Goniometer: PW3050/60 (Theta/Theta)
Resolution = Normal (0.001°)
Sample stage: PW3071/xx Bracket
Sample mode = Reflection
X-ray
Generator: MPPC
Status = On
Tension = 30 kV
Current = 10 mA
X-ray tube: PW3373/00 Cu LFF DK184097
Shutter
Status = Closed
Focus = Point focus
Port = 1
Absolute scan [AB_2_1]
counts
40
30
20
10
0
5
6
7

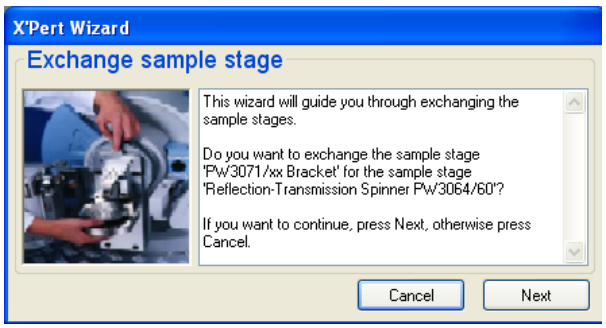
5. Typical Operation with Spinner Holder

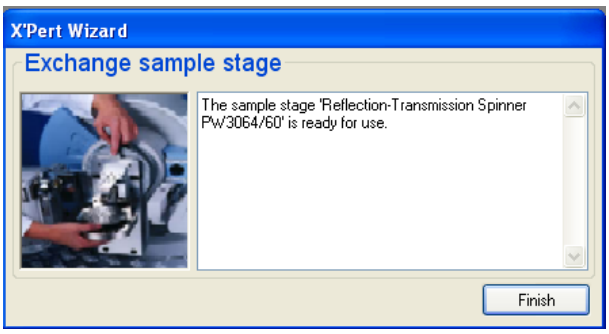
5.1.To switch sample holders you should disconnect XRD *Instrument – Disconnect*, then go to *Tools* menu → *Exchange Sample Stage*, choose *Reflection TransmissionSpinner* at *Change Sample Stage* window, press *Apply*.Follow the instructions of the. X'Pert Wizard



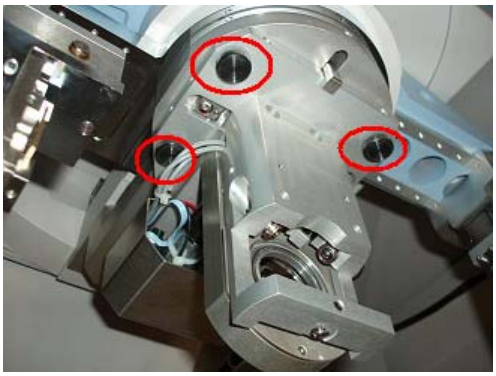




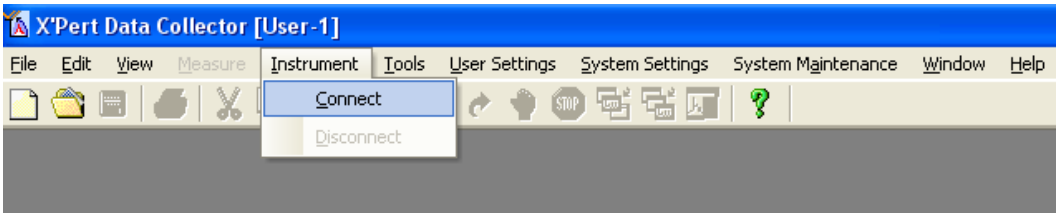


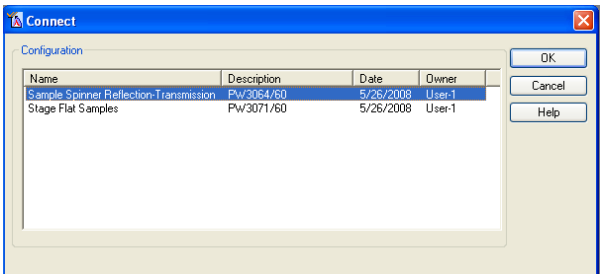


The Fixed Stage is mounted to the base by four screws. Unscrew all of them and carefully remove the stage. Take the Spinner Holder and attach to the base. Mount the holder using three screws only.

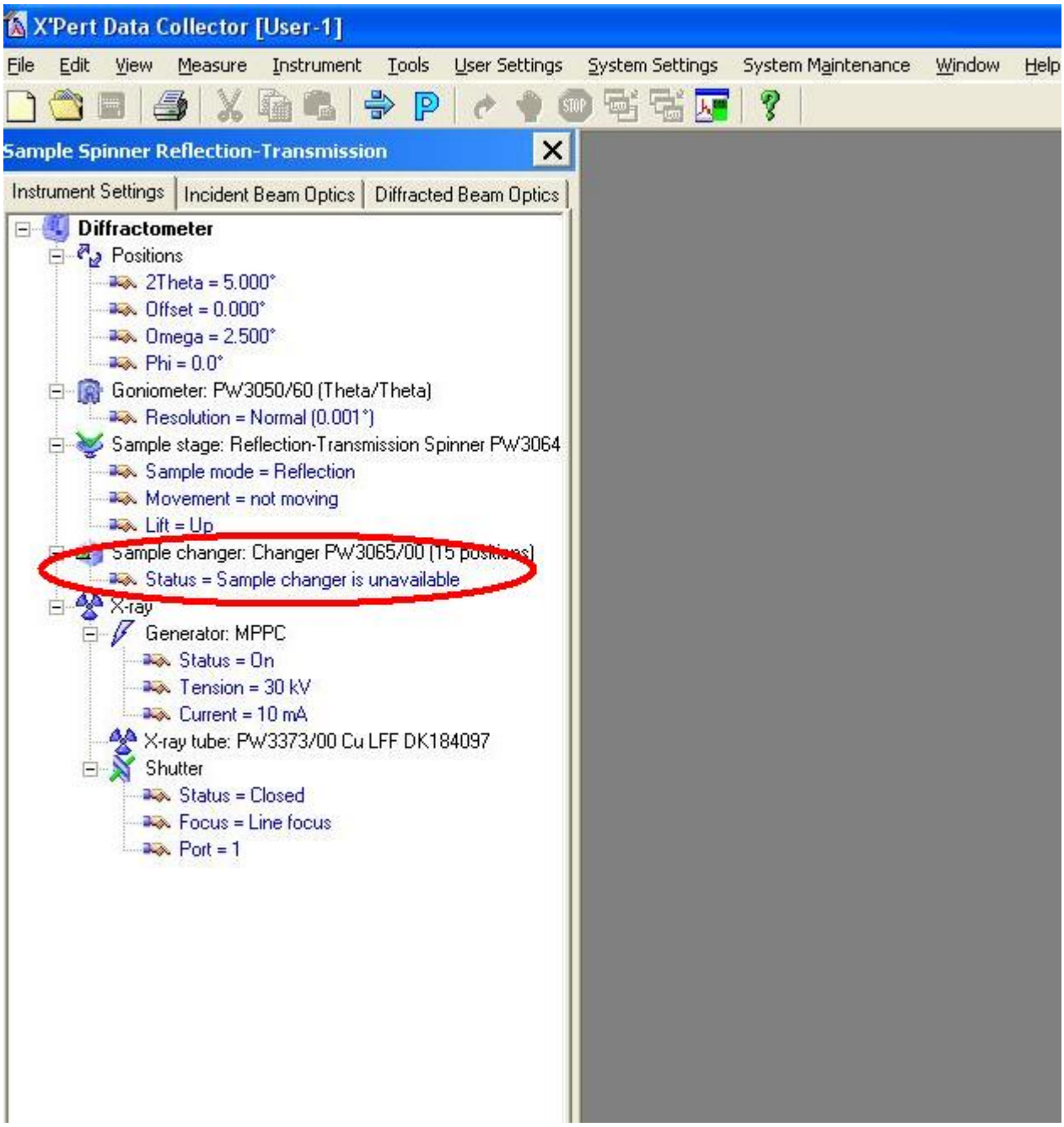


- Connect the instrument (Go to *Instrument* menu and click *Connect*).
- Select *Sample stage Reflection Transmission* in the *Connect* window and click *OK*



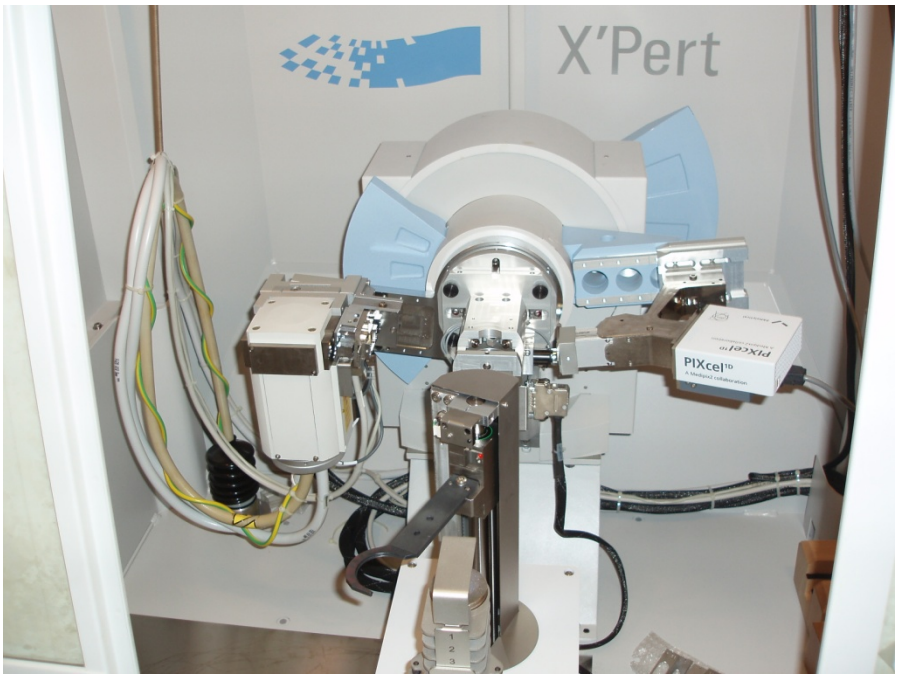


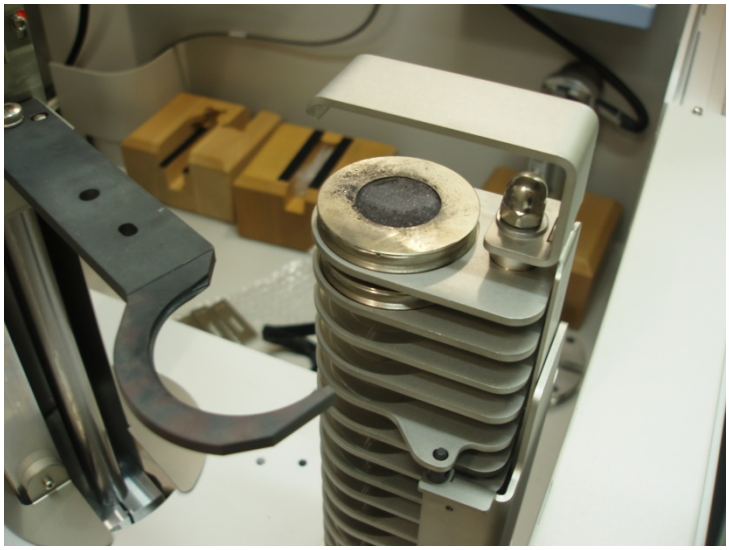
If the Sample Changer is on corner position it's status will be *Unavailable*



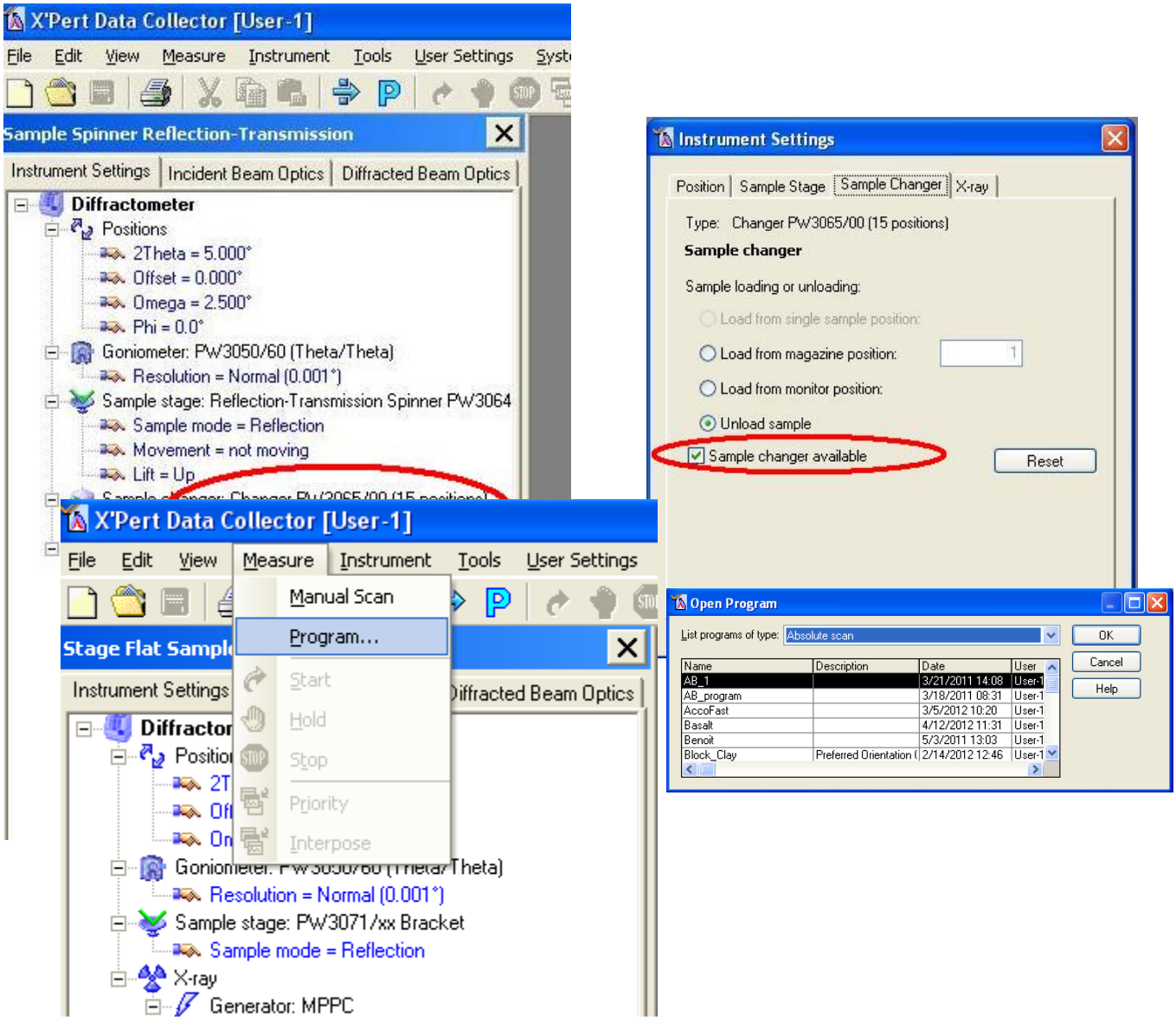
- Move Sample Changer from corner to center position

  <u>This will be done by XRD facility manager only !!!</u>





When Sample Changer will be fixed in center position it's status will be changed from *Unavalable* to *No sample loaded*. If there is no changes in Sample Changer status occurred, click on *Sample Changer* to open *Instrument setting* window, mark *Sample changer available box* and press *Apply*.



5.1. Load your sample on position 1(top) of the Sample Changer.

5.2. Go to the *File* menu then choose *Open program*. The program settings are appropriate to *samplespinnerReflection-Transmission* mode (*Configuration* – *Sample Spinner Reflection*, *Scan Axis* – *Gonio*. Click *OK*

5.3. Select *Measure* and click on *Program*, then *OK* after you choose your program.

5.4. Click the folder icon on *Start* window (If you are a new user please create a folder under your group folder in *XPert Data* directory)

5.5. Type in the file name, then click Save

5.6. Click *OK* on *Start* window. The sample will be loaded in spinner and XRD instrument starts scanning. After copmpleting measurements the Changer will unload spinner holder placing your sample back to position

6. To export data:

6.1 On the desktop, double click on *High Score Plus* icon to open analyzing software.

6.2 Go to *File* menu then *Open* select the file from your folder.

6.3 In the *ListPanel*, select *Anchor Scan Data* tab, mouse right click while marker on the excel chart. To get XYdata,select *Save list as*, and name your file. Save the data in .CSV or .txt format file.

6.4 To export data in different format, go to *File* menu and select *Save As* and select the desired data format, such as: RD, UDF, DAT…etc.

Data saved in C:\X'Pert Data\ automatically

7. To Turn off the Instrument:

7.1 Double click on X-ray on Stage Flat Sample or Sample SpinnersStageReflection-Transmission on left sideof *Data Collector* window

7.2 Change tube powerto 20 kV and 5 mA in *Instrument Settings* box.

7.3 Click *Apply* then wait for ~1-2 min, uncheck the *Generator* box and click *OK*.

(Do not turn off the key on instrument panel).

7.4 Turn off the cooling water switch on chiller (recirculator).