

PANalytical X'Pert³ Powder

Version: February 12, 2023

SAFETY INSTRUCTIONS

Radiation exposures from **X-ray** diffraction units can be extremely hazardous. Any part of the body temporarily placed in the beam would receive enough radiation to cause serious radiation burns. X-rays cause the most damage to rapidly growing, undifferentiated cells. Thus, women those are pregnant or suspect that they may be pregnant, should take special care to avoid X-rays exposure.

The maximum permissible dose exposures in one year for radiation workers without limiting the activities are: Whole body 1 mSv ; Lens of the eye 15 mSv ; Skin (1 cm²) 50 mSv. A cabinet X-ray system must contain sufficient shielding and be located so that exposure rates in unrestricted areas do not exceed 0.02 mSv/h and 1 mSv/year.

1 millisievert (mSv)= 100 millirem (mRem).

All unused ports must be securely closed to prevent accidental opening and the X-ray beam must be terminated within the enclosure at all times. All interlocks on the X-ray machine must be functional and in operation for X-ray production. X-ray diffraction machines must be operated in accordance with the basic X-ray safety guidelines in this laboratory instruction.

No individuals other than the operator(s) should be in the X-ray room while the X-ray diffractometer is prepared for operation. During operation all individuals must leave the X-ray room.

The **high voltage** power supply of X-ray machines can be particularly hazardous. Personnel must never tamper with high voltage equipment. Only properly trained personnel are permitted to install, repair, or modify high voltage equipment.



Technical info

Instrument name : PANalytical X'Pert³ Powder
X-ray source : Cu K_α , λ=1.5418 Å
Operating range : 10 – 70 °2θ
Temperature : Room temperature
Humidity : Ambient
Sample amount : App. 10 mg

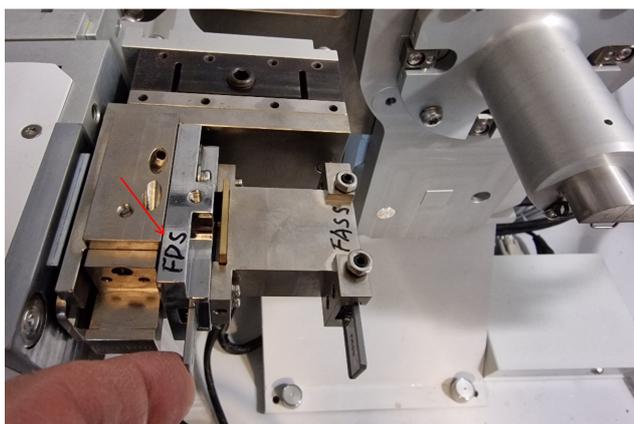


Consult lab supervisor if other sample holders, °2θ, temperature (temperature chamber available) or humidity (humidity chamber available) are required.

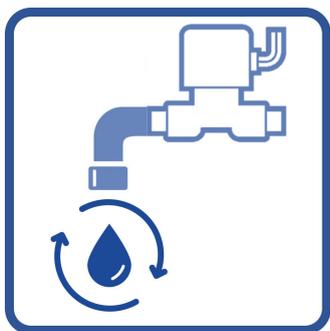
Measuring software data collector is always ON (if off then username and password is needed). When Measurement status showing "No program executing" means no measurement is currently running. Also as an indicator for no measurement running is the status of the MPPC generator is off.

1. STANDARD FLAT STAGE

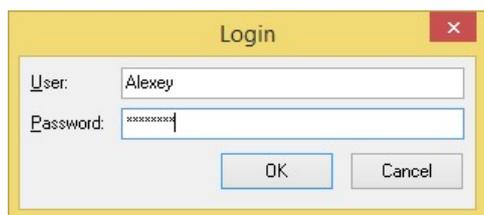
1. Check the filter used. the X-ray source filter (5.0), FASS filter (2°) and FDS (1/4°) filter. You may exchange only the FDS filter to manipulate the detected intensity (reduce or raise the intensity). Do not touch other filters.



2. Check, the door of the XRD must be closed.
3. Turn ON cooling-water circulation switch(s), then turn ON the HT key.

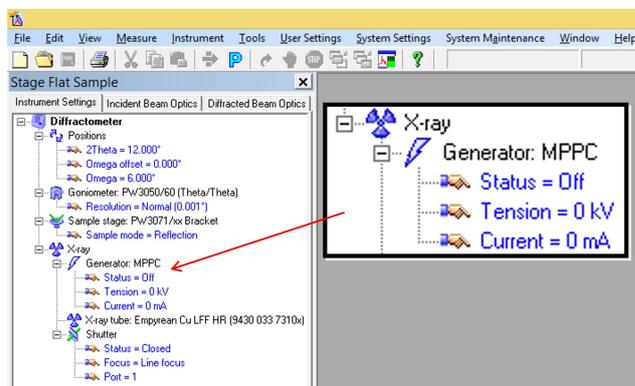
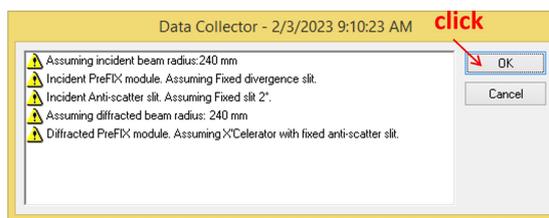
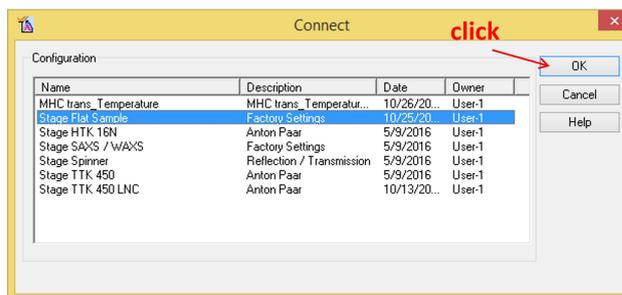
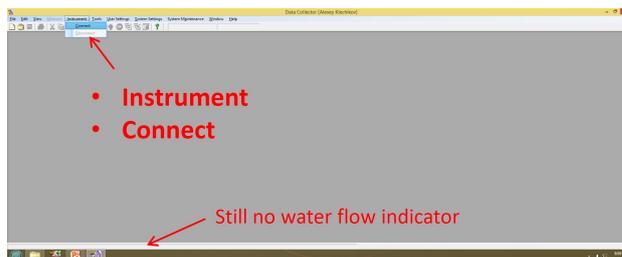


4. Start software  Data Collector, and login, if it was not already on the screen.

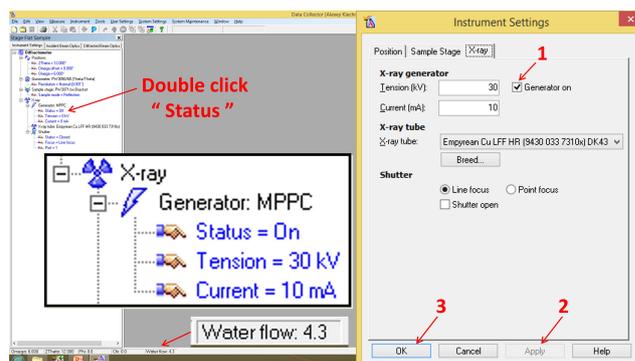


Username: Alexey , Password: Parashut

5. Go to **Instrument** menu, then **Connect**. Do not change anything just press **OK** on the pop-up windows. A column with diffractometer setting commands will appear on the left side.

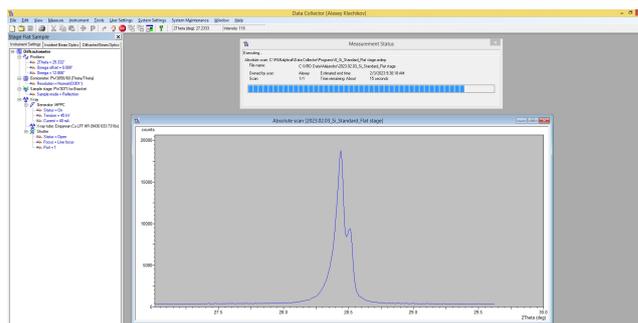
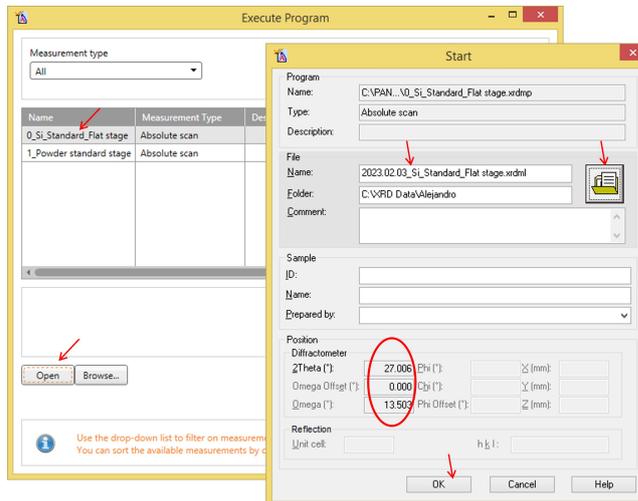
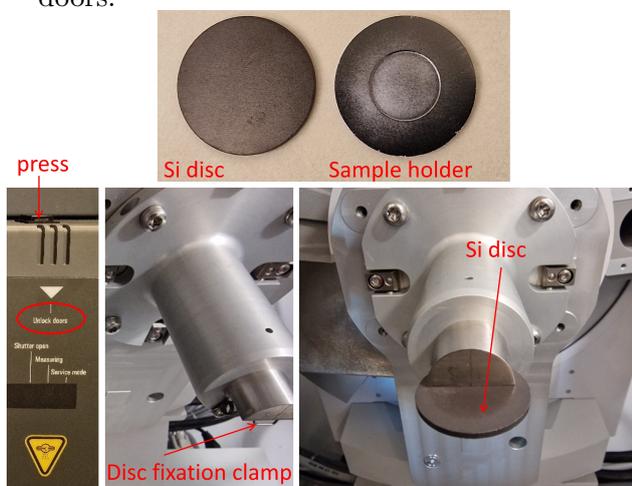


6. Double click on **Status**, click on **Generator on**, then press **Apply**, values 30 kV, 10 mA will load automatically. Wait until the values are confirmed, then press **OK**.



A test measurement before a series of sample measurements is recommended (optional). This is to test the XRD performance in general and test the detected intensity. By using the same FDS filter one must get approximately the same intensity as in previous series of measurements. This test measurement is performed by a Silicon disc which has a double peak located on $2\theta=27^\circ$, intensity about 20000 counts when $1/4^\circ$ FDS filter is used. **The Si disc, or powder sample holder disc are too fragile. Do not drop.**

- Press Unlock doors button to open the XRD doors. Push down the disc fixation clamp and load the Si disc on position. Then lock the doors.

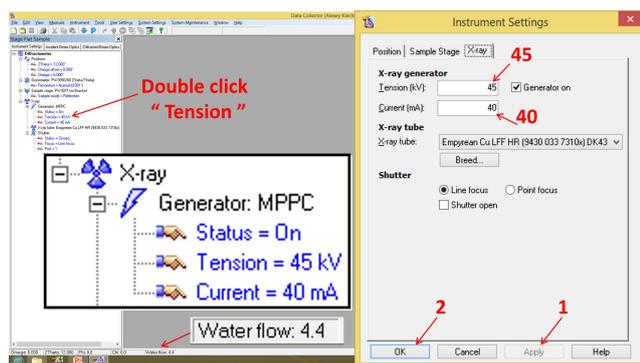


Test or performance measurement is done.

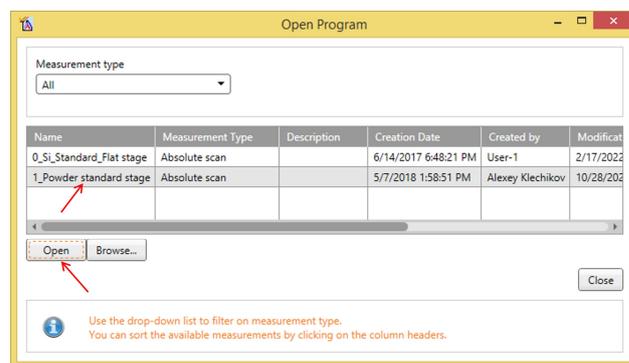
- For measurement on your sample; set tension to **30 kV** and Current to **10 mA**, then **Apply** and **OK**. Unlock the XRS doors. Take out the Si disc and replace by the disc for powders. **Load powder** and lock the doors.



- Double click on **Tension**, then exchange values to **45 kV** and **40 mA**, then **Apply**. Wait until the values are confirmed, then press **OK**.

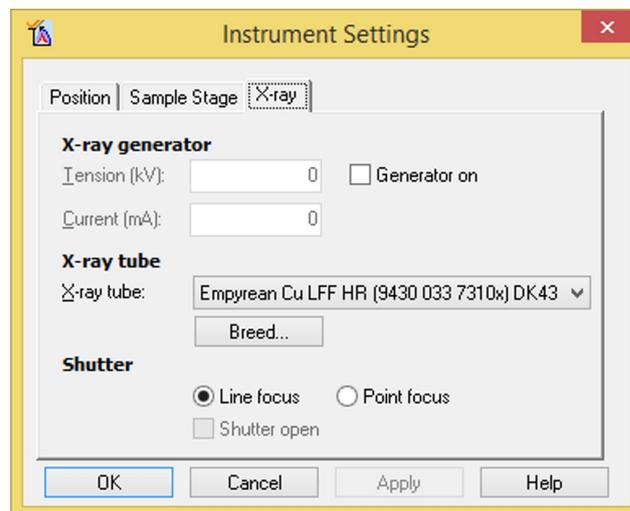
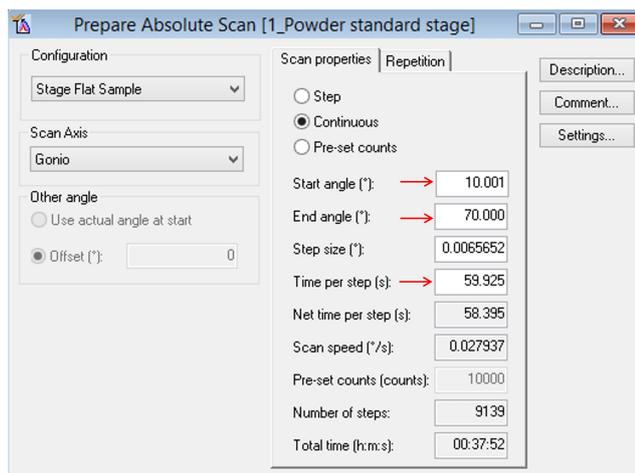


- Go to **Open** menu then select **1_Powder standard stage**, press **Open**.



- Go to **Measure** menu then **Program**, select **0_Si_Standard_Flat stage**, press **Open**. A new window will pop up, there you have to give file name and destination for saving. Here you will also can see the 2θ measurement range 13.5-27. Finally press **OK**. Measurement will start and stop automatically after about 2 minutes.

Here you can change the setting for **Start angel**, **End angle** and **Time per step**.



12. Input **Start angel**, **End angle** and **Time per step** then **close this window** .

For best quality results; the recommended values are: 10 , 70 and 130 s, this will give a total scan time of about 1 hour.

13. To measure do as given in steps 8 and 9. Exchange values to **45 kV** and **40 mA**, then **Apply** and **OK**. Go to **Measurement** menu then select **program**, select **1_Powder standard stage**, press **Open**, give file name and destination for saving, press **OK**. Measurement will start, do not disturb until it is finished.

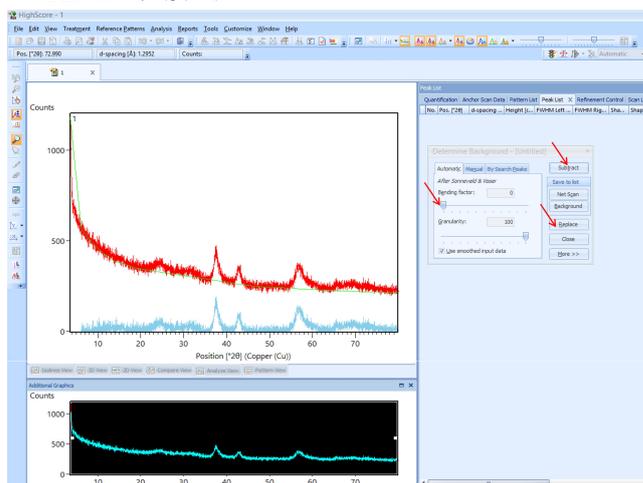
14. To measure on a second sample, do as given in **step 10** (Set tension to **30 kV** and Current to **10 mA**, then **Apply** and **OK**. Unlock the XRS doors. Take out the first sample and replace by the second sample, lock the doors). Run measurement as in **step 13** again (Exchange values to **45 kV** and **40 mA**, then **Apply** and **OK**. Go to **Measurement** menu then select **program**, select **1_Powder standard stage**, press **Open**, give file name and destination for saving, press **OK**).

15. When all measurements are done? Double click on **Status**, unclick **Generator on**, then press **Apply**, values 0 kV, 0 mA will load automatically. Wait until the values are confirmed, then press **OK**.

16. Turn OFF the HT key, then turn OFF cooling-water circulation switch(s). Do not shut down software or PC.

2. DATA ANALYSIS

1. Start software  **HighScore** , no login or password are required.
2. To save your data file as text file to be used in other data processing programs. Go to **File** ⇒ **Open** ⇒ choose your data file ⇒ Open. Then go to **File** ⇒ **Save As...** ⇒ Save. The data will be save as ASC file in the same destination as the original file.
3. To determin background, you have to loads the wanted data in HighScore software. Go to **Treatment** ⇒ **Determine Background...** ⇒ adjust the Bending factor until you satisfied ⇒ **Subtract** ⇒ **Replace** ⇒ **File** ⇒ **Save As...** ⇒ Save.



4. Some other things can be done by this software, like **Treatment** ⇒ **Search Peaks** or **Analysis** ⇒ **Search & Match...**, these can be used to identify the peaks and extract some info about the peaks but the results are not accurate. Better to be done in other software based on your experience.