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Standard Operational Procedure for Powder X-Ray Diffractometer

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Modification Statement

Revised Date	Version	Details of Revision	Revised by	Approved by	Approval Status
2020.03.24	V1.0	Trial	Xiaohe MIAO		

目 录

1. Purpose

This SOP is to standardize the operation procedure of Powder XRD Bruker D8 Advance, to ensure of a proper and regulating use

2. Scope

This SOP is applicable to all users operating Bruker D8 Advance Powder X-Ray Diffractometer.

3. Responsibilities

3.1. Users: strictly follow the instructions and immediately report to staff if any troubleshoot.

3.2. Staff: authorizing users who pass the training and qualification and standardizing their operations.

4. Safety regulations in X-Ray laboratory

4.1. All personnel entering the lab should be aware of potential X-ray radiation hazard. It's mandatory to take the HSE lab safety and radiation trainings.

4.2. All personnel entering the lab should be familiar with chemical safety knowledge, water/gas knowledge.

4.3. Strictly adhere to the safety regulations in XRD and notice the alerts. Lab coat is required in XRD lab.

4.4. Independent operation is permitted only after operational training and qualification and following SOP. It's illegal to use the facility without training. Operations beyond SOP are prohibited. It's not allowed to use the machine under the circumstance of modifying hardware or removing safety circuits.

4.5. It's strictly forbidden to transfer data with USB and get network connected.

4.6. Users should operate in strict accordance with SOP; staff should make frequent inspection to correct violations in time and avoid safety risks. Tidy up the working bench and properly make recorder after experiment.

4.7. Gloves are mandatory during sample preparation and transferring. No bare hands for sample holders. No gloves for keyboard, mouse and doorknob.

4.8. All users are personally responsible for maintaining working bench neat and organized. Tools should be returned to original containers. Smoking, eating, drinking, and storage of food are not permitted in the lab.

4.9. Be strictly subjected to University policies. All lab waste must be segregated and disposed into the appropriate waste container accordingly, such as toxic trash, reagents, utensils and sharps.

4.10. Please inspect electricity prior to leaving the lab and close the door.

4.11. Please report to the staff if you find anything wrong with the system, Do Not try to repair by yourself.

4.12. Users will be responsible for violations and penalties will be applied.

5. Usage policies of X-Ray facility

5.1. Usage policies of Bruker D8 Advance Powder XRD

This equipment is obedient to university policies for management of large-scale facilities with the principle of "concentrated investment, unified management, public open, sharing resource", to support academic research and teaching in Westlake University and cooperative R&D in industry. It's proposed appropriate charge based on machine usage. The equipment will be open to the whole society when it meets the internal needs from Westlake researchers.

Training and service of Bruker D8 Advance Powder XRD is classified into 5 ranks:

1) Training: requests from user and scheduled by staff. The training involves sample preparation, introduction of lab regulations, basic theory of PXRD, equipment configuration, sample mount procedure, SOP, software operation of Diffrac. Management and EVA, data collection and processing.
2) Independent operation (Primary): sample preparation and independent operation for data collection wide-angle diffraction, as well as data process and upload.
3) Independent operation (Intermediate): sample preparation and independent operation for complicated experiments, including low-angle diffraction and various-segment data collection as well as data process and upload.
4) Independent operation (Advanced): sample preparation and independent operation of data collection for Rietveld refinement, as well as data process and upload.
5) Service: staff analyze samples submitted by users with sufficient information and measurement requirement.

This equipment can be reserved on "LIMS" via the link below and necessary information

should be provided:

https://share.westlake.edu.cn/lims/!equipments/equipment/index.455.reserv

5.2. Reservation policy

To sufficiently use the machine to serve Westlake researchers, XRD lab has proposed 24/7 reservation policy. Herein, reservation can be made 1 day in advance, even during weekends, while 2 days in advance during public holidays, such National Day, Chinese New Year.

It's advised to adhere to reserved slot strictly, avoid of wasting machine hours. Communicate with the next user when overtime with the support of staff. Free cancellation can be made 8 hours in advance. In case of no show without no notice, the machine access will be suspended for 1 month.

Reservation hours		Reservation slot per person	Measurement content
Working hours (Monday~Friday)	09:00 am ~ 17:30 pm	Minimum 30 min each time	Independent operation, service
Non-working hours (Monday~Friday)	18:30 pm ~ 8:00 am	Minimum 30 min each time	Independent operation
Non-working hours (weekends & public holidays)	09:00 am ~ 8:00 am	Minimum 30 min each time	Independent operation

1) Internal users are permitted to operate the machine after qualification;
2) Please keep accurate records for experiments as well as machine status after experiment;
3) It's inhibited to modify, disassemble or adjust any parts of equipment.
4) Please immediately report to the staff if anything wrong with the equipment is found, and request for a prompt repair.
5) User's research group should afford the repair charge for instrument damage due to his/her individual mis-operation.
6) Please contact the staff for any operations beyond SOP.
7) Do Not delete raw data on the control computer, otherwise contact the staff.
8) Uploading raw data via NAS drive and downloading to personal computer for further data process instead of copying data by USB. Raw data will be remained on control computer for 2 months (temporarily, and can be extended if there is sufficient capacity to keep it.

9) All users are personally responsible for maintaining working bench neat and well organized. Samples will be taken away from XRD lab, otherwise discarded without notice.

5.3. Evaluation policy

Researchers like professors, graduates, postdoctoral fellows are eligible for trainings, scheduled by the staff, which contain three parts.

Part I: introduction of XRD lab safety regulations, equipment configuration and basic theory.

Part II: operational training, including sample preparation, the hand-on practical operation according to SOP and basic data process.

Part III: trainees passed the operational evaluation should practice at least twice within a week with inspection of XRD staff.

The staff consider that the trainee's operational skills achieves at relevant level, should authorize trainee with qualified access. Mis-operation by users who should afford the repair charge, also be disqualified and be charged double for repetitive training in future.

Notes: Users should close sessions of Part I and Part II of the training within 1 week, or else apply for another training. Qualified users guarantee about independent operation at least once per month. And it will be failed to reserve the machine if the usage frequency of the equipment is lower than once per month.

Requirements for trainees:

(1) Understand the theory, configuration and function of Powder XRD, strictly obey regulations, check the status and report problems.

(2) Proficient in operating Powder XRD and data format conversion; strictly follow SOP to avoid troubleshoots. Make proper record for Powder XRD after usage.

5.4. Troubleshoot report

1) Users are requested to report any problems related to the instrument or the facility (power, water, AC …) in the logbook AND contact the staff.

2) For reporting the troubleshoot, taking a screenshot and save in Error Report folder named as "PI name-user-sample identity-troubleshoot time", also specify your observation briefly in comment.

6. Standard operation procedure for Bruker D8 Advance PXRD

6.1. Introduction

- Model：Bruker D8 Advance
- Origin：Germany
- Basic information：a novel X-ray diffraction tool enabling high-throughput data collection for phase identification and Rietveld refinement with Bragg-Brentano geometry

  Equipped with Cu source, 90-seat autosampler and energy resolved LynxEye XE-T detector

6.2. Specifications:

1) 3 kW Cu source with auto air scatter, sealed tube, /2 scan
2) rotational sample stage;
3) Automatic DBO optics;
4) 90-seat autosampler;
5) Accuracy: 0.0001º; minimum step size: 0.0001º
6) discrimination of K and fluorescence by energy-resolved (380 eV) LynxEye XE-T detector

The configuration of Bruker D8 Advance Powder XRD in Figure 6-1:

Figure 6-1 Configuration of Bruker D8 Advance Powder XRD

Location: Room 112, Level 1 Building 4, Yunqi Campus

Contact： Dr. Xiaohe MIAO, Tel: 0571-87310229 Email: miaoxiaohe@westlake.edu.cn

6.3. Sample requirement

6.3.1 Non-toxic, incorrosive samples;

6.3.2 Dry and air-stable; homogeneous particles with size of ~ 20 m, ~ 0.5 g;

6.3.3 Flat and neat sample surface, block, pallet or fiber-like samples. And films deposite on substrate with thickness ≤5 mm, diameter ≤2 cm;

6.3.4 Specify the analysis requirement, for instance, 2 ranges;

6.3.5 Specify the storage condition of samples, like recycle, freezing, dry, lucifuge etc.

6.4. Sample preparation

6.4.1 Powder sample preparation;

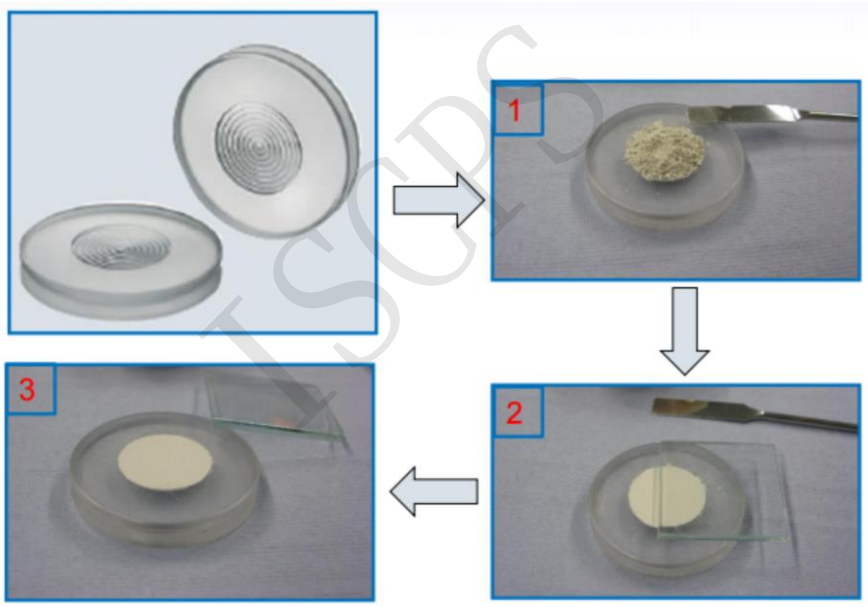


Figure 6-2 Sample preparation for regular powder samples

6.4.2 Small-amount sample preparation: use low-background holder;

Figure 6-3 Zero background holder

6.4.3 Irregular shaped samples: use deep-well holder and supported with clay at the bottom of sample;

Figure 6-4 Deep-well holder

6.4.4 Transfer sample holder and mount to the sample stage.

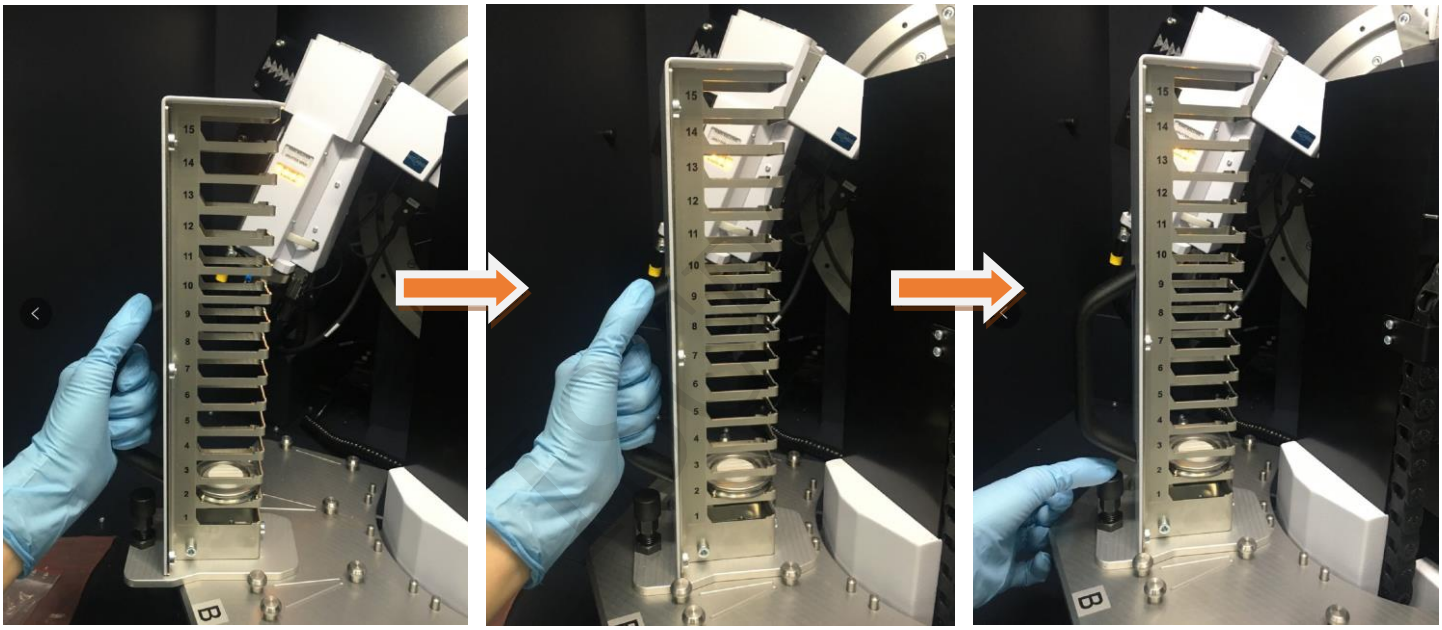


Figure 6-5 Sample tower mount

6.5. Power-on procedure

6.5.1 Switch on the water chiller and set temperature to 26 ± 2 ℃ and pressure 0.45 MPa. Please report to staff if over these values.

Figure 6-6 T and pressure for water chiller

6.5.2 Turn the handle from “O” to “I” at left side of the machine, and turn on the green button, so that trigger for initialization; subsequentially, the indicator on upper-left control panel turns as white light to “I”; press the top button to power on high voltage. Conditioning the tube is required for long-time shut down and the top button presents as  after conditioning. So the machine is ready for use.

Figure 6-7 Power buttons

6.5.3 Launch the software DFFRAC.Measurement Center and select “Lab Manager” with password.

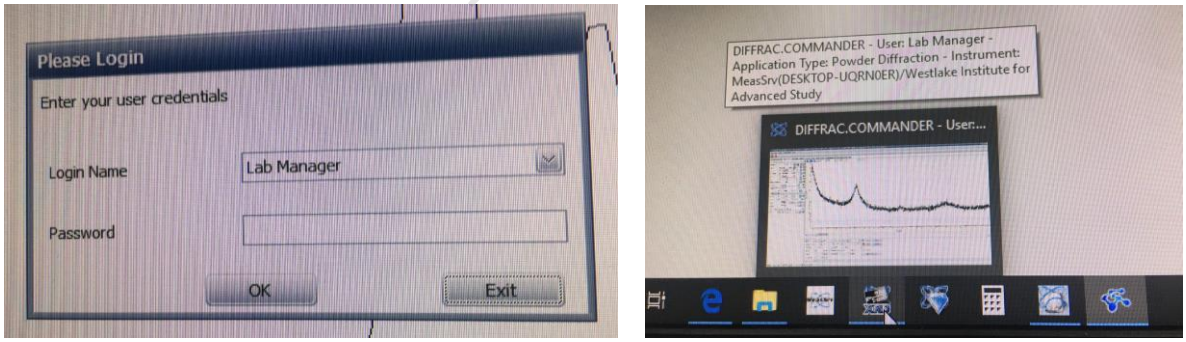


Figure 6-8 Launch DIFFRAC.SUITE

6.5.4 On “Commander” tab, check all requests and click “Initialize”, to initialize all drives. (Initialization is always required automatically when power on the machine. No initialization presents as “!” while proper initialization shows  .

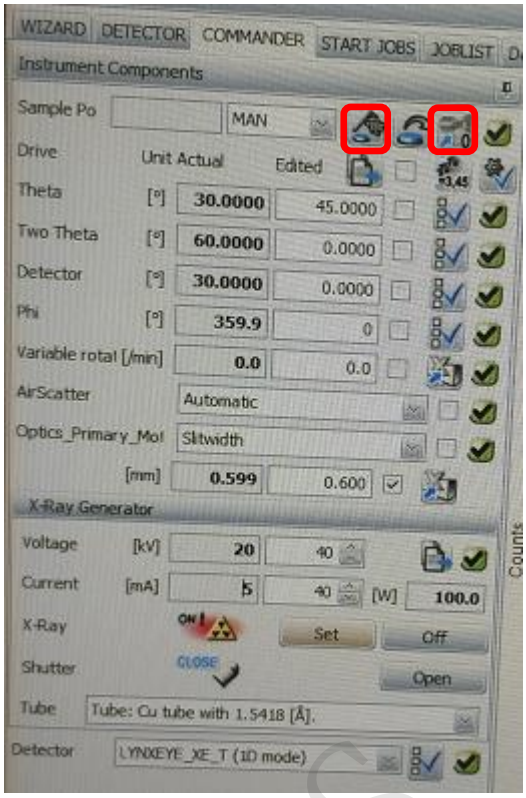


Figure 6-9 Initialize drives

6.6. Collimation

Standard sample corundum is used to measure 2 of 34.5°~36.0°, step size 0.01°/step, and Kα1 peak locates at 35.149°, with deviation of± 0.01°; please contact XRD staff if the deviation is beyond above range.

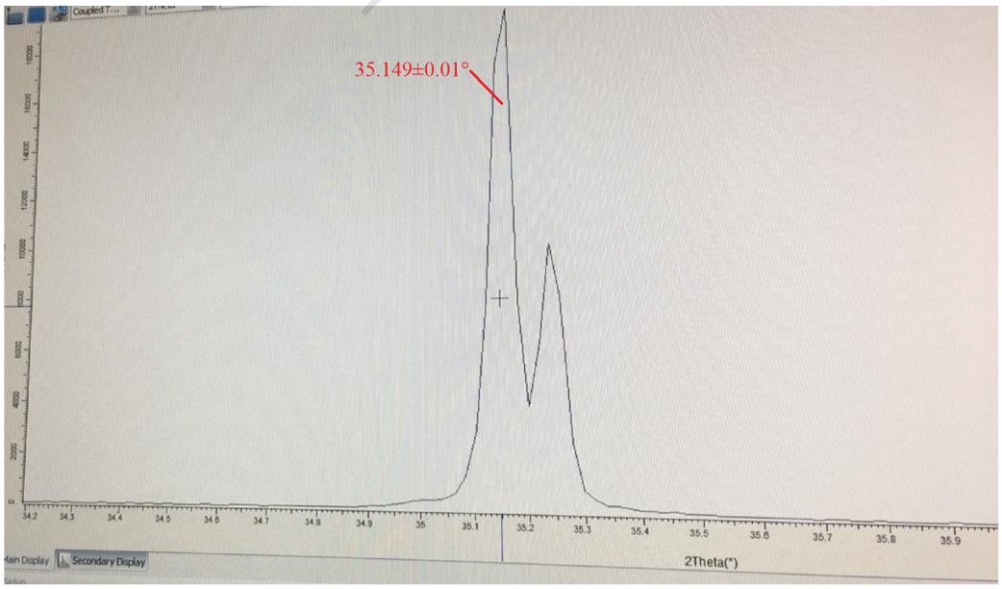


Figure 6-10 Reference peak

6.7. Single sample measurement

6.7.1 Edit sample position: pick up B02 from “Sample Po” if the sample sits on 1B02, and click  , to load the sample to stage.

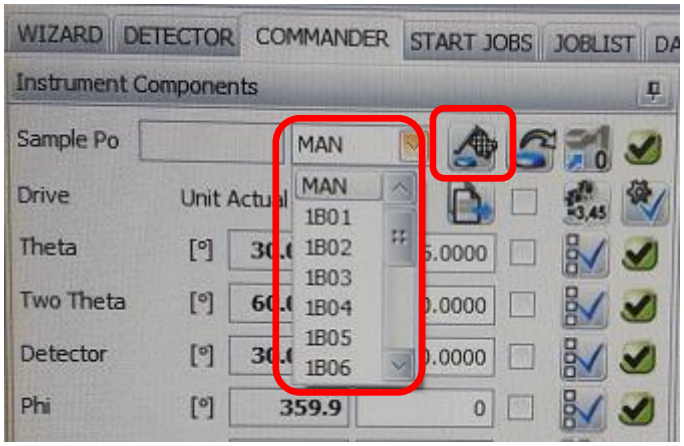


Figure 6-11 Define sample position and sample tower

Click  to unload the sample and return it to the sample tower.

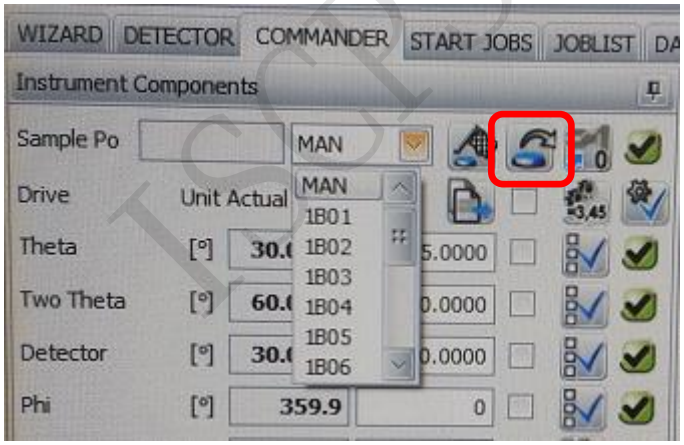


Figure 6-12 Dismount sample

6.7.2 Set rotation speed to improve the preferred orientation.

Figure 6-13 Set rotation speed

Attention: samples with low density and quality are prone to glide easily, so set rotation speed to 0.

6.7.3 Set Airscatter to Automatic mode.

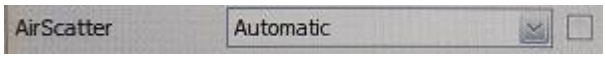


Figure 6-14 Define AirScatter mode

6.7.4 Settings of motorized divergent slit:

2 beyond 10°, set Slitwidth 0.6mm, and check it;

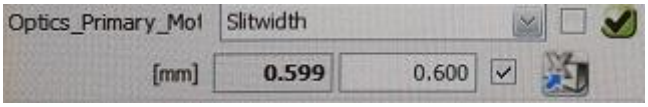


Figure 6-15 Define Slitwidth

2 starts from 5°, set Slitwidth 0.4 mm which 2 starts from 1°, set it to 0.2 mm.

6.7.5 Check tube parameters: Cu source with wavelength 1.5418 Å。

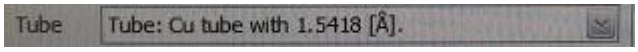


Figure 6-16 Define X-ray source

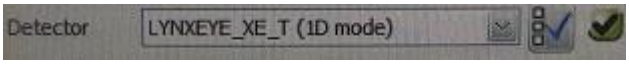
6.7.6 Set LynxEye XE-T to 1D mode: 

Figure 6-17 Define detector mode

6.7.7 Scan parameters：①2 (Start), ②2 (Stop), ③ step size (Increment), ④exposure time (Time), ⑧detector opening (PSD opening).

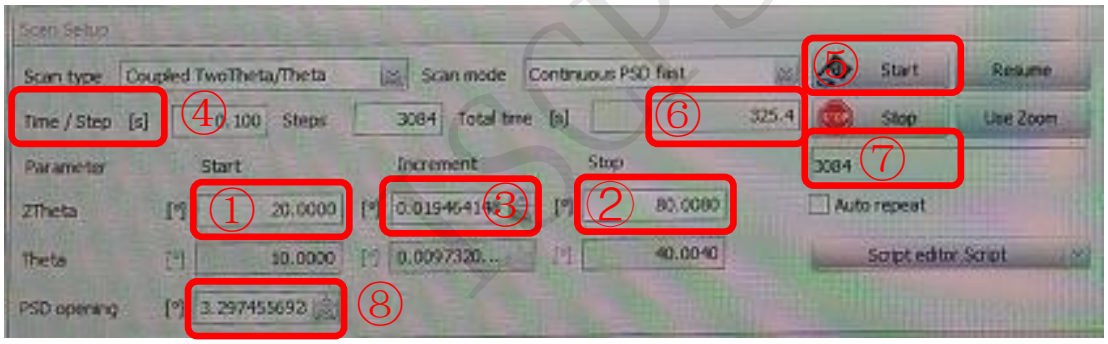


Figure 6-18 Define scan parameters

6.7.8 Complete settings and ensure the lead door is properly closed prior to launch the measurement. Click⑤Start 

, and the acquisition duration is shown in ⑥; the ending time will be presented in ⑦ when starts the measurement.

6.7.9 Save data to assigned folder in D disk after experiment:

Figure 6-19 Save data

6.8. Batch sample measurement

6.8.1 Method setting: click Wizard→ New Wizard →XRD →OK.

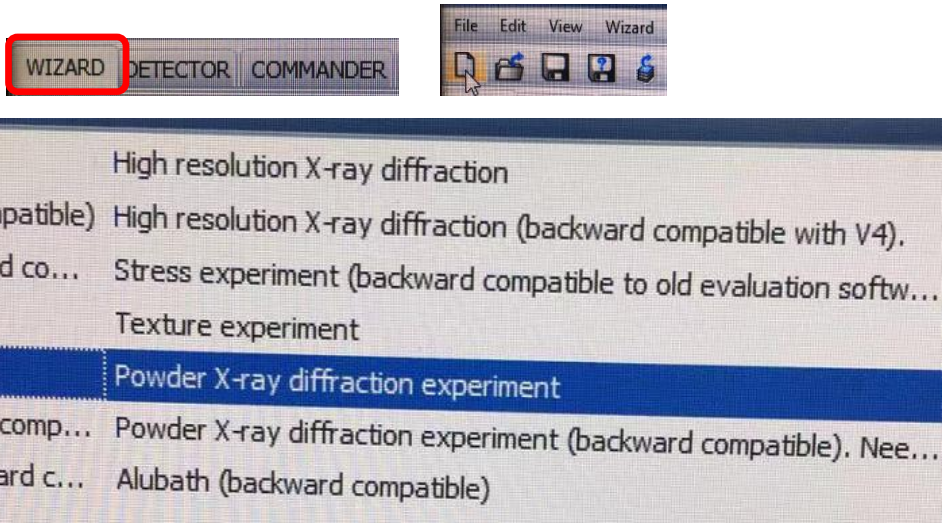


Figure 6-20 Launch Wizard

6.8.2 Hardware settings

6.8.2.1 Set tube power to: power 40 kV, current 40 mA;

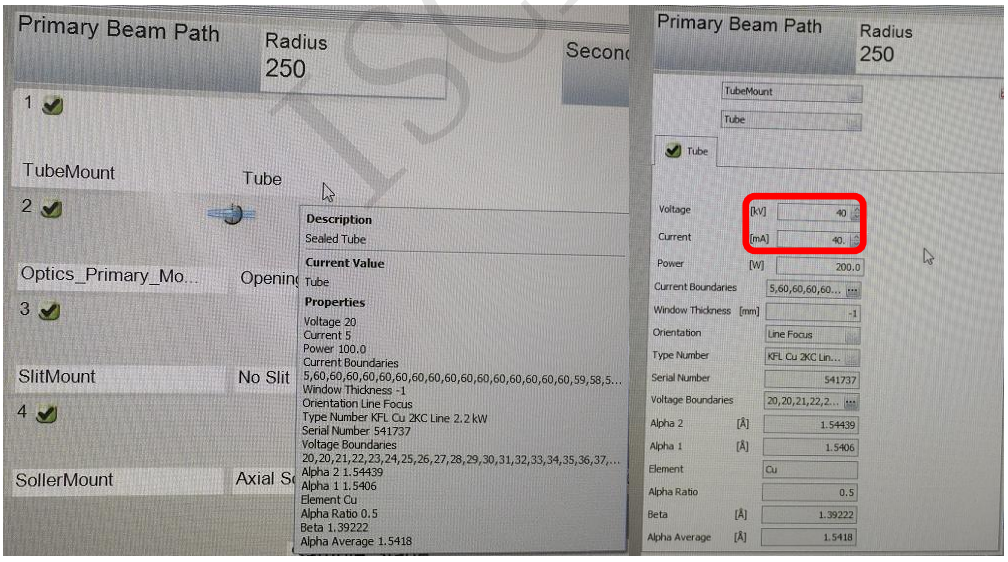


Figure 6-21 Set Voltage/Current

6.8.2.2 Set Optics Primary Mo: set Opening degree as 0.6mm；

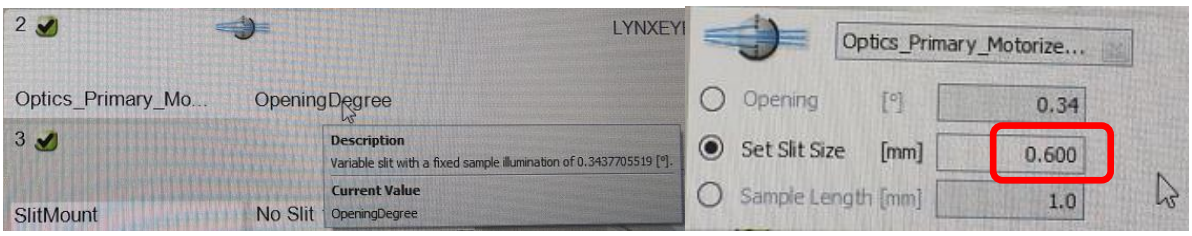


Figure 6-22 Define Slit Size

6.8.2.3 Check LynxEye XE-T details:

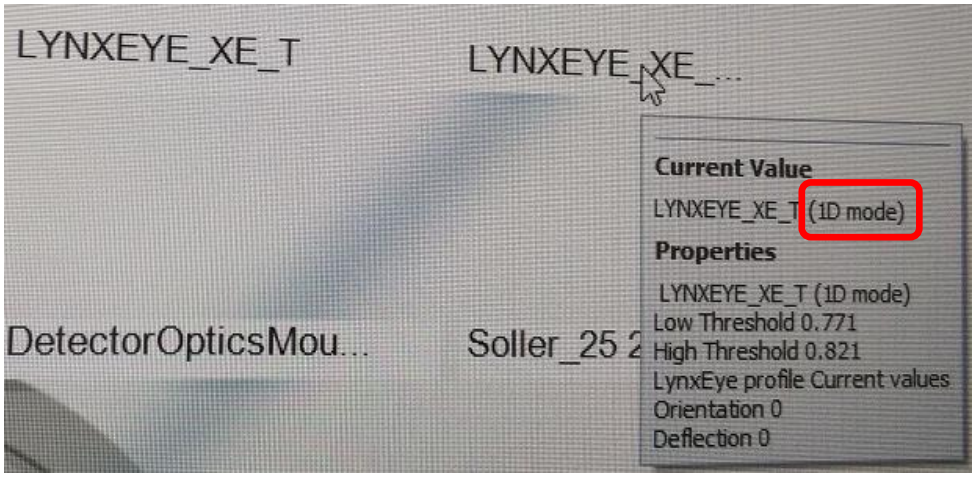


Figure 6-23 Define detector mode

6.8.2.4 Set sample stage details: click “AirScatter” and put 15 rad /min into Rotation if necessary; and set Automatic for AirScatter:

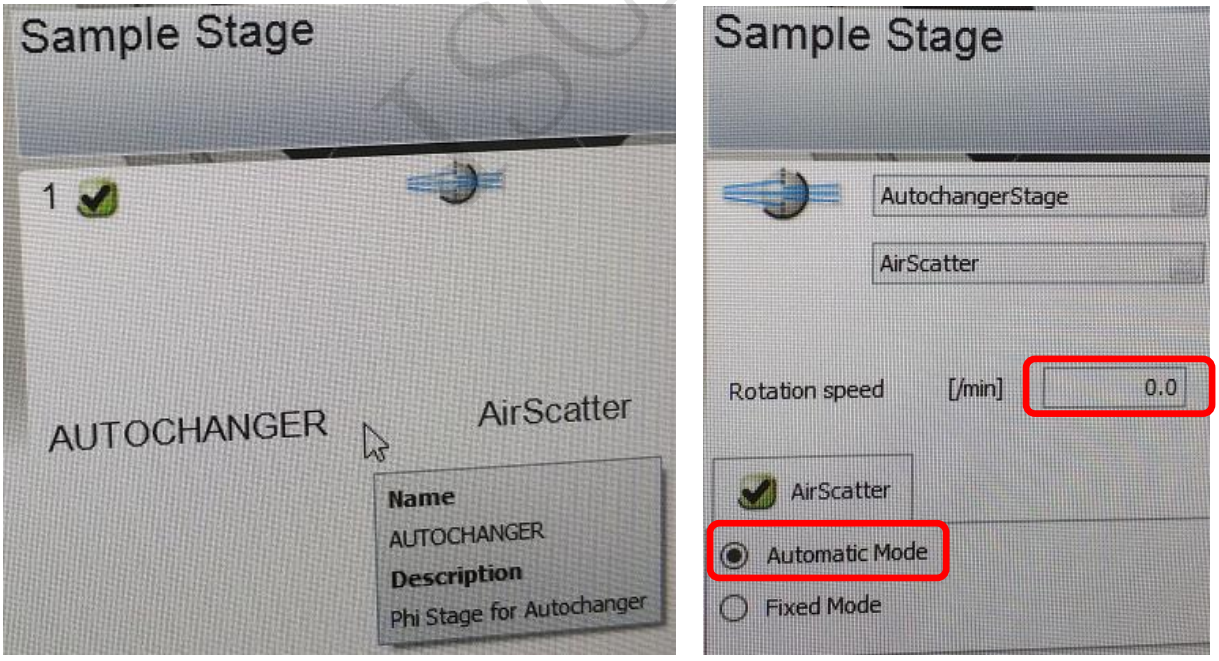


Figure 6-24 Define AirScatter mode

6.8.2.5 Straight forward with “next”， till “DAVINCI” settings and click on. Fill in blanks if necessary;

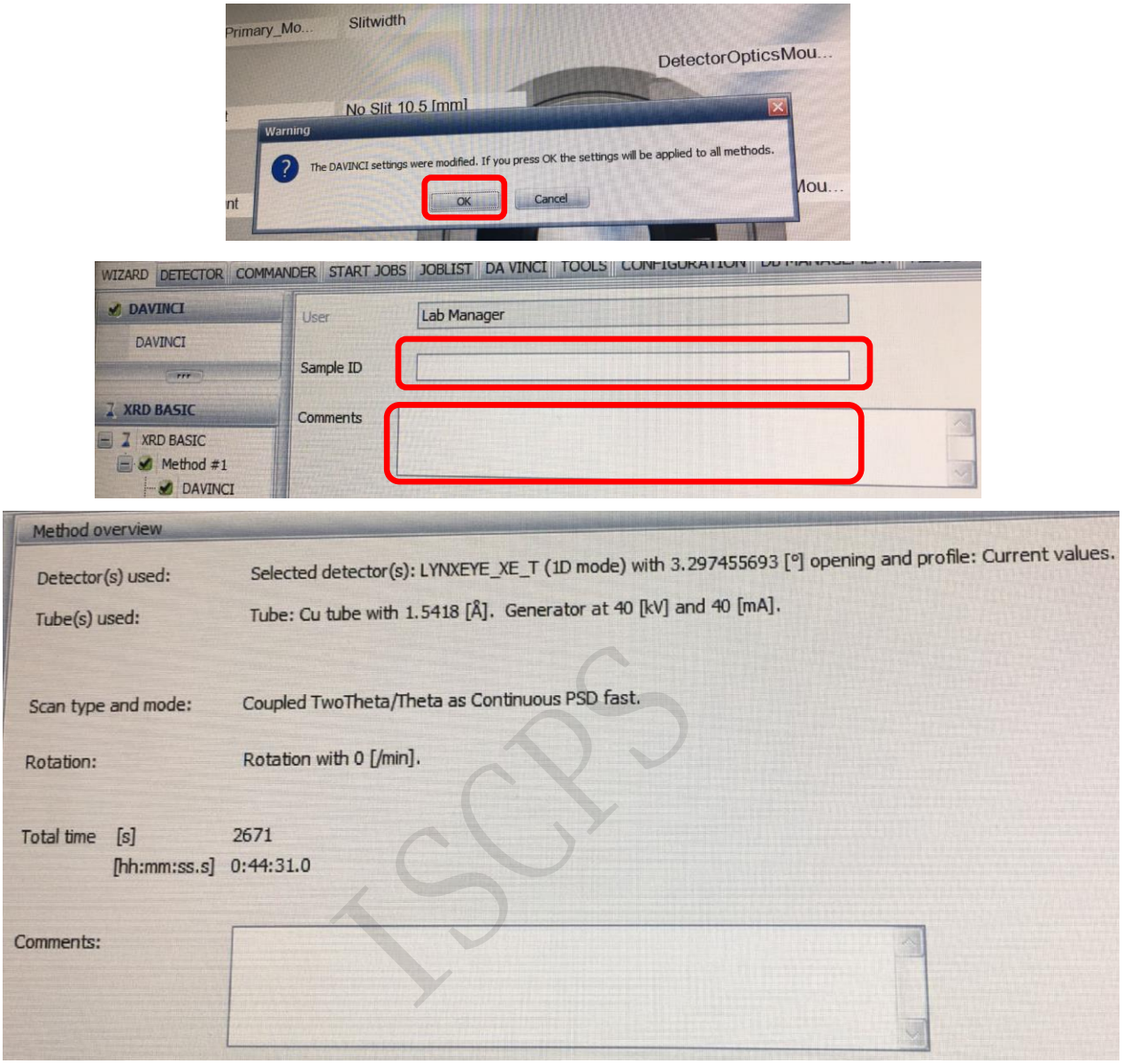


Figure 6-25 Method summary

6.8.2.6 Select “Method” in navigation pane and set “XRD setup”: ①2 (Start), ②2 (Stop), ③step size (Increment),④exposure time *(Time) and ⑤detector opening (PSD opening);

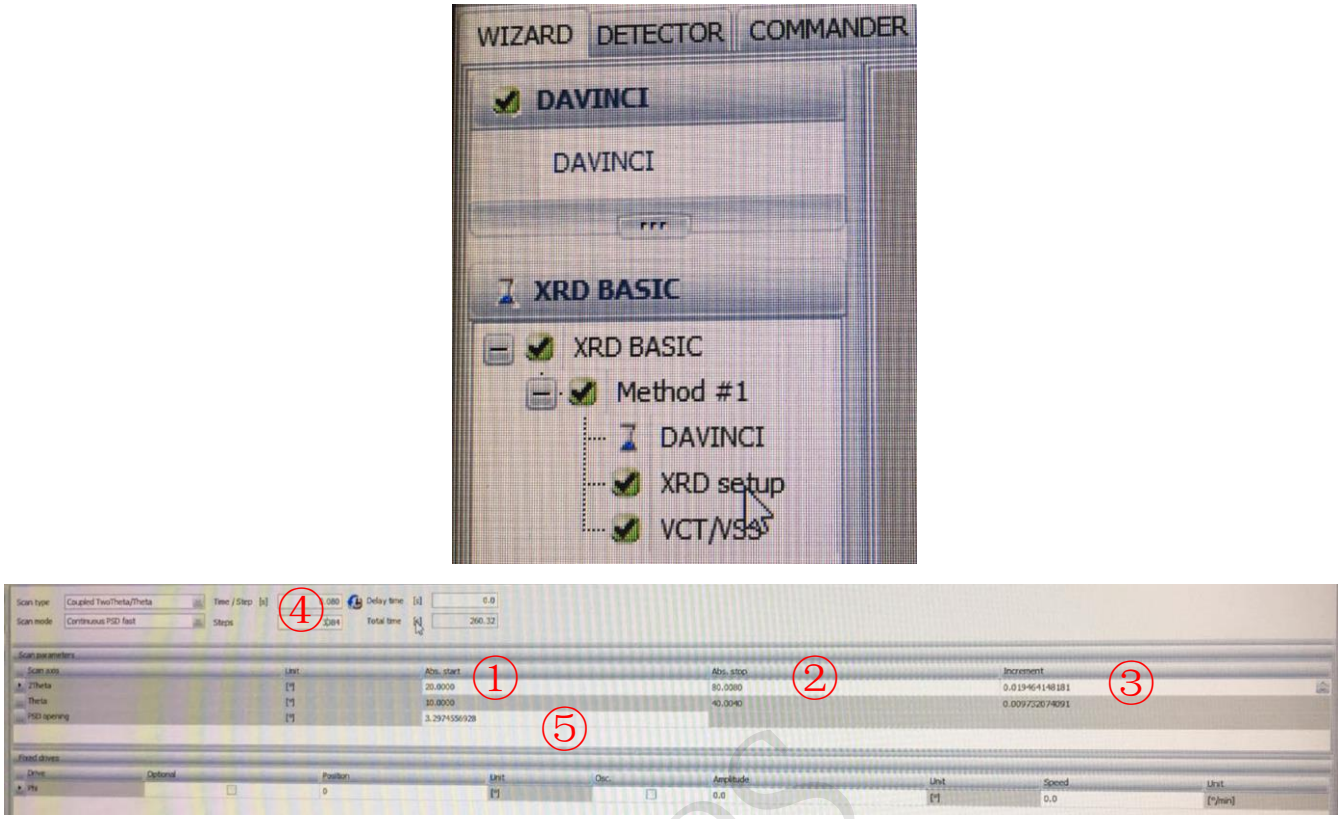


Figure 6-26 Scan parameter set up

6.6.2.6 设置结束后，最后在 Summary-Methods 里确认所设置的参数：

6.8.2.7 Move on to “Summary” in navigation pane to check all the settings and save the method to the assigned folder.

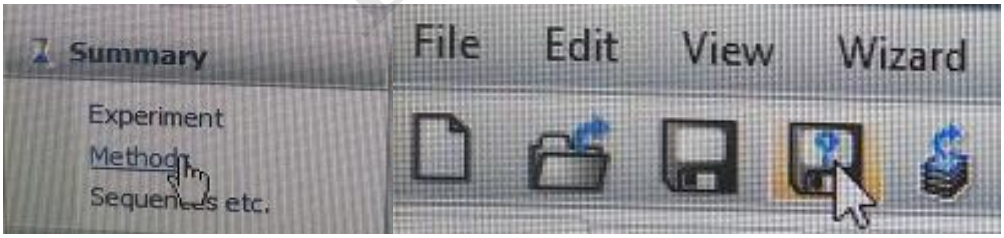


Figure 6-27 Save method in Wizard

6.8.2.8 Batch measurement: click “Start Jobs”, input Sample position, (Sample ID), choose experiment method (Experiment Name)and accordingly assigned the path for result files (Result File Name).

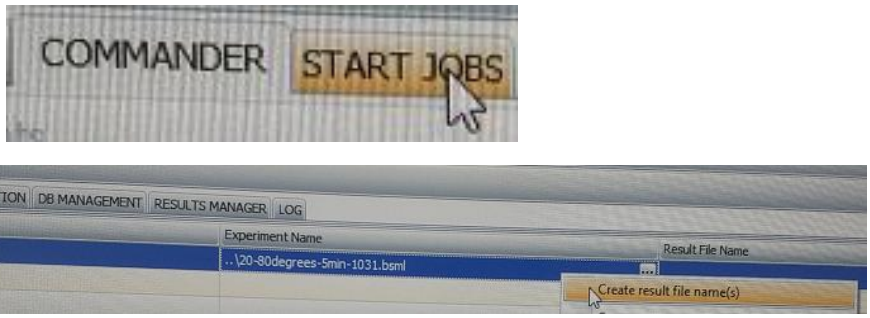


Figure 6-28 Loading method to "START JOB"

6.8.2.9 Click "Start Jobs" when all values are properly set;

Figure 6-29 "Start Job"

6.8.2.10 During data acquisition, the completion time of current experiment can be monitored in "Job list" tab:

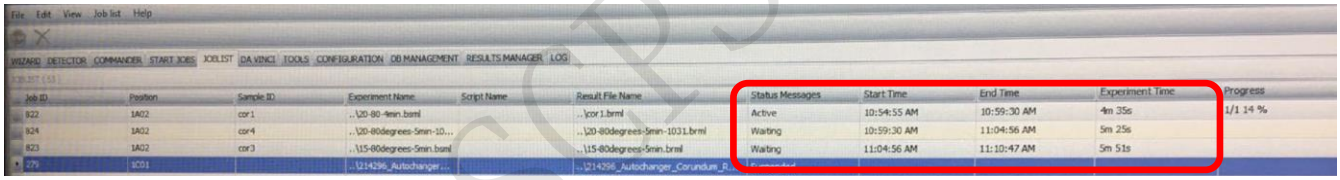


Figure 6-30 Tract job process

6.8.2.11 File format conversion

Launch File Exchange 7.3 (shown in Figure 6-31), open the interested files in left column of "Source", and navigate to the same folder in right column of "Target". Highlight these files in "source" column and click "F9 Convert" at the bottom, and the converted file is presenting in "Target" column.

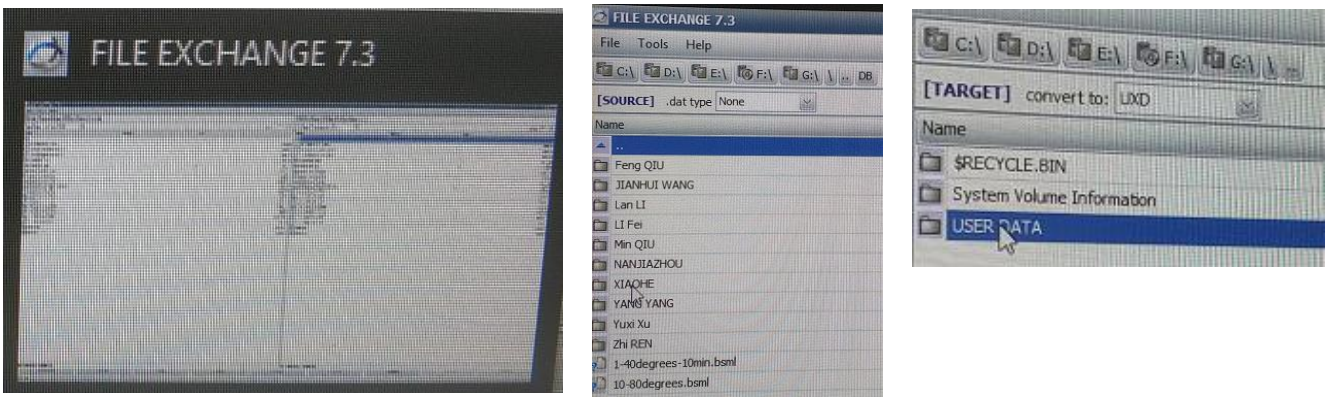


Figure 6-31

7. Relevant supporting documents

Q/WU FLHR001 format file

8. Logbook

Properly make record on Logbook V1.0 for Bruker D8 Advance PXRD.

Logbook

Date	User	PI	Sample ID	Sample details: composition and number	Test type		Sample recycle	Sample status	User contact	Comment
					Service	Independent operation				

Attention: 1) Launch the experiment only when the instrument is under good condition. Once launched, it's regarded as good condition; 2) In case of any problem, please contact staff.