IMSERC User Manual for STOE StadiP

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INTRODUCTION

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for ‘routine’ data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/crystallography-instruments.html. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at http://imserc.northwestern.edu/crystallography-instruments.html by pressing on the ‘User manual’ button. If while using the system, something happens that you do not understand, please stop, and get help. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/about-policies.html and the Crystallography specific policies at http://imserc.northwestern.edu/crystallography-policies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment
- X-Ray Safety

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room BG70. Protective eyewear is required in this room, and gloves should be removed when using the computer. Gloves are located by the Olympus optical microscope in room BG62.
DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor’s group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied in your supervisor’s group folder on ‘imsercdata.northwestern.edu’ under ‘xrd/StadiP’ every 15 minutes. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

SOFTWARE

Data reduction and analysis can be performed with ‘WinXPow’. Software is installed on the instrument computer. For offline analysis, please use any the following resources:

- For registered IMSERC users, licensed to IMSERC software can be downloaded from ‘imsercdata.northwestern.edu’ under the folder ‘public/Crystallography/Stoe/STADI-MP-P’. Software is available for Windows only. Please follow instructions under ‘Data Access’ at http://imserc.northwestern.edu/about-general-faq.html#data on how to connect to the ‘public’ folder.
- Software is installed on the communal computers located in the area outside room BG51.
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore.

DEFAULT INSTRUMENT STATUS

The default measurement mode of StadiP is room temperature measurements in high-throughput transmission geometry with flat disc holders and pure CuKα1-radiation. Please notify the right staff member well in advance if you would like to run an experiment in a different mode than the one listed above, and you are not trained to perform the required mode-switch. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/crystallography-instruments.html.
The default working condition of StadiP is as follows:

1. Computer screen is by default deactivated. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore.
2. The default ‘StadiP’ user account should be logged in. In case the computer was restarted, the password for the ‘StadiP’ account is.
3. Acquisition software should be running. Leave the acquisition software open when you are done with the measurement.
4. Green light on the safety beacon at the top left corner in the enclosure must be on (circle at the top left corner of figure 4).
5. X-ray generator (located in the middle of the instrument rack below the benchtop, circle at the bottom of figure 4) should be on with voltage and current values of 40 kV and 40 mA (figure 5), respectively.

When you are done with your measurement, please remember to:

1. Leave the acquisition software running.
2. End your reservation in NUcore.
3. In case transmission holders were used, do not unscrew all the way the screws that fix the metallic mask onto the holders, one turn counterclockwise is sufficient to release the metallic mask from the holder.
4. Leave lab tables clean and tools/accessories organized.
5. Return any unused metallic masks.

If there is an error or problem with the instrument which is not addressed under the ‘Troubleshooting’ section, please report the issue by following at least one of the steps below:
1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the ‘Stop’ sign near the instrument computer to notify users immediately after you. ‘Stop’ signs are located on the shelf above the computers in BG51.

2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the ‘Stop’ sign near the instrument computer.

3. Contact a staff member for instructions.
GENERAL STEPS FOR SAMPLE PREPARATION AND PACKING

Please follow these general steps as discussed during your training sessions. Pack your powdered sample into the appropriate holder depending on the type of measurement. In more details:

- Use the flat plate disc holder in transmission mode for qualitative analyses on powder sandwiched between acetate, polyimide, or mylar foils (high throughput option up to 30 flat plates). Use the appropriate foil based on the details discussed during training. Polyimide and acetate foils are available to all users and supplies are located on the bench near the instrument.
- Use glass capillaries in Debye-Scherrer (transmission) mode for samples in liquid or air sensitive samples. A high throughput option for measuring up to 10 capillaries is available. Capillaries can be either ordered by your group or provided by a Crystallography staff and billed as an accessory under your NUcore reservation.
- Use polyimide (or glass) capillaries in Debye-Scherrer (transmission) mode for quantitative analyses (e.g., Rietveld). Capillaries can be either ordered by your group or provided by a Crystallography staff and billed as an accessory under your NUcore reservation.
- Use glass capillaries (Pyrex, borosilicate, quartz) for high temperature experiments with any of the furnaces. Capillaries can be either ordered by your group or provided by a Crystallography staff and billed as an accessory under your NUcore reservation.

A. PACK SAMPLE IN A FLAT DISC TRANSMISSION HOLDER USING POLYIMIDE OR ACETATE TAPE

There are two different sizes of flat disc metallic masks for transmission experiments, i.e., 3 mm and 8 mm metallic masks. Use either size based on the availability of your samples. Note that the size of the X-ray beam is as wide as the 8 mm metallic mask, therefore use of a 3 mm metallic mask will result in an approximate 50% reduction in intensity in respect to the intensity obtained with 8 mm opening. Packing process of the sample into the metallic masks is the same regardless of the size of the metallic mask. Use the following steps to pack your powder into the holder for flat disc transmission measurements using polyimide or acetate tape with adhesive:

1. Select the appropriate size metallic mask based on the availability of your sample (figure 1).
2. Stick the polyimide or acetate tape on one side of the metallic mask (figure 2).
3. Flip the metallic mask with the side that has the tape facing down and pack your powder in the opening of the metallic plate (figure 3).
4. Stick the polyimide or acetate tape on the side of the metallic mask that is not covered with tape. Powder sample should be sandwiched between the metallic mask and two layers of tape (figure 4).
5. Take one of the black holders located in the plastic container by the instrument (top left of figure 5)

6. Place the metallic mask into the holder by aligning the half holes of the mask with the screws of the holder (bottom left of figure 5)

7. Rotate the mask by approximately 45 degrees and gently tighten the two screws (right of figure 5)

B. PACK SAMPLE IN A FLAT DISC TRANSMISSION HOLDER USING ACETATE FOILS

There are two different sizes of flat disc metallic masks for transmission experiments, i.e., 3 mm and 8 mm metallic masks. Use either size based on the availability of your samples. Note that the size of the X-ray beam is as wide as the 8 mm metallic mask, therefore use of a 3 mm metallic mask will result in an approximate 50% reduction in intensity in respect to the intensity obtained with 8 mm opening. Packing process of the sample into the metallic masks is the same regardless of the size of the metallic mask. Use the following steps to pack your powder into the holder for flat disc transmission measurements using acetate foils without adhesive:

8. Place one acetate foil into the black holder and under the screw heads of the black holder (figure 8)

9. Place your powder sample on top of the acetate foil and in the middle of the black holder (figure 9)

10. Place one acetate foil on top of your powder and under the screw heads of the black holder (figure 10)

11. Place the appropriate size metallic mask on top of the acetate foil and clamp both acetate foils and powder against the black holder. Rotate the mask approximately 45 degrees and gently tighten the two screws (figure 11). Sample powder should be well packed between the two acetate foils (figure 12, back side of holder)
GENERAL STEPS FOR DATA ACQUISITION

The software interface for measuring under different modes, such as transmission, reflection (available on StadiMP only), and Debye-Scherrer, is very similar regardless of the mode selection. The general steps for data acquisition described in this section illustrate the standard workflow of the software. The various sections listed in this user manual provide more details about each specific mode and its settings. Please follow these general steps as discussed during your training session:

1. Pack your powdered sample into the appropriate holder as described under the ‘General steps for sample preparation and packing’ section
2. Verify that instrument is idle by checking that the safety light (circled in figure 2) in the beacon is green. Start your reservation in NUcore to have the screen of the computer turned on
3. Start the ‘WinXPOW’ software (icon on the desktop) if it is not already running (figure 3), and
   a. (Optional) Press on ‘Change Directory’ button to select your personal working directory under your group’s folder. Create a folder for the new files
   b. Under the ‘Diffract.1’ menu, select ‘Diffractometer Control’. It will take a few seconds to initialize hardware before the control window is accessible
      i. ‘Diffractometer Control’ window (‘PowDat’ module) shown the acquisition parameters (figure 3b) of the loaded or newly created file. You can load a previous measurement (*.dat) from the menu ‘File’ and option ‘Open’, in case you want to reuse the same acquisition parameters every time
      ii. At the bottom of the ‘Diffractometer Control’ window, information about the wavelength, geometry, detector, position of omega and 2theta, and energy threshold of the detector are shown
4. Define the ‘Scan Mode’ of your measurement by selecting the option under the ‘Ranges’ menu or you can press on the ‘M’ button. Specific configuration settings for each geometry (e.g., transmission plate, capillary, etc.) are described in the next pages. Generally, ‘Scan Mode’ (labeled as number 4 in the figure) relates to the type of sample holder in use:
   - ‘Transmission’ (most common mode) scan mode for flat plate discs usually keeps a constant 2:1 between 2θ- and ω-position. However, the exact relation is defined with the ‘Scan Type’ option
   - ‘Reflection’ scan mode requires a different holder than the one used in transmission and mode adds a constant offset of 90° to the ω-position to account for the different orientation of the sample surface relative to the primary beam
   - ‘Debye-Scherrer’ requires a different holder than the one used in transmission or reflection geometry and this scan mode keeps the ω-position constant at 0° for every 2θ-position

5. ‘Scan Type’ option (labeled as number 5 in the figure) is activated only if the ‘Scan Mode’ parameter is not set to Debye-Scherrer. Options are:
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- ‘2Theta/Omega’ (most common type) invokes the normal 2:1 coupling between 2θ and ω-position during the scan
- ‘2Theta’ defines a scan with moving detector, but the ω-position kept constant at a user-defined value
- ‘Omega’ is a scan with fixed detector position and moving ω-position (rocking curve)
- ‘Independent’ let you define 2θ and ω movements independently from each other, e.g., a certain ω-offset and/or a 1:1 ratio between 2θ- and ω-step width may be entered in the ‘Edit Ranges’ dialog box

6. Set the ‘PSD Mode’ (labeled as number 6 in the figure) which controls the movement of the detector during measurement. You can select between:
   - ‘Moving’ (most common mode) where the detector will move in steps during a measurement, allowing a maximum scan range as wide as the diffractometer allows
   - ‘Stationary’ where the detector will stay at one defined by the user position. The width of the scan range in 2theta will be as wide as the detector itself (~ 19º)
   - ‘Cont. Scan’ where the detector will move continuously during the scan. Use this mode for long collections (>4 h) for better signal-to-noise ratio

7. Set the ‘Omega Mode’ (labeled as number 7 in the figure) for the position of the ω-angle. Options are:
   - ‘Fixed’ means that for every PSD 2θ-position the ω-position will be set to 2θ/2 and will remain at this position for all the measuring time of this PSD step
   - ‘Moving’ means that the ω-circle is continuously moved during data collection which usually requires longer step times and thus should only be used if additional particle randomization is required
   - ‘Gandolfi’ requires a special attachment/holder which currently is not available at IMSERC. The ‘Gandolfi’ modes enables a Debye-Scherrer measurement at a fix chi-angle which is different that 0 or 180 degrees while changing the omega- and phi- angles during collection. ‘Gandolfi’ is the ultimate method to reduce preferred orientation during collection

8. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window
   a. Press on the ‘+’ button to add a scan range
   b. Double press on the new scan range you just added and
      i. Edit the ‘2theta range’ accordingly (figure 8b) assuming a ‘PSD Mode’ of either ‘Moving’ or ‘Cont.Scan’ has been selected in step 6. In case a ‘Stationary’ ‘PSD Mode’ has been selected, the ‘Edit Scan Range’ window will not let you change directly the ‘2Theta range’ (figure 8b-i) but you can edit the center of the ‘2Theta range’ in the next step
ii. Define the ‘Step’ of the detector, typically 4º. This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015º for a single scan. Resolution can be improved with more special scans (see a Crystallography staff for more details). Our detector can cover a solid angle of ~19º with a single step, therefore with a step of 1º, the same 2theta region will be measured 19 times. In case a ‘Stationary’ ‘PSD Mode’ has been selected, the ‘Step’ is renamed to ‘Centre’ where you can select the center of the detector position in respect to the primary beam (2theta of zero). By changing the ‘Center’ value and pressing in a different field, e.g., ‘Time’ text box, the ‘2Theta’ range will change accordingly.

iii. Set the integration ‘Time/PSD Step’ in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see ‘Scan usage’ below).

iv. The ‘Omega’ range will be automatically filled assuming you have selected the ‘2Theta/Omega’ for ‘Scan Type’. In case you have selected either ‘2Theta’ or ‘Independent’ for ‘Scan Type’, provide the ‘Omega’ value you want for the experiment.

c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

9. Under ‘Ranges’ menu, select the ‘Scan Usage’ option (figure 9) accordingly:
   - Select ‘Single Sample’ if you want to measure your sample/scan range only once, or
   - Select ‘Multiple Samples’ if you would like to use any of the autosamplers (transmission or capillary), or
   - Select ‘Repetition’ if you want to measure your sample/scan range multiple times for improving the signal-to-noise ratio. You have the option to add a ‘Waiting time’ in case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.
10. Start the measurement by pressing on either ‘Data Collection’ under the ‘Measure’ menu or pressing on the button with the ‘C’ icon
11. Provide the location and filename of the collection
12. (Optional) Provide more info into the header of the file by typing a file title and/or some comments
13. Press on the ‘OK’ button and the measurement will start. An estimate time of completion will be shown on the screen during collection
RUNNING SAMPLES WITH THE FLAT DISC TRANSMISSION HOLDER

A. WITHOUT THE TRANSMISSION AUTOSAMPLER

1. Verify that the transmission stage is mounted on the diffractometer (circled in figure 1), otherwise see a Crystallography staff to set up the attachment.

2. If stage is spinning and/or occupied with a sample holder, you need to
   a. Stop any rotation by turning the ‘Sample spinner’ dial counterclockwise until it clicks. Dials are located on the left side of the goniometer base behind the arm of the detector (figure 2a). Watch the ‘stadip-transmission-sample-spinner’ video for a visual demonstration. The ‘stadip-transmission-sample-spinner’ video is also part of the (un)mounting demonstration video ‘stadip-transmission-manually-mounting-unmounting-holder’ that most likely you need to watch for the next steps described below.
   b. Remove any sample holder and leave it on the sample table outside the enclosure of the diffractometer. Watch the ‘stadip-transmission-manually-mounting-unmounting-holder’ video for a visual demonstration of how to (un)mount a holder.

3. Mount your sample holder onto the stage. Refer to the attached picture (figure 3) for the right orientation of the sample holder on the stage. The metallic mask should face the X-ray source and the side of the holder facing the detector should have only one hole visible in the middle of the holder/mask. Watch the ‘stadip-transmission-manually-mounting-unmounting-holder’ video for a visual demonstration.

4. Turn on spinning by rotating the ‘Sample spinner’ clockwise. Watch the ‘stadip-transmission-sample-spinner’ video for a visual demonstration. Spinning may or may not start right away depending on the position on the omega axis. Spinning will start though with your measurement, therefore just ensure that the sample spinner is all the way clockwise before going to the next step.
5. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 5). Watch the ‘stadip-safety-windows’ video for a visual demonstration.

6. Under ‘Ranges’ menu, select the ‘Scan Mode’ option (or press on the button with the ‘M’ icon) and set (figure 6a-e)
   a. ‘Scan mode’ to ‘Transmission’
   b. ‘Scan Type’ to ‘2Theta/Omega’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   c. ‘PSD mode’ to ‘Moving’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   d. ‘Omega mode’ to ‘Fixed’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   e. ‘Points to be added’ to ‘1’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   f. Press on the ‘OK’ button to accept all settings and close the ‘Scan Mode’ window

7. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window
   a. Press on the ‘+’ button to add a scan range
   b. Double press on the new scan range you just added and
      i. Edit the ‘2theta range’ accordingly (figure 7b)
      ii. Define the ‘Step’ of the detector, typically 4º. This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015º for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of ~19º with a single step, therefore with a step of 1º, the same 2theta region will be measured 19 times.
iii. Set the integration ‘Time/PSD Step’ in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure (see ‘Scan usage’ below)

c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

8. Under ‘Ranges’ menu, select the ‘Scan Usage’ option (figure 8) accordingly:
   o Select ‘Single Sample’ if you want to measure your sample/scan range only once, or
   o Select ‘Repetition’ if you want to measure your sample/scan range multiple times for improving the signal-to-noise ratio. You have the option to add a ‘Waiting time’ in case you are interested in a time dependent measurement, e.g., kinetics, transformations, decompositions, etc.

9. Press on the ‘OK’ button of the ‘Scan Usage’ window

10. Start the measurement by pressing on either ‘Data Collection’ under the ‘Measure’ menu or pressing on the button with the ‘C’ icon

11. Provide the location and filename of the collection

12. (Optional) Provide more info into the header of the file by typing a file title and/or some comments

13. Press on the ‘OK’ button and the measurement will start. An estimate time of completion will be shown on the screen during collection
B. WITH THE TRANSMISSION AUTOSAMPLER

1. Ensure the transmission stage and autosampler are mounted on the diffractometer (figure 1), otherwise see a Crystallography staff to set up the attachment.

2. If stage is spinning, stop any rotation by turning the ‘Sample spinner’ dial counterclockwise (figure 2). Watch the ‘stadiP-transmission-sample-spinner’ video for a visual demonstration. The ‘stadiP-transmission-sample-spinner’ video is also part of the (un)mounting demonstration video ‘stadiP-transmission-autosampler-unloading-holders’ that you need for the next steps as described below.

3. Remove any sample holder(s) from the ‘Deposit’ magazine and leave the holder(s) on the sample table outside the enclosure of the diffractometer. Watch the ‘stadiP-transmission-autosampler-unloading-holders’ video for a visual demonstration on how to remove measured samples from the autosampler.

4. Install your sample holder(s) in the ‘Stack’ magazine and make sure the ‘Deposit’ magazine is empty. Refer to the attached picture (figure 4) for the right orientation of the sample holder in the magazine. The side of the holder facing the detector should have only one hole visible in the middle of the holder/mask. Watch the ‘stadiP-transmission-autosampler-loading-holders’ video for a visual demonstration.

5. Turn on spinning by rotating the ‘Sample spinner’ fully clockwise. Watch the ‘stadiP-transmission-sample-spinner’ video for a visual demonstration. Spinning may or may not start right away depending on the position on the omega axis. Spinning will start though with your measurement, therefore just ensure that the sample spinner is all the way clockwise before going to the next step. **Turning on the sample spinner is a required step** to measure with the autosampler otherwise autosampler will never start and collection will time out.

6. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 6). Watch the ‘stadiP-safety-windows’ video for a visual demonstration.
7. Under ‘Ranges’ menu, select the ‘Scan Mode’ option and set (figure 7a-e)
   a. ‘Scan mode’ to ‘Transmission’
   b. ‘Scan Type’ to ‘2Theta/Omega’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   c. ‘PSD mode’ to ‘Moving’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   d. ‘Omega mode’ to ‘Fixed’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   e. ‘Points to be added’ to ‘1’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options

8. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window
   a. Press on the ‘+’ button to add a scan range
   b. Double press on the new scan range you just added and
      i. Edit the ‘2theta range’ accordingly (figure 8b)
      ii. Define the ‘Step’ of the detector, typically 4º. This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015º for a single scan. Resolution can be improved with more special scans (see a Crystallography staff for more details). Our detector can cover a solid angle of ~19º with a single step, therefore with a step of 1º, the same 2theta region will be measured 19 times
      iii. Set the integration ‘Time/PSD Step’ in seconds, typically 10-20 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough
   c. (Optional) Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections or add a range that could be used for different acquisition parameters between samples in the autosampler

9. Under ‘Ranges’ menu, select the ‘Scan Usage’ option
a. Select ‘Multi-sample’ in ‘Scan Usage’ (figure 9a)
b. Set the total number of samples you have loaded on the autosampler in ‘Number of Samples’
c. You have two options for measuring the queue of samples in the autosampler. Options depend on the acquisition criteria of the samples, i.e., either i) measure all samples using the same acquisition parameters and with an automated file naming scheme (default option), or ii) define filenames and/or acquisition parameters manually for each sample in the autosampler queue:
   ➢ By default, measurements will be saved as a series of files having the same filename (to be provided in step 11) plus a unique index number as a file extension whose starting value is set in ‘First File Number’. For example, if the filename is ‘sampleX’, the ‘First File Number’ is ‘5’, and the ‘Number of Samples’ is ‘9’, the 9 individual files that will be saved in your folder will have filenames ranging from ‘sampleX.005’ to ‘sampleX.013’
If you want to give a unique filename and/or assign different acquisition parameters (ranges under step 8), to each sample in the queue of the autosampler:

1st. Tick the ‘Individual Ranges / Files’ box and press on the ‘Ranges, Files...’ button (figure 9c-1st)

2nd. The ‘Individual Ranges/Files/Titles’ window will open (figure 9c-2nd) with the list of samples prepopulated based on the ‘Number of Samples’ you provided (step 9b)

3rd. Provide a file name for each sample in the autosampler queue by either double pressing on the filename or single pressing, typing in the ‘File’ field, and pressing on the ‘Set’ button

4th. Assign a specific range per sample, in case you have more than one ranges defined (optional step 8c). Note that the default range of ‘0’ will use all ranges in the list, i.e., if there are two ranges defined (step 8c), each sample will be measured twice, one measurement per range, when range is set to ‘0’

5th. (Optional) Edit the title option

6th. Press on the ‘OK’ button to close the ‘Individual Ranges / Files / Titles’ window when done editing

7th. Press on the ‘OK’ button to close the ‘Scan Usage’ window

10. Start the measurement by pressing on either ‘Data Collection’ under the ‘Measure’ menu or pressing on the button with the ‘C’ icon

11. Software will prompt for the location and filename of the collection. Since you are measuring with the autosampler, the filename provided here will contain only the information of the sample queue and acquisition parameters (step 7-9). The filename provided here will not contain any diffraction data since data for each sample in the autosampler queue will be saved in the individual files provided in step 9c. Provide a filename that is different than the filenames provided in the list of samples in the queue (step 9c)

12. (Optional) Provide more info into the header of the file by typing a file title and/or some comments

13. Press on the ‘OK’ button and the measurement will start. An estimate time of completion will be shown on the screen during collection
RUNNING SAMPLES IN CAPILLARY IN DEBYE-SCHERRER MODE

A. WITH THE CAPILLARY GONIOMETER HEAD

1. Verify that the capillary stage with the goniometer (figure 1) is mounted onto the diffractometer, otherwise see a Crystallography staff to set up the attachment for you. The autosampler for transmission flat plates may remain mounted onto the diffractometer.

2. If stage is spinning or occupied with a sample, you need to:
   a. Stop any rotation by turning the ‘Sample spinner’ dial counterclockwise
   b. Remove the beamstop. Watch the ‘stadip-beamstop’ video for a visual demonstration
   c. Remove the capillary (if any) and leave it on the sample table outside the enclosure of the diffractometer
   d. Mount your capillary onto the goniometer

3. Align the capillary on the rotation axis of the goniometer. Watch the ‘stadip-debye-scherrer-goniometer-capillary’ video for a visual demonstration. Please check ‘Appendix A’ for the tools needed and location of the screws on the goniometer head that needs to be adjustment during this alignment process:
   a. Under the ‘Diffractometer’ menu, select the ‘Drive Circles’ option or press on the button with the ‘D’ icon
   b. Press the ‘Viewing Position’ button to drive the 2theta above the dovetail mount for the camera (figure 3b)
   c. Camera is located next to the computer. Mount the optical camera onto the dovetail and connect the USB cable (figure 3c)
   d. Press the ‘XView’ button (figure 3d) to launch the video software
   e. On the video software, press on the ‘Start Video’ button
f. Adjust the X, Y, dX, and dY screws on the goniometer, so that the capillary is parallel and centered when rotated by hand 360º. Please check ‘Appendix A’ for the tools needed and location of the screws on the goniometer head that needs to be adjustment during this alignment process. Watch the ‘stadip-debye-scherrer-goniometer-capillary’ video for a visual demonstration.

g. Close the ‘XView’ software, disconnect and unmount the camera from the dovetail, and put it back onto its holder next to the computer.

h. Mount the beamstop

4. Turn on spinning by rotating the ‘Sample spinner’ clockwise. The position of the knob should be between 3 and 4 o’clock.

5. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 5). Watch the ‘stadip-safety-windows’ video for a visual demonstration.

6. Under ‘Ranges’ menu, select the ‘Scan Mode’ option (figures 6a-6e):
   a. ‘Scan mode’ to ‘Debye-Scherrer’ (figure 6a)
   b. ‘Scan Type’ is disable by default (figure 6b)
   c. ‘PSD mode’ to ‘Moving’ (most common, figure 6c). Read the ‘General steps for data acquisition’ section for details about the other custom options
   d. ‘Omega mode’ to ‘Fixed’ (most common, figure 6d). Read the ‘General steps for data acquisition’ section for details about the other custom options
   e. ‘Points to be added’ to ‘1’ (most common, figure 6e). Read the ‘General steps for data acquisition’ section for details about the other custom options

7. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window
   a. Press on the ‘+’ button to add a scan range
   b. Double press on the new scan range you just added:
      i. Edit the ‘2theta range’ accordingly (figure 7b)
      ii. Define the ‘Step’ of the detector, typically 4º. This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015º for a single scan. Resolution can be improved
with more special scans (see a Crystallography staff for more details). Our detector can cover a solid angle of ~19º with a single step, therefore with a step of 1º, the same 2theta region will be measured 19 times

iii. Set the integration ‘Time/PSD Step’ in seconds, typically 20-60 s. For quantitative analyses, you need a very good signal-to-noise ratio especially at high angles. Exposure time in this case might be several minutes and overall collection time several hours. Signal-to-noise ratio improves when you measure with a longer exposure time and/or average multiple scans that have been collected with a long enough exposure

c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

8. Under ‘Ranges’ menu, select the ‘Scan Usage’ option (figure 8)
   o Select ‘Single Sample’ if you want to measure your sample/scan range only once, or
   o Select ‘Repetition’ if you want to measure your sample/scan range multiple times for improving the signal-to-noise ration. You have the option to add a ‘Waiting time’ in case you are interested in a time dependent measurement, e.g., transformation, decomposition, etc.

9. Start the measurement by pressing on either ‘Data Collection’ under the ‘Measure’ menu or pressing on the button with the ‘C’ icon

10. Provide the location and filename of the collection

11. (Optional) Provide more info into the header of the file by typing a file title and/or some comments

12. Press on the ‘OK’ button and the measurement will start. An estimate time of completion will be shown on the screen during collection
B. WITH THE CAPILLARY AUTOSAMPLER

1. Verify that the capillary autosampler attachment is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff

2. Insert the full-length glass capillary in one of the 10 positions

3. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 3). Watch the ‘stadip-safety-windows’ video for a visual demonstration

4. Under ‘Ranges’ menu, select the ‘Scan Mode’ option and set (figure 4)
   a. ‘Scan mode’ to ‘Debye-Scherrer’
   b. ‘PSD mode’ to ‘Moving’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options
   c. ‘Scan Type’ is disable by default
   d. ‘Omega mode’ to ‘Fixed’
   e. ‘Points to be added’ to ‘1’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options

5. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window
   a. Press on the ‘+’ button to add a scan range
   b. Double press on the new scan range you just added and
      i. Edit the ‘2theta range’ accordingly (figure 5b)
      ii. Define the ‘Step’ of the detector, typically 4°. This step is different than the resolution step (2theta values in your powder pattern) which is fixed to 0.015° for a single scan. Resolution can be improved with more special scans (see a staff for more details). Our detector can cover a solid angle of ~19° with a single step, therefore with a step of 1°, the same 2theta region will be measured 19 times
iii. Set the integration ‘Time/PSD Step’ in seconds, typically 10-60 s. Signal-to-noise ratio improves if you measure the same 2theta range multiple times instead of measuring once with a longer exposure (see ‘Scan usage’ below)

c. (Optional) Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections or add a range that could be used for different acquisition parameters between samples in the autosampler

6. Under ‘Ranges’ menu, select the ‘Scan Usage’ option
   a. Select ‘Multi-sample’ in ‘Scan Usage’ (figure 6a)
   b. Set the total number of samples you have loaded on the autosampler in ‘Number of Samples’
   c. You have two options for measuring the queue of samples in the autosampler. Options depend on the acquisition criteria of the samples, i.e., either i) measure all samples using the same acquisition parameters and with an automated file naming scheme (default option), or ii) define filenames and/or acquisition parameters manually for each sample in the autosampler queue:
      ➢ By default, measurements will be saved as a series of files having the same filename (to be provided in step 8) plus a unique index number as a file extension whose starting value is set in ‘First File Number’. For example, if the filename is ‘sampleX’, the ‘First File Number’ is ‘5’, and the ‘Number of Samples’ is ‘9’, the 9 individual files that will be saved in your folder will have filenames ranging from ‘sampleX.005’ to ‘sampleX.013’
      ➢ If you want to give a unique filename and/or assign different acquisition parameters (ranges under step 5c), to each sample in the queue of the autosampler:
        1st. Tick the ‘Individual Ranges / Files’ box and press on the ‘Ranges, Files...’ button (figure 6c-1st)
        2nd. The ‘Individual Ranges/Files/Titles’ window will open with the list of samples prepopulated based on the ‘Number of Samples’ you provided (figure 6c-2nd)
3rd. Provide a file name for each sample in the autosampler queue by either double pressing on the filename or single pressing, typing in the ‘File’ filed, and pressing on the ‘Set’ button.

4th. Assign a specific range per sample, in case you have more than one ranges defined (optional step 5c). Note that the default range of ‘0’ will use all ranges in the list, i.e., if there are two ranges defined (step 5c), each sample will be measured twice, one measurement per range, when range is set to ‘0’

5th. (Optional) Edit the title option

6th. When done editing, press on the ‘OK’ button to close the ‘Individual Ranges/Files/Titles’ window

7th. Press on the ‘OK’ button to close the ‘Scan Usage’ window

7. Start the measurement by pressing on either ‘Data Collection’ under the ‘Measure’ menu or pressing on the button with the ‘C’ icon

8. Software will prompt for the location and filename of the collection. Since you are measuring with the autosampler, the filename provided here will contain only the information of the sample queue and acquisition parameters (step 4-6). The filename provided here will not contain any diffraction data since data for each sample in the autosampler queue will be saved in the individual files provided in step 6c. Provide a filename that is different than the filenames provided in the list of samples in the queue (step 6c)

9. (Optional) Provide more info into the header of the file by typing a file title and/or some comments
10. Press on the ‘OK’ button and the measurement will start. An estimate time of completion will be shown on the screen during collection

C. WITH THE PERMANENTLY ALIGNED CAPILLARY HOLDER

This pre-aligned capillary holder enables the measurement of full-length capillaries with a diameter ranging from 0.3 mm to 1.5 mm without the use of an optical camera for alignment. Capillary can be spun during the measurement. Verify that the permanently aligned capillary autosampler attachment is mounted onto the diffractometer (figure 1), otherwise see a Crystallography staff to set up the attachment for you. To perform a measurement with the permanently aligned holder, please follow these steps:

1. If stage is spinning and/or occupied with a sample holder, you need to
   a. Stop any rotation by turning the ‘Sample spinner’ dial counterclockwise
   b. Remove the capillary and leave it on the sample table outside the enclosure of the diffractometer
2. Insert the full-length glass capillary in the holder and rotate the ‘Sample spinner’ knob clockwise at a position between 3 and 4 o’clock
3. Close the safety windows of the enclosure and make sure the green light next to the top emergency button is on (figure 3). Watch the ‘stadip-safety-windows’ video for a visual demonstration
4. Under ‘Ranges’ menu, select the ‘Scan Mode’ option (figure 4) and set:
   a. ‘Scan mode’ to ‘Debye-Scherrer’
b. ‘PSD mode’ to ‘Moving’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options

c. ‘Scan Type’ is disable by default

d. ‘Omega mode’ to ‘Fixed’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options

e. ‘Points to be added’ to ‘1’ (most common). Read the ‘General steps for data acquisition’ section for details about the other custom options

5. Under ‘Ranges’ menu, select the ‘Scan Range’ option. On the ‘Edit Range’ window

a. Press on the ‘+’ button to add a scan range

b. Double press on the new scan range you just added and

i. Edit the ‘2theta range’ accordingly (figure 5b)

ii. Define the ‘Step’ of the detector, typically 2-6º. This step is different than the step of the intensity points in your powder pattern that is fixed to 0.015º. Our detector can cover a solid angle of ~19º with a single step, therefore with a step of 1º, the same 2theta region will be measured 19 times

iii. Set the integration ‘Time/PSD Step’ in seconds, typically 20-60 s. Signal-to-noise ratio improves when you measure with a longer exposure time or average multiple scans that have been collected with a long enough exposure

c. Keep adding more ranges according to your needs, e.g., add a range with longer exposure for the high angle reflections, etc.

6. Under ‘Ranges’ menu, select the ‘Scan Usage’ option

   o Select ‘Single Sample’ if you want to measure your sample/scan range only once (figure 6)

   o Select ‘Repetition’ if you want to measure your sample/scan range multiple time for improving the signal-to-noise ration. You have the option to add a ‘Waiting time’ in case you are interested in a time dependent
measurement, e.g., transformation, decomposition, etc.

7. Start the measurement by pressing on ‘Data Collection’ under the ‘Measure’ menu or pressing on the button with the ‘C’ icon

8. Provide the location and filename of the collection

9. (Optional) Provide more info into the header of the file by typing a file title and/or some comments

10. Press on the ‘OK’ button and the measurement will start. An estimate time of completion will be shown on the screen during collection
A. EXPERIMENTAL SECTION

Modify the text below according to the setup and conditions you used during the measurement:

“PXRD data were collected at room temperature on a STOE-STADI-P powder diffractometer equipped with an asymmetric curved Germanium monochromator (CuKα1 radiation, λ = 1.54056 Å) and one-dimensional silicon strip detector (MYTHEN2 1K from DECTRIS). The line focused Cu X-ray tube was operated at 40 kV and 40 mA. Powder was packed in a X (3 or 8 mm metallic mask | capillary) and sandwiched between XXXX (two polyimide or acetate layers of tape | capillary). Intensity data from YY to ZZ degrees two theta were collected over a period of XX mins. Instrument was calibrated against a NIST Silicon standard (640d) prior the measurement.”

B. ACKNOWLEDGEMENT

“This work made use of the IMSERC X-RAY facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University.”
TROUBLESHOOTING

A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default ‘StadiP’ user account should be logged in. In case the computer was restarted, the password for the ‘StadiP’ account is __________. See ‘Default instrument status’ section for more details.

C. THERE IS NO INTENSITY OR THE DIFFRACTION IS VERY WEAK DURING COLLECTION

1. Verify that all six safety doors are closed and aligned, and the safety green light is on (figure 1). If the green safety light is not on, reseat all safety doors by opening and closing each door. Watch the ‘stadip-safety-windows’ video for a visual demonstration.

2. The sample is heavily absorbing X-rays. Repack the sample and do not fill up the metallic masks with material. Use only a thin layer of powder for heavily absorbing samples.

3. Check the voltage and current settings of the X-ray generator (middle of the rack with electronics/controllers). Default operating settings for Cu-radiation are 40 kV and 40 mA (figure 3a). In case:
   a. Values on the generator are zero, contact a Crystallography staff.
   b. Settings are different than the default values, under the ‘Setup’ menu select ‘Generator’. At the ‘Generator Settings’ window (figure 3b) put the default values at the corresponding ‘Set to’ fields and press the ‘Set kV, mA’ button. It takes a few seconds for the generator to apply the new values.
D. AUTOSAMPLER WILL EITHER NOT START OR LOAD SAMPLES PROPERLY

Verify that the sample spinner dial is rotated all the way clockwise. Panel with the dial is located on the left side of the goniometer (figure D1). Watch the ‘stadip-transmission-sample-spinner’ video for a visual demonstration. When dial is not turned all the way clockwise, autosampler will either not start or start and load only the first sample in the queue.

Additionally, verify that both the ‘Stack’ and ‘Deposit’ magazines have been mounted correctly. Watch the ‘stadip-transmission-autosampler-loading-holders’ video for a visual demonstration on how to load samples on the autosampler and the ‘stadip-transmission-autosampler-unloading-holders’ video on how to remove measured samples from the autosampler.

When mounting the ‘Stack’ and ‘Deposit’ magazines, please verify that magazines have been inserted correctly and the guiding pins are touching the autosampler base (figure D2). Common mistakes of non-fully inserted magazines are shown in figures D3 and D4.

**Figure D2.** Proper position of the ‘Stack’ and ‘Deposit’ magazines with the guiding pins touching the base.
**Figure D3.** Example of improper positioning of the ‘Deposit’ magazine where the guiding pin is not touching the base. **Figure D4.** Example of improper positioning of the ‘Deposit’ and ‘Stack’ magazines.
E. SAMPLE STAGE WILL NOT SPIN

Sample spinning might not start right away after rotation of the sample spinner, but sample spinning will start with data collection. Please check the following steps:

1. Verify that the sample spinner dial is rotated all the way clockwise. Panel with the dial is located on the left side of the goniometer (figure E1). Watch the ‘stadip-transmission-sample-spinner’ video for a visual demonstration. When dial is not turned all the way clockwise, autosampler will either not start or start and load only the first sample in the queue.

2. Verify that the acquisition software is running. Acquisition software controls the spinning function and if software is closed, spinning will not start when turning the sample spinner dial clockwise. If acquisition software is already running, please restart the acquisition software. To (re)start the acquisition software, under the ‘Diffract.1’ menu of ‘WinXPow’, select ‘Diffractometer Control’. Check step 3 under the ‘General steps for data acquisition’ section for more details about restarting the acquisitions software.

F. SPECIFIC ERROR MESSAGES

Please check the error list below with the suggested actions:

1. Job stopped by an error! Error while starting data collection!

Verify that all settings for using the autosampler are correct. Verify that the sample spinner dial is rotated all the way clockwise. Panel with the dial is located on left side of the goniometer (figure D).

2. Job stopped by an error! Error while reading PSD intensities

Restart the acquisition software. If after restarting the error is persistent, please contact a Crystallography staff.

3. Unable to init the PDT-Board! Error: Unable to connect to MYTHEN interface! (‘get version’ failed)

Restart the acquisition software. If after restarting the error is persistent, please contact a Crystallography staff.

4. Unable to communicate with the generator! Generator does not respond. The program will terminate!

Restart the acquisition software. If after restarting the error is persistent, please contact a Crystallography staff.
G. THERE IS AN ERROR/PROBLEM WITH THE INSTRUMENT THAT IS NOT ADDRESSED UNDER THE TROUBLESHOOTING SECTION

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the ‘Stop’ sign near the instrument computer to notify users immediately after you. ‘Stop’ signs are located on the shelf above the computers in BG51.
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the ‘Stop’ sign near the instrument computer.
3. Contact a staff member for instructions.
APPENDICES

APPENDIX A: ALIGNMENT TOOLS AND POSITION OF ADJUSTMENT SCREWS ON THE DEBEY-SCHERRER GONIOMETER HEAD

Locate the toolbox (figure A1) in the enclosure of the diffractometer and take one of the two wrenches (figure A2) needed for adjusting the alignment screws.

![Toolbox](image1.png)  ![Wrenches](image2.png)

**Figure A1.** Toolbox containing the various accessories needed for the alignment of the goniometer head. **Figure A2.** Wrenches that can be used for adjusting the alignment screws on the goniometer head.

First, locate the ‘Lock screw’ as shown in figure A3, and secure the pin with your sample onto the goniometer head using the wrench. With the wrench, adjust the X, Y, dX, and dY screws (figures A3-A6) on the goniometer head, so that the capillary is parallel and centered when rotated by hand 360°. Watch the ‘stadi-debye-scherrer-goniometer-capillary’ video for a visual demonstration of the alignment process.
A3
Lock screw
Tilt (dx/dy)
Translation (x/y)

A4
Tilt (dx/dy)
Translation (x/y)

A5
Tilt (dx/dy)
Translation (x/y)

A6
Tilt (dx/dy)
Translation (x/y)
APPENDIX B: COMPARING POWDER PATTERNS COLLECTED AT DIFFERENT WAVELENGTHS

To compare powder patterns collected with different wavelengths/sources, such as Cu-, Mo, Ag, synchrotron-radiation, you need to convert the 2θ axis (which is wavelength dependent) to a different unit. Two popular options for wavelength-independent units are:

- \( Q = \frac{4\pi\sin(\theta)}{\lambda} \), where \( \lambda \) is the wavelength of the source (CuK\(\alpha_1 \) radiation, \( \lambda = 1.54056 \) Å | MoK\(\alpha_1 \) radiation, \( \lambda = 0.70930 \) Å | AgK\(\alpha_1 \) radiation, \( \lambda = 0.559407 \) Å), and
- \( \frac{1}{d} = \frac{2\sin(\theta)}{\lambda} \)

To use the software that came with the diffractometer (‘WinXPow’):

1. Start the ‘Graphics’ program by selecting ‘Graphics’ under ‘Raw Data’ menu on ‘WinXPow’
2. Load the patterns collected with different wavelengths by either opening (File->Open) a selection of files or pressing the ‘Add raw Data’ option under the ‘Window’ menu

Select ‘Scaling axes’ under the ‘Options’ menu and select either ‘Q’ or ‘1/D’ for the ‘X-axis’ option
**IMSERC User Manual for StadiP (v1.34)**

### REVISIONS

<table>
<thead>
<tr>
<th>Version</th>
<th>Date</th>
<th>Changes</th>
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| v1.34   | 2021/09/17 | • ‘Appendix’ section added  
• ‘Troubleshooting’ section updated  
• Training video files transferred from Box to OneDrive, and all relevant links were updated |
| v1.32   | 2020/08/23 | • Links to online video module added for demonstrating sample alignment using a capillary goniometer head  
• More pictures added under the ‘Autosampler will either not start or load samples properly’ section under ‘Troubleshooting’ |
| v1.30   | 2020/07/27 | • Improvements and more pictures added for facilitating remote training:  
  o More pictures were added under the sample preparation section which has its own section now (used to be under ‘General steps for sample packing and data acquisition’ section). The ‘General steps for data acquisition’ section was modified accordingly  
  o Links to online video modules added for demonstrating holder (un)mounting processes using modes of ‘transmission without the autosampler’, and ‘transmission with the autosampler’. Additionally, references to the videos for proper verification of alignment of safety doors and un(mounting) the beamstop were added in the corresponding sections  
• Details about custom runs using the transmission and Debye-Scherrer autosamplers were added |
| v1.20   | 2020/02/18 | • Reformatted according to the latest template. Sections about ‘Safety’, ‘Data management’, and ‘Software’ were added  
• Added more pictures to each section  
• Updated figures to reflect the new options and layout in windows for MainMenu 3.12.5, PowDat 3.6.0.6, Graphic 3.6.0.1, and RawDat 3.6.0.1 |
| v1.18   | 2018/06/24 | • Updated figures to reflect the Win10 theme and new options for MainMenu 3.12, PowDat 3.6.0.1, Graphic 3.6.0.0, RawDat 3.6.0.0, FIT 3.6.0.0, Calibrate 3.6.0.0, Index 3.6.0.0, Theo 3.6.0.0, SystEval 3.6.0.0, Cryst 3.6.0.0, and Retaust 3.6.0.0  
• Added sections for variable temperature measurements  
• Added more pictures for each section  
• Added ‘Publication’ section  
• Added ‘Troubleshooting’ section |
• Release of original version of the user manual for MainMenu 3.4.6, PowDat 3.4.8, Graphic 3.4.4, RawDat 3.4.4, FIT 3.4.0, Calibrate 3.4.2, Index 3.4.1, Theo 3.4.2, SystEval 3.4.2, Cryst 3.4.2, and Retaust 3.4.0