

Cornell Center for Materials Research *Bruker D8*
Discover Powder Diffractometer Standard
Operating Procedure

February 14, 2022

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1 Introduction

The Bruker D8 Discover Powder diffractometer is designed for efficient collection of theta-2theta powder x-ray diffraction patterns to measure lattice parameters of crystalline materials or to find the mass ratios of multiple crystalline phases in a sample. Diffraction is measured using Cu K_{α} radiation with a wavelength of approximately $\lambda = 1.54\text{\AA}$ and photon energy of $E=8.04$ keV. The source is a water-cooled fixed Cu target excited by a 40 kV x 25 mA electron gun. A Ni filter inserted near the end of the source arm is used to filter out Cu K_{β} radiation, leaving Cu $K_{\alpha 1}$ and Cu $K_{\alpha 2}$ and some remaining Bremsstrahlung radiation. A silicon strip detector (SSD160) is used to record the photon count rate (intensity) as a function of diffraction angle. This detector has a limited degree of energy discrimination, allowing it to ignore photons with energy far away from Cu K_{α} and Cu K_{β} , reducing the background level of measurements.

2 Acknowledgments

This instrument was purchased with support of the NSF MRSEC program. All publications and patent applications which include work performed on this instrument must acknowledge this support, including the current NSF grant number: DMR-1719875. For recommended wording, please see [this link](#). Use of current grant numbers is vital for funding agency support!

3 CCMR Shared Facility Policies

The CCMR operates its shared facilities in accordance with its shared facility policy. The full policy document can be found [here](#). All users are expected to follow these policies, and in particular:

- To use a particular instrument, a researcher is required to have been trained by facility staff unless specifically posted otherwise. An unauthorized researcher and anyone knowingly granting access to such person is in violation.
- Users are not allowed to share login IDs/passwords, or to reserve or enable an instrument on behalf of another user.
- Instrumentation must be reserved and enabled by the intended user prior to and during use.
- Disabling or circumventing instrument interlocks for the purpose of avoiding instrument permissions or machine time charges is strictly forbidden.
- Users are not to move or disturb the work of other researchers unless given permission by facility staff or the researcher. Notify facility staff about unattended samples.
- Users are expected to remove all samples and to leave the work area in as clean or cleaner condition compared to when they arrived.

4 Facility and Emergency Contacts

In the case of emergency, users should call 911 from any phone. Emergency phones are located in the Bard Hall stairwell on the basement level landing, and on the loading

dock in the yellow call box.

Inquiries about training and use of the Bruker D8 Discover Powder Diffractometer should be made to Mark Pfeifer at map322@cornell.edu, (607)255-4161, or in person in Thurston Hall, Room 113.

Secondary contact for this instrument should be directed to Phil Carubia at pmc228@cornell.edu, (607)255-6757, or in person in Bard Hall, Room B57.

5 Access

5.1 Becoming a CCMR shared facility user

Prior to accessing the CCMR shared facilities, instrument researchers will need to register to become facility users and train on the instruments of interest. Registration consists of four things:

- Register for a CCMR FOM account: Internal users can register in [FOM](#) directly with a valid net ID. External users can register with the [External FOM registration](#)
- Read the orientation for the appropriate CCMR facility - which can be downloaded [here](#) - and complete, sign and return the last page to the facility manager (hard copy or electronic).
- Take required safety training for access to the room/instrument of interest (see safety training section of this manual)
- Schedule training with appropriate facility manager.

5.2 Physical Access

Access to Bard Hall Room SB56 is controlled through a proximity sensor which reads Cornell ID cards. Either before, or at the beginning of training, provide the lab manager with a copy of the completed last page of the [orientation form](#). After completing training, the laboratory manager will confirm that you have completed all necessary safety training courses and then grant you door access. This process may take several days to complete.

A researcher without a Cornell ID card can request a temporary access card from the laboratory manager.

5.3 Instrument access: Facility Online Manager(FOM)

The instrument control computers are interlocked with FOM and must be enabled, through FOM, by a user with an active CCMR FOM account, before an instrument can be used. FOM can be accessed through any web browser. Users can log into FOM through the data analysis computer - provided in all CCMR Lab spaces, or any other device with a web browser - at <https://fom.ccmr.cornell.edu/fom>. If FOM is accessed through a public computer, you should either: use a private browsing session, making sure to close the browser when you have finished logging on to the instrument; or, you must log out of FOM through the browser *and* close all open browser windows. Failure to do so may grant another user access to your FOM account through the public computer.

It is the expectation of the Bard Materials Facility that researchers will reserve only time that they need, use the bulk of their reserved time, and be on time to their reservations. To allow flexibility for unforeseen events, reservations in FOM will stay active for the scheduling increment of the instrument. If the instrument is not enabled before the end of this grace period, the reservation will automatically be deleted and the instrument will become available to all trained users. Users with a reservation will always have precedent and it is always recommended that a user make a reservation for their session.

6 Safety training

Before performing any laboratory work at the Cornell Center for Materials Research Bard Facility, users must have completed the following safety training courses through CULearn:

- EHS 2555 - Laboratory Safety
- EHS 2716 - Chemical Waste Disposal
- EHS 5330 - Fire Safety
- EHS 5033 - Radiation Producing Equipment Safety

7 Preparation before coming to the facility

Standard sample holders for sample volumes of 80 μL are available, as are reduced volume 4 μL sample wells. Flat polycrystalline samples can also be studied, but may require some additional effort to mount optimally.

8 Equipment

8.1 Hardware

When not in use, the Bruker D8 Discover is left in an enabled state with the source at low power and the radiation shutter closed. The source being enabled is indicated by an amber light on the source (inside the hutch) and an amber illuminated radiation symbol on the outside of the hutch. A red light on the source is illuminated only when the radiation shutter is open. The shutter is automatically opened before a scan and closed at the end of the scan. For safety, both the shutter and the motorized motions of the diffractometer are interlocked with the door of the hutch.

8.2 Software

9 Procedure

9.1 Instrument Startup

9.1.1 Configure the data acquisition software

Start the software Bruker Diffrac.Suite on the instrument control computer. There should be an icon for it on the left corner of the status ribbon at the bottom of the screen. The user name is ‘Lab manager’ and there is no password. Once the software has started, select the ‘Commander’ tab. In the upper left portion of the window is a list of physical variables on the instrument. The left column contains the names of the variables. The middle column shows values (with gray background) read from the diffractometer. The right column contains editable values (with white background).

9.1.2 Set the source to operating power

The standard source used in this instrument is water cooled Cu source. Edit the source voltage and current to 40 kV and 25 mA for 1000 W (see Figure 1). The standby power is 20 kV and 5 mA for 100 W. After editing the voltage and current fields click the ‘Set’

button nearby. It takes under 30 seconds for the source to ramp to full power, during which time the control software is grayed out and the illuminated radiation symbol on the front of the D8 Discover blinks to indicate that the source power is not stable. Once the source power is stable, the instrument is ready for use.

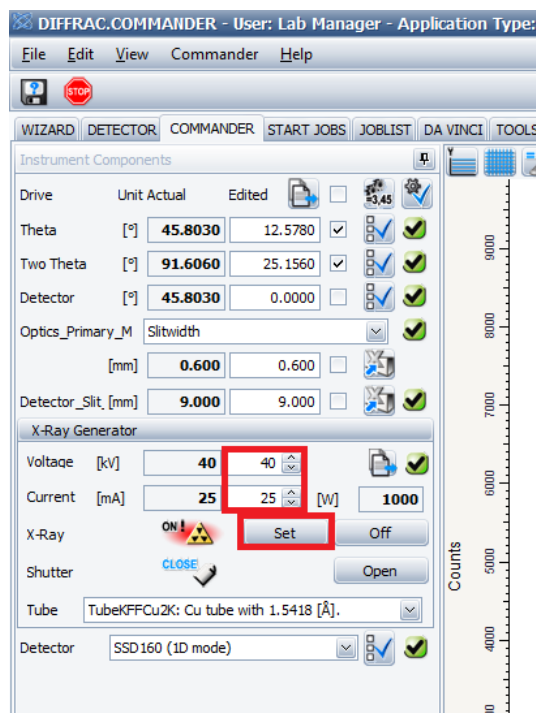


Figure 1: Setting the x-ray source voltage and power.

9.1.3 Reducing fluorescence background from Co, Fe, Mn, Cr

Elements with absorption edges below (but near) the photon energy of the source will emit photons at a lower energy (fluoresce), distributed over all angles. If the detector used for a diffraction experiment is sensitive to these photons, they will produce a background, increasing the noise in the measurement. For our system using Cu K_{α} radiation, fluorescence can be a problem from Co, Fe, Mn, and Cr. Although Ni has lower atomic number than Cu, it is close enough that its absorption edge is higher than the emission line of Cu. Figure 2 shows a typical background signal due to fluorescence; increasing from low angle up to 40° and staying high with increasing angle.

The silicon strip detector produces a voltage signal which is fed into a discriminator.

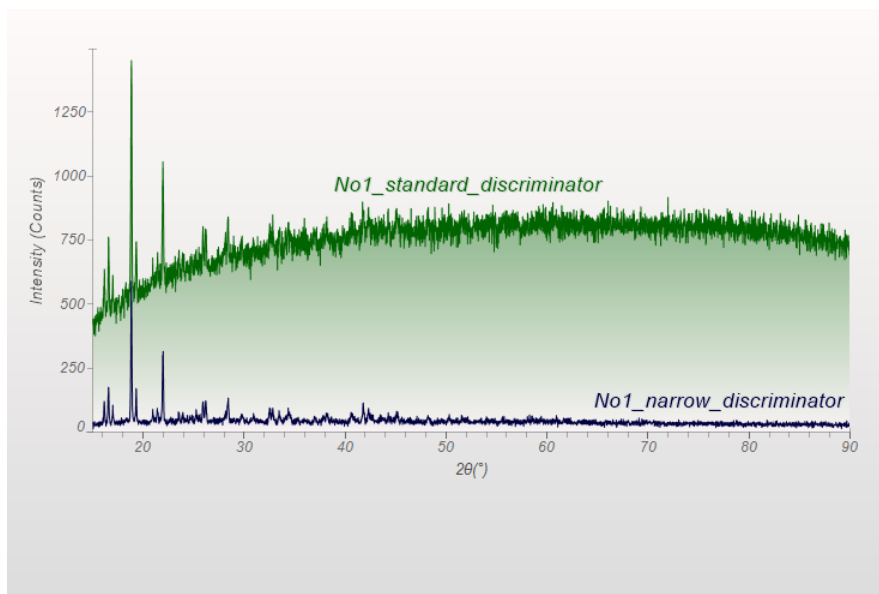


Figure 2: Diffraction patterns from Fe based metallo-organic framework using standard discriminator settings (green curve) showing strong fluorescent background and with narrower discriminator settings (blue curve) to reduce fluorescent signal.

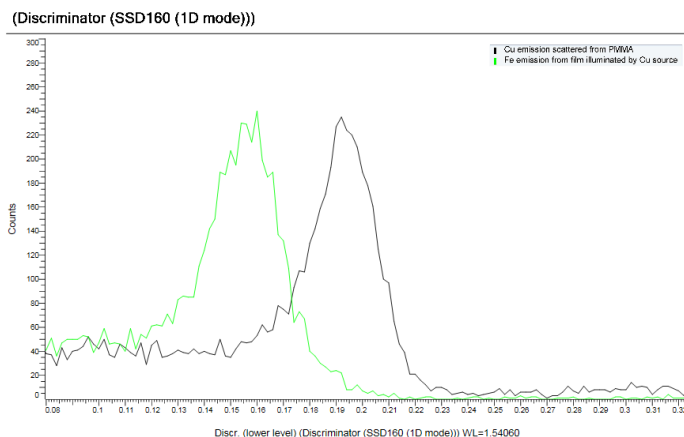


Figure 3: Intensity measured using a narrow discriminator window, as a function of discriminator lower level. The green curve, peaking near 0.15 V, was recorded using an Fe sample illuminated by Cu K_{α} . The black curve, peaking near 0.19 V, was recorded using Cu K_{α} scattered from a polymer sample.

	Standard	Fluorescence rejection
Lower Discriminator (V)	0.110	0.182
Upper Discriminator (V)	0.250	0.250

Table 1: Discriminator settings for SSD160 for standard operation and for suppression of fluorescent background when measuring Fe bearing samples with Cu K_{α} radiation.

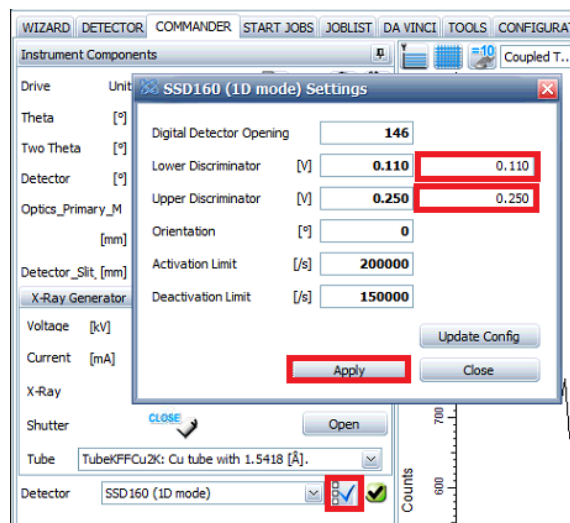


Figure 4: Setting the detector discriminator.

The pulses output by the discriminator are reported as counts (ideally detected photons). In normal operation, the discriminator has a broad voltage window (0.110 - 0.250 V) to optimize signal strength. For fluorescence rejection, the lower discriminator should be raised (0.180 - 0.250 V) as shown in Table 1. These values were obtained experimentally, and if a particular sample has a very high amount of Co or Fe, it may be useful to set the lower discriminator higher. The Cu K_{α} peak is near 0.193 V with a full width half max of 0.026 V, as shown in figure 3.

To change the discriminator settings, click on the box with the blue check-mark to the right of the box that says ‘SSD 160 (1D mode)’ next to ‘Detector’ (see Figure 4). Edit the ‘Lower Discriminator’ field and click ‘Apply’. If you modify the discriminator for an experiment, please set it back to the default values at the end of your experiment.

9.2 Preparing and mounting a sample

Powder diffraction assumes a random angular distribution of a very large number of crystallites illuminated by the x-ray beam. Traditionally this is satisfied by using a powdered sample with roughly 10 μm or smaller extent. Larger crystallites (100 μm) result in a pattern which is not representative of scattering from all orientations, but it in some cases may still be useful for phase identification. Polycrystalline solids may also be used as samples for powder diffraction, but partial orientation of grains may affect the relative intensities of Bragg peaks, potentially completely eliminating some diffraction peaks.

9.2.1 Nanomaterials

Nanomaterials in general present an unknown risk and require special handling considerations. Obtain approval from the laboratory manager before bringing any nanomaterials into the facility. Because there is no controlled environment (fume hood or biosafety cabinet) in the room with the powder diffractometer, nanomaterials may not be transferred between containers or holders in our facility. Researchers wishing to study nanomaterial powders will be required to purchase one or more zero diffraction plates (from the CCMR or from a vendor directly) so that they can load and empty sample wells in their own laboratory.

9.2.2 Choose sample well

There are a few different wells which can hold powdered samples. The powdered sample should completely fill the well so that the surface of the powder is level with the surface of the holder. The small quartz wells with 10 mm diameter have a volume of roughly 0.1 mL. If the sample does not completely fill the well, the surface of the sample will not be at the correct height when mounted in the diffractometer, resulting in an angular error in the measurement. There are also reduced volume wells with 5 mm diameter and 0.2 mm depth, for roughly 0.004 mL volume. These wells should only be used when you don't have enough volume for the larger wells (because you will get significantly less diffraction signal, and have grain size much less than 100 microns (so that you can have a well-packed powder inside the well).

The round sample wells are glass, which will give a broad background diffraction signal below roughly 30° 2-theta. The square sample wells are quartz and have no diffraction peaks in the vertical diffraction plane.

9.2.3 Grind sample if necessary

Wear gloves when handling powder both for safety concerns and for general cleanliness of the laboratory. Gloves are provided at the sample preparation table. Powder x-ray diffraction assumes that the sample contains an ensemble of crystals with every possible orientation. Samples composed of a relatively small number of large crystals will not give a representative diffraction patterns. If your sample has large grains (greater than 100 microns) then data quality will be improved by crushing your sample using the mortar and pestle provided. There is no absolute rule for maximum particles size – it depends on what information is needed and how complex the diffraction pattern is. If the goal is to distinguish between two possible phases which have completely different diffraction patterns, then large grain size may not be detrimental. If the sample is a combination of multiple phases, and the diffraction pattern will be used differentiate the sample between possibly compositions which have similar diffraction patterns, then more care must be taken in sample preparation.

9.2.4 Fill sample well and smooth surface

The sample wells should be found in a clean state. If you are highly concerned about cross-contamination of samples, particularly if you will use the same material for further analysis, clean the sample with alcohol and laboratory wipes. There are options for sample wells and sample pucks.

1. The square wells are quartz and must be slid into the sample pucks with slots. These sample wells are crystalline but to not have diffraction peaks in the vertical plane. They are therefore good general purpose sample mounts. Greater care needs to be taken in aligning the sample puck when mounting it (see next section), and the puck should be carried level because the well can slide rather freely in the slot.
2. The round wells are glass, and as such they produce a broad diffraction peak at low angle. For many experiments, particularly with highly crystalline materials and large diffraction angles (over 30°) this is usually not a concern. These wells sit in the sample pucks with round receptacles. Note that there are a few sample well thicknesses and receptacle depths. Before filling a sample well, make sure that there is a sample puck that that sample well fits in with their two surfaces flush.

Place a sheet of weigh paper under the sample well before transferring your sample. If static electricity is a problem when transferring your sample, use a small square of

aluminum foil instead of weigh paper. The sample should completely fill the sample well. If there is enough material available, a smooth sample can be obtained by overfilling the sample well and using the edge of a glass microscope slide to scrape across the surface of the well, pushing excess material onto the weight paper or foil. Place the sample well in the appropriate sample puck.

9.2.5 Mount sample puck

The diffractometer includes three pins which serve as the height reference for the sample (see Figure 5). When a sample is correctly mounted and inserted into the system, the surface of the sample is coplanar with the three reference pins. Place the sample puck

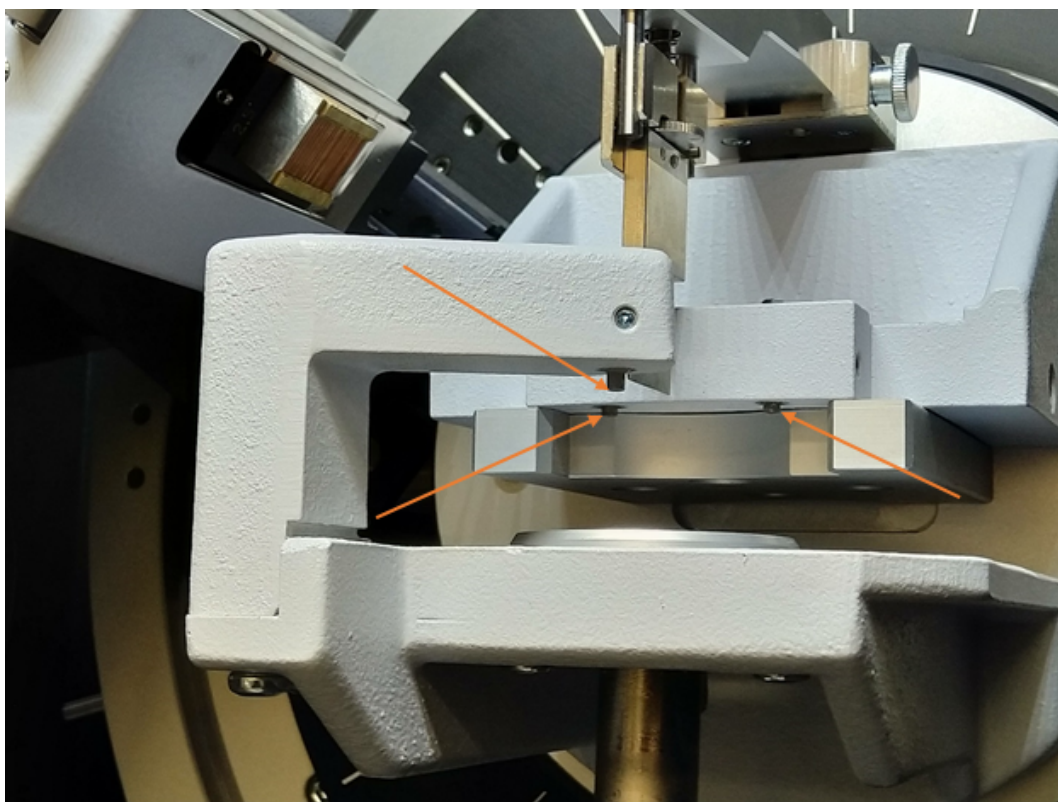


Figure 5: Sample mounting platform and three height reference pins.

on the round aluminum platform in the center of the diffractometer. Visually check that the puck is centered on the platform. Using the post that extends below the platform, push upwards until the top of the sample puck is just touching the three references

pins. Never raise the platform without a sample puck in place, because doing so can push the stage higher than it is designed to go. Do not pull down on the post; lowering should be done by supporting the post from below, and pulling the lever that is behind the post. The lever should be pulled towards the front of the instrument. Keep the lever pulled towards you as you slowly lower the post, by lowering the hand which is supporting it from below. Again, you should not need to pull on the post, and your hand should only be needed below it to keep it from falling too quickly.

After raising the platform to the height where the top face touches the three reference pins, visually check that it is touching all three. If it does not, slide the puck until it is centered under them. Failure to do this will cause you to record invalid data and likely cause the instrument to jam. Once the top of the sample puck is touching all three reference pins, push the sample post up, compressing a spring, until it stops – approximately 1 cm above the position where the puck initially makes contact with the reference pins. A properly mounted sample is shown in Figure 6.

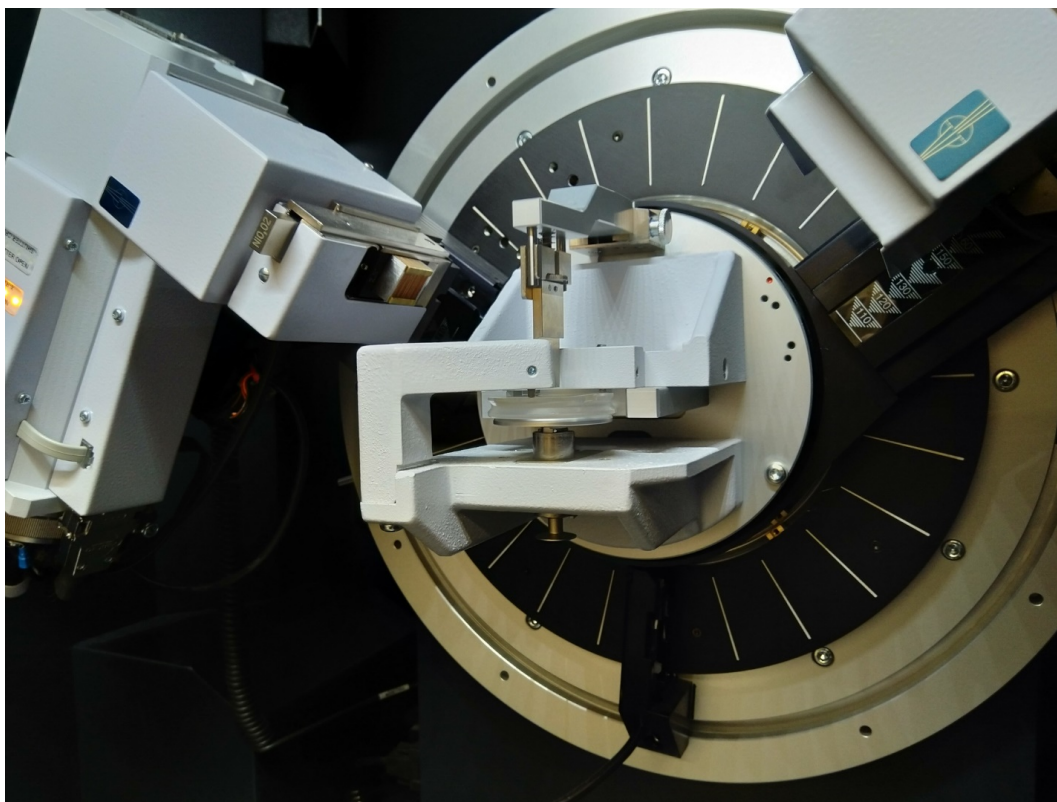


Figure 6: Sample properly mounted in diffractometer.

9.3 Measure powder diffraction pattern

9.3.1 Set the slits

The incident beam slit, detector slit, and active detector height (PSD opening) should be set based on the type of measurement to be made (see the table below and Figure 7). Decreasing the slit opening reduces the low angle scattering which causes a background in low angle scans. This should be considered when the minimum scan angle is below 15° . Reducing slits also decreases the signal available to a measurement; this is often the cause for lower than expected signal during a measurement. The PSD opening only needs to be decreased when the starting angle is below 5° to prevent exposing the detector to the direct beam.

	Standard range	Low angle
Divergent beam slit	0.6-1.0 mm	0.1-0.2 mm
Detector slit	9 mm	3 mm
PSD opening	$\approx 2.747^\circ$	Half of starting angle

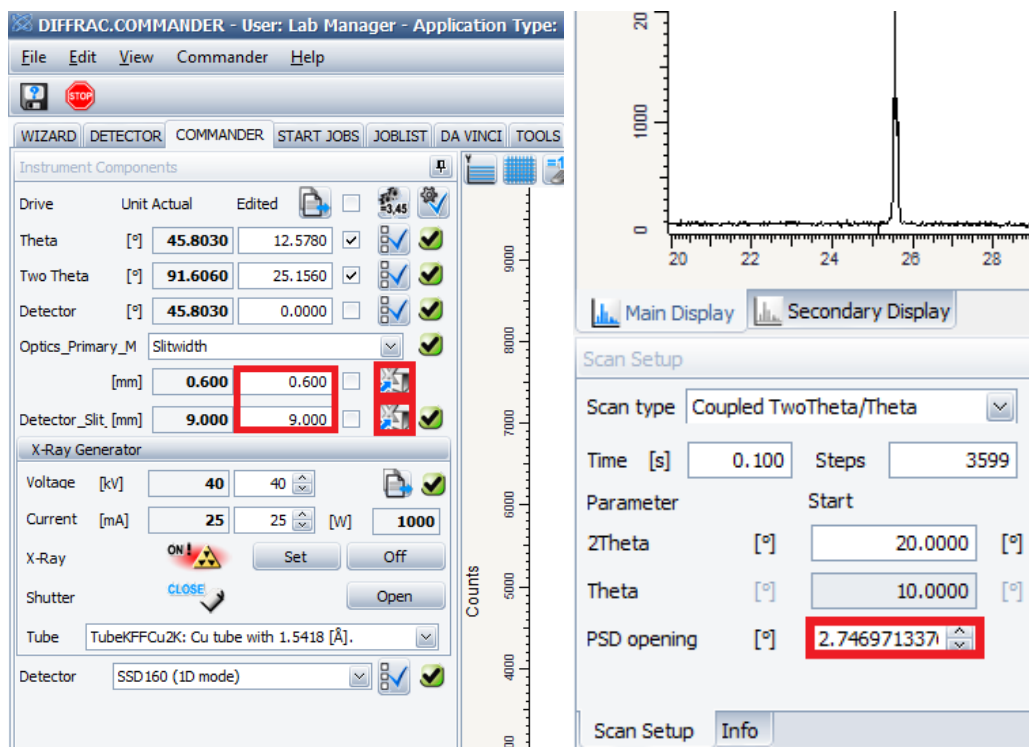


Figure 7: Setting the slits and PSD opening.

9.3.2 Scan design

Instrument users typically know in advance the general position for peaks which might be representative of their sample. Using Cu K $_{\alpha}$ radiation, minerals and metallic samples can typically be characterized by measuring peaks between 20° and 80°. Polymers and glasses may have peaks in the 5° to 30° range. Measurement noise is generally dominated by statistical noise in the counted number of photons, which is given by the square root of the number of counts. For a diffraction peak which is very strong relative to the background level, the signal to noise is therefore proportional to square root of the counting time; in order to improve signal to noise by a factor of 2, counting time (per data point) needs to be increased by a factor of 4. Where the background is large (often due to a non-crystalline phase), the background also contributes to the statistical noise. In the lower portion of the ‘Commander’ tab, confirm that the scan type is set to ‘Coupled TwoTheta/Theta’ (see Figure 6). Set the ‘Start angle’ to the desired beginning of the scan range. The default step size (approximately 0.019°) is reasonable for most measurements, and needs only to be decreased if the goal is to characterize peak shape, or increased for efficiency if the user is characterizing very broad peaks, for example from glassy phases or nanocrystals. This step size will always be a multiple of the detector pixel height. Set the ‘End angle’ to the desired end position for your scan. After changing the cursor focus, that value will be replaced by a very similar value which is an integer number of steps away from the start angle.

The software should have updated the number of steps in the scan. Click on the box for ‘Time per point’. Increasing this value will improve the signal to noise at the cost of making the measurement take longer. For most mineral and metallic samples, 0.1 s is a reasonable starting value. After changing this value, you may need to click in another box, such as number of data points, to see the calculated measurement time updated by the software. Click ‘Start scan’. The source and detector angles will move to the start position. The shutter will automatically open and the instrument will continuously sweep the source and detector angles while recording diffracted intensity.

9.4 Save data

Scans are not automatically saved by the Diffrac.Suite software. To save a scan select File→Save Result. Navigate to the ‘\rs-ccmr-coral-a\Share\Users\’ directory, and create a directory with your NetID if one does not already exist. Save your data in this directory in .brml format. If you wish to export data in a format you can analyze with your own software, open Diffrac.Eva (on either the instrument control computer or the analysis computer) and choose File→Import Data. With a single data set selected in

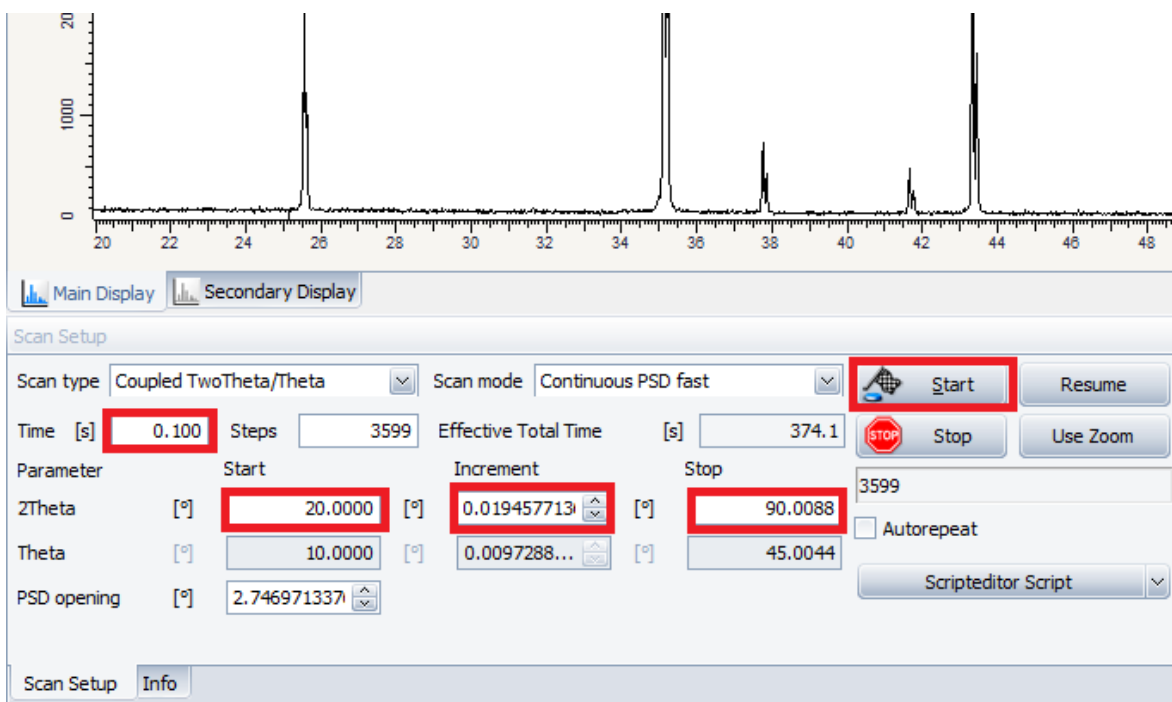


Figure 8: Typical scan configuration for highly crystalline mineral.

the software, select Export Data and save as a .xy file, which includes 1 line of header information (including the source type) and contains data in a two column (two-theta and intensity) which space separated format.

9.5 Analyze diffraction pattern

9.5.1 Jade 2010

The CCMR maintains licenses for Jade 10 and ICSD database of crystallography data. They can be accessed from the analysis computer in the room with the Bruker Powder diffractometer or the analysis computers at the Bruker GADDS or Rigaku SmartLab. CCMR diffraction users may use any of these computers for data analysis without having to log on through Coral. This document only gives a basic introduction to using Jade. Start Jade using the icon on the desktop. Navigate to your data directly by selecting Start→Open New Folder. Files in that directory will be listed in the panel on the left side of the window, organized by data format. Jade is capable of reading many data formats used in x-ray diffraction, including .brml, .raw, .asc, and .xy. Select a data

file by double clicking on it in the left panel. Many functions in Jade are organized by tab: Scans, Phases, Peaks, and Profiles.

Background fitting Jade automatically applies background correction to loaded data. To see and modify the fit background, select the button labeled ‘BC’ with a red line passing through points. This will add to the plot window a red line passing through a series of points which the software chose as background points. For data from highly crystalline samples with sharp peaks more than 10x the background level, automatic background fitting is typically satisfactory. For lower signal levels and more complex signals, the background is often overfit by the software. The background is modeled by a cubic spline passing through a set of fixed points, with a minimal curvature constraint. Right clicking on one of these points eliminates it from the model, left clicking on an open portion of the plot adds a new constraint point. Constraint points can be moved either by clicking and dragging, or hovering and moving the scroll wheel (holding Ctrl for horizontal movement).

Measuring lattice parameters Select the ‘Peaks’ tab. The software will attempt to locate peaks within the data. The parameters H, A, PK, and Filter, are used in the automatic identification of peaks. Peaks can be manually added (or removed) by selecting ‘Edit Peaks’ from the right side of the plot and left (or right) clicking on the plot. The peak list in the bottom panel includes statistics for each peak, including d-spacing, relative height, and peak full width at half maximum.

Phase identification Select the ‘Phases’ tab. Select ‘Chemistry’ to modify the chemistry filter. Select ‘Toggle All’ to select or deselect all elements. Either manually select each element which might be present, or select sets of elements (such as ‘Light elements’) from the drop-down menu and manually add elements you expect to be present. Check the ‘Unit cell’ tab and deselect all check boxes unless you want to apply constraints based on additional information known about your sample. Click ‘S/M’ (Search/Match) to select an appropriate database for the materials expected to present in the sample. For a power sample of polycrystalline material without orientational ordering (texture), ‘Severely Oriented’ should not be selected. For thin films or oriented crystallites, select ‘Severely Oriented’. When analyzing data recorded on the Bruker powder diffraction system or Bruker GADDS, ensure that ‘K-alpha 2 present’ is selected. When analyzing data recorded on the Rigaku SmartLab deselect ‘K-alpha 2 present’ if the Ge monochromator was used during data collection. After configuring the search/match function, click on ‘One-Click Analysis’. If the software is able to interpret the measured diffraction pattern as a sum of patterns from the selected crystallography databases, it

will return a list of phases with relative compositions expressed as weight percentages. In other cases, the software will return a list of potential fits.

Strain and particle shape fitting Through the ‘Profile’ tab it is possible to fit the profiles of individual peaks to obtain a more accurate measure of their relative areas and full-widths at half-maxima. Click on the position of a peak in the data to add a peak to the curve fitting function, then click and drag to change the peak height or position. Hover over the peak and use the scroll wheel on the mouse to change the peak width. When the curve is a reasonable estimate of the data, click on ‘Fit’, which will refine the curve. The table below the plot will display information about the peak area, width, and an estimate of the crystal domain size which gave rise to the diffraction pattern, if the domains are smaller than 100 nm. More accurate fits of peak shape can be obtained through the ‘Whole Pattern Fitting’ function. The button to enter this function is labeled with small line plot and the letters ‘WPF’.

9.6 Transferring Data

Data should be transferred from this computer using a secure file transfer protocol such as Cornell Box (<https://cornell.account.box.com/login>) or Cornell Dropbox (<https://dropbox.cornell.edu>). Because they are a common source of computer viruses in shared facilities, *the use of USB drives is prohibited*.

9.7 End of experiment

9.7.1 Cleaning up

This instrument is in a shared laboratory space. It is therefore important that each user take responsibility for cleanliness of the work space. Simply put, leave the lab space as clean as you found it. This includes:

- Clean any glassware and/or other lab hardware which came into contact with samples. When rinsing glassware, capture any solvents in a container which you will remove at the end of the experiment, with the exception of water used for final rinsing, which may go down the drain.
- Dispose of any pipettes (including plastic) in the trash containers labeled ‘glass only’. These containers should only be used for pipettes, broken glassware and used slides.

- If you used powder or liquid samples, wipe down any work surfaces where samples were handled or transferred.
- Remove samples. Take all samples and other materials with you.

9.7.2 Disabling the control computer through FOM

Disable the instrument through FOM. Either go to the instrument schedule page and click on your present use, or go to your User Home and click on the instrument logoff link at the top of the page. A pop-up box will appear. Select the appropriate funding source (if you have more than one option), and then click on the ‘Logoff’ button. If you access FOM through a shared computer, you must log out of FOM *and* close all instances of the web browser to avoid unintended access of your account by another user. Log out of FOM by clicking on the red ‘X’ in the top right corner of the page, next to your name, and confirm that you wish to log out.

10 Quick Review

1. Enable the instrument though FOM.
2. Set the source to **40 kV x 25 mA**.
3. Use weigh paper (or Al foil) below the sample slide when filling with powder. Mount your sample so that it is level with the surface of the puck.
4. Put the sample puck in the instrument and carefully raise it until the plastic surfaces meet all three reference pins. This is the easiest place to make a mistake, so check that it the puck meets all three pins before pushing the post up further to compress the spring.
5. Check the slit settings, the scan range, increment, time per step (0.1 s typical), and detector discriminator settings.
6. Run your scan.
7. Save you data on the shared drive in your own folder.
8. Support the post to keep the sample from dropping quickly, but **DO NOT** pull on the post. Pull the release lever behind the sample post, and keep the lever pulled as you lower the post.
9. Remove your sample and clean the work surface.

10. Set the source to **20 kV x 5 mA**.
11. Disable the instrument through FOM.
12. Make sure you cleaned the work surface and remove all of your materials.