RESEARCH INSTRUMENTATION STANDARD OPERATING PROCEDURE FOR
BRUKER D2 PHASER POWDER X-RAY DIFFRACTOMETER (OPPXRD-1)
AT NUCS FULMER

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# TABLE OF CONTENTS

1  Background ........................................................................................................... Error! Bookmark not defined.
   1.1 Weekly Calibrations ........................................................................... Error! Bookmark not defined.
2  Safety Requirements ..................................................................................... Error! Bookmark not defined.
3  Calibration Procedure ...................................................................................... 4
4  Reporting Weekly Calibration ..................................................................... Error! Bookmark not defined.
5  Sample Preparation ....................................................................................... Error! Bookmark not defined.
6  Data Collection on the Bruker D2 Phaser Powder X-ray Diffractometer .......... 8
7  Data Workup on the Bruker D2 Phaser Powder X-ray Diffractometer .............. 28
8  Powering Down the Bruker D2 Phaser Powder X-ray Diffractometer .............. 36
9  Training ............................................................................................................. 37
OPERATING PROCEDURE OPPXRD-1: BRUKER D2 PHASER POWDER X-RAY DIFFRACTOMETER

1 Background

X-ray powder diffraction is a scientific technique that uses X-ray diffraction on powder or microcrystalline samples to obtain structural information on materials. An instrument dedicated to performing such powder measurements is called a powder X-ray diffractometer. An X-ray diffractometer produces X-rays with a known wavelength and frequency, which is determined by the X-ray source. When the X-rays from the X-ray source reach the sample, the incoming beam of X-rays is either reflected off the surface, or they enter the lattice and are diffracted by the atoms present in the sample. If the atoms are arranged symmetrically with a separation distance \( d \), these waves will interfere constructively only where the path-length difference \( 2d \sin \theta \) is equal to an integer multiple of the wavelength, producing a diffraction maximum in accordance with Bragg's law, and bright spots in the diffraction pattern. The diffraction of X-rays also results in destructive interference at points between the intersections where the waves are out of phase, which are not bright spots in the diffraction pattern. The location of the spots in the diffraction pattern are used to determine the atomic spacing of the material.

Powder X-ray diffraction allows for the rapid, non-destructive analysis of multi-component mixtures without the need for extensive sample preparation. Powder X-ray diffraction is used to quickly analyze unknown materials and perform materials characterization in such fields as metallurgy, mineralogy, chemistry, forensic science, archeology, condensed matter physics, and the biological and pharmaceutical sciences.

1.1 Weekly Calibrations/QA/QC Checks

Weekly calibrations are undertaken to verify the functionality of the instrument and to head off any problems or safety concerns before they become larger problems that could result in instrument failure. A calibration standard provided by the instrument vendor is used in the weekly calibration (see Section 3).

2 Safety Requirements

X-ray powder diffraction is a scientific technique that utilizes X-rays to obtain structural information on powder samples. X-rays are ionizing radiation and X-ray photons carry enough energy to ionize atoms and disrupt molecular bonds, which makes X-rays harmful to living tissue. A very high radiation dose over a short period of time will cause radiation sickness, while lower doses will give an increased risk of radiation-induced cancer. The Bruker D2 Phaser contains radiation protective interlocks (the main door needs to be closed for operation) to prevent a user from being exposed to the X-ray radiation during the use of the powder X-ray diffractometer. The door to the sample holder is locked when voltage is applied to the X-ray tube. The yellow lights on the top of
the X-ray diffractometer and the yellow Busy light on the left side of the diffractometer is illuminated when voltage is applied to the X-ray tube and X-rays are being generated.

All users are required to provide the NUCS Core Facility Staff with records of completion of the WSU Radiation Safety Office Training Courses #1-7 & 10 ([https://rso.wsu.edu/wsu-radiation-safety-training/](https://rso.wsu.edu/wsu-radiation-safety-training/), prior to being trained on the use of the Bruker D2 Phaser Powder X-ray Diffractometer. See Section 9 for training records.

### 3 Calibration Procedure

**Materials:** Bruker AXS Corundum reference sample (A46-B29-S), Bruker D2 Phaser Powder X-ray Diffractometer

3.1 Turn on the instrument and open the diffractometer, by pushing down on the handle and pulling the latch (see Step 6.10), so that you can access the sample holder.

3.2 Place the corundum sample in the sample holder and raise the lever to place the sample into the measurement position.
3.3 Check that the divergence slit is 0.6 mm and the air screen is set at 3 mm before closing the diffractometer, see Steps 6.14-6.18 for instructions.

3.4 Collect the diffraction pattern using a Time/Step of 0.030 seconds, a Number of Steps of 8390, which will result in a data collection that will take 265.92 seconds (4.4 minutes).

3.5 When the data collection has completed and save the data in the folder: C:\Users\user\Desktop\NUCS Calibrations\Weekly Calibrations\Year (where Year is replaced with the year, eg. 2022).

3.6 Save the data file as Corundum_Calibration_mmddyy, where mm stands for the month, dd stands for the day, and yy stands for the year. For example, a filename of Corundum_Calibration_020722, was a data collection that occurred on February 7th of 2022.

3.7 After the data collection has completed, turn off the X-rays, see Step 6.38.

3.8 Remove the corundum sample and place it back into its holder.

3.9 Turn off the diffractometer, following the procedure indicated in Section 8.

3.10 Complete the Bruker D2 Phaser Weekly Calibration Report as indicated in Section 4. Make sure to include any issues encountered in both the Weekly Calibration form and the Bruker D2 Phaser Powder X-ray Diffractometer logbook.
4 Reporting Weekly Calibration

4.1 Collect powder X-ray diffraction data on the corundum reference sample as indicated in Section 3.

4.2 The report for weekly calibrations can be found on the Research drive at the following file location: M:\NSC User Facility\XRD\Phaser D2 PXRD\Calibrations\Weekly Calibrations\.

4.3 Enter the name of the NUCS Core Facility staff that collected the data and date of the data collection. Also, include the instrument parameters on the Weekly Calibration Form.

4.4 Use the DIFFRAC.SUITE EVA software to analyze the collected data. Upon importing the data, undergo a Peak Search, then Append the data to the List, and Click on Peak Column View to find the 2-Theta values, the relative intensities, and the d-spacings. See Section 7 for instructions. Place these values in the Weekly Calibration Form.

4.5 If more than three values measured d-spacings do not match to the literature values (seen on the calibration form) to within 0.5 %, then there is a reason to be concerned and inform NUCS Core Facility staff.

4.6 Please save the data and the report on the NUCS drive using the following file location: Z:\Bruker PXRD\Weekly Calibration Data and place in the respective year and folder for the data and report. Please ask the NUCS Core Facility staff if you have any questions.
5 Sample Preparation

5.1 Pick up a circular sample holder from the sample holder box on the sample prep table next to the diffractometer.

5.2 Place the sample holder on top of a Kimwipe.

5.3 Place a small amount of sample on the holder, making a 1 cm diameter (~0.5-1 gram of sample) with a spatula.

5.4 Use the side of a glass slide to tap on the powder to make the height of the sample level with the plastic outer ring of the holder. Do not drag the glass slide across the sample holder or powder will be smeared all over the holder.
6 Data Collection on the Bruker D2 Phaser Powder X-ray Diffractometer

This section is to be completed after a weekly calibration (Sections 3 & 4) has been performed.

Materials: Powder X-ray Diffraction Sampler, Bruker D2 Phaser Powder X-ray Diffractometer

6.1 Instrument time on the Bruker D2 Phaser are made on iLab (https://wsu.corefacilities.org/), prior to the user’s instrument time. Reservations are based on service, not time. Please indicate the number of data collections obtained during the reserved time. If you have questions regarding booking instrument time, please ask the NUCS Core Facility Staff.

6.2 Start by writing the date, starting time, and your name in the instrument logbook.

6.3 checking to see if the Bruker D2 Phaser powder X-ray diffractometer is on. The default state is to have the system powered down. The power switch can be found on the back of the instrument on the left side. The switch should be depressed so that the part of the switch labeled with the vertical line is depressed.
6.4 After a little bit of time, the instrument will ask for a password to login. The password is: password

6.5 When the instrument logs in, you will come to a home screen that shows the following:
6.6 Notice the lights on the top left of the instrument, only the green light indicating that the instrument is On should be illuminated. If the red alarm light is illuminated, please inform NUCS Core Facility staff.

6.7 Click on the DIFFRACT.MEASUREMENT icon on the bottom of the screen to load the measurement software. The following screen will be seen while the software is loading.
6.8 After a little while the software will ask for a password. Please use the Lab Manager account and press the OK button, as this account does not have a password.

6.9 After clicking OK, the software will say that the Measurement Server is running, this software is necessary for the instrument to talk to the data collection software.
6.10 The door of the instrument can be opened by pushing down on the handle and pulling the small handle towards you to unlatch the door. If the door does not open (slide up), pull down on the handle.

6.11 When the latch is released, the monitor will slide upwards to show the diffractometer behind it.
6.12 The sample is placed on top of the sample holder below.

6.13 The picture shown below demonstrates the instrument with the corundum standard sitting on top of the sample holder, prior to moving the sample up into the measure position.
6.14 The sample is then moved into place by lifting the lever underneath the sample holder.

6.15 Before closing the instrument, the divergence slit, the air scattering screen, the Söller slit, kβ filter, and the air scatter slits need to be adjusted, if needed. Usually just the Divergence Slit and Air Scattering Screen need to be adjusted.

6.16 The D2 Phaser has options of 0.1, 0.2, 0.6, and 1.0 mm divergence slits. The choice of divergence slit is made by picking the slit for the material that best describes your sample. A list of recommended configurations can be seen below.
6.17 The divergence slit is changed by sliding out the current slit and sliding in the desired slit.

6.18 The air scatter screen can either be set to 1 mm or 3 mm. It is changed by pulling it off and having the desired screen facing down. The air scatter screen in the picture is set to 3 mm. The air scatter screen connects to the instrument with magnets and should click into place.

6.19 Close the door to the instrument by pulling it down until it latches and does not spring back up.
6.20 You should now see the Commander window, which is the main window that will be used for data collection.

6.21 To set up a scan, click on the Scan Setup tab on the bottom of the Commander window to see the data collection parameters.
6.22 Scan Type should be set to *Coupled Two Theta/Theta*, Scan Mode should be *Continuous PSD fast*. These are the default values and should not need to be changed.

6.23 The time/step and the steps should be adjusted to give the desired experiment time. The default value is 0.5 seconds. A typical experiment should take about 30-60 minutes, but can take longer if you are trying to identify the composition of phases. For training considerations, the time step will be 0.03 seconds to have a scan completed within five minutes.

6.24 The 2theta parameters are a measure of the window for data collection. A typical window will run from 5° to 90°. Can be set as low as -5°, but useful data often occurs at 1° or higher. Lower numbers are shooting X-rays directly into the detector. The ending angle can be as high as 150°, but most samples do not show peaks beyond about 100°.

6.25 The PSD opening parameter is a slit on the side of the detector, that aids in avoiding the detector from being saturated by X-rays going directly into the detector. The maximum opening is 5°, but if the starting 2theta is less than 10°, the PSD opening should be about half of the starting 2theta.

6.26 Then, click on the Instrument Components tab.

6.27 Before running the scan, you will want to click on the check box next to the detector options to adjust the discriminators, if necessary.
6.28 If the sample contains Fe, Co, or Ni, then they will absorb the copper radiation and emit their own radiation, causing for noise in the baseline. To avoid this, the lower discriminator in the Detector Settings will need to be changed from 0.110 to 0.190. This will omit the lower energy X-rays from the Fe, Co, or Ni in the sample. Click on Apply.

6.29 If the sample needs to be rotated during the data collection, that option is taken care of here. Do not pick a value beyond 20, otherwise the powder will no longer reside in the sample holder and the instrument will need to be cleaned. Most samples will not need to be rotated.
6.30 Then go back to the commander window and click on Start to begin the data collection.

6.31 Upon clicking on start, you will hear a click and the yellow lights on top of the instrument will illuminate. A red light on the left side of the instrument will also illuminate, indicating that the X-rays are on.
6.32 After a few seconds, you can begin to see the collection of data under the Main Display tab. A light on the interment will also indicate that it is busy and you will hear the instrument moving around as the data are collected.

6.33 When the scan has completed, you will see a note on the bottom right of the window saying: "Job finished" You will also see that the Busy light on the left side of the instrument is no longer illuminated.
6.34 To save the data, Click on File -> Save Result

6.35 The save option are .brml file which is a Bruker diffraction data file that is only readable by the PXRD software. The .raw file can be used with other X-ray diffraction programs. The data can also be saved as a .txt file for analysis in Origin or Excel.
6.36 If the method used is desired to be used for other powder X-ray diffraction samples, the method can be saved by clicking on File-> Save Experiment.
6.37 Before trying to work up your data, if it is a known compound, you will want to reference your spectrum using the Reference and Offset Determination option under the Commander menu.
6.38 After selecting the reference option, a window will appear asking you to pick a peak and then define the offset of that peak.

6.39 When you have saved your data and/or your method, you will want to remove your sample. To do that, you will need to turn off the X-rays. This can be done under the instrument status tab. Click the X-rays Off icon, and the yellow light on the instrument will click off.
6.40 When the X-rays are turned off, then the instrument door can be opened by pushing down on the handle and pulling out the lever. The lever sometimes sticks, so if it doesn’t immediately release the door, please press down on the door handle a little more and pull the release lever.

6.41 The door will slide up allowing for the removal of the sample. Pull down on the sample holder latch, which will cause the sample to lower. Remove the sample after it has lowered.
4.34 After the sample has been removed, clean up any mess left over from the sample and close the door, leaving the sample holder in the down position.

6.42 If another sample is desired to run, the next sample can be inserted at this time and you can jump to step 6.12 to begin data collection.

6.43 When the data collections have been completed, enter the ending time in the instrument logbook and describe any issues that were encountered during the data collections, if any.
7 Data Workup on the Bruker D2 Phaser Powder X-ray Diffractometer

7.1 To determine the d spacings and the reflections of the collected data, the data needs to be opened in the Diffrac.suite EVA, which is opened by clicking on the icon on the bottom of the screen.

7.2 Upon opening the program, you will then need to import the data, by clicking on the File menu and selecting the Import from Files option.
7.3 To begin analyzing your data you will need to start with a Peak Search. To do this, Click on the Peak Search option on the left side of the spectrum.

7.4 This will bring up a window that will ask to set the thresholds for the peak search. The default values are often good enough. Click on the Append to List button.
7.5 To obtain data on the peaks, click on the Append to List button.

7.6 You will then see another window show up behind the 1D View window. The window will be called the Peak Column View. Click on this window to see information on the peaks found in the spectrum.
7.7 If you scroll over in the table, you can find the gross intensity of the peak, the relative intensity of the peak, you will also find the d-Value (in Å).

7.8 If the sample is known or a mixture of known samples, a database search can be done on the collected data.

7.9 To search the connected databases, click on Search/Match (peak list) option on the left of the screen under the Tool option.
7.10 A window will then open asking for selection of the databases to be searched. To select the databases to be searched, click on the arrow and select the databases to be used.

7.11 The database search also has options on whether the results are limited to certain elements or materials by clicking on the Chemical option.
7.12 After selecting the desired option, click on Match to search the databases. This will take a few minutes.

7.13 When the search has been completed, the results will be sorted by the Figure of Merit (FOM) on how well the measured data matches the data in the database.
7.14 Clicking on one of the options provides information on the unit cell parameters, the crystal system, the space group, and other crystallographic parameters.

7.15 To obtain the hkl values for the literature sample, click on the [hkl] Generator. Make sure that the sample found in the database search is selected.
7.16 A window will open listing the crystallographic properties and lattice parameters of the database sample.

7.17 Click on Make DIF and close the [hkl] Generator window.

7.18 Click on the D8 View under the Create option to obtain the hkl values for the sample found in the database.
# 8 Powering Down the Bruker D2 Phaser Powder X-ray Diffractometer

8.1 To shut down the instrument, click on the Windows icon in the bottom left corner of the computer screen to bring up the Start window. Click on the power icon and select Shut Down.

8.2 Wait until you see the following message before turning off the power switch on the back of the instrument (see step 6.3 for power switch location).
9 Training

All users are required to provide the NUCS Core Facility Staff with records of completion of the WSU Radiation Safety Office Training Courses #1-7 & 10 (https://rso.wsu.edu/wsu-radiation-safety-training/), prior to being trained on the use of the Bruker D2 Phaser Powder X-ray Diffractometer.

Instrument Trainers

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Training on the Bruker D2 Phaser Powder X-ray Diffractometer consists of the safe use of the instrument, collection of a single dataset on a corundum sample, data analysis using the Diffrac.suite EVA software, discussion of the interlock system and safety features of the Bruker D2 Phaser Powder X-ray Diffractometer, making instrument reservations in iLab, submitting an instrument problem report, powering the X-ray tube up and down, keeping records of use in the instrument logbook, and data transfer/access.

The form that is completed during a training session is seen below: