Bruker D4 XRD Quick Start Guide

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Introduction

The X-Ray Laboratory onboard the R/V *JOIDES Resolution* (Foc's'le deck) performs diffraction analyses of minerals and rock powders. The laboratory uses a Bruker AXS D4 Endeavor XRD (a.k.a. Bruker or D4) and a PANalytical Aeris XRD (a.k.a. Aeris) diffractometers (*Figure 1*).

Bruker-associated softwares, DIFFRACplus XRD CommanderEVA, and TOPAS, allow for powder diffraction analysis of minerals, including peak-matching and mineral and chemical compound identification. XRD scans from the Bruker can also be analyzed using HighScore Plus software (used for the Aeris measurements). The X-Ray lab provides scientists with a quick and reliable tool for mineral identification; particularly useful for identifying bulk mineralogy, clays, fine-grained minerals or mixtures of secondary minerals. In addition, XRD can be used to determine mineral proportion.

A.

B.

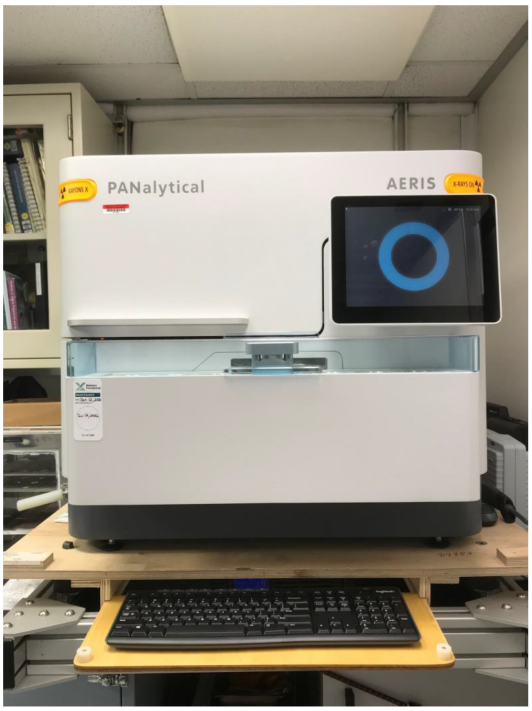

Figure 1. A. Bruker D4 Endeavor XRD. B. PANalytical Aeris XRD in the X-Ray lab

Apparatus and Materials

See Bruker D4 XRD User Guide for information on the Bruker D4 Endeavor X-Ray diffractometer and Aeris Advanced User Guide for information on the PANalytical Aeris X-Ray diffractometer.

Sample Preparation

Sample preparation techniques may include the following:
–Drying and powdering solid samples
–Mounting smear slides for small samples
–Removing carbonate from high-carbonate samples
–Separating the clay fraction from larger grain size particles
–Expanding swelling clays with ethylene glycol

Solid Sample Preparation

Solid samples are prepared for X-ray diffraction by grinding, which can be accomplished by several different methods. Appropriate method is dependent on sample matrix, size, and/or quantity:

- Soft sediment: Agate mortar and pestle
- Hard solids: Mixer Mill
- Hard solids in bulk quantities: Shatterbox. Hard samples too large for the Shatterbox are preliminarily crushed with the X-Press

Grinding Solid Samples

1.	Freeze-dry sample(s) for at least 12 hr before grinding.
2.	Grind solid samples to a fine talc-like powder using one of the following methods: –Agate mortar and pestle –Spex Shatterbox –Spex Mixer Mill (tungsten carbide, hardened steel, agate, or alumina ceramic) –X-Press
3.	Transfer the powdered sample to an appropriate labeled glass bottle or sample bag.
4.	Unless the sample material is very small (see next section); Select the steel or plastic XRD sample holder.
5.	Fill an empty XRD sample holder with enough powdered sample. Gently press the powder flush with the sample holder using a glass slide. *Note: The surface of the powder must be smooth. Remove excess powder from the sample holder edges and carefully place in the appropriate XRD slot.*

More information on bulk sample preparation is found at:

XRD Sample Preparation of Bulk Sediment

XRD Sample Preparation Hard Rock

Sample Slurry/Smear Slide Mount for Small Sample Amounts

For a very small amount of sample material (i.e., end of a tooth-pick), samples may be ground to a fine talc-like powder and smeared onto a quartz disk insert. Although not useful for semi quantitative analysis, this method is useful for rapidly determining bulk mineralogy.

1.	Grind the sample to a talc-like powder (<0.062 mm).
2.	Place a small amount of sample in the center of the zero background Si disk. Adding a quartz disks behind the Si disk will adequately fill the sample holder.
3.	Add 2–3 drops of acetone, isopropyl alcohol, or distilled water to the sample. *Note: Acetone and Isopropyl alcohol dry faster than water.*
4.	Create a thin layer of sample material using a glass rod (rolling it over the sample works well) or using a plastic or glass disposable pipette.
5.	Place sample in a desiccator to dry if water was used. Samples done with acetone or isopropyl alcohol will dry very quickly.

More information on sample slurry/smear slide for small sample amounts is found at:

XRD Sample Preparation for a small amount of material

Clay Separation Sample Preparation

Removing Carbonates before Clay Separation

In some sediments, to identify clay minerals it is necessary to dissolve carbonates. The goal is to remove as much carbonate as possible in order to analyze and isolate the material contained within.

Hydrochloric Acid Treatment

The simplest method for removing carbonate is treatment with HCl. However, treatment using strong acids can attack the structure of the clay minerals, particularly tri-octahedral minerals. Be aware that this treatment may affect clay crystallinity.

1.	Place undried sample on a glass slide or quartz disk.
2.	Using a Pasteur pipette, slowly drop 2 M HCl on the sample until bubbling/fizzing stops.
3.	Desiccate and transfer sample to sample holder for analysis.

Acetic Acid Treatment

A slightly more involved, but less destructive, method (from Kitty Milliken, UT-Austin) is as follows.

1.	Place ~2 cm^3 of undried sample into a centrifuge tube with 25 mL of acetic acid (10% solution).
2.	Mix well, and let sit until the reaction ceases (using the agitator in the chem lab helps).
3.	Shake well again to ensure the reaction has stopped (i.e., no more bubbles).
4.	Spin sample in the centrifuge (15 min at 1500 rpm)
5.	Decant the acetic acid solution and dispose of the acid solution properly.
6.	Add 25 mL of DI to the centrifuge tube and centrifuge again for 15 min at 1500 rpm.
7.	Decant the clear water.
8.	Repeat the "wash cycle" (Steps 5 and 6) with DI. Wash at least 3 times.

Separating Clay

There are various methods for separating clay from coarser material. Those listed below are methods used onboard. Discuss with the scientist(s) if other methods should be used.

"Quick and Dirty" Clay Separation Method: Not for Semi-Quantitative Analysis

1.	Add 25 ml of 1% Borax solution to the clay plug
2.	Dismembrate the sample (machine is auto set on time), to remove the >2 m clay fraction
3.	Centrifuge for 4 mins at 750 rpm, decant the suspended liquid into a separate centrifuge tube (you should end up with a ~full centrifuge tube of suspended clay)
4.	Repeat steps 1-3 on the remaining >2 m fraction
5.	Centrifuge the <2 m fraction for 15 mins at 1500 rpm to remove the Borax solution
6.	Decante and add 25 ml of nanopure water
7.	Centrifuge for 60 mins at 3000 rpm, decant the liquid before loading onto a zero background silica disk

Treating with Ethylene Glycol

Ethylene glycol can be used to expand swelling clays (e.g., smectites, montmorillonite, nontronite and beidellite), some mixed-layer clays, and vermiculite as an aid to mineral identification. There are two ethylene glycol treatment methods: Vapor treatment and Quick treatment.

Vapor Treatment

The advantage of the vapor treatment is less disturbance of the sample and less amorphous scattering of X-rays by excess liquid.
Note: Glycolation may only last 4 hours after the samples are removed from the glycolation container.

1.	Find the "Glycolator" container stored in the ICP prep sink cupboard.

2.	Pour ethylene glycol to a depth of ~1 cm in the bottom of the container.
3.	Place the samples onto the shelf
4.	Place glycolator (with samples) in oven at 60°–70° overnight.
5.	Keep samples in glycolator until ready to analyze.

Quick Treatment

1.	Using a glass rod or eye dropper, apply a drop of ethylene glycol directly to the surface of the sample mount.
2.	Samples are ready to be analyzed as soon as the glycol is uniformly absorbed. *Note: Excess ethylene glycol may be gently mopped up with a lab tissue.*

More information on Clay Separation is found at:

XRD Sample Preparation Clay Separations

Scanning Samples

Samples can be scanned with either the D4 or the Aeris. Samples are usually front-loaded for the D4 and are back-loaded for the Aeris.

As the XRD scanning procedure for these two instruments differ, the user is referred to the following guides for more information regarding how to run samples for XRD analysis.

For the Bruker D4 XRD: Bruker D4 XRD User Guide

For the PANalytical Aeris XRD: Aeris Advanced User Guide

Uploading Files to LIMS

The XRD files are uploaded using the MegaUploadaTron 5000 (MUT) program located on the desktop of the X-Ray lab computer.

- For samples scanned with the D4, there are three files for each scan to upload: a **.raw** file (readable by **Diffrac.EVA** software), a **.uxd** file (which can be read by other software such as **HighScore Plus**), and a **.png** file (diffractogram).
- For samples scanned with the Aeris, there are two files for each scan to upload: a .**xrdml** file (readable by **HighScore Plus** software), and a **.jpg** file (diffractogram).

QA/QC

The corundum standard NIST 1976 should be run every expedition for quality assurance and quality control. See the XRD Bruker User Guide for more information on running and evaluating this standard.
Several other standards (powdered concentrates of minerals) are located in the XRD standard drawer in the X-Ray laboratory. Discuss which, if any, standards the scientists would like to have measured.

See the Aeris Advanced User Guide for QA/QC.

Health, Safety, and Environment

Physical Hazard Warnings

Danger: Radiation

The direct beam of the X-ray source is very intensive. Exposure to radiation for even a fraction of a second can cause severe burns. Longer exposure can cause severe or even lethal injury.
Emitted radiation is minimized by shielding and safety equipment to be <2.5 µSv/h during operation. The enclosure of the diffraction system serves as protection against the scattered radiation produced during the measurement.

Danger: High-Voltage

Voltages up to 50 kV are generated, but they are not accessible from the outside of the system. High voltages exist in the high-voltage generator, the X-ray tube, and the high-voltage cable.

Caution: Electrical Shock

When equipment is connected to the mains supply, some terminals of the mains distribution unit may be live. Switch off the external mains supply before opening the side panel; it is not sufficient to simply turn the Power Off button.

Caution: Moving Mechanical Components

The cover of the sample magazine can be opened at any time during measurement. When the cover is open, sample handler drives stop and stay frozen until the cover is closed again; however, active measurements being made inside the X-ray enclosure will continue.
If the S604 key switch is activated, sample handler drives will not stop when the magazine cover is open. The drives inside the radiation enclosure will continue to run even if the front or rear panel is removed. Do not touch any moving components when the key switch is activated.

Danger: Injury

Goniometer components move quickly during operation. If parts of the radiation enclosure are removed, the goniometer may be accessible during operation.
When opening or closing the sample magazine, hold the cover with your hand until the final open or close position is reached. Do not release the magazine cover in an intermediate position.

Danger: Beryllium

Do not touch the front window of the X-ray detector or the X-ray tube, as they contain beryllium. Beryllium is potentially hazardous if ingested, inhaled, or absorbed through the skin.

Warning: Batteries

Disposal of batteries from electronic boards must comply with safety regulations.

Emergency Stop

- Bruker D4 Endeavor XRD: The Emergency Stop Button located on the front of the D4 Endeavor, when pressed, stops all control electronics, the high-voltage generator, and all components connected to the three mains sockets on the mains distribution unit. The X-ray source is turned off and all moving drives will stop immediately. Use only in an emergency.
- Malvern PANalytical Aeris XRD: Switch off the instrument by pressing the Power button and turning the HT Keyswitch counter-clockwise.

Chemical Hazards

Exist for Clay Separation procedure. Refer to XRD Sample Preparation Clay Separations.

Credits

This document originated from Word document XRD_QSG_374.doc (see Archived Versions below for a pdf copy) that was written by H. Barnes and K. Bronk; later edited by N. Lawler and A. Armstrong. Credits for subsequent changes to this document are given in the page history.

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