

X-ray Diffraction (XRD)

Instrument:

Rigaku D max B X-ray Diffractometer

Suggested Uses:

Analysis and identification of mineral, organic, and inorganic materials having a crystal structure. Samples may include but are not limited to:

Arson materials

Explosives

paints

powders

pyrotechnics

drugs

metals

Note: XRD manuals, logs, and reference materials are located at the XRD user station.

X-ray Diffraction Calibration Procedures

Calibration of the X-ray Diffractometer will be performed a minimum of once per month. However, calibration is not limited to once per month and can be performed as needed.

Load quartz standard slide into sample chamber.

Using “**X-ray Diffraction Operation Guidelines**”, acquire a diffraction pattern for the quartz standard. For calibration purposes, ensure that the following variables are set as follows:

kV=30.0 mA=20.0

axes to scan: coupled

2 theta start = 5.0 2 theta end = 65.0 step size = 0.05 deg/min = 6.0

scan mode: continuous scan

The auto file ID should be named by typing “cb” followed by the numerical abbreviation for the date on which the calibration is performed. For example if the calibration procedure is performed on May 7, 2000 then the auto file ID should be named “cb050700”.

Once the diffraction pattern is acquired, use “**X-ray Diffraction Operation Guidelines**” and the “**Materials Data JADE XRD Pattern Processing**” manual in conjunction with the **MDI JADE 5.0** software in order to identify the diffraction pattern. Use the **Analyze** and **Find Peaks** options to locate major phases in the diffraction pattern (the default variables are suitable for finding peaks). Use the **Identify** and **Search/Match** options to identify the diffraction pattern (search the **Inorganics Subfile** - all other default variables are suitable for identification purposes).

Print the Search/Match hit list.

Print the diffraction pattern.

Staple these printouts together and file them in the XRD Calibration Folder.

Record the date, calibration standard, search/match results, and user initials in the XRD Calibration Log.

NOTE: “X-ray diffraction Operation Guidelines”, “Materials Data JADE XRD Pattern Processing” manual, XRD Calibration Log and Folder, and other XRD reference materials and Logs are located at the XRD user station.

Approved_____Date_____

X-ray Diffraction Operation Guidelines

Data Acquisition

Read **XRD Safety Concerns**

Load the sample into the sample chamber. Ensure the radiation shield is completely closed. (See XRD Safety Concerns)

Use **DATASCAN** software. Double click on **DATASCAN** icon.

Click on **Controller**.

Click on **More Options**.

Click on **Power On**.

Click on **X-ray On**.

Under generator control, the voltage can be adjusted by typing in the appropriate setting and then clicking on **Set kV**. The XRD generator will ramp up to the specified voltage. Likewise, the current can be adjusted by typing in the appropriate setting and then clicking on **Set mA**. The XRD generator will ramp up to the specified current.

Click on **Close Dialog**.

Click on **Scan**.

Click on **Setup**.

Type in the desired scan conditions.

Click on **Close**.

Type in the **Auto File ID** using 8 characters. If need be, further identify the scan by typing a description (ie. case number, item number, physical description of sample, etc.) beside **Scan ID**.

Click on **Start Scan**.

X-ray Diffraction Pattern Processing

Use **MDI JADE 5.0** software. Double click on **MDI JADE 5.0** icon.

(Note: Data is automatically imported from DATASCAN)

Click on **File**. Click on **Patterns**.

Double Click on the file name that corresponds to the diffraction pattern to be viewed, analyzed, and/or processed. That diffraction pattern will be displayed.

To process, analyze, and/or search the diffraction pattern against the Powder Diffraction File (PDF) database see the following chapters in the **Materials Data JADE XRD Pattern Processing** manual:

Chapter 5	Basic Pattern Processing
Chapter 6	PDF Access and Retrieval
Chapter 7	Search/Match for Phase Identification
Chapter 8	Advanced Pattern Processing
Chapter 9	Quantitative Analysis

To close **MDI JADE 5.0**, click on the **X** in the upper right corner.

Save current work if necessary.

Shut Down Procedure

Use **DATASCAN** software.

Click on **Controller**.

Click on **More Options**.

Under generator control, set the current to **2.0mA** and click on **Set mA**. The XRD generator will ramp down to 2.0mA. Then set the voltage to **20.0 kV** and click on **Set kV**. The XRD generator will ramp down to 20.0 kV.

Click on **X-ray Off**.

Click on **Power Off**.

Click on **Close Dialog**.

To close **DATASCAN**, click on **X** in upper right corner.

All XRD activity must be recorded in the **XRD User Log**.

Approved _____ Date _____

X-ray Diffraction Safety Concerns

The primary safety concern in the operation of an X-ray Diffractometer is the injurious exposure to X-ray radiation. XRD utilizes an intense beam of X-ray radiation. The Rigaku D max B X-ray Diffractometer, however, is enclosed in a radiation shield which is designed to protect the human body from exposure. This radiation shield is equipped with a fail safe device. If the radiation shield is opened the fail safe will automatically close the X-ray tube shutter and sound an alarm. If entry into the radiation shield must be gained at any time during the operation of the X-ray Diffractometer, the fail safe release key must be engaged. Radiation badges and or rings should be worn at all times when operating the X-ray Diffractometer so that any exposure to X-ray radiation can be detected.

The X-ray tube utilizes a high voltage cable in the production of X-rays. Therefore, an electrical shock hazard does exist. Any maintenance on the X-ray Diffractometer should only be performed after the high voltage cable is de-energized.