

X-Ray Diffractometer: User Guide

Manual Information

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In This Manual

<i>Introduction</i>	1
<i>Apparatus, Reagents, & Materials</i>	2
<i>Sample Preparation</i>	5
<i>Running Samples</i>	7
<i>Quality Assurance/Quality Control</i>	9
<i>Health, Safety, and Environment</i>	10
<i>Maintenance & Troubleshooting</i>	12

Introduction

The X-Ray Laboratory onboard the *JOIDES Resolution* performs diffraction analyses of minerals and rock powders. The laboratory uses a D4 Endeavor X-Ray Diffractometer from Bruker AXS. Associated software, DIFFRACplus and TOPAS, allow for powder diffraction analysis of minerals and rock powders, including peak-matching and mineral and other chemical compound identification facilities. The XRD lab provides the scientist with a quick and reliable tool for mineral identification; it is particularly useful for identifying fine-grained minerals or mixtures of alteration

minerals that cannot be easily identified with other techniques onboard. In addition, XRD can be used to determine the proportion of the different minerals present.

Theory of Operation

A mineral is a three dimensional structure that forms a crystal lattice. When a focused X-ray beam is directed onto the crystal structure of a mineral, part of the beam is diffracted, much like light traveling through a prism or a water droplet producing a rainbow. X-rays are diffracted differently depending on the atomic composition and arrangement within the crystal lattice. These are the fundamental characteristics of minerals that allow mineral identification through X-ray diffraction. Each mineral has a unique fingerprint that is determined based on a characteristic set of d-spacings (space between adjacent planes of atoms in the crystal lattice). In X-ray diffraction, X-rays are generated in a vacuum tube and directed to a powdered sample. When the X-rays hit the powdered sample, they are diffracted onto a detector. The X-ray detector then detects the signal and converts signal to a count rate. The angle between the X-ray tube, sample, and detector are varied during measurement to produce an X-ray scan (counts per second versus the angle between X-ray tube, sample, and detector). Using the angle between the X-ray tube, sample, and detector (2θ , which can be measured) and the wavelength of a generated X-ray beam (λ , which is known based on material generating the X-rays) the scientist can determine the d-spacings by using Bragg's law:

$$n \lambda = 2 d \sin\theta$$

n = order of the diffracted beam

λ = wavelength of the incident X-ray beam

d = distance between adjacent planes of atoms (d-spacings)

θ = angle of incidence of the X-ray beam

The d-spacings generated by an X-ray scan provide the unique fingerprint of the mineral or minerals present in the sample.

References

Cullity, B.D., 1978. *Elements of X-Ray Diffraction*, Second Edition. Addison-Wesley Publishing Company, Inc., Massachusetts.

Bish, D.L., and Post, J.E. (Editors), 1989. *Reviews in Mineralogy Volume 20, Modern Powder Diffraction*. The Mineralogical Society of America, Washington, D.C.

Apparatus, Reagents, & Materials

Laboratory Apparatus

- Haskris water-water chiller for X-ray tube
- Cahn Model 29 or 31 microbalance
- Mettler-Toledo XS204 dual-balance system
- Powdering equipment:
 - Agate mortar and pestle
 - Ball mill
 - Shatterbox
 - Rock Labs hardened steel masher
- Glass bottles

- Sonic dismembrator
- Glass rod
- Glass beakers
- Pastuer pipette/eye dropper
- Centrifuge and glass centrifuge tubes
- Dessicator

Reagents

- Ethylene glycol (clay glyconation)
- HNO₃ (glass cleaning)
- 2 M HCl: 16.4% v/v (carbonate dissolution)
- Glacial acetic acid, 10% solution (v/v) (carbonate dissolution)
- Distilled (reagent) water (DI)
- 1% Calgon solution (w/v)

Instrument Hardware

- Bruker AXS D4 Endeavor X-ray diffractometer (XRD)
 - Goniometer
 - X-ray source (sealed vacuum tube with line focus)
 - Tube housing (ceramic body that protects the tube and shields from X-rays)
 - Mount
 - Sample holder
- Vantec-1 detector
 - Optics
 - Slit systems

X-Ray Diffractometer

The Bruker AXS D4 Endeavor XRD can analyze powder, liquid, and solid samples in a variety of sample holders. The sequence of the beam path is X-ray source > primary X-ray optics > sample > secondary X-ray optics > detector.

Goniometer

The goniometer has 2 circles: the theta circle, which sets the sample position, and the 2theta circle, which moves the detector. The position of the X-ray tube remains fixed. The goniometer center is defined by the concentric circle axes.

Goniometer Specifications

Component	Specification
Measuring circle diameter	401 mm
Operating mode	Vertical, $\theta/2\theta$
Max angular range	$-8^\circ < 2\theta < 170^\circ$
Smallest step width	0.0087°
Reproducibility of $\theta/2\theta$ angle	$\pm 0.0002^\circ$
Absolute accuracy ($\theta/2\theta$)	$< \pm 0.005^\circ$

Sample Holders

Sample holders for powdered XRD samples are steel or plastic. The selection of a sample holder depends on the volume of sample to be analyzed.

Amount of Powdered Sample	Sample Holder
Large	Steel
Small	Plastic
Smaller	Steel with quartz or zero-background Si spacer
Smallest (vein/vesicles, scrapings)	Slurry (see Sample Slurry/Smear Slide Mounting for Small Sample Amounts)

Detector

The VANTEC-1 detector features the fastest simultaneous recording of X-ray diffraction patterns within a wide 2θ angular range. For powder XRD, measurement time is reduced by a factor of up to 100 in comparison to other detectors.

Detector Specifications

Component	Specification
Active area	50 mm x 16 mm; 1600 pixels
Max 2θ range covered	12° at 435 mm diameter; 11° at 500 mm
Usable wavelength	Cr-K α to Mo-K α
Max local count rate	400,000 cps
Spatial resolution	<50 μ m; >1600 channels
Gas fill	3.04 bar Xe-CO ₂ ; no external supply needed
Power rating	120 W
Ambient temperature	41°–104°F (5°–40°C)
Operating temperature	57°–93°F (14°–34°C)
Relative humidity	Max 80%, non-condensing

Detector Optics

- Detector window slits
- Debye slits
- K β filter: suppresses characteristic K β radiation.
- Antiscatter slits: reduce primary air scatter, which influences diffraction background patterns
- Soller slits: reduces primary and secondary air scatter
- Window opening scales

Instrument Software

- DIFFRAC.SUITE.EVA, version 1.4 software for evaluation
- DIFFRAC.TOPAS version 4.2.0.1 software for diffractogram analysis

Sample Preparation

Sample preparation techniques 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 treatment

Preparing Solid Samples

Solid samples are prepared for X-ray diffraction by grinding, which can be accomplished by several different methods, depending on the sample matrix, the size of the sample, and/or quantity of prepared material needed:

Sample Matrix	Use...
Soft clay	Agate mortar and pestle
Hard solids	Ball mill
Hard solids in bulk quantities	Shatterbox
Hard sample too large to fit in the shatterbox	Rock Labs hardened steel masher

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)
 - Rock Labs hardened steel "masher"
3. Transfer the powdered sample to an appropriate labeled (EXP_SITE_HOLE_CORE_SEC_INT) glass bottle.
4. Select the steel or plastic sample holder unless the sample material is very small (see [Sample Slurry/Smear Slide Mounting for Small Sample Amounts](#)).
5. Place enough powder from the labeled sample bottle to fill an empty sample holder. Gently press the powder flush with the sample holder using a glass slide. The surface of the powder must be smooth. Remove excess powder from the sample holder edges and carefully place in the appropriate XRD slot.

Sample Slurry/Smear Slide Mounting for Small Sample Amounts

The following technique is modified from the *U.S. Geological Survey Open-File Report 01-041, A Laboratory Manual for X-Ray Powder Diffraction*. 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 one of the quartz disk inserts. Although not useful for semi-quantitative analysis, this method is useful for rapidly determining bulk mineralogy.

Preparing Smear Slurry/Smear Slide

1. Grind the sample to a talc-like powder (<0.062 mm).
2. Place a small amount of sample in the center of a quartz or single crystal quartz disk.
3. Add 2–3 drops of acetone or distilled water to the sample. **Note:** Acetone dries faster than water will.

4. Spread the sample to a thin layer with a glass rod (rolling it over the sample works well).
5. Place sample in dessicator to dry before running in the XRD.

Removing Carbonates before Clay Separation

It may be necessary to dissolve the carbonates in some sediments before the clay minerals can be identified. The goal is to remove as much carbonate as possible to isolate the material contained within the carbonate for analysis.

Hydrochloric Acid Treatment

The simplest method for removing carbonate from the sample is treatment with HCl. However, treatment with strong acids to remove carbonate can attack the structure of the clay minerals, particularly trioctahedral minerals. Be aware that this treatment may affect clay crystallinity.

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

Acetic Acid Treatment

A slightly more involved method that removes carbonates (from Kitty Milliken, UT-Austin) is as follows:

1. Place ~2 cm³ of undried sample into a centrifuge tube with 25 mL of acetic acid (10% solution). Mix well, and let sit until the reaction ceases.
2. Shake well again to ensure the reaction has stopped (i.e., no more bubbles).
3. Spin sample in the centrifuge (15 min at 1500 rpm)
4. Decant the acetic acid solution and dispose of the acid solution properly.
5. Add 25 mL of DI to the centrifuge tube and centrifuge again for 15 min at 1500 rpm.
6. Decant the clear water.
7. Repeat the “wash cycle” (Steps 5 and 6) with DI.
8. Place washed sample in a large beaker with some distilled water and a little 1% Calgon.
9. Suspend the clay material by placing the beaker in a sonic dismembrator for ~1 min.
10. Do not let the sample heat up.
11. Transfer the sample to a clean centrifuge tube and spin for 5 min at 1000 rpm to remove the >2 μm size fraction from suspension.
12. Remove the <2 μm size fraction by collecting the top 1 cm of solution with an eye dropper. If it is necessary to resuspend flocculated clay particles using the dismembrator and more Calgon solution, 15 min at 1500 rpm in the centrifuge may not settle the <2 μm particle size. Increase the speed to as much as 5000 rpm to remove the clay from the Calgon solution.
13. Make an oriented clay mount by placing 2–3 drops (enough to cover the quartz disk) of clay suspension onto a glass slide and let dry in a dessicator.

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. In centrifuge tube, mix a small amount of bulk sample (~5 mL) (fresh, not dried) with 1% Calgon solution. Use an ultrasonic bath or dismembrator, if necessary.
2. Centrifuge the Calgon solution/sample mix at 1000 rpm for 5 min to remove the >2 μm size fraction.

3. Decant the Calgon solution (containing suspended clays) into a new centrifuge tube, and spin it at 1500 rpm for 15 min to remove the remaining <math><2\ \mu\text{m}</math> clay-size fraction.
4. Decant the Calgon solution and wash the clay residue with distilled water.
5. Spin-down again at 1500 rpm for 15 min. Repeat steps 4 and 5 as necessary to remove the Calgon.
6. Make an oriented clay mount by placing 2–3 drops (or enough to cover the quartz disk) of solution onto the quartz disk and let dry in the dessicator.

Treating with Ethylene Glycol

The following techniques are modified from the *U.S. Geological Survey Open-File Report 01-041, A Laboratory Manual for X-Ray Powder Diffraction*. 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
- Quick

Vapor Treatment

The advantage of the vapor treatment is less disturbance of the sample and less amorphous scattering of X-rays by excess liquid than in the quick method.

1. Find the “Glyconator” 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 to be glyconated onto the shelf.
4. Place container (glyconator) in an oven (60°–70°C) overnight.
5. Keep samples in glyconator until ready to analyze. Glyconation may only last for 4 h after the samples are removed from the glyconation container.

Quick Method 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 on the sample mount. Excess ethylene glycol may be gently mopped up with a lab tissue.

References

Jackson, M.L., 1956. *Soil Chemical- Analysis Advanced Course* by Hsueh-Wen Yeh, Hawaii Institute of Geophysics, 1980.

Moore, D.M., and Reynolds, R.C., Jr., 1989. *X-ray Diffraction and the Identification and Analysis of Clay Minerals*: New York (Oxford University Press).

Running Samples

Running a regular XRD sample requires the following steps:

- Scan the samples on the XRD
- Convert the raw sample results to an .uxd file
- Export the raw scan to a PDF or PNG file
- Upload the raw, uxd, and PDF/PNG files to LIMS

Scanning Samples

Before beginning this procedure, discuss the desired scan parameters with the scientists.

1. Open *Commander* and initialize the Sample Changer by clicking on **Init SC**. The diffractometer's sample changer drives will adjust inside the instrument.
2. If the divergence slit needs to be initialized, check the box next to *div. slit* and click the **init drives** icon (robot-like icon with yellow labeled "init drives" on cursor hover). After all drives are adjusted, proceed to Step 3 to create a job list. If you hear a crash or the program gives an error, see the procedure for readjusting the sample handler in the Bruker manual. This is a lengthy process.
3. Open **Jobs** tab > **Create jobs** > **Positions**, and enter the position of the sample(s) (A1–A6 up to K1–K6).
4. Use the Text ID (e.g., cuts123456). A barcode scanner is connected to the XRD computer to enter the Sample ID.
5. Select the appropriate **instrument.dql** file under *Parameter*. If needed, use the **XRD Wizard** to set the desired 2θ span; for example, $4^{\circ}2\theta$ to $80^{\circ}2\theta$. Several .dql files are already created under *C:\Documents and Settings\daq\Desktop\XRD Expedition Files*.
6. Specify the path to save the raw results file, *C:\data\xrd\upload*. Use the sample's Text ID as the file name.
7. Select the appropriate instrument settings:
 - Script: *measureV4.vbs*, located in the *DIFFPLUS* folder (should open automatically)
 - Mode: **QL** (qualitative)
 - Time Scale: **1.0**
8. Check that the samples are loaded correctly and nothing is blocking the sample handler.
9. Click **Start**.

Converting Raw File to .uxd File

After the scan is completed, convert the .raw file into a .uxd file; the .raw file is only readable by the Eva software, whereas the .uxd file is a text file that can be read by other programs.

1. Click the *Raw File Exchange* software icon on the desktop.
2. Select the .raw file of interest from the dropdown menu under *input*.
3. Click **Convert**.
4. The new .uxd files will be saved in *C:/Data/upload*.

Printing Scan to PDF or PNG

1. Double click on the .raw file, which automatically opens the file in the Eva software.
2. Go to **File > Print**.
3. Export as a PNG file.
4. In the file save window, name the file with the text ID (e.g., plug12345.png) and **Save**.

Uploading Files to LIMS

The XRD files are uploaded using the MegaUploadatron 5000 program located in the IODP programs list under the *Start* menu. There are three files for each scan (.raw, .uxd., and .png):

1. Open the uploader.
2. Click **File > Open Upload Directory** to select the directory where the files are saved.
3. Ensure that all the files to be uploaded are checked in the uploader window.
4. Click **Upload**.
5. Check that these files did move to the archive directory and are uploaded into LIMS.

Quality Assurance/Quality Control

The corundum standard NIST 1976 should be run with every scan for quality assurance and quality control. At the beginning of each expedition use the NIST 1976 corundum standard to check instrument alignment and detector intensities. Use the Excel instrument verification spreadsheet saved in *C:\Documents and Settings\daq\Desktop\XRDDocs\Standard QAQC file name QAQC Corundum std Equipment Verification 3.3.xls*. This Excel spreadsheet also has instructions imbedded explaining how to analyze the scan in EVA.

Removing the Anti-AirScatter Screen

Before running the NIST standard, carefully remove the Anti-Air Scatter Screen. Failure to do this may result in a collision of the drives.

1. Carefully remove the cover over the sample staging area, making sure not to hit any wires.
2. This requires two people to lift the cover from the base (one person on the left and the other on the right). There are no screws holding the cover on.
3. Open **D4 tools** and click **Online Status**.
4. Click the computer icon to connect with the configuration files in the instrument.
5. Click **Positioning Drives** and **Sample Changer Y** direction.
6. Click **Go**, enter **360**, and hit **Enter**. The sample changer will move in the Y-direction to position 360. This allows you to open the front door and remove the Anti-AirScatter Screen.
7. Remove the Anti-AirScatter Screen without hitting the theta drives. If the theta drive is knocked, the instrument may signal a hard limit switch error.
8. Close the front door, and using the **Go** button, move the Y drive back to position 1. (Enter **1**, **Go**, and **Enter**.)

Scanning the NIST 1976 Standard and Verify QA/QC

1. Open scan (remember the QAQC parameter file *Corundum QAQC continuous 20 to 130.dql* ran without anti air scatter screen).
2. Remove background (subtract background).
3. Strip $K\alpha_2$ and Append.
4. Create Area and Append area for each of the scan angles (25.576, 35.149, 88.993, and 127.675 $2\theta^*$). This will insert Area information as a sub-tree entry under that scan. You will see two sub-tree entries: (1) Background subtracted scan and (2) Strip $K\alpha_2$ stripped scan.
 - To select the area around each peak, click **Select Area**, then manually enter the scan angle range. For example: the 25.756 peak type in *Left Angle = 24.7* and *Right Angle = 26.2*, click **Append this Area**. Do this for each of the angle ranges.
 - 25.576° 2θ (24.7°–26.2° 2θ)
 - 35.149° 2θ (34.0°–36.2° 2θ)
 - 88.993° 2θ (88.1°–89.7° 2θ)
5. Right click on the **Area List #** subheader for background subtracted scan and select **Create and Area Column View**. Do the same for the $K\alpha_2$ appended scan. This creates another tab on top with the Area data in column format.
6. Open the *QAQC Corundum std Equipment Verification 3.3.xls* Excel spreadsheet.
7. Enter your name and date and select Vantech-1 for the detector.
8. Enter the following values into the spreadsheet:
 - For 2θ Obs use the Chord. Min values from the $K\alpha_2$ appended scan areas;
 - For I Obs use Net Area from the background subtracted scan areas
 - For FWHM use the FWHM from the $K\alpha_2$ appended scan areas

9. The spreadsheet has the calculations already embedded. Remove background (subtract background).

Remember if you get the RED *ZI correction must be applied* message, you need to open the configuration program **Motor Drives > 2θ > Zero Refer**. Enter the current ZI value in the excel spreadsheet – the Corrected ZI value is what you need to update/change the configurations ZI value to. **Save** and **Download**. Always backup the config.file on the server.

Several other standards (powdered concentrates of minerals) are located in the XRD standard drawer in the XRD laboratory. Discuss which, if any, standards the scientists would like to have measured.

Health, Safety, and Environment

The Endeavor D4 has several safety issues that should be reviewed. Please see the *Bruker Manual D4 Endeavor X-Ray Diffractometer – Introductory User Manual* for the list of precautions.

Warnings

Danger: Radiation

The diffraction system has a strong X-ray source, and the direct beam of the 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. Ensure the enclosure is configured correctly as follows:

- The goniometer must be fixed in the interior of the radiation protection enclosure.
- The X-ray tube mount must be attached to the goniometer.
- The primary optics must be mounted.
- The radiation protection enclosure must be installed completely.

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.

To prevent electrical shock, turn off the D4 main power supply before:

- Touching components on the main distribution board
- Inserting/removing fuses
- Exchanging the fluorescent tube or USB hub
- Connecting/disconnecting electrical devices to the AC outlets
- Installing or removing an internal or external cooling unit
- Connecting or disconnecting an external water valve

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 and 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

The Emergency Stop Button located on the front of the D4 Endeavor, when pressed, stops all control electronics, 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.

Chemical Hazards

Ethylene Glycol

Ethylene glycol is toxic and should not be ingested. It is also harmful if inhaled or absorbed through the skin and eyes. Proper personal protective equipment should be used when handling this compound.

Calgon

This chemical largely consists of potassium sulfate and is not expected to be a health hazard.

Nitric Acid, Concentrated, or 10%–15% for the Water Bath

Concentrated nitric acid (~50%–70% HNO₃ v/v) is highly dangerous. It can cause severe tissue damage on contact, is highly toxic, and the fumes present similar risks of poisoning and chemical burns. When mixed with water, nitric acid liberates large quantities of heat, so appropriate care should be used when diluting this compound. This compound is also a strong oxidizing agent, so nitric acid waste should not be mixed with any organic materials. Note that the nitric acid used in the water bath is still dangerous and should be treated with the appropriate care.

Hydrochloric Acid, Concentrated, or 2 M for Carbonate Dissolution

Concentrated hydrochloric acid (~12M) is highly dangerous. It can cause severe tissue damage on contact, is highly toxic, and the fumes present similar risks of poisoning and chemical burns. When mixed with water, hydrochloric acid liberates large quantities of heat, so appropriate care should be used when diluting this compound. Note that the 2 M hydrochloric acid used in the carbonate dissolution procedure is still dangerous and should be treated with the appropriate care.

Acetic Acid, Glacial, or 10% for Carbonate Dissolution

Glacial acetic acid (~100%) is highly dangerous. It can cause severe tissue damage on contact. When mixed with water, glacial acetic acid liberates a lot of heat, so appropriate care should be used when diluting this compound. When diluted to ~10% concentration, it is very similar to white vinegar, so while it is still acidic and could cause tissue damage, it is not as hazardous.

Maintenance & Troubleshooting

Cleaning the Diffraction System

To clean the interior of the enclosure and exterior of the detector components, use dry cleaning utensils only. Do not use water or aggressive cleaning agents.

Air flow is critical to maintaining proper operation of the detector electronics. Do not place anything on the controllers that may restrict air flow. Regular cleaning includes removal of air flow restrictions, including dust.

Troubleshooting

Display data on computer screen has flatlined or is nonexistent

Ensure the detector type *VANTEC-1 PSD* is selected in the **Details** tab of the *COMMANDER* program.

Ensure the detector setting matches the factory setting and the setting in the *Config* program. Set the detector to factory settings using the **Set Detector** button.

Angular accuracy peak position data is greater than +0.01° of expected value.

Run the NIST 1976 standard (see [Quality Assurance/Quality Control](#)) and import the values into the instrument verification spreadsheet. Perform a Zi correction if needed. Calibration of the VANTEC-1 detector may be required. See the detector manual.

Poor instrument resolution (FWHM > 0.065° of the 104 reflection of NIST 1976)

Repeat alignment of the 2 DOF mount for the VANTEC-1 detector.

Scan outside software limits

Using fixed scan mode, the start to stop range is determined by the configuration and cannot exceed the geometry angle set in Config (max = 12°).
