

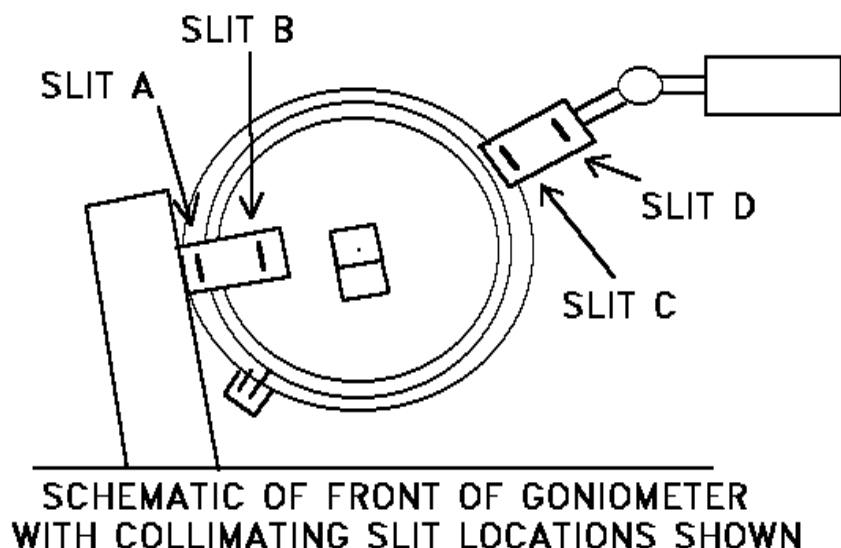
Scintag XRD Powder Diffractometer Procedures

Loading your Sample and Diffractometer Setup

Dosimeters: When working in the laboratory, always wear your dosimeter “badge” and ring(s). The badge should be clipped to clothing approximately at chest-level and the ring worn on the left hand (since this will be closest to the x-ray generator). ***Only Authorized Users who have been cleared through UNM Radiation Safety Office and are wearing their assigned dosimeters are permitted to be in the lab while X-rays are being generated.***

Samples may be loaded in a variety of sample holders depending on individual needs. These may include rectangular glass slides for slurry mounts (typically used for oriented clay mounts), plexiglass side-pack or top-pack mounts, zero-background quartz plates, or various user designed mounts for thin-film or other specialized samples. Ideal sample holders are 3 cm x 3 cm plates between 5 and 10 mm thick, but the spring mounted holder allows for considerable variation in both dimensions. The main criterion for collection of accurate data is that the top surface of your sample be at the exact same level as the top edges of the holder.

There are several user-changeable collimating slits (see diagram below with generator at left, detector at right), and it is important that users are aware of these and insert the slits appropriate to their samples and the type of data they are collecting. The default slits for most routine powder data for rock samples are as follows: Slit A: 2mm; Slit B: 4mm; Slit C: 1mm; Slit D: 0.3mm.



When your sample is loaded, make sure the door is fully closed.

Power-up and Power-down of Generator

Users must check to see that the XRD generator is powered-up before collecting data, and at the end of the day must power-down the generator. The purpose of this is to prolong tube life (since turning power on and off can shorten tube life). Please follow the procedure listed below:

1. When the first user of the day enters the lab, the power supply should be turned ***on*** and the Accelerating Voltage (***Kilovolts***) and Current (***Milliamperes***, upper left control) should be set to their idle values, ***-40 KV*** and ***10 MA***, respectively.
2. If the power supply is turned off (it shouldn't be), contact the Lab Manager (Jim Connolly) before doing anything.
3. Gradually adjust the MA (the top left knob) to ***35 MA*** over a period of a minute or so. Accelerating potential is permanently set at -40 KV. ***Do not adjust any other knobs on the power supply.*** If the KV meter does not read -40, contact the Lab Manager immediately.

Check the online calendar (<http://calendar.yahoo.com/epsxrddlab>) when done collecting data to see if someone is coming in after you. If you are the last user of the day, reduce the Milliamperes settings to the idle value of 10 MA gradually over a period of a minute or so.

Logging on and off of the EPS Network

All users of the X-Ray Diffraction system must have an account on the EPS Department Network. All computer systems in this lab run Microsoft Windows XP Professional. If you do not have an account, see the Lab Manager who will give you the appropriate paperwork to complete and create an account for you.

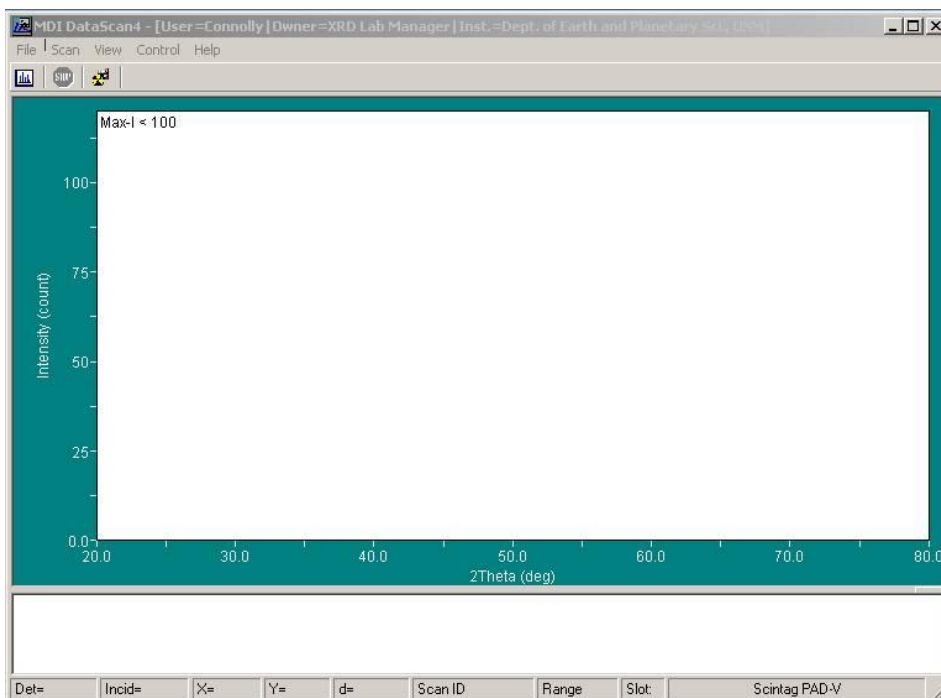
To log on for data collection, press Ctrl-Alt-Del on the data collection computer (the immediately to the right of the Diffractometer), enter your Username and Password in the appropriate boxes, then click on OK with the mouse to complete the login. Data collection (using DataScan) must be done on this system. Data analysis (using Jade software) is done on either of the other Dell systems.

When done collecting and/or analyzing data, log off the whatever systems you are using by choosing ***Log Off*** on the Start menu and complete the interaction to log off. Avoid shutting the system down; we keep all of the systems operating at all times.

One-time setting of Individual Configuration Parameters

NOTE: Each user needs to do the items in this section only once.

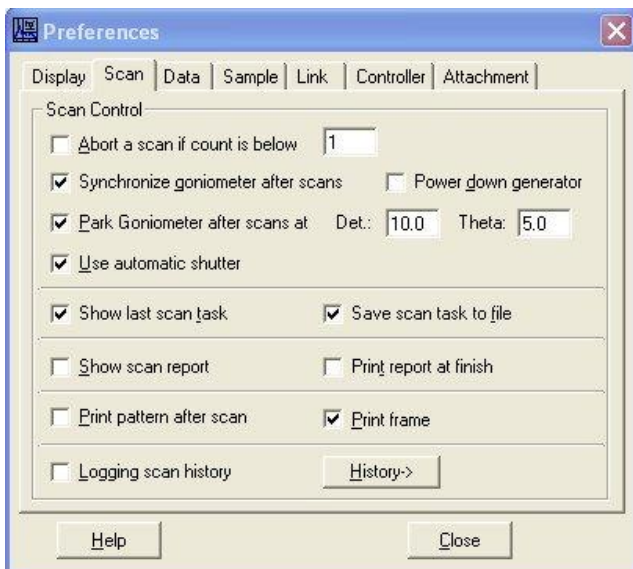
Item #1: In DataScan4 user must set their individual preferences for how the diffractometer operates. This only needs to be done one time by each user of the system, but it must be done to insure proper operation of the system. Follow the procedures below to insure that the goniometer will be parked at the 10° 2θ exchange position when



the analysis is done and to set the upper and lower angular limits.

Start DataScan4 from the “MDI DataScan4” icon on the desktop. On the startup screen (at left), select “Control” on the Menu, then “Preferences.” In this window, click the “Scan” tab

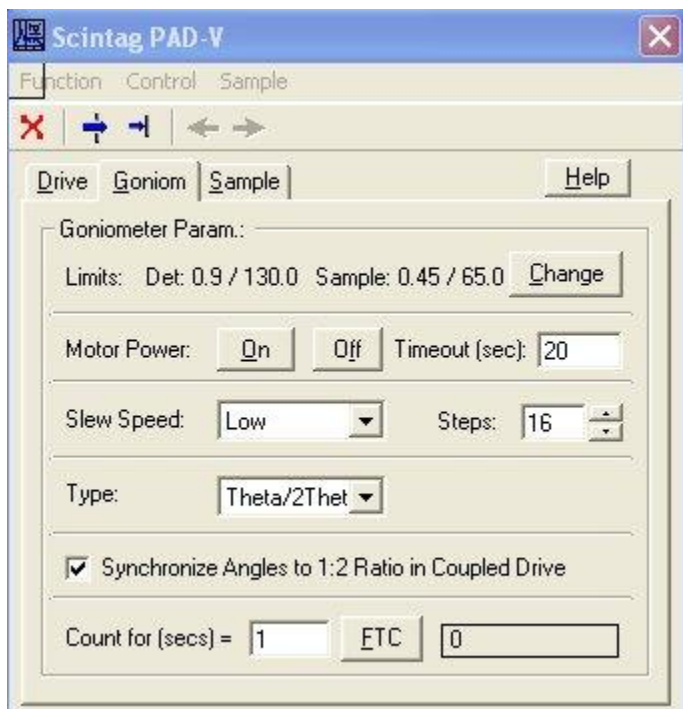
to bring up the window below.



In the window at left, **check** the box that says “Park Goniometer after Scans at” and enter 10.0 in the “Detector” box and 5.0 in the “Theta” box.

Item #2: When this has been properly set, close the “Preferences” Window to return to the main DataScan Window.

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Again select the “Control” menu in the main DataScan Window and select the “Scintag PAD-V” window shown at left.

The default detection limits will generally be set with 2° as the lower limit and 160° as the upper limit. To enable scans below 1° , click the “Change” button to bring up the screen below.



Enter the values shown at left in the boxes:
Detector: Min: 0.9; Max: 130.0
Theta: Min: 0.45; Max: 65.0

The lower limits given are the absolute minimums; setting limits lower than this can cause damage to the detector. The upper limits can be set somewhat higher ($160^\circ \pm 20$ or “Detector”, $80^\circ \pm \theta$) if required for specialized work.

Click OK to make the changes permanent. Remember, these settings only need to be changed one time for each user.

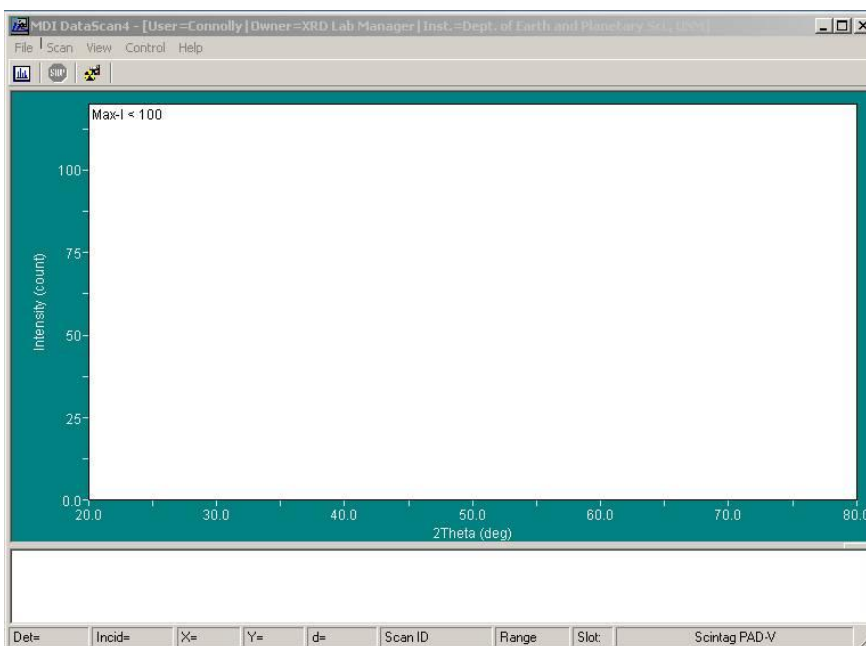
Item #3: The Windows XP “Power Scheme” must be changed so the system will not sleep if idle. Do a Right-mouse click on an open area of the desktop and select **Properties** on the menu. On the **Screen Saver** tab, select the **Power** button and choose **Always On** as your power scheme. Apply and close the Window to save your changes.

Item #4: Each user **must** establish their “Default printer” on each system they use in the XRD lab. This is **most** critical on EPSXRD2 and EPSXRD3 where Jade is installed, and **must** be done before you run Jade. It must be done on each system that you use.

From Start – Printers and Faxes, choose to “Add a Printer”. Add a Network Printer, select [\\eps2\xrdprint](#) as the printer and make it the default printer. Jade **requires** users to have a default printer set, will print **only** to the default printer and will often terminate with an error message if no printer has been defined.

Data Collection with DataScan System

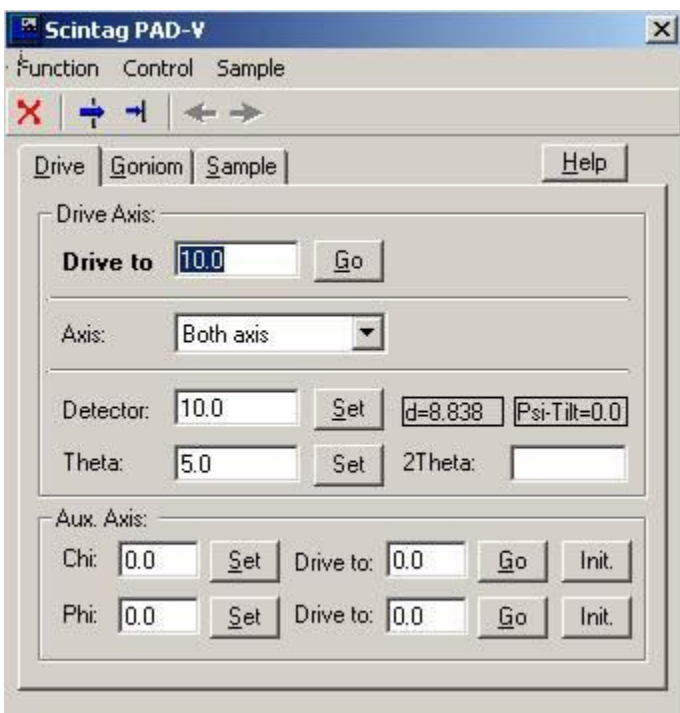
Startup and Calibration Check



After you have logged into the data collection system, double-click on the “MDI DataScan4” icon on the desktop. After a few seconds you will see a screen that looks very much like the one to the left.

The next step is to make sure that DataScan and the goniometer agree on the 2θ and θ

positions. From the DataScan menu, choose “Control” and “Scintag PAD-V”. The Window shown below should appear. Make sure the tab named “Drive” is selected.



Note the positions shown in the middle of the window for 2θ (Detector) and θ (Theta). Normally these will be 10.0 and 5.0, respectively. ***Walk over to the diffractometer*** and make sure that the positions shown are ***identical*** to those shown on the goniometer. As you face the goniometer, the inner ring reads the θ position, and the outer ring reads the 2θ position. The micrometer scales on the top of the goniometer show each degree divided into 100 divisions, the left for 2θ and the right for θ . These should both read zero (within $\pm 3/10$ of a division). ***If these conditions are met, close the***

window and skip to the section, Data Collection.

You may enter a 2θ value in the “Drive to” box to manually drive the goniometer to any desired location within the angular limits between 1° and $130^\circ 2\theta$. The blue arrows at the

top of the screen may be used to open (arrow through “window”) or close the shutter. ***All other settings in the controller window are for lab administration use only and should never be changed by users.***

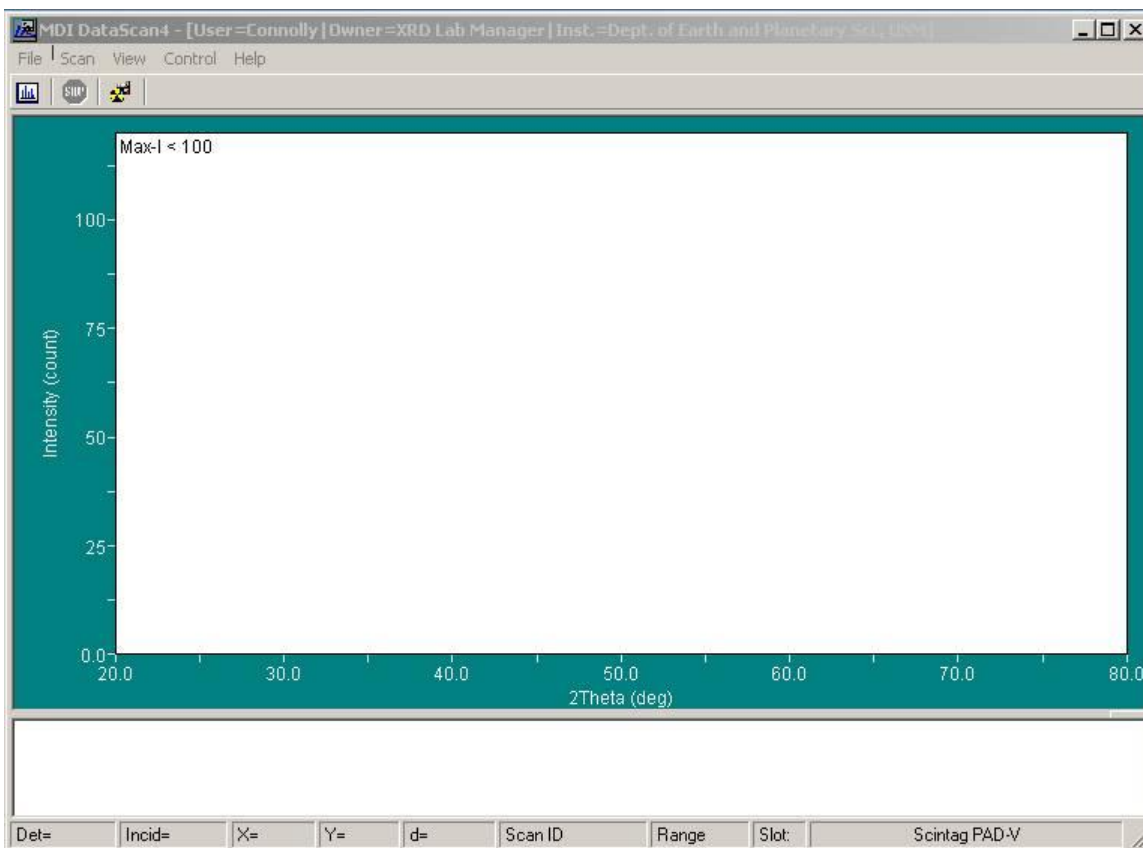
If the positions do not agree, you have two choices. You may contact the lab administrator (Jim Connolly, Connolly@unm.edu, Office Northrop 209A) who will recalibrate the angular positions or you may use the following calibration procedure:

1. In the controller window, from the “Control” menu select “Calibrate Goniom.”. The program will ask you to confirm that you want to calibrate the angle positions. A “Calibrate Angle” command will then be sent to the goniometer. This will cause θ and 2θ to be (independently) driven to the next whole-angle position.¹
2. When the goniometer has done moving, a dialog will pop up giving the angular position the software thinks are correct. Compare the actual positions with those displayed. If they do not agree, read the angular position using the large dials and the micrometer scales (using the black scales not the red ones), enter the positions to three decimal places (i.e., 10.997)². This is done for θ and 2θ independently. When you make these entries to three decimal places, the step-gearing correction will usually modify your entry in the 4th decimal place, so don’t be alarmed.
3. After this is done, enter a few angles in the “Drive to” box and check that the goniometer drives to the entered 2θ and θ angles accurately. If it is off significantly, repeat these steps until your angles stay within ± 0.003 deg (3/10 of a micrometer division) on both θ and 2θ . ***If you cannot achieve this, stop what you are doing and contact the lab manager.***
4. To check shutter function, click on Open Shutter (the big blue arrow) and check that the shutter opens (i.e., X-Ray on light comes on). Click on Close Shutter (smaller blue arrow) to close the shutter (X-Ray light goes off).
5. When done, close the Window (the red X or Function – Close on the Menu) and proceed with data collection.

¹ The software may occasionally give a communication error during this process. If it does, just close the error dialog. When it is done moving, communication will be reestablished and you can proceed with step #2.

² The DataScan 4 calibration routine will sometimes drive θ to the wrong position. If this occurs, just be careful to accurately read (and enter) the correct angular position.

Data Collection Overview



The DataScan program window above has several parts, from top to bottom:

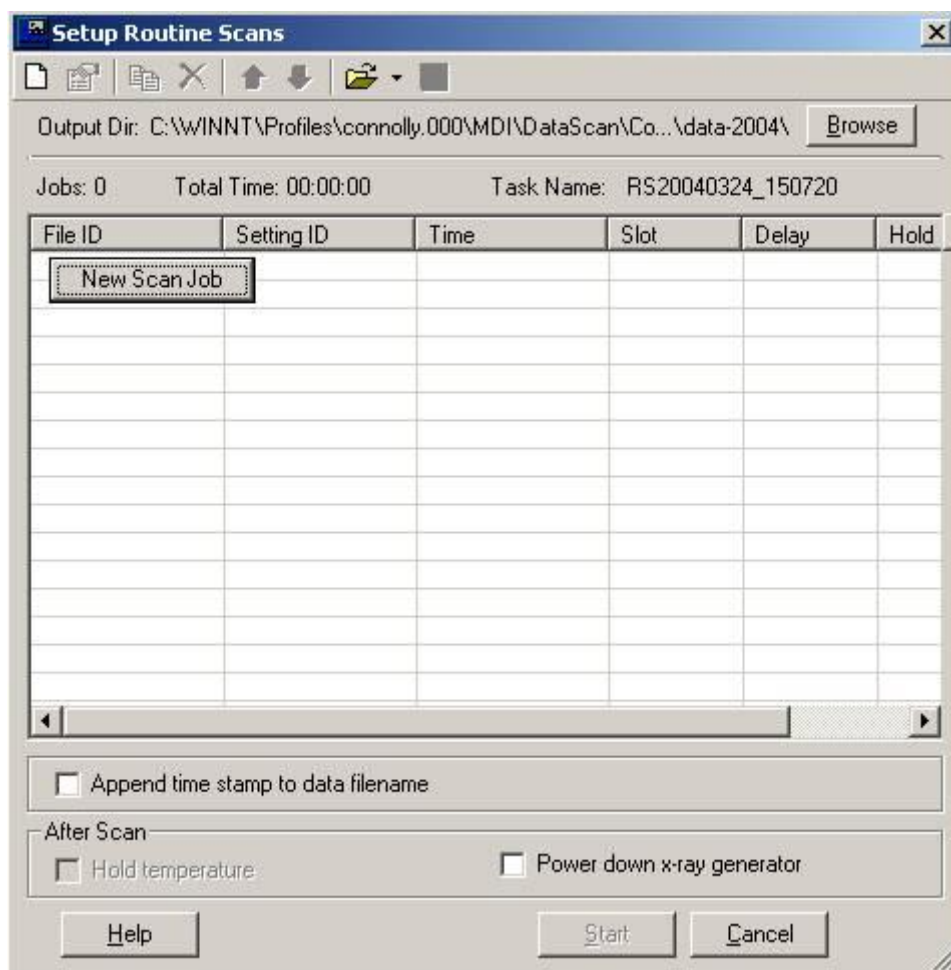
- The title line (colored, top of windows) indicates the Program Name, Current user name, and registered owner of the software.
- The Menu line below includes **File**- and Printing-related items, **Scan** (setup and start scans), **View** (items related to how data are displayed as collected), **Control** (items related to direct control and configuration – primarily for lab administrator use), and **Help** (to access Windows Help. Note that may not be up to date for the version of the program that we have).
- The icons below provide, left to right, direct access to setup a routine scan, quick stop for a scan in progress, and direct access to the controller window.
- The display window provides a graphical display of the current data collection activity
- The message window provides a real-time log of DataScan to Diffractometer communications and activities (including errors).
- The bottom line provides real-time information about active data collection including Detector (2θ) position, Incident beam (θ) position, the mouse cursor positions X (=2 θ), Y (=intensity), and d (d-spacing), the ScanID (filename) for the job, and the Range parameters for the scan. Slot is for a sample changer that we do not have.

DataScan includes 3 modes of data collection, **Quick** scan, **Routine** scan and **Area** scan.

- The **Quick** scan can be used to quickly enter run parameters in a “Scan Range” window and collect data from a specimen loaded into the diffractometer. **At the conclusion of Quick scan data collection, the user must save the file (in .MDI format) by the user with a File-Save command or it will be lost when you quit the program.** It is only useful for rapid reconnaissance to see if there is a peak located in a particular limited range prior to running a routine scan.
- The **Routine** scan creates a setup table and automatically saves data as it is collected. This is the mode that should be used by lab users. It provides more control (allowing use of saved setup routines for data collection) and lets the user enter more information about their sample into the data file. In general, **Routine scan** should always be used for all data collection.
- The **Scan Area** appears after a Quick or Routine scan and is allows the scan of a smaller area (to obtain more detail or resolution).

Routine Scan Setup

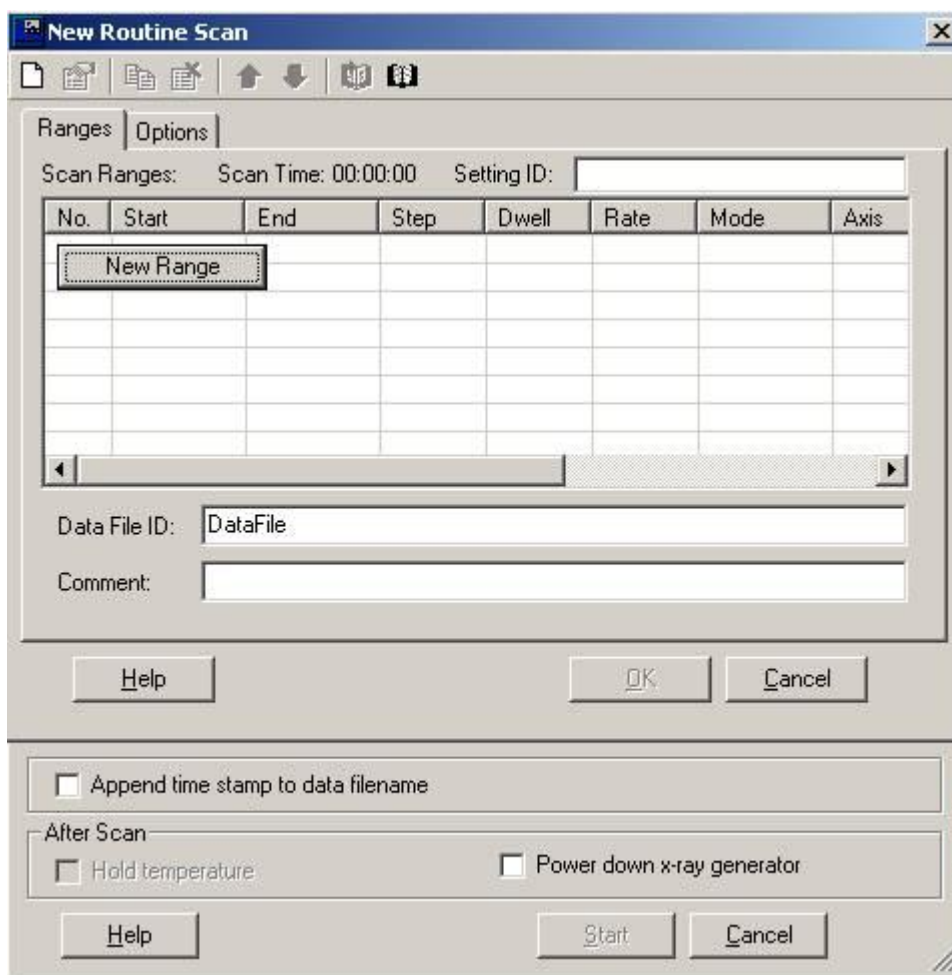
From the DataScan menu, choose “Scan” – “Routine Scan” which will bring up the scan setup window show below.



Select your Output Directory: By default, DataScan saves your data to a subfolder in Documents and Settings on the computer you are sitting at. This is a very bad place to save data since you cannot access it from either of the other systems in the lab. *The first time you use the system you should change this Directory to your “L:” drive folder.* Click on the **Browse** button, and enter “L:\” in the location window (or browse through “My Computer” until you find your L: drive) and then pick a folder in which to save your data. Entering a new folder name in the “path” (or clicking the New Folder button and entering the name) will cause the new folder to be created. Using the “L:” drive allows you to access your data from any system on the network, either data analysis system.

Important Note for Lab Users: Scan setups (i.e., how your scans are done) are customized for each user, so each user needs to recreate and save their own settings for the scans that they do routinely. What follows are the basics.

The first time you setup a routine scan, you may click on the “New Scan Job” button to define a scan. This only appears on a blank setup window. Subsequently you may use the “Page” icon in the upper left, or delete the existing line to get the “New Scan Job” button to appear:



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As with the previous window, there is a “New Range” button on a blank page, or you can click on the “Page” icon to define a new scan range (or use your right mouse button to define a “New” range): The window below will appear:



The screenshot shows a dialog box titled "New Scan Range". It contains the following fields and controls:

- Start: 18
- End: 30
- Step: 0.02
- Mode: Continuous (dropdown)
- Rate (d/m): 2.0 (dropdown)
- Axis: Both axis (dropdown)
- Psi: 0.0
- Scan Time: 00:06:00
- Buttons: Help, OK, Cancel

On the scan range dialog, the following are defined:

- Starting angle (2θ)
- Ending Angle (2θ)
- Step Size (minimum 0.01° ; 0.02° generally best for continuous scans, 0.05° for step scans)
- Data Collection Mode (Continuous, or Step Scan)
- Rate (degrees/minute for continuous mode) or Dwell (time in seconds per step in Step mode), and Axis (both axis for coupled scan).

The “Scan Time” will be calculated when sufficient parameters are entered. Click OK in window to return to the New Routine Scan window (below) with collection parameters defined.

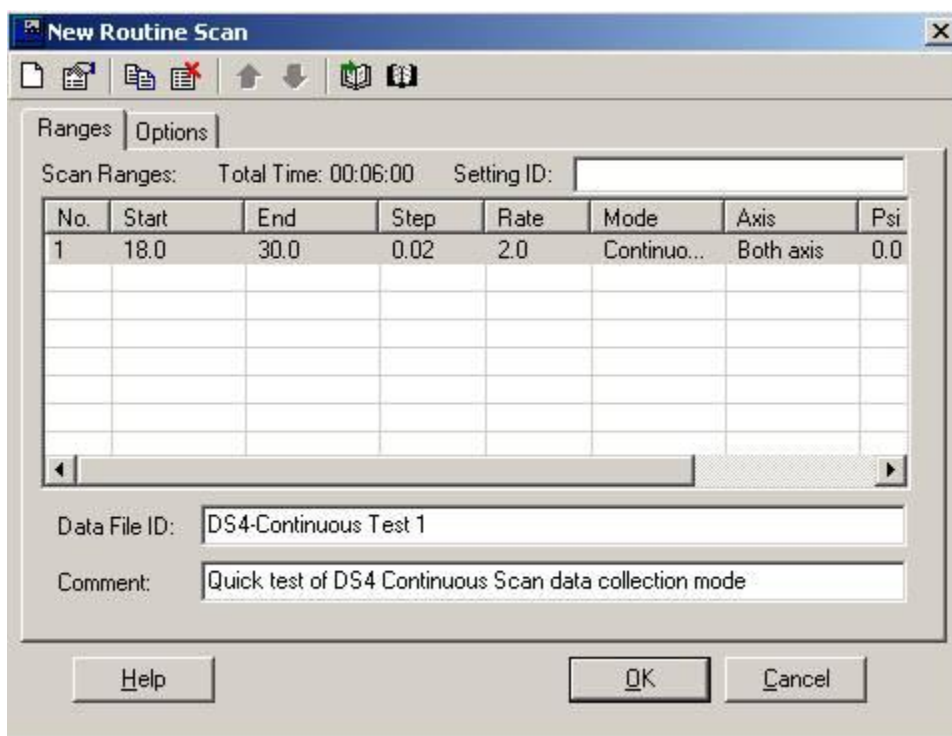
A Timing Note: For step scans, you must enter the dwell time per stop (rather than degrees per minute) as a timing parameter. For reference when entering values, a step scan with a step size of 0.02 and a dwell time of 1.2 sec/step is equivalent to a 1 deg/min continuous scan.

When to use Continuous Scan vs. Step Scan: A continuous scan collects data continuously by sampling the detector at intervals throughout the scan and as such does a kind of electronic smoothing of your data as it is collected. A step scan moves the detector in steps and counts and records data as it collected.

Rule #1: For scans slower than $\frac{1}{2}$ deg/min (with a 0.02 step size), always use the Step Scan mode. This will prevent calculation overload that can occur with DataScan when doing very slow step scans.

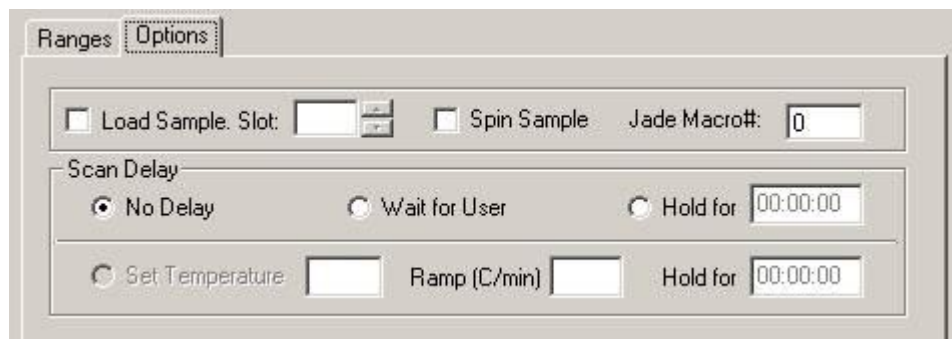
Rule #2: For scans faster than 1 deg/min (with a 0.02 step size), always use the Continuous Scan mode. This will prevent data errors associated with the mechanical “settle down” time that occurs with stepped movements of the goniometer.

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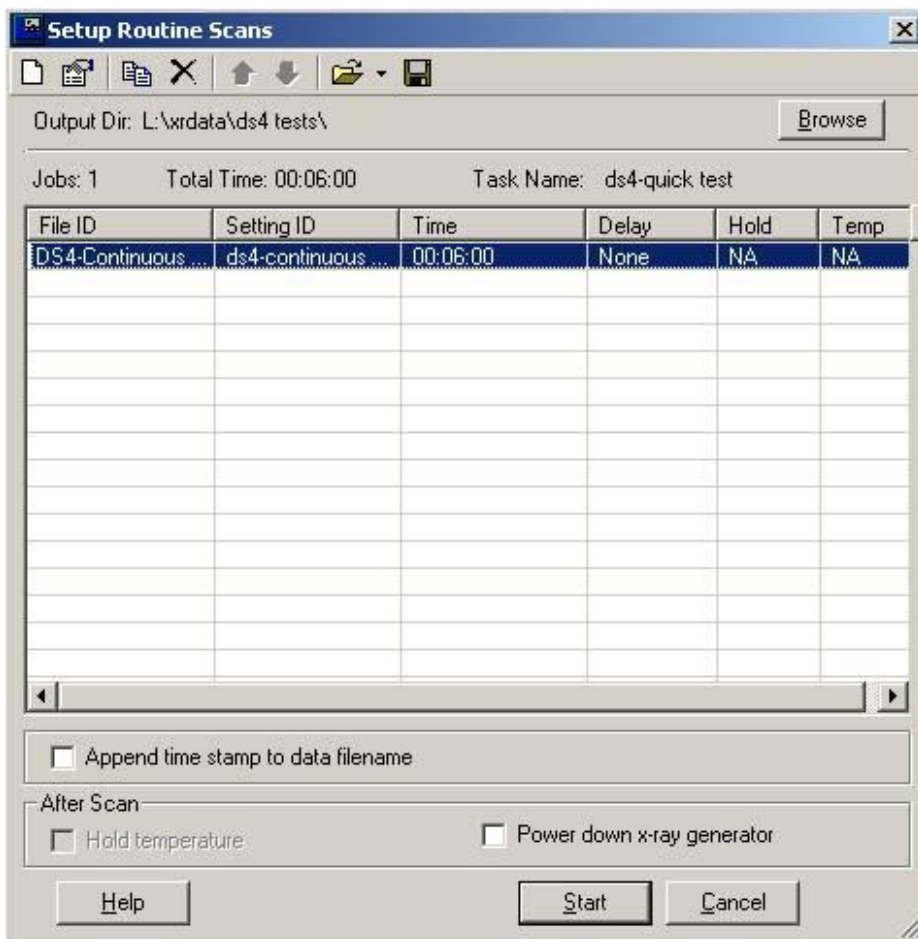


Saving Scan Parameters: The scan parameters may be saved to a separate file in your personal “Scan Book” by clicking on the save parameters icon (the open book icon with the arrow), so that the parameters may be reused for other scans. You must name your “Setting ID” to save it – use something that describes the scan parameters to make it easy to retrieve (i.e., 10-70deg, continuous, 1deg/min). The open book (without the arrow) may be used to retrieve previously saved setups from your “Scan Book”.

In the “New Routine Scan” Windows enter a Data File ID (this is the filename to be used and may be a long filename) and a Comment (this is used for your description of your specimen). The “Options” window may be used enter a “wait for user” delay should you choose to setup multiple specimen with you acting as the sample changer.



Click OK on this window to bring up the completed setup window (below):

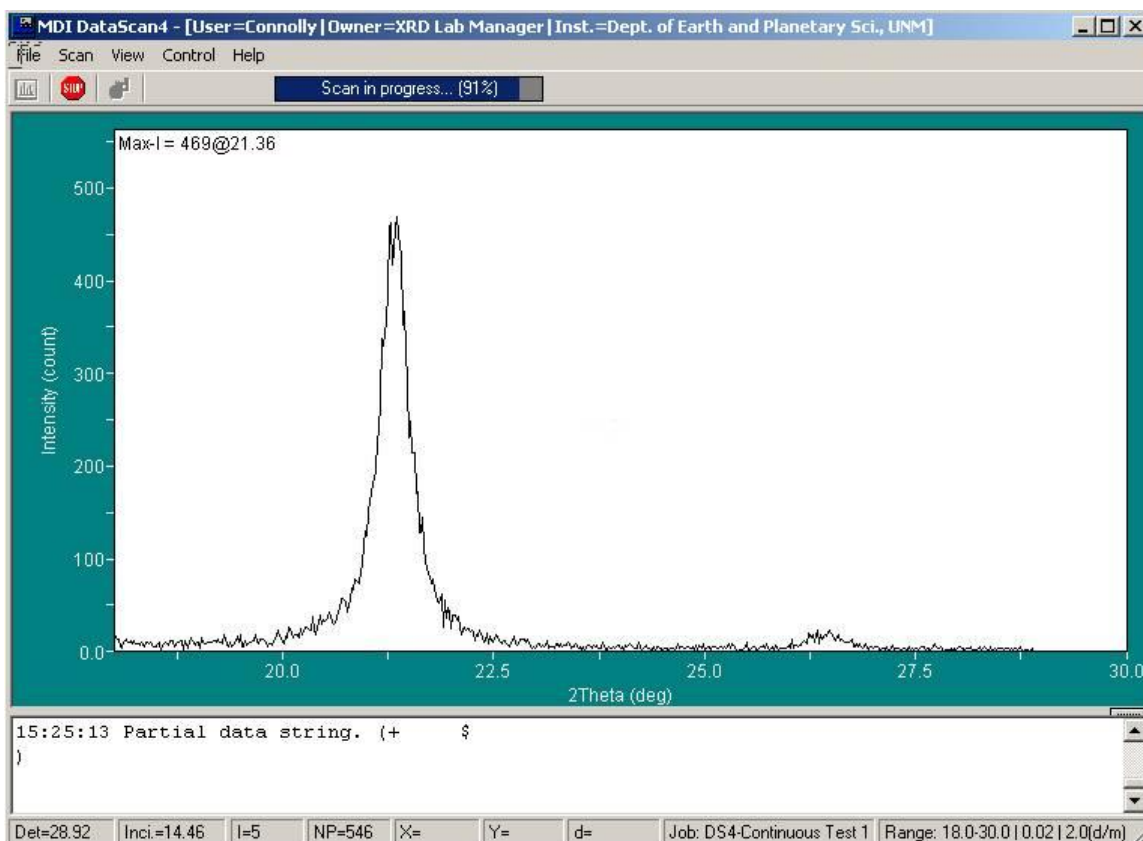


IMPORTANT NOTE:

Only one scan range (i.e., one line) should be on this screen before starting your analysis. Multiple lines are used only for systems with sample changers – something that we do not have. If you have multiple lines, remove those that are not related to the specific analysis you want to do now.

Click on the **Start** button to begin data collection. The program pops up a window confirming the current goniometer position. It is a good idea to quickly check before data collection starts that this position is correct. When this window is cleared, data collection starts and the DataScan data collection window opens. In the window below, data collection is about 90% complete.

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When data collection is done, the display written and the diffractometer is parked at the $10^\circ 2\theta$ “exchange” position*, exit the program (*File* menu – *Exit*). If you are finished using the system, log off the network to close your network connection.

* Diffractometer will only park if you have set this on your default settings; see page 3 for how to set your defaults properly.

DataScan 4 Characteristics

Some DataScan4's capabilities and characteristics (quirks?) are:

- Every user has their own settings for custom scan setups and data storage locations. These are not shared between users (as in older versions of the program).
- The program saves most of your setup parameters (goniometer parking and limits, and data location, setup from last use, etc.) between uses. This makes network storage of data easy, but also makes it fairly easy for you to accidentally overwrite the data you collected the last time you ran the program.
- "Quick Scan" can be used to quickly scan a small angular range of interest (i.e., if you are looking to see if you have peaks in a small 2θ range) before doing a full routine scan. This can be useful to see if it is necessary to run a full "Routine" scan.
- There are now some nice capabilities in refining scans and zooming in on areas of interest. The DataScan display Window is like a mini version of a Jade zoom window and you can zoom in on an area of interest by dragging a box around it with the mouse and then "Scan this range . ." from the Scan menu to get better resolution of areas of interest. This is done as a "Quick Scan" and must be saved as a separate file (if you want to keep it).
- You may print your data directly from DataScan if desired. Make sure the printer is set up to print in "landscape" mode. Printouts are basic, but give you quick printed output of your data without requiring Jade.
- The view can be modified to show dots (at each of your "step" points) or a rectangular grid overlay.
- The software now also includes a pattern viewer (File – Pattern Viewer) that opens in a new Window and lets you view your collected data patterns (not just the one you are currently working on). This pattern viewer appears to have some very useful data viewing capabilities. Feel free to explore what you can do with it.

Enter User and Scan Information in XRD Log Book

When data collection for the day is completed, fill out the logbook recording what was done. All logbook entries (at a minimum) should include

- Today's *date*,
- Your *full name* (printed legibly – not your nickname, username or partial name),
- The *number of samples run* (only include *successfully* completed runs in your total, plus run type, i.e., step scan, continuous scan, etc. These are runs for which you have a data file saved),
- Notes about condition (***OK*** if all is working appropriately, an expanded note if something needs attention. If you have any failed scans which you had to redo, please note this here. Each logbook page has a block at the bottom of the page with room for detailed operational comments.),
- Your *Supervisor* and/or Banner index number for use in billing. All users should have made billing arrangements with the lab manager. If you do work on multiple accounts, each needs to be identified on a separate line. You ***must*** print this information ***legibly and accurately*** since it is used in billing for lab use.

Note: Not making logbook entries or making false entries are cause for immediate termination of lab privileges.

Data Analysis with Jade Software

As of this writing Jade 9.0 is installed on the two data analysis computers with the ICDD 2007 database. The ICDD 2008 database will be installed in April, 2009.

This document is concerned with data collection, not data analysis, and users are referred to the Introduction to Powder Diffraction course page for an introduction to Jade. (See <http://epswww.unm.edu/xrd/xrd-course-info.htm>, Week 11.)

Jade no longer comes with a paper manual. We have put shortcuts to the Jade 9 “Help” files in both old Windows format (jade9.hlp) and new Windows compiled HTML format (jade9.chm). ***These two files are identical in content, and are the Jade 9 Users Manual.*** If individuals would like a copy of either or both files for use on their own computers, see the lab manager about how this may be accomplished.

A large suite of advanced tools are available in Jade including some for profile refinement, several types of advanced search-match, peak broadening calculations, crystal structure visualizations and many other things which your lab manager has not yet mastered. We also have the MDI Shadow program which will do various types of profile fitting, line-shape analysis, and crystallite size analysis. Users with specialized analytical needs are encouraged to read the Jade 9 Manual (online) and Shadow (paper) manuals which are found in the laboratory. If you need help getting the program to do something, please feel free to contact the lab manager. I am far from an expert with all aspects of Jade, but will help as much as I can.

Jade Data Processing and File Formats

MDI's Jade creates a large number of files when you are actually processing data. Some of these are binary control files that are of use only to the program itself, but many of them are actually useful data files in standard ASCII text format (readable with any text editor like Notepad or Wordpad). The purpose of this section is to list as many of these file types as possible and describe when they are created and how they can be recognized.

To recognize the various file types, *it is important that your Windows Explorer file lists show hidden files and file extensions*. To do this, open any explorer window (i.e., My Computer) and from the menu select Tools – Folder Options and then the View tab. In the view tab make sure “Show Hidden Files and Folders” is checked, and the “Hide Extensions for known file types” is not checked. This will let you see the file extensions for all MDI Jade data files so you can recognize what they are.

The Jade data files are listed below. In some cases, these files are created by doing some action (i.e., identify peaks, pick identified phases from a card list of search match, etc.) and saving the results in the graphical results box that is displayed on the screen. Some are created by a File – Save command.

- filename.MDI: Data file in MDI standard format. See Introduction to Jade and Datascan Software (<http://epswww.unm.edu/xrd/xrdclass/08-DataScan-Jade-Intro.pdf>) page 5 for details of this file format.
- filename.TXT: MDI data file in two column (position, intensity) ASCII format with the sample description as the first (and only) header line. This is the format most easily imported by most other XRD programs. Created by “File – Save As . . .” and choosing to save data as TXT.
- filename.PID: Text-file version detailed Peak ID Results includes 2-theta, d-spacing, BG, Height and % relative to other peaks, approximate peak Area and %, calculated FWHM. Created by identifying peaks in a data file with Jade.
- filename.PKS: Peak search configuration file (binary control file) created when a peak identification is executed in Jade.
- filename.AID: Simple ASCII text of d-spacings of identified peaks and relative intensity in the “AIDS83” format.
- filename.IDE: Text-file version of the extended peak ID created after search-match has been run and matched phases identified. Shows peak ID results and quality of match of peaks in identified phases.
- filename.PDF: Powder Diffraction File overlay list – ASCII text file lists overlaid ICDD card on the current
- filename.SAV: is a binary file that saves everything in the current workspace for later restoration. Done from the save-as menu. Multiple workspaces may be saved temporarily for the same data file by using different filenames.
- filename.DSP: is an ASCII text file of d-spacings and relative intensities in the MDI format

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- filename.XML: a variety of different data types can be saved in XML (Extensible Markup Language – a variety of HTML, the language of web pages). The XML format is one that is intended to be more prominently used in future releases of Jade.
- PDF#xx-xxx.xxxx.txt: ICDD PDF file card data in ASCII format saved from a view-card-data window with a “Save” command. Card data (but not graphical patterns) are saved.

Note that the ICDD PDF4 naming scheme is now used in Jade. In this scheme the second and third groups of digits (show as x) represent the series and card number in the old PDF2 format and the first two digits represent the database source. The sources represented in MDI formats are as follows:

The ICDD Data Formats:

- 00: Experimental PDF data (i.e. old JCPDS cards, grant-in-aid data, some calculated).
- 01: Calculated PDF data from ICSD (i.e. FIZ inorganic crystal structure database).
- 02: Calculated PDF data from Cambridge organic structure database (RDB only).
- 03: Calculated PDF data from NIST metallic and intermetallic structure database.
- 04: Reserved by ICDD for future products (e.g. MPDS, Pauling files).

The MDI/Jade Data Formats:

- 90: Customer PDF-like database (PDF1 or PDF2 created by the user).
- 91: Jade-calculated d-I list data from NIST crystal data (crystal.dat).
- 96: Jade-calculated d-I list data from PCD (copyright (c) MPDS/ASM).
- 97: Jade-calculated d-I list data from ICSD (i.e. FIZ inorganic crystal structure database).
- 98: Jade-calculated d-I list data from JCSD (structure databases created by the user).
- 99: Jade-userfile d-I list data (created by the user).

Printer Tips and Tricks

The HP Color Laserjet 2600 printer to the right of the EPSXRD2 computer is the printer for all of the computers in the lab. This printer can produce full color output on plain paper. Here are a few things to be aware of:

- ❖ **Each user must connect to the printer and set it as the default printer. Here are the steps:**
 - From Start Menu, select Printers and Faxes, then “Add a Printer”
 - When the printer wizard starts click “Next” then choose to “Connect to a Network Printer”, then Next.
 - Click the second box to Connect to this printer and enter this address in the box: <\\eps2\xrdprint>.
 - Click Next and the printer and its drivers will be loaded. If this is the only connected printer it will automatically be made the Default printer. If it is not, check the box to make it the Default printer.
- ❖ **Jade will only print to the printer set as the “default”.** <\\EPS2\xrdprint> should be set as the default printer *before* starting Jade.

Scintag XRD Powder Diffractometer Procedures

- ❖ **Paper may be found in the lab in the “in box” on top of the cabinet to the right of the printer or in the cabinet on the opposite side of the room.** Regular 20lb bond works fine in the printer. Only paper certified for Laser Printers should be used. **Warning:** Some Inkjet papers can melt in the Laser printer making it useless. Users are responsible for loading and (and unloading!) of any other paper (i.e., special photo paper or transparencies).
- ❖ The “Toner Low” message will begin to appear when about 20% of the toner is remaining. Typically this is at least 800 pages. We do not replace toner cartridges until they are *out*, but do keep spares on hand. If toner is out contact the Lab Manager, Jim Connolly, who will change the appropriate cartridge.