

Introduction to DataScan and Jade on the Scintag PADV System

(prepared by James R. Connolly, for EPS400-001, Introduction to X-Ray Powder Diffraction, Spring 2009)

Introduction

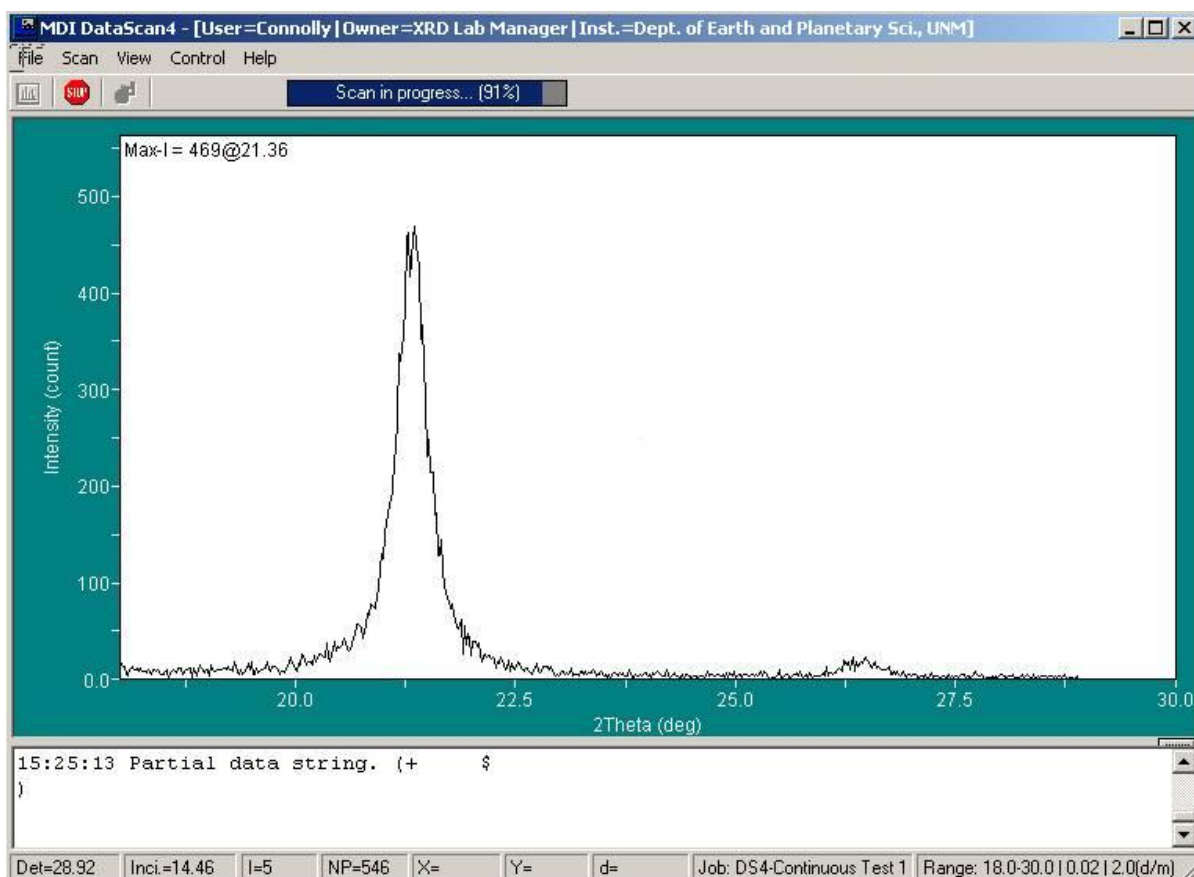
In the last 15 years, the development of sophisticated and easy to use software for data collection and analysis has made sophisticated analysis of X-ray powder diffraction data accessible to anyone with the ability to operate a personal computer. In many cases the software is so easy to use that analysts run the risk of using it as a “black box” to obtain answers not warranted by the data.

The material in this section is concerned specifically with the software installed for use with the Scintag PAD V system in the X-ray powder diffraction laboratory in the Department of Earth and Planetary Sciences at the University of New Mexico. As of this writing, the software is DataScan 4 (for data collection) and Jade 9.0 Plus (for data analysis) from Materials Data, Incorporated (MDI). MDI software is written by experts in X-ray diffraction and is relatively user friendly. Jade, in particular, is extremely powerful and well worth the time and energy spent learning to use and master its many and varied capabilities.

Most of the material in these sections is abstracted from MDI’s digital documentation for Jade, and “in-house” operating XRD Lab documents for DataScan.

Data collection with MDI’s DataScan 4

Below is the DataScan 4 interface Window referred to in the following sections:



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DataScan 4 is a 32-bit Windows program that allows each user to customize their own “default” settings for how the program operates. The program communicates with the Scintag PAD V goniometer by means of a standard (if somewhat archaic) RS-232 serial interface. This is a two way single bit-stream interface and if it is interrupted it is possible to lose communication with the diffractometer causing data collection to fail. Recent configuration changes in the system have made this a rare (but still possible) occurrence.

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 but also used by users to verify that angular positions are set correctly), and *Help* (to access Windows Help, such as it is).
- 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** (turned on and off in the View menu) provides a real-time log of DataScan to Diffractometer communications and activities (including errors).
- The **status line** (at the bottom) provides real-time information about active data collection including Detector (2θ) position, Incident beam (θ) position, the mouse cursor positions X ($=2\theta$), 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.

Data collection and operation of the diffractometer is through a series of dialogs called up through the Menu at the top of the main program window as shown on the previous page. Details of operation may be found in the XRD Lab Procedures Manual (or “XRD Cookbook”) in the laboratory. An Acrobat PDF file of this manual is available on the lab “Resources” page at <http://epswww.unm.edu/xrd/resources.htm>.

The DataScan Interface and Data Collection Setup

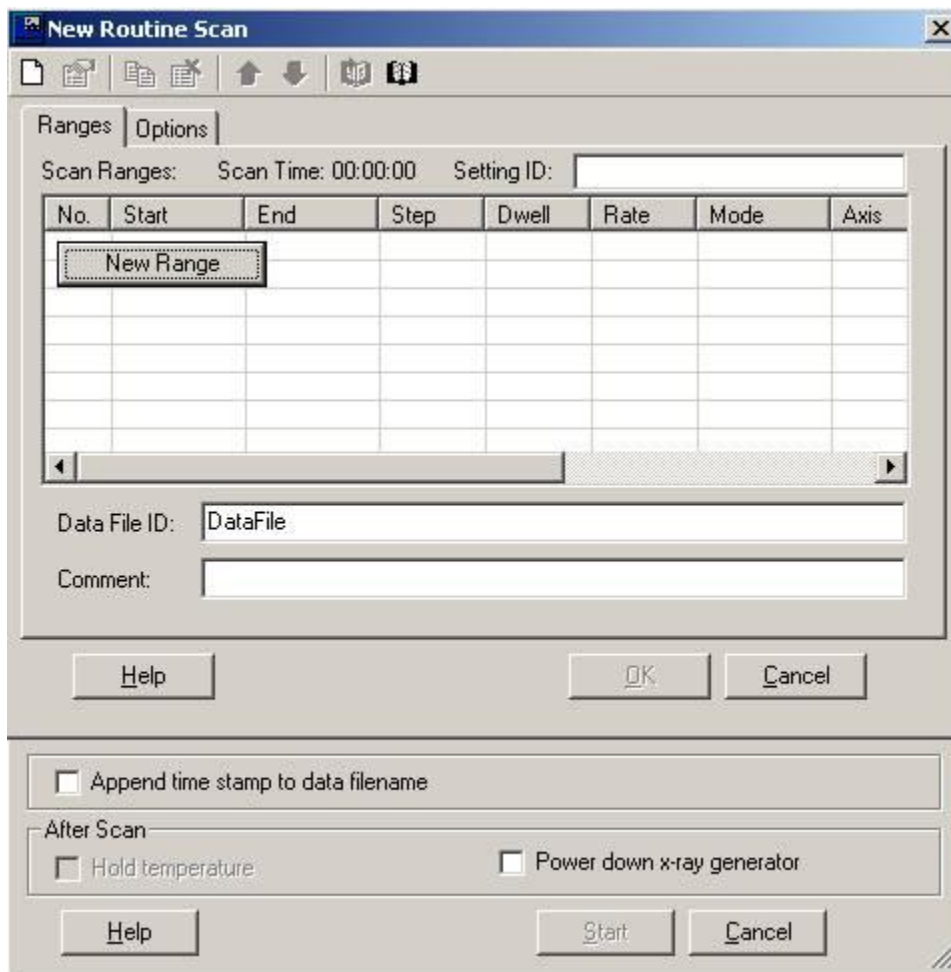
DataScan4 includes 3 modes of data collection, *Quick* scan, *Routine* scan and *Area* scan.

- 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) with a File-Save command or it will be lost when you quit the program.*** It is only useful for rapid reconnaissance (i.e., Is a particular peak located in a restricted angular range in a specimen?) prior to running a routine scan.
- The *Routine* scan uses a setup table to configure all scan parameters and automatically saves data as it is collected. 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 be used for all data collection.***

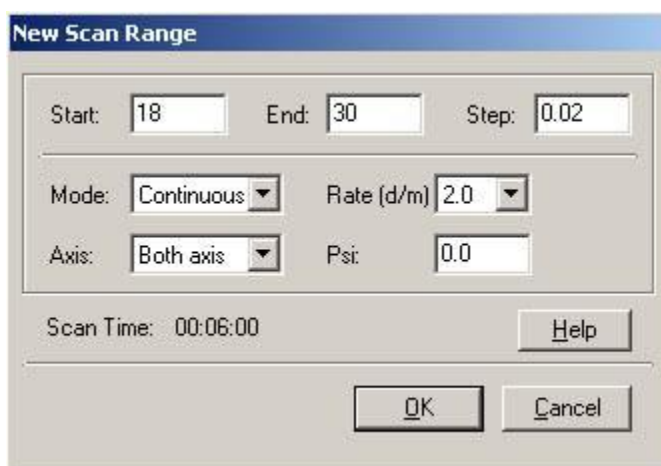
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Either “New Range” button (if no range is shown), or the last scan range used will be displayed on the Scan Range first line. Click on the “New Range” tab, the Page (upper left) icon, or double click the existing range to bring up the New Scan Range window below:



On the scan range dialog, the following are defined:

- Starting angle (2θ)

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- Ending Angle (2θ)
- Step Size (minimum 0.01° ; 0.02° is generally best for continuous scans; 0.02° to 0.05° are usable 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).

A Timing Note: For step scans, the dwell time per step (rather than degrees per minute) is entered 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 continuous scan of $1^\circ/\text{min}$.

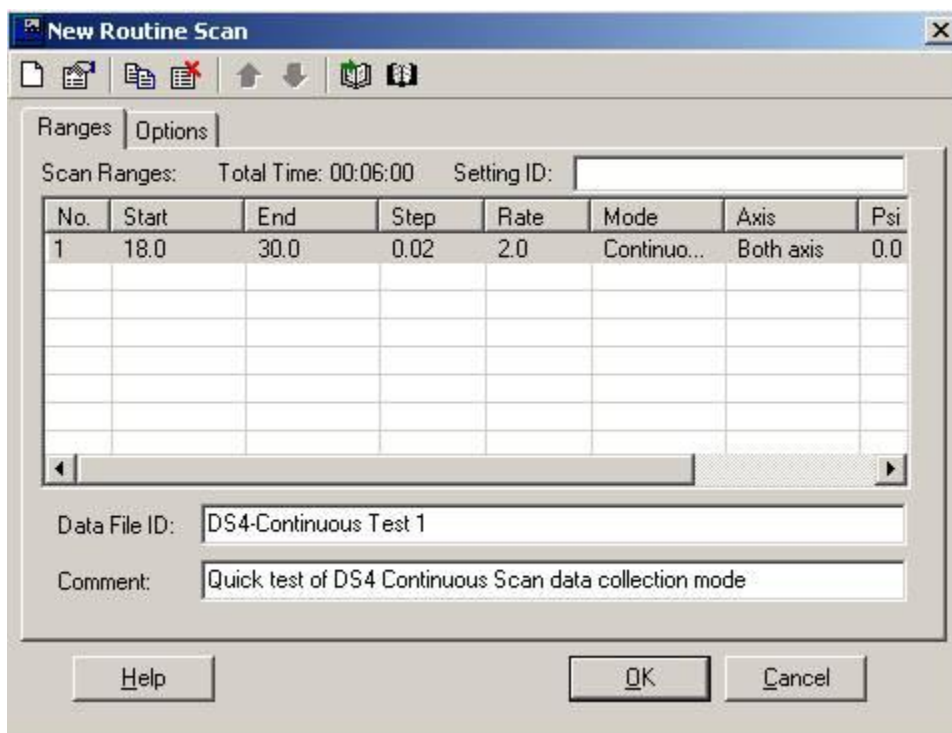
Continuous or Step Scan?: “Continuous Scan” collects data continuously by moving the detector at a specified steady rate and electronically sampling detector counts at intervals throughout the scan; as such it does a kind of electronic smoothing of your data as it is collected. A step scan moves the detector in steps and counts and records data as it collected. Step scans tend to show more variation between individual data points and record the random aspect of “raw” X-ray counts; Continuous scans tend to “average out” some of this variation by the process used in the continuous count. Continuous scans also are more computationally intensive and very slow ones can tend to overload the software (resulting in output that can lag slightly behind the movement of the goniometer). Here are the general rules for which type to use:

- **Rule #1:** For scans slower than $\frac{1}{2}$ deg/min (with a 0.02 step size), use of the Step Scan mode is recommended. 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.

The “Scan Time” is calculated when sufficient parameters are entered. Click OK in the “New Scan Range” window to return to the New Routine Scan window (next page) with collection parameters defined.

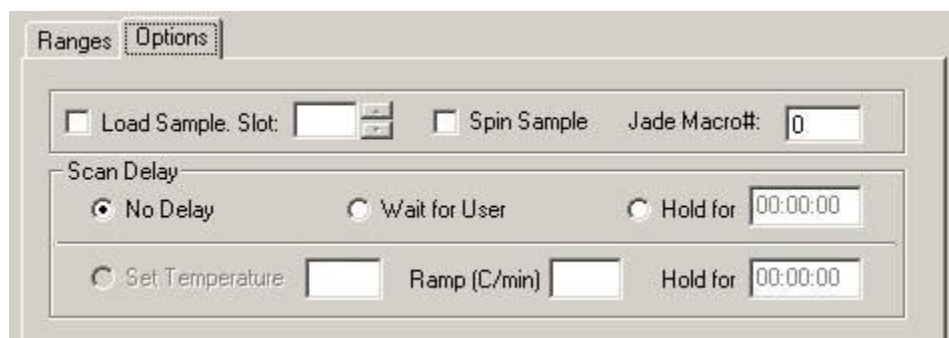
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Saving Scan Parameters: The scan parameters may be saved to each individual user's personal "Scan Book" by clicking on the save parameters icon (the open book icon with the green arrow), so that the parameters may be reused for other scans. The Scan Book entry uses the "Setting ID" as the saved name so something must be entered in that box to save it. It is suggested that a Setting ID be used that describes the scan parameters to make it easy to recognize when retrieving (i.e., 10-70deg, continuous, 1deg/min). The open book (without the arrow) may be used to retrieve previously saved setups from the "Scan Book".

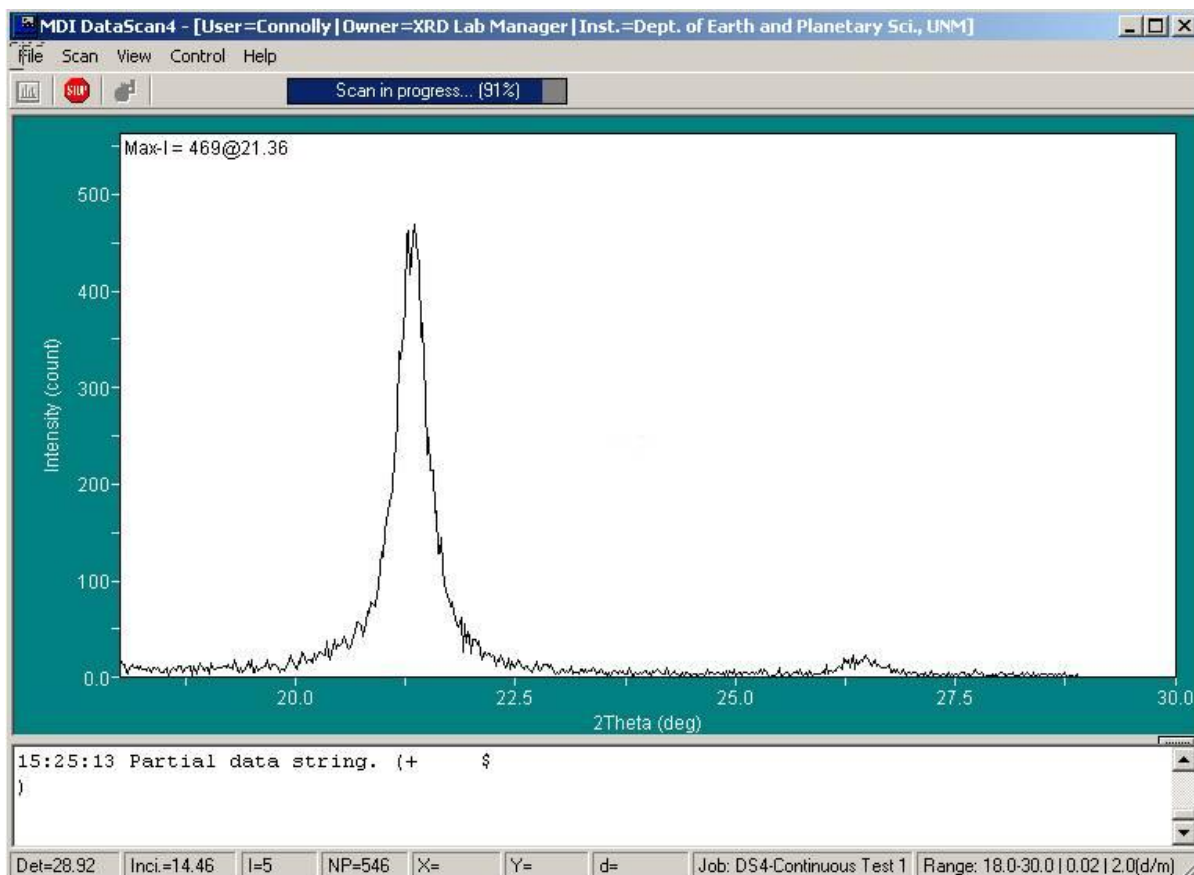
In the "New Routine Scan" Window enter a Data File ID (this is the filename) and a Comment (this is used for your description of your specimen and is included in your data file so should always be entered). The "Options" window (below) is generally not used, but may be used enter a "wait for user" delay should you choose to setup multiple specimens with you acting as the sample changer (since our system does not have one).



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

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While collecting data, the status line will show the changing detector position and counts at each point. When data collection is completed, the full pattern will be displayed, all data is written to the designated data file and the diffractometer is parked at the $10^\circ 2\theta$ “exchange” position¹. The program stays open until closed with *File* menu – *Exit*.

¹ Parking the goniometer after a run is not a default behavior of DataScan4, and must be set up by each user when they first use the program. The Lab Manual contains several items that must be set up upon initial use including the default location for your data, the parking behavior and position after data is collected and the optimal angular limits for scans. *Each user must do this or run the risk of strange behavior by the PADV system.*

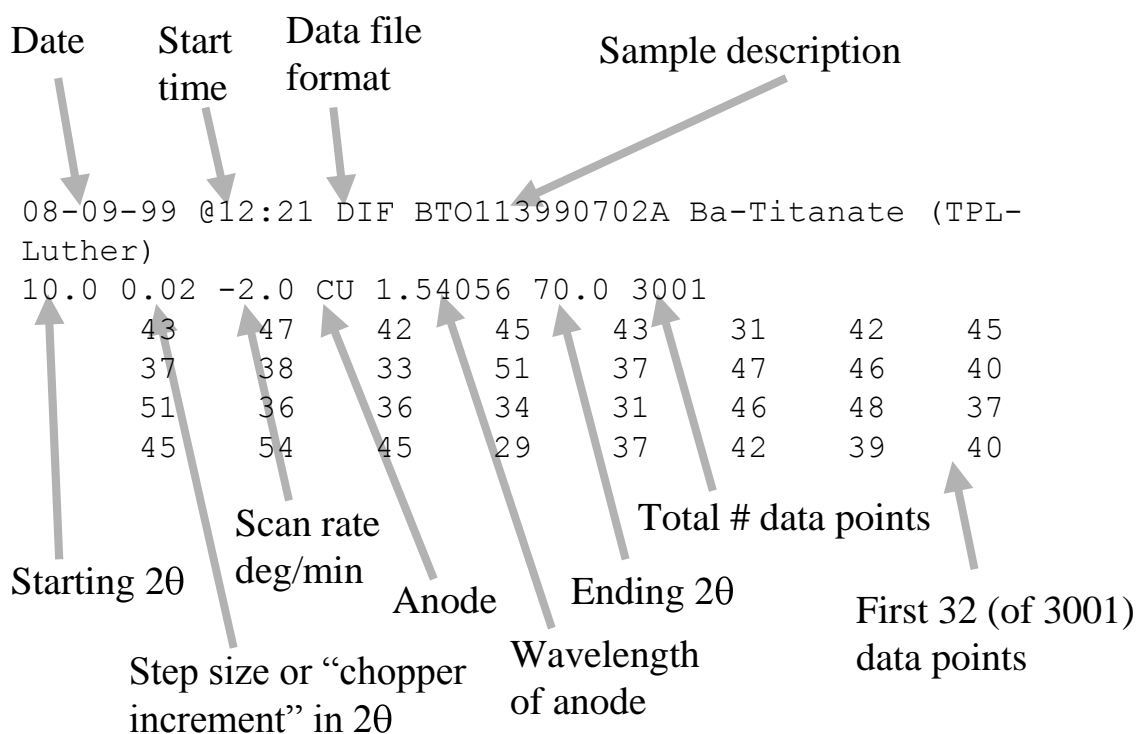
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MDI (DataScan and Jade) Data File Format

DataScan writes data files in the “MDI” file format, and files all have a “.mdi” extension. The file format is basically identical to a standard DIF (data interchange format) file which is a standard 7-bit ASCII text file.

The figure below identifies the parts of the header in the standard MDI data file. A few notes about the format as written by DataScan follow the figure.



- The Scan ID is a free-form text field, starts as the 21st character of line 1 and is up to 80 characters in length
- The date/time stamp is written at the start of data collection. Jade does not use this date in listing and sorting data files. It uses date and time data collection ended.
- DIF indicates Data Interchange File format. It may be used by other programs reading the data file, but is ignored by Jade.
- Step size in degrees 2θ (“Chopper Increment” is sometimes used when referring to steps used in continuous-mode data collection)
- Scan rate in deg/min. Minus sign indicates continuous scan (not step)
- Wavelength of the anode is whatever is defined in DataScan setup. Jade ignores this number; it reads the anode type and uses the wavelength configured for that anode in the Jade (not DataScan) program setup.
- # of data points is $([\text{end angle} - \text{start angle}]/\text{step size})+1$. (3001 points in the example above)

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- Data block is all data points; data are in counts stored as a long integer
- DataScan may be configured to record data in 8-column rows (the default, as shown above with .MDI extension) or as a single column (configured in DataScan's setup dialog with .ASC extension).

Data Analysis with MDI's Jade 9

MDI's Jade is a very powerful software system designed for XRD powder pattern processing. Some of the operations that can be completed with Jade include:

- Corrections for experimental errors, background and $K\alpha_2$ peaks
- Automated identification of peaks – Integrated and maximum intensity, 2θ
- Access to ICDD's Powder Diffraction File (PDF2 or PDF4) database and overlay of experimental patterns with those from the database
- Access to the Inorganic Crystal Structure Database (ICSD) and structural data contained in that database
- Access to new ICDD data containing structural data
- A variety of printed and digitized data reports in graphical and text formats
- Automated Search/Match of patterns with the ICDD database and manual search match with data retrieval by mineral name, chemistry, or unit cell criteria
- Automated Search/Match using whole pattern fitting algorithms (New)
- Pattern simulation from crystal data
- Quantitative analysis by reference intensity ratio (RIR) methods using internal or external standard methods (Not included in our version of the software)
- Pattern indexing and unit cell refinements
- Crystallite size and strain analysis, and residual stress analysis.
- 3D crystal modeling based on structural parameters from your experimental data

In addition to these main features, there are myriad additional capabilities that make Jade probably the single most powerful tool available for processing and understanding your diffraction data.

The program comes with both Compiled HTML (CHM) and standard Windows help files that are essentially a digital version of the printed manual and provide extremely detailed information about all aspects of the program. The printed manual for the newest version is little more than an installation guide and introductory tutorial.

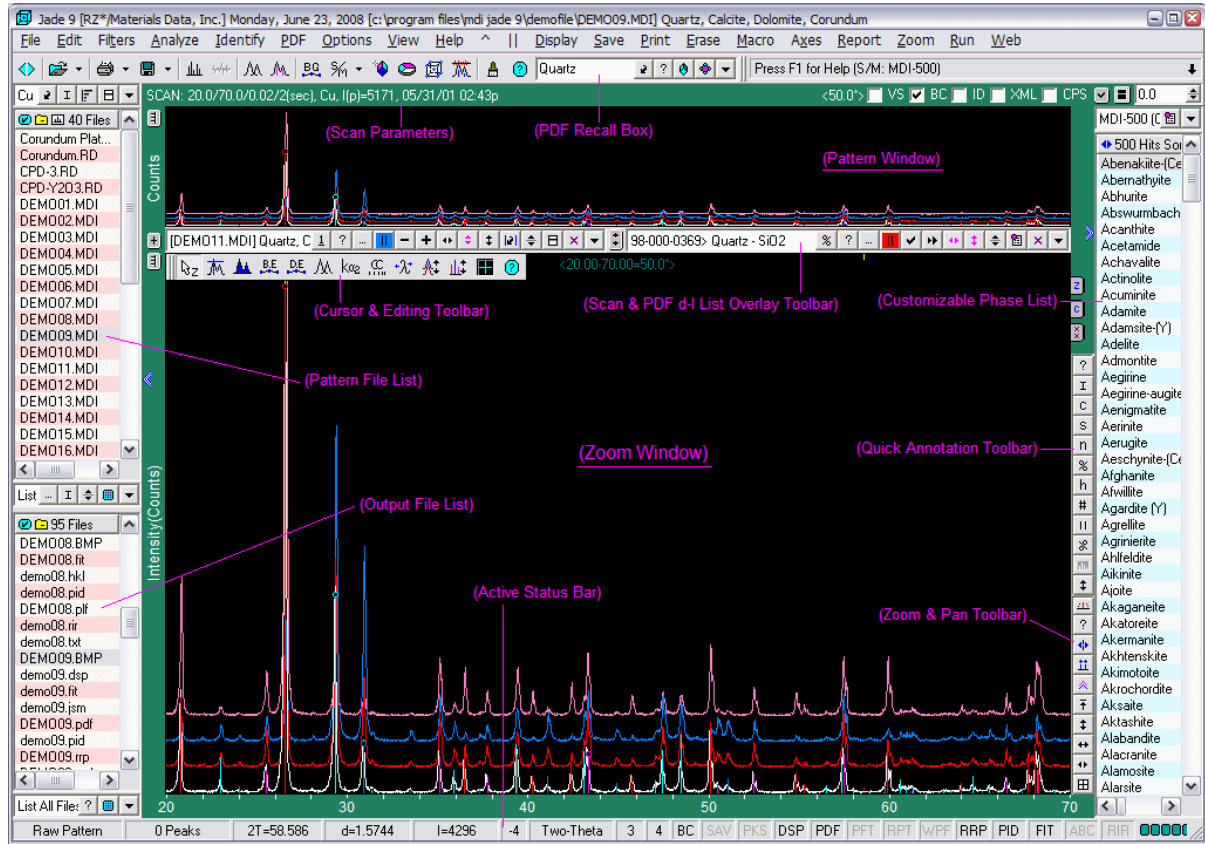
What follows is a very basic outline of Jade 9 capabilities, largely extracted from MDI's "CHM" documentation. It will barely scratch the surface, but is hopefully enough to help you get started. Use the Jade online help to fill in the blanks.

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The Jade Interface

Below is an example of Jade's main interface window with most main components labeled:



Left and Right Mouse Buttons: Jade makes extensive use of the right mouse button. In general, the left mouse button will execute a command, and the right button will call up some sort of configuration settings for that command or a context-sensitive menu (a la Microsoft Windows). For instance, a left-click on the S/M button will execute Search/Match on the current pattern with the default parameters; a right-click will bring up the Search/Match configuration window.

The Pattern Window (above the zoom window) always shows a “scaleless” version of the full pattern with a color highlight (if selected) indicating what is shown in the zoom window. The **Zoom Window** is main window in which most pattern processing takes place. It is the **zoom window** because it may be “zoomed” to examine a particular peak or group of peaks in detail that are selected in the pattern window. Many pattern processing operations may be carried out on the whole pattern or restricted to the “zoomed” portion of the window. This includes Search/Match, printing plots or data, etc. The right-mouse button in the zoom window gives a touch-screen-like access to most of the menu tasks in Jade allowing very quick operations by a practiced user.

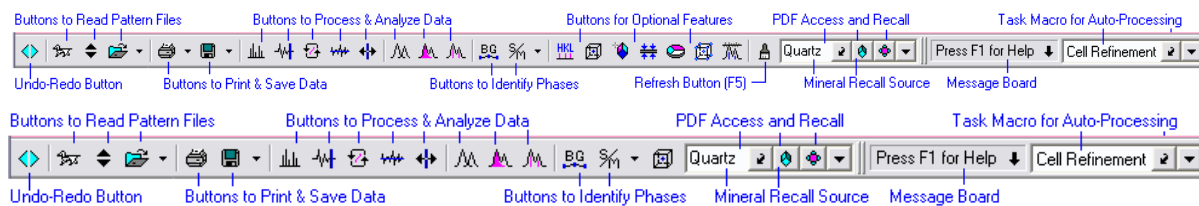
Explore Jade to Learn to Use it: Jade is an extremely powerful and complex program. While many of its functions are duplicated on the standard text-based menus (i.e., File, Edit, . . . Help), the Main toolbar (just below the menu), and a variety of floating and docked

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toolbars, there are some commands that are found only in one location. Anyone using Jade is well advised to spend some time practicing with the program, learning command locations, and reading the “Help” documentation. Every time I’m forced by program changes to revise this document for the XRD class, I find myself finding things I never knew existed.



Jade’s Main Toolbar (above) provides direct access to most XRD pattern processing functions. Students are encouraged to consult the Jade Help file for more extensive explanations of capabilities. Right click actions are in parentheses. Starting at left:

- Undo/Redo
- Fetch newest file by file date (Fetch file by name)
- Load next file on list (Load previous file on list)
- Read pattern file from Jade list (Read files from Windows file dialog)
- Print with current defaults (Print dialog to customize print)
- Save Data if work has been done (File save dialog to customize what is saved)

The buttons to process and analyze data include, from left to right:

- Automatic peak search with default parameters (Customize search parameters)
- Apply Filter to data – default is smooth data (Filter dialog)
- Swap of raw and derived data from filter (Clones derived pattern)
- Shows difference between pattern and identified phases (invert the difference pattern)
- Apply existing theta calibration or create a new one from a phase identified in the pattern (Calibration dialog)
- Apply profile fitting – peak deconvolution (Profile fitting dialog)
- Execute pattern deconvolution using current instrument parameters²
- Create and fit simulated patterns for various crystal systems or the displayed PDF overlay
- create simulated XRD pattern from crystal structure data (if available)

Buttons for optional features (all of which are available in our version of Jade+) are:

- Calculate d-spacing and Miller indices (Calculate lattice constants from peak locations and Miller indices)
- Cell Refinement from Pattern Data (Graph refined unit cell data)
- Pattern Indexing (Help with pattern indexing)
- Calculate residual stress from peak shifts (Help with RS calculation)
- Calculate XRD pattern from structural data (Structure database access)

The “Paintbrush” shows (and allows access to) any buttons hidden on the main toolbar.

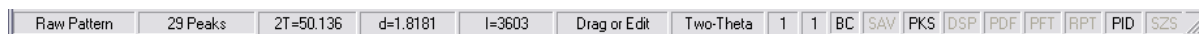
² This function requires that Jade be given appropriate machine parameters for the equipment being used and will give an error message if these parameters are not present.

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The buttons for phase identification and PDF access include, left to right:

- BG removes background and $K\alpha_2$ peaks from the pattern (BG removal dialog)
- S/M performs search match (Configure the search method and parameters)
- PDF Access and Recall allows retrieval by mineral name, database source, chemistry and a number of other parameters



Active Status Bar (above) is the bottom line of the screen, and includes information about the file as processed, the cursor position (2θ and intensity), and a list of all of the Jade file types (in black type) created for this pattern (most of which can be displayed by clicking on the file extension).

Scan Parameters (located above the Pattern Window) lists the scan parameters for the currently loaded file plus the file date and time.

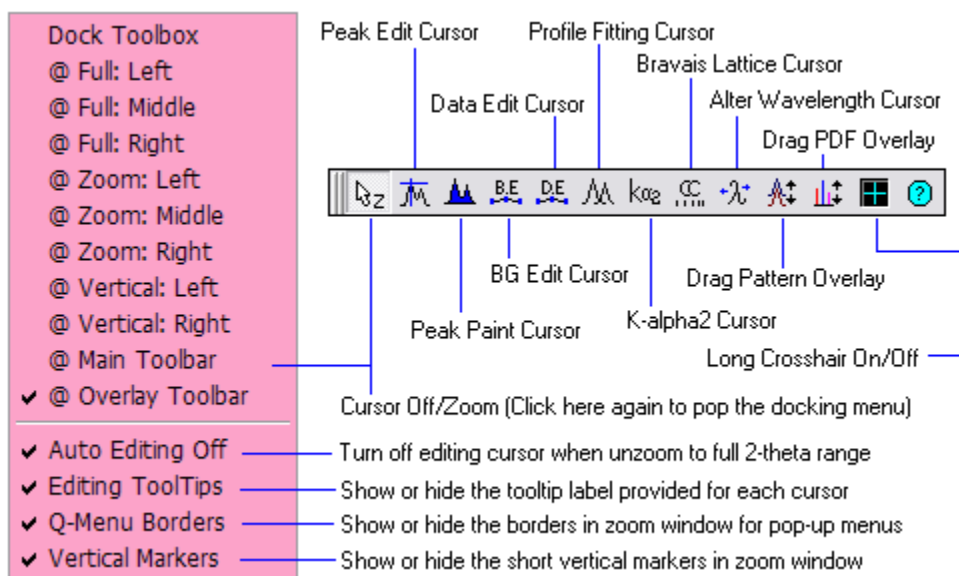
Pattern File Listing: List of all the data files found in the current directory; files may be opened (replacing the existing pattern) or added from this list.

Output File Listing: List of all files created by Jade during the analysis of particular data files. Some of the directly readable types are discussed later in this document.

PDF Recall box is part of Jade's Main toolbar (described above).

Zoom & Pan Toolbar: Allows adjustment of a variety of items including 2θ offsets of the pattern, reflection marker height adjustments, autoscale, horizontal and vertical offset adjustments, and automatic zoom and pan functions.

Phase Listing displays a defined subset of PDF (i.e., ICDD standard data) files. If Search/Match files are selected from a search, these may be displayed in a second window inserted above this one.



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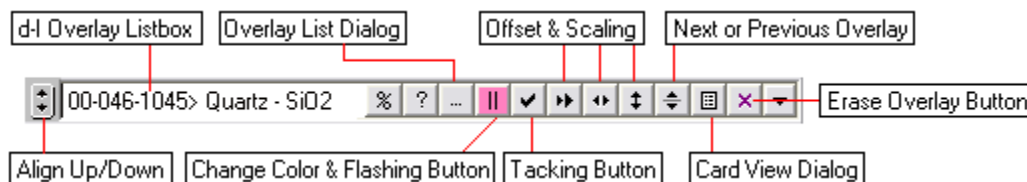
Cursor & Editing Toolbar (above) provides direct access to numerous analytical tools identified above that are applied by the selection and movement of the mouse cursor. The Dock Toolbox item is used to configure how the Toolbar is displayed. The tools are:

- **Cursor Off/Zoom:** This is the default state in which the zoom window can be “zoomed” by dragging a box around a region of interest, and the right-mouse accesses zoom window menus. By default, the toolbar “floats”; double-clicking pops off the docking menu for the toolbar (as shown).
- **Peak Edit Cursor** enables moving, editing and erasing of peak markers. This is particularly useful if the automatic peak search produces too many or too few peaks.
- **Peak Paint Cursor** enables manual graphical marking of areas under peaks by selection of areas along the baseline. By using the Ctrl-key with the Paint cursor, you can also do automatic marking of peak areas (see Jade help for details).
- **BG Edit Cursor** enables you to insert, move, and erase the tie points (dots) that control the shape of the fitted background curve. The BG curve is created with the BG dialog accessed on the main toolbar.
- **Data Edit Cursor** lets you manipulate displayed data manually. This does not modify the original data file, and is useful for eliminating aberrant spikes or other artifacts in your data. See Jade help for details of how this cursor is used.
- **Profile Fitting Cursor:** With this cursor, you can insert and edit initial profiles or remove them graphically. Generally the best approach with a complex pattern is to use the Profile Fitting tool on the main menu initially, then modify the profiles with the profile fitting cursor before further refining the pattern. See the Jade help topic on “Profile Fitting and Peak Decomposition” for a thorough discussion of this process.
- **K- α 2 Cursor:** When you move the mouse into the zoom window with this cursor, you should be able to see the K α 2 and K β lines of the active anode. You can also display the tungsten L-lines to check for filament contamination from an aging anode if you click the same button once more.
- **Bravais Lattice Cursor** creates stick patterns from your choice cubic, tetragonal and hexagonal (add your own cell parameters) Bravais lattices, that are dragged and resized as you drag them over your data in the zoom window. A good match will yield the unit cell length which can be used as input to a more precise indexing of your data (See “Unknown Pattern Indexing” in Jade help). Lattice parameters may also be chosen from matched PDF overlays as a starting point.
- **Alter Wavelength Cursor** lets you vary your X-ray wavelength dynamically and change the pattern accordingly.
- **Drag Pattern Overlay Cursor** lets you separate overlaid patterns vertically for visual comparison.
- **Drag PDF Overlay Cursor** lets you move PDF card overlays up or down in the window. For both of these “drag” cursors, it is important that unique colors are assigned to each overlay since Jade uses the colors to distinguish the overlay patterns moved.
- **Long Crosshair On/Off** changes the small cursor to a large, full-window vertical-horizontal crosshair.

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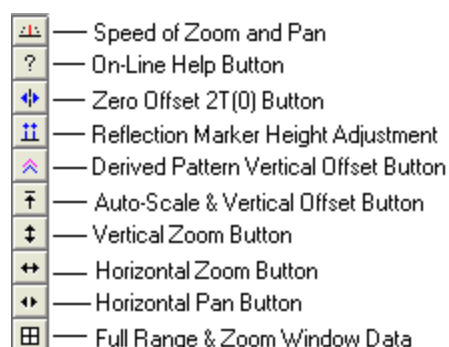
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PDF Overlay Toolbar (above) is accessible after any operations (including PDF retrieval and Search/Match) that add PDF card data to the list for the pattern in the zoom window. (See Jade Help for other ways to get custom overlay listings.) Most of the buttons are self explanatory. Of note are “Tacking” which is used to make a particular overlay permanent, “Erase Overlay” which permanently removes the selected overlay from the list, and the “Card View” which displays the PDF card data for the selected card.

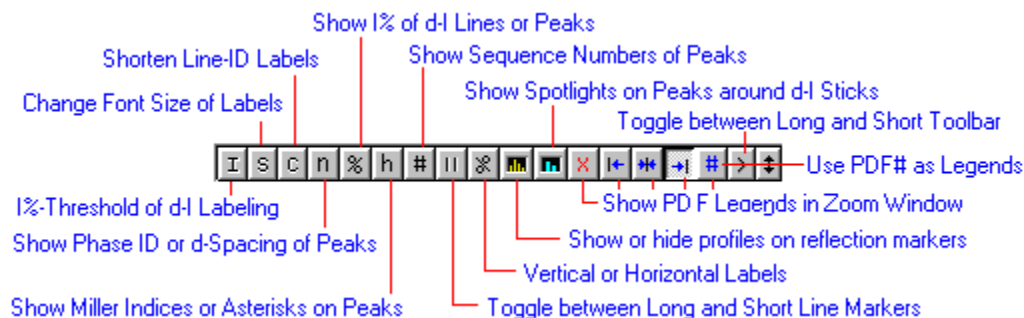
Zoom & Pan Toolbar is shown at right. It allows you to modify the pattern displayed in the zoom window in a number of ways including:

- Zero Offset is used to shift an entire pattern to correct for alignment or other errors in relation to a known standard.
- Reflection markers are produced in pattern refinements; this allows you to adjust how they are displayed.
- Derived patterns may be offset vertically for display purposes
- Auto-Scale & Vertical Offset button toggles the vertical scale of the zoom window between the maximum peaks in the zoom window and the whole pattern.
- Vertical and Horizontal Zoom Buttons zoom the pattern in or out vertically and horizontally.
- Horizontal Pan pans the zoom window left and right.
- Full Range & Zoom moves through the last few zoom window displays.



For more details about how to use the zoom and pan toolbar, consult the Jade help file.

Quick Annotation Toolbar (shown in horizontal orientation below) is used to configure how Jade displays a variety of pattern-related identification information including orientation, peak locations, font size, what is included or omitted, etc.



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A note on Modal vs. Modeless Operations: Dialog windows in Jade fall into two general groups with regard to their behavior: (a) modal dialog windows, and (b) modeless dialog windows. A modal dialog window requires you to close it to interact with its parent window. When you click outside the frame of such a dialog window, you will hear a beep from the PC speaker telling you that the click is invalid and thus ignored. A modeless dialog window has no such restriction, behaves very much like a toolbar or toolbox, and usually has no OK and Cancel buttons to dismiss it. The word 'modeless' means that the program is not constrained into a particular mode of operation which it needs to be released from. Many of the large scale operations in Jade (Search/Match, Card retrieval, profile fitting) are modeless in that they can be left open (or minimized) to interact with Jade's main window. Jade's Help contains a detailed and useful discussion of these two contrasting modes of operation.

Search Match Notes

By far the best way to learn how to use Jade's Search/Match is to study the sections on "Search Match for Phase ID" in the Jade online help. The approach is far more comprehensive than is possible here. The few suggestions below are from the author's experience with what tends to work best.

- The full pattern search/match is generally the best. For this to be effective it is important that background be removed and $K\alpha_2$ peaks stripped before the search. This can be done automatically (the program will offer to do it for you before the search) or manually using the background removal tool.
- Search/Match is very fast and can be done as many times as is necessary to produce good results. Start with the default settings, and vary them only as necessary to get good results.
- The following steps are suggested in Jade's documentation to produce good S/M results:
 1. Select the relevant PDF subfile(s).
 2. Start the initial search with the default parameters on the S/M setup dialog (right-click the Reset button).
 3. If no positive hit is found, increase the 2θ error window and do another search.
 4. If no positive hit is found, do a solid-solution search in the few % range.
 5. If no positive hit is found, focus the search on a few of the strong peaks at low angle. If there is a cluster of peaks, zoom around them and do another search.
 6. If no positive hit is found, check the 'Preferred Orientation S/M' box and do another search.
 7. If no positive hit is found, check the 'Perform Single Phase S/M' box and do another search.
- The line-based search/match is generally not as good at matching patterns exactly, but it does have a much higher tolerance for angular errors than the full pattern search. The line-based search dialog is accessed from the "Identify – Line-Based Search..." menu, and requires a peak ID before the search can proceed.

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- Use the Search/Match setup rather than the defaults in your S/M to be assured that you are searching the correct data file(s). “Inorganics” includes only experimental patterns; to select the ICSD calculated patterns be included in the search, it must be selected in the subfile list.
- Specimens containing more than a single simple phase can produce a large number of false hits in some situations. If you know the chemical composition of your specimen, you can use the chemical information to limit the permissible matches and reduce the false hits.
- The S/M process can be set to automatically shift patterns in a search by as much as $\pm 0.42^\circ 2\theta$, but large the angular deviation will still reduce the chances of a successful match result. If your S/M is not yielding good results and it appears that you may have systematic 2θ error in your data, apply, if possible, a theta calibration. This is done from the Main Toolbar and requires a recent pattern of a known standard material acquired on the system. Ask your lab manager for a recent pattern that could be used for this purpose.
- Strong preferred orientation can severely alter peak intensities in your pattern. Jade’s whole pattern S/M algorithms have trouble with strong preferred orientation. This can be alleviated somewhat by using the Preferred Orientation S/M checkbox in the search match setup. This will expand the scope of phases that the program will identify *but* will still order them based on intensities. You will still need to visually match the phases found to your data.
- One of the best pattern matching tools is the human eye. Never accept a computer S/M result without making sure that it is a good match visually.
- Bulk analyses of complex multi-phase samples (a.k.a. rocks) will sometimes identify a few major phases with automated S/M, but will never get the minor ones. For rocks, use only the Minerals and ICSD Minerals subfiles. Know your samples. One of the best techniques for working with rocks is to create a set of likely phases in a special search set and limit your searches to that set. Another option is to create a d-I list for all of the possible phases in your sample and match the pattern visually, selecting the best matches from what you compare with your data.

Jade’s documentation has tons of helpful hints and information. Make use of it and you will probably be pleased with the results.

Whole Pattern Search Match

New in Jade 9 (actually added with Jade 8.5) is a whole-pattern matching method of doing search match. It is accessed by selecting the Search/Match setup dialog and then clicking on the “S-W” button in the dialog. Here the specified subfile is searched according to the current S/M parameters and possible hits are automatically run through a whole pattern fitting routine. Jade then selects the best fitting phases, and repeats the S/M and WPF round on the residual pattern until the residual pattern disappears or three iterations are carried out.

This new addition to Jade may be used in combination with the more traditional pattern matching and line matching methods to solve complex mixtures.

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Jade Data File Formats

Jade dynamically creates a large number of data files as the data analysis process proceeds. Many of these files are used as controls in configuring the program and controlling overlays and other dynamic (but usually non-permanent) modifications to your data. All files are defined by different extensions that indicate the file type; the first part of the filename (before the extension) is usually the same for all different file types (although it is possible for operators to save files using user-selected file names).

Binary Control Files: Some of the important control files (that use binary formats) to be aware of are:

- *filename.SAV*: Used to save all work in progress (i.e., whatever has been done in the program with your data) allowing it to be recovered in a subsequent session. This is very handy when multiple sessions are needed for complete analysis. The SAV file actually creates a series of binary and ASCII data files that are read when reloading your session by opening the SAV file. Some of these include the extensions BIN, DIF, PKS, PDF, PFT, and BKG. Some of these may be saved and loaded individually through the File | Save and File | Open menus. (Hint #1: Multiple sessions may be saved by using different filenames for the SAV files, but you must be careful with multiple SAV files, because some can use the same control files and induce strange behavior. Hint #2: When saving a session, the current state of the Zoom window is saved, not the entire pattern. To save the entire pattern, the Zoom window should be expanded to show the entire pattern before saving.)
- *filename.PKS*: A binary version of the peak list file created by identifying peaks and only readable by Jade.
- *filename.JIP*: A binary thumbnail file of patterns used by Jade to find similar patterns

ASCII Text Files: Some of those file types that yield ASCII text information that may be read outside of Jade are listed and described below. Those that are useful for “stand-alone” reporting of data are shown in *bold italics*.

- ***filename.PID***: Peak ID file lists all peaks and parameters; created when peaks are identified and report viewed.
- ***filename.IDE***: Extended Peak ID file lists all peaks and ICDD standard matches; created when matches are picked after Search/Match or by manual retrieval and selection.
- ***filename.MDI***: MDI data file (format described previously) created when data collected.
- ***filename.TXT***: ASCII text data file, created by saving MDI data file in ASCII format. Makes a two column (2θ - Intensity) data file that is very easy to plot outside of Jade. The first line of the file is the sample description.
- ***filename.PDF***: PDF overlay list of all selected ICDD standard cards
- ***filename.AID***: A d-spacing vs I% listing file in ICDD AIDS83 format.
- ***filename.DSP***: A d-spacing vs I% listing file in the MDI two column (d-spacing – rel. intensity) format.
- ***PDF#00-000-0000.TXT***: Output of ICDD data file (using the new relational database naming system explained in the next section).

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The New ICDD Relational Database (RDB) Format

PDF4-RDB now employs 9-digit numbering system to accommodate large entries of calculated powder patterns. The first two digits of this 9-digit number (e.g. 01-070-0001) is the so-called database code, which designates the source or type of PDF data. Jade 9 supports this numbering system with the following MDI extensions of database codes (90 to 99):

ICDD Codes:

- 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: Calculated PDF data from Linus Pauling File structural data.

Codes Added by Jade:

- 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).
- 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).

The old ICDD flat-file database used a 6-digit number (e.g., 46-1045) and Jade 9 includes backward compatibility with older versions of the database. The new RDB structure has been forced by the explosion in the number of patterns available (over 285,000 in the newest ICDD PDF4 version), and the need to add the flexibility of a true relational database structure that allows the relatively easy addition of new data (like detailed structural data) to database entries for which these are available.

Data Acquisition and Analytical Steps for a “typical” Experiment

How data are collected and processed is totally dependent on the purpose of the analysis. You generally start with the question you are trying to answer, and devise your experiment to best help you to answer that question. A huge part of any experiment is presenting your results in a form that clearly separates your analytical data from your interpretations of that data, and transmits maximum information about what you have discovered. Below is an example of the steps involved in hypothetical project from received material to final report.

1. The client leaves a powdered sample for analysis. The material is a laboratory-produced powder that has certain known elements in it. The client is interested in what the major phase(s) are in the sample and if there are any leftover starting materials.
2. The powder is examined for uniformity. Since the work is qualitative in nature, no significant sample prep is required.
3. The ICDD data for the likely phases suggested by the client are examined using the ICDD retrieval function of Jade to examine what analytical range should yield the peaks of interest for the experiment.

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4. The specimen is side-drifted into a Plexiglas mount for analysis. It is run at $1^\circ/\text{min}$ (continuous scan, 0.02° step size) between 10° and 60° 2θ (based on information obtained in Step 3).
5. The resulting pattern is loaded in Jade and examined. Peaks generally sharp and well defined, but there are several smaller peaks that have a somewhat broader shape than the dominant peaks.
6. A peak search is done on the raw data pattern, and the printed and standard peak file list printed. Digital copies (JPEG of the pattern, a Peak-ID listing "PID" text file for peaks) are saved with the data. This provides the raw sample data for the client.
7. A full pattern search/match is done on the pattern. Initial search with default parameters yields results not reasonable given the known chemistry. A repeat search/match with a slightly larger 2θ error window yields some phases which are good matches both visually and for the known chemistry. These phases are checked in the match results list to be saved.
8. The S/M results do not contain anything that matches the small peaks in the pattern. Repeat searches with larger error windows, and preferred orientation SM still yield nothing reasonable. The match results window is closed, returning to the zoom window interface.
9. It is decided to do a manual retrieval and compare the cards visually. Before doing this, the derived pattern with the background removed is erased leaving the raw data, and the zoom window intensity rescaled to the full window. It is always a good idea to visually compare to the raw data because minor peaks that tend to be flattened into the background will be more visible to the eye.
10. A PDF retrieval is selected using Chemistry. The five possible elements in the powder are selected and a list of all matches using the Inorganic (PDF and ICSD) databases. This produces about 60 possible phases. Each of those phases is compared visually with the minor peaks in the pattern. A phase (identified as matching several calculated and experimental cards) is found that matches the minor peaks and also has minor peaks that match peaks hiding in the "noise" of the background. This phase is checked and added to the results, and the match results window closed.
11. In the main window, the PDF overlay toolbar is used to evaluate which of the selected cards best match the phases in the sample. The "Tack" button is used to select the best card matches, and the others are deleted with the Delete button.
12. From the Print – Setup window, pattern is displayed with the card file patterns overlaid on it, and/or in boxes adjacent to it. The latter is generally best to show matches of multiple phases. One or both of the output types is selected, and printed. A digital version of the "match" printout is saved as JPEG with the data.
13. At the main menu, from the View – Reports menu, the extended peak file report is selected and printed. This shows all of the peaks in the pattern and the peaks matches between the cards and the pattern. A digital version of this file is saved (plain text file with an "ide" extension) with the data.

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14. A two-column text data file (Column 1 = angle; column 2 = counts) may be saved from the Jade File | Save As menu as an "ASCII Data File." This type of file is printed easily by any program that can plot simple X-Y data.
15. From the PDF overlay tool, each matched phase is selected in succession and the PDF card for it is printed for the client. Digital versions of these cards may be saved by clicking on the save file (Diskette) icon while displayed.
16. The data as printed are assembled and a brief report of results prepared and delivered to the client. Many (most?) clients in this digital age also want a digital copy of all the results sent with the report; this may be fairly easily assembled from the saved digital data files.

This hypothetical project (based on many actual projects that I have done over the years) is a relatively simple qualitative search/match and report on results. Hopefully it gives you a general idea about how data collection and reporting should be approached.