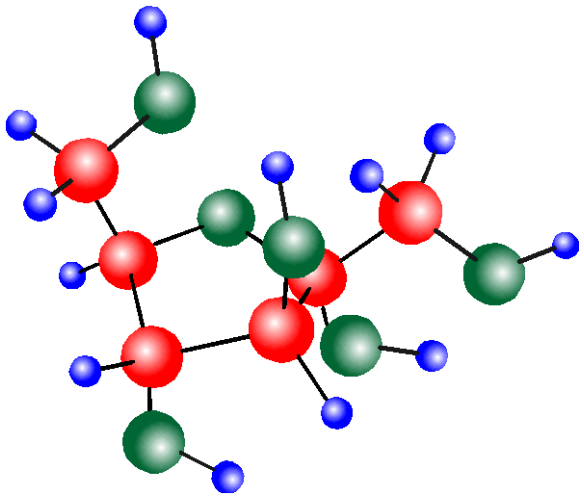
XRDCalc



MinServ

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Introduction

About XRDCalc

XRDCalc is a Windows powder diffraction utility for identifying phases, calculating and plotting powder diffraction data. XRDCalc calculates d-spacings and two-theta and plots digital scan files, two-theta or d-spacing vs intensity stick plots and peak profiles.

XRDCalc Features

Built in Spreadsheet

XRDCalc has an inbuilt spreadsheet with clipboard support for entering and editing peak data.

Builtin Mineral XRD Database

A builtin Mineral XRD Database contains the strongest three d-spacings for all minerals. The database can be searched by mineral name or d-spacing.

Plot Digital Scan Files

Import and plot ASCII, RRuff (*.rruff), Sietronics (*.cpi), dbw type (*.dat) and Scanpi(*.dat) digital scan files. Perform search/match and quantitative analysis on the plots.

XRD Peak Plots

Select from two-theta vs intensity or d-spacing vs intensity stick plots or curve plots. A choice of labels and titles are available to customize the XRD. Labels can be placed on any number of peaks. Prevent crowding of data by selecting extra wide XRD's.

On screen control includes data sensitive peaks so that each peak of the XRD is

related to a particular set of data listed within a spreadsheet.

Data Manipulation

Data can be sorted numerically or alphabetically. Both way conversions between two-theta and d-spacing are available with a click of the mouse. Calculations can be solved using any wavelengths. When two-theta for a particular wavelength is unknown, a solution using known d-spacings can be obtained.

Printing

Graphs can be customized with a choice of labels and titles. Data can be printed in a list or exported into a spreadsheet package.

About X-Ray Diffraction

X-ray diffraction is a method for analyzing the crystal structure of crystalline materials. It is in widespread use to identify crystalline materials that are difficult to characterize using other methods. Minerals, metals, alloys and organic molecules are the main materials analyzed. Some subjects that employ this method are materials science, mineralogy, physics, chemistry and metallurgy. The mining and chemical industries have routinely adopted this method of analysis.

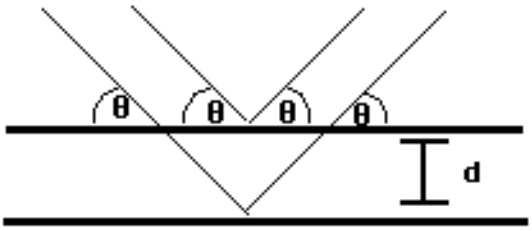
X-ray diffraction depends upon the principle of diffraction to indirectly measure the spacings (d-spacings) between lattice planes within crystals. Typically, x-rays of a specific wavelength are passed through a powdered sample mounted on a glass plate. Since the lattice planes are at the appropriate spacing to diffract certain wavelengths of x-rays, each diffracted beam corresponding to a specific d-spacing can be measured. The measured value can then be matched to reference values to identify the crystal.

With a powder diffractometer, x-rays are directed through a powdered sample that is rotated through an angle θ. The detector is rotated through 2θ on the same axis and measures intensities and 2θ angles of the diffracted rays. Output is plotted via a strip chart recorder or recorded and processed using a microcomputer system and

appropriate software.

A monochromatic beam of x-rays hits the powdered sample at an angle θ and the grating effect results in scattering. There is only one angle that satisfies Braggs Law and at which mirror reflection of the incident beam occurs for a specific d-spacing. At this angle, reflected waves from adjacent hkl planes are in phase and have a measurable intensity. The intensity and geometry of x-rays diffracted by a crystal are affected by the properties and arrangement (particularly the electronic configurations) of the constituent atoms. D-spacings are calculated from the two-theta angles using Braggs Law. From the geometry of x-ray diffraction, Braggs Law is obtained

$n\lambda = 2\, d_{hkl} \sin\theta$ where:
n = an integer equal to the diffracting lattice plane
λ = wavelength of radiation
d = d-spacing. The spacing between adjacent hkl planes
θ = angle the incident and reflected beams make with the lattice plane hkl
2θ = two-theta. The angle between incident and diffracted beams



Getting Started

XRDCalc requires a minimum system configuration for effective results. The system requirements are:

Windows 2000, XP, Vista, Windows 7

Internet Explorer 6 SP1
Devices supported by Windows
Printer supported by Windows

For automatic installation, proceed as follows:

CD SETUP:

1. Go to the Run Option on the Start Menu and Select Browse
2. Open (CDROM)D:\Disk1\Setup.exe to Setup XRDCalc
3. Setup will start. You should not have any problems, but if you do have problems, you may need to install the latest service pack for your Windows to update system files. Service packs are available from the Microsoft Windows Update Site, http://windowsupdate.microsoft.com/
4. Setup will start and copy the program files to the PC's Hard Disk
5. After Setup has concluded, you will get a [App] Setup was Completed Successfully message.
6. To Start XRDCalc, go to the XRDCalc Menu and click on XRDCalc. Enter your registration details in the Registration Window.
7. IMPORTANT: We recommend using the online help, and hardcopy manual to learn more about XRDCalc. Installation will now proceed. During installation, the user is prompted for a directory. If this is ignored, a new directory called XRDCalc will be created. If a directory path is entered, a new directory with this name will be created. The user will be notified when installation is complete. A new Menu Item containing XRDCalc and XRDCalc HELP is created. Click on XRDCalc to start the program.

Tutorial

Introduction

Sample data files are provided for initial use with the program. To load a sample peak data file, click Open from the File menu and select rhyolite.xrc in the xrdcalc.exe directory. To load a digital scan file, select Plot Scan File and Click on the Browse Button next to the Filename Box. Select one of the *.cpi files in the xrdcalc.exe directory to plot or use your own file. If help is required, press F1 to access online help . Alternatively, pull down the Help menu and select Contents.

Getting To Know XRDCalc

To load the rhyolite.xrc data file, click the Open command button or select Open from the File menu. The Open dialog box will pop up. Select rhyolite.xrc in the directory containing xrdcalc.exe. If no directory was specified by the user during installation, the default directory name is XRDCalc

Hint:

Use the Calc button as a shortcut to calculate data. The default calculation for Calc is d-spacings. To change the default setting, select a new calculation (two-theta or d-spacings) from the Calc menu.

Before proceeding with a calculation, the correct radiation must be set by the user. Pull down the Calc menu and select Set Wavelength. A value can be entered in the edit area of the list box or selected from the list. Choose CoKa from the list and click OK to accept the wavelength and return to the spreadsheet. The selected wavelength is displayed in the bottom left hand corner of the spreadsheet. Pull down the Calc menu and click the d-spacing command to calculate d-spacings from two-theta.

Entering Peak Data

Select New from the File menu to start a new data file. Data is entered in each cell by simply typing the value and pressing <enter>. Use the scroll bars, arrow, page up and page down keys to navigate through the spreadsheet. To edit a cell, press <enter> for the cell you want to edit and use the backspace/delete/insert keys to edit the text. When finished editing press <enter> the new value will appear in the spreadsheet. Whole rows can be inserted and deleted using the Insert Row and Delete Row command buttons. Blank lines are not permitted between data. Similarly, blank or zero values mixed with two-theta or d-spacing data will produce errors in the calculation.

Enter the following data:

Two-theta	D-spacing	Intensity	Crystal Name
26.66	3.34	100	Quartz
28.14	3.17	45	Plagioclase
8.77	10.1	38	Biotite
29.47	3.03	55	Calcite

To edit a cell, go back to Row 2, Col 3 and press <enter>. Type 42 to replace 45 and press <enter>.

When you have finished using the program, select Exit from the File menu.

Plotting Peak Data

Click the Set XRD Options command in the Options menu. A new window will display several options. Accept the default values and click the Plot button. A XRD of two-theta vs intensity will appear on a new form. To plot a label, Click row #3 in the grid list. This row and its corresponding peak on the XRD will now be highlighted. Click on two-theta in the labels list to the left of the grid and a two-theta label will be displayed. Try different labels. All labels can be deleted by selecting clear labels on the labels list. When finished, click the OK button and you will be returned to the

Options window.

Select two theta in the Set Labels menu and manual in the Scale Type menu. Enter 20 for Xmin and 45 for XMax. Enter an XRD title and click the Plot button. A new XRD with the selected scale and labels is displayed. Labels created are automatically printed with the XRD when Print XRD is selected. To return to the spreadsheet click OK in the Plot and Options screens.

Hint:

Use the mouse to navigate through the spreadsheet. Click a cell to change the current cell or click on the scroll bars to display out of view data. To begin editing a cell, double click the mouse.

Plotting a Digital Scan File

To plot a digital scan file, a scan file must be open in the spreadsheet or Plot Scan Options Window. To open a scan file, import a file using the Import Command. There are several different file formats supported by XRDCalc, including text files. Other file formats can be imported using the ConvX application supplied with XRDCalc and accessed with the Convert Command. For this exercise, select one of the Sietronics scan files (*.cpi) in the default XRDCalc.exe directory.

Once imported, select Plot Scan File from the Options Menu. The Plot Scan Options Window will appear. Select Plot From Spreadsheet and click on the Plot Button. The scan file should now be plotted.

Alternatively, a file can be opened directly from the Plot Scan Options Window by clicking on the Browse Button and selecting one of the supported files. A radiation can be entered for files that do not contain this information. It is much faster to plot a file directly from disk rather than using the spreadsheet. Once the scan file is plotted, peaks can be extracted and search/match can be performed.

Hint:

If an error occurs during a calculation or while plotting data, the most likely cause is incorrect data. Check for zero or blank data values. Mixed numeric and non-numeric data will also produce errors.

Performing a Search/Match

Auto Search Match

This option performs a Search/Match of the builtin mineral database using the digital scan file in the current spreadsheet or Plot Window. All peak filtering is done automatically, no manual adjustment of settings is necessary. A list of matches will be returned to the spreadsheet under the Name column. During the Search/Match, if the three strongest d-spacings are found, the mineral will be returned as a match. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option
3. D-spacing and Two-theta are formatted correctly in the Format Option

Use the Accurate Match Option in most cases. This will search the peak file for d1, d2 and d3. This option gives the best results. If you get zero likely matches, use the Expanded Match Option which searches the peak file for d1, d2 and d3, but also doubles the search width. This option will give the most matches, but beware, it will most often give false matches. If no likely matches are found, a manual search/match will probably be required. The Auto Search/Match Option may give a large number of matches. If no likely matches are found, a manual search/match will probably be required using the filters available and specifying a wider search range for d-spacings. A manual search/match can be performed following the auto search/match to further refine the search.

Manual Search/Match

This option performs a Search/Match of the builtin mineral database using the peaks in the current spreadsheet or Plot Window. This should only be performed on extracted peaks, or after the auto search/match.

1. Extract peaks, for example peaks > 5% intensity
2. Remove overlapping shoulder peaks with Refine Peaks
3. The result will be a peak file with discrete peaks

If the peaks have been read directly from a chart and entered into the spreadsheet, the previous 3 steps are not necessary. If the auto search/match has already been performed, the extracted peaks should already be displayed, the previous 3 steps are not necessary.

Specify a d-spacing search range for performing the search. All minerals matching the peak within this search range will be classified as a match. The wider the search range, the more matches obtained. A list of matches will be returned to the spreadsheet under the Name column. During the Search/Match, if the three strongest d-spacings are found, the mineral will be returned as a match. Some manual refinement of the results may be required. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option
3. D-spacing and Two-theta are formatted correctly in the Format Option

Use the Accurate Match Option in most cases. This searches the peak file for d1, d2 and d3. This option gives the best results. If you get zero matches, try widening your search. If you continue to get zero likely matches, use the Expanded Match Option. This searches the peak file for any peak that matches d1, d2 or d3, but beware, it will most often give false matches.

Backing Up Databases

The Mineral XRD Database is editable. Any changes to the database are saved to the database; however, it is highly recommended, the XRD Database is backed up periodically. The easiest way to do this is to copy the database file, xray.mdb, to a backup folder of your choice using Windows Explorer or My Computer File Manager.

XRDCalc Menu System

Introduction

When XRDCalc is started the first screen displayed contains a spreadsheet and menu system. The menu system provides access to all the features of XRDCalc. Standard Windows menu items; such as, File, Edit and Help are included. Other menu items provide access to the specialized features of XRDCalc.

File Menu

The File Menu provides many standard file management options.

New Command

Deletes data in the current spreadsheet to begin a new file

Open Command

Provides access to the Open dialog box. Opens a new file. Select the directory and file and click OK. A file can also be opened by clicking the Open command button. The Open Command is for opening XRDCalc peak files (*.xrc)

Import Command

XRDCalc can import the following digital scan files:

RRuff *.rruff
Sietronics *.cpi
DBW Type *.dat
ScanPI Type *.dat

Other file types can be plotted by using the file conversion utility in the File Menu or by importing a raw file into the spreadsheet and plotting directly from the spreadsheet.

Import Text File Command

Imports a text file. Select the type of file to import:
Space Delimited
Comma Delimited
Tab Delimited
Semi Colon Delimited
Fixed Width
Specify the number of lines to skip at the beginning of the file to avoid importation of file headings into the spreadsheet.

Convert Command

Opens the ConvX application by M. Bowden, New Zealand. ConvX converts many powder diffraction file formats. Distributed with permission.

Save Command

Saves a file to disk. The default filename is the current filename. The same result is obtained by clicking the Save command button.

Save As Command

Saves a file to disk in a directory and filename specified by the user. A Save As dialog box pops up for the user to enter filename and directory path information.

Print Command

Prints a data file. A Print dialog box pops up. Select Print to print the file. The same result is obtained by clicking the Print command button.

Print Setup Command

Specifies printer settings available to the user. A Print Setup dialog box pops up with several options to control the printer. Use this option to set the default printer and various printer settings.

Exit Command

Quits the application.

Edit Menu

The Edit Menu provides access to several standard commands for editing the spreadsheet.

Copy Command

Copies selected text in the spreadsheet to the clipboard without deleting. Highlight the text to be copied by dragging the mouse or holding down the shift key and clicking cells. Highlighted text must be contiguous. Select Copy to copy the selected text to the clipboard. Use Paste to paste text from the clipboard to the spreadsheet. Use Cut to remove highlighted text from the spreadsheet to the clipboard.

Cut Command

Copies selected text from the spreadsheet to the clipboard and clears the highlighted area in the spreadsheet. Highlight text to be cut by dragging the mouse or holding down the shift key and clicking cells. Select Cut from the edit menu to transfer selected text to the clipboard. Use Paste to retrieve cut text from the clipboard. Use Copy when you want to copy text to the clipboard without deleting the highlighted text.

Paste Command

Retrieves text from the clipboard and pastes it into selected cells. Highlight cells that are to receive the incoming text by dragging the mouse or holding down the shift key and clicking cells. Highlighted cells must be contiguous. Select Paste from the Edit

menu to transfer text from the clipboard to the spreadsheet.. Any text that already exists in the highlighted cells will be replaced. If the number of selected cells are fewer than will hold the clipboard text, the rightmost text from the clipboard will be truncated to fit the selected cells. If the number of cells selected exceed the length of the clipboard text, the remaining cells will be cleared. Use Copy and Cut to send text to the clipboard.

Search Menu

The Search Menu allows the user to find and replace user specified text.

Find Command

Finds a user specified search text in the spreadsheet. A pop up Search/Replace dialog box prompts the user for the search text. Enter the exact search text and click OK to proceed with the search.

Repeat Find Command

Repeats a search using the previous specified search text.

Replace Command

Finds a user specified search text and replaces it with a user specified replace text. A pop up dialog box prompts the user for search text (Find What) and replacement text (Replace With). The following options are available:

Replace- replace the first occurrence of the search text with the specified replace text

Replace All - replace all occurrences of the search text with the specified replace text.

Sort Menu

The Sort Menu allows the user to sort data in the spreadsheet according to two-theta, d-spacing, intensity or crystal name.

Two-theta Command

Sorts data in the spreadsheet into increasing numerical order by two-theta value.

d-spacing Command

Sorts data in the spreadsheet into increasing numerical order by d-spacing value.

Intensity Command

Sorts data in the spreadsheet into decreasing numerical order by intensity.

Crystal Name Command

Sorts data in the spreadsheet into alphabetical order by crystal name.

Calc Menu

The Calc Menu permits the user calculate two-theta values from d-spacings or d-spacing values from two-theta. The wavelength used in the calculation can be specified by the user.

2-theta Command

Calculates two-theta values from d-spacings. The correct radiation used in the calculation must be set by the user with the Set Wavelength command. Blank or zero d-spacing values mixed with data will produce an error in the calculation. If the 2-theta command has a check mark, the calculation can also be activated by clicking the Calc command button.

d-spacings Command

Calculates d-spacings from two-theta values. The correct wavelength used in the calculation must be set by the user with the Set Wavelength command. Blank or zero two-theta values mixed with data will produce an error in the calculation. If the d-spacings command has a check mark, the calculation can also be activated by clicking the Calc command button.

Database Menu

The Database Menu provides access to the builtin Mineral XRD Database or an external database specified by the user. The builtin Mineral XRD Database contains the strongest three d-spacings and mineral name for all minerals. From the Database Window a simple search can be performed and the results printed.

Backing Up Databases

The Mineral XRD Database is editable. Any changes to the database are saved to the database; however, it is highly recommended, the XRD Database is backed up periodically. The easiest way to do this is to copy the database file, xray.mdb, to a backup folder of your choice using Windows Explorer or My Computer File Manager.

Mineral XRD Database Window

The Mineral XRD Database is editable. To enable editing, goto the Edit Menu and select Enable.

A search can be performed on the database using the search text entered and clicking on search. There are several user definable options for performing searches, including searching specific fields and numeric d-spacing searches.

Options Menu

Set XRD Options Command

Accesses the Options Window to select the XRD settings prior to plotting.

Colors Command

Provides access to the Colors dialog box for selecting the plotting Colors and line styles. Select a number or click on the Color or linestyle to place the corresponding code number in the spreadsheet.

Assign Colors Command

Automatically assigns a Color to each crystal and places the appropriate Colour code into the spreadsheet. Crystal names must be entered into the spreadsheet for this option in order to identify each component.

Auto Color Command

If this option is checked, Colors are automatically calculated for each component and plotted with the stick plot. Crystal names must be entered into the spreadsheet for this option to work properly.

Format Command

This command opens the format dialog to set numeric formatting of d-spacing and two-theta. Enter the number of decimal places to round d-spacing or two-theta. Select Save to save the formatting information without formatting the data or Format to format the data immediately.

Insert Column Headings Command

Select this option to insert standard column headings into the spreadsheet. Column headings will be placed in Row 1 and in the columns set with the Define Columns command. D-Spacing, Intensity, Two-Theta and Crystal Name headings are inserted.

Intensity to Percentage Command

Converts intensity values that have been read from the powder diffractometer as

counts to percentage. Enter the upper intensity limit of the data and click on calculate to convert.

Extract Peaks Command

Extracts peak intensities and two-theta values above a specified intensity from 2-theta/Intensity digital scan files.

Typically, extracting peaks is a two part process. Peaks are first extracted from the scan file using the Extract Peaks Option. This creates a new spreadsheet or table with extracted peaks. There will be many shoulder peaks from the initial scan file and it is necessary to Refine and Filter the Peaks to obtain a final peak file. Use the Refine Peaks Option to filter out shoulder peaks.

Refine Peaks Command

This option removes adjacent shoulder peaks from the digital scan file. Specify a range for adjacent peaks and all adjacent peaks within this range will be deleted. Use the recommended values if you are unsure which values to use. Typically, extracting peaks is a two part process. Peaks are first extracted from the scan file using the Extract Peaks Option to extract peaks above a specified intensity value. This creates a new spreadsheet with extracted peaks. There will be many adjacent shoulder peaks from the initial scan file and it is necessary to Refine and Filter the Peaks to obtain a final peak file. Use the Refine Peaks Option to filter out adjacent shoulder peaks. Once the peaks have been extracted and filtered, a Search/Match can be performed. You should aim to obtain the strongest 12 peaks for multiphase samples and the strongest 4 peaks for single phase samples, in order to perform a search/match.

Search/Match Command

This option performs a Search/Match of the builtin mineral database using the peaks in the current spreadsheet or Plot Window. This should only be performed on extracted peaks, or after the auto search/match.

If the peaks have been read directly from a chart and entered into the spreadsheet, the following 3 steps are not necessary.. If the auto search/match has already been performed, the extracted peaks should already be displayed, the following 3 steps are

not necessary.

4. Extract peaks, for example peaks > 5% intensity
5. Remove overlapping shoulder peaks with Refine Peaks
6. The result will be a peak file with discrete peaks

Specify a d-spacing search range for performing the search. All minerals matching the peak within this search range will be classified as a match. The wider the search range, the more matches obtained. A list of matches will be returned to the spreadsheet under the Name column. During the Search/Match, if the three strongest d-spacings are found, the mineral will be returned as a match. Some manual refinement of the results may be required. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option
3. D-spacing and Two-theta are formatted correctly in the Format Option

Use the Accurate Match Option in most cases. This searches the peak file for d1, d2 and d3. This option gives the best results. If you get zero matches, try widening your search. If you continue to get zero likely matches, use the Expanded Match Option. This searches the peak file for any peak that matches d1, d2 or d3, but beware, it will most often give false matches.

Auto Search/Match Command

This option performs a Search/Match of the builtin mineral database using the digital scan file in the current spreadsheet or Plot Window. All peak filtering is done automatically, no manual adjustment of settings is necessary. A list of matches will be returned to the spreadsheet under the Name column. During the Search/Match, if the three strongest d-spacings are found, the mineral will be returned as a match. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option

3. D-spacing and Two-theta are formatted correctly in the Format Option

Use the Accurate Match Option in most cases. This searches the peak file for of d1, d2 and d3. If you get zero matches, try using the Expanded Match Option. This searches the peak file for d1, d2 and d3, but also doubles the search width. This option will give the most matches, but beware, it will most often give false matches. If no likely matches are found, a manual search/match will probably be required using the filters available and specifying a wider search range for d-spacings. A manual search/match can be performed following the auto search/match to further refine the search.

Search Mineral From Database

Performs a search of all peaks listed in the Plot Window Data Table and returns a match if all three mineral values match the peak values. Select a mineral from the database to search for and specify a range. All peaks that fall within the specified range are returned as a match.

Define Columns Command

When a data file in a different format is imported , columns containing d-spacing, intensity, two-theta, crystal name and/or Color must be defined using this command so that the calculations and plotting will be performed correctly. Minimum requirements are d-spacing and intensity. Enter a column number between 1 and 8 in the Define Columns dialog box that corresponds to the data type. Empty columns can be assigned to two-theta, crystal name and Color if no data is available or these can be left blank in the Define Columns dialog box. If your data file contains additional data, it will still be displayed intact and all spreadsheet functions will work.

Set Wavelength Command

Sets the radiation and wavelength used in the d-spacings and two-theta calculations. A pop up dialog box appears with a list of radiations. A value can be selected from the list by clicking a value with the mouse or entered via the keyboard in the edit window of the list box. Several options are displayed:

OK - accepts the radiation displayed in the edit window and exits the dialog box.

Add - adds the radiation displayed or entered in the edit window to the list box.

Delete - deletes the radiation highlighted in the list area of the list box. Highlight the radiation to be deleted then select delete.

Radiation must be entered in a specific format. A straight numerical value is acceptable. Alpha-numeric text with an "=" identifier preceding the numerical part is also acceptable e.g.:

1.5418 valid
CuKa=1.5418 valid
CuKa1=1.5418 valid
CuKa 1.5418 invalid

Plot Menu

Plot Command

Accesses the Options Window to select the XRD settings prior to plotting peak data. The same result is obtained by clicking the Plot command button.

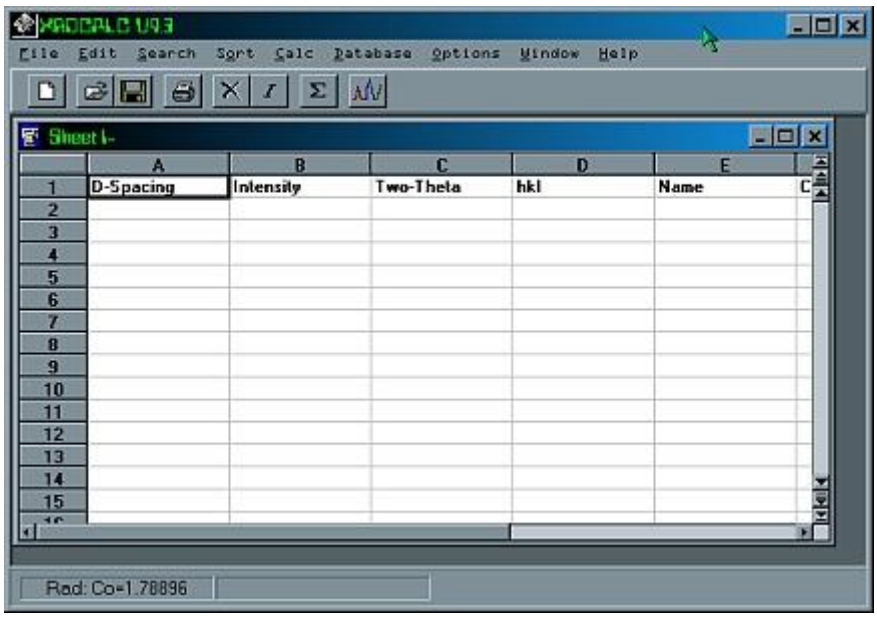
Plot Scan Command

Accesses the Plot Scan File Options Window to select the plotting settings prior to plotting a digital scan file

The Spreadsheet

Introduction

A spreadsheet is a form consisting of rows and columns of data and is the best method for displaying and manipulating data in row and column format. The custom spreadsheet supplied with XRDCalc is used for entering and editing data. Once the data is correctly entered, data can be sorted, calculated and plotted.



File Formats

XRDCalc can import the following digital scan files:

RRuff *.rruff
Sietronics *.cpi

DBW Type *.dat
ScanPI Type *.dat

Other file types can be plotted by using the file conversion utility in the File Menu or by importing a raw file into the spreadsheet and plotting directly from the spreadsheet.

The built in spreadsheet reads and writes data files in tab delimited ASCII format. This format is used by Microsoft Excel and is compatible with most spreadsheets. ASCII is short for American Standard Code for Information Exchange. ASCII files contain standard text without hidden characters. A tab delimited ASCII file is a text file with each field separated by a tab character and each line by a carriage return. XRDCalc files with the .xrc or .dat extension are tab delimited ASCII files. XRDCalc uses files containing data only with the first row reserved for column headings. Files containing any extra information; such as titles, heading information and row numbers will require editing before they can be used with XRDCalc.

The built in spreadsheet requires data to be in a specific format for plotting and converting. XRDCalc uses a custom *.xrc file format. Tab delimited ASCII files can be imported using Import in the File Menu. Imported files are displayed intact. In order to use XRDCalc's features it is necessary to correctly define which columns contain d-spacings, two-theta, intensity, crystal name and color with the Define Columns command. As a minimum, XRDCalc requires d-spacing and intensity values to work. If no data exists for some headings, leave the column value defined in the Define Columns Window blank or set to zero or assign an empty spreadsheet column. Additional data can be entered via the keyboard or using the clipboard.

Graphs created with XRDCalc can be saved as graphics files for exporting into paint programs, word processors and other applications. Graphs can be saved in the following formats:

1. bitmap (*.bmp)

Select Save XRD As from the plot menu and enter the filename with the appropriate graphics extension.

Entering Data

Data is entered in each cell by simply typing the value and pressing <enter>. Use the scroll bars, arrow, page up and page down keys to navigate through the spreadsheet. Use the scroll bars when moving around the spreadsheet. To edit a cell, press <enter> for the cell you want to edit and use the backspace/delete/insert keys to edit the text. When finished editing press <enter> the new value will appear in the spreadsheet. Whole rows can be inserted and deleted using the Insert Row and Delete Row command buttons. Blank lines are not permitted between data. Similarly, blank or zero values mixed with data for two-theta or d-spacing will produce errors while calculating or plotting. The spreadsheet can contain a maximum of 1500 rows.

Sorting Data

Data can be sorted numerically or alphabetically. To sort data, pull down the Sort menu. The following options are displayed:

- two-theta - sort two-theta into increasing numerical order
- d-spacing - sorts d-spacing into increasing numerical order
- intensity - sorts intensity into decreasing numerical order
- crystal name - sorts crystal name into alphabetical order

When an option is selected, data is sorted and displayed in the spreadsheet.

Hint:

If data was entered incorrectly an error will occur. Check for mixed numeric and non numeric data.

Calculating Data

Two types of calculation are possible. If two-theta's are known, d-spacings can be calculated. If d-spacings are known, two-theta's can be calculated using any radiation. The correct wavelength must be set before starting a calculation.

To set a wavelength, pull down the Calc menu and click Set Wavelength. A Set Wavelength dialog box will appear. Enter a wavelength in the edit area of the list box or select a wavelength from the list. Click OK to accept the wavelength and return to the spreadsheet. To proceed with the calculation, click:

d-spacings to calculate d-spacings
2-theta to calculate two-theta's

The calculation proceeds and the results are displayed in the spreadsheet.

Hint:

If an error occurs in the calculation, the most likely cause is incorrectly entered data. Check for zero, blank, duplicated or mixed numeric and non numeric data.

Printing Data

Data is printed by pulling down the File menu and selecting Print. A Print dialog box will pop up with several options. Select OK to print the data. A simple listing of current data will be printed.

Plotting Digital Scans

Plot Scan Options Window

Introduction

The Plot Scan Options Window sets options to plot a digital scan file XRD.

File Name to Plot

Click on the Browse Button to select a scan file to plot. The following scan file types can be plotted:

RRuff *.ruff
Sietronics *.cpi
DBW Type *.dat
ScanPI Type *.dat

Other file types can be plotted by using the file conversion utility in the File Menu or by importing a raw file into the spreadsheet and plotting directly from the spreadsheet.

Set Wavelength

Click on the Get Button to select a wavelength to use for converting Two-theta to D-spacings in the Plot Scan Window. If the scan file contains wavelength information then this value will be used. The wavelength can also changed after the file is plotted using the Set Wavelength Command.

Title

Enter a Title for the Scan Plot here.

Plot From Spreadsheet

If this option is checked, the current file displayed in the spreadsheet is plotted.

Generally, plotting from a file on disk is much faster, however, plotting from a spreadsheet is sometimes more convenient when working with imported files.

Plot Scan Window

The Plot Scan Window displays the XRD Scan File on screen with several options and controls. The XRD Scan File can also be printed.

Plot Scan Commands

Auto Search Match
Axes Labels
Axes Titles
Box Draw
Copy XRD
Extract Peaks
Fonts
Grid
Inset Text
Labels
Line Draw
Print Peak File
Print XRD
Quantitative Analysis
Refine Peaks
Save XRD As
Search/Match
Search Mineral From Database
Undo
XRD Color
XRD Size
XRD Width
Zoom

Auto Search/Match Command

This option performs a Search/Match of the builtin mineral database using the digital scan file in the current spreadsheet or Plot Window. All peak filtering is done automatically, no manual adjustment of settings is necessary. A list of matches will be

returned to the spreadsheet under the Name column. During the Search/Match, if the three strongest d-spacings are found, the mineral will be returned as a match. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option
3. D-spacing and Two-theta are formatted correctly in the Format Option

Use the Accurate Match Option in most cases. This will search the peak file for d1, d2 and d3 in any order. This option gives the best results. If you get zero likely matches, use the Expanded Match Option which searches the peak file for d1, d2 and d3, but also doubles the search width. This option will give the most matches, but beware, it will most often give false matches. If no likely matches are found, a manual search/match will probably be required using the filters available with the Refine Peaks and Search/Match Commands. The Auto Search/Match Option may give a large number of matches. If this occurs, perform a new search/match of the extracted peaks using the Search/Match Command and specifying a narrow search range for d-spacings.

Axes Labels

Select this option to change the axes labels font. A font dialog box pops up for selecting the font.

Axes Titles

Select this option to change the axes title font. A font dialog box pops up for selecting the font.

Box Draw

To draw a box, select box draw. The cursor will change to a cross hair. Position the cross hair where the box is to begin and drag the cursor by holding down the left mouse button. A dotted box is dragged with the mouse. Release the left mouse button when you have positioned the box correctly.

Copy XRD Command

Copies the current XRD to the clipboard for pasting into other applications.

Delete All

Deletes all drawing and text actions and redraws the plot

Extract Peaks

Extracts peak intensities and two-theta values above a specified intensity from 2-theta/Intensity scan files. Recommended to use the Plot Scan Window to extract peaks as this is much faster than importing a scan file into a spreadsheet and extracting peaks there.

Typically, extracting peaks is a two part process. Peaks are first extracted from the scan file using the Extract Peaks Option. This creates a new spreadsheet or table with extracted peaks. There will be many shoulder peaks from the initial scan file and it is necessary to Refine and Filter the Peaks to obtain a final peak file. Use the Refine Peaks Option to filter out shoulder peaks.

Fonts

To change the font for inserted text select Fonts. A Font dialog box is displayed with a list of available fonts. Select a font in the list.

Grid

Displays a list of Peak values that appear on the XRD. The peak represented by the selected text in the grid will be highlighted.

Insert Text

To insert text select Insert Text. The cursor will change to a cross hair. Position the cursor where you would like to insert text and click the left mouse button. Type the text to be inserted.

Labels

This option determines which labels appear on the XRD. Select from:

1.none - no labels appear on the XRD
2.two-theta - two-theta values
3.d-spacing - d-spacing values
4.Intensity - intensity values
5.crystal name - crystal names
6.hkl - hkl label
7. User Defined

Labels can be placed on all peaks or the current selected peak only.

Line Draw

To draw a line select Line Draw. The cursor will change to a cross hair. Position the cursor where you would like the line to begin and drag the line by holding down the left mouse button and moving the mouse. Release the left mouse button when the line is positioned correctly.

Print XRD

Prints the current XRD. A pop up Print dialog box appears. Click OK to accept the default settings and print the XRD. If labels are displayed, these will be printed with the XRD. If peaks appear to be overlapping, select a smaller scale or use landscape orientation. Full XRD scans print best with landscape orientation and normal size. The size and scale of the XRD should be adjusted to give the best result.

Quantitative Analysis

This option does a quantitative analysis of selected peaks. To do a quantitative analysis, select the required peaks from the peak table and click on the Quantitative Analysis menu item. A new options window will pop up asking for the background value. Use the automatic setting or enter a background value for the current XRD

scan. The automatic setting can only be used for scans with little background scattering; that is, with a relatively uniform background. Click on calculate to perform the analysis. Peak areas will be calculated using background corrected intensities and the result, including percentage, will be displayed in a new window. Quantitative analysis requires a high quality scan to achieve optimum results. All major peaks for the target phase must be selected to get an accurate result.

Refine Peaks

This option removes adjacent shoulder peaks from the peak file. Specify a range for adjacent peaks and all adjacent peaks within this range will be deleted. Use the recommended values if you are unsure which values to use. Typically, extracting peaks is a two part process. Peaks are first extracted from the scan file using the Extract Peaks Option to extract peaks above a specified background value. This creates a new spreadsheet with extracted peaks. There will be many adjacent shoulder peaks from the initial scan file and it is necessary to Refine and Filter the Peaks to obtain a final peak file. Use the Refine Peaks Option to filter out adjacent shoulder peaks.
Once the peaks have been extracted and filtered, a Search/Match can be performed. You should aim to obtain the strongest 12 peaks for multiphase samples and the strongest 4 peaks for single phase samples, in order to perform a search/match.

Save XRD As Command

This command saves the current XRD in a user specified directory and filename. A pop up dialog box appears which prompts the user for directory path and filename information. Graphics files can be save in bitmap (.bmp) format for exporting into a painting program for further editing.

Search/Match Command

This option performs a Search/Match of the builtin mineral database using the peaks in the current spreadsheet. Specify a d-spacing search range for performing the search. All minerals matching the peak within this search range will be classified as a match. The wider the search range, the more matches obtained. A list of matches will be returned to the spreadsheet under the Name column. During the Search/Match, if

the three strongest d-spacings are found, the mineral will be returned as a match. Some manual refinement of the results may be required. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option
3. D-spacing and Two-theta are formatted correctly in the Format Option

Search/Match is best performed on the strongest 12 peaks for multiphase samples and the strongest 4 peaks for single phase samples, otherwise multiple matches may be obtained. Use Refine Peaks Option to filter out adjacent shoulder peaks.

Use the Accurate Match Option in most cases. This will search the peak file for d1, d2 and d3 in any order. This option gives the best results. If you get zero matches, try using the Expanded Match Option. This searches the peak file for the any peak that matches d1, d2 or d3. This option will give the most matches, but beware, it will most often give false matches.

Search Mineral From Database

Performs a search of all peaks listed in the Plot Window Data Table and returns a match if all three mineral values match the peak values. Select a mineral from the database to search for and specify a range. All peaks that fall within the specified range are returned as a match.

Title

Enter a title for the XRD with this option.

Undo

To use Undo, first select the drawing action to undo then click on the undo button or the undo menu command.

XRD Color

Select this option to change the XRD Color. A Color dialog box pops up for selecting the Color of the XRD. Select a Color and click OK.

XRD Size

Select normal or small to change the default size of the printed XRD.

Zoom

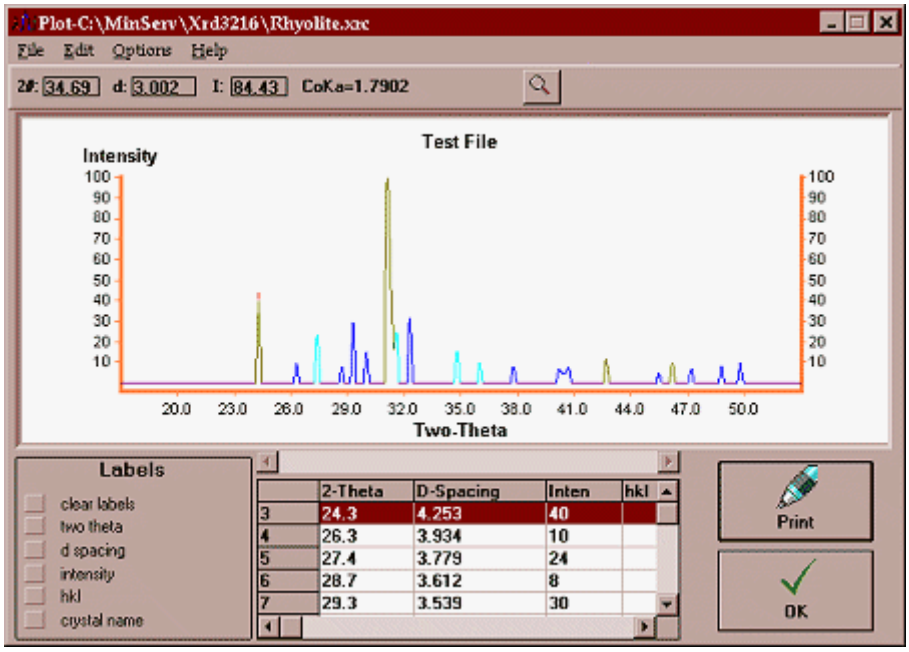
To magnify all or part of the plot click on the zoom toolbar button. Drag the mouse over the portion of the plot to be magnified. A new window will display the magnified image.

Plotting Peak Data

Introduction

Data can be plotted as d-spacing vs intensity or two-theta vs intensity stick plots or curve plots. Many options are available to customize the XRD. Publication quality graphs can be produced on a laser printer.

Before plotting data, several options must be set to control the type and appearance of the XRD. To access the Options window, pull down the Options menu and select Set XRD Options.



Options Window

The Options window has many user definable settings to control the XRD.

XRD Type

This option determines the type of XRD to be plotted. Select either:

1. two-theta vs intensity

2. d-spacing vs intensity

and

1. stick plot

2. curve plot

There is a choice of stick plots or curve plots. The appearance of the curve plot is affected by the scale and size of the plot.

Set Labels

This option determines which labels appear on the XRD. Select from:

1.none - no labels appear on the XRD

2.two-theta - two-theta values

3.d-spacing - d-spacing values

4.Intensity - intensity values

5.crystal name - crystal names

5.hkl - hkl label

Labels can also be added after the XRD has been plotted using the Labels option in the Plot window. Labels allows further control on the placement and type of labels.

Scale Type

This option determines the type of scale on the XRD. Select either:

1. Automatic - automatically calculates the scale using the

minimum and maximum data values.

2. Manual - allows the user to specify the range of values plotted

Set Scale

When a manual Scale Type is selected the user must specify the x maximum and x-minimum on the XRD. The user must enter:

1. XMin - The minimum data value plotted on the x axis

2. XMax - The maximum data value plotted on the x-axis

All values ranging between XMin and XMax are plotted.

Line Width

Sets the width of sticks on the XRD. A value between 1 (narrow) and 4 (thick) is valid.

XRD Width

Sets the width of the XRD on the screen. Select from:

1. extrawide - XRD width is twice the screen width. Use the scroll bar to view the XRD. Use with large data files to prevent crowding of peaks and labels

2. normal - XRD width is equal to the screen width

Plot Window

The Plot window displays the XRD and sends output to the printer or disk. The Plot window contains a menu bar, a graph area, grid list, labels option buttons and command buttons. The XRD contains data sensitive peaks that are linked to a grid list

of data. Each highlighted peak corresponds to a highlighted set of data within the grid. A choice of labels can be added to any number of peaks for display on screen or output to the printer. XRD's can be saved as bitmap (.bmp) graphic files for exporting to other applications.

Axes Labels

Select this option to change the axes labels font. A font dialog box pops up for selecting the font.

Axes Titles

Select this option to change the axes title font. A font dialog box pops up for selecting the font.

Box Draw

To draw a box, select box draw. The cursor will change to a cross hair. Position the cross hair where the box is to begin and drag the cursor by holding down the left mouse button. A dotted box is dragged with the mouse. Release the left mouse button when you have positioned the box correctly.

Copy XRD Command

Copies the current XRD to the clipboard for pasting into other applications.

Fonts

To change the font for inserted text select Fonts. A Font dialog box is displayed with a list of available fonts. Select a font in the list.

Grid

Displays a list of Peak values that appear on the XRD. The peak represented by the selected text in the grid will be highlighted.

Insert Text

To insert text select Insert Text. The cursor will change to a cross hair. Position the cursor where you would like to insert text and click the left mouse button. Type the text to be inserted.

Labels

Determines the type of label to display on the XRD. Choose from:

1. clear labels - clears all labels on the current XRD

2. two-theta - the current two-theta value is plotted

3. d-spacing - the current d-spacing value is plotted

4. intensity - the current intensity value is plotted

5. crystal name - the current crystal name is plotted. The full crystal name is plotted.

6. hkl - the current hkl value is plotted

To display a label, click a row in the grid. The peak corresponding to the row will be highlighted on the XRD. Now click on the label button to display the label for the highlighted row. Repeat the procedure to display labels for other peaks. When Print XRD is selected, labels will be printed with the XRD. Greater control for plotting labels is available with the Labels command in the Options Menu.

Line Draw

To draw a line select Line Draw. The cursor will change to a cross hair. Position the cursor where you would like the line to begin and drag the line by holding down the left mouse button and moving the mouse. Release the left mouse button when the line is positioned correctly.

Log Scale

Click this option to change between log linear and linear scales. Only available for stick plots.

Search/Match Command

This option performs a Search/Match of the builtin mineral database using the peaks in the current spreadsheet. Specify a d-spacing search range for performing the search. All minerals matching the peak within this search range will be classified as a match. The wider the search range, the more matches obtained. A list of matches will be returned to the spreadsheet under the Name column. During the Search/Match, if the three strongest d-spacings are found, the mineral will be returned as a match. Some manual refinement of the results may be required. If no matches are found please check the following:

1. The correct radiation has been entered in the Set Wavelength Option
2. The spreadsheet columns are defined correctly in the Define Columns Option
3. D-spacing and Two-theta are formatted correctly in the Format Option

Search/Match is best performed on the strongest 12 peaks for multiphase samples and the strongest 4 peaks for single phase samples, otherwise multiple matches may be obtained. Use Refine Peaks Option to filter out adjacent shoulder peaks.

Use the Accurate Match Option in most cases. This will search the peak file for d1, d2 and d3 in any order. This option gives the best results. If you get zero matches, try using the Expanded Match Option. This searches the peak file for the any peak that matches d1, d2 or d3. This option will give the most matches, but beware, it will most often give false matches.

Search Mineral From Database

Performs a search of all peaks listed in the Plot Window Data Table and returns a match if all three mineral values match the peak values. Select a mineral from the database to search for and specify a range. All peaks that fall within the specified

range are returned as a match.

Set Scale

This option changes the d-spacing or two-theta scale. Enter new minima and maxima and click OK to plot the new scale.

Set Wavelength

This option changes the default wavelength for calculating two-theta and d-spacings.

XRD Color

Select this option to change the XRD Color. A Color dialog box pops up for selecting the Color of the XRD. Select a Color and click OK.

XRD Size

Select normal or small to change the default size of the printed XRD.

XRD Type

Change between d-spacing or two-theta vs intensity XRD's with this option.

XRD Width

This option changes the width of the XRD between normal and extrawide width. Only available for on screen display. XRD's are displayed best with extrawide width. Use the XRD Size option to change the size of the printed XRD.

Title

Enter a title for the XRD with this option.

Undo

To use Undo, first select the drawing action to undo then click on the undo button or the undo menu command.

XRD Database

The MinServ XRD Minerals Database contains XRD data on all minerals. Over 3700 minerals with mineral name, three strongest d-spacings and intensities, chemical formula and crystal system are listed.

Using the XRD Database

The XRD Database has all entries indexed by mineral name and d-spacing and is displayed in tabular format. The table can be browsed using scroll bars. To perform a query, enter the search terms in the search option boxes. Mineral name and d-spacing can be searched and d-spacing search limits can be entered or selected.

Important: Search limits must be entered for d-spacing searches. In most cases, a narrow search pattern will give the most accurate results. If the XRD peaks are uncertain, perform a wider search, by specifying a wider d-spacing range.

Click on Search to perform the search. A list of matches is shown in the main window. Scroll through the minerals to see each record that matches the query. The results may be printed. The database can be sorted according to mineral name or d-spacing from the Edit Menu and all data can be printed.

This database is editable. Enable editing by selecting Edit Database, then Enable. Once the edit mode is enabled, any changes made to the database should be saved by using the Save Command or clicking on the Refresh Button. A new record can be entered by selecting Add New Record. A new record with empty fields is created.

Important: Blank and null entries are not allowed in the database. If no entry is desired under a specific property then enter "Empty" for that specific property. Default values for all properties are "Empty". This word is automatically added to new entries.

More information can be displayed on a mineral by clicking on the More Info button. The mineral name, formula and crystal system are displayed, if this information is available. If not, information can be added by using the Edit Menu.

Software Support

Technical support for XRDCalc is provided through the printed manual and online help. Updates can be obtained free of charge using the builtin ftp update program. If an insurmountable problem occurs further technical support is available directly from Mineral Services; however, the software must be registered to receive support.

MinServ (Mineral Services)
email: support@geologynet.com
http://www.geologynet.com

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