

# PDF-4 User's Guide to Synchrotron Data Analysis

## and Neutron Data Analysis

**International Centre  
for  
Diffraction Data**



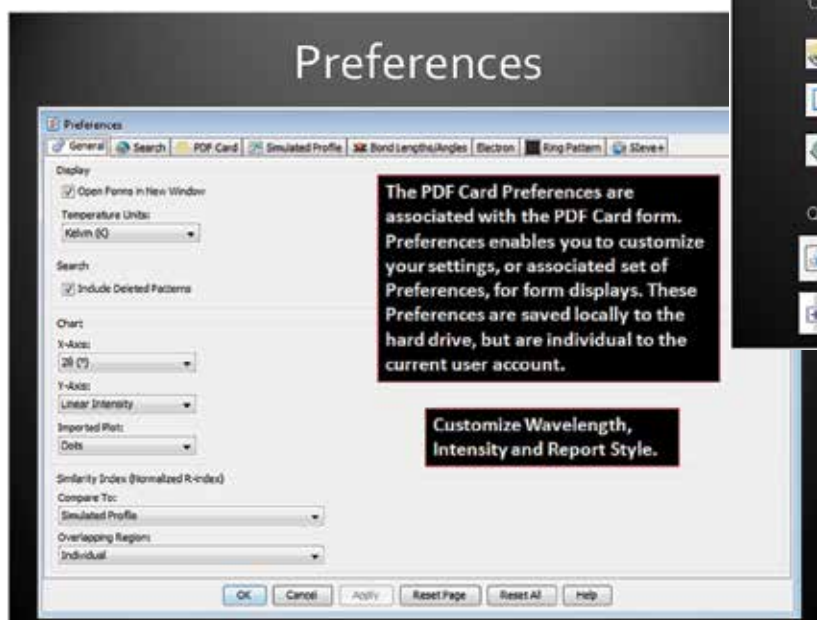
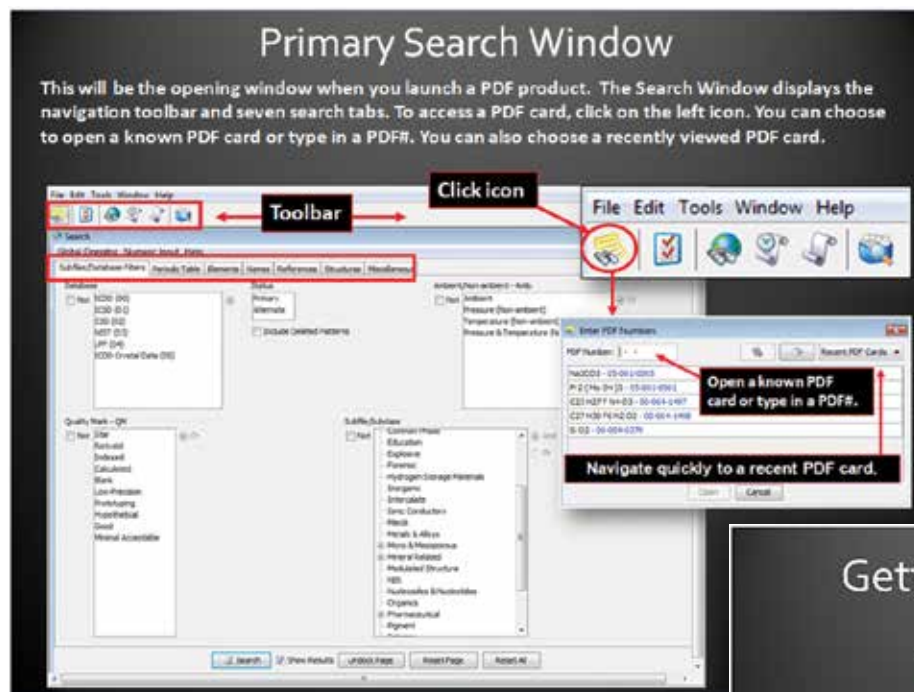
# Getting Started

**ALL** ICDD Powder Diffraction File™ databases can be opened as a stand-alone program. Our integrated software offers users flexible searches, data mining, and graphics capabilities.

## To access the PDF Database:

Choose the PDF database icon on your desktop OR

Start menu → All programs → ICDD PDF Product (folder) → ICDD PDF Product



For additional details on using PDF-4 products, please access USE PDF-4 DATABASE tutorials from the ICDD website ([www.icdd.com/resources/tutorials](http://www.icdd.com/resources/tutorials)).

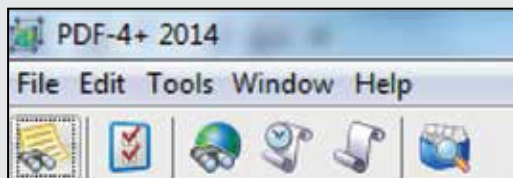
## Overview

*This guide was developed based on the content and capabilities of the ICDD 2014 PDF-4 databases. This includes PDF-4+ 2014, PDF-4/Minerals 2014, and PDF-4+/Web 2014. PDF-4/Organics 2015 includes the described synchrotron capabilities, but not the neutron diffraction capabilities.*

*This guide takes the user through the successive steps of doing a phase identification and subsequent quantitative analysis using Sleve+ for PDF-4, ICDD's optional plug-in search-indexing software for phase identification and quantitative analysis. Sleve+ is activated for a free 30-day trial period upon installation of a PDF-4 database. In order for Sleve+ to be activated beyond the free trial period, a license for Sleve+ must be purchased from ICDD or your vendor. Sleve+ is included in the PDF-4/Organics and PDF-4/Scholar licenses.*

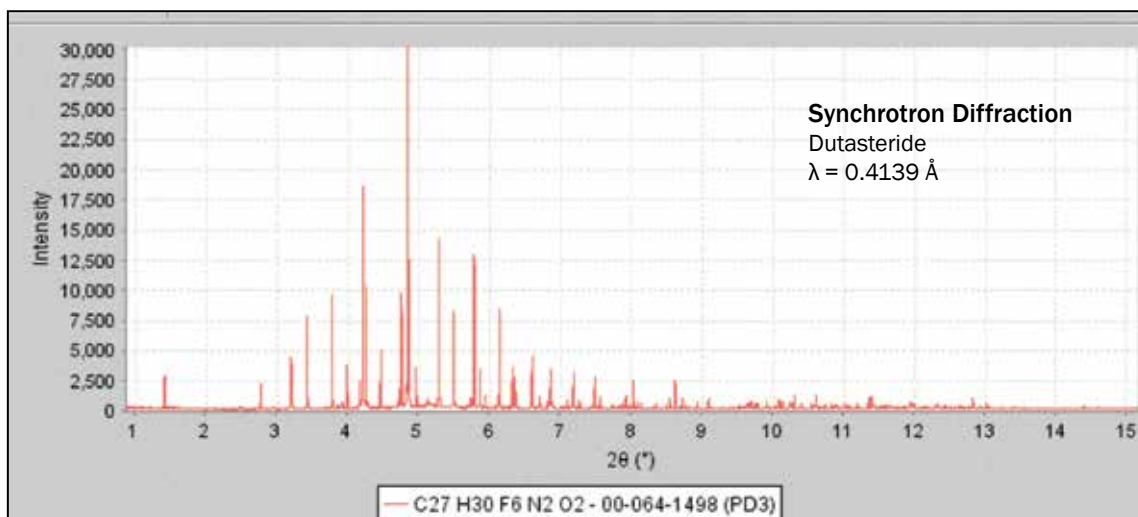
*The capabilities described within were developed in conjunction with ICDD member scientists, many of whom work at dedicated synchrotron and neutron user facilities. The growing list of user facilities has resulted in a large group of users that need to process both laboratory and user facility data. PDF-4 databases have been designed to satisfy this need.*

*We have used a select number of data sets and material types to demonstrate common features in this guide. Additional features and educational materials can be found in our Help documentation and on our website, [www.icdd.com](http://www.icdd.com). Our website contains references, publications, tutorials, and videos.*



*The navigation toolbar offers icons to access many of the features described in this guide. The figure above shows the toolbar on the opening window. The Preferences tool, Section II, is the second icon on the left and the Sleve+ tool (Sections IV and V) is the icon on the far right.*

# Synchrotron Diffraction



## Topics

- I. Introduction
- II. Reference Pattern Simulations
- III. Data Input and Processing
- IV. Phase Identification
- V. Quantitative Analysis (RIR)
- VI. Quantitative Analysis (Rietveld)
- VII. Nanomaterials, Amorphous Materials, Non-crystalline Materials
- VIII. Options
- IX. References

## I. Introduction

Worldwide synchrotron facilities offer high brilliance sources that provide very high resolution with advanced optics and detector systems. The growth of user facilities and mail-in programs has greatly expanded the general access to these facilities and the number of global users processing and analyzing synchrotron diffraction data.

The ICDD has developed a series of tools for the analysis of synchrotron data based on input from our users and ICDD members that work at synchrotron facilities around the world. Many ICDD members participate in the ICDD technical committee on Synchrotron and Neutron Methods. Their input was invaluable in database design and software capabilities that are described in this guide.

### Key Facts

*All references in PDF-4+ annual releases can be converted to synchrotron diffraction patterns through the use of pattern simulations. Instrument functions for synchrotron experiments are incorporated in the software. The high resolution and large dynamic intensity scale of most synchrotron experiments enables superior phase identification and quantitative analysis results.*



## Topics

- I. Introduction
- II. Reference Pattern Simulations
- III. Data Input and Processing
- IV. Phase Identification
- V. Quantitative Analysis (RIR)
- VI. Quantitative Analysis (Rietveld)
- VII. Nanomaterials, Amorphous Materials, Non-crystalline Materials
- VIII. Options
- IX. References

### I. Introduction

Worldwide neutron diffraction facilities offer a unique capability to analyze and resolve atomic and molecular structures based on the fundamental physics of diffraction from the nucleus. The ability of neutrons to penetrate deeply into most specimens makes it the method of choice for many types of in situ analyses. The growth of user facilities and mail-in programs has greatly expanded the general access to these facilities and the number of global users processing and analyzing neutron diffraction data.

The ICDD has developed a series of tools for the analysis of constant wavelength neutron data based on input from our users and ICDD members that work at neutron facilities around the world. Most of the tools are also applicable for the analysis of time-of-flight (TOF) neutron data. Many ICDD members participate in the ICDD technical committee on Synchrotron and Neutron Methods. Their input was invaluable in database design and software capabilities that are described in this guide.

#### Key Facts

*Neutron diffraction reference patterns can be calculated for all entries having atomic coordinates or scattering factor tables (currently > 240,000 materials). Instrument functions for neutron diffractometers are incorporated in the software, as well as scattering factors for common isotopes.*

# Synchrotron Diffraction

## II. Reference Pattern Simulations



Synchrotron data is interpreted by comparison to known reference standards. All data in the Powder Diffraction File™ (PDF®) can be simulated as synchrotron diffraction patterns using pattern simulations.

### Step 2. Input Synchrotron Wavelength

### Step 1. Set Synchrotron Defaults

The simplest way to simulate synchrotron data is to use the “Preferences – Simulated Profile” window shown above. The user needs to do a two-step procedure that sets synchrotron defaults and establishes the wavelength used for the material being analyzed. Setting the synchrotron defaults in the “Preferences” result in the following automated steps:

1. Establishes the radiation as monochromatic
2. Sets the instrumental profile parameters
3. Sets the step size for the simulation
4. Automatically selects the appropriate algorithm for the simulation calculation

The instrumental profiles are initially set to an average of published synchrotron profile parameters (Kaduk and Reid, **2011**) using a modified Thomas-Cox-Hastings (TCH) pseudo-Voigt function. The user can easily override this selection. Alternatively, the user can input an instrument profile function using several different common formats.



## II. Reference Pattern Simulations



Neutron data is interpreted by comparison to known reference standards. Approximately 240,000 entries in the Powder Diffraction File™ (PDF®) can be simulated as neutron diffraction patterns of constant wavelength.

### Step 2. Input Neutron Wavelength

The screenshot shows the 'Simulated Profile' window with the following settings:

- Radiation:**
  - ☒ Neutron Diffraction\*
    - Wavelength (Å): 2.124
    - Packing Factor: 0.6
    - Sample Can Diameter (mm): 8.0
  - ☐ Electron Diffraction
    - Energy (keV): 100.0
- Geometry:**
  - ☒ Debye-Scherrer\*
    - Polarization Fraction\*: 0.98
    - Sample Thickness (mm)\*: 0.1
  - ☐ Bragg-Brentano: Fixed Slit
- Profile:**
  - ☒ Gaussian
    - U: 0.0984
    - V: 0.13324
    - W: 0.0903
  - Significance Limit: 0.01
- Range:**
  - Step Width (°): 0.01 (To properly simulate synchrotron data, this value should be 0.005 or less.)
  - Start 2θ:
    - ☒ Longest Line Minus (°): 10.0
    - ☐ Fixed Value (°): 2.0
  - Stop 2θ:
    - ☒ Shortest Line Plus (°): 10.0
    - ☐ Fixed Value (°): 150.0

At the bottom, there are buttons: Set Synchrotron Defaults, Set Neutron Defaults, Import Instrument Parameters, OK, Cancel, Apply, Reset Page, Reset All, and Help.

### Step 1. Set Neutron Defaults

The simplest way to simulate neutron data is to use the “Preferences – Simulated Profile” window shown above. The user needs to do a two-step procedure that sets neutron defaults and establishes the wavelength used for the material being analyzed. Setting the neutron defaults in the “Preferences” result in the following automated steps:

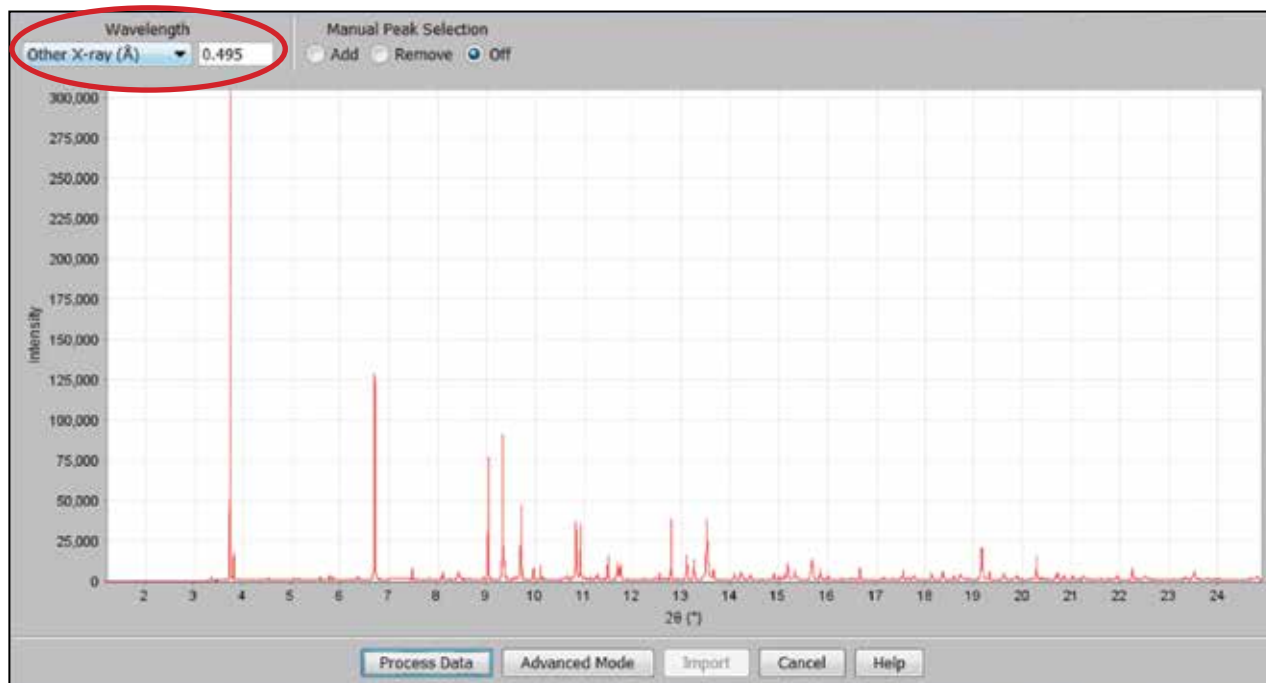
1. Establishes the radiation as monochromatic
2. Sets the instrumental profile parameters
3. Sets the step size for the simulation
4. Automatically selects the appropriate algorithm for the simulation calculation

The instrumental profiles are initially set to an average of published neutron profile parameters (Faber et. al., **2014**) using a Gaussian profile function. The user can easily override this selection. Alternatively, the user can input an instrument profile function using several different common formats.

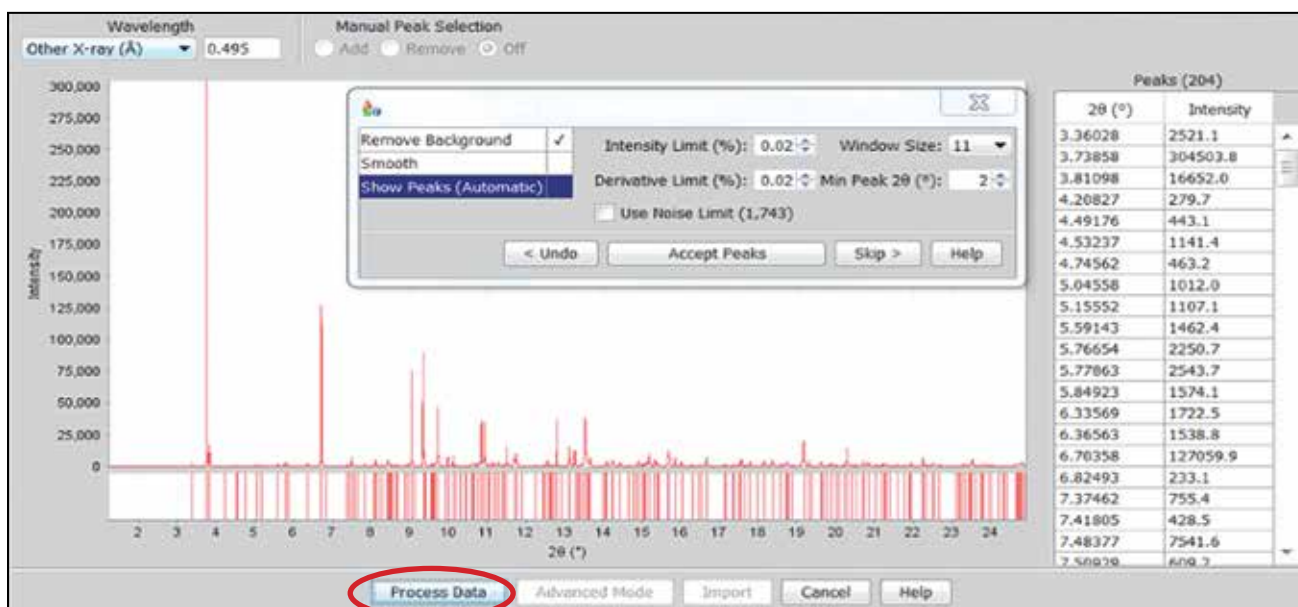
## III. Data Input and Processing



Data files with several common format types can be automatically read into PDF-4+ and Sieve+. With most synchrotron data sets the user can immediately notice the large range in intensity and high resolution of peaks with very narrow peak profiles. When importing data, the user should specify “other X-ray” in the top left entry box, titled “Wavelength”, along with the specific wavelength used in the analysis. The software recognizes this as synchrotron data, assumes the data are from monochromatic radiation ( $\alpha 1$ ), and then inputs this information into Sieve+.



This higher quality data requires special considerations during pattern processing. The user needs to carefully examine the experimental noise, establish peak detection limits, and then use more sensitive parameters for peak finding. During import, the user must remember to specify the wavelength in the import screen prior to phase identification.

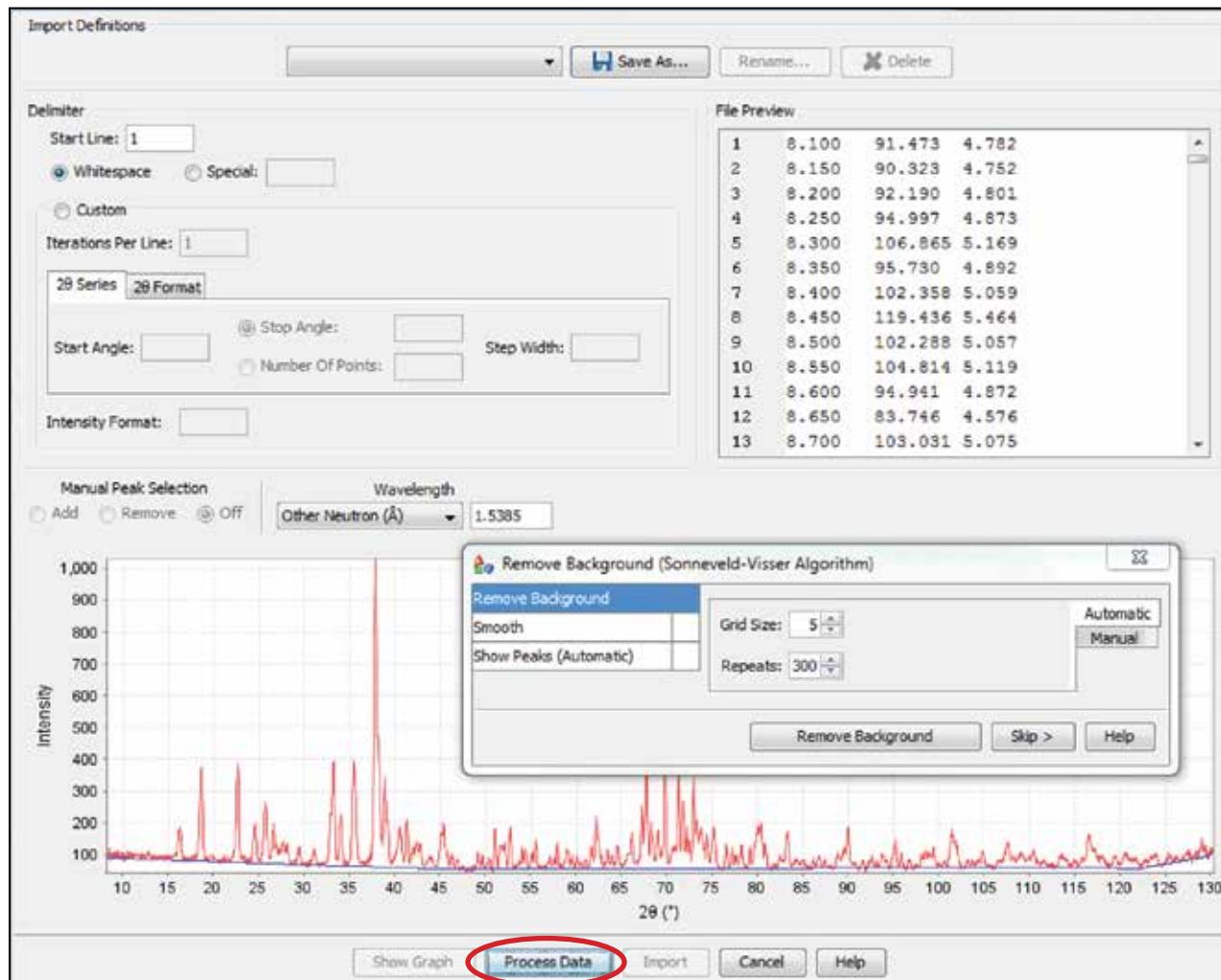




### III. Data Input and Processing



Data files with several common format types can be automatically read into PDF-4+ and Sieve+. For constant wavelength neutron diffractometers, any ASCII file can be read using the custom diffraction pattern importer (shown below). Prior to data input, time-of-flight (TOF) data needs to be converted into d-spacings, and then into  $2\theta$  based data before importing. In the near future, you should be able to import d-spacing data directly.



Throughout the world, neutron diffractometers are significantly different in design and optical configuration. Lower resolution instruments have broad peak profiles and care needs to be taken in the peak identification steps to ensure that all reasonable peaks and peak shoulders are identified.

The data input software helps by providing a calculation of the average noise level, which should be used when considering the significance of a weak peak or shoulder.



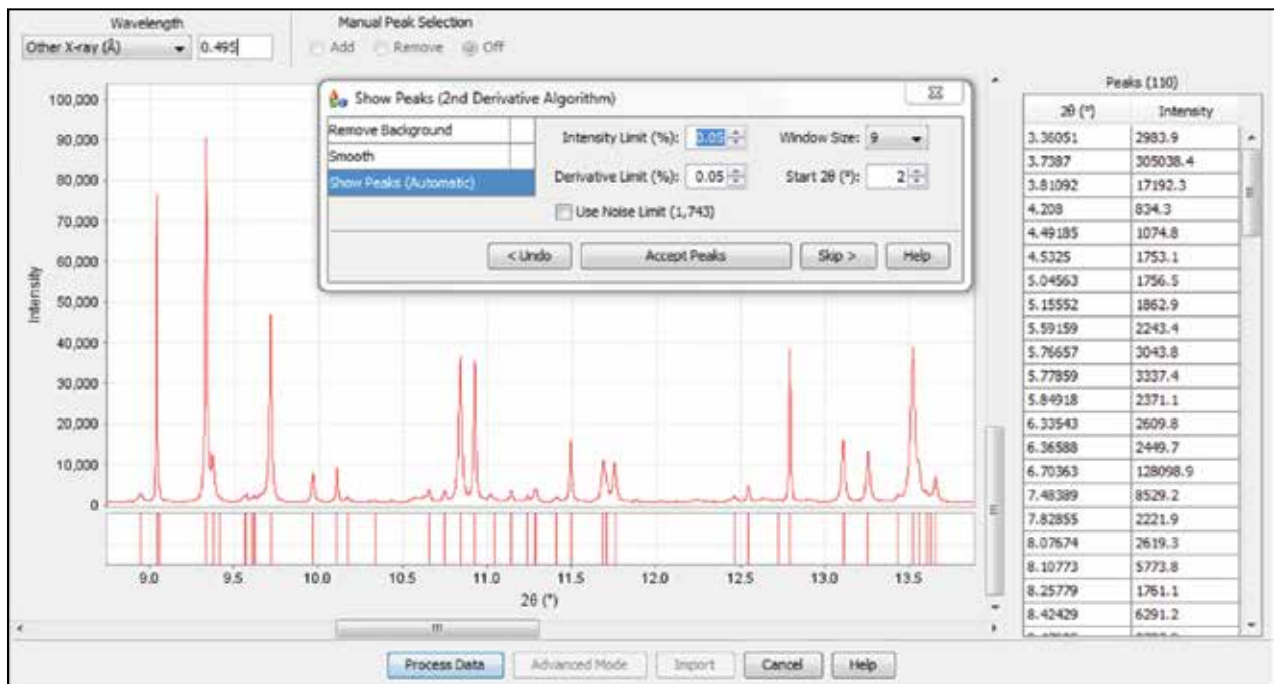
> File

> Import Diffraction Pattern

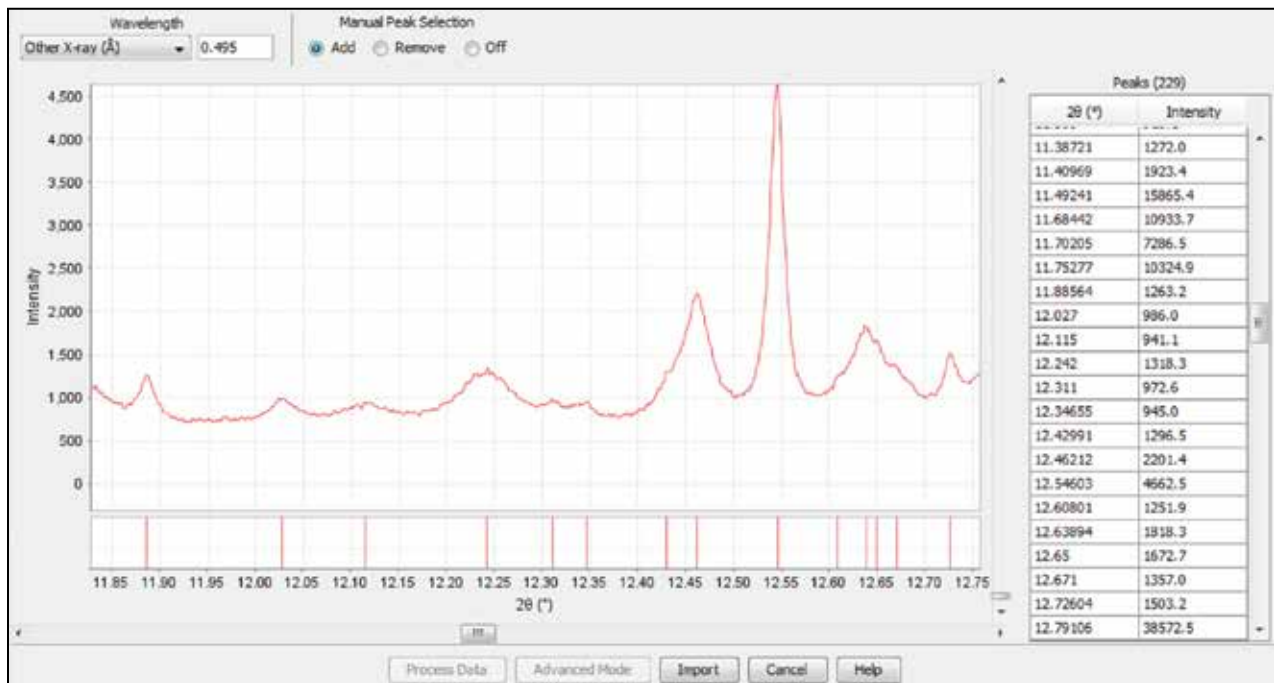
# Synchrotron Diffraction

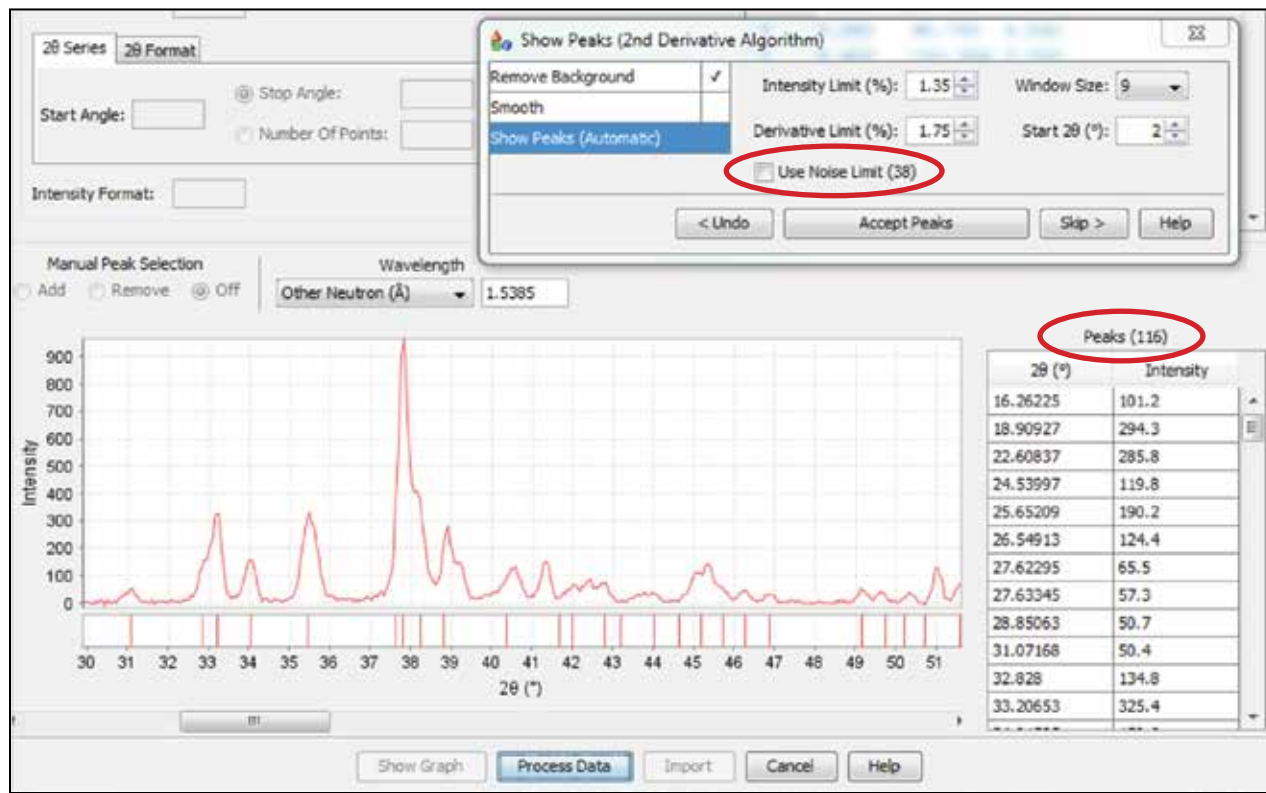
## III. Data Input and Processing (cont.)

Peak files of hundreds of d-spacings covering a large range of intensities are not uncommon in the processing of synchrotron data sets. The quality of the final results is often reflected in the care taken by the user in the peak finding step. At any point in the analysis, the user can zoom in the plot using the mouse and expand the xy scale. This may be necessary to detect very small peaks present in the pattern.



The figure above shows a zoomed in 2θ range making minor peaks visible for matching. Using manual peak selection, additional peaks were added as shown below.



III. Data Input and Processing (*cont.*)

In the example above, the software calculated a noise level of 38 counts and 116 peaks were detected in the pattern. This is the same pattern shown in the previous two figures, but the latter figure has zoomed in the plot. Close examination using the zoomed in plot shows several shoulders and weak intensity peaks. Some of these were missed by the peak finding algorithm. There is a manual peak selection option that can add or subtract peaks. Once the optimum conditions for peak selection are chosen, the program saves the settings for the next analysis.

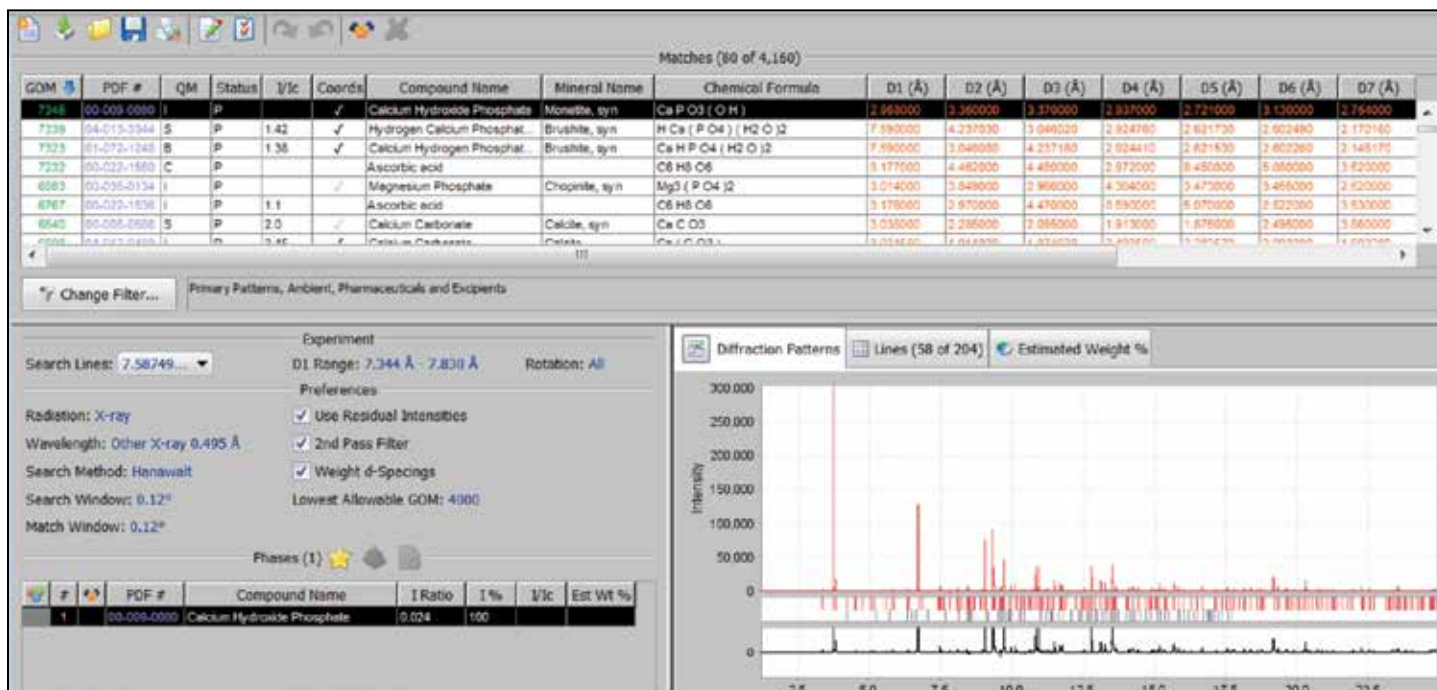
Neutron diffraction patterns generally have a lower intensity than synchrotron diffraction patterns due to the lower overall flux of neutron sources. However, the fact that atomic scattering factors for neutrons do not significantly decrease with diffraction angle means that a higher  $2\theta$  range can be used in the analysis providing important information for either a phase identification, structural determination, or microstructural analysis.

After the data are processed, sets of d-spacing and intensity pairs are generated, as shown on the right in the figure above. This information, as well as the diffraction pattern, is input into the phase identification process.

## IV. Phase Identification



Phase identification analysis is carried out through PDF-4+'s plug-in search-indexing software, Sieve+. After the data are imported, a d,I listing is generated and both the d,I listing and full digital pattern are input into the diagnostic software of Sieve+; both are utilized by the phase identification algorithms. These algorithms and filtering choices are described in full detail in the ICDD Sieve/Sieve+ Technical Bulletin.



The three-panel diagnostic screen for Sieve+ is shown above. The top panel lists candidate phases in order of phase match by GOM (Goodness of Match). The GOM is a statistical comparison of the d-spacings in the experimental pattern compared to the strongest d-spacing reference peaks. If a peak is matched, it is highlighted in red. In the screen above, nearly all the peaks match for most of the top candidates. For this particular specimen, five of the identified six phases are contained in the material. One phase is a duplicate.

The bottom right panel shows the experimental data, the location of the reference peaks, the selected candidate phase, and a difference plot. The bottom left panel specifies experimental conditions and highlights a selected candidate phase.

In practice, the GOM identifies the best statistical match by d-spacing comparison between the experiment and reference. This should always be verified by a visual comparison of the data using the graphics panel on the bottom right. In this manner, several phases can be readily identified and confirmed by visual analysis of the intensities.

The selection of an appropriate synchrotron profile function helps with the pattern simulation (Chapter II) enabling a better visual comparison and lower residuals between the experimental data and identified phases. This can be very important in the identification of minor and trace phases.

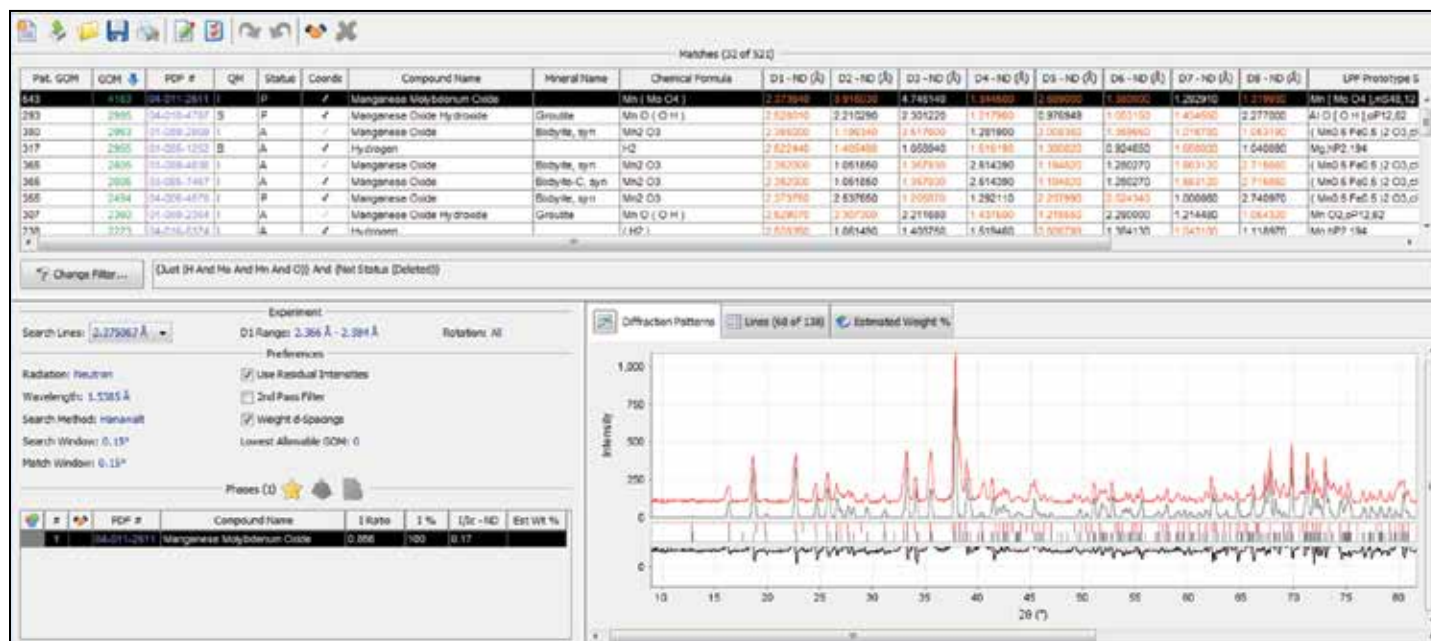


## IV. Phase Identification



Phase identification analysis is carried out through PDF-4+’s plug-in search-indexing software, Sieve+. After the data are imported, a d,I listing is generated and both the d,I listing and full digital pattern are input into the diagnostic software of Sieve+; both are utilized by the phase identification algorithms. These algorithms and filtering choices are described in full detail in the ICDD Sieve/Sieve+ Technical Bulletin.

In the example below, the numerous broad overlapping peaks do not result in a meaningful identification if all the data in the database are searched using the standard GOM as the identification algorithm.\* We used a chemistry added search of “Just H, O, Mo, Mn” to limit the search to 521 phases of the appropriate chemistry, 32 of which matched peaks found in the pattern.



It is interesting to note the comparison with the synchrotron data on the left page. The highest GOM for the synchrotron data was a score of 7,345 out of 8,000, while the highest score in the neutron analysis was 4,163 out of 8,000.

The top GOM score was obtained by matching the data to the reference of Mn (MoO<sub>4</sub>) and the reference pattern is compared to the experimental data in the bottom right diagnostic panel. Two other matching algorithms, the pattern GOM and Normalized R-Index, gave the identical reference as the top score. Finally, the visual comparison shows that the intensity matches are very close so that this phase can be positively identified with confidence.

*\*It should be noted that in the original analysis, without chemistry restrictions, a pattern scan of all primary, ambient patterns (144,653 references) does find Mn (MoO<sub>4</sub>) as the top candidate phase by either the pattern GOM or Normalized R-index algorithm. Both the GOM or Normalized R-index algorithm weigh intensity contributions more than the standard GOM and may be more effective algorithms when there are many overlapping peaks.*

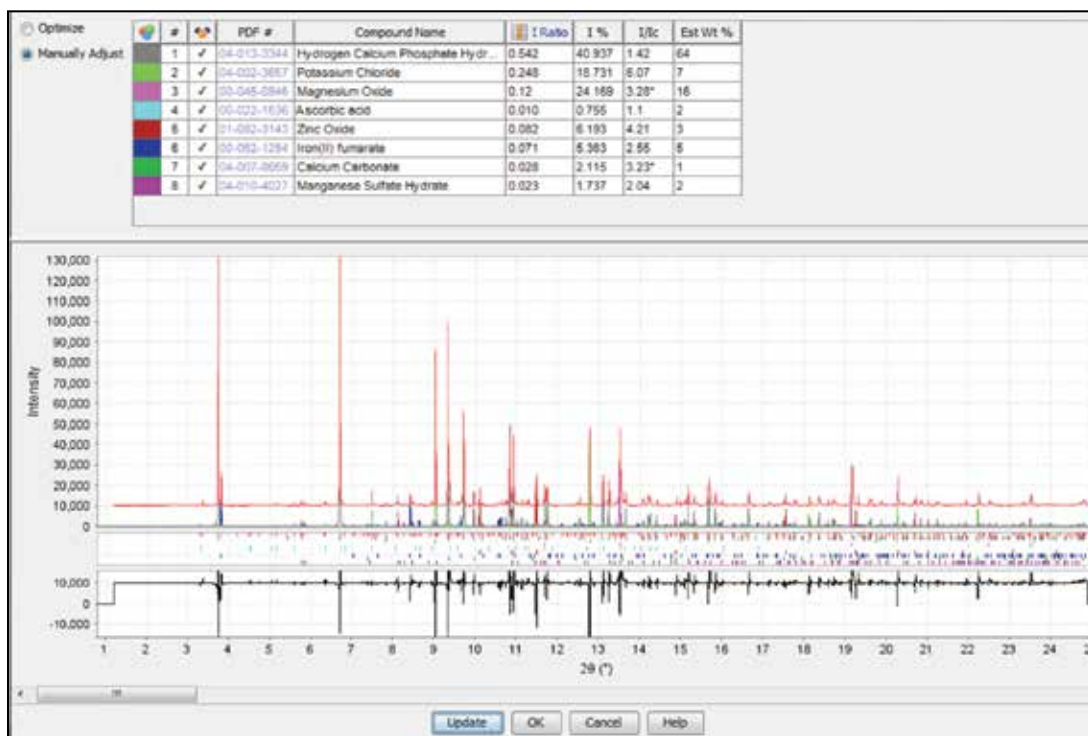


# Synchrotron Diffraction

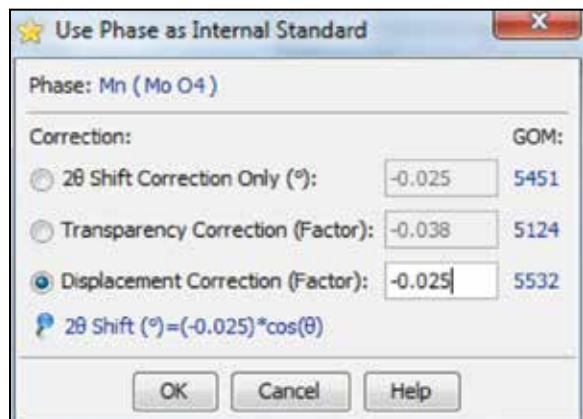
## IV. Phase Identification (cont.)



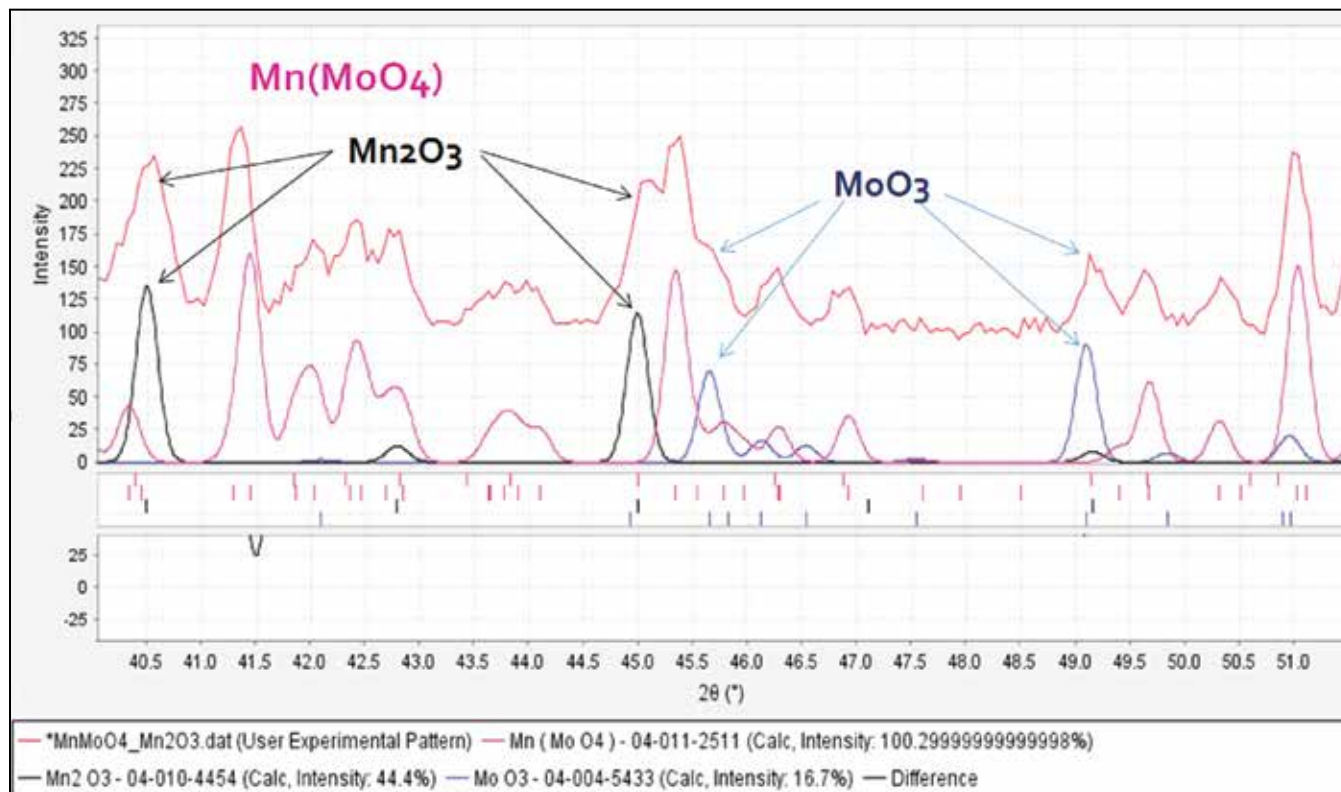
The analysis finds eight phases, all known ingredients of this vitamin pill. The eight phases account for 191 out of the 225 peaks originally identified in the experiment and all peaks above a relative intensity of one on a scale of 1-100. This would indicate that all major phases are identified, but there may be a few unaccounted for minor phases. The largest GOM candidate phase is from brushite, a phase already identified in the material. The difference pattern in the bottom right panel shows that there are large residuals from brushite. The duplicate identification results from these residuals. These are adjusted below by selecting the button at the end of the identification in the lower right panel.



## IV. Phase Identification (cont.)



We can see if there are systematic errors in the experimental data by clicking on the gold star in the bottom left diagnostic panel. This allows an identified phase to be used as an internal standard and calculates three common corrections for the experimental data. This analysis would indicate that a displacement correction ( $-0.25$  degrees  $2\theta$ ) would be appropriate and raise the GOM to 5532.

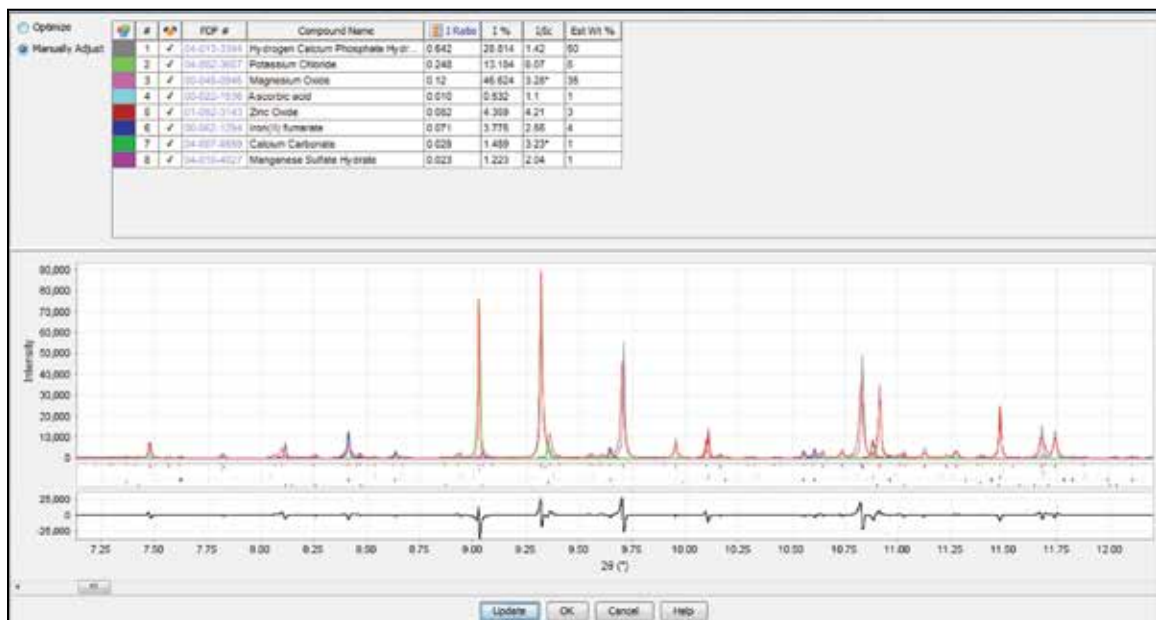


Reiteration of the phase identification process after identification of Mn ( $\text{MoO}_4$ ) identifies two additional phases,  $\text{Mn}_2\text{O}_3$  and  $\text{MoO}_3$ . An expanded view of the pattern showing these additional phases is shown above.

## V. Quantitative Analysis (RIR)

PDF-4+, in combination with the plug-in search-indexing software, Sieve+, can perform a semi-quantitative analysis using the Reference Intensity Ratio (RIR) method. The RIR method scales the intensity of all reference data to a common standard of corundum ( $I/I_c$ ).

The RIR solution and pattern fit can be visually observed using the “Adjust Phase Intensity Ratios” icon on the bottom left diagnostic panel. An adjustment can be made by a least squares method or by manual correction. This helps in cases where there is preferred orientation and the entire pattern intensity can be used in the adjustment.



The expanded view above gives the results of the adjusted RIR calculation and shows how the individual phases contribute to the experimental pattern.









This same specimen has been analyzed by other authors and published in Volume H of the International Tables of Crystallography (Gilmore et. al. **2014**). Additional phases of nicotinamide, cellulose I $\beta$  and monetite were identified by using a trace analysis technique, (Fawcett et. al., **2011**) analyzing the peaks below 1% intensity in the pattern.

## V. Quantitative Analysis (RIR)

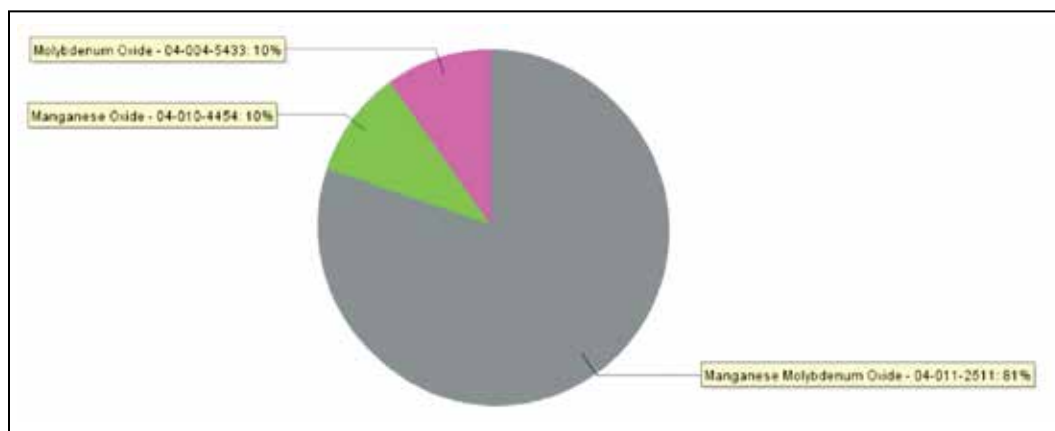
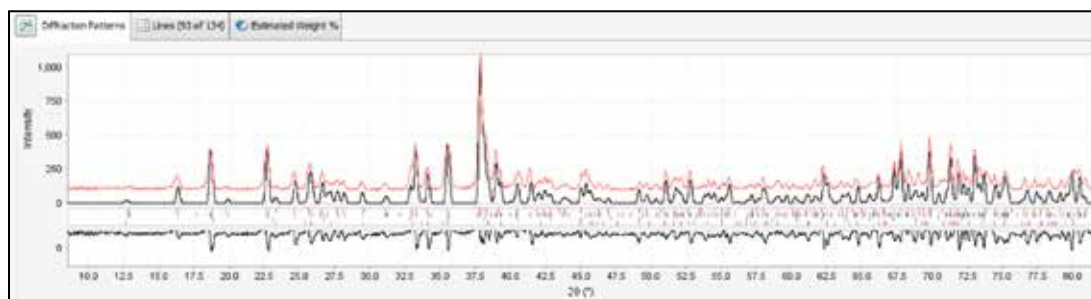
PDF-4+, in combination with the plug-in search-indexing software, Sieve+, can perform a semi-quantitative analysis using the Reference Intensity Ratio (RIR) method. The RIR method scales the intensity of all reference data to a common standard of corundum ( $I/I_c$ ).

### Key Fact

*In PDF-4+, all entries with neutron diffraction data (~ 240,000) have had  $I/I_c$  values specifically calculated from neutron scattering factors.*

Phases (3)   								
	#		PDF #	Compound Name	I Ratio	I %	I/Ic - ND	Est Wt %
	1	✓	04-011-2511	Manganese Molybdenum Oxide	1.003	62.144	0.17	81
	2	✓	04-010-4454	Manganese Oxide	0.444	27.509	0.62*	10
	3	✓	04-004-5433	Molybdenum Oxide	0.167	10.347	0.24	10

In the example used in this guide, the following data appear in the bottom left diagnostic panel. It includes the intensity of the phase, % of total pattern intensity, and  $I/I_c$  value calculated for neutron diffraction (ND). On the far right is the RIR calculation for weight % of each phase.



The figure above (left) shows the total pattern simulation using all three phases and the scaled weight percents (black) versus the experimental data (red), and a difference plot. This is not a refinement. It is a simulation and the data should be considered semi-quantitative. The calculation also used the intelligent RIR method developed by the ICDD, which replaced the  $I/I_c$  value of manganese oxide  $Mn_2O_3$  with one from a higher quality reference of the same material.



## VI. Quantitative Analysis (Rietveld)

PDF-4+ 2014 includes 239,568 entries with atomic coordinates that are displayed in the “Structure” tab.

### Key Fact

*This collection is the world's largest and most diverse for inorganic materials because these entries come from numerous data sources. It contains the world's largest collections of ceramics, metals and alloys, minerals, and even modulated structures described in super spacegroups of 4 to 6 dimensions. These data have been edited, reviewed and corrected whenever possible. Our quality review system is based on having Rietveld analysis as an objective, which means we do not publish crystallographic data with major problems, such as significant amounts of missing electron density and/or severe disorders. All entries are given Quality Marks.*

PDF-4/Organics 2015 has a collection of 66,006 atomic coordinate sets, which is only a fraction of the size of the available atomic coordinate data sets within the Cambridge Structural Database (CSD). However, the collection within PDF-4/Organics is heavily targeted and focused on commercial materials, including the world's largest collection of pharmaceuticals and excipients. Through our Grant-in-Aid program, ICDD actively collects data that are unique to the PDF databases.

The PDF-4 database family offers users the ability to achieve superior Rietveld analyses versus any other crystallographic database in the world. International round robins coordinated through the IUCr and ICDD have shown that “user errors” are the most frequent cause of poor quantitative results by the Rietveld method. (Madsen et. al. **2001**, Scarlett et. al. **2002** and Fawcett et. al., **2012**). The Quality Mark and quality review system embedded within the ICDD databases provide users with quality assessments that they can use to avoid common errors.

For example, common user errors include have selecting the wrong polymorph and selecting non-ambient structures as starting materials for Rietveld refinements. The latter is particularly easy because of the large numbers of organic and bio materials that have been analyzed by single crystal methods at low temperatures. The ICDD uses standardized nomenclature and international naming conventions (IMA, IZA, IUPAC), trade names, empirical and chemical formula, all available to help the user avoid selecting the wrong entry. Each entry has a temperature of data collection. There are search filters for temperature (ambient, non-ambient) so that all non-ambient materials can be eliminated from the selection process in the analysis of a room-temperature diffraction pattern. Alternatively, entries can be filtered within a specific temperature range for non-ambient experiments.

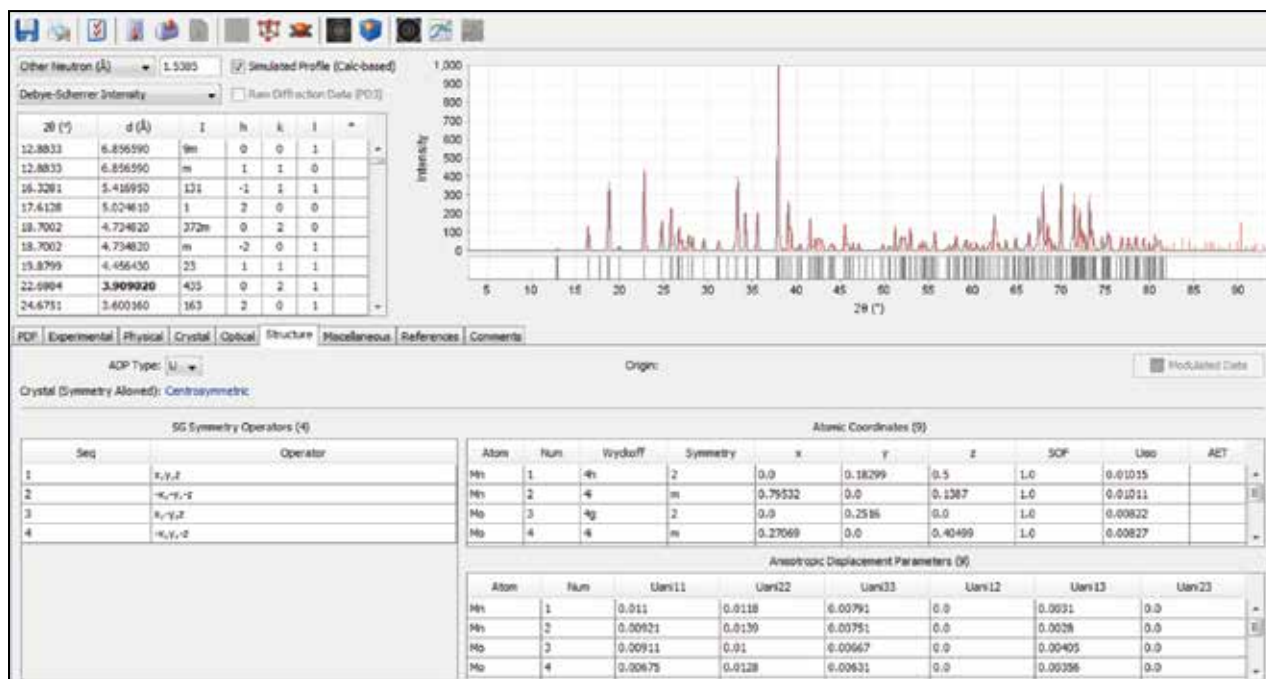
Phase identification, preceeding Rietveld refinement, would also be likely to identify very common orientation and granularity problems with the dataset. Orientation problems can be analyzed by applying a March-Dollase orientation function to the pattern simulations.



## VI. Quantitative Analysis (Rietveld)

The majority of references (209,587 out of 241,169) that have calculated neutron diffraction patterns also contain atomic coordinates required for Rietveld analyses. The remaining entries either have neutron scattering factors or are experimentally determined neutron diffraction patterns without structures.

In the example below, all three identified phases have atomic coordinates and cell parameters required for Rietveld refinement. These can be viewed from the “Structure” page of each individual entry as shown below.



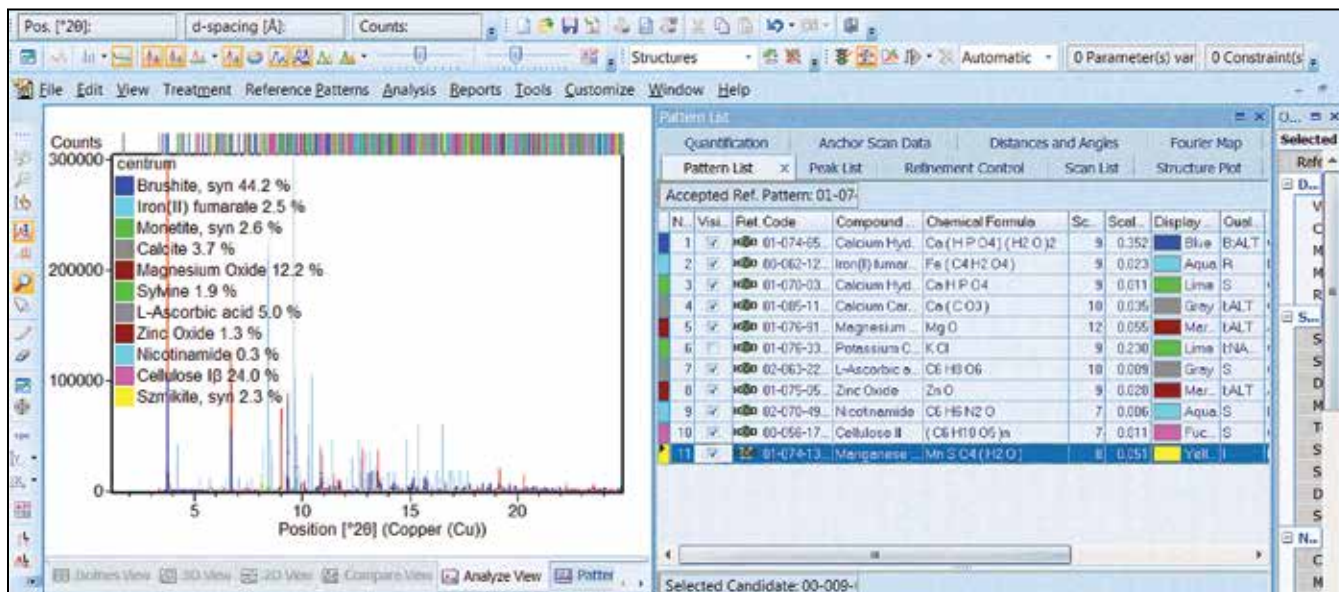
This entry also shows the space group symmetry operators, anisotropic displacement parameters, and both the indexed d-spacing list and calculated neutron diffraction pattern.

From the Simulated Pattern icon shown in the tool bar, the pattern and relevant crystallographic data can be exported from the card entries in any of the following formats (.cif, .gsas, .mdi, .xrdml, .udf, .ard, .uxd, .cpi, .xrd, .csv, .prn, .xye, as well as non-standard experimental file formats). These can be used as input into a Rietveld refinement. A Bruker TOPAS file \*.str can also be saved from the card entry under “File” shown above.

### Key Fact

*The PDF-4 database family offers users the ability to achieve superior Rietveld analyses versus any other crystallographic database in the world. The quality systems embedded in the database provides the user with information they can use to avoid well-documented common mistakes in quantitative analysis.*

## VI. Quantitative Analysis (Rietveld) (cont.)



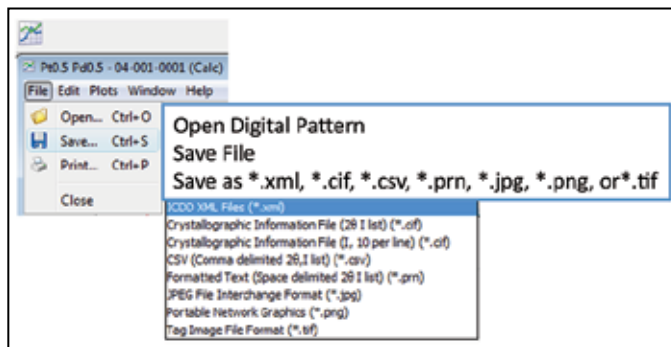
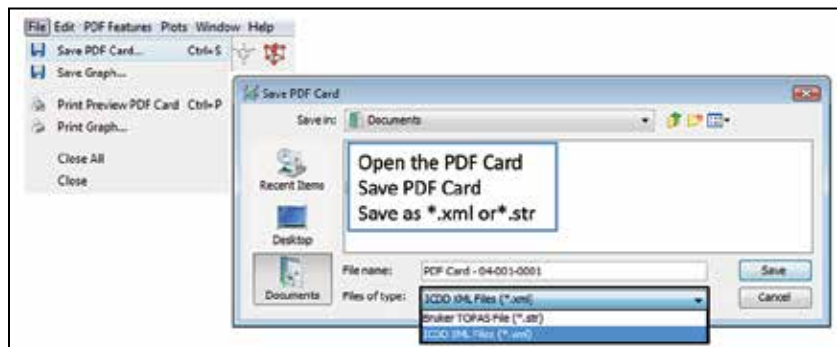
The screen capture above is taken from a commercial fully-integrated diffraction analysis software package, HighScore Plus (v.4.0), sold by PANalytical. This program performs phase identification and subsequent Rietveld analysis using interfaces to the PDF-4 database. This particular analysis used a PDF-4/Organics database and identified 11 phases from 0.3% to 44.2% concentration using synchrotron data. The advantage of using the PDF-4/Organics database is that it includes the crystal structures of vitamins B and C, as well as all the inorganic excipients that were added to this database and the cellulose structure from the ICDD. Every phase and every peak has been identified and quantified by Rietveld refinement. The advantage of using a multi-sourced database (PDF-4/Organics) is clearly seen in this 11-phase analysis as 2 phases are sourced from the ICDD (source 00), two from the CSD (source 02) and six from the ICSD (source 01). The low concentration phase, nicotinamide, was identified via Rietveld residual analyses using the methods described by Fawcett et. al. (2011).

PDF-4 software does not include a Rietveld refinement program. The commonly available freeware programs, GSAS and FullProf, are able to refine synchrotron data using \*.cif files as input. The .cif files can be output from PDF-4+.

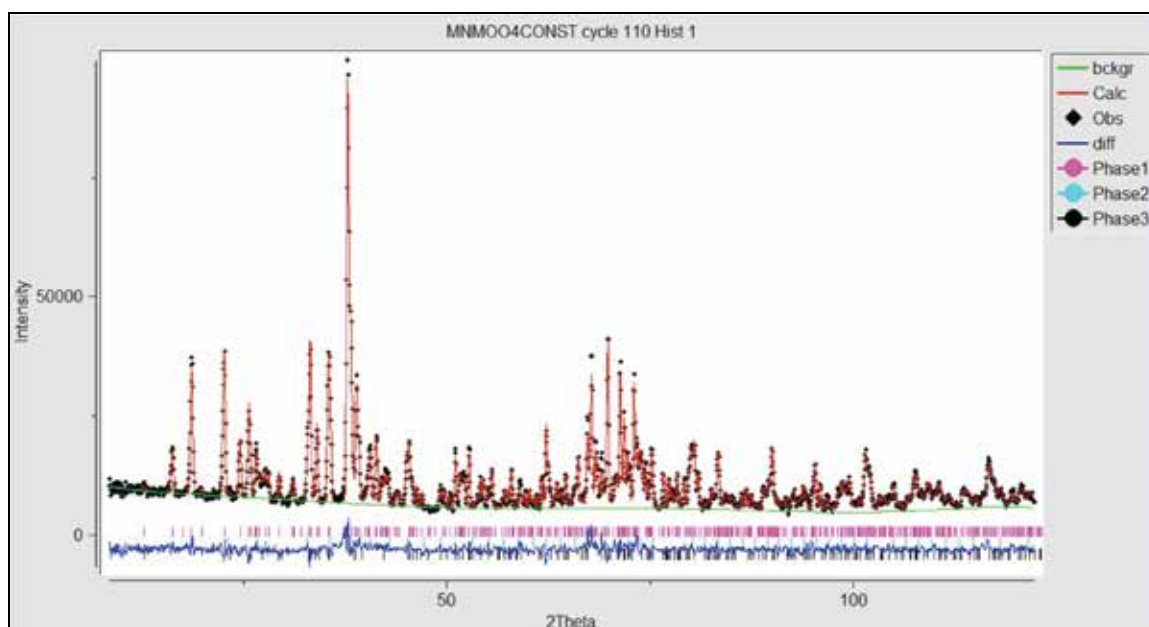
Most commercial software packages contain a Rietveld refinement option that can be used with synchrotron experimental data. You would need to consult your software supplier to see if they can accommodate synchrotron diffraction data. Large volume commercial software packages such as Jade, PDXL, and HighScore Plus have a smooth transition between the phase identification step and Rietveld refinement; import and export steps are not required. TOPAS users can use a \*.str file export from the ICDD PDF entry. If manual entry is required, most of the software systems can input standard .cif files, which are easily exported from the appropriate ICDD entry.

VI. Quantitative Analysis (Rietveld) (*cont.*)

## Two methods to save data files.



PDF-4 software does not include a Rietveld refinement program. The commonly available freeware programs, GSAS and FullProf, are able to refine neutron data using \*.cif files as input. Many commercial software packages contain a Rietveld refinement option. You would need to consult your software supplier to see if they can accommodate neutron diffraction data.

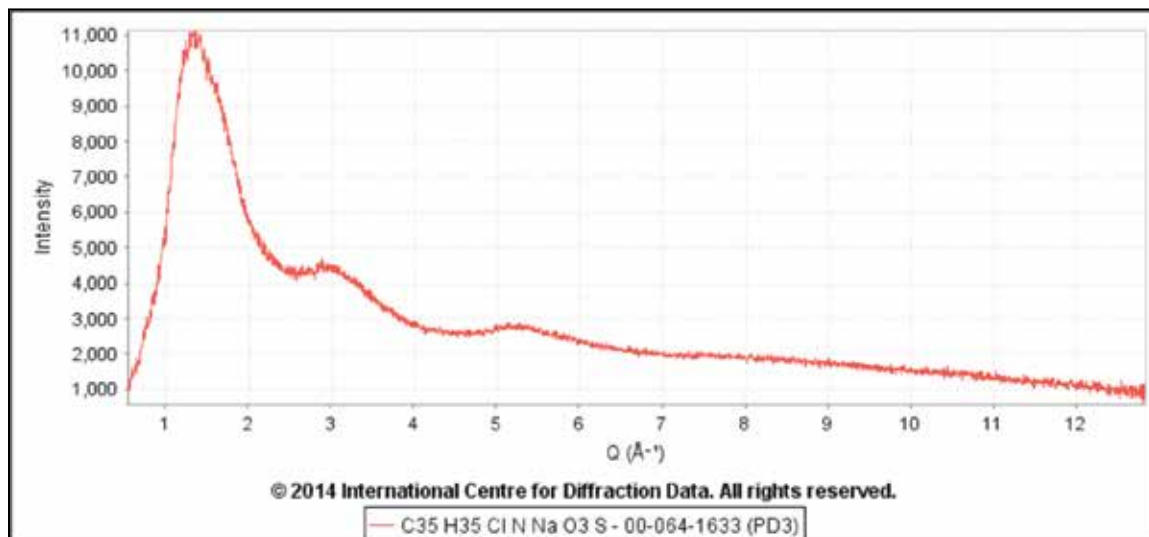


The data above are from a GSAS Rietveld refinement of the experimental neutron data. Atomic parameters used for the refinement were taken from the PDF entries identified in the search/match solution and exported from PDF-4+ into GSAS. Residuals are now minimized since the data are refined, not simulated. The quantitative phase analysis was  $\text{MnMoO}_4$  – 75.1%,  $\text{Mn}_2\text{O}_3$  – 20.2% and  $\text{MoO}_3$  – 4.7%.

Most commercial software packages contain a Rietveld refinement option that can be used with synchrotron experimental data. You would need to consult your software supplier to see if they can accommodate synchrotron diffraction data. Large volume commercial software packages such as Jade, PDXL, and HighScore Plus have a smooth transition between the phase identification step and Rietveld refinement; import and export steps are not required. TOPAS users can use a \*.str file export from the ICDD PDF entry. If manual entry is required, most of the software systems can input standard .cif files, which are easily exported from the appropriate ICDD entry.

## VII. Nanomaterials, Amorphous Materials and Non-crystalline Materials

The advantages of using synchrotron radiation for the analysis of crystalline materials relates strongly to the high resolution and high intensity flux available at most global synchrotron diffraction facilities. For the analysis of non-crystalline materials or nanomaterials, the resolution is dominated by the microstructural material characteristics, not the instrument resolution. The high flux does offer users the opportunity to collect precise data with exceptional signal-to-noise from incoherent scatter and scatter from nanomaterials having small crystallite sizes.



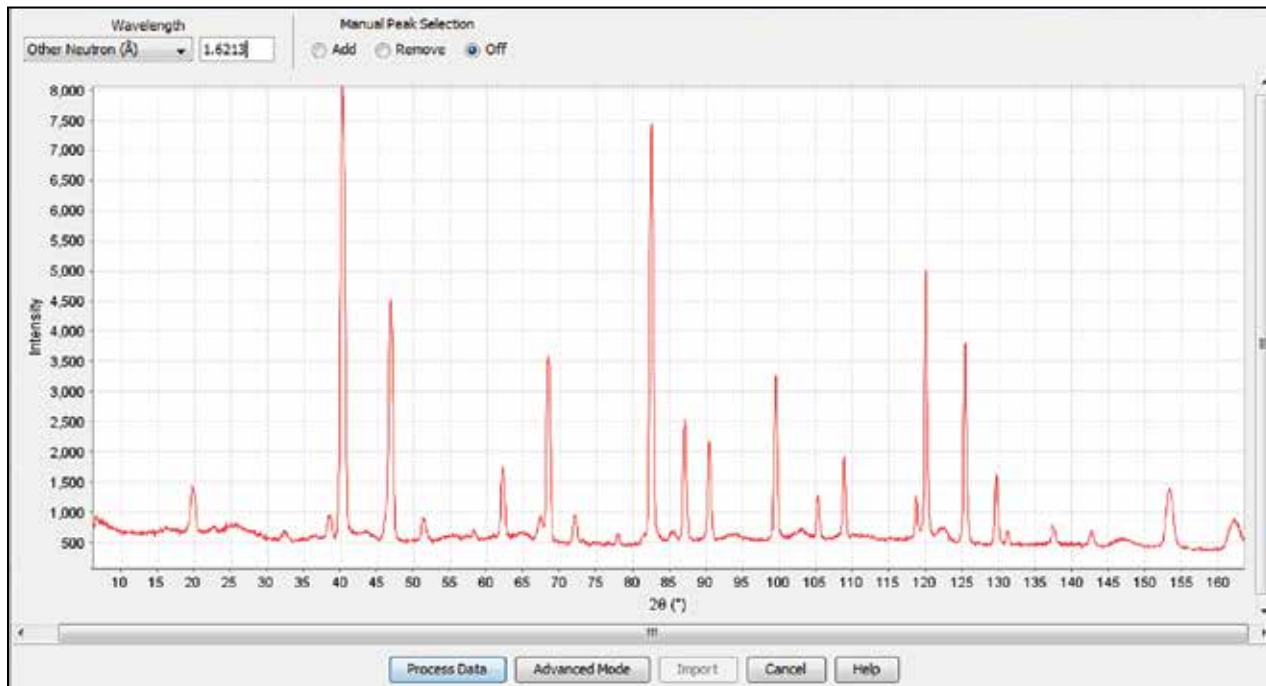
The reference above is one of several high sales volume amorphous pharmaceuticals that have been analyzed at the Advanced Photon Source at Argonne National Laboratory (Kaduk et. al., 2014). It is the pattern of sodium montelukast purchased from Key Organics LTD. Pharmaceuticals are frequently prepared in an amorphous state to improve drug solubility. The pattern is shown with  $Q$  plotted on the X-axis. Other X-axis options include  $2\theta$ ,  $d$  and  $1/d$ . Diffraction patterns expressed in  $Q$ ,  $d$  and  $1/d$  offer the ability to compare synchrotron data with data collected using different wavelengths.

The Powder Diffraction File has a collection of reference amorphous materials, nanomaterials, and mixed crystallinity references where the digital patterns can be used in whole pattern analysis methods for identification or quantitative analysis. These entries are primarily concentrated in clays, polymers, pharmaceuticals, and nano materials.

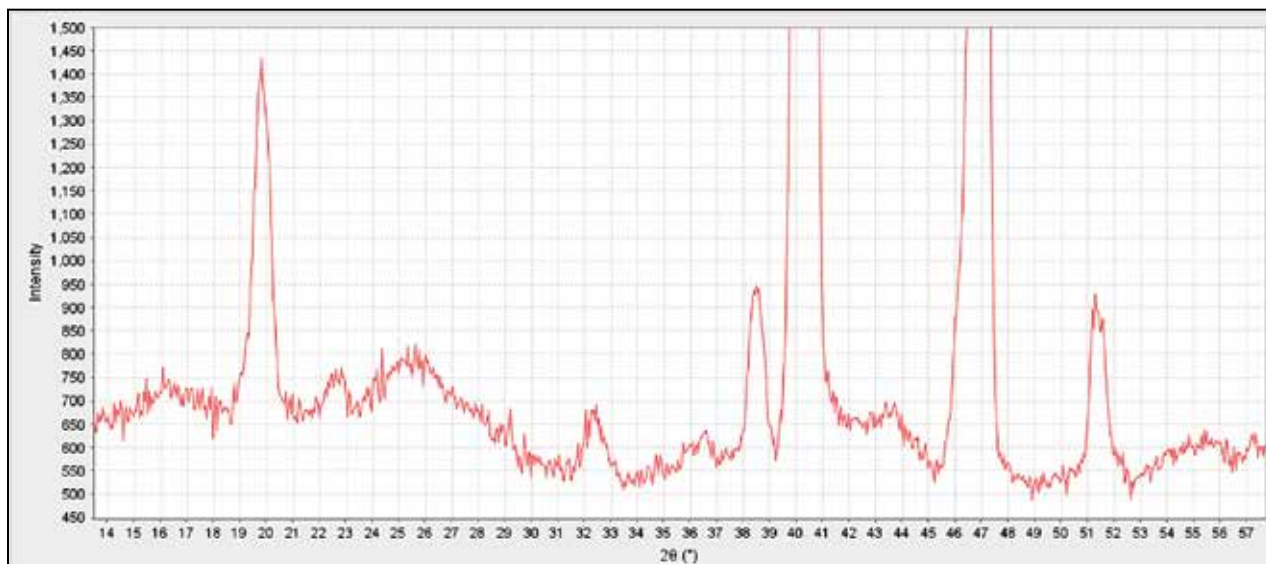
Recent publications (Fawcett et. al., **2013**) have shown how synchrotron patterns of amorphous and microcrystalline cellulose can be analyzed by their characteristic patterns. The synchrotron data are also useful for further characterization by the pair distribution function method. Pair distribution data have been used to confirm the nanocrystalline or amorphous classification in several reference materials.



## VII. Nanomaterials, Amorphous Materials and Non-crystalline Materials

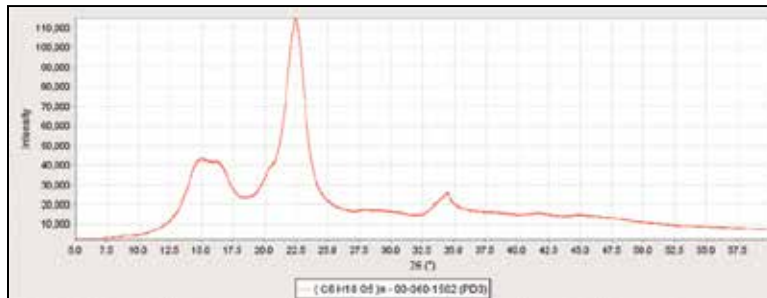
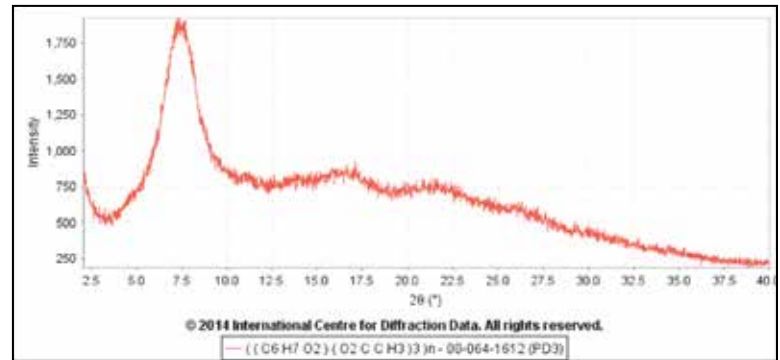
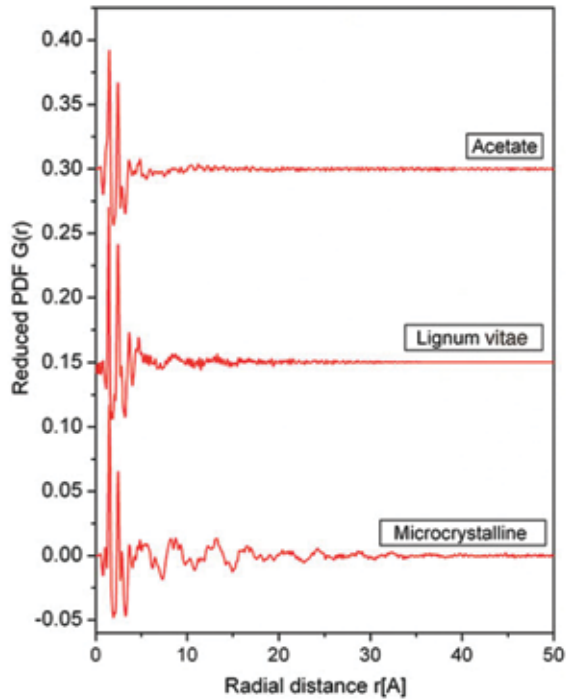


The above data were taken on the Echidna neutron diffractometer at the Bragg Institute in Sydney, Australia (Peng et.al., **2014**). The specimen is from a fuel cell cathode. Close inspection of the pattern shows some phases to be highly crystalline (sharp peak profiles), while other phase are of smaller crystallite size (broad profiles).





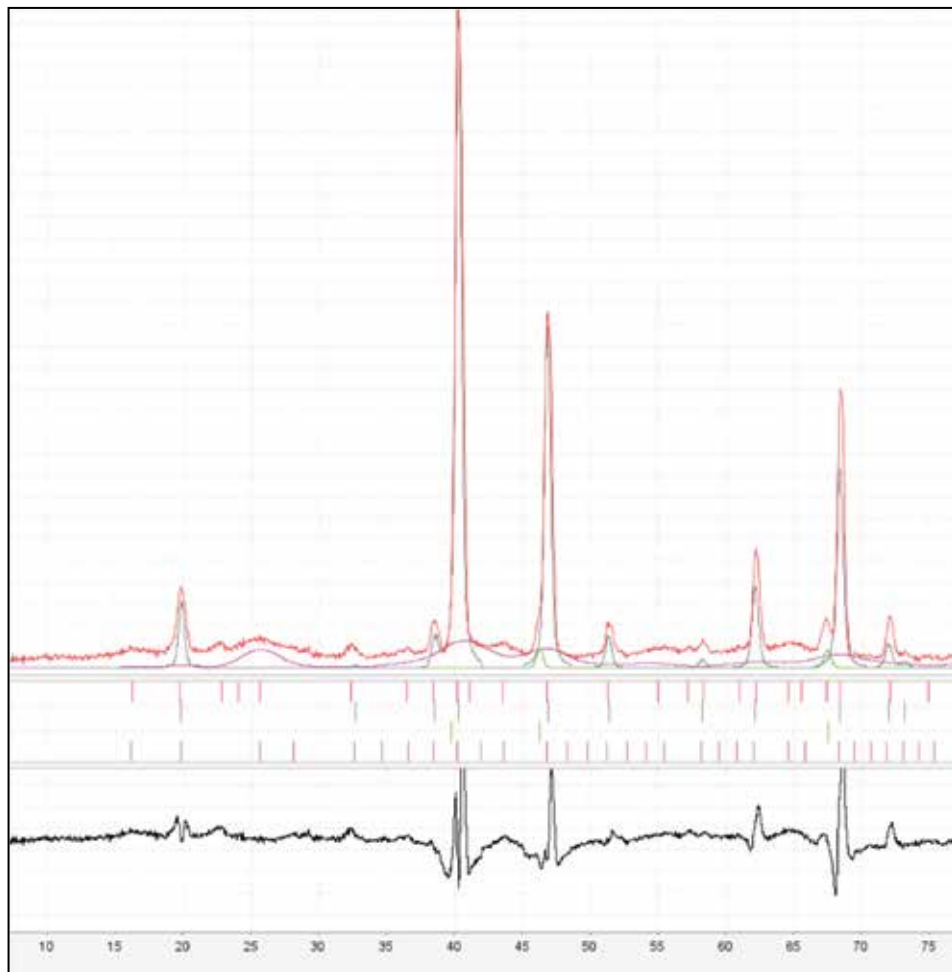
## VII. Nanomaterials, Amorphous Materials and Non-crystalline Materials (*cont.*)



The pair distribution function data above were used to confirm the designation of commercial microcrystalline cellulose as a nanomaterial cellulose I $\beta$  (bottom right, PDF 00-060-1502) and cellulose triacetate as amorphous (bottom right, PDF 00-064-1612). The microcrystalline cellulose exhibits long range order in the nanoscale; the cellulose acetate does not exhibit any long range order.

VII. Nanomaterials, Amorphous Materials and Non-crystalline Materials (*cont.*)

&gt; Crystallite Size Option

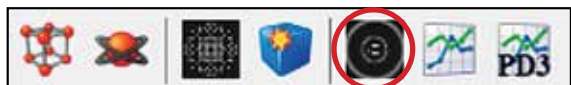


In this case, we can apply the crystallite size option available in the reference pattern simulation to account for most, but not all, of the broad features in the pattern. For this simulation, 150 Å and 180 Å were used for the two crystalline phases, PDF 01-081-9146 (Lithium manganese nickel oxide) and PDF 04-006-3534 (Lithium nickel oxide). A 30 Å crystallite size was used for the broad features, PDF 04-016-1663, a second crystallographic phase of lithium manganese nickel oxide. This last phase was also simulated with an orientation in the (210) plane by an optional March-Dollase orientation function. PDF 04-016-1663 does account for the broad peak locations (purple tick marks), but various orientation functions are required to simulate the proper intensity. The fact that we can approximate, but not exactly reproduce the intensity distribution means that there are probably other line broadening effect contributions (dislocations, vacancies) that are not accounted for by the crystallite size simulation (Scardi et. al, **2006**) and discussed by the authors (Peng et.al., **2014**).

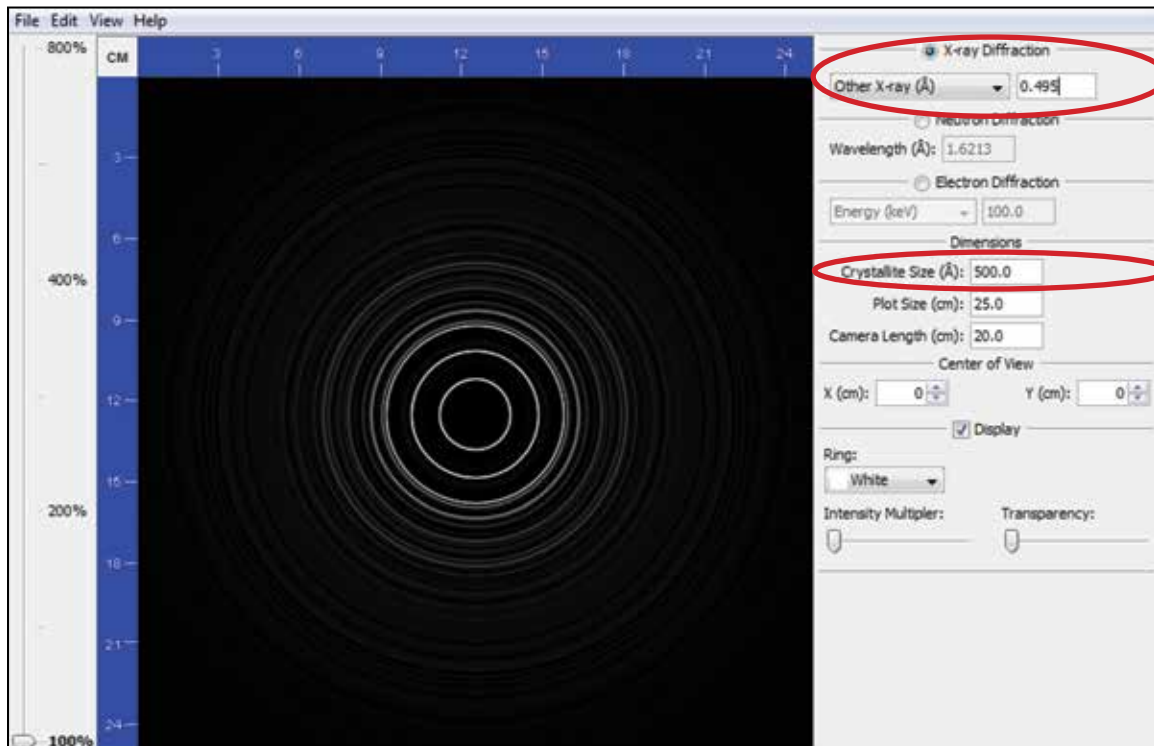
The orientation function and crystallite size simulation tools can be used to convert any of the ~240,000 neutron diffraction patterns into a nanomaterial simulation useful for microstructural analysis. As shown in Section II, the database can be converted as an entity or alternatively, each phase can be treated using a different simulation, as in the case above – various crystallite sizes.

# Synchrotron Diffraction

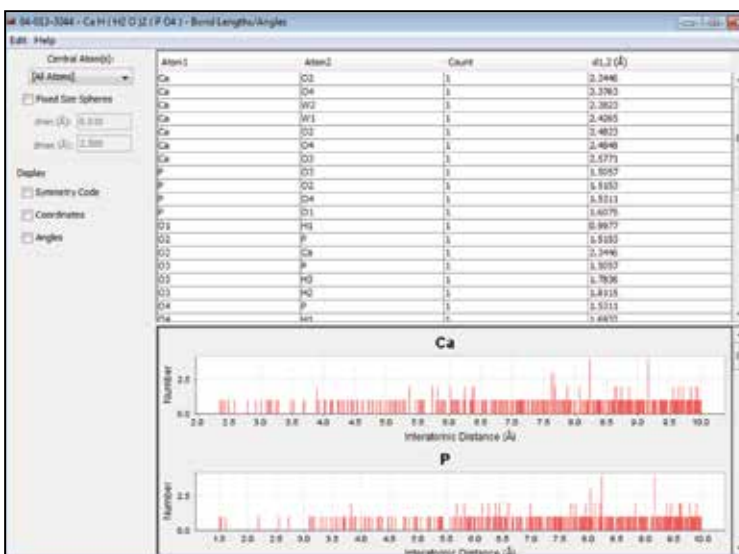
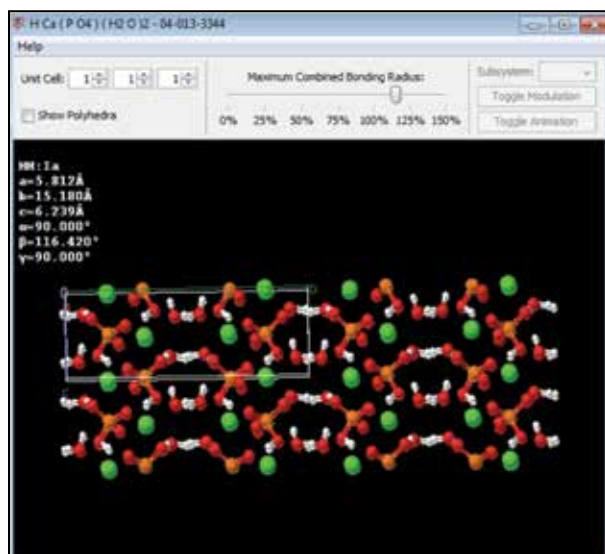
## VIII. Options



Options are accessed through the toolbar, shown above, at the top of each PDF entry display.



If you use a 2D detector, you can display synchrotron data in a ring pattern simulation. The user can adjust the focal length and crystallite size, and then import an image file for overlay. The wavelength selection for synchrotrons and crystallite size options are highlighted.

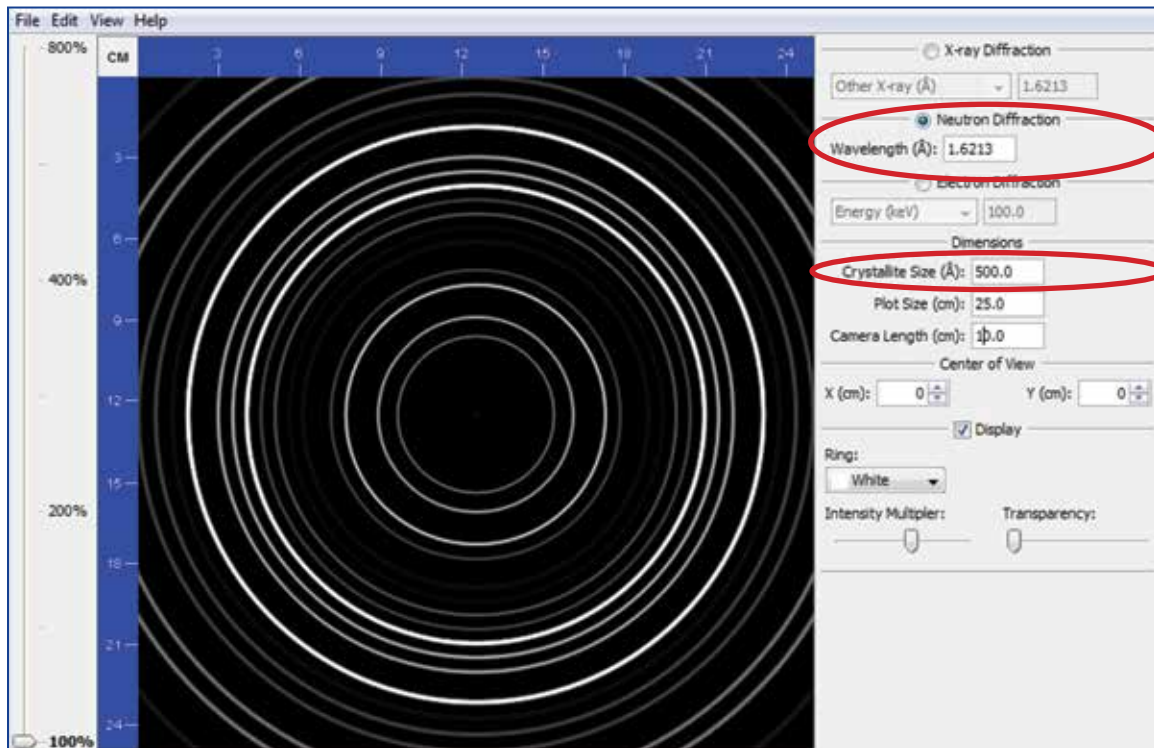


Many entries in the PDF-4 are based on single crystal determinations. For all entries with atomic coordinates (239,568 entries), the user has a choice of two different molecular graphics packages for display (Jmol shown), as well as bond length, angle, and nearest neighbor atom calculators. Data shown are for the mineral, brushite.

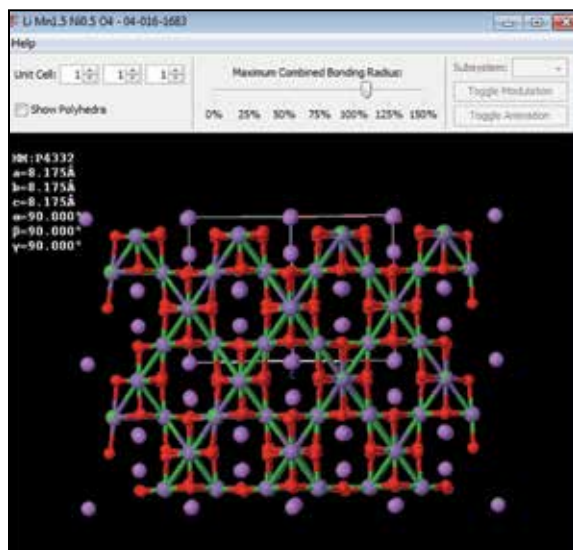
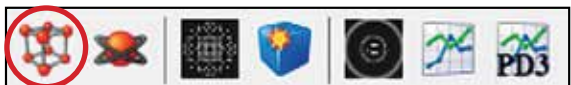
## VIII. Options



Options are accessed through the toolbar, shown above, at the top of each entry display.



If you use a 2D detector, you can display neutron data in a ring pattern simulation, adjust the focal length, crystallite size, and then import an image file for overlay. The wavelength selection for neutrons and crystallite size options are highlighted.



Many entries in the PDF-4 are based on single crystal determinations. For all entries with atomic coordinates (239,568 entries), the user has a choice of two different molecular graphics packages for display (Jmol shown), as well as bond length, angle, and nearest neighbor atom calculators. Data shown are for lithium manganese nickel oxide.

## IX References

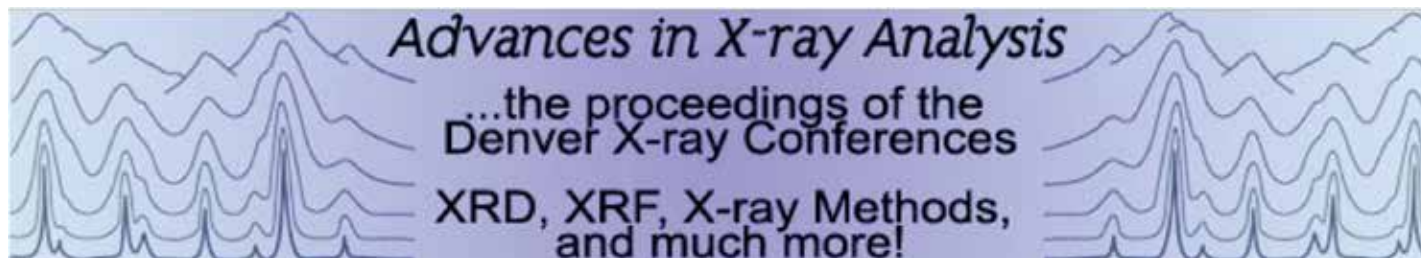
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