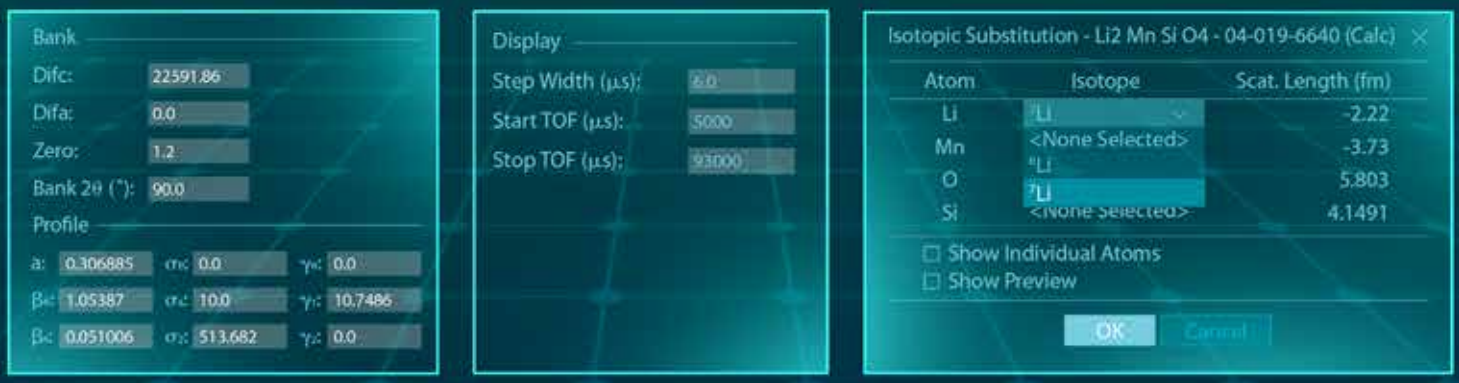
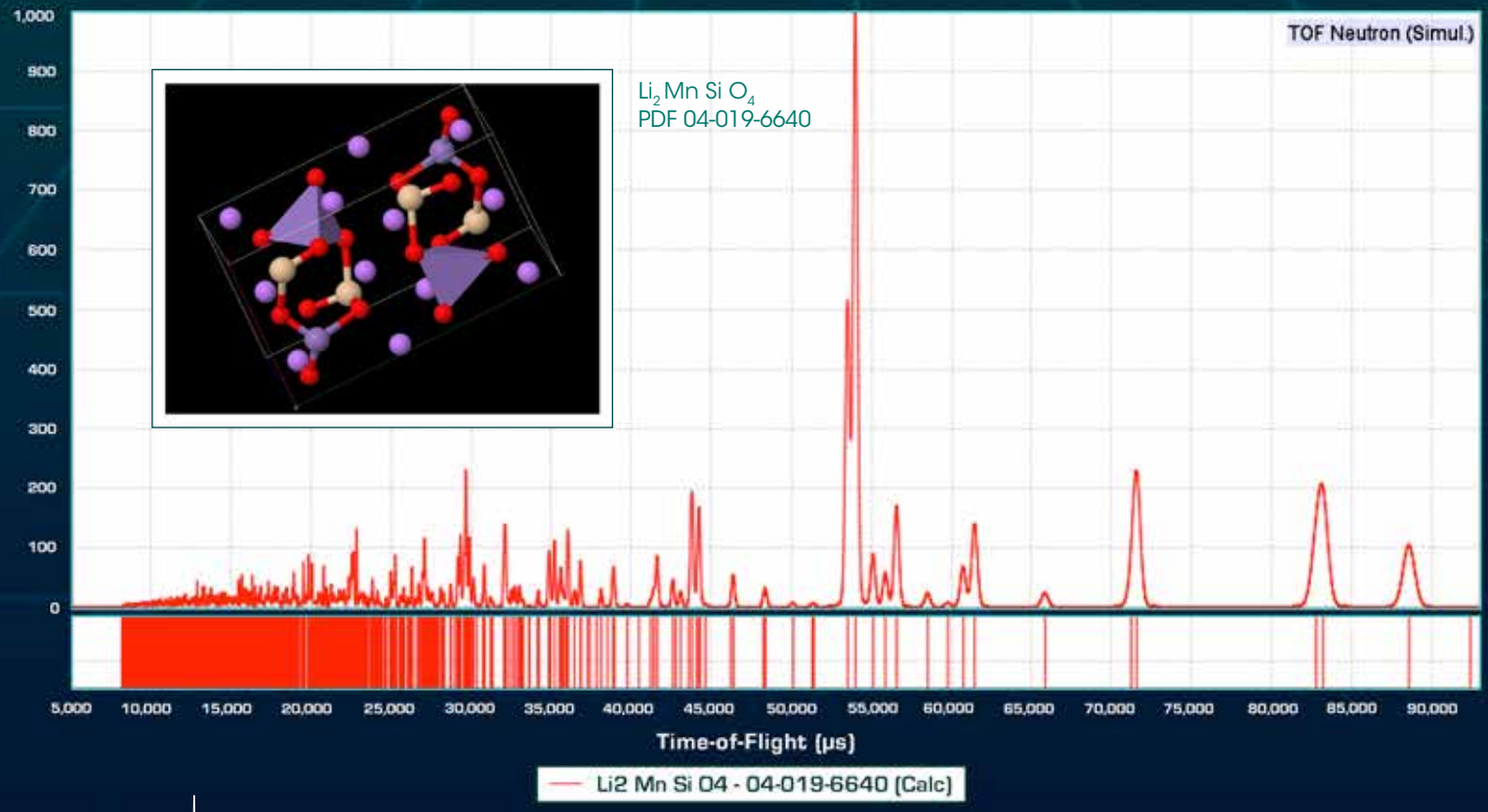
Time-of-Flight Neutron Diffraction

How to analyze Time-of-Flight using powder diffraction
and the Powder Diffraction File™





ICDD®
International Centre for Diffraction Data

ISO 9001:2008 QUALITY ASSURED COMPANY CERT.NR. 11409.1

Overview

In 2016 ICDD again enhanced the materials analysis capability of the Powder Diffraction File™ (PDF®), adding the capability of simulating Time-of-Flight (TOF) neutron diffraction patterns. Utilizing over 272,000 PDF-4+ database entries with atomic coordinates or neutron diffraction structure factors and 26,000 additional entries with cross-referenced access to atomic coordinates, TOF patterns can be simulated, displayed, and compared to imported TOF raw data. Using the Similarity Index capability of PDF-4+, full pattern matching can be performed for phase identification of major phase unknowns. This Guide presents four activities that will help with getting started using the TOF capability in ICDD PDF-4 databases.

For further information on ICDD's neutron diffraction database capabilities, please visit our website at:

- http://www.icdd.com/products/2016/neutrondiffraction.htm

Read the Time-of-Flight abstract submitted for publication for *Advances in X-ray Analysis* from the 2016 Denver X-ray Conference presentation.

- New Neutron Time-of-Flight (TOF) Capability in PDF-4+ Relational Databases: Digitized Diffraction Patterns and I/I_c for Quantitative Phase Analysis

  J. Faber, Faber Consulting, Thornton, PA, USA
  S. Kabekkodu, J. Blanton, T. Blanton, T. Fawcett, ICDD, Newtown Square, PA, USA

  http://www.dxcicdd.com/16/abstracts/D20.pdf

Online Resources

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Our goal at ICDD is to help you solve your materials problems. We provide online publications, technical bulletins, tutorials, and videos. Many tutorials focus on capabilities of the database, but there are also general tutorials that describe methods used to analyze drugs, polymers, and minerals. The tutorial page has links to free download publications, as well as instructional videos. Our website also contains over 1,000 full publications for free download from *Advances in X-ray Analysis*. Our website, tutorial page, and publication pages are there to help you!

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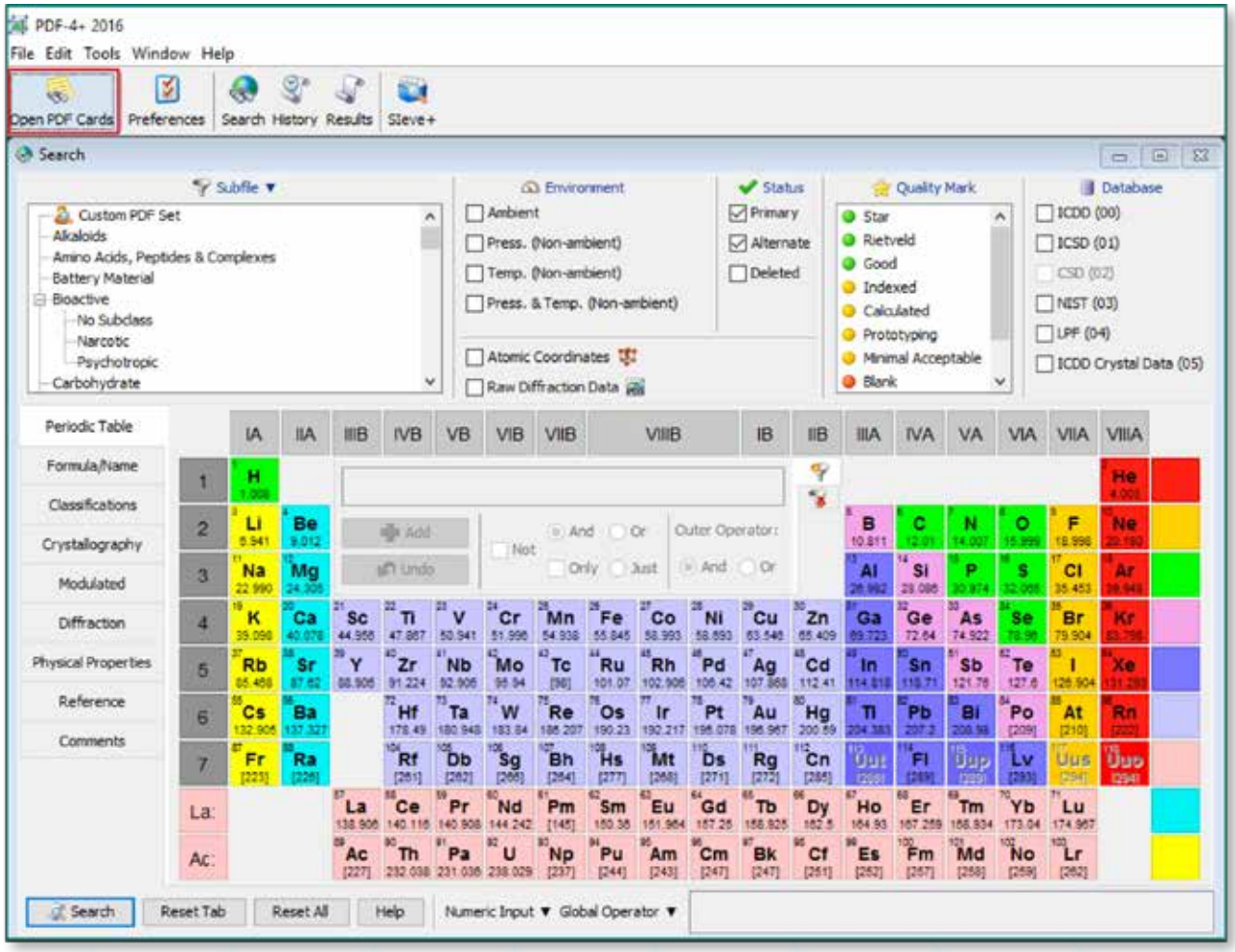
- www.icdd.com/resources/axasearch/search_based_on_vol.asp

ICDD® PDF-4+ 2016 Time-of-Flight (TOF) Neutron Diffraction Pattern Simulation and Phase Identification – Getting Started

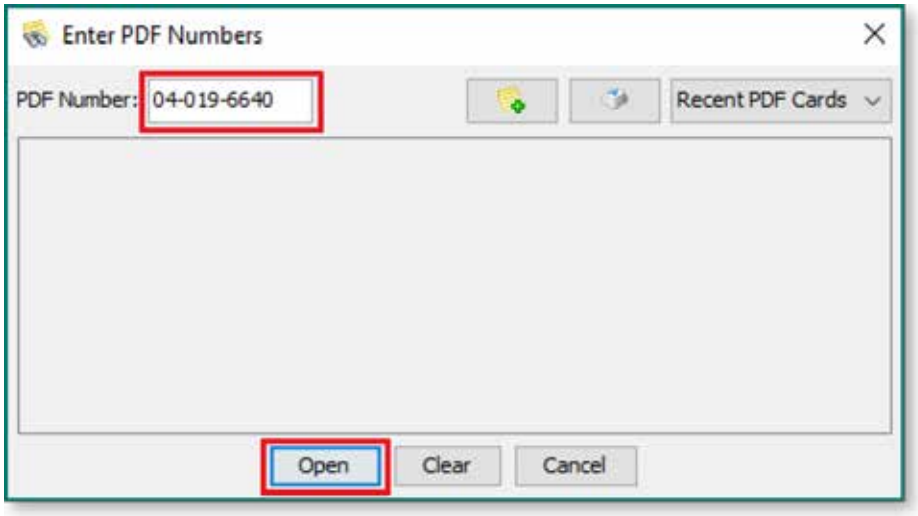
Activity 1 – Plot a simulated Time-of-Flight (TOF) pattern when you know the PDF entry you are interested in reviewing has atomic coordinates:

1 Open PDF-4+ 2016 by double clicking on the desktop icon

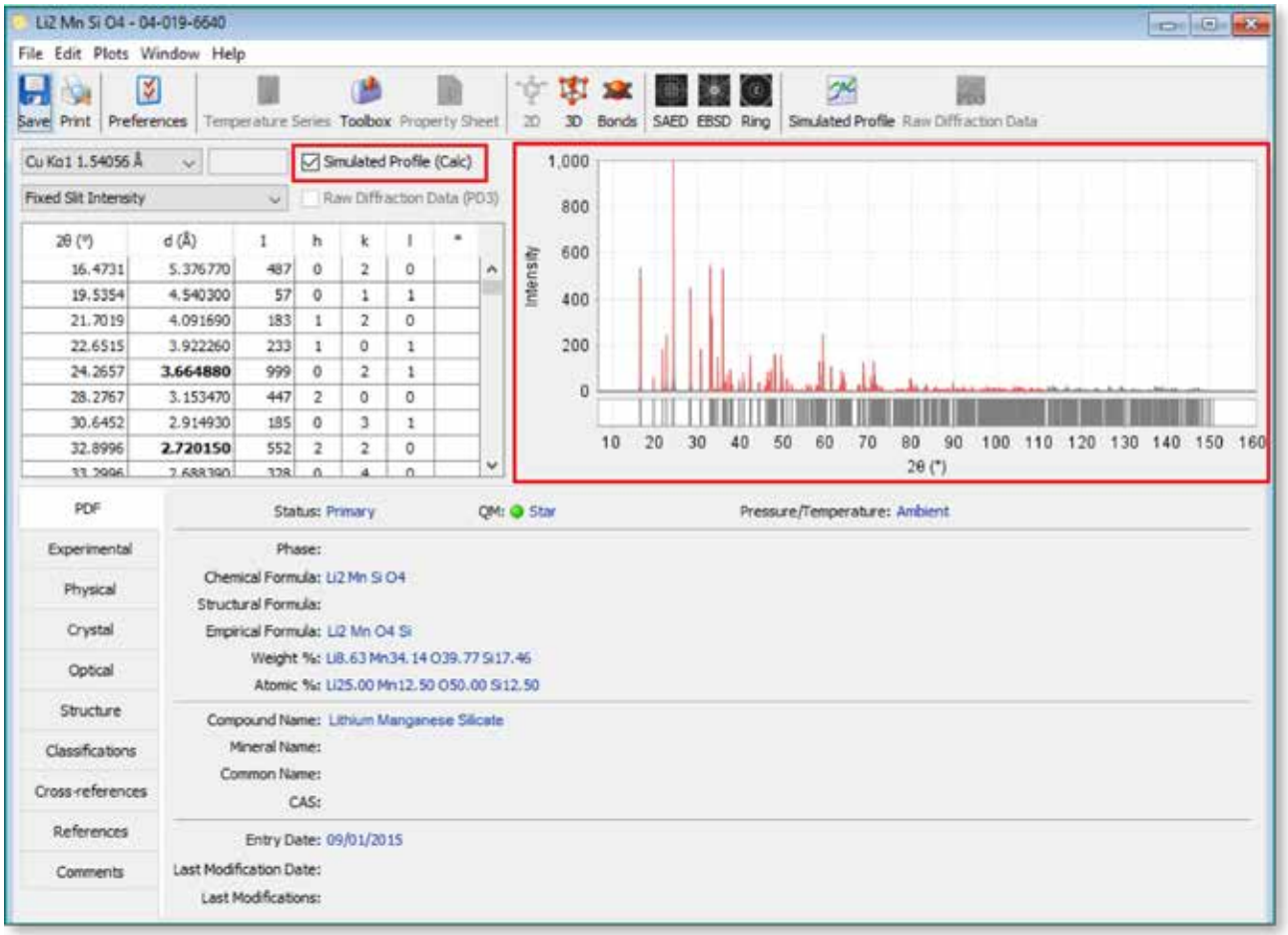
2 Enter the PDF number for a known entry with atomic coordinates by selecting the *Open PDF Cards* button



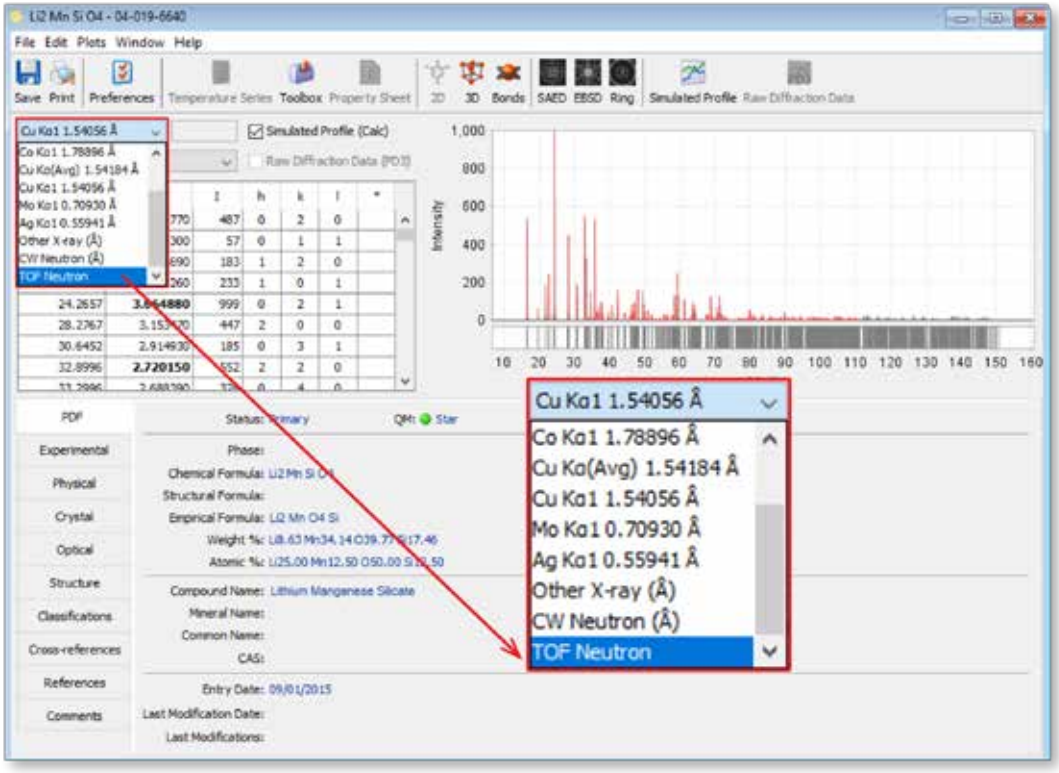
3 Enter a PDF number and click on open. For this example, use entry 04-019-6640, Li_2MnSiO_4.



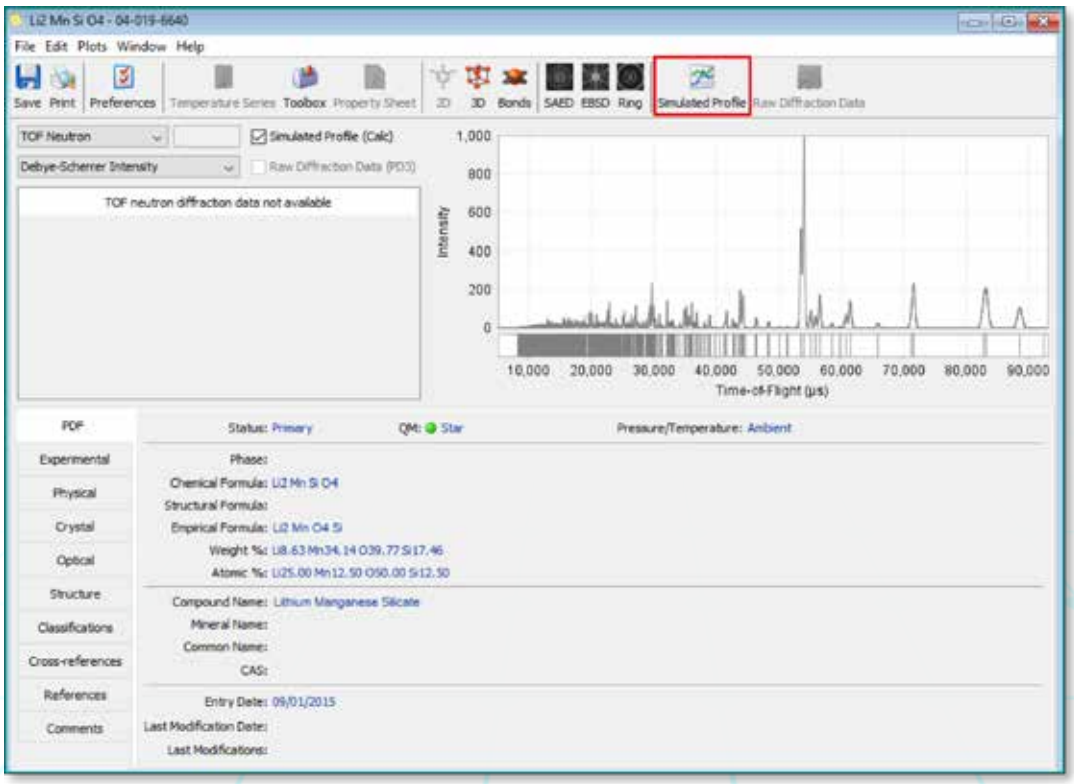
4 The PDF entry 04-019-6640 is displayed, including the option for a simulated pattern. The default is to display a powder X-ray diffraction pattern, Cu Kα1,2 radiation, pseudo-Voight peak profile.



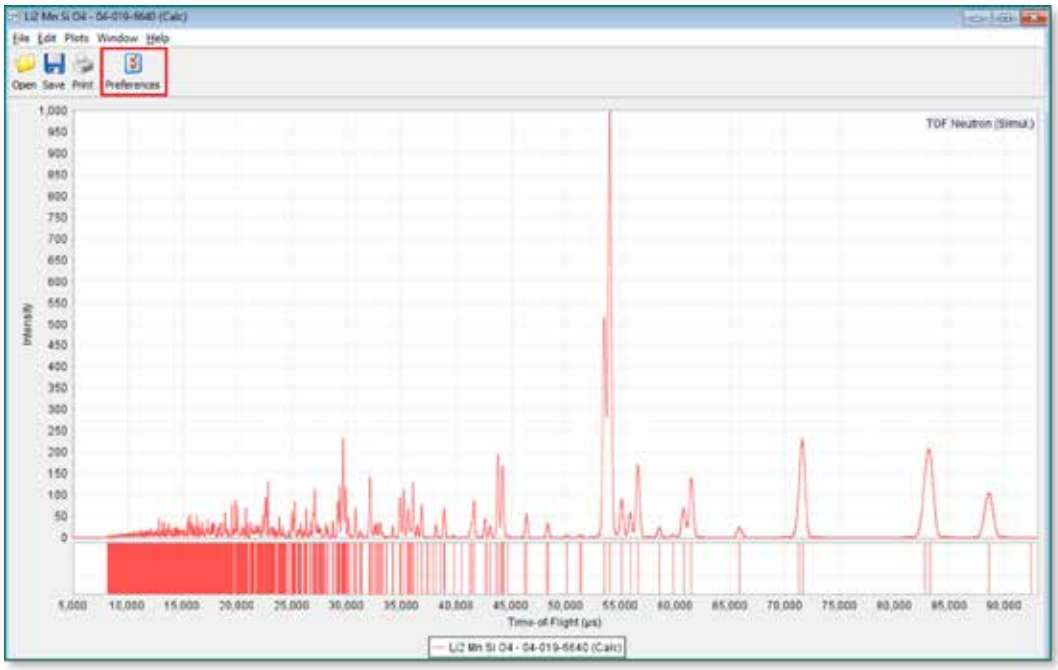
5 Click on the drop down menu for wavelength options and scroll to the bottom, click on *TOF Neutron*

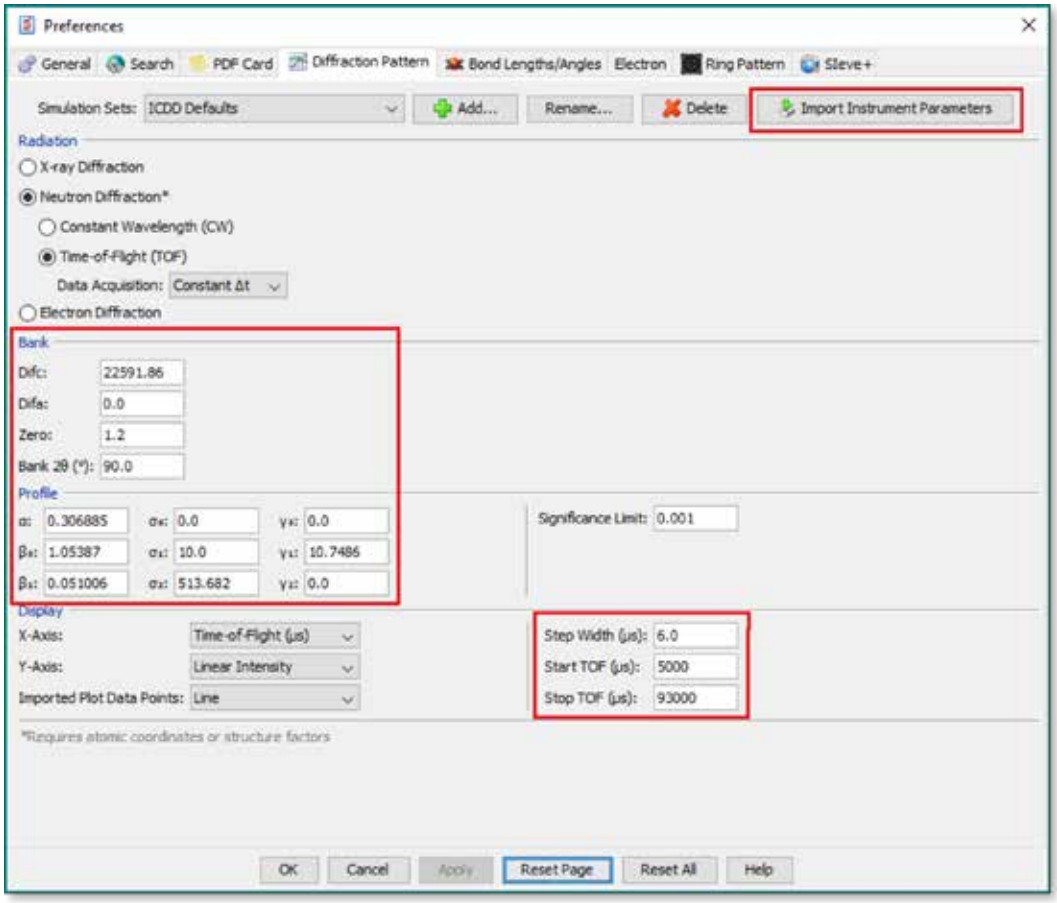


6 The simulated diffraction pattern is now an Intensity vs. Time-of-Flight plot. Currently for PDF-4+ 2016, d-spacing values will not be displayed for TOF data. Click on the Simulated Profile icon and the simulated pattern will be displayed in a larger view.

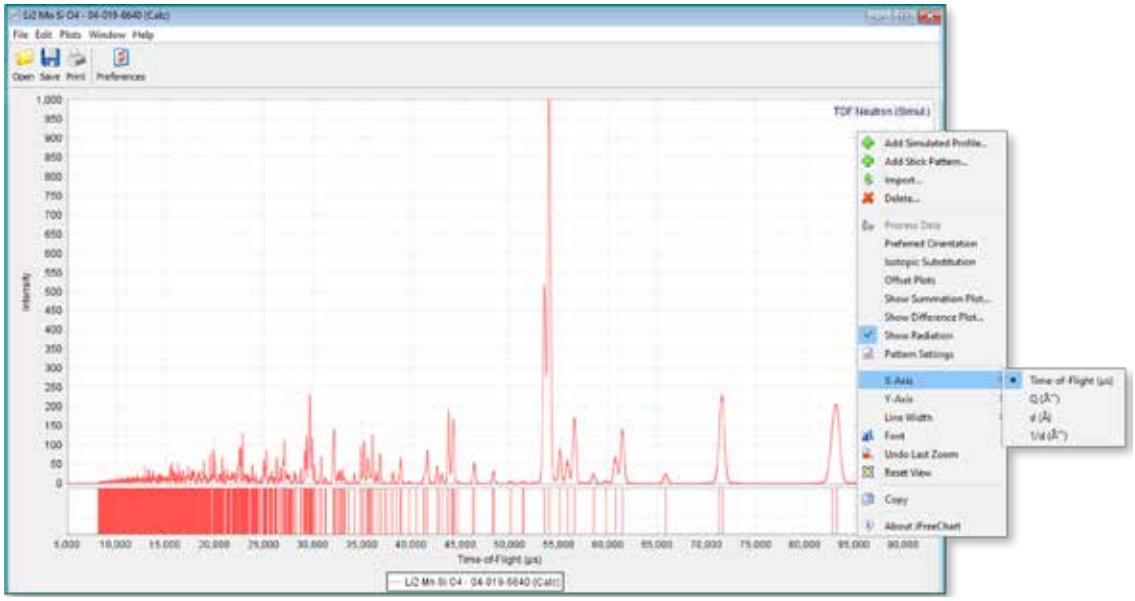


7 The TOF pattern as displayed is based on default parameters built into the PDF-4+ software. The defaults include instrument profile parameters and Time-of-Flight range. To change profile or range, click on the *Preferences* icon and adjust as necessary (Note: instrument profile parameters can be imported from different user facilities. See Activity on page 10).





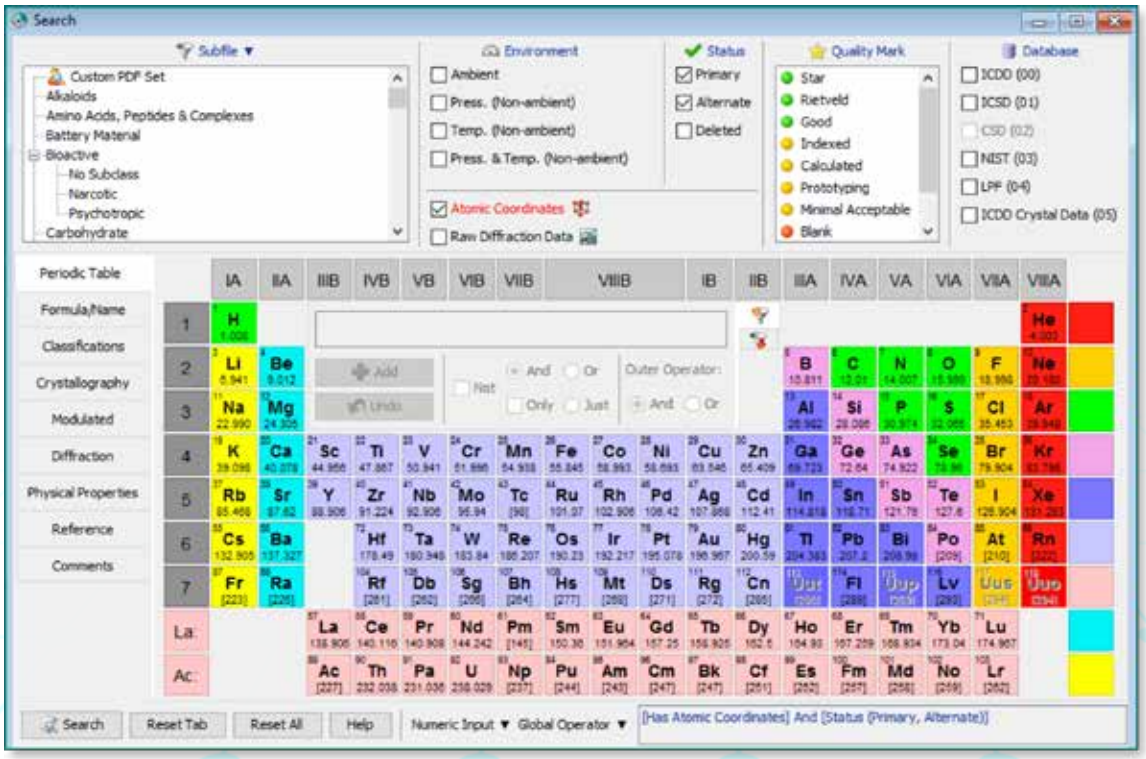
8 A right mouse click on the TOF plot will display additional pattern options, including options for X-axis units: TOF, Q, d, 1/d.



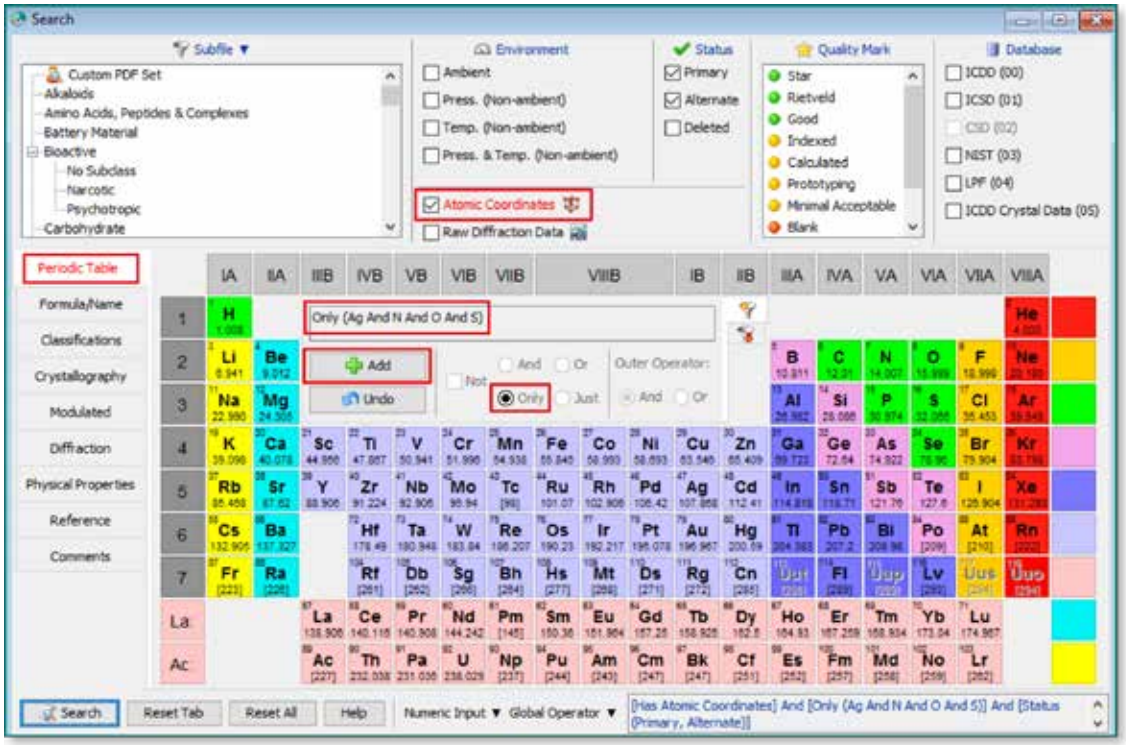
Activity 2 – Plot a simulated Time-of-Flight (TOF) pattern when you search for PDF entries that have atomic coordinates:

1 Open PDF-4+ 2016 by double clicking on the desktop icon

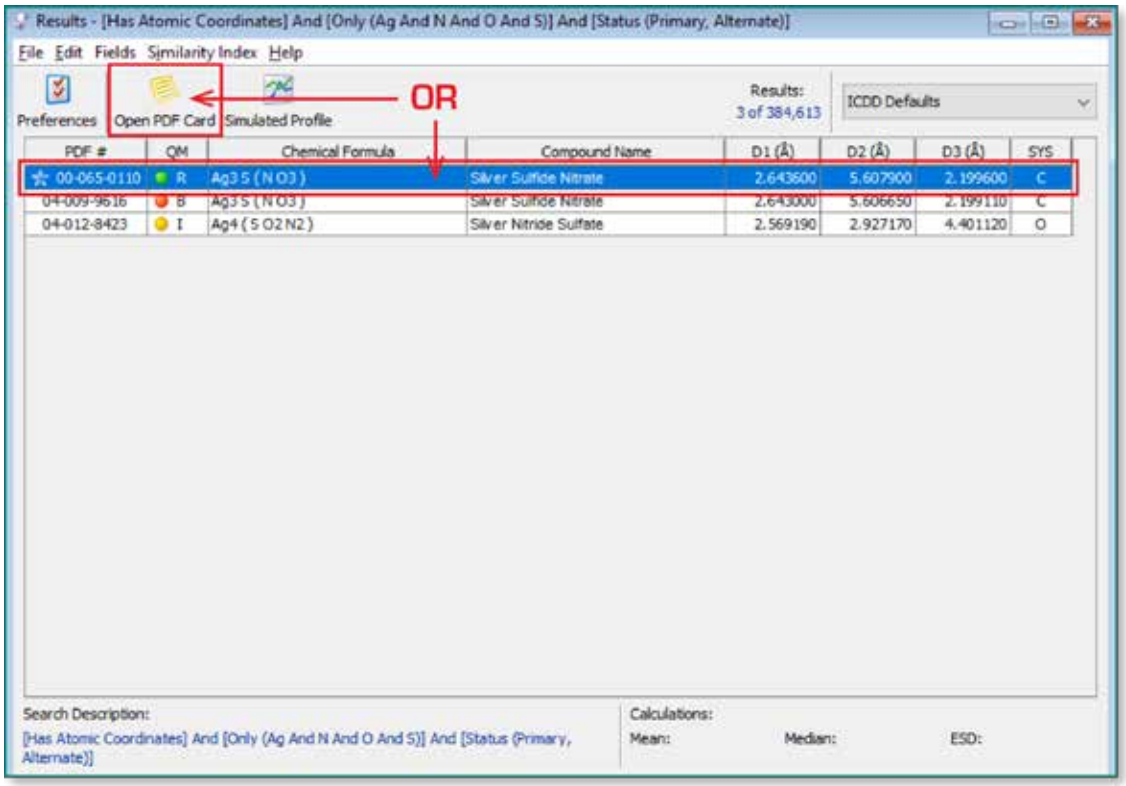
2 Select *Atomic Coordinates* on the PDF-4+ search window



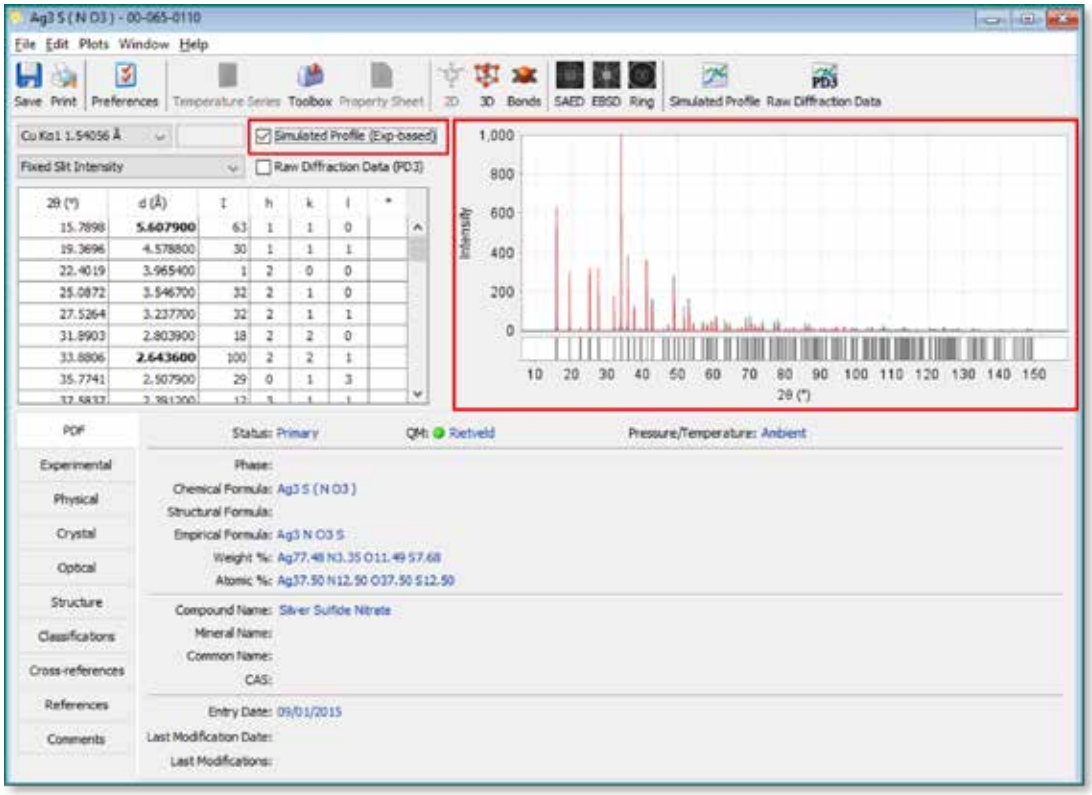
3 Select desired criteria for searching for a material type. In the example below, choose phases with atomic coordinates, plus a Periodic Table search for phases that have only elements Ag, S, N, and O, then click on *Search*.



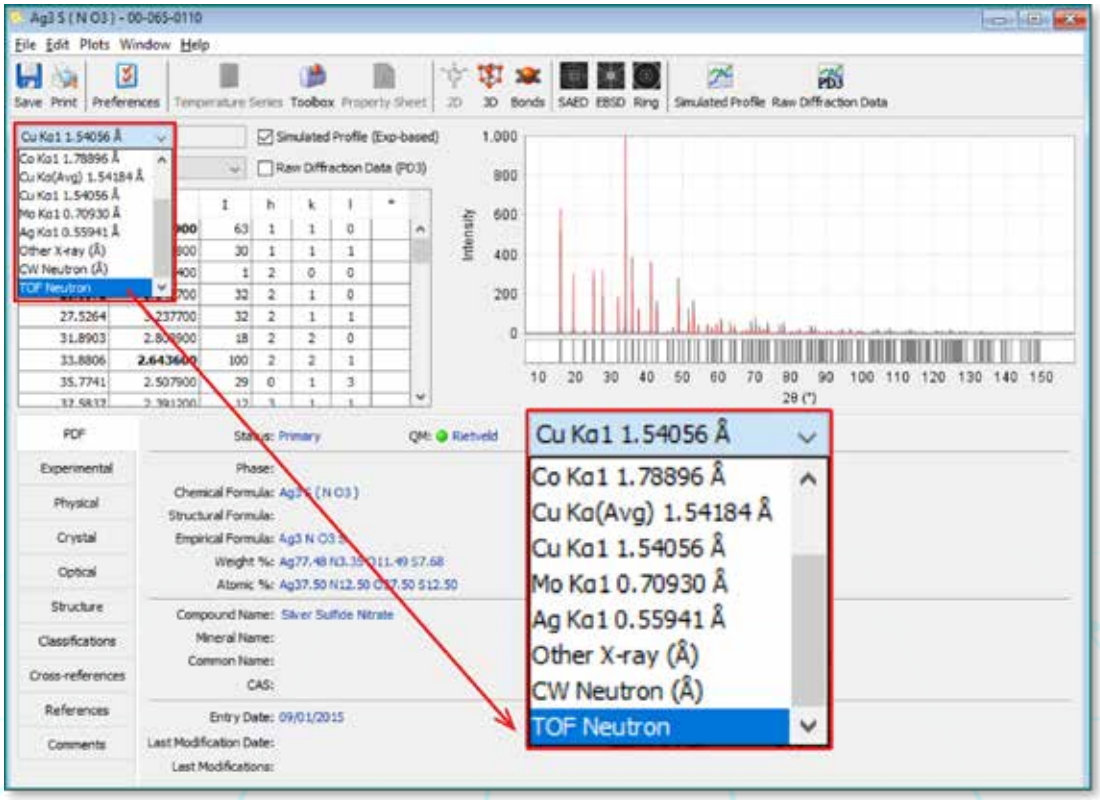
4 Three entries in the PDF meet the search criteria. Open 00-065-0110 by double-clicking on the PDF # row or clicking once on *Open PDF Card*.



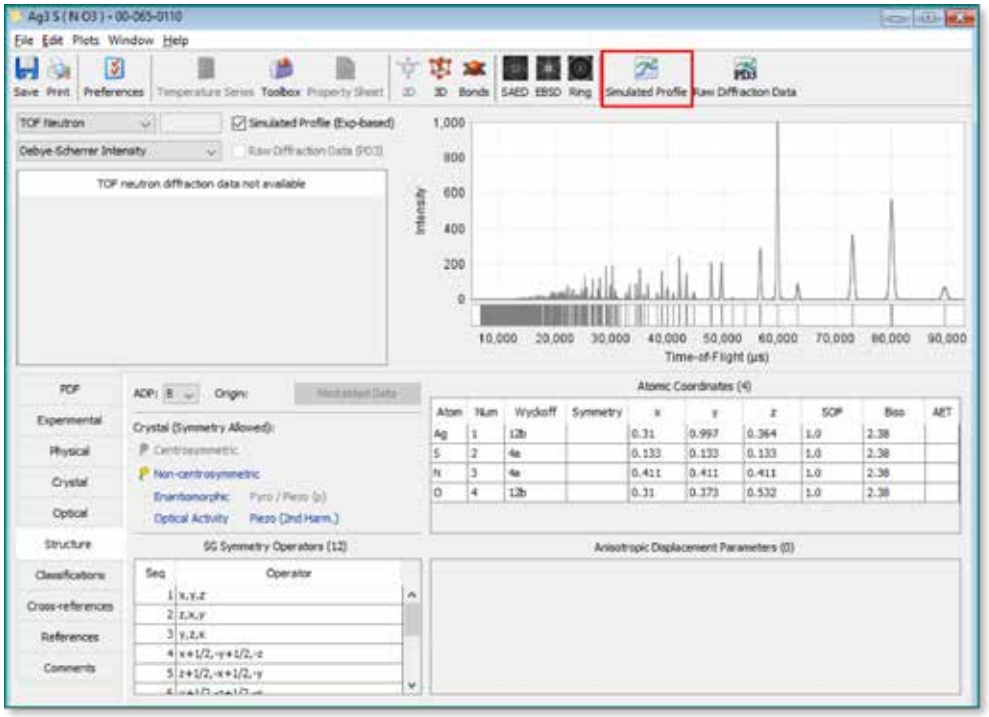
5 The PDF entry 00-065-0110 is displayed, including the option for a simulated pattern. The default is to display a powder X-ray diffraction pattern, Cu Kα1,2 radiation, pseudo-Voight peak profile.



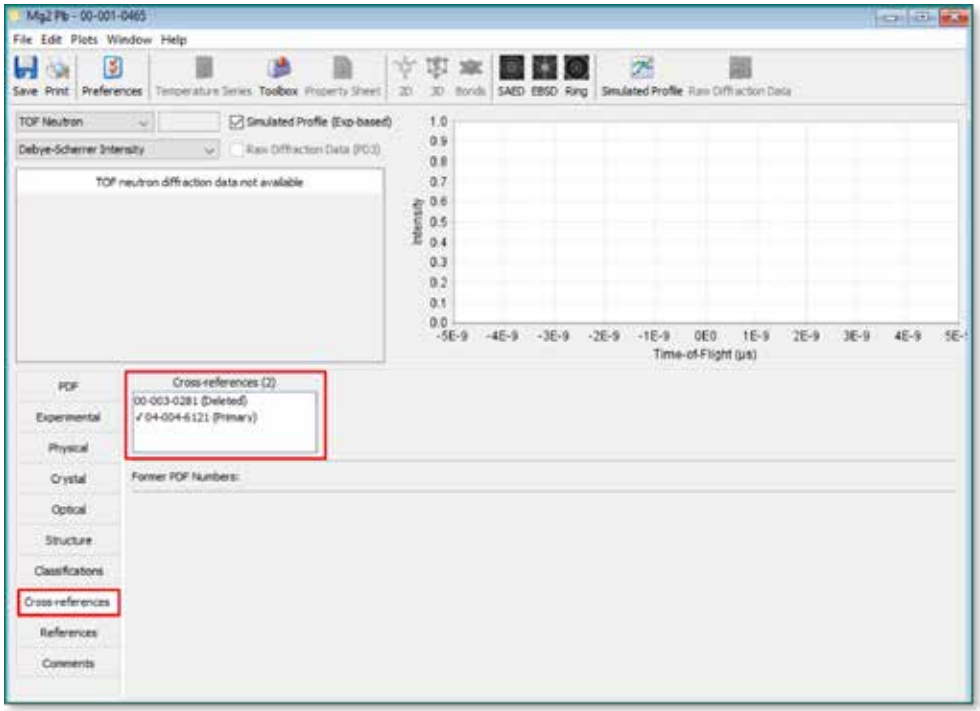
6 Click on the drop down menu for wavelength options and scroll to the bottom, click on *TOF Neutron*.



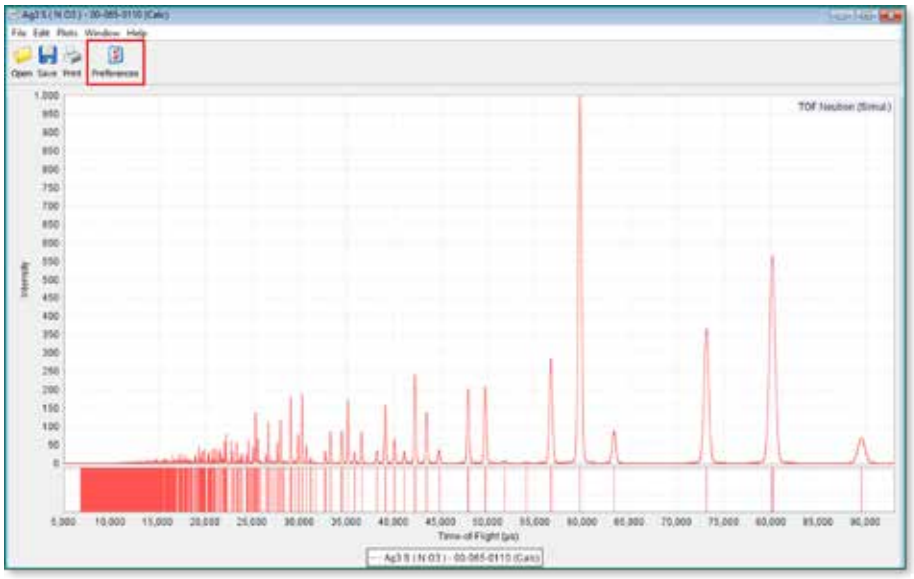
7 The simulated diffraction pattern is now an Intensity vs. Time-of-Flight plot. Currently for PDF-4+ 2016, d-spacing values will not be displayed for TOF data. Click on the *Simulated Profile* icon and the simulated pattern will be displayed in a larger view.

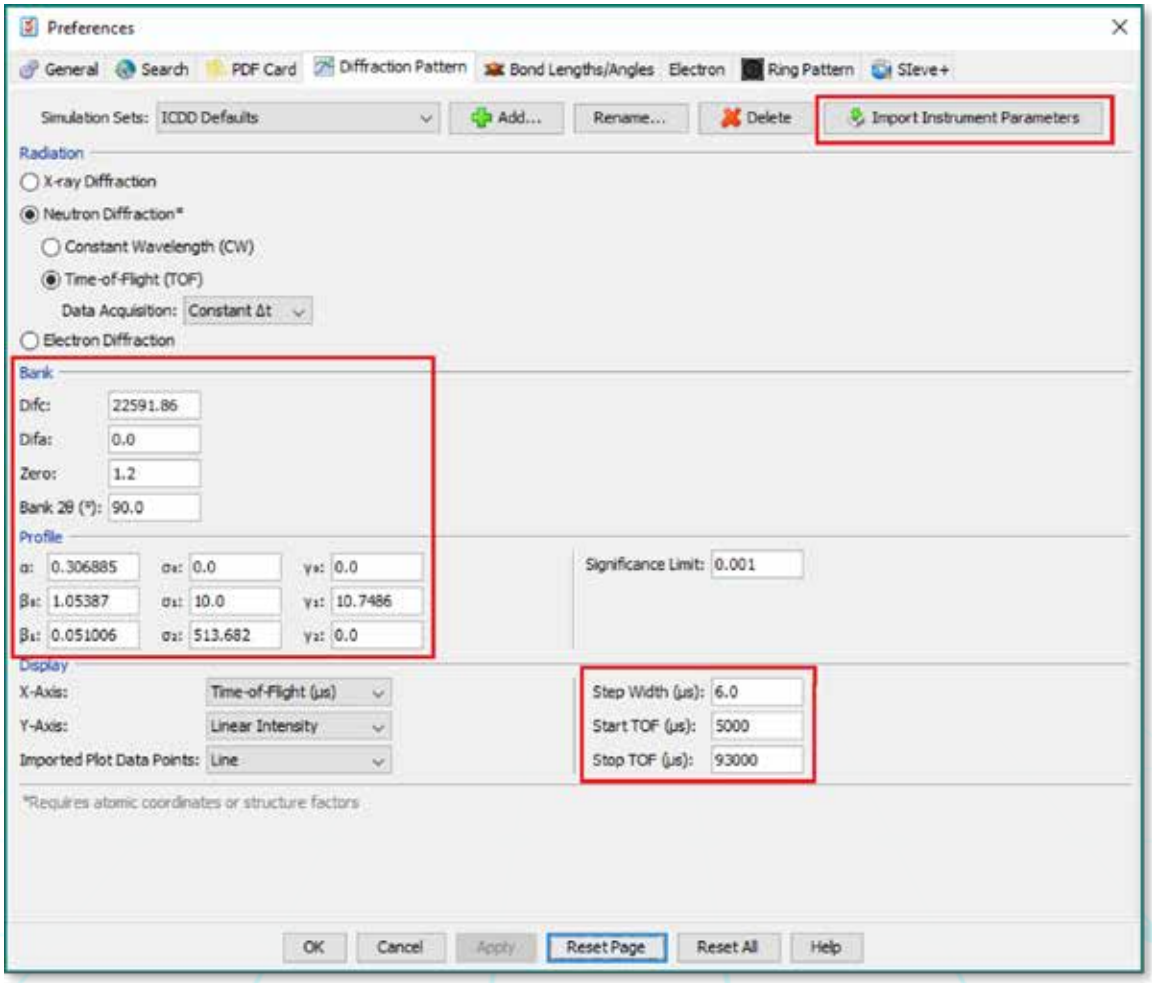


NOTE: If the simulated TOF pattern does not display (see example below), the atomic coordinates are cross-referenced for the PDF entry. Click on the *Cross-reference* tab and double click on a PDF entry noted with a √ (check mark) which is a cross-referenced PDF entry with atomic coordinates for the phase of interest. The cross-referenced entry will be displayed and the simulated TOF pattern can be viewed as described here in Activity 2.

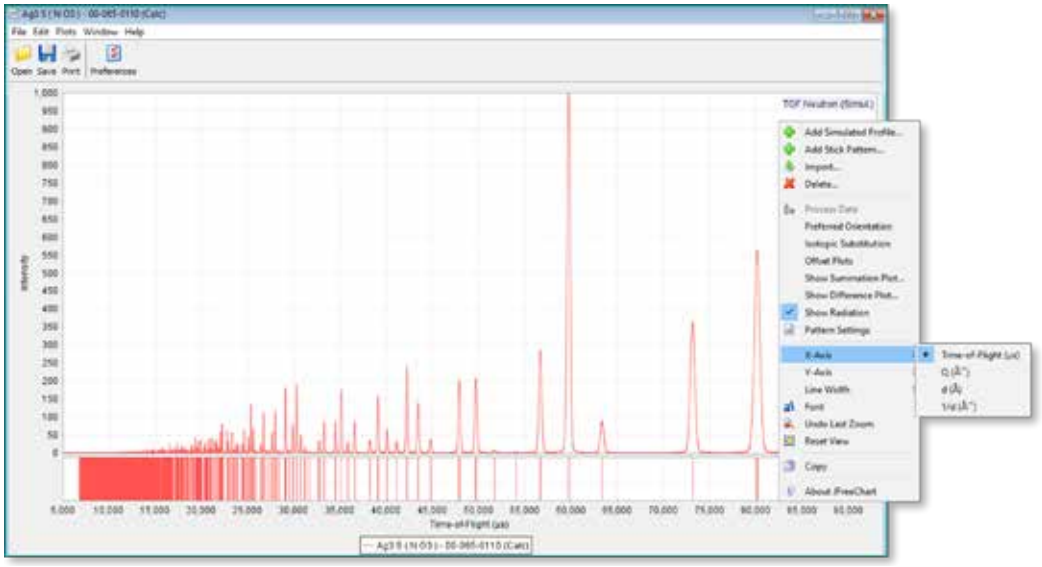


8 The TOF pattern as displayed is based on default parameters built into the PDF-4+ software. The defaults include instrument profile parameters and Time-of-Flight range. To change profile or range, click on the *Preferences* icon and adjust as necessary. (Note: instrument profile parameters can be imported from different user facilities. See Activity 3 on page 10).





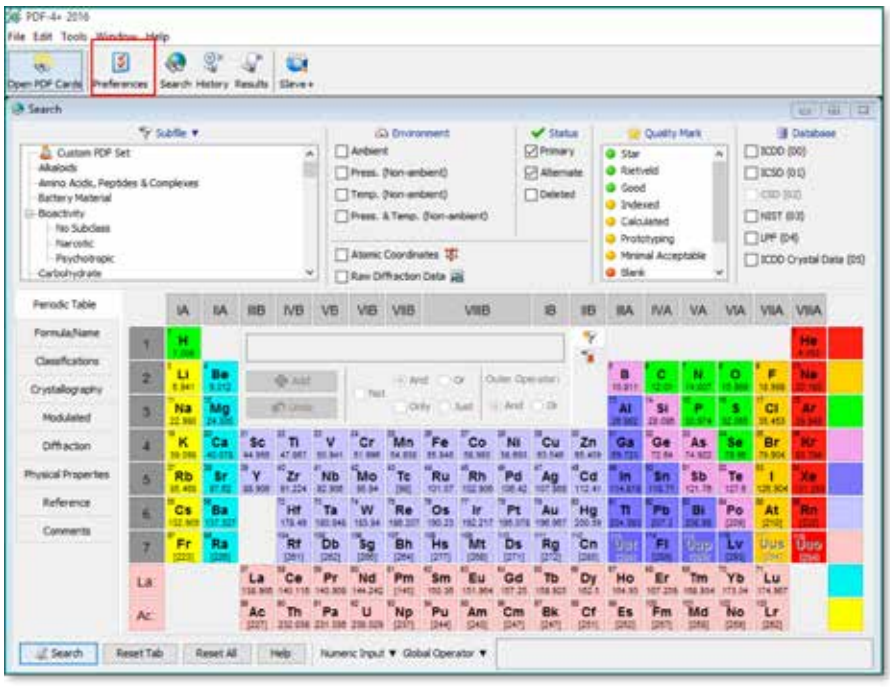
9 A right mouse click on the TOF plot will display additional pattern options, including options for X-axis units: TOF, Q, d, 1/d.

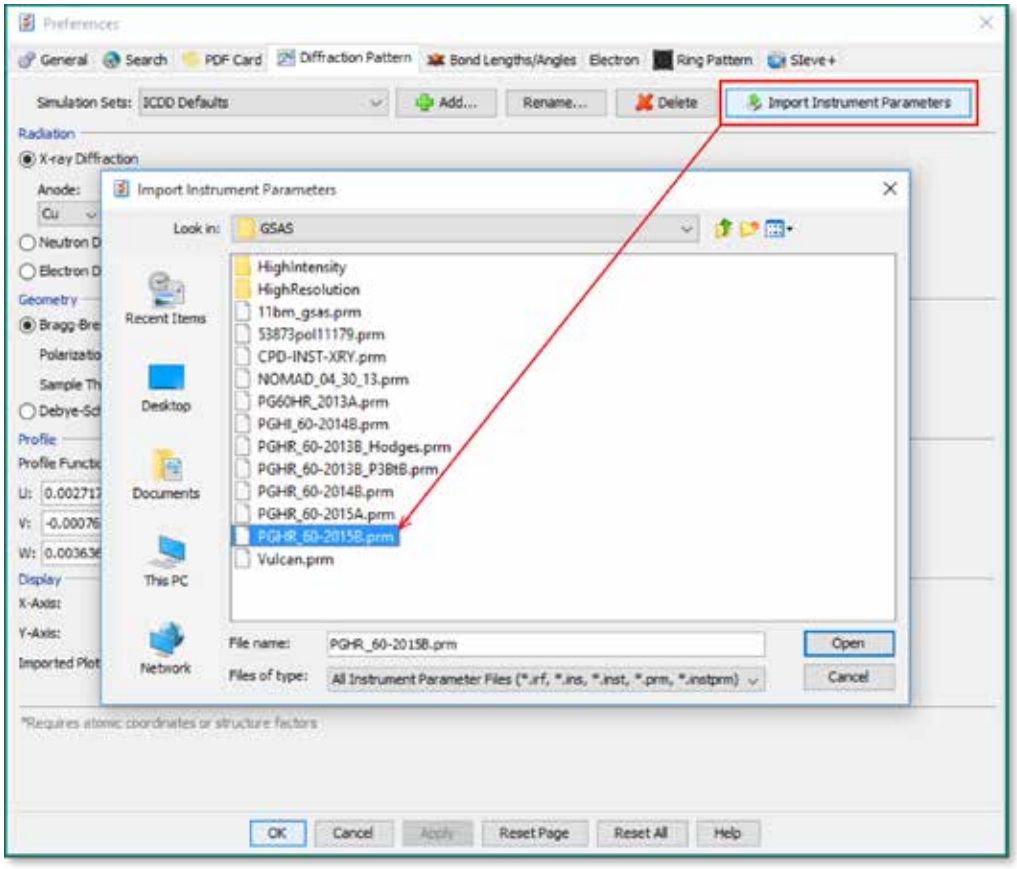


Activity 3 – Importing beamline instrument parameters, plot a simulated Time-of-Flight pattern after doing a database search, plot a Time-of-Flight pattern after doing an isotope substitution, import a raw data pattern for comparison.

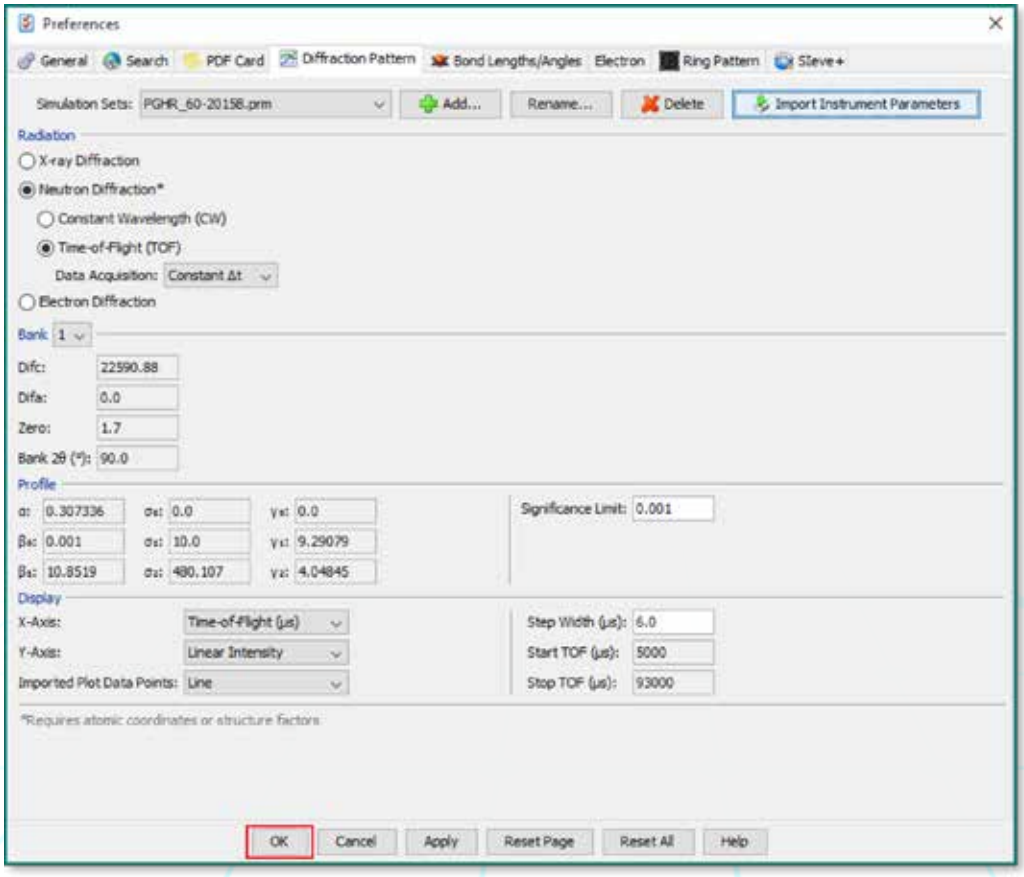
1 Open PDF-4+ 2016 by double clicking on the desktop icon

2 The PDF Search window will appear. Select the *Preferences* button on the main toolbar and select the *Diffraction Pattern* Tab. In this tab, click the Import *Instrument Parameters* button and select an instrument parameters file you have available (can be either GSAS or FullProf format).

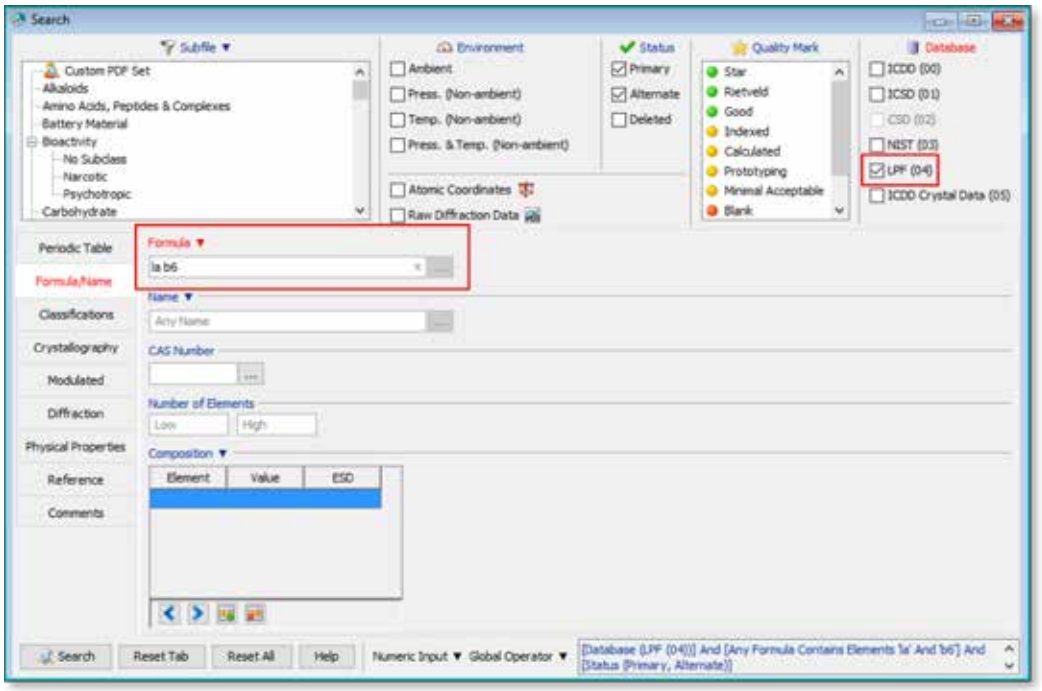




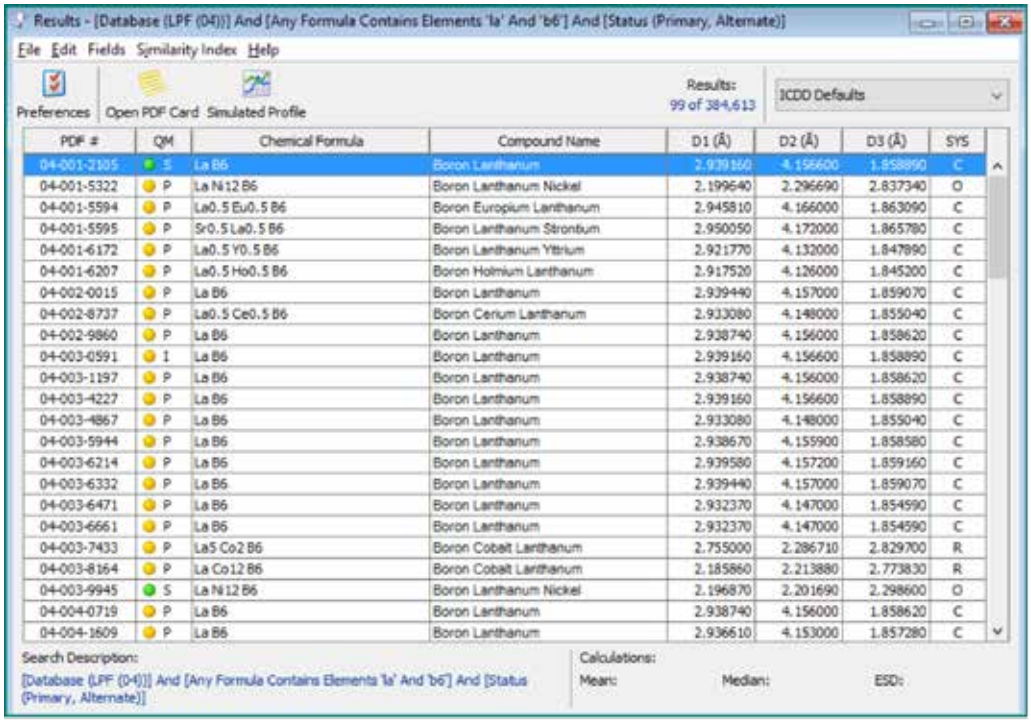
3 Once the instrument parameters have been imported, click OK.



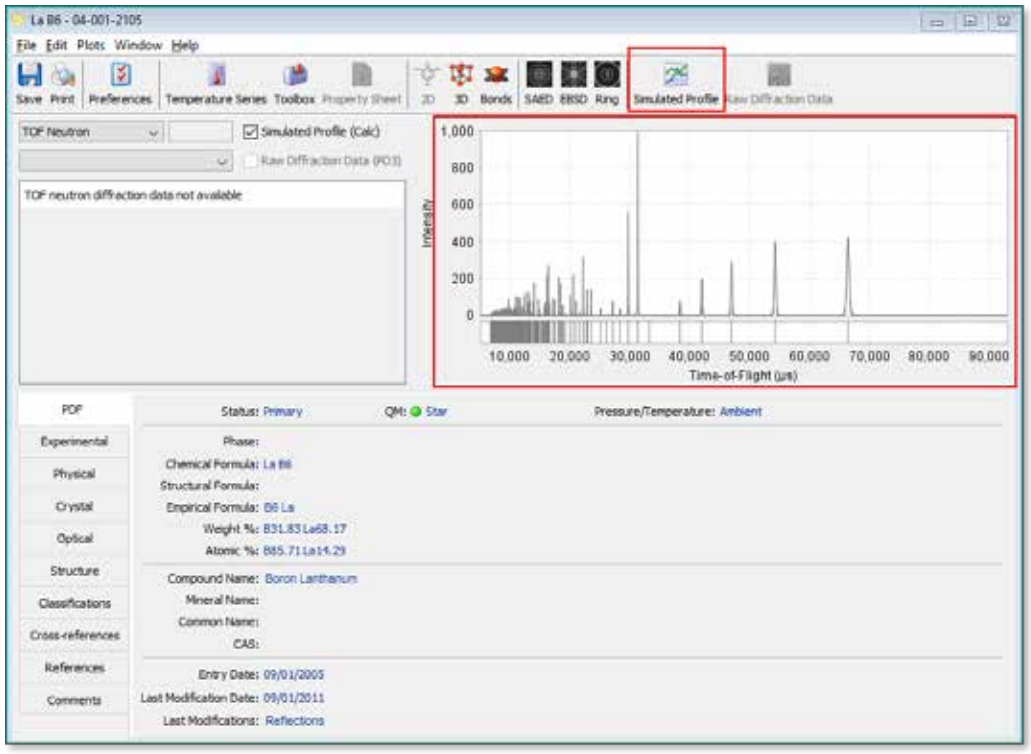
4 The next step is to perform a search and get a list of patterns in the database. This is done using the Search window that is displayed at program startup. For example, the search below is for the formula LaB_6, lanthanum hexaboride. The search also included the Linus Pauling File (LPF) database (04), as all of those entries have atomic coordinates. Patterns with atomic coordinates are required to simulate TOF neutron diffraction patterns (in PDF-4+ 2016 you can use 00, 01, 03, and 04 databases).



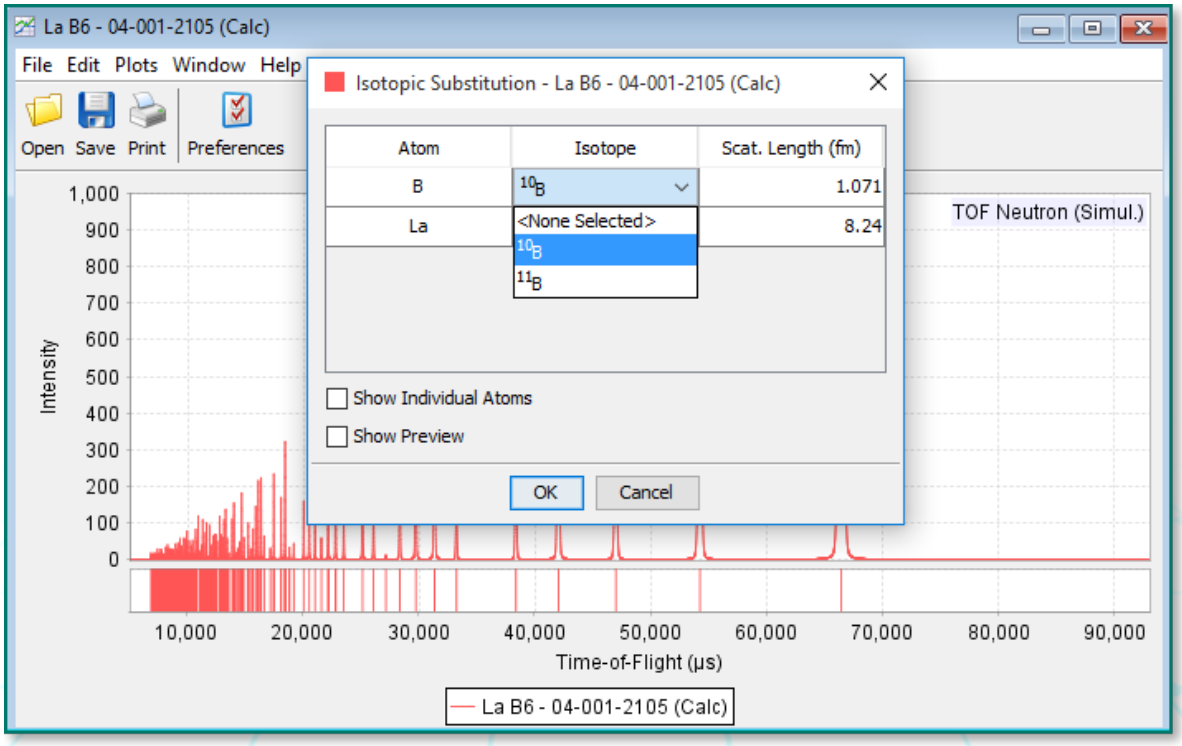
5 After clicking the *Search* button, a list of search results is shown. Double-click any of these entries to open the PDF card.



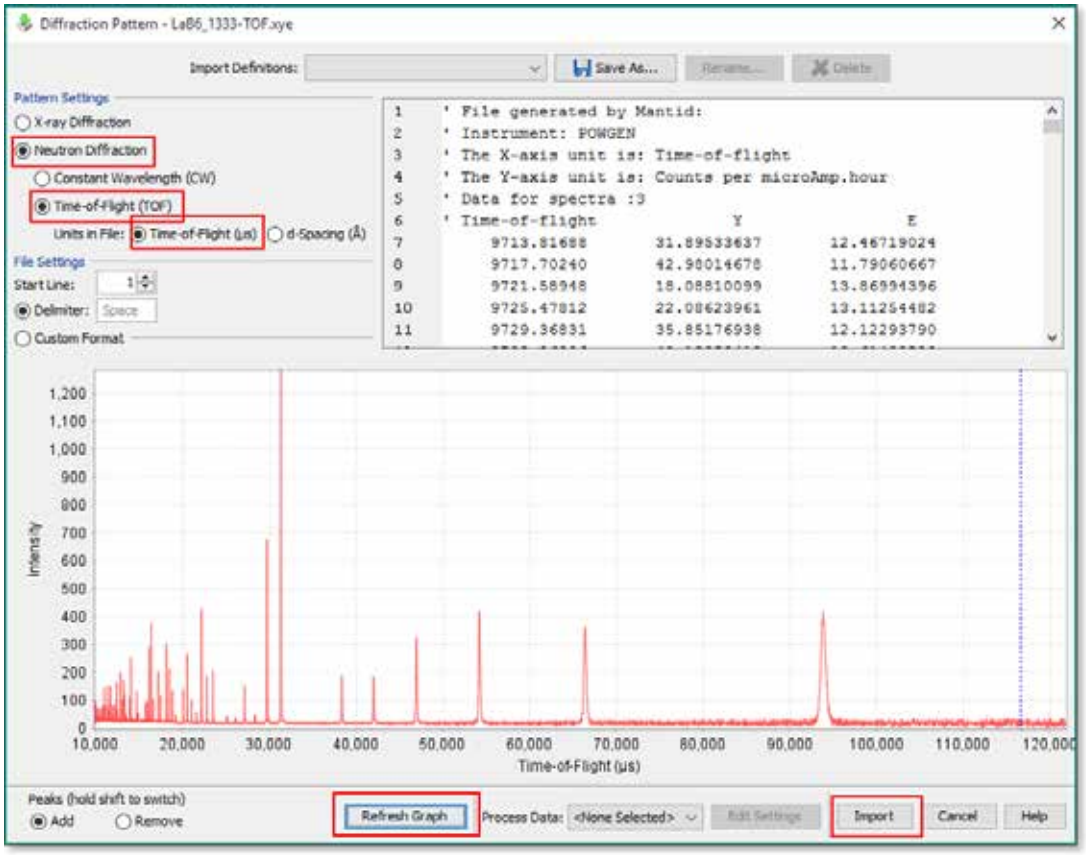
6 PDF entry 04-001-2105 is opened. There is no TOF neutron diffraction data shown in the top-left table. The list of d-spacings/intensities/etc. are not published in the 2016 PDF release (this is planned for the 2017 release). However, you can see the TOF neutron powder diffraction simulation at the top-right. Click the *Simulated Profile* button to open the simulation in a new window.



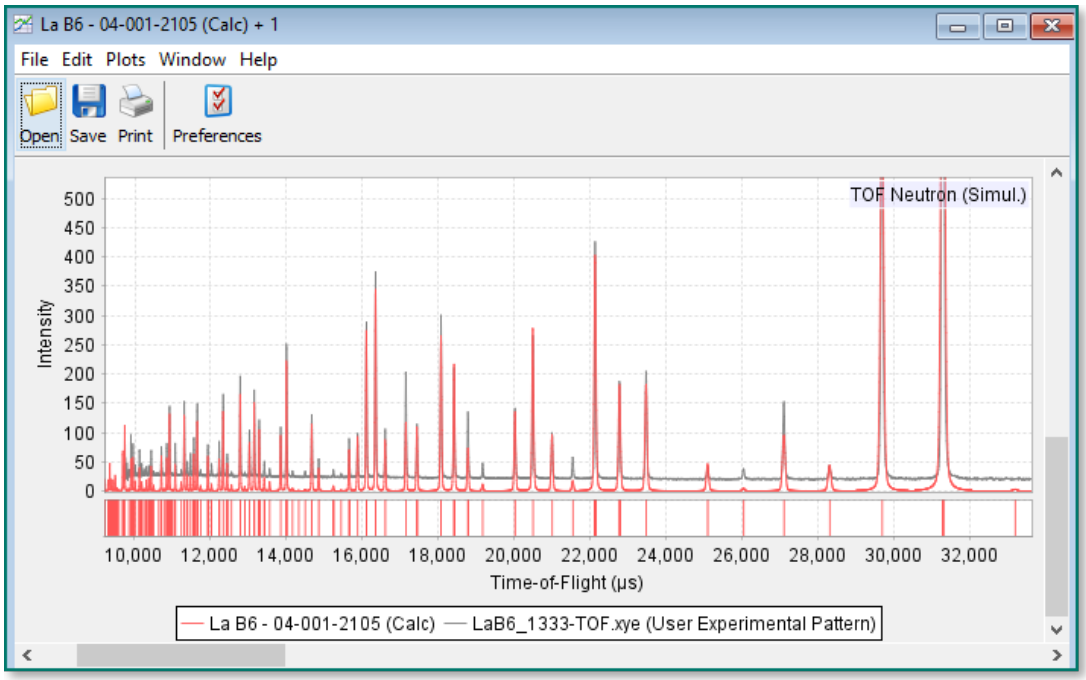
7 Isotopic substitution can be performed by clicking *Plots Menu > Isotopic Substitution*.



8 You can also import an experimental data file by clicking *Plots Menu > Import*. An example imported raw data file is below. Make certain you select the appropriate settings highlighted below. Click the *Refresh Graph* button to load the data and then click the Import button to import it.



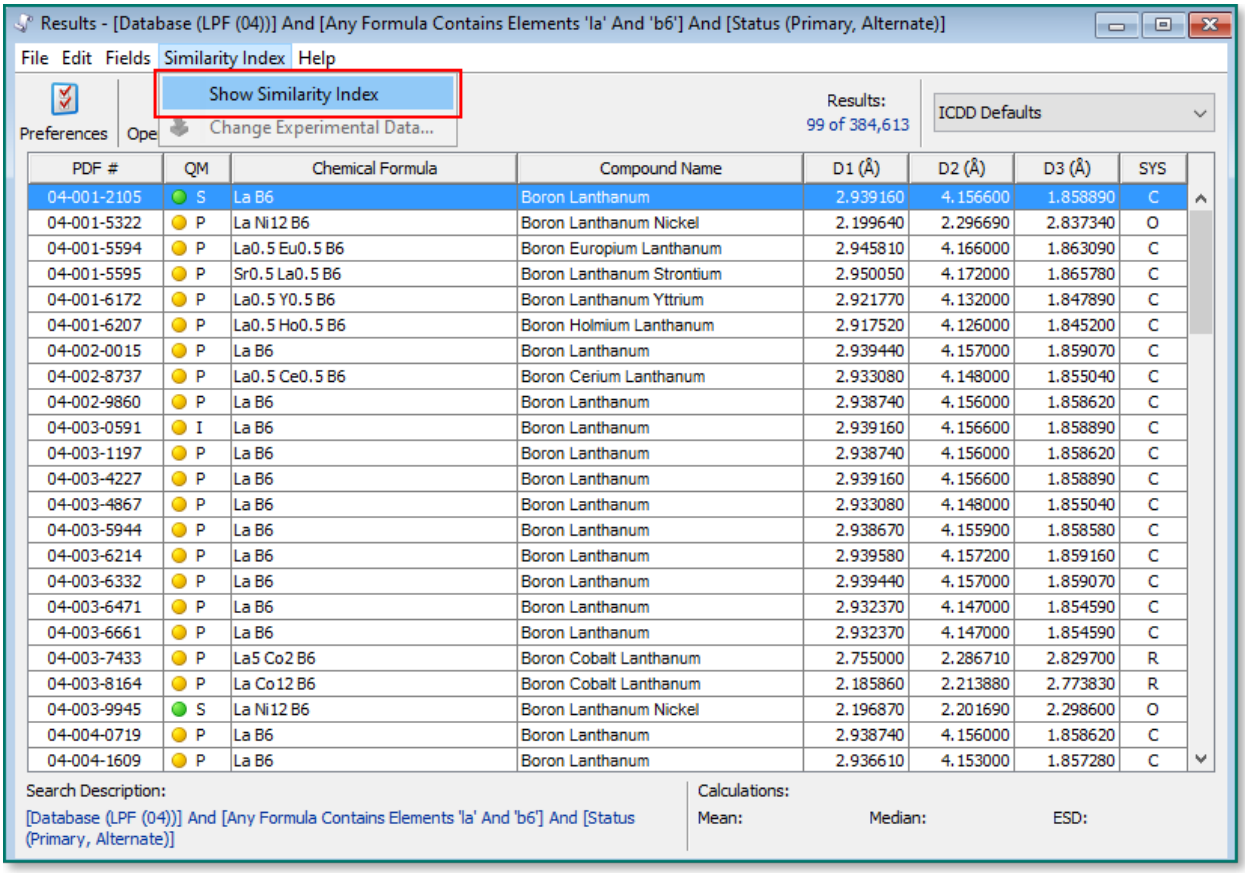
9 Here is a comparison of the simulation to the unprocessed raw data.



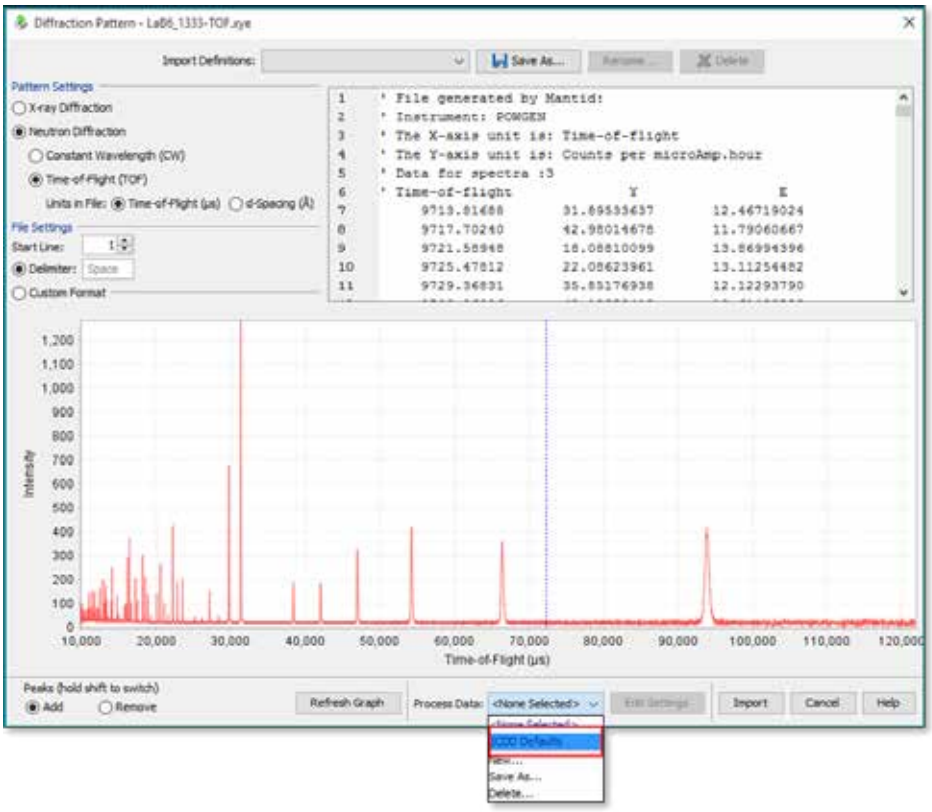
Another TOF neutron diffraction capability in the PDF-4+ 2016 is the ability to compare a TOF data file with TOF simulations in the database using full pattern matching. Full pattern matching creates similarity indexes, which are numbers that describe how well the patterns match. A similarity index of 0.0 is considered a perfect match (this is rare). The larger the number, the less likely the phase being a match. To run a Similarity Index analysis:

1. Get a list of search results you want to compare (see Activity 3, steps 1-4).

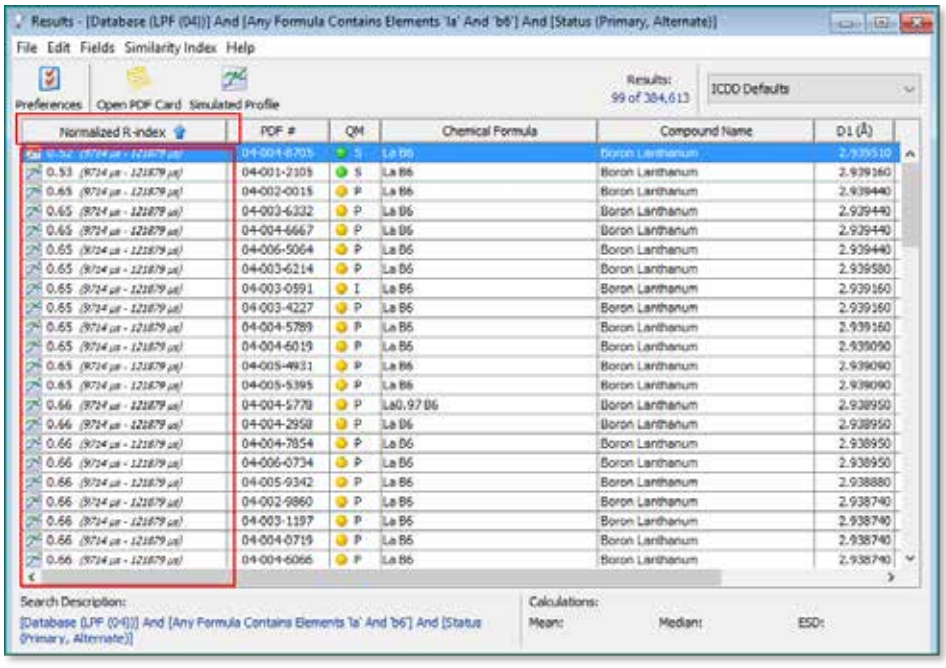
2. Click *Similarity Index Menu > Show Similarity Index*.



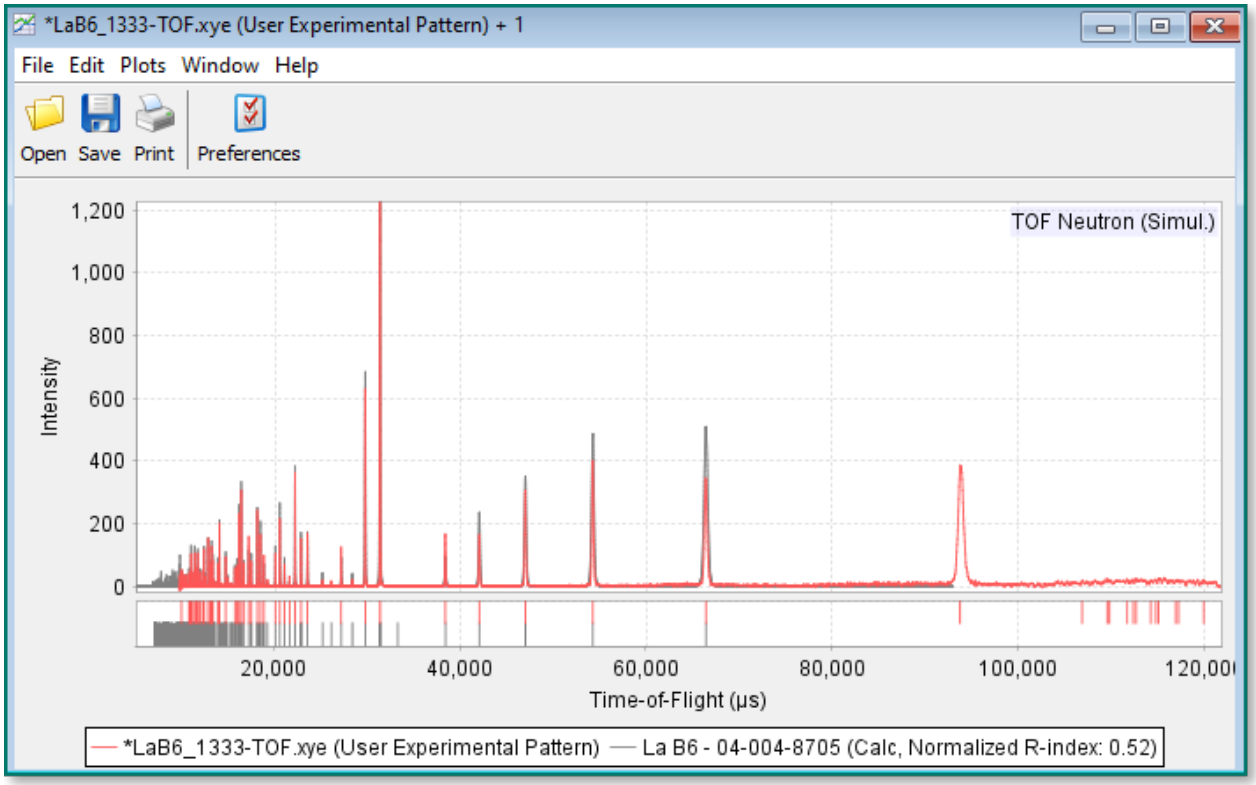
3 Import the pattern as you did in Activity 3, step 8. It is important to remove the background first, as we do not create a background in the TOF pattern simulations. Click the *Process Data* combo-box to process the experimental data. Manual refinement can be perfromed by clicking the *Edit Settings* button.



4 After clicking the *Import* button, the program will load the processed TOF data and compare it to dynamically created pattern simulations in the search results table. Click the *Normalized R-index* column to sort the data with the best matches at the top.



5 The last step is to right-click the match you want to see and select *Graph Similarity Index*. This will show the experimental data compared to the selected simulation (similar to step 9 from above).



Summary

Performing TOF neutron powder diffraction simulations (with the option of isotopic substitution) and comparing TOF experimental data patterns to these simulations are the "TOF features" added to the PDF-4+ 2016 software and database released in September 2016. Time-of-Flight simulations can also be displayed in the PDF-4/Minerals 2016 and PDF-4/Organics 2017 databases using the procedures shown in this User Guide.

The next phase of TOF analysis at ICDD is to allow the user to perform phase identification on TOF neutron diffraction data using our Sleve+ plug-in software. This is in development and is currently scheduled for the PDF-4 2017 product release.

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