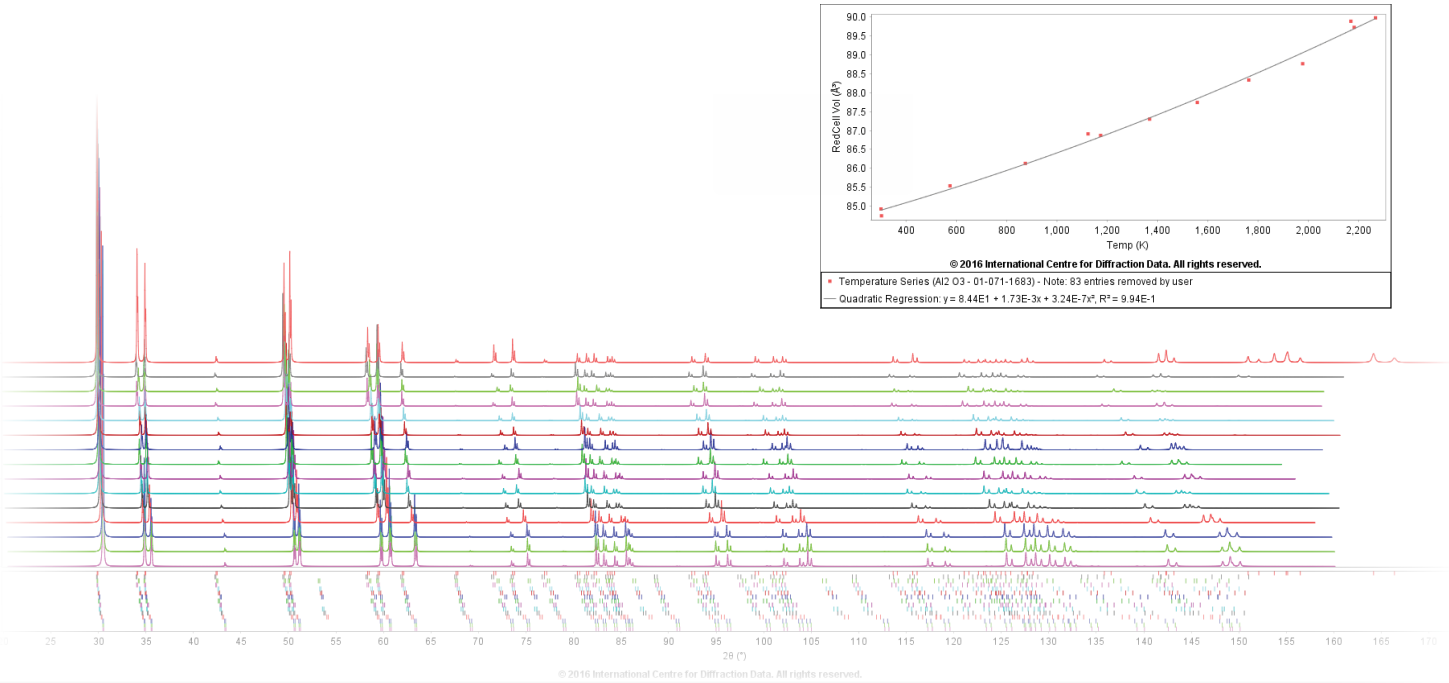
Thermal Expansion

How to Measure Thermal Expansion and Thermal Expansion Coefficients Using the Powder Diffraction File™

User Guide



Thermal expansion of corundum, from temperature 298 K to 2268 K using combined data from four literature references.

ICDD
International Centre for Diffraction Data

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

Dear Customer,

The Powder Diffraction File™ (PDF®) currently contains over 900,000 published entries from a review of approximately 1.3 million publications, grants, and donations. Each entry is comprised of >100 fields and some fields contain hundreds of data points. In the PDF-4+ family of products (PDF-4+, PDF-4/Organics, PDF-4/Minerals, and Web PDF-4+) we have organized the data into a relational database and have written extensive software to enable any user to data mine this large compilation.

Through the guidance and input of our members we started to data mine the database for information on the thermal properties of materials. This research quickly showed that the database has an enormous capability to produce unique results on the thermal expansion properties of materials. Over the last few years, the ICDD technical editors have extracted data from the database and tabulated it into organized tables, and created extensive cross-referencing systems that link materials with the same chemistry and space group. The ICDD's Engineering and Design staff have created several embedded programs (applications) so that the user can quickly identify materials of interest, display and plot the relevant information, and calculate equations of state.

This technical bulletin shows the work that has been done and the methods developed to analyze thermal expansion and thermal expansion coefficients in materials. The capabilities shown are from PDF-4+ 2016. All the plots, tables and graphs can be produced with Release 2015, but without some of the new program applications that take multi-step processes into single point and click menu option.

This work is not finished. In upcoming years we expect to add additional features. The ability to generate pressure series with point and click options is currently in development and we will add other features based on customer feedback.

Our goal is to help you solve your materials problems.

Acknowledgements

This work has been guided by ICDD members of the Non-Ambient Diffraction Subcommittee. This subcommittee has over 60 member field experts and reports to the Technical Committee of the ICDD.

In 2012, the subcommittee directed ICDD's editorial staff to extract temperature of data collection and other relevant information (i.e. experimental conditions) from past editor reviews and literature publications. Data were extracted from decades of editor's comments and placed in a separate field.

In subsequent years, a number of editorial content additions and programming applications have been written by ICDD staff members to be able to quickly review and determine thermal expansion and expansion coefficients. As the new programs enable data mining, this has led to additional capability as we try to optimize the processes and make it user friendly. Drop down menus with point and click options are used extensively with the embedded software in the database.

The ICDD is indebted to its volunteer scientific members who contribute to the field on a daily basis.

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How to Measure Thermal Expansion and Thermal Expansion Coefficients Using the Powder Diffraction File™

"Thermal expansion is the tendency of matter to change in shape, area, and volume in response to a change in temperature."

Introduction

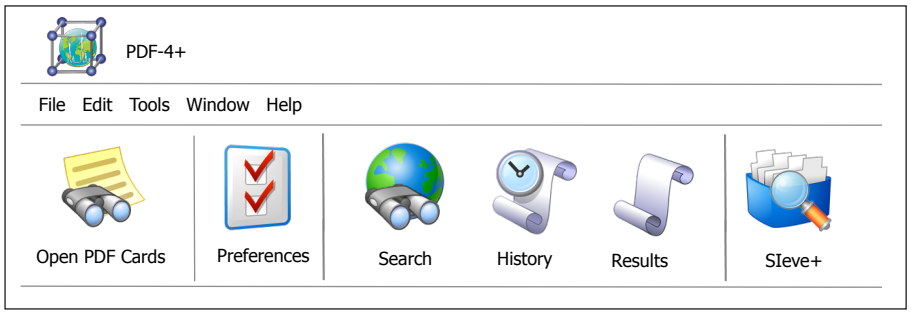
Powder diffraction is one of the fundamental methods used to determine thermal expansion and thermal expansion coefficients. A large amount of information can be extracted from a powder diffraction pattern since the peaks are related to directional vectors in the crystal. This allows directional expansion and contraction to be mapped. If the crystal structure is known, one can measure changes in bond distances, angles and lengths as a function of temperature. High and low temperature stages are common in global diffraction facilities, with both powder and single crystal diffractometers. As a result, tens of thousands of crystal structures and powder patterns are collected annually under non-ambient conditions and hundreds of thousands of non-ambient determinations have been made historically.

The International Centre for Diffraction Data (ICDD®) has developed a 5-step method that extracts this information from global literature sources contained in the Powder Diffraction File databases. Editors have extracted the temperature of data collection and crystallographic parameters that can be used to calculate thermal expansion and contraction as a function of temperature. The method was designed to be dynamic, so that new literature data are immediately classified and entered into the database and every annual release of the database contains updated data. The method incorporates ICDD quality reviews and quality marks so the user can determine whether to use all data or the highest quality references. The method has the advantage that multiple references can be combined. Individual single point measurements can be combined to develop a measurement series that did not exist in the original literature references. A series of programming applications have been embedded with the database to allow the user to easily extract information, analyze the quality, and plot the data to determine thermal expansion coefficients.

Experimental Method

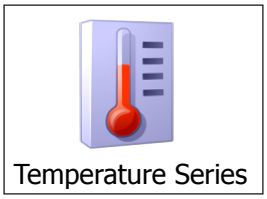
The Experimental Method uses critical fields that have been extracted by ICDD editors from the global literature. These fields can be displayed, sorted, edited and plotted. Typically one would want to plot a volumetric or directional parameter as a function of temperature. Once plotted, equations of state can be calculated from the plot to determine thermal expansion or contraction coefficients. A set of menu driven applications facilitate the selection and calculation process so that large quantities of data can be rapidly reviewed, plotted, and thermal expansion parameters calculated in just a few minutes.

Step 1: Material Selection

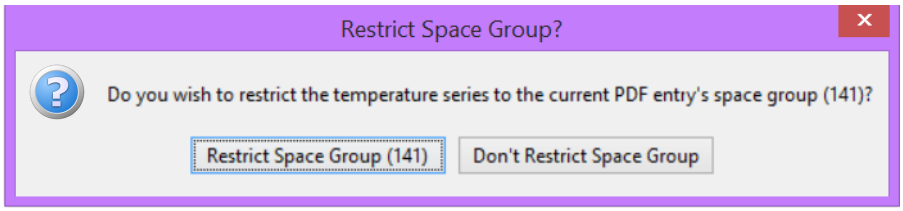


Materials can be searched and selected using any of the dozens of searches available with PDF® database products. Searches are available by clicking on the Search icon in the main toolbar. Name, formula, and common name searches are often the quickest and most productive searches.

Search results are provided in a table and any full entry can be found by double-clicking on the PDF number in the table. If the entry has the temperature series icon active in the tool bar, it is part of a temperature series collection. To access the collection, click on the Temperature Series icon.



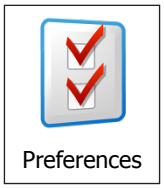
Materials are grouped by formula, so if there are multiple polymorphic forms you will see the following screen. This denotes that there may be more than one polymorph or a structural phase transition resulting in a space group change.



In the example above, we wanted to study the temperature series for a common material, ZrO_2. The entry that we selected was in space group 14, P_{21}/c, and is also known as the mineral Baddeleyite. If we don't restrict the space group, we also get entries for ZrO_2 and space groups (SG) 29, 57, 61, 111, 137 and 225. The latter space group corresponding to the mineral, tazheranite. To select another space group, just select an alternate choice from the search results.

ZrO_2 is known to crystallize in monoclinic (SG 14), orthorhombic (SG 29, 61, 62), tetragonal (SG 111, 137) and cubic forms (SG 225), and each form is polymorphic and is stable in a designated temperature range. With the space group restriction option one can study a specific structure or the entire system.

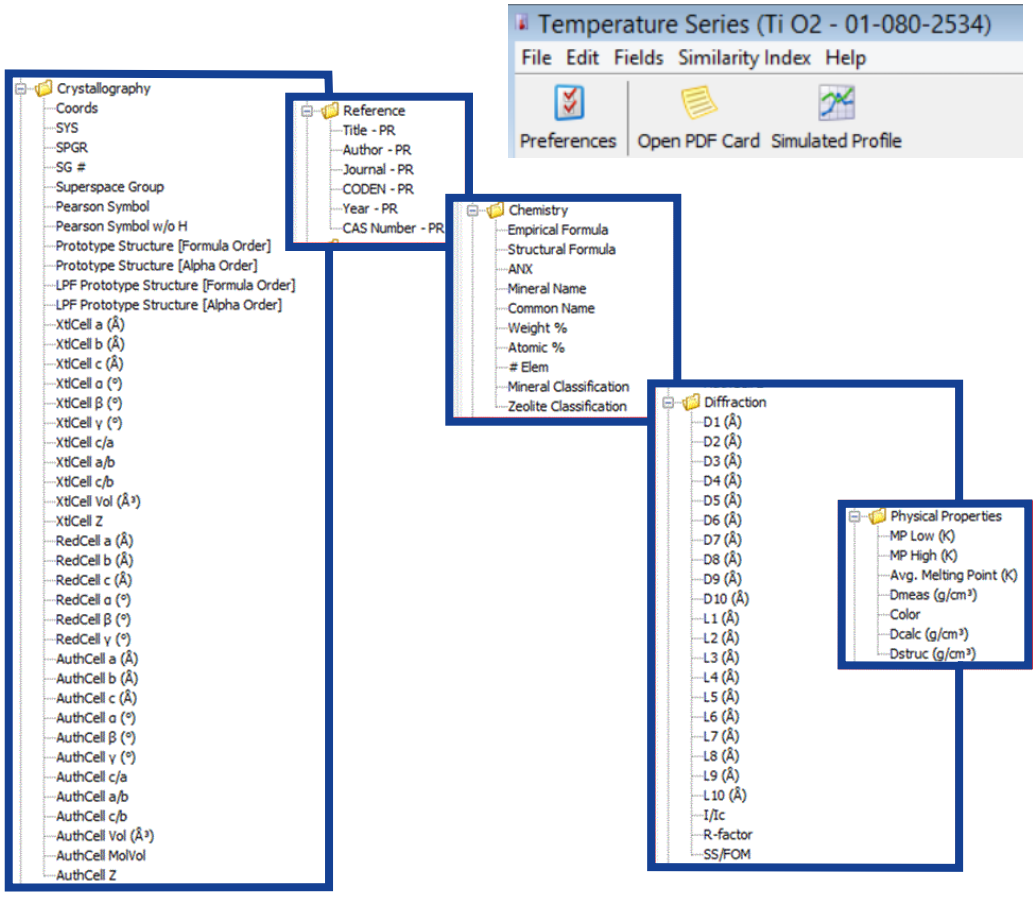
Step 2a: Selection of Parameters – Preferences



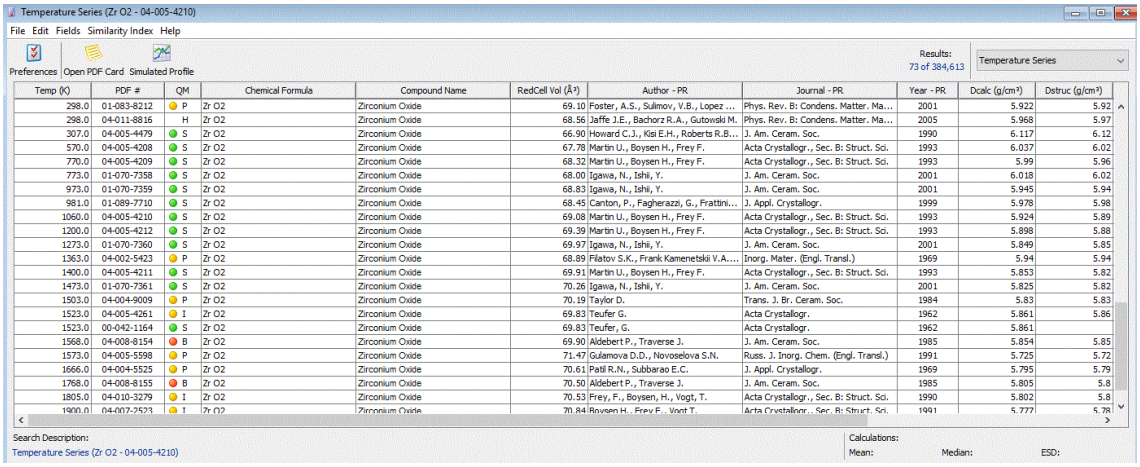
The figure above shows the "Preferences" icon on the menu bar.

When the temperature series button is selected, there is a default set of parameters. This default set contains parameters commonly selected to perform volumetric thermal expansion calculations. Parameters for the default set include, temperature of data collection, reduced cell volume, formula, quality marks, and literature reference. If these are the data that you want, you can proceed to the next step.

Current PDF products have over 100 display fields available for selection. In general, the user wants to look at various parameters as a function of temperature. To do this, use the "Preferences" menu selection in the database. Besides the default reduced cell volume, selections can also include crystal cell and author cell volumes; reduced cell, crystal cell, and author cell parameters; the ten most intense interplanar d - spacings; and the 10 longest interplanar spacings. The "Point and Click" menu selection can be used to select any of these parameters and display them in the "Preferences" table.



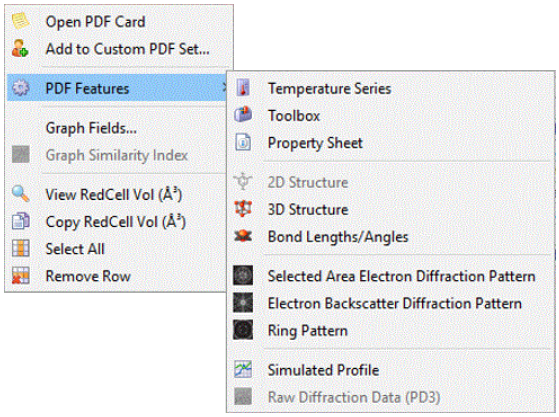
Step 2b: The Results Table



Temp (K)	PDF #	QM	Chemical Formula	Compound Name	RedCell Vol (Å³)	Author - PR	Journal - PR	Year - PR	Dcalc (g/cm³)	Dstruc (g/cm³)
298.0	01-083-8212	P	Zr O2	Zirconium Oxide	69.10	Foster, A.S., Sulimov, V.B., Lopez ...	Phys. Rev. B: Condens. Matter. Ma...	2001	5.922	5.92
298.0	04-011-8816	H	Zr O2	Zirconium Oxide	68.56	Jaffe J.E., Bachorz R.A., Gutowski M.	Phys. Rev. B: Condens. Matter. Ma...	2005	5.968	5.97
307.0	04-005-4479	S	Zr O2	Zirconium Oxide	66.90	Howard C.J., Kisi E.H., Roberts R.B...	J. Am. Ceram. Soc.	1990	6.117	6.12
570.0	04-005-4208	S	Zr O2	Zirconium Oxide	67.78	Martin U., Boysen H., Frey F.	Acta Crystallogr., Sec. B: Struct. Sci.	1993	6.037	6.02
770.0	04-005-4209	S	Zr O2	Zirconium Oxide	68.32	Martin U., Boysen H., Frey F.	Acta Crystallogr., Sec. B: Struct. Sci.	1993	5.99	5.96
773.0	01-070-7358	S	Zr O2	Zirconium Oxide	68.00	Igawa, N., Ishii, Y.	J. Am. Ceram. Soc.	2001	6.018	6.02
973.0	01-070-7359	S	Zr O2	Zirconium Oxide	68.83	Igawa, N., Ishii, Y.	J. Am. Ceram. Soc.	2001	5.945	5.94
981.0	01-089-7710	S	Zr O2	Zirconium Oxide	68.45	Canton, P., Fagherazzi, G., Frattini...	J. Appl. Crystallogr.	1999	5.978	5.98
1060.0	04-005-4210	S	Zr O2	Zirconium Oxide	69.08	Martin U., Boysen H., Frey F.	Acta Crystallogr., Sec. B: Struct. Sci.	1993	5.924	5.89
1200.0	04-005-4212	S	Zr O2	Zirconium Oxide	69.39	Martin U., Boysen H., Frey F.	Acta Crystallogr., Sec. B: Struct. Sci.	1993	5.898	5.88
1273.0	01-070-7360	S	Zr O2	Zirconium Oxide	69.97	Igawa, N., Ishii, Y.	J. Am. Ceram. Soc.	2001	5.849	5.85
1363.0	04-002-5423	P	Zr O2	Zirconium Oxide	68.89	Filatov S.K., Frank Kamenetskii V.A....	Inorg. Mater. (Engl. Transl.)	1969	5.94	5.94
1400.0	04-005-4211	S	Zr O2	Zirconium Oxide	69.91	Martin U., Boysen H., Frey F.	Acta Crystallogr., Sec. B: Struct. Sci.	1993	5.853	5.82
1473.0	01-070-7361	S	Zr O2	Zirconium Oxide	70.26	Igawa, N., Ishii, Y.	J. Am. Ceram. Soc.	2001	5.825	5.82
1503.0	04-004-9009	P	Zr O2	Zirconium Oxide	70.19	Taylor D.	Trans. J. Br. Ceram. Soc.	1984	5.83	5.83
1523.0	04-005-4261	I	Zr O2	Zirconium Oxide	69.83	Teufer G.	Acta Crystallogr.	1962	5.861	5.86
1523.0	00-042-1164	S	Zr O2	Zirconium Oxide	69.83	Teufer, G.	Acta Crystallogr.	1962	5.861	
1568.0	04-008-8154	B	Zr O2	Zirconium Oxide	69.90	Aldebert P., Traverse J.	J. Am. Ceram. Soc.	1985	5.854	5.85
1573.0	04-005-5598	P	Zr O2	Zirconium Oxide	71.47	Gulamova D.D., Novoselova S.N.	Russ. J. Inorg. Chem. (Engl. Transl.)	1991	5.725	5.72
1666.0	04-004-5525	P	Zr O2	Zirconium Oxide	70.61	Patil R.N., Subbarao E.C.	J. Appl. Crystallogr.	1969	5.795	5.79
1768.0	04-008-8155	B	Zr O2	Zirconium Oxide	70.50	Aldebert P., Traverse J.	J. Am. Ceram. Soc.	1985	5.805	5.8
1805.0	04-010-3279	I	Zr O2	Zirconium Oxide	70.53	Frey, F., Boysen, H., Vogt, T.	Acta Crystallogr., Sec. B: Struct. Sci.	1990	5.802	5.8
1900.0	04-007-2523	I	Zr O2	Zirconium Oxide	70.84	Boysen H., Frey F., Vogt T.	Acta Crystallogr., Sec. B: Struct. Sci.	1991	5.777	5.78

This is a "Results" table for tetragonal zirconia, space group 137. The temperature is displayed in the column on the far left and the reduced cell volume is displayed in a middle column.

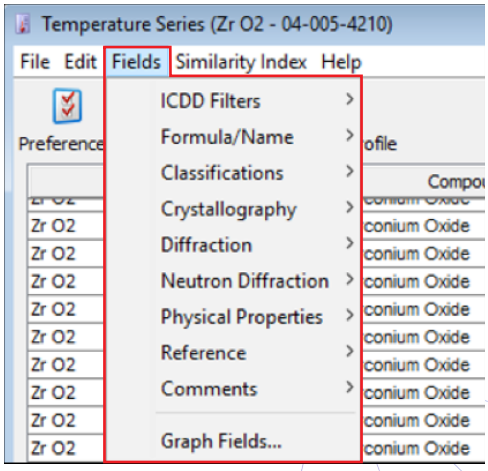
Once a materials system has been selected, the "Results" table compares the selection of parameters (Step 2a) for every available entry. Data in a "Results" table can be sorted by clicking on the top of each column. This allows the user to order the data numerically or alphanumerically, either low to high (1 click) or high to low (2 clicks). There is a right hand menu of options (shown on the right) that can be used for additional analyses. One very useful option is to remove an entry from the table. In thermal expansion calculations it is often useful to eliminate poor quality data. It is also common to see outliers in some data sets where a poor data point may occur because of poor temperature calibration of the furnace or cryogenic cooler. (See Discussion on pages 11-12).



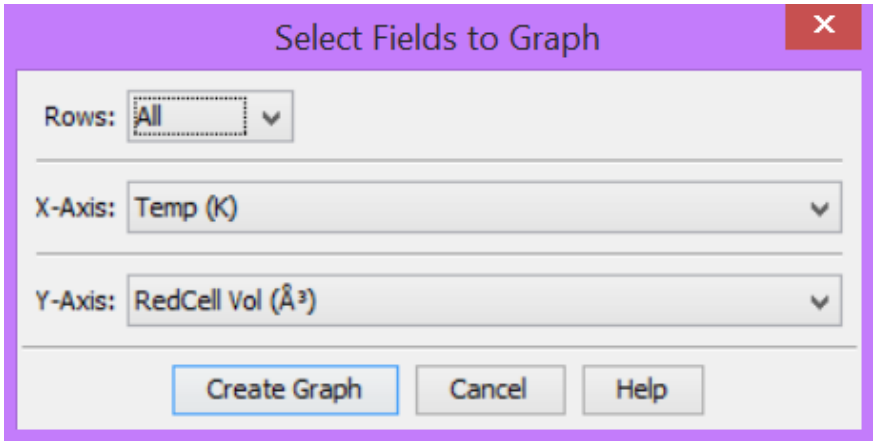
Step 3: Plots

Once you have set up the desired parameters in "Preferences", your search will output a "Results" table as described in Step 2b. In the "Results" table, any alphanumerical value can be plotted. For a volumetric thermal expansion you would want to plot the temperature on the x-axis and the volume on the y-axis.

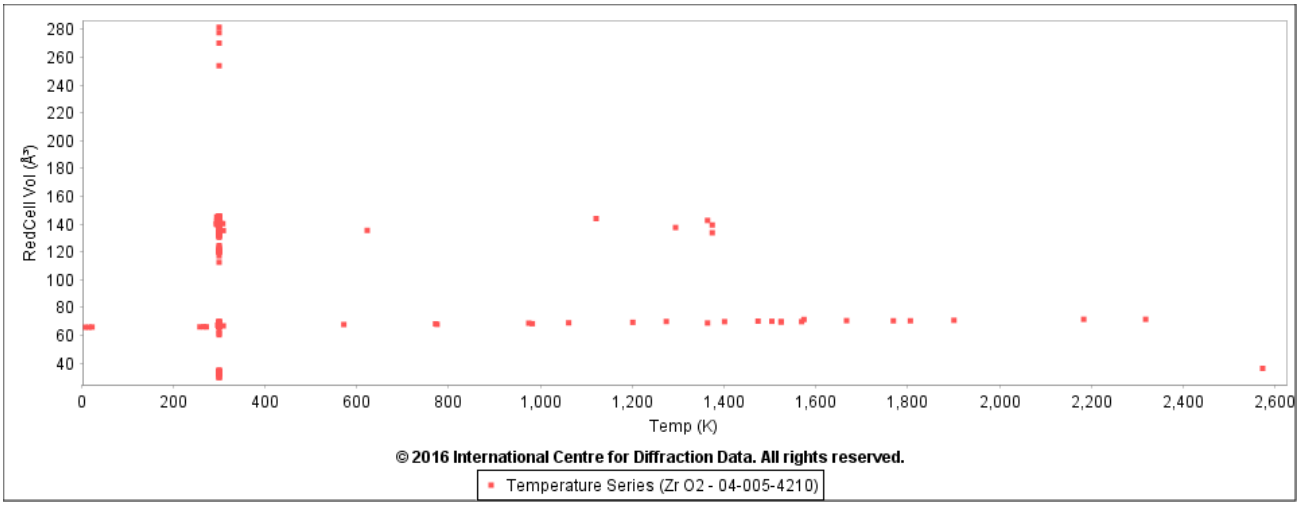
To do this, go to the top of the menu bar and select "Fields". This will show a drop down menu that has several options (similar to "Preferences") for fields to add to your "Results" table. At the bottom of the menu is an option for "Graph Fields". Select this option.



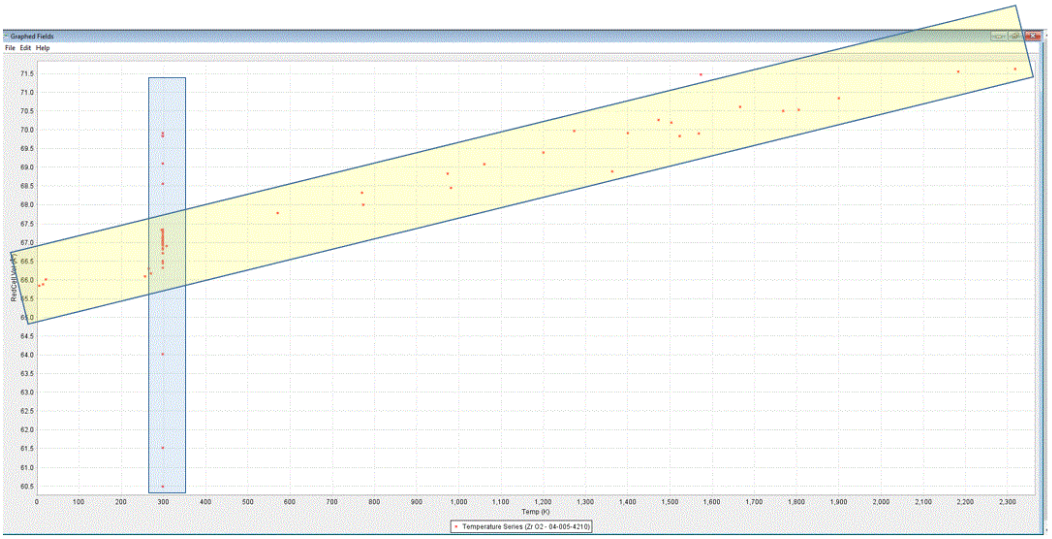
The selection of "Graph Fields" will produce the following display. For temperature series there is a default of temperature vs. reduced cell volume. However, by using the arrow on the far right any column displayed in your "Results" table can be selected.



Sometimes you get an easy to understand plot and can proceed to Step 5. Most of the time you get a complex plot that looks like the one below and you will need to go to Step 4.

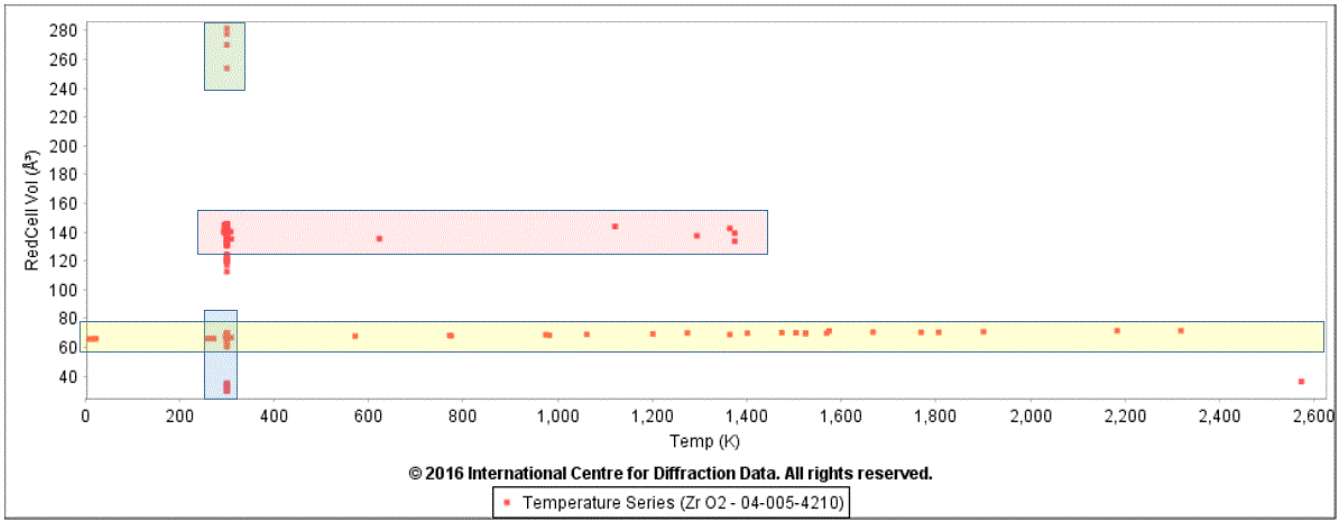


In general, these complex plots have more than one series of data. You may have temperature data, pressure data, or temperature and pressure data. Data quality may be highly variable resulting in a scatter plot versus an easily recognizable trend.



For example, if we continue with the tetragonal zirconia, space group 137, one might see at least two sets of series data from the 73 entries that comprise the series. The possible sets are highlighted in yellow and blue. The yellow set has a changing volume with temperature and is likely to be the data of interest in a thermal expansion determination. The blue set has a changing volume at constant temperature and is likely to be a pressure series.

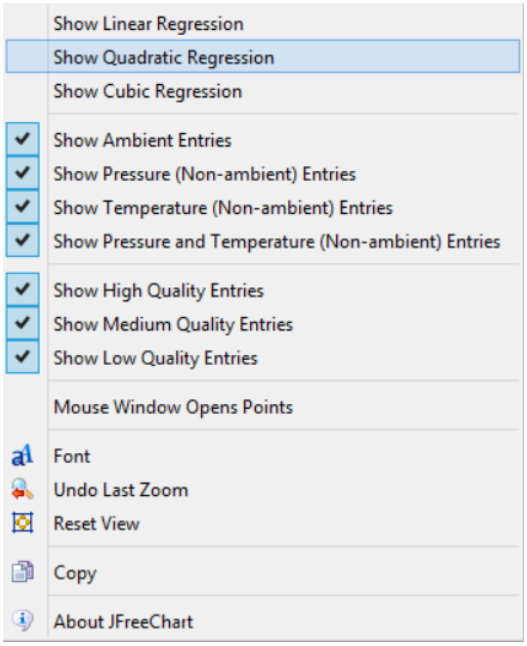
[Note: At this time we do not have a pressure series graphics output option. The pressure can be extracted from the editors comment section and plotted versus volume. We expect to have a pressure series graphics option in the near future.]



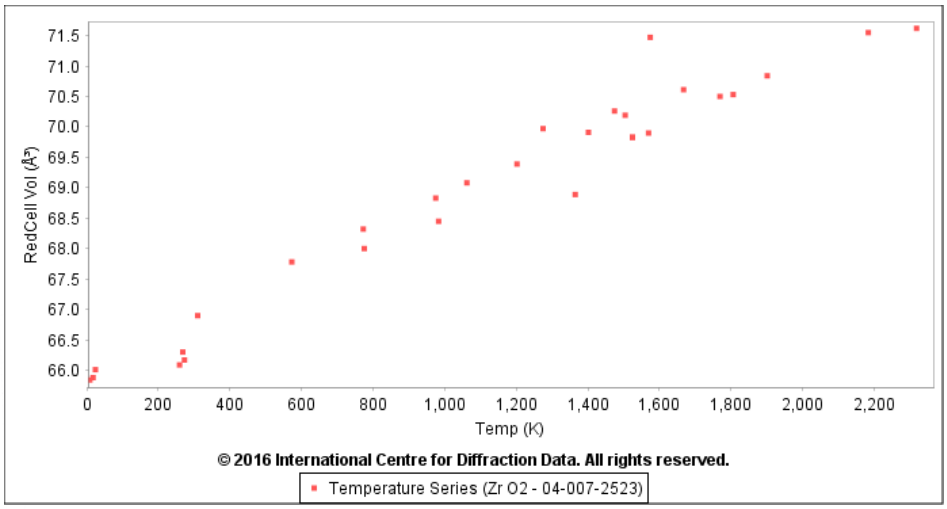
In Step 1, if we did not restrict the space group, then there would be 221 entries for ZrO_2. A plot of these entries is shown above and there are at least four series of data. We see the prior blue and yellow series from tetragonal zirconia (SG 137), as well as a green (cubic, SG 225) and red series (monoclinic, SG 14). The increase in volume with temperature is not as apparent as the two earlier graphs because of the larger y-axis scale.

Step 4. Quality Review

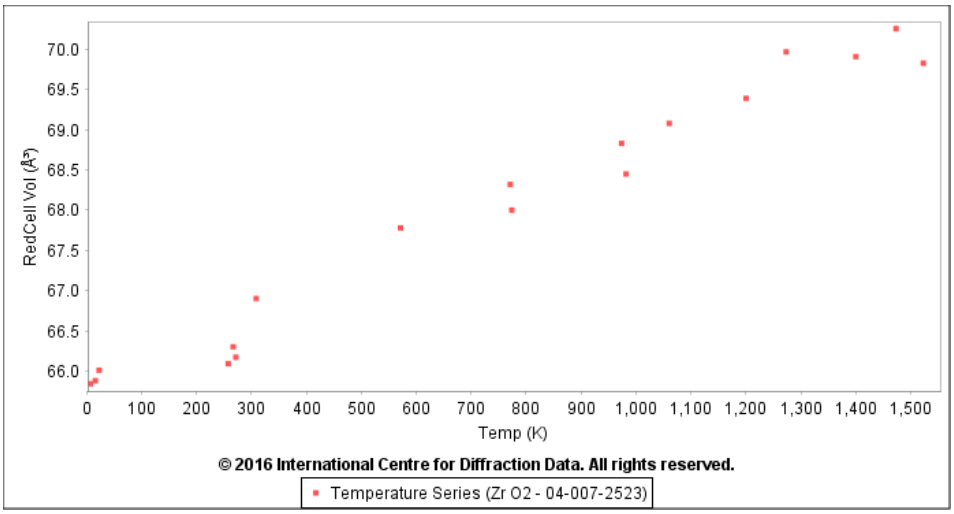
To help you perform a quality review, the ICDD has generated a menu of applications whereby you can quickly select and/or deselect several groups of data depending on types and quality. This menu is under rapid development and is not available in 2015 products. These applications are meant to interface with the plotting graphics so that you can quickly choose the data that are relevant to your research. This menu is activated by a right-click of the mouse on your screen display whenever you have a temperature plot.



This menu is an application short cut. In 2015 products you can achieve the same graphs by sorting and selecting the data from the "Results" table generated in Step 2b. You would want to remove rows of inappropriate data and then replot the data (Step 3). To choose an option just click on the arrow.



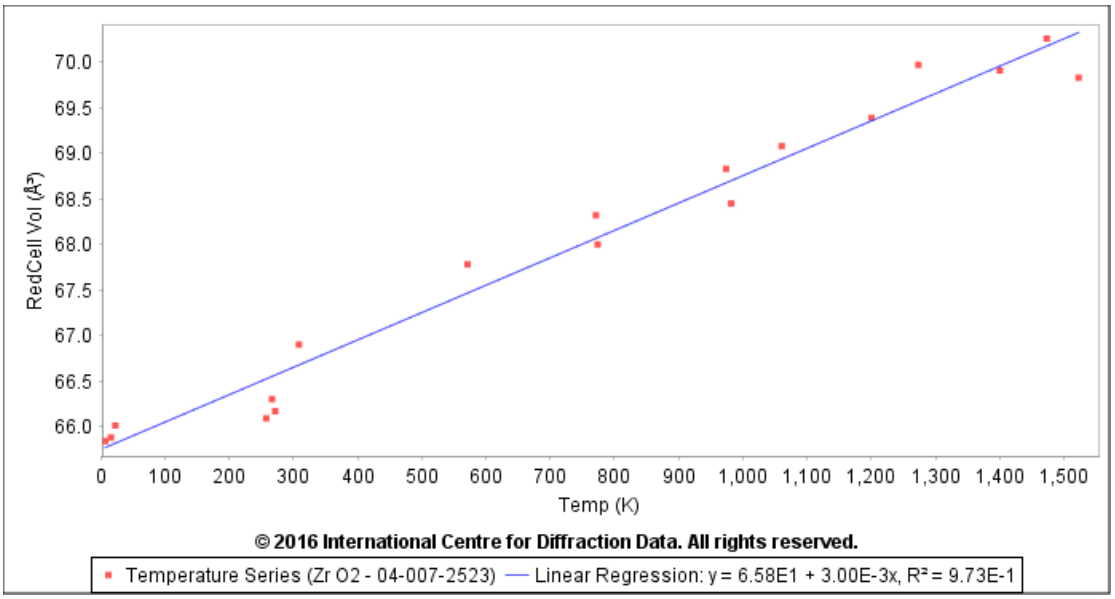
The selection above removed ambient data and pressure series data. We can now see an obvious trend where the volume is increasing with temperature. However, the data exhibits a significant amount of scatter.



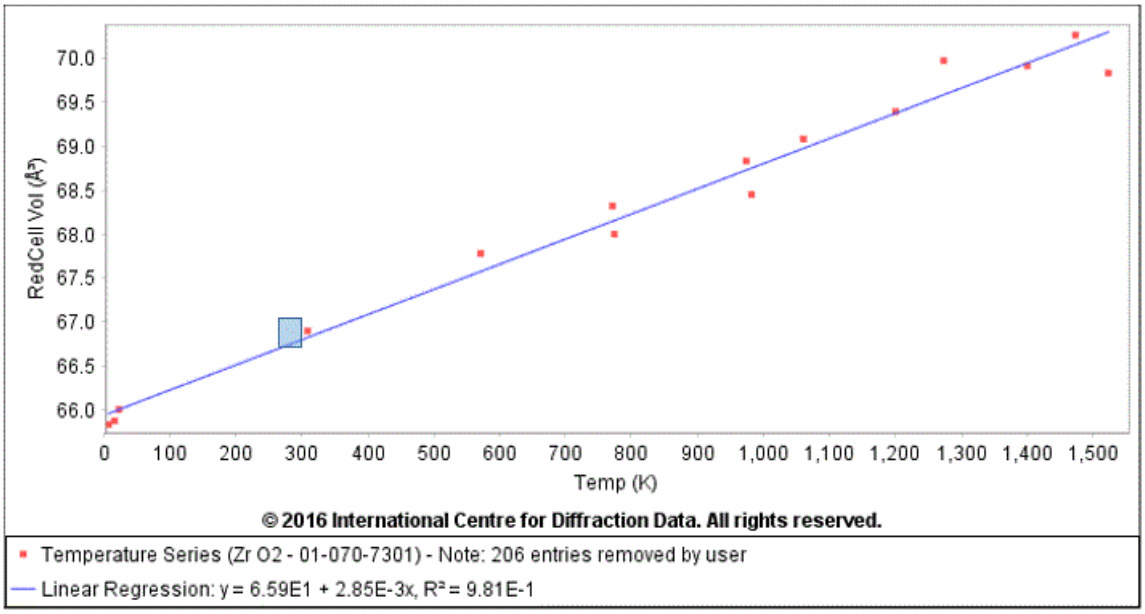
We reduce the scatter by using ICDD's quality review system so that we include just the high quality references where the data sets are without any known identified errors. This was done by eliminating medium and low quality entries by using the "Option" menu shown above. Four "B" quality references were removed from the previous graph where the editor mentions that the data were extrapolated from a graph in the literature and the cell and volume parameters were not very precise. This step also eliminated "P" (Prototyping) quality references, which were determined from prototype methods and the cell parameters did not have experimental error determinations.

Step 5. Calculation

Once you have selected and quality reviewed your data, you can now calculate equations of state and coefficients of expansion. Once again there is a series of menu driven options to assist in the calculations. They can be retrieved by right-clicking on the plot of interest. Linear, Quadratic and Cubic regression analyses can be performed singularly or by comparison with the plotted data. Each calculation is accompanied by a regression analysis fit shown at the bottom of the graph. Equations of state are also shown, from which thermal expansion coefficients can be calculated.



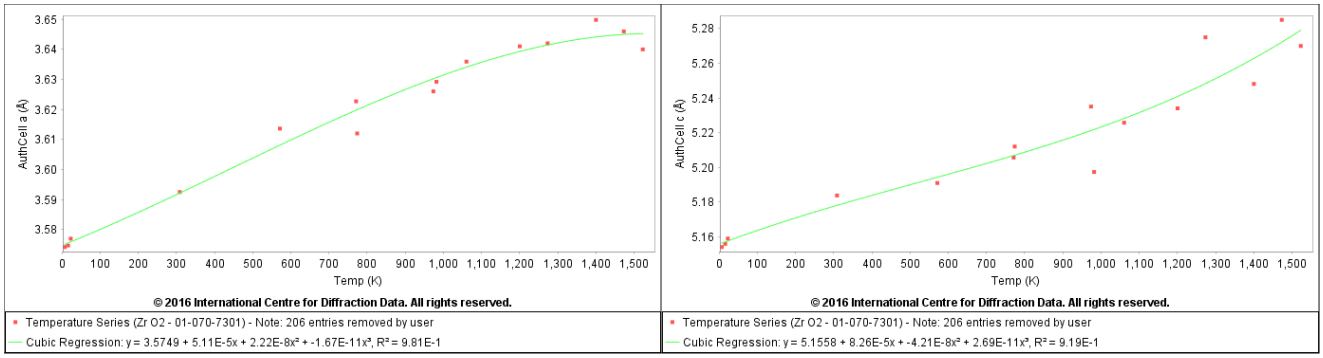
This result used 19 entries that were determined from six different literature sources. The six lowest temperature determinations were from a single source. Four independent "Star" quality room temperature determinations averaged a unit cell volume of 66.9(2) Å3. This suggests that the three determinations at ~270 K may have been atypical. In the following plot, these data were removed.



The final graph for tetragonal zirconia is shown above. While the "Star" quality room temperature data are not plotted, they are consistent with the equation shown and would be in the highlighted box.

$$\alpha_v = \frac{1}{V}\frac{dV}{dT}$$

The equation of a linear thermal expansion volume coefficient is shown above. The α_v coefficient using the volume at room temperature would be 42.6 ppm.



If the "a" and "c" cell axes are added to the "Results" table via Step 2b, then Steps 3-5 can be used to generate thermal expansion equations for the "a" and "c" cell axis. These are produced in the above graphs.

In this case a cubic regression analysis shows some anisotropy particularly at higher temperatures.

Discussion

This method has been reviewed by the scientific membership of the ICDD and specifically the members of the Non-Ambient Subcommittee of the ICDD's Technical Committee. Most members of this subcommittee are practitioners of non-ambient methods and represent experts in the field. The following review represents some of the discussion among the members.

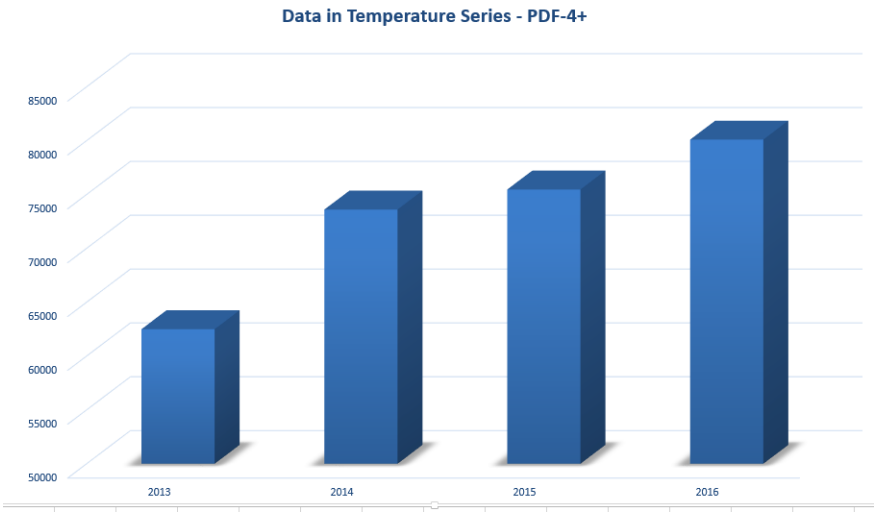
The method as described has some obvious advantages:

1) Multiple literature sources can be combined.
2) The method integrates quality review and offers quality improvements in the data series and calculated results.
3) Single point determinations can be used in a series or to make a series.
4) The method is based on classification and annual review – it is dynamic and constantly updated.
5) The method is general purpose and can be applied to over six thousand material systems.

The method also has some disadvantages:

a) Results are calculated ad hoc, not tabulated, and more effort is required by the user.
b) Quality is based on crystallographic correctness and agreement to fit; experimental procedures and best practices are not considered.
c) Most lattice determinations measure precision, not accuracy.
d) The results may not account for microstructure influences.

There was significant discussion about the merits of a dynamic system versus tabulated data. A major consideration for having a dynamic system is the large volume of data being produced globally on an annual basis, with a significant fraction of temperature variable data. This large annual volume pushed ICDD towards developing a system that could be rapidly and automatically updated and integrated with our annual editorial reviews.



Data entries that have been cross-referenced into temperature series data for the PDF-4+ Releases 2013 through 2016. In 2016 there were 6,340 series containing 80,097 data sets. Every year new data are added and new series are created from research results being published from around the world.

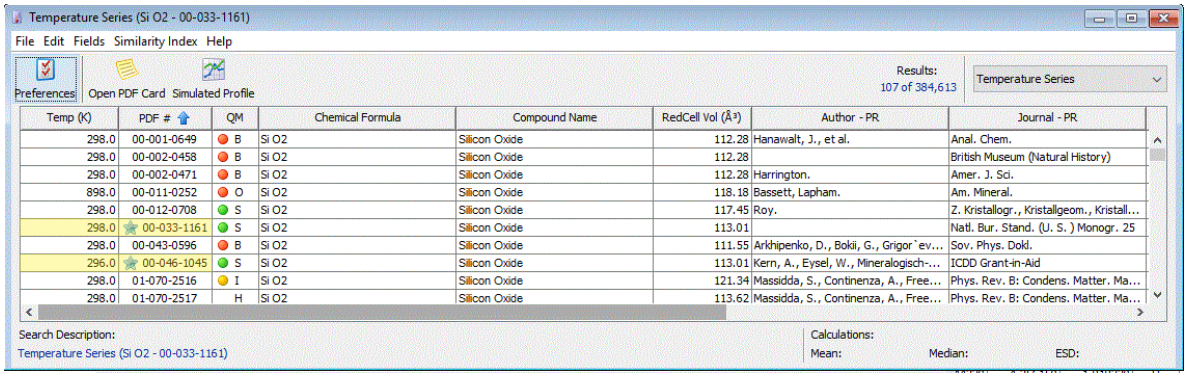
The ICDD quality review system is quite complex and considers whether the stated chemistry matches the crystallography and measured diffraction pattern and physical properties. The system also looks for and records several types of systematic errors (such as specimen displacement) commonly found in diffraction analyses. This system does not consider best practices in non-ambient experiments, nor does it consider the equipment quality and calibration. A historical review of non-ambient methods would find a tremendous range in equipment, heating and cooling methods, atmosphere control, and thermal gradients. There is also a wide range in experimental methods where specimens might become insulating or conducting, affecting whether the system is at thermal equilibrium. We strongly suspect that poorly calibrated experiments are the cause for many outliers observed in the measurement plots. While we often see outliers associated with low quality data, there can also be outliers with high quality data and we suspect this is due to experimental or specimen errors that were not captured in the database and quality review. When in doubt, we suggest that the user also review the original literature reference and evaluate the experimental conditions.

For example, in this technical bulletin we have used two systems for analysis, tetragonal zirconia and corundum. The data for corundum are shown on the cover and display much less variability than tetragonal zirconia. Tetragonal zirconia is used in many industrial applications because of the mobility of the oxygen atoms at elevated temperature. A controlled atmosphere would be required to maintain stoichiometric equilibrium in any thermal expansion experiment, but atmosphere is rarely specified and infrequently measured in most literature. In the case of zirconia, loss or gain of oxygen would change the stoichiometry and unit cell parameters. We suspect that this may be a source of variability displayed in the "a" and "c" cell axis thermal expansion data, despite the fact that only high quality references were used.

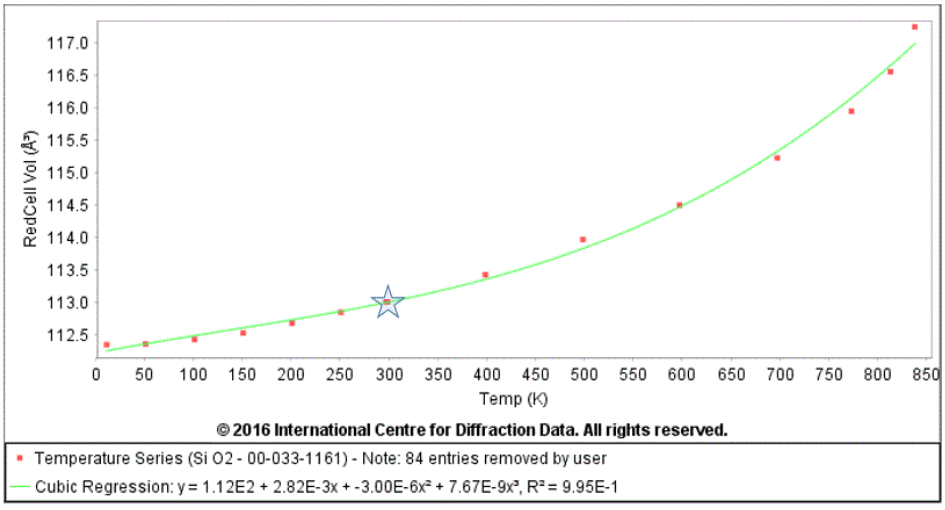
Microstructure can also influence thermal expansion calculations. Several materials, particularly mixed oxide ceramics, have been synthesized by low temperature sol-gel methods. There are literature references where a material was heated and then rapidly quenched. In these situations, thermodynamic equilibrium was rarely achieved. The materials may not be stoichiometric and defect structures are often evident. While the ICDD editors frequently extract critical synthesis and treatment methods in the editor's comments, we often do not have enough information to know if high purity defect free crystalline materials have been produced. If the material has significant defects then the energy input during the heating process may go towards annealing defects, versus thermal expansion. If you have significant outliers in a thermal expansion plot it is prudent to read the ICDD editors comments.

Accuracy and Precision – Blue Star Data

Typically, cell parameter refinements tell the user about precision, not accuracy. In the Powder Diffraction File, the ICDD always standardizes cells and verifies cell parameter calculations. In Release 2016, the ICDD has added an accuracy indicator mark, a "Blue Star", which shows determinations made by using a National Institute of Standards and Technology (NIST) certified internal standard for calibration and having all the other qualifications for a "Star" quality pattern. These patterns are both accurate and precise and can be used to "anchor" series data calculations. These patterns are relatively few in number, being a subset of "Star" quality patterns, but often are common materials used in engineering design. If the user has a wide selection among ambient data points, we recommend using "Blue Star" data points. The "Blue Star" is shown in the "Results" table next to the PDF number.



For example, looking at the temperature series for quartz we have two entries designated with a "Blue Star". PDF 00-033-1161 was produced by NIST and PDF 00-046-1045 was produced through an ICDD grant. Both data sets were calibrated with a NIST certified internal standard.



Using Steps 2-5, the above volumetric expansion graph for SiO2 was produced from 14 entries with five different literature sources. The low temperature data and high temperature data are from different sources and the two "Blue Star" references represent ambient determinations. But all five sources can be described by a single equation for volumetric thermal expansion over the 10-800 K temperature range with an R^2 fit of 99.5%.

Using Quality Marks and Quality Symbols

Quality Symbol	PDF-4+ Quality Mark	PDF-4+ 2016 No. of Entries	
S	Star	85,601	High Quality Data
R	Rietveld	4,303	
G	Good	54	
I	Indexed	130,563	Medium Quality Data
C	Calculated	4,604	
P	Prototype	64,557	
M	Minimal Acceptable	75	
B	Blank	52,806	Low Quality Data
O	Low Precision	36,037	
H	Hypothetical	6,013	

The ICDD editorial review system goes through >100 statistical checks to verify data quality. The system currently has 10 quality classifications. Some are reserved for experimental powder data, and some for experimental single crystal data, calculated data, and Rietveld refined data. Two quality marks are reserved for non-crystalline data, "Good" (G) and "Minimal Acceptable" (M), and these marks denote the quality and amount of supporting data for that particular reference. Non crystalline data sets, by definition, do not have unit cells and cell volume parameters and are unlikely to be in a temperature series. If a Rietveld or calculated data set does not meet the quality standards of the quality mark, the entry receives a lower quality mark or it is not published.

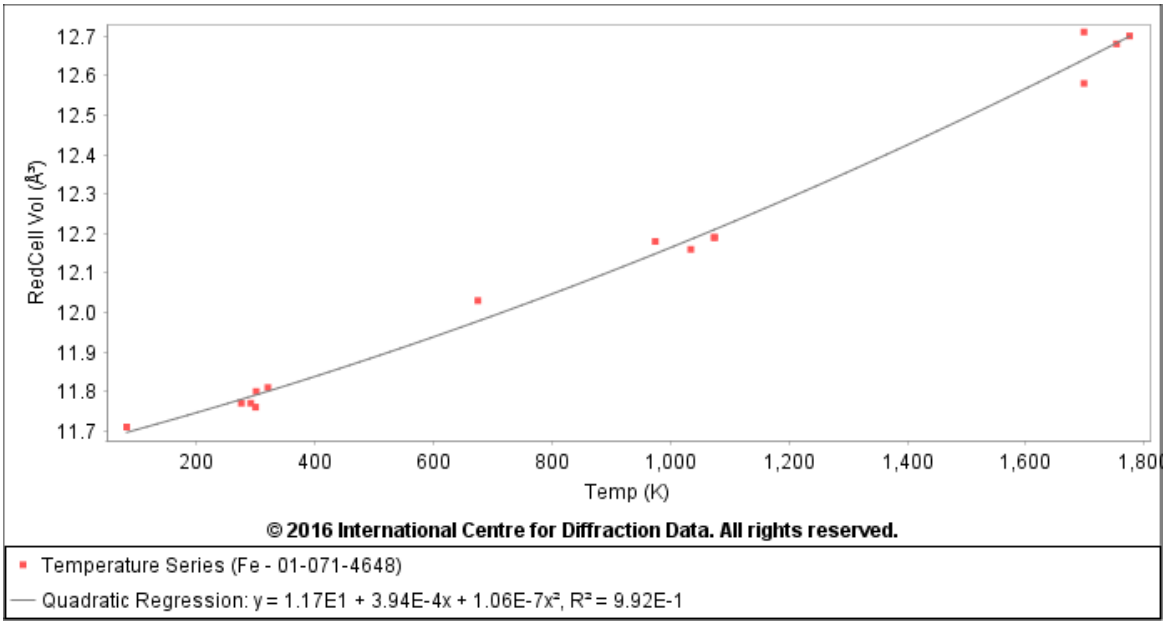
The quality review Step 4 has embedded applications that let the user select groups of high, medium, and low quality data. Whenever possible, we recommend that the user select only high quality data.

Medium quality data should be used with caution. These data have exhibited some statistical error. The error analyses are cited by the ICDD editors in the editor comment section of the entry. The quality review menu allows you to toggle the medium quality off and on to see how it influences your graphic results. The type of error should be a major factor in your review of medium quality data. A common error is when the structural density does not match the calculated density. One should evaluate the magnitude of the difference and consider that the error probably suggests that the stoichiometry is not well defined and propagates to size and volume measurements. Alternatively, sometimes medium quality data were produced on older, less precise equipment; an example is given below for the case of iron. The data may be fine but lack the precision of a modern analysis. It should be kept in mind that NIST Standard Reference Materials® (SRM's) for instrument calibration were not produced until the 1980's and not in common usage until the 1990's. In general, selecting high quality data reduces the number of entry points and results in a higher regression coefficient R^2 value.

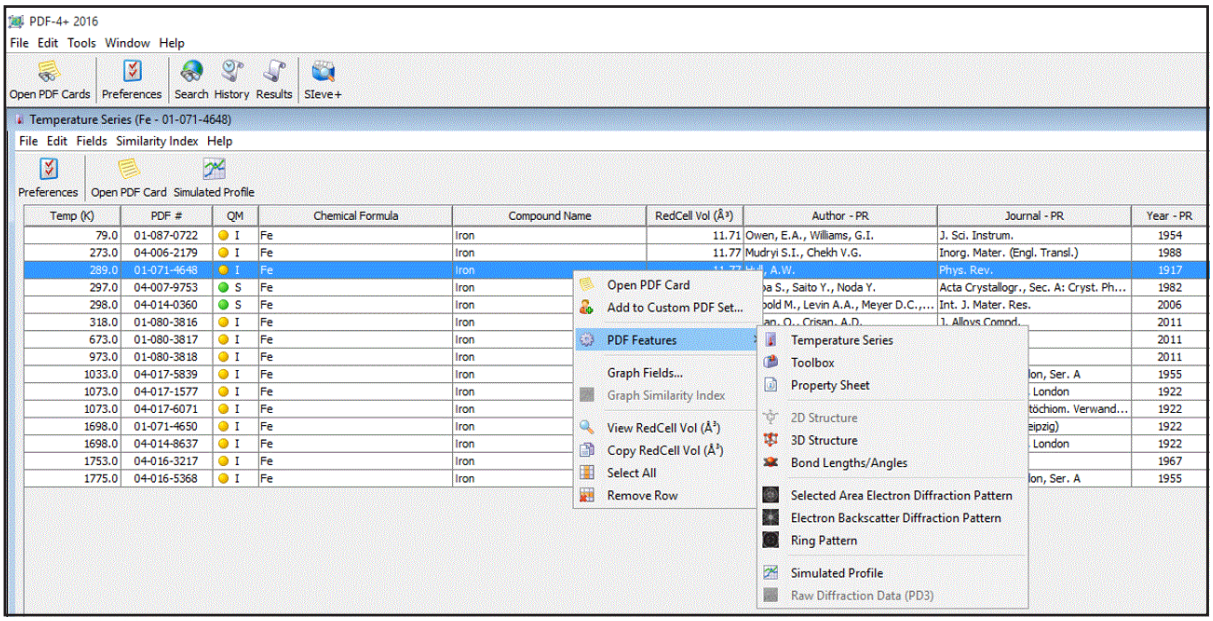
Using Hypothetical and Prototype Data

The Powder Diffraction File contains Prototype (P) data and a small amount of Hypothetical (H) data. Prototype data are often extracted in the literature from series information. Data can be editorially extracted from a table or graph or may be extrapolated from an isostructural compound. Very few graphs represent the high degree of precision that are found in experimental diffraction data. In general we recommend that this data not be used unless the user has examined the individual entry and determined the precision of the data (cell parameters and esd's). In many cases, the esd's are not available. Similarly, hypothetical data rarely reflect the precision of experimental data; esd's may not have physical significance even if calculated.

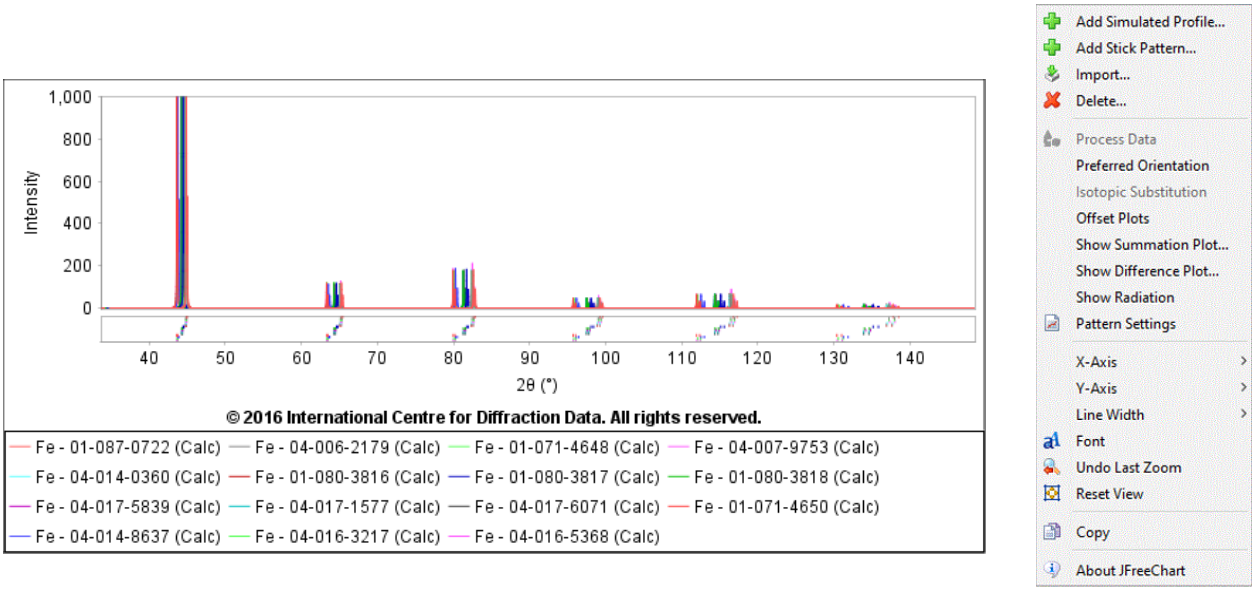
Options



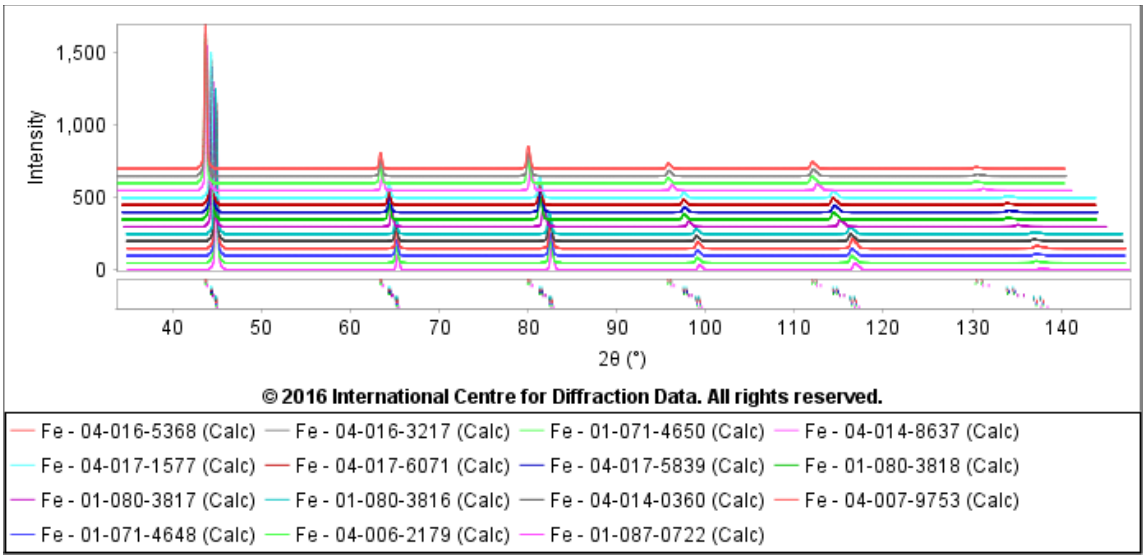
Following Steps 1-5, we can produce a graph on the thermal expansion of iron. This graph includes predominately medium quality data since high quality data were not available except at room temperature. The "Results" table includes data from 10 literature sources, including the historic A. W. Hull work, from General Electric in 1917, and some of the earliest high temperature determinations by the Swedish team of Westgram and Phragmen (U. of Stockholm) in 1922. This was one of the first crystal structures determined by powder diffraction methods.



From the "Results" table, Step 2b, right-click brings up a series of options under the field, "PDF Features", as shown above. The user can select visualization software, various pattern simulations, bond length, and angle calculations. If multiple entries are selected (using Ctrl or Shift), then multiple displays will be shown. In this manner, we can produce a temperature series plot of diffraction patterns. The entire table is selected and then the simulated profiles are chosen by a point and click option.

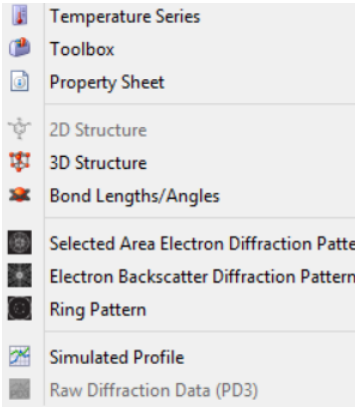


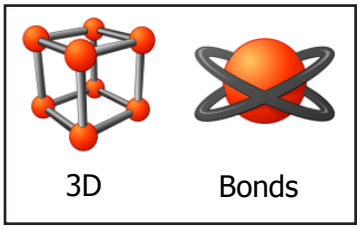
The above plot shows all the patterns in the selected temperature series superimposed on a single plot. Once again, there are a series of pattern options that can be selected from a right-click in any PDF entry number producing the menu shown on the right of the plot.



To produce this plot, we sorted the above data table by high to low temperature by clicking on the column header for temperature. Then the "Offset Plots" option was selected and the user enters a percentage offset, 5% in this case. The plot above corresponds to the thermal expansion graph with the highest temperature at the top and lowest temperature on the bottom. The shifts in the tick marks correspond to the thermal expansion of individual interplanar d-spacings.

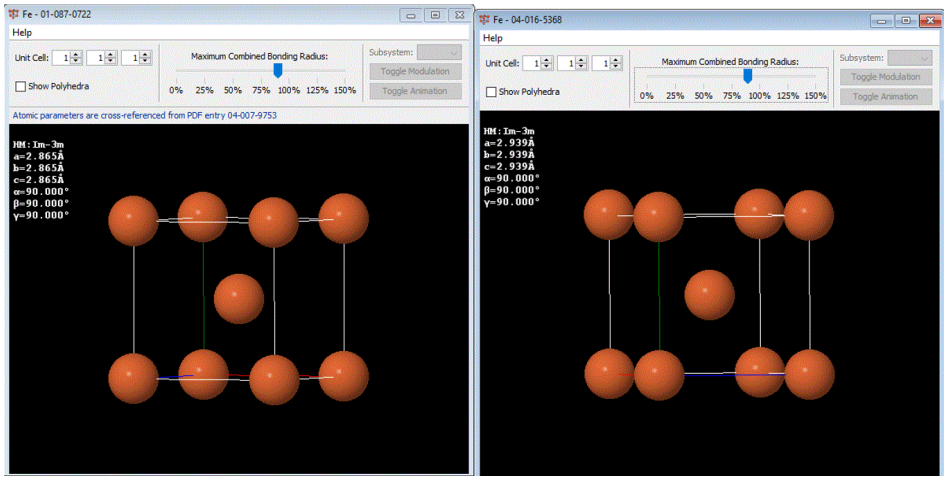
Calculating Changes in Bond Lengths and Angles with Temperature





From the "Results" table, Step 2b, the above options can be selected from "PDF Features" on the right-click menu. The selection includes 3D Structures and Bond Lengths/Angles.

Clicking on either of these icons in this menu or on the main toolbar of the individual entry will produce molecular graphics or an interactive table of bond lengths and angles. If a group of entries are chosen, then a series of displays and tables will be shown.



In the above molecular display we have the body centered cubic unit cell of iron, from determinations at 79 K and 1775 K. The unit cell dimension and space group are shown in the far left corner. A right-click on these figures produces an "Option" menu. The structure can be seen in various display types including stereographic projections and surface plots. Bond lengths and angles can be measured interactively on the graphics or seen in tabular form if the bonds icon is selected.

The Powder Diffraction File contains bond angle and length data and has molecular graphics as a function of temperature. These data can be extracted from individual files or in selected groups through the "Results" table. We currently do not have programming applications that automatically extract bond angle and distance data, and put it into the "Results" table to plot it. We do have the ability to take over 100 fields, as shown in Step 2a, add them to the "Results" table and then plot the data.

Conclusion

We present a 5-step method for extracting and calculating thermal expansion data and coefficients. The method is relatively fast through the use of tabulated cross-reference tables and embedded software applications that evaluate quality and produce calculated results.

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