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Powdercell Program Instructions for Analyzing XRD Pattern: By using a Sample of Aluminum Oxide (Al_2O_3) and Strontium Carbonate ($SrCO_3$) and Their Composite

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Abstract: The characterization of crystalline materials through X-ray diffraction (XRD) is a fundamental technique in materials science, enabling the determination of phase composition, crystalline size, and lattice parameters as well as program PowderCell is a crystallographic tool for visualization of crystal structures. This detailed study focuses on the use of PowderCell, a sophisticated software tool for simulating and analyzing XRD patterns, to investigate the crystallographic properties of pure aluminum oxide (Al_2O_3), strontium carbonate ($SrCO_3$), and their composite. This article provides step-by-step guidance on using powderCell program for the analysis of XRD patterns of these materials and it covers the setup of the software, input of crystallographic data, simulation of XRD patterns, refinement of the patterns against experimental data, and interpretation of the results. BDEC software was used to find the literature data on Aluminum oxide (Al_2O_3) and Strontium Carbonate ($SrCO_3$). By following this comprehensive guide, users will be able to efficiently utilize PowderCell to perform accurate and insightful XRD analyses.

Keywords: *Powdercell; BDEC software; X-ray diffraction; Experimental and literature data*

1. Introduction

In 1912, Max von Laue and colleagues demonstrated that for X-ray wavelengths (λ) comparable to the distance between a crystal's surfaces, crystalline materials function as a three-dimensional diffraction gratings grid system (Friedrich *et al.*, 1913). Wilhelm Conrad Roentgen's discovery of X-rays in 1895 paved the way for significant advancements across all scientific fields and the creation of new technological and medical applications (Röntgen, 2020). Nowadays, X-ray diffraction is a widely used method for studying atomic spacing and crystal formations. The basis of X-ray diffraction is the constructive interference of a crystalline sample with monochromatic X-rays. A cathode ray tube, filter, and collimated are used to produce the X-ray, and monochromatic radiation as well as concentrate and

direct to the specimen respectively (Cheng *et al.*, 2024). In X-ray diffraction, the specimen spins at an angle 2θ along the collimated X-ray beam direction, and the detector, which is sited on an arm capable of collecting the diffracted X-rays, rotations at a 2θ angle. A goniometer is used to rotate and maintain the angle of the specimen. The angles range mostly ~5º-70º at which data are acquired for common powder patterns specified in the X-ray investigation (Bunaciu *et al.*, 2015; Mishchenko *et al.*, 2018; Will, 2006).

Generally, X-ray powder diffraction is mostly used to determine unknown crystalline compounds, such as inorganic chemicals & minerals (Jabbar *et al.*, 2023). For research in geology, physics, chemical and microelectronics engineering, material engineering, biology, etc. it is essential to determine unknown solids (Epp, 2016; Moore & Reynolds, 1989). Additional uses include measuring sample purity, determining unit cell dimensions, describing crystalline solids, and recognizing difficult-to-identify fine-grained minerals, such as mixed layer clays. By using specific methods, X-ray diffraction (XRD) can be utilized to analyze thin film, and Nanoparticles specimens, measure the concentrations of elements in the specimen, investigate the structure of crystals employing refinement, and evaluate textural features like grain orientation in polycrystalline specimens (Mishchenko *et al.*, 2018). An advanced, non-destructive technique for examining a broad range of substances is XRD. These materials also include liquids, minerals, nanoparticles (Shitu *et al.*, 2023; Aldwayyan *et al.*, 2016; Aldwayyan *et al.*, 2013), metallic, polymer, drugs undoped and doped compounds, thin-film coatings, catalysts, as well plastics, ceramics, solar cells and batteries, and semiconductors. Numerous sectors, such as electronic devices, energy production, the aerospace sector, and numerous others, find effective uses for this approach. Every property related to the fundamental structure of the sample may be identified with ease using XRD analysis, including the existence of defects in a particular crystal, its size, surface texture, and degree of crystallization, as well as its stress resistance (Moore & Reynolds, 1989).

However, the PowderCell project was launched in 1992, and the structural refinement using this software was quite challenging for new researchers. A refinement operation was completed after many hours, but no tool was available to see the final crystal structure suggestion or distinguish between the experimental and refined powder patterns (Nolze, 2002). The screen just displayed a large number of running numbers and notations. However, it was decided to develop a crystallographic tool that would allow for the easy computation of diffraction patterns in addition to the observation and modification of crystal structures. In many cases, talks revealed that crystallographers who focused on solving crystal structures had preconceived notions about possible structural models (Nolze, 2017). They were searching for a tool to investigate the change in the powder pattern that was produced by translating and rotating molecules within the unit cell to build a structural model. Furthermore, non-crystallographers exhibited their own programs for diffractogram simulation during Residual Stress conferences, primarily for certain assumptions (e.g. cubic materials or metals, no convolution line intensities, no common export filters, etc.). However, W. Kraus and G. Nolze's launch of PowderCell was inspired not only by the software's excellence but also by their extensive effort spent developing a tool that ought to be freely accessible to all scientists, engineers, researchers, and technicians. There were occasions when an active assistant helped solve special problems. Examples of such specialists include M. Wendschuh-Josties (who calculated anomalous scattering factors for various radiations),

U. Mueller (who elaborated subgroup relations), G. Reck (who examined the interaction between powder data and single crystal structure investigation), and B. Mueller (who handled size and strain). Programming's first goals were creating manual movement models of structures or examining how crystal structure affected data by comparing estimated powder diffraction patterns with experimental results (Kraus & Nolze, 1996).

The objectives of this article are: to use a powdercell program for analyzing the XRD pattern of pure compound Aluminum oxide (Al_2O_3), and Strontium Carbonate ($SrCO_3$) as well as composite. A brief explanation of the idea behind XRD, the features of the equipment utilized, and sample preparation is helpful. Firstly, to investigate the orientation, composition of elements, and crystallography structure of pure compound Al_2O_3 and $SrCO_3$ as well as their composite. BDEC source was used to find the literature data as well as powerdercell tool was utilized to compare the literature and experimental data of Al_2O_3, and $SrCO_3$. Moreover, also observed the variation after refinement. In the end, find the volume percentage of Al_2O_3, and $SrCO_3$ by powdercell and also determine the variation in volume percentage of these compounds after refinement.

2.0 Experimental methods

The present work was done in the PPGEM, Department of Material Engineering, University of Sao Paulo-USP Lorena Campus, Brazil. The highest purity and good quality chemicals were utilized for XRD, Aluminum oxide (Al_2O_3, purity > 99.5%) and strontium carbonate ($SrCO_3$, purity > 99.0%). Al_2O_3 and $SrCO_3$ were ground to a fine powder using an agate mortar and pestle to get pure samples. To guarantee homogeneity, a complete grinding process was also used to create a mixture of Al_2O_3 and $SrCO_3$. The digital electronic balance, and stainless steel scoop spatula were used to measure the quantity and lift the sample respectively.

First of all, take a specific quantity of sample you want to analyze. To eliminate any water content from the sample, dry it in an oven set at the proper temperature (typically about 100°C). To turn the sample into a fine powder, use a ball mill, mortar, pestle, or other type of grinding tool. The diffraction pattern is better with a finer powder. Furthermore, make sure there are no pollutants present on the sample holder, which is often made of glass, plastic, or metal. The powder sample is put into a sample holder and also confirm the surface of the power should be flat and uniform. For this purpose, the sample was placed on a sample holder and slid to each other to form a smooth surface. There are three types of movement used to smooth the surface of a sample, front-loading, back-loading, and side-loading methods. This sample powder was also compressed by a sample holder for tightly packed but not over-compacting because it can affect the XRD pattern. At the end also check, that there are no cracks or gaps in the powder sample and surface is uniform and flat. After inserting the sample holder with the correct position in XRD and closing the door, the closed signal was activated.

In the next step, the PC with specific software was used to adjust the parameters e.g. scanning range, current, voltage, scanning speed, etc. So, the XRD started working according to the given instructions and also noted everything functionally correctly. The angles range mostly ~5^o-70^o at which data are acquired for common powder patterns specified in the X-ray scan, and it takes ~10 minutes to identify the unknown sample. For all three samples, Al_2O_3, $SrCO_3$, and their mixture followed the same processes.

3. Result and Discussion

The following data of Aluminium oxide (Al_2O_3) and Strontium Carbonate ($SrCO_3$), which was collected from the BDEC software, ICSD, and CRYSTMET portal was utilized to compare all parameters and outcomes mentioned below. Enter “BDEC” into Google; however, access to this page requires registration. Upon accessing this website, open “CRYSTMET-VERSION 6.2.0”. Subsequently, a periodic table will appear up, enabling us to select the precise elements we need to look for in the literature, click "search," and then click "result." e.g. I have selected Al and O elements, to find the literature data of Al_2O_3 as shown in **Figure 1, and 2**. We can also obtain information about lattice parameters, space group, and atomic position by clicking on “Detail” and “Coordinate” respectively as mentioned in **Figures 3 and 4**.

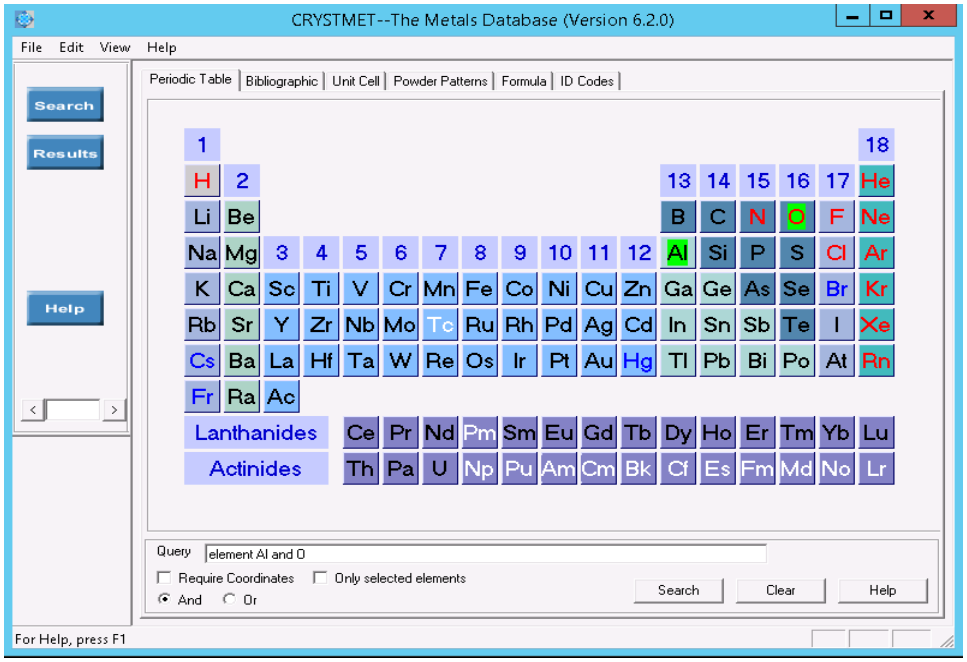


Figure 1. Selection of elements (Al and O)in CYSTMET (version 6.2.0)

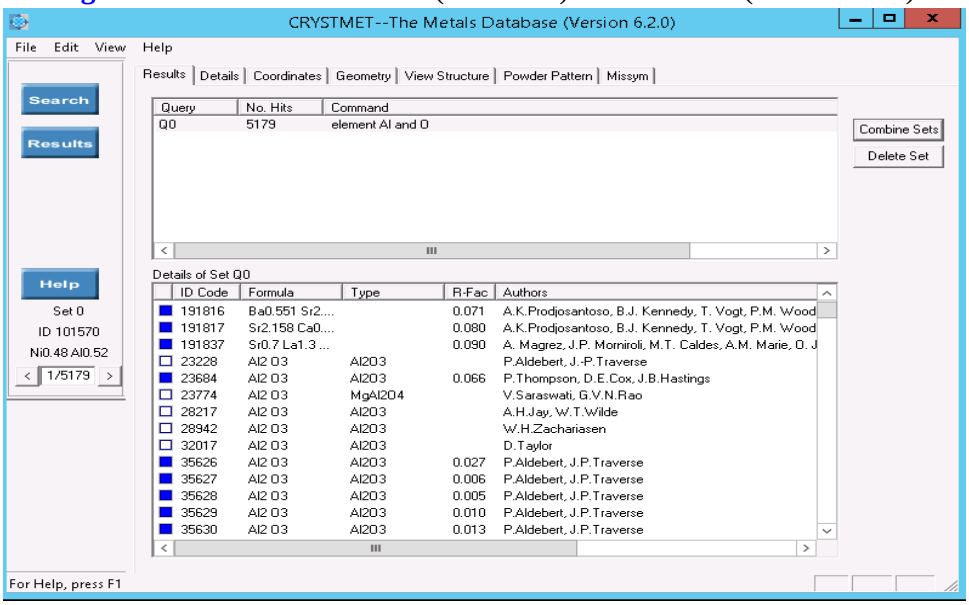


Figure 2. Results of published literature data on Al_2O_3

ID: 130134

Formula: Al_2O_3 [alpha]
Structure Type: Al_2O_3
Space Group: ***R-3c* (167)**
Pearson Symbol: hR10

Cell Dimensions

a = 4.754(1)	b = 4.754(1)	c = 12.99(2)
α = 90	β = 90	γ = 120
Volume = 254.25	Z = 2	

Temperature = 300K

Reference

N.Ishizawa, T.Miyata, I.Minato, F.Marumo, S.Iwai
Acta Crystallogr., Sect. B [ACBCAR]
Volume **B36** 228 - 230 (1980)

Figure 3. Space group, lattice parameters, of Al_2O_3 collected by CRYSTMET (version 6.2.0)

Atom	Wyckoff	Site Sym	X	Y	Z	Uiso	Occ.
Al	12c	3.	0	0	0.35228(5)	0.32	1
O	18e	.2	0.3064(3)	0	1/4	0.33	1

Figure 4: Wyckoff, coordinate collected by CRYSTMET (version 6.2.0)

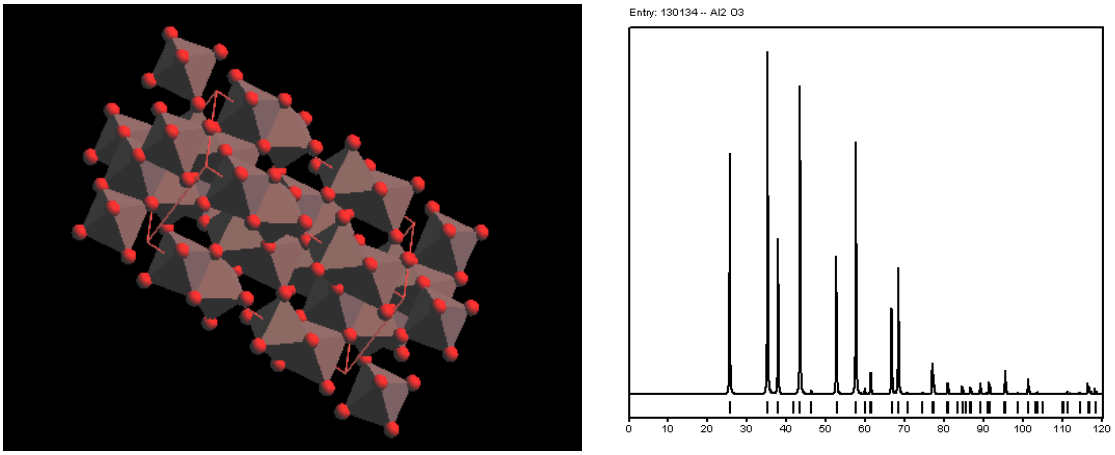


Figure 5. Structure diagram and Powder Pattern of Al_2O_3 collected by CRYSTMET (version 6.2.0)

ICSD was also used for the collection of crystallographic data. First of all, open the BDEC and click on "ICSD". It is possible to search by chemical elements in chemistry. Type the chemical elements and enter the number of elements. Next, click on "Run Query" as mentioned in **Figure 6**. For example, I have selected the elements Al and O and found the Lattice parameter, space group, Wyckoff symbol, powder pattern, and structure of Al_2O_3 collected as shown in **Figure 7**. The same method was used to find the literature data on strontium carbonate ($SrCO_3$) as shown in **Figures 8 and 9**.

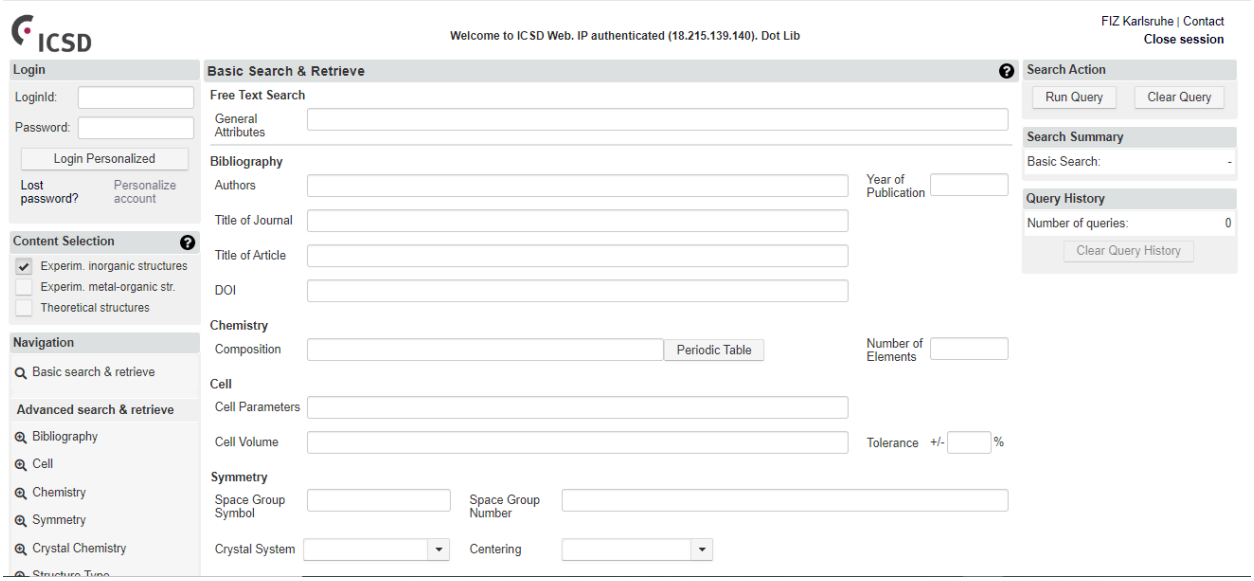
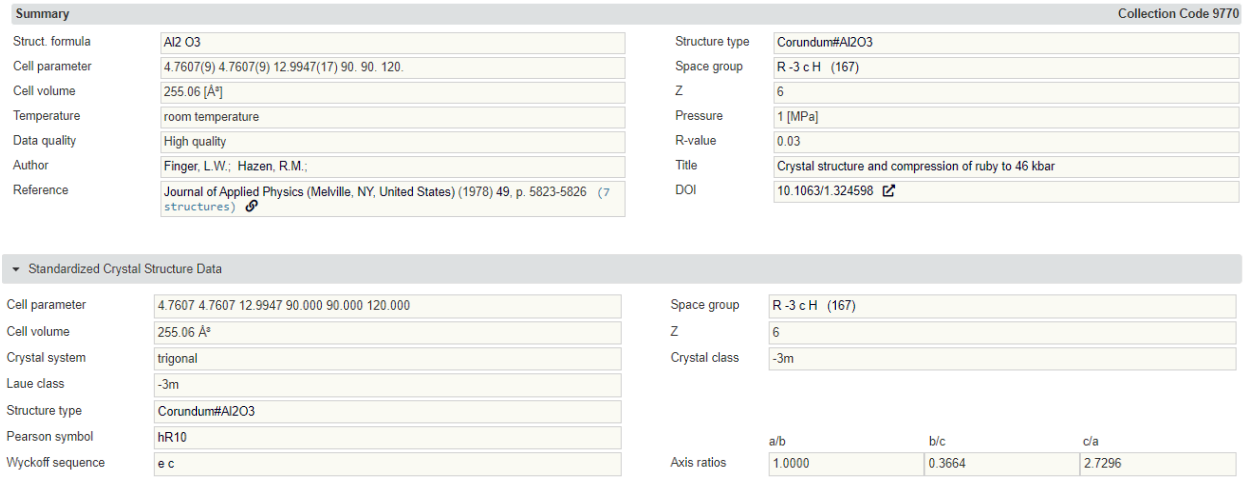


Figure 6. Crystallographic data collection by ICSD



EL	Lbl	OxState	Wyck Symb	X	Y	Z	SOF	B
Al	1	+3.00	12 c	0.0000	0.0000	0.1478	1.000000	0.3100

Visualization

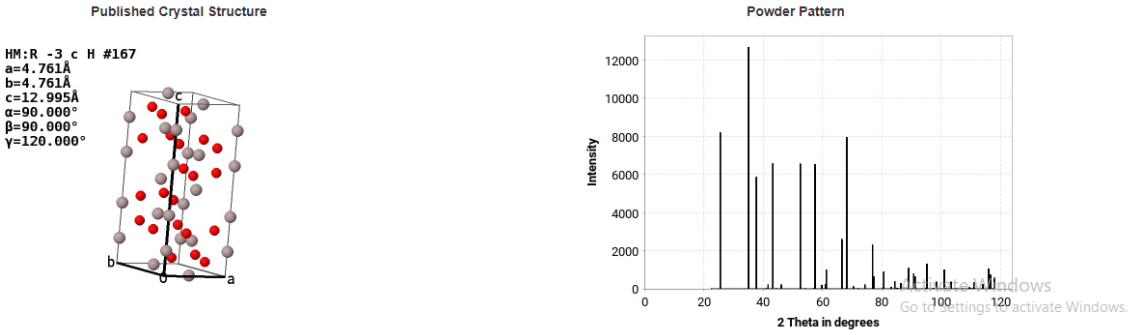


Figure 7. Lattice parameter, space group, Wyckoff symbol, powder pattern, and structure of Al_2O_3 collected by ICSD-BDEC

ID: 859748

Formula: $SrCO_3$
Space Group: ***Pmcn*** **(62)**
Pearson Symbol: oP20

Cell Dimensions

a = 6.0455	b = 5.1004	c = 8.4142
α = 90	β = 90	γ = 90
Volume = 259.45	Z = 4	

Temperature = RT

Reference

A.D.Al-Rawas, H.M.Widatallah, S.H.Al-Harthi, C.Johnson, A.M.Gismelseed, M.E.Elzain, A.A.Yousif
Mater. Res. Bull. [MRBUAC]
Volume **65** 142 - 148 (2015)

Remarks

SrCO3-99%, alpha-Fe2O3-99.9%.
Crystalline size, Mossbauer spectra, X-ray photoelectron spectra reported.
In the mixture with 15 wt% of alpha-Fe2O3(spgr=R-3c) and 32 wt% of SrFeO3(spgr=Pm

Figure 8. Space group, lattice parameters collected by CRYSTMET (version 6.2.0), BDEC

Summary — Collection Code 15195

Struct. formula	Sr (C O3)	Structure type	Aragonite#CaCO3(oP20)
Cell parameter	5.090(2) 8.358(2) 5.997(4) 90. 90. 90.	Space group	P m c n (62)
Cell volume	255.13 [Å³]	Z	4
Temperature	room temperature	Pressure	atmospheric
Data quality	High quality	R-value	0.031
Author	de Villiers, J.P.R.;	Title	Crystal structures of aragonite, strontianite, and witherite
Reference	American Mineralogist (1971) 56, p. 758-766 (3 structures)		

Standardized Crystal Structure Data

Cell parameter	5.9970 5.0900 8.3580 90.000 90.000 90.000	Space group	P n m a (62)
Cell volume	255.13 Å³	Z	4
Crystal system	orthorhombic	Crystal class	mmm
Laue class	mmm		
Structure type	Aragonite#CaCO3(oP20)		
Pearson symbol	oP20		
Wyckoff sequence	d c3	Axis ratios	a/b: 1.1782; b/c: 0.6090; c/a: 1.3937
Transformation info	REMARK Transformed from setting P m c n.--> P n m a TRANS c,a,b origin 0 1/2 0		

EL	Lbl	OxState	Wyck Symb	X	Y	Z	SOF	B
Sr	1	+2.00	4 c	0.2431	0.2500	0.5840	1.000000	0.5080
C	1	+4.00	4 c	0.0864	0.2500	0.2399	1.000000	0.5920
O	1	-2.00	4 c	0.0946	0.2500	0.0881	1.000000	0.9130
O	2	-2.00	8 d	0.0839	0.0306	0.3179	1.000000	0.7970

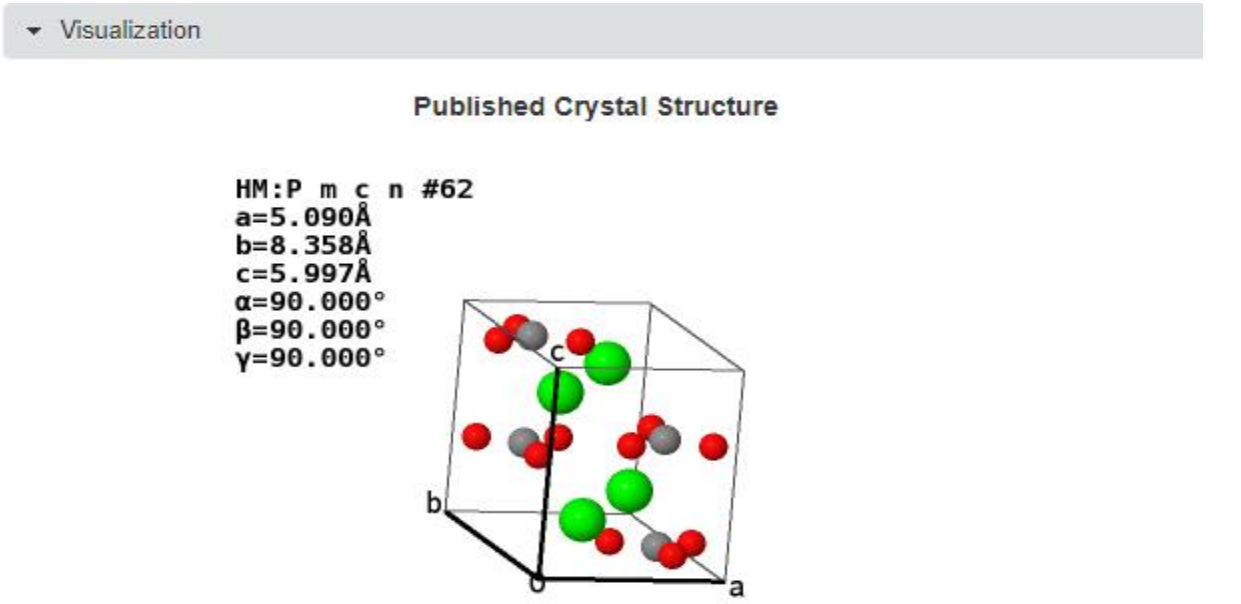


Figure 9. Lattice parameter, space group, Wyckoff symbol, structure of $SrCO_3$ collected by ICSD, BDEC

The PowderCell program 2.4 facilitates the loading and typing of data that adds to the diffractogram data analysis. The window confirms the icon for opening and selects the first button to "Open" or "Load" a structure file. This button or the equivalent entry in the File / Load main menu (shortcut key Ctr+L) may be used to access the File access dialog box, which allows the selection of a structure file with the *.cel extension as shown in **Figure 10**. Other import filters are also available, but the majority of them are no longer functional since the corresponding program has either disappeared from the app store or their format has evolved. Unfortunately, PowderCell's import filter has never been modified for the crucial CIF. The file open dialog is a typical Windows dialog; that is, the design takes advantage of the system's active Windows-specific declaration. As a result, the image displayed here may not match the color and style of your computer.

PowderCell allows you to load up to 10 phases at a time. Alternatively, you may use cell transformation or subgroup descriptions of a phase to open more phase windows. Since each one is recorded separately, it might be required to eliminate a phase by closing the corresponding tab. Please do not hesitate to contact with authors if you are having difficulty importing the file. In order to create a new structure file (import literature data collected by CRYSMET **(***version 6.2.0)* using file/new and fill the crystal information (name, space group, periodic number Z, lattice parameters, Wyckoff, coordinates, temperature, and Debye-Waller factor B) and click on “OK” and SAVE the file in your PC as shown in **Figure 11**. It is necessary to input the crystal-structure data for a real phase. Since PowderCell already understands the symmetry-specific connections between the basis vectors a, b, and c and their angles, it is often advised to begin with the space-group number and the setting number. This minimizes typing mistakes and the entry of unnecessary data.

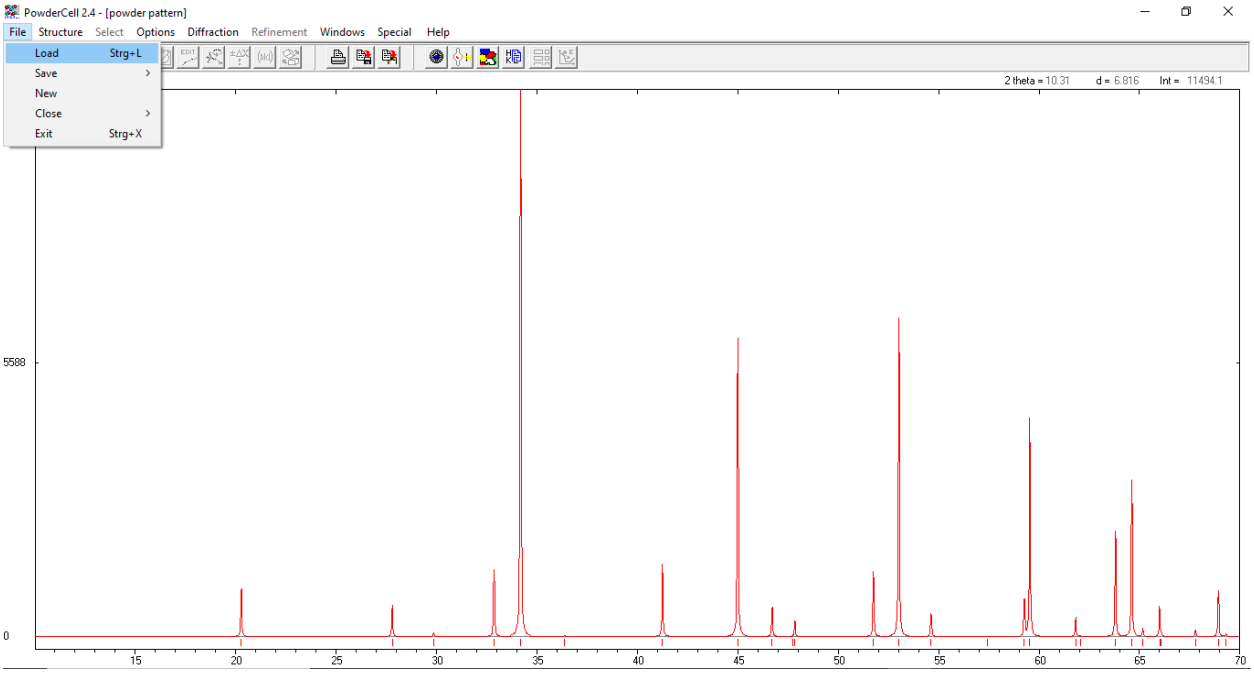


Figure 10. Import *.cel file contains crystal information of Al_2O_3 in powdercell 2.4.

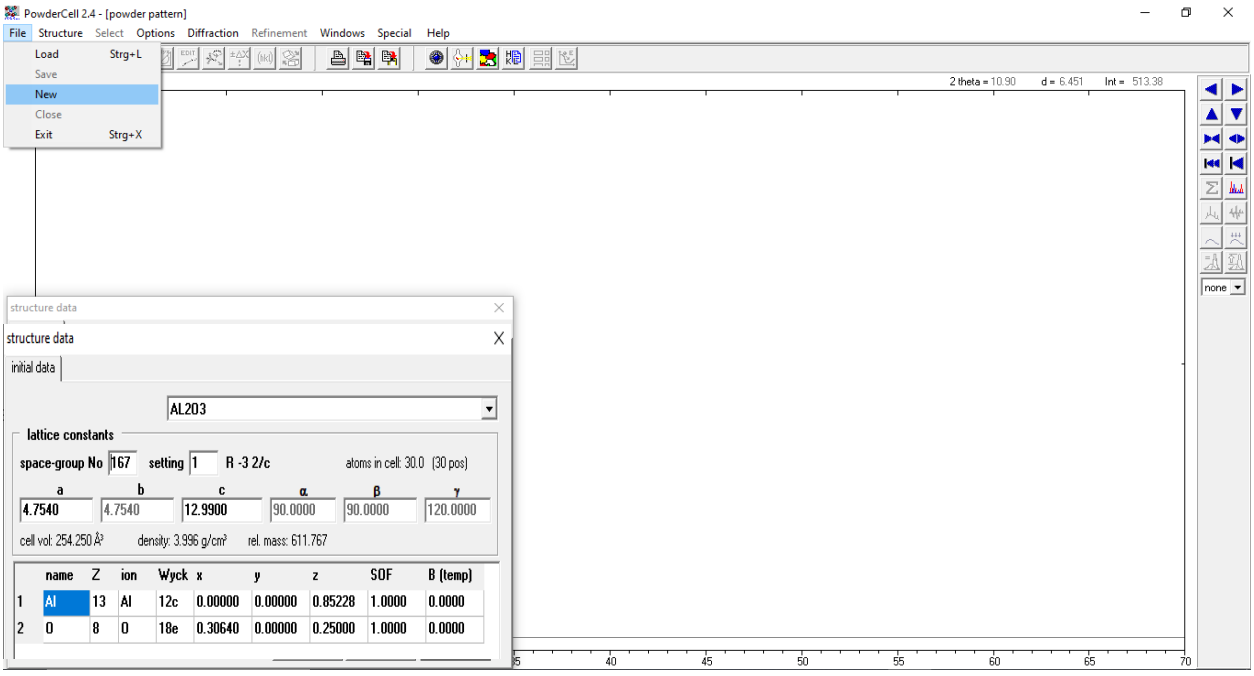


Figure 11. Literature crystal data of Al_2O_3 collected by CRYSMET (version 6.2.0)

To examine the diffraction pattern click on the "diffraction/upload powder pattern" (experimental data) file with *.x_y format. Two numerical columns of the measured data, 2theta, and intensity, are present in the text file in the.x_y format as shown in **Figure 12**. In order to change the experimental condition click on "experiment" and by the second option, the phase option can also adjust the colors, angles, and other parameters as shown in **Figure 13**.

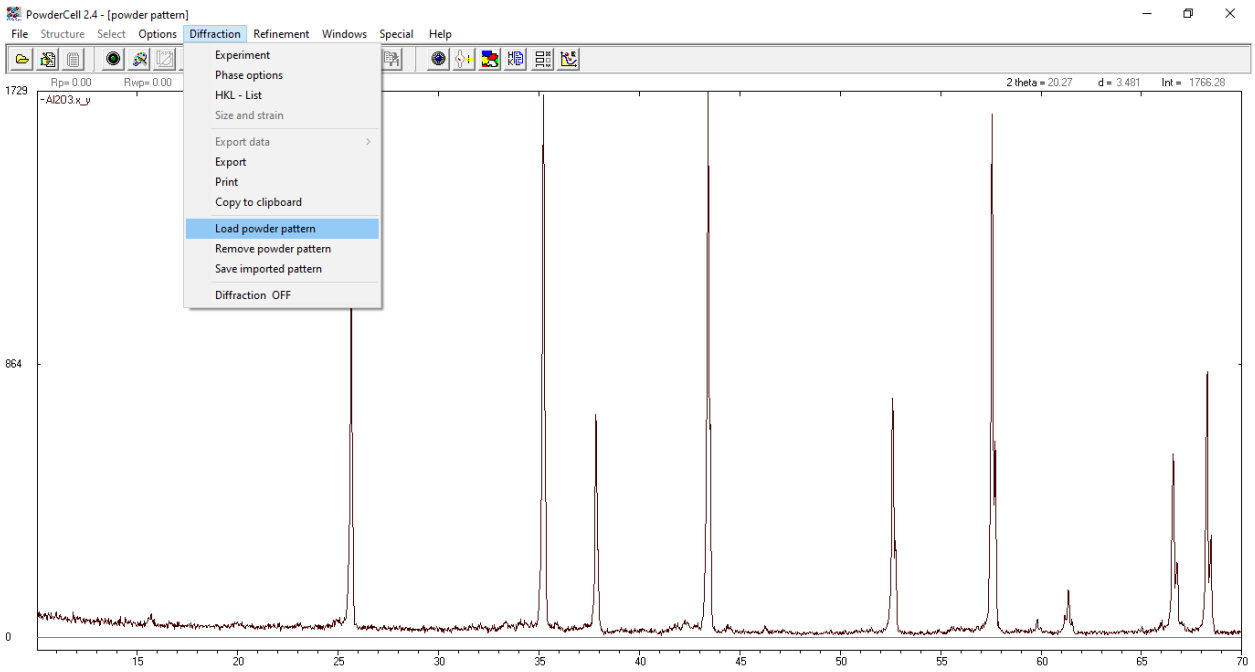


Figure 12. Import experimental data pattern of Al_2O_3

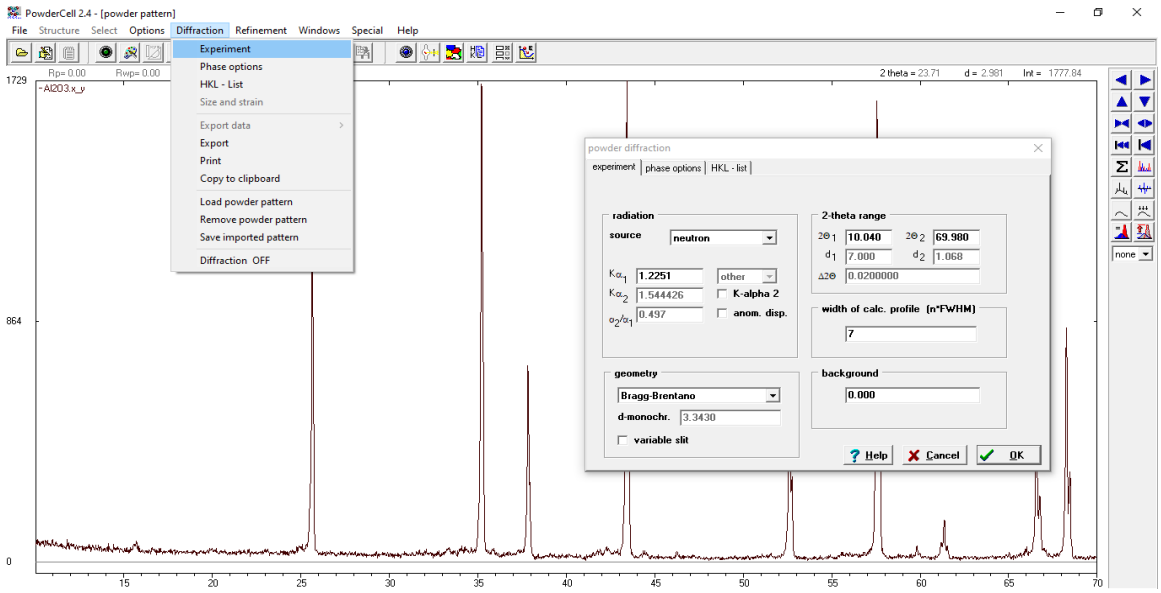


Figure 13. Variation in experimental condition

First, a comparison was made between the data measured in practice and the data found in the literature. In secondary investigations, a volumetric value was extracted from each phase that will be converted into mass and compared with the strong firstly. The composite data was entered into the PowderCell program, and diffraction techniques were employed to obtain the theoretical data as near as feasible to the real one.

In Primary analysis, Powder Cell was used to compare the experimental data and literature data of Al_2O_3. First of all, find the literature data**,** load these parameters mentioned in **Figure 11** and its peaks represented by red color, on other hand for the attained experimental graph select the "diffraction" option and load the powder pattern, and upload the experimentally measured file, its peaks represented

by black color as shown in **Figure 14**. The fact that the black and red peaks of **Figure 14**, which refers to Al_2O_3, coincide exactly shows that the compound being measured is the same as the one found in the literature, that the test conditions and measurement, software settings were appropriate, and that any errors were kept to a minimum.

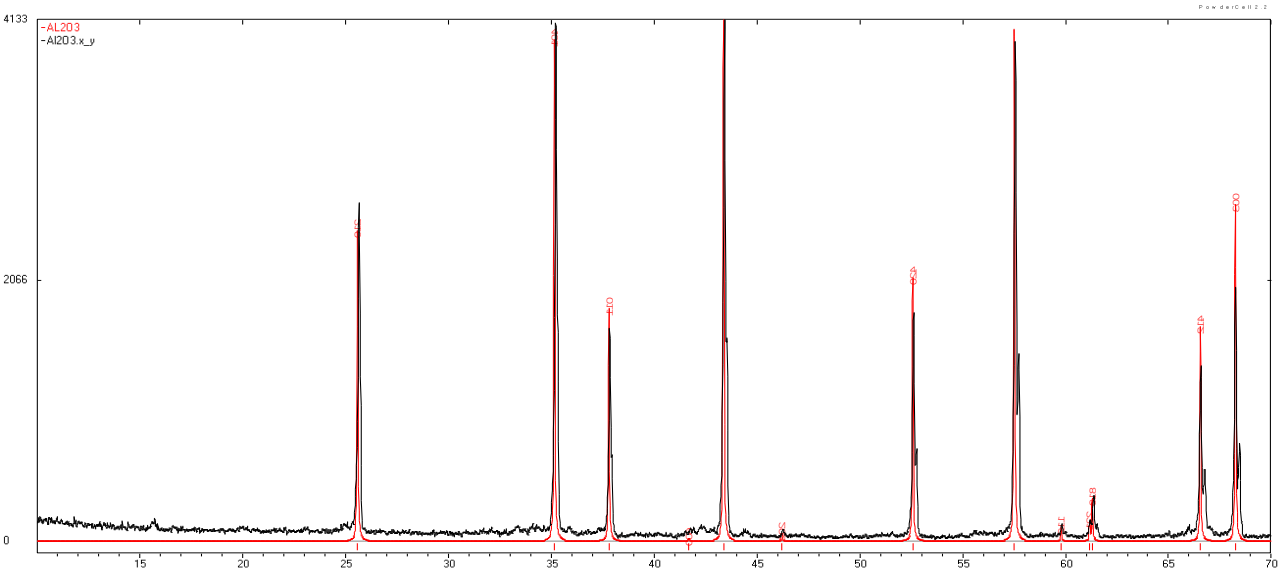


Figure 14. Comparison b/w experimentally peaks (black) and literature peaks (red) Al_2O_3 by Powdercell 2.4

In the case of strontium carbonate ($SrCO_3$) also compared the experimental data and literature data by using the same method. First of all, the literature data of $SrCO_3$ e.g. lattice parameters, space group, Wyckoff, angles, etc. mentioned in **Figure 15**. To load these all parameters on the powder cell, (follow the same steps as mentioned for the above example of Al_2O_3) its peaks are shown in green color after that also upload the powder diffraction pattern (experimentally measured data) of $SrCO_3$, its peaks represented by black color as shown in **Figure 16**. In the powder cell, adjust the value of k-alpha 1 and lattice parameters a & c until experimentally peaks (black) and literature peaks (green) of SrCO3 are strongly matched and coincide with each other and minimize the error.

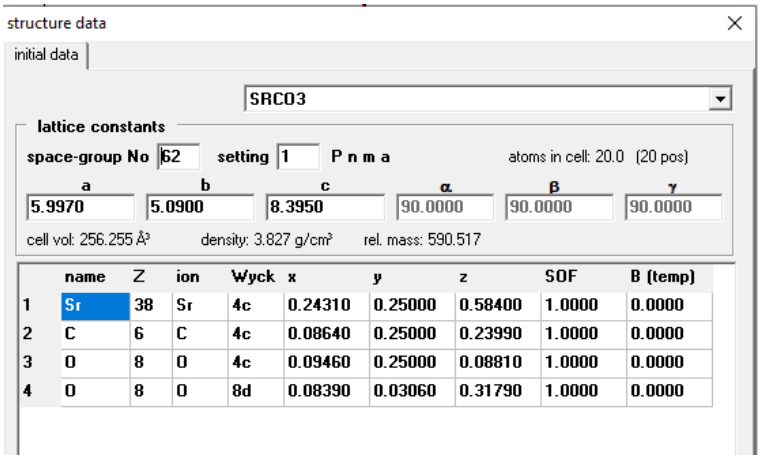


Figure 15. Space group, lattice parameters, Wyckoff space of Sr_2O_3

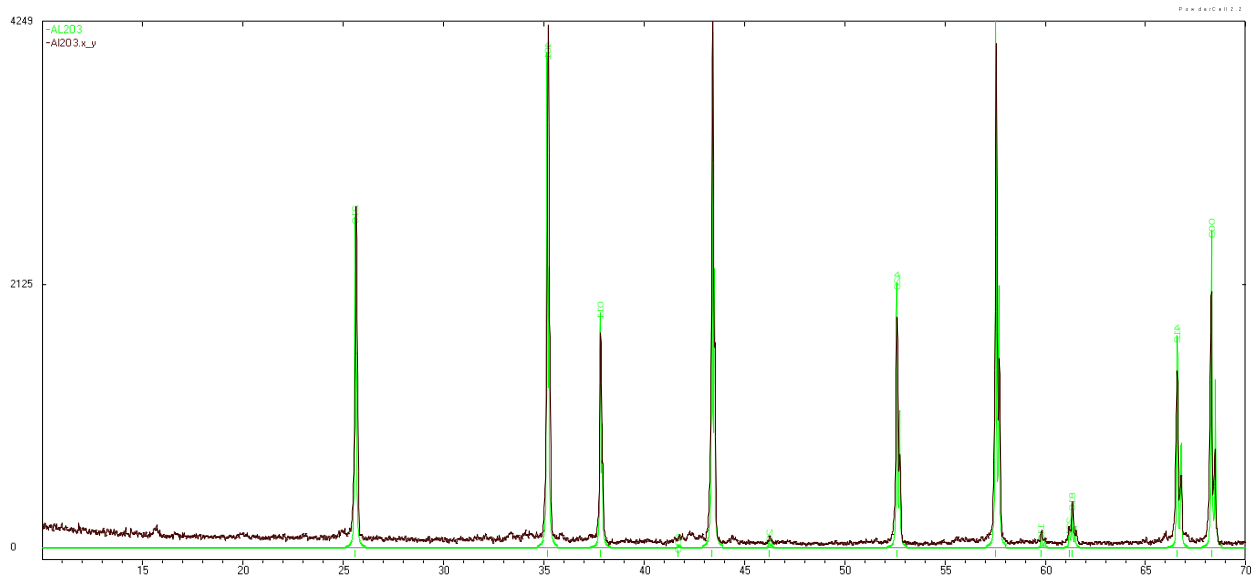


Figure 16. Comparison b/w experimental peaks (black) and literature peaks (green) of $SrCO_3$ by Powdercell 2.4

In a secondary analysis of the material, the literature spectrums of Aluminum oxide (Al_2O_3) and Strontium carbonate ($SrCO_3$) were loaded at the same time (click on "load" and load the save files), represented by red and blue peaks respectively, and compared these peaks with a mixture of black peaks of Al_2O_3- $SrCO_3$. **Figure 17** shows that aluminum oxide, strontium carbonate, and their mixture peaks are matched and coincide with each other. The volume weight percentage of Al_2O_3 and $SrCO_3$ are 50, 50% respectively. , as mentioned in the right corner of **Figure 17.**

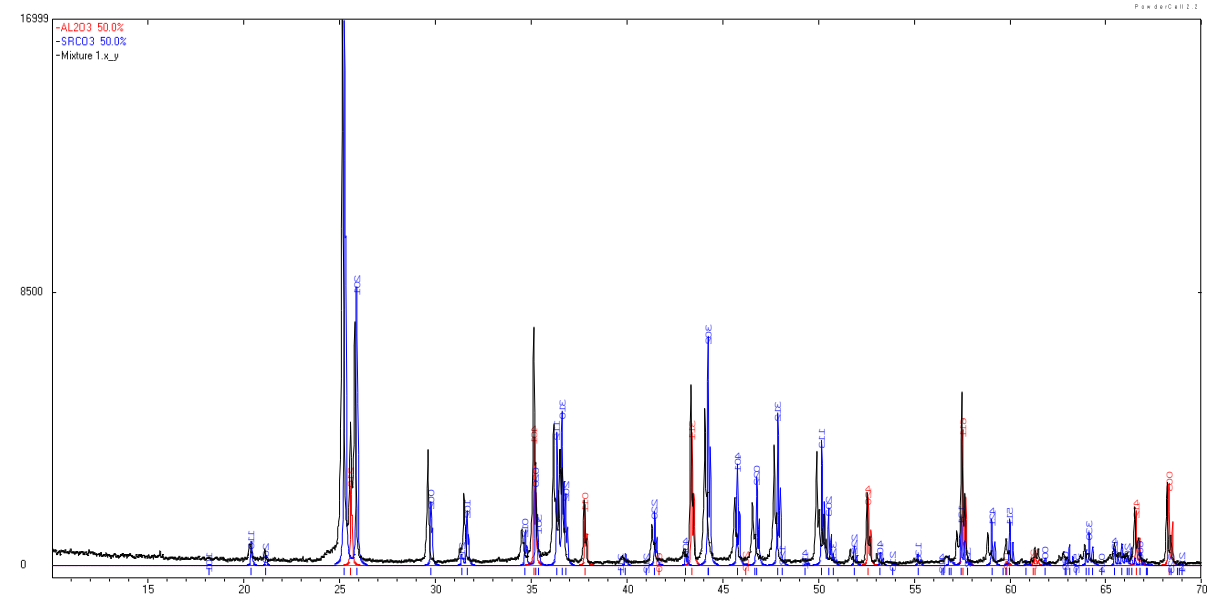


Figure 17. Volume percentage of Al_2O_3 and $SrCO_3$ collected by powdercell 2.4

PowderCell's refining process improved the program's quality, particularly from a practical perspective. One hand is used to calculate the right line intensity. On the other hand, the capacity to match the theoretical and real diffractograms. It is evident that adjusting the many parameters by hand

including the scaling factors, zero shift, background, lattice parameters, profile parameters, and desired orientation is quite challenging. As a result, an automated refinement with configurable restrictions has been put into place. The dialog window for the lattice and profile parameters is shown in **Figure 18 (a).** It is possible to choose the most suitable orientation, profile parameters, and independent lattice constants for each crystalline phase under consideration in a sophisticated manner. The basic settings (zero shift, the background, etc.), the atomic coordinate refinement, the obtained findings, and the standard parameters for the initial refinement may all be changed on different worksheets. Refinement parameters can be activated by ticking the appropriate box, as shown in **Figure 18.** During the refining process, all additional don'ts will be adjusted.

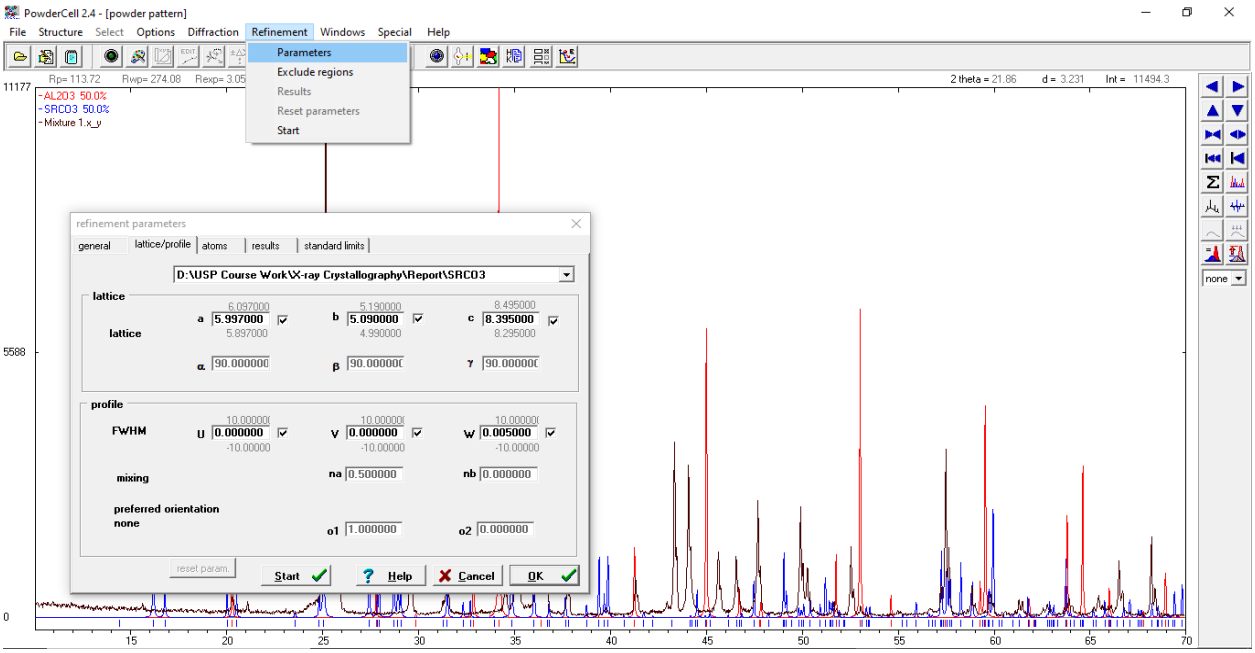


Figure 18 (a). Dialog window for defining the refinement parameters

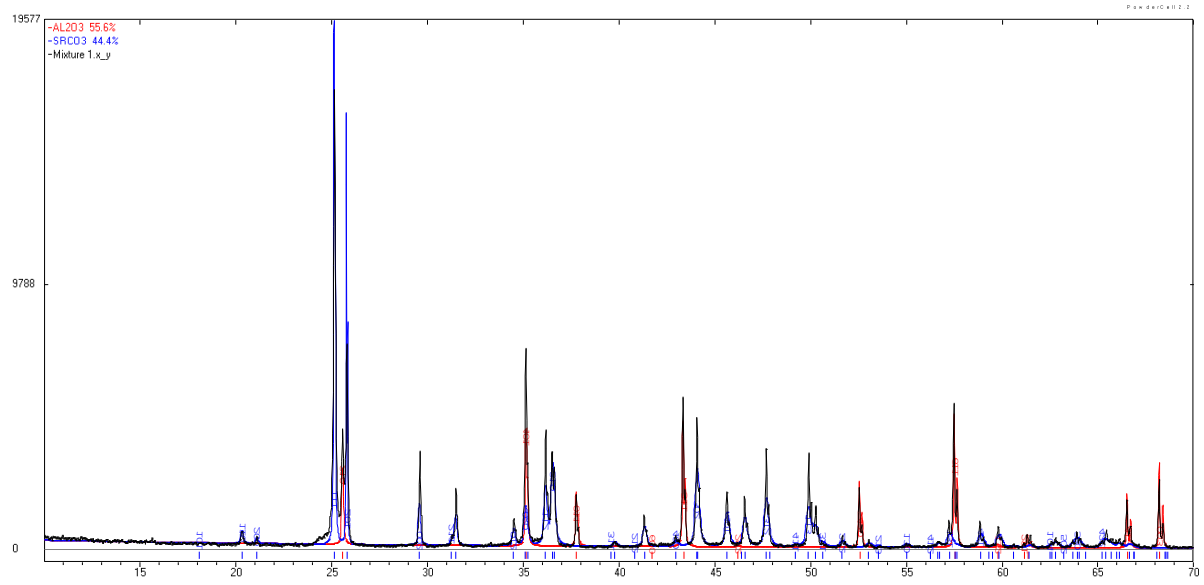


Figure 18 (b). Volume percentage of Al_2O_3 and $SrCO_3$ after refinement

Following the refinement of **Figure 17**, two distinct differences can be seen in the diffractogram data: first, blue peaks ($SrCO_3$) are more visible than red peaks, indicating that the material is more crystalline and that the scattered rate of x-ray increases by its atoms. However, the refinement red peaks of Al_2O_3 were smaller rate but the volume percentage of Aluminium oxide (Al_2O_3) was higher than strontium carbonate ($SrCO_3$). After the refinement of **Figure 17** indicates in **Figure 18 (b)** that the volume percentage of Al2O3 -55.6% and SrCO3-44.4%. The variation in parameters (e.g. scale factor, lattice parameters, zero shift, FWHM, and others) of Al2O3, SrCO3, and their mixture after refining is shown in below **Figures 19 and 20**.

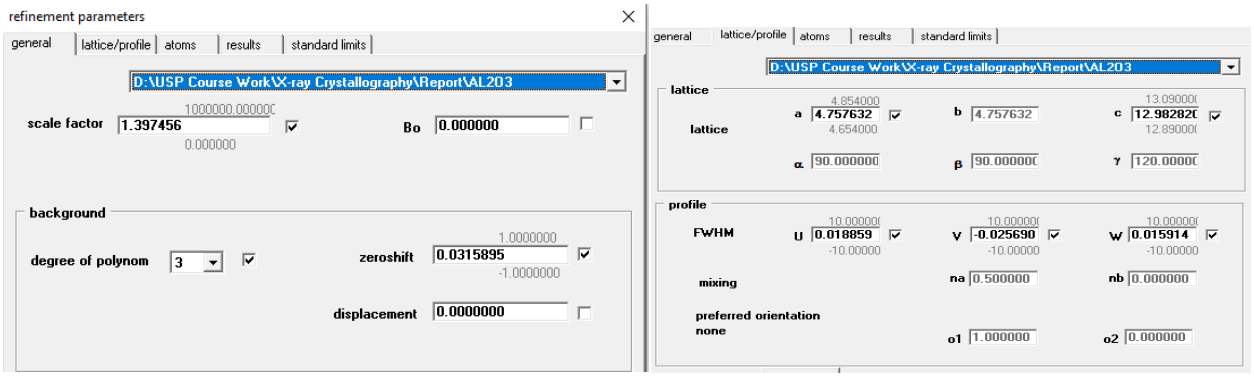


Figure 19. Refinement parameters of Al_2O_3

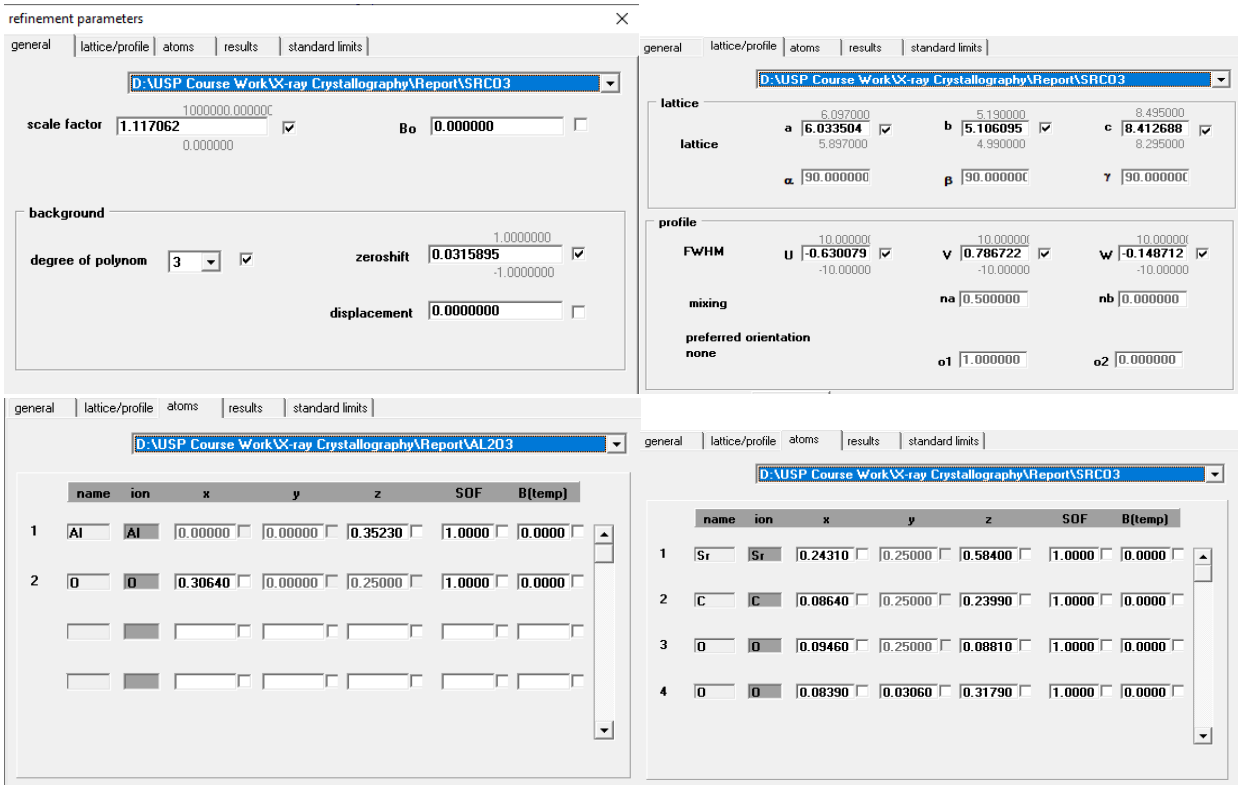


Figure 20. Refinement parameters of $SrCO_3$

```
Refinement of Mixture 1.x_y    23/06/2024  6:55:02

AL2O3        55.58 vol.%    (scf: 1.3975)
SRCO3        44.42 vol.%    (scf: 1.1171)

R-values       Rp=27.79   Rwp=37.42   Rexp=2.01
 1 iterations of 3


parameter                      old            new

AL2O3
-------------------------------------------------

lattice          a :          4.7576         4.7576
                 c :         12.9828        12.9828

profile          U :          0.0189         0.0189
PsVoigt2         V :         -0.0257        -0.0257
                 W :          0.0159         0.0159

overall B        :          0.0000              -


SRCO3
-------------------------------------------------

lattice          a :          6.0335         6.0335
                 b :          5.1061         5.1061
                 c :          8.4127         8.4127

profile          U :         -0.6301        -0.6301
PsVoigt2         V :          0.7867         0.7867
                 W :         -0.1487        -0.1487

overall B        :          0.0000              -


global parameters
-------------------------------------------------

zero shift       :          0.0316         0.0316

displacement     :          0.0000              -

backgr. polynom  :               3              3

coeff.          a0 :        524.3665       524.3665
                a1 :          -21.09         -21.09
                a2 :          0.3694         0.3694
                a3 :       -0.002349      -0.002349
```

This practice was done to confirm the accuracy of the experimental work with information available in the literature. The diffractogram display below shows that the refined theoretical peaks with the measurements of each compound peak were matched. **Figure 21** shows that the experimental and literature peaks represented by black and red color respectively, after refinement are strongly matched to each other and homogeneity. The second compound strontium carbonate ($SrCO_3$), represents the refined behavior. In this case, observe that some divergences may still be seen in the spectrum, but it is still strongly matched to the initial spectrum produced to compare the peaks. The variation after refinement has been noted in the peak positions and heights. This demonstrates that the compound's experimental measurements a little bit disagree with the information found in the literature.

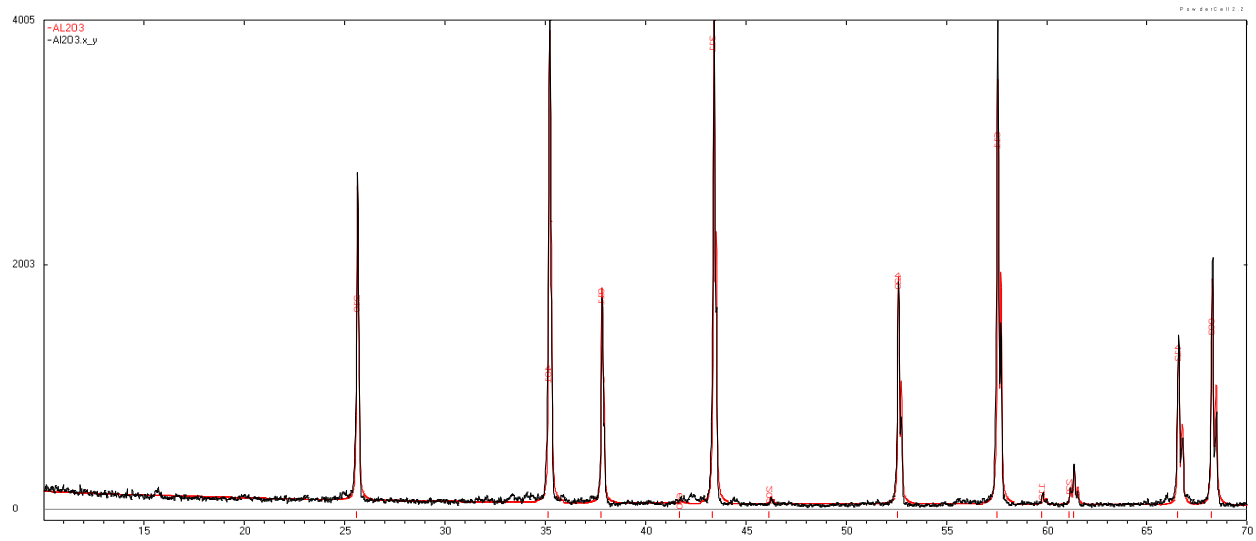


Figure 21. Comparison b/w experimental peaks (black) and literature peaks (red) of Al_2O_3 by Powdercell 2.4

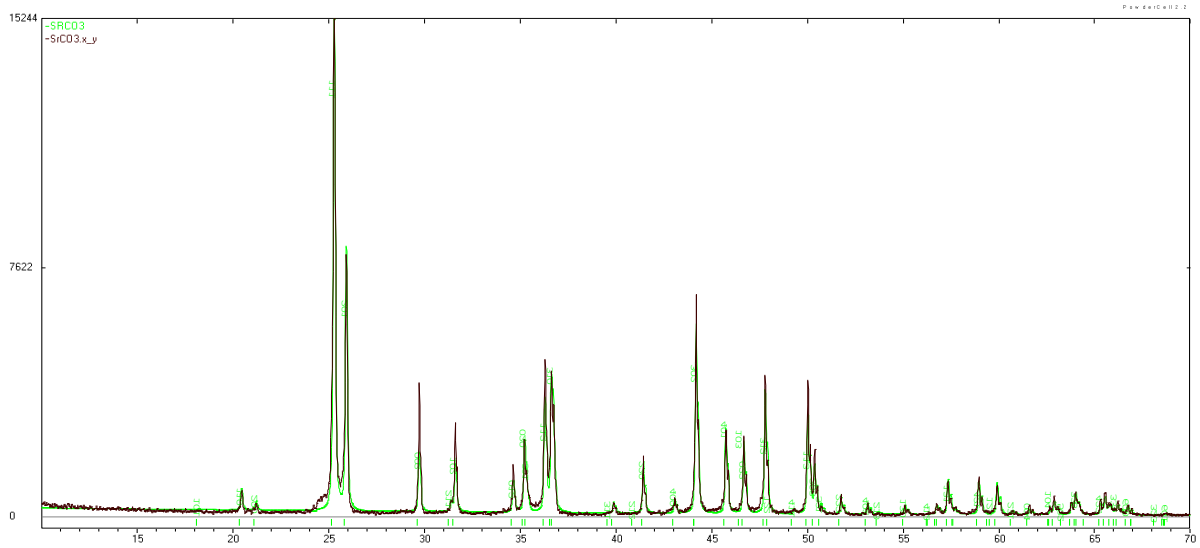


Figure 16. Comparison b/w experimentally peaks (black) and literature peaks (green) of $SrCO_3$ by Powdercell 2.4

Conclusion

The execution of this article effectively leveraged all available resources and tools that were provided and discussed for analyzing the X-ray diffraction pattern of compounds and their composite by using the Powdercell 2.4 program. The software PowderCell has a variety of applications available. It is a useful tool for both practical work and crystallographers who are curious about subgroups and how they might be applied to solve scientific problems. This application is especially user-friendly for non-specialists due to its straightforward input files and low level of experience. For qualitative or quantitative phase analysis, the refining process is a useful feature despite the rather simple profile functions that are employed. This approach is important because it shows how theoretical information may be used practically and emphasizes the external factors that may affect the outcome of the test. This allowed for reliable comparisons and inferences about the compounds' behavior and structure, both when combined and when isolated. Moreover, because the data was accurate and definitive, the technique's effectiveness was confirmed, ensuring its reliability.

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