iPowder

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Chapter 1: iPowder installation and environment configuration

iPowder requires a Python and LabVIEW runtime environment. The following will elaborate on these two points.

The iPowder installation package includes the corresponding version of the Python installer, which will automatically execute after iPowder is installed. If the user already has that version of Python installed on their computer, they can skip this step. Note that LabVIEW requires specifying the Python version when calling Python code, so iPowder will only use the specified version of Python. We will later package iPowder installation files that utilize different versions of Python to meet user needs.

Packaging the LabVIEW runtime engine with the iPowder installation package would result in a significantly larger file size, so we offer two installation packages for users to choose from. If users opt for the iPowder installation package that includes the LabVIEW runtime engine (approximately 570MB when unzipped), no additional installation will be needed. However, due to the large size of this version, it cannot be uploaded to GitHub; therefore, we have placed it in a shared drive. The download link is: https://workdrive.zoho.com.cn/folder/rnv2nf8439b9f19134d96a02e7afa9396fa26. If users choose the installation package without the LabVIEW runtime engine (approximately 50MB when unzipped), they will need to perform an additional step to install the LabVIEW runtime environment(The second setp). This installation package can be downloaded from https://github.com/iPowder/iPowder. A comparison of the two installation packages is shown in the table below:

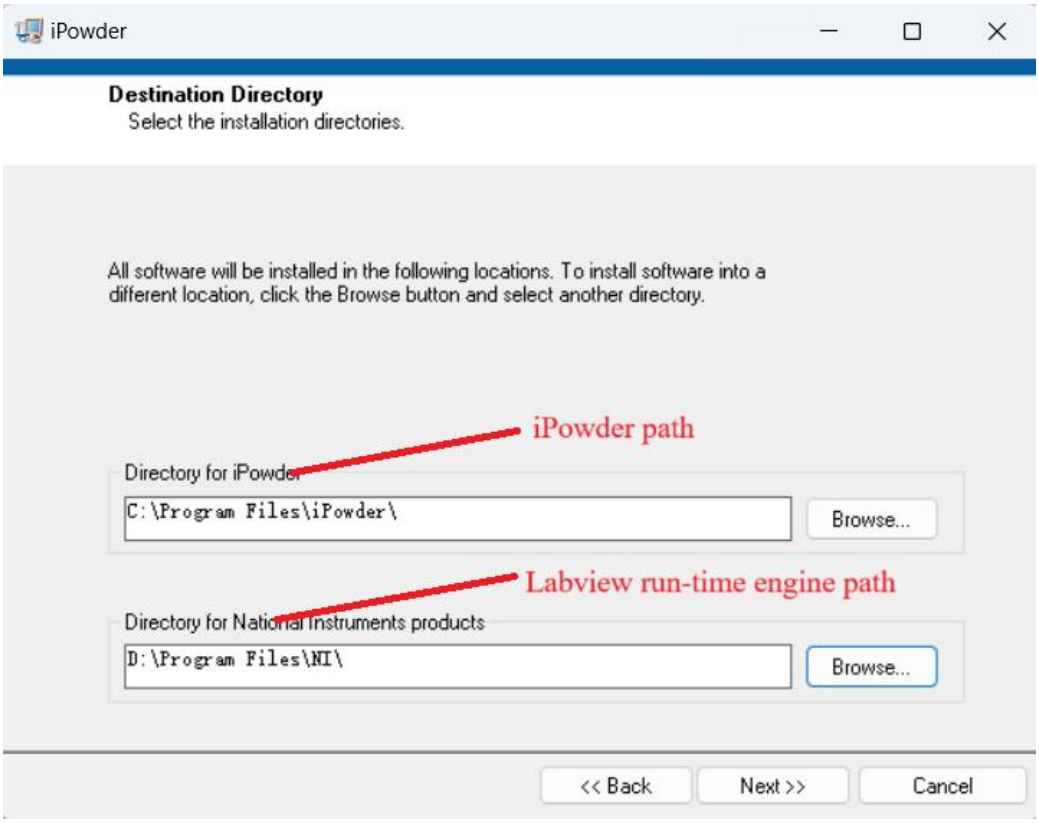
Type	LabVIEW runtime needed?	Size after decompression
iPowderInstaller-python3.10	Yes	50MB
iPowderInstaller-python3.10-(with Labview run time engine)	No	571MB

First:Install iPowder

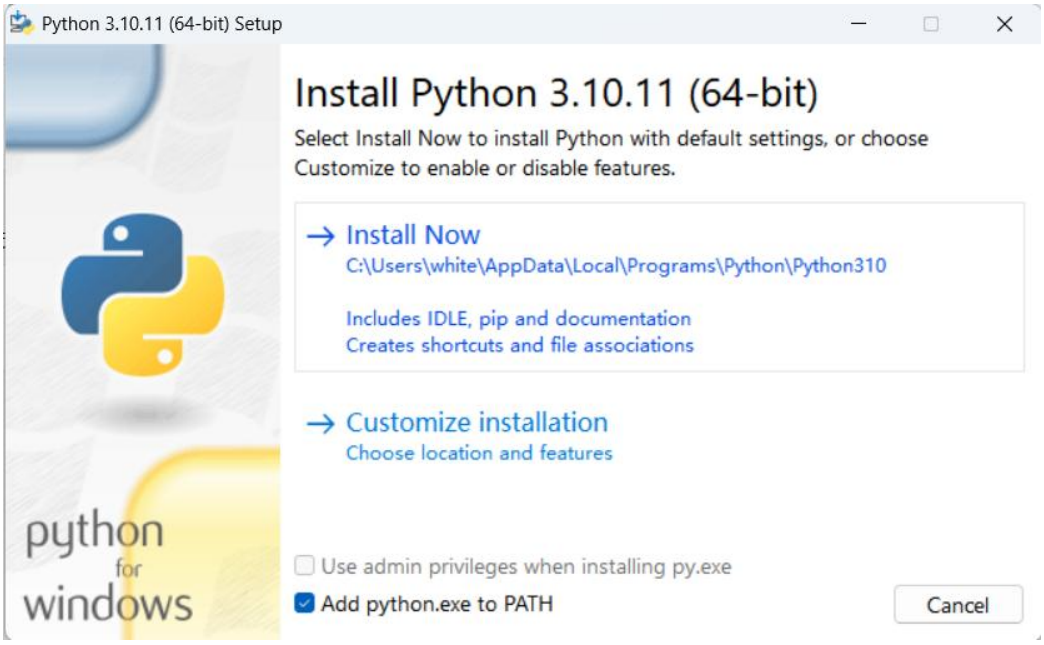
1. Click iPowderInstaller.exe in the ‘iPowderInstaller’

package.

2. In the installation window that pops up, select the installation directory and then click "Next."

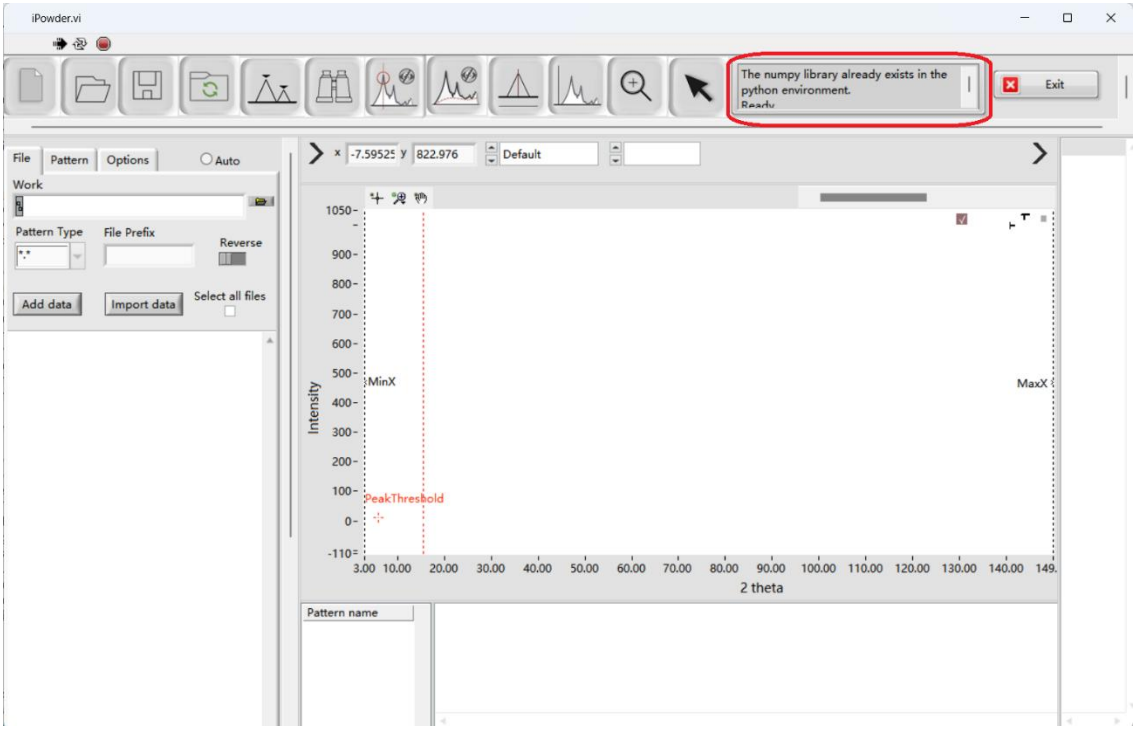


3. After iPowder is installed, a window for installing Python will automatically pop up. If the user's computer already has that version of Python, they can cancel the installation.

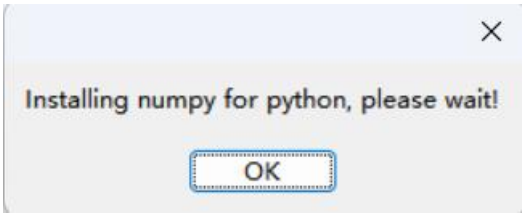


After opening the iPowder program, it will check if Python includes the NumPy library.

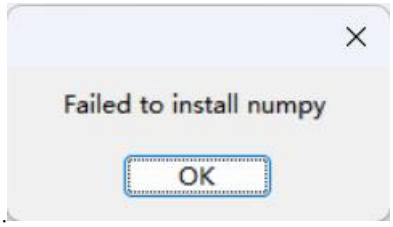
If NumPy is detected as already installed, the message will be displayed in a message box.



If the program detects that NumPy is not installed, it will download NumPy for Python. **Please ensure that the internet connection is functioning properly.**

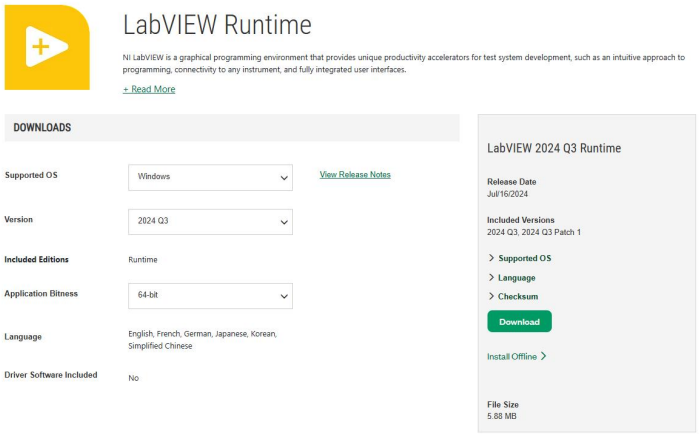


If the internet connection is not functioning properly, the installation of NumPy will fail.



Second:Install the LabVIEW runtime environment.

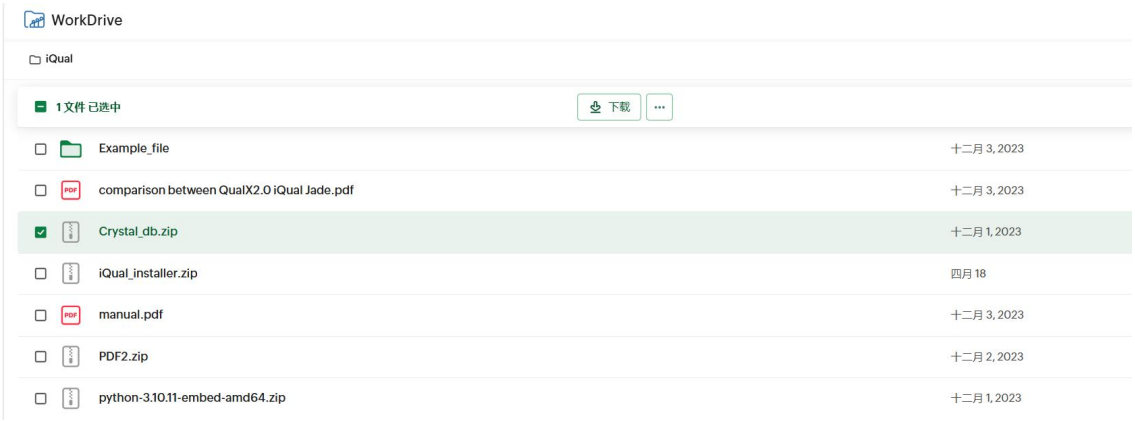
1. Visit 'https://www.ni.com/en/support/downloads/software-products/download.labview-runtime.html#544052' to download the required LabVIEW runtime environment, and then install it.



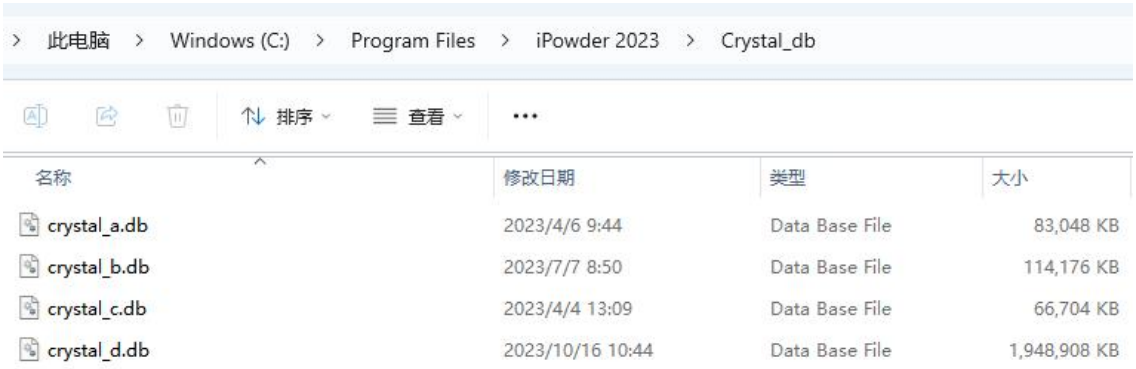
Third: Configure the Crystal_db database.

1. The Crystal_db database can be downloaded from 'https://workdrive.zohopublic.com.cn/folder/bgee25b186e54186b497097286605796b23be?layout=list'. Simply download

Crystal_db.zip.



2. Copy the four database files to the Crystal_db directory under the iPowder installation directory. For example, if iPowder is installed at C:\Program Files\iPowder 2023, then copy the four database files to C:\Program Files\iPowder 2023\Crystal_db.



Chapter 2: Data import and management.

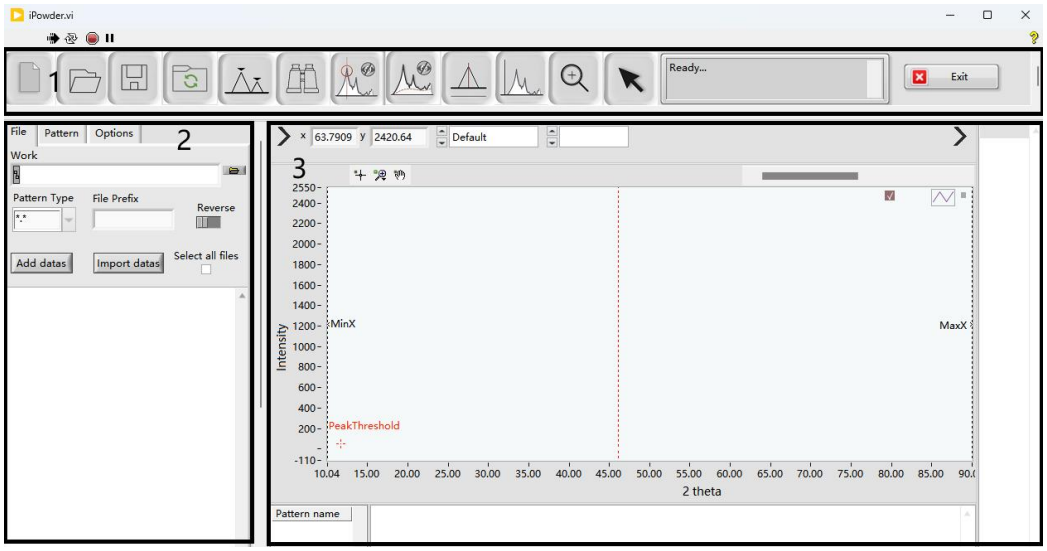


Figure 1 iPowder main interface.

As shown in Figure 1, the iPowder interface can be divided into three sections: Section 1 is the shortcut key area, Section 2 is the options area, and Section 3 is the spectrum and results list area. Data import and management are mainly performed in Section 2. Below, we will describe the methods for importing single or multiple data sets, while explaining each component involved in the process.

1.Users can import the selected data from the file list by using the "Add data" button or the "Import data" button in the File options.

Work: Clicking the Work icon allows users to **select a working directory**. The program will place the files from this directory into the list, categorizing them by file type and sorting them based on the last number in the file name. Users can click on a file to select or deselect it. **Holding down the Shift key while selecting two files will select or deselect all files between them.**

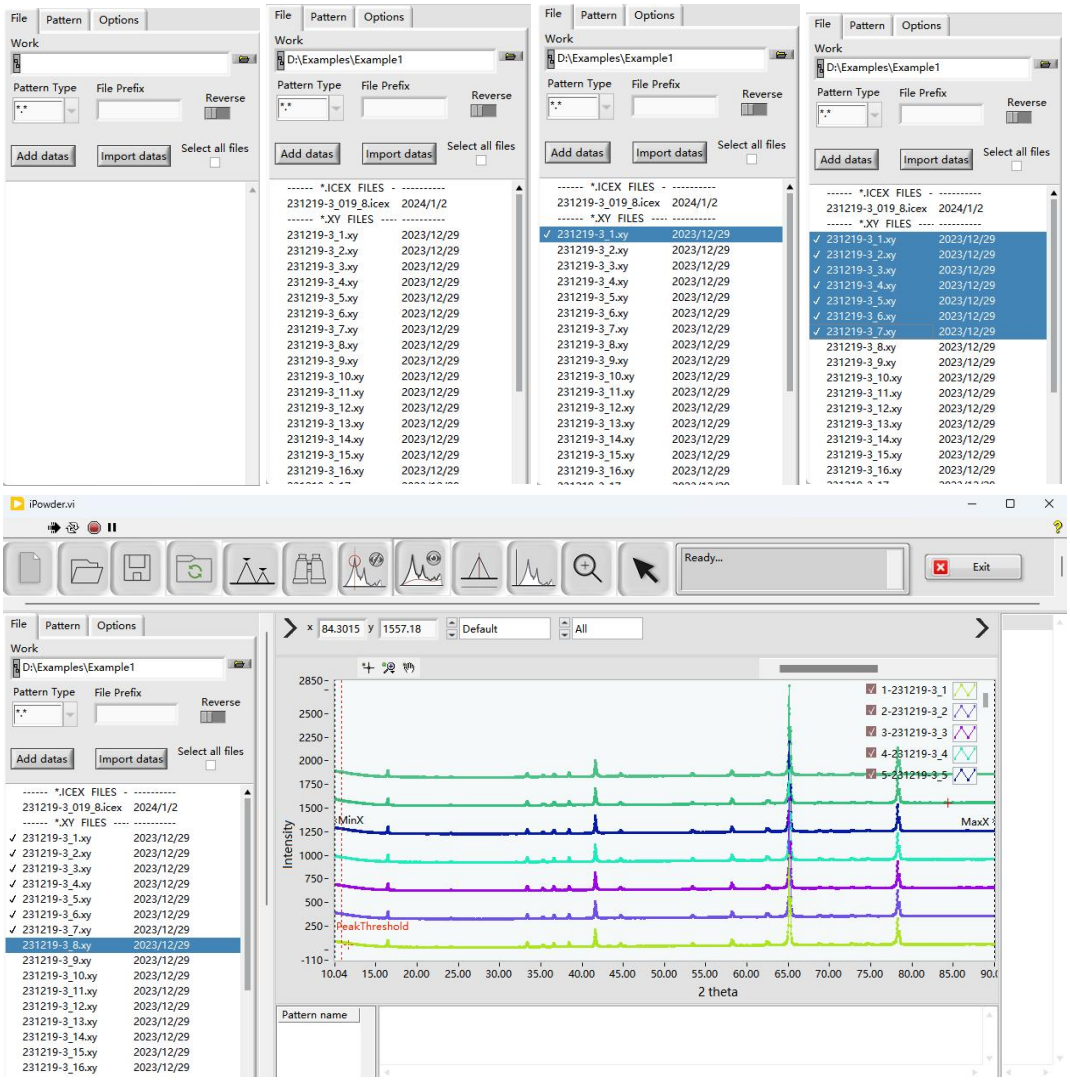
Pattern Type: This is a multi-select option for file types. After selecting a file type (e.g., ‘xy’), the list below will only display files in the ‘xy’ format.

File Prefix: Users can enter a file name prefix here. When importing files from the Work folder next time, the files in the list will be sorted after removing the prefix.

Reverse: Clicking Reverse will arrange the files in the list in reverse order.

Add data: Clicking the "Add data" button will add the selected files to the already imported data.

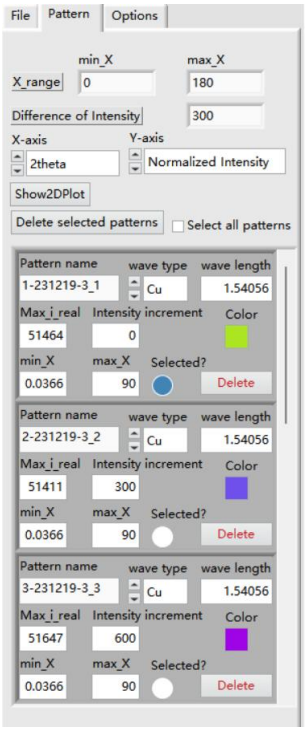
Import data: Clicking "Import data" will clear the previously imported data and then import the selected files.



2.Users can manage the imported data in the **Pattern options**, where they can modify the specific properties of each dataset in the data list, such as wavelength type, angle range, display color, and more. **It is important to note that each dataset has a "Selected" attribute, and the program will only operate on the selected data** (for example, data smoothing, background subtraction, peak finding, Ka2 peak subtraction, normalization, and search matching).

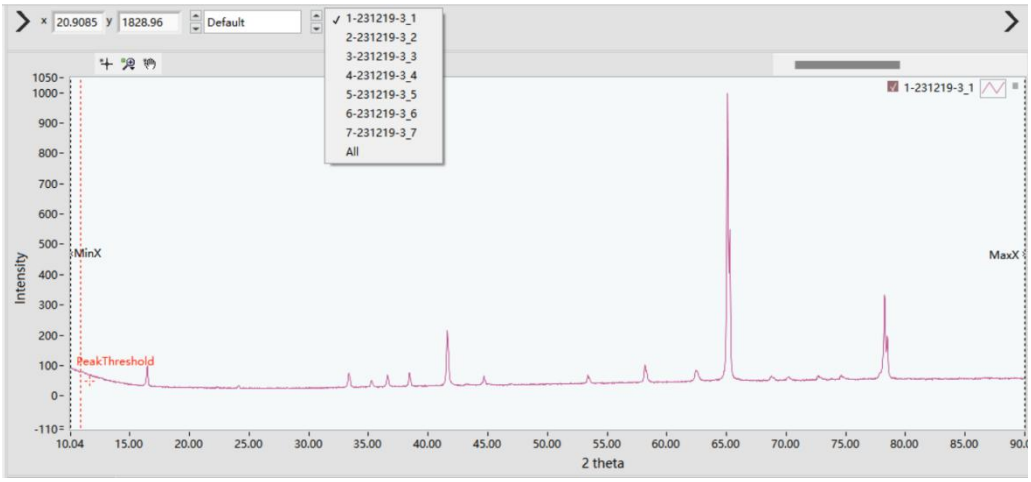
- **X_range:** Setting the 'X_range' allows users to manage the display and processing range of the spectrum (angle range).
- **Difference of Intensity:** When importing multiple spectra, this value can be adjusted to avoid overlapping spectra.

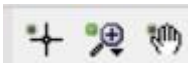
- **X-axis:** Users can choose the display type for the X-axis, supporting two display modes: "2theta" (angle) and "d" (interplanar spacing).
- **Y-axis:** Users can select the display type for the Y-axis, with three options:
    - "Normalized Intensity," where the data's intensity is normalized for display (with a maximum value set to 1000);
    - "Initial Intensity," where the data retains its original intensity;
    - "Logarithmic Intensity," where the Y-axis is displayed on a logarithmic scale.
- **Show 2D Plot:** Displays the two-dimensional image of the spectrum, ensuring that the data ranges are consistent.
- **Delete selected Pattern:** Deletes the selected data (indicated by the blue "Selected" circular light in the data list).
- **Select all pattern:** Selects all data or deselects all data.



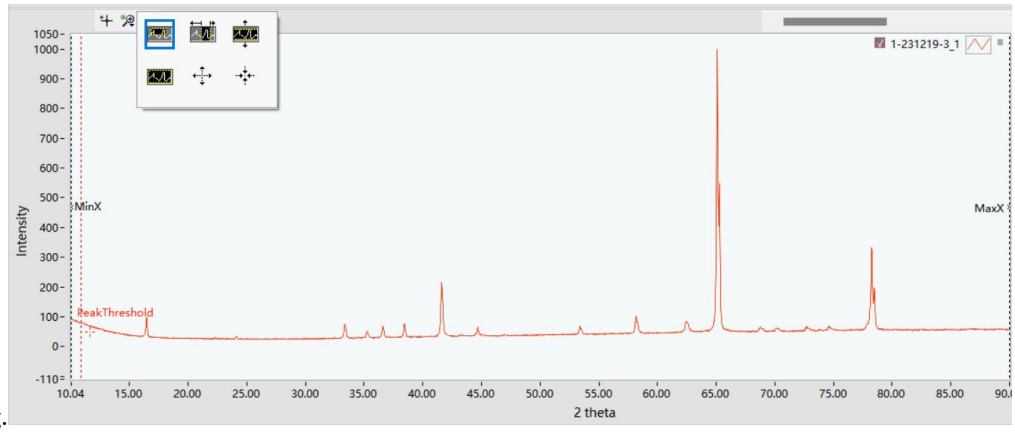
3.When users import multiple datasets, they can choose to display a specific dataset individually or display all datasets simultaneously. This operation can be achieved using the red multi-select control shown in the figure

below.





: Users can choose mouse operations for the spectrum, such as zooming in, restoring, and

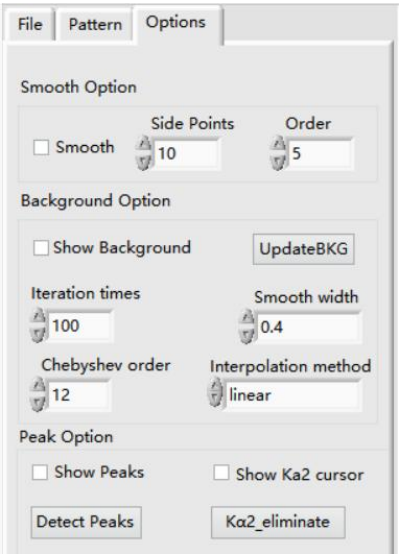


dragging.

: Right-clicking on the legend in the upper right corner of the spectrum allows users to change the properties of the corresponding curve, such as curve type (points, points + lines, lines), curve thickness, and visibility.

Chapter 3: Data preprocessing

Users can perform **various operations** on the imported data in the **Options section**, including data preprocessing, such as data smoothing (Smooth Option), background subtraction (Background Option), and peak finding (Peak Option). If the data is obtained from a Cu-target X-ray source, it may contain both Kα1 and Kα2 peaks; the program supports the subtraction of the Kα2 peak. **It is important to note that each dataset has a "Selected" attribute, and the program will only operate on the selected data** (for example, data smoothing, background subtraction, peak finding, and Kα2 peak subtraction).



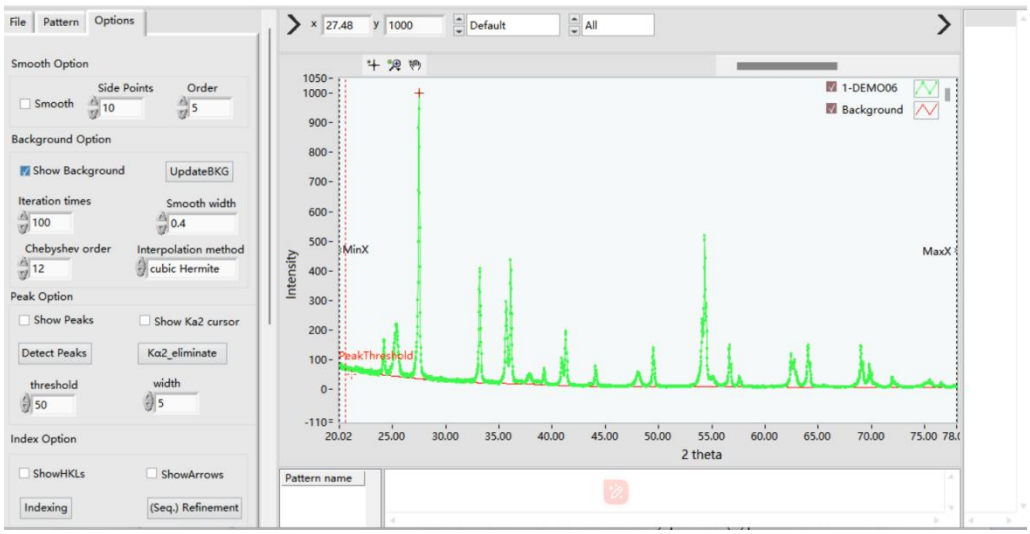
1.Smoothing Operation (Smooth Option): The smoothing method used by the program is the Savitzky-Golay smoothing method. This method works by performing polynomial fitting on a set of data points within a defined window to obtain a smooth curve. Therefore, it requires two parameters: the number of data points and the polynomial order. Users can adjust these two values to achieve better smoothing effects, but the polynomial order must be less than the number of data points.

- **Smooth:** Checking this option will apply the Savitzky-Golay smoothing to the selected data.
- **Side Points:** The number of data points within the smoothing window.
- **Order:** The polynomial order.

2.Background Operation (Background Option): The program uses two methods to obtain an appropriate background.

Brückner Method: This method iteratively searches for background points, smoothing and fitting these points to derive the background. This process can be automated; when a user selects a dataset, the program will automatically use this method to find the background.

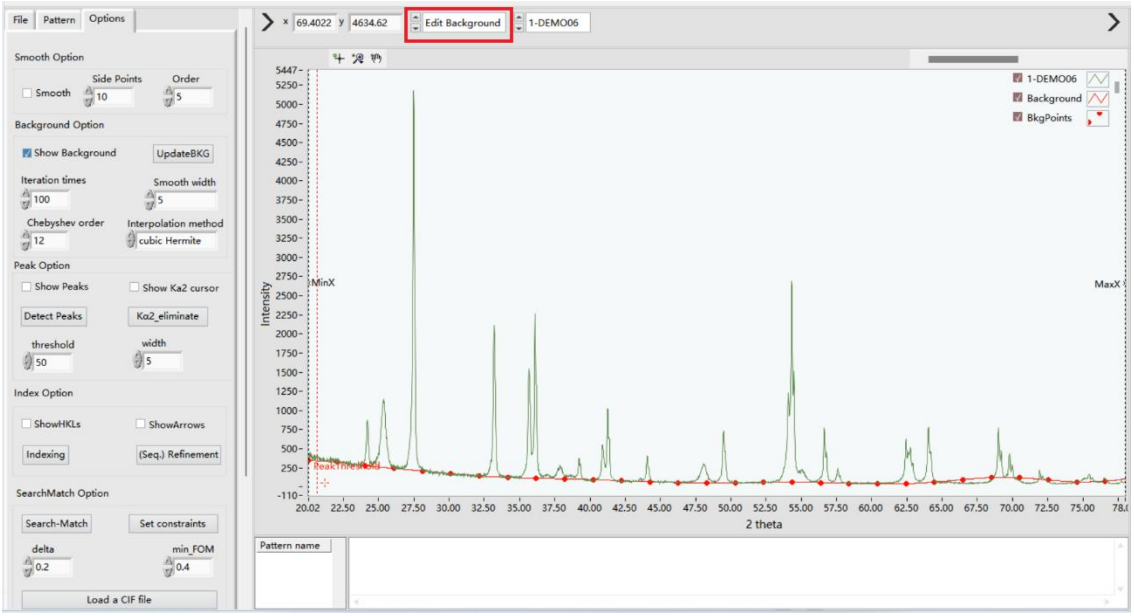
- **Show Background:** Check this option to display the background; uncheck it to hide the background.
- **Update BKG:** Click this button to update the background if you have changed the parameters for calculating the background.
- **Iteration Times:** The number of iterations for the background calculation algorithm.
- **Smooth Width:** The smoothing width applied by the background calculation algorithm.
- **Chebyshev Order:** The order of the Chebyshev polynomial used for fitting the background in the background calculation algorithm.



Another method is to manually adjust the positions of the background points. Users can enter background editing mode by selecting the "Edit Background" option in the control highlighted in red. By pressing the middle mouse button, users can add or remove background points, and holding down the mouse button on a background point allows users to drag and reposition it. This operation

can only be performed when a specific spectrum is displayed individually, and users can fit the data to obtain the background.

- **Interpolation Method:** This refers to the interpolation method used for the background points.

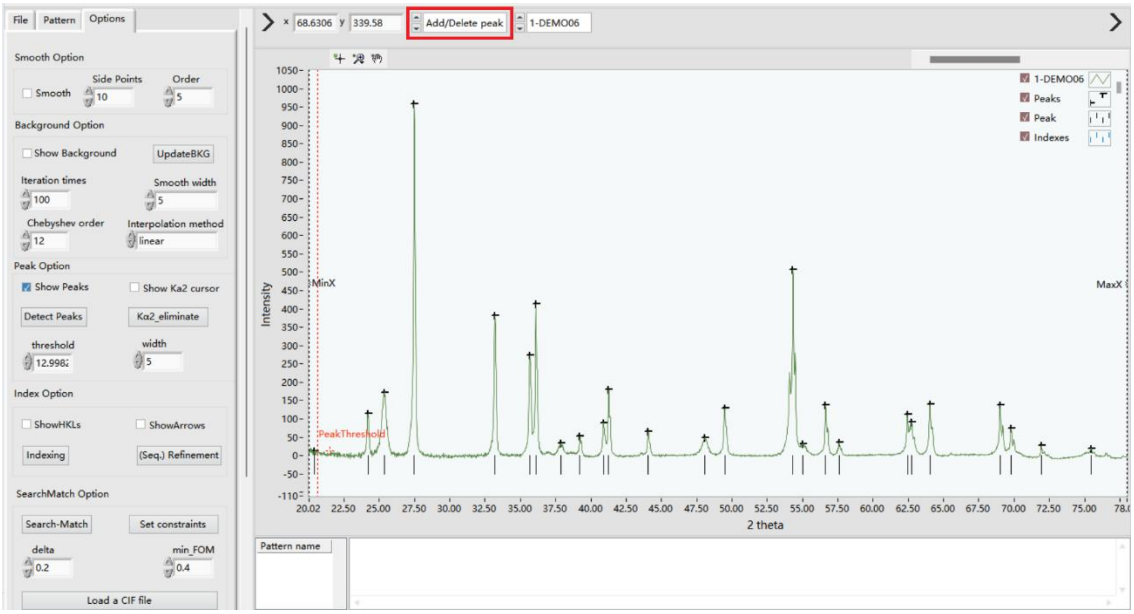


3.Peak Finding Operation (Peak Option): The program uses wavelet analysis to automatically detect peaks. When the data is set to selected status, the program will automatically find peaks based on the parameters provided, and the diffraction peaks will be displayed as black crosses on the graph (users can change this display by right-clicking the legend).

- **Show Peaks:** Check this option to display the diffraction peaks; uncheck it to hide the diffraction peaks.
- **Show Ka2 Cursor:** Display the Kα1 and Kα2 lines; users can drag the cursor to observe whether there are Kα2 peaks in the data.
- **Detect Peaks:** Clicking this button will execute the peak finding operation on the selected data.
- **Ka2 Eliminate:** Execute the Kα2 peak elimination algorithm on the selected data.
- **Threshold:** The minimum intensity for detecting diffraction peaks. This value can be input directly or adjusted by dragging the Peak Threshold cursor on the graph.
- **Width:** The width of the diffraction peak (i.e., the number of data points included in a single diffraction peak).

Users can also **manually add or delete diffraction peaks.** By selecting the "Add/Delete Peak" option in the control highlighted in red, users enter the diffraction peak editing mode. Pressing the middle mouse button allows users to add or remove diffraction

peaks.



Chapter 4: Search-Match Phase Identification

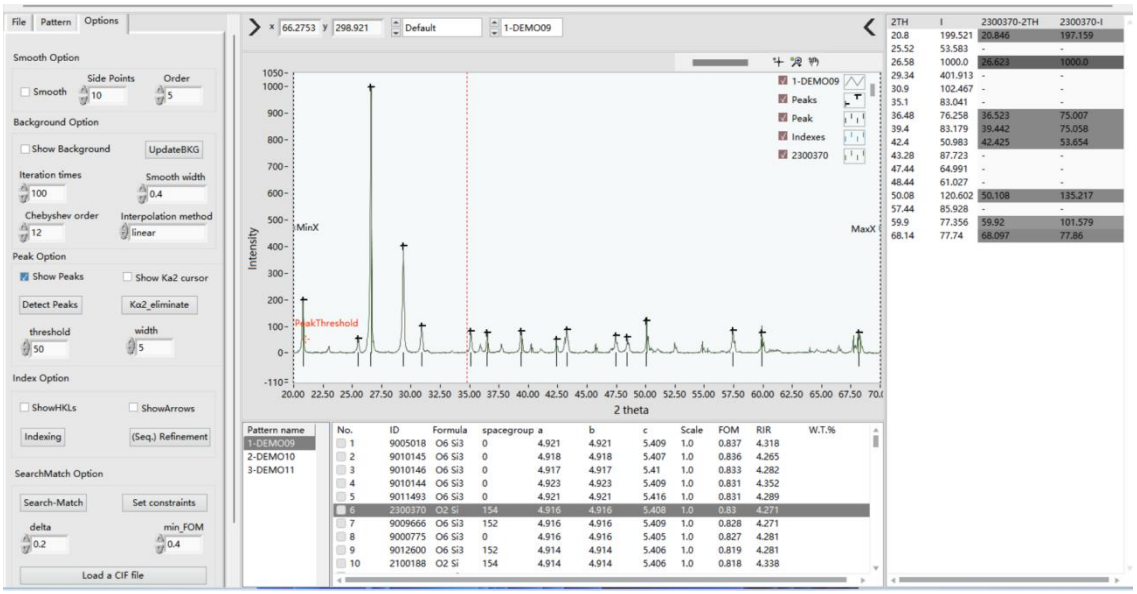
After importing the data and preprocessing it, users can perform the 'Search-Match' operation on the selected data. This operation searches the Crystal_db database for relevant phases using the three strongest diffraction peaks from the data. The program calculates a score based on a formula that compares the list of diffraction peaks from the database with the list from the data. This score reflects the degree of match between the diffraction peaks of the database phases and those in the data; the higher the score, the better the match.

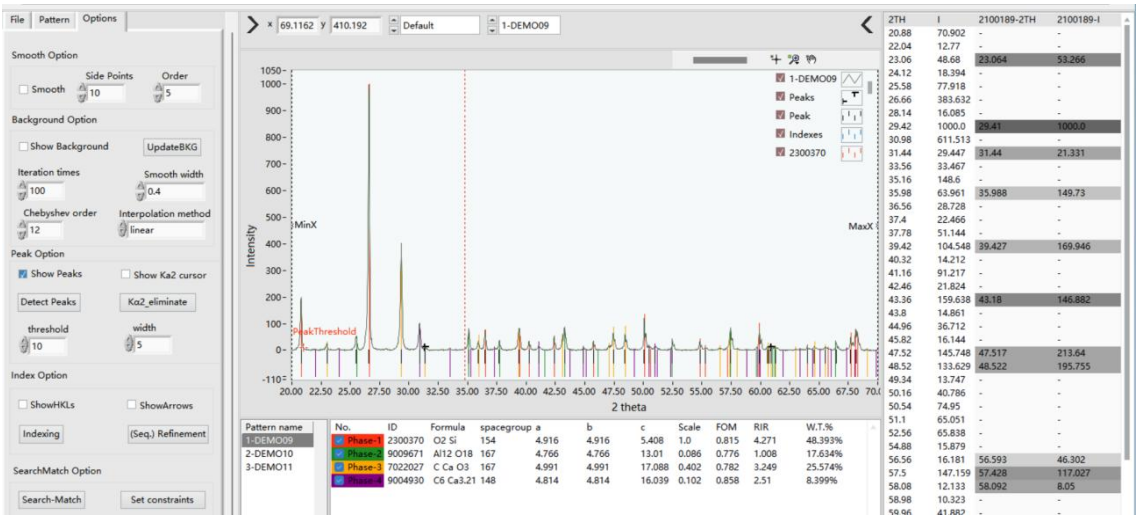
Users can compare the recommended phases in the results list below the spectrum and then select the most matching phase. If all diffraction peaks in the data match a specific peak in the diffraction peak list of that phase, the search-matching process is complete. Conversely, the program will continue the search-matching operation using the remaining unmatched diffraction peaks.

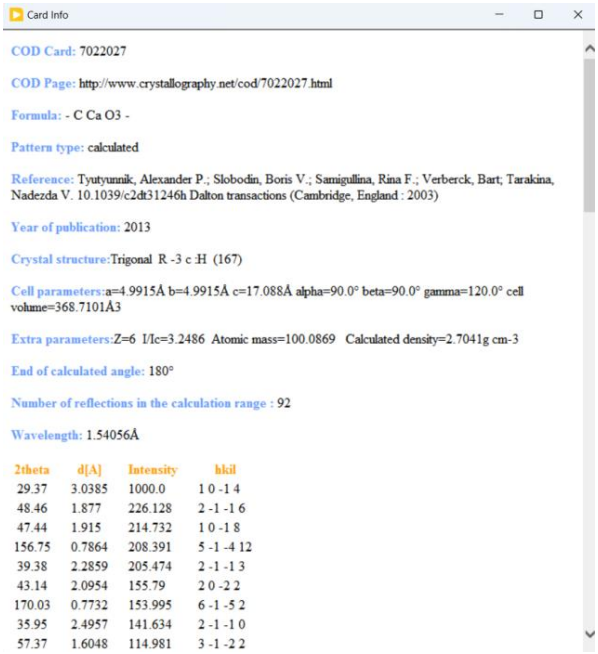
During the **search-match** process, **setting known search constraints** (such as chemical elements, crystal system, space group, and unit cell parameters) will **speed up the execution of this operation** and **allow for a more precise identification of suitable phases**. The following will explain this process in conjunction with the functions of various components of the software.

1.After clicking the "Search-Match" button, the program performs a search-matching operation on the selected data one by one, and the results will be displayed in the results list below the spectrum. As shown in the figure, **the results list is divided into two columns**: the left column records the names of the data that have undergone the search-matching operation. When one of the data entries is selected, the program will switch the spectrum to that data's spectrum, and the search-matching results on the right will also switch to the corresponding data's results. Users can click on the phases in the search-matching results list to view the diffraction peak information of that phase (which will be displayed as a black line in the figure). Users can check the appropriate phases; once checked, the program will automatically perform another search-matching operation on the remaining unmatched diffraction peaks. The list on the right side of the spectrum displays the list of diffraction peaks from the data and the corresponding matching diffraction peaks from the phases. Additionally, double-clicking on a phase in the search-matching results list will visualize the information for that phase.

- **Search-Match**: Button for performing the search-matching operation.
- **delta**: An important parameter in the search-matching process. When matching the diffraction peaks of the experimental data with those in the database, if the absolute difference between the two peaks is less than delta, they are considered to match. Therefore, the smaller the delta, the more precise the search-matching results. However, if the matching criteria are too strict, it may lead to not finding a corresponding phase, in which case the delta value should be increased appropriately.
- **min_FOM**: FOM (Figure of Merit) refers to the score indicating the degree of match between the diffraction peaks of the database phases and those of the data. If the score is less than min_FOM, that result will not be displayed in the search-matching results list.





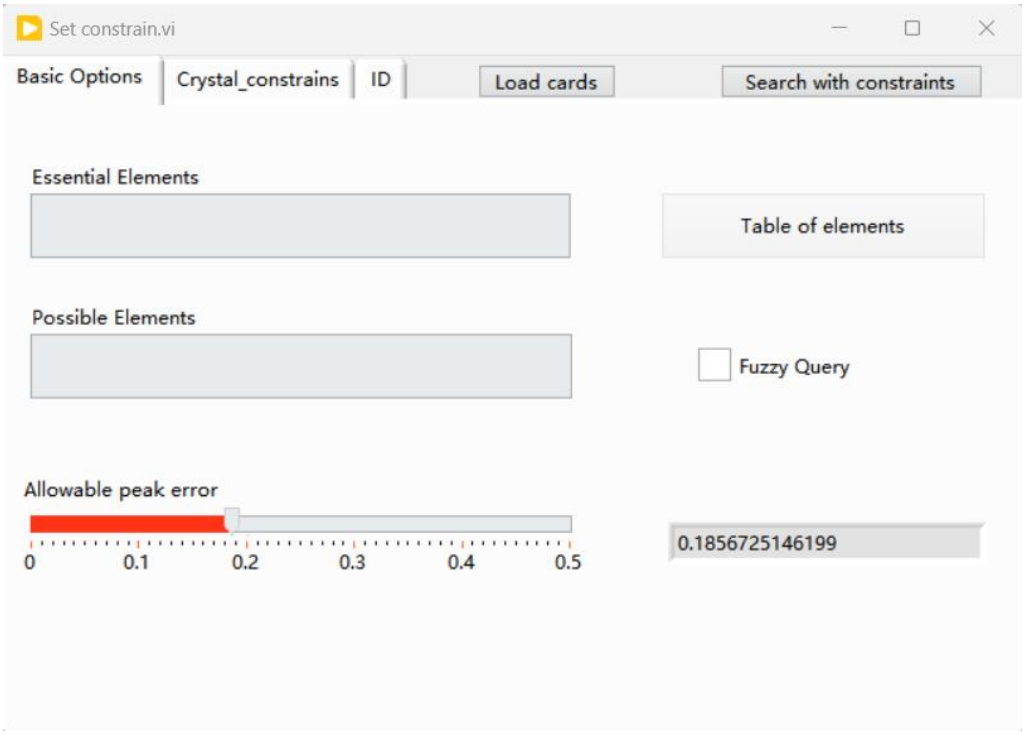


2.During the search and matching process, setting known search constraints (such as chemical elements, crystal system, space group, and cell parameters) can speed up execution and enhance the accuracy of finding suitable phases.

Set constraints: Clicking this button will pop up a window for setting search matching constraints. The constraints are divided into three sections:

1. **Basic Options:** This section primarily sets the chemical element information.
2. **Crystal Constraints:** This section is used to set crystal structure information.
3. **ID:** This part allows you to limit the phase by its serial number (corresponding to the ID in the COD database).

After configuring the search constraints, clicking the "**Load cards**" button will search for corresponding phases from the database based on the constraints and load them into the results list. Clicking "**Search with constraints**" will initiate the search and matching process incorporating these constraints.



Essential Elements:

The results must include all elements from this list.

Possible Elements:

The results should include one or more elements from this list.

Fuzzy Query:

This condition takes into account two scenarios while satisfying the above two conditions:

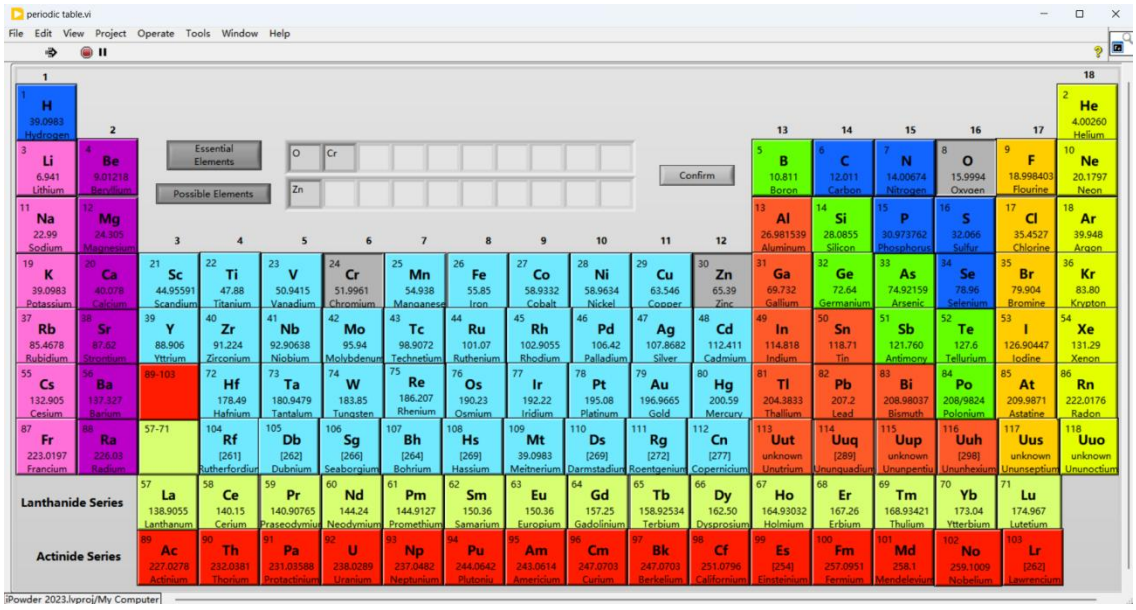
- If this option is unchecked, the results will only contain elements from the two lists above.
- If this option is checked, the results may include additional elements beyond those in the specified lists.

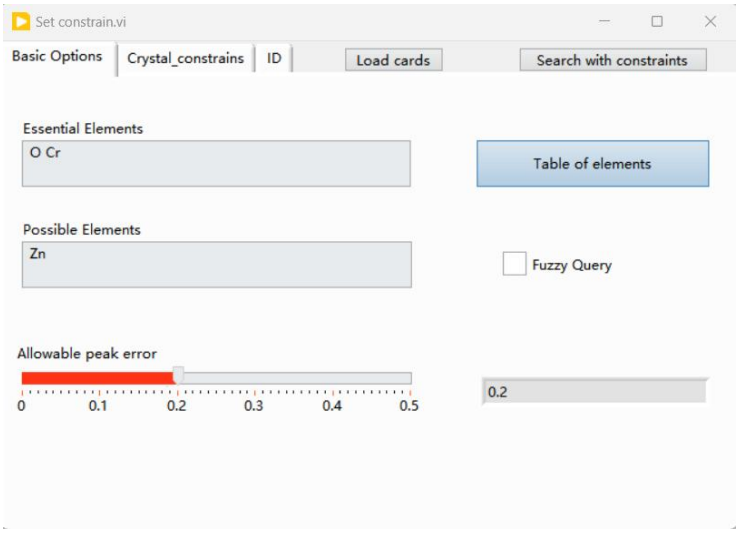
Allowable Peak Error:

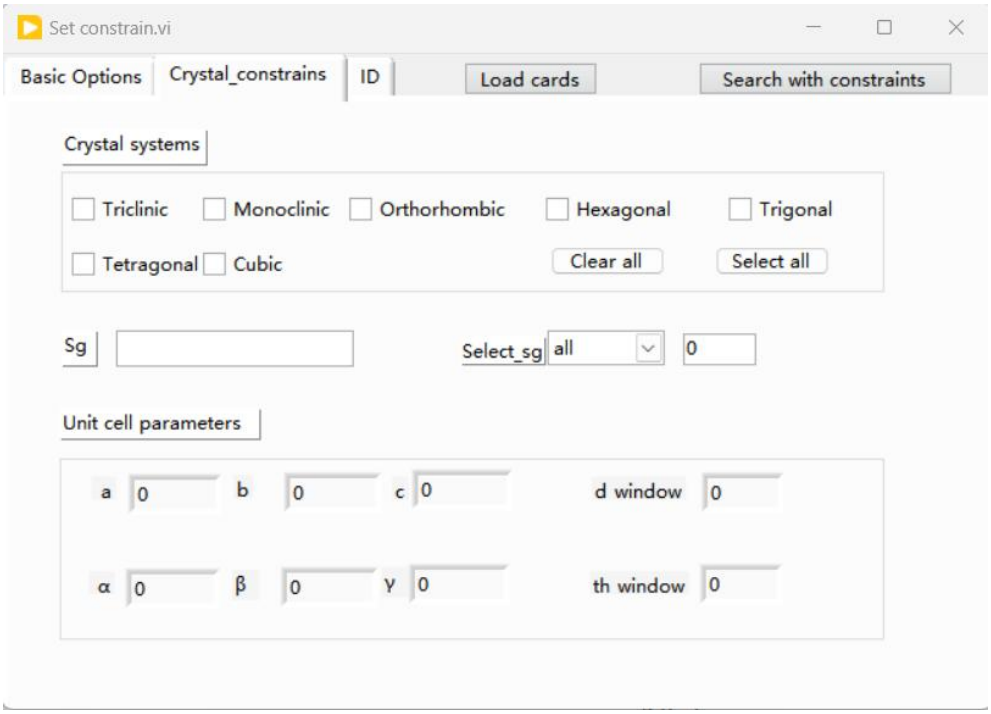
This refers to the **delta value**. It is a critical parameter during the search and matching process. When matching the diffraction peaks from experimental data with those from the database, if the absolute difference between them is less than delta, they are considered a match. Thus, the smaller the delta, the more precise the search results. However, if the matching criteria are too strict, it may lead to the inability to find corresponding phases, so it may be necessary to increase the delta value appropriately.

Table of Elements:

Clicking this button will open a **periodic table window** for selecting Essential Elements and Possible Elements. After selecting the desired elements (the icons will gray out), clicking the “Essential Elements” or “Possible Elements” button will add the selected elements to the corresponding list. For example, after selecting "O" and "Cr" and clicking "Essential Elements," the corresponding list will include these two elements. Then, selecting "Zn" and clicking "Possible Elements" will add "Zn" to the respective list. Click “Confirm” to finalize the selections, and the chosen elements will appear in the Set Constraints pop-up window.







Crystal Systems:

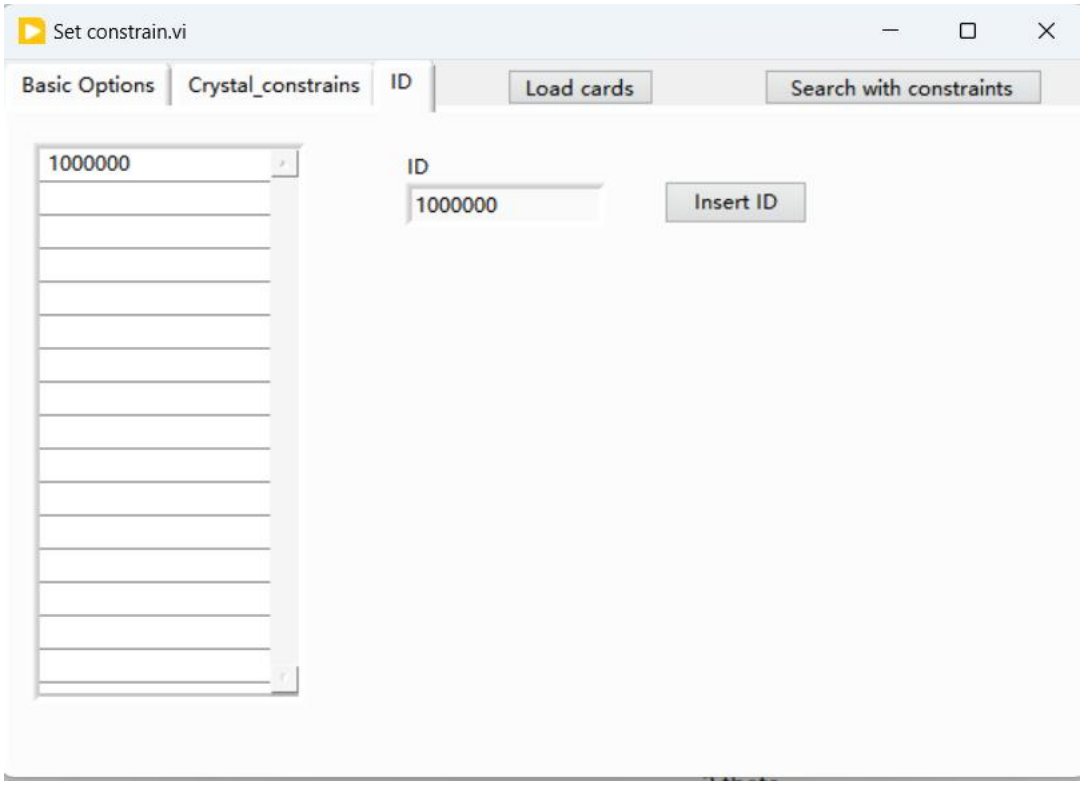
Select the type of crystal system.

Space Group (Sg):

Enter the space group number. Each space group number should be followed by a comma. For example, entering "15," indicates that the results should only include those from space group 15.

Unit Cell Parameters:

Users can input their estimated unit cell parameters along with a margin of error. For example, if a=9a = 9a=9 and the error window is set to 0.2, the results returned will include those from the database that satisfy the condition 8.8<a<9.28.8 < a < 9.28.8<a<9.2.



In the ID section, users can input the entry numbers from the COD database. Clicking the "Insert ID" button will add the specified number to the list, ensuring that the results will only include entries from this list.

Chapter 5: Indexing

When users are unable to find matching phases in the database, the indexing operation can help them obtain unit cell parameters and assign Miller indices to various diffraction peaks using the peak information from the data, especially for low-angle diffraction peaks. This will facilitate subsequent analyses.

The indexing functionality in iPowder is based on four indexing software programs (DICVOL, TREOR, ITO, McMaille), each of which employs different methods to achieve indexing. However, these programs do not provide a graphical interface, making them somewhat complex for users. iPowder automates the generation of input files for these software programs during the indexing operation and integrates their results into a results list for easier analysis.

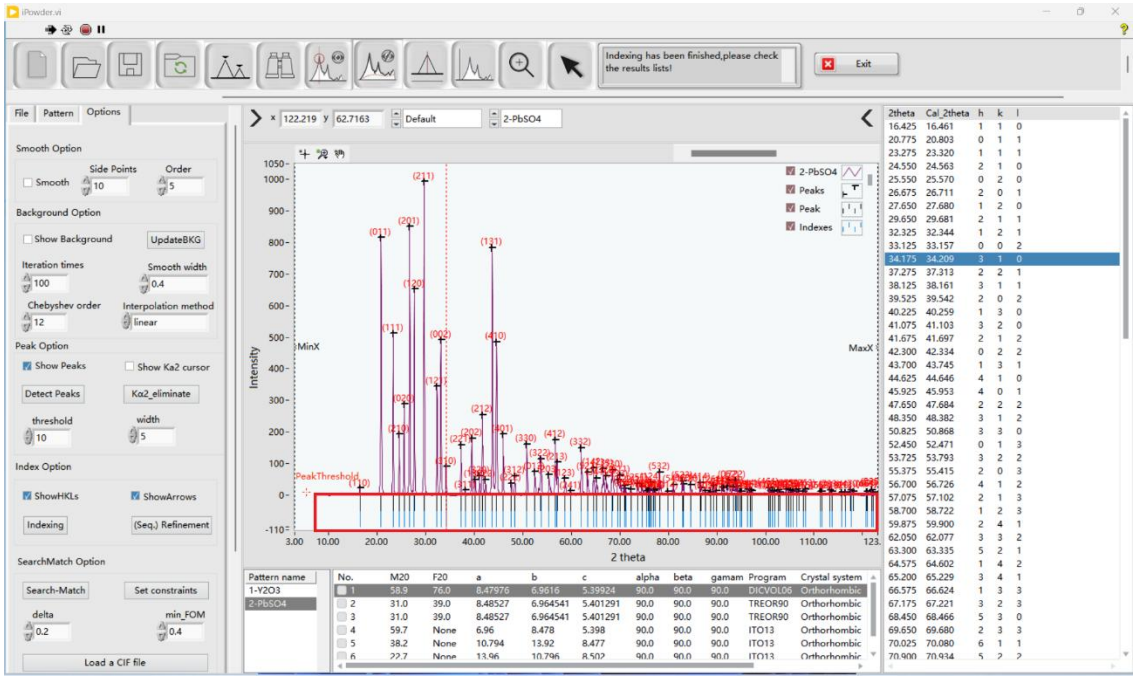
After users have preprocessed the data by smoothing, background subtraction, and peak searching to obtain suitable results, they can proceed with the indexing operation. When the user presses the "Indexing" button in the Index Option, an **indexing settings window** will pop up.

Indexing Operation Steps

1. **Select Software and Crystal System**:
    - The user selects the indexing software to be used (such as DICVOL, TREOR, ITO, or McMaille) in the indexing settings window.
    - The user also needs to choose the possible crystal system of the phase.
2. **Confirm Settings**:

- After making the selections, click the “OK” button, and the program will begin the indexing operation on the selected data one by one.

3. **Result Display**:
    - The indexing results will be displayed in the results list at the bottom of the graph. **Note that McMaille usually runs slower.**
4. **Result Sorting**:
    - The integrated indexing results in iPowder are sorted based on the **M20** score. Both M20 and F20 are scoring metrics for the indexing results; the higher the score, the more reliable the indexing results.
5. **Switching and Viewing Results**:
    - Users can switch between different data and their corresponding indexing results through the data name list at the bottom of the graph.
    - When a user clicks on a specific indexing result, the detailed indexing results will be displayed on the right side of the graph.
6. **Assessing Result Quality**:
    - Users can evaluate the quality of the indexing results by observing the alignment of the black vertical lines (representing the positions of the data diffraction peaks) with the blue vertical lines (representing the positions of the indexed diffraction peaks) in the area marked with a red box.



Show HKLs: Display the Miller indices corresponding to each diffraction peak and the indexing results.

Users can check the indexing results, and upon selection, the program will refine the indexed cell parameters and also calculate the zero-point drift of the

data.

Chapter 6: Cell parameter refinement

Cell parameters are fundamental geometric parameters that describe the crystal structure, including lattice constants and angles, and can directly reflect the structural characteristics of materials. Cell parameters may change under different environmental conditions (such as temperature, pressure, stress, etc.), and these changes can reveal important information about phase transitions, internal stresses, and defect formation in materials. Therefore, accurately extracting cell parameters from experimental diffraction data and tracking their variations is a significant requirement for researchers.

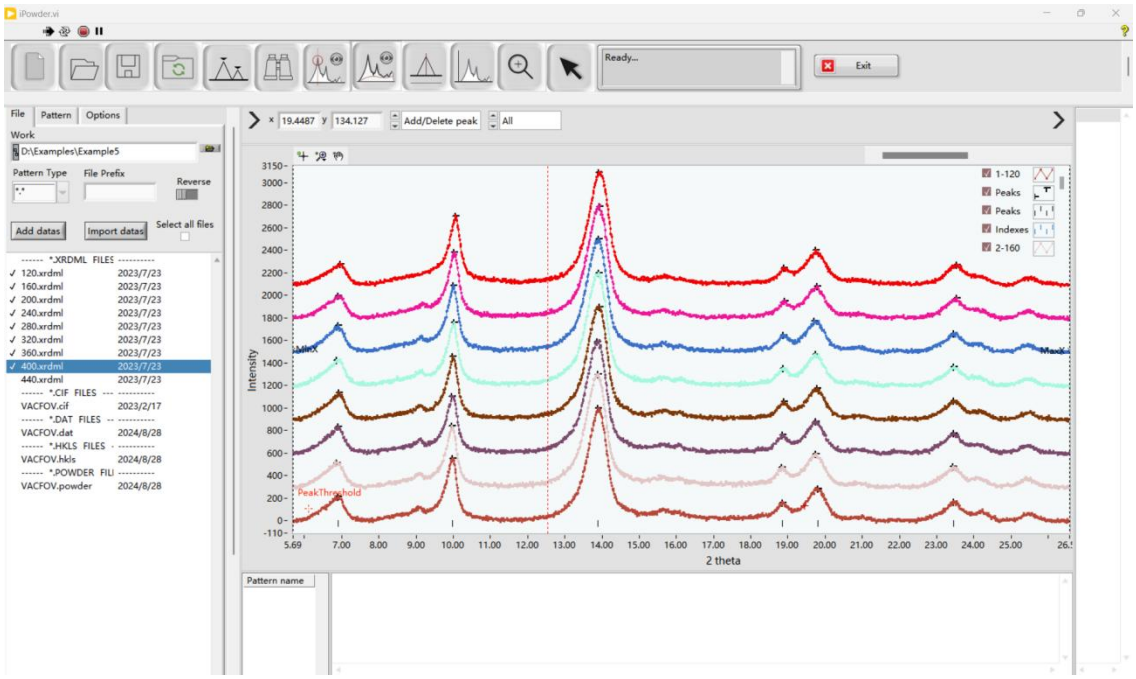
To address these issues, iPowder integrates the function of cell parameter refinement. This function optimizes the theoretical calculation of the error between the diffraction angles and the experimental diffraction angles based on the least squares method. After selecting a list of diffraction peaks, it can refine given initial cell parameters to obtain more accurate values. Additionally, the software supports the analysis of multiple datasets, enabling the capture and revelation of the patterns of cell parameter changes under varying external conditions.

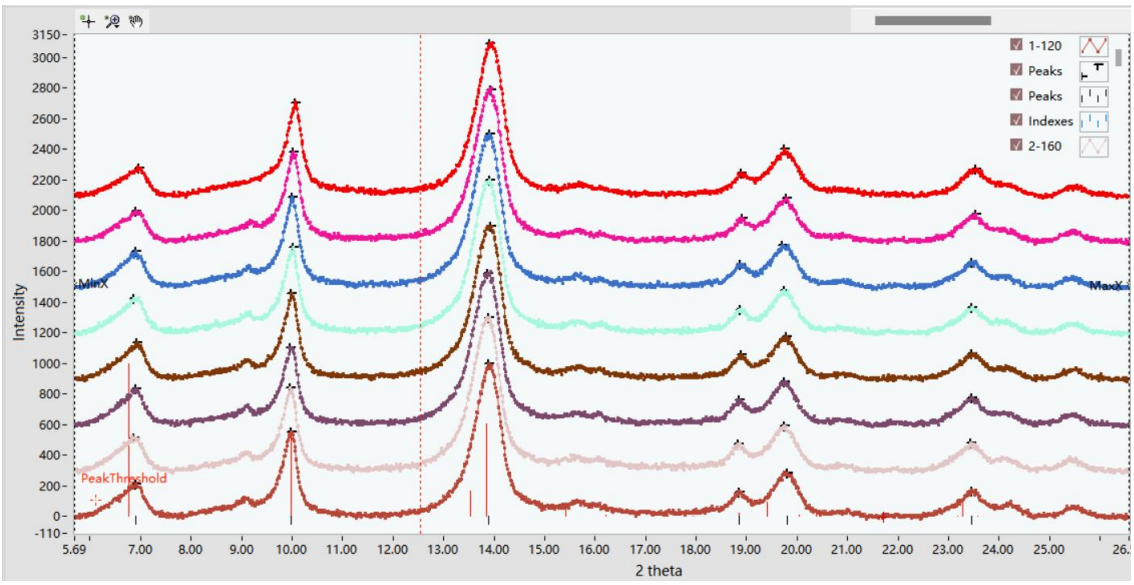
In this section, we will use the variable temperature XRD data of VACFOV, an organic crystal, from 120K to 360K (data from Example 5) as a case study to illustrate this function. In this case, the initial cell parameters are provided by a CIF file (the program also supports using the results from search-matching or indexing to provide initial cell parameters, and users can also set their own initial parameters).

- First, **import the data** and set the angular range from 5.5 to 26.5, then preprocess the data.
- Next, click the "**Load a CIF file**" button in the Options menu to import a CIF file. The program will extract the cell parameters from the file and simulate the powder diffraction pattern using these parameters, which will be used for cell parameter refinement.

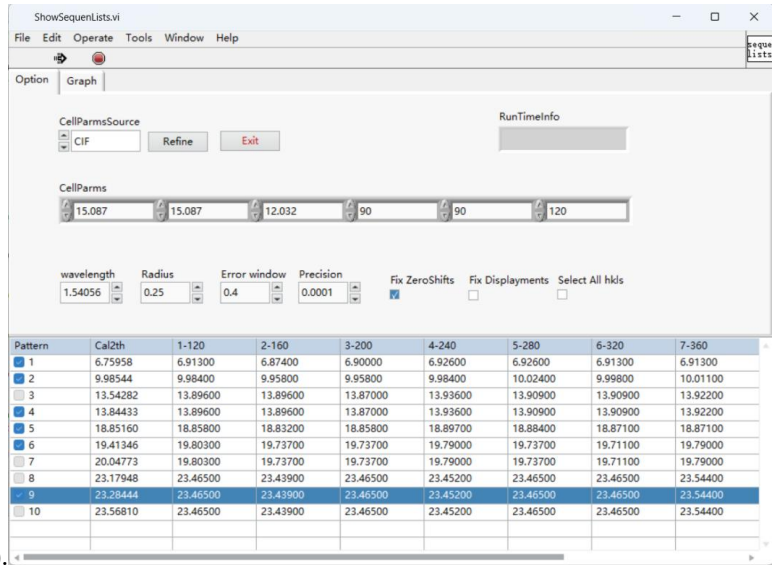
Load a CIF file: After clicking this button, you need to select a CIF file. Once the CIF file is selected, the program extracts the cell parameters and other information from it and performs a simulated calculation of the diffraction pattern. The calculated simulated powder diffraction pattern will be displayed in the graph as red vertical lines.

- Finally, click "**(Seq.) Refinement**" to enter the cell parameter refinement interface.





(Seq.) Refinement: After clicking this button, a window will pop up for refining the cell parameters. The program will match the diffraction peak list of the selected data with the calculated diffraction peaks based on proximity, forming pairs of diffraction peaks. These results will be displayed in a list. The refinement process essentially uses the least squares algorithm to minimize the absolute value of the difference between the calculated diffraction peaks and the corresponding data diffraction peaks. Select the pairs of diffraction peaks that need refinement from the list, and then click the "Refine" button to perform the refinement. It is worth noting that when refining the cell parameters for multiple datasets, it is recommended to check the "Fix ZeroShifts" option to fix the zero-point drift (since this value will not vary significantly within the same batch of XRD data).



CellParmsSource: The source of the initial cell parameters, with four options: "User define," "SearchMatch," "Indexing," and "CIF."

Refine: After clicking this button, the program will refine the cell parameters using the selected pairs of diffraction peaks from the list.

Exit: Clicking this button will exit the program.

RunTimeInfo: Displays information about the program's runtime.

CellParms: The initial cell parameters to be refined. When the CellParmsSource is set to "User define," these parameters can be edited.

Wavelength: The wavelength of the X-ray.

Radius: Instrument parameters required for calculating sample displacement. This can be omitted if sample displacement is not considered.

Error window: The error window for matching the experimental data diffraction peaks with the theoretical calculated diffraction peaks. If the absolute value of their difference is less than this value, they are considered matching and will be added to the list.

Precision: The precision of the cell parameters. The higher the required precision, the longer the calculation time.

Fix ZeroShifts: Fixes the zero-point drift during the refinement process (using the zero-point drift obtained from the first dataset for the remaining datasets).

Fix Displacements: Fixes the sample displacement during the refinement process (using the sample displacement obtained from the first dataset for the remaining datasets).

Select All HKLs: Selects all diffraction peaks in the list for refinement.

Chapter 7: Introduction to Keyboard Shortcuts

 ：Import a data file.

 ：Select the work folder.

 ：Save the diffraction peak list to the temporary working directory (in the directory where the data is located).

 ：Clear the contents of the results list.

 ：Indexing.

 ：Search-Match.

 : Show peaks.

 ：Show backgrounds.

 ：Add or remove diffraction peaks.

 ：Scale the graph to an appropriate position.

 ：Adjust the mouse function to zoom in.

 ：Adjust the mouse function to cursor.