Sleve+ Introduction

- Sleve+ is a Plug-In module integrated in the PDF-4 products. Sleve+ is licensed separately at an additional cost except for the PDF-4/Organics database. Sleve+ will activate for a free 30-day trial period or until the product is registered. A license for Sleve+ may be purchased along with a PDF-4 license or following the free 30-day trial period.

- Sleve+ accepts user data and searches the ICDD reference databases (PDF-4+, Organics, and Minerals) for comparison. There are several hundred thousand entries for both PDF-4+ and Organic databases. Therefore, various methods, especially the newly implemented features and improvements listed below are used to ensure efficient searches to obtained the accurate matches.

  - New Similarity Index with normalized R index
  - New data processing menu for background subtraction, Ka2 stripping, smoothing, and peak finding
  - New Matches Filter with Common Filters of Primary Patterns or Mineral Related or Common Phases
  - Instant display of overlapping input and reference patterns

- The matched results depend on the criteria and methods specified. This tutorial demonstrates the available methods and the operational procedures of Sleve+.

SIeve+ Purpose

SIeve+ will identify patterns of various X-ray powder diffraction (XRPD) data files:

1. **Single phase XRPD patterns**
2. **Multi-phase XRPD patterns**
3. **Data file containing only XRPD peaks (*.xml)**
4. **Raw instrumental and/or converted data file formats:**

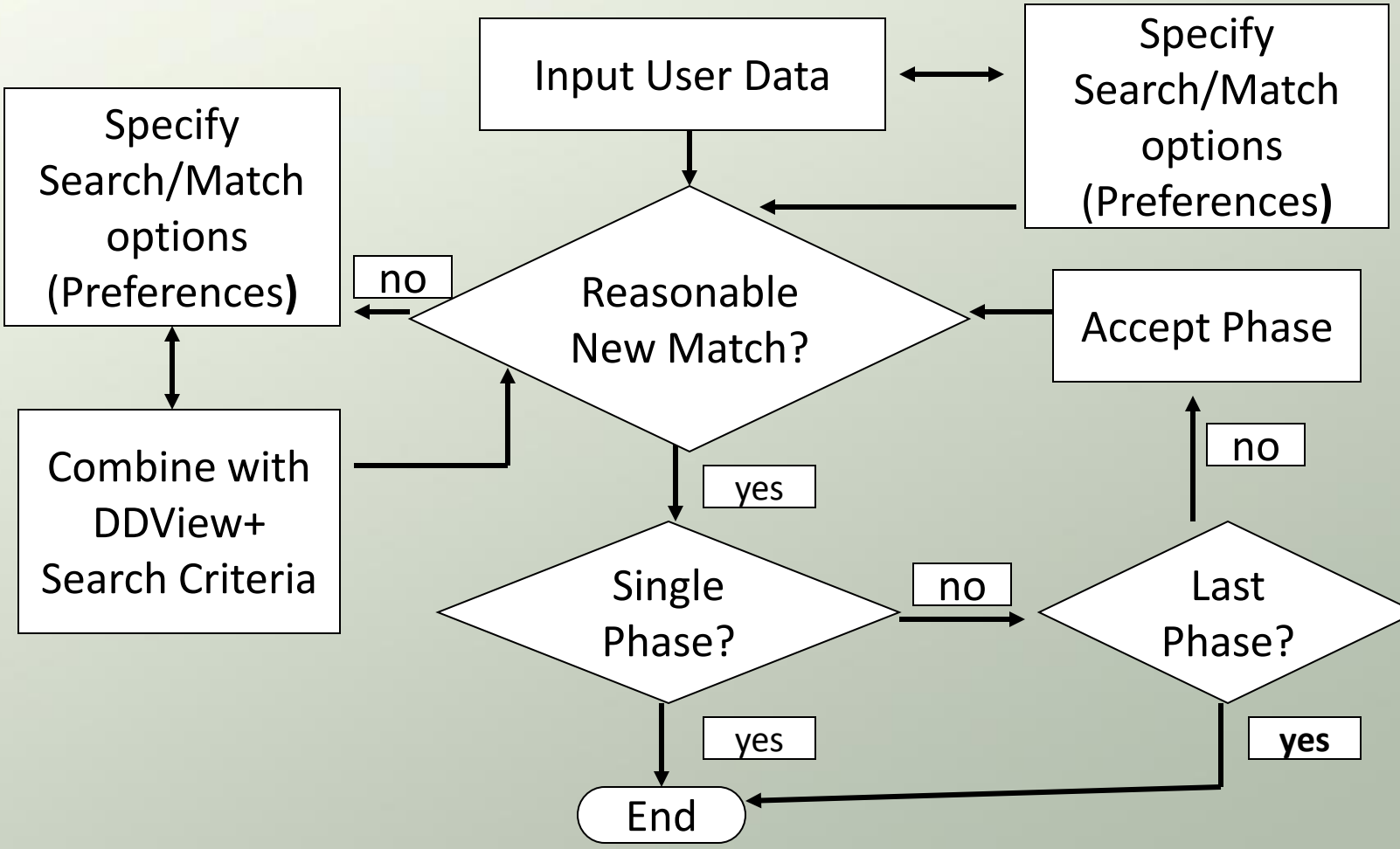
ICDD	**(*.csv, *.prn, *. *.pd3)**
CIF	**(*.cif)**
X-Y	**(*.xrd)**
GSAS	**(*.gsas, *.gss, *.gsa, *.raw, *.dat)**
Bruker	**(*.uxd)**
MDI	**(*.mdi)**
PANalytical	**(*.xrdml, *.udf)**
Scintag	**(*.ard)**
Sietronics	**(*.cpi)**

SIeve+ Methods

There are different search methods and match criteria used to get accurate results. They include:

1. Hanawalt with the option of 3 rotations – This uses the strongest line for the search. The rotation involves up to the 3 strongest lines
2. Fink with the option of 8 rotations – This uses the longest of the 8 strongest lines for the search. The rotation involves up to the 8 strongest lines.
3. Eight largest d-spacings with the option of 8 rotations – This method uses the largest of the 8 longest lines for the search.
4. Search window and Match window are options for user to specify the error margin.
5. Weighted GOM, Pattern GOM, and Similarity Index are quantities used to rank the matched reference patterns.
6. Data file import processing options – Manually add/delete points or use Cubic Spline function for background subtraction; Add diffraction peaks; Strip Ka2 peaks prior to file import.

SIeve+ Search/Match Procedure



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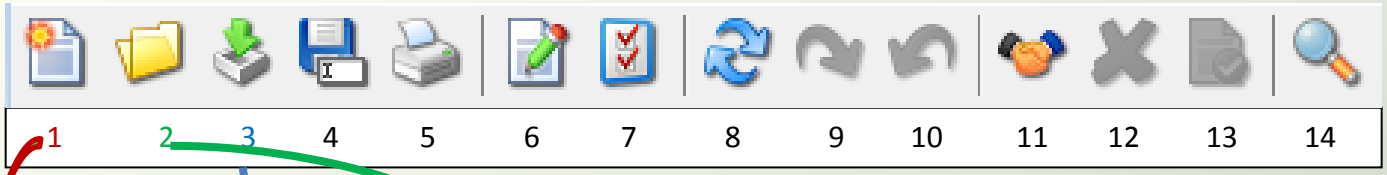
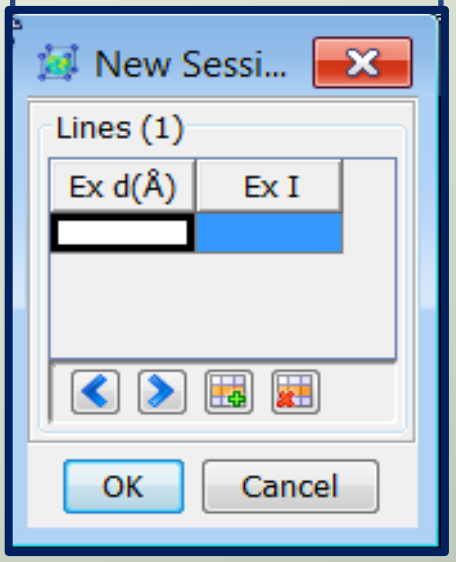
SIeve+ User Data Input

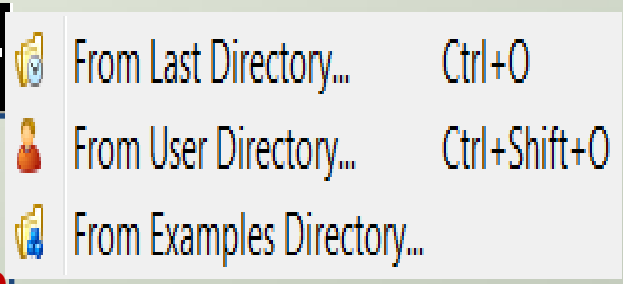
Frequently Used Icons



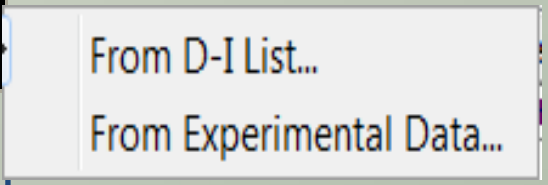
1. New Session:
Manually input peaks (or Copy/Paste)



2. Open Session:
Peak files -
56 sample multiphase peak files in the examples directory



3. Import Session:
Raw experimental digitized data files



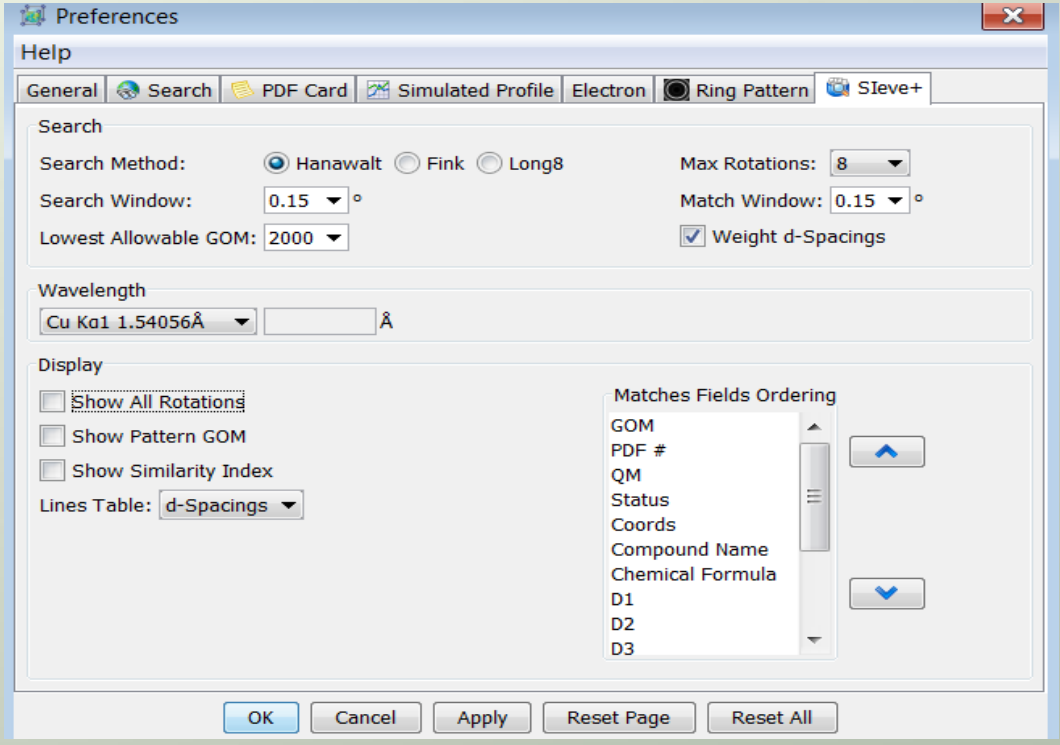
SIeve+ Miscellaneous Options

Frequently Used Icons

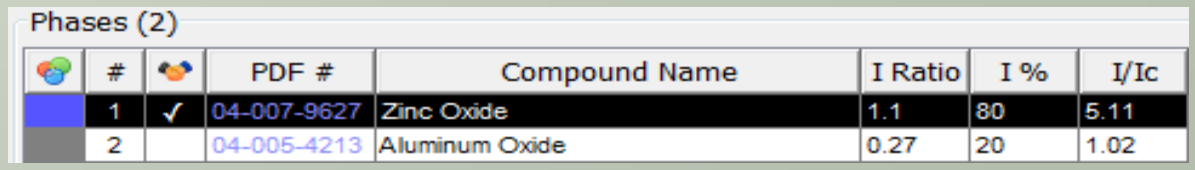
1	2	3	4	5	6	7	8	9	10	11	12	13	14

7. SIeve+ Preference:
Methods and Criteria window

- **Search/Match error margin**
- **Wavelength specification**
- **GOM limit specification**
- **Hanawalt/Fink/Long8 selection**
- **Show/Hide All Rotation**
- **Show/Hide Pattern GOM**
- **Show/Hide Similarity Index**
- **D-Spacings/2Theta selection**



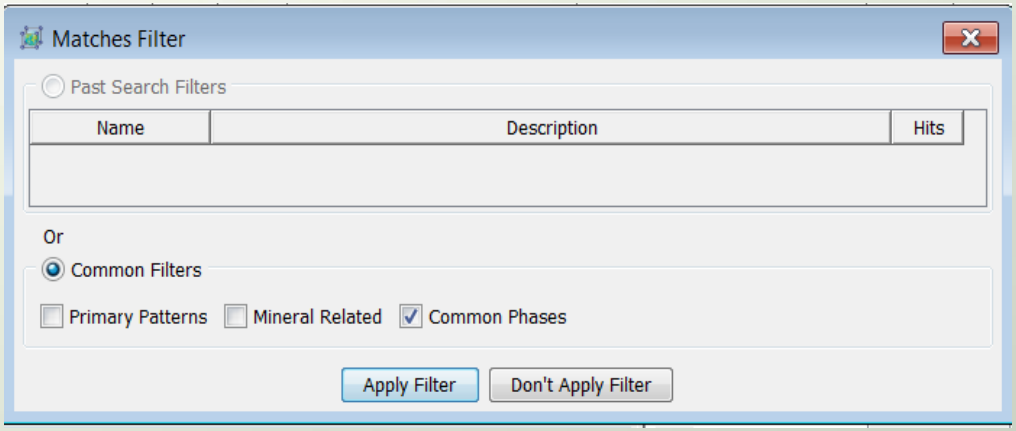
11. Accept Phase
Comparison between the user data and reference data continues after "accepting" the matched phase



#		PDF #	Compound Name	I Ratio	I %	I/Ic
1	✓	04-007-9627	Zinc Oxide	1.1	80	5.11
2		04-005-4213	Aluminum Oxide	0.27	20	1.02

SIeve+ Phases Table – Multiphase Hit List

Matches Filter:
Combine the chemical, physical, and crystallographic property from the previous search with the d-spacings/intensity search results.



Phases (3)

#		PDF #	Compound Name	I Ratio	I %	I/Ic
1	✓	04-007-9627	Zinc Oxide	1.1	49	5.11
2	✓	04-005-4213	Aluminum Oxide	0.27	12	1.02
3		04-002-5299	Calcium Fluoride	0.89	39	3.84

Three matched referenced patterns from the PDF database:

- **The mixed phases contain Zinc Oxide, Aluminum Oxide, and Calcium Fluoride**
- **Sum (intensities) of matched peaks for reference pattern 04-007-9627/ Sum (intensities) of matched peaks for unknown mixed phase = 1.1**
- **(1.1 /(1.1+0.27+0.89)) * 100 = 49**
- **I/Ic: reported reference intensity ratio in PDF database for 50/50 weight percent for [Reference Pattern]/Corundum**

SIeve+ Lines Table - Phase Comparison

d-spacings/Intensities of Multi-phases and those of reference patterns comparison table:

1st & 2nd columns: Experimental data.

3rd & 4th columns: First matched reference pattern data.

5th & 6th columns: Second matched reference pattern data.

7th & 8th columns: Third matched reference pattern data.

Diffraction Patterns | Lines (41 of 44)

Ex d	Ex I	P1 d	P1 I	P2 d	P2 I	P3 d	P3 I
3.47551	13			3.476710	64		
3.15138	82					3.153050	98
2.81186	58	2.812850	58				
2.60078	42	2.602000	45				
2.54880	23			2.548790	100		
2.47398	100	2.474510	100				
2.37763	9			2.377000	47		
2.08383	24			2.083380	98		
1.93049	89					1.930840	100
1.92175	4						
1.90973	23	1.910090	23				
1.73900	11			1.738350	49		
1.64662	27					1.646630	30
1.62391	35	1.624000	31				
1.60066	23			1.600060	94		
1.47645	30	1.476480	26				
1.40671	6	1.406430	4				
1.40377	8			1.403160	37		
1.37777	26	1.377680	22				
1.37324	14			1.372360	56		
1.36533	10					1.365310	10
1.35777	12	1.357720	10				
1.30105	2	1.301000	2				
1.25308	8					1.252890	9
1.23833	6	1.237250	3	1.238110	16		
1.23352	2			1.233150	8		
1.11489	14					1.114770	15
1.09266	8	1.092470	6				
1.06326	3	1.063160	2				

SIeve+ Case-1

Objective: Search for reference patterns in the PDF database to match mixed phase peaks in a user file

1. File => Open Session => From Examples Directory

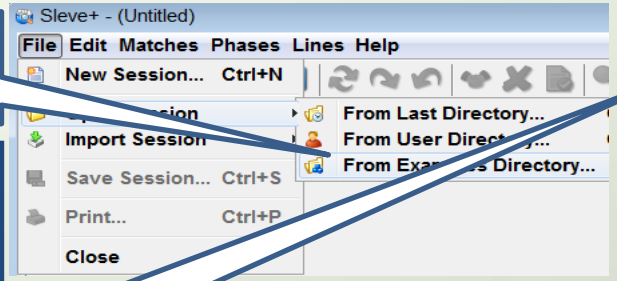
2. Select a peak file: Session_7_4_5.xml

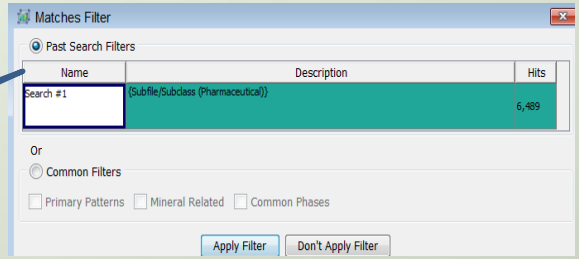
3. Select Matches Filter "ICDD Pharmaceutical" from the previous search results

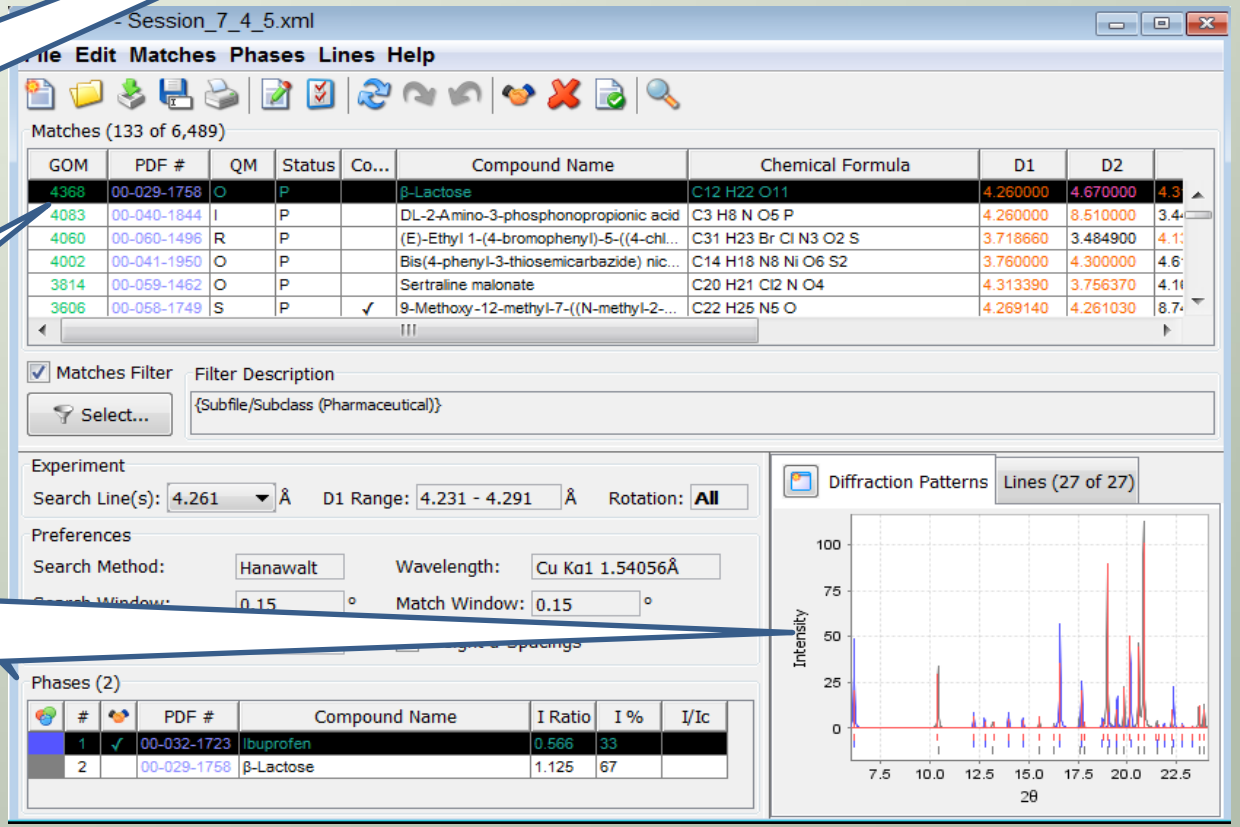
4. Accept "Ibuprofen", continue Searching

5. 2nd matched phase β –Lactose with highest GOM

6. Diffraction Patterns: unknown and matched reference patterns' d-spacings and intensities

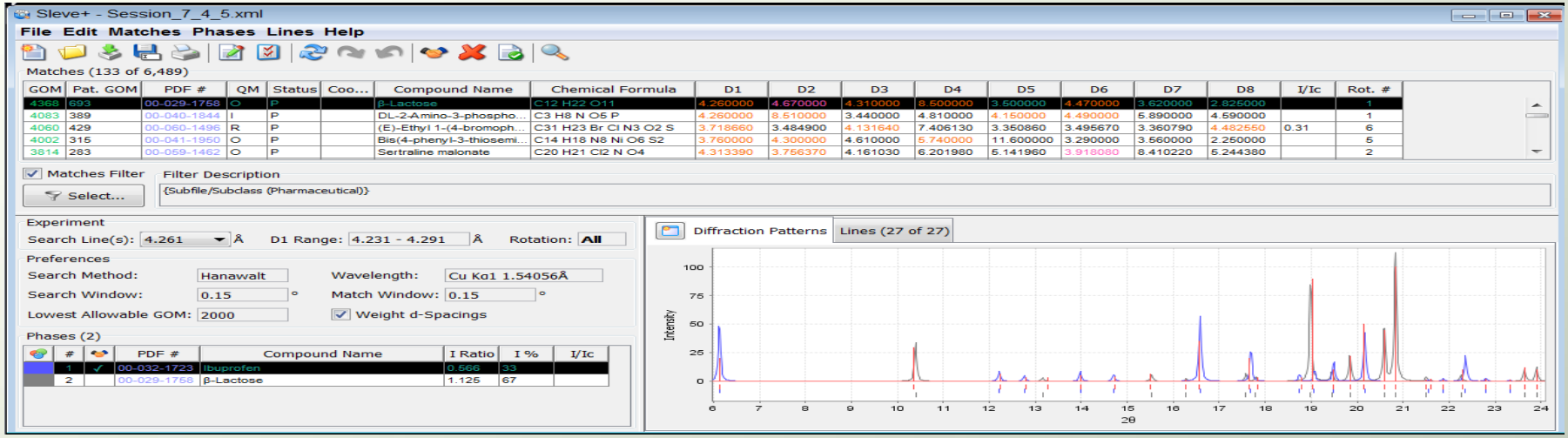






SIeve+ Case-1

Interpretation of matched results



Δ = window * d / tan(arcsin(λ / (2 * d)))

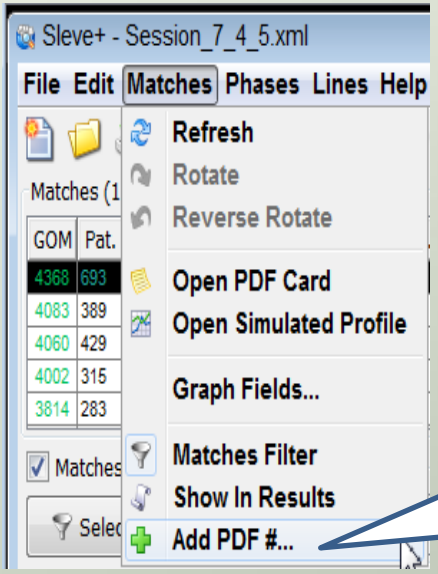
A Match: reference d-spacing <= experimental d-spacing +/- Δ

Red: matched lines; **Black**: non-matched lines; **Purple**: overlapped lines

Goodness of Match (GOM):

GOM (each line) = **(** 1 - (line error / Δ) **)** * **(** 1 - (line error / Δ) **)** * 1000

GOM (Final Weighted) = (D1 GOM * 3) + (D2 GOM * 2) +
(D3 GOM) + (D4 GOM * 0.4) + (D5 GOM * 0.4) +
(D6 GOM * 0.4) + (D7 GOM * 0.4) + (D8 GOM * 0.4)



If a suspected reference pattern is not in the “Matches” table, users can use the new option “Add PDF # “ under the “Matches” tab to manually add the pattern to the “Matches” table.

SIeve+ Case-2

Objective: Find matched patterns in the PDF database with the experimental raw data containing three phases of compounds.

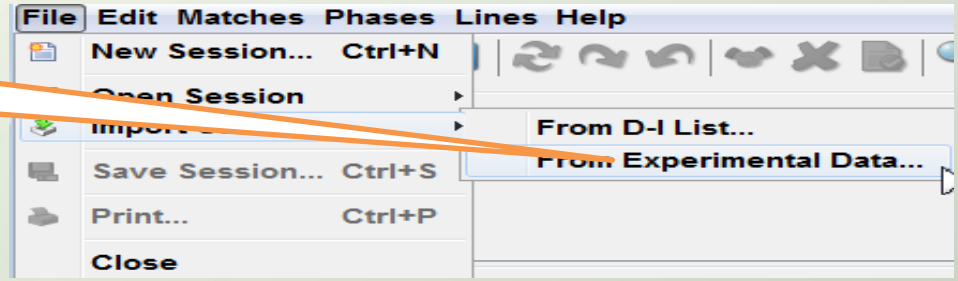
1. Import raw experimental data file – a mixture of three phases for search/match

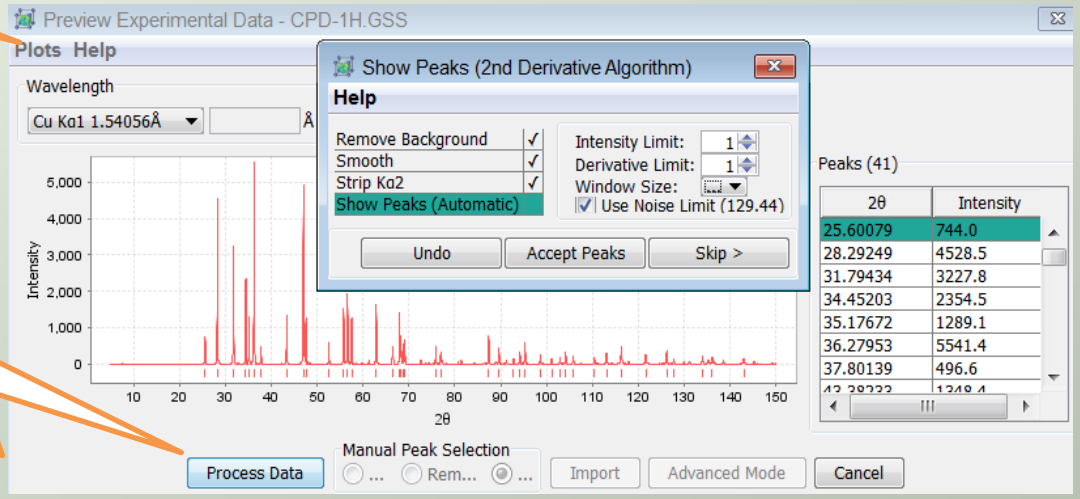
Zero point correction

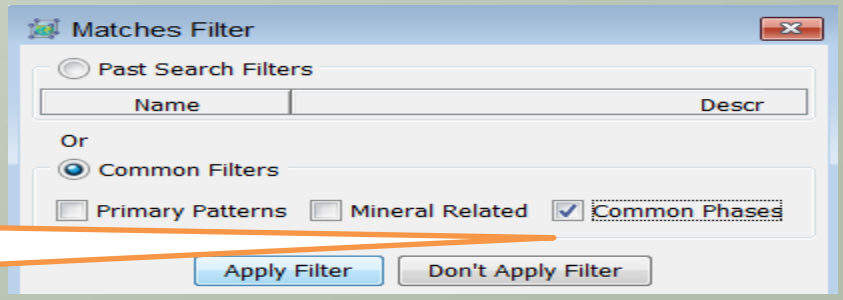
2. Process Data
- ❖ Background Subtraction
- ❖ Smoothing
- ❖ Strip Ka2 Peaks
- ❖ Show Peaks, Add/Remove Peaks

At the end of data processing, tick marks shown at the bottom of the plot indicate all peaks for the search. Also, a peak table on the right side shows the total number of peaks.

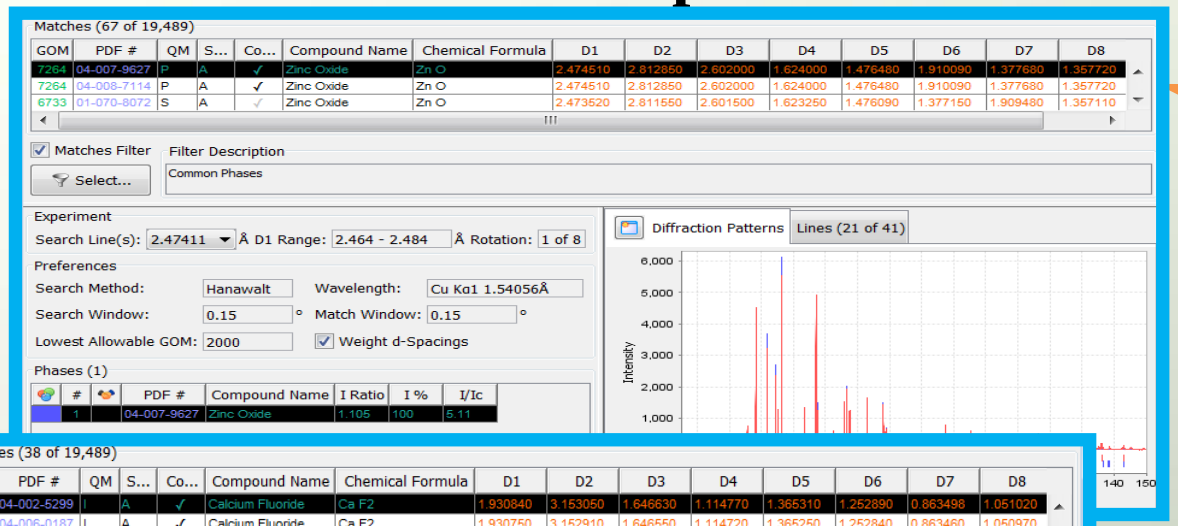
After Importing the data, “Common Phase” of the “Common Filters” was selected to reduce the number of matches.





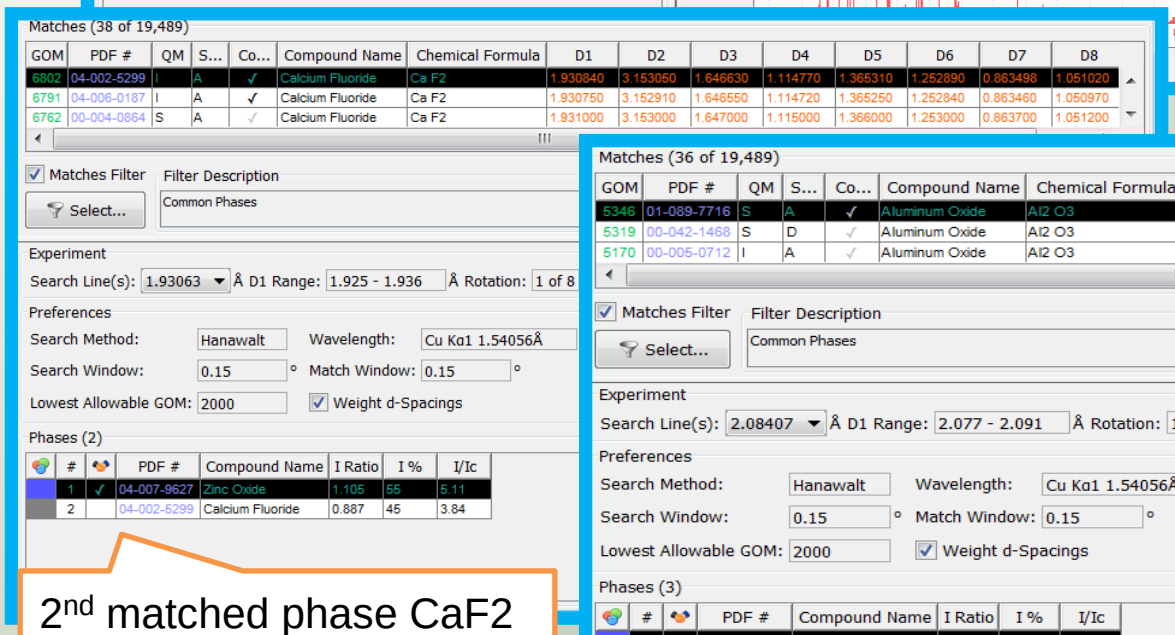


SIeve+ Case-2
Multiphase Search/Match Results

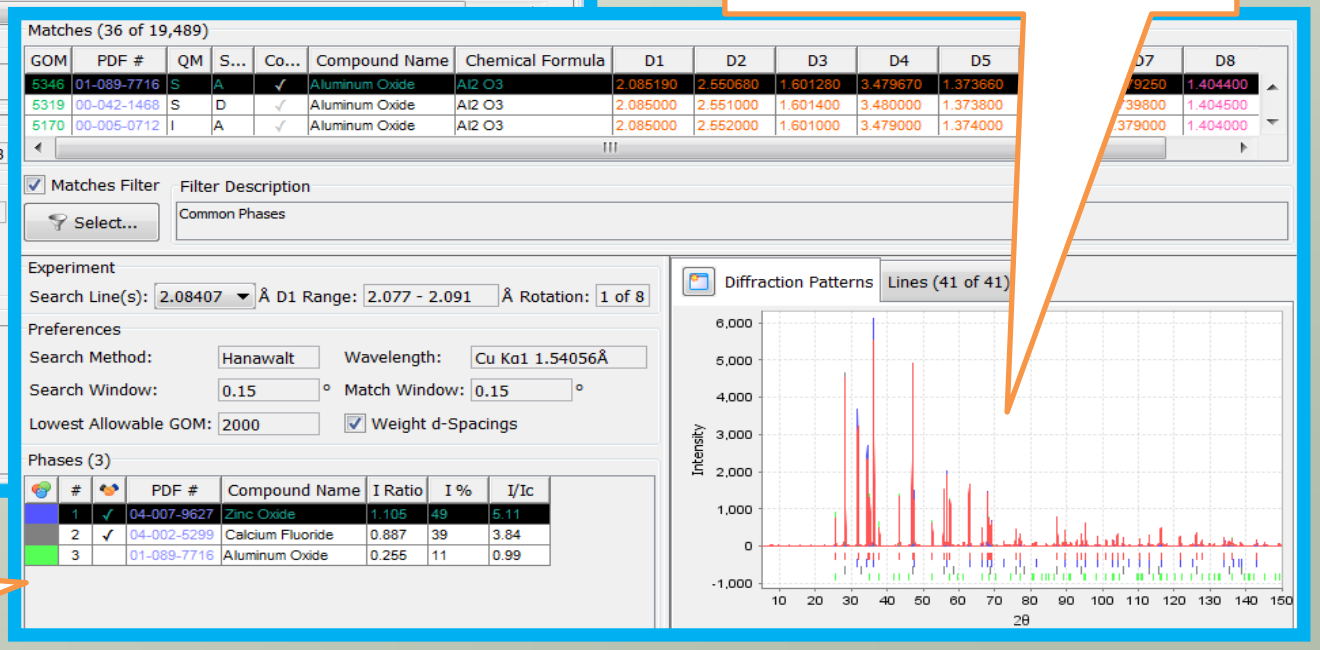


First matched phase ZnO with highest GOM

Comparison of d-spacings and intensities for the input data and those of all three reference patterns



2nd matched phase CaF2



3rd matched phase Al2O3