Advanced Identification Tools

Advanced Identification Tools

This tutorial will demonstrate how a user can increase both the speed and efficiency of the material identification process.

It demonstrates how to take advantage of embedded links between ICDD's viewing and identification programs. It also explains how to utilize ICDD's editorial classification and subfile assignments for additional benefit.

	Viewing	Identification
PDF-2	DDView	SIeve
PDF-4+	DDView+	SIeve+

Prerequisite Tutorials

- Using Subfiles
- Chemical and Structural Classification
- Identification
  - PDF-2 or PDF-4+

Advanced Identification Tools

What?

Advanced identification utilizes multiple steps:

1) Use either DDView or DDView+ to data mine the Powder Diffraction File (PDF) to a subset of data that are applicable to a problem (i.e., minerals, metals, ceramics, materials containing Fe etc.).

2) Import raw data and process it to a d/I file.

3) Use either SIeve or SIeve+ to identify the material – *using the subset that was created in step 1.*

Advanced Identification Tools

Why?

Most of the time, a researcher has some knowledge of the specimen. This might include a preparation history, an elemental analysis, or a physical property (i.e., color).
This information can be used to reduce the number of candidate materials from hundreds of thousands to a few thousand.

In complex multiphase unknowns, there can be “false” identifications due to the large number of peaks and comparison to a large number of candidate materials.

Appropriate selection of a *“properly defined”* subset can lead to increased speed and Increased efficiency.

Advanced Identification Tools

How?

The Powder Diffraction File has many subfiles, and chemical and structural classifications to help users
select a subset for analysis.

Any search can be coupled with the identification programs SIeve and SIeve+.

In PDF-4+, there are 48 Search Options – which can be combined and permuted.

Field experts are used to define subfiles.

Common Cases

Case 1: Analysis of a soil specimen (see Case 1 slides)
Use the Mineral subclassification in the Mineral Related subfile.
This includes minerals that were “naturally found”. (Note: Use the Mineral Related subfile if the specimen was synthetic or of uncertain origin.)

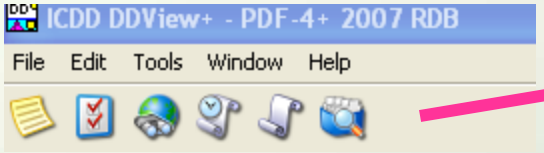
Case 2: Analysis of a drug tablet (see Case 2 slides)
Combine the Pharmaceutical subfile with the Excipients subfile.
In PDF-4/Organics, combine these results with the Bioactivity subfile.

Case 3: Analysis of evidence at a crime scene (not shown)
Use the Forensic subfile, which has been developed and compiled by law enforcement scientists.

Case 4: Analysis of concrete (see Case 4 slides)
Concrete typically consists of cement and aggregates. A combination of the Cement subfile with Minerals can effectively identify the components.

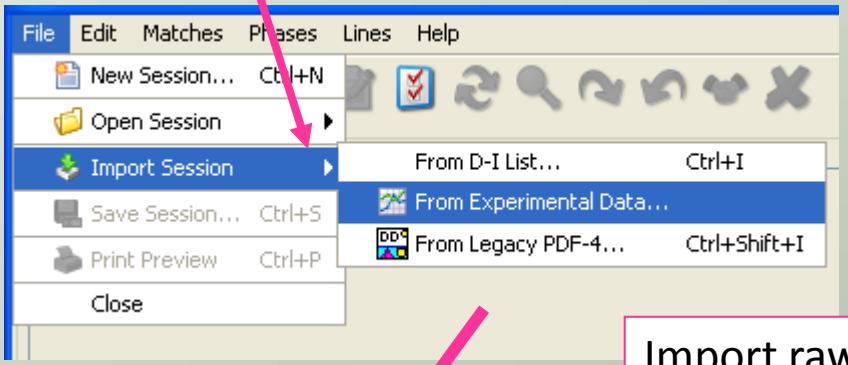
Case 1

Analysis of a Soil Specimen

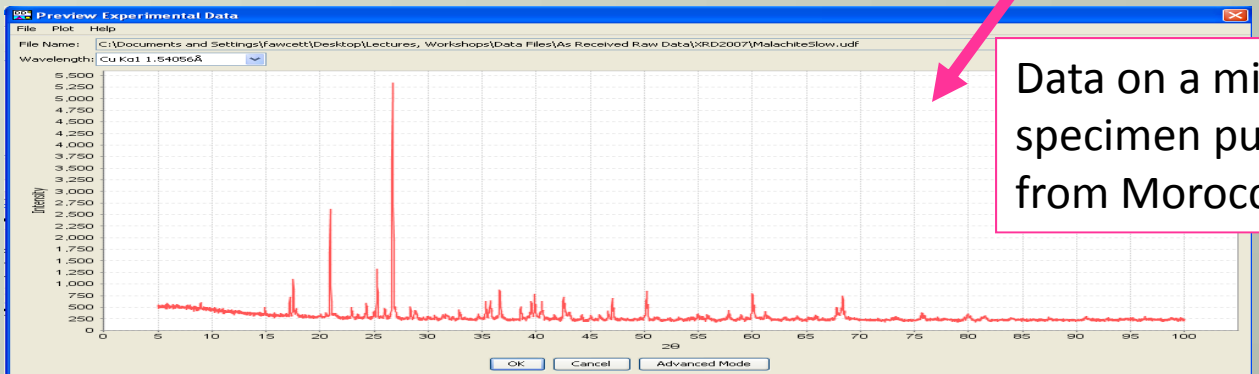


From “Toolbar”

Select SIeve+ (or SIeve)



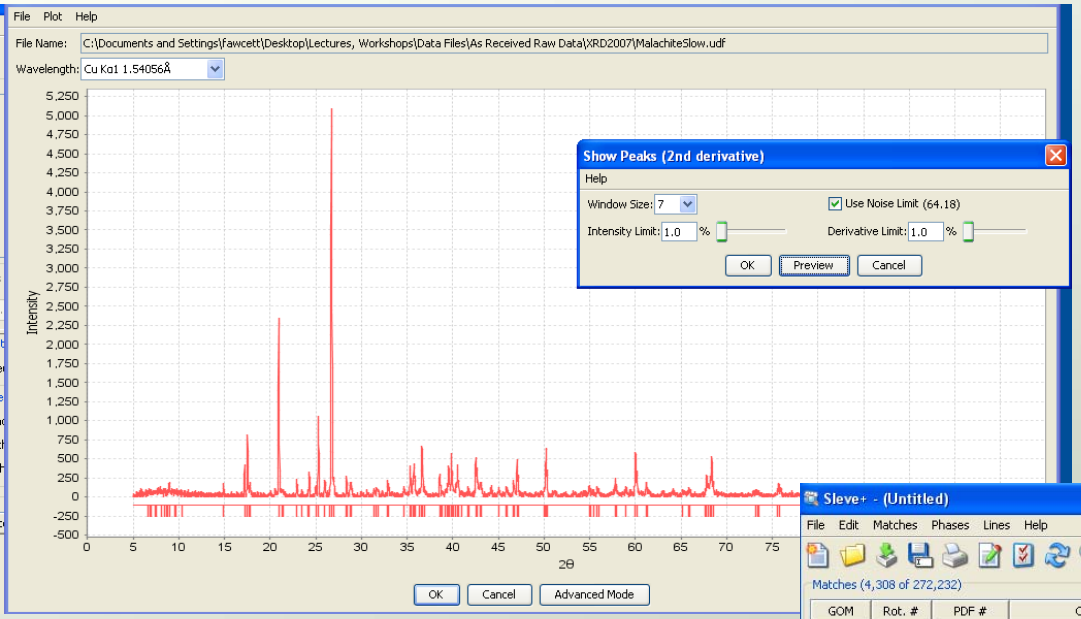
Import raw data



Data on a mineral specimen purchased from Morocco

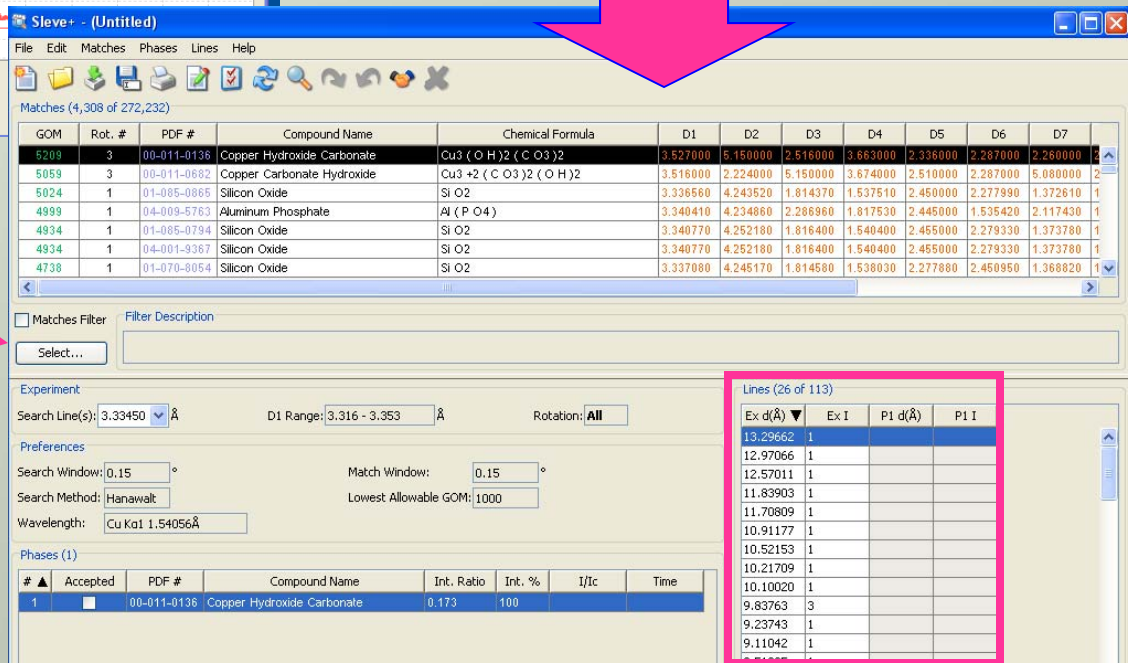
Case 1

Analysis of a Soil Specimen



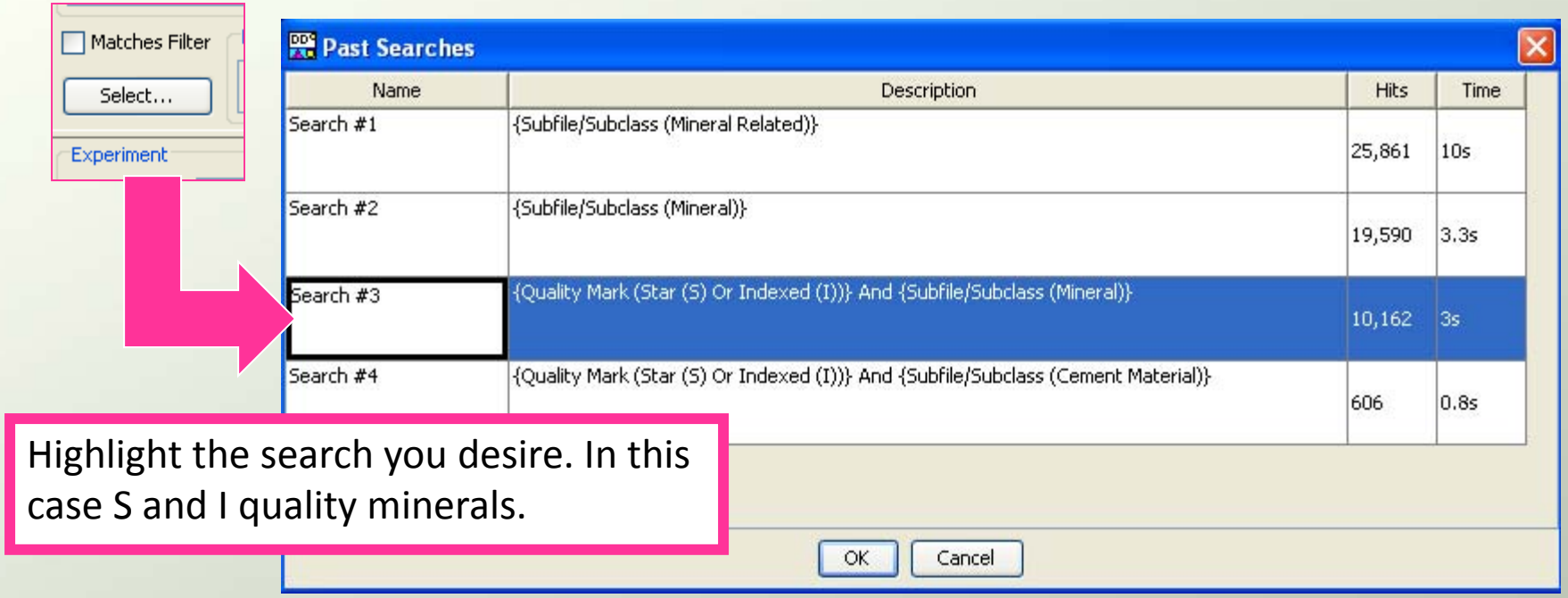
Use algorithms to find peaks and remove Background. Press “OK” to import d/I into SIeve

The “Select” button will activate if there are any active subfile searches



Case 1

Analysis of a Soil Specimen



Highlight the search you desire. In this case S and I quality minerals.

Clicking on "Select" will bring up all prior searches used in the active session, the number of entries in the search and the time it took to generate the entry subset.

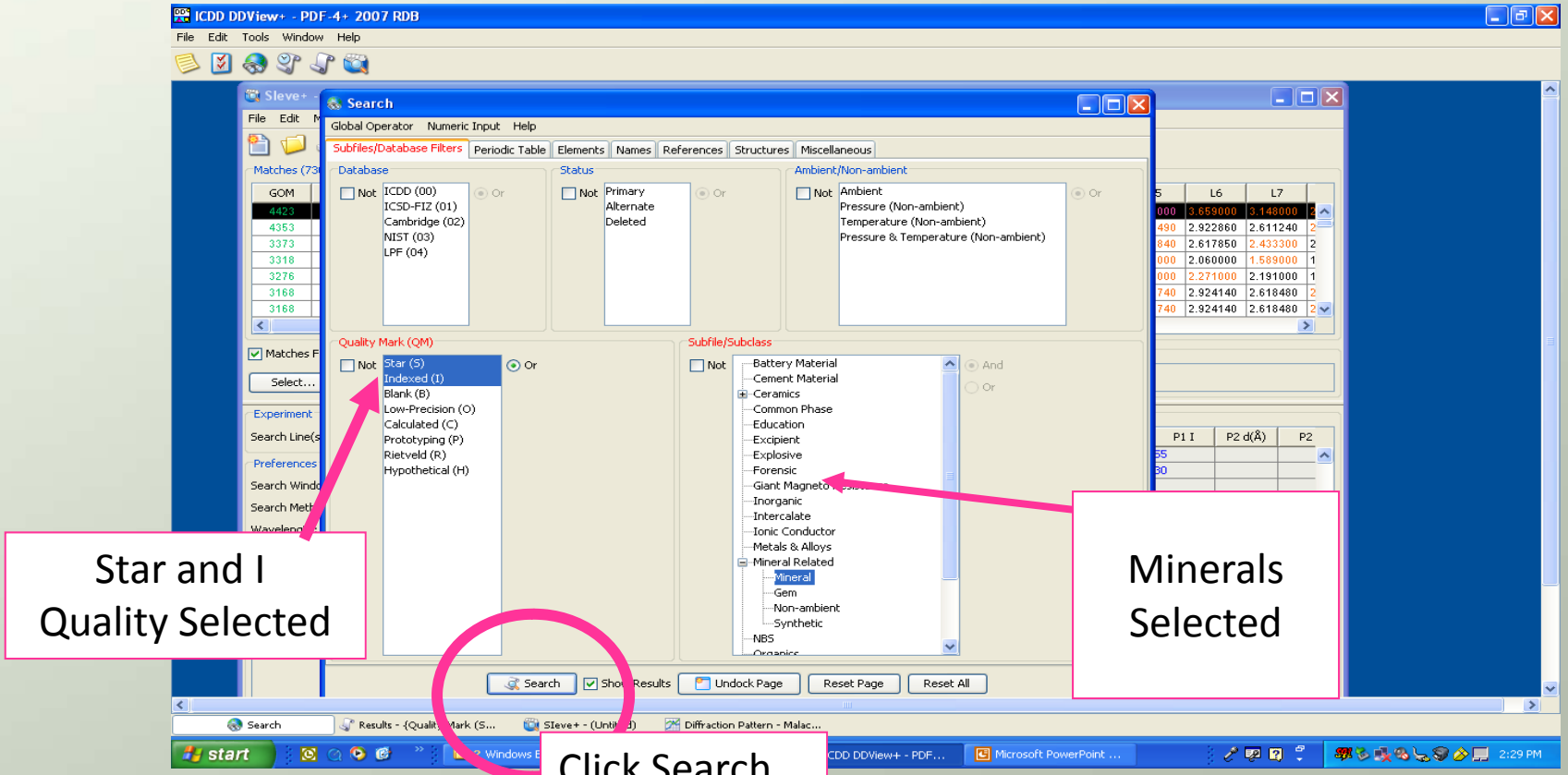
Please note: The "Select" button will only highlight if you have an active search. If you do not have an active search, you can create one and you will see that the select button automatically activates. There is no need to interrupt the session.

Case 1

Creation of Searches:

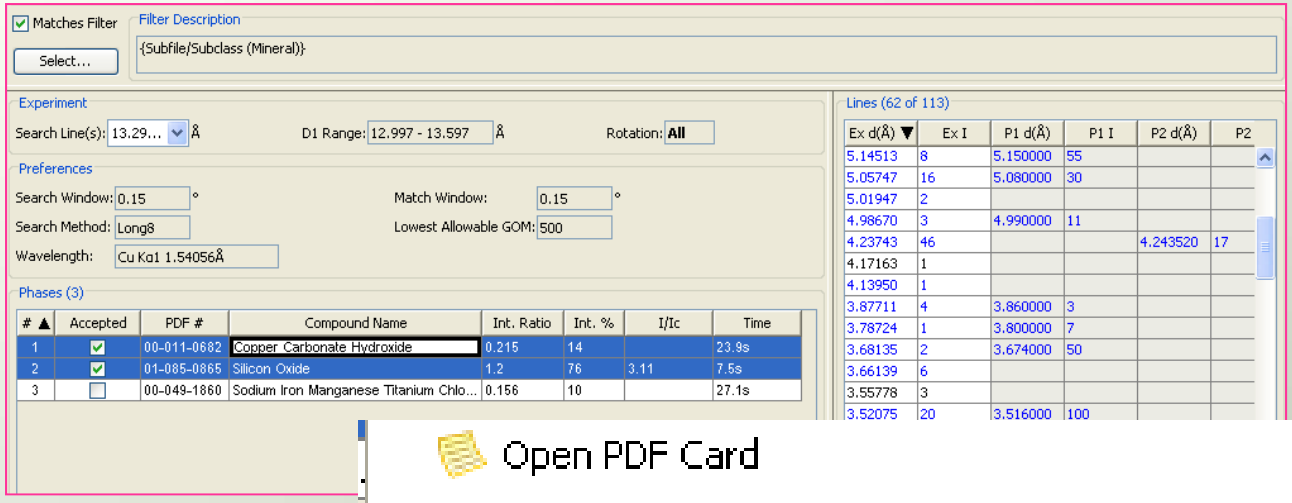
Point and Click (Highlight criteria)

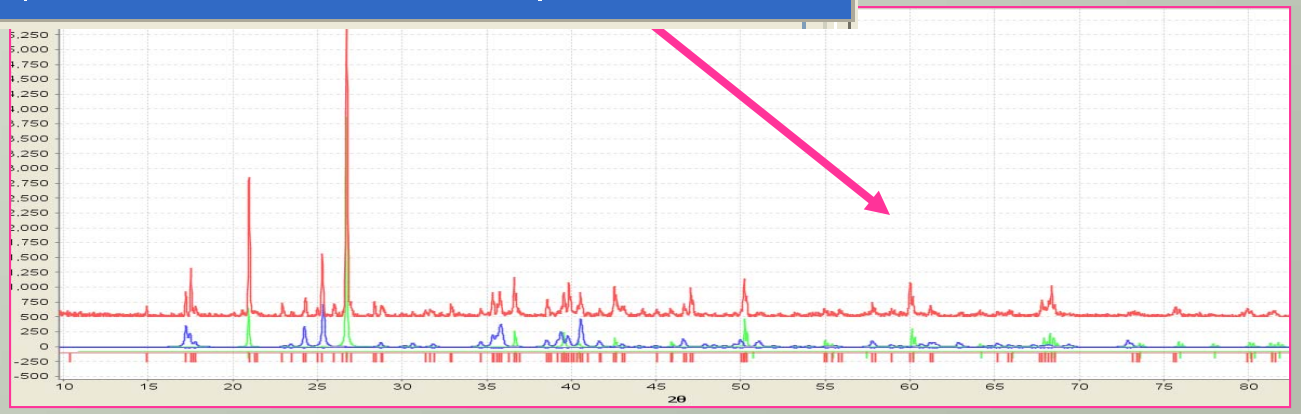
Click Search



Case 1

Analysis of a Soil Specimen





Sieve quickly identifies a blend of azurite and quartz

Top data is the Experiment. The bottom graph is the identified phases, plotted and automatically scaled.

Case 2

Analysis of a Pharmaceutical Tablet

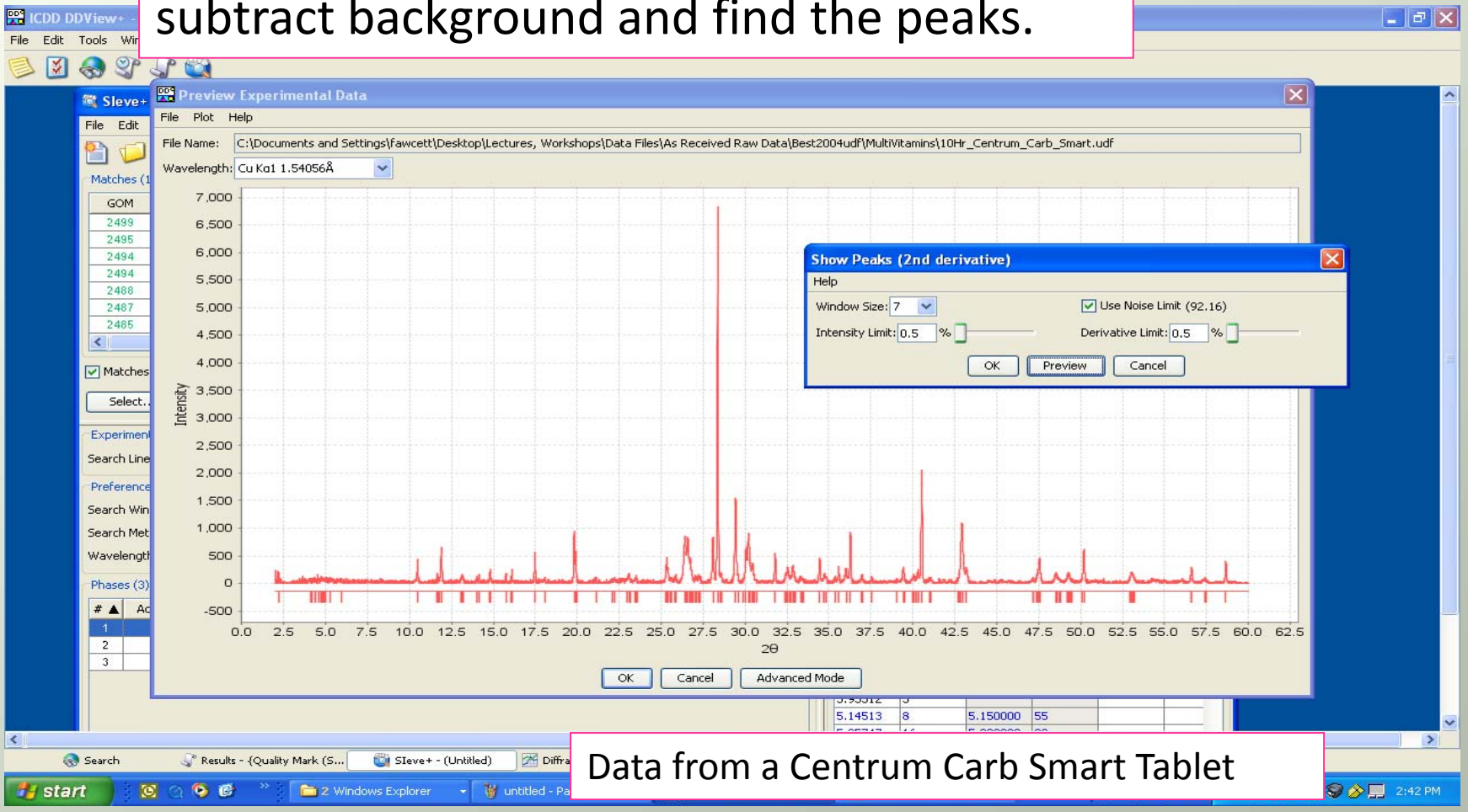
Steps:

- Import Data
- Create d/I file for SIeve+
- Select a subfile
- Plot the results

Case 2

Analysis of a Pharmaceutical Tablet

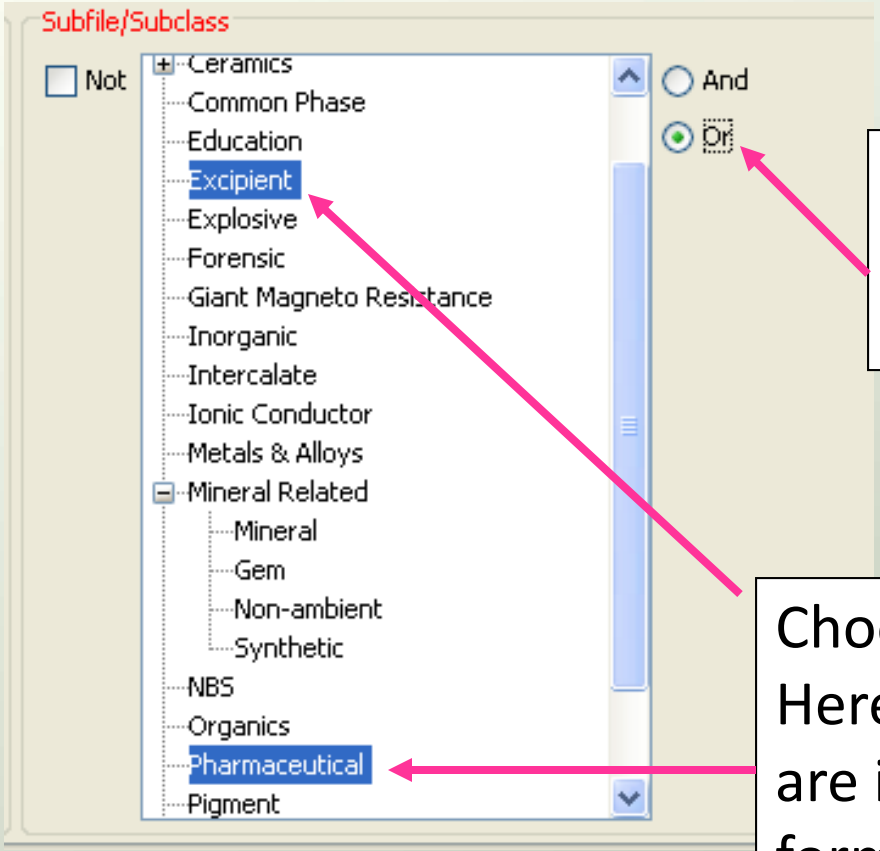
Similar to Case 1 – Import the raw data, subtract background and find the peaks.



Data from a Centrum Carb Smart Tablet

Case 2

Analysis of a Pharmaceutical Tablet

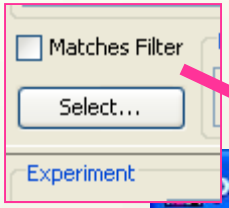


Select the Boolean "or" operator by using the Ctrl key and pressing "or"

Choose an appropriate subfile. Here we choose excipients, which are ingredients used in tablet formulations, and we choose pharmaceuticals to identify active drug substances.

Case 2

Analysis of a Pharmaceutical Tablet



Past Searches

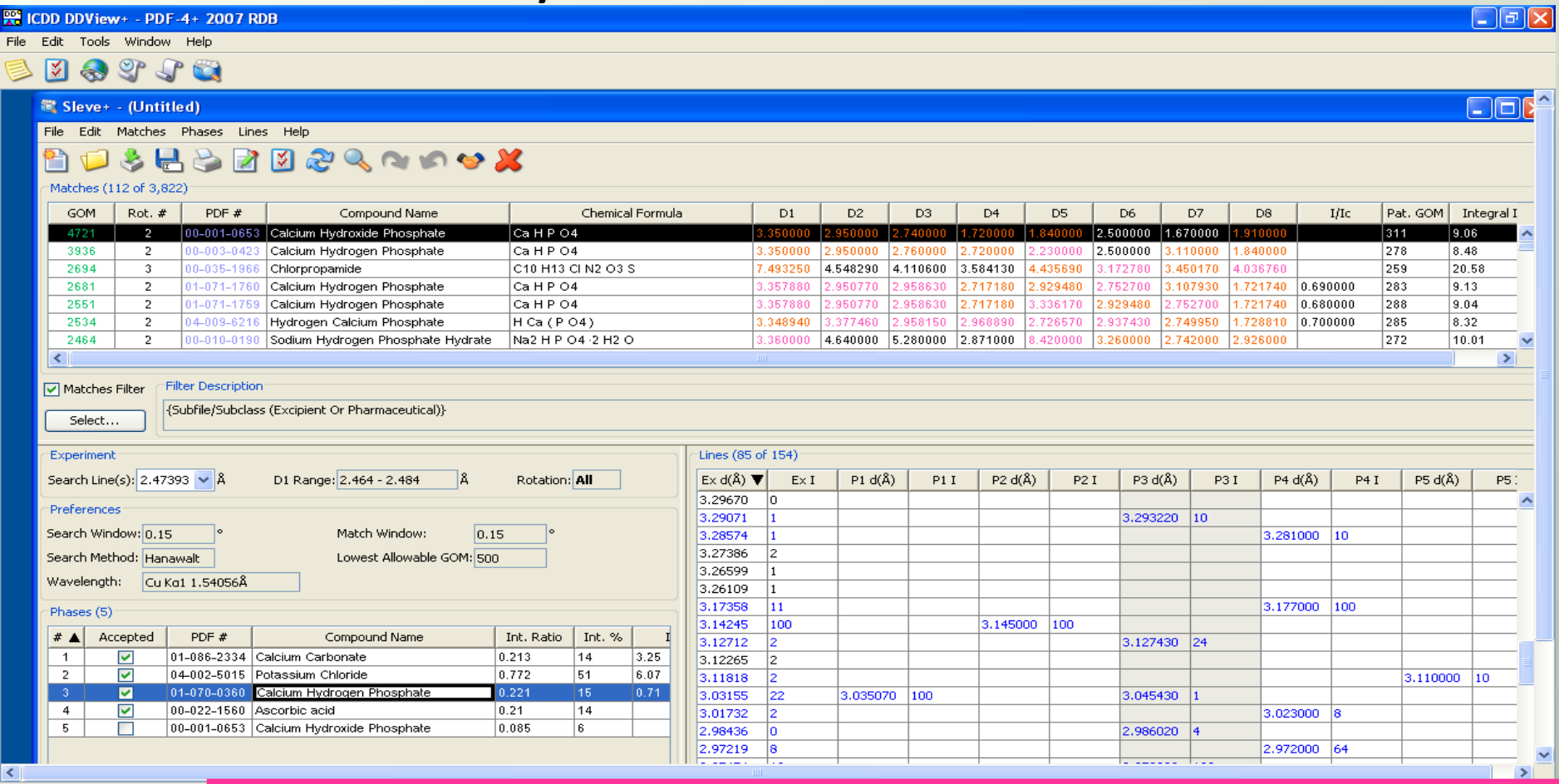
Name	Description	Hits	Time
Search #1	{Subfile/Subclass (Mineral Related)}	25,861	10s
Search #2	{Subfile/Subclass (Mineral)}	19,590	3.3s
Search #3	{Quality Mark (Star (S) Or Indexed (I))} And {Subfile/Subclass (Mineral)}	10,162	3s
Search #4	{Quality Mark (Star (S) Or Indexed (I))} And {Subfile/Subclass (Cement Material)}	606	0.8s
Search #5	{Subfile/Subclass (Excipient Or Pharmaceutical)}	3,822	29s

OK Cancel

The prior search “activates” the select button on SIeve+ and now a subfile containing pharmaceuticals and excipients can be searched.

Case 2

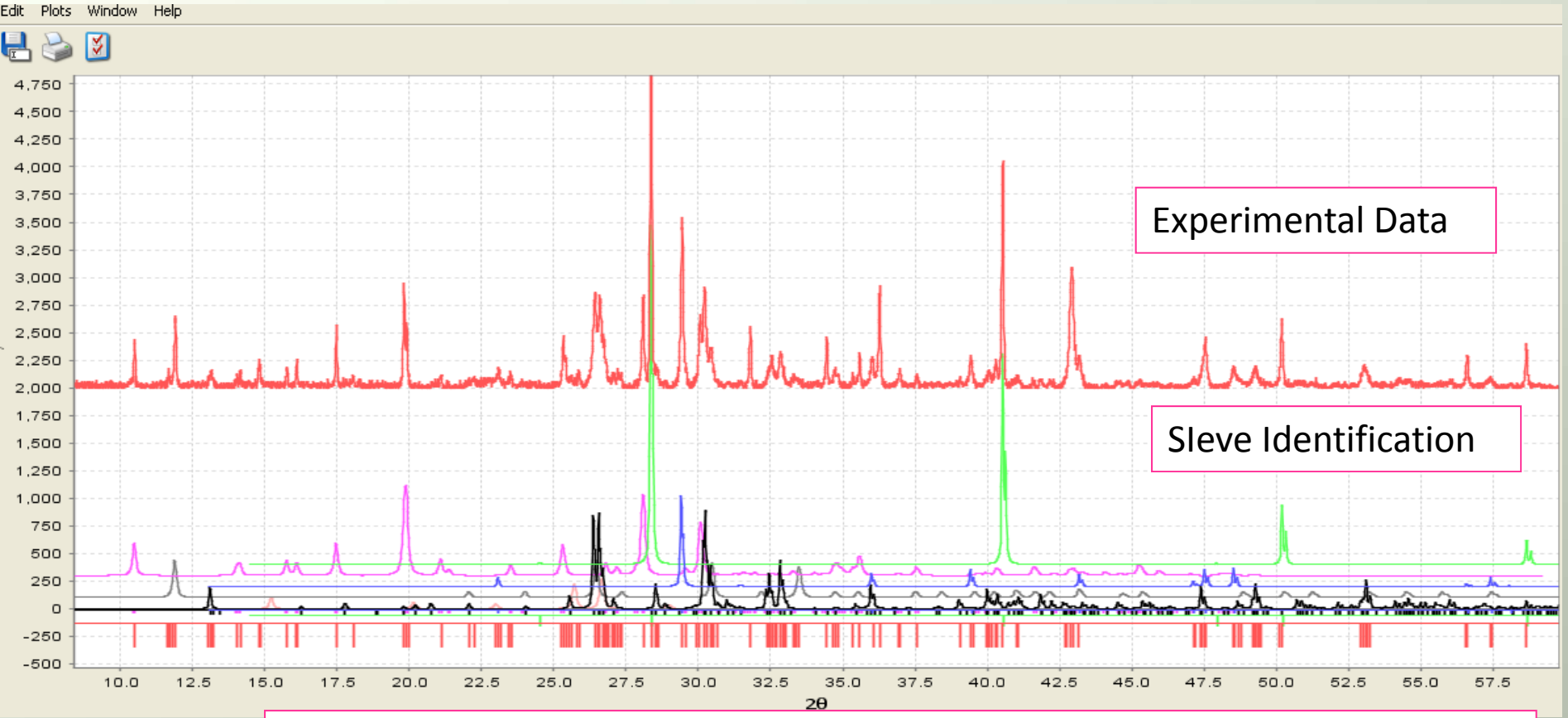
Analysis of a Pharmaceutical Tablet



Because of the targeted subset of 3,822 materials, the identification process is very fast and highly targeted; multiple phases are quickly identified.

Case 2

Analysis of a Pharmaceutical Tablet

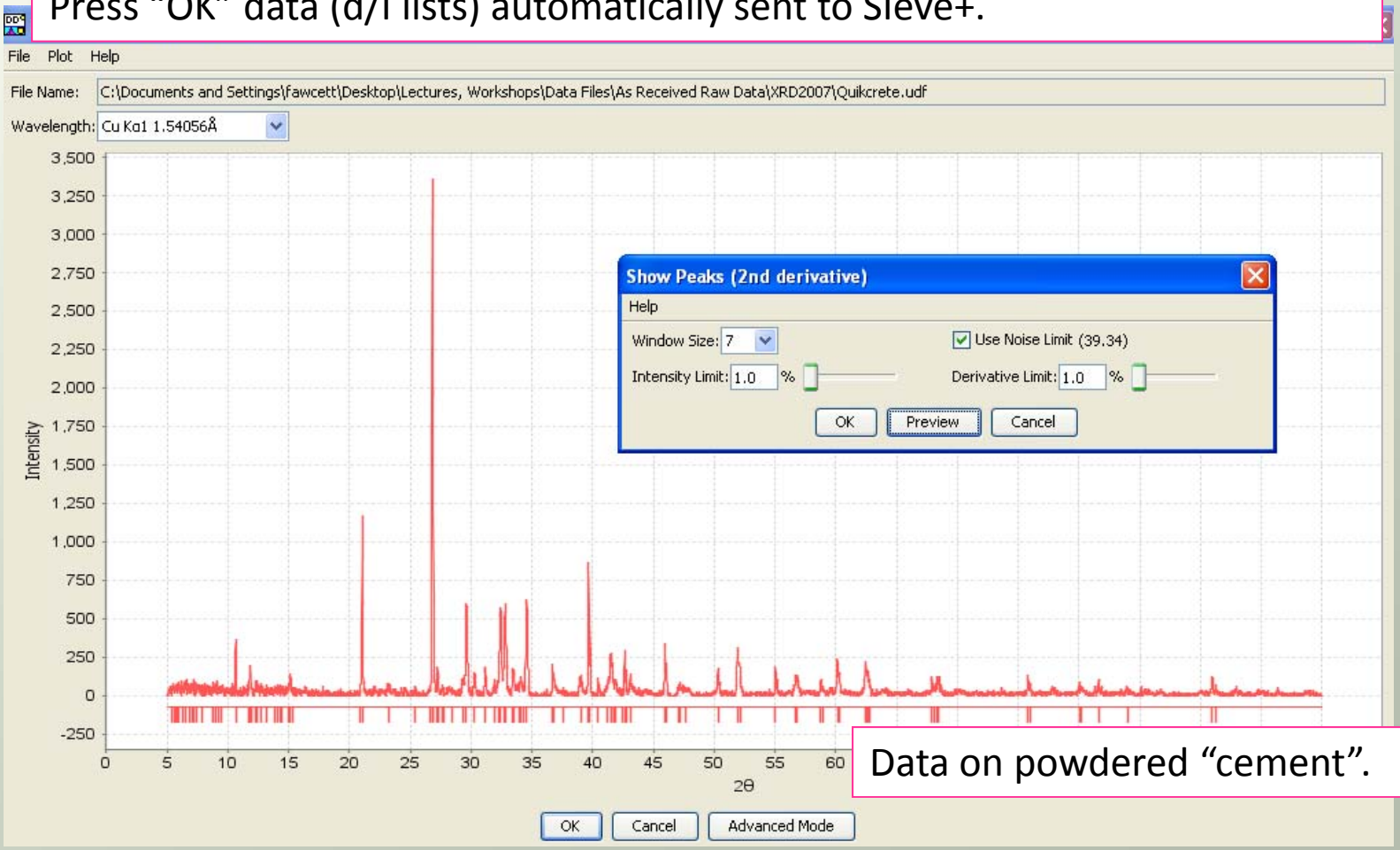


The identification finds Vitamins B and C in the tablet, as well as KCl, CaCO3, CaHPO4 and Iron Fumarate. These are plotted above with the data deliberately offset to show each phase.
Further analyses (not shown) identifies ZnO, TiO2 and MgO.

Case 4

Analysis of Concrete

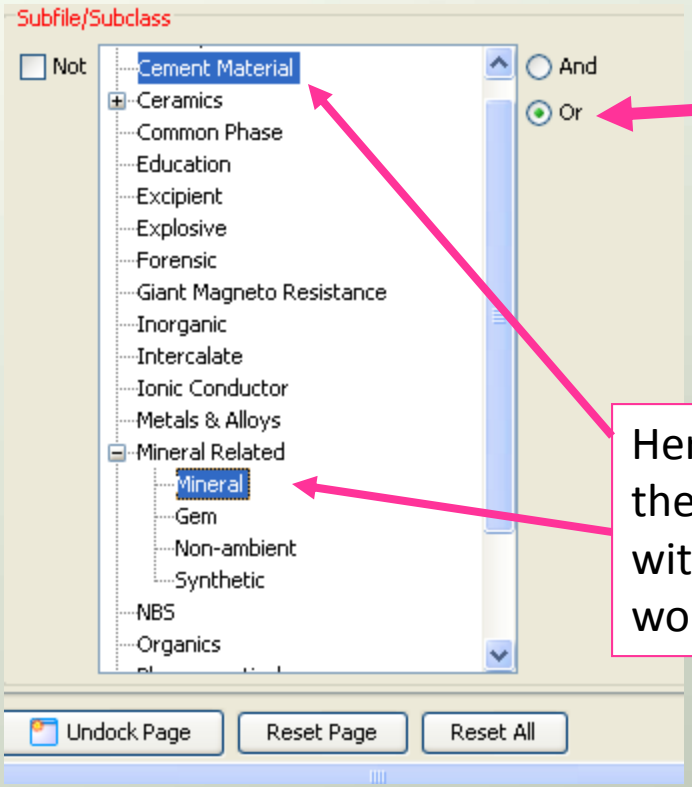
Import data, remove background, find peaks – See 1st slide of Case 1.
Press "OK" data (d/I lists) automatically sent to SIeve+.



Data on powdered "cement".

Case 4

Analysis of Concrete



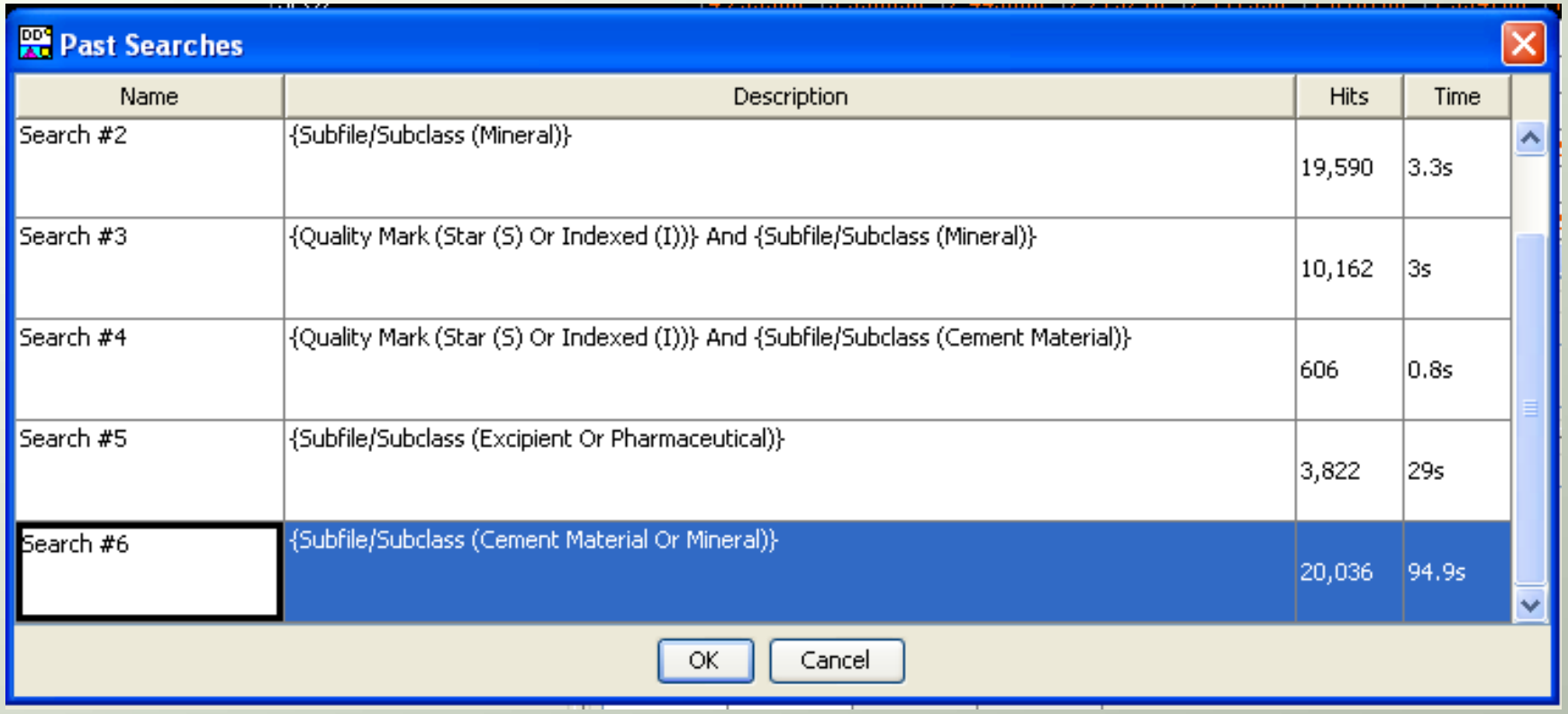
Use the Boolean operator “or” to include multiple subfiles. Use the Ctrl key to select files.

Here, the appropriate search would be of the Cement Material subfile combined with the Mineral subclass. The latter choice would be to identify any mineral aggregates.

This search produces ~20,000 materials for identification.

Case 4

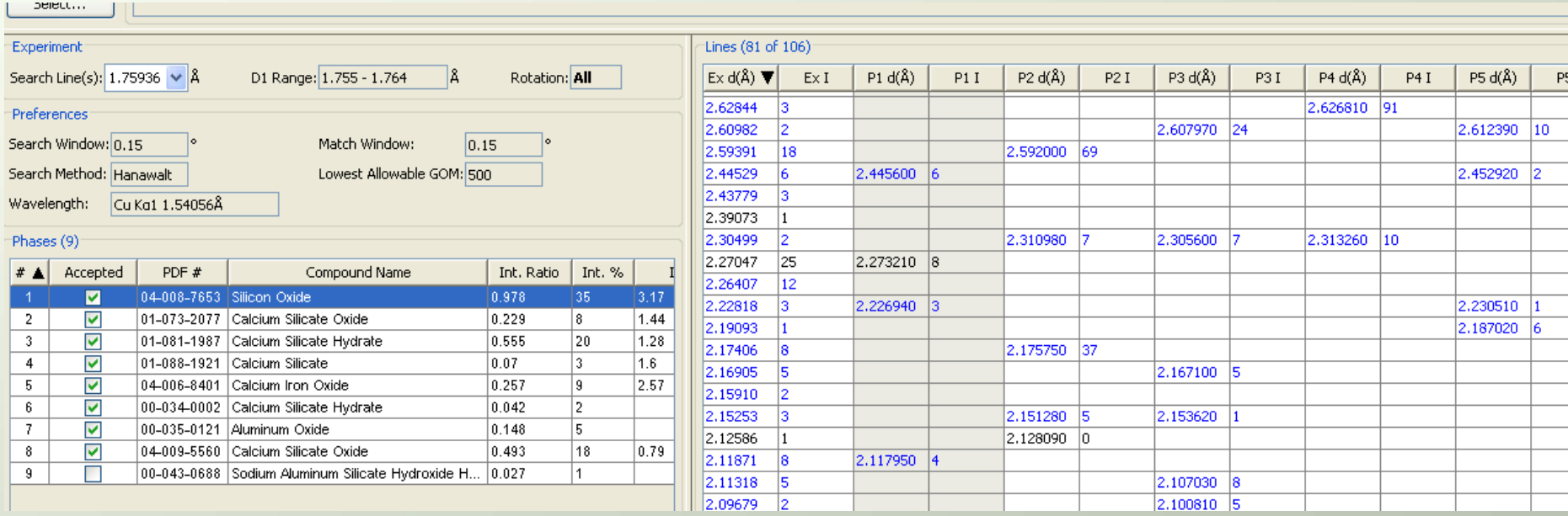
Analysis of Concrete



Select button on SIeve+ is active and the highlighted search can be chosen for identification.

Case 4

Analysis of Concrete



Experiment

Search Line(s): 1.75936 Å D1 Range: 1.755 - 1.764 Å Rotation: All

Preferences

Search Window: 0.15 ° Match Window: 0.15 °

Search Method: Hanawalt Lowest Allowable GOM: 500

Wavelength: Cu Kα1 1.54056Å

Phases (9)

#	Accepted	PDF #	Compound Name	Int. Ratio	Int. %	I
1	☑	04-008-7653	Silicon Oxide	0.978	35	3.17
2	☑	01-073-2077	Calcium Silicate Oxide	0.229	8	1.44
3	☑	01-081-1987	Calcium Silicate Hydrate	0.555	20	1.28
4	☑	01-088-1921	Calcium Silicate	0.07	3	1.6
5	☑	04-006-8401	Calcium Iron Oxide	0.257	9	2.57
6	☑	00-034-0002	Calcium Silicate Hydrate	0.042	2	
7	☑	00-035-0121	Aluminum Oxide	0.148	5	
8	☑	04-009-5560	Calcium Silicate Oxide	0.493	18	0.79
9	☐	00-043-0688	Sodium Aluminum Silicate Hydroxide H...	0.027	1	

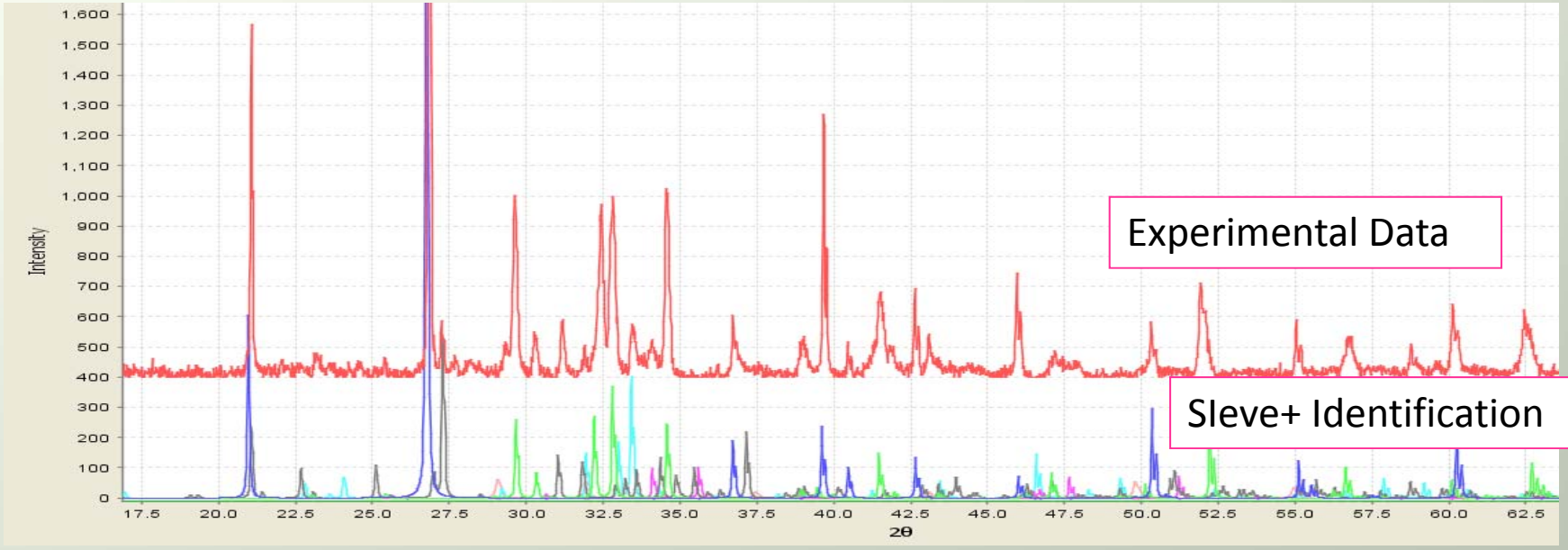
Lines (81 of 106)

Ex d(Å) ▼	Ex I	P1 d(Å)	P1 I	P2 d(Å)	P2 I	P3 d(Å)	P3 I	P4 d(Å)	P4 I	P5 d(Å)	P5
2.62844	3							2.626810	91		
2.60982	2					2.607970	24			2.612390	10
2.59391	18			2.592000	69						
2.44529	6	2.445600	6							2.452920	2
2.43779	3										
2.39073	1										
2.30499	2			2.310980	7	2.305600	7	2.313260	10		
2.27047	25	2.273210	8								
2.26407	12										
2.22818	3	2.226940	3							2.230510	1
2.19093	1									2.187020	6
2.17406	8			2.175750	37						
2.16905	5					2.167100	5				
2.15910	2										
2.15253	3			2.151280	5	2.153620	1				
2.12586	1			2.128090	0						
2.11871	8	2.117950	4								
2.11318	5					2.107030	8				
2.09679	2					2.100810	5				

SIeve results match multiple phases of calcium silicates and hydration products.

Case 4

Analysis of Concrete



The phase identification process identifies SiO2 (quartz) and the CS, C2S, C3S phases of calcium silicate, as well as monohydrates of CS and C2S. The quartz phase (blue) is oriented resulting in some intensity mismatch.

Conclusions

Coupling viewing (DDView and DDView+) software with identification software (Sleve and Sleve+) can reduce speed and increase efficiency of material identification processes.

Any search process (>40 searches in both PDF-2 and PDF-4+) can be used as well as combined searches.

Note to Users

Editorial work done to improve subfile content, nomenclature, and classification systems in the Powder Diffraction File all act to improve the accuracy and efficiency of identification processes.

Each vendor has an ability to use all or a select subset of searches in their proprietary viewing software. This frequently reduces the number of available searches and often improves speed. As a result, some of the searches demonstrated in this tutorial may not be provided in your vendor software. However, if they are provided, then they should be both fast and accurate.

Major upgrades to subfile contents were made for Release 2007, adding more high quality data to each subfile.

Thank you for viewing our tutorial.
Additional tutorials are available at the ICDD website (www.icdd.com).

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