

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.

	<u>Viewing</u>	<u>Identification</u>
PDF-2	DDView	Sleve
PDF-4+	DDView+	Sleve+

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 Sleve or Sleve+ 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 Sleve and Sleve+.

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)

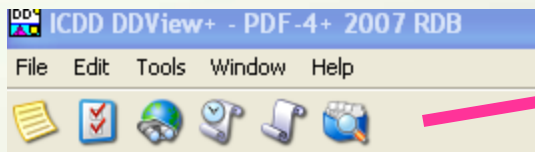
Use the Forensic subfile, which has been developed and compiled by law enforcement scientists.

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

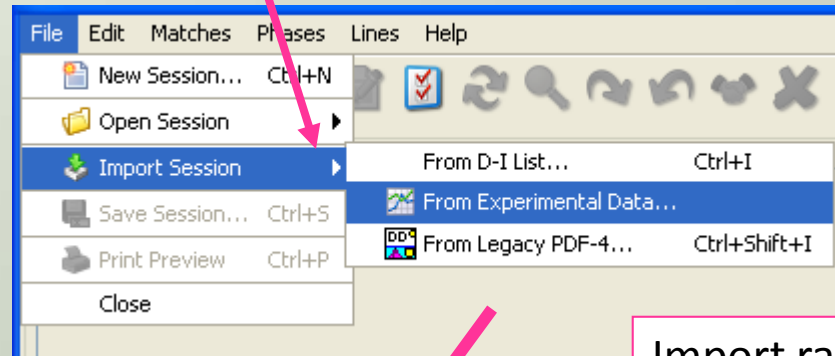
Analysis of a Soil Specimen



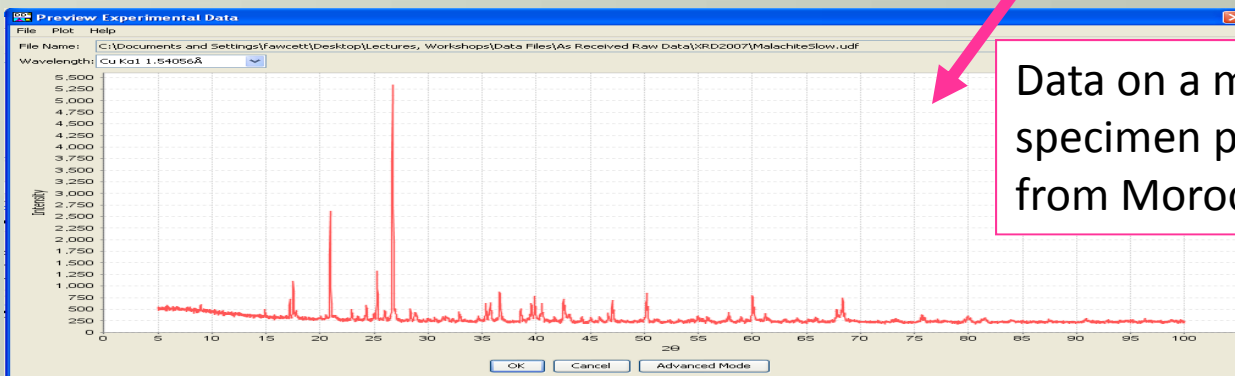
From "Toolbar"



Select Sieve+ (or Sieve)



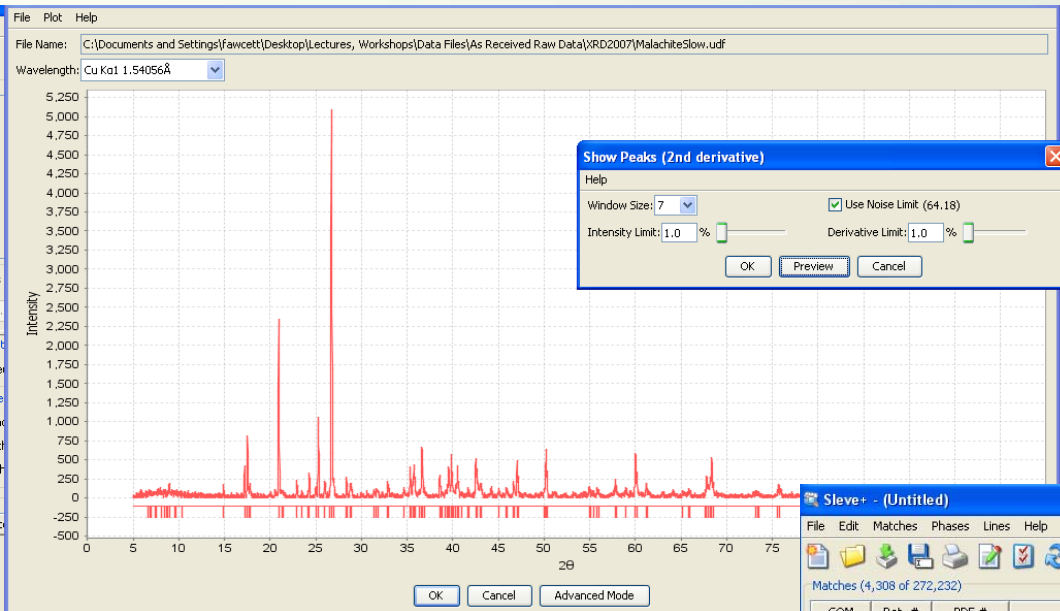
Import raw data



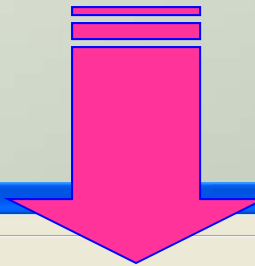
Data on a mineral specimen purchased from Morocco

Case 1

Analysis of a Soil Specimen



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



Matches Filter

Select...

Experiment

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

Sieve - - (Untitled)

File Edit Matches Phases Lines Help

Matches (4,308 of 272,232)

GOM	Rot. #	PDF #	Compound Name	Chemical Formula	D1	D2	D3	D4	D5	D6	D7
5209	3	00-011-0136	Copper Hydroxide Carbonate	Cu ₃ (OH) ₂ (CO ₃) ₂	3.527000	5.150000	2.516000	3.663000	2.336000	2.287000	2.260000
5059	3	00-011-0682	Copper Carbonate Hydroxide	Cu ₃ +2 (CO ₃) ₂ (OH) ₂	3.516000	2.224000	5.150000	3.674000	2.510000	2.287000	5.080000
5024	1	01-085-0865	Silicon Oxide	Si O ₂	3.336560	4.243520	1.814370	1.537510	2.450000	2.277990	1.372610
4999	1	04-009-5763	Aluminum Phosphate	Al (PO ₄)	3.340410	4.234860	2.286960	1.817530	2.445000	1.535420	2.117430
4934	1	01-085-0794	Silicon Oxide	Si O ₂	3.340770	4.252180	1.816400	1.540400	2.455000	2.279330	1.373780
4934	1	04-001-9367	Silicon Oxide	Si O ₂	3.340770	4.252180	1.816400	1.540400	2.455000	2.279330	1.373780
4738	1	01-070-8054	Silicon Oxide	Si O ₂	3.337080	4.245170	1.814580	1.538030	2.277880	2.450950	1.368820

Matches Filter Filter Description

Select...

Experiment

Search Line(s): 3.33450 Å D1 Range: 3.316 - 3.353 Å Rotation: All

Preferences

Search Window: 0.15 ° Match Window: 0.15 °

Search Method: Hanawalt Lowest Allowable GOM: 1000

Wavelength: Cu Kα1 1.54056Å

Phases (1)

#	Accepted	PDF #	Compound Name	Int. Ratio	Int. %	I/Ic	Time
1	<input checked="" type="checkbox"/>	00-011-0136	Copper Hydroxide Carbonate	0.173	100		

Lines (26 of 113)

Ex d(Å)	Ex I	P1 d(Å)	P1 I
13.29662	1		
12.97066	1		
12.57011	1		
11.83903	1		
11.70809	1		
10.91177	1		
10.52153	1		
10.21709	1		
10.10020	1		
9.83763	3		
9.23743	1		
9.11042	1		

Case 1

Analysis of a Soil Specimen

Matches Filter

Select...

Experiment

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

OK Cancel

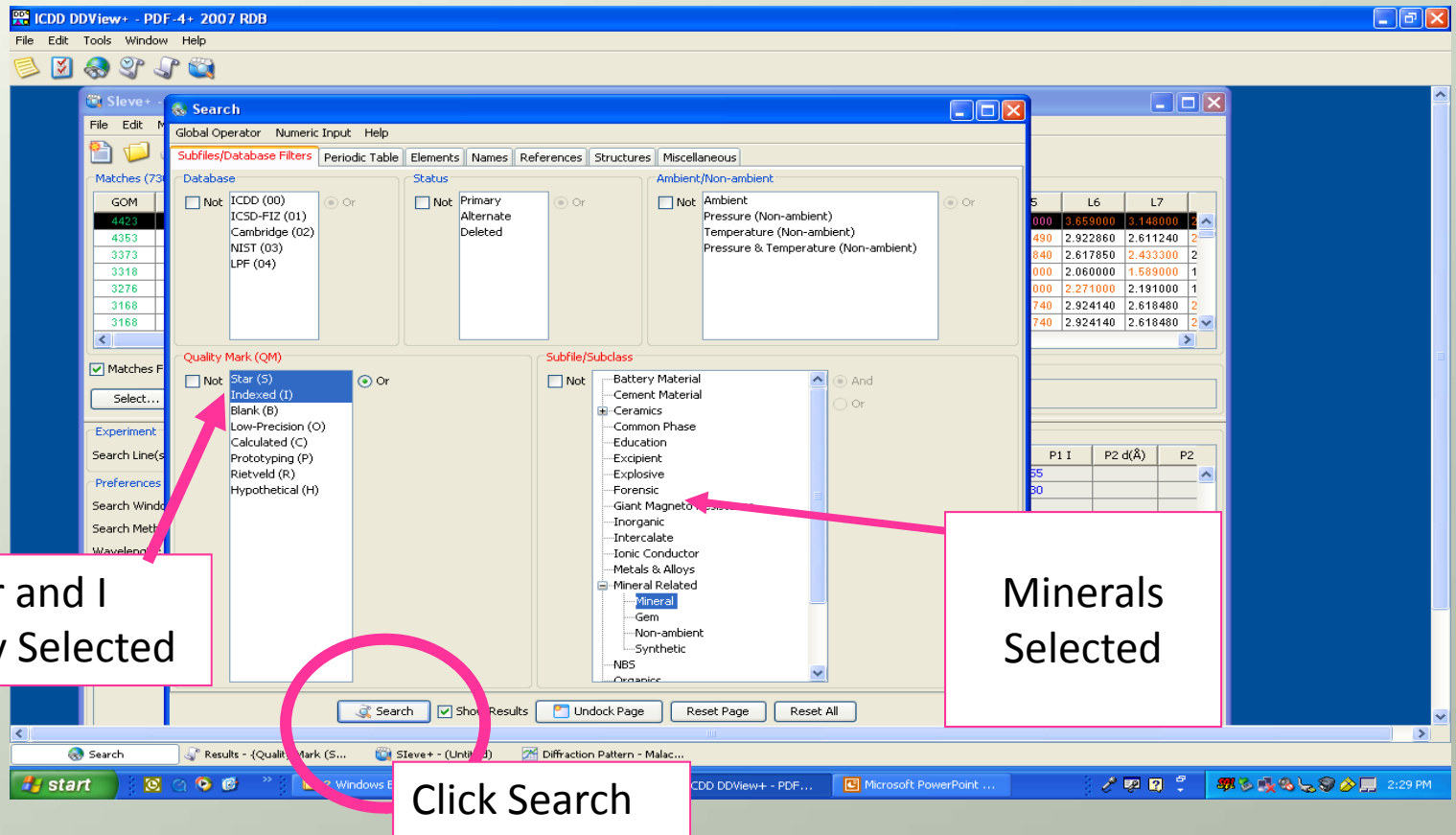
Highlight the search you desire. In this case S and I quality minerals.

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



The screenshot shows the ICDD DDView software interface with the following search criteria selected:

- Database:** ICDD (00), ICSD-FIZ (01), Cambridge (02), NIST (03), LPF (04)
- Status:** Primary, Alternate, Deleted
- Ambient/Non-ambient:** Ambient (Non-ambient), Temperature (Non-ambient), Pressure & Temperature (Non-ambient)
- Quality Mark (QM):** Star (S), Indexed (I)
- Subfile/Subclass:** Mineral

Annotations in the image:

- Star and I Quality Selected:** A pink arrow points to the 'Star (S)' and 'Indexed (I)' options under the Quality Mark (QM) section.
- Minerals Selected:** A pink arrow points to the 'Mineral' option under the Subfile/Subclass section.
- Click Search:** A pink circle highlights the 'Search' button at the bottom of the search window.

Additional interface details include a 'Matches (73)' list on the left, a 'Global Operator' menu at the top, and a data table on the right with columns P1 I, P2 d(Å), and P2.

Case 1

Analysis of a Soil Specimen

Matches Filter Filter Description
 Select... {Subfile/Subclass (Mineral)}

Experiment
 Search Line(s): 13.29... Å D1 Range: 12.997 - 13.597 Å Rotation: All

Preferences
 Search Window: 0.15 ° Match Window: 0.15 °
 Search Method: Long8 Lowest Allowable GOM: 500
 Wavelength: Cu Kα1 1.54056 Å

Phases (3)

#	Accepted	PDF #	Compound Name	Int. Ratio	Int. %	I/Ic	Time
1	<input checked="" type="checkbox"/>	00-011-0682	Copper Carbonate Hydroxide	0.215	14		23.9s
2	<input checked="" type="checkbox"/>	01-085-0885	Silicon Oxide	1.2	76	3.11	7.5s
3	<input type="checkbox"/>	00-049-1860	Sodium Iron Manganese Titanium Chlo...	0.156	10		27.1s

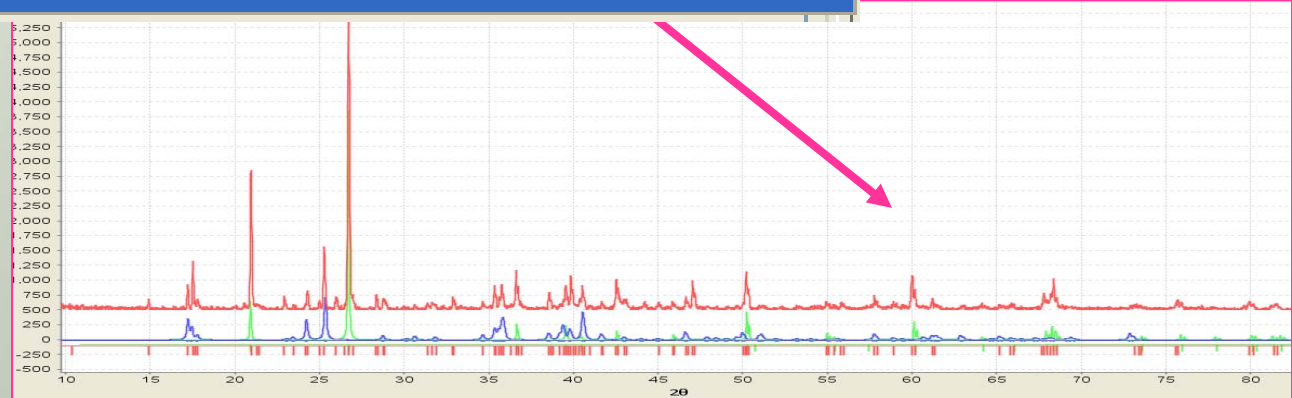
Lines (62 of 113)

Ex d(Å)	Ex I	P1 d(Å)	P1 I	P2 d(Å)	P2
5.14513	8	5.150000	55		
5.05747	16	5.080000	30		
5.01947	2				
4.98670	3	4.990000	11		
4.23743	46			4.243520	17
4.17163	1				
4.13950	1				
3.87711	4	3.860000	3		
3.78724	1	3.800000	7		
3.68135	2	3.674000	50		
3.66139	6				
3.55778	3				
3.52075	20	3.516000	100		

Sieve quickly identifies a blend of azurite and quartz

-  Open PDF Card
-  Open Diffraction Pattern
-  Open Diffraction Pattern with Experimental Data

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



Case 2

Analysis of a Pharmaceutical Tablet

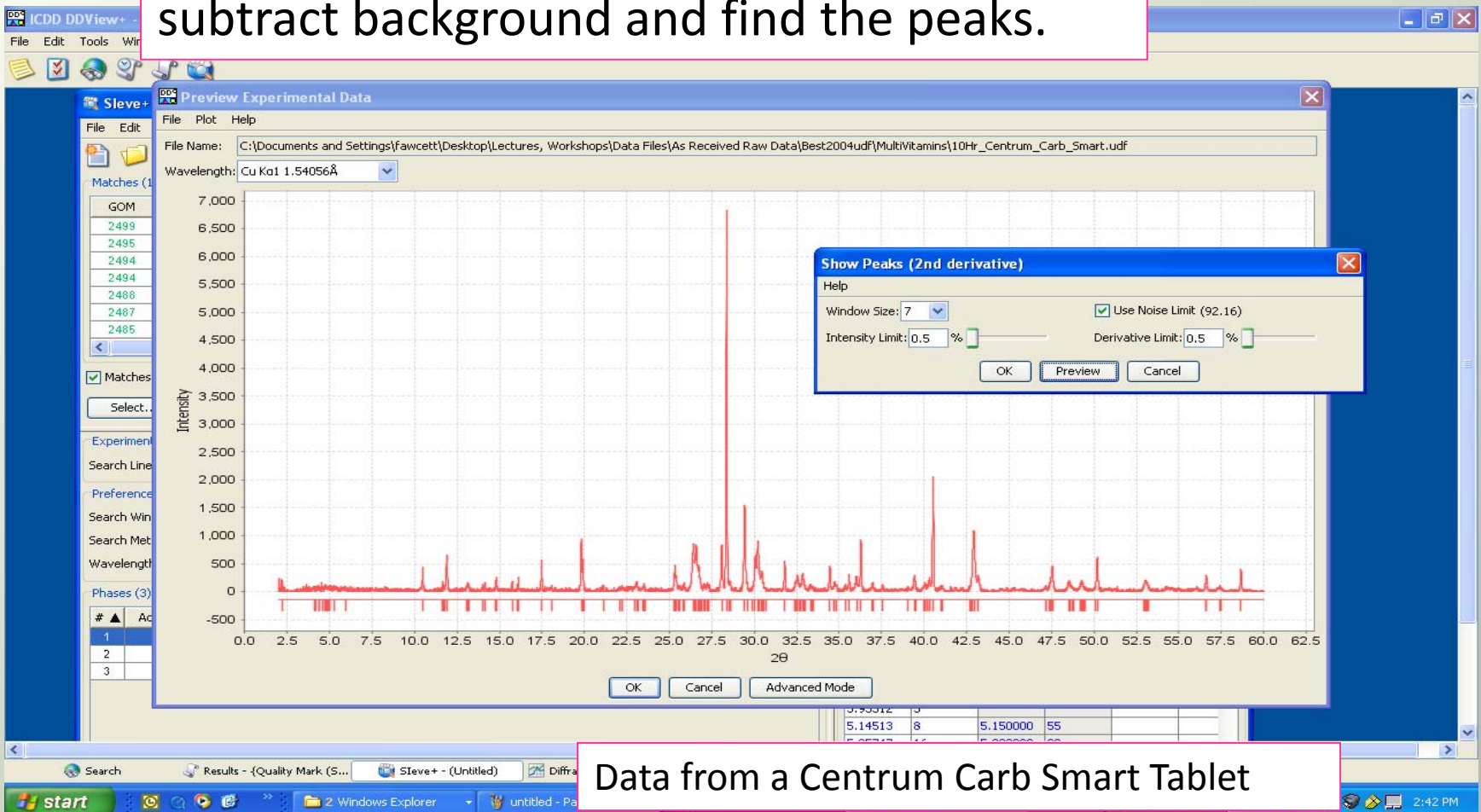
Steps:

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

Case 2

Analysis of a Pharmaceutical Tablet

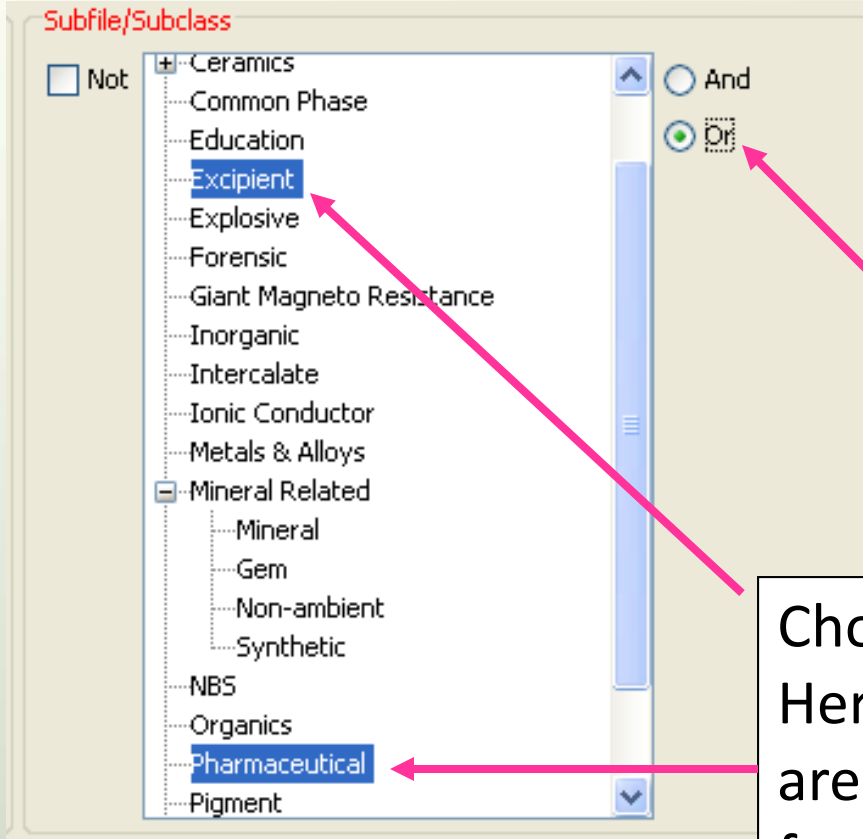
Similar to Case 1 – Import the raw data, subtract background and find the peaks.



Data from a Centrum Carb Smart Tablet

Case 2

Analysis of a Pharmaceutical Tablet

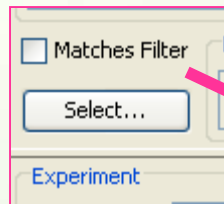


Select the Boolean “or” operator by using the Ctrl key and pressing “or”

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 Sleve+ and now a subfile containing pharmaceuticals and excipients can be searched.

Case 2

Analysis of a Pharmaceutical Tablet

ICDD DDView+ - PDF-4+ 2007 RDB

File Edit Tools Window Help

Sleve+ - (Untitled)

File Edit Matches Phases Lines Help

Matches (112 of 3,822)

GOM	Rot. #	PDF #	Compound Name	Chemical Formula	D1	D2	D3	D4	D5	D6	D7	D8	I/Ic	Pat. GOM	Integral I
4721	2	00-001-0653	Calcium Hydroxide Phosphate	Ca H P O4	3.350000	2.950000	2.740000	1.720000	1.840000	2.500000	1.870000	1.910000		311	9.06
3936	2	00-003-0423	Calcium Hydrogen Phosphate	Ca H P O4	3.350000	2.950000	2.760000	2.720000	2.230000	2.500000	3.110000	1.840000		278	8.48
2694	3	00-035-1966	Chlorpropamide	C10 H13 Cl N2 O3 S	7.493250	4.548290	4.110600	3.584130	4.435690	3.172780	3.450170	4.036760		259	20.58
2681	2	01-071-1760	Calcium Hydrogen Phosphate	Ca H P O4	3.357880	2.950770	2.958630	2.717180	2.929480	2.752700	3.107930	1.721740	0.690000	283	9.13
2551	2	01-071-1759	Calcium Hydrogen Phosphate	Ca H P O4	3.357880	2.950770	2.958630	2.717180	3.336170	2.929480	2.752700	1.721740	0.680000	288	9.04
2534	2	04-009-6216	Hydrogen Calcium Phosphate	H Ca (P O4)	3.348940	3.377460	2.958150	2.968890	2.726570	2.937430	2.749950	1.728810	0.700000	285	8.32
2464	2	00-010-0190	Sodium Hydrogen Phosphate Hydrate	Na2 H P O4 · 2 H2 O	3.360000	4.640000	5.280000	2.871000	8.420000	3.260000	2.742000	2.926000		272	10.01

Matches Filter Filter Description
{Subfile/Subclass (Excipient Or Pharmaceutical)}

Experiment
Search Line(s): 2.47393 Å D1 Range: 2.464 - 2.484 Å Rotation: All

Preferences
Search Window: 0.15 ° Match Window: 0.15 °
Search Method: Hanawalt Lowest Allowable GOM: 500
Wavelength: Cu Kα1 1.54056 Å

Phases (5)

#	Accepted	PDF #	Compound Name	Int. Ratio	Int. %	I
1	<input checked="" type="checkbox"/>	01-086-2334	Calcium Carbonate	0.213	14	3.25
2	<input checked="" type="checkbox"/>	04-002-5015	Potassium Chloride	0.772	51	6.07
3	<input checked="" type="checkbox"/>	01-070-0360	Calcium Hydrogen Phosphate	0.221	15	0.71
4	<input checked="" type="checkbox"/>	00-022-1560	Ascorbic acid	0.21	14	
5	<input type="checkbox"/>	00-001-0653	Calcium Hydroxide Phosphate	0.085	6	

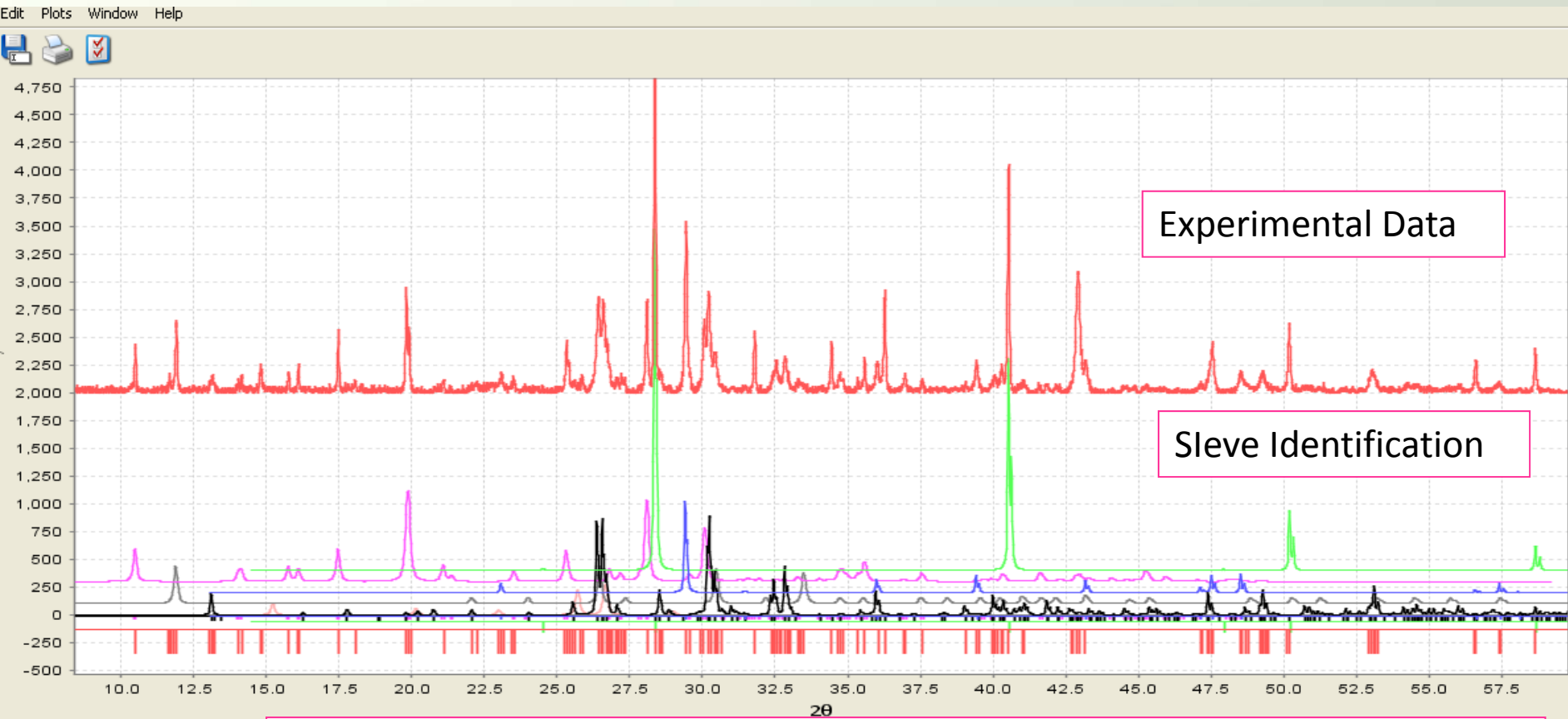
Lines (85 of 154)

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 I
3.29670	0										
3.29071	1					3.293220	10				
3.28574	1							3.281000	10		
3.27386	2										
3.26599	1										
3.26109	1										
3.17358	11							3.177000	100		
3.14245	100			3.145000	100						
3.12712	2					3.127430	24				
3.12265	2										
3.11818	2									3.110000	10
3.03155	22	3.035070	100			3.045430	1				
3.01732	2										
2.98436	0					2.986020	4	3.023000	8		
2.97219	8							2.972000	64		

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

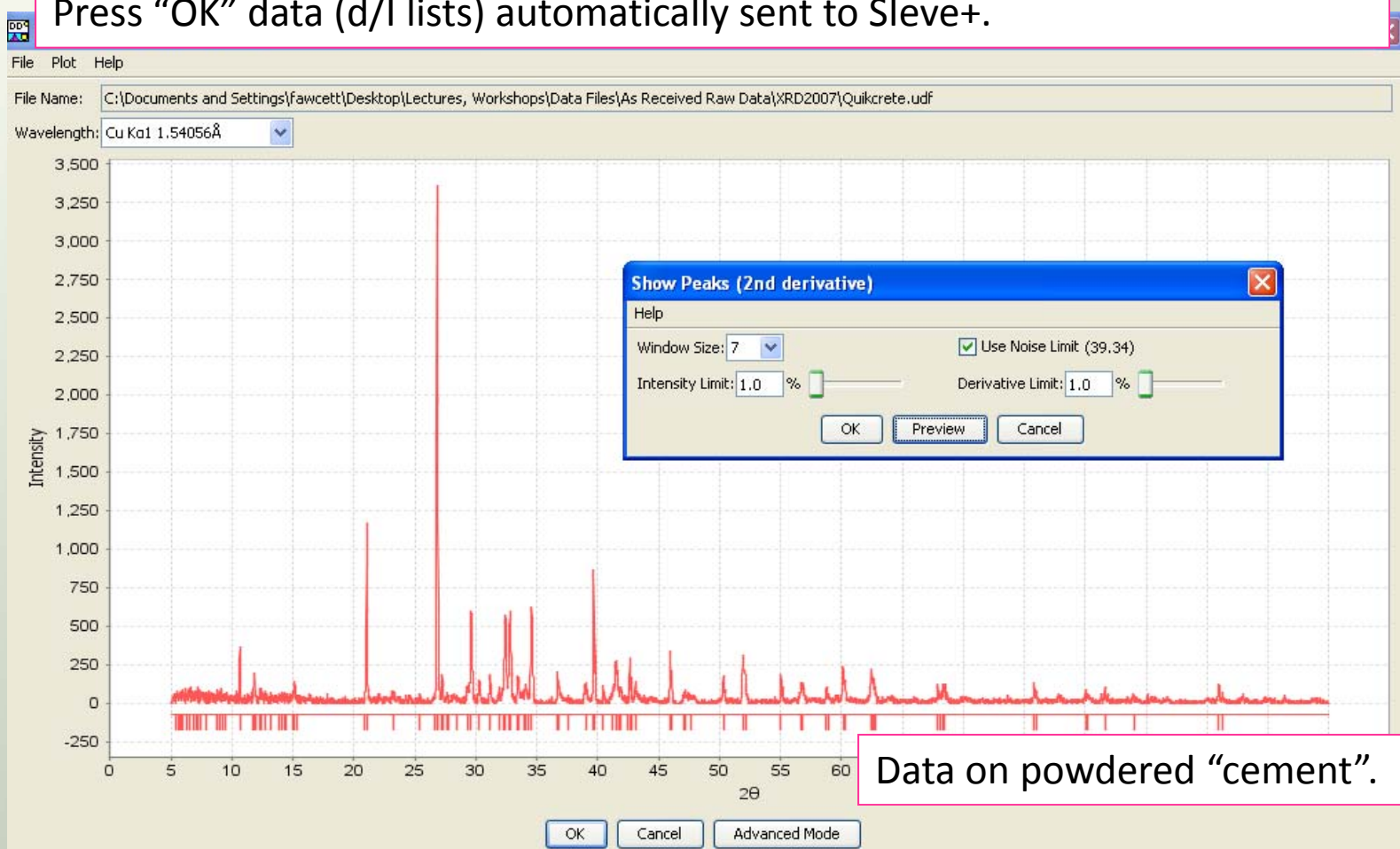


The identification finds Vitamins B and C in the tablet, as well as KCl, CaCO₃, CaHPO₄ and Iron Fumarate. These are plotted above with the data deliberately offset to show each phase. Further analyses (not shown) identifies ZnO, TiO₂ and MgO.

Case 4

Analysis of Concrete

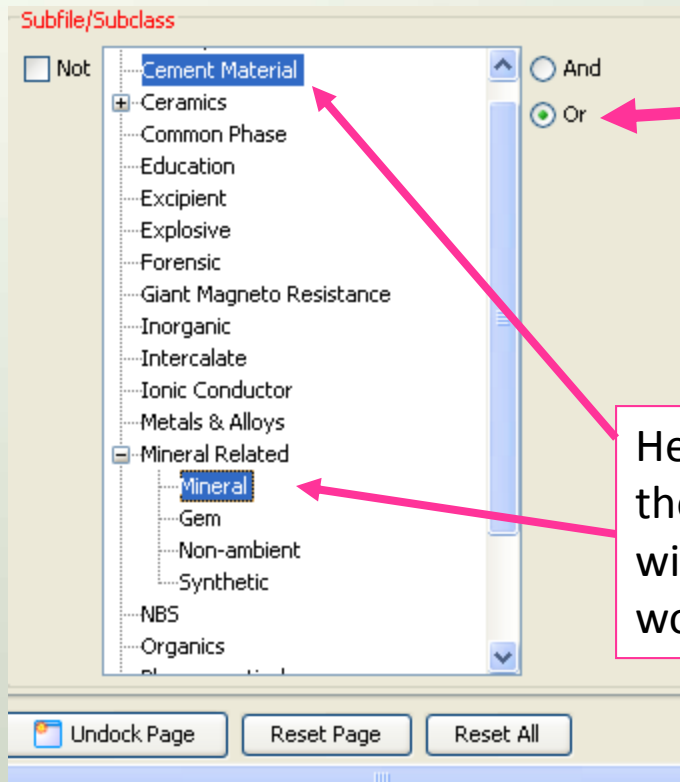
Import data, remove background, find peaks – See 1st slide of Case 1.
Press “OK” data (d/I lists) automatically sent to Sieve+.



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

Name	Description	Hits	Time
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
Search #6	{Subfile/Subclass (Cement Material Or Mineral)}	20,036	94.9s

OK Cancel

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

Case 4

Analysis of Concrete

SEARCH...

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 1.54056 Å

Phases (9)

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

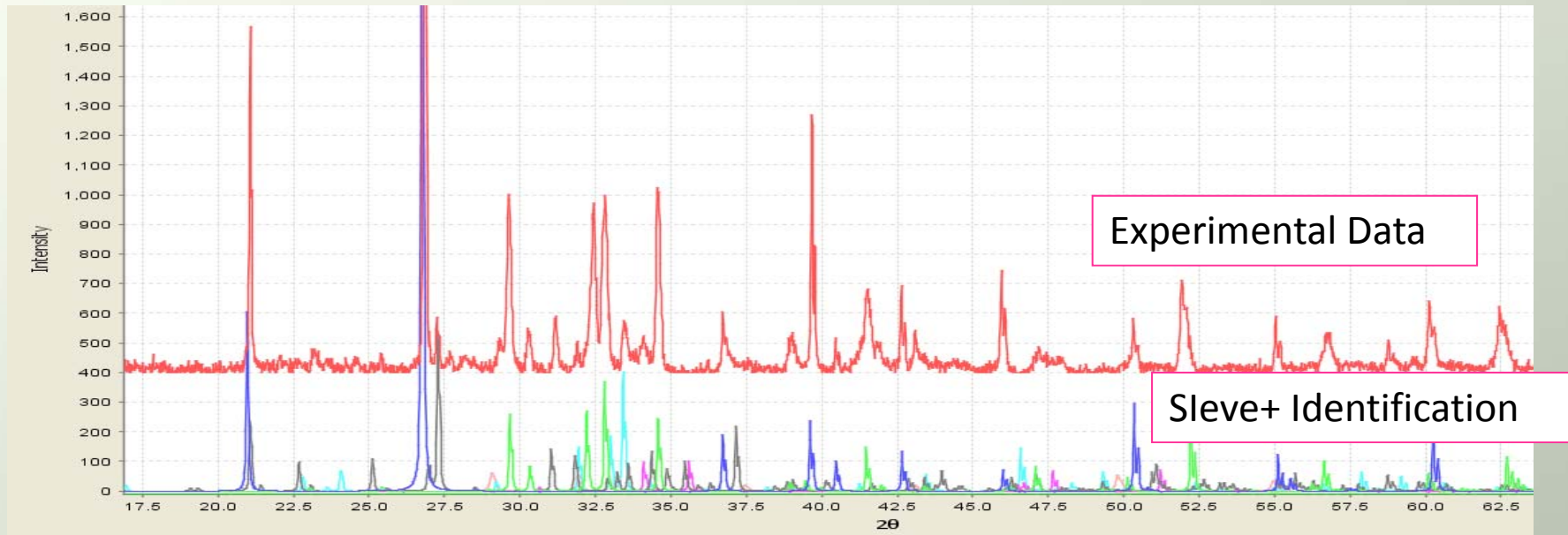
Lines (81 of 106)

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 I
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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