

PHASE IDENTIFICATION AND SEARCH/MATCH

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ACKNOWLEDGMENTS

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- Lachlan Cranswick
- Pamela Whitfield
- Nicola Döbelin
- Bussaraporn Patarachao

Special acknowledgments to:

- Mati Raudsepp
- Lee Groat and Julie (Melluish) McIntosh who started me on this XRD journey



INTRODUCTION

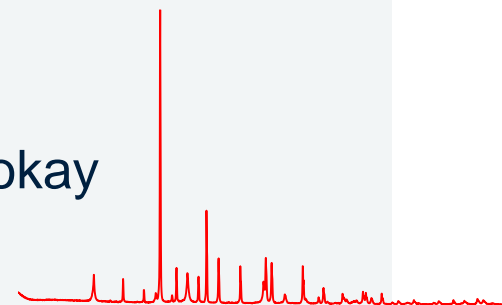


THE PROCESS OF PHASE IDENTIFICATION



What do we need?

- **Data:** acquire data for the purpose of phase ID – qualitative quality is okay
 - What do you do with it? How do you look at it?
- **Software for phase ID:** commercial, freeware; spreadsheet plot
 - Not absolutely necessary but it definitely helps
- **Database** ('fingerprints'): commercial database, free database, books, individual files
 - Historically, phase identification was done manually (Hanawalt, Fink methods)



SEARCH/MATCH



- From the DIFFRAC.EVA User Manual (DOC-M88-EXX200 V2 – 09.2011)

“The Aim of EVA Search/Match

The purpose of EVA Search/Match is to search the current scan of an **unknown** material and then identify **reference patterns** that are likely to explain the unknown scan. A Search algorithm is applied, comparing the reference patterns of a database to the scan. The algorithm gives a rank to the Patterns and lists the "best candidates". The user must compare the pattern to the scan and accept or reject the found pattern. This is called the match procedure.”

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BEFORE WE CONTINUE



Software and Databases shown and used in this presentation:

Software:

- EVA and Apex (Bruker)
- HighScorePlus (Malvern-Panalytical)
- QualX (Institute of Crystallography-CNR) – free for academic and non-profit

Databases:

- PDF-2 from the International Centre for Diffraction Data (ICDD)
- Crystallography Open Database (COD)
- American Mineralogist Crystal Structure Database (AMCSD)

DATABASES



- Inorganic and organic **databases** and crystal structure **resources**:

- PDF-5+, PDF-4 Minerals, PDF-4 Axiom and PDF-2 (Powder Diffraction File) from the **ICDD** (International Center for Diffraction Data) – commercial
- **ICSD** (Inorganic Crystal Structure Database) – commercial
- COD (Crystallography Open Database) – open access
- **AMCSD** (American Mineralogist Crystal Structure Database) – open access
- CSD (Cambridge Structural Database; organic and organometallic) – commercial

- for phase identification
- no atomic positions given so can't use for Rietveld work

- Published papers
- Unpublished data: your files; research group files – create your own database; personal communication

➤ CIF file → convert to diffraction pattern then bring it into the phase ID software to try to **match**

- For more information on databases: see International Tables for Crystallography (2019). Vol. H Chapter 3.7, pp. 304-324 (Crystallographic databases and powder diffraction by James Kaduk)

DATABASE EXAMPLE

You can search databases to find crystal structures and their information – but this is not the matching process...

... what do you need for matching?

American Mineralogist Crystal Structure Database

This site is an interface to a crystal structure database that includes every structure published in the American Mineralogist, The Canadian Mineralogist, European Journal of Mineralogy and Physics and Chemistry of Minerals, as well as selected datasets from other journals. The database is maintained under the care of the Mineralogical Society of America and the Mineralogical Association of Canada, and financed the National Science Foundation.

Logic interface

☒ AND ☐ OR

Viewing (About File Formats)

☒ amc long form ☐ amc short form ☐ cif

Download

☒ amc ☐ cif ☐ diffraction data

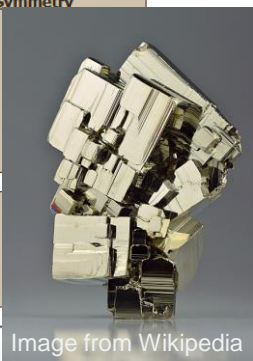


Image from Wikipedia

☐ Pyrite

Bayliss P

American Mineralogist 62 (1977) 1168-1172

Crystal structure refinement of a weakly anisotropic pyrite

cubic model

_database_code_amcsd 0000605

9 matching records for this search

☐ Pyrite

Ramsdell L S

American Mineralogist 10

The crystal structures of

Locality: natural, unknown

_database_code_amcsd 000

5.38 5.38 5.38 90 90 90

atom x y z

Fe 0 0 0

S .338 .338 .338

[Download AMC data \(View Text File\)](#)

[Download CIF data \(View Text File\)](#)

[Download diffraction data \(View Text File\)](#)

[View JMOl 3-D Structure \(permalink\)](#)

☐ Pyrite

Bayliss P

American Mineralogist 62

Crystal structure refinement of a weakly anisotropic pyrite

cubic model

_database_code_amcsd 0000605

5.4166 5.4166 5.4166 90 90 90 Pa3

atom x y z

Fe 0 0 0 .28

S .3851 .3851 .3851 .53

[Download AMC data \(View Text File\)](#)

[Download CIF data \(View Text File\)](#)

[Download diffraction data \(View Text File\)](#)

[View JMOl 3-D Structure \(permalink\)](#)

☐ Pyrite

Bayliss P

American Mineralogist 62 (1977) 1168-1172

Crystal structure refinement of a weakly anisotropic pyrite

_database_code_amcsd 0000605

5.4166 5.4166 5.4166 90 90 90 P1

atom x y z B(1,1) B(2,2) B(3,3) B(1,2) B(1,3) B(2,3)

Fe1 .0010 .0020 .0030 .0023 .0024 .0017 .0000 .0000 .0004

Fe2 .4966 .0001 .5036 .0026 .0026 .0019 .0002 .0003 .0001

Fe3 .5001 .5020 .0011 .0026 .0021 .0022 .0007 .0002 .0003

Fe4 .0006 .5013 .5038 .0026 .0026 .0016 .0001 .0002 .0001

S1 .3857 .3832 .3840 .0020 .0018 .0004 .0007 .0003 .0004

S2 .1149 .6114 .8846 .0038 .0069 .0050 .0002 .0001 .0007

S3 .8854 .1157 .6143 .0034 .0001 .0002 .0001 .0004 .0004

S4 .6153 .8865 .1141 .0039 .0075 .0039 .0003 .0008 .0010

S5 .6151 .6132 .6137 .0035 .0046 .0049 .0002 .0002 .0000

S6 .8854 .3818 .1149 .0026 .0005 .0002 .0000 .0003 .0002

S7 .1147 .8856 .3841 .0035 .0057 .0054 .0001 .0003 .0001

S8 .3857 .1161 .8842 .0014 .0004 .0011 .0004 .0001 .0004

[Download AMC data \(View Text File\)](#)

[Download CIF data \(View Text File\)](#)

CELL PARAMETERS: 5.4166 5.4166 5.4166 90.000 90.000 90.000

SPACE GROUP: Pa3

X-RAY WAVELENGTH: 1.541838

Cell Volume: 158.921

Density (g/cm3): 5.014

MAX. ABS. INTENSITY / VOLUME**2: 47.22399758

RIR: 3.067

RIR based on corundum from Acta Crystallographica A38 (1982) 733-739

2-THETA INTENSITY D-SPACING H K L Multiplicity

28.54 39.18 3.1273 1 1 1 8

33.08 95.52 2.7083 2 0 0 6

37.11 54.86 2.4224 2 1 0 12

40.81 45.47 2.2113 2 1 1 24

47.48 51.01 1.9151 2 2 0 12

56.33 100.00 1.6332 3 1 1 24

59.08 15.84 1.5636 2 2 2 8

61.75 16.11 1.5023 3 0 2 12

64.35 15.90 1.4476 3 2 1 24

64.35 5.65 1.4476 3 1 2 24

76.69 10.49 1.2427 3 3 1 24

79.06 6.56 1.2112 4 2 0 12

79.06 6.56 1.2112 4 0 2 12

81.42 8.55 1.1820 4 2 1 24

83.76 4.21 1.1548 3 3 2 24

88.41 12.51 1.1057 4 2 2 24

XPOW Copyright 1993 Bob Downs, Ranjini Swaminathan and Kurt Bartelmehs

reference, see Downs et al. (1993) American Mineralogist 78, 1104-1107.

Global

cal_name_mineral 'Pyrite'

author_name

ss P'

al_name_full 'American Mineralogist'

al_volume 62

al_year 1977

al_page_first 1168

al_page_last 1172

section_title

al structure refinement of a weakly anisotropic pyrite

model

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cal_formula_sum 'Fe S2'

length_a 5.4166

length_b 5.4166

length_c 5.4166

angle_alpha 90

angle_beta 90

angle_gamma 90

volume 158.921

crystal_density_diffrn 5.015

symmetry_space_group_name_H-M 'P a 3'

loop

_space_group_symop_operation_xyz

'x,y,z'

'1/2+z,x,1/2-y'

'z,1/2-x,1/2+y'

'1/2-z,1/2+x,y'

'-z,-x,-y'

'1/2+y,1/2-z,-x'

'1/2-y,-z,1/2+x'

'-y,1/2+z,1/2-x'

'y,z,x'

'x,1/2-y,1/2+z'

'1/2-x,1/2+y,z'

'1/2+x,y,1/2-z'

'-x,-y,-z'

'1/2-z,-x,1/2+y'

'-z,1/2+x,1/2-y'

'1/2+z,1/2-x,-y'

'z,x,y'

'1/2-y,1/2+z,x'

'1/2+y,z,1/2-x'

'y,1/2-z,1/2+x'

'-y,-z,-x'

'-x,1/2+y,1/2-z'

'1/2+x,1/2-y,-z'

'1/2-x,-y,1/2+z'

loop

_atom_site_label

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

Fe 0.00000 0.00000 0.00000 0.00355

S 0.38510 0.38510 0.38510 0.00418

HM: P a 3

a=5.417Å

b=5.417Å

c=5.417Å

α=90.000°

β=90.000°

γ=90.000°

People



Extra

REMEMBER DIFFRACTION

Crystalline materials have long range periodicity. Once you know the crystal structure of a material you can generate a list of expected diffraction peaks (peak positions/d-spacings with relative intensities) and this is like a **fingerprint** of the material. It is unique to the material although there may be similar diffraction patterns across similarly structured materials.

X-ray diffraction experiment: An X-ray beam interacts with a crystalline substance and diffraction occurs when the Bragg equation is satisfied resulting in a set of diffraction peaks. These peaks are characterized by peak positions (related to d-spacings), intensities, and peak shape.

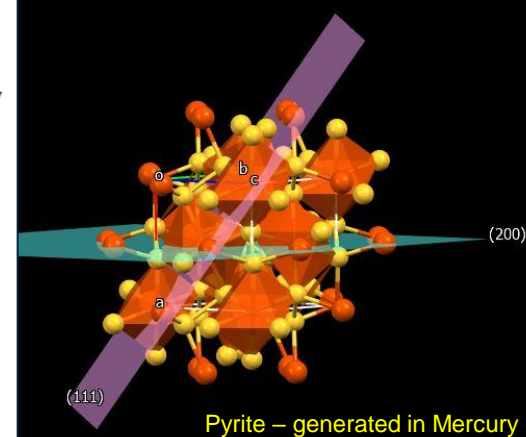
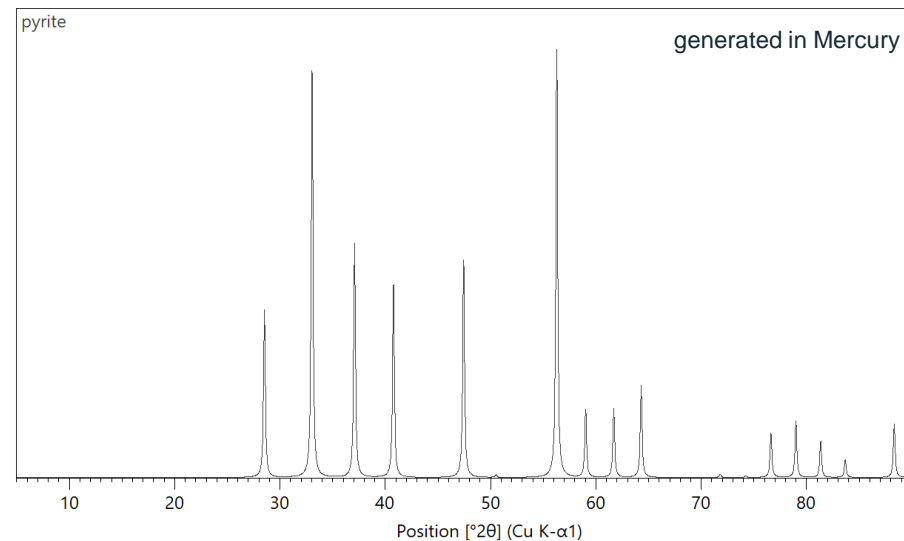
$$n\lambda = 2d\sin\theta \quad \text{Bragg equation}$$

Pyrite
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American Mineralogist 62 (1977) 1168-1172
Crystal structure refinement of a weakly anisotropic pyrite
cubic model
_database_code_amcsd 0000605

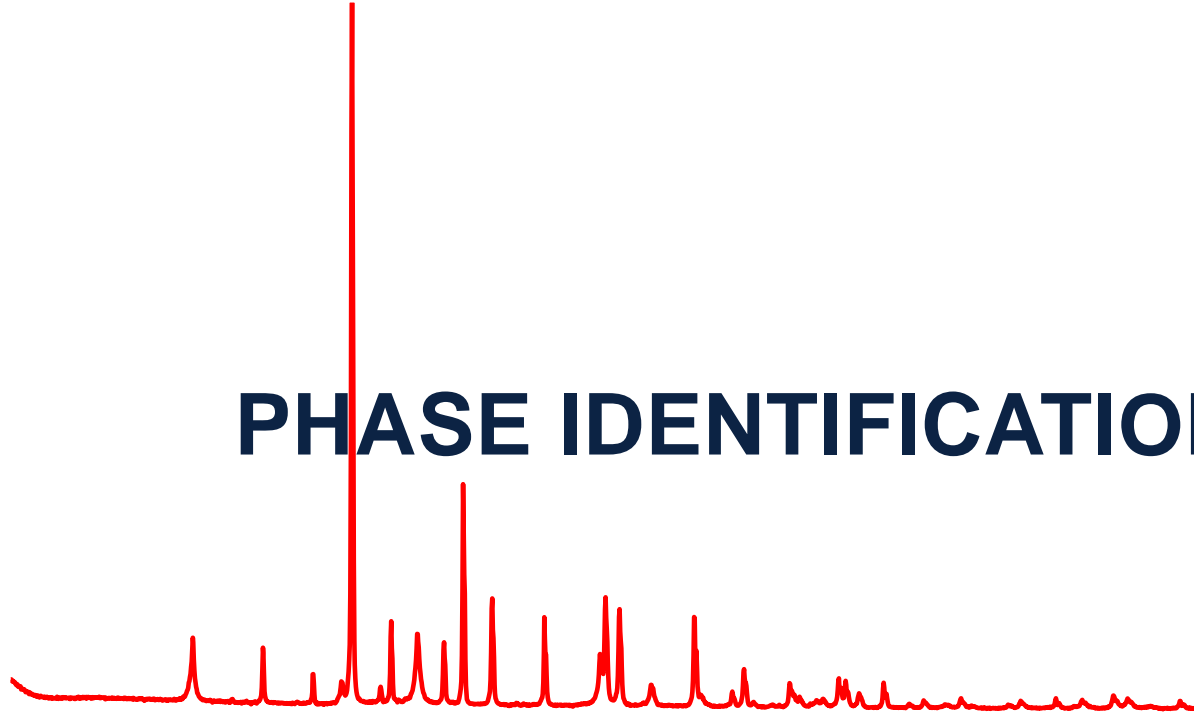
CELL PARAMETERS: 5.4166 5.4166 5.4166 90.000 90.000 90.000
SPACE GROUP: Pa3
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2-THETA	INTENSITY	D-SPACING	H	K	L	Multiplicity
28.54	39.18	3.1273	1	1	1	8
33.08	95.52	2.7083	2	0	0	6
37.11	54.86	2.4224	2	1	0	12
40.81	45.47	2.2113	2	1	1	24
47.48	51.01	1.9151	2	2	0	12
56.33	100.00	1.6332	3	1	1	24
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64.35	15.90	1.4476	3	2	1	24
64.35	5.65	1.4476	3	1	2	24
76.69	10.49	1.2427	3	3	1	24
79.06	6.56	1.2112	4	2	0	12
79.06	6.56	1.2112	4	0	2	12
81.42	8.55	1.1820	4	2	1	24
83.76	4.21	1.1548	3	3	2	24
88.41	12.51	1.1057	4	2	2	24

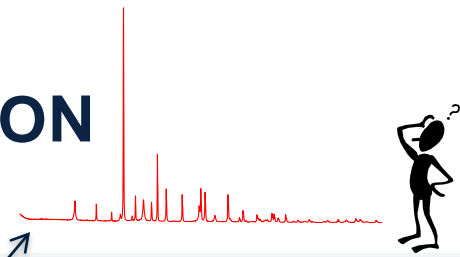
XPOW Copyright 1993 Bob Downs, Ranjini Swaminathan and Kurt Bartelmehs
reference, see Downs et al. (1993) American Mineralogist 78, 1104-1107.



PHASE IDENTIFICATION

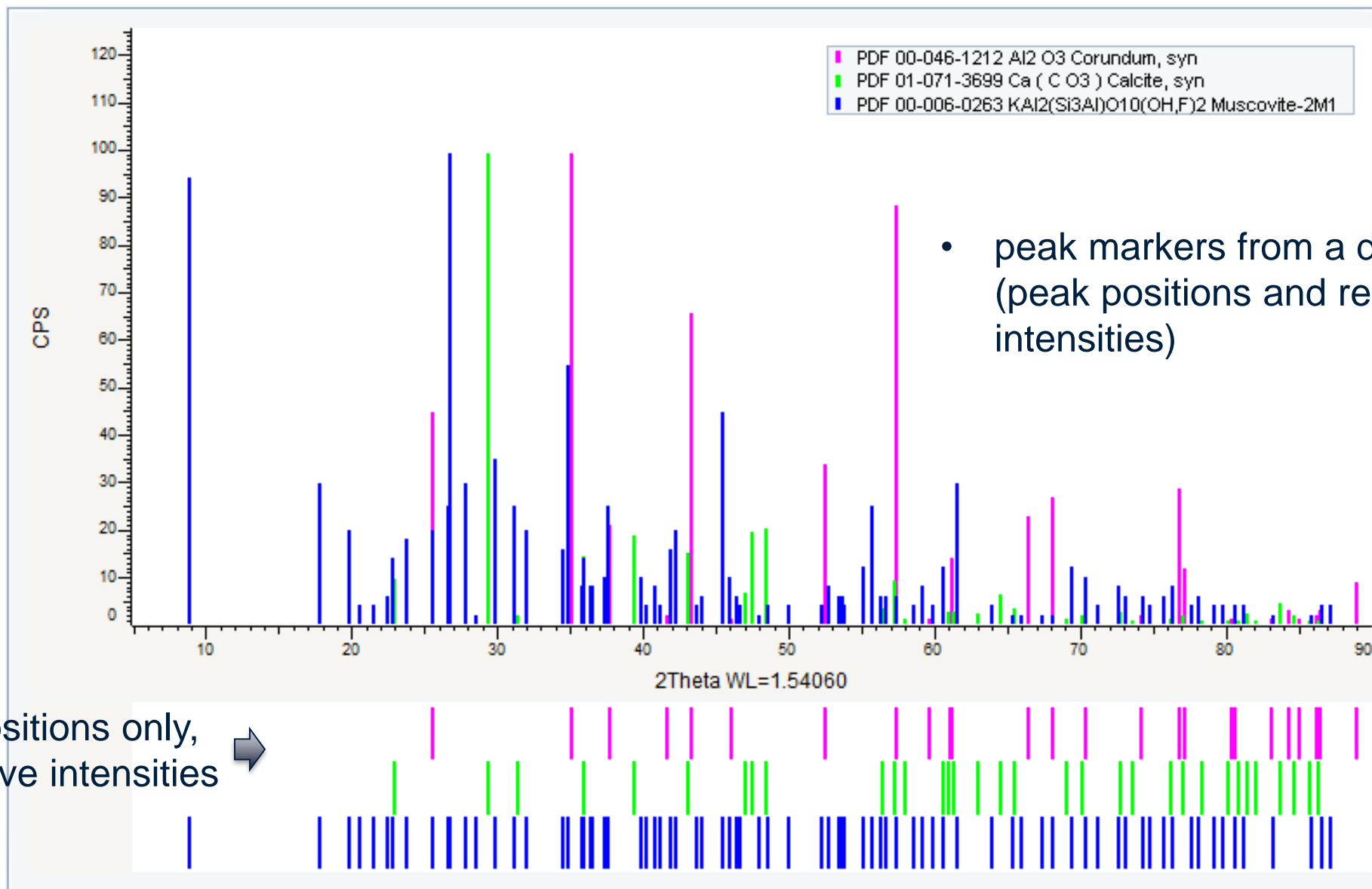


PHASE IDENTIFICATION

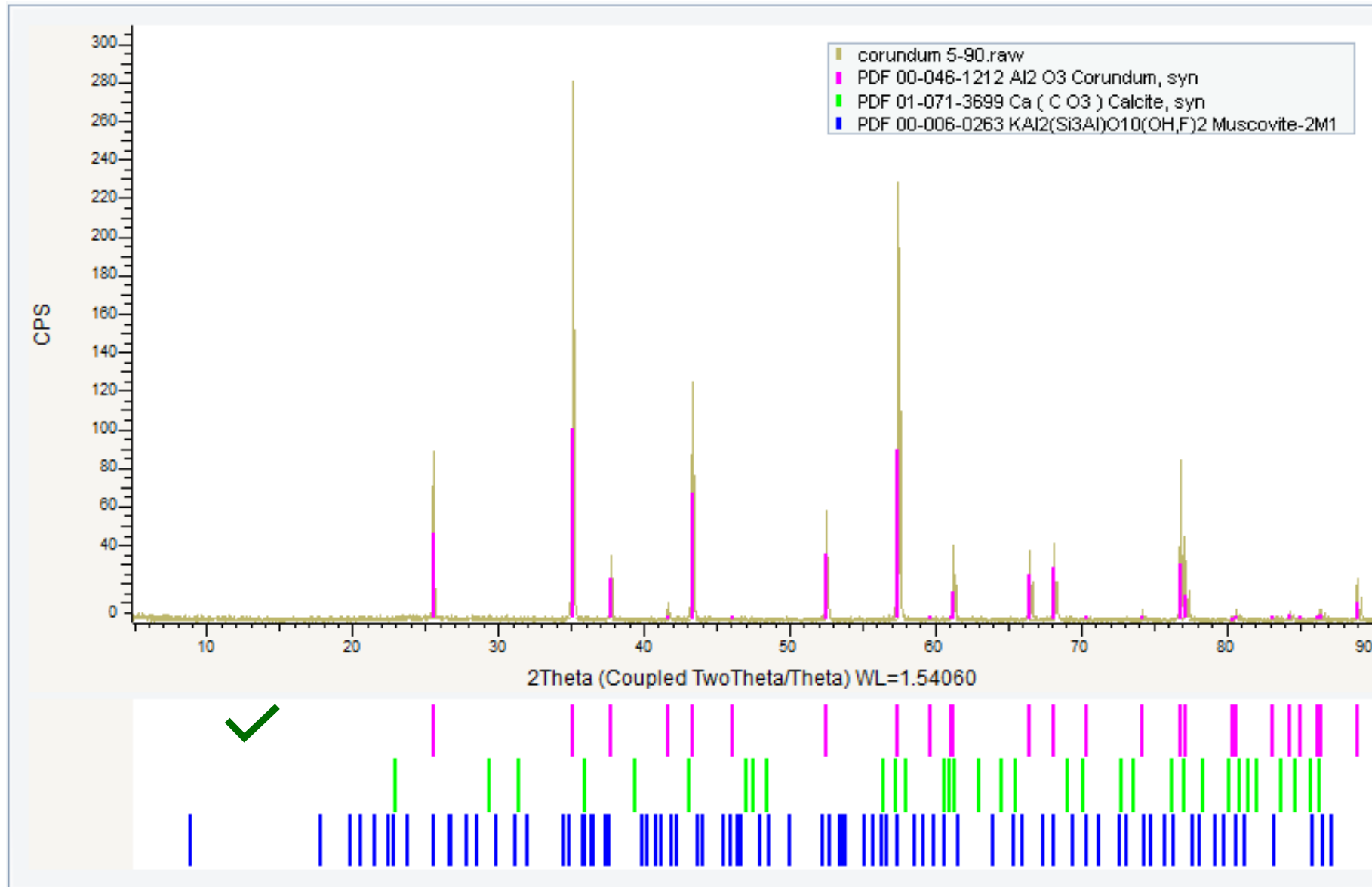


- It's like matching a fingerprint to known patterns in a database
- Peak positions are key for phase identification
 - Relative intensities are helpful but we know that the peak intensities may differ from ideal
- XRD is sensitive to structural differences; if you only have qualitative XRD data, you should be careful of drawing conclusions about the chemistry of the phases
 - chemical analyses and sample information help with phase identification
- Note: you need to do phase identification before you attempt to try quantitative phase analysis (QPA)

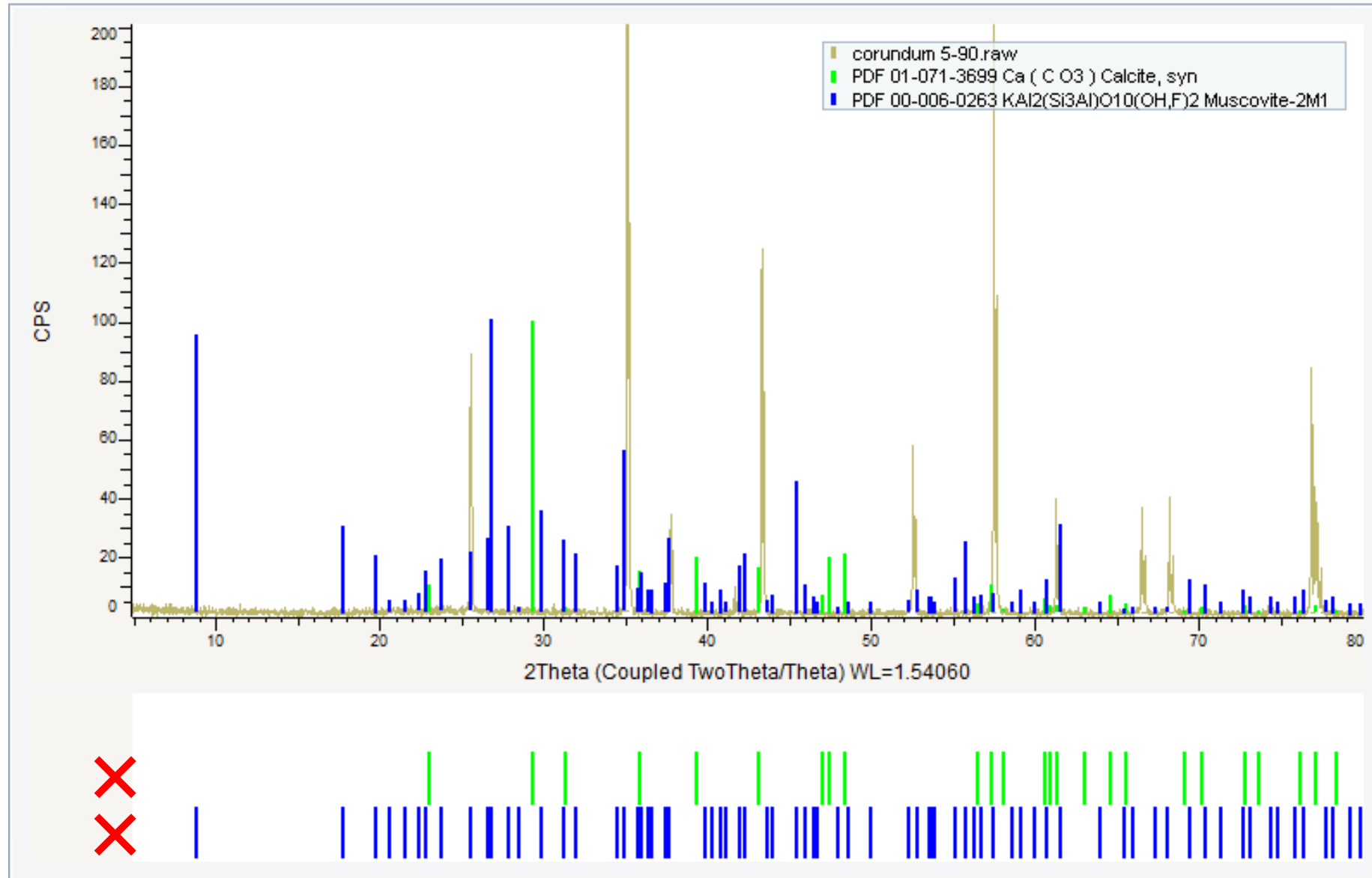
FINGERPRINT



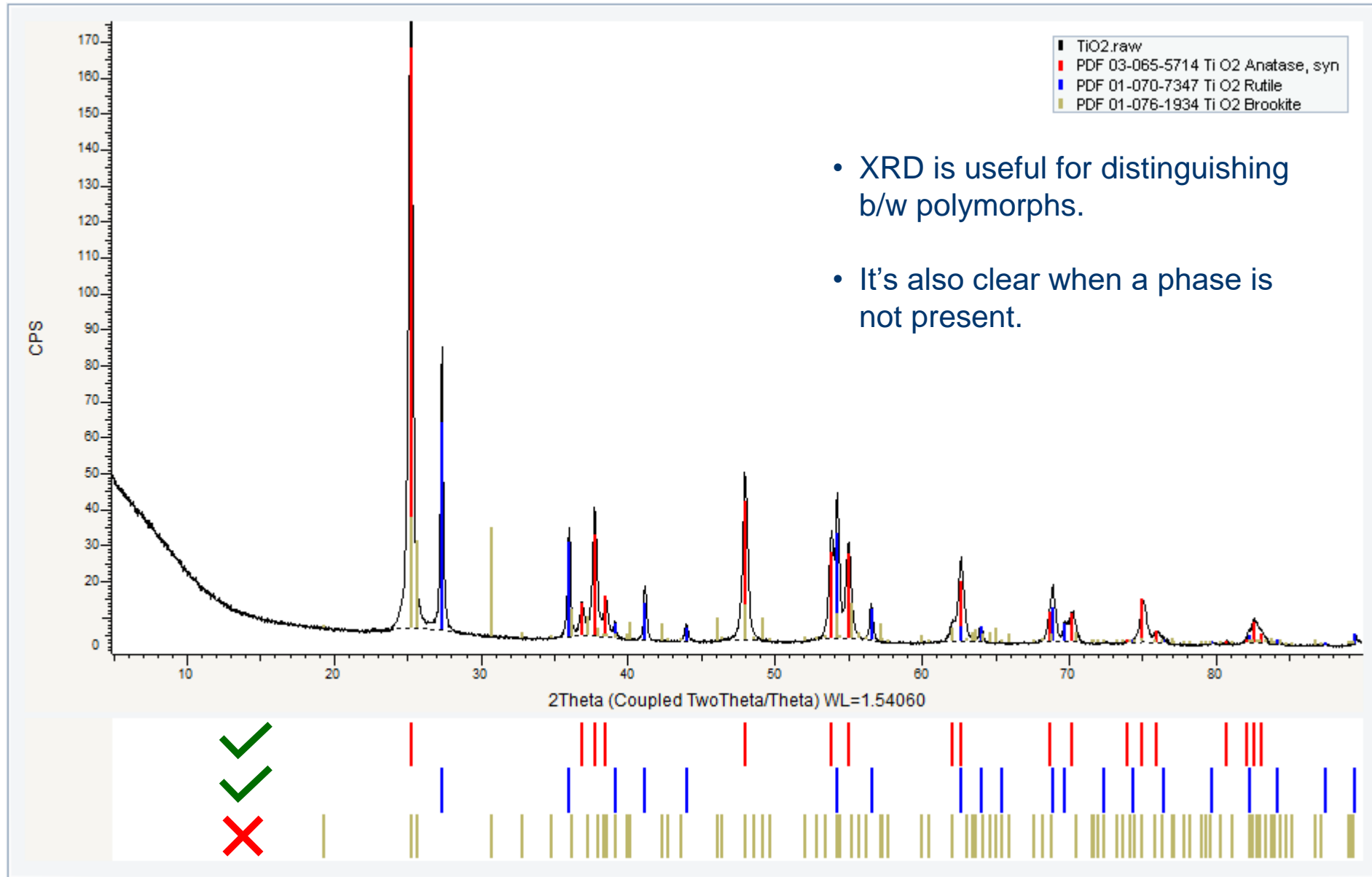
Match! – it is the set of peaks that make up the fingerprint of the pattern. You need to match the set not just one peak.

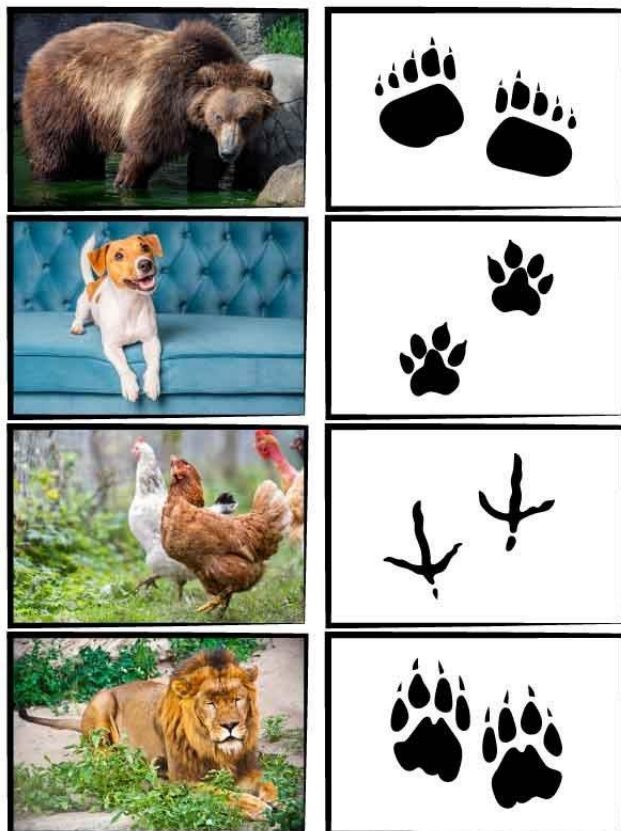


NOT a match! – it is the set of peaks that make up the fingerprint of the pattern. You need to match the set not just one peak. The absence of peaks should also match.



POLYMORPH EXAMPLE





MATCH

SEARCH/MATCH



- Search results are dependant on criteria input by the user
- Search results are suggestions or candidates
 - It is up to the researcher to select the most likely matches
 - Automatic match function in the software can be helpful but the software has no idea if the match that it selected is correct or not – that is up to you!
- An interactive process between the user and the software + database
 - User knowledge, experience, and intuition are valuable to the process

Get to work! Start practising and gaining experience!

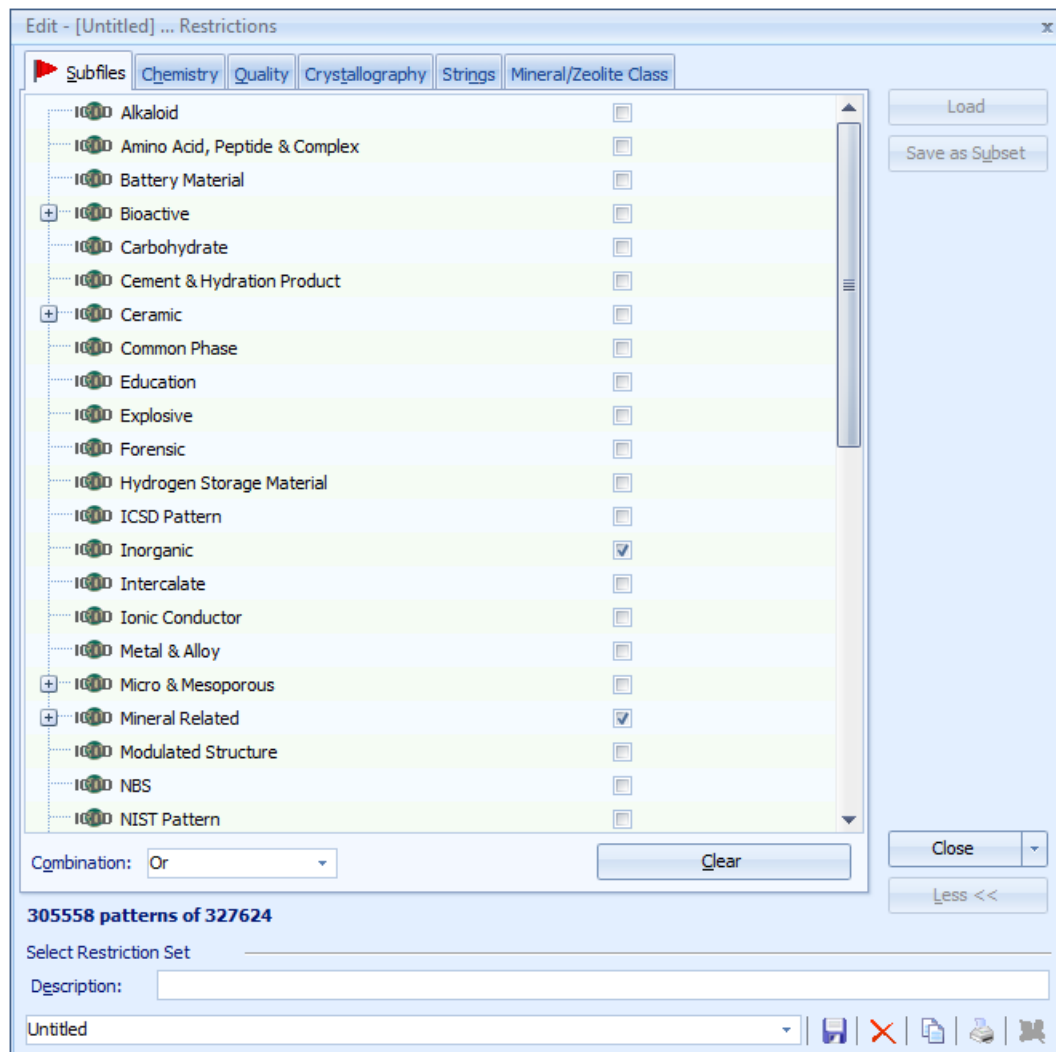
SEARCH/MATCH – SAMPLE INFORMATION



- Obtain as much information as possible about the sample
 - Where did it come from? What is likely to be in it? What are the reaction conditions? etc.
 - What industry is it from? – geological, cement, pharmaceutical, battery, etc.
 - Tip: useful to learn the language of the industry
 - Are there any chemical analyses available?
 - I usually ask the client to list **every possible element** in the system including anything the sample came into contact with such as the reaction vessel, atmosphere, wash solution, etc.
 - Have a sample with no information? – start with a light/common element search with the mineral database in the hope that it is a common substance
 - Minerals are naturally occurring crystalline materials and many are common substances (with a common name that's easy to recognize)
 - If you get stuck, go back to the client and ask for more information.

SEARCH – NARROWING THE FIELD

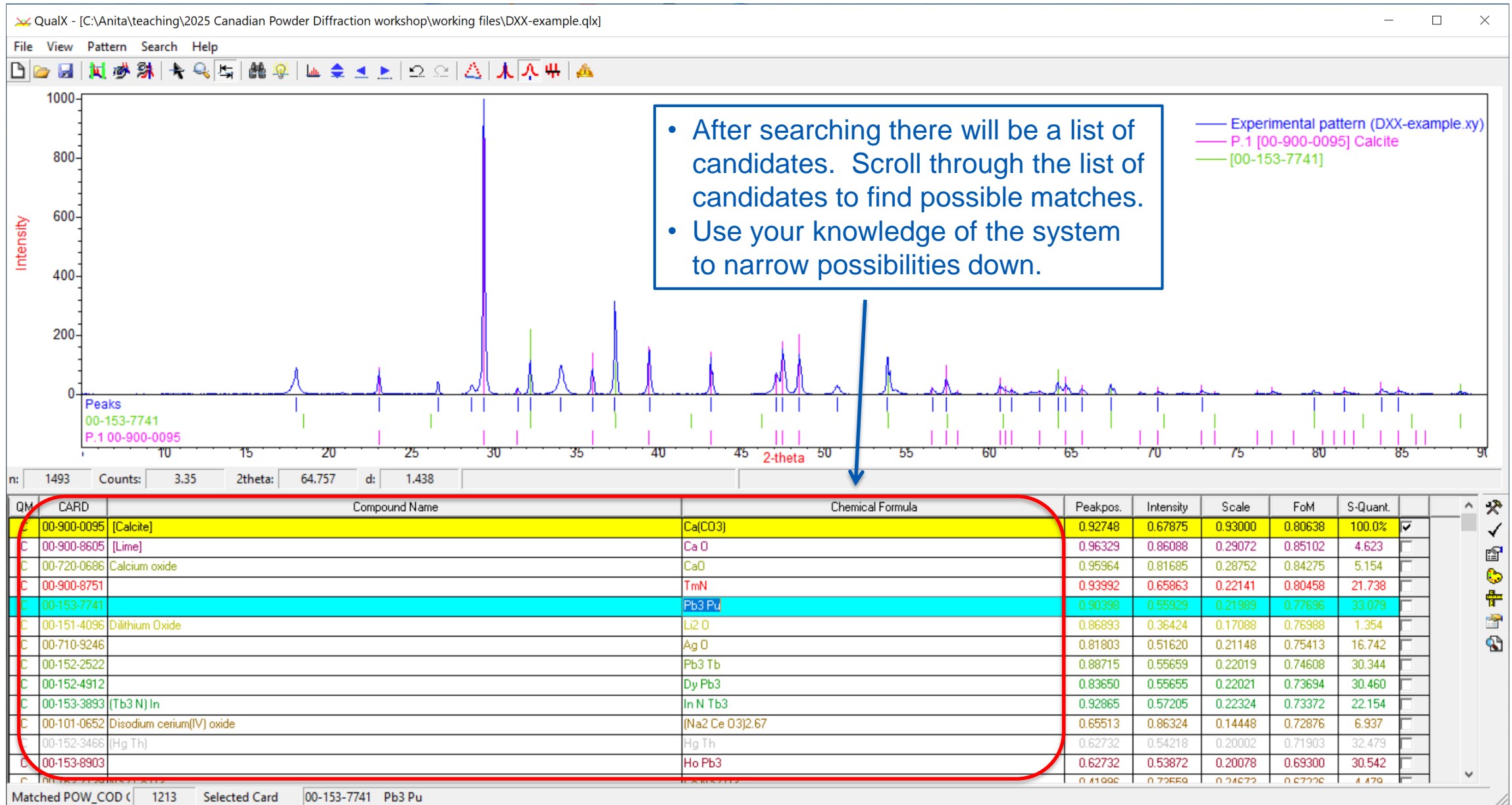
Database Subfiles:



Database Filter DXX-example.raw #1		
PDF-2 2022		
Auto Rebuild Click to filter Candidates Clear		
Import Export * Share		
Filter	Value	Candidates
<input checked="" type="checkbox"/> Full Files		
<input type="checkbox"/> Dual		27606
<input checked="" type="checkbox"/> Inorganic		277896
<input type="checkbox"/> Organic		22120
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<input type="checkbox"/> Colors		
<input type="checkbox"/> Sources		
<input checked="" type="checkbox"/> Element # in Formula		327624
<input type="checkbox"/> Min	1	≥ 1
<input type="checkbox"/> Max	25	≤ 25
<input checked="" type="checkbox"/> Density		327624
<input type="checkbox"/> Min	0	≥ 0
<input type="checkbox"/> Max	78.1	≤ 78.1
<input type="checkbox"/> Crystallographic Data		
<input type="checkbox"/> SQ Analysis		



MATCHING



SEARCHING – POSSIBLE ELEMENTS



Periodic Table

- Grey – possible elements
- Red – excluded elements
- Green – has to be there
- Blue – one of the elements has to be there

• A light element search

The image displays two screenshots of a software interface titled "Chemical Filter DXX-example.raw #1". The interface includes a dropdown menu set to "PDF-2 2022", buttons for "Auto", "Rebuild", "Click to filter Candidates", and "Clear". Below these are checkboxes for "D", "A", "M", and "N", along with "Import", "Export", and "Share" options.

The left screenshot shows a periodic table where most elements are grey. The first row (H, He) has H in grey and He in red. The second row (Li, Be) has Li in grey and Be in red. The third row (Na, Mg) has Na in grey and Mg in red. The rest of the table is grey.

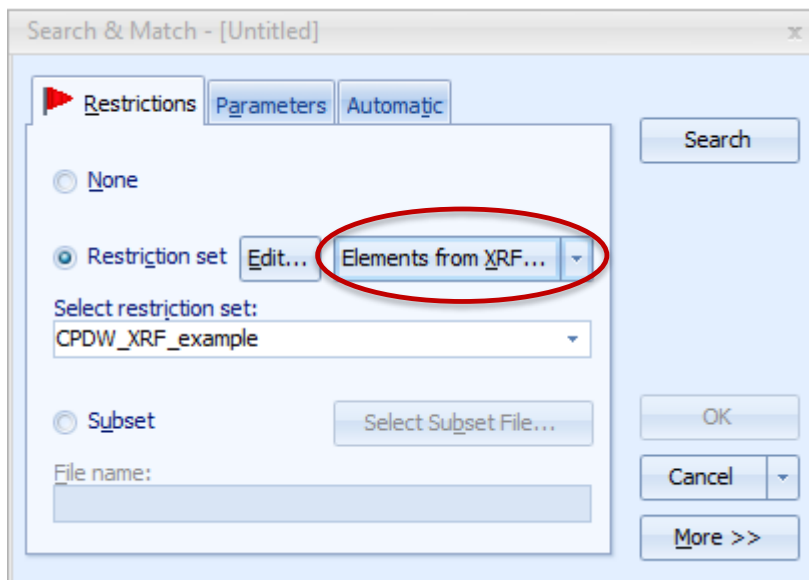
The right screenshot shows the same periodic table, but with a different filter. In addition to the red elements in the first three rows, the elements from Boron (B) to Xenon (Xe) in the p-block are also red. The rest of the elements remain grey.

SEARCHING – CHEMICAL ANALYSES

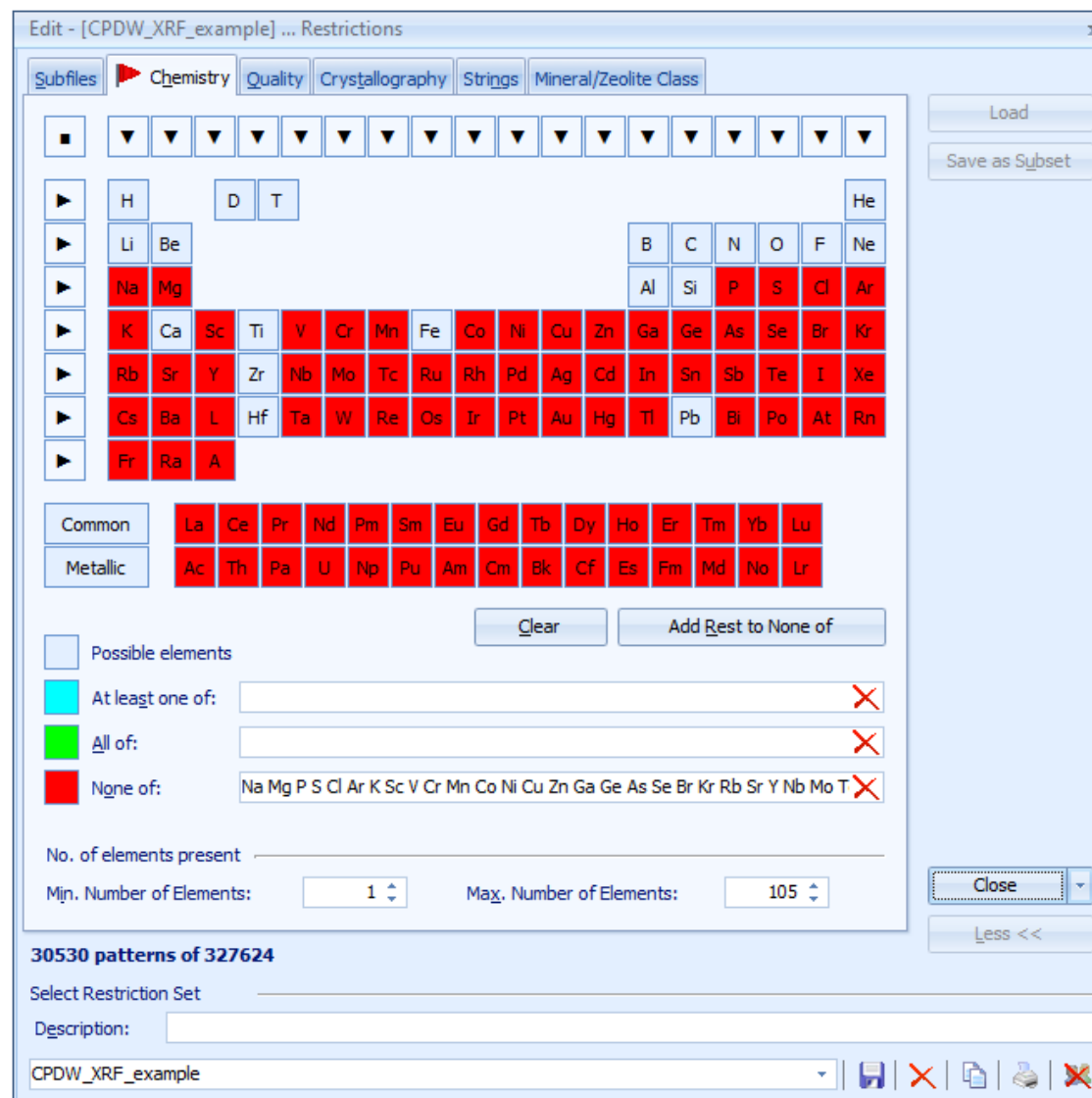


XRF results

- Grey – possible elements
- Red – excluded elements



- This allows you to search only the elements known to be in your system.



SEARCHING – ELEMENT SPECIFIC



Element focussed

- Grey – possible elements
- Red – excluded elements
- Green – has to be there

Chemical Filter unknown 2023.raw #2

PDF-2 2022

Auto Rebuild Click to filter Candidates Clear

D A M N

Share

H D T

Li Be B C N O F Ne

Na Mg Al Si P S Cl Ar

K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr

Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe

Cs Ba La Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn

Fr Ra Ac

Lanthanides / Actinides

Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu

Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No Ln

1 → Cu₂(OH)3NO₃

CuO·3H₂O

Cu_{0.4}V₂O₅

C₅₂H₅₆Cl₈CoCu₁₂

C₈H₈Cu₃Na₂O₁₆

Cu₄SO₄(OH)₆·H₂O

Cu₆Al(SO₄)(OH)₁₂Cl(H₂O)₃

C₁₅H₁₄Cu₄O₂S₂·2H₂O

Cu₄(OH)₆SO₄·H₂O

Na₄(Cu₄O₂(SO₄)₄)((Na_{0.26}Cu

C₄H₁₀Cu₃O₁₄P₄·6H₂O

C₃₈H₃₈Cu₈O₂

((N(CH₃)₄)(CuZn(CN)₄))(CCl₄)_{0.9}

Cu_{1.3}V₉O₂₂

C₂₁H₂₄CuF₁₂N₂O₆

C₂₈H₂₂CuN₂O₄

3CuO·2(P₂O₅)

Cu₂(NO₃)(OH)₃

Zn₇Cu(O₄H₇)_{0.3}(OH)₁₃((SiO(OH)₃)_{0.7}(SO₄))

Na₂Cu(C₂O₄)₂(H₂O)₂

21 → Cu₆(Al_{0.94}Fe_{0.06})(SO₄)(OH)₁₂Cl(H₂O)₃

22 → Na₂(Cu₃(CHO₂)₈)

Searching for a copper phase

Chemical Filter unknown 2023.raw #2

PDF-2 2022

Auto Rebuild Click to filter Candidates Clear

D A M N

Share

H D T

Li Be B C N O F Ne

Na Mg Al Si P S Cl Ar

K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr

Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe

Cs Ba La Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn

Fr Ra Ac

Lanthanides / Actinides

Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu

Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No Ln

Searching for an oxide

Na ₂ (CO ₃)
Na _{7.4} Al _{6.4} Si _{25.6} O ₆₄ ·O ₂
Na ₂ CO ₃
Na ₂ (CO ₃)
Na ₂ (CO ₃)H ₂ O
C ₄₈ H ₇₄ O ₁₄ ·0.5H ₂ O·0.82(C ₂ H ₅ OH)
Cu ₂ (OH) ₃ NO ₃
C ₂₀ H ₁₉ N ₅ O ₆ Na ₂ ·7H ₂ O
C ₃ H ₄ CoO ₅ ·H ₂ O
C ₂ H ₅ N ₅ O ₃ ·H ₂ O
CoLi _{1.865} (P ₂ O ₇)
C ₃ H ₃ N ₃ O ₂
13 → Na ₂ (CO ₃)
14 → CuO·3H ₂ O
15 → Na ₄ Ti ₅ O ₁₂
16 → C ₄₀ H ₅₀ N ₄ O ₄
17 → K(OH)
18 → O ₂ NC ₆ H ₄ CONHNC ₆ H ₄ N(C ₆ H ₄ CH ₃) ₂ ·H ₂ O
19 → C ₂₆ H ₂₃ N ₅ O ₂ S
20 → C ₆ H ₉ O ₆ Sc
21 → Na ₂ (CO ₃)(H ₂ O)
22 → Zn ₂ V ₂ O ₇

SEARCHING – ELEMENT SPECIFIC

Element focussed

- Grey – possible elements
- Red – excluded elements
- Green – has to be there
- Blue – one of the elements has to be there



▶	H		D	T															He
▶	Li	Be									B	C	N	O	F				Ne
▶	Na	Mg									Al	Si	P	S	Cl				Ar
▶	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr	
▶	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe	
▶	Cs	Ba	L	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn	
▶	Fr	Ra	A																
Common	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu				
Metallic	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr				

No.	Ref. Code	Chemical Formula
1	ICDD 01-082-1460	Rb Fe (Se O4)2
2	ICDD 00-068-0618	Sr Ca Ni Te O6
3	ICDD 01-085-7056	Sr3 Fe2 Te O9
4	ICDD 01-084-8465	Sr Ca Ni Te O6
5	ICDD 01-075-8547	Sr2 Fe1.33 Te0.67 O6
6	ICDD 01-075-8546	Sr2 Fe1.33 Te0.67 O6
7	ICDD 01-075-8548	Sr2 Fe1.33 Te0.67 O6
8	ICDD 01-075-8549	Sr2 Fe1.33 Te0.67 O6
9	ICDD 01-089-8379	Sr2 Cu (Te O6)
10	ICDD 01-072-4327	Cd2 (Te2 O7)
11	ICDD 00-050-0401	Cu Sr2 Te O6
12	ICDD 01-075-9489	Sr2 ((Fe1.33 Te0.67) O6)
13	ICDD 00-047-0010	Sc2 Te4 O11
14	ICDD 01-080-8053	Sr4 (V O2)2 (Se O3)4 (Se2 O5)
15	ICDD 01-083-3020	Se Mn O3
16	ICDD 01-076-3289	Sr2 Fe1.47 Te0.53 O6
17	ICDD 01-077-5338	Ag4 (Mo2 O5) (Se O4)2 (Se O3)
18	ICDD 00-055-0529	Cd2 Te2 O7
19	ICDD 01-077-5586	Cd4 V2 Te3 O15
20	ICDD 01-070-0241	Mn (Se O3)
21	ICDD 00-031-0830	Mn Se O3
22	ICDD 00-036-0881	Ca Te O3
23	ICDD 01-072-7832	Mn (Se O3)
24	ICDD 01-085-2142	Ca3 Fe2 (Se O3)6
25	ICDD 01-086-9658	Cu2 (Se O3) F2
26	ICDD 01-076-9766	Sr (Te3 O8)
27	ICDD 00-053-0208	Sr2 Fe1.33 Te0.67 O6
28	ICDD 00-029-0897	Sr2 Mn Te O6
29	ICDD 01-082-3425	Y6 Te O12
30	ICDD 01-072-7831	(Cu0.25 Mn0.75) (Se O3)

These are just a few examples – the search criteria should be tailored to your samples.

SEARCHING – CHEMICAL FILTER



Element focussed

- Grey – possible elements
- Red – excluded elements
- Green – has to be there
- Blue – one of the elements has to be there

Search using common elements – all blue is okay

Common

Metallic

Clear

Add Rest to None of

Possible elements

At least one of: H Li B C N O F Na Mg Al Si P S Cl K Ca Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se X

All of: X

None of: He Be Ne Ar Sc Kr Rb Y Tc Ru Rh Pd Te Xe Cs La Ce Pr Nd Pm Sm Eu Gd Tb D X

SEARCHING – CHEMICAL FILTER



Element focussed

- Grey – possible elements
- Red – excluded elements
- Green – has to be there
- Blue – one of the elements has to be there

Don't do this.....Why?

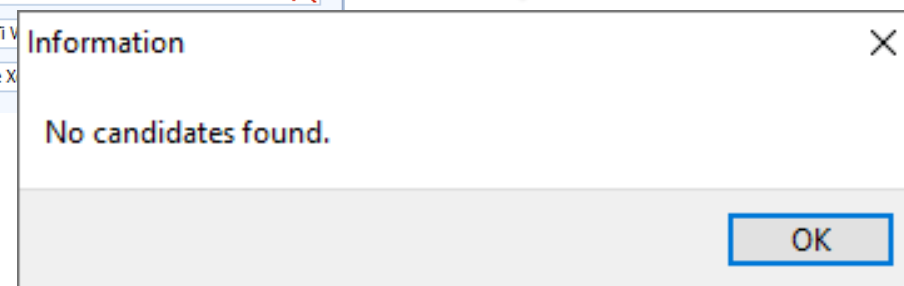
The screenshot shows a chemical filter interface with a periodic table. Elements are colored: Grey (possible), Red (excluded), Green (must be present), and Blue (one must be present). The 'None of' list is empty, which triggers an error message.

Common	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Metallic	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

Legend:

- ☐ Possible elements
- ☒ At least one of:
- ☒ All of:
- ☒ None of:

Buttons: Clear, Add Rest to None of



SEARCHING – CHEMICAL FILTERS



Element focussed

- Grey – possible elements
- Red – excluded elements
- Green – has to be there
- Blue – one of the elements has to be there

Some built-in filters are available or you can create your own.

Chemical Filter DXX-example.raw #1

PDF-2 2022

Auto Rebuild Click to filter candidates Clear

Import Export **Rock Forming Minerals** Delete Share

H	D T																He						
Li	Be																	B	C	N	O	F	Ne
Na	Mg																	Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr						
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe						
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn						
Fr	Ra	Ac																					
Lanthanides / Actinides			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu							
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Ln							

SEARCHING – BY NAME OR FILE NUMBER



Scans Search List DB Preview

	Formula	Name
1	SiO ₂	Quartz
2	SiO ₂	Quartz
3	SiO ₂	Quartz
		Quartz
		Quartz
		Quartz
		Quartz
		Quartz, low
		Quartz
		high quartz Silicon Oxide
		Quartz, syn
		Quartz, syn
		β-quartz Silicon Oxide
		quartz low, syn Silicon Oxide
		Quartz, syn
		quartz low Silicon Oxide
		quartz low Silicon Oxide
		quartz low Silicon Oxide
		quartz low Silicon Oxide
		quartz low Silicon Oxide
		quartz low Silicon Oxide
		A-Si O ₂ , quartz HP, syn Silico...
		A-Si O ₂ , quartz HP, syn Silico...
		A-Si O ₂ , quartz HP, syn Silico...

Search / Match DXX-example.raw #1

Listed 149 / 327624 Candidates in 0.0 s.

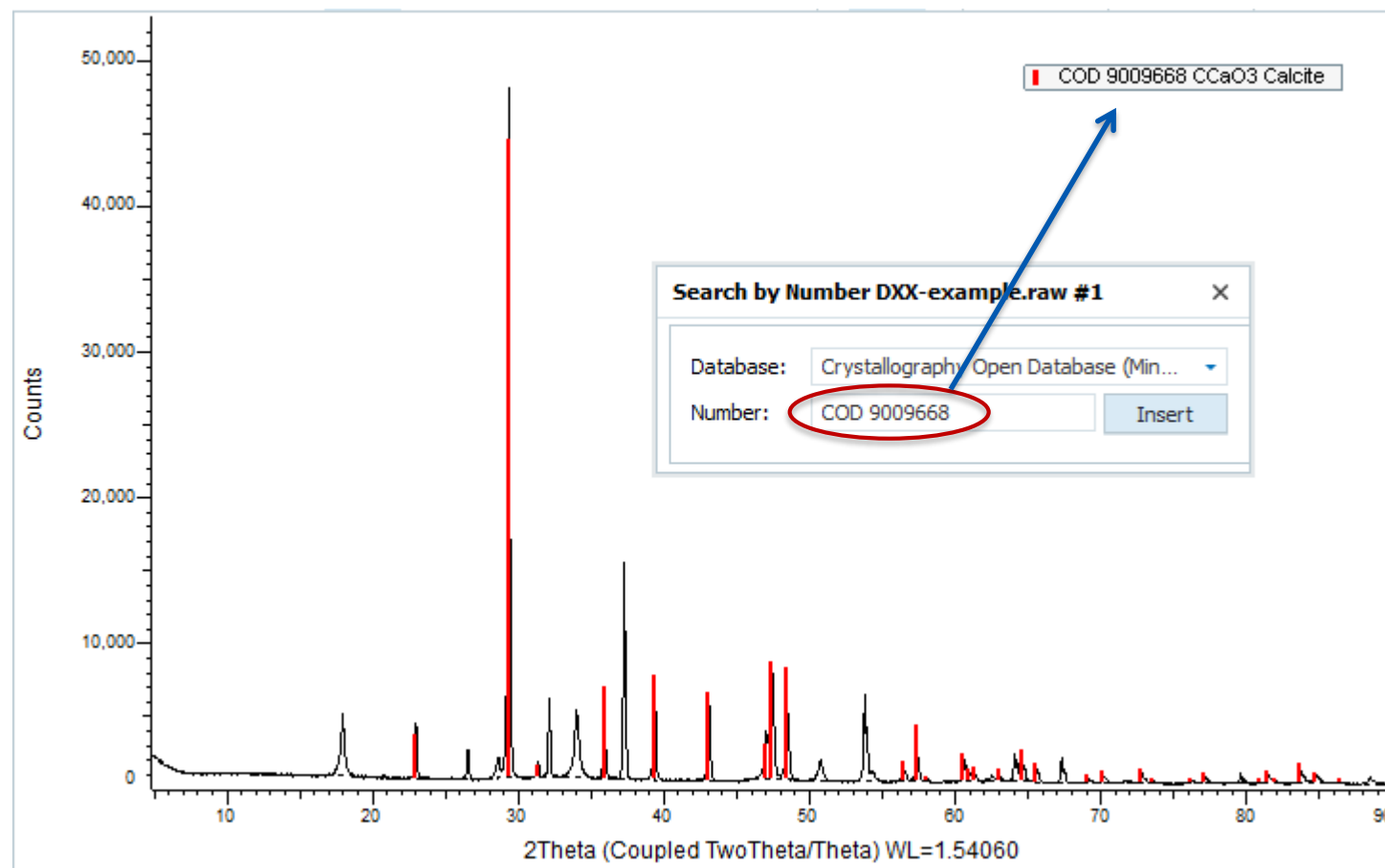
Databases Match Lists **Names** Options

quartz Use Filters ☐

Use ? and * for partial names e.g. BO*HMIT?, parenthesis for authors e.g. (BOGUE), curly brackets for formulas e.g. {CaCO₃}, dash to negate the result e.g. QUARTZ -BETA

And ☒ Or ☐

ABC Name



SEARCHING – BY CRYSTALLOGRAPHY



Edit - [Untitled] ... Restrictions

Subfiles Chemistry Quality **Crystallography** Strings Mineral/Zelite Class

Min Max Min Max

a [Å]: 5.405 5.415

b [Å]: 5.405 5.415

c [Å]: 5.405 5.415

alpha [°]:

beta [°]:

gamma [°]:

ratio a/c:

Density [g/cm³]:

No. of Formula Units (Z):

Reference Intensity Ratio:

Cell Volume [Å³]:

Space Group Number:

Crystal System: Cubic

Load

Save as Subset

Min - Max

Value - Deviation [%]

Clear

Close

Less <<

148 patterns of 327624

Select Restriction Set

Description:

Untitled

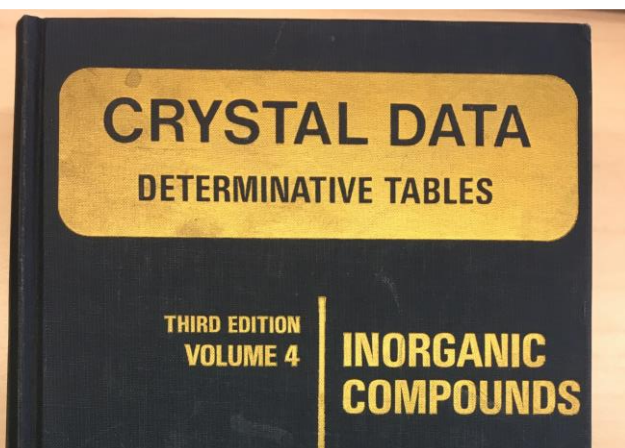
Candidates: Search

No.	Ref. Code	Cryst. Syst.	Space Gr...	Cell	Compound Name
1	ICDD 03-065-2974	Cubic	Fm-3m	a 5.406 - b 5.406 - c 5.406	Nickel Silicon
2	ICDD 00-047-1655	Cubic	F-43m	a 5.407 - b 5.407 - c 5.407	Cobalt Zinc Sulfide
3	ICDD 00-016-0576	Cubic	F-43m	a 5.405 - b 5.405 - c 5.405	Strontium Arsenic Sulfide
4	ICDD 01-085-8588	Cubic	F-43m	a 5.406 - b 5.406 - c 5.406	Zinc Sulfide
5	ICDD 01-081-5205	Cubic	F-43m	a 5.410 - b 5.410 - c 5.410	Copper Sulfide
6	ICDD 01-071-5971	Cubic	F-43m	a 5.409 - b 5.409 - c 5.409	Zinc Sulfide
7	ICDD 01-073-8755	Cubic	F-43m	a 5.409 - b 5.409 - c 5.409	Nickel Zinc Sulfide
8	ICDD 03-065-4586	Cubic	F-43m	a 5.409 - b 5.409 - c 5.409	Nickel Zinc Sulfide
9	ICDD 03-065-9585	Cubic	F-43m	a 5.409 - b 5.409 - c 5.409	Zinc Sulfide
10	ICDD 01-071-4258	Cubic	F-43m	a 5.407 - b 5.407 - c 5.407	Cobalt Iron Zinc Sulfide
11	ICDD 01-078-2500	Cubic	Fd-3m	a 5.406 - b 5.406 - c 5.406	Silicon
12	ICDD 01-072-2669	Cubic	F-43m	a 5.411 - b 5.411 - c 5.411	Nickel Zinc
13	ICDD 03-065-9470	Cubic	F-43m	a 5.411 - b 5.411 - c 5.411	Nickel Zinc
14	ICDD 00-050-0202	Cubic	Fm-3m	a 5.413 - b 5.413 - c 5.413	Cerium Gadolinium Yttrium ...
15	ICDD 01-080-6505	Cubic	Fm-3m	a 5.405 - b 5.405 - c 5.405	Calcium Cerium Oxide
16	ICDD 03-065-7441	Cubic	F-43m	a 5.411 - b 5.411 - c 5.411	Manganese Zinc Sulfide
17	ICDD 00-027-0843	Cubic		a 5.410 - b 5.410 - c 5.410	Neodymium Antimony Oxide
18	ICDD 01-081-9692	Cubic	Fm-3m	a 5.406 - b 5.406 - c 5.406	Cerium Yttrium Oxide
19	ICDD 01-080-6915	Cubic	Fm-3m	a 5.405 - b 5.405 - c 5.405	Cerium Oxide
20	ICDD 01-080-5532	Cubic	Fm-3m	a 5.406 - b 5.406 - c 5.406	Cerium Yttrium Oxide
21	ICDD 01-073-2568	Cubic	F-43m	a 5.406 - b 5.406 - c 5.406	Zinc Sulfide
22	ICDD 01-082-9831	Cubic	Fm-3m	a 5.406 - b 5.406 - c 5.406	Cerium Oxide
23	ICDD 01-075-0167	Cubic	Fm-3m	a 5.405 - b 5.405 - c 5.405	Dysprosium Cerium Oxide
24	ICDD 01-089-4958	Cubic	F-43m	a 5.411 - b 5.411 - c 5.411	Manganese Zinc Sulfide
25	ICDD 01-074-6110	Cubic	F-43m	a 5.411 - b 5.411 - c 5.411	Zinc Sulfide
26	ICDD 03-065-1691	Cubic	F-43m	a 5.411 - b 5.411 - c 5.411	Zinc Sulfide
27	ICDD 01-081-9649	Cubic	F-43m	a 5.409 - b 5.409 - c 5.409	Molybdenum Phosphide
28	ICDD 01-071-6544	Cubic	F-43m	a 5.412 - b 5.412 - c 5.412	Zinc Iron Sulfide
29	ICDD 01-083-5829	Cubic	Fm-3m	a 5.406 - b 5.406 - c 5.406	Cerium Praseodymium Oxide
30	ICDD 01-080-4831	Cubic	Fm-3m	a 5.406 - b 5.406 - c 5.406	Cerium Praseodymium Oxide

SEARCHING – BY UNIT CELL



- an aside: (single crystal) users sometimes do phase ID by checking the unit cell



ORTHORHOMBIC

a/b	c/b	a
0.5729	0.5437	9.81

Past – used
cell ratios

Information card for entry 2300571

[2300570](#) << [2300571](#) >> [2300572](#)

Preview

HM: P 21 21 21 #19
a=5.963Å
b=9.042Å
c=18.401Å
α=90.000°
β=90.000°
γ=90.000°

Coordinates [2300571.cif](#)
Structure factors [2300571.hkl](#)
Original paper (by DOI) [HTML](#)

Unit Cell:
a= 5.96Å, α=90.00°, V=993Å³
b= 9.05Å, β=90.00°, Orthorhombic P
c=18.39Å, γ=90.00°

Formula	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]	Space G
None	5.97	9.04	18.41	90.00	90.00	90.00	?
C12 H10 O2 S	5.96	9.01	18.38	90.00	90.00	90.00	P 21 21
C12 H10 O2 S	5.96	9.03	18.39	90.00	90.00	90.00	P 21 21
C12 H10 O2 S	5.96	9.04	18.40	90.00	90.00	90.00	P 21 21
C12 H10 O2 S	5.96	9.05	18.38	90.00	90.00	90.00	P 21 21
C12 H10 O2 S	5.97	9.04	18.39	90.00	90.00	90.00	P 21 21
C31 H11 O2 S	5.97	9.05	18.41	90.00	90.00	90.00	P 21 21
C11 H10 O2 S	5.97	9.04	18.41	90.00	90.00	90.00	P 21 21
C11 H10 O2 S	5.97	9.04	18.41	90.00	90.00	90.00	P 21 21
C11 H10 O2 S	5.96	9.04	18.40	90.00	90.00	90.00	P 21 21
C11 H10 O2 S	5.96	9.04	18.40	90.00	90.00	90.00	P 21 21
C23 H30 N2 O2	18.54	6.06	8.94	90.00	90.00	90.00	P 21 21
C10 H12 O2 S	6.01	9.03	18.15	90.00	90.00	90.00	P 21 21
C10 H7 N2 O2	8.81	18.36	5.86	90.00	90.00	90.00	P 21 21
C6 H2 N8 O4	5.72	8.81	18.60	90.00	91.55	90.00	P 1 21/d

Elements Filter:

☐ Only These Elements

Databases

Database	Count	Created
APEX DB	25	2019-11-19
CSD	21	2022-12-19
COD	3	2022-12-19
local CIF/RES	8	2025-07-09

Locate Structure

Automatic Mode

Start at: Collect Data

Stop after: Search

Run

Manual Mode

☒ Collect Data

☒ Harvest Spots

☒ Index

☐ Domains

☒ Bravais

☐ Refine

☒ Search

Unit cells:

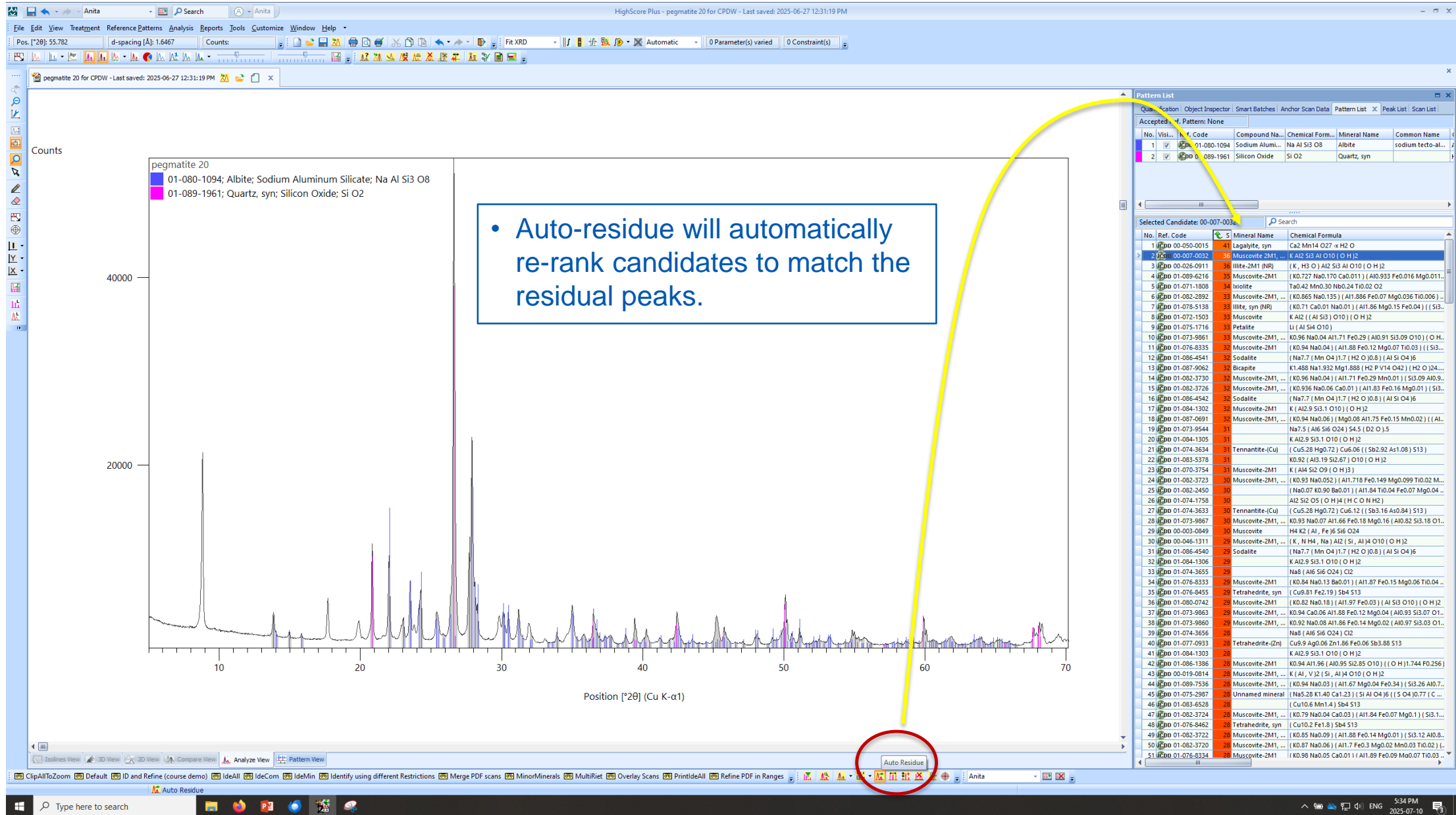
a= 5.96Å, α=90.00°, V=993Å³
b= 9.05Å, β=90.00°, Orthorhombic P
c=18.39Å, γ=90.00°

Reflections:

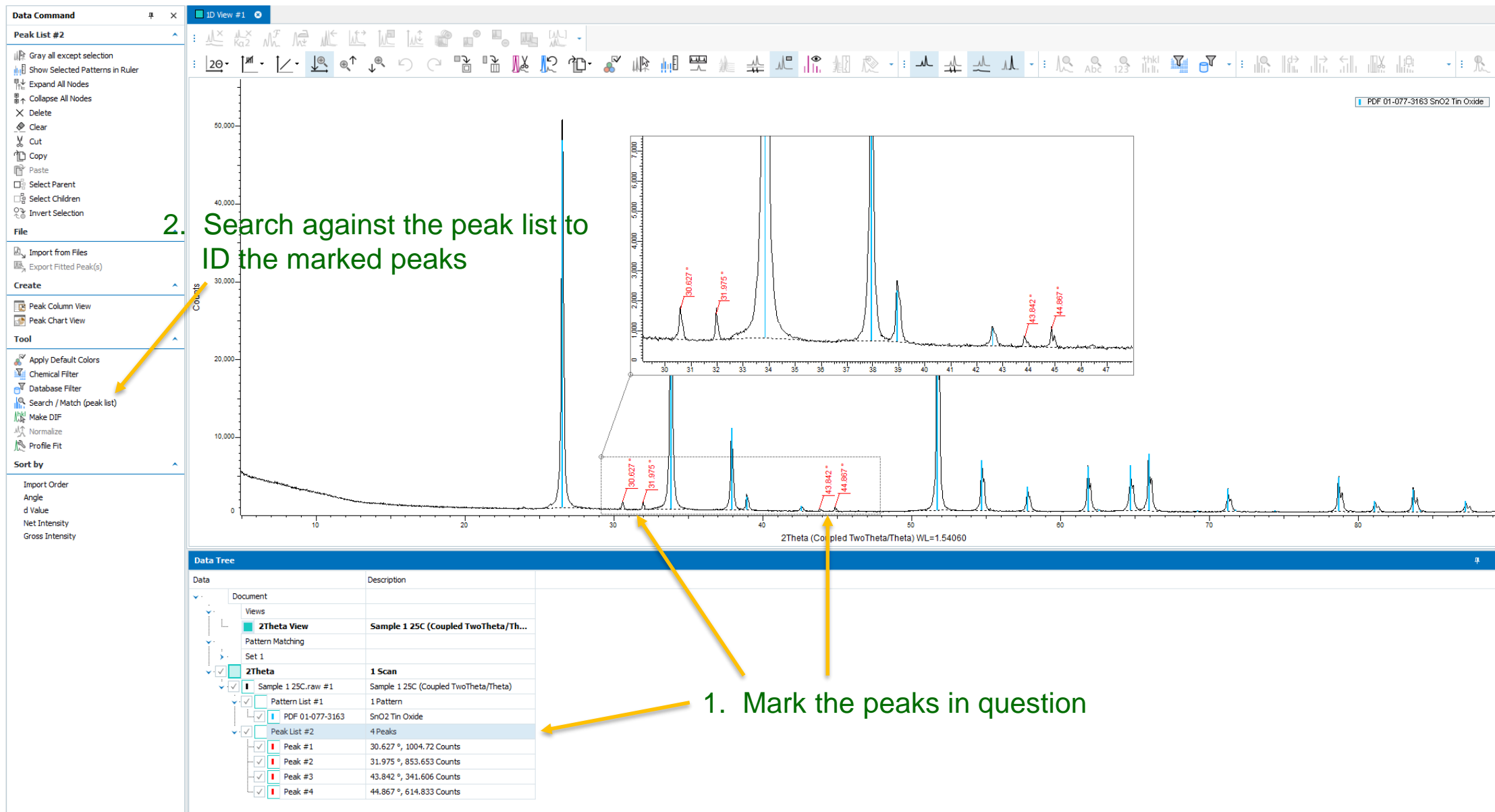
Group 0: 78 reflections

Present – search unit cell

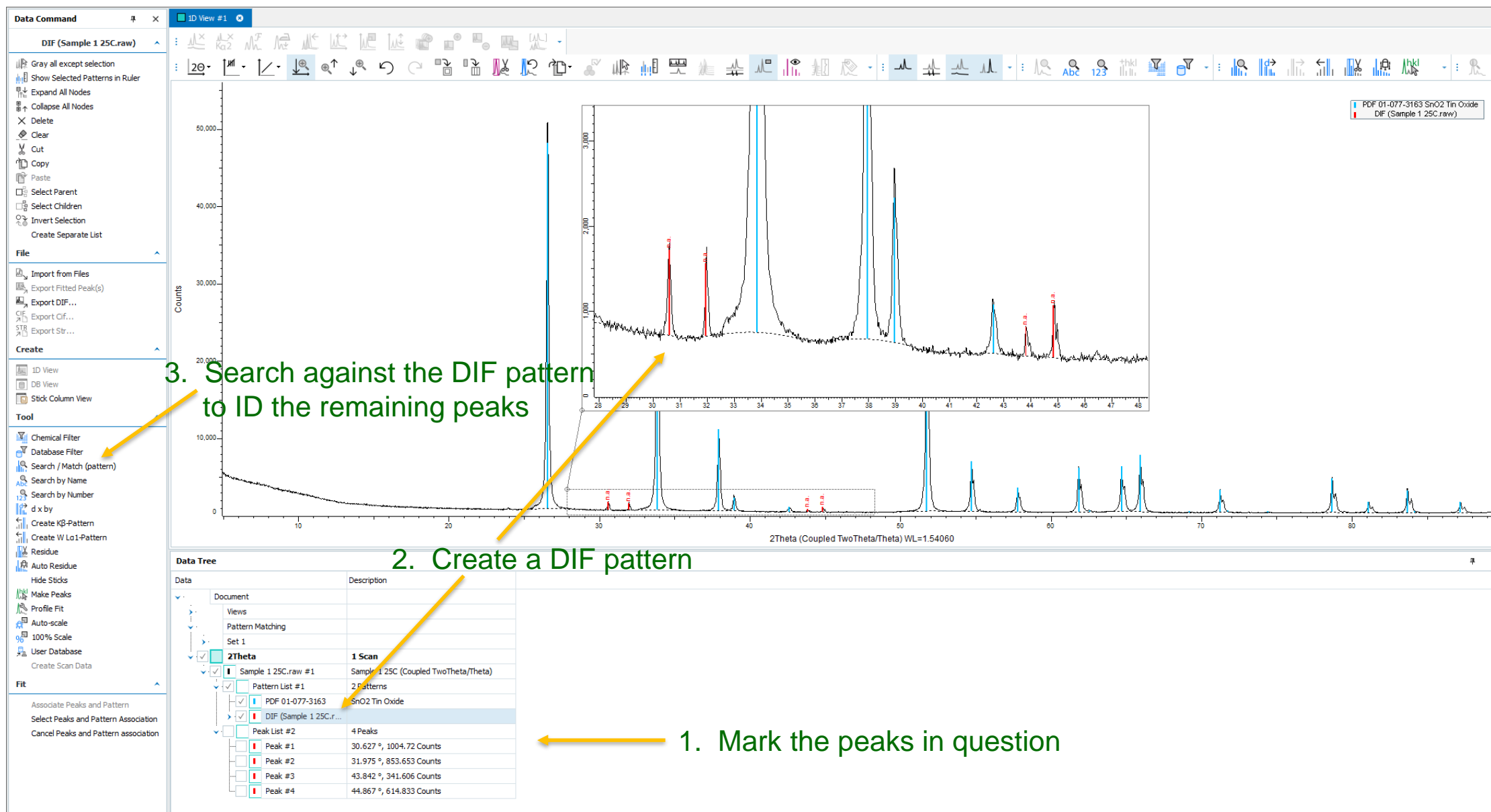
SEARCHING – RESIDUAL PEAKS



SEARCHING – SPECIFIED PEAKS



SEARCHING – SPECIFIED PEAKS, DIF PATTERN



STILL HAVING TROUBLE?

OTHER TIPS AND TRICKS



CHANGE DATABASES



- Sometimes a pattern or clue to the unidentified phase might be in a different database

The screenshot shows the 'Manage Databases' dialog box and the 'EVA Settings' window. The 'Manage Databases' dialog box has a table with the following data:

Database Name	Use	Database Location	Content Type	Properties	Convert	Writable	Database Type
ICDD PANalytical Example Database	<input type="checkbox"/>	C:\Users\User\Documents\PANalytical\HighScore\ExampleDb\	PDF-2 flat file		HighScore(Plus) V2.X de
CSD_MOFs_13k	<input type="checkbox"/>	C:\Databases\CSD_MOF_Collection\CSD_MOFs_13k.hsrdb	Converted CIF files	...			HighScore(Plus) V3.X de
COD24_HS4x	<input type="checkbox"/>	C:\Databases\COD24_HS4x.hsrdb	Converted CIF files	...			HighScore(Plus) V3.X de
CSD Database, The Cambridge ...	<input checked="" type="checkbox"/>	C:\Databases\CSD_2024_1_Database.hsrdb	CSD Database	...			HighScore(Plus) V3.X de

The 'EVA Settings' window has tabs for General, Decimal Places, Database, DB Patterns, Filters, XRF, Fit, Colors, and S. The 'Database' tab is selected, showing the 'Search / Match Databases' section with the following options:

- ☒ Enable "Deleted" PDF Patterns
- ☐ Enable "Hypothetical" PDF Patterns
- ☒ Enable "Alternate" PDF Patterns
- ☒ Auto-Search Results as Candidates
- ☐ Remember Auto-Search state

The 'Default Databases' section is also visible, with the following options:

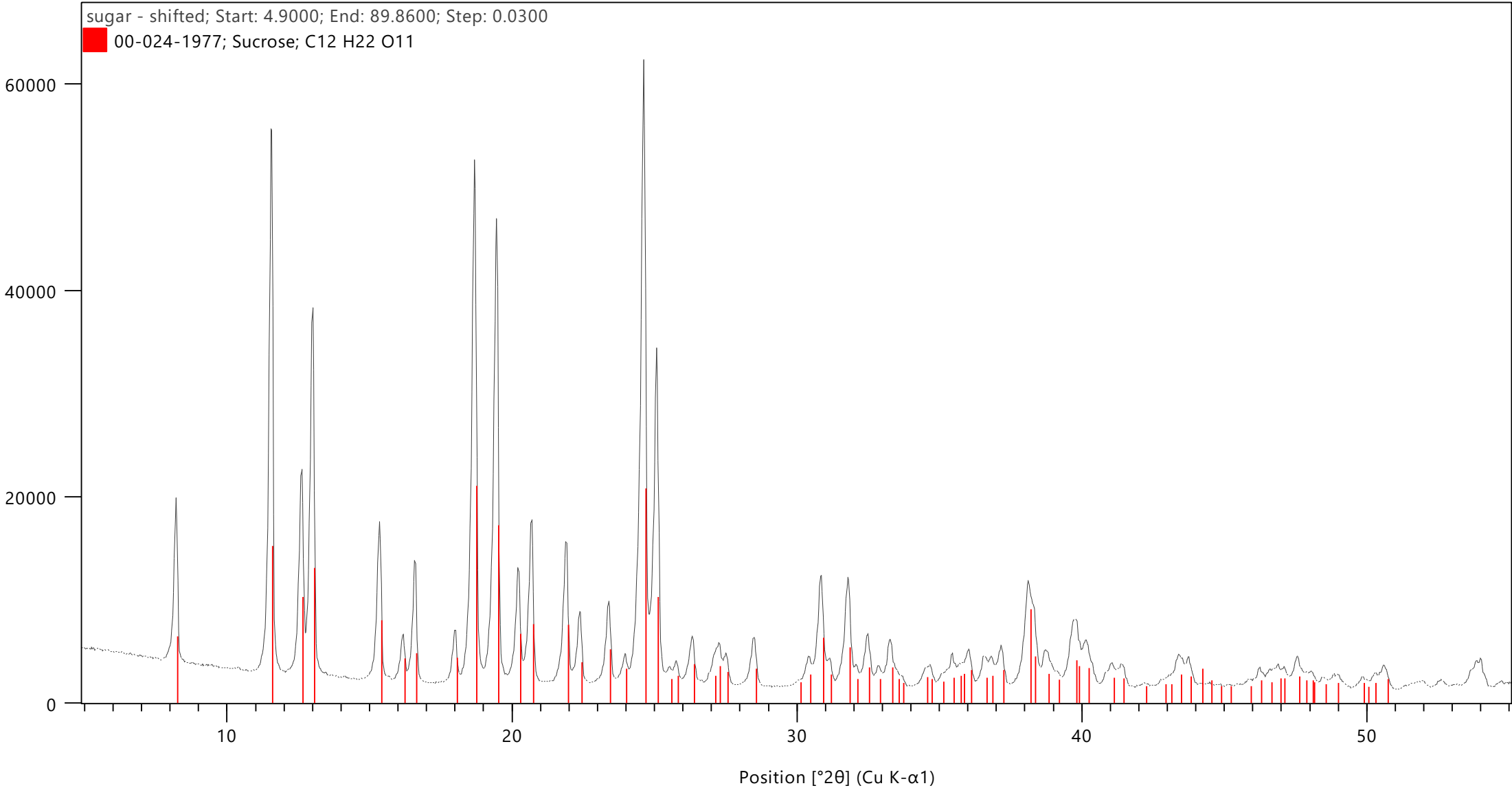
- ☐ Crystallography Open Database (rev. 269086)
- ☐ Crystallography Open Database (Minerals) (rev. 294865)
- ☒ PDF-2 2022

The 'Manage Databases' dialog box also displays a pie chart showing the distribution of patterns across databases, with a label 'CSD Da...24-544 1233377' pointing to a segment. A bar chart titled 'Disk Usage' shows the disk usage for 'Local' and 'C:\' drives, with a scale from 0 to 240 Gigabyte.

PEAK SHIFTS (see next slide)



Counts



PEAK SHIFTS (see previous slide)



Search & Match - [Untitled]

Restrictions Parameters Automatic

Data source: Peak Data

Scoring scheme: ☒ Single phase ☐ Multi phase

☐ Auto residue ☐ Demote unmatched strong

☐ Match intensity ☐ Allow pattern shift

Known Two Theta shift [°2θ]: 0.000

Search

Candidates:

No.	Ref. Code	S	Compound Name	Chemical Formula
1	ICDD 00-062-1061	50	Diaquo 1,4-bis(imidazol-1-...	Co (H2 O)2 (N2 C3 H3) C6 H4
2	ICDD 01-082-5942	48	Curium Oxide Sulfide	Cm10 O S14
3	ICDD 01-076-9357	48	Calcium Mercury	Ca9 Hg54 (Ca1.92 Hg0.08)
4	ICDD 01-085-1407	48	Sodium Calcium Iron Vana...	(Na0.7 Ca0.3) (V7.6 Fe0.4) O2
5	ICDD 01-086-6420	48	Mercury Ytterbium	Yb11 Hg54
6	ICDD 01-070-5161	48	Thallium Tin Fluoride	(Tl5 F3) (Sn F6)
7	ICDD 01-081-6277	47	Mercury Ytterbium	Yb14 Hg51
8	ICDD 01-087-2745	46	Barium Zinc Iron Oxide	Ba6 Zn5 Fe42 O74
9	ICDD 01-071-1187	46	Bromine Antimony Fluoride	(Br F2) (Sb F6)
10	ICDD 00-053-0122	46	Cadmium Chloride Thiourea	C2 H8 Cd Cl2 N4 S2
11	ICDD 00-069-1141	46	Lithium glycolate monohy...	C2 H3 Li O3 · H2 O
12	ICDD 00-070-1030	45	4,5-Dihydroxy-1-phenethyl...	C11 H14 N2 O3
13	ICDD 01-085-9599	45	Cesium Hydrogen Sulfate ...	Cs3 (H S O4)2 (H2 P O4)
14	ICDD 01-085-3221	45	Cesium Hydrogen Sulfate ...	Cs3 (H S O4)2 (H2 P O4)
15	ICDD 01-082-4378	45	Barium Mercury Zinc	Ba Hg3.40 Zn0.60
16	ICDD 01-087-1598	44	Potassium Hydrogen Germ...	K3 H (Ge7 O16) (H2 O)3.137
17	ICDD 00-041-1368	44	Calcium Manganese Silicate	Ca Mn14 +3 Si O24
18	ICDD 00-070-0293	43	Silver Zinc Iron Molybdate	Ag Zn3 Fe (Mo O4)5
19	ICDD 03-065-1166	43	Erbium Germanium Nickel	Er Ge2 Ni3
20	ICDD 01-076-9694	43	Tin Gallium Indium Titaniu...	Ga3.18 In0.82 Sn2.7 Ti0.3 O12
21	ICDD 01-073-0898	43	Tin Oxide Hydroxide Phos...	Sn3 O (OH) (PO4)
22	ICDD 01-076-1846	43	Calcium Silicate	Ca (Si O3)
23	ICDD 00-069-0886	43	Naphthalene	C10 H8
24	ICDD 01-083-0495	43	Potassium Zinc Deuteride	K3 (Zn D5)

Search & Match - [Untitled]

Restrictions Parameters Automatic

Data source: Peak Data

Scoring scheme: ☒ Single phase ☐ Multi phase

☐ Auto residue ☐ Demote unmatched strong

☐ Match intensity ☐ Allow pattern shift

Known Two Theta shift [°2θ]: 0.000

Search

Candidates:

sucrose

no candidates found

Search & Match - [Untitled]

Restrictions Parameters Automatic

Data source: Peak Data

Scoring scheme: ☒ Single phase ☐ Multi phase

☐ Auto residue ☐ Demote unmatched strong

☒ Match intensity ☒ Allow pattern shift

Known Two Theta shift [°2θ]: 0.000

Search

Candidates:

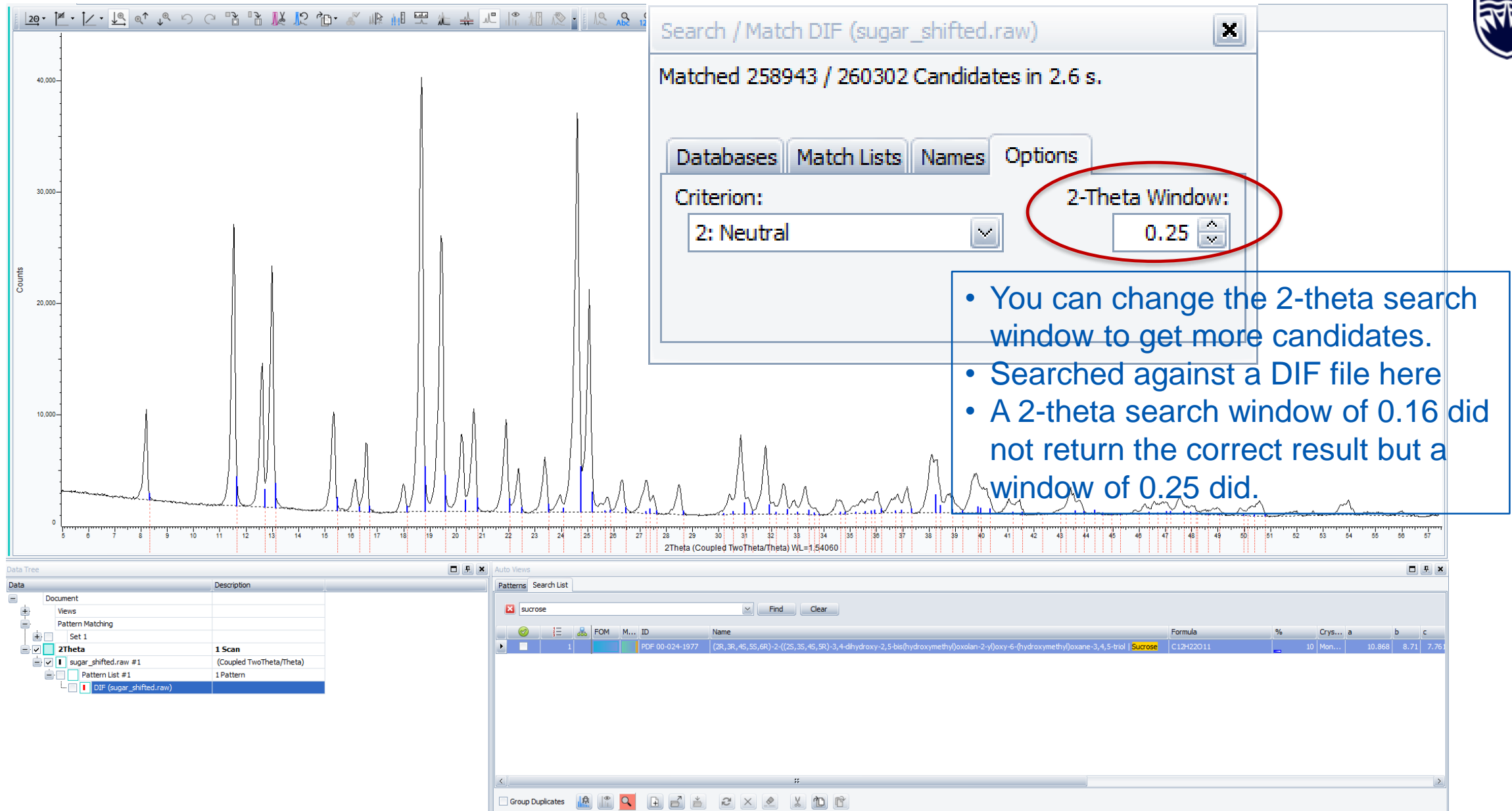
sucrose

No.	Ref. Code	S	Compound Name	Chemical Formula
47	ICDD 00-024-1917	40	Sucrose	C12 H22 O11

selected; redo search

Allowing a pattern shift brought sucrose into the candidate list

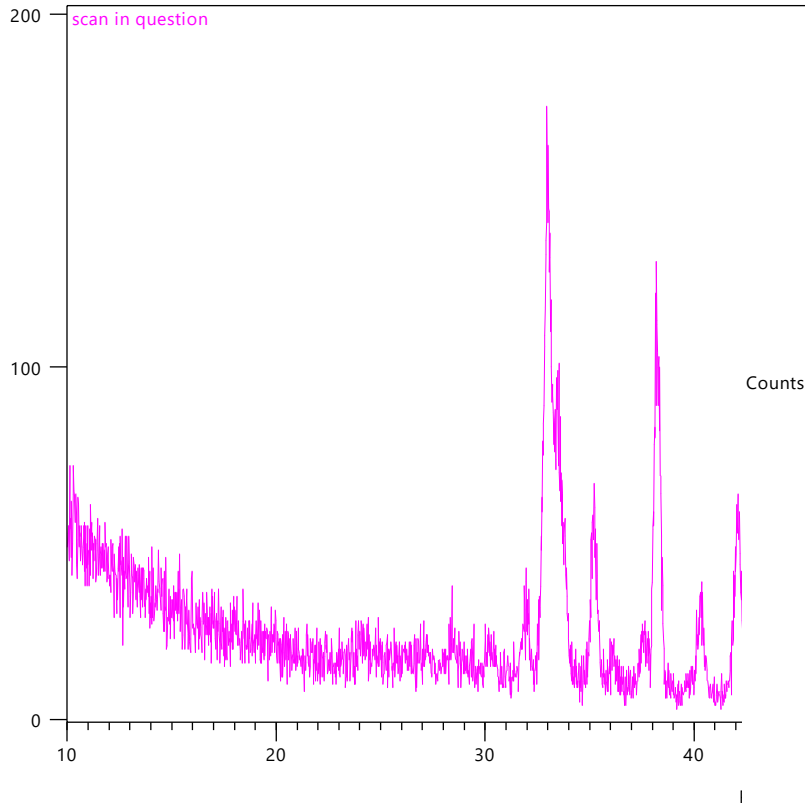
PEAK SHIFTS (see previous slide)



PEAK SHIFTS – SOMETIMES YOU HAVE TO SQUINT, part 1

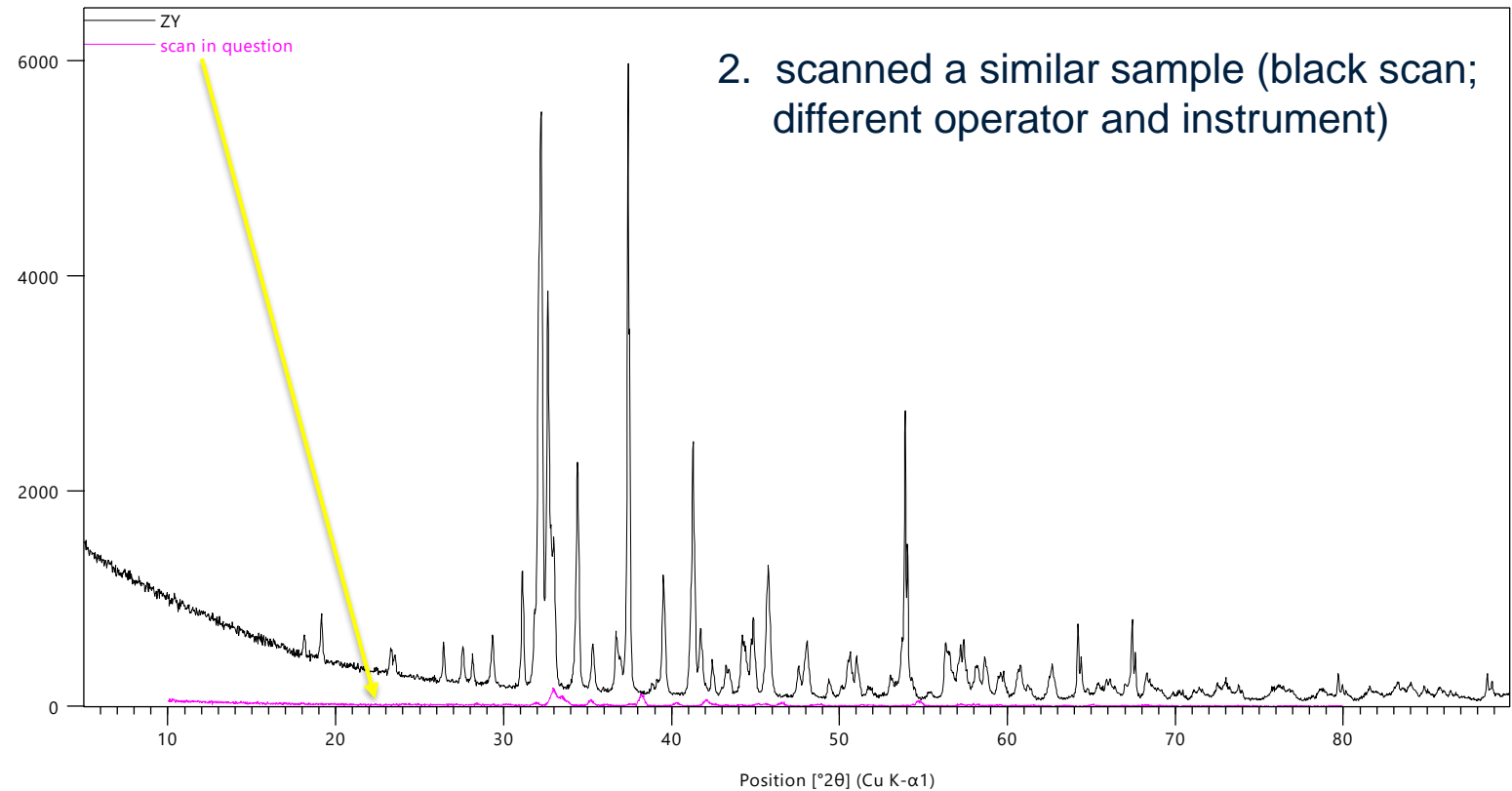


Counts



1. student run sample; had trouble identifying phase(s)

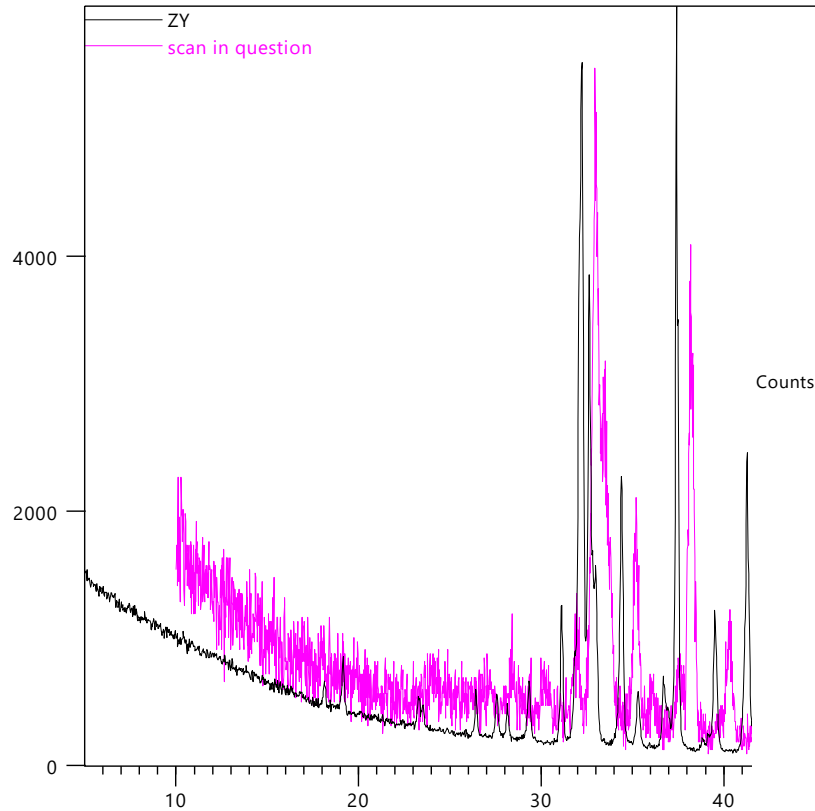
Counts



PEAK SHIFTS – SOMETIMES YOU HAVE TO SQUINT, part 2

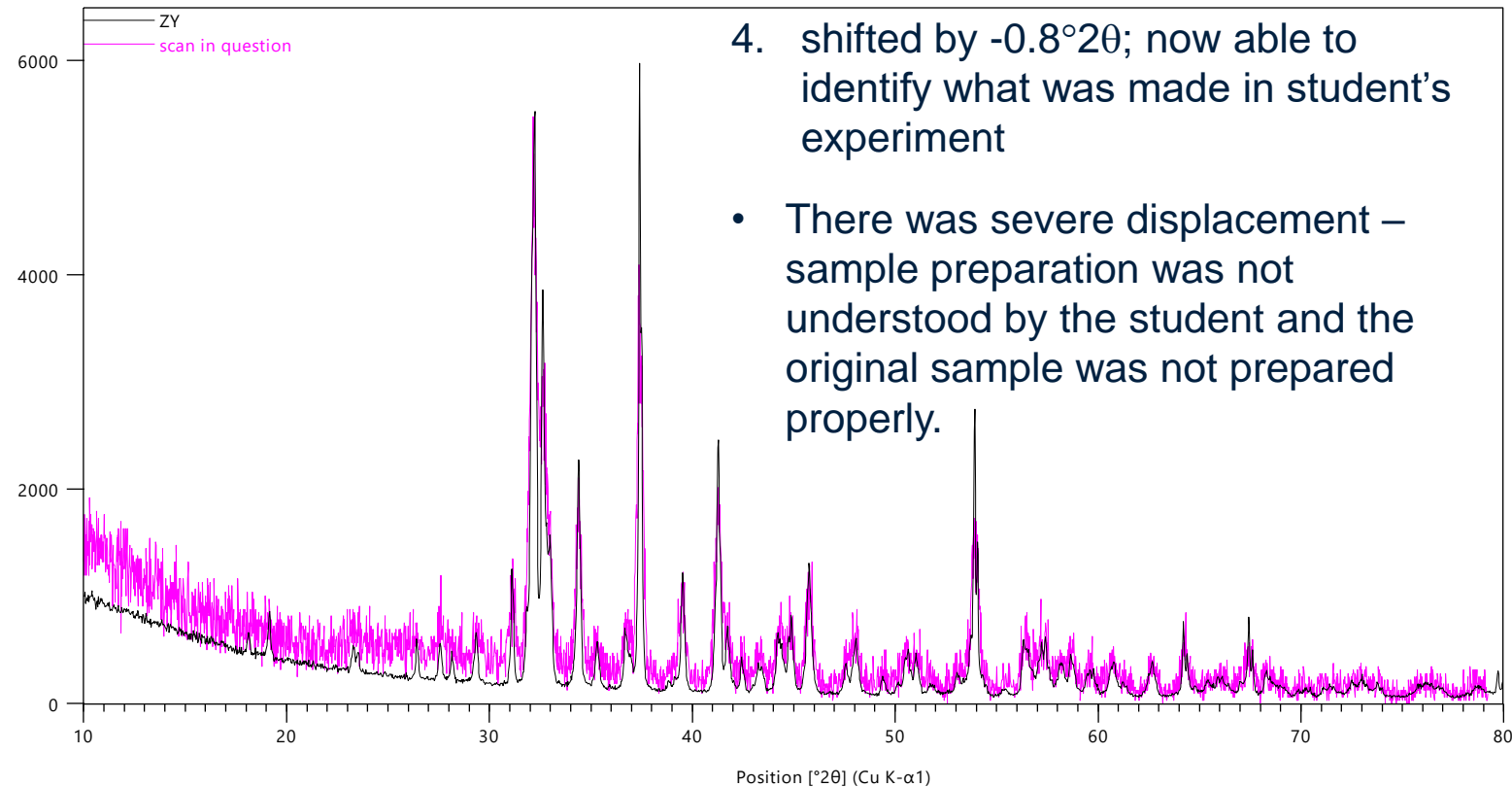


Counts



3. scaled up; if you squint you can sort of see similarities

Counts



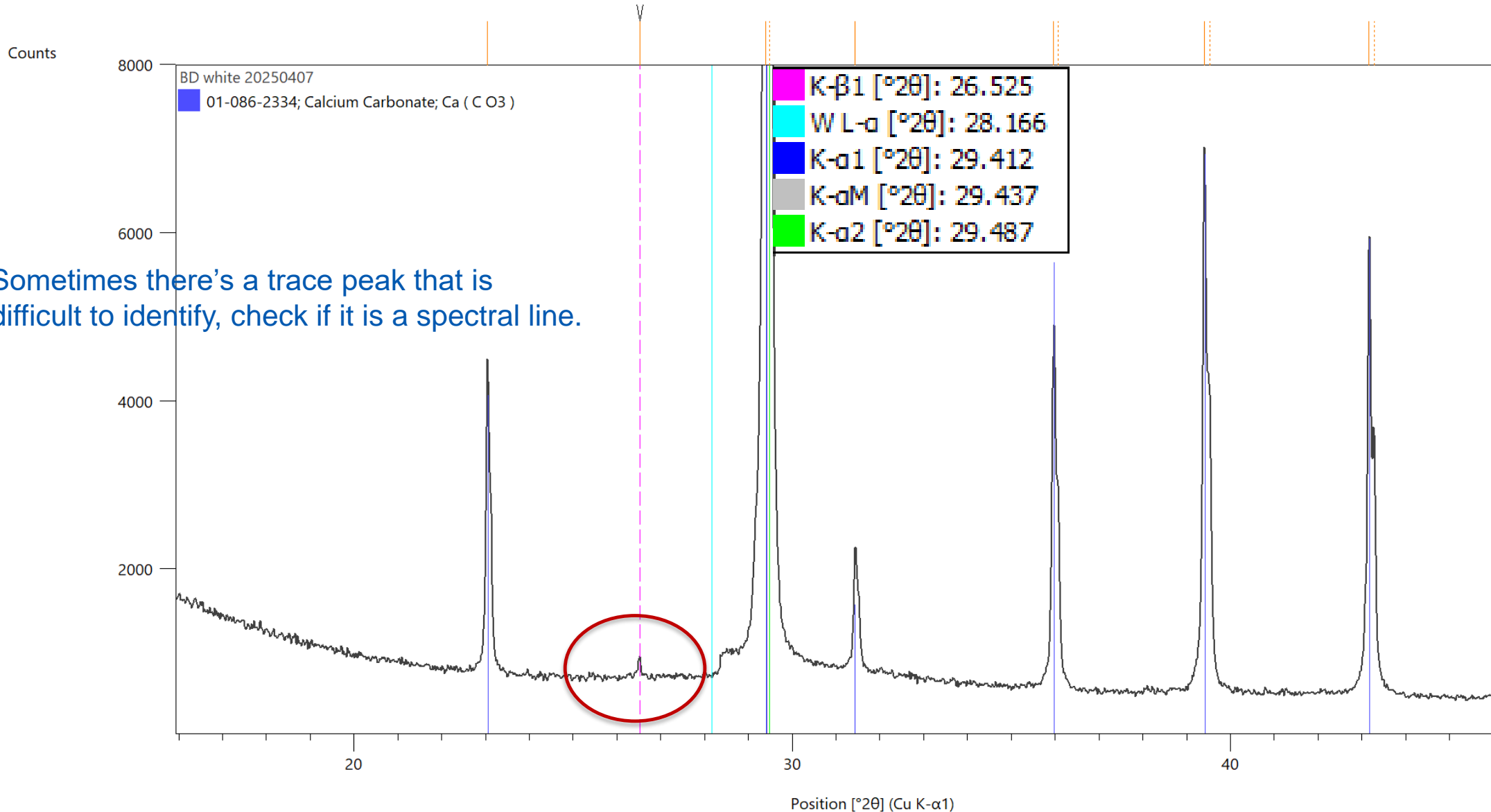
4. shifted by $-0.8^\circ 2\theta$; now able to identify what was made in student's experiment

- There was severe displacement – sample preparation was not understood by the student and the original sample was not prepared properly.

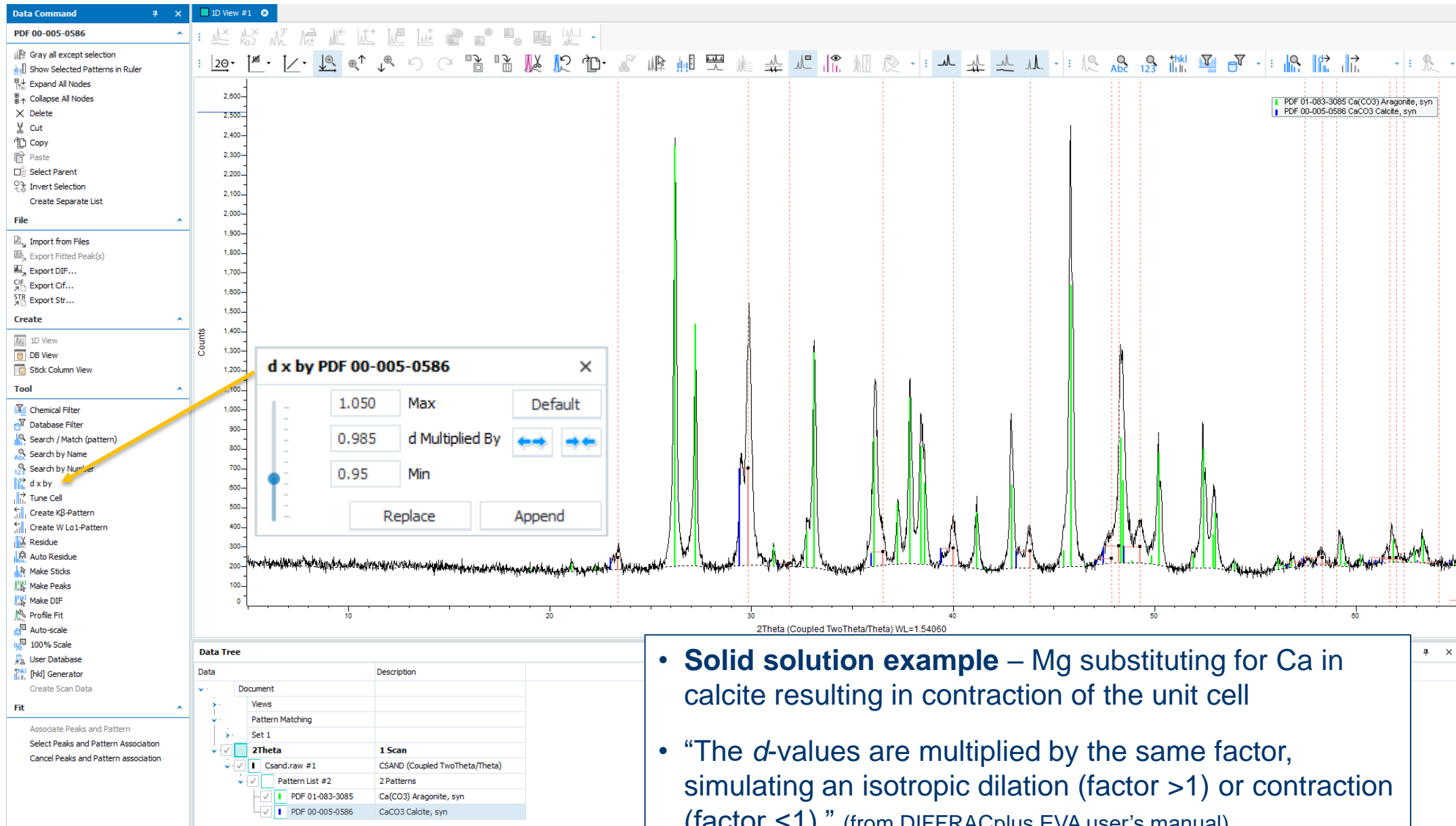
MINOR PEAKS – Check for $K\beta$ or $WL\alpha_1$ lines



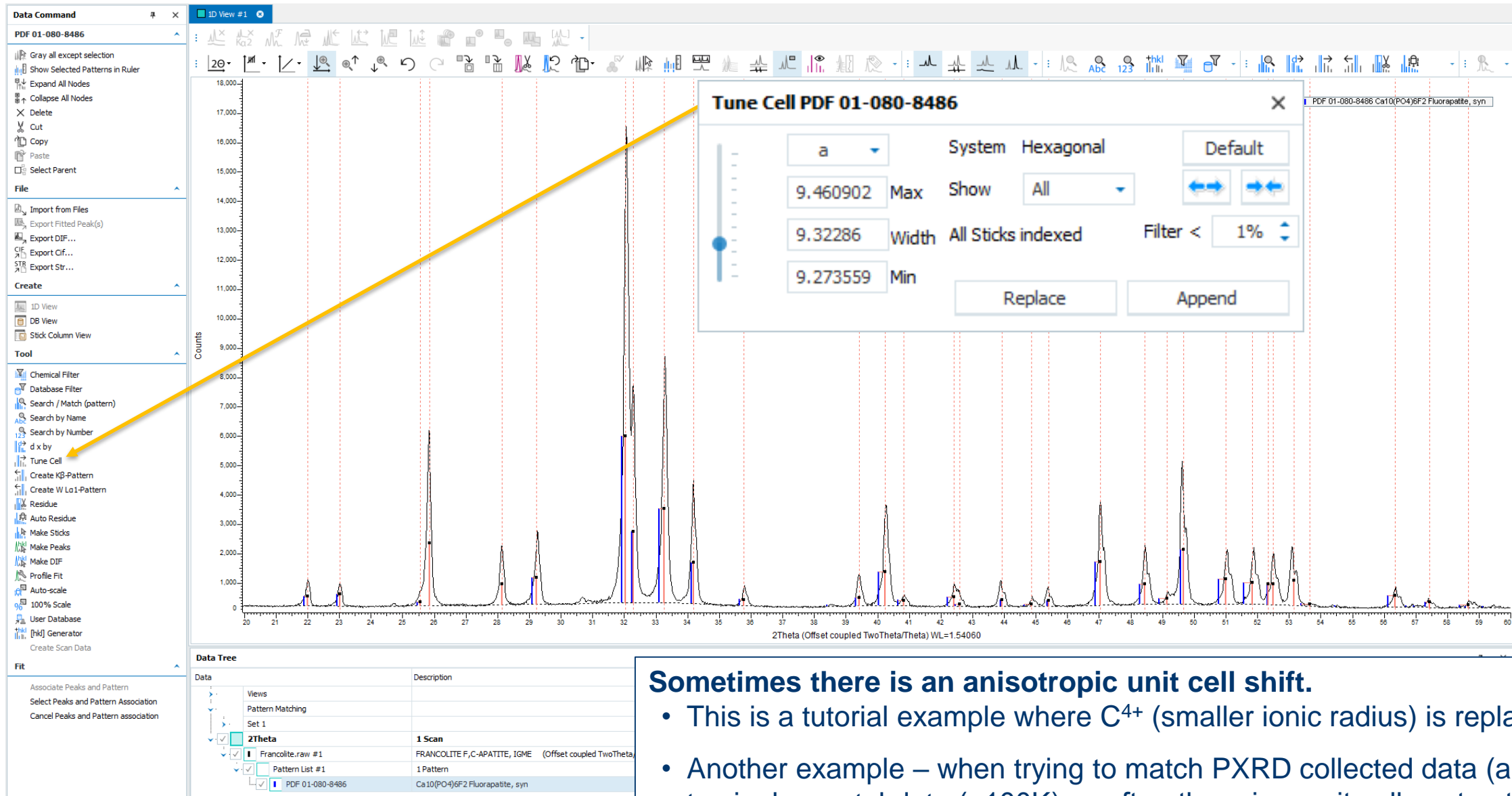
- Sometimes there's a trace peak that is difficult to identify, check if it is a spectral line.



SOLID SOLUTION – d x by (d multiplied by)



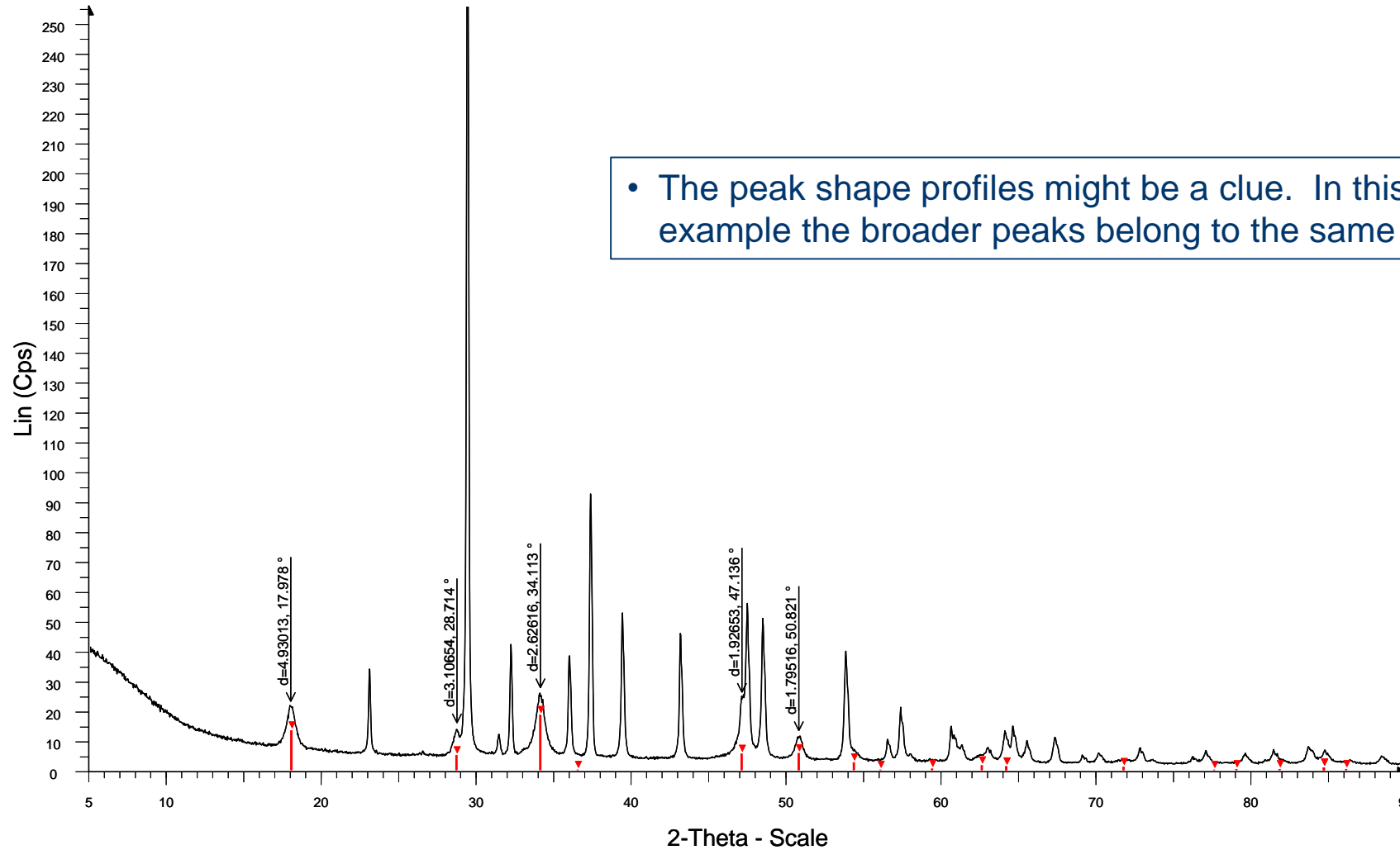
TUNE CELL – check for anisotropic unit cell shift



Sometimes there is an anisotropic unit cell shift.

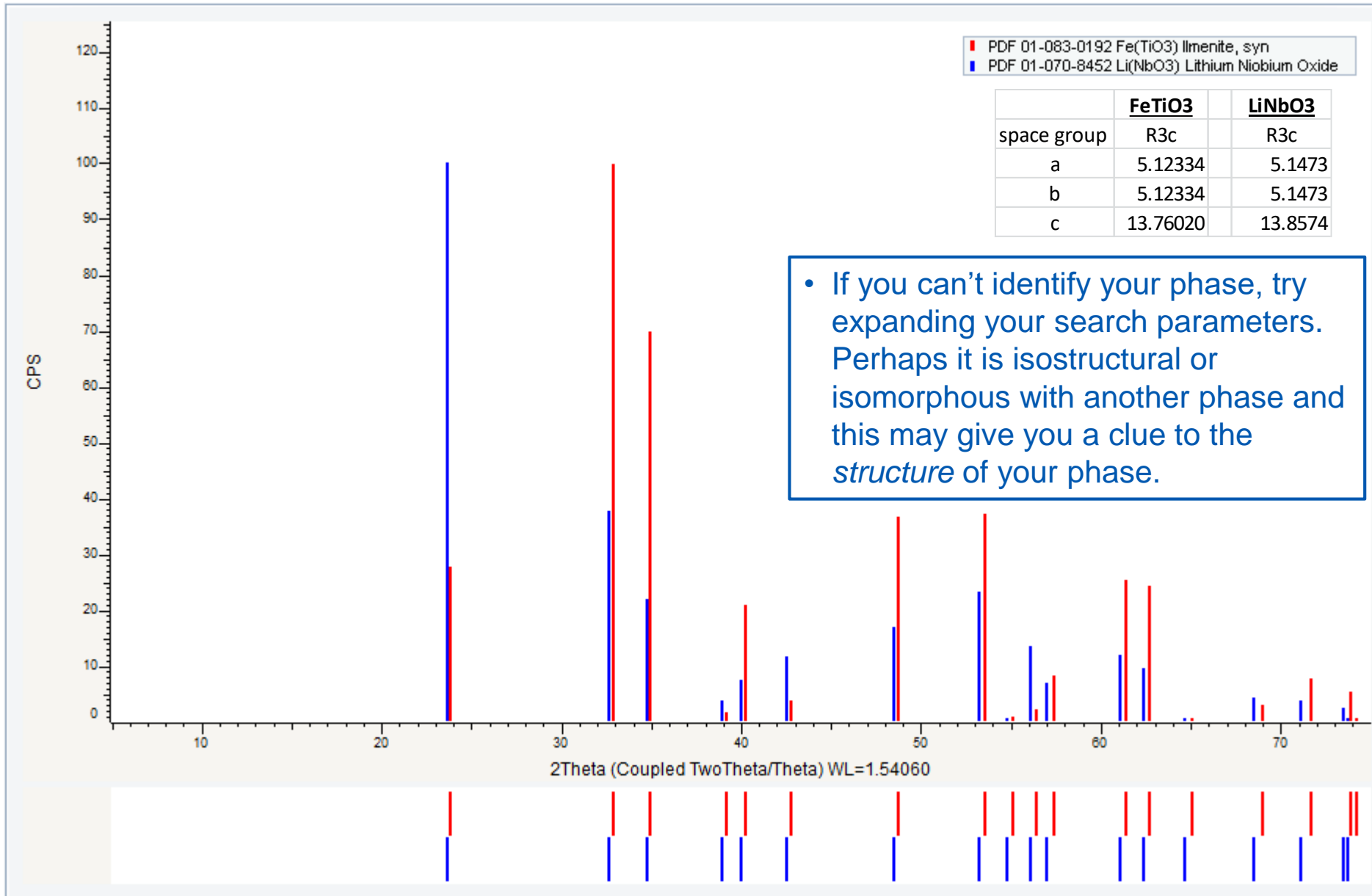
- This is a tutorial example where C^{4+} (smaller ionic radius) is replacing P^{5+}
- Another example – when trying to match PXRD collected data (ambient T) to single crystal data (~100K) ← often there is a unit cell contraction

SIMILAR PEAK SHAPES

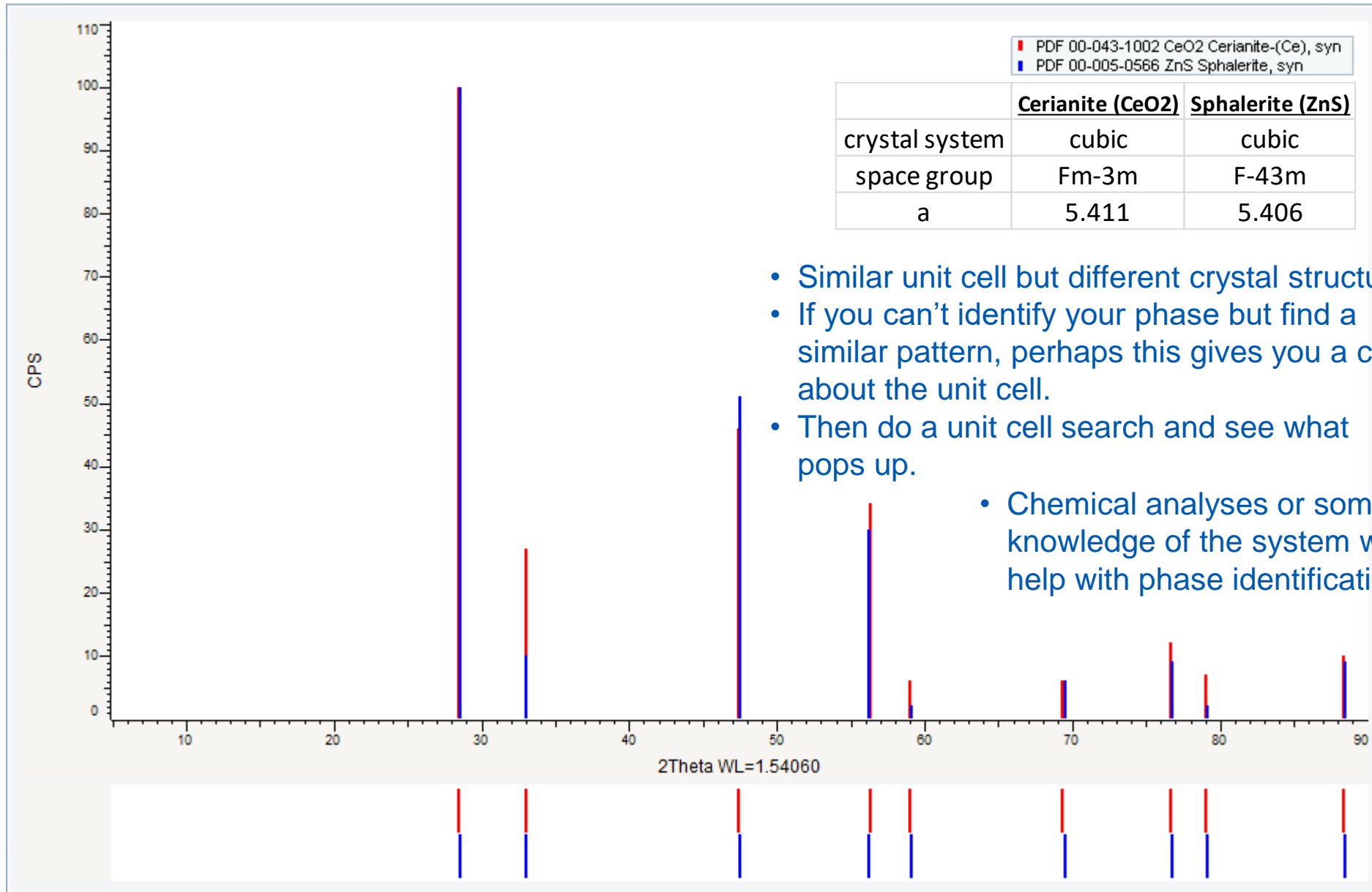


Start: 5.000 ° - End: 90.005 ° - Step: 0.030 ° - Step time: 109.2 s - Anode: Cu - WL1: 1.5406 - WL2: 1.54439 - kA2 Ratio: 0.5 - Generator kV: 40 kV - Generator mA: 40 mA - Creation: 21-Apr-2016 12:40
00-044-1481 (*) - Portlandite, syn - Ca(OH)2 - WL: 1.5406 - Hexagonal - a 3.58990 - b 3.58990 - c 4.91600 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P-3m1 (164) - 1 - 54.8665 - I/Ic PD

ISOSTRUCTURAL



SIMILAR PATTERN (but not isostructural)



PHYSICAL PROPERTIES

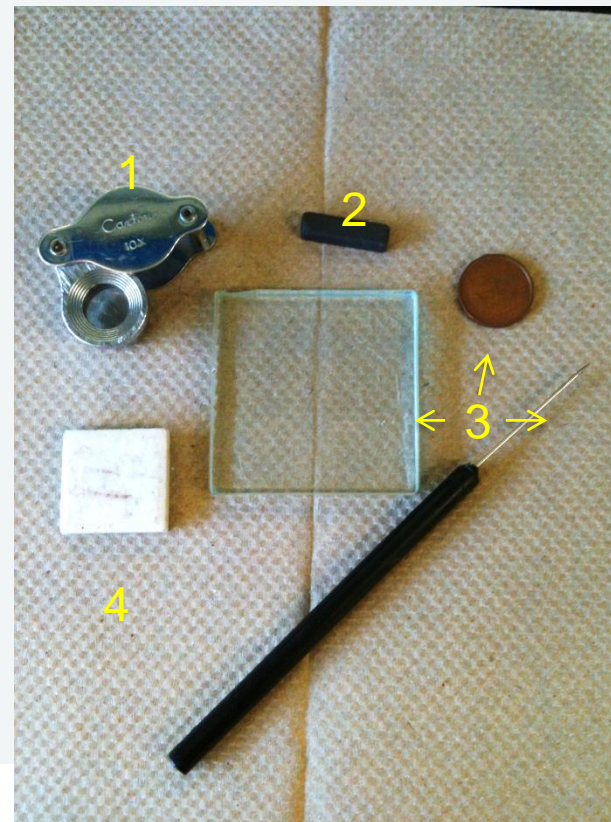


- Physical properties can be useful to help with identification

- Geologists use a basic mineral ID kit in the field

1. Visual properties – hand lens
2. Magnetism – easily tested
3. Hardness – Moh's scale
 - steel > glass > copper
4. Streak
5. Carbonate – dilute acid (not shown)

- Example



MINDSET – TIPS



Don't miss the forest for the trees

- Don't focus on specific elements too quickly
- I've had more than one student who has wasted hours trying to identify a phase when it was something simple like NaCl

Be open to possibilities

- Perhaps it is a contaminant from sample preparation or an unclean reaction vessel
- If it's a minor phase – is there a way to concentrate it or use selective dissolution?
- Maybe it's something new

Ask for help

- Staff at X-ray diffraction facilities
- Researchers working on the same or similar system
- Someone with fresh eyes

WHEN YOU'VE DONE ALL THAT YOU CAN



Sometimes no matter what you do:

- you are unable to identify a peak
 - it's okay to mark the peak as unidentified
- or several peaks remain unidentified
 - perhaps the phase is not in the database
- or sometimes two or more 'possibilities' exist (for example if you don't have much information about the system and more than one pattern may fit)
 - when I've done all that I can, I'll just give a list of reasonable possibilities to the client

Generally, it's good practice to not conclude beyond what the evidence is giving you.

SUMMARY



- Phase identification is like matching fingerprints – comparing your scan to a database of known phases
- The search/match process is an interactive process between the user and software + database
- XRD is sensitive to structural differences; if you have qualitative data, you should take care in drawing conclusions about the chemistry of the phases
 - chemical analyses and sample information help with phase identification
- User knowledge and experience is valuable to the process

THANK YOU

- UBC Chemistry Department
- Dr. Brian Patrick and Fahd Murad
- Dr. Beatriz Diaz Moreno
- And to you the audience 😊





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