

Identification and quantification of mineral phases commonly found in building materials using the software HighScore available at the Department of Mineralogy and Petrology (UGR)

Introduction

Generally, building materials contain several different minerals. More than 4000 minerals are officially recognized. Fortunately, the number of minerals that we normally find in materials such as earth, stone, bricks, mortars, alteration products, etc. is more limited. The following table shows the most common minerals in these materials and their most intense (identifying) d_{hkl} reflection. Mineral names are included in English because XRD-analysis programs use English names.

Mineral Phases

- **Earth**

(quartz 3.34 Å, phyllosilicates ~4.50 Å; calcite 3.03 Å, dolomite 2.88 Å, gypsum 7.59 Å; feldspars ~3.20 Å, hematite, goethite, rutile)

- **Clay fraction (<2µm)**

(smectite (montmorillonite, beidellite, nontronite, saponite ~13-15Å), mica/illite 10.0 Å, paragonite 9.6 Å, kaolinite 7.15 Å, chlorite ~7.15 Å, quartz, calcite)

- **Bricks**

(quartz, mica/illite, feldspars (orthoclase, plagioclase, etc.), calcite, dolomite, hematite, mullite, gehlenite, diopside, wollastonite)

- **Mortars/Plasters**

(quartz, lime (CaO), calcite, vaterite, aragonite, dolomite, portlandite, periclase, brucite, hydromagnesite, calcium silicate hydrate (cemento Portland), gypsum, bassanite, anhydrite, phyllosilicates (mica/illite))

- **Stone**

(quartz, calcite, dolomite, gypsum, feldspars, pyroxene, amphibole, olivine, phyllosilicates, phyllosilicates (mica), magnetite, hematite, goethite, pyrite, rutile)

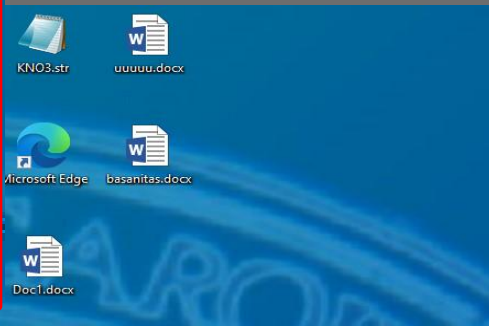
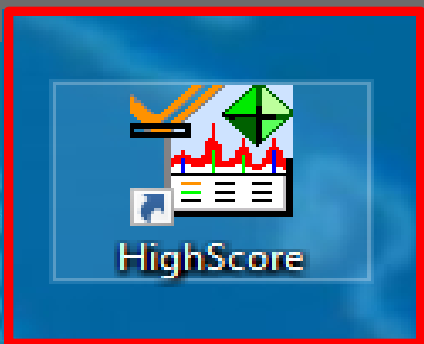
- **Alteration Products**

(bassanite, calcite, hexahydrate, anhydrite, gypsum, epsomite, halite, kaliginite, mirabilite, natron, niter, thenardite, trona, weddellite, whewellite)

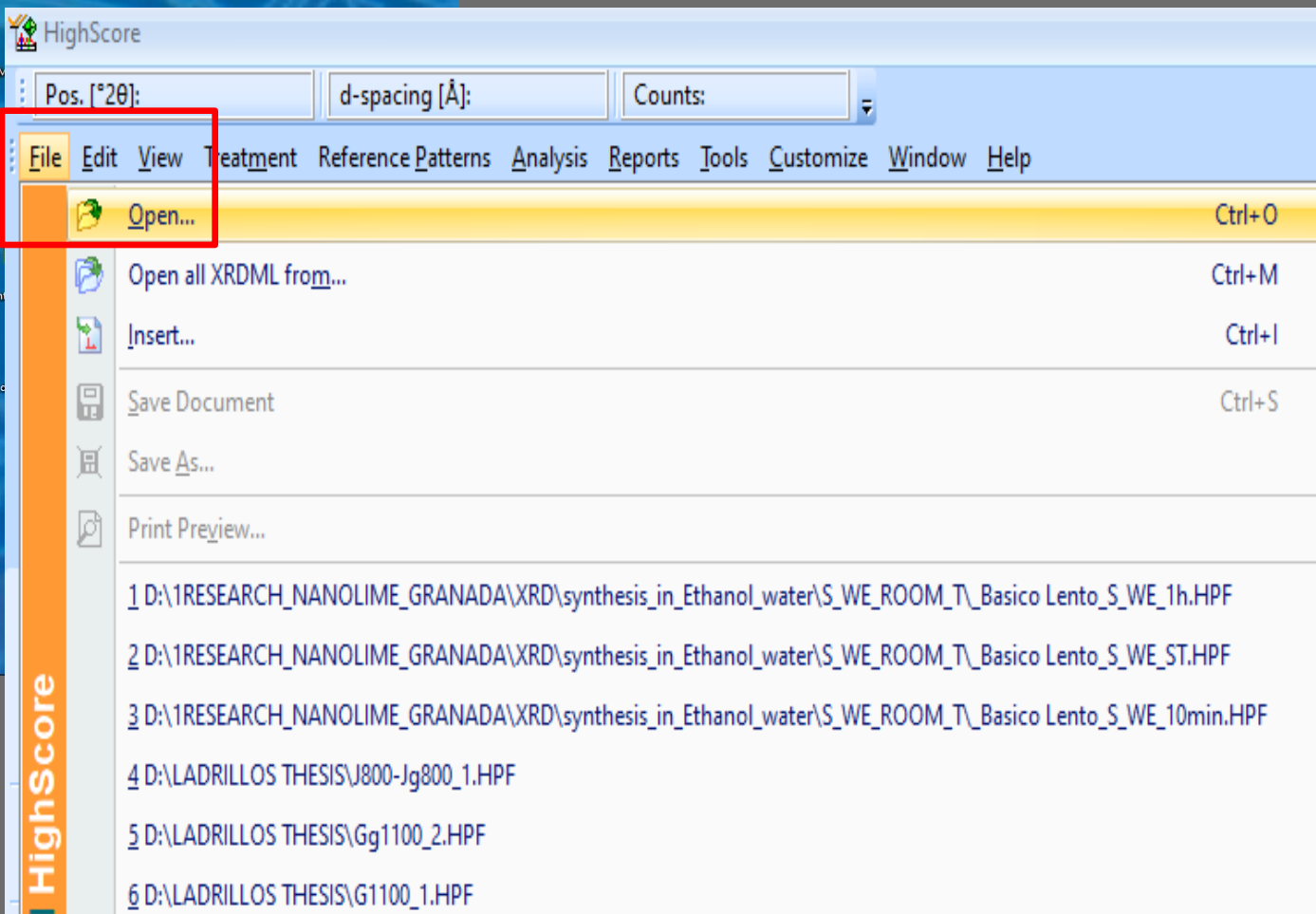
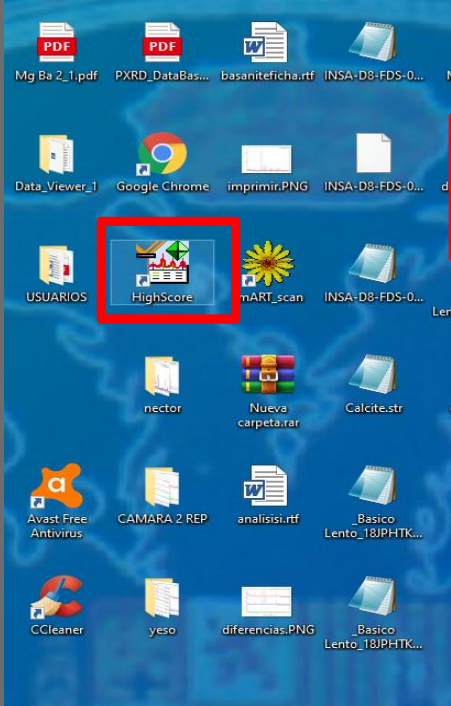
**Additional information
about minerals:**

<http://www.webmineral.com> y <https://www.mindat.org>

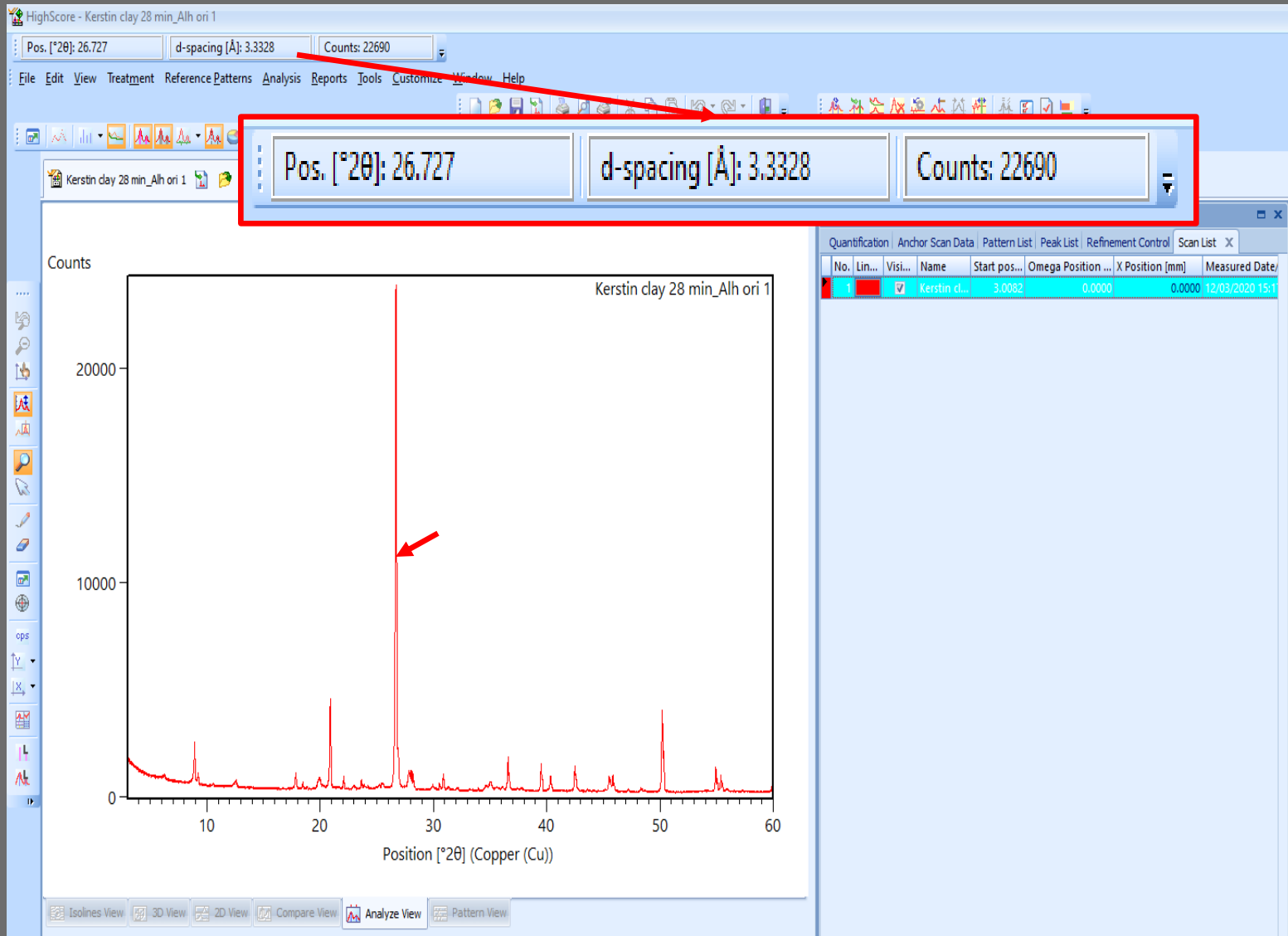
Use of the software «HighScore»



1. We open the HighScore software and a diffractogram using «File» and «Open».



2. We verify that the diffractogram is not shifted, preferably using the most intense peak of quartz at 3.34 Å or another mayor phase (due to small variations in the height of the sample during analysis the peaks may be shifted). Place the mouse above the peak and check the value.



3. If the value does not coincide with 3.34 \AA , select «Scan List» and «Shifts» and enter a negative value to shift the diffractogram to the left or positive value to shift it to the right so that the value of the quartz peak of our sample is equal to 3.34 \AA .

The screenshot displays the HighScore software interface. The main window shows a diffractogram plot with a peak at 3.0082 \AA . The Scan List table is highlighted with a red box, showing the following data:

No.	Lin...	Visi...	Name	Start pos...	Omega Position ...	X Position [mm]	Measured Date
1		<input checked="" type="checkbox"/>	Kerstin cl...	3.0082	0.0000	0.0000	12/03/2020 15:1

The Shifts panel is also highlighted with a red box, showing the following settings:

Parameter	Value
Incident Beam Mask Position...	109
Shifts	
Shift Position by [$^{\circ}2\theta$]	-2
Set Minimum [cts]	170.3551
Set Maximum [cts]	24348.13
Add [cts]	0
Scale Intensity by	1
Add Poisson Noise [ESD]	1

Red arrows indicate the relationship between the Scan List table and the Shifts panel, showing how the shift value is applied to the scan data.

4. For the analysis of our diffractogram we have to select the peaks using «Treatment» and «Search Peaks» and «Accept». If the values of «Minimum significance» and «Minimum tip width Gonio» are too high, only the highest intensity peaks are marked (if we decrease the values the program will also select the smaller peaks).

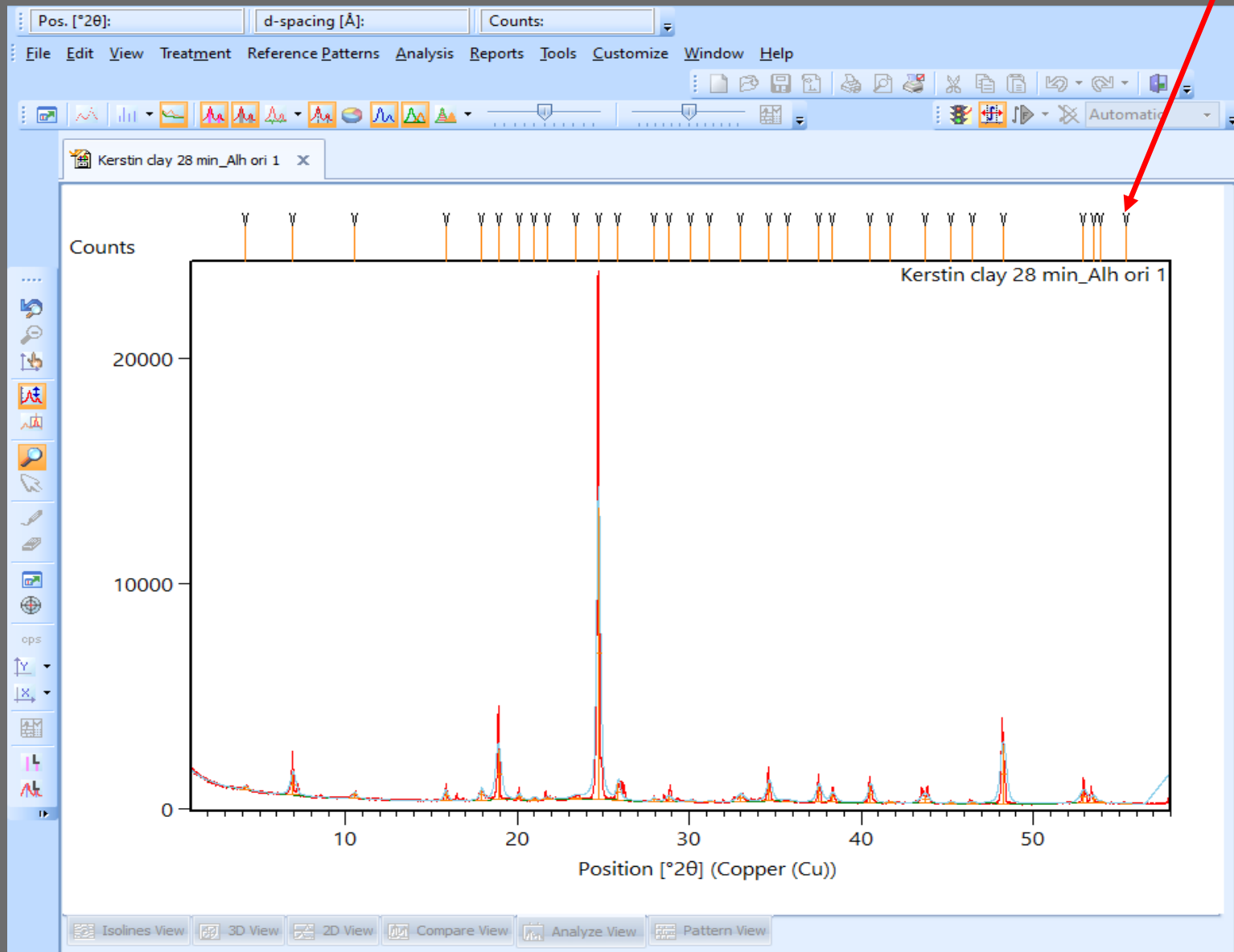
The image shows the HighScore software interface for X-ray diffraction analysis. The main window displays a diffractogram with the x-axis labeled "Position [°2θ] (Copper (Cu))" ranging from 0 to 50, and the y-axis labeled "Counts" ranging from 0 to 20,000. A prominent peak is visible at approximately 25° 2θ. The software title is "HighScore - Kerstin clay 28 min_Alh ori 1". The menu bar includes "File", "Edit", "View", "Treatment", "Reference Patterns", "Analysis", "Reports", "Tools", "Customize", "Window", and "Help". The "Treatment" menu is open, showing options like "Determine Background...", "Search Peaks...", and "Strip K-Alpha2...". The "Search Peaks..." option is highlighted with a red box. A red arrow points from the text above to the "Search Peaks..." menu item and then to the "Search Peaks" dialog box. The dialog box, titled "Search Peaks - [Untitled]", contains the following settings:

- Minimum significance: 2.00
- Minimum tip width Gonio: 0.20
- Maximum tip width Gonio: 1.00
- Peak base width Gonio: 2.00
- Method: Minimum 2nd derivative
- Trial: Search 1

Buttons for "Search Peaks", "Accept", "Close", and "More >>" are visible in the dialog box. A "Scan List" window is also open, showing a table with the following data:

No.	Lin...	Visi...	Name	Start pos...	Omega Position ...	X Position [mm]	Measured Date/
1		<input checked="" type="checkbox"/>	Kerstin cl...	1.0782	0.0000	0.0000	12/03/2020 15:1

Example of a diffractogram with selected peaks (marked by a line and a «V»).



5. For the «automatic» analysis of our diffractogram by comparison with reference data files included in the software database, we choose «Analysis» and «Execute Search & Match» and click «Search» and "OK". The program gives us a list of possible candidates, indicating the match (Score, orange column) of the peaks of each candidate with our sample.

The screenshot shows the HighScore software interface. The 'Analysis' menu is open, and the 'Execute Search & Match...' option is highlighted. A dialog box titled 'Search & Match - [Untitled]' is open, with the 'Search' button highlighted. Below the dialog, a 'Candidates' table is displayed, showing a list of mineral candidates with their scores highlighted in orange.

No.	Ref. Code	Mineral Name	Score	Compound Na...	Chemical Formula	Scal
1	ICDD 01-089-1961	Quartz low, dau...	44	Silicon Oxide	Si O2	0.
2	ICDD 01-070-2517	Quartz low - the...	43	Silicon Oxide	Si O2	0.
3	ICDD 01-070-3755	Quartz	41	Silicon Oxide	Si O2	0.
4	ICDD 01-089-8935	Quartz SGA	41	Silicon Oxide	Si O2	0.
5	ICDD 01-083-0539	Quartz	41	Silicon Oxide	Si O2	0.
6	ICDD 01-078-1252	Quartz low, syn	41	Silicon Oxide	Si O2	0.
7	ICDD 01-089-8936	Quartz SGA	41	Silicon Oxide	Si O2	0.
8	ICDD 01-077-1060		40	Silicon Oxide	Si O2	0.
9	ICDD 01-086-1560	Quartz	39	Silicon Oxide	Si O2	0.
10	ICDD 01-085-0457	Quartz low	39	Silicon Oxide	Si O2	0.
11	ICDD 01-083-2465	Quartz low, syn	39	Silicon Oxide	Si O2	0.

6. Once the reference mineral with the positions and intensities of peaks that fit best those of our sample (generally the phase with the highest «Score», **orange column**) has been selected, we drag it to the list of «Accepted Ref. Pattern Name». The program indicates the peaks of our sample that still need to be assigned (V) and automatically selects the next phase in the list whose peaks coincide with unidentified peaks.

First selected phase included in the «Accepted Ref. Pattern Name» list: Quartz

No.	Ref. Code	Compound Na...	Chemical Form...	Score	Scale ...	Display Co...
1	ICDD 01-089-1961	Silicon Oxide	Si O2	44	0.933	Blue

No.	Ref. Code	Mineral Name	Score	Compound Na...	Chemical Formula	Scal
1	ICDD 00-050-0015		33	Calcium Manganite	Ca2 Mn14 O27 1x H...	0.
2	ICDD 01-080-1286		29	Strontium Cesium	Sr4.0 Cs1.1 (Al12 Si...	0.
3	ICDD 00-052-2044		29	Anthralin	C14 H10 O3	0.
4	ICDD 00-007-0042	Muscovite-3\ITT...	29	Potassium Alumina	(K, Na) (Al, Mg, ...	0.
5	ICDD 00-048-1952		28	Lanthanum hydroxide	C H3 La O6 P2 I4 H...	0.
6	ICDD 00-037-0409		28	Sodium Zinc Silicate	Na1.625 Zn0.8125 ...	0.
7	ICDD 00-039-0101		28	Sodium Alumina	(Na2 O)0.33 Na Al ...	0.
8	ICDD 00-007-0032	Muscovite 2M1, ...	28	Potassium Alumina	K Al2 Si3 Al O10 (O...	0.
9	ICDD 01-073-2363		27	Zinc Platinum Oxide	Zn2 Pt O4	0.
10	ICDD 01-084-1306	Muscovite 2\ITM...	26	Potassium Alumina	K Al3 Si3 O10 (O H...	0.
11	ICDD 01-077-0018		26	Zinc Platinum Oxide	Zn2 Pt O4	0.

Peaks of our sample

Peaks of the selected phase

«Manual» analysis

In many cases the automatic analysis gives satisfactory results. However, we have to consider that the software chooses candidate minerals based on similarity of the position and intensity of their peaks. We have to use common sense to select the «true» minerals from the list and exclude those with «exotic» compositions. In the case of more complex diffractograms, it may be necessary to do an additional «manual» analysis, looking for specific mineral phases. In the case of construction materials, it is useful to first look for the maximum intensity peaks of the most common minerals (see below) using their d_{hkl} and mark all peaks of the identified phase. Then you would search for phases that match the peaks that have not been assigned to any of the «common» minerals.

Quartz (3.34 Å)

Calcite (3.03 Å)

Dolomite (2.88 Å)

Feldspars (~3.20 Å)

Gypsum (~7.60 Å)

Clays (~4.50 Å), including smectites, illite, kaolinite etc.

«Manual» analysis

7. There is also the possibility to search for a specific phase by choosing «Reference Patterns» and «Restrictions». We select «Strings» and we introduce the name of the mineral phase (Quartz) and press «Load».

The screenshot displays the HighScore software interface. The main window shows a diffraction pattern plot with intensity on the y-axis (0 to 10000) and position on the x-axis (0 to 20). The plot is titled 'Kerstin clay 28 min_Alh ori 1'. The 'Reference Patterns' menu is open, showing 'Retrieve Pattern by' and 'Restrictions...'. The 'Restrictions' dialog box is open, with the 'Strings' tab selected. The 'Mineral Name' field contains the text 'Quartz'. The 'Load' button is highlighted with a red box. The 'Exact Match' checkbox is checked. The 'Save as Subset' button is also visible.

Reference Patterns Analysis Reports Tools Customize Window Help

Retrieve Pattern by

Restrictions...

Reference Code...

Counts

Kerstin clay 28 min_Alh ori 1

Reference Patterns Analysis Reports Tools Customize Window Help

Retrieve Pattern by

Restrictions...

Reference Code...

Restrictions - [Untitled]

Subfiles Chemistry Quality Crystallography Strings Mineral/Zeolite Class

Compound Name: ... X

Mineral Name: Quartz ... X

Formula: ... X

Color: ... X

Author: ... X

Journal: ... X

Exact Match

Load

Save as Subset

Counts

10000

0

10 20

Pe

Additional Graphics

ClipAllToZoom Default IdeAll IdeCom IdeMin IdMine2 Merge PDF scans MinorMinerals MultiRiet Overlay Scans PrintIdeAll

Retrieve Patterns by Restrictions

8. All the files of the particular mineral in the database appear in the list «Accepted Ref. Pattern Name». We should choose the most «suitable» candidate. Duplicates can be removed by selecting them and pressing «Delete».

The screenshot displays the HighScore software interface. The main window shows an XRD pattern for 'Kerstin clay 28 min_Alh ori 1' with 'Counts' on the y-axis and 'Position [°2θ] (Copper (Cu))' on the x-axis. A pattern list window is open, showing a table of reference patterns. A red arrow points from the text above to the first entry in the list, which is highlighted.

No.	Visi...	Ref. Code	Compound Na...	Chemical Form...	Score	Scale ...	Display Co...
1	<input checked="" type="checkbox"/>	ICDD 00-001-0649	Silicon Oxide	Si O2	24	0.205	Blue
2	<input type="checkbox"/>	ICDD 00-002-0458	Silicon Oxide	Si O2	Un...	0.099	Lime
3	<input type="checkbox"/>	ICDD 00-002-0471	Silicon Oxide	Si O2	26	0.055	Gray
4	<input type="checkbox"/>	ICDD 00-003-0419	Silicon Oxide	Si O2	28	0.092	Mar..
5	<input type="checkbox"/>	ICDD 00-003-0427	Silicon Oxide	Si O2	16	0.133	Aqua
6	<input type="checkbox"/>	ICDD 00-003-0444	Silicon Oxide	Si O2	5	0.083	Fuc...
7	<input type="checkbox"/>	ICDD 00-007-0346	Silicon Oxide	Si O2	Un...	0.016	Yell...
8	<input type="checkbox"/>	ICDD 01-070-3755	Silicon Oxide	Si O2	39	0.342	Red
9	<input type="checkbox"/>	ICDD 01-074-1811	Silicon Oxide	Si O2	15	0.015	Blue

9. The program allows the comparison of the similarity of the peaks of our sample with several reference mineral files at the same time by choosing the reference files from the list «Accepted Ref. Pattern Name» and selecting «Pattern View».

The screenshot displays the HighScore software interface. The main window shows three stacked XRD patterns labeled "Muestra", "Ficha 1", and "Ficha 2". The x-axis is labeled "Position [°2θ] (°)" with values 10, 20, and 30. A "Pattern List" window is open, showing a table of reference patterns. A red arrow points to the second row of the table, which is selected. The "Pattern View" button in the bottom toolbar is also highlighted with a red arrow.

No.	Visi...	Ref. Code	Compound Na...	Chemical Form...	Score	Scale ...	Display Co...
1	<input checked="" type="checkbox"/>	ICDD 00-001-0649	Silicon Oxide	Si O2	20	0.296	Blue
2	<input checked="" type="checkbox"/>	ICDD 00-002-0458	Silicon Oxide	Si O2	Un...	0.139	Lime
3	<input type="checkbox"/>	ICDD 00-002-0471	Silicon Oxide	Si O2	21	0.043	Gray
4	<input type="checkbox"/>	ICDD 00-003-0419	Silicon Oxide	Si O2	25	0.129	Mar..
5	<input type="checkbox"/>	ICDD 00-003-0427	Silicon Oxide	Si O2	13	0.193	Aqua
6	<input type="checkbox"/>	ICDD 00-003-0444	Silicon Oxide	Si O2	5	0.047	Fuc...
7	<input type="checkbox"/>	ICDD 00-007-0346	Silicon Oxide	Si O2	Un...	0.010	Yell...
8	<input type="checkbox"/>	ICDD 01-070-3755	Silicon Oxide	Si O2	36	0.548	Red
9	<input type="checkbox"/>	ICDD 01-074-1811	Silicon Oxide	Si O2	18	0.034	Blue

10. The program allows to view the information of the reference mineral files (including the d_{hkl} of each peak), by double clicking with the right mouse button on the Ref. Code of the selected mineral file.

The screenshot shows the HighScore software interface. The main window displays an XRD pattern for 'Kerstin clay 28 min_Bacteria' with a prominent peak at approximately 26.6 degrees 2θ. A red arrow points from this peak to the 'Peak list' window, which shows the reference pattern for Quartz (01-079-1910). The peak list table is as follows:

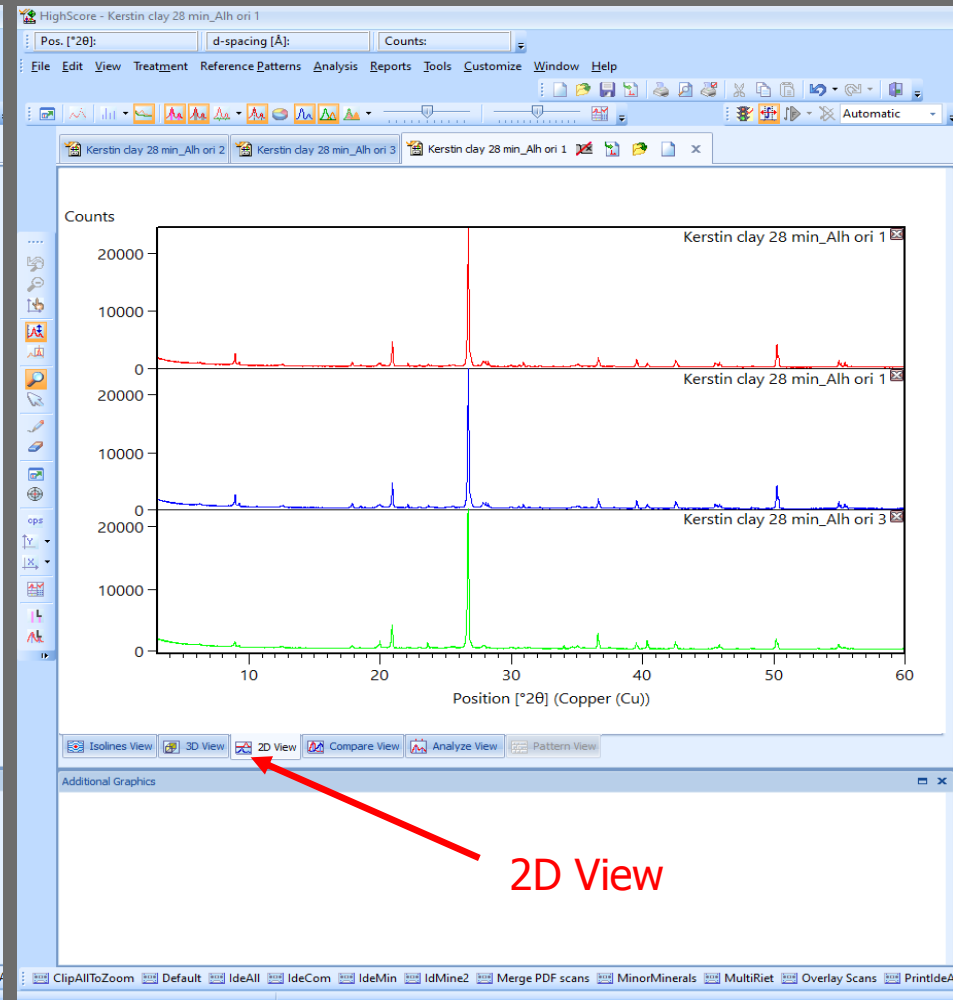
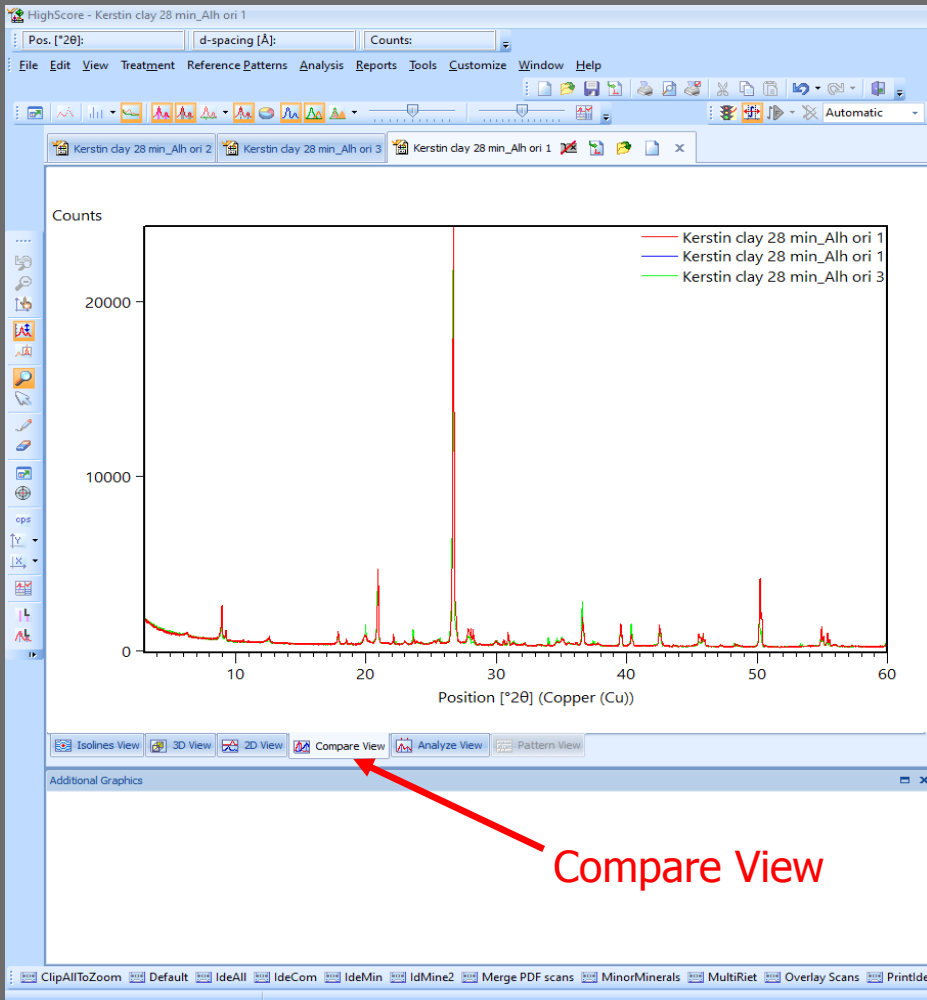
No.	h	k	l	d [Å]	2θ [°]	I [%]
1	1	0	0	4.25565	20.857	21.0
2	1	0	1	3.34387	26.637	100.0
3	1	1	0	2.45700	36.542	6.7
4	0	1	2	2.28166	39.462	6.6
5	1	1	1	2.23681	40.287	2.9
6	2	0	0	2.12782	42.448	4.9
7	0	2	1	1.97997	45.790	2.7
8	1	1	2	1.81812	50.134	11.0
9	0	0	3	1.80200	50.614	0.4
10	2	0	2	1.67193	54.868	3.3
11	1	0	3	1.65937	55.318	1.4
12	2	1	0	1.60848	57.227	0.2
13	2	1	1	1.54169	59.953	7.7
14	1	1	3	1.45309	64.026	1.4

The peak at 26.637 degrees 2θ in the reference pattern corresponds to the peak at 26.6 degrees 2θ in the sample pattern. The peak list table also shows the d-spacing (3.34387 Å) and intensity (100.0%) for this peak.

11. To analyze several samples at the same time use «File» and «Insert» to open additional sample files.

The screenshot displays the HighScore software interface. The main window title is "HighScore - Kerstin clay 28 min_Alh ori 1". The status bar shows "Pos. [°2θ]: 6.411" and "d-spacing [Å]: 13.7752". The menu bar includes File, Edit, View, Treatment, Reference Patterns, Analysis, Reports, Tools, Customize, Window, and Help. The File menu is open, and the "Insert..." option is highlighted with a red box. A red arrow points to the File menu. The background shows an XRD pattern plot for "Kerstin clay 28 min_Alh ori 1" with the x-axis labeled "Position [°2θ] (Copper (Cu))" ranging from 30 to 60. The y-axis represents intensity. The plot shows a prominent peak at approximately 26.5° 2θ and several smaller peaks between 35° and 55° 2θ. At the bottom, there are buttons for "Analyze View" and "Pattern View".

12. There are several options to compare different samples, for example: «Compare View» (you see the diffractograms stacked on top of each other) or «2D View» (you see the diffractograms separately, one above the other).



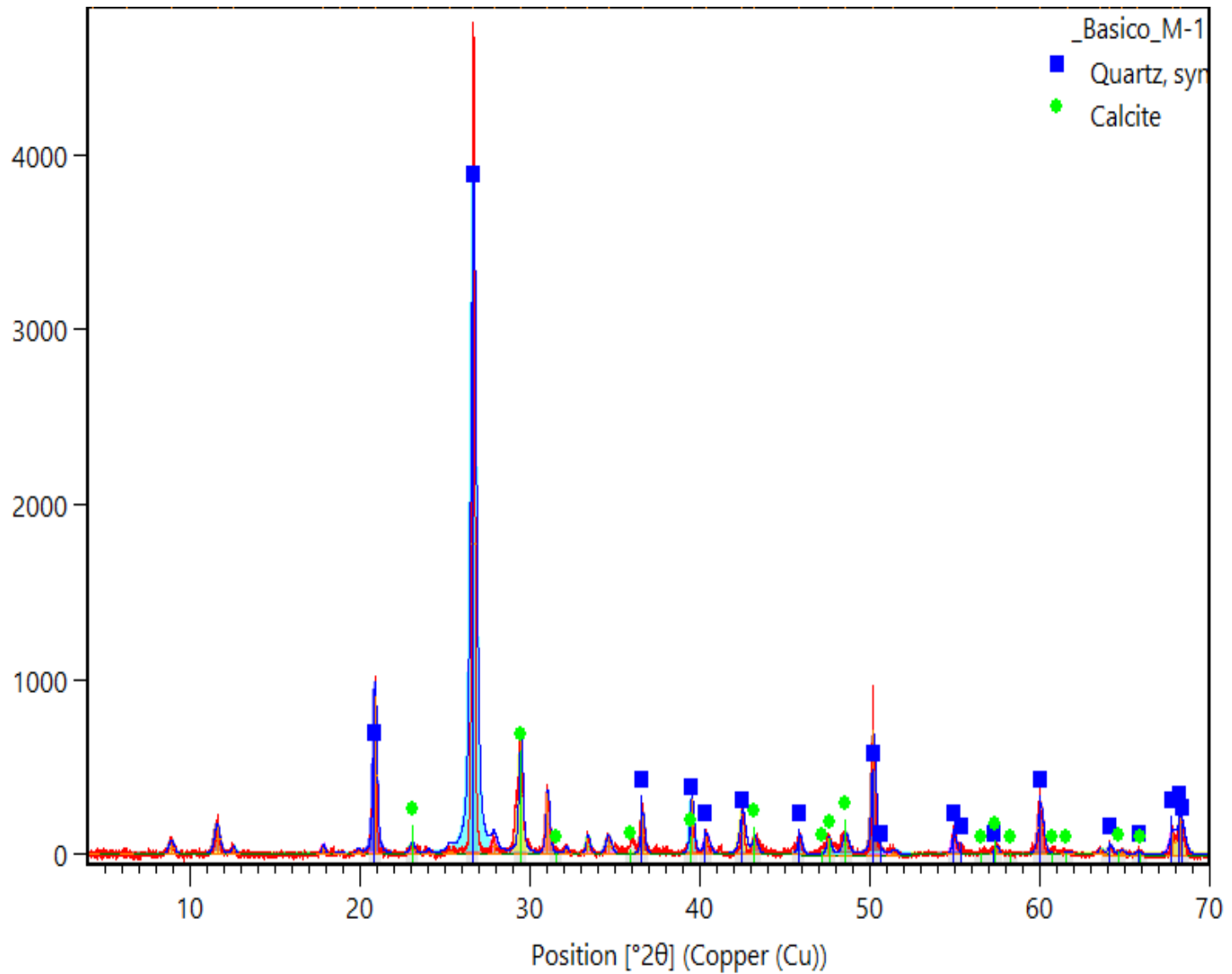
13. By default the program analyzes the first sample. If you want to analyze a different sample, you have to select it with a double «clic» of the right mouse button and choose «Take as Anchor Scan».

The screenshot displays the HighScore software interface. The main window shows a diffraction pattern plot with 'Counts' on the y-axis (0 to 20000) and 'Position [°2θ] (Copper (Cu))' on the x-axis (0 to 40). The plot title is 'Kerstin clay 28 min_Alh ori 1'. A 'Scan List' table is visible in the upper right, with a context menu open over it. The 'Take as Anchor Scan' option is highlighted with a red box. A red arrow points from the text above to this option.

No.	Lin...	Vis...	Name	Start pos...	Omega Position ...	X Position [mm]	Measured Date/
1		<input checked="" type="checkbox"/>	Kerstin d...	3.0082	0.0000	0.0000	12/03/2020 15:1
2		<input checked="" type="checkbox"/>	Kerstin d...	3.0082	0.0000	0.0000	12/03/2020 15:1
3		<input checked="" type="checkbox"/>	Kerstin d...	3.0082	0.0000	0.0000	12/03/2020 15:1

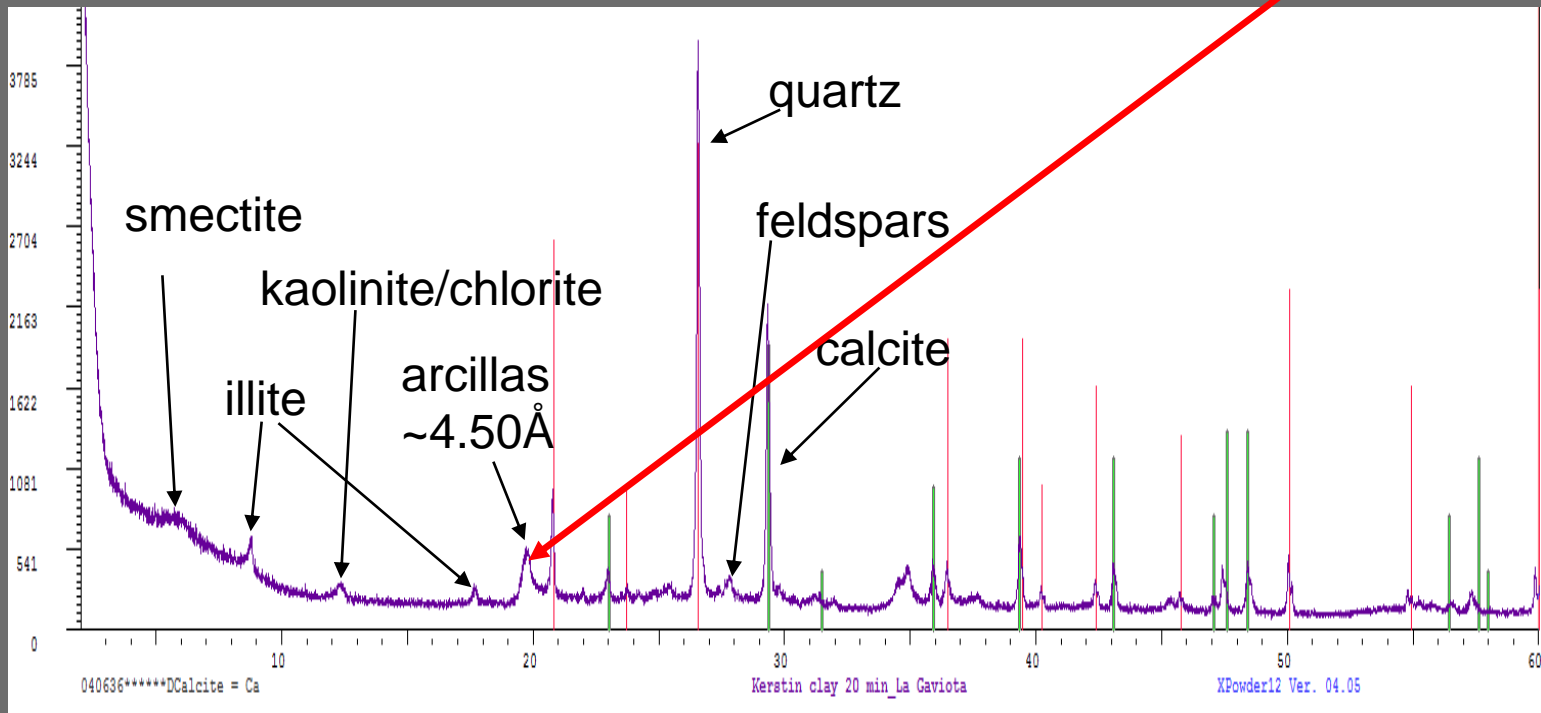
- Edit Scan Parameters...
- Copy To
- Re-Apply Color Scheme
- Make Cluster Visible
- Copy Representative Scans to New Document
- Take as Anchor Scan** **Ctrl+T**
- Take as Background
- Subtract existing Background
- Remove Scan
- Duplicate Scan
- Toggle visible
- Add selected Scans to Reference Database
- Simple Sum...

The analyzed diffractogram can be stored as a pdf file.

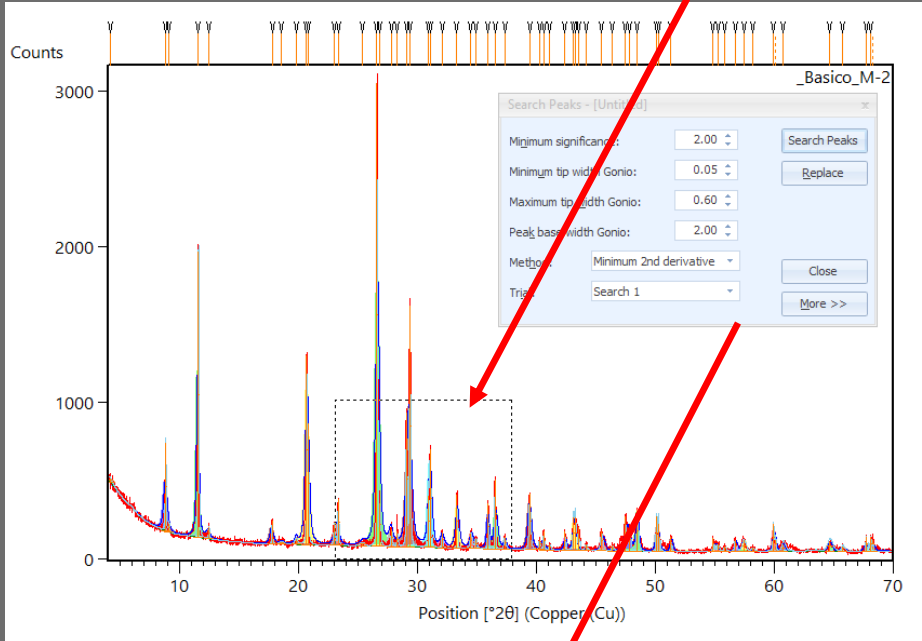
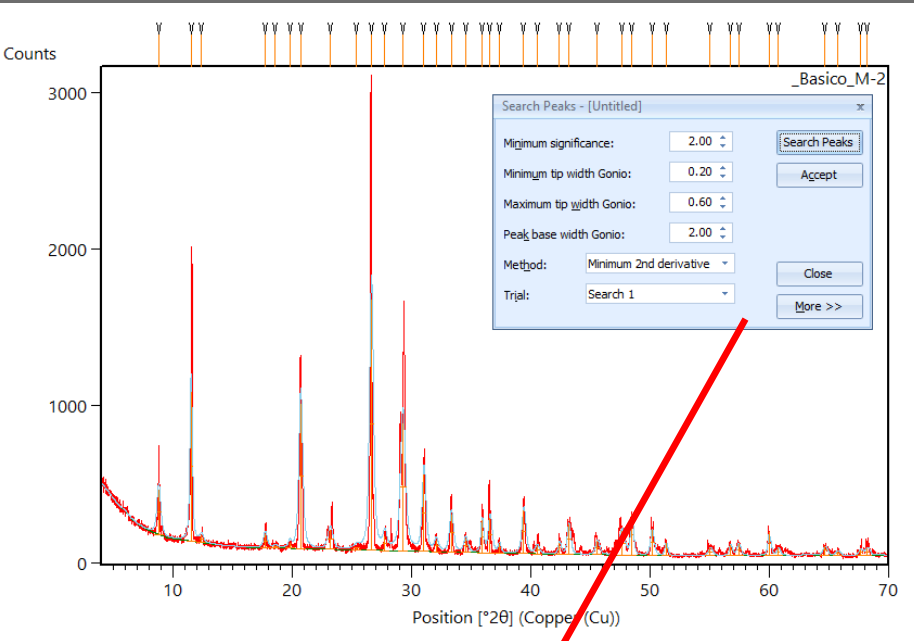


Quantification of mineral phases using XRD

Generally, commercial software allows a more or less reliable automatic quantification. A high precision quantitative analysis requires the application of the Rietveld method (advanced XRD analysis). In general, the XRD analysis is semiquantitative and in many cases we have to assume an error of approximately ± 5 wt% (in the case of clay minerals up to ± 10 wt%). Considering that many building materials are quite complex and contain several mineral phases including clays, a «semimanual» quantification is recommended. The automatic and semimanual methods are described below, using experimental reflective power values and the general peak of clays at $\sim 4.50 \text{ \AA}$ (if the sample contains clay minerals) in the case of the latter.



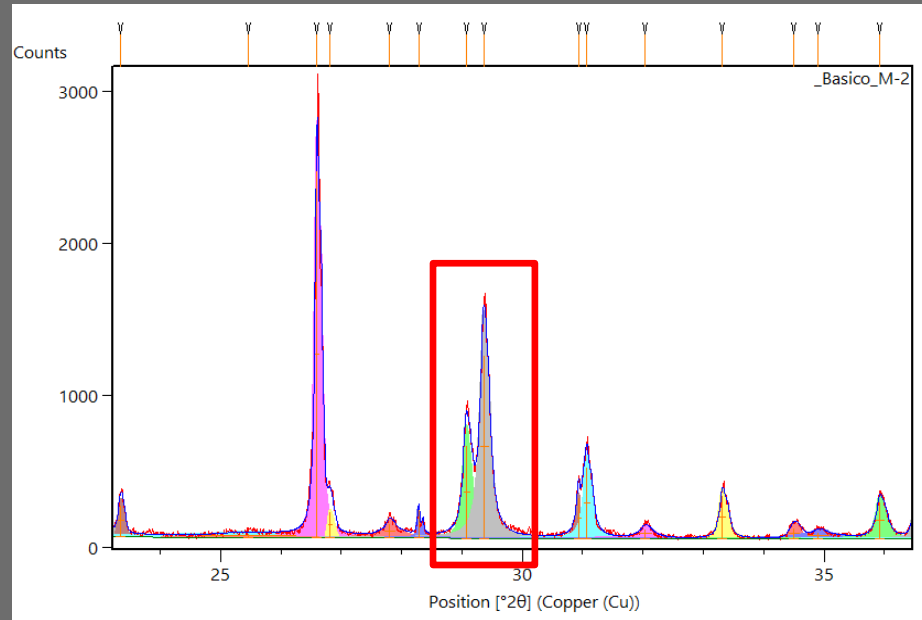
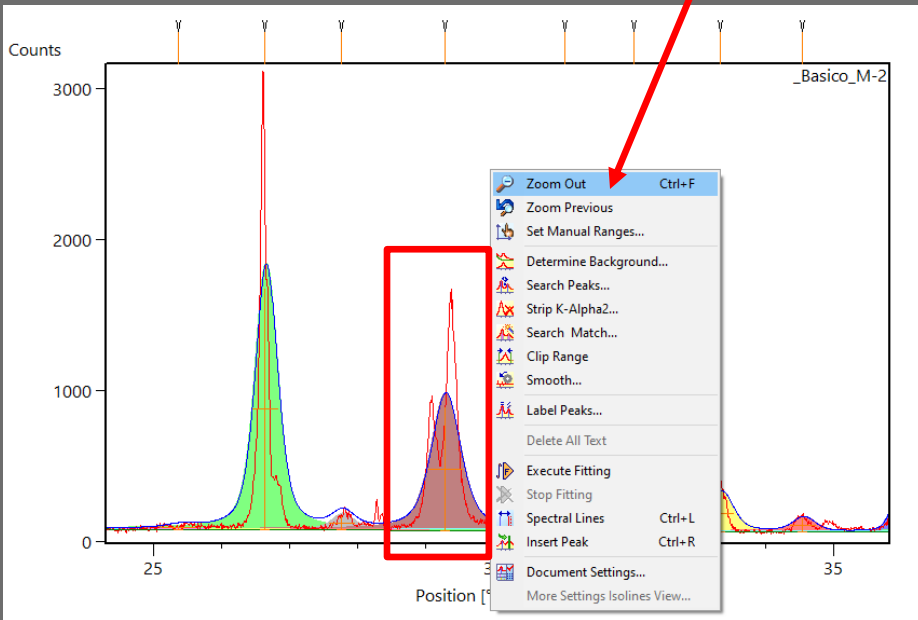
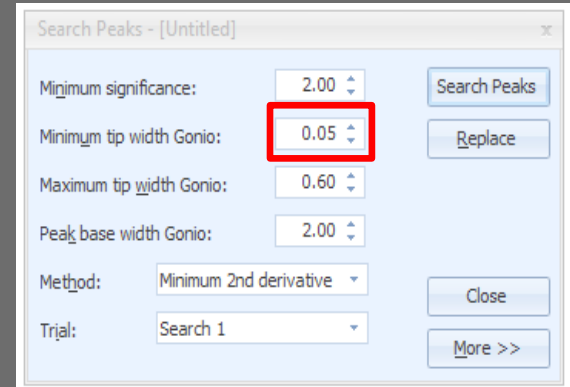
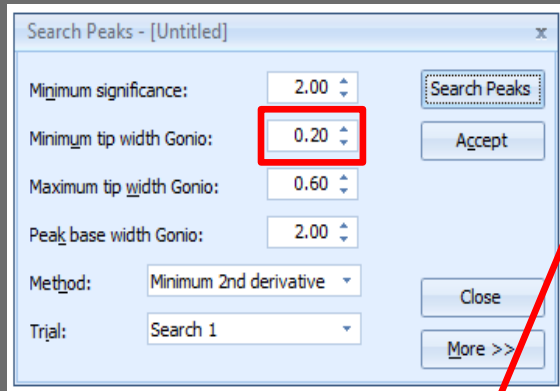
14. For a correct quantification we have to ensure that all peaks are selected and separated adequately. This can be verified by selecting a specific zone using the left mouse button.



This is a close-up view of the search peak dialog box. The 'Minimum tip width Gonio' field is highlighted with a red box and has a red arrow pointing to it from the XRD plot above. The other settings are: Minimum significance: 2.00, Maximum tip width Gonio: 0.60, Peak base width Gonio: 2.00, Method: Minimum 2nd derivative, and Trial: Search 1.

This is a close-up view of the search peak dialog box. The 'Minimum tip width Gonio' field is highlighted with a red box and has a red arrow pointing to it from the XRD plot above. The other settings are: Minimum significance: 2.00, Maximum tip width Gonio: 0.60, Peak base width Gonio: 2.00, Method: Minimum 2nd derivative, and Trial: Search 1.

Here we see the effect of a reduction in the «Minimum tip width Gonio» on the peak separation. To obtain the full view of the diffractogram again we use the right mouse button and select «Zoom Out».

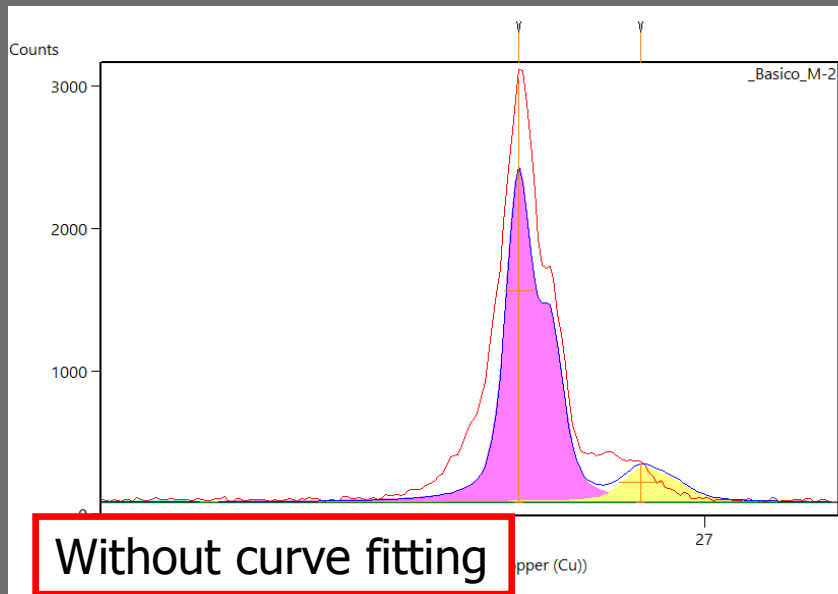


15. Once we have selected all peaks, we click on «Execute Fitting» in order to improve the curve fitting for each peak.

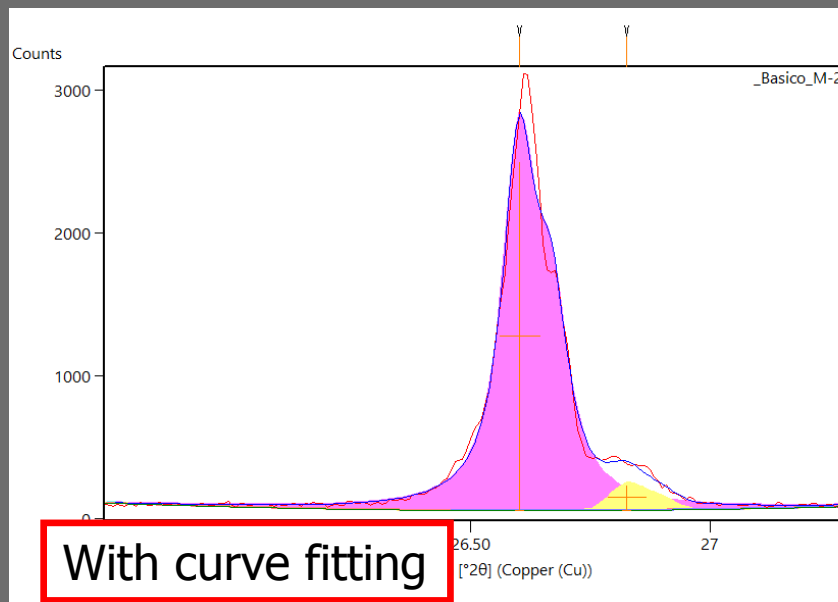
The screenshot displays the software interface for XRD data analysis. The main window shows a plot of Counts vs. Position [°2θ] (Copper (Cu)) with several peaks. A red arrow points from the text above to the 'Execute Fitting' button in the top right toolbar. An inset window titled 'R-Values' shows a graph of R-values (%) vs. Cycles (1-10) with three curves: Rexp (red), Rpr (blue), and Rwpr (green).

Cycles	Rexp (%)	Rpr (%)	Rwpr (%)
1	8.5	15.5	20.0
2	8.5	12.5	16.0
3	8.5	10.5	13.5
4	8.5	9.5	12.5
5	8.5	9.2	12.2
6	8.5	9.1	12.1
7	8.5	9.0	12.0
8	8.5	9.0	12.0
9	8.5	9.0	12.0
10	8.5	9.0	12.0

Below we see how the curve fitting influences the value of the peak area (counts). This information can be obtained by selecting «Peak List».

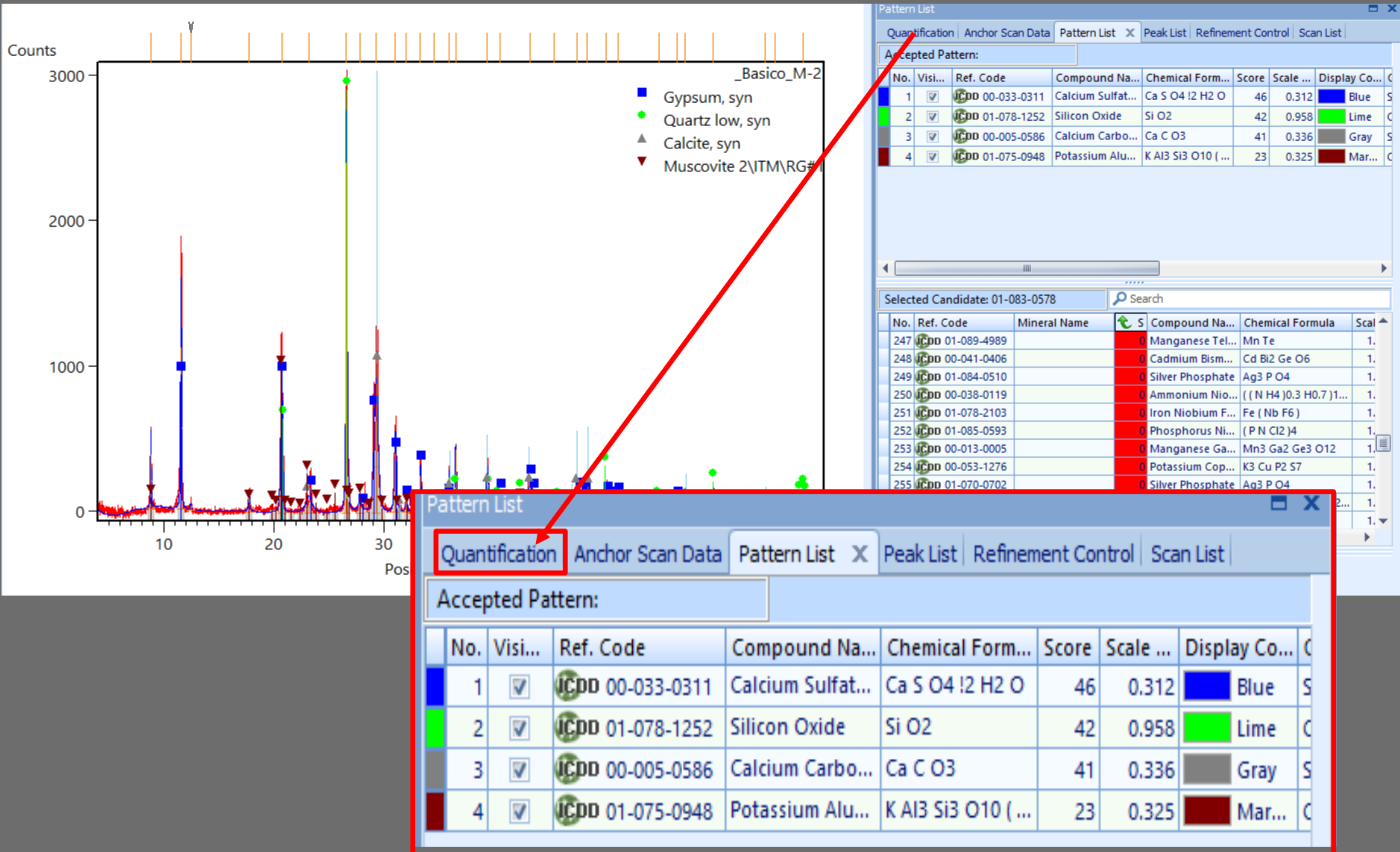


No.	Pos. [°2θ]	d-spacing [Å]	Height [c...]	Sha...	Area [cts**2θ]	Status	Crystallite Size only [Å]
1	4.2042	21.01773	146.17	0.600	54.48	Incl...	24
2	8.8595	9.98151	575.72	0.600	44.71	Incl...	210
3	9.1360	9.68002	57.13	0.600	5.32	Incl...	133
4	11.6197	7.61587	1872.05	0.600	145.37	Incl...	207
5	12.5004	7.08126	78.12	0.600	7.28	Incl...	132
6	17.8077	4.98097	154.38	0.600	9.59	Incl...	2827
7	18.5897	4.77315	15.21	0.600	5.67	Incl...	24
8	19.8153	4.48061	38.59	0.600	9.59	Incl...	37
9	20.7000	4.29106	1242.01	0.600	96.45	Incl...	201
10	20.8393	4.26271	800.38	0.600	49.72	Incl...	2840
11	23.0329	3.86146	139.34	0.600	21.64	Incl...	63
12	23.3584	3.80839	305.34	0.600	23.71	Incl...	200
13	25.3734	3.51033	21.07	0.600	10.47	Incl...	18
14	26.6098	3.34996	3035.81	0.600	188.59	Incl...	2870
15	26.822	3.328	78.12	0.600	7.28	Incl...	132
16	27.834	3.18	154.38	0.600	9.59	Incl...	2827
17	28.311	3.14	15.21	0.600	5.67	Incl...	24
18	29.077	3.08	38.59	0.600	9.59	Incl...	37
19	29.367	3.05	1242.01	0.600	96.45	Incl...	201
20	30.925	2.92	800.38	0.600	49.72	Incl...	2840
21	31.081	2.91	139.34	0.600	21.64	Incl...	63
22	32.080	2.83	139.34	0.600	21.64	Incl...	63
23	33.328	2.70	139.34	0.600	21.64	Incl...	63
24	34.504	2.61	139.34	0.600	21.64	Incl...	63
25	34.899	2.58	139.34	0.600	21.64	Incl...	63
26	35.936	2.48	139.34	0.600	21.64	Incl...	63
27	36.514	2.43	139.34	0.600	21.64	Incl...	63
28	37.339	2.38	139.34	0.600	21.64	Incl...	63
29	39.4310	2.28527	370.33	0.600	51.76	Incl...	75

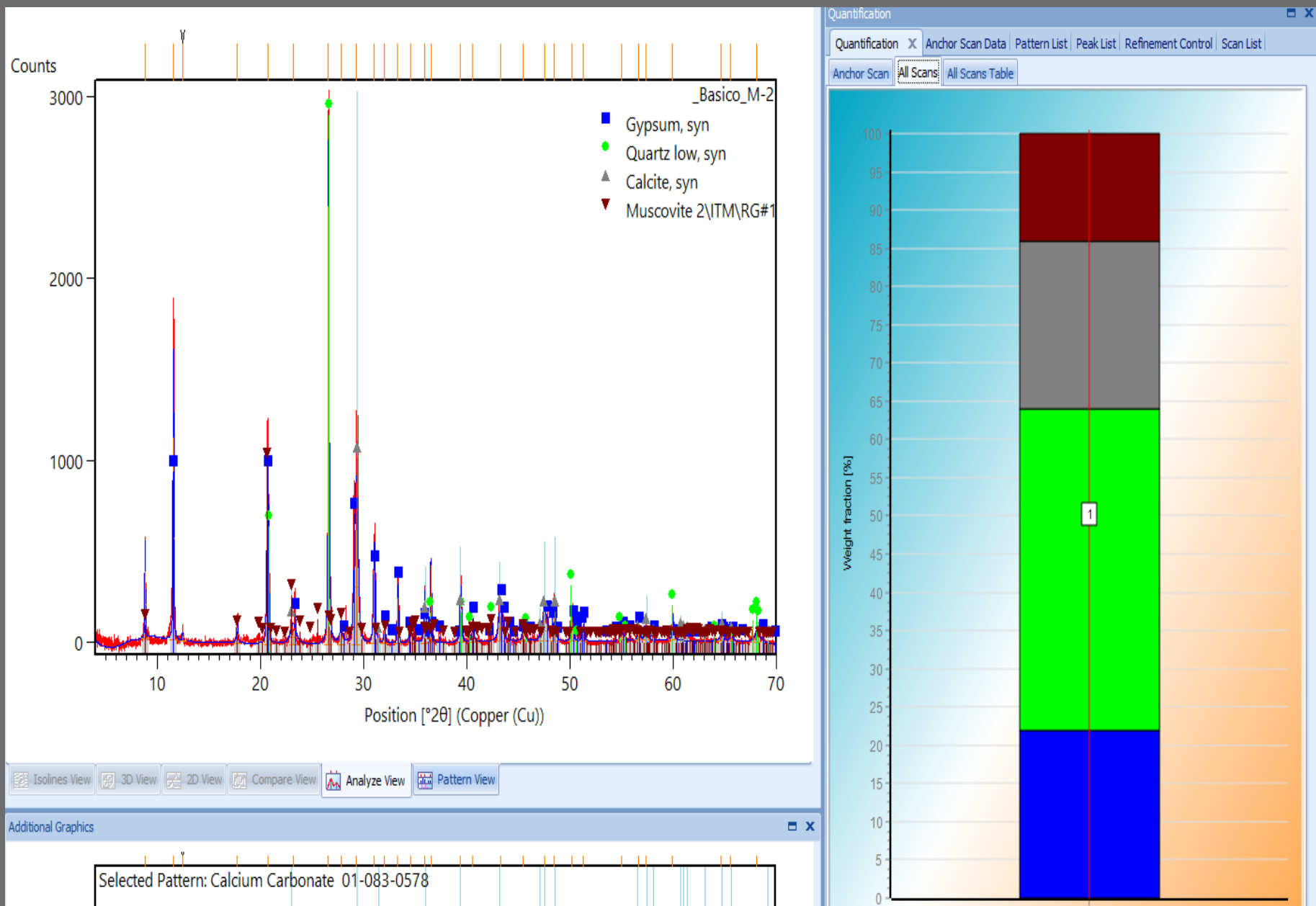


No.	Pos. [°2θ]	d-spacing [Å]	Height [c...]	Sha...	Area [cts**2θ]	Status	Crystallite Size only [Å]
1	4.2042	21.01773	146.17	0.600	54.48	Incl...	24
2	8.8595	9.98151	575.72	0.600	44.71	Incl...	210
3	9.1360	9.68002	57.13	0.600	5.32	Incl...	133
4	11.6197	7.61587	1872.05	0.600	145.37	Incl...	207
5	12.5004	7.08126	78.12	0.600	7.28	Incl...	132
6	17.8077	4.98097	154.38	0.600	9.59	Incl...	2827
7	18.5897	4.77315	15.21	0.600	5.67	Incl...	24
8	19.8153	4.48061	38.59	0.600	9.59	Incl...	37
9	20.7000	4.29106	1242.01	0.600	96.45	Incl...	201
10	20.8393	4.26271	800.38	0.600	49.72	Incl...	2840
11	23.0329	3.86146	139.34	0.600	21.64	Incl...	63
12	23.3584	3.80839	305.34	0.600	23.71	Incl...	200
13	25.3734	3.51033	21.07	0.600	10.47	Incl...	18
14	26.6007	3.34831	2488.77	0.959	322.34	Incl...	180
15	26.822	3.328	78.12	0.600	7.28	Incl...	132
16	27.834	3.18	154.38	0.600	9.59	Incl...	2827
17	28.311	3.14	15.21	0.600	5.67	Incl...	24
18	29.077	3.08	38.59	0.600	9.59	Incl...	37
19	29.367	3.05	1242.01	0.600	96.45	Incl...	201
20	30.925	2.92	800.38	0.600	49.72	Incl...	2840
21	31.081	2.91	139.34	0.600	21.64	Incl...	63
22	32.080	2.83	139.34	0.600	21.64	Incl...	63
23	33.328	2.70	139.34	0.600	21.64	Incl...	63
24	34.504	2.61	139.34	0.600	21.64	Incl...	63
25	34.899	2.58	139.34	0.600	21.64	Incl...	63
26	35.936	2.48	139.34	0.600	21.64	Incl...	63
27	36.514	2.43	139.34	0.600	21.64	Incl...	63
28	37.339	2.38	139.34	0.600	21.64	Incl...	63
29	39.4310	2.28527	370.33	0.600	51.76	Incl...	75

16. The program HighScore allows an automatic quantification considering all fases included in «Accepted Pattern».



Example of an automatic quantification considering all phases included in «Accepted Pattern».



In order to obtain the values (weight percent) of the semiquantification of each phase, we have to select «Pattern List» and the file of the corresponding phase. Below we see the semiquantification for calcium carbonate (calcite).

The image shows a software interface with two main panels: 'Pattern List' and 'Object Inspector'.

Pattern List Panel:

Quantification | Anchor Scan Data | **Pattern List** | Peak List | Refinement Control | Scan List

Accepted Ref. Pattern: 00-005-0586

No.	Visi...	Ref. Code	Compound Na...	Chemical Form...	Score	Scale ...	Display Co...
1	<input checked="" type="checkbox"/>	ICDD 00-033-0311	Calcium Sulfat...	Ca S O4 I2 H2 O	46	0.312	Blue
2	<input checked="" type="checkbox"/>	ICDD 01-078-1252	Silicon Oxide	Si O2	42	0.958	Lime
3	<input checked="" type="checkbox"/>	ICDD 00-005-0586	Calcium Carbo...	Ca C O3	41	0.336	Gray
4	<input checked="" type="checkbox"/>	ICDD 01-075-0948	Potassium Alu...	K Al3 Si3 O10 (...)	23	0.325	Mar...

Selected Candidate: 01-083-0578

No.	Ref. Code	Mineral Name	S	Compound Na...	Chemical Formula	Scal
247	ICDD 01-089-4989		0	Manganese Tel...	Mn Te	1.
248	ICDD 00-041-0406		0	Cadmium Bism...	Cd Bi2 Ge O6	1.
249	ICDD 01-084-0510		0	Silver Phosphate	Ag3 P O4	1.
250	ICDD 00-038-0119		0	Ammonium Nio...	((N H4)0.3 H0.7)1...	1.
251	ICDD 01-078-2103		0	Iron Niobium F...	Fe (Nb F6)	1.
252	ICDD 01-085-0593		0	Phosphorus Ni...	(P N Cl2)4	1.
253	ICDD 00-013-0005		0	Manganese Ga...	Mn3 Ga2 Ge3 O12	1.
254	ICDD 00-053-1276		0	Potassium Cop...	K3 Cu P2 S7	1.
255	ICDD 01-070-0702		0	Silver Phosphate	Ag3 P O4	1.
256	ICDD 00-046-0495		0	Thallium Cobal...	Tl2 Co (P O3 F)2 I2...	1.
257	ICDD 00-046-0283		0	Lithium Rubidi...	Li Rb Zn O2	1.

Object Inspector Panel:

Selected object: Accepted Pattern

Search

Reference Code: ICDD 00-005-0586

Display:

- Visible:
- Color: Gray
- Manual Shift: [Slider]
- Manual Scale: [Slider]
- Reset Pattern: ...
- Marker: Triangle

Search & Match:

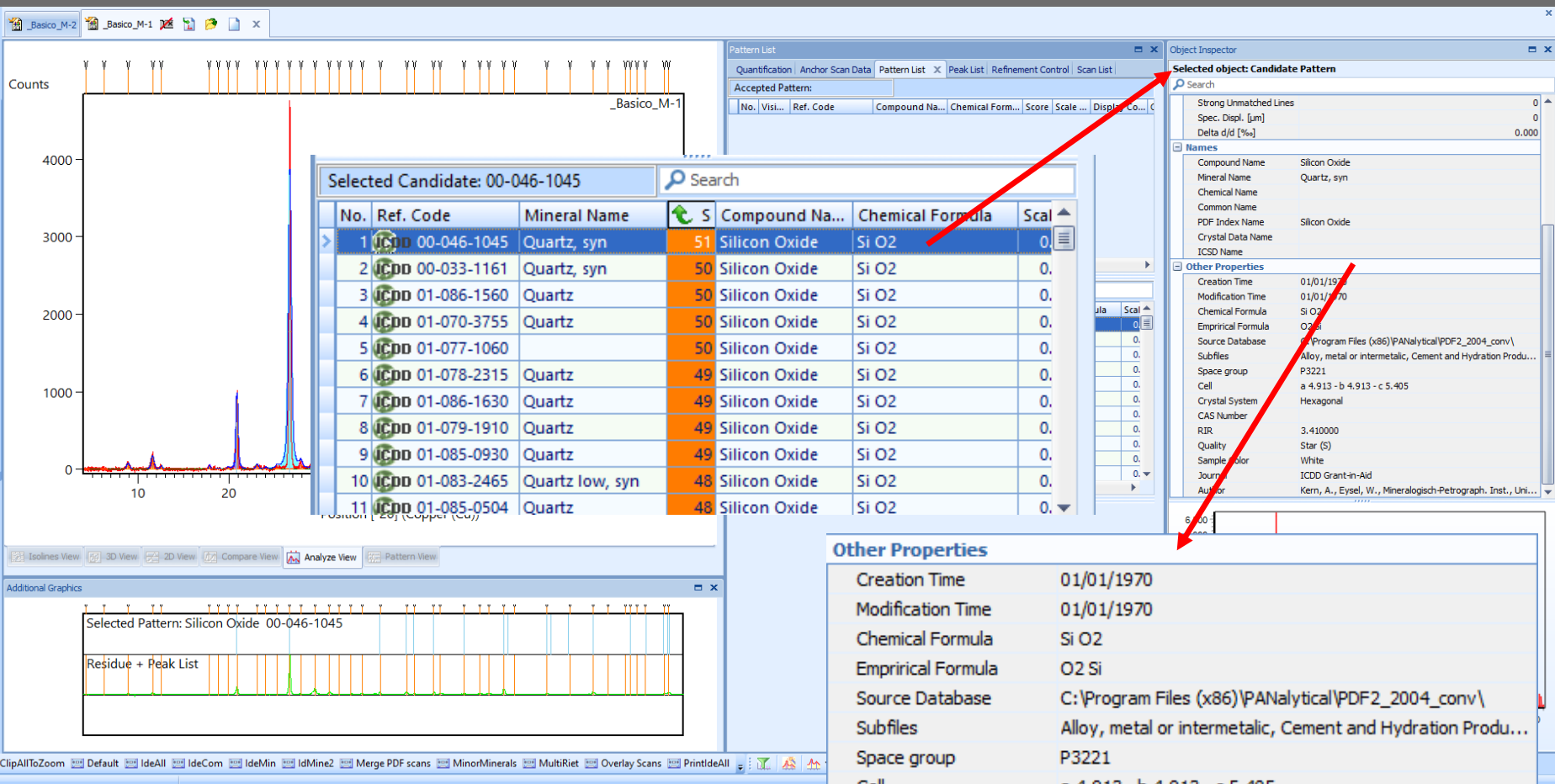
- Score: 41
- Scale Factor: 0.336
- Displacement [°2Th.]: 0.000
- Matched Lines: 12
- Total Lines: 17
- Strong Unmatched Lines: 0
- Spec. Displ. [µm]: 0
- Delta d/d [%]: 0.000
- Semi Quant [%]: 22**

Names:

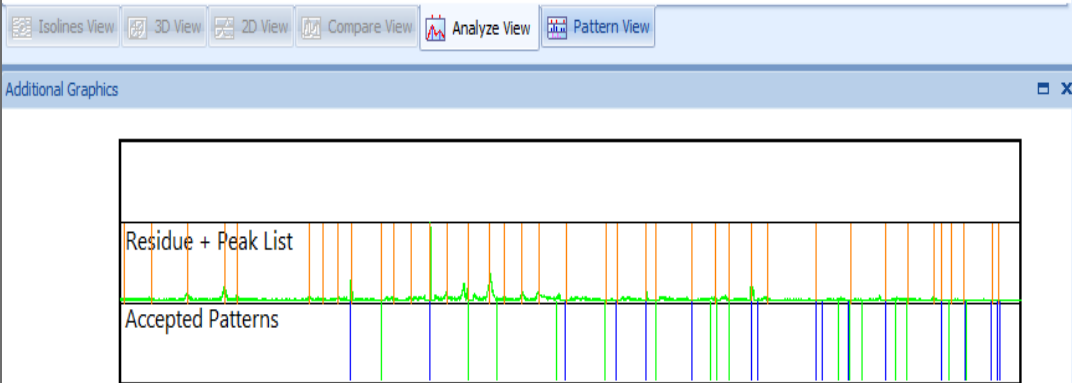
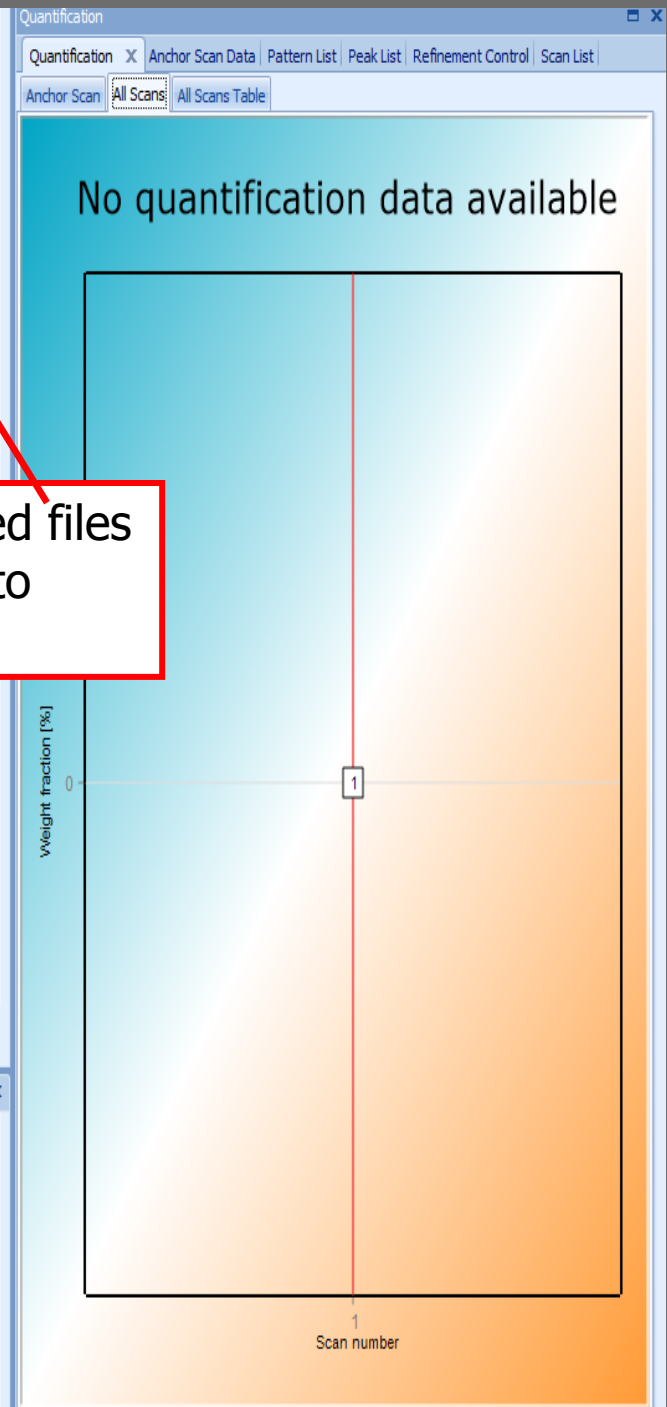
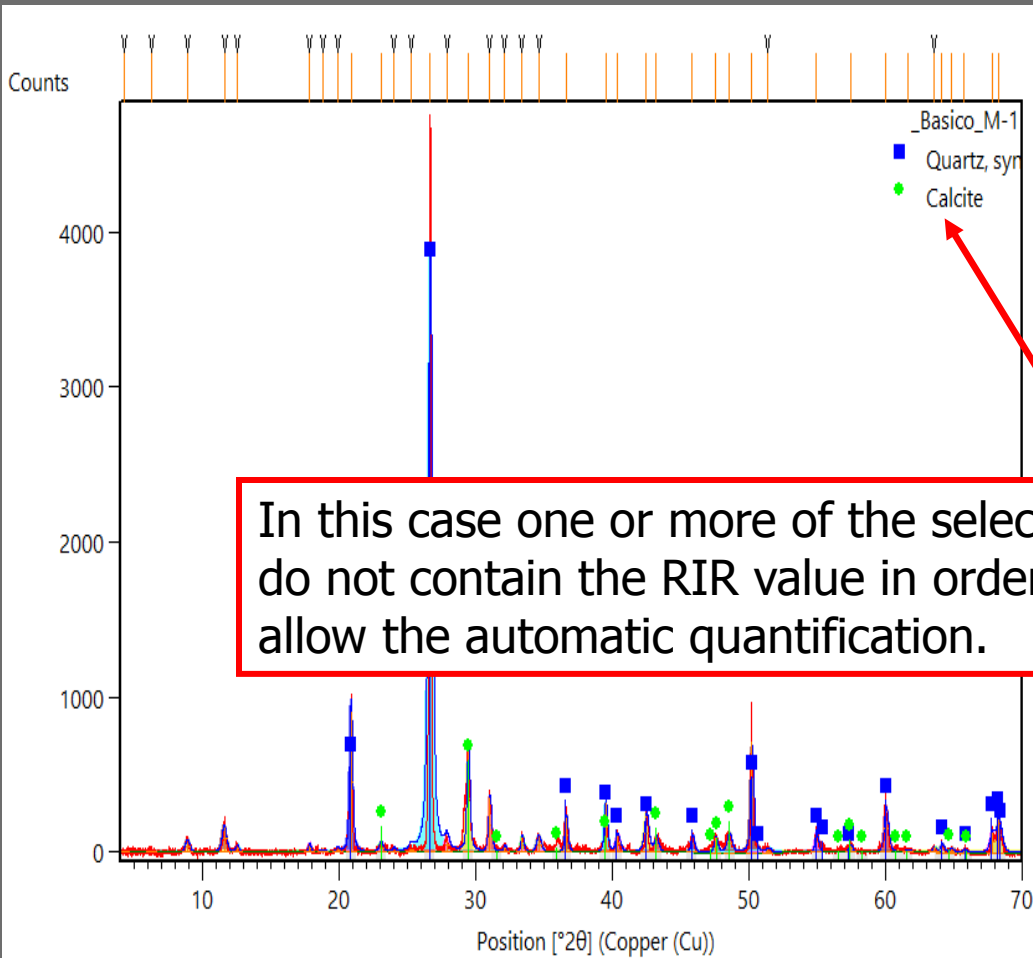
- Compound Name: Calcium Carbonate
- Mineral Name: Calcite, syn
- Chemical Name:
- Common Name:
- PDF Index Name: Calcium Carbonate
- Crystal Data Name:
- ICSD Name:

Other Properties:

We have to ensure that all selected files contain the RIR values in order to obtain an automatic quantification. We can obtain this information by selecting «Pattern List» and clicking on the selected file.



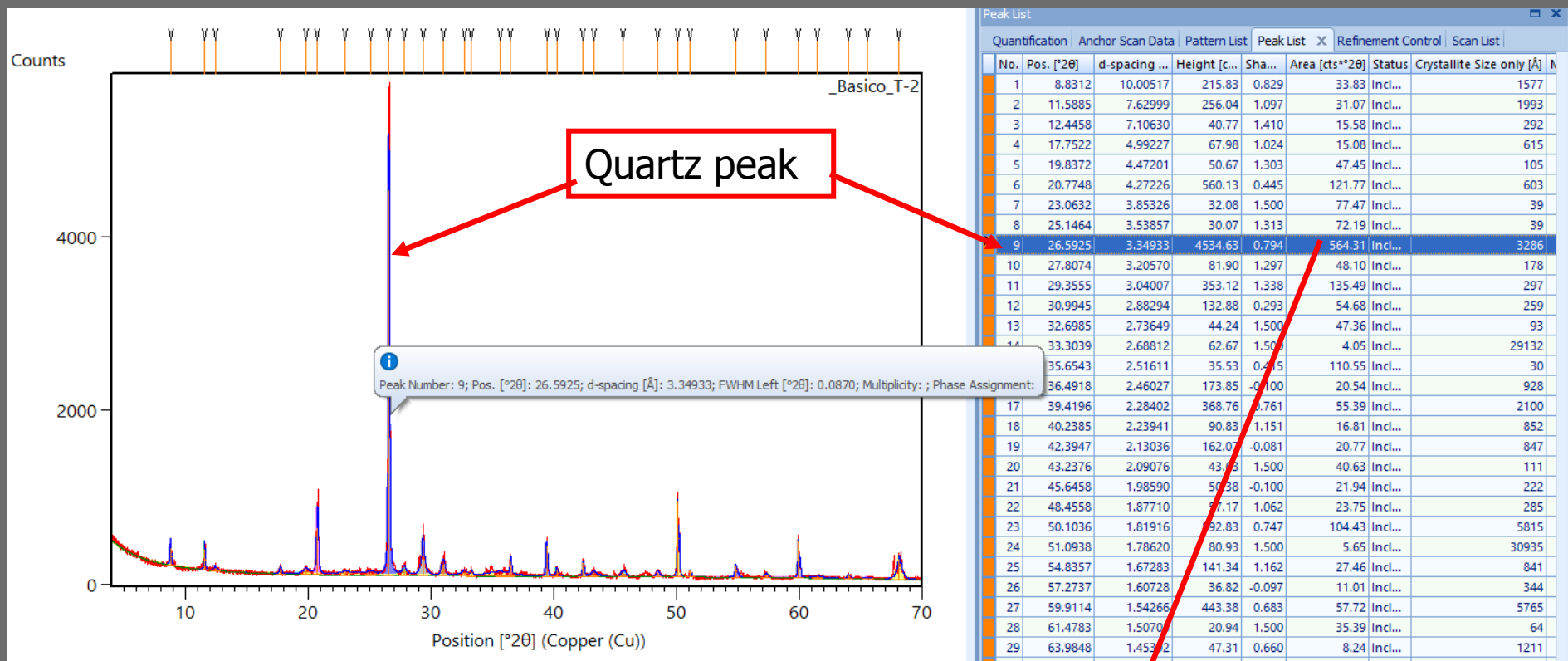
Other Properties	
Creation Time	01/01/1970
Modification Time	01/01/1970
Chemical Formula	Si O2
Empirical Formula	O2 Si
Source Database	C:\Program Files (x86)\PANalytical\PDF2_2004_conv\
Subfiles	Alloy, metal or intermetallic, Cement and Hydration Produ...
Space group	P3221
Cell	a 4.913 - b 4.913 - c 5.405
Crystal System	Hexagonal
CAS Number	
RIR	3.410000
Quality	Star (S)
Sample Color	White
Journal	ICDD Grant-in-Aid
Author	Kern, A., Eysel, W., Mineralogisch-Petrograph. Inst., Uni...



The values of the «Reference Intensity Ratio» (RIR, similar to the reflective power), which are used by the program in order to perform the automatic quantification might vary for each file. Consequently, the quantification will vary depending on the selected files.

Gypsum											
Patterns		9		Offset		<input checked="" type="checkbox"/>		Max offset		0.15	
								Convergence		0	
Set-Fil	Phase name	Q	Fract	RIR.	% W Unc	Ab	m/rho	% W Xtal	% W Xtal+A	min	%
700982	Gypsum	1	0.683	1.90	14.5 (0.3)		60.8	14.6 (0.3)	14.1 (0.3)	000.0	
700983	Gypsum	1	0.415	1.70	09.8 (0.5)		60.8	09.9 (0.5)	09.6 (0.4)	000.0	
700984	Gypsum	1	0.682	1.70	16.2 (0.3)		60.8	16.3 (0.3)	15.8 (0.3)	000.0	
720596	Gypsum	1	0.250	1.90	05.3 (0.5)		60.8	05.3 (0.5)	05.2 (0.5)	000.0	
741433	Gypsum	1	0.682	1.60	17.2 (0.3)		60.8	17.3 (0.3)	16.8 (0.3)	000.0	
741904	Gypsum	1	0.415	1.70	09.8 (0.5)		60.8	09.9 (0.5)	09.6 (0.4)	000.0	
741905	Gypsum	1	0.415	1.90	08.8 (0.5)		60.8	08.9 (0.5)	08.6 (0.4)	000.0	
761746	Gypsum	1	1.000	5.00	08.1 (0.2)		60.8	07.5 (0.2)	07.3 (0.2)	000.0	

17. For a «semimanual» quantification, instead of measuring the height and calculating the absolute intensity as we have done in the case of the XRD exercises, we are going to use the number of counts corresponding to the area of the peak with the maximum intensity of each phase. If we place the mouse on top of the peak the program will mark the corresponding data in the «Peak List».



9	26.5925	3.34933	4534.63	0.794	564.31	Incl...
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Semimanual quantification

For the quantification, we divide the number of counts corresponding to the area of each phase (peak of max. intensity) by the corresponding reflective power (R.P.). Note: Mineral names are included in English because identification programs use English names.

Phase	R.P.	d_{hkl} (Å)
Quartz	1.43	3.34
Calcite	1.05	3.03
Dolomite	1.03	2.88
Gypsum	0.70	7.56
Feldspars	0.98	~3.20
Strontianite	0.60	3.53
Celestite	0.52	2.97
Fluorite	2.00	3.16
Galena	1.50	2.96
Clays (mica, illite, kaolinite, smectite, ect.)	0.09	~4.50

18. For a semiquantitative analysis we create an EXCEL (or other spreadsheet software) document with the following columns: mineral name, d_{hkl} , reflective power, area counts, area counts divided by reflective power (AC/RP), weight percentage (wt%) and semiquantitative percentage (± 5 wt%). We simply have to enter the number of counts corresponding to the area of each mineral (red column), add all AC/RP values (green column), divide the AC/RP value of each mineral by the sum of AC/RP (blue column), and adjust the semiquantitative values by rounding to multiples of five.

Mineral	d_{hkl}	Reflective Power	Area Counts	AC/RP	Percentage (wt%)	Percentage semicuant. (wt%)
Phyllosilicates	4.49	0.09	512	(512/0.09 =) 5689	(5689/14039 =) 40	40
Quartz	3.34	1.43	11011	7700	55	55
Calcite	3.03	1.05	0	0	0	0
Dolomite	2.88	1.08	279	258	2	<5
Gypsum	7.05	0.70	0	0	0	0
Feldspars	3.21	1.03	404	392	3	<5
				Sum 14039		

19. We also have the possibility to present the results of an XRD analysis in a table, describing the abundance of each phase using the following terms: very abundant, abundant, less abundant and trace (see table). We have to consider that some minerals have very low reflective power, resulting in a relatively small peak despite a considerable amount of this mineral in the sample (for example smectites) or that the position of peaks of two minerals overlap (for example the 003 reflection of illite and 101 reflection of quartz), which may cause an underestimation of smectite or an overestimation of quartz if quantification is done by a «visual» estimate.

Example

Sample	Phyllosilicates	Quartz	Calcite	Dolomite	Gypsum	Feldspars
Alhambra 1	+	+++	+	+	-	+
Alhambra 2	tr	++	++	+	-	+
Alhambra 3	+	+++	+	tr	tr	tr
Alhambra 4	++	++	+	tr	tr	tr
Alhambra 5	tr	++	++	+	+	-
Alhambra 6	-	++	++	-	tr	-

+++ = very abundant

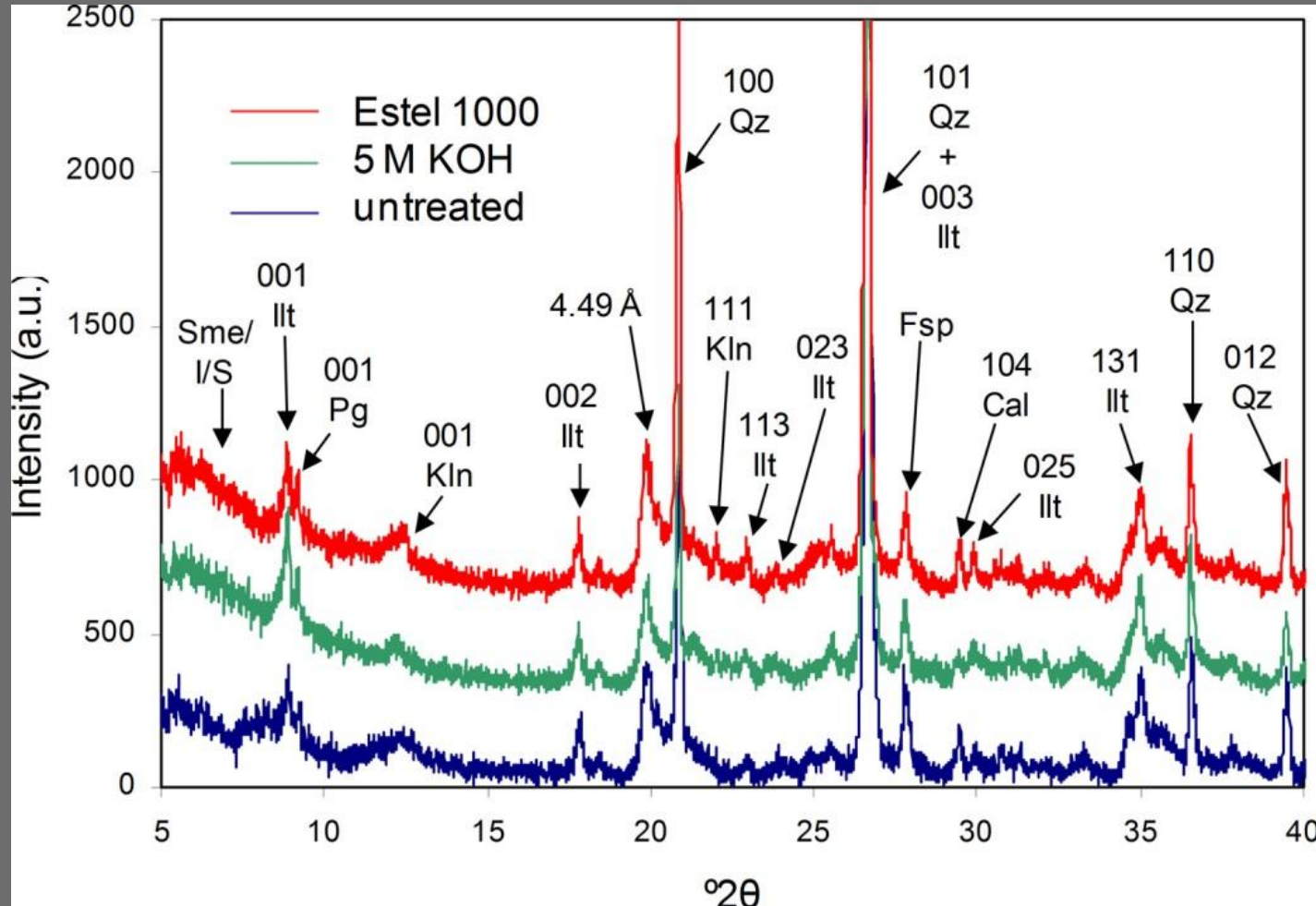
++ = abundant

+ = less abundant

tr = trace

- = not detected

20. For the presentation of the XRD data it is recommended to use a spreadsheet program such as EXCEL or ORIGIN and include the names of the minerals (official abbreviation *) and their d_{hkl} (see reference mineral files). To be able to open the files in these programs, you must convert them to text or xy using programs such as POWDLL.



*D.L. Whitney y B.W. Evans, Abbreviations of names of rock-forming minerals, Amer. Miner. 95 (2010) 185–187.