



DIFFRAC.EVA



FIRST Newsletter

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## DIFFRAC.EVA – What's New 2018?

By *Karsten Knorr, Global Product Manager Industrial Markets – XRD, Bruker AXS GmbH, Karlsruhe, Germany*

The latest evolution of our successful software for basic XRD data evaluation is in fact a revolution. DIFFRAC.EVA Versions 4.3 and 5 came with numerous improvements and new functionality. Working with the new EVA becomes as easy as never before. The current EVA combines ease-of-use for the occasional user with full flexibility and increased productivity for the experts. Welcome to the next level in powder diffraction.

### New Hardware Support

EVA 5 fully supports the new 2-dimensional (2D) detector that recently was integrated to the D8 platform, the EIGER2 R 500K. All scan types are supported, including the new 2D continuous scan mode and variable detector opening.

### BRAGG2D

BRAGG2D combines the para-focusing beam geometry with a new two-dimensional data processing algorithm to assess effects of sample preparation. The illumination of a large specimen area with the full X-ray beam allows rapid assessments while only a one-dimensional detector is required. For details of this technique refer to Application Note XRD 615: [BRAGG2D – Two Dimensional Diffraction with the D2 PHASER](#).

### All News at a Glance

- Full support of new detector hardware the EIGER2 R 500K with all scan modes including panoramic two-dimensional continuous scanning.
- BRAGG2D, two-dimensional visualization of powder XRD sample effects, such as grain size issues or texture/preferred orientation.
- SIP-X the next level in phase identification. Phase ID is at the heart of powder diffraction. Therefore, this feature is made available to all current EVA users as a complementary upgrade – regardless of the actual license level.
- Profile analysis (peak and pattern fit) for peak shape-evaluation, semi-quantitative phase analysis and crystallite size determination.
- SQUALL: Semi-quantitative full pattern matching analysis, and positive materials identification (PMI). This includes automated operation in batch, and console mode for all SQUALL functionality.
- Extended database support with access to the greatly improved Results Manager. This makes reporting multi sample properties as easy and smooth as never before.

### SEARCH/MATCH, An Entire Species Identification Protocol

Phase identification is one of the biggest obstacles in powder diffraction. To date about 400.000 species are found in the PDF4+ database and another 500.000 in the PDF Organics database. This high diversity makes the identification of phases (species)

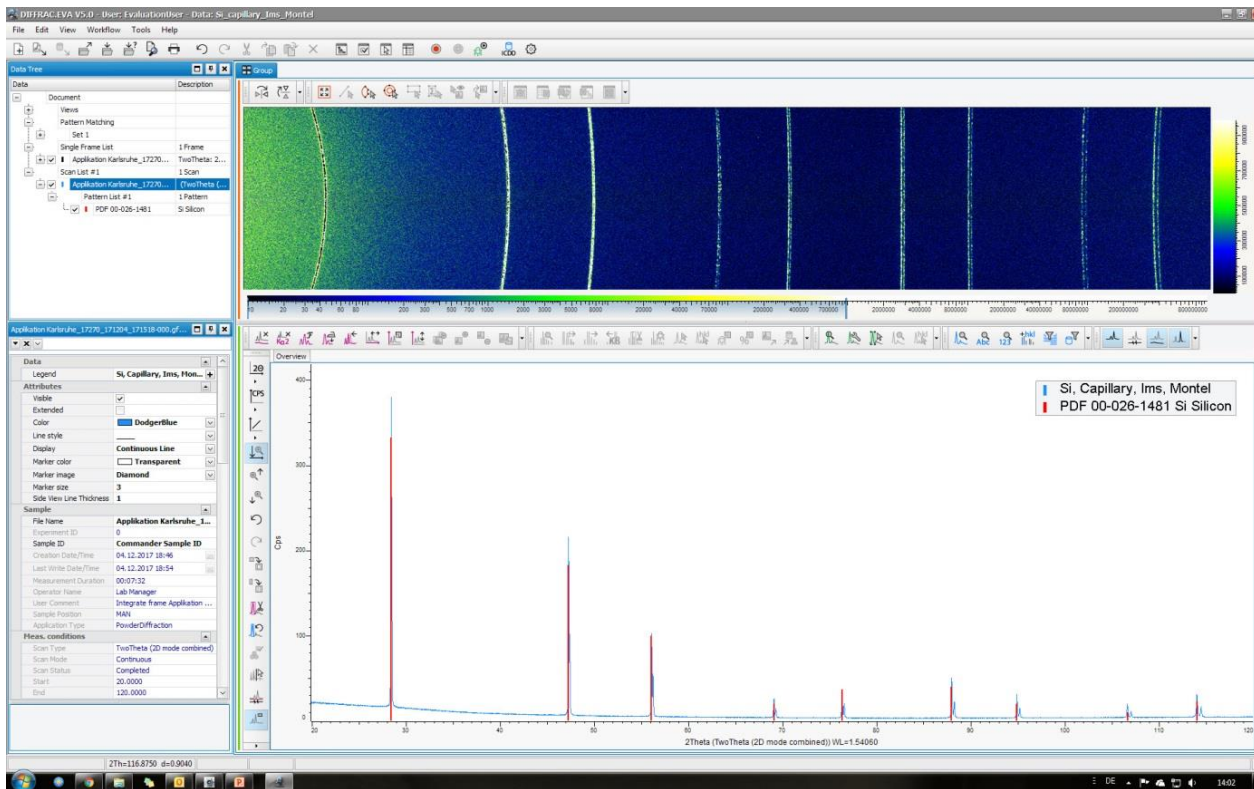


Fig 1. Example for an EIGER2 R 500K panoramic 2D continuous-scan image and integrated 1D data from a Si sample, prepared in a capillary and measured with a Cu micro-focus source and focusing Montel optics.

a highly complex task. DIFFAC.EVA provides all the tools that are required to follow a species identification protocol that is naturally structured along common best-practice guidelines:




In a first step the background is automatically subtracted. No further user interaction is required. Many users appreciate that EVA is the only third generation Search/Match software on the market. They enjoy the comfort that peaks do not need to be defined before the actual phase ID starts. Those who prefer second generation phase ID using beforehand determined peaks can do so as well.



The process of phase ID is divided into five logic steps that structure the process most naturally. This

is what we call SIP-X, Species Identification Protocol for X-rays. In a nutshell SIP-X comprises of the following steps:



SIP-X goes beyond the traditional Search/Match. The entire implementation has been restructured based on a thorough analysis of user behavior when performing phase identification. The result is a strictly workflow driven implementation.

 <p>1 Condense</p>	<p>Complexity reduction to reduce the size of the search space. The related tools are <i>Database Filter</i>, <i>Chemical Filter</i> and <i>Match Lists</i>.</p> <p>New 2018:</p> <ul style="list-style-type: none"> <li>• The tools for database and chemical filters were separated from the search tool</li> <li>• Improved filter defaults and administration of filters. All filters can easily be stored, edited and applied to new projects.</li> <li>• Filters act locally at the scan level. However, they can be made globally available and used conveniently as preset for future analysis.</li> <li>• Filters were added to the global settings to choose customer specific presets.</li> <li>• Support of PDF4 AXIOM database</li> <li>• Support of new ICDD data base subfiles, availability of RIR values, crystallographic information for the database filter</li> </ul>
 <p>2 Identify</p>	<p>Searching the database and matching candidates against the scan data for scoring. Available tools are <i>Search/Match</i>, <i>Search by Name</i>, <i>Search on Match List</i>, and <i>Auto Search</i>.</p> <p>New 2018:</p> <ul style="list-style-type: none"> <li>• Simplified GUI. The search tool has been redesigned with minimum number of mouse-clicks to result in mind.</li> <li>• Extended use of Match-List functionality. Those lists contain already identified candidates for typical materials like ores or products and byproducts of some typical chemical reactions.</li> <li>• Match Lists and User Databases provide the strongest reduction of the search complexity. Therefore, they provide the best source for <i>Auto Search</i>, a fully automated run through steps 2 to 4 of SIP-X.</li> </ul>
 <p>3 Select &amp; Complete</p>	<p>Evaluation and selection of the best-scoring candidates from the Search List and Completion of the search on the unexplained regions (residual scans) until the entire scan is explained. Related tools are: <i>Search Table</i>, <i>Database View</i>, <i>Scissors</i>, <i>Residual</i>, <i>Auto-Residual</i>, <i>Wavelength Cursor</i>, <i>Search Peaks</i>, <i>Elemental Analysis</i>.</p> <p>New 2018:</p> <ul style="list-style-type: none"> <li>• <i>Auto Search Table</i>. This new results table provides fast search list with configurable column layout, background indexing of the results for quick sorting and full-text search on the solutions. The table is completely sort and searchable.</li> <li>• Copy and paste functionality for working with <i>Match Lists</i></li> <li>• <i>Group by Column</i> and <i>Group by Box</i> adds new view possibilities to the Auto Search Table that allows for more efficiently data mining the PDF databases</li> <li>• Automatic <i>Database View</i> of the search entries</li> <li>• Improved identification of minor and trace phases by introducing a new <i>Search-Peak</i> algorithm. It puts higher sensitivity to the unexplained intensities</li> <li>• Push-button <i>Auto Residual</i> scan calculation</li> <li>• <i>Wavelength Cursor</i> for the visual identification of contaminant peaks that originate from characteristic K-beta and Tungsten L radiation</li> <li>• An icon bar at the bottom of the search list provides quick access to frequently used commands related to Search lists and residual search.</li> </ul>

	<p>All selected phases are automatically quantified, based on database or user supplied RIR values (Semi Quantitative (SQ) analysis) and automatically determined scale factors. Other related tools/features are: <i>Auto Scale</i> and <i>Internal Standard</i> for amorphous phase quantification.</p> <p>New 2018:</p> <ul style="list-style-type: none"> <li>• SQ analysis using profile fitting of the full pattern profiles instead of stick patterns</li> </ul>
	<p>The final step of the protocol involves generation of charts, printing and export of results. Related tools are <i>Pattern Column View</i>, <i>Pattern Chart View</i>, and <i>Print Preview</i>.</p> <p>New 2018:</p> <ul style="list-style-type: none"> <li>• Optional export of species and concentrations to instrument database</li> <li>• Direct printing</li> </ul>

### Profile Analysis

EVA5 provides profile fitting capabilities. It uses analytical profile shape functions (Gauss, Lorentz, symmetric and split Pseudo-Voigt peaks) together with a Chebychev polynomial for modelling the background. The software can cope with individual

peaks as well as entire patterns of peaks. This allows using them for semi-quantitative phase analysis. Furthermore, the crystallite size can be calculated from the full width at half maximum applying the Scherrer formula (e.g. LC determination of anodes according to ASTM D.5187).

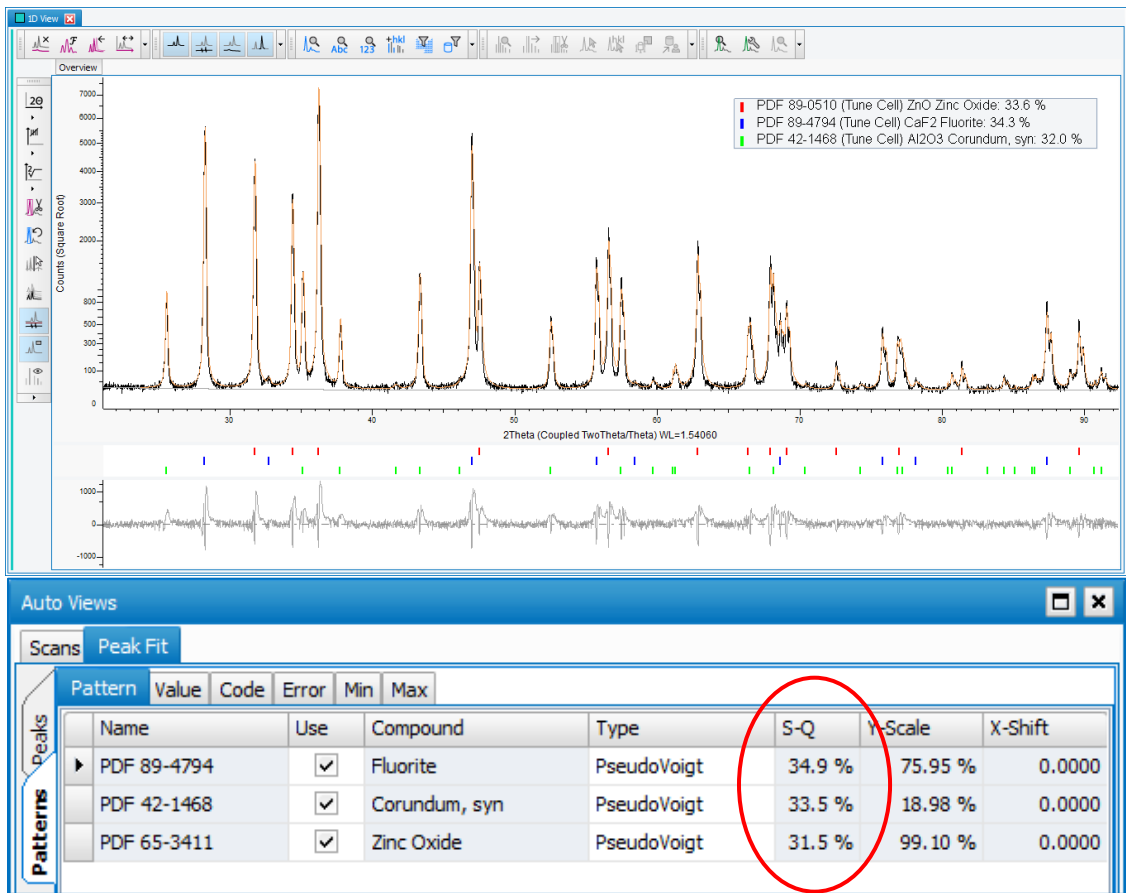


Fig 2. Pattern fit for semi-quantitative phase analysis.

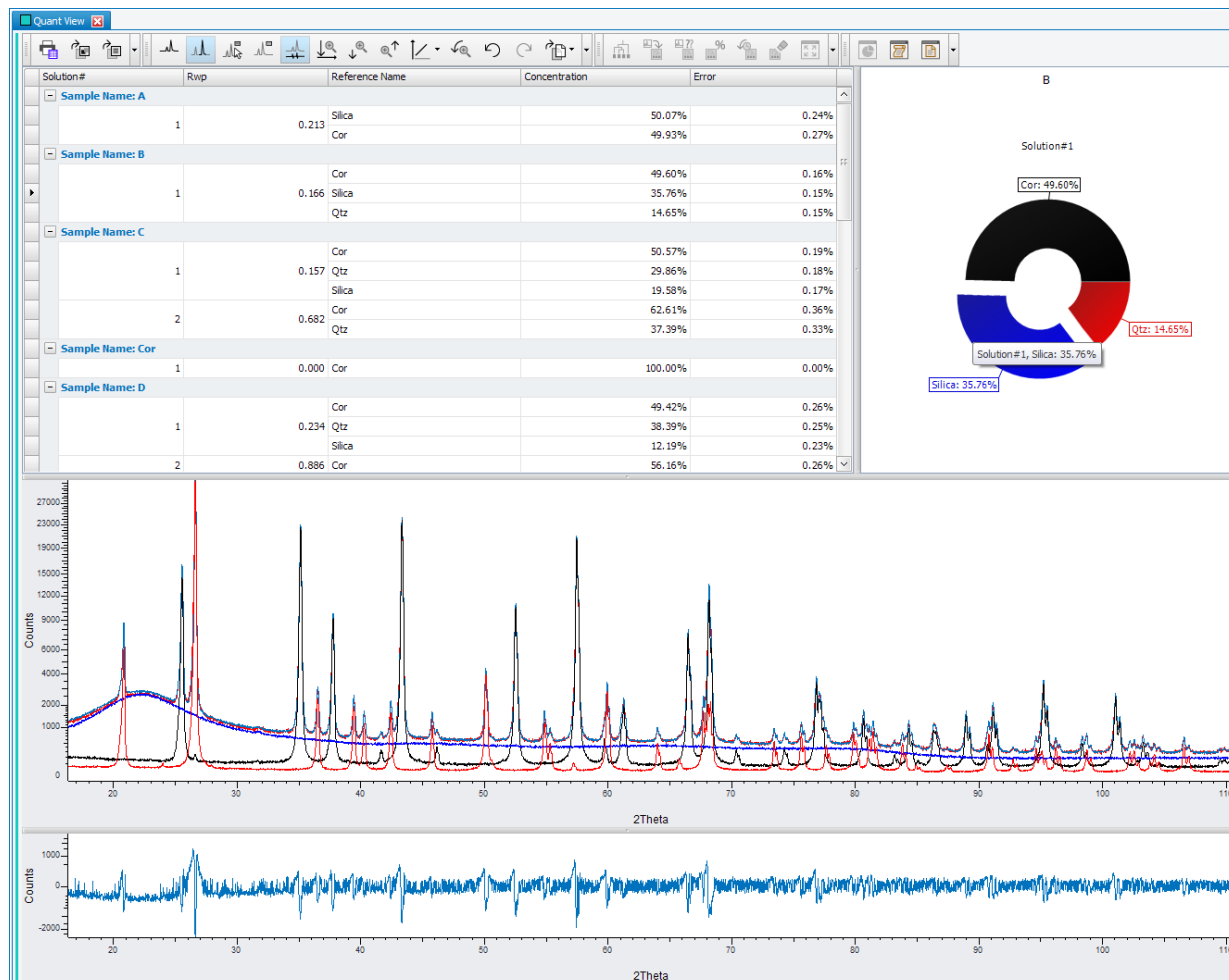
## SQUALL, Semi-Quantitative Full Pattern Matching

The program SQUALL performs database searching and semi-quantitative analysis on mixtures using a reference database of powder diffraction patterns (full scans) or spectra supplied by the user. It uses the full data profile and not just individual peaks. This has advantages in cases where:

- Users cannot or do not want to evaluate data in detail but a comparison to reference scans is sufficient
- No satisfying analytical description of the peak shapes is available. This can be the case for materials that exhibit stacking faults, e.g. clay minerals.

SQUALL overcomes a fundamental problem in powder diffraction, the correlation of the individual components (the references) that form a mixture. This is achieved by modern subset-selection regression techniques. These are: correlation pre-filters, ridge regression, forward selection and backward elimination and partial least squares (PLS). In SQUALL they are combined in a flexible, modular way to perform both, the database search and the subsequent semi-quantitative analysis.

SQUALL projects are defined and tested in the graphical user interface of EVA. This also allows fully interactive analysis of unknown samples. Furthermore (like quantitative analysis with DIFFRAC.DQUANT), those projects may also be run on batches of scans. SQUALL expert users have the full flexibility while an unexperienced operator cannot do any harm to the evaluation recipes.



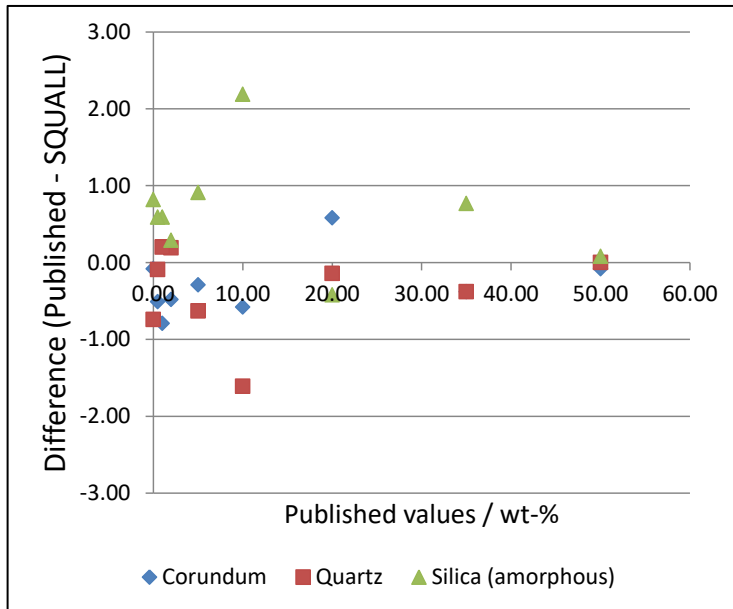


Fig 3. The SQUALL benchmark for an amorphous phase determination shows agreement better than 2 wt% over a wide concentration range. Data from Madsen et al (2011) Z. Kristallogr. 226, 944-955.

### Database Connection

Results from peak fit (peak properties, crystallite size and concentrations) as well as other area- and pattern-properties can be stored in the instruments database for subsequent reporting using the results manager. The results manager offers not only charts

(Pi, horizontal and vertical bar charts for absolute and relative data) but also trend charts for statistical process control (SPC) including floating average, standard deviations and limits. Also, numeric data can be shown together with limit control visuals for process monitoring.

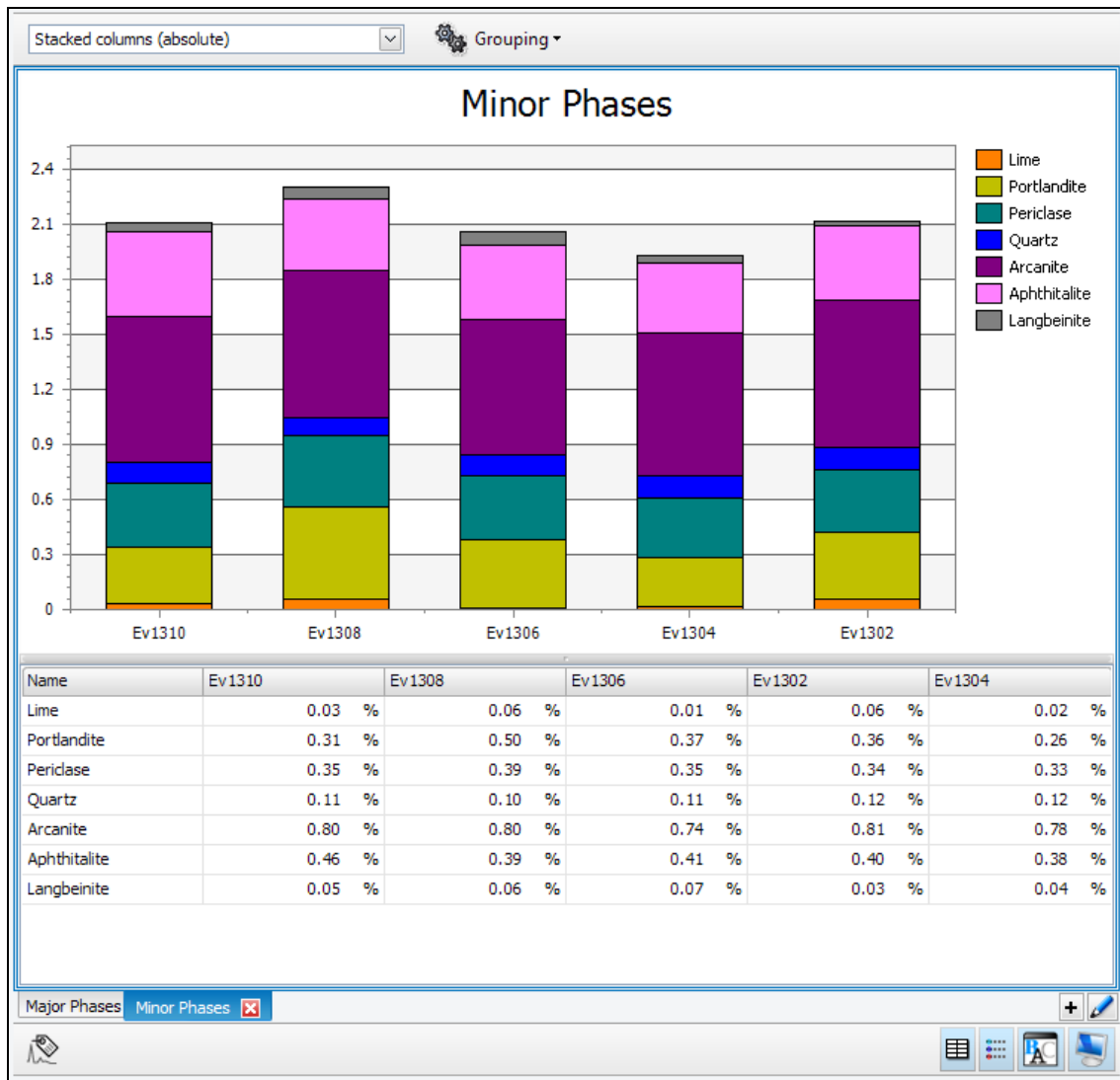


Fig 4. Example of a multi-sample reporting chart of the Results Manager. It shows a stacked column plot for minor phases of 5 different clinker samples.