UniQuant®5

Let's compare...

Peak-based versus Scan-based

Ever since WD-XRF spectrometers were introduced in the fifties, for quantitative analysis, intensities have been measured at fixed spectral positions. XRF lines are measured at their peaks whilst backgrounds are measured at interference-free off-peak positions. The latter can usually be pre-determined because each application is dedicated to a particular family of samples with a known set of elements, usually 10 to 35 elements.

In the late nineties, methods were introduced for analyzing totally unknown samples in which any of 79 elements may be present at a detectable level. One of the problems that had to be solved was that of background measurements, as this was not anymore a matter of measuring at pre-defined off-peak positions. With totally unknown samples, there is not even a single spectral position that is not potentially interfered by one or more XRF lines. In addition to the lack of pre-definable background positions, there is the huge problem of potential spectral interferences (line overlaps) of the analyte XRF lines themselves. Depending on selected spectral resolution, the total number of potential line overlaps is 1500 to 3000.

We saw two directions of development. All newly developed programs, except one, were based on fine step scanning of the entire spectrum. They are scan-based as opposed to peak-based. The exception was the UniQuant program that prescribes intensity measurements at up to 115 predefined spectral positions, all being analytical XRF line positions. UniQuant is peak-based as are all conventional quantitative WD-XRF programs.

At first sight, scanning of the entire spectrum is the most obvious approach since one would expect it to solve the problems of background and line overlap corrections with totally unknown samples. In reality, the scanning method poses its own set of problems. The most obvious one is that times spent at the analyte XRF lines are so short that elements may remain undetected when their concentration is less than 50 ppm. Lower limits of detection and determination are an order of magnitude larger than with measurements at fixed positions. Line overlap corrections are not as straight forward as one would expect. None of the scan based methods solves the problems rigorously and trace analysis is often unreliable.

To partially solve the trace analysis problem, some programs had later added the facility of measuring trace elements at their peak positions, a kind of hybrid solution. However, this does not work well with totally unknown samples, since the analyst does not know which trace elements are present or absent to start with.

Comparison of peak-based methods

From the start in 1989, UniQuant used several entirely new concepts. These addressed the problem of background measurements and line overlap corrections without the need of time consuming scanning. In the years that followed, the algorithms have been so much improved that trace analysis is now of the same quality as with conventional methods, the difference being that with UniQuant, unexpected trace elements are also determined in totally unknown samples. UniQuant calculates the background continuum taking into account any jumps that are due to absorption edges. Reliable and accurate trace analysis is a big challenge in X-ray spectrometry, if not the biggest. Sometimes, the best algorithms cannot compete with visual inspection. For this reason, UniQuant 5 offers full support by graphics, which is useful for critical trace analysis when setting up dedicated applications. Graphics also includes a breakdown of the gross peak of each analyte line into continuum, spectral impurity, line overlaps and net peak, all expressed in ppm, which is far more appealing than expression in cps.

Peak-based programs do not only differ in handling trace elements. There are also differences in the method of calculation of concentrations and in flexibility, such as for dealing with non infinitely thick samples down to thin layers, see Table of Comparison.

Table of Comparison

	UniQuant® 5	Conventional programs
Standardless mode included (for up to 79 elements)	✓	
Regression analysis when using standards	✓	✓
Few standards are sufficient in all cases	✓	
Good quality of trace analysis	✓	✓
Trace analysis with totally unknown samples	✓	
Line overlap corrections. All 1200+ line overlap coefficients are pre-calibrated for use in any application	✓	
Passes 'acid test' of many mutual line overlaps with mixture of lanthanide elements	✓	
Interelement corrections by using X-FP = Extended form of Fundamental Parameters MM = Mathematical models, using first and higher interelement correction coefficients	X-FP	MM
Need for a theoretical alphas program for setting up a new application (calibration)		✓
Mass (thickness) of samples may vary (beads and light matrix samples are not 'infinitely thick' for many XRF lines)	✓	
Variable (catch) weights for sample and flux	✓	✓
Variable (catch) weight for liquid samples	✓	
Determination of LOI or GOI (Loss or Gain on Ignitions with beads)	✓	✓
Determination of mass/area (thickness) and composition of mono-layer, possibly on a fluorescent substrate.	✓	
Each result is reported with a practical confidence interval	✓	
One single monitor program for drift correction serves all calibrated analytical programs	✓	
Determination of user defined compounds	✓	