

The Promises when WDS Supports the EDS X-ray Analysis in SEM and the Evaluation Algorithms do Merge

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The electron beam X-ray microanalysis is performed with two base kinds of spectrometers, EDS and WDS. Despite using the same quantification models in principle (because only the X-rays detection system is different), the two scientific communities drove both technologies forward, but along quite different paths. It is true that both spectrometer types have specific advantages [1] and the last decade development in EDS-SDD technology has pushed EDS towards WDS precision and accuracy [2,3]. But is then WDS still required? It maintains superiorities with a better base energy resolution, which separates X-ray lines which cannot be resolved by EDS [1]. This advantage also in principle produces higher P/B values, a benefit in trace element analysis with the detection limits being about a factor of 10 lower than normal EDS detection limits.

The EDS/WDS in SEM was promoted by several vendors to be a combined method, unifying the use and access in commercial software. When a dedicated EDS spectrum energy area needs a more detail view, a WDS measurement for that selected energy range (the spectrum “magnifying glass”) can support element identification or assess element ratios where the EDS spectrum is bad resolving. It is a straightforward and common way that a standards-based quantification can be performed with measurement of net-counts based k-ratios between the unknown specimen elements and standards. The EDAX eZAF-FSQ method does this as well [4], following the classical strategy based on k-ratios measurements of X-rays. And it makes no basic difference whether the k-ratios data were obtained from EDS-spectra or from WDS spectrometer.

Ideas were already mentioned about how the WDS spectrometer data can be really used to merge both technologies into the usual analytical algorithms for EDS spectra evaluation [5]. As an example, the automatic element identification algorithm EXpertID for EDS spectra [6] is not able in some cases to decide whether the element-lines are not present in spectrum, in cases it is not good enough resolved from another element major line and also corresponding lines are not available to decide (e.g., about Si if W is strong in sample, about S if Mo or Pb are much, etc.). In those cases when the element cannot be excluded, the EXpertID algorithm keeps the element in the list with the aim that the final quantitative deconvolution and results error determination can finally decide whether the element is perhaps below the detection limit. But depending on heights of overlapping lines, partially the errors are high, and the qualitative analysis decision uncertainties are then as well. But these cases could trigger a dedicated WDS measurement in this energy region to clarify the qualitative analytical result.

But it is obvious there are deconvolution challenges also in WDS spectra (Fig. 1). We use now the same Bayesian probability theory deconvolution algorithm [7,8], also for WDS spectra, even with identical line-series database [9]. Finally, the EDS Bayesian deconvolution algorithm is used with the ability to consider boundary conditions. The measured WDS line-ratios can be used for the EDS spectrum deconvolution. This is then even possible with standardless Quant, utilizing WDS results (WDS really standardless used). Another way is that WDS based determined weight fractions (e.g., from traces) can be used for EDS spectrum processing and evaluation with internal simulation [9] of the peaks. They are

then known EDS spectrum components and considered similar ways, like the escape-peaks and the pile-up is subtracted before the deconvolution performs. The new ways, to utilize outside obtained data and results for the benefit of the pure EDS spectrum evaluation, it was first time already applied for EDS/ μ XRF method coupling [10], now it used for combined EDS/WDS.

As an example, the quantification is affected by challenges with the used deconvolution algorithm, if many line series do overlay and everything is not well resolved. Fig. 2 shows a well-known case, a challenge for EDS, but with the corresponding WDS spectrum the lines are much better resolved. Indeed, the EDS deconvolution algorithm is able, to handle the example in almost all cases, but it remains the question about small concentrations or even trace elements. The element Pm was analysed with pure EDS spectrum evaluation to be with more than 1%. But the WDS better resolved detail view shows it is likely not present. The standardless EDS spectra evaluation, deconvolution supported by WDS, provides then below 0.1%, more than one order of magnitude less. Pr was not identified in EDS spectrum auto-ID (with used sensitivity), but there is clear evidence with WDS spectrum, at least obvious with well resolved Pr-L β line and the concentration is about 3% finally.

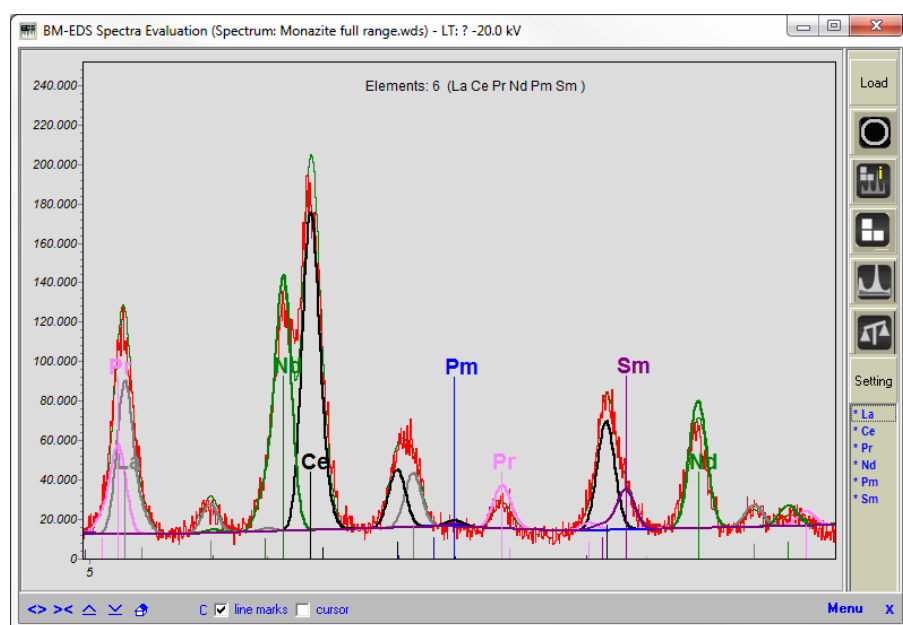


Figure 1. The individual element line-series results after Bayesian deconvolution was also applied to WDS spectra, only the estimation about resolution was changed. It is a real deconvolution approach based on probability-theory, not a parameter fitting. A linear background subtraction was applied in this example.

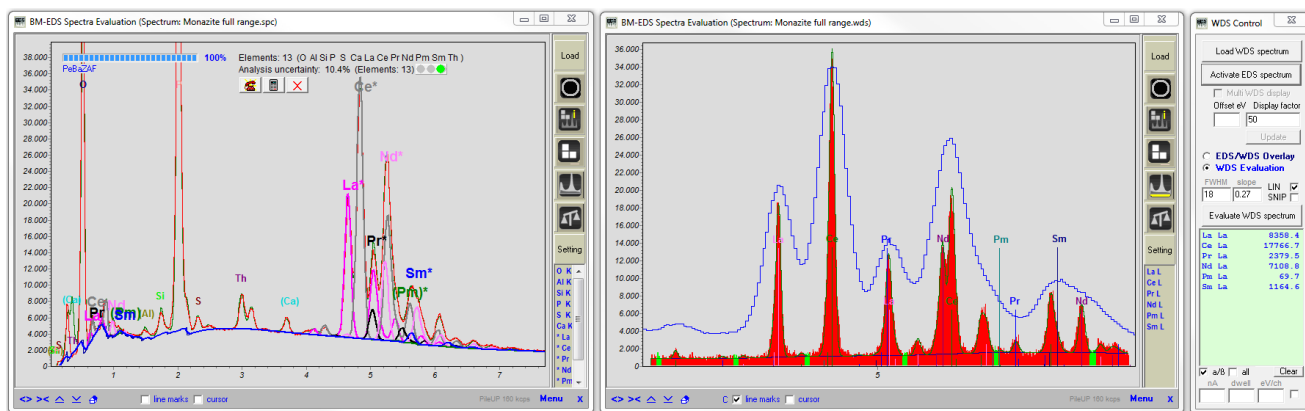


Figure 2. Full standardless parallel evaluation of EDS spectrum from a Monazite specimen (left) and WDS spectrum from same but with a selected energy range (right). The WDS measured net-counts were obtained with an automatic element identification and with Pm manually added. Bayesian deconvolution was also applied for the WDS spectrum. The element symbols with * in EDS spectrum mark that these were supported in deconvolution by the WDS measured net-counts.

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