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High Count Rate Standardless and Standard-Based Quantification in EDS – Practical Limits and Root Causes for Deviations in the Result

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With the introduction of SDDs, their extremely high count rate capability gave rise to very high speed, but low energy resolution elemental mapping and has been accepted as a major advantage for analyzing microstructures [1]. As pulse processing methods improved, the stability of optimum resolution even at high count rates has shown the additional benefits of SDDs. Additionally, developments in SDD technology have resulted in superior light element performance compared to Si(Li) detectors. Recent questions ponder over the benefit of having best resolution up to such high count rates of 100 kilo counts per second (kcps), when quantification is always performed at low count rates of around 5,000 cps. This is the question this group is focused on answering, as well as elaborating on the practical limits and root causes for deviation in quantification results at high count rates.

It is known, when performing qualitative and quantitative analysis with Si(Li) detectors, that dead-time must be kept below 30% [1], and this is generally still true for SDD detectors. However, modern hybrid pulse processing techniques, adapted to the SDD's unique characteristics, allow much lower dead time for a given resolution, or have a superior resolution for a given pulse shaping time. This allows quantitative analysis at much higher count rates than previously thought possible.

When performing a standard based φ(ρz) quantification on iron oxide minerals of hematite (Fe_2O_3) and magnetite (Fe_3O_4), the results only vary negligibly when comparing spectra with input count rates of 5.8 kcps and 27 kcps. This can be explained by the cancelling of the effects of pile-up and peak/background (P/B) ratio deterioration – P/B reduces significantly as dead-time increases – this is due to an increased background (due to background pile-up) and a peak intensity reduction (as coincident x-rays of that line end in the sum peak). However, the challenge in standardless quantification is greater, as the P/B ratio is used to calculate the relative quantities of each element. However, the standardless P/B ZAF quantification results show a result very close to stoichiometry at low count rates and deviate less than 3% at 27 kcps, fitting the accuracy expectation of standardless analysis [2]. Dead-times for the above conditions were about 5% and 28% respectively.

The rule for using less than 30% dead-time still applies, but with hybrid signal processors, the maximum count rate for quantification at best resolution (as low as 123eV @ Mn Kα) can be pushed up to 30 kcps input count rate without large deviation in quantification results. Moreover, when utilizing multi-channel detectors, these count rates can be multiplied by the number of channels. Hence the ability to collect, correctly identify and quantify with minimal deviation, has been made possible for count rates exceeding 120,000 cps. This brings new possibility for super short spectra acquisitions and true quantitative mapping applications with precise and accurate results.

References

[1] J.I. Goldstein et al., *Scanning Electron Microscopy and X-ray Microanalysis*, Plenum, New York, 2003.

[2] D.E. Newbury, *Microsc. Microanal.* 8 (Suppl. 2) (2002) 1464CD

TABLE 1. Results from 9 measurements at. % hematite, magnetite (standardless P/B ZAF).

Fe_2O_3	**O**	**Fe**	Fe_3O_4	**O**	**Fe**
Mean:	60,6	39,4	Mean:	57,7	42,3
Recovery	101,0	98,4		101,1	98,6

15kV, 1.7nA, **5.8kcps**, 43s

TABLE 2. Results from 9 measurements at. % hematite, magnetite (standardless P/B ZAF).

Fe_2O_3	**O**	**Fe**	Fe_3O_4	**O**	**Fe**
Mean:	59,2	40,8	Mean:	56,2	43,8
Recovery	98,6	102,0		98,3	102,2

15kV, 10nA, **28kcps**, 9s

TABLE 3. Results from 9 measurements at. % hematite, magnetite (standard-based φ(ρz)).

Fe_2O_3	**O**	**Fe**	Fe_3O_4	**O**	**Fe**
Mean:	60,0	40,0	Mean:	57,1	42,9
Recovery	100,0	99,9		100,0	100,0

15kV, 1.7nA, **5.8kcps**, 43s

TABLE 4. Results from 9 measurements at. % hematite, magnetite (standard-based φ(ρz)).

Fe_2O_3	**O**	**Fe**	Fe_3O_4	**O**	**Fe**
Mean:	59,8	40,2	Mean:	56,8	43,2
Recovery	99,6	100,6		99,4	100,9

15kV, 10nA, **28kcps**, 9s

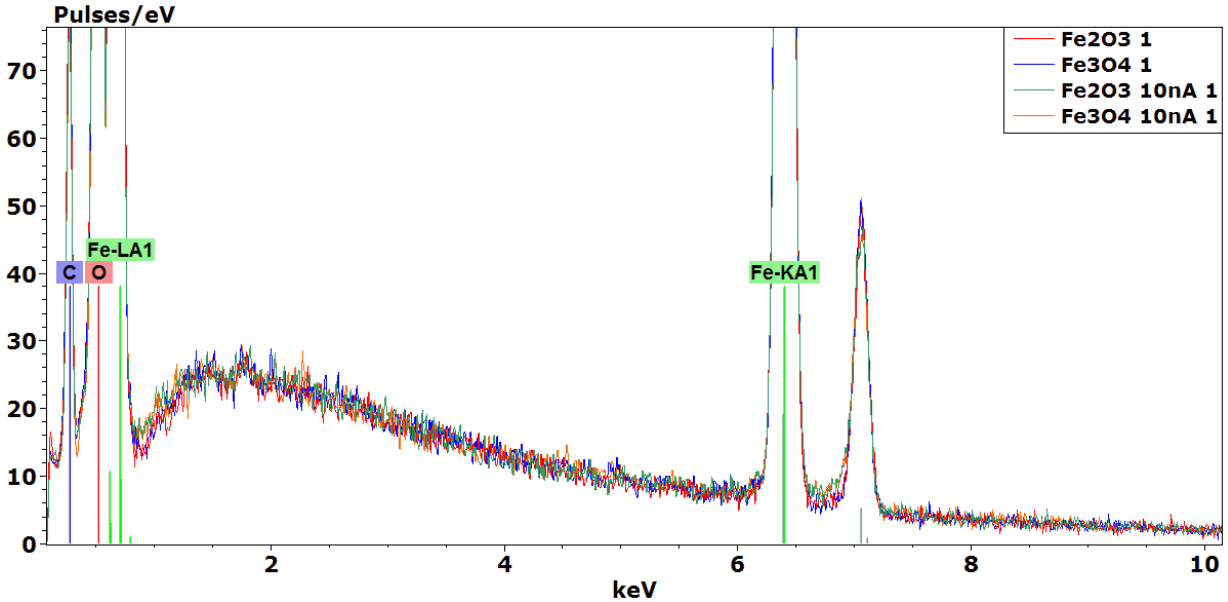

FIG. 1. Spectra overlay of hematite and magnetite spectra with 1.7nA and 10nA, with some indication of pile up on the high energy side to the iron Kα and Lα lines at 28 kcps.

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