**Technical Note** 

**1N-002** 

# Pitfalls of Energy-Dispersive X-Ray Spectroscopy: Dead time and Peak Artifacts



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Energy-dispersive x-ray spectroscopy (EDS) in the scanning electron microscope (SEM) or the electron microprobe is a very useful analytical method to rapidly characterize the chemistry of unknown samples; However, there are some characteristics of how the silicon drift detectors detect and process x-rays that are useful to understand in order to be able to interpret EDS spectra.

#### Dead time

The silicon drift detector pulse processor electronics take a finite amount of time to process an incoming x-ray. With a small x-ray flux (e.g., at low beam currents) this usually has little effect on the processing of x-rays and the incoming x-ray count is more or less equal to the processed x-rays. As the x-ray flux increases there will be more periods of time that the pulse processor is busy and cannot register incoming x-rays. This time during which the pulse processor cannot process x-rays is known as dead time and is often expressed as a percentage of the total count time (Fig. 1).

Figure 1. Dead time indicated as a percentage (under these conditions the detector cannot process x-rays 15% of the time).



## Sum peaks

When two x-rays hit the detector at exactly the same time the detector pulses pile-up and the detector may be fooled into thinking they are a single x-ray. The detector will process these x-rays as a single x-ray with an energy equaling the sum of the individual x-ray energies. This phenomenon leads to the formation of sum peaks. Sum peaks are more prevalent at high incoming x-ray count rates. For example, if two Si K $\alpha$ x-rays are detected at the same time, we will see a peak form at 3.48 keV (1.74 keV + 1.74 keV). Figure 2 shows the EDS spectrum collected from quartz (SiO2) at a high count rate on the EPMA.

If you see a peak in your spectrum, particularly at high count rates, that cannot be identified, it is likely you have a peak artifact (it is unlikely you have discovered a new element!).



Figure 2. EDS spectrum of quartz (SiO2) showing sum peaks.

### Escape peaks

Incoming x-rays may have the energy to generate Si x-rays within the silicon drift detector. When this occurs, the detector will appear to detect an x-ray with the energy of the incoming x-rays minus the energy of the excited Si x-ray (1.74 keV in the case of Si K $\alpha$ ) (Fig. 3). For example, if a Ni K $\alpha$  x-ray excites a Si K $\alpha$  x-ray in the silicon drift detector, the pulse processor will register an x-ray with an energy of 5.707 keV (i.e., 7.447 keV – 1.740 keV). With most modern EDS software, the sum and escape peaks will be automatically labelled or removed.



Figure 3: EDS spectrum of rutile (TiO<sub>2</sub>) showing Ti-Si escape peak.

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