Improved Trace Element Sensitivity with the Apollo XRF ML-50 Silicon Drift Detector on the Orbis Micro-XRF Elemental Analyzer

Materials Challenge

The measurement of trace elements is important across a wide variety of materials characterization problems. When measuring small glass fragments collected from crime and accident scenes, forensics experts analyze trace strontium (Sr) and zirconium (Zr) typically unintentionally incorporated into the glass during manufacturing as one point of identification or comparison. Traces of lead (Pb) and cadmium (Cd) are measured in a wide variety of commercial materials for verification of adherence to environmental regulations such as the European Union's Restriction of Hazardous Substances (RoHS) directive. Trace chlorine (Cl) is depth profiled in concrete to quantify ion permeation from deicing agents. In these cases and many others, improvements in detector performance can lead to improvements in micro-XRF sensitivity and faster data collection, thereby providing users with faster, more accurate results.

Orbis Detectors

Over the last decade or so, XRF detector developments have moved towards electrically cooled Silicon Drift Detectors (SDDs) providing comparatively better spectral resolution at significantly higher throughput rates. These detectors operate at temperatures much higher than Si{Li} detectors. Therefore, SDDs can be cooled by electrically powered Peltier coolers, eliminating the need for liquid nitrogen, which was used to cool most Si{Li} detectors.

The Orbis micro-XRF analyzer can be configured with either a 30 mm^2 SDD (standard) or a 50 mm^2 SDD (optional):

Standard 30 mm^2 SDD	Optional 50 mm^2 SDD
Electrically cooled	Electrically cooled
Good active area	Largest active area
Thin 8 µm Be window	12.5 µm Be window
High spectral resolution	High spectral resolution
High signal throughput	High signal throughput

The Apollo XRF-ML-50 detector is an SDD with improved active area yielding a larger solid angle of signal collection while maintaining high spectral resolution and throughput. This allows for improved overall sensitivity and measurement speed in many applications.

Orbis Micro-XRF Results

Improvements in speed and sensitivity of analysis can be achieved with detectors and associated signal processing electronics by improving spectral resolution, enhancing signal collection by size and positioning of the detector or increasing signal processing speed, also referred to as "throughput". The general goal in optimizing and improving these detector parameters is to avoid augmenting one at the sacrifice of the others. For example, it would do little good to increase the detector size while making significant sacrifices in detector resolution or throughput. Such a detector could collect more signal but would be unable to provide a high resolution spectrum or process the additional signal collected. This application note will explain the benefits of the 50 mm^2 Apollo XRF-ML50 detector and describe in which analytical situations increased speed and sensitivity can be realized.

Improvements in speed and sensitivity begin with the amount of sample X-ray signal collected by the detector, which is referred to as the "solid angle" by system designers. Detector solid angle is proportional to the signal collection area of the detector, known as active area, and inversely proportional to the square of the distance between sample and detector as shown in Equation (1).

Detector Solid Angle α (Active Area)/(Distance)^2 *Eqn [1]*

In general, augmenting active area leads to larger solid angles and increased X-ray signal collection as long as the distance between sample and detector can be optimized. In Table 1, a comparison of performance parameters is made between the two detectors which are available on the Orbis system.

Detector	Active Area	Type	Resolution [eV]	Throughput [kCPS]
Apollo XRF-ML30	30 mm^2	SDD	≤ 135	200
Apollo XRF-ML50	50 mm^2	SDD	< 137	200

Table 1. Comparison of Orbis Detector Structural and Performance Parameters

The structural and performance characteristics of the 30 mm^2 and 50 mm^2 SDD detectors are very similar except for the active area.

The additional solid angle of the 50 mm^2 detector yields improvements in speed and sensitivity in analytical problems where the 30 mm^2 detector would be signal starved. When a detector is signal starved, the detector's processing electronics still have available capacity to process more signal. In these cases, having a detector with a greater solid angle allows for more X-ray signal to be passed to the processing electronics. As long as the larger SDD's spectral resolution and throughput are similar to that of the smaller SDD, which is the case here, improvements in speed and sensitivity can be achieved with the larger detector. Typical materials analysis problems which fall into this category include measurements of light element matrices such as glasses, plastics and aluminum alloys; measurements of trace elements where heavier filters are needed for best sensitivity such as trace Pb and Cd in solders and plastics and measuring thin residues, corrosion and coatings on light element matrices. Table 2 compares the limits of detection for several elements spanning the XRF spectral range.

For the data collected in Table 2, the SDD detectors were operating in the signal starved regime where the X-ray tube is run at full power for maximum excitation. Improvements in sensitivity of the 50 mm^2 over the 30 mm^2 SDD are approximately consistent with the increase in solid angle of the 50 mm^2 over the 30 mm^2 SDD.

Detector	Std/Options	Cl(K)	Pb(L)	Sr(K)	Cd(K)
Apollo XRF-ML30	Standard	17	3		9
Apollo XRF-ML50	Option	14	2	5.5	8
Sample Matrix		Glass	Plastic	Glass	Plastic

Table 2. Limits of Detection [ppm] for Detectors Available on Orbis

In applications where improved sensitivity is not needed, the 50 mm^2 SDD can be used for increased productivity. The proportionality between sensitivity as described by the Limit of Detection [LOD] and measuring time is shown in Equation [2].

$$\text{LOD} \propto 1/(\text{Time})^2 \qquad \textit{Eqn [2]}$$

Hence, the increased sensitivity provided by the 50 mm^2 SDD can be traded for faster measuring time. For example, using Eqn [2] and the improvements in measuring sensitivity shown in Table 2, the 50 mm^2 SDD can achieve the same measurement sensitivity obtained with the 30 mm^2 SDD with a nominal 40% reduction in measuring time.

The additional solid angle of the 50 mm^2 detector yields improved sensitivity and faster measuring times in materials analysis where the 30 mm^2 detector would be signal starved, but not in cases where the 30 mm^2 detector is saturated. Saturation refers to when the detector's rate of signal collection exceeds the throughput of the analyzer electronics. Hence, sensitivity and measurement speed are limited by throughput. In this analytical situation, the X-ray tube is generally running below maximum power limiting the input XRF signal to the analyzer throughput. Typical materials analysis problems which fall into this category include measurement of major elements in steels, heavier transition metal alloys and measuring thin residues, corrosion and coatings on substrates comprised of heavier transition metal alloys. Having equal throughputs, both the 30 mm^2 and 50 mm^2 SDDs provide similar speed and sensitivity for samples which can saturate the detectors; however, the 50 mm^2 SDD allows for a greater reduction in X-ray tube power, which in turn can improve tube life.

Recommended EDAX Solution

The Orbis micro-XRF analyzer with a 50 mm^2 SDD detector is recommended for engineers and scientists who require the best overall detection limits or faster measurements from a non-destructive elemental analysis technique for measuring small samples or distributional analysis on larger samples. The Orbis with a 30 μm poly-capillary exciting optic can provide highest overall sensitivity on an extremely small measurement area which would be suitable for example in analyzing small glass and metal fragments or electrical contacts in electronics. In addition, the Orbis can be equipped with optional 1 mm and 2 mm collimators to provide measurements over larger sample areas where measurements with a finer spatial resolution are not necessary or where there is a need to properly average the effects of sample inhomogeneity which occurs, for example, in low gold alloys, traces in plastics, and mixtures of particles of varying composition.