

Determination of the Effective EDS Detector Area Using Experimental and Theoretical X-ray Emission Yields

Mathias Procop^{1,2}, Vasile-Dan Hodoroaba¹ and Ralf Terborg³

¹ BAM Federal Institute for Materials Research and Testing, Division 6.8 Surface Analysis and Interfacial Chemistry, Berlin, Germany.

² present address: IfG-Institute for Scientific Instruments, 12489 Berlin, Germany.

³ Bruker Nano GmbH, 12489 Berlin, Germany.

An energy dispersive X-ray spectrometer operating with a semiconductor detector should be specified in compliance with the ISO standard 15632 [1]. Requirements for specification are: a) a general description of the spectrometer to evaluate its performance, b) the energy resolution with corresponding dead time, c) the P/B ratio in the Fe⁵⁵ spectrum and, finally, d) the L/K intensity ratio in a Ni or Cu spectrum to estimate spectrometer efficiency at low energies. Items b) to d) can be easily checked by the user. Related procedures are recommended in the annexes of the standard.

The solid angle Ω is another important parameter as this defines the collection efficiency. It is defined as: $\Omega = F / d^2$ with F being the active area of a spherical detector and d being the distance between the radiation origin and the center of the detector. Ω is not an intrinsic spectrometer property and can only be defined in combination with a specific SEM/EDX system. The detector area is one of the most interesting spectrometer properties since large area detectors have been available. Commonly, this area is given with the general description, but it is not specified. In the present paper a procedure was developed to check the detector area. The procedure is simple and can be applied by any spectrometer user.

Measured intensity (count rate in cps) I of any X-ray line depends on the X-ray yield Y defined as the probability of emission of a photon per incident electron and per steradian:

$$I = \Omega \varepsilon j_e Y A = \frac{F}{d^2} \varepsilon j_e Y A \quad (1)$$

ε denotes the spectrometer efficiency (counts per incident photon), j_e the beam current (electrons per s), and A the absorption correction to be applied for the given take-off angle. Y was determined by various authors for selected elements [2-5]. Equation (1) enables the determination of the solid angle from count rate measurements, known efficiency and absorption correction as well as published or calculated X-ray yields.

The detector-specimen distance d can be found from a simple experiment. Count rates have to be measured at different detector positions $d + \Delta d_i$, whereat Δd_i being the retraction distance. A plot of

$$\frac{1}{\sqrt{I_i}} = \frac{1}{\sqrt{F d I_i / d \Omega}} (d + \Delta d_i) \quad (2)$$

vs. $(d + \Delta d_i)$ gives the distance d . Fig. 1 shows an example. Knowing d , the detector area F can be calculated from equation (1).

The test procedure has been applied to 3 SDD and 1 Si(Li) spectrometers from different manufacturers. Selected specimens were Cu, Ti and Si, for which X-ray yields have been published [2-5]. Spectra were measured at 20, 10 and 5 kV SEM high voltages, respectively. Cu has the advantage of lowest uncertainty of the spectrometer efficiency ε as well as published X-ray yields Y . Beam currents were measured with a calibrated Keithley Amperemeter. Table 1 gives the results for the spectrometers tested until now. Determined areas are distinctly smaller than given in the spectrometer description. The reason for the differences is likely the aperture in front of the detector

crystal shadowing its outer region. F determined according to equation (1) is hence the “active” or “effective” area of the detector.

For three spectrometers the size of the aperture was known enabling a cross checking of the procedures accuracy. Determined areas F are within the $\pm 5\%$ range for all three elements when experimentally determined yield data from ref. [5] and in case of Cu also from ref. [4] are used.

Calculated areas F agree at best for Ti when theoretical X-ray yields are used. Because absorption and fluorescence corrections are small for the high voltages used, the selected model should not influence the result. However, calculated areas F depend on the selected model for the stopping factor, especially on the selected cross section. The new theoretical cross sections [6] were tested, but best results could be achieved with cross sections calculated with Casnati’s empirical formula [7].

In summary it was demonstrated that the effective detector area F can be determined by the described procedure. Associated measurement uncertainty is below 10%, if published experimental yields [5] for Si-K, Ti-K α and Cu-K α or calculated yields with Casnati cross section are used.

References:

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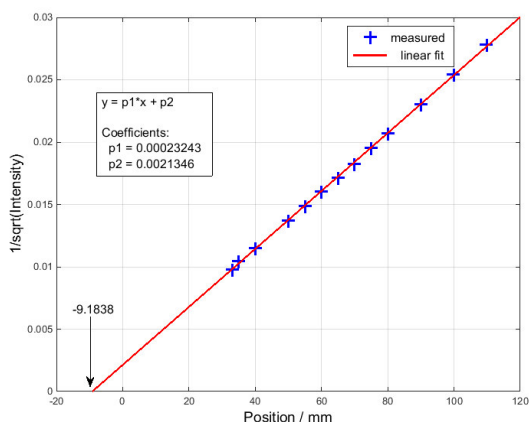


Figure 1. Determination of the detector specimen distance. At detector position 40 mm the distance amounts to $40+9.2=49.2$ mm.

Table 1: F in mm² for tested spectrometers

Detector	Si exp. Y	Si theor. Y	Ti exp. Y	Ti theor. Y	Cu exp. Y	Cu theor. Y
SDD 10 mm ²	8.5	8.8	8.7	8.5	8.8	8.1
SDD 30 mm ²	21.0	25.5	22.7	23.3	22.0	20.3
SDD 100 mm ²	77	79	75	76	79	73
Si(Li) 10 mm ²	9.2	9.4	-	-	10.1	10.8