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Instrumentation & Techniques

SXRF Spectroscopy of Reference Materials using White Light and Broad Bandpass Excitation

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Introduction

The VESPERS beamline 07B2-1 at the Canadian Light Source was designed to use bend magnet radiation to conduct simultaneous XRF and Laue X-ray diffraction experiments on near micrometre-size regions of a sample. Separate alternate optical pathways provide "white" radiation and monochromated radiation with different bandpass settings. Two sets of multilayer mirrors provide bandpasses of 1% and 10% and <111> silicon is used to provide an ultimate resolution of 0.01%. Using the 10% multilayer mirror a selective spectral region can be irradiated with a beam flux considerably higher than that typically used by other facilities with simpler optics. In addition, both optical pathways use mirror coatings that allow radiation with sufficient energy to efficiently excite the K and L lines for many elements in the periodic table. Excitation by white radiation is another attractive excitation mode, provided that any background radiation from reflection and Compton scattering can be kept acceptably low and compensated mathematically by background subtraction.

We have compared the use of 10% bandpass and white radiation in the XRF excitation of a number of differing sample matrices with known compositions. Spectral analysis has been carried out using the Science Studio software program Peakaboo. It is found that the elemental sensitivities with either mode are sufficiently high that spatial mapping of trace element distributions can be obtained in acceptably short intervals (1 – 2 h). White light excitation is considerably more intense and has few offsetting disadvantages in the lower energy range from 1 – 10 KeV that covers the K lines for elements from Na to As Above this energy, excitation using the 10% bandpass mirrors set in the 20 – 25 keV range, becomes competitive. Interference from pileup peaks is less and the excitation cross sections for L and K lines of the higher Z elements is greater. Using either mode, readily interpretable XRF spectra should be able to be acquired within a period of a few seconds to allow such elements to be detected in "maps" of their spatial distribution.

Science

As part of this study, standard reference material (SRM) glasses 610 and 612 and Montana Soil 611 prepared by NIST have been studied , along with a 6061 Al alloy and Alloy 600 that had been analyzed independently. Samples were mounted at an angle of 45˚ to the photon beam and to the SDD detector which was positioned at 3 cm from the sample.

In Figure 1, a white light-excited spectrum of NIST calcium silicate glass 610 is shown. Most elements were added to the homogenized glass at the 500 ug/g level. K line structures for each element are fitted according to literature sources but with the relative β/α intensity adjusted to our energy conditions using metallic reference materials. Using the present peak fitting algorithm in Peakaboo, most (but not all) of the overlaps between the elements present in the glass can be fitted successfully. The more complex L line spectra of the higher Z elements can also be fitted, but are better treated for reasons given above, using the 10% bandpass excitation mode. Figure 2 shows such a spectrum for excitation at 25 keV where K lines from Ge-Cd can be detected, along with L lines from W-U. Again, not all lines can be fitted simultaneously.

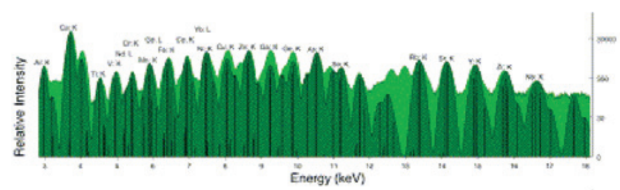

Figure 1: Logarithmic scale XRF spectrum of NIST SRM 610 glass (120 s) using VESPERS white light excitation. Most elements have concentrations near 500 ppm. A polynomial filter removes all but 4% of the background in most spectral regions. Spectral noise is reduced using a Savitsky-Golay filter. Only fitting for the K lines is shown.

The known (or expected) bulk concentration values in the reference glasses were used to develop sensitivity factors (S) for each element detected in SRM 610 for a standard collection time of 1 minute using the present VESPERS optical geometry. The K series calculated sensitivity coefficients (S), measured above their calculated backgrounds, are shown in Figures 3 for both white light and the 10% bandpass mode centred at 15 and 19 keV . Typically, S values for white light are some 20 times higher than for the 10% bandpass, except for the elements with Z above 40. From the white light data, it is possible to estimate a Minimum Detection Limit (MDL) of 5 ug/g for selenium during a 5 second exposure; this would be a typical dwell time in one spot during acquisition of a spatial distribution map. A 10% bandpass spectrum (energy centred at 20 keV) for Montana Soil 2611 (Figure 4), also illustrates the ability of the Peakaboo software to distinguish simultaneously a large number of K and L lines for elements with very different concentrations.

Conclusion

This recently developed software should facilitate the analysis of complex XRF spectra, particularly for beginning users.

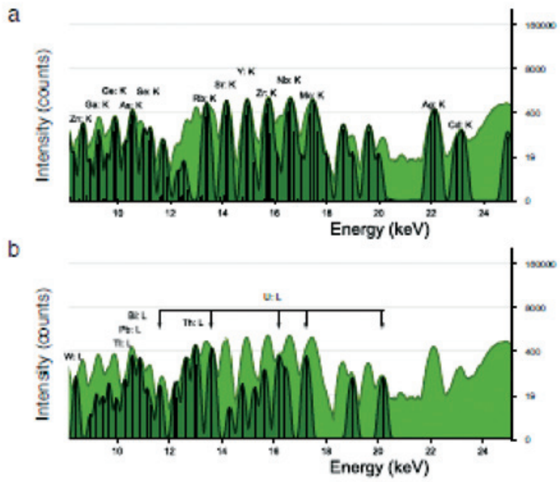

Figure 2: Logarithmic scale XRF spectrum of NIST SRM 610 glass excited with VESPERS 10% bandpass radiation centred at 25 keV for 600s. K (a) and L (b) lines are fitted separately. The complexity of the L spectra for U and Th can be seen.

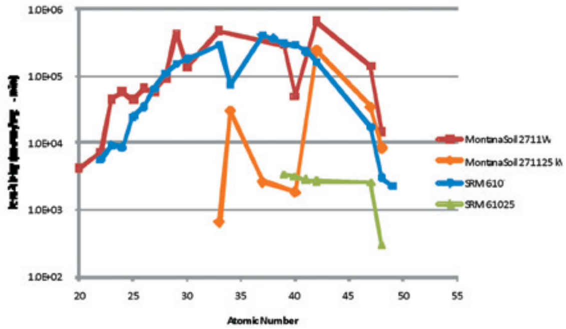

Figure 3: Comparison of Sensitivity Coefficients for SRM 610 and Montana Soil 2711 using white radiation and 10% bandpass radiation centered at 25 keV. Sensitivity coefficients acquired with white radiation agree more closely than do those using limited bandpass.

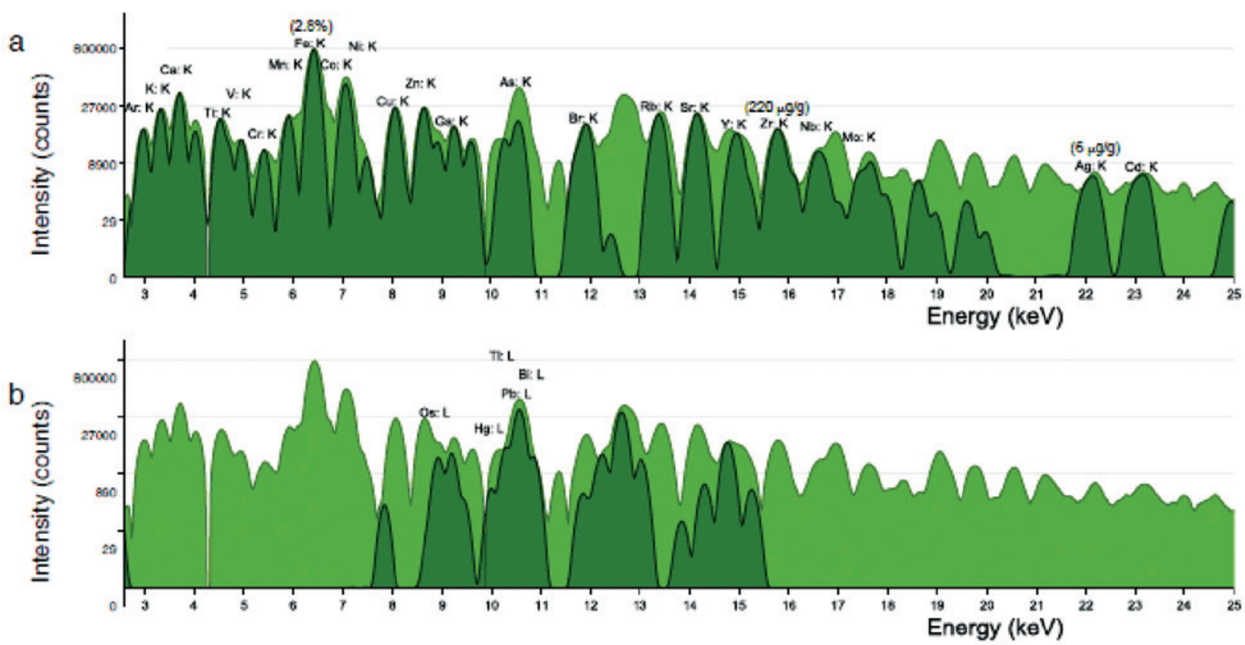

Figure 4: Log scale spectrum of Montana Soil 2611 excited by a 10% bandpass radiation centred at 20 keV. The spectrum is filled using K and L lines of twenty different elements. Some of the principal elemental line series are indicated along with their known concentration. (in ppm or %). A mathematical filter has removed all but 10%of the background.