I. Scope

Scanning electron microscopy/energy dispersive spectrometry (SEM/EDS) is a non-destructive analytical technique used to identify elements present in solid, powdered or liquid samples. EDS is capable of detecting elements from sodium (Na) (down to boron (B) in windowless mode of EDS) to uranium (U), at trace levels as low as 1% in a mixture.

The technique does have limitations. Since the transitions obtained are dependent upon the voltage (kV) used for the analysis, changing the applied voltage will result in a limitation on the transitions detected.

II. References:

Practical X-Ray Spectrometry, Jenkins and deVries, distributed by Springer Verlag

Scanning Electron Microscopy: A Student's Handbook, Postek et al, 1980.

X-Ray Spectrochemical Analysis, Birks, published by Interscience Publishers

Visual Lines for Spectroscopic Analysis, Smith, published by Hilger and Watts, Ltd.

An Introduction to X-Ray Spectrometry, Jenkins, published by Heyden and Son, Ltd.

Worked Examples in X-Ray Analysis, Jenkins and deVries, published by The Macmillan Press Limited

Forensic Investigation of Explosions, Beveridge (ed), published by Taylor & Francis Ltd

III. Safety Precautions:

No special precautions are required.

IV. Sampling and Sample Preparation EDS:

All samples should be secured to the stub with a conductive medium (carbon tape/tab, carbon paint, Duro-Tak, etc.), unless a beryllium planchet is used. Caution should be used if copper tape is used - refer to section VI for considerations. Nonconductive materials can be carbon coated and/or analyzed with the SEM in the low vacuum mode to prevent charging.

V. Analytical Procedure:

The operating conditions for the instrument used in casework shall be included on the print-out or on a separate parameter sheet that will be included in the case jacket.

Calibration

The instrument will be calibrated within one month (up to 31 days) of the analysis using the aluminum/copper standard. Additionally, the instrument should be calibrated anytime a change in normal operations is detected

See instrument manual or work instructions for calibration instructions. The date of the calibration and the instrument-specific calibration parameters to be recorded shall be available in each instrument's Calibration Log Book. A printout of the calibration spectrum may also be included.

Running Samples

To ensure a good quality EDS, the spot size should be adjusted as required.

VI. Possible Sources of Error:

Copper tape should not be used if Cu is suspected in the sample, if the Cu energy lines would interfere with other elements (possibly Na), or if semi-quantification is being performed (e.g. for comparison purposes). Prior to identifying Cu in any sample, that sample is required to be further analyzed using non-copper mounting material.

No image or a fuzzy image may be seen on SEM monitor if the electron beam is out of alignment, the aperture is off-center, or the filament has burned out.

The counts may be low if the spot size is too small.

Extraneous peaks may be seen if the count rate is too high (sum peak).

In low vacuum mode, small Al peaks from the stub may be seen if the sample is close to the edge of the mounting tape.

VII. Quality Assurance:

Through regular calibration, use of known standards, and upkeep of instrument logbooks, the quality of the EDS method is maintained.