# 9.6 Scanning Electron Microscope - Energy Dispersive Spectrometry

## 9.6.1 Introduction

The Scanning Electron Microscope/Energy Dispersive Spectrometer Technique permits the analyst to obtain elemental information about the sample. The SEM serves as a dual role of providing the energy to generate X-rays and isolating small regions for analysis. The Energy Dispersive Spectrometer system collects and processes X-rays for elemental identification.

## 9.6.2 Safety Considerations

If the x-ray detector uses liquid nitrogen, care must be used to protect the eyes and skin while working with liquid nitrogen.

## 9.6.3 Preparations

- Sample stubs for SEM.
- Carbon tape, discs, or colloidal suspensions for mounting sample.
- Scalpel
- Tweezers

## 9.6.4 Instrumentation

- Scanning Electron Microscope with attached Energy Dispersive Spectrometer system (Lansing Laboratory).
- Stereomicroscope

## 9.6.5 Procedure or Analysis

*Note: Also see TRACE-PM 7.4 EDAX EDS System*

### 9.6.5.1
Sample each layer or make a cross section

### 9.6.5.2
Mount chips onto carbon mounting medium.

### 9.6.5.3
If applicable, make a drawing of the surface of the stub in order facilitate locating the paint chips in the SEM.

### 9.6.5.4
If applicable, place stub(s) into an SEM stub box and label the box with contents.

### 9.6.5.5
If applicable, send box and drawing via U.S. Mail, UPS or carrier to the Trace unit of the Michigan State Police Lansing Laboratory.

Refer to **Appendix 9.6 A: Request for SEM Examination-Lansing Laboratory**

### 9.6.5.6
Guidelines for EDS Qualitative Analysis Interpretation

#### 9.6.5.6.1
Terminology: (Also see ASTM E2809)

**Sum Peak:** —peak occurring at the sum of the energy of two individual peaks.

**Escape Peak:** peak resulting from incomplete deposition of the energy of an X-ray entering the EDS detector. —This peak is produced when an incoming X-ray excites a silicon atom within the detector crystal and the resulting silicon (Si) K-alpha fluorescence X-ray exits the detector crystal. It occurs at the principal peak energy minus the energy of the Si K-alpha fluorescence X-ray (1.74 KeV). The escape peak intensity is about 1 to 2 % of the parent peak.

**Compton Scattering:** Occurs with low Z elements when low energy (KV) is used. Low energy causes an Auger electron (an electron from the outer shell) to be ejected. The result is a Compton peak occurring at approximately 200 eV before the element's peak.

**9.6.5.6.2**

General Guidelines:

Once an X-ray spectrum is collected, a qualitative analysis is performed to determine the elements present.

A conclusion regarding similarity results from the comparison of images and elemental composition of individual layers. Collect all data and micrographs in the same manner. Spectra may be critically compared by overlaying them.

If a comparative analysis did not demonstrate significant differences, then no differences were indicated in structure and composition within the limits of the analytical capability of SEM/EDS.

If a comparative analysis demonstrates significant differences between samples regarding structure and composition, then it can be concluded that the samples are different.

If ratio differences between peaks exist, it can be concluded that these differences may result from either actual differences in the bulk composition of the materials or from the analysis of a small sample (or area) whose chemistry is not representative of the bulk composition of an inhomogeneous specimen. The latter shall only be concluded following an extensive investigation of the heterogeneity of the known specimens and demonstration that the range of variation present in the known sample encompasses that observed in the questioned sample.

If there are no differences in major peak ratios but there are differences in minor/trace peaks, it can be concluded that no differences in major elemental constituents are indicated, although some differences in the bulk composition are evident. For example, if the sample was a fragment or smear and unable to be adequately cleaned, a small amount of foreign material may have been present during the analysis. Consequently, some of the minor elemental peaks present in the spectrum may have been produced from elements in the foreign material and not from elements in the questioned material. Equally so, the observed differences may be due to actual differences in the composition of the samples. Therefore, with respect to the elemental composition of these samples, an inconclusive result for this technique is indicated.

## 9.6.6 Documentation

Case notes shall include a copy of all of the instrumental data that was used to reach a conclusion. All data shall include a unique sample designation, the operator's name/initials, and the date of analysis.

Case notes shall also include a description of the evidence analyzed by SEM, the method of sample preparation, the analytical instrumentation used, and its operating parameters. The case notes shall also include a statement or data confirming instrument function verification.

## 9.6.7 References

ASTM E2809-13 - Standard Guide for Using Scanning Electron Microscopy/X-Ray Spectrometry in Forensic Paint Examinations

Reeve, V.; Keener, T. "Programmed Energy Dispersive X-ray Analysis of Top Coats of Automotive Paint"; Journal of Forensic Sciences, 21, 4, 883-907.

Meinhold, R.H.; Sharp, R.M. "The Application of A Multistylus Recorder to the Energy Dispersive Analysis of Paint Flakes in the Scanning Electron Microscope"; Journal of Forensic Sciences, 1978, 23, 2, 274-281.

Gardiner, L.R. "The Discrimination of Modern Household Paints Using the Scanning Electron Microscope Energy Dispersive X-Ray Analyzer"; Home Office Central Research Establishment, Report No. 405, 1981.

Gardiner, L.R. "The Homogeneity of Modern Household Paints Using the Scanning Electron Microscope Energy Dispersive X-Ray Analyzer"; Home Office Central Research Establishment, Report No. 408, 1981.

Goldstin, J.I.; Newbury, D.E.; Echlin, P.; Joy, D.C.; Riori, C.; Lifshin, E. Scanning Electron Microscopy and X-Ray Microanalysis; Plenum Press: New York, 1981; pp 59-74, 267-269.