Training Outline for SEM/EDX Elemental Analysis

1.0 Purpose – This document provides an outline for training in elemental analysis utilizing a Scanning Electron Microscope with an Energy Dispersive X-Ray Detector (SEM/EDX). This outline also includes the requisite competency testing.

2.0 Scope – This training outline shall be followed by all trainees in SEM/EDX elemental analysis, regardless of experience level.

3.0 Module I – Introduction to the SEM/EDX System

3.1 Objectives: Through completion of this module, the trainee shall have developed and demonstrated theoretical knowledge of:

3.1.1 The principles of optics and the microscope (light vs. electron microscopy).

3.1.2 X-Ray production, detection and display.

3.1.3 The sensitivity and care required for an SEM/EDX system.

3.1.4 Preparation of specimens for SEM/EDX elemental analysis utilizing micromanipulation techniques for particles.

3.1.5 The peak identification of elemental analysis using SEM/EDX.

3.2 Reading Assignments

3.2.1 Culbreth, K.L. *Personal Notes and Guides for Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis*.

3.2.2 Goldstein, Joseph and Harvey Yakowitz (ed.). *Practical Scanning Electron Microscopy*.

3.2.3 Hearle, J.W.S. and J.T. Sparrow. *The Use of the Scanning Electron Microscope*.

3.2.4 Kevex Corporation. *Energy Dispersive X-Ray Microanalysis – An Introduction*. SEM Computer.

3.2.5 LEO SEM User Manual and Tutorial. SEM Computer.

3.2.6 Oxford EDX Guide to X-Ray Microanalysis. CD-ROM Tutorial.

3.2.7 Kruesemann, Hans. "Innovations in Scanning Electron Microscopy. Preserve Sample Integrity and Validity in Forensic Analysis." *Forensic Magazine* (December 2008/January 2009): 1-5.

3.3 Exercises

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3.3.1 Read the required literature to understand the principles of:

- Electron beam formation.
- Specimen excitation and interaction with the electron beam.
- SEM image formation.
- X-Ray spectra production.
- X-Ray spectra interpretation.

3.3.2 Identify the areas of the SEM column, control panels, EDX system and the automated stage controller.

3.3.3 Demonstrate proficiency in venting the system, changing samples in the SEM, and manipulating samples in the SEM. Saturate the filament and align the image for optimal performance.

3.3.4 Review the principles of changing a filament and cleaning the cathode and anode assembly (if present on the instrument). Practice these principles and place the new assembly in the SEM. Warm up new filament, saturate the beam, and align the beam for elemental analysis.

4.0 Module II – Overview of Elemental Analysis

4.1 Objectives: Through completion of this module, the trainee shall have developed and demonstrated the theoretical knowledge of:

4.1.1 The morphology and elemental composition of particles.

4.1.2 The interpretation of data from an SEM/EDS analysis.

4.1.3 The archiving of data and printing of images and spectra.

4.1.4 Reporting and documenting results from elemental particle analysis.

4.2 Reading Assignments

4.2.1 McCrone, Walter & John Delly. *The Particle Atlas*. 2nd Ed., Vols. I-VI.

4.2.2 McCrone, Walter. *Techniques, Instrumentation, and Accessories for Micro-Analysts: A User's Manual.*

4.2.3 Kosanke, K.L., et al. "Characterization of Pyrotechnic Reaction Residue Particles by SEM/EDS." *Journal of Forensic Sciences* 48.3 (2003): 1-7.

4.2.4 MacQueen, H.R., et al. "The Application of Scanning Electron Microscopy to the Forensic Evaluation of Vehicular Paint Samples." *Journal of Forensic Sciences* 17.4 (1972): 659-667.

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4.3 Exercises

4.3.1 Read the required literature to understand the principles of elemental particle analysis and demonstrate an understanding of its principles.

4.3.2 Examine known particles and demonstrate familiarity with any morphological distinctions and elemental compositions. Particles to be analyzed include, but are not limited to, the following: powders, metals, paints, and explosive debris.

4.3.3 Analyze the powders, metals, paints, and explosive debris samples for a minimum of 300 seconds and vary the accelerating voltage to excite the various lines of the elements optimally.

4.3.4 Examine several samples and generate X-Ray spectra. Identify the elements present in the samples and save the spectra.

4.3.5 Vary the following parameters on each sample and observe the effects on the results:

- kV.
- Spot size.
- Magnification.
- Working distance.
- Secondary versus Backscattered Electron Detection.
- Scan speed.
- Variable pressure.

4.3.6 Once each x-ray spectra is collected, elementally identify each peak, label appropriately, and archive the x-ray spectra. Print out x-ray spectra and an image of each type of particulate.

4.3.7 Document and report results.

4.3.8 Compare results to other instruments used in the Laboratory for elemental analysis.

4.4 Evaluation

4.4.1 Given ten unknown particulate samples, correctly identify the elemental composition of each.

5.0 Module III – Casework

5.1 Objectives: Through completion of this module, the trainee shall have developed and demonstrated the theoretical knowledge of:

5.1.1 Evidence handling, evidence collection, evidence packaging, and chain of custody for elemental analysis evidence.

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5.1.2 Recognizing and preserving other items of potential evidentiary value.

5.1.3 Performing a complete elemental analysis from receiving the evidence to writing the laboratory report.

5.1.4 Documenting evidence condition, analytical methods used, and reasons for conclusions in the case file in a method understandable to fellow analysts.

5.1.5 Properly writing a clear, accurate, concise Laboratory Report consistent with Laboratory and Unit guidelines.

5.2 Exercises

5.2.1 The trainee shall perform casework with a qualified Forensic Scientist during the course of this training program. The following shall be discussed and practiced with the trainee:

- Proper procedures for taking notes and marking evidence.
- Proper procedures for handling, preparing and analyzing standards and unknown samples.
- Proper procedures for collecting particles from evidence.
- Proper packaging for evidence.
- Proper conclusions and report writing.

5.2.2 Discuss the interpretation of elemental analysis and its relevance and weight in conclusions.

5.3 Evaluation

5.3.1 The trainee shall be given a mock case consisting of a minimum of one known standard and two unknown samples. This exercise will include generating an entire case record and report. The trainee must accurately identify and present proper conclusions for their analysis.

6.0 Module IV - Final Evaluation and Preparation for Court

6.1 Objectives: Through completion of this module, the trainee shall have developed and demonstrated the theoretical knowledge and/or practical skills to**:**

6.1.1 Demonstrate courtroom procedures.

6.1.2 Present the results of an elemental analysis examination in court effectively.

6.1.3 Describe the legal and ethical obligations of an expert witness.

6.1.4 Describe the admissibility standards set by *Daubert* and *Frye*.

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6.1.5 Accurately present findings and conclusions as an expert witness in a courtroom setting.

6.2 Reading Assignments

6.2.1 CVs or Statements of Qualifications of other Forensic Scientists.

6.2.2 *Daubert v. Merrill Dow Pharmaceuticals,* 509 U.S. 579 (1993).

6.2.3 *Frye v. United States,* 293 F. 1013 (DC Cir. 1923).

6.2.4 Feder, H.A. and M.M. Houck. *Succeeding as an Expert Witness,* 4th ed. Boca Raton: CRC Press, 2008.

6.2.5 Kogan, J.D. "On Being a Good Expert Witness in a Criminal Case." *Journal of Forensic Sciences* 23.1 (1978): 190-200.

6.2.6 Philipps, K.A. "The Nuts and Bolts" of Testifying as a Forensic Scientist." *Journal of Forensic Sciences* 22.2 (1977): 457-463.

6.2.7 Ron Smith and Associates, Inc. "Courtroom Testimony Techniques: Success Instead of Survival." Collinsville, Mississippi.

6.2.8 Tanton, R.L. "Jury Preconceptions and Their Effect on Expert Scientific Testimony." *Journal of Forensic Sciences* 24.3 (1979): 681-691.

6.3 Exercises

6.3.1 Read literature pertaining to this module.

6.3.2 Prepare or update a CV or Statement of Qualifications reflective of experience in elemental analysis.

6.3.3 Prepare a series of qualifying questions and answers to those questions for use in a voir dire.

6.3.4 Observe pretrial conferences and courtroom testimony of qualified Forensic Scientist, if possible.

6.4 Final Evaluation

6.4.1 Using the mock case from Module III, successfully complete a moot court or roundtable discussion.

6.4.2 Successfully complete a final competency test covering all training materials.

7.0 Records

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- Training File
- Training Checklist

8.0 Attachments – N/A

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Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
10/18/2013	2	Added issuing authority to header
08/29/2014	3	Updated header to Physical Evidence Section – Trace Unit, issuing authority to Physical Evidence Section Forensic Scientist Manager. Updated all references in procedure from Trace Evidence Section to Trace Unit.

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