

## **Training Outline for Elemental Analysis using X-Ray Fluorescence (XRF)**

**1.0 Purpose** – This document provides an outline for training in the examination of inorganic elemental evidence, including arsenic examinations, and covers the requisite competency testing.

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

### **3.0 Module 1: Introduction to the XRF System**

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

**3.1.1** The principles of XRF.

**3.1.2** The method of X-Ray production, detection, and display.

**3.1.3** The interpretation of data produced from XRF analysis.

**3.1.4** The correlation of data from XRF with data from other instruments and analytical procedures.

**3.1.5** An awareness of other current methods for elemental analysis of materials, such as SEM/EDX, ICP, and XRD analysis.

**3.1.6** The current use of XRF as a tool for the forensic scientist as it relates to samples submitted to the North Carolina State Crime Laboratory.

### **3.2 Reading Assignments**

**3.2.1** Agarwal, B.K. *X-ray Spectroscopy: An Introduction*. Springer Series in Optical Sciences, Vol 15. New York: Springer-Verlag, 1979.

**3.2.2** Bertin, E.P. *Introduction to X-Ray Spectrometric Analysis*. New York: Plenum, 1978.

**3.2.3** Jenkins, R. *Introduction to X-Ray Spectrometry*. London: Heyden and Son, 1974.

**3.2.4** Azaroff, L.V., ed. *X-Ray Spectroscopy*. New York: McGraw-Hill, 1974.

**3.2.5** Herglotz, H.K. and L.S. Birks, eds. *X-ray Spectrometry*. Practical Spectroscopy, Vol.2. New York: Marcel Dekker, 1978.

**3.2.6** Jenkins, R. and J.L. DeVries. *Practical X-Ray Spectrometry*. New York: Springer-Verlag, 1967.

**3.2.7** Liebhafsky, H.A., et al. *X-Rays, Electrons, and Analytical Chemistry*. New York: Wiley-Interscience, 1972.

**3.2.8** Woldseth, R. *X-ray Energy Spectrometry*. Kevex Corporation, Burlingame, CA 1973.

- 3.2.9 Jenkins, R., R.W. Gould and Gedcke. *Quantitative X-Ray Spectrometry*. New York: Marcel Dekker, 1981.
- 3.2.10 Muller, R.O. *Spectrochemical Analysis by X-Ray Fluorescence*. New York: Plenum, 1972. Out of print.
- 3.2.11 Tertian, R. and F. Claisse. *Principles of Quantitative X-Ray Fluorescence Analysis*. London: Heyden and Son, 1982.
- 3.2.12 Dzubay, T.G., ed. *X-Ray Fluorescence Analysis of Environmental Samples*. Ann Arbor: Ann Arbor Science Publishers, 1977.
- 3.2.13 Heinrich, K.F.J., et al. *Energy Dispersive X-ray Spectrometry*. NBS Special Publication 604, National Bureau of Standards, Washington, DC, 1981.
- 3.2.14 Lachance, G.R. *Introduction to Alpha Coefficients* (Corp Scientifique Claisse, Inc., Quebec, Canada).

### 3.3 Exercises

- 3.3.1 Read pertinent literature and become acquainted with the following principles:
  - X-Ray formation principles.
  - Specimen excitation and interaction with X-Rays.
  - X-ray spectra production.
  - X-ray spectra interpretation.
- 3.3.2 Perform a literature search for new articles/literature and record these references.
- 3.3.3 Discuss with trainer the importance of XRF as a forensic tool and its appropriate use.

## 4.0 Module 2 – XRF Instrumentation and Application

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

- 4.1.1 Proper operation of the XRF.
- 4.1.2 Maintenance issues and quality control checks.
- 4.1.3 Sensitivity and care required for XRF system.
- 4.1.4 Sample preparation methods, sample holders, and films.
- 4.1.5 Proficiency in the elemental composition of various materials, including metals, glass samples, and clandestine laboratory samples.

### 4.2 Reading Assignments

4.2.1 Appropriate instrument manual.

#### 4.3 Exercises

4.3.1 Identify the areas of the XRF. Know the function of each component.

4.3.2 Demonstrate how to prepare liquids and solids for XRF analysis.

4.3.3 Prepare samples using different sample holders and films and analyze.

4.3.4 Learn how to vary the following different parameters on each sample and observe the effects on results.

- KV (excitation voltage).
- Current.
- Use of different Filters.
- Vacuum versus Atmosphere versus helium.

4.3.5 The trainee shall be given glass samples, metal samples, and clandestine laboratory samples to examine on the XRF. The trainee shall generate x-ray spectra, identify the elements present in the samples, store and print the results.

4.3.6 Perform analysis of materials on case samples with instructor's supervision for a sufficient period of time to become proficient in setup procedures for automated examinations, review and interpretation of data, archiving of results, and report writing.

#### 4.4 Evaluation

4.4.1 The trainee shall receive a combination of a minimum of 15 unknown solids and/or liquids and correctly identify each in its elemental composition. These samples shall be a conglomerate of typical forensic samples encountered in the Trace Unit (e.g., glass, clandestine laboratory samples, metals, powders, and unknown liquid samples).

### 5.0 Module 3– Reinsch Test (optional block of instruction)

5.1 **Objectives:** Through completion of this module, the trainee shall have developed and demonstrated the theoretical knowledge, practical skills, and application of the Reinsch test.

#### 5.2 Reading Assignments

5.2.1 Moffat AC, ed. *Clarke's Isolation and Identification of Drugs*, 2<sup>nd</sup> edition. London: The Pharmaceutical Press, 1986: 56-63.

#### 5.3 Exercises

5.3.1 Demonstrate the ability to perform the Reinsch test following the [Technical Procedure for the Examination of Arsenic Evidence](#).

**5.3.2** Demonstrate the proper examination of the copper wires using XRF analysis for the presence of any metals.

**5.3.3** Discuss the proper reporting guidelines and conclusions of the Reinsch Test.

#### **5.4 Evaluation**

**5.4.1** A mock case consisting of a minimum of 3 unknown samples in both solid and liquid form to perform the Reinsch test and also XRF analysis, if needed. This exercise shall include generating an entire Case Record and Report.

### **6.0 Module 4 – Casework**

**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** Complete an elemental examination of evidence using XRF instrumentation from receiving the evidence to writing the laboratory report.

**6.1.2** Demonstrate how to handle, collect and package elemental evidence.

**6.1.3** Understand how to recognize and preserve other trace items of potential evidentiary value (e.g., paint, hair, fiber).

**6.1.4** Demonstrate the proper way to document evidence condition, analytical techniques, and render a conclusion in the case file.

**6.1.5** Write a clear, concise Laboratory Report consistent with Laboratory and Unit guidelines.

#### **6.2 Exercises**

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

**6.2.1.1** Proper procedures for taking notes and marking evidence.

**6.2.1.2** Proper procedures for handling and analyzing elemental known and unknown samples.

**6.2.1.3** Proper procedures for sample selection, collecting particles from clothing, shoes and other objects.

**6.2.1.4** Proper packaging for elemental evidence.

**6.2.1.5** Determination of examination(s) applicable to each individual case.

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

## 7.0 Module 5 - Final Evaluation and Preparation for Court

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

- 7.1.1 Demonstrate courtroom procedures.
- 7.1.2 Effectively present the results of a paint examination in court.
- 7.1.3 Describe the legal and ethical obligations of an expert witness.
- 7.1.4 Describe the admissibility standards set by *Daubert* and *Frye*.

## 7.2 Reading Assignments

- 7.2.1 CVs or Statements of Qualifications of other Forensic Scientists.
- 7.2.2 *Daubert v. Merrill Dow Pharmaceuticals*, 509 U.S. 579 (1993).
- 7.2.3 *Frye v. United States*, 293 F. 1013 (DC Cir. 1923).
- 7.2.4 Feder, H.A. and M.M. Houck. *Succeeding as an Expert Witness*, 4<sup>th</sup> ed. Boca Raton: CRC Press, 2008.
- 7.2.5 Kogan, J.D. “On Being a Good Expert Witness in a Criminal Case.” *Journal of Forensic Sciences* 23.1 (1978): 190-200.
- 7.2.6 Philipps, K.A. “The Nuts and Bolts of Testifying as a Forensic Scientist.” *Journal of Forensic Sciences* 22.2 (1977): 457-463.
- 7.2.7 Ron Smith and Associates, Inc. “Courtroom Testimony Techniques: Success Instead of Survival.” Collinsville, Mississippi.
- 7.2.8 Tanton, R.L. “Jury Preconceptions and Their Effect on Expert Scientific Testimony.” *Journal of Forensic Sciences* 24.3 (1979): 681-691.

## 7.3 Exercises

- 7.3.1 Prepare or update a CV or Statement of Qualifications reflective of experience in XRF examination.
- 7.3.2 Prepare a series of qualifying questions and answers to those questions for use in a voir dire.
- 7.3.3 Observe pretrial conferences and courtroom testimony of qualified Forensic Scientist, if possible.

#### **7.4 Final Evaluation**

- 7.4.1** Using the mock cases from Modules 2 and/or Module3, successfully complete a moot court or roundtable discussion.
- 7.4.2** Successfully complete a final competency test covering all training materials (Module 3 – Reinsch Test is an optional Module).

#### **8.0 Records**

- Training file
- Training checklist

#### **9.0 Attachments – N/A**

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.