Technical Procedure for the Examination of Arsenic Evidence

1.0 Purpose – This technical procedure shall be followed for the examination of arsenic evidence.

2.0 Scope – This document shall be used for the examination of common foodstuffs and/or liquids for the presence of arsenic.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- Homogenizer and/or mortar and pestle
- X-Ray Florescence System (XRF)

4.2 Materials

- Two Erlenmeyer flasks (50 mL), plus one for each specimen
- Boekel Unheated Shallow Concentric Ring Bath or Pyrex dish
- Serological pipettes (5 mL)
- Copper wire, #20 gauge wire
- Homogenizer and/or mortar and pestle
- Magnetic stir bar
- 100 mL volumetric flask
- 200 mL volumetric flask
- Glass rod

4.3 Reagents

All chemicals shall be analytical reagent grade, stored at room temperature, and expire one year from date of preparation. A current reagent standard shall be used for each examination.

- 1N hydrochloric acid, ACS or higher grade
- Ethanol
- 10 % nitric acid, ACS or higher grade
- 0.1 mg/mL arsenic trioxide
- Distilled water

5.0 Arsenic Procedure

5.1 Reinsch Test

5.1.1 The Reinsch test is based on the fact that metallic arsenic, antimony, bismuth and mercury will deposit on a copper wire placed within a sample matrix that is acidified and heated. This deposit is visually recognized as a black or silvery staining of the copper wire. The analysis is followed by a confirmation technique utilizing XRF analysis for the particular element.

5.1.2 Standard Preparation is listed below:

5.1.2.1 1N hydrochloric acid: In a 200 mL volumetric flask add 16.6 mL of concentrated hydrochloric acid to 50 mL of distilled water. Fill the flask with distilled water to the 200 mL volume mark. This solution can be stored at room temperature in the volumetric flask.

5.1.2.2 Arsenic standard: A 0.1 mg/mL standard solution of arsenic trioxide is prepared by placing 10 mgs (0.01 grams) of arsenic trioxide in a 100 mL volumetric flask. This shall be diluted with 1N hydrochloric acid to the 100 mL mark.

5.1.2.3 Dissolution takes place with magnetic stirring (1-2 hours). Prepare annually (as needed) and store at room temperature.

5.1.2.4 10 % nitric acid: Add 10 mL of nitric acid to 90 mL of distilled water.

5.1.3 Prepare a water bath using either a Pyrex dish filled with water on a hot plate or Boekel Unheated Shallow Concentric Ring Bath. Heat the water bath until boiling. Be sure to watch water levels when boiling; additional water may need to be added to prevent boiling to dryness.

5.1.4 Prepare a copper wire by wrapping the wire around a glass rod 10 times. Leave an additional 4 to 6 inches of straight copper wire and cut the wire from the roll. The straight piece will serve as the handle of the copper wire. Prepare a second copper wire and one for each sample to be tested in the same manner.

5.1.5 Label one wire POSITIVE, one wire NEGATIVE, and each sample wire with its Laboratory number and item number. Obtain a large test tube or small beaker that will fit each copper wire (one for each sample and the positive and negative controls). Add 10 % nitric acid solution to each test tube/beaker. Place each copper wire in a test tube/beaker. Observe the effervescence for approximately 30 seconds. Remove the copper wire from the beaker or test tube. Terminate chemical reaction by rinsing the wire with distilled water, then ethanol, then distilled water. The clean coil should have a shiny appearance. If not, repeat procedure.

5.1.6 Place each copper wire in separate 50 mL Erlenmeyer flasks containing 4 mL of concentrated HCl.

5.1.7 Thoroughly homogenize the submitted specimen to ensure a representative aliquot is sampled. This may be accomplished by grinding in a mortar and pestle (i.e. candy), grinding by hand (i.e. cakes, cookies), or vigorously shaking the specimen (i.e. beverages). Add 10 grams or mL of uniform specimen to the flask followed by 10 mL of distilled water. A negative and positive control shall be processed similarly.

5.1.7.1 Note – If a specimen was initially homogenized with an equal volume of distilled water, use 20 grams of homogenate as the sample with no further addition of distilled water.

5.1.7.2 Note – A positive control that is generated in the specimen matrix also uses 20 grams of sample (10 grams specimen/10 mL analyte standard solution) with no further addition of distilled water.

5.1.8 Cover the flask with aluminum foil and swirl the contents to ensure uniform distribution of the acid.

5.1.9 Partially submerge the Erlenmeyer flask in a steam bath or on the top of the equivalent Boekel Unheated Shallow Concentric Ring Bath for one hour. Stir the contents by swirling the flask occasionally.

5.1.10 After the elapsed time, remove the wire and rinse with distilled water. Visually inspect each wire for a deposited black or silvery coating. Photographs shall be taken of the copper wires to document negative and/or positive results. Document results in notes. (For other elements: antimony and bismuth will yield a similar black stain while mercury coats the wire with a silvery deposit.)

5.1.11 If negative result occurs on the questioned item(s), a report shall be prepared indicating such a result. If a positive result occurs on the questioned item(s), proceed to ***5.2 Elemental Analysis***.

5.2 Elemental Analysis

5.2.1 If the Reinsch test yields a positive result for the questioned samples, all copper wire samples (positive, negative, and questioned) shall be analyzed using the XRF method for solids. Refer to the Trace Evidence Section Technical Procedure for X-ray Fluorescence Spectrometer.

5.2.1.1 The K alpha line for arsenic will appear at 10.54 keV, the K beta line will be at 11.72 keV.

5.2.2 Print all spectra for the known and unknown items. Determine if there are any elemental differences between the known and unknown spectra by performing spectral overlays.

5.3 Guidelines for Arsenic Result Statements

5.3.1 Positive

5.3.1.1 When both Reinsch Test results are positive and XRF spectra reveal the presence of arsenic.

5.3.1.1.1 Example: Examination of Item A revealed the presence of arsenic.

5.3.2 Negative

5.3.2.1 When the results of the Reinsch Test are negative and/or XRF analyses were negative for arsenic, antimony, bismuth and mercury.

5.3.2.1.1 Example: Examination of Item A did not reveal the presence of arsenic.

5.3.3 Document the presence of any other elements in the notes.

5.4 Calibrations – No additional calibrations or performance checks are required. See associated technical procedures for instrumental performance checks.

5.5 Maintenance – No additional maintenance is required. See associated technical procedures for instrumental maintenance.

5.6 Sampling and Sample Selection

5.6.1 No sampling is performed. When sample selection occurs, it shall be based on the Forensic Scientist's training and experience.

5.6.2 All items submitted for arsenic examinations shall be analyzed.

5.7 Calculations – N/A

5.8 Uncertainty of Measurement – N/A

6.0 Limitations

6.1 The Reinsch Test readily detects the metal cations arsenic, antimony, bismuth and mercury at concentrations of about 0.01 % (w/w) in food. The primary value of this test is exclusionary.

6.2 The deposit of a fixed (water insoluble) black or silvery stain on the copper wire only indicates and does not identify the presence of arsenic, antimony, bismuth or mercury. Tarnishing of the copper wire may occur from the presence of other metals such as selenium or tellurium. XRF shall be used to target the individual metal for qualitative data.

6.3 This test is a qualitative analysis and not a quantitative analysis. This means that the tests performed are designed to identify the presence of a particular heavy metal but not to determine how much is present.

7.0 Safety

7.1 Boiling water can scald. Care shall be taken when using hot water.

7.2 Strong acids can burn or irritate the skin. Proper protection shall be worn and all work done in a hood to prevent splashes, spills, and burns.

7.3 Metal accumulation is cumulative in the body, and care shall be taken to avoid skin contact with standard solutions.

7.4 Care shall be taken around the X-ray Fluorescence (XRF). All users must read the safety section of the XRF manual. NEVER open the lid while the XRF is operating.

8.0 References

Curry, A. *Poison Detection in Human Organs.* 2nd Edition. Springfield, IL: Charles C. Thomas, 1969.

Ellenhorn M.J., Barceloux D.G., *Medical Toxicology: Diagnosis and Treatment of Human Poisonings.* New York: Elsevier, 1988, pp.1012-1016.

Kaye, S. *Handbook of Emergency Toxicology*. Springfield, IL: Charles C. Thomas, 1954, pp. 32-40.

Moffat, A.C., ed. *Clarke's Isolation and Identification of Drugs.* 2nd edition. London: The Pharmaceutical Press, 1986, pp. 56-63.

Sunshine, I., ed. *Methodology for Analytical Toxicology*. Boca Raton, FL: CRC Press, 1975, pp. 395-398 (chapter on Heavy Metals by Sidney Kaye).

9.0 Records – N/A

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document