Examinations for Association or Origin

Table of Contents

Examinations for Association or Origin

1 INTRODUCTION

Manufactured goods and the items (e.g., fragments) derived from them bear characteristics indicative of their processing history and subsequent use in service. Metallurgical examinations of such characteristics can be used to identify a product type, determine a potential source of an item, or distinguish among items which are nominally of the same class. Individual characteristics, or sets of characteristics, of a product can also be evaluated and compared to specifications to assess conformance to expected properties. An extremely wide variety of metallurgical factors produce observable and measurable characteristics that can be used to identify and compare evidentiary items. The following procedure outlines the basic analyses most commonly performed to examine an item to ascertain its origin or evaluate an association to other item(s).

This technical procedure fully adopts the following reference (see section References):

- *United States Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline*, latest revision

2 SCOPE

This document applies to case working personnel using the associated instrument(s) and supporting equipment in support of metallurgy examinations.

Well-established metrological inspection techniques for which Chemistry-Metallurgy has not issued technical procedures can be applicable in some situations. In these instances, a full validation may not have to be completed, however appropriate performance characteristics will be validated contemporaneously.

3 EQUIPMENT

A list of items commonly used in these examinations follows. Not every item is used for all association and origin investigations. The instrumentation and equipment used will depend on the nature of the evidence to be examined and compared.

- Photography equipment for macro- and micro-documentation
- Observation enhancing tools, such as:
  - magnifying tools (e.g., borescope, magnifying glass, jewelers' loupe)
  - light microscopes (e.g., stereomicroscope, digital microscope, comparison microscope)
  - scanning electron microscope (SEM)
- Digital radiography system*
- Measurement tools, such as:
  - micrometers, calipers, measuring tape
  - optical measuring microscope (e.g., SmartScope FOV*)
  - balances
  - magnet
- Miscellaneous hand tools, clamps, and fixtures for specimen manipulation and support

- Certified reference materials (CRMs), reference materials, and standardization materials
- Digital multimeter
- Specimen cleaning and protection equipment and materials, such as:
    - compressed air
    - lint free wipes
    - cleaning brushes
    - cellulose acetate replication tape
    - EvapoRust™ rust remover
    - solvents
    - ultrasonic cleaner
    - desiccant
    - vacuum chamber
- Compositional analysis instruments, such as:
    - Energy dispersive X-ray fluorescence spectrometer (EDXRF)*
    - Spark discharge-in-argon optical emission spectrometer (SDAR-OES)*
    - Scanning electron microscope with energy dispersive X-ray spectrometer (SEM/EDS)
- Metallographic specimen preparation and examination equipment*
- Non-destructive testing equipment, such as:
    - magnetic particle inspection equipment
    - liquid dye penetrant and developer
    - ultrasonic inspection equipment
- Mechanical testing instruments, such as:
    - Hardness* and microhardness* testers
    - Tensile*, torsion, fatigue, impact, and wear testers
- Statistical software, such as MINITAB, Microsoft Excel, or equivalent

* When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy technical procedure for additional equipment.

4 STANDARDS AND CONTROLS

The standards and control materials used in this procedure will depend on the specific analytic methods employed and the nature of the item under analysis. Any instrument used in this procedure will employ such standards as required under its specific technical procedure. If available, exemplars for evidentiary items (i.e., known materials) may be obtained and examined to establish the expected variability of manufactured characteristics.

5 SAMPLING

5.1 Visual Inspection of All Items

Visual examinations are performed on every item examined under this procedure. Further testing is based on the suitability of individual items, or portions of items, for relevant examination techniques. Case notes will describe which examinations were performed on which items. If initial examinations reveal that an analyzed characteristic can vary on a single item, the means of selecting a location to test the characteristic will be noted in the case file.

5.2 Specimen Selection

5.2.1 Representative testing

A. The attribution of a trait measured at a single location of an object or on an extracted sample to the whole will depend on the manufactured characteristics of the object. For example, the composition of a visually uniform metal coating on an object may be analyzed in a single location and that composition may be attributed to the entire visually uniform coated surface.
B. Specimens or sections may be taken from an item for analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested. Destructive specimen removal should be approved by the contributor prior to modification of the evidence.

5.2.2 Sampling

A. If an item contains multiple visually indistinguishable objects that are suitable for one analysis technique, a subset may be selected for testing by non-statistical or statistical means. The manner of selection will be recorded in the case notes.
    1. For non-statistical specimen selection, the report will attribute the measured characteristic only to the specimen(s) tested. This can be facilitated by sub-dividing the evidence and reporting the specific analysis results for the sub-divided portion only, or by otherwise documenting the specific piece or location tested.
    2. If a sampling plan will be used to make an inference about the entire set of visually similar items (i.e., population), then the plan will be based on a statistically valid approach. A hypergeometric distribution can be used to make an inference about the population when not every item is tested, see Appendix A: Hypergeometric Table. Appendix A assumes that all results are consistent (i.e., ‘success’.) If inconsistent results are encountered (i.e., ‘failure’), either:
        i. Estimate the probability of observing the trait in the population via an appropriate statistical calculation, or
        ii. Limit conclusions regarding that characteristic to the specimens (or specific locations on a specimen) that were tested.
B. Statistical inferences will be clearly documented and reported as such.

6 PROCEDURE

6.1 Objectives

6.1.1 Origin determination

Origin determination: Metallurgical properties of components, or pieces of components, can be examined to potentially identify the original product, its means of manufacture, and its post-manufacture modification. Production characteristics such as alloy type, distribution of microstructural phases, component shape and dimensions, fabrication marks from forming or assembly, welds, coatings, identification markings, and other features can often be used to distinguish between places of manufacture and sometimes between different production lots by the same manufacturer. Post-manufacturing damage, including fracture, wear, thermal damage, and corrosion can indicate whether two objects endured similar service conditions or environmental exposure. One or more exemplars that share some characteristic(s) with the evidentiary items can often provide useful information.

6.1.2 Association evaluation

Items suspected of being from common sources are compared against each other in their relevant compositional and physical characteristics using the examination techniques that are most appropriate. These techniques typically include visual and microscopic examination, dimensional measurements, and compositional analysis of the items. Two items which are not distinguished from each other on these bases are considered to demonstrate an association and possibly a common origin. For example, two pipe sections from different sources can be distinguished based on their diameter, method of fabrication, alloy content, the nature of plating materials on them, or the presence or absence of fabrication marks. Conversely, two sections cut from a common length of pipe would be expected to be indistinguishable in all of these characteristics (except for differential degradation after separation.)

6.2 Analysis Techniques

6.2.1 Required

A. Perform a preliminary visual evaluation of the item(s).
   1. If loose debris is present, gently shake the item(s) over clean paper (e.g., Kraft) and collect and retain the debris.
   2. Evaluate the fabrication method(s), fracture and/or damage morphology, materials processing characteristics, material transfer, and any other characteristics deemed to be of value.
   3. Low magnification microscopy can be used to support this initial evaluation.

B. Photograph the submitted or in situ items in the “as received condition” (ARC).
   1. Additional photography should be conducted during the metallurgical examinations to record any features or characteristics upon which a conclusion is likely to be based.
   2. Whenever practicable, include a scale in the photograph or apply a verified micron marker to the photograph. See section Photography.

6.2.2 Optional

The remaining steps and/or tests are not required in every situation and will vary depending on circumstances and the evidence. Additionally, the sequence below serves only as a general guideline. The techniques selected and the sequence in which they are performed should be established by the nature of the evidence and the facts and circumstances of the case. In instances where evidence is to be evaluated for association to a known source, the characteristics of the unknown item are typically evaluated first, then compared with those of the object(s) representing the known source. Data gathered during examinations will be included in the case notes.

A. Evaluate the physical properties of the items by measuring appropriate features, such as dimensions, mass, and magnetic response.
    1. Use calibrated, traceable measurement tools for measurements that are reported or are used to substantiate conclusions that are reported. See section Verify Instrument Performance.
    2. See METAL-320 for precision optical measurements using the SmartScope.
B. Perform a radiographic examination of the specimens looking for internal structure(s), contaminants, defects, and any other appropriate characteristics suitable for evaluation by this technique. (See METAL-330.)
C. Conduct visual and low power magnification examinations for significant features, such as:
    - forming characteristics (e.g., shape, size, material(s), fabrication marks, anomalies)
    - processing characteristics (e.g., surface porosity, texture, heat tint, oxidation)
    - post-manufacture modifications
    - degradation (e.g., service abuse, characteristics of environmental interaction, existence of fractures and/or damage, manner of separation or failure, exogenous residues/deposits (composition and manner of deposition), and any other characteristics of value.
D. Perform higher magnification evaluations and comparisons of pertinent features (e.g., fabrication and materials processing characteristics, morphological features, fracture surfaces, exogenous deposits, and damage sites.) Such evaluations can be conducted using a variety of instruments, including stereomicroscope, digital microscope, comparison microscope, or SEM.
E. Assess the characteristics of environmental interaction(s) as appropriate for the determination(s) requested. For compared items, apparent differences in corrosion behavior should be reconciled with the facts or feasible explanations of material behavior and/or environmental parameters.
F. Perform qualitative or quantitative compositional analysis of any materials observed during examinations under this protocol which may assist in associating (or disassociating) specimens and characteristics and/or determining possible origin.
    1. Qualitative compositional analysis includes comparing the spectrum of an item of interest to that of another item, reference material, or specification to identify alloy class or product category.
        i. For EDXRF, see METAL-410 and the appropriate associated instrument-specific technical procedure.
        ii. For SEM/EDS, see section Compositional Analysis by SEM/EDS and IOSS-771.
    2. Quantitative compositional analysis may be used to report specific alloying content (including estimated measurement uncertainty) or to evaluate the possibility of a common production source (using a statistical t-test comparison, see section Calculations.)

i. For SDAR-OES, see METAL-400 and the appropriate associated technical procedure for the material and size of the item(s).
ii. For quantitative application of EDXRF or SEM/EDS, validate an appropriate technique in accordance with LAB-100 and CHEM-100 for the specific alloy matrix and analyte(s) of interest.

G. The above examinations can be augmented by various inspection and testing techniques, including non-destructive inspection, mechanical property testing (i.e., hardness, microhardness, or tensile testing) and metallography. See the appropriate Chemistry Unit metallurgy technical procedures.
H. Any destructive testing should be performed with regard to minimizing material loss and retaining informative features. Care should be taken to carefully document any features which may be lost prior to conducting destructive testing.
I. Research the possible origin of pertinent manufacturing or inspection marks. Reference the information resource(s) used to identify such marks in the case notes. For example, if a trademark is identified in an external database, record the database name and the organization that maintains the database (e.g., US Patent and Trademark Office, Underwriters Laboratory) in the case notes. If the owner or meaning of the mark will be reported, also reference the information resource in the *Laboratory Report*.
J. Report findings after evaluation of all gathered data.

6.3 Supporting Operations

The following additional instrumental conditions will be applied:

6.3.1 Photography

Macro- and micro-photographs will contain a reference scale whenever feasible, however these are included for general reference, and measurements will not be made from the images. Micron markers that are automatically generated by camera or microscope software are to be considered approximate and also will not be used to measure features within the image unless the marker is verified against a calibrated scale. Record the photograph filename in a photo log, along with identifying information such as camera or instrument identification, magnification, and feature depicted.

6.3.2 Sectioning

When possible, cutting and grinding operations will be lubricated to prevent overheating that can change the metallurgical characteristics of the specimen. If lubrication is not possible, the metallurgical changes imparted by the process must be considered during analysis.

6.3.3 Verify Instrument Performance

A. The following instruments will be verified according to the appropriate Chemistry Unit technical procedure prior to their first use to acquire traceable quantitative data on any given day. Record the instrument identifier, the calibration status, and that the verification was performed in the case notes.

- traceable micrometers/calipers – these instruments are calibrated annually by a service provider that meets FBI Laboratory quality assurance requirements. Perform the daily verification on calibrated gauge blocks of appropriate size for the particular instrument, typically 0.05 inch, 0.5 inch, and either 1 inch or 2 inch. Compare the measurements to acceptance criteria posted on the instrument-specific log sheet. Record the verification data and PASS/FAIL status on the instrument log.
- traceable balances – per CHEM-100
- SmartScope – per METAL-320
- Universal mechanical test machine – per METAL-530
- Hardness tester – per METAL-510
- Microhardness tester – per Metal-520
- SDAR-OES – per METAL-400 and the appropriate associated material-specific technical procedure

B. The following instruments will be verified prior to their first use any given day. (These instruments are not typically used to report quantitative data for metallurgy examinations. However, should a validated quantitative method be developed, the method will include steps to perform a quantitative verification on CRMs.)
- XRF – per METAL-410 and the appropriate associated instrument-specific technical procedure
- SEM/EDS – per IOSS-771

6.4 Scanning Electron Microscopy (SEM)

6.4.1 <u>Compositional Analysis by SEM/EDS</u>

A. Prior to the first use to acquire case data on any given day, run the instrument performance verification routine according to IOSS-771. File one copy with the instrument performance records and retain one copy in the case notes.

B. Prepare and insert the specimen(s) ensuring electrical continuity with the specimen stage.

C. Adjust the instrument conditions to image the region of interest for analysis. Backscattered electron imaging can be helpful to locate features that differ in mean atomic number from their surroundings.

D. Acquisition duration will depend on the conditions chosen and the specimen area exposed to the incident beam. The acquisition time can be extended to optimize spectrum clarity or shortened to enhance collection efficiency based on the case requirements.

E. Label the elemental peaks on the acquired spectrum, considering peak shapes and energy positions, the relative heights of adjacent peaks and system-generated peaks. Many SEM/EDS systems have software that can accurately identify the escape and sum peaks in a spectrum. The peak identification system resident in the instrument software can be augmented by analyzing CRMs of similar composition to the specimen of interest.

F. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.

6.4.2 *Image Acquisition by SEM*

A. Images acquired to document SEM/EDS spectrum acquisition location are not typically noted in a photo log but are saved and printed with the spectrum file.
B. For images acquired to document features contributing to analyses, record the photograph filename in a photo log, along with identifying information including:
   - instrument identification
   - magnification
   - excitation mode
   - feature depicted

7 CALCULATIONS

7.1 Quantitative Analysis

Calculate measurement uncertainty for values that are reported or are used to substantiate conclusions that are reported, see section Measurement Uncertainty. In many instances, no other calculations are required to perform this procedure. Calculations associated with the use of a particular instrument will be found in the appropriate technical procedure.

7.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a two-tailed, Welch's t-test statistic is used for the comparison of the sample means. Two samples are deemed to be "indistinguishable" in the property under consideration if the absolute value of the t-statistic calculated from the data sets is less than the preselected critical t-value ($t_{critical}$). The critical t-values are typically chosen so that an overall (Bonferroni-corrected) significance of $\alpha = 0.05$ can be achieved for the analysis and are determined by the degrees of freedom associated with the measurement. An $\alpha = 0.05$ means there is a 5.0% chance of incorrectly concluding two samples are from different sources when they are not.

A. Acquire data. An individual trait (such as mass, dimension, or the composition of an element in an alloy) or combinations of traits may be evaluated. Typically, five or more measurements per specimen are used for performing comparisons.
B. Establish the t-test significance, α. The Bonferroni correction is applied when more than one trait is evaluated on the same objects (e.g., mass and diameter of two sets of pellets, or the concentrations of multiple elements in two pieces of steel).

   1. For a single trait, use $\alpha = 0.05$.
   2. To evaluate multiple traits simultaneously, apply the Bonferroni correction to achieve $\alpha_{total} = 0.05$ by making each comparison using $\alpha_{individual}$:

      $$\alpha_{individual} = \alpha_{total}/E$$

      Where:

      E is the number of non-zero traits to be simultaneously compared
   3. For example, when comparing the compositions of two metal objects, E is the number of elements present above the LOQ in both specimens.

C. Perform a Welch's two-tailed statistical test of the two sample means for each trait.

1. The Excel macro "t-Test: Two-Sample Assuming Unequal Variances" performs the required calculations for the selected data sets and selected significance, α.

2. To proceed without using the Excel macro, calculate the mean and variance of each test data set as follows:

   i. Sample mean, $\bar{x}_a$:

   $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ or, in Excel: "=AVERAGE(x_i)

   ii. Sample variance, s_a^2:

   $s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x}_a)^2}{n_a}$ or, in Excel: "=VAR.S(x_i)"

   Where:
   $\bar{x}_a$ is the average value of the measurements on sample "a",
   x_i is each individual measurement in data set a, i = 1 through n_a, and
   n_a is the number of measurements made on sample "a"

3. The mean and variance of the data from sample "b" are calculated in the analogous manner.

4. Calculate the degrees of freedom, ν, as:

$$v = \frac{\left(\frac{s_a^2}{n_a} + \frac{s_b^2}{n_b}\right)^2}{\frac{1}{(n_a - 1)}\left(\frac{s_a^2}{n_a}\right)^2 + \frac{1}{(n_b - 1)}\left(\frac{s_b^2}{n_b}\right)^2}$$

5. Calculate the t-test statistic:

$$t_{stat} = \frac{(\bar{x}_a - \bar{x}_b)}{\sqrt{\frac{s_a^2}{n_a} + \frac{s_b^2}{n_b}}}$$

6. Determine $t_{critical}$ from the appropriate t-distribution curve (α_{total} and ν).

   i. To find the $t_{critical}$ value from a table, round down ν to the nearest integer.

   ii. In Excel: "=T.INV.2T($\alpha_{individual}$, ν)" .

7. If $|t_{stat}| > t_{critical}$ for any trait of comparison, the samples are concluded to have a statistically significant difference. If not, the samples are concluded to be indistinguishable.

8 ACCEPTANCE CRITERIA – INSTRUMENT PERFORMANCE

For instruments that require verification, standardization, or energy adjustment, a copy of the appropriate record(s) will be included in the case notes. For each instrument noted (*) in section Equipment, follow the procedures in the appropriate Chemistry Unit Metallurgy technical document.

9 MEASUREMENT UNCERTAINTY

9.1 Comparisons

When gathered, quantitative data are generally used for comparative purposes, as detailed in the Calculations section. Expanded measurement uncertainty should not be used for these inter-comparisons because it increases the probability two samples will appear to be analytically indistinguishable and therefore increases the likelihood of Type II errors (false inclusion).

9.2 Instrumental Results

If it is necessary to estimate the measurement uncertainty of an instrumental result, it will be done in accordance with CHEM-100. Instrumental measurement uncertainty is addressed in the individual instrument technical procedures and will be calculated and reported when appropriate.

- For mechanical testing and compositional analysis instruments, each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated.
- For calibrated, traceable dimensional measuring equipment, the repeatability component will be updated annually.
- Often the variation present in a part production run or allowed in a part specification is substantially larger than the uncertainty contribution from the measuring instrument. In these instances, instrument measurement uncertainties may not be calculated per an uncertainty budget because they are effectively negligible.

10 LIMITATIONS

The limitations of a particular analysis are determined by the type of item(s) being analyzed, the condition of the items, the case-specific information requested, and the specific examinations required in the situation under consideration. Specific limitations cannot therefore be predicted within this protocol but will be reported when appropriate.

11 METALLURGICAL CONCLUSIONS

Metallurgical conclusions are drawn from the whole of the analyzed and researched data.

11.1 Identification of Product/Origin

An examination for identification purposes evaluates the physical and chemical nature of the evidence. Conclusions are normally limited to statements of fact describing the item.

11.2 Failure Analysis Conclusions

A failure analysis examines a damaged component or assembly, to determine how it came to be in its present state. The strength of failure analysis conclusions is sometimes limited by the information available from the object(s) being analyzed and from the circumstances of the event(s) leading to failure. See METAL-220 Analysis for Failure, Damage, and Fracture.

11.3 Comparison Analysis Conclusions (Associations)

When conducting metallurgical examinations to compare evidence to specifications or to other evidence, the examiner assesses whether characteristics are in agreement or disagreement in order to come to a conclusion. Normally, all examinations conducted on questioned items must yield comparable results to the known item or specification if an association is to be reported. However, observed differences which can reasonably be explained within the established factual framework of a particular case do not preclude an association from being made. Conclusions will be expressed in reports and testimony according to current FBI Laboratory requirements. Refer to METAL-904 and the Metallurgy ULTR.

The results of examinations for association can be expressed as ‘physical fit’, ‘inclusion’, ‘exclusion’, or ‘inconclusive’ conclusions:

11.3.1 <u>Physical Fit</u>

A. ‘Physical fit’ is an examiner’s conclusion that two or more metallurgy items or materials were once part of the same object. This conclusion is an examiner’s opinion that two or more metallurgy items or materials show sufficient correspondence between their observed characteristics to indicate that they once comprised a single object and insufficient disagreement between their observed characteristics to conclude that they originated from different objects. This conclusion can only be reached when portions of two or more metallurgy items or materials physically fit together.

B. The basis for a “physical fit” conclusion is an examiner’s opinion that the observed characteristics of the items or materials provide extremely strong support for the proposition that they were once part of the same object and extremely weak support for the proposition that the items or materials originated from different objects.

11.3.2 <u>Inclusion</u>

A. ‘Inclusion’ is an examiner’s conclusion that two or more metallurgy items or materials with indistinguishable characteristics could have originated from the same source or process. An examiner may conclude that two or more items or materials originated either from the same metallurgy source or process or from another source or process that is substantially similar to the examined items or materials in all observed characteristics. An item or material may be included within a broad general population of items or materials (such as those that are mass-produced), or to a less frequently encountered population of items or materials, based on their physical and chemical characteristics.

B. The basis for an ‘inclusion’ conclusion is an examiner’s opinion that two or more items or materials do not exhibit any differences in observed characteristics that would not be expected from items or materials that originated from the same source or process.

11.3.3 Exclusion

A. ‘Exclusion’ is an examiner’s conclusion that the metallurgy items or materials could not have originated from the same source or process.
B. The basis for an ‘exclusion’ conclusion is an examiner’s decision that two or more items or materials exhibit exclusionary differences in observed characteristics that would not be expected from items or materials that originated from the same source or process.

11.3.4 Inconclusive

A. ‘Inconclusive’ is an examiner’s conclusion that no determination can be reached as to whether two or more metallurgy items or materials could have originated from (or be excluded as originating from) the same source or process.
B. The basis for an ‘inconclusive’ conclusion is the examiner’s opinion that there is insufficient quantity and/or quality of observed characteristics to determine whether two or more items or materials could have originated from the same source or process (or be excluded as originating from the same source or process.)

12 METALLURGY LABORATORY REPORTS

Not every metallurgy examination scenario can be anticipated. This section provides guidelines for the structure of metallurgy reports. The scope and amount of detail included in metallurgy Laboratory Reports can vary widely depending on the specific request and case scenario.

12.1 Format

A metallurgy Laboratory Report will contain the administrative requirements set forth in the FBI Laboratory Operations Manual (LAB-200). Metallurgy Laboratory Reports will also comply with the language limitations detailed in the FBI Approved Standards for Scientific Testimony and Report Language for Metallurgy (the Metallurgy ASSTR, METAL-904) and the Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline (the Metallurgy ULTR). The Metallurgy ULTR will be included in the report by reference.

12.2 Results of Examinations Section

The Results of Examinations section will include a summary of the results of the metallurgy examinations conducted on each item of evidence examined and a succinct statement of any conclusion drawn from the examination results. See Metallurgical Conclusions.

- Limitations that prevent forming conclusions related to the incoming request must be included in this section. Other limitations may be deferred to the Methodology, Interpretation, and Discussion section of the report under a *Limitations* heading if it is deemed to improve the clarity of the report.

- For a comparative examination, this section will indicate the general nature of any conclusion (e.g., ‘physical fit’, ‘inclusion’, ‘exclusion’, or ‘inconclusive’), the characteristic(s) on which the conclusion is based, and an indication of the significance of the conclusion.
- For an ‘inconclusive’ comparison, the reason for the inconclusive result will be expressly stated (e.g., “Due to the small size of the particle, there is an insufficient quantity of observable characteristics to determine whether it originated from a bullet.”)

12.3 Methodology, Interpretations, and Discussion Section

In addition to the Results of Examinations section, each metallurgy report will include a “Methodology, Interpretations, and Discussion” section that specifies the instruments and procedures that were employed. This section also provides additional information intended to help the reader understand the results of the examinations. To this end, this section will include the following, when appropriate:

- information on both the strengths and the limitations of the examinations performed.
- for “inclusion” conclusions, an explanation of the population of other materials that possess characteristics similar to those exhibited by the evidence. For example, when an ‘inclusion’ is made to a broad, general population of items or materials (such as mass-produced items), the report will explain that the chance of finding coincidentally indistinguishable materials may be high.
- explanations of the principles on which conclusions were based.
- recommendations for remedial action to prevent future failures.
- supplementary data and the sources of such data (e.g., externally managed databases like those maintained by the United States Patent and Trademark Office.)
- details regarding the nature and strength of any associations other interpretations, opinions or predictions that can be inferred from the results of the examinations, and the bases for these opinions.

13 Safety

- Wear an X-ray film badge or dosimeter when operating instruments that generate X-rays.
- X-ray generating instruments have protective enclosures and internal safety interlocks to prevent inadvertent X-ray radiation exposure. Never bypass or disable safety interlocks on instruments.
- Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting, chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock, wearing a face guard to prevent impact from flying debris.) Review instrument technical procedures and pertinent Safety Data Sheets (SDS) prior to conducting examinations.

14 References

- LAB-100, Quality Assurance Manual, FBI Laboratory, latest revision
- LAB-200, Operations Manual, FBI Laboratory, latest revision
- CHEM-100, Quality Assurance and Operations Manual, FBI Laboratory, Chemistry Unit, latest revision
- METAL-220, Analysis for Failure, Damage, and Fracture, Chemistry Unit, latest revision
- METAL-221, Functionality Examinations, Chemistry Unit, latest revision
- METAL-222, Examinations of Timing Mechanisms, Chemistry Unit, latest revision
- METAL-223, Lamp Bulb Examinations, Chemistry Unit, latest revision
- METAL-320, Operation of the SmartScope FOV Video Measurement System, Chemistry Unit, latest revision
- METAL-330, Digital Radiography, Chemistry Unit, latest revision
- METAL-400, Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision
- METAL-401, Analysis of Carbon and Low Alloy Steel Samples by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision
- METAL-402, Analysis of Small Carbon and Low Alloy Steel Specimens by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision
- METAL-403, Analysis of Copper by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Chemistry Unit, latest revision
- METAL-410, Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), Chemistry Unit, latest revision
- METAL-411, Operation of the Thermo QUANT'X X-Ray Fluorescence Spectrometer, Chemistry Unit, latest revision
- METAL-412, Operation of the Bruker M4 Tornado X-Ray Fluorescence Spectrometer, Chemistry Unit, latest revision
- METAL-413, Operation of the Olympus Delta Handheld X-Ray Fluorescence Spectrometer, Chemistry Unit, latest revision
- METAL-450, Metallography, Chemistry Unit, latest revision
- METAL-510, Rockwell Hardness Testing, Chemistry Unit, latest revision
- METAL-520, Microhardness Testing, Chemistry Unit, latest revision
- METAL-530, Operation of the Instron Model 3382 Universal Testing Machine, Chemistry Unit, latest revision
- METAL-904, FBI Approved Standards for Scientific Testimony and Report Language for Metallurgy, Metallurgy Manual Metal 901, Chemistry Unit, latest revision
- IOSS-771, Performance Monitoring Protocol (QA/QC) for the Scanning Electron Microscope (SEM) / Energy Dispersive X-ray Spectrometer (EDS), Chemistry Unit, latest revision
- United States Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline, latest revision

15 Revision History

Revision	Issued	Changes
10	09/30/2022	Revised to comply with new formatting requirements. Removed expository information to retain as training material. Consolidated Introduction and Principle sections. Added references to technical procedures. Added Reporting Results section with procedural content and associated references from prior document METAL-250 (formerly Metal-900 General Approach to Report Writing in Metallurgy.)
11	08/15/2024	Relocated informational content from Scope to Introduction and from Principles to Analysis/Objectives sections. Added incorporation of external document. Added 'physical trait' to application of the hypergeometric sampling table and added text regarding statistical sampling. Updated terms to reflect ULTR changes effective 5/8/2024. Added documentation requirements for photography and instrument verification. Corrected SDAR-OES document number. Added SEM imaging section. Relocated and reorganized Report Writing section and conclusion information. Changed quantitative comparison model from Student's t-test to Welch's t-test to accommodate data sets with unequal variances. Reformatted lists and step sequences. Updated reference section. Corrected Bonferroni equation.

Appendix A: Hypergeometric Table

The hypergeometric table below shows the minimum number of samples that need to be analyzed (and yield consistent results) to obtain a 95% confidence level that at least 90% of the population contains a given substance or possesses a given physical trait.

Table 1. Hypergeometric Table

Total Number of Units	Number of Units to be Sampled
1-10	All (no inferences)
11-13	10
14	11
15-16	12
17	13
18	14
19-24	15
25-26	16
27	17
28-35	18
36-37	19
38-46	20
47-48	21
49-58	22
59-77	23
78-88	24
89-118	25
119-178	26
179-298	27
299-1600	28
more than 1600	29