

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. These characteristics include the specific alloy type, the specific composition of a particular alloy, the physical dimensions, fabrication marks from the forming process, the presence of welds, the microstructure, damage suffered in service such as fracture, wear, thermal damage, and numerous others. These characteristics can be used to determine a potential source of an item or distinguish among items which are nominally of the same class. 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, the presence or absence of fabrication marks and so on. Similarly, two sections cut from a common length of pipe would be expected to be indistinguishable in all of these characteristics.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. In metallurgy, there is an extremely wide variety of components, metals, treatments, fabrication techniques, service abuse, types of failure and damage, applications, and exposure environments that are variables affecting evidentiary materials. The following procedure outlines the basic analyses most commonly performed to examine an item for association or origin.

3 Principle

Items can be examined to potentially identify their source or origin of manufacture. Compositional and physical characteristics can often be used to distinguish between places of manufacture and possibly even between different production lots by the same manufacturer. These characteristics can include chemical composition, physical marks imparted by fabrication tooling, dimensions and other design and fabrication considerations. Often one or more exemplars that share some characteristic(s) with the evidentiary items can provide useful information.

Items suspected of being from common sources are compared against each other in their relevant compositional and physical characteristics using those techniques which are most appropriate. These typically include visual and microscopic examination of the surfaces and other characteristics, dimensional measurements, evaluation of the fabrication characteristics, 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.

4 Specimens

Nearly any metal object and many nonmetallic objects can be examined using the steps outlined in this procedure.

5 Equipment/Materials/Reagents

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 to be employed will depend on the nature of the item(s) to be examined and compared. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) for additional supplies (see section 15 References).

- a. Photography equipment for macro- and micro-documentation
- b. Observation enhancing tools, such as:
 - i. borescope, magnifying glass, jewelers' loupe
 - ii. visible light microscopes (stereomicroscope, digital microscope, comparison microscope)
 - iii. scanning electron microscope (SEM)
- c. Radiography system*
- d. Measurement tools, such as:
 - i. micrometers, calipers, measuring tape
 - ii. optical measuring microscope (e.g., SmartScope FOV*)
 - iii. balances
 - iv. magnet
- e. Miscellaneous hand tools
- f. Certified reference materials and calibration standards as needed
- g. Digital multimeter
- h. Specimen cleaning and protection equipment and materials:
 - i. compressed air
 - ii. lint free wipes
 - iii. cleaning brushes
 - iv. cellulose acetate replication tape
 - v. EvapoRust™ rust remover
 - vi. Solvents: water, alcohol, etc.
 - vii. ultrasonic cleaner
 - viii. desiccant
 - ix. vacuum chamber

- i. Compositional analysis instruments:
 - i. Energy dispersive x-ray fluorescence spectrometer (EDXRF)*
 - ii. Spark discharge-in-argon optical emission spectrometer (SDAR-OES)*
 - iii. Scanning electron microscope with energy dispersive x-ray spectrometer (SEM/EDS)
- j. Metallographic sample preparation and examination equipment*
- k. Non-destructive testing equipment, such as:
 - i. magnetic particle inspection equipment
 - ii. liquid dye penetrant (LDP) and developer
 - iii. ultrasonic inspection equipment
- l. Mechanical testing instruments, such as:
 - i. Hardness* and microhardness* testers
 - ii. Tensile*, torsion, fatigue, impact and wear testers

6 Standards and Controls

The standards and control samples 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 standard operating procedure. Exemplars for evidentiary items may be obtained and examined to establish the expected variability of manufactured characteristics.

7 Sampling

Visual examinations are performed on every item examined under this protocol. 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 may vary on a single item, the means of selecting a location to test the characteristic will be noted in the case file.

If an item contains a large number of visually indistinguishable objects that are suitable for one analysis technique, a subset may be selected for testing by (1) non-statistical or (2) statistical means. Any sampling plan and corresponding procedure used will be recorded in 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.

(2) If a sampling plan will be used to make an inference about the entire set of visually similar items, then the plan will be based on a statistically valid approach. A hypergeometric distribution can be used to describe the probability of encountering deviations within a set of items when not

every item is tested. (See Appendix A.) Appendix A assumes that all results are consistent. If inconsistent results are encountered, metallurgy conclusions regarding that characteristic will be limited to the specimens tested.

8 Procedure

The following 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, and the examinations selected should be established by the facts and circumstances of the case. Data gathered during examinations will be included in the case notes. This procedure will not be taken as a substitute for sound engineering judgment.

- a. Perform a preliminary visual and low magnification microscopic evaluation of the item(s) to evaluate the fabrication method(s); fracture and/or damage morphology; materials processing characteristics; material transfer; and any other characteristics deemed to be of value.
- b. Photograph submitted or in-situ items in the “as-received condition” (ARC). Additional photography should be conducted during the metallurgical examinations to record any features or characteristics upon which a conclusion is likely to be based. Whenever practicable, include a scale in the photograph or apply a verified micron marker to the photograph.
- c. Evaluate the physical properties of the items by measuring appropriate features, such as dimensions, mass and magnetic response.
- d. 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.
- e. Conduct visual and low power magnification examinations for characteristics of shape, size, material(s), fabrication characteristics/marks, anomalies, processing characteristics, modifications for service or other post-purchase use, service abuse, non-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.
- f. Perform higher magnification examinations and/or comparisons of fabrication and materials processing characteristics, morphological features, fracture surface evaluations, exogenous deposit characterization, damage site evaluation, and any other higher magnification examination deemed appropriate.
- g. Microscopic inspections may be augmented with scanning electron microscopy (SEM). Typically, this should be done when optical instruments are unable to resolve sample features which the examiner deems are important in reaching a conclusion.

- h. Assess the characteristics of environmental interaction(s) as appropriate for the determination(s) requested. Apparent differences in corrosion behavior should be reconciled with the facts or feasible explanations of material behavior and/or environmental parameters.
- i. Perform qualitative, semi-quantitative, or quantitative compositional analysis of any materials observed during examinations under this protocol which may assist in associating specimens and characteristics and/or determining possible origin. Samples or sections may be taken from the items for chemical analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested.
- j. The above examinations may be augmented by various inspection and testing techniques, including non-destructive inspection, mechanical property testing (i.e., hardness, tensile, impact testing) and metallography.
- k. Any destructive testing should be performed with regard to minimizing material loss and retaining informative features.
- l. Report findings after evaluation of all gathered data.

9 Instrumental Conditions

For instruments that require verification, standardization or energy adjustment, a copy of the appropriate record(s) will be included in the case notes.

9.1 Analytical Instruments (for SEM/EDS see 9.3)

For each instrument noted (*) in 5 Equipment/Materials/Reagents, follow the appropriate CU Metallurgy SOP (see section 15 References).

9.2 Supporting Equipment

The following additional instrumental conditions also will be applied:

- a. 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.
- b. 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.

- c. The following instruments will be verified according to the appropriate CU Instrument Operations Systems Support (IOSS) SOP (see 15 References) prior to their first use to acquire case data on any given day:
 - i. traceable micrometers/calipers
 - ii. traceable balances

9.3 SEM/EDS

Compositional analysis by SEM/EDS will be conducted as follows:

- a. Prior to the first use to acquire case data on any given day, run the instrument performance verification routine according to the appropriate IOSS SOP (see 15 References). File one copy with the instrument performance records.
- b. Prepare and insert the specimen(s) ensuring electrical continuity with the sample 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 sample 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.

10 Decision Criteria

A conclusion that a particular item is from a particular origin, or may be associated with another known item is based on a series of direct comparisons with that known item. Normally, all examinations conducted on known and questioned items must yield comparable results 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 (see section 15 References).

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

‘Fracture 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 decision 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.

‘Inclusion’ is an examiner’s conclusion that two or more metallurgy items or materials 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. The basis for an ‘inclusion’ conclusion is an examiner’s decision that two or more items or materials exhibit substantially similar observed characteristics with no unexplainable differences.

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

‘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. The basis for an ‘inconclusive’ conclusion is the examiner’s decision 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 process (or be excluded as originating from the same process.)

11 Calculations

In most instances, no calculations are required to perform this procedure. Calculations associated with the use of particular instruments will be found in the appropriate SOP.

Where quantitative data from two specimens are being compared, a pooled, two-tailed Student’s t-test statistic of the sample means is typically used for the comparison. Two samples are deemed to be “indistinguishable” in the property under consideration if the two samples differ by less than the preselected critical t value (t_{critical}). The critical t values are typically chosen so that an overall value 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 rejecting a match between two samples when one actually exists.

To perform this test, the means and variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where: \bar{x}_a is the average value of the measurements on sample

“a”, $\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample.

The variance of the individual measurement values from sample “a” is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

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

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 \left(\frac{1}{n_a} + \frac{1}{n_b} \right)} \right)} \right| > t_{critical}$, the samples are concluded to have a statistically significant

difference. If not, the samples are concluded to be indistinguishable.

In general, the number of individual measurements required per sample is determined by the population data distribution. If the sample populations are known to be or can reasonably be assumed to be normally distributed (Gaussian), as few as three measurements per sample can be used to compare the results. In the majority of instances where the measurement populations are not normally distributed, 5-10 measurements per sample will result in sample means which are approximately normal and will be adequate for the comparison outlined above. For heavily skewed population distributions, a minimum of 30 measurements per sample may be required to achieve this. Heavily skewed data distributions will normally be detectable on inspection of the sample data. Statistical tests also exist for determining whether data are Gaussian or non-Gaussian and can be employed as they are needed. Commonly, a normal probability plot is constructed for this purpose using statistical software packages such as MINITAB or equivalent.

12 Measurement Uncertainty

When gathered, quantitative data are generally used for comparative purposes. Expanded 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).

In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*. Instrumental measurement uncertainty is addressed in the individual instrument SOPs and will be calculated and reported when appropriate. Each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated. 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 cases, instrument measurement uncertainties will not be reported because they are considered negligible.

13 Limitations

The limitations of a particular analysis (if any) are determined by the type of sample(s) being analyzed, the condition of the samples, the specific determinations being made, and the specific examinations required in the situation under consideration and cannot therefore be predicted within this protocol but will be reported when appropriate. See also section 10 Decision Criteria.

14 Safety

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

15 References

ASM International Handbook Committee, *ASM Handbook, Volume 8 - Mechanical Testing and Evaluation*, ASM International 2000, or latest revision

ASM International Handbook Committee, *ASM Handbook, Volume 10-Materials Characterization*, ASM International 1992, or latest revision

Oberg, E., Jones, F. D., Horton, H. L., and Ryffel, H. H., *Machinery's Handbook*, 25th edition, Industrial Press Inc. 1996

Encyclopedia of Forensic Sciences, Editor-in-Chief Siegel, J.A., Academic Press 2013

Forensic Science Handbook, 2nd Edition, Editor Saferstein, R., Prentice-Hall Press 2002

Anderson, R. C., *Inspection of Metals, Volume I: Visual Examination*, American Society for Metals 1983

Milton, J. S. and Arnold, J. C., *Introduction to Probability and Statistics - Principles and Applications for Engineering and Computer Sciences, Fourth Edition*, McGraw-Hill Higher Education, 2003

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

General Approach to Report Writing in Metallurgy, Metallurgy Manual Metal 900, Chemistry Unit, latest revision

Chemistry Unit (CU) FBI-Approved Standards for Scientific Testimony and Report Language for Metallurgy, Metallurgy Manual Metal 901, Chemistry Unit, latest revision

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

Digital Radiography, Metallurgy Manual Metal 303, Chemistry Unit, latest revision

Operation of the SmartScope FOV Video Measurement System, Metallurgy Manual Metal 302, Chemistry Unit, latest revision

Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), Metallurgy Manual Metal 500, Chemistry Unit, latest revision

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Metallurgy Manual Metal 400, Chemistry Unit, latest revision

Metallographic Examinations, Metallurgy Manual Metal 800, Chemistry Unit, latest revision

Operation of Rockwell Hardness Testers, Metallurgy Manual Metal 701, Chemistry Unit, latest revision

Operation of Microhardness Testers, Metallurgy Manual Metal 702, Chemistry Unit, latest revision

Operation of the Instron Model 3382 Universal Testing Machine, Metallurgy Manual Metal 703, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Balances, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Micrometers and Calipers, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Scanning Electron Microscope (SEM)-Energy Dispersive X-ray Spectrometer (EDS), Instrument Operations Systems Support, Chemistry Unit, latest revision

Rev. #	Issue Date	History
6	12/21/2018	Reformatted and expanded section 5. Added section 8c. Augmented section 9 to include specific instrument procedures. Made minor editorial corrections throughout document. Added references to section 15 and corrected revision information.
7	01/29/2019	Added definitions of 'fracture fit', 'inclusion', 'exclusion', and 'inconclusive' conclusions to section 11. Updated titles of referenced documents.
8	02/18/2020	Revised section 7 and added Appendix A. Expanded section 8 to refer to techniques that use equipment already listed in section 5 and add minimization of destructive testing.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader:

Date: 02/13/2020

Chemistry Unit Chief:

Date: 02/13/2020

Appendix A: *Hypergeometric Table*

The hypergeometric table listed 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.

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

Analysis for Failure, Damage, and Fracture

1 Introduction

Failure analysis is a long-established practice with its roots in all of the engineering sciences. In fact, modern industry could not exist in its present form without failure analysis being used to advance technology. Metallurgy in particular is a mature engineering field extensively researched and covering a broad spectrum of subjects. Failure analysis examinations use this vast knowledge to try to determine why a metal component failed to perform adequately in use.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. The field of metallurgical failure analysis encompasses all of metallurgy and materials science and engineering, from raw material production to end product use. There are an extremely wide variety of types of parts, alloys, post-manufacture treatments, service conditions, types of loading, applications, environments, and combinations of all of these. In addition, there are nearly unlimited questions that can be asked and determinations that can be requested with regard to the failure and/or damage exhibited.

The metallurgical failure analysis practitioner recognizes that each examination and testing procedure is situational and requires sound engineering judgment and discretion. Not all examination steps in this procedure are required in every case but several techniques are listed for examiner consideration. This procedure outlines analyses which may be performed in examination(s) for failure/damage/fractures. The standard operating procedures (SOPs) for each of the instruments used for a given set of examinations should also be consulted.

3 Principle

Failure analysis is fundamentally an engineering-based investigation of an event or a series of events. By application of engineering principles, an item can be examined to determine if fundamental design problems exist or if a defect may have been artificially introduced. For example, a sharp change in cross-section can result in a stress concentration and crack development under conditions not otherwise expected for the assumed operating stresses. Fracture mechanics permits analysis of such a stress concentration to predict its potential effects on the component's operating life.

Fractography can be applied to determine the mechanism responsible for crack growth and to determine the crack initiation site. Some fracture mechanism examples include simple tensile overload, fatigue, stress corrosion cracking, and creep failure. Each fracture surface also provides information on the nature of the applied stress (i.e., torsional, shear, tensile, mixed mode). The metallographic and/or compositional analysis of an object may reveal deficiencies in material or

manufacturing process as a cause of failure. Numerous other methodologies are also available for exploring a particular aspect of a failure and determining its cause.

4 Specimens

Nearly any metal object and many nonmetallic objects can be examined using the procedures outlined in this protocol.

5 Equipment/Materials/Reagents

A list of items commonly used in these examinations follows. Not every item is used for all failure and damage investigations. The instrumentation and equipment to be employed will depend on the nature of the items to be examined. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) for additional supplies (see 15 References).

- a. Photography equipment for macro- and micro-documentation
- b. Observation enhancing tools, such as:
 - i. borescope, magnifying glass, jewelers' loupe
 - ii. visible light microscopes (stereomicroscope, digital microscope)
 - iii. scanning electron microscope (SEM)
- c. Radiography system*
- d. Measurement tools, such as:
 - i. micrometers, calipers, measuring tape
 - ii. optical measuring microscope (e.g., SmartScope FOV*)
 - iii. balances
 - iv. magnet
- e. Miscellaneous hand tools
- f. Certified reference materials and calibration standards as needed
- g. Digital multimeter
- h. Specimen cleaning and protection equipment and materials:
 - i. compressed air
 - ii. lint free wipes
 - iii. cleaning brushes
 - iv. cellulose acetate replication tape
 - v. EvapoRust™ rust remover
 - vi. Solvents: water, alcohol, etc.

- vii. ultrasonic cleaner
- viii. desiccant
- ix. vacuum chamber
- i. Compositional analysis equipment:
 - i. Energy dispersive x-ray fluorescence spectrometer (EDXRF)*
 - ii. Spark discharge-in-argon optical emission spectrometer (SDAR-OES)*
 - iii. Scanning electron microscope with energy dispersive x-ray spectrometer (SEM/EDS)
- j. Metallographic sample preparation and examination equipment*
- k. Non-destructive testing equipment, such as:
 - i. magnetic particle inspection equipment
 - ii. liquid dye penetrant (LDP) and developer
 - iii. ultrasonic inspection equipment
- l. Mechanical testing equipment, such as:
 - i. Hardness* and microhardness* testers
 - ii. Tensile*, torsion, fatigue, impact and wear testers

6 Standards and Controls

The standards and control samples to be employed in this procedure will depend on the specific analytic methods employed and the nature of the items under analysis. Any instrument used in this procedure will use the standards required under its specific SOP. Exemplars for evidentiary items will be obtained as needed.

7 Sampling

Visual examinations are performed on every item examined under this protocol. 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 may vary on a single item, the means of selecting a location to test the characteristic will be noted in the case file.

If an item contains a large number of visually indistinguishable objects that are suitable for one analysis technique, a subset may be selected for testing by (1) non-statistical or (2) statistical means. Any sampling plan and corresponding procedure used will be recorded in 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.


(2) If a sampling plan will be used to make an inference about the entire set of visually similar items, then the plan will be based on a statistically valid approach. A hypergeometric distribution can be used to describe the probability of encountering deviations within a set of items when not every item is tested. (See Appendix A.) Appendix A assumes that all results are consistent. If inconsistent results are encountered, metallurgy conclusions regarding that characteristic will be limited to the specimens tested.

8 Procedure

The following analysis sequence was derived from guidelines established by ASM International and augmented for forensic metallurgical applications. Each of the listed analyses are not required in every situation. Further, this protocol will not be taken as a substitute for sound engineering judgment. Data gathered during examinations will be included in the case notes.

- a. Perform a preliminary visual and low magnification microscopic evaluation of the nature of comparison or item, fabrication, coating, service use/abuse, type(s) of failure, possible contamination, and any other characteristics deemed valuable. This preliminary exam serves to formulate a general concept for the approach to examination, sampling, and testing. Care should be exercised to ensure that mating fracture surfaces are not brought into contact with each other to “see if they fit” to avoid possible destruction of valuable surface information.
- b. Photograph the submitted or in-situ items in the “as-received condition” (ARC) prior to any extraction or retrieval for laboratory examinations. These photographs will record component positions, in-situ conditions, fracture and failure orientation relative to its environment and to other components, service conditions, service abuse, and any other characteristic, condition or information to be considered during the failure/damage analysis. Whenever practicable, include a scale in the photograph or apply a verified micron marker to the photograph. Additional photographs can be taken during various stages of the examinations to record more detailed characteristics of morphology, microstructure, contaminants, or other features relevant to the analysis. If the evidence is altered for examination, note the modification in the case notes.
- c. Evaluate the physical properties of the items by measuring dimensions, mass and magnetic response.
- d. Sample selection should be effected with as little damage as possible. Record with notes and photographs any information derived from the preliminary examinations which may potentially be needed to reach a conclusion but could be considered damaged or eliminated due to sample or specimen removal. Provide adequate protection of all fracture surfaces and damaged regions to prevent contact with each other, with other portions of the same component, or with other objects or items during transport and/or examination. If appropriate and feasible, package with desiccant to reduce degradation by corrosion.

- e. If available and appropriate, acquire and analyze documentary information to assist in the reconstruction of the sequence of events leading to the damage and/or failure. While such documentation can help to narrow the focus of an analysis and provide useful guidance, it is not a substitute for the physical evidence generally required to definitively establish a failure mode. Ordinarily, such information is collected by the submitting agency and may include fabrication, manufacturing and processing information; service history; interviews of eyewitness individuals; interviews with individuals whose duties, behavior or failure to act may have induced, or may have affected, the material behavior in question; as-received sample photos; site/in-situ photographs; repair history; environmental details (e.g., temperatures, loading conditions, load magnitude(s), environment chemistry); and similar component history.
- f. Prior to any specimen cleaning, perform visual and low power magnification examinations of fracture surfaces, secondary cracks, relevant surface phenomena, gross deformation, thermal damage, and any other metallurgical or environmental characteristic deemed appropriate.
- g. If appropriate and feasible, samples should be taken from the items for chemical analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested. It may be necessary to do some or all specimen retrieval prior to, or between stages of, cleaning. Analyses such as EDXRF and SEM/EDS can often be helpful in identifying the chemical compounds present on a failed component.
- h. If appropriate, clean specimen(s) using methods that progress from least to most aggressive (see Table 1 for examples) until contaminant is removed. Preserve any replica(s) or contaminant sample for appropriate analysis (see 8.g).

Table 1: Specimen Cleaning Procedures	
Increasingly Aggressive	Dry air blast
	Soft artist's brush
	Stiff non-metallic brush
	Aqueous rinse
	Organic solvent (e.g., isopropanol, hexane)
	Chelating Agent (EvapoRust™)
	Ultrasonic cleaning
	Plastic replication

- i. After cleaning, the specimen should be subjected to the same examinations referenced in step 8.e.
- j. As needed, stereomicroscopic and SEM examinations may be performed to further characterize fracture, surface features, characteristics, and any exogenous material present.
- k. Nondestructive testing (i.e., magnetic particle, LDP, ultrasonic, x-ray, and other electromagnetic evaluations) may be performed following the appropriate instrument SOP and protocol. Non-destructive testing can find unopened cracks. Such cracks can sometimes be used to make inferences about the failure mode when the fracture surfaces are too damaged to allow interpretation. They can also be used to determine how widespread a failure mechanism is in a given system. For example, a fracture in a gas pipeline may be accompanied by other, unopened cracks. If unrepaired, these can potentially lead to additional failures following repair of the known defect.
- l. As appropriate, destructive testing (i.e., hardness, tensile, impact testing) may be conducted to characterize mechanical and material properties. When possible, ASTM International standard methods should be used.
- m. If needed, metallographic examinations may be performed either before or after mechanical testing (step 8.j) for the evaluation of inclusions; microstructural segregation or inhomogeneities; decarburization; carbon pick-up; improper heat treatment; corrosion; grain size; type, distribution and morphology of microstructural constituent(s); or any other characteristics present of metallurgical interest.

- n. Consideration should be given to supplementary examinations by SEM/EDS for qualitative and/or semi-quantitative evaluation subsequent to metallographic preparation and examination. EDS can be utilized to identify elements present in different microstructure regions of interest.
- o. An evaluation of the data and facts accumulated from the above analyses performed should allow for the determination of the fracture mode or cause(s) of the damage exhibited.
- p. If appropriate, a mathematical analysis of mechanical factors leading to fracture may be used to:
 - i. predict flaw size which caused catastrophic fracture at a load below that expected to cause failure
 - ii. evaluate manufacturing flaws
 - iii. establish a quantitative framework for evaluating structural reliability
 - iv. assist in the design and prediction of service life
- q. Consideration should be given to testing similar specimens (exemplars) under simulated conditions when the history and service conditions of the questioned specimen are known.
- r. Report findings after evaluation of all gathered data.
- s. Although it is not typical for criminal cases, it may be prudent to suggest corrective measures to prevent future failures.

9 Instrumental Conditions

For instruments that require verification, standardization or energy adjustment, a copy of the appropriate record(s) will be included in the case notes.

9.1 Analytical Instruments (for SEM/EDS see 9.3)

For the instruments noted (*) in 5 Equipment/Materials/Reagents, follow the appropriate CU Metallurgy SOP (see 15 References).

9.2 Supporting Equipment

The following additional instrumental conditions also will be applied:

- a. 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.

- b. 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.
- c. The following instruments will be verified according to the appropriate CU Instrument Operations Systems Support (IOSS) SOP (see 15 References) prior to their first use to acquire case data on any given day:
 - i. traceable micrometers/calipers
 - ii. traceable balances

9.3 SEM/EDS

Compositional analysis by SEM/EDS will be conducted as follows:

- a. Prior to their first use to acquire case data on any given day, run the instrument performance verification routine according to the appropriate IOSS SOP (see 15 References). File one copy with the instrument performance records.
- b. Prepare and insert the specimen(s) ensuring electrical continuity with the sample 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 sample 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.

10 Decision Criteria

The conclusions derived from this procedure are based on careful interpretation of all factual information gathered from completed testing and investigation. A valid conclusion is one that reasonably explains the observations made during the various stages of examination. This decision will depend strongly on the nature of the evidence and the available factual information. Furthermore, more than one possible failure scenario may contribute to explain a given set of

observations. If a unique scenario does not explain the failure, all possibilities deemed relevant by the examiner will be reported in the conclusion. Conclusions will be expressed in reports and testimony according to current FBI Laboratory requirements (see 15 References).

Analysis for failure, damage, and fracture contributes to evaluating whether two or more items or materials were once part of the same object. 'Fracture 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 decision 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.

11 Calculations

A wide range of possible calculations can be encountered in a failure analysis. These are case-specific and potentially span multiple fields of engineering. The most commonly encountered types are determination of the applied structural stresses and strains, fracture mechanics calculations, and corrosion-related calculations. The references listed in section 15 of this procedure contain useful information sources for these calculations.

12 Measurement Uncertainty

When gathered, quantitative data are generally used for comparative purposes. Expanded 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).

In the event that it should be necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*. Instrumental measurement uncertainty is addressed in the individual instrument SOPs and will be calculated and reported when appropriate. Each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated.

13 Limitations

The limitations of a particular failure analysis are determined by the type, amount and condition of sample(s) being analyzed; the available background information; the specific examinations required; and subsequent determinations made, and therefore cannot be predicted within this protocol but will be reported in the case notes.

14 Safety

- a. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.
- b. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review instrument SOPs and pertinent material Safety Data Sheets (SDS) prior to conducting examinations. If additional guidance is required, contact the Laboratory Health and Safety Group.
- c. Mechanical hazards in the form of mechanical testing equipment and other machinery may require special precautions. Refer to the equipment manufacturer's guidelines regarding personal safety protocol and/or consult the Laboratory Health and Safety Group.

15 References

ASM International Failure Analysis Committee, *Handbook of Case Histories in Failure Analysis*, ASM International 1992

Wulpi, D. J., *Understanding How Components Fail*, 2nd edition, ASM International 1999

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Anderson, R. C., *Inspection of Metals, Volume I: Visual Examination*, American Society for Metals 1983

Shames, I. H. and Pitarresi, J. M., *Introduction to Solid Mechanics*, 3rd edition, Prentice-Hall Inc. 2000

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Meyers, M. A. and Chawla, K. K., *Mechanical Behavior of Materials*, 2nd edition, Cambridge University Press 2008

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Operation of the SmartScope FOV Video Measurement System, Metallurgy Manual Metal 302, Chemistry Unit, latest revision

Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), Metallurgy Manual Metal 500, Chemistry Unit, latest revision

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Metallurgy Manual Metal 400, Chemistry Unit, latest revision

Metallographic Examinations, Metallurgy Manual Metal 800, Chemistry Unit, latest revision

Operation of Rockwell Hardness Testers, Metallurgy Manual Metal 701, Chemistry Unit, latest revision

Operation of Microhardness Testers, Metallurgy Manual Metal 702, Chemistry Unit, latest revision

Operation of the Instron Model 3382 Universal Testing Machine, Metallurgy Manual Metal 703, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Balances, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Micrometers and Calipers, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Scanning Electron Microscope (SEM)-Energy Dispersive X-ray Spectrometer (EDS), Instrument Operations Systems Support, Chemistry Unit, latest revision

Rev. #	Issue Date	History
5	12/21/2018	Reformatted and expanded section 5. Added section 8c. Augmented section 9 to include specific instrument procedures. Made minor editorial corrections throughout document. Added references to section 15 and updated revision information.
6	01/29/2019	Added definition of 'fracture fit' conclusion to section 10. Updated titles of referenced documents.
7	02/18/2020	Revised section 7. Removed reference to x-ray diffraction analysis in section 8.g.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader:

Date: 02/13/2020

Chemistry Unit Chief:

Date: 02/13/2020

Appendix A: *Hypergeometric Table*

The hypergeometric table listed 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.

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

Functionality Examinations

1 Introduction

Many manufactured devices function on the basis of well-established engineering and design criteria. Consequently, when a device fails to function as intended, it is usually possible to discern the cause of the failure by careful inspection of the device's components. A common example of such activity is the diagnosis and repair of a faulty system in a motorized vehicle by an automobile mechanic. Similarly, an electronics technician might replace a faulty power supply or hard drive in order to restore a computer to proper working order.

In general, these sorts of repairs require a working knowledge of the device's operating principles. The possible causes of a particular malfunction can be diagnosed by examining the subsystems of the device and determining which of them may be responsible for the observed problem. Correction of the problem indicates it has been appropriately diagnosed.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. There are a wide variety of mechanisms, components, metals, treatments, conditions, types of damage, applications, environments, and combinations of these, as well as an unpredictable range of determinations that can be requested with regard to the failure or damage sustained and exhibited, in any particular evidence submission. The following procedure outlines the basic analyses most commonly performed in functionality testing an item or assembly.

3 Principle

Generally, it is possible to establish whether a mechanism is operating correctly by supplying the appropriate energy source to it. These can include dry cell batteries, electrical power, wind, gasoline, sunlight, pressurized gas, or another source, depending upon the device's requirements.

When the device is a sensor, it is also necessary to supply its stimulating species in order to verify its proper function. For example, a smoke detector can be tested by exposing it to a smoke source. Similarly, a radiation detector can be tested using an appropriate radioactive source material.

When a device is not functioning, it is often possible to determine why by examining it closely. For example, a broken wire in an electrical device could prevent it from working as intended. If replacement of a failed component restores its function, it may be deduced that it was the cause

of the malfunction, provided that it can also be demonstrated that another defective component did not contribute to the failure of an otherwise sound component.

4 Specimens

Nearly any mechanical device and many non-mechanical devices can be examined using this procedure.

5 Equipment/Materials/Reagents

A list of items commonly used in this examination follows. Not every item is used for every investigation. The instrumentation and equipment used will depend on the configuration of the item or mechanism to be examined for functionality. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) (15 References) for additional equipment/materials/reagents.

- a. Nikon D200 digital camera and/or Nikon DXM1200 digital camera (or equivalent)
- b. Stereomicroscope with a fiber optic light source and a magnification of at least four (4) diameters
- c. Faxitron CS-100 or NSI 5000 radiography unit (or equivalent)*
- e. Miscellaneous hand tools
- f. Digital multimeter
- g. Manually operated gas pump
- h. Graduated cylinder (500 ml)
- i. Leak detecting solution (Snoop[®] or equivalent)
- j. Miscellaneous components for substitution of missing or damaged components
- k. Compressed non-reactive gases

6 Standards and Controls

The standards and control samples to be employed in this procedure will depend on the specific analytic methods employed and the nature of the items under analysis. Exemplars for evidentiary

items will be obtained as needed. Any instrument used in this procedure will employ such standards as are required under its specific SOP (see section 15 References).

7 Sampling

Not applicable.

8 Procedure

- a. Perform a preliminary examination and note any apparent shipping damage as well as any material transfer from shipping and/or handling. Record any dial/gauge readings and make an evaluation as to type of device including the nature of the input and output (e.g., gas, electrical power, fluid). Record any valuable manufacturer's markings, visible or restorable.
- b. Photograph the "as received condition" (ARC) of the specimen, noting the overall condition of the appliance or item, any damage exhibited, and the spatial relationship of the controls and components.
- c. If appropriate, perform radiographic examination of the internal components of the device.
- d. If feasible and appropriate, obtain a comparable, undamaged device (exemplar) for examination and comparison.
- e. Conduct both visual and low power magnification examinations to assess the totality of the item and/or system, its integrity and the specific relative position(s) of controls, control components, and other functional components. Examine any exhibited damage, exogenous debris, material, or item not present by design and any post-production modifications. In addition, note any missing components and any other characteristic of interest or value.
- f. Check electrical, flow, or other appropriate continuity with a multimeter, pump or other appropriate detection device(s). Make note of any relevant circuit, system, and/or fluid behavior.
- g. If the device is electrically activated, gradually apply input voltage until device activation or full-rated power is reached. Observe voltmeter, ammeter, and/or ohmmeter at various locations in circuits as necessary. Note the threshold voltage of activation if testing an audible alarm device.

- h. If the device is flow-activated, pass gas through the device to detect any sources of leakage. Leakage in the pressurized device may be detected by submersion, audibly, or by spraying the pressurized portions of the item with soapy water¹ or other leak detection solution (e.g., Snoop®). Note: If leakage is detected, measure the volume of escaping gas and note duration of collection (elapsed time). One method of doing this involves using a graduated cylinder which has been submerged then inverted over the leak. If no leak is detected, pressurize the system to its rated pressure and observe for leak(s) and/or proper functioning.
- i. Summarize findings based on all collected data of value.

9 Instrumental Conditions

- a. The instrumental conditions of imaging systems are generally adjusted by the operator to achieve sufficient resolution for analysis. See section 15 References for SOPs containing additional recommendations of instrumental conditions for digital radiography.
- b. Macro- and micro-photographs will contain a reference scale whenever feasible, however these are included for general reference, and measurements will not be taken 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.

10 Decision Criteria

The conclusions derived from this procedure are based on careful interpretation of all of the factual information gathered from testing and investigation. A valid conclusion is one which reasonably explains the observations made during the various stages of examination. In some cases, the proper functioning of a device is self-evident. In other instances, it is possible to infer the functioning (or malfunctioning) of a device based on an analysis of its physical remains. The uncertainty associated with such an analysis will depend strongly on the nature of the evidence submitted and the available factual information. Conclusions will be expressed in reports and testimony according to current FBI Laboratory requirements (see section 15 References).

11 Calculations

The range of possible calculations that may be encountered in functionality evaluations is case-specific and potentially spans all engineering disciplines. No particular calculation is routinely

¹ The use of soap solutions may tend to remove any residues which may be present on the surfaces of the device.

used for this examination. Examples of some of the types of calculations which may be encountered are available in the listed references.

12 Measurement Uncertainty

When quantitative data are gathered to define the limits of functionality of an item or assembly, measurement uncertainty will be calculated in accordance with the *Procedures for Estimating Measurement Uncertainty* in the *Chemistry Unit Quality Assurance and Operations Manual*.

13 Limitations

The limitations of a particular functionality test are determined by the device condition and type, the available background information and numerous other factors specific to the situation under consideration. Although specific limitations cannot be predicted within this procedure, any limitations encountered during functionality examinations will be recorded in the case notes, and, if appropriate, included in the *Laboratory Report*.

14 Safety

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

15 References

Wolf, S., *Guide to Electronic Measurements and Laboratory Practice*, 2nd edition, Prentice-Hall, Inc. 1983

Scatler, N., *Mechanisms and Mechanical Devices Sourcebook*, McGraw-Hill 2011

Parmley, R. O., *Machine Devices and Components Illustrated Sourcebook*, McGraw-Hill 2004

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Digital Radiography, Metallurgy Manual Metal 303, Chemistry Unit, latest revision

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

Rev. #	Issue Date	History
4	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 3 and is now designated Metal 201. Added personnel to section 2. Made minor editorial corrections throughout document. Deleted obsolete equipment in section 5. Augmented section 6. Deleted section 7 and renumbered subsequent sections. Removed example from section 10. Added measurement uncertainty statement to section 12. Updated safety requirements in section 14. Added references to section 15.
5	12/21/2018	Added paragraph regarding equipment use to section 5 heading. Added reference to instrument SOPs in sections 5, 6 and 9. Removed micrometers and added digital radiography to section 5. Removed reference to NIST standards for dimensional measurement from section 6. Added 8c to include radiography. Renumbered following steps in section 8. Augmented section 9 to include specific instrument procedures. Added statement regarding conclusions to section 10. Added SOP references to section 15.

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Approval

Metallurgy Technical Leader

Date: 12/20/2018

Chemistry Unit Chief

Date: 12/20/2018

QA Approval

Quality Manager

Date: 12/20/2018

Examinations of Timing Mechanisms

1 Introduction

Watches, appliance timers and similar devices are encountered in a wide variety of criminal circumstances. For example, altered watches are commonly used as timers in explosive devices. Similarly, appliance timers are frequently encountered in arson investigations. Careful examination of such a device can reveal a variety of useful information regarding the timer and its possible alteration.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. There are a large variety of watches or other timing mechanisms. These are subject to a myriad of conditions of environmental exposure, types of damage (for example, impact, corrosion, and/or compression damage), and applications (wristwatches, kitchen timers, appliance timers, etc.). Combinations of all of these variables are considered in these examinations. Principles of various aspects of materials science may be required to evaluate material and component behavior depending on the determination(s) requested.

3 Principle

Watches, appliance timers and similar devices are encountered in a wide variety of circumstances in criminal matters. Commonly, the timers are relatively simple mechanical or electro-mechanical devices. Careful inspection of the device permits a number of characteristics to be determined. These include the manufacturer (and/or brand) of the item, the time and date the mechanism ceased functioning, the cause of mechanism cessation, and the identity of jeweler's or owner's markings. Comparison of the device with exemplars also makes it possible to determine if the mechanism has been altered. Moreover, it is often possible to deduce the reasons such alterations may have been made.

4 Specimens

This procedure is designed for the examination of time and interval indicating devices (e.g., alarm clocks, analog wristwatches, electrical timers) and the components comprising such mechanisms.

5 Equipment/Materials/Reagents

A list of items commonly used in this examination follows. Not every item is used for every investigation. The instrumentation and equipment used will depend on the configuration of the timing mechanism to be examined. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) (see section 15 References) for additional equipment/materials/reagents.

- a. Nikon D200 digital camera and/or Nikon DXM1200 digital camera (or equivalent)
- b. Stereomicroscope having a fiber optic light source and a magnification of at least four (4) diameters
- c. Faxitron CS-100 or NSI 5000 radiography unit (or equivalent)*
- d. Fluke digital multimeter (or equivalent)
- e. Long (~365 nm) and short (~254 nm) wavelength ultraviolet (UV) light source(s)
- f. Alternative light source with filters and goggles
- g. Miscellaneous hand tools
- h. Battery

6 Standards and Controls

The standards and control samples to be used in this procedure will depend on the specific analytic methods employed and the nature of the items under analysis. Any instrument used in this procedure will employ such standards as are required under its specific SOP (see 15 References). Exemplars for evidentiary items will be obtained as needed.

7 Sampling

Not applicable.

8 Procedure

- a. Conduct a preliminary evaluation of specimen condition to assess the condition of crystal and face, the time displayed by mechanism immediately upon receipt, and any apparent

shipping damage or material transfer due to shipping and handling. Also, note if mechanism is functioning or attempting to function.

- b. Photograph the specimen(s) in the “as received condition” (ARC). Photodocumentation should depict a fair and accurate representation of characteristics deemed to be significant to the determination requested.
- c. If deemed appropriate, perform x-ray radiography of the mechanism to evaluate the movement type, the component positions, any internal damage, the power source, as well as any other information deemed to be of value for the determinations requested.
- d. Conduct stereomicroscopic examinations of the specimen(s) in ARC to evaluate any damage present, the mechanism type, any identification markings, any exogenous deposits, as well as any other information deemed of value for the determinations requested.
- e. Examine the timer for, and note any damage consistent with, impulsive loading if appropriate. These examinations are conducted using multiple lighting conditions. First examine the timer under ambient light to look for any characteristics associated with hand/dial interaction caused by forced contact. Next, examine the relevant timer surfaces with fiber optic lighting using as many incident angles as possible (particularly low angle) looking for indications of hand/dial interaction by forced contact to determine the time displayed by the mechanism when impulsively loaded. Then repeat the surface examinations in a darkened room using long and short wavelength UV lighting looking for characteristics of hand/dial interaction by forced contact, including luminous or phosphorescent material transfer between the surfaces.
- f. Note the presence and nature of corrosion products, if any.
- g. Perform radiographic examination of the internal components of the device.
- h. Conduct an internal visual examination of the movement and cavity noting any jeweler's markings, movement plate markings, and the condition of the mainspring or the internal power source. Note any calendar (day/date) position(s) as well as any component damage, blockage and/or misalignment.
- i. If appropriate, remove timer power source and measure the source's terminal (no load) voltage.
- j. Loss of power due to winding down of the mechanism or an exhausted battery can be ascertained by replacing the battery or winding the mechanism as appropriate.
- k. Prepare findings after evaluation of all data determined to be of value.

9 Instrumental Conditions

- a. The instrumental conditions of imaging systems are generally adjusted by the operator to achieve sufficient resolution for analysis. See section 15 References for the CU Metallurgy SOP that contains additional recommendations of instrumental conditions for digital radiography.
- b. 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 image features unless the marker is verified against a calibrated scale.

10 Decision Criteria

A conclusion that a damaged timer stopped at a particular time can often be read directly from the device if it remains intact. In other cases, damage to the timer from fire, explosion, corrosion or other effects will leave physical marks on the mechanism that allow the hand positions to be inferred from the observed characteristics. Conclusions will be expressed in reports and testimony according to current Laboratory requirements (see section 15 References).

11 Calculations

Not applicable.

12 Measurement Uncertainty

Not applicable.

13 Limitations

Time and date determinations on non-functioning timepieces are typically limited to those having analog-type displays.

14 Safety

- a. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent

inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.

- b. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review instrument SOPs and pertinent material Safety Data Sheets (SDS) prior to conducting examinations. If additional guidance is required, contact the Laboratory Health and Safety Group.

15 References

Penman, L., *The Clock Repairer's Handbook*, Skyhorse Publishing 2010

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Digital Radiography, Metallurgy Manual Metal 303, Chemistry Unit, latest revision

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3	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 3 and is now designated Metal 202. Added personnel to section 2. Made minor editorial corrections throughout document. Deleted obsolete equipment in section 5. Added exemplar statement to section 6. Deleted section 7 and renumbered subsequent sections. Updated safety requirements in section 14. Added references to section 15.
4	12/21/2018	Added paragraph regarding equipment use to section 5. Added reference to instrument SOPs in sections 5, 6 and 9. Augmented section 9 to include specific instrument procedures. Added statement regarding conclusions to section 10. Added SOP references to section 15.

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Approval

Metallurgy Technical Leader .

Date: 12/20/2018

Chemistry Unit Chief .

Date: 12/20/2018

QA Approval

Quality Manager .

Date: 12/20/2018

Lamp Bulb Examinations

1 Introduction

In automotive accidents, the question of whether both parties had their headlights or taillights illuminated frequently arises. Similarly, the question of whether a vehicle was moving in reverse may arise under certain circumstances and can sometimes be answered by examining the back-up lights in the rear of the vehicle. Examinations of automotive lamps frequently allow strong inferences to be made regarding the operating condition of the lamps at the time of an accident.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. Material response to an applied force typically varies with temperature, strain rate, and conditions of static and dynamic loading. Examination of a lamp that has been subjected to impact loading can often support an inference as to its operating condition (on/off) during the event. When in service, lamp bulbs operate at such extremes of temperature that impacts during accidents can permanently alter the physical characteristics of its components. Exposure of the lamp's internal components to the atmosphere can also produce characteristic changes. Alterations to the lamp components caused by impact and any resultant environmental exposure can be observed by forensic examination and used to infer the lamp's operating condition at the time of an impact.

3 Principle

Vehicle lamp filaments are made of tungsten and are normally brittle at room temperature but are quite ductile at operating temperatures. By examining the physical characteristics of a lamp filament, it is often possible to determine the operating condition when it was subjected to an impact of the type encountered in a collision. For example, a cleavage (brittle), intergranular or fibrous fracture indicates the lamp was off when the filament broke. Severe deformation of a filament similarly indicates that it was hot when impacted since tungsten is usually not ductile at room temperature.

Moreover, tungsten is not oxidation resistant. Consequently, if the glass surrounding a tungsten filament breaks while the lamp is on, severe oxidation of the filament will occur. Furthermore, small fragments of the glass envelope will commonly melt, then become fused to the filament when it cools. By careful inspection of these characteristics, it is frequently possible to determine the operating condition of a lamp when it was subjected to an impact.

4 Specimens

Specimens for this examination generally consist of various lamp bulbs (e.g., automotive, household, commercial, aviation, marine, electronic).

5 Equipment/Materials/Reagents

A list of items commonly used in this examination follows. Not every item is used for every lamp investigation. The instrumentation and equipment used will depend on the configuration of the items to be examined. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) (See section 15 References) for additional equipment/materials/reagents.

- a. Nikon D200 digital camera and/or Nikon DXM1200 digital camera (or equivalent)
- b. Stereomicroscope having a fiber optic light source and a magnification of at least four (4) diameters
- c. Ethyl or methyl alcohol (laboratory grade)
- d. Faxitron CS-100 or NSI X5000 radiography unit (or equivalent)*
- e. Glass-scoring instrument
- f. Oxyacetylene mini-torch
- g. Tissue, napkins or suitable water-retaining and shapeable medium
- h. Needle-nose pliers
- i. Welder's glasses
- j. Fire retardant apron
- k. Puncture resistant gloves
- l. Rotating dish or 'Lazy Susan'-type platform (optional)
- m. Electric charcoal starter (optional)
- n. Fluke digital multimeter (or equivalent)
- o. Scanning electron microscope (SEM)

- p. SEM with energy dispersive X-ray spectroscopy (SEM/EDS)

6 Standards and Controls

The standards and control samples to be employed in this procedure will depend on the specific analytic methods used and the nature of the item under analysis. Any instrument used in this procedure will employ such standards as required under its specific SOP (see section 15 References). Exemplars for comparison to evidentiary items will be obtained as needed if possible.

7 Sampling

Not applicable.

8 Procedure

- a. Conduct a preliminary evaluation of the specimens noting the type of lamp bulbs represented, the condition of lamp bulbs or remnants, and the nature of any damage exhibited.
- b. Review any supplied photographs of the accident and/or damaged vehicle(s) to evaluate the spatial relationship between the lamp bulb damage and the damaged regions of vehicle(s) in order to infer possible impact energy transmission and attenuation.
- c. Photodocument the specimens to record the “as-received condition” (ARC) of the submitted lamp bulbs or remnants to characterize the presence and condition of both the glass envelope and the resistance element (filament). Also record the 3-dimensional spatial relationship of any electrical components present and the presence of any exogenous debris that may be detected by macroscopic and/or microscopic examination.
- d. X-radiographic examinations should be considered for any automotive sealed beam headlamps (or any other lamp type for which it is deemed appropriate) with an unbroken glass envelope or intervening material obstructing direct visual and microscopic observation of any electrical elements present or of the location(s) such elements are expected to occupy.
- e. Perform visual and low power magnification examinations of the lamp bulbs to observe and record the condition of envelope, the type of bulb and any manufacturer's markings on the bulb or base. Record any observations of the symmetry and deformation exhibited

by filament(s), any filament discoloration, as well as any other information deemed to be of value.

- f. After the state of the filament(s) has been definitively determined, check the electrical resistivity (or conductivity) of the electrical element(s) with a multimeter for static and dynamic electrical continuity. The lamp should be moved along each of its three principal axes during the dynamic continuity test to account for the possible presence of mechanically contacting fracture surfaces on the filament. The three principal axes are typically oriented parallel to the length of the lamp, parallel to the length of the filament, and in the direction which is mutually perpendicular to these two axes. Exact orientation is not critical. However, filament fracture may not be detected if motion is only effected parallel to the long axis of the filament.
- g. Microscopic examinations should be conducted of the lamp bulb filament(s) at higher magnification in the ARC for the presence of deformation, fused glass, fracture(s), bulbous ends, wire recrystallization (to evaluate service life), characteristics of arcing, welds, and any other information of foreseeable value.
- h. If the lamp bulb glass envelope is apparently intact, or if microscopic examination of the fracture surfaces is desired, careful removal of the glass envelope to expose the electrical elements for microscopic examination may be required. This will be done in a manner least likely to damage or alter any electrical elements or residues present within the bulb. The following technique has proven to be effective and introduces no detectable damage or alteration to critical components if performed properly. However, it is only one of several techniques available for effective envelope separation.
 - i. If the specimen is an automotive lamp bulb, equalize the internal protective environment pressure by breaking the gas inlet site (generally prominent between the lugs) with needle-nose pliers or other suitable tool. If the specimen is a different type of bulb, a pinhole may be drilled into the metal base to allow pressure equalization. For safety reasons, equalizing the internal pressure is a very important step.
 - ii. Score the circumferential periphery of the glass envelope completely to establish a localized triaxial state of stress.
 - iii. Wrap a cool, moist tissue paper around the bayonet base or other site where low melting temperature material (for example, solder) is present from fabrication. This will assist in establishing a steep thermal gradient in the vicinity of the stress concentration while simultaneously protecting low melting temperature material.
 - iv. Using the charcoal starter or oxyacetylene torch, heat the stress concentration region as quickly as feasible. If an oxyacetylene torch is used, the lamp must be rotated on a rotating table to evenly distribute the heat.

- v. Remove the glass envelope when it cracks.
- vi. Conduct a second stereomicroscopic evaluation as detailed in step 8.f.
- i. If the fracture mode of the filament cannot be determined with a stereomicroscopic examination, SEM imaging will be considered to characterize any filament failure as to the ductile-brittle behavior, the fractographic features and any other characteristic which may indicate or otherwise assist in determination of conditions existing at failure. In addition, SEM/EDS may be used to analyze the composition of the various components and any deposits.
- j. Make a determination based on results of all of the examinations.

9 Instrumental Conditions

9.1 Imaging Systems

- a. The instrumental conditions of imaging systems are generally adjusted by the operator to achieve sufficient resolution for analysis. The SEM is most often operated at 25 kV in the secondary electron imaging mode, but accelerating voltage may be reduced to resolve fracture surface features. Backscattered electron imaging can be useful for locating transfers of material having a different average atomic number than the substrate. See section 15 References for the SOP that contains additional recommendations of instrumental conditions for SEM and digital radiography.
- b. 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.

9.2 SEM/EDS

Compositional analysis by SEM/EDS will be conducted as follows:

- a. Run the instrument performance verification routine. File one copy with the instrument performance records.
- b. Prepare and insert the specimen(s) ensuring electrical continuity with the sample 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 sample 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.

10 Decision Criteria

The conclusions derived from this procedure are based on careful interpretation of all factual information gathered from testing and investigation. A valid conclusion is one that reasonably explains the observations made during the various stages of examination. The uncertainty associated with such an analysis will depend strongly on the condition of the evidence. Occasionally, more than one possible scenario may explain a given set of observations. If a unique scenario does not explain the observations, all reasonable possibilities should be appropriately reported in the conclusion.

Although it may be possible to determine that a bulb was lit at the time of impact due to its deformation/damage, it is often not possible to determine if it was due to the impact in question or a previous accident.

Conclusions will be expressed in reports and testimony according to current Laboratory requirements (see section 15 References).

11 Calculations

Not applicable.

12 Measurement Uncertainty

Not applicable.

13 Limitations

This protocol is not suitable for the examination of lamps not having filaments (e.g., LED, fluorescent light bulbs).

14 Safety

- a. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.
- b. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review pertinent material Safety Data Sheets (SDS) prior to conducting examinations. If additional guidance is required, contact the Laboratory Health and Safety Group.
- c. Observe fire safety precautions when oxyacetylene torch equipment is used for envelope separation: use a spotter, ensure the availability of fire extinguishers in close proximity and wear a flame-retardant apron and gloves. Fire safety precautions are more specifically described in the *FBI Laboratory Safety Manual* maintained by the Laboratory Health and Safety Group.
- d. The glass envelopes of lamp bulbs are usually pressurized or evacuated. Appropriate eye protection and gloves must be worn when breaching the envelope.

15 References

Baker, J. S., Aycock T. L., and Lindquist, T., *Lamp Examination for ON or OFF in Traffic Accidents*, Topic 823 of Traffic Investigation Manual, Northwestern University Traffic Institute, 1985

Noon, R. K., *Engineering Analysis of Traffic Accidents*, CRC Press, pp. 83-91, 1994

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Methodology for Report Writing in the Metallurgy Discipline, Metallurgy Manual Metal 900, Chemistry Unit, latest revision

Chemistry Unit (CU) FBI-Approved Standards for Scientific Testimony and Report Language for the Metallurgy Discipline, Metallurgy Manual Metal 901, Chemistry Unit, latest revision

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

Digital Radiography, Metallurgy Manual Metal 303, Chemistry Unit, latest revision

Rev. #	Issue Date	History
4	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 5 and is now designated Metal 203. Added personnel to section 2. Made minor editorial corrections throughout document. Deleted obsolete equipment in section 5. Added statement added to section 6. Deleted section 7 and renumbered subsequent sections. Clarified section 10. Updated safety requirements in section 14. Added references to section 15.
5	12/21/2018	Added paragraph regarding equipment use to section 5. Added reference to instrument SOPs in sections 5, 6 and 9. Moved scene documentation review to earlier in the examination process (8b) and renumbered following paragraphs. Augmented section 9 to include specific instrument procedures. Added statement regarding conclusions to section 10. Added SOP references to section 15.

Redacted - Signatures on File

Approval

Metallurgy Technical Leader _

Date: 12/20/2018

Chemistry Unit Chief _

Date: 12/20/2018

QA Approval

Quality Manager _

Date: 12/20/2018

Operation of the SmartScope FOV Video Measurement System

1 Introduction

The SmartScope FOV (Optical Gaging Products, Inc.) is a video measuring microscope system that uses calibrated optics and a high precision linear translation table to produce non-contact, dimensional measurements of objects. The instrument produces measurements in three dimensions which are both more accurate and more precise than those obtainable using standard mechanical devices such as micrometers and calipers.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. The SmartScope FOV video measurement system is used for measuring physical dimensions and surface features of a wide variety of objects.

3 Principle

The instrument determines planar dimensions by reference to a highly accurate and precise linear translating stage on which the object being measured is placed. Calibrated lenses and software algorithms permit accurate measurement of planar spacing and height differential between selected points or features. Although lenses of various magnifying capacity are available to provide different image areas (fields of view), the selected points to be measured do not have to be within a single field of view (FOV). Illumination options provide the user the ability to control the intensity, direction and incidence angle of light and optimize the image for measurement. The software algorithms use the calibrated magnification factors to determine the size of the region within the instrument FOV at a given magnification. Physical measurements are made by selecting two or more points on a feature of interest and comparing the relative distance between them to the known dimensions of the FOV and the difference in lens height at optimal focus. Distances greater than the FOV are determined by reference to it and to the stage displacement. Edge detection and automated focusing routines permit highly reproducible feature selection.

4 Specimens

Imageable features of solid objects that are small enough to fit between the sample stage and lens apparatus can be analyzed by this method. The features to be measured must be perpendicular to the optical axis. Rough surfaces and irregular edges can affect measurement accuracy. Specimens should be free of any loose debris where in contact with the stage and at all measurement points.

5 Equipment/Materials/Reagents

- a. Certified microrule
- b. Calibrated grade B-standard thickness block set
- c. Microsoft Excel, MINITAB software or other statistical calculating tool

6 Standards and Controls

The instrument contains a built-in, NIST (National Institute of Standards and Technology) traceable, optical reference filar. The instrument determines its magnification factor automatically by reference to this filar. This internal calibration process is verified by reference to measurements made on a certified microrule and/or grade B-standard thickness blocks.

7 Calibration and Verification

The system is calibrated annually by a certified and licensed service provider that meets the FBI Laboratory Operations Manual (LOM) requirements. During normal operation, the lens magnification is auto-calibrated by the instrument within its measurement routine. Prior to the first use of a particular lens to make a significant measurement on a given day, the user will verify the calibration and enter the verification results in the instrument log book. Thickness reference standards are used to verify height differential, and a certified microrule is used to verify planar distance.

8 Sampling

Sampling of items examined under this protocol is determined by the nature of the evidence and can consist of multiple items or one or multiple regions of interest on one item. If large numbers of physically indistinguishable items are received for testing, a sampling plan may be employed. If the sampling plan will be used to make an inference about the entire set of items, then the plan will be based on a statistically valid approach. Every item may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be documented in case notes.

9 Procedure

- a. Select the appropriate lens. Image the feature of interest (FOI) on the sample (or reference material) using the MeasureMind 3D MultiSensor software tools. Adjust the magnification as needed using the joystick or software controls.

- b. Adjust the lighting to optimize the image using the appropriate light source. Available light sources include coaxial lighting from above, a ring light with selectable lighting directions and incidence angles, or transmitted light from below. Adequate brightness and contrast can typically be achieved by adjusting the light level to approximately 40 - 65%.
- c. Focus the image using the joystick or select the focus tool that is appropriate for the FOI from the Toolbox icons.
- d. To set the stage reference position, move an appropriate point of the FOI to the screen centerline, focus and zero the X, Y and Z values.
- e. Select the measurement mode and a measurement tool from the Toolbox icons. (The specific function of each icon can be determined by hovering over the icon with the cursor.)
- f. Move the stage to the first point to be recorded. (The software contains edge finder functions which allow optimal selection of the feature edge being measured). Record the point using the "Enter" button on the joystick platform. Relocate the stage to the next point of interest and record the next point until the feature is represented.
- g. When the correct number of points has been entered, the software will automatically display the result. For example, if three points are entered along the edge of a circle, the software will fit a curve through the points and generate a diameter measurement. To collect an additional measurement of the same FOI, select "Again".
- h. To measure planar (X-Y) dimensions, no refocusing is required between point acquisitions. For Z (height) measurements, use the autofocus function to focus on the surface at each height of interest.
- i. Record the measurement values in the case notes.

10 Instrumental Conditions

The principal instrumental condition to be set is the lighting level. Multiple lighting sources are present on the instrument (see section 9 Procedure). The one which gives the best FOI image should be used in a given situation. A light level between approximately 40 and 65% typically produces adequate contrast for the autofocus and edge finder routines to properly function. The light level is displayed on screen and can be adjusted in live time.

11 Decision Criteria

The instrument is considered to be functioning correctly if the mean value of five or more measurements generated on a standard gauge block falls within the 99.7% confidence interval for its certified values or 0.0001 inches, whichever is larger. If the measured values do not agree adequately, a second series of five measurements will be taken. If these also do not agree within the specified tolerance, the instrument will be serviced and recalibrated by a certified and licensed service provider that meets the LOM requirements.

12 Calculations

12.1 Quantitative Analysis

- a. Feature characteristics (such as diameter, line length, or radius of curvature) are automatically calculated by the instrument software from the position of the stage and lens at each of the points entered for that feature. These readings, and their associated measurement uncertainty, may be used to report a range or series of measurements.
- b. To report averaged measurements, collect at least five values. Calculate and report the mean and expanded measurement uncertainty.

Mean is calculated as: $\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$, where $\sum_{i=1}^n x_i$ is the sum of the measurements, n is the number of measurements and \bar{x} is the mean dimension.

12.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed Student-t test statistic of the sample means is typically used for the comparison. Two samples are deemed to be “indistinguishable” in the property under consideration if the two samples differ by less than the preselected critical t value (t_{critical}). The critical t value is typically chosen so that a value of $\alpha = 0.05$ can be achieved for the analysis and is determined by the degrees of freedom associated with the measurements. An $\alpha = 0.05$ means that there is a 5.0% chance of incorrectly rejecting a match between two samples when one actually exists.

To perform this test, the sample means and sample variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where \bar{x}_a is the average value of the measurement on sample “a”,

$\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample. The variance of the individual measurement values from sample “a” is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

The mean and variance of the sample “b” data are calculated in an analogous manner.

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 \left(\frac{1}{n_a} + \frac{1}{n_b} \right)} \right)} \right| > t_{critical}$, the samples have a statistically significant difference. If not, the

samples are deemed to be indistinguishable.

In general, the number of individual measurements required per sample is determined by the population data distribution. If the sample populations are known to be, or can reasonably be assumed to be, normally distributed (Gaussian), as few as three measurements per sample can be used to compare the results. However, an increased number of measurements will provide an improved measurement standard deviation estimate. Typically physical dimension measurements are normally distributed.

In the majority of instances where the measurement populations are not normally distributed, 5-10 measurements per sample will result in sample means that are approximately normal and will be adequate for the comparison outlined above. For heavily skewed population distributions, a minimum of 30 measurements per sample may be required to achieve this. Heavily skewed data distributions will normally be detectable on inspection of the sample data. Statistical tests also exist for determining whether data are Gaussian or non-Gaussian and can be employed as they are needed. Commonly, a normal probability plot is constructed for this purpose using statistical software packages such as MINITAB.

13 Measurement Uncertainty

Quantitative data from this procedure are typically used for comparative purposes. Expanded 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). Should quantitative reporting be required, the measurement uncertainty will be estimated in accordance with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty* in the CU QAM.

14 Limitations

Since measurement relies on imaging the specimen, the instrument cannot measure internal cavities. In order for a feature to be measured accurately, it must be perpendicular to the objective lens. Accurate height (Z) measurements require surfaces that respond well to the autofocus software routine.

15 Safety

Standard safety precautions, such as wearing protective gloves, should be observed when handling evidentiary materials. Electrical or mechanical hazards may require special precautions.

This instrument SOP has the following specific safety requirements:

- The motorized stage is computer-driven. Ties and other loose clothing should not be worn when operating it, and long hair should be tied back. If entanglement occurs, a red panic button labeled “STOP” can be used to interrupt the instrument power and halt the stage motion.
- The halogen sample illumination lamps are extremely hot when operating and should never be touched unless adequate time has been allowed for cooling.

16 References

MeasureMind Graphical Measurement Software Reference Guide, Part No. 790040, 5th Printing, Optical Gaging Products, 850 Hudson Ave., Rochester, NY 14621-4896

MeasureMind 3D MultiSensor Software Fast Start Guide, Part No. 790321, Optical Gaging Products, 850 Hudson Ave., Rochester, NY 14621-4896

SmartRing Service and Maintenance Guide, Part No. 790136, Optical Gaging Products, 850 Hudson Ave., Rochester, NY 14621-4896

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, most recent revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, most recent revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, most recent version

Rev. #	Issue Date	History
3	04/24/2014	Minor grammatical changes made to sections 3, 7 and 12. Language in section 6 updated. Service interval in section 7 changed from 6 months to annually. Calculation for sample standard deviation simplified in section 12. Corrections made to the remaining equations in section 12. Section 13 has been rewritten to reflect updated measurement uncertainty requirements. References updated in section 16.
4	12/21/2018	Renumbered Metallurgy SOP Manual documents; this document was formerly Metal 13 and is now designated Metal 302. Added personnel to section 2. Made minor editorial corrections throughout document. Updated section 7 to include verification. Added requirements for sampling plan in section 8. Revised measurement steps in section 9. Updated confidence interval and service requirements in section 11. Updated measurement uncertainty policy in section 13. Revised sections 4, 5, 14 and 15. Added additional references to section 16.

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Approval

Metallurgy Technical Leader

Date: 12/20/2018

Chemistry Unit Chief

Date: 12/20/2018

QA Approval

Quality Manager

Date: 12/20/2018

Digital Radiography

1 Introduction

X-ray radiography is useful for non-destructive inspections of the interior of objects. This technique can reveal internal components of assemblies and defects in materials for examination and assessment. Features are exposed based on differences in transmitted intensity of an x-ray beam through variations in thickness and material.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: firearms/toolmarks examinations and general physical and chemical analysis in support of metallurgy or anthropology examinations. Radiography is a powerful imaging technique that can be applied to a wide variety of objects and assemblies. Because of this versatility, the conditions associated with imaging a particular object are optimized during the operation of the radiograph. The following procedure outlines only the basic steps to acquire a digital radiograph (2-dimensional) with the Faxitron CS-100AC and NSI X5000 radiography systems and a computed tomography (CT) scan (3-dimensional) with the NSI X5000 system. Additional information and options for computed tomography are available from the extensive software manual which accompanies the NSI X5000 system. Under no circumstances should either x-ray system ever be independently operated by untrained personnel.

3 Principle

Digital radiography utilizes the same operating principles as traditional film radiography. An object of interest is exposed to an intense source of x-rays. These are absorbed by the components comprising the object to a degree which depends on the atomic weight and thickness of the components. An electronic detector behind the object measures the spatial distribution of the transmitted x-rays and produces an electronic image whose brightness and contrast depend on the point-to-point variation of the x-ray intensity. This electronic image can then be used to make inferences regarding the internal structure of the object without opening it.

CT uses a series of acquired x-ray images to generate a virtual 3-D representation of the x-ray transparency variations throughout the object. Visualization software allows this 3-D volume to be examined from any angle and at any depth from the object's surface.

The Faxitron CS-100AC uses a micro-focus x-ray source with fixed spot size for high-resolution 2-D imaging. The distances of the source and detector from the sample are adjustable, allowing optimization of field of view (FOV), focus and magnification. The maximum voltage of the x-ray tube is 90kV. Typically, this system can successfully image fine components within thin metal casings.

The NSI X5000 system provides capacity for both 2-D and 3-D x-ray imaging using either of two x-ray sources: a 225 kV micro-focus x-ray tube or a 450kV x-ray tube. The distances of the sample and the detector from the source are adjustable, allowing optimization of FOV, focus and magnification. The detector and source height are also adjustable. Automated positioning software allows acquisition of mosaic images of large specimens in 2-D. Combined with automated sample rotation, helical image acquisition can be used for 3-D CT scans of large specimens.

4 Specimens

Specimen size is limited by the x-ray system chamber size and the weight capacity of the sample platform. The material of construction is only important insofar as it affects the ability of the x-ray beam to penetrate the object. If the object construction is excessively thick-walled, a satisfactory image may not be obtained. See also 13 Limitations in this document.

The CS-100AC accommodates samples up to 18" x 24" x 8 ½" height on a moveable stage and has a 90kV maximum x-ray energy with a very small focal spot (10 microns or less.)

The NSI X5000 can accommodate larger samples, up to 200 lbs. Stage, detector and source(s) are all moveable to allow an area of interest in a large part to be imaged. The 225 kV microfocus tube has focal spot <6 µm at 320W and the 450 kV tube has a focal spot ~0.4 mm at 700W.

5 Equipment/Materials/Reagents

- a. Digital x-ray radiography system (Faxitron CS-100AC, NSI X5000, or similar):
Each unit consists of a lead-lined chamber, x-ray tube(s), power supply and controller, sample positioning table, a digital x-ray detection panel and a computer workstation. The NSI X5000 has an additional computer to construct 3-D CT images
- b. X-ray filters (optional) – typically copper or aluminum sheets, but other materials may be

used at the operator's discretion. Optimum sheet thickness depends on imaging conditions.

- c. Tools for supporting specimen(s) on the sample table – these may include modeling clay, clamps, Styrofoam blocks, sandbags or other assorted objects as needed. Beeswax is a useful low atomic number mounting medium
- d. For CT:
 - i. anti-vibration mat to place under specimen
 - ii. alignment reference standard

6 Standards and Controls

Although standards and controls are not required for basic operation and subsequent examination of 2-D digital radiographs, thickness and curvature standards of similar atomic number to the materials being examined can provide useful comparisons during analysis.

The NSI X5000 detector requires gain adjustment to remove background electronic noise and improve the appearance of the acquired digital radiograph. Failure to adjust can result in electronic artifacts appearing in the image. Gains are adjusted with respect to the conditions at which the object will be imaged, so changing the imaging conditions may require readjustment of the gains in order to ensure optimal image quality. The NSI X5000 software also accommodates removal of dead pixels from the image. The pixel clamping routine should be run daily when the instrument is in use.

Computed tomography requires alignment using reference materials that are provided by the manufacturer and exclusive to the particular software used to generate the 3-D reconstruction.

7 Sampling

If accommodated by the x-ray system, an entire object or region of interest can be imaged. For CT scans, limiting the image acquisition to a minimum region of interest can significantly reduce acquisition/computation time, allowing possible improvement in resolution (smaller increments between acquired images) or throughput. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

- 8.1** Warm up the x-ray tube.
- 8.2** Power down the x-ray tube in order to place or exchange samples in the chamber.
- 8.3** When inserting the sample(s), consider imaging orientation. For symmetrical objects, angle the planes of symmetry in the beam path (source-to-detector) in order to provide more information than parallel or perpendicular alignment.
- 8.4** Close the x-ray sample chamber in order to energize the x-ray tube.
- 8.5** Adjust voltage and current (amperage) to obtain a satisfactory image (see 9 Instrumental Conditions).
- 8.6** Adjust FOV, magnification, focus, distance to source and/or detector, power and other parameters to obtain the optimal image for the application. See 10 Decision Criteria for further information.
- 8.7** A .tif file format is recommended for image file saving to retain the most digital information.
- 8.8** Power off the system components when the imaging session is complete.
- 8.9** Record usage in instrument log book.

9 Instrumental Conditions

The instrumental operating conditions are determined by the examiner in reference to the on-screen image. In general, increasing the current will increase the image brightness, and increasing the x-ray source voltage will increase the radiation penetration through the object. The voltage should be increased if the object appears opaque. Filtering the primary radiation source decreases the average x-ray wavelength reaching the sample and increases beam penetration through the sample. Filtering can be used in conjunction with voltage increases to improve object penetration. In general, any combination of operating parameters which produces a useful image is acceptable.

10 Decision Criteria

An x-ray radiograph is considered acceptable when the details of interest are visible in the image. This result is typically self-evident and is at the discretion of the operator. Interpretation of x-ray images is largely a matter of experience and training. X-ray scattering effects must be considered when interpreting the shapes of edges and corners and as a source of artifacts within the detected image. Atomic number and consequent x-ray absorption must also be considered when comparing relative thickness of the separate parts of multi-component and/or multi-material assemblies. Appropriate interpretation may require additional background investigation, including radiography and/or disassembly of exemplar components for comparison.

11 Calculations

Not applicable.

12 Measurement Uncertainty

Not applicable.

13 Limitations

An x-ray source is limited to a maximum operating voltage depending on the instrument in use. Thick metal sections may not permit sufficient flux of x-rays for useful imaging. The exact thickness limit will be a function of the object's geometry and its materials of construction. For example, according to published references, steel up to 40 mm thick can be satisfactorily examined with 300 kV x-rays.

Light items such as plastic and paper may not be visible due to poor x-ray absorption if they are inside a high atomic number material such as steel. However, it is usually possible to image plastics, tape, and other low atomic number materials if they are not otherwise shielded by higher atomic number materials.

14 Safety

- a. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.
- b. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review instrument SOPs and pertinent material Safety Data Sheets (SDS) prior to conducting examinations. If additional guidance is required, contact the Laboratory Health and Safety Group.
- c. For the NSI X5000, never close a person inside the chamber. The door is mechanically driven and may not open in case of power loss.

15 References

Brogdon, B. G., *Forensic Radiography*, CRC Press LLC 1998

Cartz, L., *Nondestructive Testing, Radiography, Ultrasonics, Liquid Penetrant, Magnetic Particle, Eddy Current*, ASM International 1995

Halmshaw, R., *Industrial Radiology, Theory and Practice*, 2nd ed., Chapman & Hall 1995

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
0	06/21/2006	New document that replaces a previous document also entitled <i>Digital Radiography</i>
1	03/02/2018	Renumbered Metallurgy Standard Operating Procedure (SOP) Manual documents. This document was formerly Metal 9 and is now designated Metal 303. Updated sections 1, 2 and 3 for clarity. Added personnel to section 2. Made minor editorial corrections throughout document. Updated section 4 to include maximum specimen sizes. Removed obsolete equipment from section 5. Incorporated section 7 into section 6 and renumbered subsequent sections. Added sampling statement in section 7. Redacted parts of section 8 specific to a particular instrument and revised. Updated safety requirements in section 14. Deleted obsolete reference in section 15 and added additional references.

Approval

Redacted - Signatures on File

Metallurgy
Technical Leader

Date: 02/28/2018

Firearms/Toolmarks
Technical Leader

Date: 02/28/2018

Anthropology
Technical Leader

Date: 02/28/2018

Chemistry
Unit Chief

Date: 02/28/2018

Firearms/Toolmarks
Unit Chief

Date: 02/28/2018

Scientific Analysis
Unit Chief

Date: 02/28/2018

Trace Evidence
Unit Chief

Date: 02/28/2018

QA Approval

Quality Manager

Date: 02/28/2018

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)

1 Introduction

The concentrations of selected elements in metallic objects can serve to chemically characterize their source. The concentrations of the elements intentionally added to an alloy are controlled by manufacturers to impart specific metallurgical properties required for a given product. Intentionally added elements are commonly specified to fall within a particular concentration range for a given alloy. Other elements are specified at maximum levels which the manufacturer may not exceed but which may otherwise vary considerably. Such variations in elemental concentrations provide a means for differentiating among the same alloy produced by different manufacturers and for distinguishing among specific production runs made by a single manufacturer.

Spark Discharge in Argon-Optical Emission Spectroscopy (SDAR-OES) is a destructive technique that can be used for quantitative compositional analysis of alloys. The SDAR-OES is used to produce validation data for alloy systems, establishing a limit of quantitation (LOQ) for various components within those systems. In an alloy system for which validation data has been generated, SDAR-OES can measure elements above the established LOQ, typically in the parts per million (ppm) range.

2 Scope

This document applies to case working personnel who perform metallurgical analyses. This procedure serves as a general guideline for all FBI Laboratory procedures that involve the use of SDAR-OES.

General procedures for quantitative compositional analysis by SDAR-OES are described below. Operational procedures for specific alloy classes or sample sizes are detailed in supplemental standard operating procedures (SOPs).

3 Principle

Application of this SOP to forensic materials requires knowledge of general concepts of optical emission spectroscopy (OES) analysis and specific instrument operation instructions. This knowledge can be obtained from the references provided, training from manufacturers or principal operators within the Lab, or schools covering theory and/or instrument operation.

SDAR-OES has its basis in quantum mechanics. Electricity is used to heat a small surface region of the sample to a plasma state where elemental photons of characteristic wavelengths (energies) are ejected as visible or ultraviolet (UV) light. Photons are filtered to form a parallel beam which impacts a grating and is reflected at unique angles depending on the wavelengths present. Each photon strikes one of many detectors depending on its angle of reflection. The electronic reaction is converted to a digital signal which is processed to determine the energy of the striking photon. Many such emission and collection events are measured during a single experiment. Quantitative determination of individual elemental concentrations is achieved by comparing measured emission intensities to the manufacturer-determined calibration curves resident on the instrument's computer system.

The analysis is performed by machining a flat surface on the specimen, placing it on the plasma spark stand and completing an electrical circuit arc-spark protocol predetermined by the instrument software for the alloy class being analyzed.

4 Specimens

This procedure can be used for the analysis of metal alloy classes for which the instrument has been validated. Samples must be large enough to completely cover the analysis area to permit their examination by the method outlined here. The surface to be analyzed must also be flat with any nonmetallic debris removed.

5 Equipment/Materials/Reagents

- a. SpectroLab LAV M10 spectrometer, or equivalent
- b. Optic re-profiling standard
- c. Standardization samples provided by the instrument manufacturer
- d. Sample preparation equipment specific to the alloy class being analyzed (see 8.1 Prepare Samples)
- e. High purity argon
- f. One or more certified reference materials (CRMs) matrix-matched to the alloy class being tested

- g. Spark stand insert (waveguide), tungsten carbide electrode and wire brush dedicated to the alloy class being tested
- h. Small sample spark stand plate with insert, 6 mm diameter opening (optional)
- i. Vacuum cleaner with precision nozzle attachment
- j. Lint-free wipes

6 Standards and Controls

Manufacturer-provided standards are used for re-profiling and standardization. The standardization samples to be employed in this procedure are specific to the alloy class being analyzed. Proper standardization is verified using one or more CRMs in the same alloy class. These CRMs are chosen by the operator to demonstrate adequate performance of the instrument on the elements of interest in a particular analysis.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Prepare Samples

Specimens measured by SDAR-OES must be flat and debris-free over the entire region of analysis. The method of specimen preparation for any specific alloy class is included in the method validation. Typically, steels are ground on zirconium oxide abrasive paper, cast irons are abraded using a swing grinder with Al_2O_3 abrasive, and softer, non-ferrous materials are turned on a lathe. Other methods of preparation may be suitable, but quantitative analysis must be validated for each preparation method. Optic re-profiling standards follow the steel preparation technique. Standardization samples follow the preparation method for the appropriate alloy class.

8.2 Clean Instrument

- a. Turn off power (“Source”) to the spark stand. (Turn back on when cleaning/instrument check is complete.)
- b. Assure that there is sufficient water in the system and that the air filter is not saturated with soot. Fill or exchange as necessary.
- c. Inspect the spark stand components for soot accumulation. Vacuum if necessary.
- d. If the spark stand components are changed in order to run a different alloy class, the spark stand must be thoroughly cleaned.
- e. Prior to running the re-profiling program, run a ‘dummy’ sample of the same alloy class (steel) to flush out any residual oxygen and nitrogen in the system. This is especially important if the instrument has just been cleaned. More than one dummy analysis may be run to ensure that the elemental readings are stable.

8.3 Re-profile Optic

- a. Verify that the proper spark stand components for the re-profiling standard (electrode and spark stand insert) are installed in the spark source stand.
- b. Follow the manufacturer’s instructions to run the re-profile procedure to optimize alignment of the optics. A minimum of five measurements is recommended.
- c. Print the re-profiling results and retain them in the log accompanying the instrument.

8.4 Standardize

- a. Run the standardization procedure within the appropriate method for the alloy class chosen based on the evidence to be analyzed. A minimum of five readings per standard is recommended.
- b. Print the standardization results and retain them in the associated case file. Record the results of the standardization in the instrument log.

8.5 Analyze

- a. Verify standardization with CRM(s) using the appropriate, standardized method

for the selected alloy class. Follow surface preparation in 8.1 Prepare Specimens, if needed. A minimum of five readings per standard is recommended.

- b. If the CRM results do not agree with the certification, the instrument may be re-standardized, or re-profiled then re-standardized, to optimize performance.
- c. Print the CRM results and retain them in the associated case file.
- d. Prepare an appropriately sized area on the evidentiary item(s) (see 8.1 Prepare Specimens).
- e. Follow the instrument's instructions for sample analysis. Take at least five measurements on each sample. Save and print the concentration data from individual measurements as well as the average and standard deviation of the collected measurements.
- f. Rerun one or more of the CRMs, taking a minimum of five measurements per CRM, to create pre- and post-test comparison(s) to determine if any instrument drift has occurred.
- g. Print the post-test CRM results and retain them in the associated case file.

9 Instrumental Conditions

The instrumental conditions are set by the instrument manufacturer and are not normally changed or modified by FBI Laboratory personnel.

10 Decision Criteria

In general, it is expected that the mean concentration values determined by the instrument on a given CRM will be within the range of values indicated on the certificate of analysis. If a measured element fails to give adequate agreement with the CRM, the instrument can either be re-standardized to improve the agreement or, if the value is not critical, it may be regarded as a semi-quantitative value. The following process is used for the comparison of data from samples thought to share a common origin. *If only the alloy composition is required, this section can be omitted.*

The average weight concentrations (wgt %) of a given element in two samples are inter-compared using a pooled t-test statistic of the type detailed in 11 Calculations. The

concentrations are deemed to be "analytically indistinguishable" if the concentrations in the two samples differ by less than the preselected critical t value. This procedure is repeated for each of the elements present in the sample. Critical t values are chosen so that an $\alpha = 0.05$ can be achieved for the overall analysis. Only elements above the LOQ are considered in calculating " α ". $\alpha = 1 - [P(|t| \leq t_{\text{critical}})]^r$ where "r" is the number of elements present above the quantitation limit, P is the probability of observing a t-value of less than or equal to t_{critical} and α represents the approximate cumulative probability of calling two samples analytically distinguishable when they are not.

For example, suppose 15 elements are above the LOQ, then choosing ($t_{0.9966} = t_{\text{critical}}$) yields an $\alpha = 0.05$. This means there is a 5.0% chance of incorrectly rejecting a match between any two samples when one actually exists. Note that repetitive means testing requires a relatively high critical value of t. In contrast, for a $t_{0.99}$, $\alpha = 0.14$ leading to a relatively high rate of false rejection (14%).

Segregation, surface depletion and microstructural banding may also need to be considered in evaluating alloy sources. Constituents that do not completely solutionize are especially prone to inhomogeneous distribution. Consequently, these may potentially exhibit unusually high t-test values. Because these effects depend on the prior processing history, generalizations often cannot be applied. The examiner may make a determination that two samples are associated to a given degree based on inspection of the analytic data and considerations of the possible sources of compositional variation in the material.

11 Calculations

11.1 Quantitative Analysis

The measured emission intensity of each element of interest is compared to the emission intensity of a selected matrix line and the system calibration curves. This data is used to determine the weight percentage of each of the elements present. Quantitation of the data is performed automatically by the system program and is not directly controlled by the user.

11.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student's t-test statistic of the sample means is typically used for the comparison. Two samples are deemed to be "indistinguishable" in the property under consideration if the two samples differ by less than the preselected critical t value (t_{critical}). The critical t values are typically chosen so that an overall, (Bonferroni-corrected) value 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 rejecting a match between two samples when one actually exists.

To perform this test, the means and variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where \bar{x}_a is the average value of the measurements on sample "a",

$\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample. The variance of the individual measurement values from sample "a" is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

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

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 \left(\frac{1}{n_a} + \frac{1}{n_b} \right)} \right)} \right| > t_{critical}$ for any point of comparison, the samples are concluded to have a

statistically significant difference. If not, the samples are concluded to be indistinguishable.

Typically five or more measurements per sample are used for performing comparisons.

12 Measurement Uncertainty

Quantitative data from this procedure are generally used for comparative purposes. Expanded 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).

When quantitative data are compared to a particular specification or when quantitative elemental compositions are reported and it is necessary to determine the expanded uncertainty of a measurement, it will be calculated in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*. Measurement uncertainty associated with a particular element can vary depending on alloy class, concentration of the element of interest, and the concentrations of other elements present in the alloy, so uncertainties must be calculated on a case-by-case basis.

13 Limitations

The estimated limits of quantitation (LOQ) have been determined for each alloy class validated and are listed in the specific alloy class SOPs. In general, these values have been found to correlate well with those observed in practice using CRMs (where these are available). However, some day-to-day variation is expected and values approaching the LOQ are best regarded as semi-quantitative unless standards are available to confirm the LOQs in a particular instrumental run. Data below the LOQ are to be used only in a qualitative fashion due to the inherent uncertainty associated with them. For example, the absence of manganese from a steel sample would be an important qualitative result due to the ubiquitous presence of this element in steels.

For elements in the range 0.1-100 wgt %, the relative standard deviation (rsd) will typically be less than 5%. For elements in the range of 0.01-0.1 wgt %, the rsd is typically less than 15%. Below 0.01 wgt % but above the LOQ, rsd's of up to 30% are to be expected though less than 10% is typical. Insoluble elements will frequently give higher than expected standard deviations due to their inherently inhomogeneous distribution in a given sample.

If higher relative standard deviations than those cited above are experienced, attempts can be made to correct the problem by re-machining the surface of the sample and retesting.

14 Safety

- a. Review pertinent material Safety Data Sheets (SDS) prior to machining and testing samples. Analyze the composition of an unknown metal sample by non-destructive means (e.g., x-ray fluorescence analysis) prior to SDAR-OES analysis. If additional guidance is required, contact the Laboratory Health and Safety Group.
- b. Wear safety glasses when machining samples and cleaning and operating the SDAR-OES instrument to prevent eye injury. Additionally, wear lab coat and gloves when cleaning the instrument and changing the air filter.

15 References

Thomsen, V. B. E, *Modern Spectrochemical Analysis of Metals - An Introduction for Users of Arc/Spark Instrumentation*, ASM International 1996

Slickers, K., *Automatic Atomic-Emission-Spectroscopy, 2nd Ed.*, Bruhlsche Universitätsdruckerei, Germany 1993

Milton, J. S. and Arnold, J. C., *Introduction to Probability and Statistics - Principles and Applications for Engineering and Computer Sciences, Fourth Edition*, McGraw-Hill Higher Education 2003

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
0	03/02/2018	Original issue. Relevant general information was taken from 401-7 <i>Analysis of Carbon and Low Alloy Steel Samples by Spark Discharge in Argon Optical Emission Spectroscopy</i> , 402-4 <i>Analysis of Small Carbon and Low Alloy Steel Samples by Spark Discharge in Argon Optical Emission Spectroscopy</i> , and 403-4 <i>Analysis of Copper by Spark Discharge in Argon Optical Emission Spectrometry</i> .

Approval

Redacted - Signatures on File

Metallurgy Technical Leader .

Date: 02/28/2018

Chemistry Unit Chief .

Date: 02/28/2018

QA Approval

Quality Manager .

Date: 02/28/2018

Analysis of Carbon and Low Alloy Steel Samples by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)

1 Introduction

The concentrations of selected elements in steel objects can serve to chemically characterize the source of the steel. The concentrations of the elements intentionally added to steel are controlled by manufacturers to impart the specific metallurgical properties required for a given product. Intentionally added elements are commonly specified to fall within a specific concentration range for a given alloy. Other elements are specified at maximum levels which the manufacturer may not exceed but which otherwise may vary considerably. Such variations in the elemental concentrations provide a means of differentiating among steels made by different manufacturers and for distinguishing among specific production runs made by a single manufacturer.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy examinations. This procedure determines the concentrations of several elements, if present above the limits of quantitation (LOQ), in carbon and low alloy steels. These elements include C, Si, Mn, S, P, Cr, Mo, Al, Ni, Co, Cu, Nb, Ti, V, Sn, As, Zr, Sb, Ta, B, and N. Extension of the procedure to other elements is possible if additional validation is completed. Operation of the SDAR-OES instrument will follow procedures defined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)* and the specific parameters described below.

3 Principle

Compositional analysis of steel alloys by SDAR-OES requires using the pre-determined spark sequence to generate characteristic light emissions from the elements present in the sample. Quantitative determination of individual elemental concentrations is achieved by comparison of measured emission intensities to the manufacturer-determined calibration curves resident on the instrument's computer system. The analysis is verified by demonstrating adequate performance on certified reference materials (CRMs) with similar composition to the evidentiary items.

4 Specimens

This procedure can be used for the analysis of plain carbon and low alloy steel sheets, plates, bars, pipes and any other object having sufficiently large physical dimensions. Generally, samples must be large enough to cover the 13mm diameter analysis area to permit their examination by the method outlined here. Any sample which completely covers the opening in the plasma source stand plate is of adequate size.

5 Equipment/Materials/Reagents

- a. SpectroLab LAV M10 spectrometer
- b. Spectro RH 18/30 optic re-profiling standard
- c. Spectro standardization samples RE 12/80, RN 19/24, and BS XCCS
- d. 60 - 120 grit zirconia grinding paper dedicated for steel OES preparation
- e. Bench grinder or other metallurgical grinder
- f. High purity argon
- g. One or more steel CRMs
- h. Spark stand insert (waveguide), tungsten carbide electrode and wire brush dedicated to steel alloys
- i. Vacuum cleaner with precision nozzle attachment
- j. Lint-free wipes

6 Standards and Controls

A re-profiling standard is provided by the instrument manufacturer. The standardization samples used in this procedure are specific to the steel alloy class and are specified in 5 Equipment/Materials/Reagents. Appropriate CRMs are selected by the operator to demonstrate adequate performance of the SDAR-OES instrument.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Prepare Samples

Specimens measured by SDAR-OES must be flat and debris-free over the entire analysis region. Re-profiling, standardizing and CRM materials may require surface preparation before testing. This is accomplished by dry grinding on 60 – 120 grit zirconium oxide abrasive paper dedicated for steel use.

8.2 Perform Analysis

Steel analysis uses the “Fe-10 method” that resides on the SpectroLab LAV M10 spectrometer. Follow the procedures detailed in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)* to clean, re-profile, and standardize the instrument using the appropriate stage and standardization samples. Select CRMs that contain concentrations of elements of interest that appropriately bound the concentrations present in evidentiary samples.

9 Instrumental Conditions

The instrumental conditions are set by the instrument manufacturer and are not normally changed or modified by FBI Laboratory personnel.

10 Decision Criteria

In general, it is expected that the mean concentration values determined by the instrument on a given CRM will be within the range of values indicated on the certificate of analysis. If a measured element fails to give adequate agreement with the CRM, the instrument can either be re-standardized to improve the agreement or, if the value is not critical, it may be regarded as semi-quantitative.

The process used to compare data from samples thought to share a common origin is detailed in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*.

11 Calculations

11.1 Quantitative Analysis

The measured emission intensity of each element of interest is compared to the emission intensity of a selected matrix line and the system calibration curves. This data is used to determine the weight percentage of each of the elements present. Quantitation of the data is performed automatically by the system program and is not directly controlled by the user.

11.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student's t-test statistic of the sample means is typically used for the comparison, as described in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*.

12 Measurement Uncertainty

Quantitative data from this procedure are generally used for comparative purposes. Expanded 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). In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*.

13 Limitations

Redacted

Redacted

Wear safety glasses when machining samples and cleaning and operating the SDAR-OES instrument to prevent eye injury. Additionally, wear lab coat and gloves when cleaning the instrument and changing the air filter.

15 References

Thomsen, V. B. E, *Modern Spectrochemical Analysis of Metals - An Introduction for Users of*

Arc/Spark Instrumentation, ASM International 1996

Slickers, K., *Automatic Atomic-Emission-Spectroscopy, 2nd Ed.*, Bruhlsche
Universitätsdruckerei, Germany 1993

Milton, J. S. and Arnold, J. C., *Introduction to Probability and Statistics - Principles and
Applications for Engineering and Computer Sciences, Fourth Edition*, McGraw-Hill Higher
Education 2003

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation,
Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest
revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory
Division, latest revision

*Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-
OES)*, Metallurgy Manual 400, Chemistry Unit, most recent revision

Rev. #	Issue Date	History
6	04/24/14	Minor grammatical changes in sections 12 and 15. Formula used for calculating sample variance in section 13.2 has been simplified. Section 14 has been rewritten to reflect updated measurement uncertainty requirements. References updated.
7	03/02/18	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 8 and is now designated Metal 401. Removed information that has been relocated to Metal 400: <i>Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)</i> throughout, renumbered sections and designated dependence on this document. Added personnel to section 2. Made minor editorial corrections throughout document. Added requirement for sampling plan retention in section 7. Added CRM verification in section 3.. Changed units in LOQ table in section 13. Augmented section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader

Date: 02/28/2018

Chemistry Unit Chief

Date: 02/28/2018

QA Approval

Quality Manager

Date: 02/28/2018

Analysis of Small Carbon and Low Alloy Steel Samples by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)

1 Introduction

The concentrations of selected elements in steel objects can serve to chemically characterize the source of the steel. The concentrations of the elements intentionally added to steel are controlled by manufacturers to impart the specific metallurgical properties required for a given product. Intentionally added elements are commonly specified to fall within a specific concentration range for a given alloy. Other elements are specified at maximum levels which the manufacturer may not exceed but which otherwise may vary considerably. Such variations in the elemental concentrations provide a means of differentiating among steels made by different manufacturers and for distinguishing among specific production runs made by a single manufacturer.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy examinations. This procedure determines the concentrations of several elements, if present above the limits of quantitation (LOQ), in carbon and low alloy steels. These elements include C, Si, Mn, S, P, Cr, Mo, Al, Ni, Co, Cu, Nb, Ti, V, Sn, As, Zr, Ta, and B. Extension of the procedure to other elements is possible if additional validation is completed. Operation of the SDAR-OES instrument will follow procedures defined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)* and the specific parameters described below.

3 Principle

Compositional analysis of steel alloys by SDAR-OES requires using the pre-determined spark sequence to generate characteristic light emissions from the elements present in the sample. Quantitative determination of individual elemental concentrations is achieved by comparison of measured emission intensities to the manufacturer-determined calibration curves resident on the instrument's computer system. The analysis is verified by demonstrating adequate performance on certified reference materials (CRMs) with similar composition to the evidentiary items.

4 Specimens

This procedure can be used for the analysis of plain carbon and low alloy steel sheets, plates, bars, pipes and any other object having sufficiently large physical dimensions. Generally, samples must be large enough to cover the 6mm diameter analysis area to permit their examination by the method outlined here. Any sample which completely covers the opening in the insert of the small sample spark stand plate is of adequate size.

5 Equipment/Materials/Reagents

- a. SpectroLab LAV M10 spectrometer
- b. Small sample spark stand plate with insert, 6 mm diameter opening, dedicated for steel use
- c. SpectroLab LAV M10 standardization samples RE 12/80, RN 19/24, and BS XCCS
- d. 60 - 120 grit zirconia grinding paper dedicated for steel OES preparation
- e. Bench grinder or other metallurgical grinder
- f. High purity argon
- g. One or more steel CRMs
- h. Spectro RH 18/30 optic re-profiling standard
- i. Spark stand insert (waveguide), tungsten carbide electrode and wire brush dedicated to steel alloys
- j. Vacuum cleaner with precision nozzle attachment
- k. Lint-free wipes

6 Standards and Controls

A re-profiling standard is provided by the instrument manufacturer. The standardization samples used in this procedure are specific to the steel alloy class and are specified in 5 Equipment/Materials/Reagents. Appropriate CRMs are selected by the operator to demonstrate

adequate performance of the SDAR-OES instrument.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Prepare Samples

Specimens measured by SDAR-OES must be flat and debris-free over the entire analysis region. Re-profiling, standardizing and CRM materials may require surface preparation before testing. This is accomplished by dry grinding on 60 – 120 grit zirconium oxide abrasive paper dedicated for steel use.

8.2 Perform Analysis

Small sample steel analysis uses the “Fe-10 method” that resides on the SpectroLab LAV M10 spectrometer. Follow the procedures detailed in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)* to clean, re-profile, and standardize the instrument using the appropriate stage, spark source components and standardization samples. Select CRMs that contain concentrations of elements of interest that appropriately bound the concentrations present in evidentiary samples.

9 Instrumental Conditions

The instrumental conditions are set by the instrument manufacturer and are not normally changed or modified by FBI Laboratory personnel.

10 Decision Criteria

In general, it is expected that the mean concentration values determined by the instrument on a given CRM will be within the range of values indicated on the certificate of analysis. If a

measured element fails to give adequate agreement with the CRM, the instrument can either be re-standardized to improve the agreement or, if the value is not critical, it may be regarded as semi-quantitative. The process used to compare data from samples thought to share a common origin is detailed in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*.

11 Calculations

11.1 Quantitative Analysis

The measured emission intensity of each element of interest is compared to the emission intensity of a selected matrix line and the system calibration curves. This data is used to determine the weight percentage of each of the elements present. Quantitation of the data is performed automatically by the system program and is not directly controlled by the user.

11.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student's t-test statistic of the sample means is typically used for the comparison as described in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*.

12 Measurement Uncertainty

Quantitative data from this procedure are generally used for comparative purposes. Expanded uncertainty should not be used for these inter-comparisons because it increases the probability that two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion). In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*.

13 Limitations

Redacted

Redacted

14 Safety

Wear safety glasses when machining samples and cleaning and operating the SDAR-OES instrument to prevent eye injury. Additionally, wear lab coat and gloves when cleaning the instrument and changing the air filter.

15 References

Thomsen, V. B. E., *Modern Spectrochemical Analysis of Metals-An Introduction for Users of Arc/Spark Instrumentation*, ASM International 1996

Slickers, K., *Automatic Atomic-Emission-Spectroscopy, 2nd Ed.*, Bruhlsche Universitätsdruckerei, Germany 1993

Milton, J. S. and Arnold, J. C., *Introduction to Probability and Statistics-Principles and Applications for Engineering and Computer Sciences, Fourth Edition*, McGraw-Hill Higher Education 2003

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Metallurgy Manual 400, Chemistry Unit, most recent revision

Rev. #	Issue Date	History
3	04/24/14	Minor grammatical changes made in sections 5, 10.3 and 12. Formula used for calculating sample variance in section 13.2 has been simplified. Section 14 has been rewritten to reflect updated measurement uncertainty requirements. References in section 16 have been updated.
4	03/02/18	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 19 and is now designated Metal 402. Removed information that has been relocated to Metal 400: <i>Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)</i> throughout, renumbered sections and designated dependence on this document. Added personnel to section 2. Made minor editorial corrections throughout document. Added requirement for sampling plan retention in section 7. Added CRM verification in section 3. Changed units in LOQ table in section 13. Augmented section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader

Date: 02/28/2018

Chemistry Unit Chief

Date: 02/28/2018

QA Approval

Quality Manager

Date: 02/28/2018

Analysis of Copper by Spark Discharge in Argon Optical Emission Spectrometry (SDAR-OES)

1 Introduction

The concentrations of selected elements in copper serve to chemically characterize it. The elements analyzed in copper can occur as impurities though intentional additions are also made to create alloys. Usually, the impurities are specified not to exceed some maximum level which depends on the copper grade. Below this threshold, significant variation is permissible. Such variation provides a potential means of differentiating among different sources and/or manufacturing processes of copper.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy examinations. This procedure provides the examiner with a method for determining the concentrations of several elements, if present above the limits of quantitation (LOQ), in relatively pure copper. These elements include Zn, Sn, Mn, Pb, P, Ni, Mg, Cr, Co, Fe, Ag, Te, As, Sb, Cd, Bi, Al, S, Ti, Se, Si, and O. Extension of the procedure to other elements is possible if additional validation is completed. Operation of the SDAR-OES instrument will follow procedures defined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)* and the specific parameters described below.

3 Principle

Compositional analysis of copper by SDAR-OES requires using the predetermined spark sequence to generate characteristic light emissions from the elements present in the sample. Quantitative determination of individual elemental concentrations is achieved by comparison of measured emission intensities to the manufacturer determined calibration curves resident on the instrument's computer system. The analysis is verified by demonstrating adequate performance on certified reference materials (CRMs) with similar composition to the evidentiary items.

4 Specimens

This procedure can be used for the analysis of copper sheets, plates, bars, pipes and any other object having sufficiently large physical dimensions. Generally, samples must be large enough to cover the 13mm diameter analysis area to permit their examination by the method outlined here. Any sample which completely covers the opening in the spark source stand is of adequate size.

5 Equipment/Materials/Reagents

- a. SpectroLab LAV M10 spectrometer
- b. Spectro RH 18/30 optic re-profiling standard
- c. Spectro standardization samples: RC 11/4, RC 12/12, and RC 14/20
- d. Lathe, bench-top or other
- e. Carbide or diamond-tipped cutting tool for lathe dedicated for use on copper
- f. High purity argon
- g. One or more copper-based CRMs
- h. Spark stand insert (waveguide), tungsten carbide electrode and wire brush dedicated to copper alloys
- i. Vacuum cleaner with precision nozzle attachment
- j. Lint-free wipes

6 Standards and Controls

A re-profiling standard is provided by the instrument manufacturer. The standardization samples used in this procedure are specific to the copper alloy class and are specified in 5 Equipment/Materials/Reagents. Appropriate CRMs are selected by the operator to demonstrate adequate performance of the SDAR-OES instrument.

7 Sampling

If large numbers of samples are received solely for quantitative compositional analysis, a hypergeometric random sampling scheme can be employed for their testing. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Prepare Samples

Specimens measured by SDAR-OES must be flat and debris-free over the entire analysis region. Re-profiling, standardizing and CRM materials may require surface preparation before testing. This is accomplished by mounting the sample in a lathe and machining the surface to be analyzed to produce a bright finish. To prevent re-oxidation of the surface prior to analysis, samples should be tested within a short time (several hours) after machining. If significant time elapses, the sample should be re-machined. This is especially important for oxygen measurement, in which case the sample should be tested immediately (within one hour) after machining.

8.2 Perform Analysis

Copper analysis uses the "Cu-10 method" that resides on the SpectroLab LAV M10 spectrometer. Follow the procedures detailed in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)* to clean, re-profile, and standardize the instrument using the appropriate stage, spark source components and standardization samples. Select CRMs that contain concentrations of elements of interest that appropriately bound the concentrations present in evidentiary samples.

9 Instrumental Conditions

The instrumental conditions are set by the instrument manufacturer and are not normally changed or modified by FBI Laboratory personnel.

10 Decision Criteria

In general, it is expected that the mean concentration values determined by the instrument on a given CRM will be within the range of values indicated on the certificate of analysis. If a

measured element fails to give adequate agreement with the CRM, the instrument can either be re-standardized to improve the agreement or, if the value is not critical, it may be regarded as semi-quantitative. The process used to compare data from samples thought to share a common origin is detailed in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*.

11 Calculations

11.1 Quantitative Analysis

The measured emission intensity of each element of interest is compared to the emission intensity of a selected matrix line and the system calibration curves. This data is used to determine the weight percentage of each of the elements present. Quantitation of the data is performed automatically by the system program and is not directly controlled by the user.

11.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student's t-test statistic of the sample means is typically used for the comparison as described in *Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)*.

12 Measurement Uncertainty

Quantitative data from this procedure are generally used for comparative purposes. Expanded 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). In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*.

13 Limitations

Redacted

Redacted

14 Safety

Wear safety glasses when machining samples and cleaning and operating the SDAR-OES instrument to prevent eye injury. Additionally, wear lab coat and gloves when cleaning the instrument and changing the air filter.

15 References

Thomsen, V. B. E., *Modern Spectrochemical Analysis of Metals - An Introduction for Users of Arc/Spark Instrumentation*, ASM International 1996

Slickers, K., *Automatic Atomic-Emission-Spectroscopy, 2nd Ed.*, Bruhlsche

Universitätsdruckerei, Germany 1993

Milton, J. S. and Arnold, J. C., *Introduction to Probability and Statistics - Principles and Applications for Engineering and Computer Sciences, Fourth Edition*, McGraw-Hill Higher Education 2003

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FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Metallurgy Manual 400, Chemistry Unit, most recent revision

Rev. #	Issue Date	History
3	04/24/14	Minor grammatical changes in sections 10.1 and 12. Formula used for calculating sample variance in section 13.2 has been simplified. Section 14 has been rewritten to reflect updated measurement uncertainty requirements. References in section 17 updated.
4	03/02/18	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 21 and is now designated Metal 403. Added personnel to section 2. Removed information that has been relocated to Metal 400: <i>Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES)</i> throughout, renumbered sections and designated dependence on this document. Made minor editorial corrections throughout document. Added requirement for sampling plan retention in section 7. Added CRM verification in section 3. Changed units in LOQ table in section 13. Augmented section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader

Date: 02/28/2018

Chemistry Unit Chief

Date: 02/28/2018

QA Approval

Quality Manager

Date: 02/28/2018

Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF)

1 Introduction

Energy dispersive x-ray fluorescence spectrometry (EDXRF) is a technique that can be used for the non-destructive elemental analysis of a wide variety of materials for elements ranging in atomic number from fluorine to uranium. Microspot x-ray fluorescence spectrometry (micro-XRF) is used for the analysis of samples that are too small for standard x-ray fluorescence analysis methods. Both EDXRF and micro-XRF can be used to characterize the major, minor, and trace elemental constituents present in solid and liquid samples.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy, anthropology or general chemistry examinations. This procedure serves as a general guideline for all FBI Laboratory procedures that involve the use of x-ray fluorescence spectrometry (XRF) analyses using energy dispersive detectors. (Wavelength dispersive detectors are not included in this standard operating procedure, SOP.)

Application of this SOP to forensic materials requires knowledge of general concepts of XRF analysis. Methods requiring analysis by XRF require operational proficiency of each instrument employed. This knowledge can be obtained from the references provided, training from manufacturers or principle operators within the unit, or schools covering theory or instrument operation.

General procedures for compositional analysis by EDXRF and micro-XRF are described below. Operational procedures for specific instruments are detailed in supplemental SOPs.

3 Principle

XRF has its basis in quantum mechanics. An incident x-ray beam is used to eject core electrons from the atoms comprising the sample. Subsequent relaxation from the excited state results in emission of x-ray photons (x-rays), each with an energy that is characteristic of the element emitting it. Each photon striking the detector generates an electronic reaction that is converted to

a digital signal which is processed to determine the energy of the incident photon. Many such emission and collection events are measured during a single experiment. The spectral output is a histogram displaying the number of photons (or “intensity”) collected at each energy level over the time interval of the test. These relative intensities are the basis for determining the concentrations of each detected element in the sample.

Micro-XRF is a modification of standard EDXRF in which the x-ray beam is focused down to a small spot size. This can be accomplished using a beam collimator or a poly-capillary or mono-capillary x-ray lens. Such configurations permit elemental analysis of samples as small as the incident spot size.

4 Specimens

Virtually any liquid or solid sample can be analyzed by this method. Particles as small as 50 μm in diameter can be examined using micro-XRF. Since the incident x-rays will interact with the sample surface, any surface coating or contamination will be measured during the procedure. Specimens may need to be cleaned or ground to measure underlying layers or base material.

For quantitative analysis, specimens must be prepared so that they have a flat, smooth surface and be sufficiently thick to accommodate the entire x-ray interaction volume.

In the case of liquid samples, a plastic cup can be used to contain the sample. Depending on the orientation of the x-ray tube and detector, either the flat surface of the liquid will provide the required analysis surface or the sample will be measured through a layer of polymer film.

5 Equipment/Materials/Reagents

- a. Energy dispersive x-ray fluorescence spectrometer, such as:
 - Thermo QUANT’X X-ray Fluorescence Spectrometer
 - Bruker M4 Tornado Micro X-ray Fluorescence Spectrometer
 - Olympus Delta Premium Handheld X-ray Fluorescence Spectrometer

The choice of instrument to use will depend on the desired measurement environment, the available amount and particle size of the sample, and the elements to be measured. Each EDXRF instrument has different capabilities to measure major and minor alloying elements and trace elements in different matrices. Refer to the individual instrument SOP for known limitations. Additional capabilities can be demonstrated on a case-by-case basis by appropriate validation.

- b. Calibration standard(s) appropriate for the specific instrument used.
- c. Mounting materials (optional, depending on sample) such as:
 - Graphite stubs
 - Durotak sample mounting adhesive
 - Plastic sample cups (liquid samples)
 - Chemplex X-Ray Mylar (2.5µm thick) or polypropylene film (6.3 µm thick) or similar
- d. Certified reference materials (CRMs), if an alloy class comparison, elemental quantitation, or the absence of a specific element is to be reported.

6 Standards and Controls

Each EDXRF instrument requires energy adjustment (sometimes referenced as “calibration” or “validation” by the instrument manufacturer) to coordinate the electronic signal from the detector with known emission energies from standard reference materials and to verify the energy resolution. The required performance check standard is listed in each instrument SOP.

CRMs are often used to demonstrate the discernment of major, minor, and trace elements in alloys similar to the sample under the same measurement conditions. For quantitative analysis, validation will be performed according to the *FBI Laboratory Operations Manual (LOM)* and *Chemistry Unit Quality Assurance and Operations Manual (CU QAM)* using appropriate CRMs.

XRF instruments are not calibrated by Chemistry Unit personnel. Adequate instrument performance is demonstrated prior to case work by performing a verification or energy adjustment routine. At a minimum, such a performance check is completed each day prior to the instrument being used for case work. In addition, energy adjustment should be performed when the spectrum peaks become shifted from their theoretical energy positions by more than 0.05 keV. A record of the performance check is retained near the instrument, in either electronic or paper form, to track historical performance.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. For bulk samples, like soil or fines, the

material should be mixed until it appears visually homogeneous before performing sampling. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Basic Operation

The basic operation of each individual instrument is detailed in the instrument SOP. The general steps for performing XRF analysis follow:

- a. Run the instrument performance check routine. File one copy with the instrument performance records.
- b. Prepare and insert the specimen(s).
- c. Apply the desired atmospheric environment: air, helium, or vacuum. Do not perform evacuation on unconstrained fine powders or on liquid samples. Fine powder can be protected from disseminating under vacuum if contained in a sample cup with a perforated film cover.
 - i. Where low atomic number (low Z) elements such as sodium and silicon are present, attenuation of the fluorescent x-rays produced will occur in air and dramatically increase the minimum detectable concentration of these elements. Typically, samples are analyzed under a vacuum of 1 torr or less (standard atmospheric pressure is 760 torr) to optimize the detection limits of low Z elements and to eliminate argon x-ray fluorescence which occurs as the x-rays pass through air.
 - ii. Helium is useful for measuring low Z elements in liquids because it is a poor x-ray absorber and does not contribute interfering signal.
 - iii. In samples where evacuation is not possible (e.g., fine powder or liquid), analysis will be conducted in air or helium atmosphere.
 - iv. For samples where low Z elements (F through Ca) are not present or relevant, analysis in air is acceptable.
- d. Adjust instrument conditions to best reveal the analytes (elements) of interest. This may be an iterative process. Selection of x-ray tube voltage and filtering is dependent upon the sample composition and the objective of the analysis. Choice of the optimal conditions for a given analysis requires experience and a basic knowledge of x-ray physics. Suitable tube current is dictated primarily by the need to have a sufficient count rate to collect a spectrum.

- i. If the compositions of two specimens are to be compared, analyze them both under the same instrumental conditions.
 - ii. If a specimen is to be compared to an alloy class or classes, analyze appropriate CRMs under the same instrumental conditions as the unknown specimen.
 - iii. If the absence of an analyte is under consideration, establish a limit of detection (LOD) for that analyte in a similar matrix under the same instrumental conditions.
 - iv. If quantitative compositions are to be reported, validate the analytes of interest in a similar matrix using CRMs according to the LOM and CU QAM.
- e. Acquisition duration will depend on the conditions chosen and the sample area exposed to the incident beam, but must be adequate to acquire sufficient counts for analysis. The acquisition time can be extended to optimize spectrum clarity or shortened to enhance collection efficiency based on the requirements of the case.
- f. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.

8.2 Spectral Analysis

Determination of the elements detected in a spectrum requires analysis of the peak shapes and energy positions, the relative heights of adjacent peaks, consideration of the effects of secondary and tertiary fluorescence and other x-ray/specimen interactions. System peaks, sum and escape peaks, Rayleigh and Compton scattering and diffraction may also contribute peaks to the spectrum and should be considered when interpreting spectra. Some XRF 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.

8.3 Quantitation of Data

In general, x-ray spectra are not used to generate quantitative data. However, the spectra acquired from a sample can be processed to yield weight concentrations of the elements detected. Different XRF instruments employ different software routines to quantitatively process spectral data. If numerical compositional data is to be reported, validation must be performed on the same instrument under the same operating conditions using CRMs with a matrix similar to that of the specimen of interest.

9 Instrumental Conditions

Instrument conditions will vary depending on the instrument and application. See individual instrument SOPs and 8.1 Basic Operation.

10 Decision Criteria

10.1 Instrument Performance

Instrument performance is demonstrated during the instrument performance check. Energy adjustment of the instrument will be undertaken whenever the measured x-ray peak positions differ by more than 0.05 keV from their theoretical positions.

10.2 Qualitative Analysis

Peak identification on XRF spectra requires a thorough understanding of x-ray physics, instrument performance and potential artifacts. Although instruments provide peak identification software, the analyst is required to interpret the validity of the suggestions produced by programs using libraries of x-ray energy lines. In addition:

- a. Comparisons between specimens must consider possible surface or embedded contamination, variations in surface topography between specimens and any variation in the as-manufactured homogeneity of pristine specimens.
- b. Comparison to an alloy class or classes must consider the factors above and the possibility of overlapping compositions among alloy classes. Although it may be possible to demonstrate that a specimen is of similar composition to a particular alloy class, it may be impossible for XRF to reveal whether a material was produced to a particular specification.
- c. If the absence of an analyte is under consideration, the validated LOD for that analyte in a similar matrix under the same instrumental conditions will determine the decision criteria.

10.3 Quantitative Analysis

If quantitative compositions are to be reported, a CRM standard (selected by alloy) is used to check the accuracy of the quantitative analytical results (see 8.3 Quantitation of Data). If the measured concentrations of the major elements of interest deviate by more than 15% from the

certified values, the analysis should be rerun as systematic errors may be present. It is also noted that some element peaks may be overlapped by a major constituent rendering them indistinguishable. For example, the manganese peaks in stainless steel alloys are masked by those of chromium and iron. In such instances, any concentration results generated for the overlapped, minor constituent element should be regarded as no more than semi-quantitative in nature. If quantitative results are required, another method of analysis will be chosen.

11 Calculations

Calculations for establishing relative concentrations of elements in a measured sample are embedded in quantitation software routines of each XRF instrument. The internal routines are verified by CRMs as described above.

The expected position of escape and sum peaks on the energy axis can be calculated if they are not automatically generated by instrument software:

- a. For instruments using silicon-based detectors, an “escape peak” may appear at 1.740 keV less than the energy peaks of the major constituents.
- b. “Sum peaks” may appear at an energy equal to the sum of the peak energies of major constituents. For example, in a brass alloy with high zinc content, sum peaks can be seen at double the Cu K α energy, double the Zn K α energy and at the Cu K α + Zn K α energy.

12 Measurement Uncertainty

Typically, XRF is not used for quantitative analysis. Should quantitative reporting be required, the measurement uncertainty will be estimated in accordance with *Chemistry Unit Procedures for Estimating Measurement Uncertainty* in the CU QAM.

13 Limitations

The physical interactions of incident and emitted x-rays with the different components of an x-ray measurement system, including the measured sample, must be taken into account when analyzing XRF spectra:

- a. The detection limit of an element is dependent upon its atomic number, the matrix material, the analysis atmosphere, the presence of overlapping element energy peaks,

acquisition time, any filters used and other factors. Parts per million (ppm) level detection limits are readily obtained in some situations. More commonly, the practical detection limits in metal alloys range from ~0.01 - 0.1 weight percent depending on the matrix and the element being analyzed.

Where required, specific detection limits under a given set of analytical conditions can be estimated by analysis of compositionally similar CRMs containing known levels of the analyte of interest using identical instrumental conditions. The first eight elements of the periodic table (H, He, Li, Be, B, C, N, O) cannot be detected by XRF systems at any concentration.

- b. Unusually thin samples may result in skewing of the relative heights of the various energy peaks and thus cannot be reliably quantified. Similarly, a layered sample may produce a spectrum containing signal from the multiple layers.
- c. Peak identification in a spectrum can be complicated by the presence of diffraction peaks, scatter peaks associated with the x-ray tube target material, and other system peaks as discussed in 8.2 Spectral Analysis. For example, using Mylar film to support a specimen may contribute a Ca peak to the spectrum. Organic matrices also generate high background scatter. Analyzing a known material with a matrix similar to the specimen can often help identify these artifact features in a spectrum.
- d. On-board peak identification routines compare measured energy intensities with the known characteristic energies emitted by elements. The analyst must take into account the likelihood of the presence of any particular element. For example, in a manganese standard, an automated peak identification routine may indicate that an energy peak at 6.49keV could be a Dy $L\alpha$ line or a Mn $K\beta$ line. The analyst should recognize that dysprosium is not likely to be present based on its rarity in the earth and upon the absence of additional Dy spectral emissions.
- e. Different types of x-ray detectors have different limitations of performance. Solid state detectors using lithium-drifted silicon crystals (SiLi) require sufficient cooling to retain chip integrity and provide adequate energy resolution (e.g., Peltier cooling). Silicon drift detectors (SDD) do not need to be cooled as severely to achieve adequate resolution, but they provide relatively poor sensitivity for high energy x-rays. This effect can be mitigated somewhat by choosing optimum tube excitation and filtering conditions during measurement.
- f. X-ray fluorescence energy spectra are subject to numerous, but well documented, interferences from other element characteristic energies. The following common

interferences are known for spectra collected using SiLi or SDD energy dispersive detectors. The presence of these interfering species can usually be determined from examination of the affected peak series, the peak height ratios and the presence of higher energy peaks that will normally accompany the interfering species.

Element	Interferences
F (K lines)	Fe (L lines)
Na (K lines)	Cu (L lines)
Mg (K lines)	As (L lines); Tb (M lines)
Al (K lines)	Br (L lines); Tm, Yb (M lines)
Si (K lines)	Rb (L lines); Sr, W (M lines)
P (K lines)	Zr (L lines); Ir (M lines)
S (K lines)	Mo (L lines); Pb (M lines)
Cl (K lines)	Rh (L lines); Ru (L lines)
K (K lines)	In (L lines)
Ca (K lines)	Te (L lines); Sb (L lines)
Ti (K lines)	V-K _α ; Ba (L lines); La (L lines)
V-K _β line	Cr-K _α line
Cr-K _β line	Mn-K _α line
Mn-K _β line	Fe-K _α line
Fe-K _β line	Co-K _α line
Co-K _β line	Ni-K _α line
Ni-K _β line	Cu-K _α line
Cu-K _β line	Zn-K _α line
As (K lines)	Pb (L lines)

14 Safety

- Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.
- XRF detector windows are comprised of beryllium and are extremely delicate. In the event of damage to the window, the beryllium dust created could pose an acute health

hazard. If this occurs, seal the chamber and seek assistance from the Laboratory Health and Safety Group.

15 References

Jenkins, R., *X-ray Fluorescence Spectroscopy*, Wiley Interscience, New York 1988

Jenkins, R., Gould, R.W. and Gedcke, D., *Quantitative X-ray Spectrometry*, 2nd ed., Marcek Dekker, Inc., New York 1995

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FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
0	08/18/2014	Original issue. Relevant general information was taken from 18-1 Operation of the EDAX Eagle III XXL Micro X-Ray Fluorescence Spectrometer and 20-1 Operation of the Thermo QUANT'X X-Ray Fluorescence Spectrometer for specific EDXRF instruments.
1	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 25 and is now designated Metal 500. Included referenced acronym in title. Added personnel to section 2. Made minor editorial corrections and terminology changes throughout document. Deleted obsolete equipment in section 5. Incorporated section 7 into section 6 and renumbered subsequent sections. Added requirement for sampling plan retention in section 7. Added section 8.1.e to clarify operation. Revised description of instrument performance check in section 10.1. Updated section 13 to clarify instrument limitations. Deleted obsolete section 14.c. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy
Technical Leader

Date: 02/28/2018

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Operation of the Thermo QUANT'X X-Ray Fluorescence Spectrometer

1 Introduction

The Thermo QUANT'X is an energy dispersive x-ray fluorescence spectrometer (EDXRF) used for non-destructive compositional analysis of solids and liquids ranging in atomic number from sodium to uranium. The instrument can operate under an air, helium, or vacuum atmosphere.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy, anthropology or general chemistry examinations. The operation of the Thermo QUANT'X X-ray Fluorescence Spectrometer will follow procedures defined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by X-ray Fluorescence Spectrometry (EDXRF)* and the specific parameters described below.

3 Principle

The Thermo QUANT'X uses a Rh-target x-ray tube to generate the incident x-ray beam and a Peltier-cooled, lithium-drifted silicon detector (PCD) to collect and measure photons emitted by the sample. These components are arranged in a bottom-up configuration so that specimens are placed face down over the stage portal for measurement. Elements with atomic number equal to and greater than 11 (sodium) can be measured. Measurements can be made in an air, vacuum, or helium environment. Instrument control and data analysis are performed using WinTrace software running on a personal computer that is connected to the analyzer.

4 Specimens

Any liquid or solid sample can be analyzed on this instrument, provided the cover can be closed. Larger specimens may have to be sectioned to provide access to an area of interest to be analyzed. Although the stage portal is approximately 1 inch in diameter, the portal does not have to be completely covered for qualitative measurement. Typically, a few grams of liquid or solid will provide satisfactory results. If quantitative results are desired, it is necessary to prepare the samples so that they have a flat, smooth surface. In the case of fine powder or liquid samples, the thin support film (e.g., 2.5 μm Mylar) used to contain the sample provides the required surface.

5 Equipment/Materials/Reagents

- a. ThermoScientific ARL QUANT'X EDXRF Energy Dispersive X-Ray Fluorescence Spectrometer
- b. Calibration standard(s) such as "Thermo OFHC" or a copper Certified Reference Material (CRM), purity $\geq 99\%$ Cu
- c. CRMs of alloys similar to the type(s) under analysis, if elemental quantitation is required or limit of detection (LOD) is being established
- d. One inch diameter (or larger) plastic sample cups
- e. Chemplex X-Ray Mylar (2.5 μm thick) or polypropylene film (6.3 μm thick) or similar
- f. Helium gas, purity $\geq 99.95\%$ He, optional
- g. "Excitation Filter Guide" chart, provided by ThermoScientific

6 Standards and Controls

The instrument performance check (energy adjustment routine) requires a pure copper disc. Normally, the Thermo OFHC standard is used for this procedure, but any CRM pure copper disc is adequate for this purpose. For quantitation, alloy class comparison or to establish the absence of an element, CRMs as similar as possible to the material under analysis are used.

The energy adjustment routine is used to fine tune the signal processor. Initiation of the automated routine is described in 8.1 Basic Operation below. In the event the energy adjustment is unsuccessful, the routine should be rerun. If repeated failure of the energy adjustment routine occurs and the operator cannot correct the problem, then the instrument must be serviced. The instrument is fine-tuned each day prior to being used for casework. One copy of the Energy Adjustment Report is kept with the instrument and one copy with the applicable case notes.

For quantitative analysis, a calibration curve is generated for each analyte (element) to be quantified. The calibration curve(s) are matrix-dependent and so must be developed from materials as similar as possible to the matrix of the unknown sample.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding

procedure used will be recorded in case notes.

8 Procedure

8.1 Basic Operation

The basic operation from a powered-down condition consists of the following steps.

- a. Turn on computer and instrument. From a cold start, the red “Detector” light on the front of the instrument will blink until the detector is cooled sufficiently to operate.
- b. Position the Thermo OFHC or other pure copper disc for analysis.
- c. In the WinTrace software, select the “Energy Adjustment Shortcut” from the desktop or open Acquisitions Manager and select “Analyze” > “Energy Adjustment”.
- d. The energy adjustment routine normally uses the parameters listed in Table 1. Three acquisition rates (AR) are available. The energy adjustment must be run at the AR to be used for acquiring the spectrum from the unknown, typically Medium.

Table 1: Typical Operating Parameters for Energy Adjustment Routine

<u>Parameter</u>	<u>Value</u>
Line Energy	8.041 keV
Filter	Pd Medium
Voltage	20 kV
Current	Auto
Acquisition Range	40 keV
Acquisition Rate	Medium
Spinner	OFF

- e. Select “OK” to start the energy adjustment routine. The software self-adjusts the current to achieve an appropriate dead time of 30-59%.
- f. Upon successful completion, the Energy Adjustment Report appears with diagnostic information. The results of the energy adjustment (pass/fail) will be recorded in the instrument log accompanying the instrument. The report will be retained in the logbook for system performance diagnostics.
- g. The report will be compared to previous energy adjustment results:
New and old Gain DAC settings should change by no more than 100 on a weekly basis.
1) FWHM should not exceed 195 eV for the pure copper disc measured at the medium AR with the PCD detector.

- 2) Counts (cps/mA): This indicator of x-ray tube performance should be compared with previous values. Significant changes might be a sign of normal x-ray tube aging or of stability problems.

If any of the above conditions is unacceptable, consult a service representative.

- h. If the energy adjustment is unsuccessful, the routine will be performed again. If repeated failure of the energy adjustment occurs and the operator cannot correct the problem, the instrument must be serviced.

8.2 General Analysis

- a. Place the specimen on the stage face down over the stage portal. The surface to be analyzed should be flush with the stage surface, although the portal need not be completely covered for qualitative analysis. Center the area to be analyzed over the center of the hole. No part of the specimen should ever protrude down into the portal.
- b. Program the run by opening a qualitative or quantitative “tray list” (8.3 Qualitative Analysis or 8.4 Quantitative Analysis) and selecting the parameters for each sample and condition to be run.
 - i. To run one specimen at multiple analysis conditions, enter the identical specimen ID on additional lines, selecting the new desired condition for each.
 - ii. To measure multiple specimens under the same analysis condition, enter the different specimen IDs on each line, selecting the same condition. Altering a condition that appears more than once in a tray list will alter all instances of that condition. As the analysis proceeds, the instrument will prompt for the required sample change.
 - iii. Define each operating condition. Optimum x-ray tube voltage(s) and mode of irradiation (filter type or unfiltered) depend on the sample composition and the objective of the analysis. Combinations that typically generate good intensities for specific elements are suggested on the “Excitation Filter Guide” provided by the manufacturer. However, since matrix interactions can be severe, the choice of the optimal conditions for a given analysis may deviate from these recommendations. The tube current is dictated primarily by the need to have a sufficient count rate to collect a spectrum, corresponding to deadtime of ~50% (30-59%), and is automatically adjusted by the instrument control software.
 - iv. Select the appropriate measurement atmospheric condition: air, helium, or vacuum. (Air is the default setting for each standard condition.)
- c. Identify the peaks present in the spectrum using the WinTrace Spectrum Viewer program. The Auto Peak Identification routine is activated by the thumbprint icon.

Software-generated identifications must be interpreted by an experienced operator to prevent misidentification of peaks. The “MLK” function overlays sets of characteristic emission lines on the spectrum to aid in x-ray peak identification. The interactive periodic table (found under “Tools” > “Peak Identification” > “Setup”) can be used to adjust the sensitivity of the labeling routine and to force or remove element labels. Double-clicking on an element label on the spectrum display will remove an individual peak label without blocking the entire line series for that element. Refer to CU Metallurgy SOP Manual *Compositional Analysis by EDXRF* for more details regarding peak identification.

8.3 Qualitative Analysis

- a. Spectra for qualitative analysis are collected in WinTrace “Acquisitions Manager” using a Qualitative Tray List. Before acquisition, be sure to specify the location the spectra will be saved by selecting “Edit” > “Spectrum Location”.
- b. Open a Qualitative Tray List; enter the sample ID; select filter conditions then click off of the row for that sample. Click back onto the sample row and select the “Edit Conditions” icon to change the acquisition settings and environment if desired.
- c. To begin acquisition, select the “Go” icon.
- d. After data collection, the x-ray tube automatically powers down. The Peltier detector requires electricity for cooling. As long as the instrument remains plugged in, power is applied to the cooling circuit, even if the power switch is turned off.
- e. Use the Auto Peak Identification (thumbprint) and “MLK” icons to aid in x-ray peak identification. Hold down the Control key and press an Arrow key, ← or → , to change the element for which the MLK lines are shown.
- f. Spectra can be viewed, annotated, and overlaid in the WinTrace “Spectrum Viewer” application.
- g. A copy of the spectrum, with peaks labeled and acquisition conditions listed, will be retained in the case notes.

8.4 Quantitative Analysis

8.4.1 Method Development

Quantitative evaluation of spectra requires acquisition of the unknown spectra, acquisition of CRM calibration standards, acquisition of peak profiles for pure elements and selection of a mode of analysis that will be conducted in WinTrace “Method Explorer”.

- a. Determine favorable analysis conditions for each element of interest empirically by running qualitative analyses and/or by using the “Excitation Filter Guide”.

- b. Pure element peak profiles are collected in WinTrace “Acquisitions Manager” using a Reference Tray List. Before acquisition, be sure to specify the location the spectra will be saved by selecting “Edit” > “Spectrum Location”. These spectra will later be imported into the chosen Method. If “L” series spectral lines are of interest for quantitation, the conditions for acquisition of pure element peak profiles must be identical to those used to collect the unknown spectra. If only “K” series spectral lines will be analyzed, the acquisition conditions need not match.
- c. CRM spectra can be collected in WinTrace “Acquisitions Manager” using a Reference Tray List or can be collected directly into the Method developed for analysis. If collecting in “Acquisitions Manager”, before acquisition be sure to specify the location the spectra will be saved by selecting “Edit” > “Spectrum Location”. These spectra will later be imported into the chosen Method.
- d. Spectra for quantitative analysis can be collected in WinTrace “Acquisitions Manager” using a Quantitative Tray List or can be collected directly into the Method developed for analysis. Before acquisition, be sure to specify the location the spectra will be saved by selecting “Edit” > “Spectrum Location”. These spectra will later be imported into the chosen Method.

8.4.2 Analysis

The instrument software “Method Explorer” contains analysis techniques for semi-quantitative and quantitative analysis. Two of these are described briefly below. Select the analysis technique by selecting “File” > “Settings”.

- a. “Fundamentals Parameters (alpha)” is a fundamental parameters-based quantitation routine. This technique requires only a limited number of standards to generate calibration curves for the analytes of interest. It provides quantitative analysis over a wide range of concentrations and can be used with standards that are dissimilar from the unknown samples. The resulting calculated concentrations can be viewed as absolute or normalized values.
- b. “Intensity Correction” is an empirical technique that uses a suite of calibration standards to generate calibration curves. The standards must be similar in matrix to the unknown in order to account for matrix interactions. This analysis requires that the standards bracket the compositions of the analytes of interest in the unknown.
- c. Each analysis technique offers different options for spectrum processing. Typically, “Force through zero” should be selected (compensation for background is included in the software routine). The default setting for spectrum processing for Fundamental Parameters – Alpha is “XML” for every element. If a large peak dwarfs a smaller adjacent peak, setting the spectrum processing mode for the element with the larger peak to “Derivative” can improve the analysis of the smaller peak.

- d. Once all of the data are entered, the spectra are collected and the analysis parameters are chosen, select “Calibrate”, then select a sample list to “Analyze”. Results from the calibration and analysis are saved in the Method. These results can be exported in .csv format to a database program like Excel for statistical evaluation. If quantitative data are to be reported, the “Calibration” and “Analysis” reports will be retained in validation records and in the case file.

9 Instrumental Conditions

- a. The “Excitation Filter Guide” chart, provided by ThermoScientific, contains recommendations for analysis conditions that have been found to be useful for the preliminary screening of materials. If the specimen is a liquid sample or a vacuum sensitive material, choose a helium or air atmosphere as appropriate to the samples and the analysis objectives. Instrumental conditions can be adjusted at the discretion of the analyst to optimize the analyses.
- b. Acquisition times will depend on the conditions chosen and the sample area exposed to the incident beam to acquire sufficient counts for analysis.
- c. Two x-ray collimators are available, 1.0 mm and 8.8 mm. These are used to limit the x-ray beam size incident on the sample. For routine operation, the 8.8 mm filter wheel collimator is typically used.
- c. Three acquisition rates are available: Low, Medium, and High. The Medium rate normally provides sufficient resolution for qualitative or quantitative analysis. If the Low or High rate is preferred for a particular analysis, the Energy Adjustment routine must be run for the appropriate acquisition rate.

10 Decision Criteria

10.1 Instrument Performance

Energy adjustment of the instrument will be undertaken whenever the measured x-ray peak positions differ by more than 0.05 keV from their theoretical positions.

10.2 Spectral Analysis

General decision criteria for peak identification, spectral comparison and quantitative EDXRF analysis are described in *Compositional Analysis by EDXRF*. Additional considerations particular to this instrument are:

- a. The Thermo QUANT’X EDXRF generates a low intensity system peak at the energy of

aluminum $K\alpha$ that interferes with identifying minor amounts of aluminum in alloys.

- b. The on-board peak identification software does not present marker lines for escape peaks* or sum peaks. This requires the operator to calculate and identify these peaks associated with the major constituents of a sample. (*Labels generated by the peak ID routine can identify the general vicinity of escape peaks.)

11 Calculations

In dilute solutions, the measured x-ray intensity of a particular element is typically directly proportional to its concentration in solution. If several samples of differing concentrations are analyzed to create a calibration curve, a standard linear regression analysis can be used to calculate the best fit line to the data. Typically, such a curve can be established using four solutions with known concentrations of the element of interest and a matrix blank containing below the level of detection of the element of interest. Concentrations of unknown samples are then determined by reference to the linear fit of the data.

12 Measurement Uncertainty

Typically, the Thermo QUANT'X is not used for quantitative analysis. Should it be required, the measurement uncertainty will be estimated in accordance with *Chemistry Unit Procedures for Estimating Measurement Uncertainty* in the CU Quality Assurance and Operations Manual.

13 Limitations

Compositional Analysis by EDXRF contains general limitations of compositional measurement by x-ray fluorescence spectrometry.

Samples of adequate size (usually a few grams) are required for optimal results. Where only smaller samples are available, the use of a microspot EDXRF is recommended.

14 Safety

- a. The ThermoScientific QUANT'X produces x-rays. Wear an x-ray film badge or dosimeter when operating this instrument. The instrument has a protective enclosure with internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable the safety interlocks on this instrument.
- b. The detector and tube windows are comprised of beryllium and are extremely delicate. In the event of damage to a window, the beryllium dust created could pose an acute

health hazard. If this occurs, seal the chamber and seek assistance from the Laboratory Health and Safety Group.

15 References

Jenkins, R., *X-ray Fluorescence Spectroscopy*, Wiley Interscience, New York 1988

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FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
3	12/23/2015	Helium designated as optional in section 5.f. Operating parameter corrected in section 9.1.d. Peak labeling instruction added to section 9.2.c. Reorganized section 12 to make calibration curve procedure applicable to both liquid solutions and alloys. Editorially revised to clarify instructions throughout.
4	03/02/18	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 20 and is now designated Metal 501. Added personnel to section 2. Made minor editorial corrections throughout document. Clarified application in section 5.c. Incorporated section 7 into section 6 and renumbered subsequent sections. Added requirement for sampling plan retention in section 7. Removed reference to check sample in section 8.4.2.b, since verification requirements are addressed during method validation. Clarified definition in section 11. Updated safety requirements in section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy
Technical Leader

Date: 02/28/2018

General Chemistry
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QA Approval

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Operation of the Bruker M4 Tornado X-Ray Fluorescence Spectrometer

1 Introduction

The Bruker M4 Tornado is an energy dispersive x-ray fluorescence spectrometer with fine focus and microspot analytical capabilities. These techniques are used for the analysis of samples that are too small for bulk x-ray fluorescence analysis methods.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy, anthropology or general chemistry examinations. The operation of the Bruker M4 Tornado will follow the procedures outlined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF)* and the specific parameters described below.

3 Principle

The Bruker M4 Tornado has two excitation options to generate the incident x-ray beam:

- 1) a rhodium target, microfocus x-ray tube (50 kV, 30W max) with a polycapillary lens delivering a 25 µm spot (micro-XRF), or
- 2) a molybdenum target, fine focus x-ray tube (50 kV, 40W max) with a 1.0 mm fixed collimator (collimated XRF).

A silicon drift detector (SDD) measures photons emitted by the sample. These components are arranged in a top-down configuration, with a working distance of approximately 10 mm. Specimens are placed face up on an adjustable stage for measurement. Elements with atomic number equal to and greater than 11 (sodium) can be measured. Measurements can be made in air or under vacuum.

Video cameras provide sample surface images at approximately 10X and 100X magnification and a chamber image to aid in positioning the samples for measurement. Instrument control and data analysis are performed using ESPRIT software running on a personal computer that is connected to the analyzer.

4 Specimens

Samples as small as 25 μm in diameter can be examined using the instrument. Smaller samples may produce sufficient x-ray emission for measurement if they are positioned within the circle on the video screen aligned with the x-ray optic. If quantitative results are desired, it is necessary to prepare the samples so that they have a flat, smooth surface. The samples must also be sufficiently thick to allow reliable quantitation. In the case of liquid samples, a plastic cup can be used to contain the sample. The flat, upper surface of the liquid will provide the required analysis surface.

5 Equipment/Materials/Reagents

- a. Bruker M4 Tornado X-Ray Fluorescence Spectrometer
- b. Calibration standards:
 - i. Bruker “Nano” standardization block or any commercially pure manganese Certified Reference Material (CRM)
 - ii. FBI-B002 reference block or other zirconium CRM
- c. Mounting materials (optional):
 - i. Graphite planchet
 - ii. Durotak sample mounting adhesive
 - iii. Plastic sample cups (liquid samples)
 - iv. Chemplex X-Ray Mylar (2.5 μm thick) or polypropylene film (6.3 μm thick) or similar
- d. CRMs of alloy similar to the material being analyzed

6 Standards and Controls

Manganese and zirconium standards are used to adjust energy output and to verify adequate performance of the instrument. For quantitation, alloy class comparison, or to establish the absence of an element, CRMs as similar as possible to the material under analysis are used.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Basic Operation

The basic operation from a powered-down condition consists of the following steps.

- a. From a cold start, turn on the power to the computer and instrument.
- b. Open the control software ("M4" icon) and follow the on-screen prompts. Several modes that define regions of acquisition are available including Point, Line and Area modes (tabs at top of screen).
- c. If the instrument power has been off longer than 2 weeks, condition each tube to be used at 15 kV, 300 μ A for 5 minutes followed by 25 kV, 300 μ A for 5 minutes before operating at higher power.
- d. Position a specimen using the software drag bars or by entering known position locations. WARNING: The stage motion controller adjusts z height first. Assure that specimens will clear the tube guard by moving in x and y directions at low z (z=50mm typical), then adjust the z-height separately. Should the proximity sensor on the tube guard be activated, reset the motion controller by selecting "Reference all" on the "Stage" tab. Sample height must be incorporated in determining stage height to achieve desired working distance.
- e. Verify energy scale linearity:
 - i. Use either tube with no filter, in air or vacuum.
 - ii. In Point mode, acquire signal for 100 s live time on a Zr standard (FBI-002 block or other Zr CRM.)
 - iii. Identify the position of the maximum height of the Zr $K\alpha$ peak. If this energy differs by more than ± 0.05 keV from the expected peak energy of 15.746 keV (the

- weighted average of the Zr K α 1 and Zr K α 2 emissions), then perform an energy adjustment.
- iv. Energy adjustment: Open the System tab, then Spectrometer tab. Highlight Zr K α , select pulse rate of 130,000 cps, and energy ranges of 20 kV and 40 kV. Select Medium (precision), then “START”.
 - v. Verify the position of the Zr K α peak maximum as above. If repeated failure of the energy adjustment routine occurs, the instrument may have to be serviced to correct the problem.
 - vi. Record the verified Zr K α peak position in the instrument log.
- f. Verify energy resolution:
- i. Use either tube at full power with no filter, in air or vacuum.
 - ii. Position a Mn standard (Bruker “Nano” standardization block or other Mn CRM) for measurement.
 - iii. Open the System tab, then Spectrometer tab. Select Mn K α , 130,000 cps pulse rate, 40 kV, Medium (precision), then “TEST”.
 - iv. Run long enough to allow the FWHM (full width-half maximum) value to stabilize to one decimal place. If the FWHM value exceeds 148eV, instrument service may be required.
 - v. Record the Mn K α FWHM value in the instrument log.
- g. Verification of the energy linearity and resolution are performed each day prior to the instrument being used for casework. Instrument performance can be re-verified during a long course of testing at the operator’s discretion. Energy adjustment of the instrument will be undertaken whenever the x-ray peak positions differ by more than ± 0.05 keV from their theoretical positions.

8.2 General Analysis

- a. Insert the specimen(s). In Point mode, align the area of interest at the center of the circle displayed on the video image at 10X magnification. Rough surfaces must be placed so that a working distance of ~ 10 mm can be achieved at the region to be analyzed without obstruction from neighboring high points. For a liquid specimen, the surface of the liquid should be within 2 mm of the top of the sample cup.
- b. Select the appropriate measurement atmospheric condition: air or vacuum. Liquids or vacuum sensitive materials must be tested in an air atmosphere. To test in vacuum, select the vacuum icon.

- c. Focus the area to be measured at 100X magnification. An autofocus function is available through an icon adjacent to the video output.
- d. Select the appropriate x-ray tube, tube voltage and current to achieve a dead time of approximately 30% when collecting a spectrum. Acquisition parameters and filters can be adjusted to optimize the analysis conditions. An aluminum alloy, carbon steel and a silver alloy, for example, would all require different operating conditions for optimal results. A set of useful, basic analysis conditions can be found in 9 Instrumental Conditions. It is typical to analyze materials of atomic number 22 (Ti) and below under vacuum atmosphere.
- e. Acquire a spectrum for an appropriate duration to collect sufficient counts for analysis. Acquisition times will depend on the conditions chosen, sample material and the sample area exposed to the incident beam.
- f. Identify the peaks present in the spectrum using the interactive periodic table in the analysis software. Software-generated identifications must be interpreted by an experienced operator to prevent misidentification of energy peaks. The system software automatically filters the escape peaks of predominant elements from the spectral display. Refer to CU Metallurgy SOP *Compositional Analysis by EDXRF* for more details regarding peak labeling.
- g. Print an individual spectrum using the innermost IMPORT-EXPORT menu, print command. This command will generate the filename and acquisition conditions on the printout. Write the resulting percent dead time on this printout. (Although dead time is saved within the raw data file, it will not print automatically.) To print multiple overlaid spectra, highlight and checkmark all of the spectra from the viewing window list before selecting the print command from the innermost IMPORT-EXPORT menu. If not printed on separate, individually printed spectra, record the acquisition conditions for the multiple, overlaid spectra in the case notes.
- h. Ensure the tube power returns to standby (20kv, 50μA) after testing is completed. If the instrument will be unused for two or more days, shut off the x-ray tube. Close the software.

8.3 Quantitation of Data

In general, x-ray spectra are not used to generate quantitative data. However, the spectra acquired from a sample can be processed to yield weight concentrations of the elements detected. The ESPRIT analysis software contains several methods for semiquantitative and quantitative

analysis, including standardless fundamental parameters-based and standards-based analysis that can be applied to bulk materials or coating systems. Validation of any of these methods will include analysis of CRMs similar to the material under examination to assess the reliability of the data being generated.

9 Instrumental Conditions

Table 1 contains a set of basic analysis conditions that have been found to be useful for preliminary screening of materials. Other conditions are permissible and will be determined by the operator based on analytical objectives.

Table 1: Basic Operating Parameters for Analysis

<u>Control Tab</u>	<u>Parameter</u>	<u>Value</u>
Spectrometer	Maximum energy	40keV
	Mode	Normal operation
	Cooling	Thermostat
X-ray tube 1 (microfocus tube)	Voltage	40 kV
	Current	250 μ A
	Filter	none
Acquire	Range	40 keV (provides ~10 ev/channel)
	Preset	100 seconds
(Top left icon)	Atmosphere	Vacuum

10 Decision Criteria

10.1 Instrument Performance

Energy adjustment of the instrument will be undertaken whenever the x-ray peak positions differ by more than ± 0.05 keV from their known positions.

10.2 Spectral Analysis

General decision criteria for peak identification, spectral comparison and quantitative EDXRF analysis are described in *Compositional Analysis by EDXRF*. Additional considerations particular to this instrument are:

- The Bruker M4 Tornado microfocus x-ray tube with polycapillary lens generates a

Bremsstrahlung excitation spectrum that tails off at the higher end of the spectrum. This inhibits fluorescence of lines with absorption energies greater than 30keV, regardless of any further increase in tube voltage. This can result in significantly higher limits of detection for K line emissions from elements with atomic number of approximately 48 and higher. Conditions may be adjusted to optimize the L and M lines emissions from these elements to aid detection.

- b. Each tube will produce its own characteristic energy peaks, e.g., the rhodium x-ray tube should not be used if Rh is expected to be in the sample and conversely, Rh should not be reported to be present in the sample unless verified. The Mo x-ray tube can be used for verification.
- c. The geometry of the tube-sample-detector arrangement creates diffraction peaks from crystalline materials (including metals). These artifacts in the compositional spectra can usually be readily differentiated from x-ray fluorescence peaks because diffraction peaks appear broader across the energy axis and because their positions often differ from known atomic emissions. When using the microfocus polycapillary tube to analyze steels, the Bruker M4 Tornado often presents a small diffraction peak near 3.5 keV.

11 Calculations

Not applicable.

12 Measurement Uncertainty

Typically, the Bruker M4 Tornado is not used for quantitative analysis. Should it be required, the measurement uncertainty will be estimated in accordance with *Chemistry Unit Procedures for Estimating Measurement Uncertainty* in the CU Quality Assurance and Operations Manual.

13 Limitations

Compositional Analysis by EDXRF contains general limitations of compositional measurement by x-ray fluorescence spectrometry. Additional limitations specific to this instrument are:

- a. Vacuum sensitive samples, like liquids, that are suspected of containing elements with atomic number $Z < 22$ (Ti) cannot be measured in the Bruker M4 Tornado. These can be measured in a system offering a helium overpressure atmosphere.

- b. Spectral artifacts, including diffraction peaks, must be considered during analysis. (See 10.2 Spectral Analysis).

14 Safety

- a. The Bruker M4 Tornado produces x-rays. Wear an x-ray film badge or dosimeter when operating this instrument. The instrument has a protective enclosure with internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable the safety interlocks on this instrument.
- b. The detector and tube windows are comprised of beryllium and are extremely delicate. In the event of damage to a window, the beryllium dust created could pose an acute health hazard. If this occurs, seal the chamber and seek assistance from the Laboratory Health and Safety Group.

15 References

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FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
1	03/15/2016	Added references to a second x-ray tube option (Sections 1, 3 8.2d-e, 9-Table 1, 10.2b and 14b). Clarified spectrometer verification routine (Section 8.1c-g). Corrected terminology (Section 8.2h).
2	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 27 and is now designated Metal 502. Added personnel to section 2. Clarified application of instrumental conditions, Section 9. Made minor editorial corrections throughout document. Added requirement for sampling plan retention in section 7. Revised sections 8.1.e and 8.1.f to clarify instrument verification procedure. Added instrument shut-down directions in section 8.2.h. Added section 10.2.b to clarify x-ray tube influence. Updated safety requirements in section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy
 Technical Leader

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General Chemistry
 Technical Leader

Date: 02/28/2018

Anthropology
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Date: 02/28/2018

Chemistry
 Unit Chief

Date: 02/28/2018

Trace Evidence
 Unit Chief

Date: 02/28/2018

QA Approval

Quality Manager

Date: 02/28/2018

Operation of the Olympus Delta Premium Handheld X-Ray Fluorescence Spectrometer

1 Introduction

The Olympus Delta Premium Handheld X-ray Fluorescence Spectrometer is a battery-operated, portable energy dispersive x-ray fluorescence spectrometer (EDXRF) used for non-destructive compositional analysis of solids ranging in atomic number from magnesium to uranium. The instrument operates in air.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy or anthropology examinations. The operation of the Olympus Delta Premium will follow procedures defined in the current revision of Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) *Compositional Analysis by Energy Dispersive X-ray Fluorescence Spectrometry (EDXRF)* and the specific parameters described below.

3 Principle

The Olympus Delta Premium uses a Rh target x-ray tube (4W, 200 μ A maximum) to generate the incident x-ray beam and a silicon-drifted detector (SDD) to collect and measure the photons emitted by the sample. These components are contained in a portable housing. Light elements (such as Mg, Al, and Si) are measured by optimizing beam conditions, acquiring for extended times, compensating for atmospheric pressure (altitude), and measuring over a short, protected beam path.

Electronic control and analysis software is contained within the handheld instrument but is also accessible through integrated software that can be run externally from a personal computer. To protect personnel from emitted x-rays, the instrument has a proximity interlock that aborts a test if a sample is not in front of the measurement portal.

The instrument is configured in the “Alloy Plus” mode. This mode allows several beam options for measurement. The analysis software uses a fundamental parameters routine to calculate elemental compositions that are then compared to libraries of alloy grades. The software is designed to dynamically report alloy matching. Qualitative alloy class matches can be verified

using appropriate CRMs. However, to use this matching functionality for quantitative analysis, relevant validation must be performed for specific elements of interest in the specific matrix of interest.

4 Specimens

Any solid sample can be analyzed on this instrument, provided a flat area of the sample can be presented to the measurement window of the analyzer. The measurement window is covered by a thin polymer film. Care should be taken so that protruding surfaces do not puncture this film. The entire portal does not have to be completely covered for qualitative measurement. If quantitative results are desired, it is necessary to prepare the samples so that they have a flat, smooth surface.

5 Equipment/Materials/Reagents

- a. Olympus Delta Premium Handheld X-Ray Fluorescence Spectrometer
- b. Calibration standard – AISI Grade 316 stainless steel (316 SS) standardization coupon
- c. CRMs of alloys similar to the type(s) under analysis, if alloy class comparison or elemental quantitation is required
- d. Delta docking (optional) – This station provides convenient storage and recharging for the instrument. It also houses a calibration standard, communication port and an extra charging station for a reserve battery.

6 Standards and Controls

Basic standardization of the instrument requires an AISI Grade 316 stainless steel standardization block. A standard is permanently mounted in the docking station, but any 316 SS CRM can be used to standardize the instrument. For quantitation, alloy class comparison or to establish the absence of an element, CRMs as similar as possible to the material under analysis are used.

Standardization is prompted daily when the instrument is powered up. This performance check is accomplished by repeated analysis of a 316 SS standardization disc. Initiation of the automated routine is described in 8.1 Basic Operation.

7 Sampling

If large numbers of physically indistinguishable samples are received for compositional analysis, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

8 Procedure

8.1 Basic Operation

The instrument can be used as a stand-alone unit or controlled by an external computer. For either control method:

- a. Remove uninvolved personnel from the vicinity where the instrument will be operated (to beyond ~5m radius or behind walls).
 - b. When the CalCheck message is present, place the unit in the docking station or present a 316 SS standardization coupon to the measurement window. Select the green ✓ Cal icon to start the standardization routine. If the CalCheck is unsuccessful, the routine will be performed again. If repeated failure of the CalCheck occurs and the operator cannot correct the problem, the instrument must be serviced by an appropriately qualified person (see 14 Safety).
 - c. Select the measurement beam condition(s). The “Alloy Plus” configuration allows three different beam conditions to be used in four different combinations:
 - i. Beam Conditions:
 - Beam 1 (40kV excitation voltage)
 - Beam 2 (13kV)
 - Beam 3 (8kV)
 - ii. Combinations:
 - Single Beam with light element (LE) suppression – Beam 1 only
 - Single Beam with LE – Beam 1 only; software infers LE presence
 - Two Beams – Beam 1 followed by either Beam 2 or Beam 3
 - SmartSort – Beam 1 always; adding Beam 2 or Beam 3 only as needed.
- Specific grades are set up to automatically extend testing time. Specific elements trigger the use of an additional beam condition.

- d. Fill out the specimen identification field(s) prior to each test or maintain a log recording the auto-generated test number and the corresponding specimen identification. (Warning: Post-test editing of specimen information is not enabled on this system.)
- e. Acquire data; label spectra; print results to .pdf files.

8.1.1 Stand-alone Operation

- a. Insert a charged battery into the analyzer handle and turn on the instrument.
- b. Read the radiation safety notice screen and acknowledge user certification.
- c. Perform CalCheck.
- d. Select beam conditions from “Test Condition” > “Mode”. Enter specimen ID via “Setup” > “Label Defaults”.
- e. Acquire sample measurements by holding the instrument against the area of a specimen to be analyzed. Start a test from the green arrow on the touch screen or pull the trigger. The red x-ray warning light stops flashing when the test is complete.
- f. Test session results are grouped by date. Innov-X software on an external computer can be used to import the spectra for analysis and printing reports.

8.1.2 Computer Acquisition

- a. Turn on the computer first, letting it become fully functional. Connect a USB cable between the computer and the handheld unit. Turn on the handheld unit. The Windows Mobile application will start. Choose “Connect without setting up device”.
- b. Open the Innov-X control software on the computer. Select “Close Device App”. Select “Import to PC”. Select “Start”.
- c. Perform CalCheck. Select beam conditions from “Test Conditions” > “Mode”. Enter specimen ID via “Setup” > “Results Test Info”.
- d. Acquire sample measurements by holding the instrument against the area of a specimen to be analyzed. Start a test from the green arrow on the computer screen or pull the trigger. The red x-ray warning light stops flashing when the test is complete.

8.2 Qualitative Analysis

- a. Verify the peak identification performed by the instrument by inspecting the spectrum. Annotate the spectrum using the interactive periodic table. (Refer to *Compositional Analysis by EDXRF* for more details regarding peak identification.)
- b. Set report parameters on the “Print” tab under “Test Conditions” > “Setup”. (The multi-print function is not enabled.) From the “View Data” page, save each result as a .pdf using the “Print” icon.

8.3 Quantitative Analysis

Although the Olympus Delta Premium is configured to output quantitative compositions, at best these can be considered approximations unless specific alloy classes have been validated.

9 Instrumental Conditions

The instrument offers limited options for operational parameters. The Alloy Plus two-beam mode provides adequate results for qualitative alloy classification and comparison.

10 Decision Criteria

General decision criteria for peak identification, spectral comparison and quantitative EDXRF analysis are described in *Compositional Analysis by EDXRF*. Options specific to this instrument include:

- a. Spectra are automatically analyzed and quantified by the Olympus Delta Premium. Results are presented in table format. These compositional results should be considered as qualitative information to determine element components within the measured specimen, unless a quantitative validation has been performed for the specific matrix. To verify the accuracy of the peak identification, the graphical spectrum can be examined by the analyst. Peak labels can be generated by selecting the instrument-identified elements from the interactive periodic table chart. The software does not present marker lines for escape peaks or sum peaks. This requires the analyst to calculate and identify these peaks which are usually associated with the major constituents of a sample.
- b. The nominal chemistry functionality looks for “invisible” elements based on grade

identification, including Al in Beam 1 or elements like Be or C that are not detectable by XRF. These inferred elements can be used by an analyst to direct investigation (e.g., to seek an alternate identification method) but should not be used for reporting qualitative or quantitative results.

11 Calculations

Not applicable.

12 Measurement Uncertainty

Typically, the Olympus Delta Premium is not used for quantitative analysis. Should it be required, the measurement uncertainty will be estimated in accordance with *Chemistry Unit Procedures for Estimating Measurement Uncertainty* in the CU Quality Assurance and Operations Manual.

13 Limitations

Compositional Analysis by EDXRF contains general limitations of compositional measurement by x-ray fluorescence spectrometry. The Olympus Delta Premium is restricted to operating in air, thus it cannot detect fluoresced x-rays from any element lower in atomic number than Mg.

Flat samples of adequate size are required for optimal results. Where only smaller samples are available, the use of a micro x-ray fluorescence spectrometer is recommended.

14 Safety

- a. Only trained operators may use this instrument. A sign stating “TO BE OPERATED ONLY BY TRAINED PERSONNEL” will be stored with the device.
- b. The Olympus Delta Premium produces x-rays that may propagate many meters in open air. Operate the handheld XRF spectrometer with respect for the direction of the emitted x-ray beam. Assure no personnel are present in the region in front of the instrument in the direction of the emitted beam when operating.

- c. The unit is equipped with a proximity sensor that shuts off the x-ray tube if a sample is not in place in front of the measurement window. Also, a software trigger lock will engage if five minutes pass between tests. Both situations require user intervention to unlock/restart testing. All personnel operating the spectrometer routinely are monitored via personal radiation monitors (dosimeters), administered at the unit level and tracked by the Health and Safety Group.
- d. Internal components within the handheld device contain beryllium windows. These are extremely delicate and, if damaged, the beryllium dust created could pose an acute health hazard. If this occurs, isolate the instrument and seek assistance from the Laboratory Health and Safety Group.
- e. The Prolene window covering the measurement window can be exchanged by the instrument operator following the instructions provided by the manufacturer. Never open the main housing of the instrument. Only a qualified manufacturer's representative or radiological technician should attempt to service this instrument.

15 References

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FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
0	08/18/2014	Original issue.
1	03/02/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 26 and is now designated Metal 503. Added personnel to section 2. Made minor editorial corrections throughout document. Incorporated section 7 into section 6 and renumbered subsequent sections. Added requirement for sampling plan retention in section 7. Updated safety requirements in section 14. Added additional references to section 15.

Approval

Redacted - Signatures on File

Metallurgy
Technical Leader

Date: 02/28/2018

Anthropology
Technical Leader

Date: 02/28/2018

Chemistry
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Date: 02/28/2018

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Unit Chief

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QA Approval

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Date: 02/28/2018

Operation of Rockwell Hardness Testers

1 Introduction

The Rockwell hardness of a material is a measure of its ability to resist permanent deformation when in contact with an indenter under load. Hardness measurements can aid in determining material production characteristics (alloy class and heat treatment), degradation (during service or post-service exposure) and the uniformity of the material throughout a component. Components can also be tested to determine conformance with specified hardness requirements.

Due to fairly accurate quantitative relationships between hardness and other mechanical properties of materials, hardness tests can be used to estimate the tensile strength of some metals without the need to perform more destructive mechanical testing.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy examinations. Hardness can be tested on components of any shape, as long as the part can be mounted in a fixture that provides sufficient support to isolate the test load application from any other load and that prevents contact between the component and any part of the indenter other than its tip.

3 Principle

The Rockwell hardness test is based on the difference of indenter depth from two load applications. An indenter is pressed into the material under an initial minor load to establish a zero point. A major load is then added, causing additional penetration into the specimen. After the specified dwell time for the major load, it is removed while still keeping the minor load applied. The resulting Rockwell number represents the difference between the zero level established by the minor load prior to the major load being deployed and the depth associated with the minor load after the major load is withdrawn.

There are two general classes of Rockwell test: Rockwell and superficial Rockwell. In Rockwell testing, the minor load is 10 kgf and the major load is 60, 100 or 150 kgf. In superficial Rockwell testing, the minor load is 3 kgf and major load is 15, 30 or 45 kgf. In both types of test the indenter may be either a diamond cone or a tungsten carbide ball. Each Rockwell scale is defined by the use of a specific indenter and major load. The appropriate scale will depend upon the size of the component to be tested and on its actual hardness.

4 Specimens

At a minimum, a specimen subjected to Rockwell hardness testing must be large enough to receive three hardness indentations. The centers of all indentations must be at least three indentation diameters apart and not less than two and one half diameters from all edges. The specimen thickness must be a minimum of 10 times the indentation depth for a homogeneous material and may be more for unfavorably oriented material. Tables correlating the minimum required specimen thicknesses with the measured hardness are available (see section 16 References). Adjustment of the test loads or indenter type may be necessary during testing to accommodate these requirements.

For accurate and precise measurement, the test surface presented to the indenter must be flat and perpendicular to the loading axis. The specimen may be ground using methods that do not impart elevated temperature or cold work that could alter the material hardness. Finish grinding is usually sufficient to provide reproducible measurements, but superficial testing may require a lapped or polished finish. Correction tables exist for measurements made on smooth, curved surfaces such as pipes and can be used when surface grinding is undesirable (see section 16 References). Prior to hardness testing of case samples, the user will verify the instrument performance using one or more CRM test blocks. The performance check procedure can be found in section 9 Procedure.

5 Equipment/Materials/Reagents

- a. Rockwell Hardness tester, such as Wilson Model 524T Rockwell Hardness Tester, or equivalent, with diamond brale 120° cone or 1/16 inch diameter carbide ball indenter
- b. Certified reference material (CRM) test block(s) appropriate for the hardness scale(s) in use
- c. Various geometries of platens and specimen holders
- d. Lint-free wipes

6 Standards and Controls

Adequate instrument performance is verified using CRM test blocks. CRMs will be tested and the results recorded every time the instrument is powered up for use or the load or indenter is changed. CRM blocks must be certified for the indenter and load (scale) of test performed in order to assure the indenter is seated properly, it contains no defects, and the load application is reliable. At least one CRM in the expected hardness range (within 15 Rockwell points if possible) of the sample must be tested prior to analysis. Additional blocks may be tested at the

examiner's discretion, typically one above and one below the expected hardness range of the sample to be measured.

7 Calibration and Verification

The instrument is calibrated annually by a certified and licensed service provider that meets the LOM requirements. Prior to hardness testing of case samples, the user will verify the instrument performance using one or more CRM test blocks. The performance check procedure can be found in 9 Procedure.

8 Sampling

Whole components, or sections thereof, are typically presented to the indenter for hardness testing. Hardness readings should be interpreted to apply only to that material in the immediate vicinity of the indentation. Since cold work and thermal gradients during heat treatment can alter hardness, regions of a component with differing thickness, geometry and/or surface treatment should be examined separately.

Sampling of items examined under this protocol is determined by the nature of the evidence received and can consist of multiple items or one or multiple regions of interest on one item. Sometimes the physical nature of the sample will best lend itself to hypergeometric sampling or other standard random sampling methods. If large numbers of physically indistinguishable samples are received for Rockwell hardness testing, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be recorded in case notes.

9 Procedure

9.1 Instrument Performance Check

- a. Select an appropriate combination of indenter type and load (scale) for the samples to be tested. Some of the available tests include the following:

Rockwell Scale	Indenter	Load (kgf)
A	C diamond	60
B	1/16" carbide ball	100
C	C diamond	150
N-15	N diamond	15
N-30	N diamond	30
N-45	N diamond	45
T-15	1/16" carbide ball	15
T-30	1/16" carbide ball	30
T-45	1/16" carbide ball	45

For homogeneous bulk materials, either the Rockwell B or C scale is typically appropriate depending on the material properties. Superficial tests (N and T scale) may be run when the sample is too thin to use a B or C scale or when the indents produced would be too destructive. The A scale is used primarily for testing tungsten carbide and other very hard materials. The list above is not inclusive, and other Rockwell tests are available.

- b. To ensure that the indenter is seated properly, make at least one hardness measurement on a suitable test piece using the maximum load for the indenter to be used; do not record these results. Repeat this step any time the indenter is changed during use.
- c. Select a CRM test block for the hardness range and test scale being run. It is recommended that the hardness of a standardized test block and the sample fall within +/- 15 Rockwell points for carbide ball or diamond brale indenter tests. Alternatively, two standardized test blocks; one with a higher hardness and one with a lower hardness than the sample of interest, within the same scale, may be used for verification. Additional CRM test blocks may also be tested at the examiner's discretion.
- d. Place the appropriate CRM block onto the specimen stage (platen). Measure only the certified side of the test block. Indentations made on the opposite side are not only invalid but may also deleteriously affect readings subsequently taken on the correct certified side of the block.
- e. Raise the stage until the test block contacts the indenter and the minor load is applied. Adjust the zero offset of the scale if necessary.

- f. Apply the major load. The instrument will indicate the end of the appropriate dwell time. For manual machines, release the major load. Read the Rockwell Hardness Number (HR) from the correct scale on the analog dial face. For an automatic tester, the major load is removed without intervention. The HR will appear in the electronic readout.
- g. Repeat the test to obtain at least two readings from the CRM, adjusting the sample position away from the center of any previous indentations by at least three indentation diameters to avoid any plastically deformed or damaged material.
- h. Ensure that the hardness readings are consistent with the hardness range reported on the CRM certification sheet. Alternatively, see 16 References (ASTM E18) to calculate the error E and repeatability R for comparison to the maximum tolerances given. If consistent hardness readings are not obtained, wipe the platen surface and the back of sample with a clean cloth to remove any debris from the standard and retest to verify. If this is not successful, replace the indenter and re-verify. If all efforts to alleviate inconsistent readings are unsuccessful, the instrument will be serviced and recalibrated by a certified and licensed service provider that meets the LOM requirements.
- i. Record the date, operator, measured values, verification result and other appropriate information on the instrument log.

9.2 Sample Testing

- a. Clean any loose debris or scale from both the specimen test surface and the support surface. Align the specimen so the test region is flat relative to the indenter geometry and the test region and support surface are perpendicular to the load axis. Curved surfaces may be tested subject to the limitations described below. Small components may be mounted in polymer molds to accommodate polishing. Mounted samples that rely on the mounting material to provide the support surface must be tested using a superficial hardness scale.
- b. For steels, consult section 16 References (ASTM E18) for minimum thickness guidelines for the selection of the appropriate Rockwell scale. For other materials, the sample thickness should be a minimum of ten times the indentation depth. In general, materials with a surface hardness gradient (e.g. due to carburizing or alloy depletion) should be tested using the microhardness method.
- c. For convex cylindrical surfaces, consult section 16 References (ASTM E18) for minimum diameter guidelines.
- d. Mount the sample beneath the indenter on an appropriate platen or gripping fixture. Assure that the manner of mounting will support the applied test load, and the specimen will not move when load is applied.

- e. Apply the minor load and inspect to assure that only the indenter tip will contact any part of the specimen.
- f. Apply the major load. The instrument will indicate the end of the appropriate dwell time. For manual machines, release the major load. Read the measurement from the correct scale on the analog dial face. For an automatic tester, the major load is removed without intervention. The measurement will appear in the electronic readout.
- g. If the measured hardness is outside of the range initially verified, repeat the performance check procedure in 9.1 with appropriate test block(s).
- h. Repeat to acquire a minimum of three test readings. If comparative statistics are required, acquire additional readings in the areas of interest. Typically, 5 to 10 measurements per area will provide sufficient data for meaningful statistical comparisons.

10 Instrumental Conditions

The instrument must not be subject to vibration when the test is underway. The indenter must be seated securely and must be free of foreign material. Indenters must be free of nicks, broken edges or flat areas. Compromised indenters must be replaced, a new indenter securely seated, and the instrument must be performance checked..

The test load and indenter type used are dependent upon the hardness and shape of the material in the test region.

11 Decision Criteria

Verification is acceptable when the hardness values determined by the instrument on a given CRM fall within the range of values indicated on its certificate or if calculated E and R are within the maximum tolerances allowed (see step 9.1.h Instrument Performance Check). If the verification remains unsuccessful after assuring that the test block and indenter are sound, then the instrument must be serviced and re-calibrated by a certified and licensed service provider that meets the LOM requirements.

Variability in hardness measurements may be significant. If this occurs the results should not be averaged but should be reported individually or as a range for the area tested.

When evaluating whether sets of test readings from different locations on a single specimen or from different specimens are statistically distinguishable, a Student t-test of the local means will be employed.

12 Calculations

12.1 Quantitative Analysis

- a. Hardness values are automatically calculated by the instrument software. These readings, and their associated measurement uncertainty, may be used to report a range or series of measurements.
- b. To report averaged hardness measurements collected over a broader area, collect at least five values. Calculate and report the mean and expanded measurement uncertainty.

Mean is calculated as: $\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$, where $\sum_{i=1}^n x_i$ is the sum of the measurements, n is the number of measurements and \bar{x} is the mean hardness value.

- c. In order to determine whether a part is of sufficient thickness, the depth of a Rockwell indentation can be calculated as follows (HR = Rockwell Hardness Number):

For a diamond indenter:

$$\text{Depth (mm)} = (100 - \text{HR}) \times 0.002 \text{ mm}$$

For a ball indenter:

$$\text{Depth (mm)} = (130 - \text{HR}) \times 0.002 \text{ mm}$$

For Rockwell superficial tests, regardless of indenter used:

$$\text{Depth (mm)} = (100 - \text{Superficial Hardness Number}) \times 0.001 \text{ mm}$$

Such values can also be interpreted from a table that relates maximum allowed hardness for a given thickness on a specific scale (see section 16 References, ASM Handbook vol. 8).

- d. General correlation between hardness scales, e.g. Rockwell to Brinell, can be read from appropriate conversion charts (see step 14.e Limitations).

12.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student-t test statistic of the sample means is typically used for the comparison. Two samples are deemed to be “indistinguishable” in the property under consideration if the two samples differ by less than the preselected critical t value (t_{critical}). The critical t value is typically chosen so that a value of $\alpha = 0.05$ can be achieved for the analysis and is determined by the degrees of freedom

associated with the measurement. An $\alpha = 0.05$ means there is a 5.0% chance of incorrectly rejecting a match between two samples when one actually exists.

To perform this test, the means and variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where \bar{x}_a is the average value of the measurements on sample “a”,

$\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample. The variance of the individual measurement values from sample “a” is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

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

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 \left(\frac{1}{n_a} + \frac{1}{n_b} \right)} \right)} \right| > t_{critical}$, the samples are concluded to have a statistically significant

difference in hardness. If not, the samples are concluded to be indistinguishable in hardness.

Typically five or more measurements per sample are used for performing comparisons.

13 Measurement Uncertainty

In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accordance with the Chemistry Unit *Procedures for Estimating Measurement Uncertainty*. Each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated. 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 cases, instrument measurement uncertainties will not be reported because they are considered negligible.

Quantitative data are sometimes used for comparative purposes. Expanded uncertainty should not be used for these inter-comparisons because it increases the probability that two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion).

14 Limitations

- a. There is no upper hardness limit when using the diamond indenter for Rockwell testing. Because Rockwell diamond indenters are not calibrated below 20 HRC (Rockwell hardness C) points, they should not be used when the readings fall below this level. Rockwell scales using ball indenters range from 0 to 130 points; however, readings above 100 points should be avoided since the ball can be easily damaged. When testing with a ball indenter above 100 points, it is necessary to change the ball frequently to avoid measurement errors.
- b. The material immediately surrounding the indentations is cold worked due to the plastic flow of material caused by the indentation process. Specimen geometry in this test region must not interrupt this normal flow. For example, measurements made on concave or convex surfaces will be altered by additional constraint, or lack thereof. Such measurements should be considered approximate. Estimated correction factors for some geometries are provided in section 16 References (ASTM E18).
- c. Inhomogeneities in the plastic flow region of an indentation will adversely affect the hardness measurement. Materials with large inclusions or a surface hardness gradient may be better characterized using a microhardness method.
- d. Cold work typically extends below an indentation to approximately ten times the indentation depth. It is recommended that the thickness of the specimen be a minimum of 10 times the indentation depth. For specimens of insufficient thickness to meet this requirement for an appropriate Rockwell scale, superficial Rockwell or microhardness testing may be considered. For steels, see section 16 References (ASTM E18) for minimum thickness guidelines for the selection of the appropriate Rockwell scale. These guidelines for appropriate specimen dimension are not to be substituted for the sound engineering judgment of the trained operator.
- e. Conversion tables relating hardness values measured on different scales are only approximate and never mathematically exact due to the difference in cold working response of a material to the indenter shape and load applied. All conversion tables of hardness scales, including those in section 16 References, are based on the assumption that the metal tested is homogeneous to a depth several times as great as the indentation. If not, different loads and different indenter shapes would penetrate, or encounter the resistance of, metal of varying hardness depending upon the indentation depth.

Nevertheless, conversion is of considerable value when comparing different hardness scales in a general way.

- f. Estimates of the tensile strength of carbon steels based on hardness measurements are generally considered to be accurate to $\pm 10\%$ of the estimated strength.

15 Safety

Standard safety precautions, such as wearing protective gloves, should be observed when handling evidentiary materials of hazardous nature. Electrical or mechanical hazards may require special precautions.

This instrument SOP has the following specific safety requirements:

- Wear safety glasses when making hardness indentations
- Wear personal protective gear and use engineering controls that are appropriate for the task being performed when preparing specimens.

If additional guidance is required, contact the Laboratory Health and Safety Group.

16 References

ASM Handbook, Volume 8, Mechanical Testing and Evaluation, ASM International, Materials Park, OH 2000, or latest revision

ASTM Method E18: *Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials*, ASTM International, West Conshohocken, PA, latest revision

ASTM Method E140: *Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness*, ASTM International, West Conshohocken, PA, latest revision

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FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
3	02/05/2014	Section 4 updated to align required indentation spacings with those of ASTM E18-12. Updated section 5 to reflect requirement to use a carbide ball in testing. Section 9.1 updated to include requirement to test specified test blocks when performing verification of the tester. Text amended to require verification measurements to be recorded in the instrument verification log. Minor formatting change made. Section 9.2 includes minor change in terminology. Minor grammatical change made in section 10. Section 11 updated to include requirement to include expanded estimate of uncertainty when reporting hardness values. Section 12 includes minor language updates. Formula for sample variance simplified. Typographical error in formula for variance of mean corrected. Section 13 has been rewritten to better reflect requirements on measurement uncertainty. Section 14 updated and reference source for correction factors added. Section 16. References have been updated.
4	12/21/2018	Renumbered Metallurgy SOP Manual documents; this document was formerly Metal 14 and is now designated Metal 701. Added personnel to section 2. Made minor editorial corrections throughout document. Added item to section 5. Revised sampling statement in section 8. Created a table in section 9.1 to clarify procedure. Separated quantitative and comparative calculations in 12. Added α and $t_{critical}$ selection process to 12. Added additional detail to section 14. Updated measurement uncertainty policy in section 13. Revised PPE requirements in section 15. Updated references section 16.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader .

Date: 12/20/2018

Chemistry Unit Chief .

Date: 12/20/2018

QA Approval

Quality Manager .

Date: 12/20/2018

Operation of Microhardness Testers

1 Introduction

Microhardness testing is an indentation hardness method that can aid in evaluating production characteristics, degradation (during service or post-service exposure), and the uniformity of the material throughout a component. Common applications include determining:

- local hardness in a limited region (such as case depth, multiple layers, or surface degradation)
- hardness of metals embedded in a secondary material (metallographic mounts)
- hardness of parts too thin or too small to register valid results via standard (macro) indentation testing.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy examinations. The following procedures outline the basic process for Knoop or Vickers microhardness testing using equipment with a digital filar optical measuring system and automated hardness calculation capability.

3 Principle

To measure microhardness, a diamond indenter of specified shape is pressed, under load of up to 1000 grams, into a polished metal surface. The indenter shape is specific to the type of test performed, Knoop or Vickers. The size of the resulting impression is measured microscopically and related through the appropriate correlation equations to hardness scales. Since the Vickers and Knoop indenters are shaped differently, the choice of test type will depend on the shape of the feature(s) to be characterized.

4 Specimens

At a minimum, a specimen subjected to microhardness testing must be large enough to receive three hardness indentations, all of which are sufficiently spaced to prevent interactions between the measurements. The specimen thickness must exceed two times the depth of the indentations for a homogeneous material and potentially more for unfavorably oriented material. An appropriate adjustment of the test loads and/or the indenter shape must be chosen to accommodate these requirements.

For accurate and precise measurement, the test surface presented to the indenter must be flat, smooth and perpendicular to the loading axis. The specimen should be ground and polished using methods that do not impart high temperatures or result in cold work that could locally alter the hardness of the material. (See the CU Metallurgy SOP *Procedure for Metallographic Examinations*.)

5 Equipment/Materials/Reagents

- a. Microhardness tester with integral diamond indenters, magnifying objectives, optical filar measuring system and automated hardness calculation
- b. Certified Reference Material (CRM) test block(s) appropriate for the parameters being used; i.e. only use Vickers hardness standards with the Vickers diamond indenter and Knoop hardness standards with the Knoop diamond indenter
- c. Specimen fixture or fixturing material, e.g., beeswax or clay or universal specimen holder

6 Standards and Controls

Adequate instrument performance is demonstrated using CRM test blocks. CRMs will be tested and the results recorded every time the instrument is powered up for use or the load or indenter is changed. CRM test blocks must be certified for the type (indenter and load) of test performed in order to assure that the indenter is seated properly, contains no defects and the load application is reliable. At least one CRM in the expected hardness range of the sample must be tested prior to sample analysis. Additional blocks may be tested at the examiner's discretion.

7 Calibration and Verification

The instrument is calibrated annually by a certified and licensed service provider that meets the LOM requirements. Prior to microhardness testing of case samples, the user will verify the instrument performance using one or more CRM test blocks. The performance check procedure can be found in 9 Procedure.

8 Sampling

Whole components, or sections thereof, are typically presented to the indenter for microhardness testing. Microhardness readings should be interpreted to apply only to that material in the immediate vicinity of the indentation. Since cold work and thermal gradients during heat treatment can alter hardness, regions of a component with differing thickness, geometry and/or surface treatment should be examined separately.

Sampling of items examined under this protocol is determined by the nature of the evidence received and can consist of multiple items or one or multiple regions of interest on one item. If large numbers of physically indistinguishable samples are received for testing, a sampling plan may be employed. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be documented in case notes.

9 Procedure

9.1 Instrument Performance Check

- a. Affix the appropriate CRM test block onto the specimen stage with wax, clay or a sample fixture.
- b. Focus the surface region to be tested with the objective lens that will be used for measuring the indentation, typically 5X, 10X or 50X magnification. Adjust the illumination to permit comfortable viewing.
- c. Assure the ocular filars are sharply in focus. Adjust the ocular if necessary, then refocus on the specimen surface.
- d. Select the test load to be applied as indicated by the CRM test block certificate (typically 500 grams), and select the appropriate diamond indenter (Knoop or Vickers) for the test being performed.
- e. Apply the test load. Never attempt to rotate the turret during a test as it will damage the instrument. When the test is complete, the measurement objective moves over the specimen automatically.
- f. Observe the location of the indentation in the field and refocus if necessary. The indentation should be straight, symmetrical, and approximately centered on the ocular filar. If it is not, the test is invalid and must be repeated on a location sufficiently spaced from this indentation. If a second indentation is not straight and symmetrical, check the specimen mounting to assure a flat, perpendicular plane is presented to the indenter and the underside is well supported.
- g. Place both filars at one vertex (end point) of the indentation (long indentation axis for Knoop) side-by-side with no space between them and zero the measurement scale. Adjust the measurement filar to the opposite vertex of the indentation. To mitigate errors from any rotational slack in the dial, always move the filar into position from the same direction of dial rotation. Press the button on the ocular to accept the length value. For Vickers testing, rotate the filar ocular 90 degrees, and measure the second diagonal. The

tester will automatically compute and display the indentation length(s) and the hardness on the digital read-out.

Calculations for determining Knoop or Vickers hardness number from the linear indentation dimensions are performed by the instrument software.

- h. Repeat the test two more times. Adjust the sample position away from any previous indentations using the stage micrometers to avoid any plastically deformed or damaged material.
- i. Ensure that the microhardness readings are consistent with the hardness range reported on the CRM certification sheet. Alternatively, see section 16 References (ASTM E384) to calculate the error E and repeatability R for comparison to the maximum tolerances given. If consistent hardness readings are not obtained, wipe the specimen holder surface and the back of sample with a clean rag to remove any debris from the standard. Ensure that the sample surface is perpendicular to the indenter loading axis. If efforts to alleviate inconsistent readings are unsuccessful, the instrument must be serviced and recalibrated by a certified and licensed service provider that meets the FBI Laboratory Operations Manual (LOM) requirements.
- j. Record the date, operator, test type (Knoop or Vickers), test load, measured values, performance check result and other appropriate information in the instrument log. Record the hardness, load used and test type in the case notes.

9.2 Sample Testing

- a. Samples should have a good quality surface finish so that a clearly defined indentation may be obtained. Polishing is recommended if feasible. Small components may be mounted in polymer to accommodate polishing. (See the CU-Metallurgy SOP *Procedure for Metallographic Examinations*.)
- b. The surface being tested must be perpendicular to the direction of indenter motion and the back surface must be securely supported. Significant deviations from perpendicular are readily detected by a lack of symmetry in the indentation. If this is observed, the sample must be refinished prior to proceeding or the back of the sample mount must be ground flat. For example, a difference in length of greater than ~10% in the two portions of the long diagonal to either side of the short diagonal of a Knoop test requires leveling the sample surface.
- c. For a Vickers indentation, if one half of either diagonal is more than 5% longer than the other, or if all four corners of the indentation do not focus simultaneously, then the sample surface requires leveling before proceeding. If leveling the specimen does not correct the problem, the indenter should be replaced and the performance check repeated prior to proceeding.

- d. Affix the test sample onto the specimen stage with wax, clay or a sample fixture. Assure that the manner of mounting will support the applied test load. Test as in steps 9.1.c through 9.1.h Performance Check, and record the microhardness measurements in the case notes.
- e. If comparative statistics are required, take additional readings in the areas of interest to provide typically 5-10 readings per area.

10 Instrumental Conditions

The instrument must not be subject to vibration when the test is underway. The indenter must be seated securely and free of foreign material. Indenters must be free of nicks or broken edges. Damaged indenters must be replaced, the new indenter reseated securely, and the performance check repeated.

The test load and indenter type used are dependent upon the hardness and shape of the material in the test region.

The Knoop indenter's geometry creates indentations of accurately measurable lengths with light testing loads (1000 g or less). The indenter is very sensitive to surface flatness and perpendicularity to the indenter movement and to surface finish. The penetration depth of a Knoop diamond indent is only about one-thirtieth of the longer diagonal length. This shallow indentation makes it well-suited for measuring the hardness of thin plating layers, case hardening, thin metal and foils, decarburized regions and hard, brittle materials.

Because the indentation area is smaller, the Vickers test is better suited for testing microscopic particles. It can be used when the region of interest is too small to accommodate the elongated Knoop indentation. Microhardness Vickers indentations are limited to loads of 1000 g or less.

11 Decision Criteria

The performance check is acceptable when the microhardness values determined by the instrument on a given CRM fall within the range of values indicated on its certificate or if calculated E and R are within the maximum allowable tolerances (see 9.1.i Instrument Performance Check). If the performance check remains unsuccessful after assuring that the test block and indenter are sound, then the instrument must be serviced and re-calibrated by a certified and licensed service provider that meets the LOM requirements.

Variability in microhardness measurements made on a single sample beyond that demonstrated by the verification CRM test block is significant. If this occurs, the results should not be averaged but should be evaluated individually or as a range for the area tested.

When evaluating whether sets of test readings from different locations on a single specimen or from different specimens are statistically distinguishable, a Student t-test of the local means will be employed.

12 Calculations

12.1 Quantitative Analysis

- a. Microhardness values are automatically calculated by the instrument software. These readings, and their associated measurement uncertainty, may be used to report a range or series of measurements, e.g., the decrease of microhardness from the surface to the interior of a casehardened specimen.
 - i. The hardness number for Knoop (HK) is calculated as:

$$HK = 14,229 \times (P/d^2)$$
 where:
 d is the length of the long diagonal in microns (μm) and
 P is the test load (force) in grams-force (gf).
 - ii. The hardness number for Vickers (HV) is calculated as:

$$HV = 1854.4 \times (P/d^2)$$
 where:
 d is the mean diagonal length in microns (μm) and
 P is the test load (force) in grams-force (gf).
- b. To report averaged microhardness measurements collected over a broader area, collect at least five values. Calculate and report the mean and expanded measurement uncertainty.
 Mean is calculated as: $\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$, where $\sum_{i=1}^n x_i$ is the sum of the measurements, n is the number of measurements and \bar{x} is the mean microhardness value.
- c. General correlation to a macrohardness number, e.g. Rockwell or Brinell, can be read from appropriate conversion charts (see 14 Limitations).

12.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student-t test statistic of the sample means is typically used for the comparison. Two samples are deemed to be “indistinguishable” in the property under consideration if the two samples differ by less than the preselected critical t value (t_{critical}). The critical t value is typically chosen so that a value of $\alpha = 0.05$ can be achieved for the analysis and is determined by the degrees of freedom

associated with the measurements. An $\alpha = 0.05$ means there is a 5.0% chance of incorrectly rejecting a match between two samples when one actually exists.

To perform this test, the means and variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where \bar{x}_a is the average value of the measurements on sample “a”,

$\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample. The variance of the individual measurement values from sample “a” is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

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

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 \left(\frac{1}{n_a} + \frac{1}{n_b} \right)} \right)} \right| > t_{critical}$ for any point of comparison, the samples are concluded to have a

statistically significant difference. If not, the samples are concluded to be indistinguishable.

Typically five or more measurements per sample are used for performing comparisons.

13 Measurement Uncertainty

In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the Chemistry Unit *Procedures for Estimating Measurement Uncertainty*. Each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated. 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 cases, instrument measurement uncertainties will not be reported because they are considered negligible.

Quantitative data are sometimes used for comparative purposes. Expanded uncertainty should not be used for these inter-comparisons because it increases the probability that two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion).

Uncertainties are calculated for the diagonal measurements in microns. The formulas given in section 12 Calculations can be used to convert these measurements into equivalent microhardness values.

14 Limitations

The guidelines for appropriate specimen dimensions stated above are not to be substituted for the sound engineering judgment of a trained operator. Extremely soft materials may flow non-uniformly, preventing accurate microhardness measurement of even large, flat specimens. For loads of 100 g or less, a high-quality metallographic polish is required. The hardness readings obtained are dependent upon load if below 500 g for the Knoop test and below 100 g for the Vickers test and are therefore generally used for comparative purposes only.

All conversion tables of hardness scales, including those in section 16 References, are based on the assumption that the metal tested is homogeneous to a depth several times greater than the indentation. If not, different loads and indenter shapes would penetrate, or encounter the resistance of, metal of varying hardness depending upon the indentation depth.

Conversion tables relating hardness values measured on different scales are only approximate and never mathematically exact due to the difference in cold-working response of a material to the indenter shape and load applied. Nevertheless, conversion is of considerable value when comparing different hardness scales in a general way.

15 Safety

Standard safety precautions, such as wearing protective gloves, should be observed when handling evidentiary materials. Assemblies or components that have electrical or mechanical hazards may require special precautions to disassemble and prepare specimens for microhardness testing.

This instrument SOP has the following specific safety requirements:

- Wear safety glasses when making microhardness indentations
- During specimen preparation, wear personal protective gear and use engineering controls that are appropriate for the task being performed

If additional guidance is required, contact the Laboratory Health and Safety Group.

16 References

ASM Handbook, Volume 8, Mechanical Testing and Evaluation, ASM International, Metals Park, OH, 1992

Chandler, H., ed., *Hardness Testing*, 2nd ed., ASM International, Materials Park, OH, 1999, or latest revision

ASTM Method E384, *Standard Test Method for Microindentation Hardness of Materials*, ASTM International, West Conshohocken, PA, latest revision

ASTM Method E140: *Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness*, ASTM International, West Conshohocken, PA, latest revision

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
3	02/05/2014	Section 4 updated to amend requirements for indentation spacing. Section 9.1 updated to include requirement to test CRM block JH97 when performing verification of the tester. Text amended to require verification measurements to be recorded in the instrument verification log. Section 9.2 spelling correction made. Subsection e deleted. Language in section 10 updated. Section 11 updated to include requirement to include expanded estimate of uncertainty when reporting hardness values. Paragraph from section 10 relocated to section 11. Section 12 includes minor language updates. Formula for sample variance simplified. Typographical error in formula for variance of mean corrected. Formula for calculation of Knoop hardness added. Section 13 has been rewritten to better reflect requirements on measurement uncertainty. Section 16. References updated.
4	12/21/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 15 and is now designated Metal 702. Added personnel to section 2. Added relation between test type and feature size to section 3. Made minor editorial corrections throughout document. Added reference to CU Metallurgy SOPs in sections 4 and 8. Added item to section 5. Removed reference to specific CRM in sections 6 and 8. Revised sampling statement in section 8. Revised section 9 to clarify procedure. Separated quantitative and comparative calculations in 12. Added α and $t_{critical}$ selection process to 12. Updated sections 13 and 15. Updated reference section 16.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader _ Date: 12/20/2018

Chemistry Unit Chief _ Date: 12/20/2018

QA Approval

Quality Manager _ Date: 12/20/2018

Operation of the Instron Model 3382 Universal Testing Machine

1 Introduction

The mechanical properties of metallic materials stem from their elemental composition, the manufacturing processes employed in forming them and post-production exposure to load and environment. Mechanical property measurement can aid in the determination of a material's alloy class, heat treatment, degradation (during service or post-service exposure) and conformance to material specifications.

The Instron Model 3382 Universal Testing Machine can impose tensile, compressive, shear or bending stresses depending on the test fixture configuration and samples used. These types of mechanical tests can be performed under various atmospheric and temperature conditions.

2 Scope

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical analysis in support of metallurgy examinations. Mechanical testing and the response of metals to such tests are the subjects of several college-level courses within typical materials science/engineering curricula. Performance and interpretation of meaningful mechanical testing requires a well-trained, experienced operator. It is essential that an operator has attended such course(s) or has equivalent experience prior to attempting to operate the uniaxial load frame. The following procedure outlines only the basic process for performing uniaxial load (tension, compression, bending or shear) tests using the Instron Universal Testing Machine. Published ASTM standards provide guidelines for a variety of test configurations.

3 Principle

The mechanical test system is designed to apply, read and record a calibrated load along a central load axis. The type of stress imparted to a specimen depends upon the configuration of components (load cell, test fixtures, and specimen) along this load axis, called the load train. The validity and usefulness of the data generated will be determined by the alignment of this load train, so effective design is critical. An electrically powered screw mechanism drives the crosshead during testing. Test speeds are controlled to isolate the effect of strain rate, which can be substantial for some materials. Deformation of the test specimen during and/or after the test can be monitored and recorded.

- a. Tension: The load train is configured to apply a tensile force, usually directly pulling on the ends of a test specimen with a reduced cross-sectional area referred to as a gage section. Force-extension data is monitored using an extensometer, allowing generation of

a stress-strain curve and determination of the modulus of elasticity (E), Poisson's ratio (ν), yield strength (YS), tensile strength (TS), elongation, reduction in area (RA) and the strain hardening characteristics.¹

- b. Compression: The load train is configured to apply a compressive force, usually by directly pushing on the ends of a right circular cylindrical test specimen with a length-to-diameter (l/d) ratio of less than 2.0. Force-extension data is monitored using an extensometer, allowing generation of a stress-strain curve and determination of the bulk elastic modulus, yield strength, and strain hardening characteristics.¹
- c. Bending: The load train is configured to apply a bending force, usually to a flat, rectangular test specimen or beam. Bend ductility tests determine the smallest radius around which a specimen can be bent without cracks being observed on the tensile surface. Bending strength tests determine the modulus of elasticity in bending and the bending strength. For both of these types of testing, a wide variety of test fixtures are available.
- d. Shear: The load train is configured to apply a shear force to a test specimen. For example, tests are often performed on fasteners, oriented to the load as they would be in service, to measure their shear strength. Translational shear tests are performed on sheet materials to characterize shear anisotropy. Torsional (rotational) shear tests are not covered by this procedure. For all types of shear tests, a wide variety of test fixtures are available.
- e. A variety of other mechanical properties, such as stress relaxation and spring constant, can also be determined using the universal testing machine. Guidelines for performing such tests are available from standards-producing organizations like ASTM International.

4 Specimens

Specimens must be designed to mount into the test frame such that the appropriate stress mode is conveyed to the desired portion of the specimen. Entire components may be tested if the load train configuration can accommodate the component alignment. ASTM provides test specimen size and shape specifications for many mechanical tests (see section 16 References).

The orientation and location of specimens taken from parent materials must be recorded, particularly for anisotropic materials. Proper orientation of the stresses with respect to the principal axes of microstructural orientation is an important test consideration.

¹ In situations where breaking stress (or load) is the only desired output, the crosshead displacement can be substituted for the strain signal.

5 Equipment/Materials/Reagents

- a. Instron Model 3382 Universal Testing Machine
- b. Computer with Instron Bluehill 2 software (version 2.0 or later)
- c. Externally calibrated load cell
- d. Extensometer (optional)
- e. Test fixtures (grips) appropriate for the test specimen(s)
- f. Standard weights with calibration certificates from supplier: 1, 2, 5, 10, 20 and 50 lbs and 2, 5, 10 and 20 kg .
- g. Dimensional measuring device(s) such as a micrometer, caliper, traveling microscope, etc.
- h. Environmental chamber (oven, cooling jacket, etc.) (optional)

6 Standards and Controls

Prior to use in casework, the individual load cell to be used will be verified using supplier-certified calibration weights, and the strain gage or crosshead displacement will be verified using a calibrated linear measurement device such as a micrometer, caliper or traveling microscope according to the procedures outlined in section 9 Procedure.

7 Calibration and Verification

The instrument components (load cells and strain gages along with crosshead displacement and speed) are calibrated annually by a certified and licensed service provider that meets the FBI Laboratory Operations Manual (LOM) requirements. The user will verify the performance of the load cell and strain or displacement measuring device each day that the configuration is used to perform tests. The performance check procedures can be found in section 9 Procedure.

8 Sampling

Whole components, or sections from a component, can be affixed into the load train for mechanical testing. These sections are accepted to represent the mechanical properties of that component. The orientation and location of specimens taken from the parent component must be recorded, particularly for anisotropic materials. Proper orientation of the stresses with respect to

the principal axes of microstructural orientation is an important test consideration. Depending on the test specimen size and configuration, one or more test specimens may be machined from a single component.

Typically, all submitted components, or sections from each component, are tested. However, if large numbers of physically indistinguishable samples are received for mechanical property measurement, a sampling plan may be employed for testing. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. Any sampling plan and corresponding procedure used will be recorded in the case notes.

9 Procedure

9.1 Load Cell Performance Check

- a. Select, install and connect the appropriate load cell for the type of test and material being tested. The maximum capacity of the cell must exceed the breaking strength of the test specimen. Attach a fixture to accommodate hanging weights.
- b. Apply power to both the load frame and computer. Allow the system to warm up for 15 minutes to stabilize the load cell before verifying it.
- c. In the software, zero the load channel. (Press key 2 “Balance Load” or, under “Load Cell Setup Dialog”, “Load” tab, click “Balance”.)
- d. Verify the load cell by hanging standard weights from it, it, using at least 1% of the load cell capacity. When applying the weights, never exceed the maximum capacity of the load cell.
- e. Assure that the value displayed by the load channel matches the applied load within the accuracy limit required for the test. If it does not, the load cell and output channel must be serviced and recalibrated by a certified and licensed service provider that meets the LOM requirements.
- f. Record the results of the performance check in the instrument log. Retain the data in the examiner’s case notes.

9.2 Strain Extensometer Performance Check (optional)

- a. Select the appropriate extensometer for the specimen geometry and expected elongation range to be tested. Connect it to the test frame and ensure the correct scale is chosen in the software.

- b. Mount the extensometer on the verification device (e.g., calibrated micrometer) in the same manner as it is normally attached to the test specimen. Position the extensometer at approximately the neutral position (not compressed or extended). View and zero the strain channel in the software.
- c. Dial the full range of the extensometer extension on the micrometer. Adjust the scale factor in the software to assure the electronic output on the strain channel matches this extension.
- d. Verify the extensometer's linearity by returning it to zero in appropriate increments. Use at least ten increments which span the entire extensometer range. Record the micrometer spacing versus extensometer output.
- e. Repeat for compression, if the extensometer is so rated.
- f. Perform a linear regression of the data. The linearity required will depend on the test to be conducted. If the linearity over the entire extensometer range is insufficient, verify similarly over a smaller range and limit subsequent tests to this limited range. Various classes of extensometers are required to meet different levels of accuracy so consult the appropriate ASTM test method. For yield strength determination, a Class II extensometer should be chosen so the fixed error does not exceed 0.0002 in/in, and the variable error does not exceed 0.5% of strain, over the entire range used for subsequent testing.
- g. If the extensometer cannot be adjusted to achieve linearity over the required test interval, the extensometer and output channel must be serviced and recalibrated by a certified and licensed service provider that meets the LOM requirements.
- h. Record the performance check results in the instrument log. Retain the data and regression equation in the case notes.

9.3 Crosshead Displacement Performance Check (optional)

- a. Mount a traveling microscope onto the load frame base and mark a point on the crosshead for observation.
- b. Open the Setup Dialog box and zero the displacement channel.
- c. Move the crosshead to the extreme range expected to be encountered during the test and measure the interval with the traveling microscope.
- d. Assure that the value displayed by the displacement channel matches the interval measured by the traveling microscope within the accuracy limit required for the test.
- e. Verify linearity by returning to zero in appropriate increments (at least ten increments

over the entire interval). Record the distance traveled by the microscope versus the displacement transducer output.

- f. Plot distance vs. output. Calculate the best linear fit to the data using a regression analysis. The degree of linearity required will depend upon the test to be conducted. If the data linearity is insufficient, the transducer must be serviced and recalibrated by a certified and licensed service provider that meets the LOM requirements.
- g. Record the performance check results the examiner's case notes.

9.4 Sample Testing

The guidelines below apply for methods that have been pre-programmed in the software to perform an appropriate test sequence, calculate desired values and output data in a specific format. Newly created methods must be validated in accordance with the LOM.

- a. Select and install the appropriate components and fixtures for the type of test and material being tested, including an environmental chamber if required, and assure the measuring components are verified, as in 9.1 through 9.3 in section 9 Procedure.
- b. Adjust the crosshead position to achieve appropriate grip separation for the test being performed using the crosshead controller.
- c. Adjust crosshead stop and opposite limit stops to the positions required by the test by sliding them up or down.
- d. Select the appropriate display units.
- e. Open (or create) the appropriate method for the test to be performed. Typically a prompted test method will be used.
- f. Measure the required dimensions of the specimen and enter them into the appropriate software data fields.
- g. Mount the specimen in the grips.
- h. (Optional) Attach and align the extensometer on the specimen.
- i. Zero the transducers using the software. Click on the appropriate instrument set-up dialog box and select "Balance", "Zero" or "Reset". Alternatively, click on key 1 "Reset Gauge Length" and key 2 "Balance Load" to zero the values prior to starting each new test run (each new specimen).

- j. From the “Test” tab, click on "Start". The machine will now prompt the user for information, perform the test and display the test progress.
- k. Observe the progression of the test. If using an extensometer, pause the test and remove the extensometer before displacement threatens to surpass its rated extension or compression range. Otherwise press stop when the specimen fractures or when the desired data has been acquired.
- l. Remove the specimen from the grips; then return the crosshead to the starting position and repeat the test for the desired number of specimens.
- m. Save the test data. Print a report of the test results and retain the report in the case notes.

10 Instrumental Conditions

- a. The instrumental operating conditions are determined by the examiner in accordance with the desired test parameters.
- b. Load cells and extensometers are to be used only within their rated capacities. Capacities are listed on certification sheets stored near the instrument.
- c. Grips and fixtures must be rated for any environmental exposure conditions encountered during testing. (e.g., do not use a room temperature-rated strain gage extensometer for elevated temperature tests.)

11 Decision Criteria

Load cells, extensometers, and displacement transducers will be considered within calibrated limits if the performance check results meet the requirements of the applicable ASTM specification(s) or the manufacturer-specified instrument accuracy.

The conclusions derived from this procedure are based on careful interpretation of all of the data gathered during testing. The applicability of the test results depends on the uniformity of the material response during testing, the material homogeneity, and how well the test specimens represent the component, among other factors.

In general, problems with a given test, such as the specimen slipping in the test fixtures, will be reflected in the stress-strain curves generated during data collection. The factors responsible for unexpected curve non-linearities or erratic curves will be investigated and the tests rerun if problems are found.

12 Calculations

- a. For the evaluation of component design strength, mechanics-based design equations can often be used to predict the approximate behavior of materials under complex stresses from the measurements of mechanical behavior under uniaxial tension or compression. See section 16 References for a list of references that are appropriate for this purpose.
- b. The mean value of a property, such as tensile strength, is calculated as: $\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$, where $\sum_{i=1}^n x_i$ is the sum of the individual tensile strength measurements, n is the number of measurements and \bar{x} is the mean tensile strength of the test specimens.

12.1 Quantitative Analysis

- a. A variety of mechanical properties (such as E, YS, TS, and elongation) can be automatically calculated by the instrument software from the load and displacement data. These readings, and their associated measurement uncertainty, may be used to report a range or series of measurements.
- b. To report averaged measurements, collect at least five values. Calculate and report the mean and expanded measurement uncertainty.

The mean value of a property, such as tensile strength, is calculated as: $\bar{x} = \frac{\sum_{i=1}^n x_i}{n}$, where $\sum_{i=1}^n x_i$ is the sum of the measurements, n is the number of measurements and \bar{x} is the mean value of the property.
- c. For the evaluation of component design strength, mechanics-based design equations can often be used to predict the approximate behavior of materials under complex stresses from the measurements of mechanical behavior under uniaxial tension or compression. See section 16 References for a list of references that are appropriate for this purpose.

12.2 Comparative Analysis

- a. Where quantitative data from two specimens are being compared, a pooled, two-tailed Student-t test statistic of the sample means is typically used for the comparison. Two samples are deemed to be “indistinguishable” in the property under consideration if the two samples differ by less than the preselected critical t value ($t_{critical}$). The critical t value is typically chosen so that a value of $\alpha = 0.05$ can be achieved for the analysis and is

determined by the degrees of freedom associated with the measurements. An $\alpha = 0.05$ means that there is a 5.0% chance of incorrectly rejecting a match between two samples when one actually exists.

To perform this test, the sample means and variances are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where \bar{x}_a is the average value of the measurement on

sample “a”, $\sum x_i$ is the sum of the individual measurements and n_a is the number of

measurements made on that sample. The variance of the individual measurement values from sample “a” is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

The mean and variance of the sample “b” data are calculated in an analogous manner.

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 \left(\frac{1}{n_a} + \frac{1}{n_b} \right)} \right)} \right| > t_{critical}$, the samples have a statistically significant difference. If not,

the samples are deemed to be indistinguishable in the property being compared.

- b. When multiple mechanical properties are being evaluated to compare two items, the critical t values for the Student-t test are typically chosen so that an overall (Bonferroni corrected) value of $\alpha = 0.05$ can be achieved for the combined analysis.

13 Measurement Uncertainty

The quantitative data from this procedure is sometimes used for comparative purposes. Expanded 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). In the event that it is necessary to calculate the

expanded uncertainty of a measurement, it will be done in accordance with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*.

14 Limitations

Mechanical testing is, by its nature, destructive testing. Destructive tests on evidence should only be considered if absolutely necessary and must be pre-approved by the contributor. Application of this procedure is limited by the material availability, component geometry and numerous other factors specific to the situation under consideration.

15 Safety

Standard safety precautions, such as wearing protective gloves, should be observed when handling evidentiary materials. Electrical or mechanical hazards may require special precautions.

This instrument SOP has the following specific safety requirements:

- Safety glasses must be worn during load application to prevent possible eye injury.
- High loads on test pieces with large cross-sections may generate projectiles when they fracture. If such loading conditions are anticipated, install a shield between the test piece and the operator.
- Ties and other loose clothing should not be worn when operating this instrument, and long hair should be tied back. If entanglement occurs, a red “EMERGENCY STOP” button can be used to interrupt the instrument power and halt the screw drive motion.
- When testing at elevated temperatures, wear appropriate heat resistant protective gloves and clothing.

16 References

Bluehill 2 – Extended System and Software Introduction and Test Method Development Manual, Application Guide, Manual M18-14443-EN Revision A, Instron Corporation 2004

ASM Handbook, Volume 8, Mechanical Testing and Evaluation, ASM International, Metals Park, OH, 2000

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ASTM Method E9, *Standard Test Methods of Compression Testing of Metallic Materials at Room Temperature*, ASTM International, West Conshohocken, PA, latest revision

ASTM Method A370, *Standard Test Methods and Definitions for Mechanical Testing of Steel Products*, ASTM International, West Conshohocken, PA, latest revision

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date:	History:
2	07/29/2016	Reinstated with editorial revisions in accordance with new formatting guidelines. Added Section 3e covering component property testing. Removed reference to specific calculation tools in Sections 5i and 7.2e. Clarified extensometer calibration in Sections 7.2 and 7.2a. Corrected and simplified equation in Section 12b. Changed Section 13 to reflect updated measurement uncertainty requirements.
3	12/21/2018	Corrected software title in section 1. Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 16 and is now designated Metal 703. Added personnel to section 2. Made minor editorial corrections throughout document. Added common acronyms and ASTM references in section 3. Clarified where to find sample size specifications in section 4. Generalized section 7 and moved specific verification (performance check) procedures to section 9. Added Sampling section 8.. Moved ASTM test recommendation from section 9 to section 2. Reworded sections 11 and 15. Added additional references to section 16.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader _

Date: 12/20/2018

Chemistry Unit Chief _

Date: 12/20/2018

QA Approval

Quality Manager _

Date: 12/20/2018

Metallographic Examinations

1 Introduction

The internal structure of metallic materials contains useful information about composition, processing, quality, service history and mechanical properties of these products. Metallographic examination permits determination of macro- and micro-structural details and can yield information not readily obtainable by other methods.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. Techniques for successful metallographic sample preparation and examination depend highly on the form and composition of the metal under investigation. Appropriate preparation techniques are often developed empirically. This procedure outlines the general concerns to be addressed during all metallographic investigations and cites references for specific preparation techniques that have been found to be successful on particular alloy systems. Sample preparation is typically destructive and requires thoughtful integration into an examination sequence.

3 Principle

Metallographic preparation is a technique used to reveal the internal structure of metals and alloys. These structural details include the size and shape of the crystals (grains) comprising the material, the presence of multiple phases, casting defects (e.g., gas porosity), and material flow lines from plastic deformation, as well as many other characteristics. Large scale features may be visible to the unaided eye while examination of smaller microstructural features may require magnification using appropriate instrumentation.

In the simplest cases, metallographic preparation may require nothing more than buffing a specimen to remove surface contamination and expose the underlying surfaces to the unaided eye. In more complex situations, it may require sample selection, sectioning, mounting, grinding, polishing and etching steps to facilitate high magnification observation of features as small as a few nanometers. Metallographic preparation is also used to produce test specimens for microhardness testing. A wide variety of techniques are available for every step of the preparation process, and many tools are available for the examination process. It is not uncommon for several different metallographic sequences to be used to expose all of the pertinent information contained in one specimen.

4 Specimens

Metallographic techniques can be applied to any size specimen; however, depending on the information desired, specimens may require sectioning to permit examination. The orientation and location of specimens taken from parent material must be recorded. This will allow the orientation of microstructure features to be related back to the original object orientation.

5 Equipment/Materials/Reagents

A list of items commonly used for metallographic examinations follows. Not every item is used for every metallography investigation. The instrumentation and equipment to be employed will depend on the nature of the items to be examined.

- a. Sectioning equipment: band saw, shears, abrasive cutters, drills, cutting lubricants
- b. Mounting equipment: epoxy resin systems, thermosetting or thermoplastic mounting media, mold release agents, specimen support clips, compression mounting press, vacuum desiccator
- c. Grinding equipment: handheld, bench or belt grinders; silicon carbide (SiC), zirconia or diamond abrasive disks or papers; coolants
- d. Polishing equipment: SiC or alumina (Al_2O_3) abrasive paper of various grit sizes, diamond or Al_2O_3 polishing compounds or solutions, colloidal silica, polishing cloths of various naps, coolants, clean compressed air
- e. Chemical etchants for exposing macro or micro features: see ASTM standards E340 and E407 and other references
- f. Electrolytic polishing/etching equipment: electrolyte, power source, connectors, cathodes
- g. Rinse solutions: reagent grade alcohol, deionized water
- h. Ultrasonic cleaner with cleaning solution
- i. Observation equipment: stereomicroscope, metallograph, scanning electron microscope (SEM)

6 Standards and Controls

Certified reference materials (CRMs) or reference photomicrographs may be used to evaluate the identity and distribution of constituent(s).

Calibrated gage blocks are used to verify micrometers and calipers for measuring sufficiently large features. Examinations that report microconstituent size should be performed on a microscope with a calibrated reticle or compared to a micron marker that has been verified against a NIST traceable rule.

7 Sampling

7.1 Sample Selection

Whole components, or sections from a component, can be examined metallographically.

- a. To study the macro-scale properties of a metal, a representative sample of the bulk material must be selected from a region some distance away from free surfaces (e.g., metal plate edges) and other metallurgically heterogeneous regions.
- b. To study individual characteristics associated with production or service, the region(s) of interest may be examined in-situ or sectioned from the evidence if destructive testing is allowed.
- c. If sectioned, the orientation of the section to the original object must be recorded. Refer also to section 10 Decision Criteria.

7.2 Sampling Plan

If large numbers of physically indistinguishable samples are received for testing, a sampling plan may be employed. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be documented in case notes.

8 Procedure

The following steps describe elements of typical sequences for preparing and observing metallographic specimens. These are guidelines only and not all steps may be necessary; however, step 8.1 As-Received Documentation is mandatory. The exact method(s) used, or developed empirically, depend on many variables including alloy system, sample size, surface finish required, desired feature(s) to observe and numerous other factors. The preparation procedure must produce a surface that accurately represents the structure as it existed in the metal before sectioning and/or grinding. Any localized damage/artifacts introduced in the surface must be removed in subsequent preparation steps.

8.1 As-Received Documentation

Photodocument the evidence in the as-received condition (ARC) before beginning any metallographic procedures.

8.2 Sectioning

- a. Record, by sketch or photograph, the intended section to be removed in a manner that documents its orientation to the original object.
- b. Choose a suitable sectioning method to minimize damage to both the retained portion of evidence and the piece to be metallographically examined. Cutting damage depends on the material being sectioned, the cutting device and parameters used, and the amount and type of coolant. Employ a sectioning method that produces minimal surface damage that can be removed during subsequent grinding and polishing. Coolant can be critical because elevated temperature may locally alter the microstructure near the cut.
- c. Section the object and document the sectioning method in case notes. Deburr as needed.

8.3 Mounting

Mounting material must protect and preserve the specimen, preventing physical damage and microstructural alteration. The medium must penetrate into or flow around physical features of the specimen, providing edge retention without pores (air bubbles). The grinding and polishing characteristics of the medium should be similar to those of the specimen, and the mount must resist any solvents, lubricants and etchants used. Consequently, the choice of mounting method will depend on the specimen material and on the desired features to be revealed. For embedded specimens, placing shot or small bearings of material similar in hardness to the specimen around the edge of the mount may assist in maintaining flatness during grinding and polishing.

- a. Prior to mounting, remove any residue from the specimen by immersing in cleaning solution in an ultrasonic cleaner for 2-5 minutes for hard materials or <30 seconds for soft materials. Rinse with deionized water and dry in air, heated air or clean pressurized air. An alcohol rinse after water rinse may be used to aid sample drying.
- b. To mount mechanically, devise a clamping system to securely hold and expose the desired surface for grinding and polishing. For ferromagnetic samples, a strong permanent magnet may be used.
- c. To embed the specimen in castable resin, prepare a mold form over a glass plate and coat with mold release. Place the specimen face down within the mold and support with a clip or other fixture if necessary. Choose a resin system with desired characteristics and prepare according to the system instructions, agitating as gently as possible to minimize air bubble production. Pour carefully to cover the specimen without introducing bubbles.

Readjust specimen alignment if necessary. Vacuum impregnation can remove air bubbles and allow epoxy to enter crevices, assuring complete bonding. Place mold in a vacuum chamber and evacuate, cycling from vacuum to air pressure in one minute intervals until bubbles are removed. Five cycles are usually sufficient to assure impregnation; however, total time under vacuum should not be extended any more than necessary since some resin system constituents evaporate more readily than others. Allow to harden according to resin system instructions before removing from mold.

- d. To compression mount the specimen, coat the interior of the mounting press compression chamber with mold release agent. Place the specimen face down on the piston and support with a clip if necessary. Lower the piston to about double the depth of the sample to accept the mounting material. Pour powder carefully to avoid misaligning the specimen. Lower the piston completely. Secure the piston head and start the operation cycle.

8.4 Grinding

Use successively finer abrasives to create a flat, smooth surface for polishing, etching or immediate examination. Assure that each step in the sequence removes material to a depth sufficient to eliminate scratches and underlying deformed layer created by the previous step. Several typical sequences follow:

- a. To reveal macro features, such as weld heat-affected zones, on large components that have not been sectioned, grind off the surface scale or cladding with a hand grinder and buff with a rotating tool. Proceed with etching and examination.
- b. To reveal macro features, such as obliterated serial numbers, on small components or sections of components, grind the surface area using coolant if possible (e.g., on a metallographic belt grinder). Perform the minimum amount of grinding required to produce a flat, smooth surface since number restorations typically depend upon the layer of disturbed material created when the number was imparted to the material. If too much material is removed, the characters cannot be restored. Proceed with polishing, etching and examination.
- c. To reveal microstructural features such as grain size or phase distribution, grind the specimen on a wet belt grinder to create a flat surface. Sequentially grind through a series of Al_2O_3 or SiC abrasive papers of 240, 320, 400, and 600 grit size. Starting with the coarsest grit (e.g., 240), grind in a direction perpendicular to the scratches imparted by the belt grinding operation. Grinding should continue until the scratches from the previous step are no longer visible. Next, rotate the sample 90° to the previous grinding direction and remove the scratches left by the prior grinding step using the next finer grit size in the series. Care should be taken to maintain a flat surface. If beveling occurs and is objectionable, the sample should be reground to remove the bevel. An automatic system can also be used for grinding and polishing.

8.5 Mechanical Polishing

Use successively finer abrasives on polishing paper or cloth to create a smooth surface for etching or immediate examination. Assure that each step in the sequence removes material to a depth sufficient to eliminate scratches and the deformed layer created by the previous step. Several typical sequences follow:

- a. For revealing macro scale features, such as serial number restorations, on small components or sections of components, polish the surface of the area of interest by either applying part to abrasive or abrasive to part. Rinse polishing compound off with suitable solvent (e.g., water or alcohol) and dry with forced air. Proceed with etching and/or examination.
- b. For revealing microstructural features such as grain size or phase distribution, sequentially polish through a series of abrasive polishing compounds typically ending with a final polish with 0.05 micron Al_2O_3 . These polishing agents are typically applied with lubricant to a dedicated polishing cloth mounted on a rotating wheel. Remove scratches from each previous step using one of the following techniques:
 - i. Turn the sample so that the scratches from the previous grit size are perpendicular to the wheel motion and polish until the scratches from the previous grit size are removed.
 - ii. Rotate the sample counter to the polishing wheel direction until all scratches appear to be of uniform depth.
- c. Rinse the polishing compound off with suitable solvent (e.g., water or alcohol) and dry with forced air between each step and when polishing is complete. It may be necessary to ultrasonically clean the sample between each polishing step for about one minute to prevent cross-contamination. Proceed with etching and/or examination.
- d. Should an initial polishing attempt prove inadequate, revert to the previous particle size and repeat the polishing procedure. If necessary, go back to the final grinding step (e.g., to 600 grit) then repeat the polishing procedure. Change polishing cloths if any cross-contamination occurs.
- e. Chemical/mechanical polishing: Final polishing to produce a totally scratch-free surface is rarely necessary for forensic examinations; however, this may be accomplished by lapping on a polishing wheel or on a vibratory polishing system using an appropriate polishing solution. Acidic polishing solutions are typically used for ferrous materials and basic solutions (such as colloidal silica) for non-ferrous material.

8.6 Electropolishing

Electrolytic polishing is useful for metallographic preparation of metals that are difficult to polish by mechanical methods detailed in 8.2-8.5 (e.g., Mg, Zr). Electropolishing is not typically appropriate for preparation of multiphase materials. This method can be used to eliminate mechanical deformation induced by conventional mechanical polishing or directly after the final grinding step to replace mechanical polishing. See section 15 References for optimal current/voltage relations and electrolyte solutions to use for the particular metal under examination. Similar information can often be found in literature from metallographic supply companies.

- a. Establish a DC variable voltage current source, electrical circuit and electrolytic cell. This requires a DC power supply, wires with alligator clips or other electrical connectors and an electrolyte container. Typically, commercially manufactured electrolytic equipment is used.
- b. Prepare specimen surface to a 600 grit or finer finish.
- c. Provide stirring or air agitation if necessary to prevent localized heating of the surface (e.g., a magnetic stir bar in the electrolyte solution over a stir plate).
- d. Connect electrodes so that the specimen is the anode (connected to the “ + ” side of the DC power source) and the cathode is a metal component connected to the “ - ” side of the power source, and both are immersed in the electrolyte.
- e. Adjust the voltage to achieve adequate current density. “Adequate” will be determined empirically for each metal because the current density generated depends on sample size.
- f. Should an initial polishing attempt prove inadequate, regrind the specimen to 600 grit or finer and repeat the electropolishing procedure using a different applied voltage.
- g. Should artifacts (such as furrowing or dimpling due to gas evolution) be created, regrind the specimen and repeat the electropolishing procedure with additional solution agitation.
- h. Complete any necessary photodocumentation of the as-polished specimen surface prior to etching.

8.7 Chemical Etching

Etching proceeds by selective dissolution according to the electrochemical characteristics of the microstructural constituents. Suggested solutions for chemical etching are given in ASTM E 340 or E 407 and in 15 References. Be sure to follow safety and hazardous waste disposal guidelines for dealing with the chemicals used. A typical chemical etching sequence follows:

- a. Assure that the specimen surface has been appropriately prepared. Macroetching may only require grinding to a 600 grit finish then buffing. Microetching usually requires a finer finish (e.g., 0.05 micron).
- b. Swab with, or immerse specimen in, the etchant until sufficient contrast is produced. Rinse with appropriate solvent to halt the reaction, typically with deionized or distilled water. Multiple steps of etching and rinsing may be required.
- c. Should an initial etching attempt prove inadequate, regrind the specimen to remove the etched surface (e.g., to 1 micron abrasive for microetched materials or 600 grit for macroetched materials). Sometimes samples etch more readily immediately after polishing. Etching can also happen preferentially depending on whether the sample is immersed or swabbed. If two metals are present it may be necessary to mask one so it will not etch to the exclusion of the other.

8.8 Electrolytic Etching

An electrical potential is applied to the specimen using an electrical circuit to promote removal of metal ions from the specimen surface. Refer to ASTM E 340 or E 407 or other appropriate guidelines (see 15 References) for suggestions of optimal current/voltage relations and electrolytes for the particular metal under examination. Follow safety and hazardous waste disposal guidelines for dealing with the chemicals used. The basic steps are identical to those in section 8.6 Electropolishing, but different parameters are used to effect etching rather than polishing. Document the parameters used in the case notes.

8.9 Observation

Identification of microstructural features and their significance requires metallurgical knowledge of the metal being examined. For unknown materials, reference micrographs are provided in ASM Handbook Vol. 9, *Metallography and Microstructures* as well as in many additional resources. Identify and use the appropriate ASTM procedure for quantifying microstructural features (such as grain size or inclusion density) if required (see section 15 References).

Photodocument the as-polished and/or etched macro and/or micro-structures as needed for case notes.

9 Instrumental Conditions

The cooling reservoirs of wet cutting and grinding equipment must be maintained to assure adequate coolant levels and cutting fluid cleanliness according to procedures found in the appropriate instrument manuals.

Microscopes and cameras must be maintained to prevent artifacts from being introduced during observation and photodocumentation. Annual maintenance is usually sufficient; however, if artifacts are observed, the instrument(s) should be cleaned immediately.

10 Decision Criteria

The conclusions derived from this procedure are based on careful interpretation of all of the data gathered from examination. In general, it is possible to identify the phases present in a material by careful comparison with published microstructures. When this is not possible, additional testing methods such as x-ray diffraction, transmission electron microscopy or SEM/EDS may be required for phase identification.

The degree of certainty to be applied to test results will depend on the material homogeneity and how well the prepared specimen represents the sample, among other factors. Careful consideration of the specimen geometry and its other physical features generally allows an experienced examiner to select appropriate representative samples for metallographic purposes. Where doubt exists, additional sampling should be considered.

Accurate microstructure interpretation requires a well-trained, experienced metallurgist. It is essential that the analyst has received training in microstructural development as it is influenced by solidification, phase transformations, heat treatment, and plastic deformation. The analyst must also recognize production defects and corrosion products and must understand the effects of microstructural constituents and their distribution on mechanical properties. Training may consist of attending topical courses and/or possessing equivalent experience prior to attempting to interpret microstructural information.

11 Calculations

Calculations regarding microstructures are typically limited to estimations of phase distribution, case depth or grain size. When required, an appropriate method will be validated, applied, and documented in the case notes.

12 Measurement Uncertainty

Normally, metallography is non-quantitative in nature. However, quantitative analysis of a set of metallurgical features is sometimes possible and could potentially be used in a comparison of one metal sample to another or to a published specification, or to establish its approximate mechanical properties and/or processing history. Should it be required, the measurement uncertainty associated with such a procedure will be established in accordance with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*.

13 Limitations

Metallographic preparation is, by nature, destructive. Destructive tests on evidence should only be considered if absolutely necessary and must be pre-approved by the contributor and principal investigator.

There are numerous potential sources for artifact generation when preparing metallographic specimens. These include overheating during sectioning, embedded abrasive material, smearing of soft materials (e.g., pure Al or Cu), and pitting or preferential attack during polishing and/or etching. The examiner analyzing the revealed microstructure must also be cognizant of the possibility of stress or thermally-induced phase transformations when evaluating the material.

Whenever possible, grinding operations should be performed with adequate coolant such as water, glycerin or lubricating oil to avoid potential false microstructure changes due to localized heating.

14 Safety

Standard safety precautions, such as wearing protective gloves, and eyewear, must be observed when handling evidentiary materials of a hazardous nature. In addition, wear an acid-resistant apron and chemical splash goggles when polishing and/or etching with hazardous chemicals. Wear impact-resistant eye protection during sectioning and rough grinding operations to protect from flying debris. When consulting Safety Data Sheets (SDS's) for the components of an etchant or electrolytic solution, always follow the safety guidelines for the mixture's most hazardous component. Review all pertinent SDS's prior to using any potentially hazardous chemicals.

Dispose of hazardous chemicals according to the guidelines established by the FBI Laboratory, consulting the Chemistry Unit Safety Officer for proper procedures.

Any materials that have contacted hydrofluoric acid (HF) containing solutions must be thoroughly rinsed before handling or observing under glass optics because of the potential for HF-containing liquid and fumes to attack bones (decalcify) and etch glass.

To apply the above procedures to materials with significant concentrations of hazardous metals such as Pb, Be, Te, Ag, Hg and Cd, rinses and etchants contaminated with these metals must be collected and disposed of as hazardous waste. Do not cut or grind these metals on machines with closed-loop cooling systems. Collect chips generated from sawing or grinding and any grinding belts, abrasive papers, polishing cloths, and other solid materials embedded with metal debris, and dispose of them as hazardous waste. When using automatic polishing equipment, run the drain line into a collection vessel. Transfer waste from this collection to the proper container in the 90-day waste accumulation site established by the Safety Officer.

15 References

ASM Handbook, Vol. 9 – *Metallography and Microstructures*, ASM International, USA, current revision

Vander Voort, G. F., *Metallography: Principles and Practice*, McGraw-Hill Publishers 1984

ASTM Annual Book of Standards, Vol. 03.01 (current revision), multiple standards for metallographic preparation and analysis, ASTM International, West Conshohocken, PA

ASTM Method E3 (current revision), *Standard Guide for Preparation of Metallographic Specimens*, ASTM International, West Conshohocken, PA

ASTM Method E7 (current revision), *Terminology Relating to Metallography*, ASTM International, West Conshohocken, PA

ASTM Method E45 (current revision), *Standard Test Method for Determining the Inclusion Content of Steel*, ASTM International, West Conshohocken, PA

ASTM Method E112 (current revision), *Standard Test Methods for Determining Average Grain Size*, ASTM International, West Conshohocken, PA

ASTM Method E340 (current revision), *Test Method for Macroetching Metals and Alloys*, ASTM International, West Conshohocken, PA

ASTM Method E407 (current revision), *Practice for Microetching Metals and Alloys*, ASTM International, West Conshohocken, PA

ASTM Method E883 (current revision), *Guide for Reflected-Light Photomicrography*, ASTM International, West Conshohocken, PA

ASTM Method E930 (current revision), *Standard Test Method for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)*, ASTM International, West Conshohocken, PA

ASTM Method E1077 (current revision), *Standard Test Methods for Estimating the Depth of Decarburization of Steel Specimens*, ASTM International, West Conshohocken, PA

ASTM Method E1181 (current revision), *Standard Test Methods for Characterizing Duplex Grain Sizes*, ASTM International, West Conshohocken, PA

ASTM Method E1351 (current revision), *Standard Practice for Production and Evaluation of Field Metallographic Replicas*, ASTM International, West Conshohocken, PA

ASTM Method E1558 (current revision), *Guide for Electrolytic Polishing of Metallographic Specimens*, ASTM International, West Conshohocken, PA

ASTM Method E2014 (current revision), *Standard Guide on Metallographic Laboratory Safety*, ASTM International, West Conshohocken, PA

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, current revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, current revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, current revision

Rev. #	Issue Date	History
2	04/24/2014	Updated required equipment in section 5. Minor grammatical changes made to text in sections 3, 5 and 9.1 and 9.2. Section 13 has been rewritten to reflect the current measurement uncertainty requirements. Equipment referenced in section 15 has been updated. References have been updated in Section 16.
3	12/21/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 17 and is now designated Metal 800. Added personnel to section 2. Made minor editorial corrections throughout document. Revised throughout to remove specific brand names/equipment. Incorporated section 7 into section 6 and renumbered subsequent sections. Added requirement for sampling plan retention in section 7. Updated optional procedures in section 8. Augmented section 14. Added additional references to section 15.

Redacted - Signatures on File

Approval

Metallurgy Technical Leader .

Date: 12/20/2018

Chemistry Unit Chief .

Date: 12/20/2018

QA Approval

Quality Manager .

Date: 12/20/2018

General Approach to Report Writing in Metallurgy

1 Introduction

The report issued by a metallurgy examiner is a summary of analytical findings and an explanation of the interpretations of these findings. As a result of the variety of requests and evidence received, this standard operating procedure is only a general guideline for report writing. Its intent is to assist examiners in formulating reports that are both factual and contain enough explanatory information to allow rational evaluation of the conclusions drawn and the limitations of the examinations performed. Further, the wording must be approved by an authorized reviewer during the technical review process.

2 Scope

This document applies to Chemistry Unit caseworking personnel who write *Laboratory Reports* that convey the results of metallurgy examinations and the interpretations and opinions that are based on these examinations.

3 Equipment/Materials/Reagents

Not applicable.

4 Standards and Controls

Not applicable.

5 Sampling or Sample Selection

Not applicable.

6 Procedure

Metallurgy *Laboratory Reports* will contain the administrative requirements set forth in the FBI Laboratory Operating Manual (LOM). Metallurgy *Laboratory Reports* will also comply with the language limitations detailed in the Chemistry Unit (CU), FBI Approved Standards for Scientific Testimony and Report Language for Metallurgy and the *Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline (Metallurgy ULTR)*

6.1 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. Any pertinent limitations of the evidence that prevent forming conclusions related to the incoming request may be included in this section or may be deferred to the Methodology, Interpretation and Discussion section of the report if it is deemed to improve the clarity of the report.

6.2 For a comparative examination, the Results of Examinations section will indicate the general nature of any conclusion (e.g., ‘fracture fit’, ‘inclusion’, ‘exclusion’, or ‘inconclusive’), the characteristics on which the conclusion is based, and an indication of the significance of the conclusion. An explanation of the significance of an ‘inclusion’ will describe 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. 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.”)

6.3 Any quantitative result reported in the Results of Examinations will include an estimate of the uncertainty for the value that will be reported at 99.7% confidence or higher. The (estimated) confidence level will also be reported.

6.4 In addition to the Results of Examinations section, each metallurgy report will include a Methodology, Interpretation and Discussion section that specifies the instruments and procedures that were employed. This section may also provide additional information intended to help the reader understand the results of the examinations. To this end, the section may include:

- information on both the strengths and the limitations of the examinations performed
- 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.

6.5 The Remarks section will contain a statement that incorporates the *Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline* into the *Laboratory Report* by reference. In addition, the following will be included in the Remarks section when applicable and appropriate and when this information may assist the reader of the report:

- a listing of evidence collected but not examined in metallurgy
- an explanation of how to properly collect, mark and preserve metallurgical evidence in the future
- any other information that will assist the reader that does not belong in another section of the report.

6.6 The language used to convey this information should be accurate, clear, unambiguous and objective. The composition will be decided upon by the metallurgy examiner and agreed to by the technical reviewer before the *Laboratory Report* is issued. Documentation of this agreement is maintained in the case record (e.g., Case Record Report, Case Record Reviews, Technical: “Completed”, or initials on a hardcopy of expedited results.) Copies of metallurgy *Laboratory Reports* will be maintained in a central, designated area in order to facilitate consistency in language over time and among examiners.

6.7 Exemplar reports illustrating the basic document layout to be employed in writing metallurgy *Laboratory Reports* are contained in Appendix 1.

7 Calculations

Not applicable.

8 Measurement Uncertainty

Not applicable

9 Limitations

Not every metallurgy examination scenario can be anticipated. This document provides guidelines for the structure of metallurgy *Laboratory Reports*, but is not intended to limit the report author to any specific vocabulary, scope or amount of detail.

10 Safety

Not applicable.

11 References

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Approved Standards for Scientific Testimony and Report Language for Metallurgy, Metallurgy Manual Metal 901, Chemistry Unit, latest revision

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

Rev. #	Issue Date	History
0	06/08/2010	New document.
1	01/29/2019	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 22 and is now designated Metal 900. Changed title and removed references to metallurgy “discipline” throughout. Revised Section 2 Scope. Removed specific titles of LOM documents. Revised Section 6 Procedure by incorporating relevant documents by reference and adding and revising language to correspond to the relevant documents. Clarified documentation of technical reviewer agreement with report language. Also added documents to Section 11 References. Revised examples in Appendix 1.
2	02/18/2020	Changed the description of the technical reviewer qualification to “authorized” to conform with LOM language in sections 1 and 6.6. In section 1, changed “inferences” to “conclusions” to avoid confusion with sampling requirements. Added descriptive headings, increased specificity of Example 2 results and added Example 4 to the Appendix.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader:

Date: 02/13/2020

Chemistry Unit Chief:

Date: 02/13/2020

Appendix 1: Examples of the Types of Content to be included in a Metallurgy Report

In the examples below, comments regarding the report language are enclosed in { } and are not part of the sample text.

Example 1: Comparison

Results of Examinations:

Items 1, 7 and 10 are sections of red-insulated wire. Each has a single-strand copper conductor. The diameters of the wire sections compare favorably with AWG 22 wire.

The Item 2-5 assembly contains one length of red-insulated, two-conductor cord. Each conductor is a single strand of copper that compares favorably to AWG 22 wire.

The Item 32 assembly contains two lengths of red-insulated, two-conductor cord. Each conductor is a single strand of copper that compares favorably to AWG 22 wire.

Based on metallurgical examinations of the insulated copper wires in Items 1 and 7, it was concluded that these were manufactured with the same fabrication tooling (extrusion die) as one of the wires attached to an electrical plug from Item 32. The observed characteristics indicate that the Item 1 and 7 wires were either cut from the same item as one wire in Item 32 or from some other length of wire produced using the same extrusion die. [Inclusion] {This statement explains that Items 1 and 7 could also have originated from another length of wire that did not include Item 32.} Due to large production runs, a substantial amount of wire would be expected to bear the same observable characteristics. {This paragraph explains the potential source population.}

Metallurgical examinations of the Item 10 insulated copper wire determined it to have the same manufacturer's information printed on its surfaces as the section from Item 2-5 and one of the sections from Item 32. The observed characteristics indicate that Item 10, the section from Item 2-5 and the section from Item 32 were either cut from the same length of wire or from similar lengths of wire bearing the same manufacturer's information. [Inclusion]

Further, the contents of the printed markings on the wire from Item 2-5, Item 10 and one wire from Item 32 differ slightly from those printed on the wires in Items 1, 7 and the other wire from Item 32. This means that at least two distinguishable sources of physically similar, insulated wires are represented within the items. [Exclusion] {The two types of wire differ from each other.}

Methodology, Interpretation and Discussion:

The items were examined for association/origin by visual inspections, dimensional measurements of the wire insulation and the internal conductors, x-ray fluorescence spectrometry (XRF) of the metal conductors and microscopic comparisons of the fabrication marks on the wire

insulation.

{ Include a list of Metallurgy SOPs used. }

Because wire producers commonly have many extrusion dies of the same size, the presence of extrusion marks on the wire insulation can be used to distinguish wire produced with a particular die from wire of the same size, color and type produced using a different die.

Modern fabrication tooling can produce an unknown but very large quantity of insulated wire having identical fabrication marks and printed manufacturer's information. Although many manufacturers worldwide make a myriad of types, sizes and colors of wire, large concentrations of a particular product in a limited geographic area can and do sometimes occur. Consequently, it is extremely difficult to estimate the number of individuals who might possess seemingly identical products within a limited geographic area at any given time. { This paragraph contains details regarding the limited significance of the *Inclusions* presented in the Results section. }

Example 2: Identification and Comparison

Results of Examinations:

The Item 1-1, 1-2 and 1-3 pipe fragments and the largest metal fragment from Item 6-1 were analyzed and determined to be fabricated from galvanized, plain carbon steel. Based on the similarities in the compositions and physical characteristics of the four fragments, metallurgy examinations determined that they either all originated from the same length of pipe or from multiple pipes fabricated from identical grades of steel having highly similar physical dimensions, chemistries, and plating layers. [*Inclusion*] { This paragraph includes the bases for the *Inclusion* conclusion. }

In addition to the fragment analyzed, Item 6-1 contains four additional, smaller fragments. These fragments appear to have originated from a source such as an endcap based upon the visual and microscopic visual examinations of them. No compositional analysis of these fragments was conducted.

Methodology, Interpretation and Discussion:

Items 1-1, 1-2 and 1-3 and the largest fragment from Item 6-1 were analyzed for association/origin with a binocular microscope, physical examinations, x-ray fluorescence spectrometry (XRF) and spark-discharge-in-argon optical emission spectrometry (SDAR-OES). { Include a list of Metallurgy SOPs used. }

The binocular microscopy, XRF and mechanical measurements served primarily as evaluations of the class characteristics of the fragments to include: the general alloy class, the type of surface plating, wall thickness, shape, and surface finish. Because of the damaged nature of the fragments, such evaluations are primarily qualitative in character. { This paragraph explains limitation of the examinations. }

Sections were cut from Items 1-1, 1-2, 1-3, and the largest fragment from Item 6, and the galvanized coating was ground off of one surface of each section. Quantitative compositional comparisons of the sections were performed using SDAR-OES. Commonly, carbon steel samples contain fifteen or more elements above the limits of detection of the instrument. Statistical comparisons of the measured compositions of two steel samples are made using a Bonferroni corrected, Student's t-test with a null hypothesis that the steels have identical compositions. Using this scheme, the comparison of two steel samples that actually have identical compositions will result in a failure to associate them approximately 5% of the time.

The likelihood of incorrectly associating two steel samples that actually have different elemental compositions is more complicated to calculate. Thousands of different compositional specifications exist which govern the production of the many different types of steel. Moreover, there is considerable compositional variation between individual heats of steel produced to the same specification. Consequently, the number of potentially distinguishable compositions of steel that could be encountered is very large but it is not unlimited. Logically, it follows that there is a possibility of encountering steel components that cannot be distinguished from each other but which are from different manufactured sources.

Because a single heat of steel commonly exceeds one hundred tons in weight, many thousands of pipes having indistinguishable compositions can potentially be produced from a single heat. Also, large concentrations of a particular product in a limited geographic area can and do sometimes occur. Consequently, it is extremely difficult to estimate the number of individuals who might possess seemingly identical products within a limited geographic area at any given time. {The above paragraphs explain the limitations of the significance of the *Inclusion* conclusion.}

Example 3: Identification of Material

Results of Examinations:

Based on microstructural evaluations of the Item 1 copper disc, it was concluded that the cone is unlikely to have been fabricated from an oxygen free grade of copper. [*Exclusion*] This conclusion is supported by compositional analysis of microscopic inclusions. The Item 1 material contains significantly more oxygen-bearing microscopic inclusions than would be expected in oxygen free copper.

Methodology, Interpretation and Discussion:

The microstructure of the disc was evaluated by cutting and polishing copper sections to a less than 0.1 micron surface roughness using metallographic diamond polishing compounds containing known particle sizes. Some of the inclusions contained in the copper from the Item 1 specimen were analyzed by scanning electron microscopy with energy dispersive spectrometry (SEM/EDS) to determine their elemental compositions.
{Include a list of Metallurgy SOPs used.}

Oxygen is not highly soluble in copper and its presence can be seen in the form of inclusions having a different color and reflectivity under microscopic examination than the polished metal surface surrounding them. Comparison of such polished surfaces with the published images of polished copper available in standard references was used to investigate the types of inclusions present in the Item 1 sample. Based on such comparisons, the sample from Item 1 contains numerous inclusions which closely resemble the oxide inclusions known to be present in oxygen bearing grades of copper. Oxygen free grades of copper commonly contain less than 10 parts per million oxygen by weight. As a consequence of this, oxide particles are not readily observed in oxygen free grades of copper.

Several chemically distinct types of inclusions can occur in copper. These may include sulfides, phosphides, and oxides, as well as other types. Since the chemistry of the inclusions affects their shapes and colors, it is usually possible to distinguish among different inclusion types based on visual microscopic examinations. The elemental composition of these inclusions can be evaluated by SEM/EDS.

Some extreme service conditions are capable of changing the microstructural characteristics of materials. For example, heating an oxygen free grade of copper at high temperature in air for a very prolonged period would permit the diffusion of oxygen into the sample. However, it is highly probable this would result in a much higher density of oxide particles near the surface than in the sample core unless extensive, additional thermal and mechanical processing of the sample was subsequently undertaken. No such gradient of oxide particles was observed in Item 1. {This paragraph describes limitations of the examination.}

Example 4: Lamp Examination

Results of Examination:

Metallurgical examinations of the damaged Item 3 headlight lamp determined that some of its observable characteristics almost certainly resulted from the lamp sustaining a severe impact while its low beam filament was illuminated. Notably, the filament of Item 3 was extensively stretched out.

The remains of the Item 1, Item 2 and Item 4 lamps were too limited to determine if any of these lamps were incandescent at the time of the reported collision. {This paragraph explicitly states the reason the examination of these items was *Inconclusive*.}

Methodology, Interpretation and Discussion:

Lamp examinations consist of evaluations of the characteristics that distinguish whether a lamp was exposed to impact and/or air while at either high temperature or at ambient temperature. The lamps in this case were examined visually and with a binocular microscope for physical changes which would indicate their operating condition at the time of the reported collision. The Item 3 lamp was also checked with an ohmmeter to verify its electrical continuity.

The forensic examination of automotive lamps is based on the knowledge that the tungsten metal used to produce lamp filaments has dramatically different chemical and physical properties at room temperature and at the elevated temperatures that characterize an incandescent filament. Tungsten is typically brittle at room temperature but capable of highly ductile behavior at elevated temperatures. Consequently, extensive stretching of a lamp filament usually indicates that the filament was deformed while at elevated temperature.

It is frequently not possible for these examinations to determine when a particular lamp became damaged. In some instances, the observed changes in a lamp could have occurred in an incident prior to that under investigation since some damaged lamps can continue to function.

When slight apparent filament deformation is present, it is not always possible to unambiguously determine its cause. For example, unused (new) filaments can exhibit slight ductility when cold. In addition, automotive lamps are occasionally manufactured with filaments that have a slight bow in them. Similarly, long, fine gauge filaments may stretch downward somewhat during prolonged normal operation due to the effects of gravity. This phenomenon is known as "age sag". Despite the possible occurrence of such characteristics, it is usually possible to identify the damage induced by a severe impact on an incandescent filament.

The stretching of the Item 3 filament is not typical of deformation resulting from age sag, cold deformation of a new filament or with as-manufactured characteristics. Rather, it is more typical of the deformation that occurs during a severe impact while a filament is at elevated temperature.

Example 4: Characterization of Product

Results of Examination:

Metallurgy examinations determined that the Item 1 collection contains ten coins marked "silver". Compositional analysis determined that the exterior of each coin is unalloyed silver. One of the coins, Item 1-1, was sectioned and the composition was analyzed through the thickness of the coin. The composition through the entire thickness of the sectioned coin is unalloyed silver."

{ Avoid attributing characteristics determined by analysis to items (or locations on items) that were not analyzed. Do not express results that may be misconstrued as describing ALL of the pieces, such as, "The Item 1 collection contains silver coins." }

Chemistry Unit (CU)

FBI Approved Standards for Scientific Testimony and Report Language for Metallurgy

1 Purpose

This document provides examples of the scientifically-supported conclusions and opinions approved for reporting examination conclusions and offering expert opinion statements during testimony by metallurgy examiners within the Chemistry Unit (CU). Metallurgy is an engineering field of very broad scope. Accordingly, these examples are not intended to be all inclusive. When conclusions can be drawn from sound engineering practices and substantiated through the technical review process, topics other than those listed may potentially be discussed in court testimony. Specific limitations may also be imposed by the judge or locality in which a testimony is provided. Such restrictions may require modification of the following examples. Further, these examples are not intended to serve as precedent for other forensic laboratories and do not imply that statements by other forensic laboratories are incorrect, indefensible, or erroneous.

2 Scope

This document applies to CU employees who prepare an *FBI Laboratory Report* (7-1 or 7-1 LIMS) and/or provide expert testimony in metallurgy. This document does not apply to CU employees who provide fact witness testimony.

3 Responsibilities

3.1 The examiner will ensure that a *Laboratory Report* is consistent with the approved statements contained within this document when applicable.

3.2 The examiner will ensure that his/her testimony related to metallurgy examinations is consistent with the approved statements contained within this document when applicable.

3.3 The Technical and Administrative reviewers will ensure compliance of the metallurgy *Laboratory Report* with the statements contained within this document.

3.4 The authorized evaluator who reviews testimony in accordance with the FBI Laboratory Operations Manual (LOM), Practices for Testimony Related Activities will assess if metallurgy testimony complies with the statements contained within this document and the Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline (Metallurgy ULTR).

4 Statements Approved for FBI Metallurgy Examination Testimony and/or Laboratory Reports

For more detailed guidance on writing metallurgy reports, see the CU, General Approach to Report Writing in the Metallurgy standard operating procedure.

- An examiner may report and/or state the elemental composition of an item. For example, an examiner may report that a piece of jewelry is a gold-copper alloy. If the results are quantitative in nature, the estimated measurement uncertainty will also be reported.
- An examiner may report and/or state the alloy an object is comprised of, based on an elemental analysis of the item and comparison with published standards. For example, an examiner may state a drill bit is comprised of W2 tool steel and not M3 tool steel.
- An examiner may report and/or state the mechanical properties of an item, if supported by appropriate testing, and may opine as to whether these results are consistent with a particular published or contractual specification.
- An examiner may report and/or state the production process used to manufacture an item when the physical characteristics permit such an inference. For example, an examiner may indicate that fine wire is produced using a drawing process.
- An examiner may identify the company that is assigned a particular trademark, barcode, Underwriters Laboratory (UL) listing code, etc., by reference to an appropriate source (e.g., a database). The examiner will identify the source of the information.
- An examiner may report the physical mechanism(s) responsible for a particular failure (e.g., fatigue, creep, hydrogen embrittlement).
- An examiner may report the exclusion of a particular failure mechanism when supported by the physical features of the evidence, even if the actual failure mechanism remains undetermined.
- An examiner may report and/or state an estimate of the forces required to produce a given fracture. The examiner may also discuss the basis of the estimate.
- An examiner may report and/or state other factors which contributed to the failure such as inclusions, improperly machined notches, corrosive environment, etc.
- An examiner may report and/or state a determination that automotive lamps were operating or were off when subjected to an impact if the physical changes in the lamp are sufficient to support such a conclusion.
- If the physical characteristics are inconclusive, the examiner may report and/or state the operating condition of an automotive lamp could not be determined.
- An examiner may report and/or opine a *Fracture Fit* when portions of two or more items

physically fit together and their chemical and physical characteristics show sufficient correspondence to indicate that they once comprised a single object.

- An examiner may report and/or opine an *Inclusion* conclusion that items could have originated either from the same metallurgy source or process or from another source or process that is substantially similar to the examined items in all observed characteristics. For the large majority of such cases, these comparisons are limited to the class characteristics of such items and, as such, are not individualizing.
- The examiner will report and/or state a description of the relative strength of an *Inclusion* conclusion based on reference to relevant data. For example, when an *Inclusion* is made to a broad general population of items or materials (such as mass-produced items), the chance of finding coincidentally indistinguishable materials may be high (e.g., two Coca Cola cans). As the population of items or materials narrows, the chance of finding coincidentally indistinguishable materials decreases. .
- When relevant data for evaluating the relative strength of agreement between two or more items, materials or processes is unknown, the examiner must explain this limitation in reports and/or testimony.
- An examiner may report and/or opine an *Exclusion* determination that two items or materials could not have originated from the same source or process due to substantial dissimilarities in their observed characteristics.
- An examiner may report and/or opine an *Inconclusive* conclusion that no determination could be reached as to whether two or more items or materials could have originated from the same source or process based on insufficient quality and/or quantity of corresponding information. The reason(s) for the *Inconclusive* conclusion must be included in the report and testimony.
- The examiner may report and/or state the limitations of his/her examinations and expert opinion.

5 Statements Not Approved For FBI Metallurgy Testimony and/or Laboratory Reports

- An examiner may not report or state an opinion that any metallurgical examination is infallible or has a zero error rate.
- An examiner will not report or state a conclusion that includes a statistical or numerical degree of probability except when based on relevant and appropriate data.
- Under most circumstances, an examiner may not report or state an opinion that the damage to an automotive lamp was the result of the particular accident under investigation.
- An examiner may not report or state an opinion that two or more items were once part of

the same object unless portions of the items physically fit together.

- When offering a *Fracture Fit* conclusion, an examiner may not assert that the fragments originated from the same source object “to the exclusion” of all other metal objects. This may wrongly imply that a *Fracture Fit* conclusion is based on statistically-derived or verified measurement or actual comparison of the items to all other metallurgy sources in the world, rather than on the examiner’s opinion.
- An examiner may not report or state an opinion that a particular company must have produced an item based on the markings present on it. For example, Rolex watches are widely counterfeited. Therefore, the presence of a Rolex trademark does not mean Rolex produced it.
- An examiner may not report or state an opinion that a mass-produced item can be exclusively associated with others of its type in the possession of an individual. For example, the examiner may not say that a screw from an explosive device must have originated from a defendant’s box of screws.
- An examiner may not report or state the results of calculations pertaining to evidence that is presented for the first time to the examiner in the courtroom. The examiner will respectfully decline to perform such calculations on the grounds that such work requires technical verification. Estimates based on prior analyses are permissible.
- An examiner may not cite the number of forensic metallurgy examinations performed in his or her career as a direct measure for the accuracy of a proffered conclusion. (However, such information may be cited for the purpose of establishing, defending or describing the examiner’s qualification or expertise.)
- An examiner may not use the expressions “reasonable degree of scientific certainty,” “reasonable scientific certainty,” or similar assertions of reasonable certainty in either report or testimony unless required to do so by a judge or applicable law.

6 Laboratory Report Reviews

The content of a metallurgy *Laboratory Report* will be reviewed per the appropriate LOM documents and the CU Quality Assurance and Operations and Operations Manual, Case Record and Review Procedures to ensure compliance with the approved statements in this document.

7 Testimony Reviews

Metallurgy testimonies will be reviewed and evaluated following the FBI LOM, Practices for Testimony Related Activities. The evaluation will ensure compliance with the statements in this document.

8 References

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

General Approach to Reporting Metallurgy, Metallurgy Manual Metal 900, Chemistry Unit, latest revision

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

Rev. #	Issue Date:	History:
0	05/23/2014	New document.
1	01/29/2019	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 24 and is now designated Metal 901. Changed title. Updated report title to Laboratory Report throughout. Removed references to metallurgy “discipline” throughout. Revised the order of permissible statements. Revised the terminology regarding comparison conclusions. Incorporated the Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline by reference. Reformatted other references. Updated titles of referenced documents.

Redacted - Signatures on File

Approval

Metallurgy Technical Leader

Date: 01/28/2019

Chemistry Unit Chief

Date: 01/28/2019

QA Approval

Quality Manager

Date: 01/28/2019