

Guidelines for a Material Verification Program for New and Existing Assets

1. SCOPE

1.1. Purpose

The purpose of this recommended practice (RP) is to provide the guidelines for the owner-user to develop and implement a material verification program (MVP) as part of a quality assurance program. The MVP uses PMI and other methods to verify that the nominal composition of an asset, an asset component, or weldment within the pressure envelope is consistent with the selected or specified construction materials. ~~to minimize the potential for catastrophic release of toxic or hazardous liquids or vapors.~~

A well designed and implemented MVP is one of the most important tools used to minimize the potential for catastrophic release of toxic or hazardous liquids or vapors, and other general products contained.

1.2. About this Document

This RP provides the guidelines for material verification programs involving ferrous and non-ferrous alloys during the construction, installation, maintenance, and inspection of new and existing process equipment. This RP applies to metallic materials purchased for use either directly by the owner/user or indirectly through vendors, fabricators, or contractors and includes the supply, fabrication, and installation of these materials. ~~Carbon steel components specified in new or existing piping systems are not specifically covered under the scope of this document unless minor/trace alloying elements are critical to component corrosion resistance or similar degradation.~~

1.3. Material Verification Programs in General

Methods for managing materials are part of an overall Quality Assurance program or Quality Management system.

Purchasing agreements should include clearly stated requirements related to chemical composition (including allowed trace or tramp elements), mechanical properties, critical dimensions, and other acceptance criteria.

The personnel involved in material manufacturing and related quality assurance process must be suitably trained and qualified. Personnel must also have clear requirements related to their duties and up to date acceptance thresholds.

A clearly defined and documented process needs to be in place and understood by all personnel involved in the production, testing, and acceptance of material. Periodic audits should be

conducted at all stages to ensure that processes are being followed: that the techniques used to validate the materials meet requirements, and that processes are delivering consistent results.

When MVP techniques identify materials that do not meet requirements, it is imperative to follow existing quality processes, and correct root causes identified processes that led to the nonconforming product.

Using the above with a well thought out and documented MVP process, PMI is invaluable in the overall achievement of 100% material assurance.

2. REFERENCES

2.1. Normative

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

- API 510, Pressure Vessel Inspection Code: In-service Inspection, Rating, Repair, and Alteration
- API 570, Piping Inspection Code: Inspection, Repair, and Rerating of In-service Piping Systems
- API Recommended Practice 574, Inspection Practices for Piping System Components
- API Recommended Practice 575, Guidelines and Methods for Inspection of Existing Atmospheric and Low-pressure Storage Tanks
- API Recommended Practice 576, Inspection of Pressure-Relieving Devices
- API Recommended Practice 577, Welding Inspection and Metallurgy
- API Recommended Practice 751, Safe Operation of Hydrofluoric Acid Alkylation Units
- API RP 939-C, Guidelines for Avoiding Sulfidation Corrosion Failures in Oil Refineries

2.2. Informative

The following referenced documents are informative in the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

- API RP 571, Damage Mechanisms Affecting Fixed Equipment In the Refining Industry
- API Recommended Practice 580, Risk-Based Inspection
- API 579-1/ASME FFS-1 Fitness-For-Service

- ASME¹ Boiler and Pressure Vessel Code: Section II

Part A: Material Specifications

Part B: Ferrous Materials

Part C: Nonferrous Materials

Part D: Welding Rods, Electrodes, and Filler Metals

- ASME PCC-2, Repair of Pressure Equipment and Piping

- ASME B31.3, Process Piping

CSB² Chemical Safety Board Bulletin 2005-04-B, “Positive Material Verification: Prevent Errors During Alloy Steel Systems Maintenance”

NACE³ Paper No 03651, “Specification for Carbon Steel Materials for Hydrofluoric Acid Alkylation Units”

PFI⁴ ES22, Recommended Practice for Color Coding of Piping Materials

Footnotes

¹ASME International, 3 Park Avenue, New York, New York 10016-5990, www.asme.org.

²U.S. Chemical Safety and Hazard Investigation Board, Office of Prevention, Outreach, and Policy, 2175 K Street NW, Suite 400, Washington, D.C. 20037-1848, 202-261-7600, www.csb.gov. Most CSB publications are posted on, and can be downloaded from the CSB web site, http://www.csb.gov/safety_publications/docs/SB-Nitrogen-6-11-03.pdf.

³NACE International, 1440 South Creek Drive, Houston, Texas 77218-8340, www.nace.org.

⁴Pipe Fabrication Institute, 511 Avenue of the Americas, #601, New York, New York 10011. www.pfi-institute.org.

3.0 DEFINITIONS AND ACRONYMS

3.1 Definitions

3.1.1 Alloy Material - Any metallic material (including welding filler materials) that contains alloying elements, such as chromium, nickel, or molybdenum, which are intentionally added to enhance mechanical or physical properties and/or corrosion resistance. Alloys may be ferrous or non-ferrous based.

3.1.2 Distributor - A warehousing supplier for manufacturers or suppliers of materials or components.

3.1.3 Fabricator - One who fabricates piping systems, or portions of a piping system, as defined by ASME B31.3

3.1.4 Heat - A batch of metal made at the same time, able to be traced from its original constituents and manufacturing process

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- 3.1.5 Inspection Lot - A group of items or materials of the same type from a common source from which a sample is to be drawn for examination. NOTE: An inspection lot shall not include items from more than one heat.
- 3.1.6 Level of examination - The specified percentage of the number of items to be examined in an inspection lot.
- 3.1.7 Lot size - The number of items available in the inspection lot at the time a representative sample is selected.
- 3.1.8 Material manufacturer - An organization that performs or supervises and directly controls one or more of the operations that affect the chemical composition or mechanical properties of a metallic material.
- 3.1.9 Material nonconformance - A positive material identification (PMI) test result that is not consistent with the specified material.
- 3.1.10 Material supplier - An organization that supplies material furnished and certified by a material manufacturer, but does not perform any operation intended to alter the material properties required by the applicable material specification.
- 3.1.11 Material verification program - A documented quality assurance procedure used to assess materials (which can include weldments and attachments when specified) in order to verify conformance to a specification as designated by the owner/user. The program should include a description of methods for material testing, component marking, and record-keeping.
- 3.1.12 Mill Test Report (MTR) - A document issued and certified by the material manufacturer identifying an item according to its heat and confirming conformance to specification. Mill test reports include the results of specification-required testing including the original chemical analysis.
- 3.1.13 Owner-user - The organization that exercises control over the operation, engineering, inspection, repair, alteration, pressure testing, and rating of the piping systems.
- 3.1.14 Pressure-containing components - Items that form the pressure-containing envelope of the piping system.
- 3.1.15 Positive Material Identification (PMI) - A physical evaluation or test of a material performed to confirm that the material which has been or will be placed into service is consistent with what is specified by the owner/user. These evaluations or tests may provide either qualitative or quantitative information that is sufficient to verify the composition.
- 3.1.16 Random - Selection process by which choices are made in an arbitrary and unbiased manner.
- 3.1.17 Representative sample - One or more items selected at random from the inspection lot that are to be examined to determine acceptability of the inspection lot.

3.1.18 Standard reference materials - Sample materials for which laboratory chemical analysis data are available and are used in demonstrating the accuracy and reliability of a test instrument.

3.2 Acronyms

API American Petroleum Institute

ASME American Society of Mechanical Engineers

IDL Instrument Detection Limit

LOD Limit of Detection

LOQ Limit of Quantization

MDL Method Detection Limit

MTR Mill Test Report

MVP Material Verification Program

NACE National Association of Corrosion Engineers

PFI Pipe Fabrication Institute

PMI Positive Material Identification

RSD Relative Standard Deviation

SD Standard Deviation

4.0 APPLICABILITY CONSIDERATIONS AND GENERAL CONCERNS

4.1 Applicability

<A paragraph suggesting applicability key points in upstream, midstream and downstream sectors is required>

4.2 Carbon Steel Substitutions in Low Alloy Steel Systems

When considering the likelihood of material non-conformances, it is worth noting that historically the greatest number of material non-conformances with serious consequences have involved carbon steel components inadvertently placed into low alloy steel (e.g. 1¼Cr-½Mo, 2¼Cr-1Mo, 5Cr-½Mo, 9Cr-1Mo) piping systems. There have been fewer non-conformances in stainless steel and nonferrous systems because of appearance and weldability issues.

4.3 Alloy Substitutions for Carbon Steel

When determining the need to perform material verification on carbon steel, the owner/user should evaluate the effect that the process stream could have on substituted materials. In some cases, the substitution of hardenable alloy materials for carbon steel has resulted in failure and loss of containment. Examples of such systems include those handling wet hydrogen sulfide (H₂S), hydrofluoric acid (HF), or sulfuric acid (H₂SO₄). Where material substitution could lead to failure and/or loss of containment, the extent of verification should be planned and/or increased in effectiveness to validate the materials specified for use

4.4 Residual Elements In Carbon Steels In Hydrofluoric Acid Alkylation Units

Carbon steels in some specific locations in HF acid service have been reported to suffer increased corrosion rates based on the residual element (RE) content in the steels (refer to API RP 751.) PMI methods can be used to assess the suitability of materials for HF service in carbon steels according to RE content and the discussion within the context of this document. Consideration should be given to the ability of the PMI method to detect the various elemental concentrations, which are miniscule in comparison to the bulk analysis.

4.5 Process Units Susceptible To Sulfidation

Carbon steels with low silicon (<0.10%) content can corrode at an accelerated rate when exposed to hydrogen-free sulfidation conditions. These phenomena are discussed more extensively in API RP 571 and API RP 939-C. Owner/users with assets at risk from this type of

degradation should consider the risks and the requirements to apply PMI control in order to determine silicon levels and the effect on predicted corrosion rate.

4.6 Gasket Materials

Gaskets in incompatible service may cause premature failings. The principles outlined in this document can also be applied to gasket materials. The owner/user is required to ascertain the potential material variations and select analytical methods capable of providing the required data. The actual test procedures can be more detailed due to the geometric structure of the gasket materials.

4.7 Refractory (anchors)

Selection of refractory anchor metals shall be suitable for the anticipated operating conditions. PMI testing to verify the refractory anchors match specifications should be considered by the owner/operator. Material specifications and maximum design temperatures for selected materials are discussed API Standard 560.

5.0 MATERIAL VERIFICATION PROGRAMS

5.1 General

The owner/user shall establish a written material verification program indicating the extent and type of PMI testing to be conducted during the construction of new assets, retroactively on existing assets, and during the maintenance, repair, or alteration of existing assets.

For higher-risk systems, the owner/user should consider the need for employing a higher level of examination (up to 100%) rather than random sampling which may be more appropriate for lower-risk systems. Inadvertent material substitution problems tend to be sporadic; therefore, small sample sizes may not locate all inadvertent alloy substitutions. The owner/user should also consider the need to conduct examinations after fabrication is complete, to ensure that incorrect substitutions did not occur at the work site.

5.2 Components Covered in a Material Verification Program

Examples of some pressure-containing components that are found in equipment and systems which are included in this RP:

- pipe lengths;
- pipe fittings, such as tees, elbows, reducers, caps, special pipe components, blinds and plugs;
- flanges;

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- special forgings;
 - valves – process valves, control valves, relief valves
 - pressure-containing welds;
 - instruments (all pressure containing parts);
 - weld overlays or cladding;
 - bolting;
 - expansion joints and bellows;
 - gaskets

5.3 Document concerns in an MVP

5.3.1 Mill Test Reports

Mill test reports should not be considered a substitute for a PMI test. However, mill test reports are an important part of an overall material quality assurance program.

5.3.2 Material Certifications

Material Certifications or Certificates of Compliance should not be considered a substitute for PMI testing, but may be an important part of an overall quality assurance program.

5.4 New Construction MVP

5.4.1 In General

This section covers equipment during fabrication either in the shop or in the field prior to the items being placed into service. It is not restricted to only pressure-containing boundaries, unless explicitly stated by the owner-user's documented material verification program

5.4.2 Roles and Responsibilities

A material verification program may involve participation of several groups within the operating plant or the shop of a contractor, vendor, or fabricator. When establishing a material verification program, consideration should be given to the roles and responsibilities that each group has within the specific organization. These roles and responsibilities should be clearly defined and documented. In the operating plant, this can include those groups responsible

for purchasing, engineering, warehousing/receiving, operations, reliability, maintenance, and inspection.

It shall be the responsibility of the owner/user or designee:

- (1) to determine the extent of examination required,
- (2) to verify that the implementation of the material verification program,
- (3) to verify the material verification program is properly performed according to this RP, and
- (4) to ensure the documentation of the material verification program is according to this RP.

It shall also be the responsibility of the owner/user to verify that the materials subsequently placed into service are as specified.

5.4.3 Material Verification Test Procedure Review

When PMI testing is performed by the material supplier or third-party agency, the owner/user or designee shall review and approve the adequacy of the material verification program and testing procedure of the fabricator or material supplier prior to testing.

5.4.4 Scheduling of Material Verification Testing

PMI testing should be performed at the point in time that ensures that proper materials have been used in the fabrication of an identifiable assembly.

- #### 5.4.5 PMI Testing of Components Supplied by a Distributor
- A higher degree of PMI testing verification should be conducted on materials supplied by stocking distributors due to the potential for inadvertent material substitutions as a result of frequent handling.

5.5 Existing Assets MVP

5.5.1 In General

This section covers assets that are already in service where the material verification program procedures for the construction are not documented or not completed according to this RP. Material verification coverage is only limited by the owner-user's written program, but shall meet the minimum

requirements as stated in this RP. It is important to recognize that previous maintenance activities, as well as new construction practices, may influence the likelihood of inadvertent materials substitutions.

5.5.2 Responsibilities

The owner/user shall be responsible:

- (1) for determining if a retroactive material verification program is appropriate for the assets in question,
- (2) for prioritizing the identified assets requiring retroactive PMI testing, and,
- (3) for determining the extent of PMI testing required.

5.5.3 Prioritizing Assets for Retroactive PMI Testing

5.5.3.1 General Factors to Consider

If the owner/user elects to prioritize **equipment or systems** for the material verification program or needs to determine whether PMI testing is needed at all, the owner/user should consider the following:

- (1) Likelihood of inadvertent material substitutions during previous projects and maintenance activities. The effectiveness of the material verification program when these activities occurred is an important consideration.
- (2) Consequences of a failure due to improper material being installed. Flammability and potential for spreading fire, toxicity, proximity to other equipment or community, temperature, pressure, mode of failure, and size of release should be considered.
- (3) Reason for a specific material specification (i.e., corrosion resistance or product purity). This refers to the criticality of the material of choice.
- (4) Historical data relating to inadvertent material substitutions. This may be related to previous experience with material non-conformities

Taken together, these factors can be used to determine the risk associated with possible material non-conformances in a process unit, a piping system, or an asset.

The owner/user should establish a methodology for estimating the relative priority for PMI testing within a given unit. This methodology may be based on a

qualitative or quantitative risk analysis. API RP 580 discusses risk-based approaches and what should be considered when conducting a risk analysis (such as material, service conditions, service fluid, and mode of failure).

5.5.3.2 Site Specific Factors to Consider

Site-specific or experienced-based factors should be considered when prioritizing equipment or systems:

(1) Construction and maintenance practices. In assessing the likelihood of material non-conformances, the owner/user should also consider the materials handling, material control, and any PMI testing procedures followed during construction of the process unit. Process-unit maintenance procedures are also important. Process units in which rigorous procedures for material verification are used would be expected to have a lower likelihood of non-conformances.

(2) Reason for alloy specification. In some cases, alloys are used in piping systems for reasons other than corrosion resistance or structural integrity. In these cases, the structural integrity of the system may not be harmed by material non-conformances. A material verification program may not be necessary in these systems. An example would be stainless steel lube oil systems in which stainless steel is used for maintaining oil purity.

5.5.3.3 Component Prioritization Factors and pipe

Based on experience, some types of piping system components can have a higher likelihood of inadvertent substitution of a non-specified material. This can provide a basis for prioritizing specific equipment in a given system or process unit. Examples are:

- (1) warm-up and bypass lines on pumps or check valves;
- (2) small bore piping, including welds (2 NPS and below)
- (3) valves and other removable devices such as rupture discs, spacer blinds, or ring joint gaskets;
- (4) thermowells;
- (5) bolting;
- (6) piping as a part of a packaged system;
- (7) components without recognized marking;

(8) specific process systems with known corrosion issues

5.5.3.4 Factors to Consider When Determining the Extent of PMI Testing

The owner/user should determine the extent of PMI testing. Factors to consider when determining the extent of PMI testing for an existing process unit include:

- (1) historical inspection and material verification program records,
- (2) number of plant modifications,
- (3) materials control during original construction and during modifications,
- (4) material verification program quality during construction and fabrication,
- (5) consequence of release,
- (6) likelihood of corrosion/degradation.

5.6 The MVP as an Element of Process Safety Management

5.6.1 In General

The principles associated with materials verification should also be applied to provide confidence that proper materials are being used as part of a comprehensive process safety management (PSM) system. The concepts noted previously should be reviewed and applied as applicable to PSM.

In particular, the PSM program usually addresses materials verification through the inspection function of the company. The inspection function should cover new assets, existing assets, and the related maintenance activities that could affect the mechanical integrity of the assets.

5.6.2 Responsibilities

It shall be the responsibility of the owner/user to evaluate maintenance systems so that material verification programs can be designed and implemented to effectively support PSM. The owner/user should establish a written procedure for the material verification program to be used for repair of assets during maintenance activities. This procedure shall be documented by the owner/user.

5.6.3 Control of Incoming Materials and Warehousing

A material verification program should be directly applied to activities associated with receiving materials into a warehouse system. PMI testing may

be performed as part of this receiving function, or, when appropriate, may be performed at the supplier's location as a condition of release for shipment. The material verification program that is adopted should provide for proper documentation and methods for indicating which materials have been tested and are approved for use.

The use of material verification program principles to check materials received into a warehouse system should be regarded as a Quality Assurance practice to minimize the potential for discovering an alloy material discrepancy during subsequent PMI testing. PMI testing within the warehouse should not be regarded as an alternative to PMI testing of the fabricated piping system when testing is specified.

5.6.4 Maintenance Activities

There are a number of in-service maintenance activities where material verification should be established. Some examples include, but are not limited to:

(1) Temporary removal of piping spool pieces needs to be managed in such a way that material mix-ups cannot occur. Incidents have occurred when same size spool pieces were removed during turnarounds and substituted into the wrong locations during re-installation. Consideration should be given to a firm control system or verification prior to re-installation to prevent these incidences from occurring. A process where a system of 'tagging' spools as they are removed to ensure correct replacement should be considered.

(2) Replacement of small bore threaded pipe nipples, frequently found as drains and vents in process areas. An immediate need may necessitate installation of nonconforming materials in a temporary repair. It is important to recognize even small changes need to be documented and reported for possible future follow-up

(3) < examples needed >

It is important that repair procedures include consideration of PMI testing as part of obtaining satisfactory alloy materials to be used for the repair. As appropriate, this may include any of the components noted in 5.2.

Much of this can be controlled through awareness of the issues at all levels within the repair process. Consulting with an Inspector prior to commencement of work can ensure that systems, processes and activities are in place to ensure good material control.

6.0 MATERIALS VERIFICATION TEST METHODOLOGY AND TECHNOLOGY

6.1 In General

A variety of PMI test methods are available to determine the identity of alloy materials. The primary methods include: portable spectroscopy and laboratory chemical analysis. A description of several test methods is listed below. In addition to these methods, there are a variety of alloy sorting techniques that may be appropriate for the purposes of this RP, including magnetic testing to differentiate between ferritic and austenitic materials. It is important that users verify what the objectives and accuracies are required from the PMI tool they wish to use. All of the tools have benefits and limitations on the elements they can or cannot detect, in addition to the accuracy and ability to differentiate between different material grades which have only slight variations in alloying elements.

6.2 MVP Test Method Objectives

The test methods outlined in this RP are intended to identify materials and are not intended to establish the exact conformance of a material to a particular specification. Depending on the test method selected, the method may identify the nominal composition of materials. Identification of materials by visual stamps/markings alone should not be considered as a substitute for PMI testing but may be an important component of an overall quality assurance program.

6.3 PMI Test

The PMI test procedure should identify and include:

- (1) the techniques used,
- (2) equipment calibration elements,
- (3) qualification requirements for PMI test personnel,
- (4) the testing methodology,
- (5) acceptance criteria and
- (6) the documentation requirements

6.4 Documentation Review

<Needs verbiage>

6.5 Personnel Qualifications

The person(s) performing the PMI test should be knowledgeable about all aspects of operation of PMI test equipment and the PMI test method used. Qualifications of the person performing the PMI test, including training and experience, should be submitted for review and approval by the owner/user.

Owner/users should ensure that personnel using testing devices are adequately trained not only in the specific instrument but also educated about the materials they will be examining. In some cases, (critical applications) a formal documented program and some form of testing and certification of personnel may be required. The higher the degree of operator analysis the more important this aspect of the whole procedure becomes.

6.6 Chemical Analysis Techniques

6.6.1 Chemical Laboratory Chemical Analysis 6.6.1

Owner/user-approved material analysis laboratories using X-ray emission spectrometry, optical emission spectrometry, or wet chemical analysis can provide the most accurate analytical results for all elements. The accuracy is typically much higher than is normally needed for PMI testing. Laboratory analysis may involve the removal of significant amounts of material, and is typically slower than field PMI test techniques.

6.6.2 Chemical Spot Testing

The chemical spot test is typically accomplished by electrochemically removing a minute amount of surface metal and depositing it onto moistened filter paper. Reagents dropped onto the paper produce distinct colors that are indicative of the presence of specific elements in the sample tested. Chemical spot testing is much slower than the other field PMI test methods and interpretation is subjective.

6.7 Sorting Analysis Techniques

6.7.1 Resistivity Testing

The principle employed in the test method is known as the Seebeck Effect, or thermoelectric principle. A heated junction of dissimilar metal is created when the heated probe [300°F (150°C)] and the metal being tested are in contact with each other. The voltage generated at this junction is representative of the chemistry and crystalline structure of the metal being tested. Every alloy of a given crystalline structure will generate the same voltage regardless of the geometry or size of the piece being tested or the pressure applied. By references to known standards, these instruments are capable of sorting and identifying a wide range of ferrous and nonferrous materials. Alloy sorters have not proved to be consistently capable of sorting low alloy (<5 % Cr) and austenitic stainless steels.

6.7.2 Other Techniques

Techniques such as eddy-current sorters, electromagnetic alloy sorters, triboelectric testing devices (e.g., ferrite meters), and thermoelectric tests are qualitative and as such may only be appropriate for limited sorting applications and not for specific alloy identification.

6.8 Spectrometer Technology

6.8.1 Portable X-ray Fluorescence

There are several variants of portable X-ray fluorescence (XRF) spectrometers available. The principle of operation is that one or more gamma ray or X-ray sources are used to generate a beam of low energy radiation to excite the material under analysis. The material under analysis then emits a characteristic radiation spectrum which can be analyzed both qualitatively and quantitatively to determine which elements are present and in what quantity. The results of this analysis can be reported in either of the following formats:

- (1) as a match against one of many reference spectra stored in the instrument (i.e. '316 Stainless Steel' or '5 Cr –½ Mo Steel'); and/or,
- (2) each element present is reported as a percentage (i.e., 'Iron = 87.5%' or 'Iron = 0.875' as part of an entire elemental list that should be normalized against 100%).

A number of PMI instruments are available. These instruments should have the sensitivity to determine the elemental levels that meet the ASTM limits. Advancements in XRF technology, such as 50KV, x-ray tubes, and Silica Drift Detectors (SDD), allow the user to detect light elements down to very low concentrations in steel substrates. For example, the latest generation of handheld X-ray fluorescence analyzers now measure light elements (Mg, Al, Si, P, and S) to very low limits of detection and can provide for field measurement of silicon in steel down to a very low concentrations.

These advancements in technology allow for accurate, non-destructive measurements to be conducted. However, in order to get this near laboratory-quality measurement, some surface preparation is required. Most assets are exposed to environments that promote surface corrosion and contamination which can interfere with the analysis. Typically a small test area is prepared with a portable grinding disk (60 to 80 grit Zirconium Aluminum Oxide disposable abrasive disk) to properly clean the sample location prior to analysis.

Because of the inherent limitations of the technique it is not possible to detect all elements. XRF analyzers are capable of detecting elements from magnesium (Mg) to

Uranium (U) in the periodic tables. This may exclude some of the important elements in carbon steels such as carbon and boron. It is important to decide exactly what elemental analysis is required and select an appropriate instrument.

6.8.2 Portable Optical Emission Spectrometry

In optical emission spectrometry (OES), an electric arc stimulates atoms in the test sample to emit a characteristic spectrum of light for each element in the sample. The combined light spectra from different elements are passed through a light guide to the optical analyzer. In the analyzer, the light is dispersed into its spectral components, and then measured and evaluated against stored calibration curves.

These devices fall into two groups:

(1) The first is a light-weight, portable and operator evaluated device that can typically identify up to sixteen (16) elements but depends upon operator evaluation of the light spectra. These devices do not directly indicate alloy grade or composition, but produce an output in the form of visible light spectra that permits semi-qualitative alloy identification. This technique is highly sensitive to operator skill and experience.

(2) The second group refers to field portable, laboratory grade analyzers. These were originally difficult to use due to their size and weight; however, modern units are now available that can be considered light-weight, including the small Argon cylinders required for operation. Some of these analyzers operate in a pure arc mode for routine PMI applications while the more sophisticated units have a spark mode allowing laboratory quality analysis. The significant advantage of these instruments is the expansion of elements that can be analyzed including carbon. An advantage of these advanced instruments is that they are not subject to operator interpretation.

Similar to X-ray fluorescence devices, results can be reported in either a spectral match or elemental percentage mode. As these techniques generate arcs and sparks, a potential ignition source occurs during their operation and therefore prior to use of this technique in the field a review shall be conducted in order to determine if gas testing and hot work permits are required.

6.8.3 Laser Induced Breakdown Spectroscopy

Laser-induced breakdown spectroscopy (LIBS) is an atomic emission spectroscopy technique which uses highly energetic laser pulses. The laser is focused to form a plasma, which atomizes and excites samples. Plasma light emissions provide the spectral signature of chemical composition for practically any material.

When calibrated for metal analysis, LIBS can provide both qualitative and quantitative information to determine which elements are present and in what quantity. The results of this analysis can be reported as metal chemistry and based on stored grade library also as metal grade identification.

Considerable progress has been made during the last few years in LIBS technology. Handheld LIBS metal analyzers are becoming available which can provide fast in situ chemical analysis with a good precision and detection limits. The significant advantage of LIBS technology is its wide element range. In principle, LIBS can detect all elements, limited by the power of the laser as well as the sensitivity and wavelength range of the spectrograph & detector. The LIBS technology also can provide low level carbon analysis, which in principle enables LIBS to positively identify a wide range of ferrous alloys.

6.8.4 Equipment Calibration

Persons performing the PMI testing should calibrate and/or verify the test equipment performance as specified by the equipment manufacturer. The PMI test procedure should provide the frequency interval for this calibration/verification. If calibration procedures are not provided by the equipment manufacturer, they should be established by the owner/user. Typically, these procedures should include calibration/verification using certified standards.

6.8.5 Equipment Precision

The precision of the test equipment should be consistent with the established test objectives (see 5.2). When component composition is desired, the owner/user should establish the acceptable precision and repeatability.

Accuracy and the method in which accuracy is determined need to be understood. For example, in some tools the sensitivity may depend on duration of the test in order to improve signal averaging algorithms. Failure to understand these issues may produce inaccurate results.

6.9 Safety Issues

6.9.1 In General

The specific requirements for each PMI test technique should be clearly reviewed as to the amount of mechanical preparation. Consideration should be given to the anticipated thickness of the sample before mechanical methods are used to prepare the sample. In addition, considerations for electrical arcing and

“hot spots” should be considered as well as appropriate electrical and hot work permits. Chemical spot testing involves the use of a variety of chemicals. Appropriate safety precautions should be taken when handling these chemicals.

6.9.2 XRF Analyzers

6.9.2.1 Intrinsic safety

XRF Analyzers are not intrinsically safe. Pertinent Hot Work procedures should be followed in areas where non-intrinsically safe equipment is to be used.

6.9.2.2 Radiation Safety

The XRF Analyzer was designed so that virtually no measurable radiation external to any part of the analyzer can escape when the shutter is closed. Some XRF analyzers contain an x-ray tube, which emits no radiation at all unless the user turns the x-ray tube on.

Human exposure to radiation is typically measured in REMs, frequently reported in units of one-thousandths of a REM, called milliREMs (mREM). For a given source of radiation, three factors will determine the radiation dosage received from the source:

(1) Duration of Exposure

The longer one is exposed to a source of radiation, the more radiation strikes the body and the greater the dose is received. Dosage increases in direct proportion to time of exposure.

(2) Distance from the Source

The closer one is to a source of radiation, the more radiation strikes the body. The dosage increases in inverse-squared relation to distance from the source of radiation. For example, the radiation dose one inch from a source is nine times greater than the dose three inches from the same source, and 144 times greater than the dose 12 inches from the source. Always keep hands and all body parts away from the front end of the analyzer when the shutter is open to minimize exposure.

(3) Shielding

The analyzer emits no radiation at all unless the x-ray tube is turned on, and virtually no radiation with the x-ray tube on and the internal shutter closed (as during calibration). When the x-ray tube is on and the shutter is open, as during a measurement, the analyzer emits a directed radiation beam. Always hold the

analyzer so to avoid the radiation beam. Consider using a test stand to add shielding for analysis.

Note: Wearing a dosimeter badge does not protect against radiation exposure. A dosimeter badge measures exposure.

Note: Pregnant workers may want to take special precautions to reduce their exposure to radiation. It has been recommended that the radiation dose to pregnant women should not exceed a total of 500 mREM/gestation period. [Refer to U.S. NRC Regulatory Guide 8.3 "Instruction Concerning Prenatal Radiation Exposure," which lists typical radiation doses encountered in daily life and the annual occupational radiation dosage limits for adults set forth in 105CFR120.200].

The allowable limit in the U.S. for occupational exposure is 5,000 mREM/year (50 mSv/years) for a whole-body and 50,000 mREM (500 mSv) for shallow penetration of extremities. Extremity exposure from a properly used XRF analyzer will be less than 100 mREM per year, (1.0 mSv per year) even if the analyzer is used as much as 2,000 hours per year, with the shutter open and the tube energized continuously.

6.10 Testing of Welds and Weldments

6.10.1 PMI Testing of Welding Consumables

When welding is conducted, one electrode or wire sample from each lot or package of alloy weld rod should be positively identified. The remainder of the lot should be compared to the sample to verify that the markings of the wires/electrodes are correct.

Some weld rods have the alloying elements contained in the flux, and do not meet the alloy specification until welded. PMI testing of weld metal (e.g. deposited weld metal or undiluted weld "buttons") is an acceptable alternative to PMI testing of an electrode or wire sample provided. It is conducted immediately prior to welding or during the welding process (especially when changing lot numbers of electrodes or welding wire).

6.10.2 Longitudinal Pipe and Fitting Welds

Where there is reason to suspect problems, longitudinally welded alloy pipe and fittings should receive random PMI testing verification of the base metal and weld metal.

6.10.3 Autogenous Welds

If the owner/user determines that material verification testing is required on autogenous-welded (with no added filler metal) alloy pipe or fittings, it is necessary to conduct testing on only the base metal.

6.10.4 Dissimilar Metal Welds and Weld Overlays

Results from testing dissimilar metal welds should take into account the effects of dilution, which occurs during weld deposition. The owner/user should establish the minimum compositional requirements of the as-deposited weld metal necessary for the intended service.

7.0 EVALUATION OF TESTING RESULTS

7.1 Material Acceptance Methods

The owner/user may elect any one of the following methods of material acceptance.

- 1) Materials can be confirmed to contain the nominal amounts of alloying elements specified in the relevant materials specification (e.g., ASME Section II or ASTM specifications).
- 2) Materials can be classified through a qualitative sorting technique (see 6.7) to establish the conformance with the intended material.
- 3) When PMI testing indicates alloying elements are outside the ranges indicated in the material specification, the owner/user may still choose to allow the use of the tested materials in situations where a person knowledgeable of the appropriate damage mechanisms confirms that the material will perform satisfactorily in the service.
- 4) If testing using one of the portable or qualitative analysis methods leads to the potential rejection of a component, a more accurate analysis may be used to determine component acceptance (see section 6).

7.2 Follow-up PMI Testing after Discovery of a Nonconformity

If any one of a representative sample is rejected, all items of that inspection lot should be considered suspect. A more extensive inspection of the remaining lot should be considered.

8.0 MATERIALS IDENTIFICATION

8.1 Identification Process

Alloy materials should be identified by their alloy designation or nominal composition. Examples of some acceptable identification methods are:

- 1) color coding by alloy,

2) a low-stress stamp marking indicating that the test has been performed,

3) documentation showing both the PMI test results and the PMI test locations.

Test locations should be shown on appropriate drawings so that each test site can be traceable to the fabricated piping components.

8.2 Color Coding/Marking

8.2.1 If the material verification program procedure established by the owner/user requires a visual identification such as color coding or marking, the owner/user should maintain a record of the alloy material/color code combinations. PFI Standard *ES22* is an example of one such system. Materials identification by color coding is not a substitute for permanent manufacturers' markings required by applicable ASTM or other materials specifications.

8.2.2 Where reliance on color marking is used, persons responsible for reading the colors should be confirmed as being able to distinguish difference between the colors being used.

8.3 Marking of Components

If the owner/user's documentation process requires physical marking of piping components, it should specify one of the following.

1) Whether or not the marking system should remain legible for the expected life of the component without deterioration due to corrosion or elevated temperature.

2) Whether or not the marking system is only temporary to facilitate proper handling and identification from the point of PMI testing to final installation. This marking can be semi-permanent paint applied to each item. The markers should not contain additives such as metallic pigments (Al, Pb, or Zn), sulfur, or chlorides.

8.4 Traceability to Field Components

The information listed in 7-5 [9.3] should be reported in such a manner that it is traceable to the point of installation.

9.0 DOCUMENTATION AND RECORDKEEPING

9.1 Shop and Field PMI Test Documentation

Those individuals performing PMI testing should obtain and follow the PMI test procedure approved by the owner/user. This procedure should include the technique used, equipment calibration, the qualification requirements of PMI test personnel, the testing methodology, and documentation requirements.

When documentation, such as drawings, is used in lieu of physical marking, the documentation should allow the owner/user to identify which components were tested.

9.2 New and Existing Equipment or System Documentation

When PMI testing is conducted on new or existing **assets and systems**, records of the results should be kept as long as the **equipment** exists in its original location. If equipment or portion of an equipment system that has not received material verification is relocated, the owner/user should consider the need for PMI testing prior to placing the relocated components into service.

9.3 PMI Test Records

Typical PMI test records should contain the following:

- 1) Reference to the PMI test procedure(s) used.
- 2) Date of testing.
- 3) Test instrument identification number or serial number, where appropriate.
- 4) Name of each person performing the tests, with identification of their respective employer.
- 5) Results of the tests.
- 6) Basis and action for resolving and documenting PMI test non-conformances including those that have been left in service.
- 7) Documentation of the criteria used for prioritization of piping systems and extent of PMI testing performed.

Alternately, the owner/user may choose to include this within the written material verification procedure. When included in the owner/user's written material verification procedure, the date and edition number of the written procedure should be documented in the test record.

Annex A (informative)

Statistical Terminology

Below are listed some of the terminologies regarding statistics that may be encountered when using the methodology and/or technology discussed within this document.

Accuracy - The closeness of agreement between the test result and the accepted reference value

Precision - The closeness of agreement between independent tests results The Precision depends only on the distribution of random errors and does not relate to the accepted standard certified value.

Bias - The difference between the expectation of the test results and an accepted reference value. Bias is a systematic error in contrast to a random error. There may be one or more systematic error components contributing to the Bias.

Limit of Detection (LOD) - the lowest concentration of analyte that can be reliably distinguished from zero concentration in a sample. By defining a 99% confidence to show that the analyte concentration is greater than zero. Therefore, 3-sigma calculations must be used to determine LOD. See SD, below.

Instrument Detection Limit (IDL) - the lowest (best) detection limit generated by an instrument in a clean matrix (blank).

Method Detection Limit (MDL) - the typical detection limit obtained when running a typical sample, it is matrix dependent, and will vary from matrix to matrix.

Limit of Quantization (LOQ) - the lowest concentration that can be reliably measured to allow quantitative readings. It is typically defined as 10-sigma (or 3.33 times the LOD).

Standard Deviation (SD) - The standard deviation is a statistic that defines how tightly all the data is clustered around the mean in a data set. It essentially measures the variability (spread or dispersion) from the mean (average) and is defined mathematically. For "normal" distributions, we can apply an empirical rule that states that 68% of the data is within one standard deviation of the mean, 95% of the data is within two standard deviations of the mean, and 99.7% of the data is within three standard deviations of the mean

Relative Standard Deviation (RSD) - The standard deviation is divided by the mean of the data and multiplied by 100 to give a % value. The bigger the % value, the "noisier" the signal and less confidence in the final value.

Repeatability - The duplicate (or more) analysis within the shortest possible time, performed by the same person using the same method. The standard of many analyses using repeatability conditions indicates the minimum deviation achievable in the particular conditions.

Reproducibility - This is a duplicate (or more) analysis performed by a different person preferably using a different method or (different instrument).