

# **Material Verification Program for New and Existing Assets**

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# Material Verification Program for New and Existing Assets

## 1 Scope

### 1.1 Purpose

The purpose of this document is to provide recommended practices for an owner/operator to develop and implement a material verification program (MVP) as part of an asset integrity program. The MVP uses positive material identification (PMI) and other testing and administrative methods to verify that the nominal composition of an asset, an asset component, or weldment (or any other tested item) is consistent with the selected or specified construction materials.

A well-designed and well-implemented MVP is an important management system used to minimize the potential for release of hazardous substances due to nonconforming materials of construction.

### 1.2 About This Document

This recommended practice addresses MVPs involving ferrous and nonferrous alloys during the construction, installation, maintenance, and inspection of new and existing process equipment. It applies to metallic materials purchased for use either directly by the owner/operator or indirectly, through distributors, fabricators, or contractors, and includes the supply, fabrication, and installation of these materials.

This recommended practice is applicable to all refining and petrochemical industries and may be applied in other industries and or businesses at the discretion of the owner/operator. It is intended to be applied by any owner/operator wishing to verify and/or validate that the materials of construction received, fabricated, and/or installed are in accordance with purchase documents and/or company specification(s).

## 2 Normative References

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any addenda/errata) applies.

API Recommended Practice 571, *Damage Mechanisms Affecting Fixed Equipment in the Refining Industry*

API Recommended Practice 580, *Risk-based Inspection*

API Recommended Practice 751, *Safe Operation of Hydrofluoric Acid Alkylation Units*

API Recommended Practice 939-C, *Guidelines for Avoiding Sulfidation (Sulfidic) Corrosion Failures in Oil Refineries*

ASME<sup>1</sup> Boiler and Pressure Vessel Code (BPVC), *Section II: Materials (Part A: Ferrous Material Specifications; Part B: Nonferrous Material Specifications; Part C: Specifications for Welding Rods, Electrodes, and Filler Metals; Part D: Properties)*

## 3 Terms, Definitions, Acronyms, and Abbreviations

### 3.1 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

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<sup>1</sup> American Society of Mechanical Engineers, Two Park Avenue, New York, New York 10016, [www.asme.org](http://www.asme.org).

**3.1.1****alloy material**

Any metallic material (including welding filler materials) that contains alloying elements, such as chromium, nickel, or molybdenum, that are intentionally added to enhance mechanical or physical properties and/or corrosion resistance.

NOTE 1 Alloys may be ferrous or nonferrous.

NOTE 2 Carbon steels are not considered alloys for the purposes of this document.

**3.1.2****asset**

Equipment owned by a company that is either directly or indirectly involved with the manufacturing process.

**3.1.3****distributor**

A warehousing supplier for manufacturers or suppliers of materials or components.

**3.1.4****extent of examination**

The specified percentage of the number of items to be examined in an inspection lot.

**3.1.5****fabricator**

An organization that utilizes the materials of construction affected by this recommended practice to create an asset.

**3.1.6****heat**

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

**3.1.7****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 does not include items from more than one heat.

**3.1.8****lot size**

The number of items available in the inspection lot at the time a representative sample is selected.

**3.1.9****material certifications****certificates of compliance**

See "mill test report."

**3.1.10****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.

**3.1.11****material nonconformance**

A PMI result or other documented certification that does not conform with material specified.



**3.1.12****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.

**3.1.13****material verification program****MVP**

A documented work practice that uses PMI and other testing and administrative methods to verify that the nominal composition of an asset, equipment item, an asset component, or weldment within the pressure envelope is consistent with the selected or specified construction materials.

**3.1.14****mill test report****MTR****certified mill test report****certified material test report****mill test certificate****inspection certificate****certificate of test**

A quality assurance document used in the steelmaking industry that certifies a material's compliance with appropriate standards, including physical and chemical specifications, and applicable dimensions.

NOTE The MTR also includes a date of production and testing and may include notation about method of fabrication. A "mill test report" is also known by other names; MTR is the term used in this document.

**3.1.15****owner/operator**

The organization that exercises control over the operation, engineering, inspection, repair, alteration, pressure testing, and rating of the assets.

**3.1.16****positive material identification****PMI**

A physical evaluation or test of a material performed to confirm that the material that has been or will be placed into service is consistent with what is specified by the owner/operator.

NOTE These evaluations or tests may provide either qualitative or quantitative information that is sufficient to verify the composition.

**3.1.17****pressure-containing components**

Items that withhold the fluid contents of the equipment system and prevent the fluid from being released to the environment.

**3.1.18****quality assurance****QA**

All planned, systematic, and preventative actions specified to determine if materials, equipment, or services will meet specified requirements so that equipment will perform satisfactorily in service.

**3.1.19****quality control****QC**

Those physical activities that are conducted to check conformance with specifications in accordance with the quality assurance plan.

**3.1.20****random**

Selection process by which choices are made in an arbitrary and unbiased manner.

**3.1.21****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.

**3.1.22****retroactive positive material identification****retro-PMI**

The term commonly used for a PMI on existing material or equipment.

**3.1.23****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.

**3.1.24****weld button****button**

A sample of welding filler material deposited on relevant base metal to provide basis for PMI of the filler.

**3.2 Acronyms and Abbreviations**

LIBS	laser-induced breakdown spectroscopy
LOD	limit of detection
MTR	mill test report
MVP	material verification program
OES	optical emission spectrometry
PMI	positive material identification
QA	quality assurance
QC	quality control
XRF	X-ray fluorescence

**4 Examples of Where Material Verification Program May Be Needed****4.1 Carbon Steel Substitutions in Low-alloy Steel Systems**

When considering the likelihood of material nonconformances, it is worth noting that, historically, the greatest number of material nonconformances with serious consequences has involved placing unapproved carbon steel components into low-alloy steel (e.g., 1¼Cr–½Mo, 2¼Cr–1Mo, 5Cr–½Mo, 9Cr–1Mo) piping systems. Carbon steels visually appear to be the same as the low-alloy steels but behave much differently in the services where low-alloy steels are specified.

**4.2 Alloy Substitutions for Carbon Steel**

When determining the need to perform material verification on carbon steel, the owner/operator 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 (e.g., stress cracking). Examples of such systems include those handling wet hydrogen sulfide (H<sub>2</sub>S), hydrofluoric acid (HF), or sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). The extent of verification should be evaluated and appropriate for the risk associated

with an unintended substitution, and greater levels of verification may be used for situations where the material substitution can lead to a failure and/or loss of containment.

### 4.3 Stainless Steel and Nonferrous Substitutions Within High-alloy Systems

There is an assortment of stainless steels with varying compositions and varying corrosion resistances to different process streams. PMI should be considered by the owner/operator depending upon the level of risk (probability and consequence of failure) based upon the potential damage mechanisms and damage rates associated with the installation of an unapproved high alloy during fabrication and installation.

Examples of such situations include, but are not limited to, the following:

- substitution of stainless steels for Alloy 400 in HF alkylation services;
- substitution of a nonstabilized grade of stainless steel for a stabilized grade in a high-temperature service;
- substitution of Type 304 stainless steel for Type 316 stainless steel where the molybdenum content is important for corrosion resistance;
- substitution of austenitic stainless steel for duplex stainless steel.

### 4.4 Residual Elements in Carbon Steels in Hydrofluoric Acid Alkylation Units

Carbon steels in some specific locations in HF acid service can experience increased corrosion rates based on the residual element (RE) content in the steels, e.g. Cr, Ni, Cu (see API 751). PMI methods can be used to assess the suitability of materials for HF service. Consideration should be given to the ability and/or accuracy 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 ( $\text{Si} < 0.10\%$ ) content can corrode at a greater rate compared with carbon steels with higher silicon ( $\text{Si} > 0.10\%$ ) content when exposed to hydrogen-free sulfidation conditions. These phenomena are discussed more extensively in API 571 and API 939-C. Owner/operators with assets at risk from this type of degradation should consider the risks and the requirements needed to apply PMI material control to verify silicon levels and the effect on predicted corrosion rate. Alternatively, the owner/operator may want to consider locating and conducting thickness measurements on all potentially susceptible carbon steel materials in a circuit exposed to hot sulfidation to determine if low-silicon components originally installed may be resulting in higher corrosion rates than the rest of a piping circuit.

### 4.6 Gasket Materials

Gaskets in incompatible service may result in premature failures. The principles outlined in this document can be applied to gasket materials. The owner/operator should define the material control methods to be used. The actual test procedures may be complex due to the construction of the gasket.

### 4.7 Refractory Installation Systems (Anchors)

Testing to verify that the refractory anchors match specifications should be considered by the owner/operator. Material specifications and maximum design temperatures for selected materials are presented in API 560.

### 4.8 Bolting Materials

When specialized bolting materials are specified for a variety of reasons, owner/operators should consider the need to add requirements to their MVP to validate that the specified bolting materials were received and installed.

One example is the use of B7M bolting where there may be susceptibility of stress cracking in service. In such cases, owner/operators should require methods of validating that B7M studs were actually received. That validation may include checking for appropriate certifications, markings, receiving paperwork, and hardness testing. Other applications may require high-alloy bolting that should also be verified as received and installed (e.g., exchanger floating head bolting and other applications of internal bolting failures could lead to significant reliability or process safety incidents).

## 5 Material Verification Programs

### 5.1 General

The owner/operator shall establish a written MVP indicating the extent and type of PMI and/or other testing and administrative methods 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/operator should consider the need for employing a higher extent of in-process and final examination (up to 100 %) rather than random sampling, which may be more appropriate for lower-risk systems. The owner/operator should also consider the need to conduct examinations after fabrication is complete at the point of installation to ensure that inadvertent substitutions did not occur.

### 5.2 Asset Components Included in an MVP

Examples of pressure-containing components exposed to process conditions that are found in equipment and systems that may require an MVP or PMI include, but are not limited to, the following:

- a) pipe lengths;
- b) pipe fittings, such as tees, elbows, reducers, caps, special pipe components, blinds, and plugs;
- c) fired heater tubes (plain);
- d) heater/cooler finned tubes;
- e) flanges;
- f) forgings;
- g) plate material;
- h) valves—process valves, control valves, and relief valves;
- i) welds;
- j) instrumentation;
- k) weld overlays, liners, and cladding;
- l) bolting;
- m) expansion joints and bellows;
- n) gaskets;
- o) rotating equipment.

### **5.3 Mill Test Reports and Usage in an MVP**

MTRs should not be considered a substitute for PMI because such documents have historically not always been accurate. However, MTRs are an important part of an overall material quality assurance program.

### **5.4 Roles and Responsibilities**

An MVP may involve participation of several groups within the operating plant or the shop of a contractor, distributor, or fabricator. When establishing an MVP, 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.

The owner/operator or designee should specify the following:

- a) extent of examination/verification of new construction and existing assets, with consideration for the number of items to be examined/verified;
- b) acceptable method(s) of examination/verification;
- c) locations for examination/verification, if applicable;
- d) examination/verification results acceptance criteria;
- e) timing of examination/verification in the work process;
- f) process for managing material nonconformances;
- g) qualification requirements for personnel performing PMI;
- h) method of documenting/identifying items acceptable for service.

### **5.5 New Construction MVP and PMI**

#### **5.5.1 General**

This section addresses MVP activities (e.g., PMI, MTR review, comparison to owner/operator specifications, product markings, certifications, etc.) during or after the fabrication stage, either in receiving, in the shop, or in the field, prior to the items being placed into service.

#### **5.5.2 Material Verification Test Procedure Review**

The owner/operator or designee should review and approve the MVP requirements and the testing procedure(s) of the fabricator, material supplier, or third-party agency prior to fabrication.

#### **5.5.3 Timing of Material Verification During Receiving or Fabrication**

MVP and PMI should be performed at the point in time that helps determine that proper materials and materials quality assurance/quality control documentation have been conducted and/or verified during the fabrication of a component or assembly.

#### **5.5.4 Positive Material Identification of Components Supplied by a Distributor**

A higher degree of MVP activities, including PMI, should be conducted on materials supplied by stocking distributors due to the potential for unapproved material substitutions as a result of frequent handling by several parties.

## 5.6 Retroactive PMI of Existing Installed Assets

### 5.6.1 General

This section addresses assets that are already in service where the MVP procedures for the construction are not documented or not completed according to this recommended practice. It is important to recognize that previous maintenance activities, as well as new construction practices, may influence the likelihood of unapproved materials substitutions.

### 5.6.2 Prioritizing Assets for Retro-PMI

#### 5.6.2.1 General Factors to Consider

If the owner/operator elects to prioritize equipment or systems for the MVP or needs to determine whether PMI is needed at all, the owner/operator should consider the following.

- a) Likelihood of unapproved material substitutions during previous projects and maintenance activities—The effectiveness of the MVP when these activities occurred is an important consideration.
- b) 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.
- c) Reason for a specific material specification (i.e., corrosion resistance or product purity).
- d) Historical data/information relating to unapproved material substitutions—This may be related to previous experience with material nonconformities in the process unit/operating plant or within published information available within the company and industry.

Taken together, these factors can be used to determine the risk associated with possible material nonconformances in an asset.

The owner/operator should establish a methodology for estimating the relative priority for retro-PMI within a given process unit. This methodology may be based on a qualitative or quantitative risk analysis. API 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). The owner/operator may also want to consider the opportunity to conduct PMI relative to upcoming planned maintenance opportunities (e.g., outages, turnarounds).

#### 5.6.2.2 Site-specific Factors to Consider for Retro-PMI

Site-specific and/or experience-based factors should be considered when prioritizing equipment or piping systems.

- Construction and maintenance practices—In assessing the likelihood of material nonconformances, the owner/operator should also consider the materials handling, material control, and any PMI 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 nonconformances.
- Reason for alloy specification—In some cases, alloys are used in equipment systems for reasons other than corrosion resistance or structural integrity. In these cases, the mechanical integrity of the system may not be compromised by material nonconformances. A retro-PMI may not be necessary in these systems. Two examples would be stainless steel lube oil systems in which stainless steel is used for maintaining oil purity or stainless steel in a chemical manufacturing process where corrosion of carbon steel might cause product discoloration where any grade of stainless steel would be an acceptable substitution.

Based on experience, some types of components can have a higher likelihood of unapproved substitution of a nonspecified material. This can provide a basis for prioritizing specific equipment in a given system or process unit. Examples are as follows:

- a) warm-up and bypass lines on pumps or check valves;
- b) small-bore piping (2 NPS and below);
- c) valves, valve assemblies, and valve bonnets;
- d) removable devices such as rupture disks, spacer blinds, blind flanges, plugs, or ring joint gaskets;
- e) thermowells;
- f) bolting;
- g) piping as a part of a packaged system;
- h) components without recognized marking;
- i) process systems with history of frequent maintenance requirements;
- j) welds;
- k) threaded components.

### **5.6.2.3 Factors to Consider When Determining the Extent of PMI**

Factors to consider when determining the extent of PMI for existing assets include the following:

- a) historical inspection and MVP records;
- b) number of plant modifications;
- c) material control during original construction, equipment modifications, and maintenance activities;
- d) MVP quality during construction and fabrication;
- e) failure mode and consequence of a loss of containment;
- f) likelihood of corrosion/degradation.

## **5.7 MVP as an Element of Maintenance Systems**

### **5.7.1 General**

The principles associated with materials verification as part of an installation of new equipment shall also be applied as part of maintenance activities to provide confidence that proper materials are being installed. The roles and responsibilities noted in [5.4](#) should be reviewed and applied as applicable to the maintenance function.

### **5.7.2 Responsibilities**

It shall be the responsibility of the owner/operator to evaluate maintenance systems so that MVPs can be designed and implemented to effectively support the mechanical integrity needs of existing assets. The owner/operator shall establish a documented procedure for the MVP to be used for repair of existing assets during maintenance and turnaround activities.



### 5.7.3 Control of Incoming Materials and Warehousing

An MVP should be directly applied to activities associated with receiving materials into a warehouse system. PMI 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 MVP that is adopted should provide for proper documentation and methods for indicating which materials have been PMI tested and are approved for use.

The use of MVP 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. Any identification within the warehouse should not be regarded as an alternative to the supplier's certified PMI of the fabricated assets when specified.

### 5.7.4 PMI of Welding Consumables

#### 5.7.4.1 General

Performing PMI of a weld cap does not ensure that the root pass or subsequent weld passes are made with the specified chemistry. An acceptable method to address in-process PMI during welding is explained as follows.

- Prior to use in fabrication, sample "buttons" should be welded using each heat of bare wire, lot of covered electrodes, or flux-cored electrodes. PMI can then be used to confirm that weld metal meets specification. The size of the weld button should be adequate to ensure accurate test results.
- Some weld rods have the alloying elements contained in the flux and do not meet the alloy specification until welded.
- It is not necessary to test a button of bare wire if the test chosen can identify the composition of the wire before welding.

#### 5.7.4.2 Longitudinal Pipe and Fitting Welds

Where there is reason to suspect incorrect weld metal or that such welds are included in a scheduled PMI effort, longitudinally welded alloy pipe and the welds for fittings in pipe systems should receive PMI verification of the weld metal in addition to the base metal. Typically, only the cap pass is subject to PMI that will not necessarily apply to the root and subsequent passes.

#### 5.7.4.3 Autogenous Welds

If the owner/operator determines that material verification testing is required on autogenous-welded (with no added filler metal) alloy pipe or fittings, it is appropriate to conduct testing on only the base metal. If repairs are made during original fabrication, the fabricator should be directed by the owner/operator to conduct testing on the base metal and the weld metal involved in the repairs (e.g., for type 316 stainless steel that may include Ni, Cr, and Mo).

#### 5.7.4.4 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/operator should establish the minimum compositional requirements of the as-deposited weld metal necessary for the intended service.

### 5.7.5 MVP for Maintenance Activities

There are several in-service maintenance activities where material verification should be established. Some examples include, but are not limited to, the following.

- a) Temporary removal of piping spool pieces, including the removal of blind flanges used for access: These activities need to be properly managed to minimize the potential for unapproved material substitutions.



Incidents have occurred when same-size spool pieces were removed and reinstalled in the wrong locations. Consideration should be given to a material control system, such as “tagging” spools as they are removed, or the use of PMI prior to reinstallation to prevent these incidences from occurring.

- b) Replacement of small-bore threaded pipe nipples and plugs, frequently found as drains and vents in process areas: An immediate need may necessitate the 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.
- c) Replacement of welded-in valves: Manufacturers/distributors may substitute low-alloy valves for carbon steel valves in higher-temperature/pressure services, and if this is not communicated, the craftsmen may end up using the incorrect welding procedure during installation. This error may produce cracks in the new welds.
- d) In turnaround situations where many heat exchangers in varying services are disassembled for cleaning, inspection, and repair: It is essential that all original components, or correct replacement components, are returned to the same exchanger during reassembly. An adequate marking and tracking system, as well as PMI, can be used to ensure that the proper components are returned to the correct service.
- e) When tower internals, such as tray parts (e.g., clips, tray flapper valves or bubble caps, and fasteners) are replaced, one may consider performing point-of-installation PMI (or other material control program) to ensure that the replacement parts are as specified.

It is important that repair procedures include consideration of PMI and appropriate other aspects of MVP and quality assurance/quality control to help ensure that the right materials are always used. Much of this can be controlled through awareness of the issues at all levels within the repair process. Consulting with those involved with implementing the programs where the repair will be performed, prior to commencement of work, can help ensure that systems, processes, and activities are in place to provide material control and verification.

## **6 Positive Material Identification Methodology and Technology**

### **6.1 General**

A variety of PMI 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 recommended practice, including magnetic testing to differentiate between ferritic and austenitic materials. It is important that users define the objectives and accuracies required of the PMI tool they wish to apply. All of the tools have benefits and limitations on the elements that can be detected, in addition to the accuracy and ability to differentiate between different material grades that have only slight variations in alloying elements. (See [Annex A](#).)

### **6.2 MVP Test Method Objectives**

The test methods outlined in this document are intended to identify the grades of materials and are not necessarily intended to establish the exact conformance of a material to a particular specification. However, some PMI methods are capable of establishing conformance of materials. It is important that the owner/operator establish the purpose and objectives of the MVP, including PMI, before the technology and methodology are selected. Depending on the test method selected, the method may identify only the nominal composition of materials. Identification of materials by visual stamps/markings alone should not be considered as a substitute for PMI but may be an important component of an overall quality assurance program.

### **6.3 PMI Procedure**

The PMI procedure should include the following.

- a) The techniques to be applied; see [6.4](#).
- b) Equipment calibration.
- c) Personnel qualifications:

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

The owner/operator is responsible for ensuring that all individuals performing PMI are trained and qualified in accordance with the applicable procedure approved and applied during the examination. Certification and qualification are two of the most effective aspects of the PMI procedure to enhance the likelihood that all requirements of the PMI procedure will be met.

- d) Surface preparation requirements.
- e) Testing methodology applied.
- f) Acceptance criteria.
- g) Documentation requirements.

## **6.4 Sorting Analysis Techniques**

### **6.4.1 Resistivity Testing**

The principle employed in this 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 are not widely used and have not proved to be consistently capable of sorting low-alloys (< 5 % Cr) and austenitic stainless steels.

### **6.4.2 Other Alloy Sorting Techniques**

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

## **6.5 Spectrometer Technology**

### **6.5.1 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 that 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:

- as a match against one of many reference spectra stored in the instrument (i.e., “316 Stainless Steel” or “5 Cr-½Mo Steel”), and/or
- each element present 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 %).

Several PMI instruments are available. These instruments can 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 XRF analyzers now measures light elements (Mg, Al, Si, P, and S) to very low limits of detection (see [Annex A](#)) and can provide for field measurement of silicon in steel down to very low concentrations.

These advancements in technology allow for accurate, nondestructive measurements to be conducted. However, to get this near-laboratory-quality measurement, significant care regarding surface preparation and cleanliness is required, as contamination of the prepared surface can lead to inaccurate results. 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, minimum zirconium-aluminum-oxide disposable abrasive disk) to properly clean the sample location prior to analysis.

The inherent limitations of the technique mean that 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 excludes some of the important elements in carbon steels, such as carbon and boron. It is important to define exactly what elemental analysis is required and select an appropriate instrument.

### 6.5.2 Portable Optical Emission Spectrometry

In optical emission spectrometry (OES), an electric arc, spark, or laser 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 three groups.

- 1) The first is a lightweight, portable, and operator-evaluated device that can typically identify up to 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—which 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 lightweight, including the small argon cylinders required for operation. Some of these analyzers operate in a pure arc mode for routine PMI applications, whereas 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. Another advantage of these advanced instruments is that they are not subject to operator interpretation.

Similar to XRF 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; therefore, prior to use of this technique in the field, a review shall be conducted to determine if gas testing and hot work permits are required.

- 3) The third group is known as laser-induced breakdown spectroscopy (LIBS), in which an atomic emission spectroscopy technique uses highly energetic laser pulses. The laser is focused to form 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.

Considerable progress has been made during the past few years in LIBS technology. Handheld LIBS metal analyzers are becoming available that can provide fast in situ chemical analysis with appropriate precision and detection limits (see [Annex A](#)). 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 and 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.

**NOTE** The accuracy of carbon equivalent (CE) elements is highly dependent upon calibration within the range of interest, especially to minimize bias. So, it is advisable to perform a "Type Cal" on the upper and lower bounds of the anticipated elemental CE boundaries.

This technique generates a laser during operation, which is a potential ignition source. Therefore, before using this technique in the field, a review shall be conducted to determine if gas testing and hot work permits are required.

### 6.5.3 Equipment Calibration

Persons performing PMI should calibrate and/or verify the test equipment performance as specified by the equipment manufacturer. The PMI 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/operator. Typically, these procedures should include calibration/verification using certified standards. The owner/operator may consider requirement of equipment verification checks on a routine basis (e.g., once per shift) using certified standards of the same alloy family.

### 6.5.4 Equipment Precision

The precision of the test equipment should be consistent with the established test objectives (see [6.2](#)). When elemental composition is desired, the owner/operator should establish the acceptable precision and repeatability.

Accuracy and the method in which it is determined need to be understood (see [Annex A](#)). For example, in some tools, the sensitivity may depend on duration of the test to improve signal averaging algorithms. Failure to understand these issues may produce inaccurate results.

## 6.6 Chemical Analysis Techniques

### 6.6.1 Laboratory Chemical Analysis

Owner/operator-approved material analysis laboratories using X-ray emission spectrometry, OES, 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 but may be necessary where field PMI techniques cannot accurately quantify small amounts of alloying or contaminating elements (e.g., C, S, Si, V, Cu, etc.). Laboratory analysis may involve the removal of significant amounts of material and is typically slower than field PMI 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 (e.g., this test is sometimes used to verify that an austenitic stainless steel contains molybdenum and is more likely to be Type 316 as opposed to Type 304). Chemical spot testing is much slower than the other field PMI methods, and interpretation is subjective and very qualitative.

## 6.7 Safety Issues

### 6.7.1 General

The specific requirements for each PMI 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.

## 6.7.2 XRF Analyzers

### 6.7.2.1 Intrinsic Safety

XRF analyzers are not intrinsically safe. Pertinent hot work procedures should be followed in areas where nonintrinsically safe equipment is to be used.

### 6.7.2.2 Radiation Safety

The user should be aware of potential radiation exposure hazards when operating an XRF device. The user is expected to observe local jurisdictional requirements that govern radiation safety and consult the manufacturer's instructions for safe operation.

## 7 Evaluation of Testing Results

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

- a) Materials can be confirmed to contain the nominal amounts of alloying elements specified in the relevant materials specification (e.g. ASME BPVC Section II or ASTM specifications).
- b) Materials can be classified through a qualitative sorting technique (see [6.4](#)) to establish the conformance with the intended material.
- c) When PMI indicates that alloying elements are outside the ranges indicated in the material specification, the owner/operator 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.
- d) 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](#)).
- e) When the test results indicate an unacceptable material, then the material involved is rejected and replaced with the correct material.
- f) 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 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 as follows:

- a) color coding by alloy;
- b) a low-stress stamp marking indicating that the test has been performed;
- c) documentation showing both the PMI results and the PMI locations.

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

## 8.2 Color Coding/Marking

**8.2.1** If the MVP procedure established by the owner/operator requires a visual identification such as color coding or marking, the owner/operator should maintain a record of the alloy material/color code combinations. PFI ES-22 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 have their eyesight tested to confirm that they are able to distinguish the difference between the colors being used (i.e., not be color-blind).

## 8.3 Marking of Components

If the owner/operator's documentation process requires physical marking of equipment components, it should specify one of the following:

- the marking system should remain legible for the expected life of the component without deterioration due to corrosion or elevated temperature;
- the marking system is only temporary to facilitate proper handling and identification from the point of PMI to final installation; this marking can be semipermanent paint applied to each item. The markers should not contain additives such as metallic pigments (Al, Pb, or Zn), sulfur, or chlorides.

# 9 Documentation and Recordkeeping

## 9.1 Shop and Field PMI Documentation

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

When documentation (e.g., drawings) is used in lieu of physical marking, the documentation should allow the owner/operator to identify which components were tested.

## 9.2 New and Existing Equipment or System Documentation

When PMI 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 a portion of an equipment system that has not received material verification is relocated, the owner/operator should consider the need for PMI prior to placing the relocated components into service.

Where applicable, master PMI piping isometric drawings to track the components tested and test results may be considered. Since a single documentation system eliminates the need to search for data in multiple locations, such drawings would best be maintained and updated as piping modifications occur from projects or process changes.

## 9.3 PMI Records

Typical PMI records should contain the following:

- reference to the PMI procedure(s) used;
- date of testing;
- test instrument identification number or serial number, where appropriate;



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- name of each person performing the tests, with identification of their respective employer;
  - results of the tests;
  - basis and action for resolving and documenting PMI nonconformances, including those that have been left in service;
  - documentation of the criteria used for prioritization of equipment or systems and extent of PMI performed.

The information listed should be reported in such a manner that it is traceable to the point of installation.

Alternatively, the owner/operator may choose to include this within the written material verification procedure. When included in the owner/operator'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 some of the terms regarding statistics that may be encountered when using the methodology and/or technology discussed within this document. PMI inspectors should understand the listed terms to properly appreciate what each of the analyzer types (XRF, OES, LIBS) is capable of measuring to produce accurate results. Understanding these will help the PMI inspectors accomplish their goals in performing PMI for the owner-user's MVP.

- Accuracy—The closeness of agreement between the test result and the accepted reference value.
- Precision—The closeness of agreement between independent test results. The precision depends only on the distribution of random errors and does not relate to the accepted standard certified value.



Figure A.1—Illustration of Statistical Accuracy

- 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 definition for standard deviation 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 quantitation (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 are 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 are within one standard deviation of the mean; 95 % of the data are within two standard deviations of the mean; and 99.7 % of the data are within three standard deviations of the mean.
- Relative standard deviation—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.



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- 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.

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<sup>3</sup> Center for Chemical Process Safety, 120 Wall Street, Floor 23, New York, New York 10005, [www.aiche.org/ccps](http://www.aiche.org/ccps).

<sup>4</sup> Chemical Safety Board, 1750 Pennsylvania Avenue, NW, Suite 910, Washington, DC 20006, [www.csb.gov](http://www.csb.gov).

<sup>5</sup> National Association of Corrosion Engineers (now the Association for Materials Protection and Performance), 15835 Park Ten Place, Houston, Texas 77084, [www.nace.org](http://www.nace.org).

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