NONMANDATORY APPENDIX B
MATERIAL AND WELD EXAMINATION/INSPECTION DOCUMENTATION

See Forms MEL-1, MER-1, and WEL-1 beginning on next page.

Form MEL-1 Material Examination Log

Project Number/Name: _____ From (Supplier or Manufacturer): _____

A/E Project Number: _____ Date Received: _____

Received by (Company): _____ Person Receiving Material (Printed): _____

Staged Location of Material: _____ Person Receiving Material (Signature): _____

List only material received from a single source.

Manufacturer	Material or Alloy Type	Sample Description	Heat No. or Lot No.	Wall Thickness	O.D. Tolerance	Surface R_a	Visual [Note (1)]	Product Marking [Note (2)]	MTRs Verified	Quantity Received	Quantity Examined	Qty. Accept Qty. Reject	Examiner's Initials	Date Examined	NCR Number

Comments: _____

GENERAL NOTES:
(a) "A" or Acc. indicates a conformance with the applicable sections of Part DT.
(b) "R" or Rej. indicates a nonconformance with the applicable sections of Part DT.

NOTES:
(1) Per applicable sections of Part SF.
(2) Markings verified to be in accordance with DT-11.

☐ Attachments: ( ) Pages Approving Supervisor: _____ Date: _____

Copies: ☐ Owner ☐ A/E ☐ Contractors ☐ Consultants ☐ _____ ☐ _____ ☐ _____ ☐ _____ ☐ _____ ☐ File

BPE **Form MER-1 Material Examination Record**

Project Number/Name: _____ A/E Project Number: _____

Record Number: _____ Received by: _____ Date Received: _____

Owner/User: _____ Name and Date of Approval: _____

Customer Company Name: _____
(Enter the name of the company receiving the material.)

Address: _____
(Enter the address of the company receiving the material.)

Contact Names and Numbers: _____
[Enter the name(s) and contact information of personnel receiving the material.]

Supplier/Manufacturer Name: _____
(If receiving a product or material, enter the name of the company supplying the material.)

Address: _____
(If receiving a product or material, enter the address of the company supplying the material.)

Contact Information: _____
[If receiving material, enter the name(s) and contact information of personnel supplying the material.]

Project Information: _____ NCR Number: _____
(Related Specifications, Codes, and Standards) *(NCR report number if needed)*

Heat Number/Heat Code: _____ Material Specification: _____
[Record heat number(s) for the sample.] *(ASTM spec., customer spec.)*

P.O. Number: _____
(Enter associated purchase order number here.)

Packing List Number: _____
(Enter packing list and/or tracking number here.)

Lot Number: _____
(For multiple lot shipments, enter associated lot number here.)

Examiner's Information: _____
(Enter the name of the examiner, company of examiner, etc.)

Material/Alloy Type: _____
[Enter the type or grade of material (UNS S31603, N08367, etc.)]

Material Description: _____
[Enter the size, material product form (tubing, 90, 45, TEE, ferrule, valve, etc.)]

DT-11 Compliant: _____
(Record Accept or Reject after markings verification.)

Wall Thickness: _____
[Record Accept or Reject after physical examination of the lot (if required).]

O.D. Tolerance: _____
[Record Accept or Reject after physical examination of the lot (if required).]

Surface R_a: _____
[Record Accept or Reject after physical examination of the lot (if required).]

Visual Examination: _____
(Record Accept or Reject after physical examination.)

MTR(s) Verified: _____
(Record Accept or Reject for MTR compliance with specifications.)

Quantity Received: _____ Quantity Accepted: _____ Quantity Rejected: _____ Date Examined: _____

Comments: _____
(Record any notes for examination, and attach additional sheets if needed.)

Copies: ☐ Owner ☐ A/E ☐ Contractors ☐ Consultants ☐ _____ ☐ _____ ☐ File

Form WEL-1 Weld and Examination/Inspection Log

Project Number/Name: _____ Fabricating Contractor: _____

A/E Project Number: _____ Isometric/Drawing Number: _____

Owner/User: _____ Name and Date of Approval: _____

Weld Log				Examination				Inspection				
Weld No.	Weld Date	Welder or Welding Operator ID	Size	Type [Note (1)]	Accept/ Reject	Initials	Date	Type [Note (1)]	Accept/ Reject	Initials	Date	Comments [Note (2)]/Reject Cause

GENERAL NOTES:

(a) Welds shall be uniquely identified per applicable drawings.
(b) Rejected welds that can be rewelded shall use the same number with an "R" after it.
(c) Rejected welds that must be cut out and replaced with a new weld shall be assigned the same identifier followed by an "A" for the first, "B" for the second, etc. in consecutive order.
(d) If a weld is cut out for a reason other than a rejected weld, it shall be recorded as a "Deleted" weld in the Comments section. The replacement weld shall be assigned the next available identifier.
(e) Coupon welds may be listed with production welds or listed separately. Either way, the coupon weld should be identified with the symbol "CPN" as either a prefix or a suffix to the user's weld numbering format.

NOTES:

(1) VO = Visual O.D. only; VI = Visual O.D. and I.D.; B = Visual O.D. and Borescope I.D.
(2) Blind welds shall be indicated as "Blind" in the Comments section. Manual welds shall be indicated as "Manual" in the Comments section. Videotaped welds shall be indicated as "Video" in the Comments section. Any other requirements may be indicated in the Comments section.

Copies: ☐ Owner ☐ A/E ☐ Contractors ☐ Consultants ☐ _____ ☐ _____ ☐ _____ ☐ _____ ☐ _____ ☐ File

NONMANDATORY APPENDIX C
SLOPE MEASUREMENT AND JOINT MISALIGNMENT

C-1 GENERAL

(a) Slope measurement shall be made with a digital level or a digital protractor. The instrument used should be capable of displaying slope in degrees, percent, and in./ft (mm/m).

(b) Refer to the owner's manual for the proper procedure to perform the self-calibration routine. This must be performed immediately prior to use.

(c) Slope measurements shall only be made under the following conditions:

(1) before insulation has been installed

(2) after hangers/pipe supports have been installed, adjusted, and fixed in place

(3) before the introduction of any fluids, such as liquids or process gases (pure oxygen, nitrogen, steam, etc.)

(4) when the system is at ambient pressure and temperature

(d) For piping or tubing systems, slope measurements shall be made at the following locations:

(1) between hangers/pipe supports

(2) at each change in direction

(3) at any other location deemed necessary by the inspector, such as between welds or any apparent change in slope

(e) Slope should be measured only on runs that are approximately horizontal.

(f) Slope measurements may be made on either the top or bottom of the tubing/piping.

(g) For slope measurements made on skids or modules, ensure that the base is level in all directions. Then, make sure that all slope measurements are made relative to the base.

(h) Slope shall be verified after the fabricator has completed, or corrected, the piping installation and set the slope.

C-2 JOINT MISALIGNMENT

In order to meet O.D. misalignment criteria in this Standard, the accumulated tolerances in piping, tubing, and fittings may result in a welded joint with an I.D. misalignment. Should this occur, the owner/user, installation contractor, and inspection contractor shall apply good engineering judgment to determine the best solution for the application considering flow, orientation, and drainability.

The orientation of the piping, tubing, or fittings should be considered prior to final disposition of the weld joint prior to welding.

(a) Vertical Orientation

(1) Misalignment should be uniformly distributed around the circumference.

(2) Direction of flow should be considered when assembling the components.

(b) Horizontal Orientation. *Horizontal Orientation.* Misalignment should be oriented to maximize drainability, normally accomplished by minimizing the I.D. misalignment at the bottom.

NONMANDATORY APPENDIX D
ROUGE AND STAINLESS STEEL

D-1 GENERAL

This Appendix provides methods to measure rouge in a system both in the process solution and on the actual product contact surface. It also suggests various fabrication and operation practices to minimize rouge formation and methods/techniques for its remediation.

For the definition of rouge and its classification, see GR-8.

D-2 CONSIDERATIONS FOR REDUCING ROUGE FORMATION

Tables D-2-1 and D-2-2 provide guidance on different variables and how they may contribute to the presence of rouge in a high-purity system. They are listed in the following categories:

(a) Category 1: Little Influence on the Formation of Rouge. There are theories that suggest other factors that may have a role in the formation of rouge. These variables are not listed in Tables D-2-1 and D-2-2.

(b) Category 2: Moderate Influence on the Formation of Rouge. There is industry data supporting these variables, and they should be considered.

(c) Category 3: Strong Influence on the Formation of Rouge. There is well-established industry data supporting these variables, and they should be considered.

D-2.1 System Fabrication

See Table D-2-1 for a discussion of fabrication variables that affect the amount of rouge formation.

D-2.2 System Operation

See Table D-2-2 for a discussion of operation variables that affect the amount of rouge formation.

(19) D-3 EVALUATION METHODS TO MEASURE ROUGE

Rouge can be measured by its presence in the process fluid and/or its presence on the process contact surface.

D-3.1 Process Fluid Analyses

Fluid analyses provide a means of identifying the mobile constituents within a subject process system. They represent the current quality status of the media and the result of rouging.

Table D-3.1-1 provides descriptions, pros, and cons of various tests for the identification of mobile constituents.

D-3.2 Solid Surface Analyses

Surface analyses provide information on the nature, microstructure, and composition of surface layers. They may represent the future status of the media and the possible threat of rouging to the water quality.

Table D-3.2-1 provides descriptions, pros, and cons of various tests for the identification of the composition of surface layers.

D-4 METHODS TO REMEDIATE THE PRESENCE OF ROUGE IN A SYSTEM

Remediation (derouging) processes are designed to remove iron oxide and other surface constituents of rouge while minimizing damage to the surface finish. Rouge occurs on the surface, from corrosion, or precipitates onto the surface after migrating from other locations. These conditions are easily categorized by using the standard Classes I, II, and III rouge. The following sections describe remediation processes and the conditions under which they are performed.

D-4.1 Class I Rouge Remediation (19)

Class I rouges are weakly attached to the surface and are relatively easily removed and dissolved. This rouge is generally hematite or red ferric iron oxide with low levels of other oxides or carbon content. Phosphoric acid is useful to remove light accumulations and may be blended with other acids and compounds including citric, nitric, formic, or other organic acids and surfactants to assist in its derouging effectiveness. Citric acid–based chemistries with additional organic acids are effective at rouge removal. The use of sodium hydrosulfite (i.e., sodium dithionite) is also fast and effective at removal of Class I rouge.

These chemistries are processed at elevated temperatures from 104°F to 176°F (40°C to 80°C) for between 2 hr and 12 hr. The process time and temperatures may depend on the severity of rouge accumulation, the system component's material of construction, and the concentration of chemistries. The concentration of each chemistry is based on proprietary testing and process design criteria.

Electrochemical cleaning is an alternative method of rouge removal that uses phosphoric acid and applied direct current where the process contact surface is anodic. As a cathode is moved over the process contact surface to be cleaned, rouge is readily removed. This process is very effective in removing all three classes of rouge but is limited to accessible parts of a system and is primarily performed on the product contact surfaces in vessels.

For specific Class I rouge remediation processes, refer to Table D-4.1-1.

D-4.2 Class II Rouge Remediation

Class II rouge consists mostly of hematite or ferric iron oxide with some amount of chromium and nickel oxides as well as small carbon content. It is removed with chemistries that are very similar to the above processes with the addition of oxalic acid, which improves the effectiveness in removal of this type of rouge. All of the above chemistries remove the rouge without damage to the surface finish with the exception of oxalic acid, which may etch the surface depending on conditions and concentration processed. Class II rouges are more difficult to remove than Class I and may require additional time, even though these processes are often run at slightly higher temperatures and increased concentrations.

For specific Class II rouge remediation processes, refer to Table D-4.1-1.

D-4.3 Class III Rouge Remediation

Class III rouge is much more difficult to remove compared to Class I and Class II rouge, due to both its chemical composition difference and its structural difference. These high-temperature deposits form magnetite iron oxide with some substitution of chromium, nickel, or silica in the compound structure. Significant amounts of carbon are generally present in these deposits due to reduction of organics present in the water, which sometimes produces the “smut” or black film that may form during derouging. The chemistries used to remove Class III rouge are very aggressive and will affect the surface finish to some degree. Phosphoric acid-based derouging systems are generally only effective on very light accumulation of the rouge. The strong organic acid blends with formic and oxalic acid are effective on some of these high-temperature rouges, and, being less aggressive, they produce much less potential for etching of the surface finish.

Citric and nitric blends with hydrofluoric acid or ammonium bifluoride will remove these Class III rouges more quickly but will definitely etch the surface wherever the base metal is subjected to the derouging fluid. The amount of etching or increase in surface finish roughness is dependent on process conditions, chemical concentration, and variability of the rouge thickness and level of surface finish roughness initially. The condition of use for these processes is highly variable in both temperature and time required to effectively remove all of the rouge and leave the surface prepared for cleaning and passivation. The less-aggressive chemistries are used at higher temperatures [140°F to 176°F (60°C to 80°C)] and require longer contact time (8 hr to 48-plus hr); the nitric acid-based fluoride solutions are often used at lower temperatures [ambient to 104°F (40°C)], while the citric acid-based fluoride solutions are used at higher temperatures and shorter contact times (2 hr to 24 hr).

For specific Class III rouge remediation processes, refer to Table D-4.1-1.

D-4.4 Remediation Variables (19)

The times and temperatures given in D-4.1 through D-4.3 are in direct relation to the percent by weight of the base reactant(s). A change in a formulation will change the corresponding requirements. Different application methods include fluid circulation, gelled applications for welds or surfaces, and spraying methods for vessels and equipment. Rinsing of the surface after processing as well as proper waste disposal planning is critical to the derouging process. The waste fluids generated by these processes can be classified as hazardous due to chemical constituents or heavy metals content.

Rouge can effectively be removed from process contact surfaces to reduce the potential for oxide particulate generation into the process fluids. These derouging processes are required prior to proper cleaning and passivation of the stainless steel surface for restoration of the passive layer after corrosion. Analytical testing of utility fluids may be useful in identifying the level of particulate generation and levels of metal oxides contained in these fluids as corrosion degrades the surface.

(19) **Table D-2-1 Considerations That Affect the Amount of Rouge Formation During the Fabrication of a System**

Variables	Considerations
Category 3 — Strong Influence on the Formation of Rouge [Note (1)]	
Alloy selection	Selection of the proper alloy (e.g., 316L-type or 6 moly-type stainless steel) should address the corrosive effects of the process conditions. For example, low-carbon stainless steel (316L-type) has better corrosion resistance than higher-carbon stainless steels (316-type). Beneficial alloys can mitigate premature or accelerated corrosion. Higher nickel content will enhance corrosion resistance.
Mechanical polishing/buffing	Striations from cold working techniques may include residual grinding/polishing debris in lapping inclusions. Cumulative increase of interior area due to surface finish inconsistency proportionally exposes more alloy to the mechanisms of corrosion and burden of passivation.
Electropolishing	Minimizes the exposure area of the alloy to oxidizing fluids or halides and minimizes the origins for micropitting by corrosion mechanisms.
Passivation	Impedes or retards corrosive development of stainless steel surfaces. The effectiveness of passivation methods in terms of depth and enhancement of surface alloy ratios (i.e., chrome to iron) determines the eventual propensity of an alloy to corrode and the rate of corrosion.
Alloy composition	(% molybdenum, chromium, nickel, etc.) The microstructure quality affects precipitation of impurities at grain boundaries. Migration of impurities to the alloy surface can either support corrosion cells or seed downstream corrosion. Weld joints on tubing and/or components with dissimilar sulfur concentrations may result in lack of penetration due to weld pool shift. The resulting crevice may become a corrosion initiation site.
Welding, welding conditions, purging, etc.	Improper welds can result in chromium-depleted heat-affected zones (HAZs) and other conditions that reduce corrosion resistance. Weld discontinuities create opportunities to trap fluid-borne impurities. Cracks resulting from poor welds will create breaches in the passive layer and form active corrosion cells. Proper purging prevents weld contamination by heat tint oxides and the concurrent loss of corrosion resistance. Passivation cannot reverse the effects of improper purging.
Product form and fabrication methods	The ferrite content can be greatly affected by the forming process (e.g., a forging will typically have much lower ferrite percentages than a casting). Barstock endgrain voids at the surface can enhance the potential of the alloy to pit and corrode. Minimization of differences in sulfur content will enhance the potential for successful welding.
Category 2 — Moderate Influence on the Formation of Rouge [Note (1)]	
Installation/storage environment	Unidentified corrosion due to the storage or installation environment, including carbon steel contamination, scratching, exposure to chemical contaminants, stagnated condensation or liquids, etc., may warrant a derouging step prior to passivation. Failure to detect instances of corrosion will marginalize the effect of a normal passivation.
Expansion and modifications to an established system	Oxide formations in newly commissioned systems can form at different rates than in older systems and initially generate migratory Class I rouge. Where oxide films exist in established systems, they are likely to be more stable, producing less migratory iron or chrome oxides. Because the newer system can generate and distribute lightly held Class I migratory hematite forms throughout the system, the corrosion origin and cause can be difficult to identify.

NOTE: (1) There is well-established industry data supporting this, and it needs to be considered.

Table D-2-2 Considerations That Affect the Amount of Rouge Formation During the Operation of a System

Variables	Considerations
Category 3 — Strong Influence on the Formation of Rouge [Note (1)]	
Corrosive process fluid (bleach, halides, etc.)	Corrosion cell inceptions at breaches in the passive layer, as in chloride corrosion cells, will progressively catalyze the corrosion mechanism. This has a very strong influence for applications such as high-salt buffer tanks, etc.
High shear/velocity environment (pump-impeller, sprayball, tees, etc.)	Erosive forces deplete or erode the passive layer and introduce base metal composition particles to the remainder of the system. Severe instances can cause pitting on the tips of pump impellers or fluid impingement spots on vessel walls. In pure steam systems, high-velocity sections can scour tubing walls, either preventing sustained buildup of stable magnetite layers or sloughing off fragments from developing oxide formations that are then transported downstream for possible corrosion seeding.
Operating temperature and temperature gradients	Operating temperature and temperature gradients will affect the eventual nature of oxide formations (e.g., Class I hematite versus Class III magnetite), the ease of removal, the propensity to become stationary, stable, or lightly held and migratory. The nature of restoration by passivation and derouging may be largely determined by the operating temperature of the system. Established magnetite formations in pure steam systems may require a derouging step prior to the passivation steps.
Gaseous phase composition, including dissolved gases (O_2 and CO_2)	For monographed fluids (PW, WFI, and pure steam), the constituency of dissolved gases is generally believed to have an influence on rouge formation when within established conductivity and total organic carbon (TOC) limits in systems that have an adequate passive layer. It is possible for impurities to migrate across distillation and pure steam generation processes as dissolved gases. A variety of analytic spectrometry methods are available to identify these species. (Refer to Tables D-3.1-1 and D-3.2-1.)
Application, process media (pure steam, WFI, buffer, media, CIP, etc.), frequency of operation	The nature of the oxide formation, potential for corrosion, remedial methods, and period of formation are greatly influenced by the application as noted in the other impact descriptions (temperature, corrosive process, etc.). In steam-in-place (SIP) systems, velocity, temperature, and trapping can have impacts on the composition and locations of rouge formations and migratory deposits. Adequately designed systems can minimize this impact. Poorly trapped pure steam headers, regularly exposed to pressure gradients, can introduce corrosion mechanisms and products through steam condensate. Long hold periods in high-salt buffer tanks and the effectiveness of the tank agitation can promote or accelerate rouge formation. SIP following inadequate CIP can create corrosion mechanisms and further aggravate removal methods.
System CIP, cleaning methods	Exposure to CIP cycles and the specific chemical cleaning solutions strongly affects the potential for rouge occurrence. System sections exposed to a cyclic CIP regime will be less likely to form or collect rouge. Significant factors include whether there is an acid or hot acid CIP cycle in the CIP recipe. The duration and temperature of the acid cycle can be important. Acid cycles with mild concentrations (e.g., 2% to 20% phosphoric acid) have been shown to maintain and restore passive layers.
Redox potential	The use of ozone to sanitize purified water or WFI systems has also demonstrated beneficial effects in impeding alloy corrosion.
Maintenance of the system	System components such as worn pure steam regulator plug seats, improper or misaligned gaskets, worn regulator and valve diaphragms, pump impellers (with worn tips), and eroded or cracked heat exchanger tube returns are believed to be sources of Class I rouge.
Stagnant flow areas	A moving oxidizing fluid can maintain the passive layer. (Studies with nitrogen-blanketed WFI storage tanks have shown negative effects on passive layers as a result of minimizing oxygen in the fluid.) Liquid condensate that is not immediately removed from a pure steam conduit or that collects from improper valve sequencing can concentrate and transport surface oxidation products or steam contained solubles. These can concentrate and deposit at a branch terminus such as a vessel sprayball, dip tube, etc. These deposits are typically lightly held forms of hematite. Though easily removed, they can be difficult to remove in large vessels and appear to go against the common stipulation of “visually clean.”
Pressure gradients	Pure steam systems only. Pressure changes in the distribution system will affect the amount of steam condensate as well as the quality of the steam. If system sections are exposed to pressure ranges, condensate that is not effectively removed from horizontal sections can be revaporized at higher pressures, which will lower the steam quality and transport any impurities borne in the steam condensate.
System age	This depends on how the system has been maintained in regard to frequency of passivation or derouging, CIP exposure, and formation of stable oxide layers. New systems have been observed to generate disproportionate amounts of Class I rouge formations in contrast to established systems. In pure steam systems, although oxide formations become stable with

Table D-2-2 Considerations That Affect the Amount of Rouge Formation During the Operation of a System (Cont'd)

Variables	Considerations
	age, they can also thicken and be prone to particle sloughing in high-velocity sections. It should be noted that system time in use can have both beneficial and negative effects in regard to rouge formation and that regular system monitoring is important in identification of incipient corrosion.

NOTE: (1) There is well-established industry data supporting this, and it needs to be considered.

Table D-3.1-1 Process Fluid Analyses for the Identification of Mobile Constituents of Rouge

		Test Criteria	
Type of Test	Test Description	Pros	Cons
Ultra trace inorganic analysis (by ICP/MS)	Concentrations of trace metals in process solutions including pure water/steam are directly analyzed by inductively coupled plasma mass spectrometry (ICP/MS).	Noninvasive sample acquisition. Highly quantitative information. Provides strong ability to trend data	Baseline must be determined for each system analyzed.
Standard particulate analysis (via light)	A liquid sample is subjected to a laser light, which scatters on contact with particles. The scattered light is collected, processed, segregated by channel, and displayed as a specific count for each size range analyzed.	Noninvasive sample acquisition. Highly quantitative information. Provides strong ability to trend data	Baseline must be determined for each system analyzed.
Ultra trace inorganic analysis (by SEM/EDX)	Fluids are filtered via vacuum filtration, and particles are collected on a fine-pore filter medium. The particles are then analyzed by scanning electron microscopy for size, composition, and topographical features.	Provides highly detailed physical observation and elemental composition data for mobile particulates	Limited with respect to organic particulate identification
Fourier transform infrared spectroscopy (FTIR)	Organic analysis of liquid samples or extracts from wipe samples. Used to identify possible organic films or deposits	Potentially noninvasive sample acquisition. Allows for organic identification of elastomers or alternate organic contaminants	Organic contaminants must be profiled in a specific target compound library.

Table D-3.2-1 Solid Surface Analyses for the Identification of Surface Layers Composition

		Test Criteria	
Type of Test	**Test Description**	**Pros**	**Cons**
Microscopic and human visual analysis	Visual analysis via polarized light microscopy (PLM), scanning electron microscopy (SEM), or alternative microscopy instrumentation	Good test for morphology determination. Can be coupled with energy dispersive X-ray (spectroscopy) (EDX) analysis for elemental composition information	Invasive test. Requires periodic removal of solid samples (e.g., coupons)
Scanning auger microanalysis (SAM) or auger electron spectroscopy (auger)	Surface metal elemental composition analysis. Provides for detailed qualitative elemental composition data on both the surface itself and the subsurface (or base metal)	Highly accurate method for positive identification and qualification of the surface metal composition. Used to determine the depth and elemental composition of the surface including the passive layer itself	Invasive and destructive test. Requires periodic removal of solid samples (e.g., coupons)
Small spot electron spectroscopy for chemical analysis (ESCA) or X-ray photoelectron spectroscopy (XPS)	The sample is subjected to a probe beam of X-rays of a single energy. Electrons are emitted from the surface and measured to provide elemental analysis of the top surface layers.	Highly accurate method for the qualification and quantification of the surface metal composition. Used to determine the depth and compositional analysis of the passive layer. Provides excellent elemental analysis of the top surface layers, including which oxide(s) are present	Invasive and destructive test. Requires periodic removal of solid samples (e.g., coupons)
Reflection grade ellipsometry	Multicolor interferometry using light and its diffractive properties to assess surface conditions	Nondestructive analysis. Known diffractive characteristics of elements could provide for qualitative analysis of surface chemistry properties.	Invasive test. Requires periodic removal of solid samples (e.g., coupons). Field qualification of this method is still ongoing.
Electrochemical impedance spectrometry	The analysis of electrochemical noise in order to quantify the state of corrosion of a metallic surface	Noninvasive, real-time quantification of metallic corrosion. Provides strong ability to trend data	Field qualification of this method is still ongoing.

(19)

Table D-4.1-1 Rouge Remediation Processes Summary

Derouging Processes: Specific				
Class of Rouge	**Description [Notes (1), (2)]**	**Comments [Notes (3), (4)]**	**Chemistry [Note (5)]**	**Conditions of Process [Notes (6), (7)]**
Class I Removal	Phosphoric acid	Effective at removing iron oxides without etching the process contact surface	5% to 25% phosphoric acid	2 hr to 12 hr at 40°C to 80°C
	Citric acid with intensifiers	Effective at removing iron oxides without etching the process contact surface	3% to 10% citric acid with additional organic acids	2 hr to 12 hr at 40°C to 80°C
	Phosphoric acid blends	Can be used at a variety of temperatures and conditions	5% to 25% phosphoric acid plus either citric acid or nitric acid at various concentrations	2 hr to 12 hr at 40°C to 80°C
	Sodium hydrosulfite (i.e., sodium dithionite)	Effective at removing iron oxides without etching the surface but may generate sulfide fumes	Up to 10% sodium hydrosulfite	2 hr to 12 hr at 40°C to 80°C
	Electrochemical cleaning	Useful in removing stubborn rouge without risk of etching the process contact surface	25% to 85% phosphoric acid	Limited to accessible parts of systems, primarily vessels. Process times are approximately 1 min/ft^2.
Class II Removal	Phosphoric acid	Effective at removing iron oxides without etching the surface	5% to 25% phosphoric acid	2 hr to 24 hr at 40°C to 80°C
	Citric acid with organic acids	Effective at removing iron oxides without etching the surface	5% to 10% citric acid with additional organic acids	2 hr to 24 hr at 40°C to 80°C
	Phosphoric acid blends	Can be used at a variety of temperatures and conditions	5% to 25% phosphoric acid plus either citric acid or nitric acid at various concentrations	2 hr to 24 hr at 40°C to 80°C
	Oxalic acid	Effective at removing iron oxides; may etch electropolished surfaces	2% to 10% oxalic acid	2 hr to 24 hr at 40°C to 80°C
	Electrochemical cleaning	Useful in removing stubborn rouge without risk of etching the process contact surface	25% to 85% phosphoric acid	Limited to accessible parts of systems, primarily vessels. Process times are approximately 1 min/ft^2.
	Sodium hydrosulfite (i.e., sodium dithionite)	Effective at removing iron oxides without etching the surface but may generate sulfide fumes	Up to 10% sodium hydrosulfite	2 hr to 12 hr at 40°C to 80°C
Class III Removal	Phosphoric acid blends	Can be used at a variety of temperatures and conditions	5% to 25% phosphoric acid plus either citric acid or nitric acid at various concentrations	8 hr to 48+ hr at 60°C to 80°C
	Oxalic acid	May etch metallic surfaces	10% to 20% oxalic acid	8 hr to 48+ hr at 60°C to 80°C
	Citric acid with organic acids	May etch metallic surfaces	5% to 10% citric acid with additional organic acids	8 hr to 48+ hr at 60°C to 80°C
	Citric acid with intensifiers	Will etch metallic surfaces	5% to 10% citric acid with additional organic acids and fluorides	8 hr to 48+ hr at 60°C to 80°C

(19)

Table D-4.1-1 Rouge Remediation Processes Summary (Cont'd)

Derouging Processes: Specific				
Class of Rouge	**Description [Notes (1), (2)]**	**Comments [Notes (3), (4)]**	**Chemistry [Note (5)]**	**Conditions of Process [Notes (6), (7)]**
	Nitric/HF or nitric/ ammonium bifluoride	Will etch metallic surfaces	15% to 40% nitric acid with 1% to 5% HF or 1% to 5% NH_4HF_2	1 hr to 24 hr at ambient to 40°C
	Electrochemical cleaning	Useful in removing stubborn rouge without risk of etching the process contact surface	25% to 85% phosphoric acid	Limited to accessible parts of systems, primarily vessels. Process times are approximately 1 min/ft^2.

NOTES:

(1) All of these derouging processes should be followed with a cleaning and passivation process of the treated surface.

(2) Application methods include fluid circulation, gelled applications for welds or process contact surfaces, and spraying methods for vessels and equipment.

(3) These derouging processes may produce hazardous wastes based on metals content and local and state regulations.

(4) Oily or loose black residue due to carbon buildup may be present on the process contact surfaces after derouging and may require special cleaning procedures to remove.

(5) Chemical percentages are based on weight percent.

(6) The time and correlating temperatures given above are in direct relation to the percent by weight of the base reactant(s). A change in a formulation will change those corresponding requirements.

(7) A deionized water rinse shall immediately follow each of the above chemical treatments.

NONMANDATORY APPENDIX E
PASSIVATION PROCEDURE QUALIFICATION

(19) ## E-1 GENERAL

This Appendix provides basic information and offers guidelines for owner/users, equipment manufacturers, and service providers for newly manufactured or installed systems in accordance with the requirements of GR-1. This Appendix covers the preparation and execution of procedures associated with the initial water flushing, chemical cleaning and degreasing, passivation, and final rinse(s) of specialized systems, as well as bioprocessing equipment after assembly, erection, or modification. These procedures will apply to UNS S30400, UNS S30403, UNS S31600, and UNS S31603 stainless steels. Superaustenitic stainless steels and nickel alloys may require a modified procedure.

This Appendix defines a method for qualifying the passivation process used for system and process component surfaces.

This Appendix provides information on passivation procedures and testing of the surface resulting from various passivation procedures.

E-2 PURPOSE OF PASSIVATION TREATMENTS

Passivation, or the forming of a passive layer on the surface of stainless steel alloys, is a naturally occurring phenomenon on a clean surface when oxygen is present. The passive layer may be augmented by chemical treatment of the stainless steel surface.

A critical prerequisite in preparation for chemical passivation processes is a cleaning procedure. This procedure includes all operations necessary for the removal of surface contaminants (oil, grease, etc.) from the metal to allow the chemical passivation to be most effective. The purpose of the final chemical passivation process is to enhance the passive layer and provide an alloy surface free of free iron or other contaminants, allowing the alloy to be in the most corrosion-resistant state.

For improved corrosion resistance in the standard stainless steel grades (e.g., UNS S31603), the passivation treatment is most beneficial and important. With superaustenitic stainless steels and nickel alloys, passivation is less critical, provided the surfaces are clean and free of contaminants. At the owner/user's option, passivation may be performed to remove any free iron on process contact surfaces and to facilitate the formation of the passive layer.

In a discussion on passivation, it should be realized that the best passivation treatment or any surface treatment only puts the alloy in its most corrosion-resistant state for a particular environment. In other words, there are inherent corrosion-resistance limitations for any alloy, and the best passivation treatment does not replace the need for a more corrosion-resistant material for certain applications.

E-2.1 Why Passivation Is Necessary

Although stainless steel components may be clean and the passive layer intact prior to installation, welding destroys the passive film on the weld bead and the heat-affected zone (HAZ) of the weld. The distribution of elements across the weld and HAZ, including chromium, iron, and oxygen, are disturbed when the metal is melted so that the concentration of iron is elevated, while chromium, which is normally of higher percentage than iron in the passive layer, is reduced.

Discoloration and contamination (especially free iron) introduced during fabrication may also compromise corrosion resistance unless removed. Passivation after welding, by removing free iron, helps to restore the passive layer. It does not remove discoloration. Removal of discoloration requires a more aggressive acid than the usual nitric or citric acids used for passivation. Since the only postweld treatment normally used for installed piping systems is passivation, welding procedures that minimize discoloration are specified (see Part MJ of this Standard).

Fabrication, cutting, bending, etc., can result in contamination that leads to loss of corrosion resistance. Examples are embedded iron, heat tint, welding flux from covered electrodes, arc strikes, and painting/markings. Exposure to carbon steel or iron is particularly detrimental. By removing contamination, especially free iron, a passivation treatment can help to restore the natural passivity of stainless steel that is damaged by fabrication.

E-2.2 When Passivation Is Necessary

(a) after welding and fabrication
(b) after welding of new components into a system

Table E-3.2-1 Minimum Surface Requirements for Process Qualification Samples

Material	Test Method	Cr/Fe Ratio	Oxide Depth
UNS S31600 or UNS S31603	AES	1.0 or greater	15 Å, min.
UNS S31600 or UNS S31603	GD-OES	1.0 or greater	15 Å, min.
UNS S31600 or UNS S31603	XPS/ESCA	1.3 or greater	15 Å, min.

GENERAL NOTES:
(a) XPS/ESCA readings of metal oxides typically obtain higher values than readings of metals.
(b) Additional alternative testing methods for cleanliness and passivation are shown in Table E-5-1.

E-3 PASSIVATION PROCEDURE (SEE SF-2.6)

E-3.1 Procedure Description

The passivation provider shall obtain welded and nonwelded sample component(s) or coupons from each passivation method used (e.g., circulation, spot, bath) for the purpose of demonstrating that the procedure is capable of providing the required surface characteristics, namely, cleanliness, surface chemistry, and corrosion resistance.

The passivation process used on the qualification component(s) or coupons shall be reproducible in the system for which it is intended.

The procedure description and qualification document shall be available for review by the owner/user or his designee. The owner/user shall be responsible for verifying that the passivation procedure to be used on their system or components has been qualified.

E-3.2 Procedure Qualification

The passivation provider shall develop a passivation procedure for each method used. The procedure shall be developed to ensure that essential variables used to obtain the qualification samples can effectively remove free iron and meet the requirements of Table E-3.2-1. Procedure qualification, as a minimum, shall include the following:

(a) Process Description. The following steps shall be described as a minimum (Table E-3.2-2 may be used as a guide):

(1) prepassivation survey and preparation
(2) flushing
(3) cleaning
(4) passivation
(5) final rinsing
(6) verification

(b) Essential Variables (Conditions Under Which the Samples Were Processed). The following essential variables shall remain within the designated range:

(1) process time
(2) temperature of solution during process
(3) general chemistry of process fluids
(4) process endpoint determination
(5) conductivity of final deionized rinse water

(c) Procedure Qualification Coupon Testing

(1) AES (auger electron spectroscopy) testing at the weld, including the worst discoloration area in the weld and heat-affected zone, and on the base metal to meet the requirements of Table E-3.2-1

(2) GD-OES (glow discharge–optical electron spectroscopy) testing at the weld, including the worst discoloration area in the weld and heat-affected zone, and on the base metal to meet the requirements of Table E-3.2-1

(3) XPS (X-ray photoelectron spectroscopy), also known as ESCA (electron spectroscopy for chemical analysis), testing at the weld, including the worst discoloration area in the weld and heat-affected zone, and on the base metal to meet the requirements of Table E-3.2-1

Qualification of method shall be supported by documentation for each procedure. The actual values of the essential variables and coupon testing listed above shall be documented and maintained as part of the procedure.

E-3.3 Procedure Documentation Requirements

The passivation provider shall generate and provide the following documentation, as a minimum:

(a) process descriptions
(b) essential variables
(c) ESCA/XPS or AES or GD-OES testing for each procedure qualification sample produced

E-4 PASSIVATION QUALITY CONTROL

E-4.1 Quality Control Surveillance

Quality control surveillance to ensure the written and qualified passivation procedure has been followed is essential. A thorough rinse with deionized or owner/user-approved water should follow the chemical treatment. It is good practice to continue rinsing until, as determined by conductivity analysis, the ionic contaminants, process chemicals, and by-products have been removed. This document shall be available for review by the owner/user or his designee.

(a) Written documentation that all requirements of the qualified procedure have been followed.

(b) Final rinse shall meet pre-established conductivity (quality) requirements.

Table E-3.2-2 Passivation Processes

Process Type	Process Description [Notes (1), (2)]	Comments [Note (3)]	Conditions of Process [Notes (4), (5)]	Chemistry [Note (6)]
		Precleaning		
Water flushing/ filtration processes	High-velocity DI (or owner/user-chosen) water flushing for removal of particles and construction debris	Removes debris prior to the passivation process	Ambient temperature for 5 min to 30 min per section; generally includes filtration of fluids	DI water
	High-velocity water flushing	Removes debris prior to the passivation process. Chlorides in water are detrimental to austenitic stainless steels	Ambient temperature for 15 min to 60 min per section	DI water (recommended)
		Cleaning		
Cleaning/ degreasing processes	Phosphate cleaners	Removes light organic deposits. Can leave phosphate surface contamination	1 hr to 4 hr at heated conditions depending on the solution and contamination level	Blends of sodium phosphates [monosodium phosphate (MSP), disodium phosphate (DSP), trisodium phosphate (TSP)] and surfactants
	Alkaline cleaners	Can be selected for specific organic contaminates		Blends of nonphosphate detergents, buffers, and surfactants
	Caustic cleaners	Effective at removal of heavy organic contamination or degreasing		Blends of sodium and potassium hydroxides and surfactants
	Isopropyl alcohol (IPA)	Effective as a degreaser. Volatile. Highly flammable and sensitive to static discharge	Hand swab or wipe surface at ambient conditions	70% to 99%
		Passivation		
Passivation processes	Nitric acid	Proven method under ASTM A380/A967. Can be processed at ambient conditions depending on formulation	30 min to 90 min at ambient temperature or higher, depending on concentration used	10% to 40% nitric acid
	Phosphoric acid	Effective at removing iron oxides in addition to free iron	1 hr to 4 hr at heated conditions	5% to 25% phosphoric acid
	Phosphoric acid blends	Can be used at a variety of temperatures and conditions		5% to 25% phosphoric acid plus either citric acid or nitric acid at various concentrations
	Citric acid	Specific for free iron removal. Should be processed at elevated temperatures. Takes longer to process than mineral acid systems. Meets or exceeds ASTM A967		10% citric acid
	Chelant systems	Should be processed at elevated temperatures. Takes longer to process than mineral acid systems. Removes iron oxides in addition to free iron. Meets or exceeds ASTM A967		3% to 10% citric acid with various chelants, buffers, and surfactants

Table E-3.2-2 Passivation Processes (Cont'd)

Process Type	Process Description [Notes (1), (2)]	Comments [Note (3)]	Conditions of Process [Notes (4), (5)]	Chemistry [Note (6)]
		Passivation (Cont'd)		
	Electropolishing	This process is generally limited to components rather than installed systems. Process should be performed according to a qualified procedure. This process removes metal from the surface. Electropolishing should be performed in such a way as to meet or exceed ASTM B912.	Exposure time must be calculated to ensure 5 µm to 10 µm material removal from all surfaces requiring passivation. Rinsing must include a step to ensure removal of residual film that may adversely affect the appearance or performance of the product.	Phosphoric acid-based electrolyte
		Oxidation		
Oxidation processes	Hydrogen peroxide	Oxidizes metal surface and sanitizes	30 min to 2 hr at ambient to 40°C	3% to 10% hydrogen peroxide
	Hydrogen peroxide with peracetic acid blends	Oxidizes metal surface and sanitizes		1% to 2% blend

NOTES:
(1) Application methods include fluid circulation, gelled applications for welds or surfaces, and spraying methods for vessels and equipment.
(2) Special attention should be directed to removal of metal shavings and construction debris from locations such as sprayballs, diaphragm valves, heat exchangers, etc.
(3) These passivation processes may produce hazardous wastes based on metals content, and local and state regulations.
(4) The time and correlating temperatures in the Table are in direct relation to the percent by weight of the base reactant(s). A change in a formulation may change those corresponding requirements.
(5) A deionized water rinse shall immediately follow each of the chemical treatments.
(6) Chemical percentages are based on weight percent.

E-4.2 Certificate of Passivation Compliance

The passivation provider shall supply a Certificate of Compliance for each system or set (type) of component(s) that shall include, but not be limited to

(a) customer's name
(b) description of system or component(s)
(c) vendor company name
(d) qualified passivation method used
(e) documentation of passivation process, as follows:
(1) written qualified procedure
(2) documentation of process control of essential variables
(3) instrument calibration records
(4) certificates of analysis for all chemicals used
(5) process testing and verification
(f) postpassivation verification method(s) used

E-5 EVALUATION OF CLEANED AND PASSIVATED SURFACES

There are no universally accepted tests to ensure that a component or system has been passivated or is in a passive condition. If the system/component has received the proper chemical passivation treatment, the documentation generated during the process (listed in E-4.2) should provide assurance that the components or system has received the specified treatment. As a guide to owner/users and others, to help determine whether an acceptable surface has been achieved following a particular cleaning or chemical passivation procedure, Table E-5-1 has been developed.

E-5.1 Acceptance Criteria for Cleaned and/or Passivated Process Contact Surfaces (See Table SF-2.6-1) (19)

Table E-5-1 may be used as a guide for acceptance criteria for cleaned and/or passivated components or systems. This matrix is a simplified compilation of testing methodologies that an owner/user may want to use in selecting a test or as a means to interpret a proposal from a testing company.

The matrix is divided into groups of four types of testing methods

(a) gross inspection of cleaned and passivated parts per ASTM A380/A967 (Pass/Fail)
(b) precision inspection of cleaned and passivated parts under ASTM A380/A967 (Pass/Fail)
(c) electrochemical field and bench tests
(d) surface chemical analysis tests

Groups 1 and 2 of Table E-5-1 reflect the two main divisions in ASTM A380 and ASTM A967. The most obvious type of examination of these methods is visual. The

(19) **Table E-5-1 Test Matrix for Evaluation of Cleaned and/or Passivated Surfaces**

Type of Test	Test Description	Pros	Cons
1. Gross Inspection of Cleaned and/or Passivated Parts per ASTM A380/A967 (Pass/Fail)			
Visual examination [CT (test for cleanliness), RT (test for the presence of rouge)]	Bench or field test. Visual examination is the direct or remote visual examination of, in this case, a passivated metallic surface.	Can be performed with minimal preparation and equipment. Good general appearance review	Not quantitative. Subjective interpretation of findings
Wipe test ASTM A380 (CT, RT)	Bench or field test. This test consists of rubbing a test surface with a clean, lint-free, white cotton cloth, commercial paper product, or filter paper moistened with high-purity solvent.	Useful for testing surfaces that cannot be readily accessed for direct visual examination. Removable surface contamination can be easily identified and compared.	Not quantitative. Difficult to inspect hard-to-reach areas of large tube diameters. There is also a risk of leaving errant fibers behind from the wipe or plug. Can be detrimental to electropolished surfaces
Residual pattern test ASTM A380 (CT)	Bench or field test. After finish-cleaning, dry the cleaned surface per ASTM A380. The presence of stains or water spots indicates the presence of contaminants.	A simple test with rapid results	Not quantitative. Not very sensitive
Water-break test ASTM F22	The water-break test is performed by withdrawing the surface to be tested, in a vertical position, from a container overflowing with water. The interpretation of the test is based on the pattern of wetting.	General cleanliness of surface is easily determined. Useful in detecting hydrophobic contamination	Not quantitative. This test identifies the presence of retained oils and greases. The test is not applicable on all surfaces including, but not limited to, electropolished surfaces.
ASTM A380 water-wetting and drying; ASTM A967 water immersion practice A [PT (test for passivation)]	Bench or field test. Immersed in, or flushed with distilled water then air dried. A modified version of this test requires a solution of 3% to 7% salt water, with a final rinse prior to inspection, using DI-quality water or better.	Staining is evidence of free iron, which is detected through visual examination. Identifies possible pitting corrosion sites or embedded iron	Not quantitative
High-humidity test ASTM A380 and ASTM A967 Practice B (PT)	Bench test. Sample coupon is immersed or swabbed with acetone or methyl alcohol then dried in an inert atmosphere. The coupon is then subjected to 97% humidity at 100°F for 24 hr or more.	Staining is evidence of free iron, which is detected through visual examination	Not quantitative. Not used for installed tubing. Sample coupons can be used, but does not prove complete coverage. Lengthy test. Containment cabinet required
Salt spray test ASTM A967 Practice C (PT)	Bench or field test. This test is conducted in accordance with ASTM B117 subjecting the test area to a 5% salt solution for a minimum of 2 hr.	Rust or staining attributable to the presence of free iron particles embedded in the surface will become noticeable on visual examination of the metal surface	Not quantitative. Longer-term testing is required to test for passive film quality or corrosion resistance. However, exposures over about 24 hr may show light staining resulting from differences in micro finish texture.
2. Precision Inspection of Cleaned and/or Passivated Parts Under ASTM A380/A967 (Pass/Fail)			
Solvent ring test ASTM A380 (CT)	Bench test. Place a single drop of high-purity solvent on the surface to be evaluated, stir briefly, then transfer to a clean quartz microscope slide and allow the drop to evaporate. If foreign material has been dissolved by the solvent, a distinct ring will be formed on the outer edge of the drop as it evaporates.	Good test for organic contamination on the test surface	Not quantitative

(19) **Table E-5-1 Test Matrix for Evaluation of Cleaned and/or Passivated Surfaces (Cont'd)**

Type of Test	Test Description	Pros	Cons
2. Precision Inspection of Cleaned and/or Passivated Parts Under ASTM A380/A967 (Pass/Fail) (Cont'd)			
Black light inspection ASTM A380 (CT)	Bench test. This test requires the absence of white light and a flood-type ultraviolet light.	Suitable for detecting certain oil films and other transparent films that are not detectable under white light. Good test for organic contamination on surface	Not quantitative. Not practical when testing for passivation
Atomizer test ASTM A380 (CT)	Bench test. This test is conducted in accordance with ASTM F21 using DI-quality water or better. A variation of the water-break test, this test uses an atomized spray, rather than a simple spray or dip to wet the surface.	Test for presence of hydrophobic films. This test is more sensitive than the water-break test.	Not quantitative. Requires direct visual examination
Ferroxyl test for free iron ASTM A380/potassium ferricyanide-nitric acid ASTM A967 Practice E (PT)	Bench or field test. Apply a freshly prepared solution of DI-quality water or better, nitric acid, and potassium ferricyanide to the coupon using an atomizer having no iron or steel parts. After 15 sec a blue stain is evidence of surface iron. Remove solution from the surface as soon as possible after testing, per ASTM A380 or ASTM A967. Test nonsystem coupons only.	Identification of free iron contamination on surface. Very sensitive test	Not quantitative. This test will only identify free iron on the surface and will not directly measure the improvements of the passive oxide layer. This is a very sensitive test and must be performed by personnel familiar with its limitations. Either a sacrificial coupon is used for this test, or the test area is cleaned as described in the respective ASTM practice and/or specification. Safety and disposal issues exist with the test chemical. Easy to get a false-positive result
Copper sulfate test ASTM A380/ASTM A967 Practice D (PT)	Bench test. Prepare a 250-cm^3 solution consisting of 1 cm^3 of sulfuric acid (s.g. 1.84), 4 g copper sulfate, and the balance in DI-quality water or better. Apply this to a sacrificial coupon using a swab. Keep the surface to be tested wet for a period of 6 min with additional applications as needed.	Identification of free iron contamination on the test surface. Is effective in detecting smeared iron deposits	Not quantitative. Embedded iron is detected, but difficult to detect small discrete iron particles
3. Electrochemical Field and Bench Tests			
Cyclic polarization measurements	This technique uses cyclic polarization measurements similar to the ASTM G61 test method to measure the critical pitting potential (CPP). The more noble (more positive) the CPP, the more passive the stainless steel surface. Similar results may be obtained with the ASTM G150 test that measures critical pitting temperature (CPT).	This test method provides a direct measurement of the corrosion resistance of a stainless steel surface. The measured CPP provides a quantitative measurement of the level of passivation. The test equipment is relatively inexpensive.	The method requires a potentiostat and corrosion software package to make the measurements. To ensure reliable results, operators should be trained in electrochemical test techniques.

(19) **Table E-5-1 Test Matrix for Evaluation of Cleaned and/or Passivated Surfaces (Cont'd)**

Type of Test	Test Description	Pros	Cons
3. Electrochemical Field and Bench Tests (Cont'd)			
Electrochemical pen (ec-pen) (PT)	The result is based on preset values. Being the size and shape of a writing instrument, the ec-pen makes electrolytic contact when placed on the test surface. Capillary action causes electrolyte to flow from the reservoir to the surface through a porous polymer body while preventing the electrolyte from leaking out of the pen. There is a stable electrode inside the pen mechanism. By simply positioning the ec-pen on the sample surface, electrolytic contact is established and electrochemical characterization is possible. The measured area is typically 1.5 mm^2.	Easy to handle, short sample preparation time, real-time results, and the possibility to run experiments on virtually any size object with various surface geometries. The ec-pen is a portable instrument for the measurement of corrosion potential suitable for field use.	This test does not quantify the passive layer, but instead provides a pass-fail indication of passivity. The local test area needs to be cleaned and repassivated after testing.
Koslow test kit 2026/3036 (PT)	Similar to the ec-pen, in that it measures the corrosion potential of the metal surface, the Koslow 2026/3036 consists of a meter, a probe, and an interconnecting cable. An electrical charge is first applied to the test piece, after which a moist pad is placed on the surface of the same test piece. The probe is pressed into the moist pad to complete the circuit. Within a couple of seconds the cell voltage result appears on the digital meter.	Measures corrosion potential at the surface	User sensitive
4. Surface Chemical Analysis Tests			
Auger electron spectroscopy (AES) (PT, RT)	Secondary and auger electrons, in the targeted area of the test coupon, are bombarded with a primary electron beam, which is used as an excitation source. Photoelectrons are subsequently ejected from the outer orbital of atoms in the target material. The ejected photo-electrons are then detected by means of electron spectroscopy. The method by which the ejected photo-electrons are detected and analyzed is AES. This test is useful for surface analysis from 2 Å to a depth greater than 100 Å.	Provides quantitative analysis. Using a scanning primary beam, secondary electron images yield information related to surface topography. Auger electrons, when analyzed as a function of energy, are used to identify the elements present. Elemental composition of the surface to a depth of 2 Å to 20 Å is determined and can be used in depth profiling applications.	The specimen chamber must be maintained at ultra-high vacuum (UHV). The specimen must be electrically conductive. Instrument is not readily available. Expertise is needed for data interpretation.

(19) **Table E-5-1 Test Matrix for Evaluation of Cleaned and/or Passivated Surfaces (Cont'd)**

Type of Test	Test Description	Pros	Cons
4. Surface Chemical Analysis Tests (Cont'd)			
Electron spectroscopy for chemical analysis (ESCA) also known as X-ray photoelectron spectroscopy (XPS) (PT, RT)	Using X-ray as an excitation source, photoelectrons are ejected from the inner-shell orbital of an atom from the target material. The ejected photoelectrons are then detected by means of XPS. The method by which the ejected photoelectrons are then detected and analyzed is ESCA (or XPS). Useful for surface analysis to a depth of 10 Å to 100 Å.	Provides quantitative analysis in measuring the following: *(a)* Elemental composition of the surface (10 Å to 100 Å usually) *(b)* Empirical formula of pure materials *(c)* Elements that contaminate a surface *(d)* Chemical or electronic state of each element in the surface *(e)* Uniformity of elemental composition across the top of the surface (also known as line profiling or mapping) *(f)* Uniformity of elemental composition as a function of ion beam etching (also known as depth profiling)	The specimen chamber must be maintained at ultra-high vacuum (UHV). Instrument is not readily available. Expertise is needed for data interpretation.
GD-OES (glow discharge–optical emission spectroscopy) (PT, RT)	GD-OES uniformly sputters material from the sample surface by applying a controlled voltage, current, and argon pressure. Photomultiplier tube detectors are used to identify the specific concentrations of various elements based on the wavelength and intensity of the light emitted by the excited electrons in each element when they return to the ground state.	The GD-OES method is particularly useful for rapid, quantitative depth profiling of thick- and thin-film structures and coatings	Relatively expensive. Instrument not widely available

examiner shall look for a clean surface free of oxides, scale, weld discoloration/heat tint, stains, dirt, oil, grease, or any deposits that could prevent the chemical passivation solution from reaching the metal surface.

The test results from ASTM A967, which are exclusively for passivation, are all based on visual detection of staining or discoloration indicative of the presence of free iron. These test results are subjective and nonquantifiable. However, for some applications this may be all that is required. The visual acceptance criteria in ASTM A380 and ASTM A967 apply.

Groups 3 and 4 of Table E-5-1 reflect two distinct methods of quantitative testing. These tests are not contained in either of the ASTM standards. These tests are designed to provide a more quantifiable analysis of a passivated surface. The electrochemical field and bench tests in Group 3 in Table E-5-1, with the exception of cyclic polarization, are suitable for field tests such as those used for postpassivation testing of installed piping systems and passivated welded surfaces.

Passivation is capable of dramatically increasing the chromium-to-iron (Cr/Fe) ratio on the surface of 316L-type stainless steel when properly applied. One measurement of the degree of enhancement of the layer following a chemical passivation treatment is the Cr/Fe ratio as determined by AES, GD-OES, or ESCA. The procedure is not readily adapted to field use but may be useful in developing the passivation procedure.

A Cr/Fe acceptance ratio, regardless of test method, should be 1.0 or greater (see Table E-3.2-1); because of variability in accuracy, identical results obtained with the different test methods are not expected. The surface chemical analysis tests in Group 4 in Table E-5-1 include methods for evaluation of the thickness and chemical state of the passive layer on stainless steel. Cyclic polarization measurements (Group 3 in Table E-5-1) may also be used to provide a quantitative evaluation of the level of passivation. Cyclic polarization as well as the methodologies in Group 4 in Table E-5-1 might be applied to sacrificial coupons placed in systems subject to the complete passivation process.

NONMANDATORY APPENDIX F
CORROSION TESTING

F-1 GENERAL

Corrosion testing may be used to determine whether the material manufacturer has used the appropriate processing variables during the fabrication of the raw product form. These variables include those primarily related to thermomechanical processing and heat treatment. The material can be evaluated based on weight loss or electrochemical response, or it can be measured by destructive testing techniques such as toughness testing. The standard ASTM tests that are commonly used are shown in Table F-1-1. However, there is no guarantee that a tested alloy will be appropriate for a specific environment even if it performs well in an industry-accepted test.

It is often appropriate to test a number of candidate alloys in a specific environment. Ideally the test selected should reflect the corrosion mode anticipated in production. These corrosion modes include general corrosion, crevice corrosion, pitting corrosion, and stress corrosion cracking.

F-2 CORROSION TESTS

For general corrosion, the most commonly used test method is ASTM G31, Standard Practice for Laboratory Immersion Corrosion Testing of Metals.

To rank materials based on their resistance to localized corrosion, such as pitting corrosion, the two most commonly used electrochemical methods are ASTM G61, Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion, and ASTM G150, Standard Test Method for Electrochemical Critical Pitting Temperature Testing of Stainless Steels.

Other methods used to screen for more specific metallurgical problems such as the presence of sigma phase, chromium carbides, or improper heat treatment are described in Table F-1-1.

F-3 PITTING RESISTANCE EQUIVALENT (PRE) NUMBER

Where testing is not possible or desired, owner-users may use the PRE number as a guide to rank a material's corrosion resistance. Relative PRE number values for some wrought stainless steel and nickel alloys are shown in Table F-3-1. Notice that although different equations are used to calculate the PRE number for the two different alloy systems [see Table F-3-1, Notes (1) and (2)], the numbers may still be used to compare alloys for ranking purposes.

Since the PRE numbers are calculated based on composition, the listed values in Table F-3-1 are based on nominal composition only and are not representative of the ranges of PRE numbers that could result from the compositional ranges permitted by the applicable material specification. The values listed in Table F-3-1 are not representative of values that may be obtained by compositions specified by the owner/user. The owner/user is cautioned that PRE numbers should be developed from the specific composition of the heat intended for use in order to accurately rank or estimate the alloy's resistance to pitting. Consideration should be given to other factors that might reduce the corrosion resistance such as

(a) improper heat treatment
(b) surface finish and quality
(c) deleterious second phases
(d) welding

Table F-1-1 ASTM Corrosion Tests

ASTM Standard		Purpose of Test	Data Obtained	Typical Alloys Tested
ASTM A262	Practice A (oxalic acid test)	Qualitative test to determine susceptibility to intergranular attack associated with chromium carbide precipitates. Tests the effectiveness of final heat treatment. Used to screen specimens intended for testing in Practices B, C, and E	Comparative, visual examination of microstructure after testing only	Austenitic stainless steels
	Practice B (ferric sulfate–sulfuric acid test)	Quantitative test measuring weight loss due to intergranular corrosion associated with chromium carbide precipitates. Also tests for sigma phase in 321-type alloys. Tests the effectiveness of final heat treatment	Report weight loss only	Austenitic stainless steels
	Practice C (nitric acid test)	Quantitative test measuring weight loss due to intergranular corrosion associated with chromium carbide precipitates. Also tests for sigma phase in 316-, 316L-, 317-, 317L-, 321-, and 347-type alloys. Tests the effectiveness of final heat treatment	Report weight loss only	Austenitic stainless steels
	Practice E (copper–copper sulfate–sulfuric acid test)	Qualitative test to determine susceptibility to intergranular attack associated with chromium carbide precipitates. Tests the effectiveness of final heat treatment	Pass or fail	Austenitic stainless steels
ASTM A923	Method A (sodium hydroxide etch test)	Detection of the presence of detrimental intermetallic phases. Used to screen specimens intended for testing in Method B and Method C	Visual examination. Pretest for subsequent methods	Duplex stainless steels
	Method B (Charpy impact test)	Used to test toughness characteristics that may result from processing irregularities	Impact toughness energy	Duplex stainless steels
	Method C (ferric chloride test)	Detects a loss of corrosion resistance associated with a local depletion of Cr and/or Mo as a result of the precipitation of chromium-rich and possibly molybdenum-rich phases	Report weight loss only	Duplex stainless steels
ASTM G48	Methods A and B (ferric chloride test)	Resistance to pitting and/or crevice corrosion	Report weight loss	Stainless steels, Ni-based alloys, and Cr-bearing alloys
	Methods C and D (ferric chloride test)	Resistance to pitting and/or crevice corrosion. Define the minimum temperature at which pitting or crevice corrosion initiates. Test the effects of alloying elements, final heat treatment, and surface finish of final product.	Report critical temperature	Ni-based and Cr-bearing alloys
	Methods E and F (ferric chloride test)	Resistance to pitting and/or crevice corrosion. Define the minimum temperature at which pitting or crevice corrosion initiates. Test the effects of alloying elements, final heat treatment, and surface finish of final product.	Report critical temperature	Stainless steels
ASTM G28	Method A	Tests the susceptibility to intergranular attack associated with composition and processing	Report weight loss only	Ni-based alloys
	Method B	Tests the susceptibility to intergranular attack associated with composition and processing, specifically subsequent heat treatments	Report weight loss only	Ni-based alloys

Table F-3-1 PRE Numbers for Some Alloys

UNS or EN Designation	PRE Number
Austenitic Stainless Steels [Note (1)]	
S30400	20
1.4301	19
S30403	20
1.4307	19
S31600	23
1.4401	23
S31603	23
1.4404	23
1.4435	26
S31703	28
1.4438	29
6% Mo Superaustenitic Stainless Steels	
N08367	43
S31254	42
1.4547	42
Duplex Stainless Steels	
S32205	35
1.4462	31
Nickel-Based Alloys [Note (2)]	
N06625	41
N10276	45
2.4819	45
N06022	46
2.4602	46

GENERAL NOTE: The above are industry-accepted formulas. Other formulas may be used at the owner's discretion.

NOTES:
(1) For stainless steels: PRE Number = %Cr + 3.3 [%Mo + 0.5(%W)] + 16(%N).
(2) For nickel alloys: PRE Number = %Cr + 1.5 (%Mo + %W + %Nb).

NONMANDATORY APPENDIX G
FERRITE

G-1 GENERAL

Ferrite is a phase that may precipitate during solidification of austenitic stainless steels depending on the ratios of the alloying elements. The ferritic phase consists of crystals with a body-centered cubic (bcc) lattice in contrast to the face-centered cubic (fcc) lattice of the austenitic matrix. The presence of ferrite in austenitic stainless steel welds may reduce the corrosion resistance in some corrosive environments. However, a minimum ferrite level may be required to maintain specific properties of particular product forms (e.g., castings) or is deemed necessary to prevent hot cracking of heavy wall weldments (e.g., vessels made from plate).

The ferrite level of austenitic stainless steel base metal strongly depends on heat analysis, primarily the chromium to nickel ratio, product form, and final heat treatment. Whereas wrought 316L-type stainless steel materials in the solution annealed condition typically show very low ferrite levels of 0 vol.%–3 vol.%, CF8M and CF3M stainless steel castings may contain 10 vol. %–20 vol.% of ferritic phase in the austenitic matrix.

As-solidified austenitic stainless steel welds typically have higher ferrite levels than the base metal. This is caused by rapid cooling that prevents the ferrite to austenite transformation from proceeding to thermodynamic equilibrium. The ferrite level of as-solidified austenitic stainless steel welds can be determined from the WRC-1992 Constitution Diagram for Stainless Steel Weld Metals[1] using a chromium equivalent Cr (eq) = %Cr + %Mo + 0.7%Nb and a nickel equivalent Ni (eq) = %Ni + 35%C + 20%N + 0.25%Cu. Postweld heat treatment (e.g., solution annealing of welded tubing) reduces the amount of ferrite in the weld.

It should be recognized that many austenitic stainless steels with high nickel content and nickel alloys do not contain any ferrite in as-solidified welds.

Measuring of ferrite in production welds shall be in accordance with AWS A4.2M:2006 (ISO 8249:2006MOD).

G-2 INFLUENCE OF FERRITE IN BIOPHARMACEUTICAL SERVICE

Ferrite in the base metal and welds can have a beneficial or a negative effect depending on the particular service, but generally offers little concern for biopharmaceutical services. Laboratory corrosion tests in severe biopharmaceutical service have shown that increased amounts of weld metal ferrite somewhat lowers corrosion resistance.[2] However, in high-purity water systems, there have been no reported system failures related to delta ferrite content in welds.

G-3 CONTROL OF FERRITE CONTENT IN WELDS OF AUSTENITIC STAINLESS STEELS

Ferrite in welds of austenitic stainless steels can be controlled by one or more of the following methods:

(a) postweld solution annealing

(b) use of weld filler with increased nickel content

(c) increase of nickel equivalent by addition of approximately 1 vol.%–3 vol.% nitrogen to shielding gas

(d) selection of heats of materials with high nickel to chromium ratios, such as the European steel grade 1.4435 (see Table MM-2.1-1) with a restricted Cr(eq) to Ni(eq) ratio[3] as per BN2[4]

[1] D. J. Kotecki and T. A. Siewert, "WRC-1992 Constitution Diagram for Stainless Steel Weld Metals: A Modification of the WRC-1988 Diagram," *Welding Journal* 71(5), p. 171-s, 1992.

[2] R. Morach and P. Ginter, "Influence of Low δ-Ferrite Content on the Corrosion Behaviour of Stainless Steels," *Stainless Steel World*, September 1997.

[3] Cr(eq) — 0.91 Ni(eq) ≤ 7.70, with

(a) Cr(eq) = %Cr + 1.5%Si + %Mo + 2%Ti, and

(b) Ni(eq) = %Ni + 0.5%Mn + 30%C + 30(%N–0.02)

[4] Basler Norm BN2 (N 11.265), Nichtrostender Stahl nach BN2, 1997.

NONMANDATORY APPENDIX H
ELECTROPOLISHING PROCEDURE QUALIFICATION

(19) ## H-1 SCOPE

This Appendix defines a method for qualifying the electropolishing process used for electropolishing component (s) surfaces that will be exposed to the process fluids in bioprocessing and pharmaceutical systems and ancillary equipment.

H-2 PURPOSE

This Appendix is intended to provide general guidelines for qualification of the electropolish methods used to achieve required surface improvements. Electropolishing is used to impart a surface that

(a) shall be free of oxide contamination and undesirable metallurgical conditions

(b) takes advantage of a material's surface chemical characteristics minus any damage or degradation from the component(s) manufacturing process

(c) exhibits a surface that is free of the surface irregularities that result from prior machining and forming processes

(d) optimizes corrosion resistance

H-3 ELECTROPOLISH PROCEDURE QUALIFICATION

H-3.1 Method Procedure

This Appendix is intended to provide general guidelines for qualifying the electropolish process used to provide the surface improvements of component(s) required.

The electropolish vendor shall produce sample component(s) or coupons from each electropolish method used (e.g., submersion, spot, in situ) for the purpose of demonstrating that the method is capable of providing the required surface characteristics.

The electropolish vendor should also demonstrate the ability to reproduce the method used on the qualification component(s) or coupons on the production component (s) and/or equipment for which the method is being qualified.

The electropolish vendor shall have a written quality control program that shall describe, as a minimum, the following:

(a) prepolish inspection process

(b) precleaning process

(c) specific gravity at operating temperature of electrolyte bath (minimum and maximum)

(d) bath analysis data (last date analyzed, iron/water concentrations of electrolyte, adjusted specific gravity value)

(e) resistivity of final deionized rinse water (minimum and maximum)

Qualification will be supported by internal documentation for each method. The actual values of the essential variables listed above shall be documented, maintained, and available for customer review.

H-3.2 Essential Variables

The electropolish vendor shall develop an electropolishing procedure for each method used. The procedure will be developed to ensure that essential variables used to produce the qualification samples can be reproduced. The electropolishing procedure, as a minimum, shall include the following essential variables:

(a) amperage/time (minimum and maximum)

(b) temperature range of bath during process (minimum and maximum)

(c) electropolish process

(d) final rinsing/cleaning process

(e) final inspection requirements

H-3.3 Vendor Documentation

The electropolish vendor, as a minimum, shall generate and maintain the following additional information:

(a) scanning electron microscope (SEM) records for each process qualification sample produced.

(b) XPS (ESCA) records for each process qualification sample produced. These results must meet the criteria of Table H-3.3-1.

(c) actual sample(s) used to qualify the process.

(d) process control records.

(e) the electropolish procedure used.

(f) final R_a (if required).

(g) copies of Certificate of Compliance (C of C) for each job.

H-3.4 Certificate of Compliance

The electropolish vendor, if requested by the customer, shall provide a Certificate of Compliance with each type of component(s) that shall include but is not limited to

(19) **Table H-3.3-1 Minimum Surface Requirements for Process Qualification Samples**

Material	Cr/Fe Ratio	Depth [Note (1)]	Surface Photo [Note (2)]
UNS S31600 UNS S31603	1 to 1 or greater	15 Å minimum	150X

NOTES:
(1) Test method: X-ray photoelectron spectroscopy (XPS/ESCA) analysis.
(2) Scanning electron microscopy (SEM).

(a) vendor's company
(b) customer's name
(c) description of component(s)
(d) identification of the electropolish procedure used
(e) final surface finish report (R_a if required by the customer)

NONMANDATORY APPENDIX J
VENDOR DOCUMENTATION REQUIREMENTS FOR NEW INSTRUMENTS

(19)

J-1 OVERVIEW

J-1.1 Section 1: VDR Definitions

This section identifies the vendor documentation requirements (VDR) number, document title, and definitions for the documentation (see Table J-1.1-1).

J-1.2 Section 2: Instrument Types and Required Documents

This section identifies the major instrument types and the required documentation, by VDR number (see Table J-1.2-1).

J-2 INSTRUCTIONS FOR USE

Together, these two sections are intended to be used by end-users, design and procurement agents, and vendors, to identify the documents required to support commissioning/qualification, installation, operation, and maintenance of instrumentation for the biopharmaceutical industry.

These documentation requirements may be modified, as necessary, to reflect the actual documents required for a particular instrument, based on the instrument's complexity, application, end-user's specific requirements, etc.

Table J-1.1-1 Vendor Documentation Requirements for New Instruments: Section 1, VDR Definitions

VDR #	Documentation	Definitions
1	Certified Arrangements/ Assembly Drawings	Provide Certified Arrangements/Assembly Drawings for the tagged component [or tagged packaged equipment (skid)] specified on the P.O. A "Certified Arrangement" or "Assembly Drawing" means that a statement, signed and dated by an authorized company representative, is included on (or with) the drawing, certifying the component (or skid) has been manufactured in accordance with stated applicable federal and state or internationally recognized regulatory requirements and the designated component (or skid), by tag number, complies with the established industry standards and product specifications.
2	Catalog information, cut sheets, product bulletins	This information shall include the supplier's literature for the component being purchased. The literature shall include dimensions, materials of construction, and layout considerations such as orientation, typical utility requirements, power, and instrument air.
3	Detailed parts list/bill of material	Provide a complete listing of all subassemblies, parts, and raw materials that compose the final (finished) component (or skid). Include the quantity of each item.
4	Installation, operation, maintenance, and lubrication manual(s)	Provide manuals for the components (or skid) being purchased. Manuals should include installation guidelines, detailed operating instructions with operating ranges, settings, etc. Also, include step-by-step startup, operating, and shutdown procedures and maintenance procedures for all required maintenance/repairs and lubrication schedule.
5	Recommended spare parts for 1 yr normal maintenance	Spare parts list will include the vendors' recommended listing of spare parts required for 1 yr, assuming that the system is cycled once a week (50 times/yr); the list is to include the tag number (if applicable), a description of each part sufficient for ordering, and the vendor's part number.
6	Certified Performance Report	Provide a Certified Performance Report that states that the instrumentation by tag number and serial number complies with the stated process ranges established in the stated specification. The Certified Performance Report must be signed and dated by an authorized person from the manufacturer or subsupplier who performed the test. Typically this testing is a destructive test, and the instrumentation being purchased was produced by the same manufacturing process with the same material supplier(s).
7	Wiring schematics	Provide drawings that show the following: *(a)* terminal strip/wiring numbering *(b)* starter, overloads, protective devices *(c)* ALL electrical components *(d)* instrumentation (electrical connections)
8	Instrument calibration reports	Calibration certificates or reports must be traceable to NIST or other internationally recognized and agreed-on calibration standards. They must also include the procedure used, calibration data/results, the calibration date, the person who performed the calibration, along with the serial number(s) of the standards or equipment used in the calibration process. NOTE: All calibration certificates or reports must contain the instrument serial number.
9	Sizing calculations	Given two or three of the following parameters, provide the sizing calculations, designated by tag number, for the design flow: *(a)* type of liquid and viscosity *(b)* piping size *(c)* flow For relief devices, the calculation to show the relieving flow, set pressure, back pressure, vacuum, specific gravity, viscosity, coefficient of discharge, back pressure coefficient, viscosity (or other applicable) coefficient, calculated required area in square inches; summary to show the manufacturer, model number, and the selected area.
10	Material Test Report for metallic materials	The Material Test Report for process contact metallic materials shall comply with the requirements listed in Part GR. REQUIRED ONLY FOR HYGIENIC APPLICATIONS
11	Certificate of Compliance for elastomers	The Certificate of Compliance for process contact elastomer materials shall comply with the requirements listed in Part GR. REQUIRED ONLY FOR HYGIENIC APPLICATIONS
12	Certificate of Compliance for surface finish	The Certificate of Compliance for surface finish must be uniquely identified by tag number, serial number, and/or model number; state the associated surface finish value in R_a or BPE designation per Part SF; and whether any polishing compounds were used to meet the stated specification. If polishing compounds are used they shall be inorganic and animal source material-free as stated on the Certificate of Compliance. The Certificate of Compliance must be signed and dated by an authorized person from the manufacturer. REQUIRED ONLY FOR HYGIENIC APPLICATIONS
13	Certificate of Compliance for polymer-based materials	The Certificate of Compliance for process contact polymer-based materials shall comply with the requirements listed in Part GR. REQUIRED ONLY FOR HYGIENIC APPLICATIONS

Table J-1.2-1 Vendor Documentation Requirements for New Instruments: Section 2, Instrument Types and Required Documents

Instrument Types	Required Documents (VDR Number)
Analytical element: condition/density/pH/resistivity	2, 3, 4, 5, 7, 10, 11, 12, 13
Conservation vent valve	1, 2, 3, 4, 5, 6, 9, 10, 11, 12, 13
Control damper: flow/humidity/pressure/temperature	2, 4
Control valve: analytical/flow/humidity/level/pressure/temperature	1, 2, 3, 4, 5, 7, 9, 10, 11, 12, 13
Controllers, indicating controllers	2, 3, 4, 5, 7
D/P transmitter: flow/level/pressure	2, 4, 7, 8, 10, 12
Damper actuator	2, 4, 7, 9
Electrical components	2, 4, 7
Flow element	1, 2, 3, 4, 5, 7, 8, 9, 10, 11, 12, 13
Flow orifice	2, 4, 10, 11, 12, 13
Flow switch: thermal	2, 3, 4, 5, 7, 10
Flow valve, automated valve assembly	1, 2, 3, 4, 5, 7, 10, 11, 12, 13
Indicator: flow/level	2, 3, 4, 5, 10, 11, 12, 13
Indicator: humidity/pressure/temperature	2, 4, 8, 10, 12
Level element	2, 3, 4, 5, 7, 10, 11, 12, 13
Level transmitter: microwave	2, 4, 7, 10, 11, 12, 13
Lighting	2, 4, 5, 7, 10, 12
Miscellaneous instruments: alarm/element/switch/transmitter	2, 4, 7
Positioner/transducer I/P: pressure/speed/temperature	2, 4, 7
Pressure element, pressure safety element	2, 4, 6, 10, 11, 12, 13
Pressure port	2, 4
Pressure safety (relief) valve	1, 2, 3, 4, 5, 6, 9, 10, 11, 12, 13
Recorder, indicating recorder	2, 4, 5, 7
Regulator valve: temperature/pressure	1, 2, 3, 4, 5, 10, 11, 12, 13
Sight glass	2, 3, 4, 5, 10, 11, 12, 13
Smoke detector, motion detector	2, 3, 4, 5
Solenoid valve	2, 4, 7
Switch: current/limit	2, 4, 7
Switch: analytical/flow/level/pressure/vibration	2, 3, 4, 5, 7, 10, 11, 12, 13
Temperature element: RTD	2, 4, 7, 8, 10
Temperature switch	2, 4, 7, 10, 12
Thermowell	2, 10, 12
Transmitter: analytical/flow/humidity/level/pressure/temperature/weight	2, 3, 4, 5, 7
Weight element	2, 3, 4, 5, 7, 8

NONMANDATORY APPENDIX K
STANDARD PROCESS TEST CONDITIONS (SPTC) FOR SEAL PERFORMANCE EVALUATION

(19)

K-1 SEALING COMPONENT PERFORMANCE EVALUATION

K-1.1 Material and Component Testing

Standard process test conditions are presented here in order to assess sealing components (hygienic union seal materials and diaphragm valve component seals). Typical steam operating parameters are presented in K-1.2.1. A simulated clean-in-place (CIP) and steam-in-place (SIP) test cycle is presented in K-1.2.2. Other process considerations are presented in K-1.2.3. Any specific process conditions that fall outside the design of this standard test should be evaluated separately.

The specific material composition(s) used in the test article shall be evaluated against the process conditions to which it may be exposed, including routine sterilization and cleaning. For diaphragm valve testing, the test article should reflect the specific valve configuration to be used in the anticipated application. Other conditions or process parameters/chemicals, such as allowable extreme process upset conditions and nonroutine treatments (e.g., passivation, derouging), should also be considered. Form S-1, Application Data Sheet, defines a number of operational conditions (e.g., chemistry, temperature, pressure) to consider when developing nonstandard performance tests.

Before testing

(a) verify that the material/component's service temperature and pressure rating meet the desired process conditions, including sterilization and cleaning.

(b) verify that the material/component is compatible with the intended process and cleaning chemicals at the routine concentrations used, including consideration for extreme allowable process conditions, per Part PM.

K-1.1.1 Test Article Requirements

(a) The following information, at a minimum, shall be included in test reports:

(1) seal type
(2) seal size
(3) sample size
(4) maximum rated pressure of valve or seal
(5) for valves only
(-a) actuator model number and spring pressure
(-b) air pressure supplied to actuator
(-c) valve type
(-d) valve size
(-e) valve model number

(b) Test samples shall be representative of a certain model or product range of seals and shall be chosen randomly from those fabricated with the standard manufacturing process. Any modifications that may impact performance shall be included in the test report, e.g., travel stops. Knowledge of factors that impact material performance may help determine the minimum selection criteria of samples. Appropriate study design or supporting data are required to support all conclusions. Any differences or factors that impact material performance across the commercial product range shall be addressed. This includes, but is not limited to, seal materials of construction, size, shape, and manufacturing process.

K-1.2 Exposure Testing

Sections K-1.2.1 and K-1.2.2 present two example test cycles that can be performed on test articles. Simulated SIP testing in K-1.2.1 incorporates a typical steam sanitization cycle. K-1.2.2 provides simulated CIP and SIP testing and additional information for valve cycling. Components can be evaluated with either or both of these tests based on application requirements. Actuation requirements in K-1.2.1 and K-1.2.2 apply to valve testing only.

K-1.2.1 Simulated Steam-in-Place Testing. Expose the material to multiple SIP cycles to establish a life expectancy for the application and configuration. The testing cycles should occur without intervention (e.g., retorquing of clamps or fasteners), beyond initial installation procedures. All deviations identified during the test program should be documented and analyzed, including their impact on the test results and conclusions.

The cycle will consist of the following:

(a) Initial Installation and Preparation. This typically includes assembly, cleaning, performance verification (routinely includes a thermal exposure cycle to allow the seals to set), seating of seals, and retorquing of valve clamps and fasteners, etc., per the manufacturer's procedures.

(b) Initial Performance Evaluation. Verify the initial performance of the sealing component per K-1.2.4.

(c) Steam-in-Place. Expose the system to a simulated SIP with saturated USP pure steam or equivalent (e.g., steam generated from DI/RO water or equivalent).

(1) System Temperature. Above 266°F (130°C).

(2) System Pressure. Saturated steam pressure.

(3) Test System Volume. A fixed volume of less than 2.6 gal (10 L) is recommended.

(4) Test Exposure Time. Minimum of one continuous hour greater than 266°F (130°C).

(5) Actuations. Minimum of ten actuations per cycle (at SIP temperature).

(d) Cool Down. Cool the system with ambient clean dry air.

(1) System Temperature. Ambient, as close to 77°F (25°C) as possible.

(2) System Pressure. 0 psig to 45 psig (0 bar to 3.1 bar).

(3) Cool-Down Target. Until the system reaches 77°F (25°C).

(e) Performance Evaluation. Assess the performance of the sealing component at appropriate intervals (e.g., initial, 10, 100, 500 cycles and final) per K-1.2.4.

(f) Repeat Steps. Repeat steps in paras. (c) and (d) .

(g) Final Performance Evaluation. Assess the performance of the sealing component at the completion of testing per K-1.2.4.

K-1.2.2 Simulated Combined CIP and SIP Testing. Expose the component to multiple CIP and SIP cycles to determine relative performance. Because this cycle is designed to assess material degradation, actual performance in service may differ. The testing cycles should occur without intervention (e.g., retorquing of clamps or fasteners) beyond initial installation procedures. All deviations identified during the test program should be documented and analyzed, including their impact on the test results and conclusions.

The cycle will consist of the following:

(a) Initial Installation and Preparation. This typically includes assembly, cleaning, performance verification (routinely includes a steaming cycle), seating of seals, and retorquing of valve clamps and fasteners, etc., per the manufacturer's procedures.

(b) Initial Performance Evaluation. Verify the initial performance of the sealing component per K-1.2.4.

(c) DI/RO Water Rinse — 5 min

(1) System Temperature. ≤104°F (≤40°C)

(2) System Pressure. 72.5 psig ± 7.3 psig (5 bar ± 0.5 bar)

(3) Actuations. 15 (10 sec open, 10 sec closed)

(d) Chemical Wash 1 — 30 min

(1) System Temperature. 176°F ± 9°F (80°C ± 5°C)

(2) System Pressure. 72.5 psig ± 7.3 psig (5 bar ± 0.5 bar)

(3) Actuations. 30 (5 sec closed, 55 sec open)

(4) Chemical. Sodium hydroxide (0.5N, 0.5M, 2% w/w)

(e) DI/RO Water Rinse — 5 min

(1) System Temperature. ≤104°F (≤40°C)

(2) System Pressure. 72.5 psig ± 7.3 psig (5 bar ± 0.5 bar)

(3) Actuations. 15 (10 sec open, 10 sec closed)

(f) Chemical Wash 2 — 30 min

(1) System Temperature. 176°F ± 9°F (80°C ± 5°C)

(2) System Pressure. 72.5 psig ± 7.3 psig (5 bar ± 0.5 bar)

(3) Actuations. 30 (5 sec closed, 55 sec open)

(4) Chemical. Phosphoric acid (0.36N, 0.12M, 1.2% w/w)

(g) DI/RO Water Rinse — 5 min

(1) System Temperature. ≤104°F (≤40°C)

(2) System Pressure. 72.5 psig ± 7.3 psig (5 bar ± 0.5 bar)

(3) Actuations. 15 (10 sec open, 10 sec closed)

(h) SIP — 30 min

(1) System Temperature. Saturated USP pure steam or equivalent (e.g., steam generated from DI/RO water or equivalent) ≥266°F (≥130°C)

(2) System Stabilization Time. 5 min

(3) Actuations. 150 (5 sec open, 5 sec closed)

(i) Cool Down. Cool system to <194°F (<90°C).

(j) Performance Evaluation. Assess the performance of the sealing component at appropriate intervals (e.g., initial, 10, 100, 500 cycles and final) per K-1.2.4.

(k) Repeat Steps. Repeat steps in (c) through (i) until the completion of testing or failure.

(l) Final Performance Evaluation. Assess the performance of the sealing component at the completion of testing per K-1.2.4.

K-1.2.3 Other Process Testing Considerations

K-1.2.3.1 Vacuum. The ability of the system to hold vacuum should be considered for routine process equipment, where applicable. Specific applications that require vacuum, such as autoclaves and lyophilizers, shall require the addition of a vacuum hold test requirement.

K-1.2.3.2 Additional Cleaning Chemicals. Additional integrated CIP test exposures should also be considered as part of the testing cycles. Specific cleaning chemicals and concentrations are determined by the process applications. Some systems, such as CIP systems, may be exposed to multiple cleaners. In addition to those listed in K-1.2.2, other cleaning agents such as sodium hypochlorite (0.67N, 0.67M, 0.5% w/w) should be assessed as required.

K-1.2.4 Performance Testing and Acceptance Criteria. Test the sealing components to evaluate their ability to maintain the component's integrity before, during, and after exposure (e.g., initial, 10, 100, 500

cycles and final), using the test procedures and acceptance requirements of EN-12266-1.

(a) For all sealing components, use test procedure and acceptance requirements for shell tightness, Test P11 of EN-12266-1.

(b) For valve sealing components, use test procedure and acceptance requirements of seat tightness, Test P12 of EN-12266-1 with maximum allowable seat leakage rate A.

K-1.3 Test Acceptance Criteria

K-1.3.1 Hygienic Fittings. The seal will be classified as a Level 10, 100, or 500 seal, if all of the following acceptance criteria are met after the corresponding number (10, 100, or 500) of SIP exposure/cool-down cycles:

(a) Pressure hold test shall be passed after the 10th, 100th, and/or 500th SIP exposure cycle.

(b) Compliance with SG-4.2 shall be established after the 10th, 100th, and/or 500th SIP exposure cycle (Intrusion Category I or II) for gaskets.

(c) The condition of the seal shall be examined after the 10th, 100th, and/or 500th SIP exposure cycle, and any direct visible changes (e.g., surface defects, compression marks, discoloration, or erosion) shall be recorded. Cracks, tears, or holes will be considered failures.

(d) Inspection at 0 (initial), 10 (through outlet), 100 (through outlet), and 500 (through disassembly of fittings) cycles.

K-1.3.2 Valve Diaphragms. The purpose of this section is to establish recommendations for evaluating diaphragm service life under specified process conditions in order to

(a) provide acceptance criteria for hygienic performance of diaphragms when conducting the performance evaluation test per this Appendix.

(b) provide additional observations that may be recorded after performing the test or when evaluating valve diaphragms that are in service.

K-1.3.2.1 General Requirements for Performance Evaluation Test for Valve Diaphragms, per This Appendix. Prior to testing, ensure that

(a) the manufacturer's installation and operational procedures are followed.

(b) a new diaphragm and new backing (if applicable) are used.

(c) the valve mating surfaces are dry and free of scratches and any residual material.

(d) the manual bonnet or actuator compressor is undamaged.

Figures K-1.3.2.1-1 through K-1.3.2.1-5 provide visual reference to the terminology used in this section.

K-1.3.2.2 Test Acceptance Criteria — Required Criteria for Hygienic Performance of Diaphragms

(a) After conducting performance evaluation testing per this Appendix, the following criteria will determine if the diaphragm has passed or failed the test:

(1) Valve passes pressure test criteria per this Appendix.

(2) Diaphragm stud is attached.

(3) Product contact surface is free of surface splits, tears, cracks, or blisters.

(4) Diaphragm flexes and inverts without displaying any cracks or tears to product contact surface.

(5) Reinforcement fabric, if present, does not penetrate into product contact surface.

(6) No visually apparent adherence of diaphragm material to valve seat.

(b) The following checks can also be recorded as observations after performing the test, where applicable. These are also useful when evaluating valve diaphragms that are in service.

(1) Check that the marking on the diaphragm, according to SG-3.3.2.3(b)(4), is present and legible.

(2) Check that the diaphragm stud is firmly attached, not rotated, and undamaged. The bayonet pin should be centered to the stud.

(3) Check for creases or marks that traverse the sealing bead. Some flattening of the bead is a normal occurrence from compression during assembly and/or operation. Uneven compression indicates improper assembly.

(4) Check that deformation into valve cavities aligns with the center bead and is not excessive.

(5) Check that compressor deformation marks on the backing align with the center bead and are not excessive. Extreme deformation is an indication of overclosure.

(6) Check that the backing is free of splits and cracks.

(7) Check that bolt holes are round and not elongated. Elongated bolt holes indicate overclosure and/or improper assembly or excessive service life.

(8) Check for any diaphragm discoloration. Discoloration of diaphragms may be unrelated to diaphragm performance (e.g., rouging) or an indication that the diaphragm is not suitable for the application (e.g., unsuitable diaphragm material selection).

(9) Check that there is no evidence of adherence of diaphragm material to the body/bonnet flange.

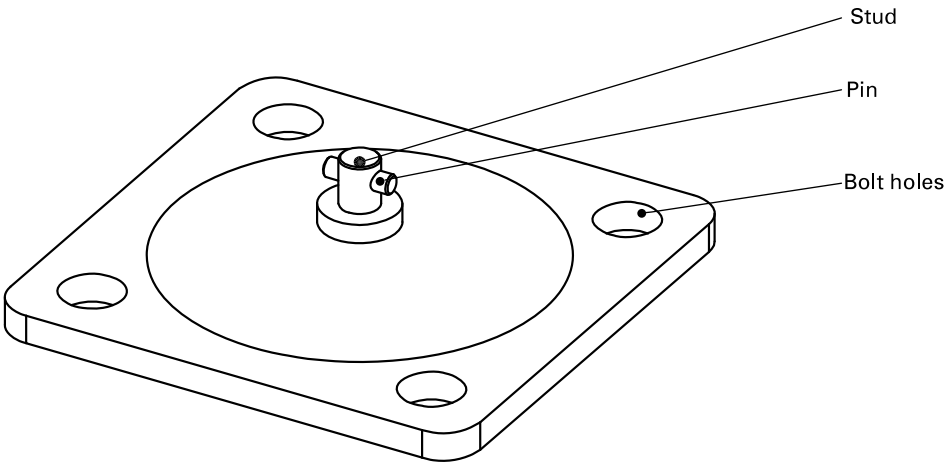
K-2 MECHANICAL SEAL PERFORMANCE EVALUATION

K-2.1 Mechanical Seal Performance Evaluation

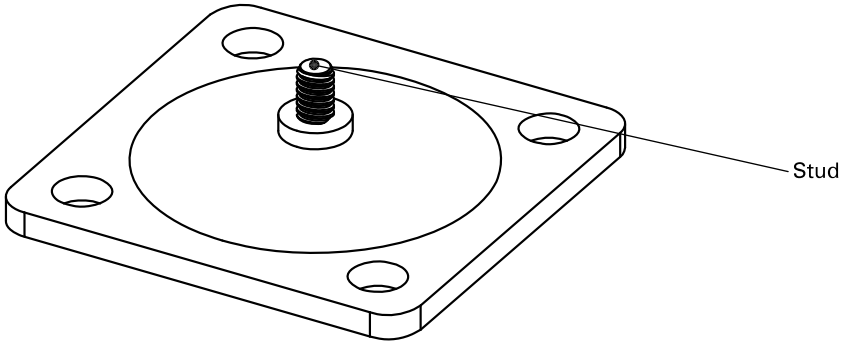
SG-4.3.2 of this Standard enumerates the various points, from manufacture to owner/user, that seal performance may be tested. This Standard recommends that the performance test of the supplier/manufacturer be accepted at each point. The reason for this is twofold, as follows:

(a) A mechanical seal is a complex piece of equipment, and seal designs have proven to be very reliable directly from the supplier/manufacturer.

Figure K-1.3.2.1-1 Weir-Style Diaphragm

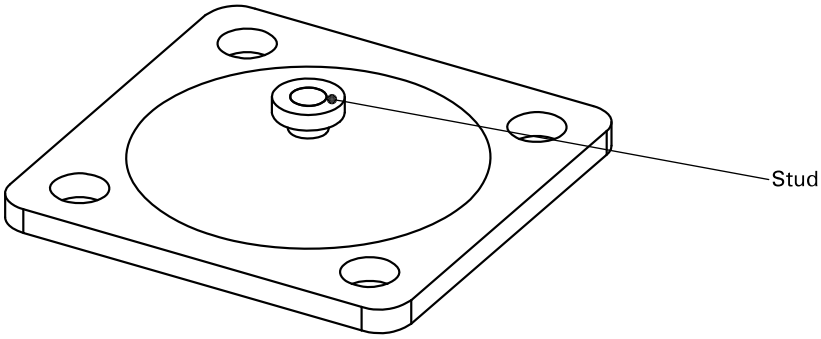


(a) Bayonet Style

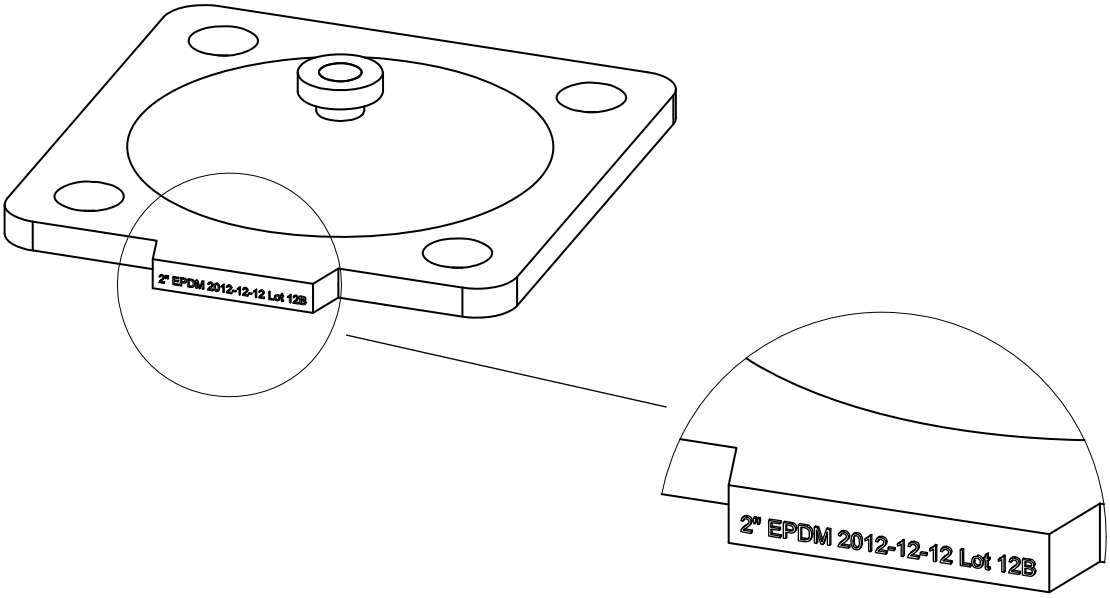


(b) Threaded Style

Figure K-1.3.2.1-1 Weir-Style Diaphragm (Cont'd)

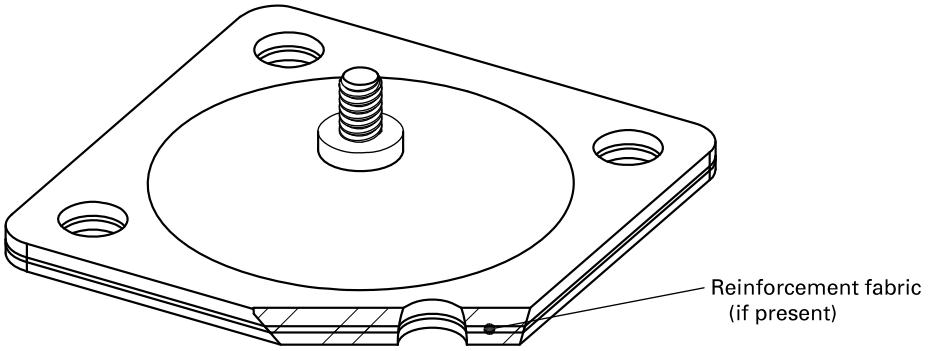


(c) Button Style

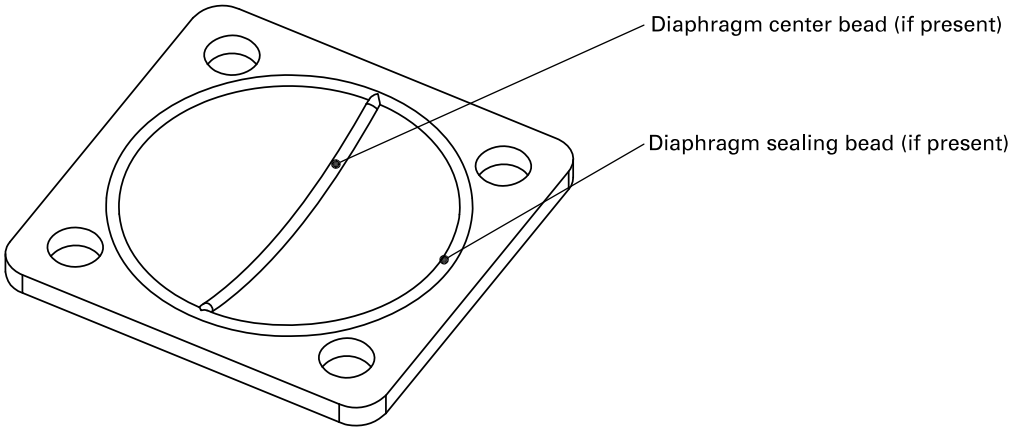


(d) Example of Diaphragm Marking

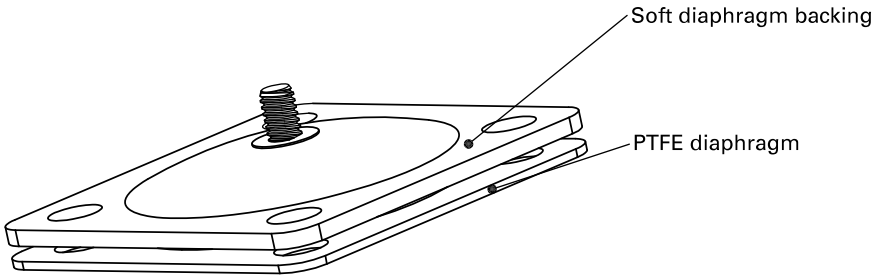
Figure K-1.3.2.1-1 Weir-Style Diaphragm (Cont'd)



(e)



(f)



(g)

Figure K-1.3.2.1-2 Weir-Style Body

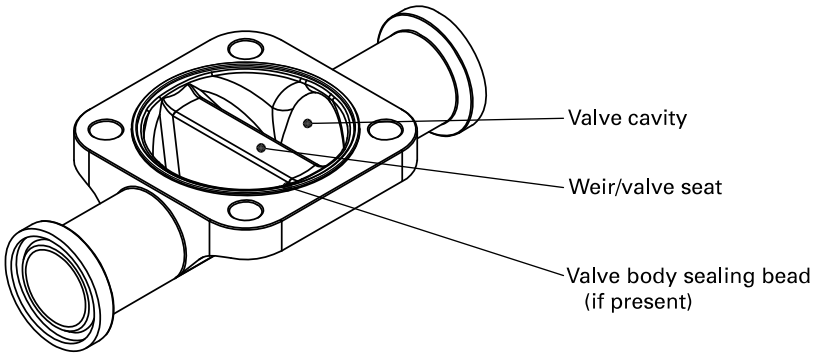


Figure K-1.3.2.1-3 Radial-Style Body

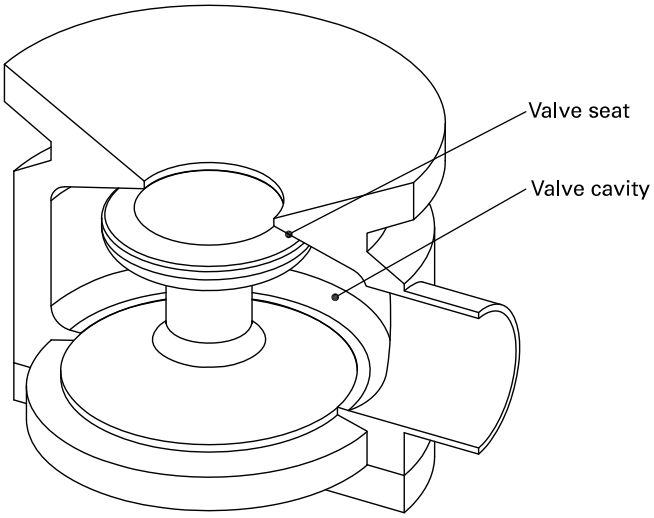


Figure K-1.3.2.1-4 Manual Bonnet

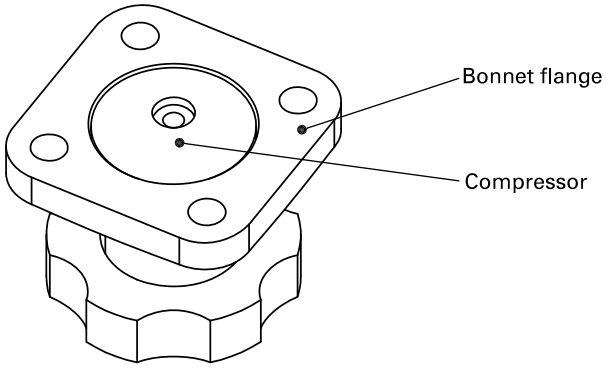
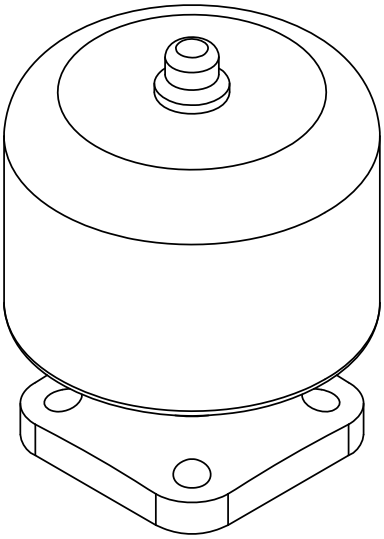


Figure K-1.3.2.1-5 Pneumatic



(b) A performance test conducted in an environment other than the process operating conditions and the specific piece of equipment provides little more than generalized results.

K-2.1.1 Factors Affecting Seal Performance. Seal performance may vary significantly depending on the environment in which the seal will operate. Mechanical seal designers shall take into account many factors, including the following:

(a) shaft speed (revolutions per time)
(b) shaft size
(c) process pressure
(d) process temperature
(e) tribological characteristics of the lubricating fluid
(f) weepage expectation
(g) equipment on which the seal operates
(h) start-stop operation
(i) barrier or buffer fluid availability
(j) multiprocess characteristics like CIP and SIP

K-2.1.2 Design Parameters. Once all process information is understood, the seal designer shall determine the following:

(a) seal face material(s)
(b) secondary seal material(s)
(c) type of lubrication such as boundary lubrication or full fluid film lubrication
(d) seal balance
(e) color of wearing materials
(f) flush (piping) plan

All of these factors affect operating life and weepage of the mechanical seal.

Seal performance is dictated by many factors. A properly designed, installed, and operated seal can exceed operational expectations. Many mechanical seals do not meet their operational life because of a variety of errors. Exceptions to normal seal wear that lead to failure are listed, in part, in K-2.2.

K-2.2 Exceptions to Normal Seal Performance

It is rare that end face mechanical seals "wear out." For a seal to be "worn out" implies that one or both of the primary seal faces have worn away due to normal rubbing friction. Three groups of examples to the exceptions to normal seal performance are listed in K-2.2.1 through K-2.2.3.

K-2.2.1 Event-Based Operational Failures. Examples of event-based operational failures are

(a) pressure reversals
(b) dead-heading pump
(c) process upset conditions
(d) tampering with seal support system or support system upset
(e) lubricating fluid becomes contaminated
(f) dry seal runs wet or liquid seal runs dry
(g) faces glue together during shut-off
(h) shock-induced failure — shaft

K-2.2.2 Design- and Application-Based Failures. Examples of design- and application-based failures are

(a) running seal dry when a liquid seal was designed or running seal wet when a dry seal was designed
(b) operation outside of seal design parameters
(c) improper selection of materials
(d) insufficient cooling
(e) dynamic secondary seal hang-up

K-2.2.3 Equipment-Based Failures. Examples of equipment-based failures are

(a) excessive run-out/deflection
(b) bolting distortion/equipment mounting flange flatness
(c) equipment alignment
(d) pipe strain and pipe support issues
(e) vibration
(f) bearing failure

K-2.3 Mechanical Seal Integrity Tests

Performance of mechanical seals may be tested several different ways. Test methods may vary between companies, and sometimes within a company. K-2.3.1 and K-2.3.2 provide a framework from which a test procedure may be drawn followed by a reasonable assessment of the test results. Unless otherwise specified, the test fluid is water for the liquid seals and oil-free compressed air or nitrogen for the gas seals. These tests will only verify the integrity of the seal faces and secondary seals. These tests do not reveal any information about the validity of the seal selection, expected seal life, or dynamic seal performance. The tests describe examples of effective methods to verify seal integrity. The owner/user may modify the test or pass/fail criteria.

K-2.3.1 Single Mechanical Seals: Liquid Lubricated by Process

(a) Wet Test. A single-cartridge seal or noncartridge seal shall be installed in equipment for a performance test. A liquid seal is tested dynamically with liquid lubrication. Visible leakage is a typical quantifier to verify integrity of a seal.

(1) Dynamic Wet Test for Liquid-Lubricated Single Mechanical Seal

Step 1. Follow all safety rules and regulations during assembly and testing of the seal.

Step 2. After the seal has been installed, flood the equipment with the test liquid, paying special attention that the seal chamber has been completely flooded.

Step 3. Confirm that the equipment is capable of withstanding the test pressure.

Step 4. Operate and pressurize the equipment.

Step 5. Observe the seal. If the test criteria have been met, the seal passes the test.

Step 6. If the seal fails the test criteria initially, consider operating the equipment longer to see if the seal wears in and passes the test.

Step 7. Document the results.

Step 8. If the seal does not pass the test, follow the procedure for resolution.

(2) Static Wet Test for Liquid-Lubricated Single Mechanical Seal

Step 1. Follow all safety rules and regulations during assembly and testing of the seal.

Step 2. After the seal has been installed, flood the equipment with the test liquid, paying special attention that the seal chamber has been completely flooded.

Step 3. Confirm that the equipment is capable of withstanding the test pressure.

Step 4. Pressurize the equipment.

Step 5. Observe the seal. If the test criteria are met, the seal passes the test.

Step 6. If the seal fails the test criteria initially, consider rotating the shaft manually a few turns.

Step 7. Document the results.

Step 8. If the seal does not pass the test, follow the procedure for resolution.

(b) Dry Test. A single-cartridge liquid mechanical seal or noncartridge liquid mechanical seal shall be installed in equipment for a performance test. A liquid seal tested dry with gas pressure, typically air or nitrogen, is tested statically. Gas flow across the seal is the typical quantifier to test the integrity of the seal. If the flow rate of gas across the seal is greater than 1 std. ft^3/hr/in. (1.1 L/hr/mm) shaft diameter at 30 psi (2 bar) the seal may be considered failed. Other pass/fail criteria may be applied if required.

(1) Static Dry Test for Liquid-Lubricated Single Mechanical Seal

Step 1. Follow all safety rules and regulations during assembly and testing of the seal.

Step 2. Confirm that the equipment is capable of safely withstanding the test pressure.

Step 3. Install an appropriately sized flowmeter and pressure regulator to the equipment.

Step 4. Seal all other openings in the equipment.

Step 5. Pressurize the equipment to 30 psi (2 bar). Do not block off the source of pressure. Hold the pressure constant.

Step 6. Let the pressure stabilize in the equipment and read the flowmeter.

Step 7. If the flowmeter reads less than 1 std. ft^3/hr/in. (1.1 L/hr/mm) of shaft diameter, the seal passes the test.

Step 8. If the seal does not meet the test criteria, consider turning the shaft slowly by hand to see if the flow rate is reduced.

Step 9. Document the results.

Step 10. If the seal does not pass the test, follow procedures for resolution.

(2) Static Dry Test for Liquid-Lubricated Single Mechanical Seal

Step 1. Follow all safety rules and regulations during assembly and testing of the seal.

Step 2. Confirm that the equipment is capable of safely withstanding the test pressure.

Step 3. Attach a pressure source and a pressure regulator to the equipment.

Step 4. Seal all other openings in the equipment.

Step 5. Pressurize the equipment to 30 psi (2 bar).

Step 6. Note time, pressure, volume, and temperature of the equipment.

Step 7. Block off the source of pressure.

Step 8. Wait for a designated time.

Step 9. Note the ending time, pressure, volume, and temperature of the equipment.

Step 10. Use the ideal gas law and the measured time to calculate the volume flow of gas over the seal per hour.

Step 11. If the calculations reveal less than 1 std. ft^3/hr/in. (1.1 L/hr/mm) of shaft diameter, the seal passes the test.

Step 12. If the seal does not meet the test criteria, consider turning the shaft slowly by hand to see if the flow rate is reduced.

Step 13. Document the results.

Step 14. If the seal does not pass the test, follow standard procedures for resolution.

K-2.3.2 Dual Mechanical Seals: Liquid Lubricated by Barrier Fluid (Dual Pressurized) or by Process and Buffer Fluid (Dual Unpressurized)

(a) Wet Test. Dynamic and static barrier and buffer fluid tests may be used to check the integrity of a dual mechanical seal. A dual-cartridge mechanical seal may be capable of being bench tested. A dual-component seal shall be installed in equipment for a performance test. However, it is not possible to view the inboard seal, and in many cases it is not possible to view the outboard seal. This means that a static or dynamic liquid test will only reveal if secondary seals are installed properly and if proper face contact is occurring.

(1) Static Wet Bench Test for Liquid-Lubricated Dual-Cartridge Mechanical Seals

Step 1. Follow all safety rules and regulations during assembly and testing of the seals.

Step 2. Confirm that the equipment and seal cartridge are capable of safely handling the test pressure.

Step 3. Find and plug appropriate ports in the seal cartridge.

Step 4. Connect the pressure line to the appropriate port in the seal cartridge.

Step 5. Important: Double check that bolting in the seal cartridge is adequate to hold the test pressure.

Step 6. Fill the seal cavity with test liquid, usually water, taking special care to purge the cavity of all air.

Step 7. Pressurize the seal to 30 psi (2 bar).

Step 8. Observe both ends of the seal cartridge. If no visible leakage occurs, the seal passes the test.

Step 9. Document the results.

Step 10. If the seal does not pass the test, follow the procedure for resolution.

(2) Static and Dynamic Wet Test for Dual Liquid Mechanical Seal Installed in Equipment. In this test it is not possible to view the inboard seal of the dual seal. Therefore the inboard seal will not be observed for visible leakage. It might be possible to view the outboard seal. Therefore these tests will only reveal if inboard secondary seals have been installed properly and are undamaged and if the inboard seal faces are in proper rubbing contact.

Step 1. Follow all safety rules and regulations during assembly and testing of the seals.

Step 2. Confirm that the equipment and seal are capable of safely handling the test pressure.

Step 3. For the dynamic test, confirm that the pressure, temperature, and flow rate of the barrier fluid or the buffer fluid are appropriate for the test.

Step 4. Connect the pressure line and pressure gauge to the appropriate port in the seal cavity.

Step 5. Fill the seal cavity with test liquid, usually water, taking special care to purge the cavity of all air.

Step 6. Pressurize the seal to 30 psi (2 bar).

Step 7. Shut "IN" and "OUT" valves to the seal to isolate the pressure for no more than 5 sec to avoid seal damage.

Step 8. Observe the pressure drop. If the pressure does not immediately drop, the seal parts are in place and operating and pass the test.

Step 9. Document the results.

Step 10. If the seal does not pass the test, follow the procedure for resolution.

(b) Dry Test for Liquid Mechanical Seal, Static Test Only. This is a bench test of a dual-cartridge seal or an installed-in-equipment test for a cartridge or component seal.

Step 1. Follow all safety rules and regulations during assembly and testing of the seal.

Step 2. Confirm that the equipment is capable of safely withstanding the test pressure.

Step 3. Attach a source of gas pressure, a pressure regulator, and a flowmeter to the seal.

Step 4. Plug all other openings in the equipment.

Step 5. Pressurize the seal to 30 psig (2 bar).

Step 6. Let the pressure equalize.

Step 7. Note the gas flow rate on the flowmeter.

Step 8. If the flowmeter reveals less than 1 std. ft^3/hr/in. (1.1 L/hr/mm) of shaft diameter, the seal passes the test.

Step 9. If the seal does not meet the test criteria and is installed in the equipment, consider turning the shaft slowly by hand to see if the flow rate is reduced.

Step 10. Document the results.

Step 11. If the seal does not pass the test, follow standard procedures for resolution.

(c) Dry Test for Gas Mechanical Seal Designed, Static, or Dynamic Test. This is a bench test of a dual-cartridge mechanical seal or an installed-in-equipment test for a cartridge or component mechanical seal.

Step 1. Follow all safety rules and regulations during assembly and testing of the seal.

Step 2. Confirm that the equipment is capable of safely withstanding the test pressure.

Step 3. Attach a source of gas pressure, a pressure regulator, and a flowmeter to the seal.

Step 4. Plug all other openings in the equipment.

Step 5. Pressurize the seal to 30 psi (2 bar) [for lift-off seals 50 psi to 60 psi (4 bar)].

Step 6. Let the pressure equalize.

Step 7. If it is a dynamic test, and it is safe to do so, operate the equipment.

Step 8. Note the gas flow rate on the flowmeter.

Step 9. If the flowmeter reveals less than 1 std. ft^3/hr/in. (1.1 L/hr/mm) (for lift-off seals 2 std. ft^3/hr/in.) of shaft diameter, the seal passes the test.

Step 10. If the test is performed statically and does not meet the test criteria and is installed in the equipment, consider turning the shaft slowly by hand to see if the flow rate is reduced.

Step 11. Document the results.

Step 12. If the seal does not pass the test, follow standard procedures for resolution.

K-2.4 Mechanical Seal Testing Notes

(a) Safety Precaution. If testing dual-cartridge mechanical seals that are not installed in the equipment, it is necessary to review the seal design. The seal cartridge shall be capable of containing the pressure injected into the seal chamber. Using compressible fluids can be a very dangerous method for bench testing dual seals.

(b) When gas is used as a test fluid, the volume of gas passing across the mechanical seal determines the seal integrity. Determining the volume of gas passing across seal faces is the preferred method when using a compressible fluid as a test fluid.

(c) Pressure drop of compressible fluid tests are acceptable when used consistently and checked against operational sealing success. Equipment manufacturers and assemblers use pressure drop tests that have been proven repeatable in the field. Experience of the OEM and assembler allows for accurate and repeatable interpretation of the results.

(d) Volume flow across a seal may be calculated using the ideal gas law if all the following information is known in the test system:

(1) initial pressure and final pressure

(2) initial temperature and final temperature

(3) volume of the system; constant and known

(4) the system is dry

(19)

NONMANDATORY APPENDIX L
STANDARD TEST METHODS FOR POLYMERS

L-1 STANDARD TEST METHODS FOR THERMOPLASTIC POLYMERS

ASTM C177, Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus
ASTM D256, Standard Test Method for Determining the Izod Pendulum Impact Resistance of Plastics
ASTM D543, Standard Practices for Evaluating the Resistance of Plastics to Chemical Reagents
ASTM D570, Standard Test Method for Water Absorption of Plastics
ASTM D638, Standard Test Method for Tensile Properties of Plastics
ASTM D648, Standard Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position
ASTM D785, Standard Test Method for Rockwell Hardness of Plastics and Electric Insulating Materials
ASTM D789, Standard Test Methods for Determination of Solution Viscosities of Polyamide (PA)
ASTM D790, Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electric Insulating Materials
ASTM D2240 or ISO 48, Standard Test Method for Rubber Property—International Hardness or Durometer Hardness
ASTM D3418, Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
Publisher: American Society of Testing and Materials (ASTM International), 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959 (www.astm.org)

L-2 STANDARD TEST METHODS FOR THERMOSET POLYMERS

ASTM D395 or ISO 815, Standard Test Methods for Rubber Property — Compression Set
ASTM D412 or ISO 37 — Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers — Tension
ASTM D471 or ISO 1817, Standard Test Method for Rubber Property — Effect of Liquids
ASTM D624, ISO 34, or ISO 816, Tear Strength
ASTM D2240 or ISO 48, Standard Test Method for Rubber Property — International Hardness or Durometer Hardness
Publisher: American Society of Testing and Materials (ASTM International), 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959 (www.astm.org)

L-3 THERMOSET POLYMER TEST PROPERTIES

Refer to Table L-3-1.

L-4 INTERPRETATION OF THERMOSET MATERIAL PROPERTY CHANGES

Refer to Table L-4-1.

L-5 TESTING PROTOCOLS FOR THERMOSET POLYMERS

L-5.1 Samples

Sample parts shall be prepared according to ASTM D412 (ISO 37). Samples tested per this specification shall be from the same formulation as finished parts.

L-5.2 Immersion Fluids

Test fluids and test temperatures for fluid immersions are as follows:

(a) sodium hydroxide — (0.5N, 0.5M, 2% w/w) at 176°F (80°C)

(b) phosphoric acid — (0.36N, 0.12M, 1.2% w/w) at 176°F (80°C)

(c) sodium hypochlorite — (0.67N, 0.67M, 0.5% w/w) at 176°F (80°C)

(d) saturated clean steam at 266°F (130°C)

Rinse samples with water to a neutral pH, or minimum conductivity, and dry before testing.

Additional test fluids and conditions may be specified.

Table L-3-1 Thermoset Polymer Test Properties

Property Change (From Original Value)	Test Designation	Description and Application (Reference Table L-4-1)
Fluid immersion (70 hr, 166 hr, and 502 hr at specified temperature) [Note (1)]	...	...
Compression set	ASTM D395B or ISO 815	Measure of recovery after deformation
Volume and/or weight	ASTM D471 or ISO 1817	Absorption of solvent or extraction of soluble constituents from elastomer
Hardness, IRHD, or Shore	ASTM D1415, D2240, or ISO 48	Changes related to solvent and/or temperature; higher numbers indicate harder material
100% modulus	ASTM D412 or ISO 37	Measure of force required to extend sample by 100%
Tensile strength at break	ASTM D412 or ISO 37	Force needed to stretch part to breaking point
Elongation at break	ASTM D412 or ISO 37	Percent elongation at break
Tear strength	ASTM D624 or ISO 34	Elastomer resistance to tear

NOTE: (1) Test duration times are 0/+2 hr.

L-5.3 Qualification Testing

Qualification testing should be performed on samples from each product formulation. Product properties shall be tested in accordance with the specifications listed in L-2, as applicable.

L-5.4 Elastomer Testing: Property Retention

Elastomer material testing requirements are listed in Table L-3-1. The test durations are 70 hr, 166 hr, and 502 hr. These tests indicate a minimum standard of acceptance and provide a guide regarding property changes with exposure time.

Table L-4-1 Interpretation of Thermoset Material Property Changes

Property Change	Measurement	Test Result Interpretation	Additional Comments
Hardness	Shore A, shore D scale, shore M scale (O-rings), or IRHD hardness. Usually measured in units of points	May indicate fluid absorption (increase) or extraction of ingredients (decrease); however, both absorption and extraction may occur simultaneously. A significant change in hardness may also indicate attack on the polymer backbone.	Relatively easy test to run. A significant decrease in hardness may result in increased abrasion. This is a macro measurement.
100% modulus	This is the stress required to reach 100% elongation.	Change may be caused by heat aging (increase) and/or chemical attack (decrease). Chemical absorption (decrease) or ingredient extraction (increase) can also affect modulus. Excessive increase in modulus may be a sign of polymeric embrittlement. Related to tensile strength and inversely related to elongation	Requires specialized equipment for measurement. Evaluates the elastomer on the micro level. Elastomer modulus should not be confused with modulus measurements for metals.
Tensile strength at break	Ultimate tensile strength recorded at material breakage	May indicate exposure to excessive heat (increase) and/or chemical attack (decrease)	Requires specialized equipment for measurement. Evaluates the elastomer on a micro level
Elongation at break	Ultimate elongation of sample measured at material breakage	May indicate exposure to excessive heat (decrease) and/or chemical attack (increase). Elongation (macro) and localized is important for sealing to avoid elastomer splits and cracks.	Requires specialized equipment for measurement. Evaluates the elastomer on a micro level. Especially important for flexing applications such as diaphragms
Compression set	Measures the ability of an elastomer to recover dimensionally after being subjected to compressive load, at a temperature, over time	Compression set is an indication of whether an elastomer is able to maintain sealing force. In general, the lower the compression set value, the better, especially if the application will involve temperature cycling. In this case, the elastomer has to maintain sealing through thermal expansion cycles.	Relatively easy test to run. Preferred to run at application temperature. Most important for applications involving O-rings or gaskets
Volume/weight	Measure weight gain/loss or volume increase/decrease	Volume swell and weight gain typically track together. Fluid exposure can result in fluid absorption (increase) or extraction of elastomer ingredients (decrease). Absorption of process fluid may or may not be a reversible process.	Weight and volume change are relatively easy to measure and may be the best indicators of performance. An increase due to absorption can result in product failure due to nibbling and extrusion. A decrease can result in leakage around the seal.
Tear strength	The ease at which a tear can be initiated and propagated	May indicate fluid absorption (decrease) or extraction of ingredients (increase). Property is typically related to change in elongation	Requires specialized equipment for measurement. May be useful data for applications involving diaphragms

NONMANDATORY APPENDIX M
SPRAY DEVICE COVERAGE TESTING

(19)

M-1 SCOPE

This Appendix defines an acceptable method for performing spray device coverage testing for bioprocessing equipment.

M-2 PURPOSE

The purpose of a spray device coverage test is to document fluid coverage of the process contact surfaces of bioprocessing equipment. The test provides information about fluid coverage and the conditions necessary to achieve this coverage as a prerequisite for cleaning of the process equipment. The coverage test is not intended to demonstrate cleanability, but rather the ability to deliver cleaning solutions to the target surfaces. Cleanability is verified using a full CIP protocol during cleaning validation.

M-3 MATERIALS

(a) A concentration of 0.08 g/L to 0.22 g/L riboflavin (vitamin B2) aqueous solution provides visible fluorescence under ultraviolet light. The riboflavin should be free of animal-derived ingredients (ADI). Riboflavin is water soluble, noncorrosive, and nonreactive on materials commonly used to manufacture bioprocessing equipment (e.g., stainless steel, polymers, and ceramics). Riboflavin fluoresces with exposure to long-wavelength ultraviolet (UV) light with peak intensity at 365 nm. Note that if other fluorescent materials are used, the UV wavelength for optimum visibility may be different.

(b) UV lamps are available with different wavelengths and intensities. A lamp with a peak wavelength of 365 nm and an intensity of 4 000 $\mu W/cm^2$ at a distance of approximately 15 in. (38 cm) is optimal to observe riboflavin fluorescence. Ultraviolet lamp intensity is inversely proportional to the square of the distance from the source. Ultraviolet lamps of this intensity may present a safety hazard to the eyes and skin. Personal protective equipment (PPE) is recommended. UV lamps of other wavelengths can be used, but stronger concentrations of riboflavin may be required for detection.

(c) An extension mirror or borescope camera may be useful for visual inspection of hard-to-reach areas.

(d) The quality of water used for the formulation of the riboflavin solution and for coverage testing shall be agreed to by the owner/user and manufacturer. The minimum acceptable water quality is noncompendial purified water (e.g., reverse osmosis or deionized).

M-4 PROCEDURE

M-4.1 Equipment Preparation

(a) All internal appurtenances should be installed (e.g., agitators, level probes, and dip tubes) during the spray device coverage testing. If conducting the test with all interior appurtenances in place is not practical, alternative means to simulate shadowing should be agreed on with the owner/user (e.g., dummy shafts and dip tubes may be used). If the agitator is installed, it should be rotated at the same rate as planned for CIP.

(b) All internal surfaces and appurtenances shall be clean prior to the coverage test. Contaminated surfaces (e.g., with grease or oil) may produce inconclusive results.

(c) Verify that the spray device(s) are installed in the designed location and orientation (where applicable).

M-4.2 Application of Fluorescent Solution

(a) The test shall be performed by spraying the fluorescent solution as a mist on all targeted surfaces of the bioprocess equipment including walls, nozzles, baffles, and other appurtenances. The solution application should minimize droplet formation and runoff. Care should be taken to avoid applying the fluorescent agent to areas that are outside of the process boundary (e.g., the side of the manway gasket that is not exposed to the process). Note that the inside of dip tubes or similar hollow members not targeted by the spray device may require a separate rinse path during the test.

(b) Using an ultraviolet light permits visual verification that the targeted surfaces have been wetted with the fluorescent solution. Fluorescent agents such as riboflavin typically fluoresce only when they are wet.

(c) The riboflavin application inspection methods shall be consistent with the postrinse inspection methods.

M-4.3 Execute Rinse

(a) The rinse should be performed with ambient (or colder) temperature water to allow for immediate inspection of wet surfaces. The use of other temperatures shall be agreed on with the owner/user.

(b) The rinse should be performed before the riboflavin solution has dried, as the test is designed to confirm coverage and not cleaning.

(c) The rinse shall be performed in a once-through mode.

(d) Conditions such as flow rate, pressure, and time shall be recorded during the coverage test as described in M-6.

M-4.4 Inspection

(a) Inspection should be performed before the surfaces dry. Surfaces must be wet to detect riboflavin fluorescence.

(b) If surfaces are dry at the time of inspection, the surfaces shall be gently rewetted from bottom and up with ambient or cold water to observe any residual riboflavin fluorescence. Rewetting and inspecting lower surfaces first and higher surfaces next will reduce the likelihood of misidentification of the location of residual riboflavin.

(c) Ambient light should be minimized to improve the visibility of riboflavin fluorescence.

(d) The postrinse inspection methods shall be consistent with the riboflavin application inspection methods.

(e) For large enclosures (e.g., vessels with manways) confined space entry may be necessary to conduct a thorough inspection.

(f) The inspection sequence should be designed to avoid false results due to transfer of residual riboflavin from internal or external sources.

M-5 ACCEPTANCE CRITERIA

(a) Acceptance criteria and coverage test protocol shall be agreed on with the owner/user before the coverage test.

(b) A typical acceptance criterion is removal, to the limit of visual detection, of the riboflavin solution from all targeted areas or as otherwise agreed on with the owner/user.

(c) If areas of residual riboflavin are present, they should be documented, and a corrective action plan should be established with the owner/user.

M-6 RECOMMENDED DOCUMENTATION

(a) Test configuration sketch (reference the OEM drawing) and description (with, e.g., line size, instrument locations, elevation)

(b) Spray Device Data

(1) Model, make, serial number, and tag number.

(2) Verify correct installation, orientation, down pipe, and down pipe length.

(3) Recommended pressure and flow conditions (data sheet).

(c) Instrument Data

(1) Data sheets (instrument ranges)

(2) Calibration certificates for instruments

(d) Riboflavin Solution Data

(1) Riboflavin catalog number and lot number

(2) Expiration date

(3) Amount of riboflavin

(4) Amount of water and quality

(5) Time and date of preparation

(6) Time and date of application and preinspection

(7) Time and date of rinse and postinspection

(e) UV Lamp Data. Model number and data sheet

(f) Temperature of Rinse Water

(g) For initial flow path and each subsequent transition to a different flow path, document

(1) Flow rate

(2) Time (burst/delay sequence, if applicable)

(3) Pressure (measured as close to the spray device as practical)

(4) For dual-axis dynamic spray devices, time, flow, and pressure to complete a pattern

(h) Test Results

(1) Pass/fail

(2) If applicable, residual riboflavin location(s) and descriptions

(3) If applicable, corrective actions taken

NONMANDATORY APPENDIX N
COMMENTARY: UNS S31603 WELD HEAT-AFFECTED ZONE DISCOLORATION ACCEPTANCE CRITERIA

(19)

(a) The acceptance criteria for discoloration on weld heat-affected zones were developed by making autogenous square groove welds on 2-in.-diameter UNS S31603 stainless steel tube-to-tube butt joints whose inside diameters were purged with argon containing controlled amounts of oxygen. The oxygen levels reported were measured on the downstream side of the welds. For the sample numbers listed in Figures MJ-8.4-2 and MJ-8.4-3, the oxygen contents were as follows:

(1) #1a and #1b — 10 parts per million (ppm)
(2) #2 — 25 ppm
(3) #3 — 35 ppm
(4) #4 — 50 ppm
(5) #5 — 80 ppm

(b) All welds were made with the gas-tungsten arc welding (GTAW) process using 95% argon — 5% hydrogen shielding gas.

(c) The electropolished tubing used for the test welds had an SF4 surface finish (15 μin. R_a max.) and the mechanically polished tubing had an SF1 surface finish (20 μin. R_a max.).

(d) The photos shown in Figures MJ-8.4-2 and MJ-8.4-3 were taken using a camera having direct visual access to the weld surfaces.

(e) The corrosion resistance of the welded samples was determined by both ASTM G150, Critical Pitting Temperature Test, and the modified ASTM G61, Potentiodynamic Polarization Corrosion Test.

(f) ASTM G150 determines the voltage-independent critical pitting temperature (CPT) by way of a potentiostatic technique that determines the temperature above which pitting corrosion proceeds on its own under standardized test conditions. Higher CPTs indicate increased resistance to pitting corrosion.

The modified ASTM G61 determines the voltage (potential) at which the anodic current increases rapidly during a standardized cyclic polarization test at room temperature. The voltage determined, referred to as the E_{PIT}, is a measure of resistance to pitting corrosion. Higher, or more noble, values of E_{PIT} indicate increased resistance to pitting corrosion.

Neither the CPT nor the E_{PIT} values determined are material properties per se; rather, they are the result of standardized tests designed to rank different materials or different surface finishes of the same material in their resistance to the stable propagation of pits in a standard test environment.

(g) The acceptable levels of discoloration identified in Figures MJ-8.4-2 and MJ-8.4-3 are based on corrosion resistance, not on the oxygen levels of the internal purge gas used during welding. As a result, the photographs in Figures MJ-8.4-2 and MJ-8.4-3 should be used to identify the degree of discoloration by number, but not to specify the amount of oxygen in the backing gas.

(h) All welds were tested in the as-welded condition, with no postweld conditioning.

(i) For the electropolished tubing in Figure MJ-8.4-2, acceptable levels of heat-affected zone discoloration were those that exhibited corrosion resistance similar to unwelded, electropolished UNS S31603 base metal in the ASTM G150 test.

(j) For the mechanically polished tubing in Figure MJ-8.4-3, acceptable levels of heat-affected zone discoloration were those that exhibited corrosion resistance similar to that of a cold-rolled, mill-finished, UNS S31603 base metal.

(k) It is generally accepted that as-welded heat-affected zones on mechanically polished tubing having the same level of discoloration as weld heat-affected zones on electropolished tubing will exhibit lower resistance to pitting than the heat-affected zone on electropolished tubing.

(l) The user is cautioned that the amount of discoloration and its appearance can be influenced by factors other than oxygen, as listed below:

(1) High levels of moisture in the backing gas can increase the degree of discoloration.

(2) Other contaminants, such as hydrocarbons, moisture, and some types of particulates on the surface prior to welding, can all affect discoloration levels.

(3) Hydrogen in the argon backing gas can significantly reduce the amount of discoloration.

(4) The metal's surface finish can also affect the appearance of the discoloration.

NONMANDATORY APPENDIX O
GUIDANCE WHEN CHOOSING POLYMERIC AND NONMETALLIC MATERIALS

(19)

O-1 GENERAL

Polymer materials can be divided into two general classes: thermoplastics and thermosets. The composition, form, and construction of these materials determine their suitability for use in their various applications, and the systems designer should be aware of their strengths and limitations.

Polymer materials may be manufactured from a single monomer (homopolymer) or multiple monomers (copolymers). They may be filled or unfilled. They may be elastomeric or rigid. They may exist in an amorphous, crystalline, or semicrystalline state. They may consist of either single or multiple microphases, be manufactured as composites, and include adhesive materials.

Nonmetallic materials may be rigid or flexible, amorphous or crystalline, exist in single or multiple microphases, and formed into complex mixtures and composites. These materials can offer a range of unique properties (e.g., extreme hardness, chemical inertness, self-lubrication, or transparency). The system designer and owner/user should be aware of the broad range of physical and chemical properties of these materials.

O-1.1 Gamma Irradiation

Gamma irradiation of polymers may change the physical properties of the polymeric material (see Mandatory Appendix III-11.1). Other effects may include discoloration and generation of low-molecular-weight compounds. Antioxidants or stabilizers may have been added to the polymeric material to minimize changes in physical properties.

O-2 PARTICULATES

O-2.1 Characterization

Particulates are characterized by several attributes including, but not limited to, size, morphology/shape, hardness, and chemical composition. These attributes may affect the ability to detect, measure, capture, identify, and control the particulates.

(a) Physical Form. Particulates may be of varying shapes. Some may be long and thin while others may be short and round. A majority of the particulates are irregular shapes with complex geometries.

(b) Material Makeup. Intrinsic particulates are native to the single-use system and include materials of construction or ingredients in the process formulation. Extrinsic particulates are foreign to the single-use system and come from the manufacturing environment or process personnel.

(c) Hardness. Particulates may be rigid or soft.

(d) Chemical Composition

O-2.2 Levels of Acceptance

Acceptable levels of particulate quantity and size distribution in single-use components and assemblies should be determined by their intended use. Owner/users should use established industry standards for the end product to quantify acceptable particulate criteria. Some of the industry standards available as acceptance criteria for drug products include USP <1>, USP <787>, USP <788>, USP <790>, EP 2.9.19, EP 2.9.20, JP 6.06, and JP 6.07.

NONMANDATORY APPENDIX P
GENERAL BACKGROUND/USEFUL INFORMATION FOR EXTRACTABLES AND LEACHABLES

(19)

P-1 REFERENCES

21 CFR 211.94, Code of Federal Regulations, Part 211, Current Good Manufacturing Practice for Finished Pharmaceuticals

Guidance for Industry-Container Closure Systems for Packaging Human Drugs and Biologics, FDA/CDER/CBER

ICH Q3A, Guidance for Industry, FDA, "Impurities in New Drug Substances"

ICH Q9, Guidance for Industry, FDA, "Quality Risk Management"

Publisher: U.S. Food and Drug Administration (FDA), 10903 New Hampshire Avenue, Silver Spring, MD 20993 (www.fda.gov)

"Accumulation of Organic Compounds Leached From Plastic Materials Used in Biopharmaceutical Process Containers"

Publisher: Parenteral Drug Association (PDA), Bethesda Towers, 4350 East West Highway, Suite 600, Bethesda, MD 20814 (www.pda.org)

"BPSA Recommendations for Testing and Evaluation of Extractables From Single-Use Process Equipment," Bio-Process Systems Alliance, 2010

Publisher: Bio-Process Systems Alliance (BPSA), 1400 Crystal Drive, Suite 630, Arlington, VA 22202 (www.bpsalliance.org)

EP 3.1.5, Polyethylene With Additives for Containers for Parenteral Preparations and for Ophthalmic Preparations

EP 3.1.9, Silicone Elastomers for Closures and Tubing

EP 3.1.13, Plastic Additives

Publisher: European Directorate for the Quality of Medicines & Healthcare (EDQM Council of Europe), 7 allée Kastner, CS 30026, F-67081 Strasbourg, France (www.edqm.eu)

"Extractables and Leachables: Challenges and Strategies in Biopharmaceutical Development"

"Recommendations for Extractables and Leachables Testing, Part 1: Introduction, Regulatory Issues, and Risk Assessment"; Part 2: "Executing a Program"

"Toward Industry Standardization of Extractables Testing for Single-Use Systems: A Collective BPSA Perspective"

Publisher: BioProcess International, 2 International Place, Boston, MA 02110-2601 (www.bioprocessintl.com)

ISO 10993-18: 2005(E), "Biological Evaluation of Medical Devices," Part 5: "Tests for In Vitro Cytotoxicity"; Part 13: "Identification and Quantification of Degradation Products From Polymeric Medical Devices"; Part 16: "Toxicokinetic Study Design for Degradation Products and Leachables"; Part 17: "Establishment of Allowable Limits for Leachable Substances"; Part 18: "Chemical Characterization of Materials"

Publisher: International Organization for Standardization (ISO), Central Secretariat, Chemin de Blandonnet 8, Case Postale 401, 1214 Vernier, Geneva, Switzerland (www.iso.org)

Jenke, D., Compatibility of Pharmaceutical Solutions and Contact Materials Safety Assessments of Extractables and Leachables for Pharmaceutical Products

Publisher: John Wiley & Sons, 111 River Street, Hoboken, NJ 07030-5774 (http://www.wiley.com)

PQRI — Safety Thresholds and Best Practices for Extractables and Leachables in OINDP

Publisher: Product Quality Research Institute (PQRI), 1500 K Street, N.W., 4th Floor, Washington, DC 20005-1209 (www.pqri.org)

"Standardization of Single Use Components' Extractable Studies for Industry"

"Standardized Extractables Testing Protocol for Single-Use Systems in Biomanufacturing"

Publisher: International Society for Pharmaceutical Engineering (ISPE), 3109 W. Dr. Martin Luther King, Jr. Blvd., Tampa, FL 33607 (www.ispe.org)

USP <88>, Biological Reactivity Tests, In Vivo

USP <232>, Elemental Impurities Limits

USP <661.1>, Plastic Materials of Construction

Publisher: U.S. Pharmacopeial Convention (USP), 12601 Twinbrook Parkway, Rockville, MD 20852-1790 (www.usp.org/usp-nf)

P-2 RECOMMENDED CONDITIONS FOR A POLYMERIC MATERIAL-SPECIFIC EXTRACTION

(a) Surface Preparation. Materials of construction with a high likelihood for contact.

(b) Sample Size. 60 cm^2/20 mL of extract fluid or 0.2 g/1 mL of extract fluid.

(c) Test Temperature. When a soxhlet extractor is used, fluid temperature is controlled by the condenser, near room temperature. For other methods of solvent extraction, the temperature may be elevated compared with the anticipated actual use conditions.

(d) Test Solvents. Use of one compatible polar solvent (e.g., ethanol, DI water) and one nonpolar solvent (e.g., hexane or toluene) to maximize extraction.

(e) Test Time. 24 hr.

P-3 RECOMMENDED CONDITIONS FOR AN EXTRACTION STUDY IN BIOPROCESS MODEL SOLUTIONS

(a) Surface Preparation. Final article and contact surface should be used.

(b) Sample Size. 60 cm^2/20 mL of fluid or 0.2 g/1 mL of fluid.

(c) Test Temperature. 40°C.

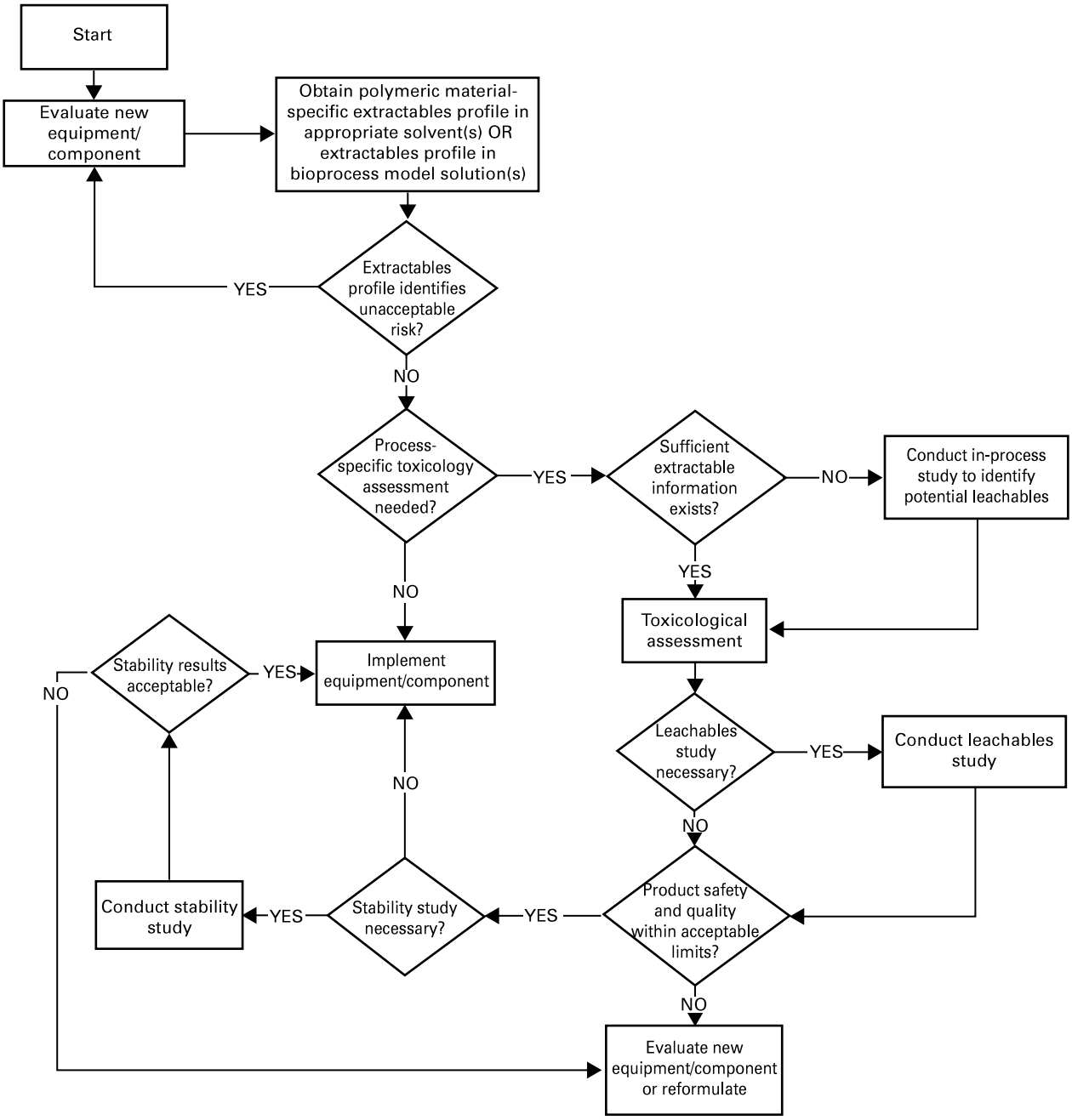
(d) Test Fluids. 20% ethanol (v/v), 0.1 M phosphoric acid (H_3PO_4), 0.1 M sodium hydroxide (NaOH), 5.0 M salt solution (NaCl), and/or purified water (meets USP requirements, at a minimum).

(e) Test Time. 30 days, unagitated.

P-4 OVERVIEW OF BIOPROCESSING EQUIPMENT/ COMPONENT EVALUATION RELATED TO EXTRACTABLES AND LEACHABLES CHARACTERIZATION

See Figure P-4-1.

Figure P-4-1 Flowchart of Bioprocessing Equipment/Component Evaluation Related to Extractables and Leachables Characterization



NONMANDATORY APPENDIX Q
(19) TEMPERATURE SENSORS AND ASSOCIATED COMPONENTS

Q-1 GENERAL

This Appendix presents additional information not addressed in PI-7 on temperature sensors and various influences on sensor performance.

Q-1.1 General Considerations

Platinum-based resistance temperature detectors (RTDs) are the most commonly used temperature-sensing technology. Alternative temperature measurement technologies are available that may be selected based on system design and owner/user preference. Manufacturers of the temperature sensors can confirm that the selected instrument meets the specified performance requirements in the environmental conditions at the installation location.

Q-2 EXTERNAL SUPPORT COMPONENTS

Temperature sensor assemblies may include components external to the sensor that will influence the measurement accuracy. Assemblies typically may include enclosures, wire and cables, and transmitters. The instrument manufacturer can provide guidance on appropriate external components needed to meet the required measurement accuracy of the system.

Q-3 MEASUREMENT ACCURACY

Total measurement accuracy includes, but is not limited to, sensor accuracy, installation effects on accuracy, wiring and cabling influences, electronics accuracy, process influences, and ambient influences.

Q-3.1 Sensor Accuracy

The sensor manufacturer's stated accuracy represents the sensor performance as verified by the manufacturer's calibration laboratory. The sensor can be expected to meet the interchangeability criteria based on the tolerance class stated by the manufacturer and defined by the industry standards per PI-7.4.1.

A multipoint calibration can be performed to define the actual resistance vs. temperature relationship of the specific RTD. This calibration data can be used to improve the measurement accuracy.

Q-3.2 Wiring and Cabling

Sensor wiring configuration and wire length will influence the measurement accuracy due to resistance variation between lead wires and the lead wire compensation technique used.

(a) For RTDs, accuracy is influenced by lead wire compensation, specifically when long cable lengths are used. Four-wire designs provide the most effective compensation. Three-wire designs are effective for shorter cable lengths. Two-wire designs are generally not recommended by sensor manufacturers unless a closely mounted transmitter is incorporated into the instrument assembly.

(b) RTDs with long cable runs typically use shielded cable to minimize the influence of electrical noise along the cable run.

(c) For commonly used thermocouples, it is best to match the extension wire type with the thermocouple type. For thermocouple types R, S, and B (platinum based), alternate extension wire is commonly used. The manufacturer can provide guidance regarding extension wire choices, including when to use shielded wire.

Q-3.3 Electronics

The accuracy and stability of electronic devices and control systems should be included in the assessment of the total measurement accuracy.

Matching electronics to sensor calibration data is an effective way to improve measurement accuracy and is preferred for process measurements where process system accuracy better than ±3°F (1.5°C) is required.

Electronics have an input resistance limitation that limits sensor cable lengths. Input resistance should not exceed the capability of the electronics. Refer to the manufacturer's specifications.

Q-3.4 Process Influences

Stagnant flow, heating/cooling sources, valves, tubing construction, and other instruments near the fluid temperature measurement location can also influence the measurement accuracy.

Q-3.5 Ambient Influences

(a) Environmental Influences. Moisture and other environmental conditions near the measurement location can influence the measurement and/or damage the sensor. Sensor installation should incorporate wire/cable connector and/or an enclosure (connection head). The enclosure rating should meet or exceed the NEMA rating (or international equivalent) for the installation location as defined by the owner/user. Selection of an appropriate enclosure should be based on ambient conditions during system operation and cleaning/sterilization cycles.

(b) Ambient Temperature Effects. For sensor assemblies with an internal transmitter, the maximum operating temperature of the transmitter should be considered when insulating the assembly to minimize the influence of the ambient temperature (see PI-7.4.4).

Q-4 SELECTION

Q-4.1 Sensor Selection

(a) Consider the cable/wire length necessary to support removal for calibration or other maintenance activities.

(b) RTD-based sensors will generally provide the most accurate temperature measurement.

(c) A thermocouple-based sensor may be an effective choice where the accuracy requirements can be achieved and the control system accepts a millivolt (mV) input.

(d) Bimetallic, mechanical temperature sensors are typically used only for local indication due to the limited measurement range and accuracy.

(e) Liquid-in-glass (LiG) temperature sensors are not an effective choice due to various performance, capability, and environmental concerns.

Q-4.2 Transmitter Selection

When a transmitter is required, a sensor/transmitter combined calibration is preferred to achieve the best accuracy.

Q-5 MAINTENANCE

Sensor maintenance should include calibration verification and general sensor inspection.

Q-5.1 Sensor Calibration Verification

(a) Methods. Specific calibration verification approaches should be according to the manufacturer's recommendations and specific process system performance requirements.

Sensor/transmitter combined calibration should be verified as a system. The association between sensor/transmitter system components should be maintained through tagging and/or serialization.

When verifying sensor or sensor/transmitter system accuracy, the typical verification temperature is the midpoint of the process operating temperature. At a minimum, sensors should be verified at 32°F (0°C).

Bimetallic sensors should be calibrated in the orientation of final use.

(b) Frequency. Sensor calibration should be verified annually. Alternate verification frequency may be specified based on criticality of the measurement and historical sensor verification data.

Q-5.2 Sensor Inspection

An insulation resistance test (sensor lead wire to sensor body) on all RTDs and ungrounded thermocouples should be performed during periodic verification. Insulation resistance should be tested per the manufacturer's specification.

Physical inspection should be performed at each periodic verification event per the manufacturer's specification for the specific sensor, including an examination of overall condition and cleanliness of the sensor. The manufacturer's recommendations should be followed regarding cleaning, repair, or replacement if the sensor or thermowell exhibits indications of wear, damage, or other conditions that may affect performance or the useful life of the instrument.

(19) NONMANDATORY APPENDIX R INSTRUMENT RECEIVING, HANDLING, AND STORAGE

R-1 INTRODUCTION

This Appendix is a supplement to PI-3.

R-2 INSTRUMENT RECEIVING

The instrument(s) shall be verified against the packing slip prior to items leaving quarantine for release to inspection and/or storage.

R-2.1 Warnings and Documentation

Refer to the product manual for any warnings and/or notices (e.g., ANSI Z535.6) regarding the instrument and comply accordingly. Documentation, such as calibration reports, material traceability, etc., should be kept with the instrument or handled per the owner/user's document control procedures.

R-2.2 Incoming Inspection

Incoming inspection shall be performed to check for manufacturing defects per Parts DT and SF and/or other standards and internal quality criteria.

R-3 INSTRUMENT HANDLING

Many instruments are assemblies of components. If it is necessary to disassemble the instrument to component level, component control is required.

R-3.1 Instrument Assembly Segregation

When disassembling an instrument, each instrument assembly shall be segregated or kitted from other instrument assemblies to avoid mixing of components.

R-3.2 Component Labels

Each component should, when possible, have a printed, individual, waterproof component label that includes information such as the instrument name, component part description, serial number, P & ID location, or barcode. Each component should have a unique part number. The preferred method for doing this is to use the serial number followed by (A) — first component; (B) — second component; etc.

R-4 STORAGE

After receiving and inspections, instruments shall be packaged for storage to protect them from environmental conditions and contamination. The outside of the packaging shall be labeled to clearly identify the stored instrument.

NONMANDATORY APPENDIX S
APPLICATION DATA SHEET

(19)

See Form S-1 beginning on the next page.

Form S-1 Application Data Sheet

Identification # _____
Tag # _____
P & ID # _____

Client: _____
Project: _____
Location: _____
Date: _____

Application/ Operation	Service	Application Details		Normal Operating Temperature, °F or °C [Note (1)]	Operating Low Temperature, °F or °C	(High) Design Temperature, °F or °C	Normal Operating Pressure, psig or bar [Note (2)]	(High) Design Pressure, psig or bar	Shutoff Pressure (0%/100%), ΔP [Note (3)]	Cycle Frequency, per day/wk/yr	Duration, hr/day
	Size										
	Rupture Disk Set Pressure	Pressure _____ psi (bar)									
		Service Descriptions	**Concentration, %**								
	Process Fluid										
Steam	Continuous ☐ Yes ☐ No Intermittent ☐ Yes ☐ No										
Purified Water/WPI	☐ Yes ☐ No										
Other	☐ Yes ☐ No										
CIP	Sodium hydroxide ☐ Yes ☐ No										
	Phosphoric acid ☐ Yes ☐ No										
	Sodium hypochlorite ☐ Yes ☐ No										
	Other ☐ Yes ☐ No										
SIP	☐ Yes ☐ No										
Autoclave	☐ Yes ☐ No										
Passivation	Check one:										
	Citric acid ☐ Yes ☐ No		Concentration, % _____								
	Nitric acid ☐ Yes ☐ No		Concentration, % _____								
	Phosphoric acid ☐ Yes ☐ No		Concentration, % _____								
	Other ☐ Yes ☐ No		Concentration, % _____								
Solids	☐ Yes ☐ No		Concentration, % _____								
Valve Operation											
Mode of Operation: Valves	Manual ☐ Yes ☐ No										
	Automated ☐ Yes ☐ No		Minimum air pressure ____ psi or bar Maximum air pressure ____ psi or bar								
Diaphragm material currently using? (please state in the space below)											

NONMANDATORY APPENDIX T
GUIDANCE ON POLYMER APPLICATIONS: CHROMATOGRAPHY COLUMNS AND FILTRATION

(19)

T-1 CHROMATOGRAPHY COLUMNS

T-1.1 General

Chromatography columns are used in processes to purify products or isolate substances of interest from other components contained within process solutions. Typical examples include large-scale purification of biopharmaceuticals and fine chemicals.

T-1.2 Column Construction

A typical chromatography column is comprised of a cylindrical shell (the "tube") closed at each end. The space between the ends of the column is filled with a medium (referred to as the "stationary phase" or "bed") in which the chromatography separation takes place. The liquid that travels axially through the column is referred to as the "mobile phase."

At each end of the column, a "bed support" retains the medium within the tube. This device is a porous disk typically constructed of a rigid, finely woven mesh or a semipermeable material. Behind each bed support is a "flow distributor." The flow distributor ensures uniform distribution of the mobile phase. The distributor is typically attached to a "rigid support plate," which is a pressure-retaining component that includes hygienic connections.

Usually, one end of the column is designed in such a way that it can be inserted into the column tube rather than fixed to the end (much like a piston in a syringe), thereby allowing adjustment of the bed height. This assembly is referred to as the "adapter." A column is typically installed with its axis oriented vertically and the adapter positioned at the top.

Chromatography columns may include additional mechanical features, such as the ability to add and remove the medium without opening the column and the ability to mechanically compress the bed.

T-1.3 Cleaning

The purpose of cleaning a chromatography column is to prevent environmental contamination, product-to-product carryover, or cross-contamination.

T-1.4 Sanitization

Chemical sanitization processes are used to reduce bioburden.

T-2 FILTRATION

T-2.1 General

Liquid or gas filtration is used
(a) to isolate processes
(b) to purify substances of interest
(c) to concentrate
(d) to exchange buffer/diafilter
(e) for viral removal
(f) for sterilization/bioburden reduction

T-2.2 Filtration Formats

Typical filtration formats include large-scale filtration and separation of biopharmaceuticals and fine chemicals using direct flow filtration (DFF) and tangential flow filtration (TFF). The filter elements used in both modes may be hydrophobic or hydrophilic. The principal difference between DFF and TFF is the direction of flow with respect to the filter surface. In TFF, the process fluid flow is tangential to the filter media with only a portion of the process fluid passing through it. This results in one inlet stream and two outlet streams. In DFF, the process fluid flow is perpendicular to the filter media with all of the process fluid passing through the filter media. This results in one inlet and one outlet.

T-2.2.1 Direct Flow Filtration. In DFF, the process fluid passes through the filtration media in a single pass. These filters are typically used for clarification, gas and vent filtration, virus reduction, and process fluid sterilization. This mode of filtration is also known as dead-end filtration, depth filtration, and normal flow filtration.

T-2.2.1.1 Filtration Elements. A typical DFF element may be one of the following acceptable designs:

(a) Pleated or Wrapped Design. This design is comprised of a cylindrical shell, which can have the media either pleated or wrapped. The flow path is typically from the outside of the cartridge through the wrap or pleat

pack to the core. One end of the cylindrical shell can be either sealed (capped) or left open (referred to as "double open end"). Housing connections are typically flat gasket or various O-ring configurations. For submicron filtration, single-open-end designs are preferred with a secured double O-ring connection.

(b) Lenticular Design. This design is comprised of two sheets of filtration media cut in a circular design sandwiched and supported by a flow channel. These formats are usually stacked on top of each other. One end is closed for flow while the other end is connected either by a flat gasket seal or by an O-ring connection. Sealing between the stacked formats is accomplished by either compression or welding.

(c) Hollow Fiber Design. This design is comprised of small-diameter permeable tubular membranes that are sealed on opposite ends by "potting." The flow path can be from either direction (from inside to outside or vice versa) of the tubes to either end of the potting. One end of the potting is capped or sealed and the other end has single-open-end designs with Code 7 connection being preferred for submicron filtration.

T-2.2.2 Tangential Flow Filtration. In TFF, also known as cross-flow filtration, the bulk of the process fluid flow is tangential to the filter media surface and is typically referred to as "feed" or "retentate." The portion of the process fluid that flows through the filter media is typically referred to as "permeate" or "filtrate" and is captured as a separate flow stream. TFF filter media are usually classified as microfiltration, ultrafiltration, or reverse osmosis based on the filter media pore size. TFF is typically used for clarification, diafiltration (buffer exchange), and/or concentration of a substance of interest.

T-2.2.2.1 Filtration Elements. A typical TFF element may be one of the following acceptable designs:

(a) Hollow Fiber. This design is comprised of one or more permeable membrane tubes, which are typically sealed into a housing on both ends by potting in a shell-and-tube design. The "shell" side of the membranes and the inside of the tubes are generally the two distinct different portions of the device as they can communicate only through the membrane of the tube. Hollow fiber elements are typically 0.079 in. (2 mm) or less in inner diameter. The two typical operation arrangements are inside-out and outside-in. In the inside-out arrangement, the process fluid is pumped into the inside of the tubes; in this case, the retentate will emerge out the other end of the tube, and permeate will be collected on the shell side of the device. In the outside-in arrangement, the process fluid is pumped into one end of the shell, and the retentate emerges out the other end; in this case, permeate is collected from the inside of the tubes.

(b) Tubular. A tubular membrane is characterized by its discrete tubes and large lumen sizes, which are typically 0.5 in. (12 mm) or greater inner diameter. These lumens are usually metallic or polymeric and provide strong structural support for the membranes. Tubular membranes flow from the inside out, with turbulent flow in the retentate stream keeping the membrane surface clean. Permeate flows through the walls of the tubes and is collected outside the modules. Modules are constructed of metallic or polymeric materials, to accommodate user needs. Solids levels of up to 5% by weight are commonly handled by tubular membranes.

(c) Plate and Frame. Plate-and-frame or flat-sheet filters are characterized by an envelope-type structure formed out of two pieces of membrane. In this type of device, process fluid is passed over the outside of the envelope and through the membrane into the inside of the envelope; the remaining material that did not pass through the membrane is the retentate. In a plate-and-frame arrangement, there is usually a rigid or semirigid frame that supports the membrane so that there is always a flow path on the inside and outside of the envelopes. This frame may be a rigid metallic or polymeric frame or something as simple as a screen or straps.

(d) Spiral Wound. Spiral-wound membrane devices are constructed from an envelope of membrane that is wound around a hollow core. The inside of the envelope is connected to the hollow core in such a way as to allow fluid from inside the envelope to pass through the center of the hollow core. The feed material is passed into one end of the device and over the outside of the envelope. The permeate passes through the membrane into the inside of the envelope and eventually outside the hollow core. The retentate passes out the opposite end of the spiral device from the feed.

(e) Monolith. A monolith membrane is characterized by a porous rigid body with long flow paths (lumens) that pass through the middle. These lumens are coated on the inside diameter with a highly controlled porous surface or membrane. Typically, these devices are made of ceramic material, which withstands high temperatures or aggressive chemicals. The process fluid is fed into one end of the device and the retentate passes out the other end; the permeate passes through the membrane surface and porous body and is collected on the outside of the device.

T-2.3 Housings and Encapsulation

Housings are designed with different options, such as the capacity for handling multiple elements and various inlet and outlet options. Encapsulated filtration elements are designed for handling purposes or in place of metallic housings.

T-2.3.1 Holders. Holders are used to stack or hold in place the filter elements and to manage or direct the process fluid. The holders are designed to ensure proper sealing between the inlet and outlet connections, as well as between the encapsulated elements.

T-2.4 Design for Cleaning

T-2.4.1 Cleaning. The purpose of cleaning a DFF system is to prevent environmental contamination, product-to-product carryover, and cross-contamination. For TFF, flow rates are also an important design parameter for effective cleaning.

T-2.5 Normalized Water Permeability (NWP) Testing

T-2.5.1 To conduct an NWP test, the TFF system is controlled at one or more operating points with respect to feed flow and transmembrane pressure (TMP) using pyrogen-free purified water and/or a buffer or cleaning agent of suitable quality. The permeate pressure should be as close as possible to 0 psig (atm) to allow the permeate flow rate to reach its maximum. During the test, the permeate flow rate (flux) is measured, along with the temperature of the solution.

The water permeability is calculated using the following equation:

water permeability = permeate flux (l/m^2-h)/TMP (psi or bar)

The resulting flow rates are normalized to a standard temperature by applying a temperature correction factor that accounts for the change in density. A baseline measurement of the membrane's permeability shall be obtained when the membrane is new. For all subsequent measurements, the NWP test is performed following the cleaning of the membrane at operating conditions as close as possible to the original test.

NONMANDATORY APPENDIX U
(19) GUIDANCE FOR THE USE OF U.S. CUSTOMARY AND SI UNITS

U-2 GUIDELINES USED TO DEVELOP SI EQUIVALENTS

The following guidelines were used to develop SI equivalents:

(a) SI units are placed in parentheses after the U.S. Customary units in the text.

(b) The table designation (e.g., table number) is the same for both the U.S. Customary and SI tables, with the addition of suffix "M" to the designator for the SI table, if a separate table is provided. In the text, references to a table use only the primary table number (i.e., without the "M"). For most tables, where interpolation is not required, SI units are placed in parentheses after the U.S. Customary unit.

(c) Separate SI versions of graphical information (charts) may be provided as necessary, except that if both axes are dimensionless, a single figure (chart) is used.

(d) In most cases, conversions of units in text were done using hard SI (approximate) conversion practices, with some soft conversions (exact) on a case-by-case basis, as appropriate. This was implemented by rounding the SI values to the number of significant figures of implied precision in the existing U.S. Customary units. For example, 8 in. has an implied precision of one significant figure. Therefore, the conversion to SI units would typically be 200 mm. This is a difference of about 1.6% from the "exact" or soft conversion of 203.2 mm. However, the precision of the conversion was determined by the Committee on a case-by-case basis. More significant digits were included in the SI equivalent if there was any question.

(e) Minimum thickness and radius values that are expressed in fractions of an inch were generally converted according to the following table:

	Proposed SI	
Fraction, in.	Conversion, mm	Difference, %
1/32	0.8	−0.8
3/64	1.2	−0.8
1/16	1.5	5.5
3/32	2.5	−5.0
1/8	3	5.5
5/32	4	−0.8
3/16	5	−5.0
7/32	5.5	1.0
1/4	6	5.5
5/16	8	−0.8
3/8	10	−5.0
7/16	11	1.0
1/2	13	−2.4
9/16	14	2.0
5/8	16	−0.8
11/16	17	2.6
3/4	19	0.3
7/8	22	1.0
1	25	1.6

(f) For nominal sizes that are in even increments of inches, even multiples of 25 mm were generally used. Intermediate values were interpolated rather than converted and rounded to the nearest millimeter. See examples in the following table:

Size, in.	Size, mm
1/4	6
3/8	10
1/2	13
3/4	19
1	25
1 1/2	38
2	50
2 1/2	64
3	75
4	100
6	150

(g) For all pressures, the SI units are in kPa (even when it is 1 000 kPa). Rounding was to one significant figure (two at the most) in most cases. See examples in the following table. (Note that 14.7 psi converts to 101 kPa, while 15 psi converts to 100 kPa. While this may seem at first glance to be an anomaly, it is consistent with the rounding philosophy.)

Pressure, psi	Pressure, kPa
0.5	3
2	15
3	20
10	70

Pressure, psi	Pressure, kPa
14.7	101
15	100
30	200
50	350
100	700
150	1 000
200	1 500
250	1 700

(h) In most cases, temperatures (e.g., for PWHT) were rounded to the nearest 5°C. Depending on the implied precision of the temperature, some were rounded to the nearest 0.1°C or 10°C or even 25°C. Temperatures colder than 0°F (negative values) were generally rounded to the nearest 0.1°C. The examples in the following table were created by rounding to the nearest 5°C, with one exception.

Temperature, °F	Temperature, °C
70	20
100	38
120	50
150	65
200	95
250	120
300	150
350	175
400	205
450	230
500	260
550	290
600	315
650	345
700	370
750	400
800	425
850	455
900	480
925	495
950	510
1,000	540
1,050	565
1,100	595
1,150	620
1,200	650
1,250	675
1,800	980
1,900	1 040
2,000	1 095
2,050	1 120

U-3 CHECKING EQUATIONS

When a single equation is provided, it has been checked using dimensional analysis to verify that the results obtained by using either the U.S. Customary or SI units provided are equivalent. When constants used in these equations are not dimensionless, different constants are provided for each system of units. Otherwise, a U.S. Customary and an SI version of the equation are provided. However, in all cases, the Standard user should check the equation for dimensional consistency.

U-4 SOFT CONVERSION FACTORS

The following table of "soft" conversion factors is provided for convenience. Multiply the U.S. Customary value by the factor given to obtain the SI value; similarly, divide the SI value by the factor given to obtain the U.S. Customary value. In most cases, it is appropriate to round the answer to three significant figures.

U.S. Customary	SI	Factor	Notes
in.	mm	25.4	...
ft	m	0.3048	...
$in.^2$	mm^2	645.16	...
ft^2	m^2	0.09290304	...
$in.^3$	mm^3	16,387.064	...
ft^3	m^3	0.02831685	...
U.S. gal	m^3	0.003785412	...
U.S. gal	L	3.785412	...
psi	kPa	6.89475729	...
°F	°C	5/9 × (°F–32)	Not for temperature difference
°F	°C	5/9 × °F	For temperature differences only
lbm	kg	0.4535924	...
lbf	N	4.448222	...
psi	P	6 894.75729	...
µin.	m	0.0254	...
gal/min	L/min	3.785	...
ft/sec	m/s	0.3048	...
in./in./°F	mm/mm/°C	1.8	...

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NONMANDATORY APPENDIX W
POSITIVE MATERIAL IDENTIFICATION

W-1 GENERAL

Positive material identification (PMI) is a form of product (check) analysis in which a partial chemical composition is determined and used to verify material identification.

As discussed in this Standard, the scope of PMI is limited to alloy verification using portable, handheld X-ray fluorescence (XRF) equipment. Typically, this equipment cannot detect or measure elements having an atomic number less than 22 (titanium). For this reason, elements such as carbon, sulfur, and phosphorous cannot be detected. This makes it impossible to use this equipment in lieu of a complete chemical compositional analysis or to prove that a specific alloy conforms to a material specification. This equipment, therefore, is not suitable for material acceptance as a stand-alone analysis. Without the support of a referee analysis or a Material Test Report (MTR), PMI should not be used to accept materials, except for forensic situations in which unknown materials are being analyzed.

PMI results should not be the sole reason for material rejection either. When PMI results indicate that a material may be other than that specified, a referee analysis, acceptable to the owner/user, purchaser (as appropriate), and organization performing the PMI should be performed, unless the owner/user permits rejection of material based solely on PMI results. Rather, PMI should be used in conjunction with MTRs or Certificates of Compliance from the material manufacturer.

W-2 GENERAL REQUIREMENTS

PMI shall be performed in accordance with a written procedure as described in W-4. The alloying elements to be tested shall be either those necessary to identify the alloy in question or those having a significant effect on the performance characteristics of the material. Unless otherwise specified in the contract, the elements to be analyzed shall be at the discretion of the organization performing the PMI. The instrument employed shall be capable of detecting the alloying elements of interest. Records of PMI tests shall be documented and provided to the owner/user on request.

The analytical technique to be used shall be mutually agreed on by the purchaser (as appropriate) and owner/user and shall be in accordance with ASTM E1476, Standard Guide for Metals Identification, Grade Verification and Sorting, or another recognized industry standard method. The technique should also comply with this Appendix.

XRF analysis may be performed using either the analysis mode or the alloy identification (alloy matching) mode.

In the analysis mode, the PMI instrument will produce chemical composition data, including an accuracy tolerance, for each alloying element. This will result in a range of values for each alloying element. For a reading to be considered acceptable, one of the following two conditions shall be met:

(a) The nominal values displayed on the PMI instrument, plus the accuracy tolerances displayed on the instrument, shall be within the corresponding chemical composition ranges in the applicable material specification.

(b) The nominal values displayed on the PMI instrument shall fall within the chemical composition ranges in the applicable material specification as modified by the product (check) analysis tolerances in the material specification incorporated by reference.

When used in the alloy identification mode, the instrument will display a specific alloy type, based on a comparison between the chemical composition data obtained during testing and the data ranges in the instrument's internal library. When using this mode, the user is cautioned that any alloy identified by the PMI instrument is defined by a single material specification in the unit's internal library whose chemical composition ranges may not coincide with those in the applicable material specification or contract requirement. As a result, the user of the PMI instrument shall take into consideration potential differences between the material specification ranges loaded into the PMI unit's internal library and those specified for the material, component, or surface being tested.

Since the material specifications loaded into the PMI unit's internal library may not coincide with those specified for the part being analyzed, the recommended mode of operation is the analysis mode.

W-3 CALIBRATION

Periodic calibrations shall be performed at frequencies recommended by the equipment manufacturer. These calibration tests shall be documented, although the specific data does not need to be recorded.

In addition, at the beginning of each shift, or prior to use on each shift, the instrument calibration shall be verified using three consecutive tests against a known standard for each alloy category to be inspected during the shift, using the technique to be used during the shift. An acceptable calibration verification check shall be valid to the end of the shift.

In the event of a nonconforming result, the PMI instrument should be again verified against the known standard and the suspect material should be checked again with three consecutive checks. If any of the three consecutive rechecks are found to be nonconforming, refer to W-6 and W-7.

All these instrument verification checks shall be performed under environmental conditions similar to those of the test location. All such standards used in the instrument calibration verification shall be clearly identified as to nominal alloy composition. A certified reference material (CRM) that is an Analytical Reference Materials International (ARMI) or National Institute of Standards and Technology (NIST) traceable standard is recommended. As an alternative, the known sample could be provided with an MTR and a chemical composition analysis performed by an independent test laboratory.

W-4 PROCEDURE

A written procedure shall be used, and it shall include the following elements:

(a) the instrument to be used

(b) the analytical technique to be used

(c) sampling plans for each material, component, or surface

(d) material, component, or surface to be inspected

(e) documentation requirements

(f) material identification requirements

(g) requirements for instrument calibration and instrument calibration checks

(h) personnel qualification requirements

(i) control of rejected materials

The procedure may also include segregation procedures for the tested material, marking, and small-percentage random sampling.

W-5 PERSONNEL TRAINING AND QUALIFICATION

Individuals performing PMI shall be trained and qualified. The training shall address the following as a minimum:

(a) requirements for surface preparation prior to testing

(b) minimum exposure times for adequate data collection

(c) proper activation of the analyzer window shutter to ensure that it opens completely during exposure and data collection

(d) assurance that the surface to be analyzed completely covers the analyzer window

(e) precautions to take when using the alloy analyzer on curved or contoured surfaces

(f) special instructions or ancillary equipment needed for analyzing welds

(g) demonstration of capabilities, if required by the owner/user

Records of training shall be maintained by the employer.

W-6 ACCEPTANCE/REJECTION

When an MTR is available, material may be accepted based on PMI results, if the PMI results for the required elements are acceptable and the data for all other required elements on the MTR are acceptable as well.

Gross deviations that are clearly outside the material specification limits, as modified by the product analysis tolerance (e.g., UNS S31603 for which PMI testing shows 1.50% molybdenum), shall be cause for immediate quarantine of the material. Referee analysis, agreeable to all parties, should be conducted to verify this unless the owner/user elects to permit rejection based solely on PMI results. When the referee analysis shows similar gross deviations, the material shall be rejected.

When no MTR is available, acceptance may be based solely on the results of alloy identification analysis when permitted by the owner/user.

W-7 PMI REFEREE METHODS

PMI is not meant to replace an MTR. If PMI testing indicates a nonconforming result, the suspect material shall be quarantined, but it should not be immediately rejected. A quantitative referee method, acceptable to all parties, should be used, unless the owner/user decides otherwise. Examples of such methods may be found in ASTM E1476. Any remaining disputes shall be resolved by a third party that is accredited by the American Association for Laboratory Accreditation (A2LA) or an equivalent, if available.

W-8 PMI SPECIAL CONDITIONS

Generally, PMI of bare welding filler wire or inserts can be achieved with proper procedures.

The users of PMI are cautioned that most equipment is not typically suitable for analysis of small areas or small volumes of material, such as welds, and is not capable of

accounting for dilution factors in welds. Special equipment adaptations, such as reduced-size detection windows, or special methods may be required to perform analysis on small areas. The equipment manufacturer should be consulted for such applications.

Analysis of welds, with or without filler metal, for weld acceptance is not recommended.

Analysis of coatings may require special analytical methods and is outside the scope of this Appendix.

NONMANDATORY APPENDIX Y
PROCUREMENT SOURCES

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To procure ASTM specifications, contact ASTM International at www.astm.org.
To procure ASME specifications, contact ASME at www.asme.org.
To procure DIN specifications, contact DIN at www.din.de.
EN specifications may be obtained from any of the following member organizations:

Country	Country Standards Development Organization	Website
Austria	Austrian Standards Institute (ASI)	www.austrian-standards.at
Belgium	Bureau de Normalisation/Bureau voor Normalisatie (NBN)	www.nbn.be
Bulgaria	Bulgarian Institute for Standardization (BDS)	www.bds-bg.org
Croatia	Croatian Standards Institute (HZN)	www.hzn.hr
Cyprus	Cyprus Organization for Standardisation (CYS)	www.cys.org.cy
Czech Republic	Czech Office for Standards, Metrology and Testing (UNMZ)	www.unmz.cz
Denmark	Dansk Standard (DS)	www.ds.dk
Estonia	Estonian Centre for Standardisation (EVS)	www.evs.ee
Finland	Suomen Standardisoimisliitto r.y. (SFS)	www.sfs.fi
Former Yugoslav Republic of Macedonia	Standardization Institute of the Republic of Macedonia (ISRM)	www.isrm.gov.mk
France	Association Française de Normalisation (AFNOR)	www.afnor.org
Germany	Deutsches Institut für Normung (DIN)	www.din.de
Greece	National Quality Infrastructure System (NQIS/ELOT)	www.elot.gr
Hungary	Hungarian Standards Institution (MSZT)	www.mszt.hu
Iceland	Icelandic Standards (IST)	www.stadlar.is
Ireland	National Standards Authority of Ireland (NSAI)	www.nsai.ie
Italy	Ente Nazionale Italiano di Unificazione (UNI)	www.uni.com
Latvia	Latvian Standard Ltd. (LVS)	www.lvs.lv
Lithuania	Lithuanian Standards Board (LSD)	www.lsd.lt
Luxembourg	Organisme Luxembourgeois de Normalisation (ILNAS)	www.portail-qualite.lu
Malta	The Malta Competition and Consumer Affairs Authority (MCCAA)	www.mccaa.org.mt
Netherlands	Nederlands Normalisatie-instituut (NEN)	www.nen.nl
Norway	Standards Norway (SN)	www.standard.no/
Poland	Polish Committee for Standardization (PKN)	www.pkn.pl
Portugal	Instituto Português da Qualidade (IPQ)	www.ipq.pt
Romania	Romanian Standards Association (ASRO)	www.asro.ro
Slovakia	Slovak Office of Standards Metrology and Testing (UNMS)	www.unms.sk
Slovenia	Slovenian Institute for Standardization (SIST)	www.sist.si
Spain	Asociación Española de Normalización y Certificación (AENOR)	www.aenor.es
Sweden	Swedish Standards Institute (SIS)	www.sis.se
Switzerland	Schweizerische Normen-Vereinigung (SNV)	www.snv.ch
Turkey	Turkish Standards Institution (TSE)	www.tse.org.tr
United Kingdom	British Standards Institution (BSI)	www.bsigroup.com

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NONMANDATORY APPENDIX Z
QUALITY MANAGEMENT SYSTEM

Z-1 SCOPE AND PURPOSE

This Appendix identifies the elements that shall be addressed in a Quality Management System (QMS) for ASME BPE component manufacturers. These elements identify the procedures, describe the processes, and list the resources necessary to implement the QMS. The QMS shall identify and describe the authority of the individuals responsible for ensuring that the quality activities necessary for the manufacture of ASME BPE-compliant components is done in a consistent and controlled manner. These activities shall ensure that the requirements of the ASME BPE Standard are met through quality planning, quality control, quality assurance, and quality improvement.

A QMS established by a manufacturer that intends to meet the requirements of the ASME BPE Standard shall be suitable for the types of components being manufactured and the types of activities performed in the manufacture of those components.

Z-2 GENERAL

The manufacturer shall establish and maintain a QMS that ensures compliance with all applicable requirements of the current ASME BPE Standard. Upon the issuance of a revised ASME BPE Standard, the certificate holder shall review his QMS and, where required, revise it to be in conformance with the revised Standard. Implementation of the revised QMS shall be within 6 months of the date of issue of the revised Standard.

Z-3 QUALITY MANAGEMENT SYSTEM MANUAL

The QMS shall be documented in a written QMS manual. There is no specific format or particular arrangement required for the manual, as long as all applicable elements have been addressed and the various topics are arranged in a logical and easy-to-interpret manner. The elements identified in Z-3.1 through Z-3.15, as applicable to the manufacturer's scope of work, shall be addressed in the manual. The manual shall be ASME's guide for surveying and auditing the manufacturer's activities and documentation for conformance to its QMS. The manual shall be an auditable document that identifies the controls and processes used to ensure that the activities performed in the manufacture of ASME BPE components will be in conformance with the ASME BPE Standard. The manual shall not be a reiteration of the ASME BPE Standard, but shall, instead, describe or identify what, when, where, and how processes are conducted.

Z-3.1 Quality Management System

(a) Management personnel shall establish objectives for measuring effective implementation of the QMS and are responsible for obtaining the desired results. A policy statement indicating management authority and responsibility shall be included.

(b) A description of the components being manufactured in accordance with the ASME BPE Standard shall be provided.

(c) The authority and duties of those responsible for implementing the QMS shall be clearly established. Individuals performing quality assurance and quality control functions shall not be under the direct supervision of those in charge of areas being evaluated. They shall also have sufficient and well-defined responsibility, authority, and organizational freedom to

(1) identify quality-related problems

(2) initiate, recommend, and provide solutions to quality-related problems

(3) verify implementation of those solutions

(4) assure that further processing, delivery, or use of a suspect material or product is halted until proper disposition of any potentially nonconforming, deficient, or unsatisfactory condition has occurred

(d) Activities required to be performed by qualified personnel shall be identified. Minimum qualifications for such personnel shall be established. Controls shall be established to ensure that only those personnel who have the specified qualifications are permitted to perform those activities.

(e) Auditing activities shall be performed by trained and qualified auditors. The qualification, experience, and training requirements for auditors shall be specified in the QMS manual. Auditing competence includes, at a minimum, demonstrated knowledge and understanding of

(1) the ASME BPE Standard

(2) applicable regulations

(3) the QMS program

(4) auditing techniques for examining, questioning, evaluating, and reporting, including identifying audit findings, following up on corrective actions, and the closing out of audit discrepancy findings

(f) Services may be outsourced. Outsourced services shall be performed by an approved and qualified supplier in accordance with Z-3.8. Services that are required to be performed by qualified personnel may also be outsourced to a third party provided the QMS manual describes the manner in which it verifies the individuals' qualifications.

Z-3.2 Organization

(a) The QMS manual shall include an organization chart that shows the structure of functional groups, their responsibilities, levels of authority, lines of communications and identify within each group by job title the individuals involved with activities affecting quality. The purpose of this chart is to identify and associate the various organizational groups with the function for which they are responsible. The organization may modify this chart as necessary to suit changes in its scope of activities as long as those changes are reflected in the QMS manual.

(b) Quality assurance and quality control activities shall be performed by personnel whose functions are sufficiently independent of cost and schedule and independent of departments responsible for production or service processes.

(c) Specific personnel shall be designated for each of the following functions as appropriate to the scope of work performed by the organization:

(1) design

(2) purchasing/procurement

(3) contract review

(4) document control

(5) material control

(6) manufacturing/production

(7) quality control/assurance

(8) examination/inspection. A manufacturer may perform either examination functions or both examination and inspection functions. However, the individuals performing the final examination to assess the component's conformance to the ASME BPE Standard shall be independent of all departments responsible for production or service activities. Examination personnel may be responsible to production management only if a third-party inspection agency is performing the final evaluation for conformance to the ASME BPE Standard.

(9) maintenance of equipment

Z-3.3 Drawings, Design Calculations, and Specification Control

Controls shall be established to ensure that only the latest applicable drawings, design calculations, specifications, instructions, and authorized changes thereto are used for manufacturing, examination, inspection, and testing.

Z-3.4 Material Control

(a) Controls shall be established for material receipt to ensure that the material received is the material ordered, that it is properly identified, and that it has sufficient documentation. This documentation shall include, as a minimum, Material Test Reports (MTRs) for metallic materials and Certificates of Conformance (C of Cs) for polymeric and other nonmetallic materials that show that the material received satisfies the requirements of the ASME BPE Standard.

(b) The material control system shall ensure that only the intended material is used in the manufacture of ASME BPE components. Effective material control includes

(1) identification during receipt inspection [e.g., positive material identification (PMI) for metallic materials, laboratory testing, or verification of MTRs or C of Cs]

(2) traceability

(3) segregation of nonconforming materials as necessary

(4) proper marking

Z-3.5 Examination, Inspection, and Testing Program

Provisions shall be in place to establish acceptance criteria for the examinations, inspections, and testing necessary to prove conformance with the requirements of the ASME BPE Standard. In addition, the type and extent of examinations, inspections, and testing shall be specified as well as the step(s) during the manufacturing sequence at which these activities are to be performed.

Unless otherwise specified in the ASME BPE Standard, examination, inspection, and testing shall be conducted at frequencies (extent) specified in the applicable referenced product specification(s), but not less than 10%.

Z-3.6 Control of Special Processes

Special processes shall be identified and performed as a controlled activity. Special processes include, but are not limited to

(a) Material Joining. Measures shall be established to ensure that all joining processes are performed by qualified personnel using qualified procedure specifications. The qualification of personnel and procedure specifications shall be in accordance with the ASME BPE Standard.

(b) Heat Treatment. Measures shall be established to ensure that heat treatment of components conforms to the requirements of the ASME BPE Standard.

(c) Nondestructive Examination (NDE). Measures shall be established to ensure that NDE is performed by personnel whose qualifications meet the requirements of the ASME BPE Standard. These measures shall also ensure

that all NDE activities are conducted in accordance with procedures as specified in the ASME BPE Standard.

(d) Surface Finish/Treatment. Measures shall be established to ensure that surface finish treatments and the procedures used to apply them conform to the requirements of the ASME BPE Standard.

(e) Special Controlled Environments. Provisions shall be enacted to identify activities that require special controlled environments, and appropriate measures are established to achieve and maintain the desired conditions.

Z-3.7 Control of Equipment and Tooling

(a) Provisions shall be established to create a maintenance schedule to ensure the use of equipment, fixtures, machinery, and tooling that are properly maintained.

(b) Provisions shall be established for the safe storage of equipment and tooling that are not in service.

Z-3.8 Control of Outsourced Items and Services

(a) Controls shall be established to ensure that outsourced items and services meet the specified requirements of the ASME BPE Standard. Provisions shall be established for the qualification and approval of suppliers and for the correction or elimination of nonconformances in outsourced items and services.

(b) The method used to qualify, approve, and monitor the performance of suppliers shall be described.

(1) For outsourced items supplied by an ASME BPE Certificate Holder, the method employed shall include the following:

(-a) a review of the certificate's scope and/or the QMS manual to determine the Certificate Holder's capability of supplying the item.

(-b) receipt of the supplied item with documentation certifying that the work was performed in accordance with the ASME Certificate of Authorization with the certificate number identified.

(2) For items and services supplied by an organization that does not hold an ASME BPE Certificate of Authorization, the method employed shall be based upon one or more of the following:

(-a) The supplier's history of providing items or services that conform to specified requirements or that perform satisfactorily in service. This can be based on past performance or a current third-party certification from a recognized accreditation body. Additionally, when qualification and approval are based upon third-party certification, the scope of that certification must be verified to be appropriate for the item or service being supplied.

(-b) Verification upon receipt inspection that the item or service conforms to specified requirements. Inspections shall be performed by qualified personnel using an inspection plan that details the characteristics to be inspected, the method of inspection, and the acceptance criteria. The results of the inspection shall be documented.

(-c) Verification during fabrication that the item or service performs satisfactorily in service. When adequate service performance cannot be verified through inspection activities on the completed component, hold points shall be established to evaluate the conformity of the supplied item or service. Work shall not proceed until the inspector has verified that the work performed is in conformance with the specified requirements.

(-d) On-site surveillance of the supplier by a qualified technical expert or an auditor to verify the conformance of the supplied item or service.

(c) Controls shall be established for the segregation of outsourced items that are determined to be nonconforming.

Z-3.9 Control of Measurement and Test Equipment

Controls shall be established for the calibration of examination, measuring, and test equipment used in quality control activities.

(a) Procedures. Procedures shall be established to ensure that tools, gauges, instruments, and other measuring and testing devices used to verify compliance with the specified requirements are calibrated at regular intervals. Periodic checks on these devices shall be performed to determine that calibration is current. These periodic checks shall be documented.

(b) Calibration. Calibration shall be conducted using certified samples having documented traceability to primary standards, where such standards exist. If no primary standard exists, the standard or basis for calibration shall be documented.

(c) Control Measures. Control measures shall include provisions for identification of these devices and for determining calibration status by either equipment marking or records traceable to the equipment.

(d) Out-of-Tolerance Devices

(1) When a device fails to calibrate during a planned periodic calibration, provisions shall be established to ensure that appropriate corrective action shall be taken. These provisions shall include a method for reviewing all measurements or tests performed with that device since the last successful periodic calibration to determine if applicable requirements have been met.

(2) When a device fails to calibrate during a periodic check, provisions need only address measurements or tests performed since the last successful periodic check provided the method and frequency used for the periodic check are described in calibration procedures.

Z-3.10 Nonconformances and Corrective Actions

(a) Items and services that do not conform to specified requirements shall be controlled to prevent inadvertent installation or use. Controls shall address identification, documentation, evaluation, segregation when practical, and disposition of nonconforming items, as well as the notification of affected organizations.

(b) Measures shall be established to ensure that conditions that do not conform to specified requirements, such as failures, malfunctions, deviations, defective material and equipment, nonconformances, and other quality system deficiencies, are identified, reported to appropriate levels of management, and promptly corrected. These measures shall also ensure that the root cause (s) responsible for these nonconformances be determined and corrected.

(c) The identification of conditions causing recurring nonconformances, the cause of these conditions, and the corrective action taken shall be documented and reported to appropriate levels of management.

Z-3.11 Storage, Shipping, Handling, and Packaging

Controls shall be established for the cleaning, preservation, packaging, storage, and shipping of finished components.

Z-3.12 Control of Documents and Record Retention

(a) Provisions shall be established to ensure that documents that are to be maintained throughout the design and manufacture of the component are identified. A minimum retention period of 3 yr from the date the component is shipped shall be established.

(b) These provisions shall also require that manufacturer's Data Reports, MTRs, and C of Cs shall be retained for a minimum period of 5 yr from the date the component is shipped.

(c) Document retention requirements shall extend to records of personnel training, qualification, and certification, for which the minimum retention time shall be 5 yr after their employment ceases. For personnel providing outsourced services in accordance with Z-3.1(f), provisions shall be established for the certificate holder to have access to their personnel records, as needed.

Z-3.13 Sample Forms

The use of forms shall be described in the QMS manual. Typical examples, referred to as "exhibits" shall be included and marked "Sample." These samples should be completed in a manner typical of that expected for actual production documents.

Z-3.14 Internal Audits

Requirements shall be established for internal audits. Those requirements should address the following, as a minimum:

(a) The frequency of internal audits shall be specified. These audits shall be conducted in accordance with a written procedure by qualified auditors not having direct responsibility for the areas being audited. All elements in the QMS shall be internally audited at least once during each certification period.

(b) Audit results shall be documented by auditing personnel and reviewed by management having responsibility over the areas being audited. This documentation shall bear the signatures of the responsible management personnel.

(c) Corrective actions taken in response to deficiencies or nonconformances shall be documented. Follow-up actions shall be required after corrective actions have been taken to ensure the problem has been corrected. These follow-up actions may include a re-audit of deficient areas.

Z-3.15 Management Performance Assessments

Management personnel with assigned responsibility shall review the organization's QMS at least annually to ensure its continued suitability, adequacy, and effectiveness. The input from management review shall include information on the results of audits, process performance and product conformity, the status of preventive and corrective actions, and customer feedback, as appropriate.

NONMANDATORY APPENDIX AA
STATIC SEALS APPLICATION GUIDE FOR COMPENDIAL WATER SYSTEMS

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AA-1 GENERAL

Table AA-1-1 provides a guide to static seal selection, which is intended to give readers general information about chemical, physical/mechanical, and maintenance considerations related to selecting materials suitable for static seals used in compendial water systems. Evaluations presented in the Table generally describe compounds in the specified material class. Different compounds within a material class, different compounds from particular suppliers, and articles manufactured from the same compounds in different ways may vary significantly. Seal location within the system may also affect seal performance. Some performance data for this application can be developed using test methods listed in Nonmandatory Appendix L. Chemical compatibility/suitability of a particular compound may be verified through the material supplier. Maintenance characteristic evaluations are expert opinions based on testing (see Nonmandatory Appendix K) and experience using these compounds. The scope of this Appendix cannot address all possible materials. Materials other than those listed may be suitable for compendial water applications provided they meet the requirements of Part PM. The intent of this Appendix is to guide communication between the owner/user and the supplier to find a balance of properties needed for the application.

This information may give initial guidance for material selection. However, due to the inherent variability of materials, compound processing, and use, the owner/user should qualify individual materials and suppliers based on performance data.

AA-2 MATERIAL CLASS DESCRIPTIONS[1]

AA-2.1 Synthetic Rubbers (Elastomers)

EPDM (Ethylene Propylene Diene Rubber) – peroxide-cured systems: EPDM is one of the more widely used and varying materials in bioprocessing equipment, including compendial water systems.

VMQ (Silicone) – peroxide-cured and platinum-cured systems: Silicone is often preferred for single-use applications due to its relative simplicity of formulation, low toxicity, and optical clarity. Peroxide-cured silicone systems generally have more reaction by-products, which may be extractable, than platinum-cured systems.

FKM (Fluoroelastomers): FKM materials can be separated into types, based on the monomers used and their ratio(s). In general, the higher the fluorine level, the better the chemical resistance. Different curing systems within the same type can affect performance significantly.

AA-2.2 Plastics and Composites

PTFE (Polytetrafluoroethylene) and Composites: PTFE has long been used in bioprocessing equipment due to its relative high purity, chemical inertness, nonstick properties, and long seal life. However, as a plastic, PTFE seals are susceptible to irreversible deformation under load/compression (cold-flow/creep). PTFE seals can vary in geometry due to warping and/or process variation, and fit may be less predictable than elastomeric seals. PTFE seals and their variants generally require higher compressive forces (fastener torque) to form a seal than their elastomeric counterparts. They are more susceptible to degradation due to gamma-irradiation (sanitization) than other commonly used materials. PTFE seal properties can also vary with PTFE grades (molecular weight and particle size distribution) and manufacturing variables.

PTFE and its composites are used widely for compendial water applications.

AA-2.3 Modified PTFE

Modified PTFE has lower vapor phase transmission, without sacrificing chemical resistance, than standard PTFE. Modified PTFE is often substituted for PTFE in static seals (see AA-2.2).

[1] Reference ASTM D1418, Standard Practice for Rubber and Rubber Latices — Nomenclature.

Table AA-1-1 Static Seals for Use in Compendial Water Systems (SG-2.2.1)
Recommendations are for Static Seals Located Within the Compendial Water Envelope (SG-5.3)

	Characteristics													
	Chemical [Note (2)]					Maintenance			Physical/Mechanical Properties					
	C	S	S	S	S	I	C	I	S	C	I	C	S	S
Material [Note (1)]	Hot DI Water	Acidic	Alkaline	Oxidizing/ Ozonation	Steam	Ease of Installing/ Removal/ Cleaning	Length of Service	Need for Retightening	Resistance to Cold Flow (Creep) During Thermal Cycling and/or Compression	Resistance to Erosion	Tear Resistance	Ease of Forming Seal (Sealability/ modulus)	Permeability	Compression Set
EPDM (Ethylene Propylene Diene Rubber)	●	●	●	●	●	◒	◒	◒	++	++	+	++	++	+
VQM (Silicone)														
Peroxide-Cured	◒	○	◒	◒	◒	●	◒	●	++	+	-	++	-	++
Platinum-Cured	◒	◒	◒	◒	●	●	◒	●	++	-	-	++	-	++
PTFE (Polytetrafluroethylene)	●	●	●	◒	●	●	●	◒	-	-	++	-	++	-
Modified PTFE	●	●	●	◒	●	●	●	◒	+	+	++	-	++	-
PTFE composites														
Elastomer/PTFE envelope	●	●	●	◒	●	●	●	◒	+	+	++	+	++	-
Elastomer/PTFE bonded	●	●	●	◒	●	●	●	◒	+	+	++	+	++	-
PTFE/inorganic fillers	●	●	●	●	●	●	●	◒	+	++	++	-	++	-
FKM (Fluoroelastomers)														
Type 1: low fluorination ~66%	●	●	○	●	●	●	◒	●	++	+	+	++	++	++
Type 2: medium fluorination ~68%	●	●	○	●	●	●	◒	●	++	+	+	++	++	++
Type 2: high fluorination ~70%	●	●	◒	●	●	●	◒	●	++	+	+	++	++	++
	● Preferred ◒ Acceptable ○ Unacceptable								++ Best + Good - Caution					

GENERAL NOTE: Relative importance to this application (see SG-5.3) is defined as follows:
C = critical
I = important
S = secondary (importance depends on owner/user specifics)

Table AA-1-1 Static Seals for Use in Compendial Water Systems (SG-2.2.1)
Recommendations are for Static Seals Located Within the Compendial Water Envelope (SG-5.3) (Cont'd)

NOTES:
(1) Comments made here are generally true with compounds in this class currently available. Different compounds within a class vary between seal suppliers. Different compound numbers from a particular supplier may also vary. Communication between user and supplier is recommended to find the balance of properties needed for the owner/user.
(2) Passivation chemicals are occasionally used in compendial water systems. Consult with supplier.

NONMANDATORY APPENDIX BB
MECHANICAL SEAL FACE MATERIAL SELECTION FOR COMPENDIAL WATER PUMPS

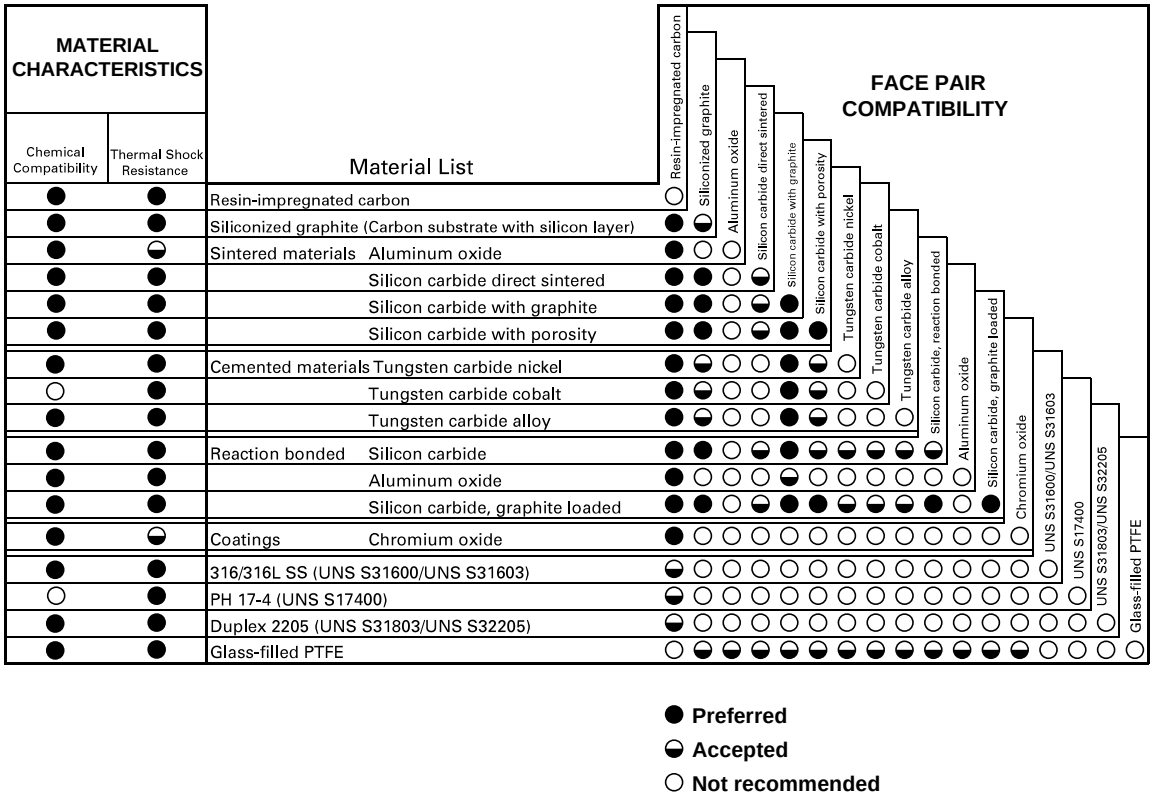
(19)

BB-1 GENERAL

Figure BB-1-1 will help the reader select a face pair (consisting of a rotating face and a stationary face) for a compendial water pump mechanical seal.

The most common mechanical seal face materials are listed under the "Material List" column in Figure BB-1-1. The materials listed are generic and do not specify the many grades available. Other seal face materials and combinations not listed in the Figure may be suitable. For information about material availability, face pair

Figure BB-1-1 Mechanical Seal Face Material Selection for Compendial Water Pumps



GENERAL NOTES:

(a) Solid circles indicate the material is preferred for the column characteristic. Half-filled circles indicate accepted material for operating in compendial water. Performance issues may exist specific to the characteristic listed in the column heading. Open circles indicate materials should not be used.

(b) Solid circles indicate the preferred face pairs for operating in compendial water. Half-filled circles indicate accepted face pairs for operating in compendial water. This face pair can operate in a satisfactory manner for a period of time but will likely have more wear, friction, heat, and debris over a shorter period of time than the preferred face pairs. Open circles indicate materials and face pairs that are not recommended for operating in compendial water. It is expected that this face pair will cause damage to one or both seal faces with the greatest amount of particle generation, heat, and wear.

combinations, or specific grades, contact the equipment vendor.

(a) The "Material Characteristics" column offers insight into application-specific attributes of the material. The materials for both faces in the face pair should have preferred or accepted ratings in the Material Characteristics column.

(b) The "Face Pair Compatibility" column of Figure BB-1-1 addresses the tribological characteristics of the face pair. The face pair is lubricated by the process fluid. The grading system is a direct indication of expected seal performance including service life, weepage rate, and particle generation.

(c) The grading system for material characteristics and face pair compatibility is explained in the General Notes for Figure BB-1-1.

(d) The instructions to select mechanical seal face materials using Figure BB-1-1 are as follows:

(1) Select the material for one of the seal faces in the "Material List" column.

(2) Verify the material selection is suitable using the grading system in the "Material Characteristics" column.

(3) Select a mating face material based on the grade indicated in one of the "Face Pair Compatibility" columns.

(4) Verify the mating face material is preferred or accepted in the "Material Characteristics" columns.

NONMANDATORY APPENDIX CC
(19) EXAMINATION, INSPECTION, AND CROSS REFERENCES

Type	Topic	Paragraph/Figure/Table
	General, Personnel, and Procedures	
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	Welding operator coupons	MJ-6.3
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	Piping — Examination procedures	MJ-7.1.2
	Tubing — Examination procedures	MJ-7.1.3
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	Copper tubing/piping — Examiners qualification	MJ-7.2.5
	Polymeric materials — Examination procedures	MJ-9.6.1
	Polymeric materials — Examiners qualification	MJ-9.6.2
Inspection	Inspector definitions	GR-4.1
	Inspector's Delegate	GR-4.2
	Levels of qualification — Inspector's Delegate	GR-4.2.1
	Qualification requirements — Inspector's Delegate	GR-4.2.2
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	Recertification — Inspector's Delegate	GR-4.2.4
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	Piping — Inspectors and Inspector’s Delegates qualification	MJ-7.2.2
	Tubing — Inspectors and Inspector’s Delegates qualification	MJ-7.2.3
	Tube attachments — Inspectors and Inspector’s Delegates qualification	MJ-7.2.4
	Copper tubing/piping — Inspectors and Inspector’s Delegates qualification	MJ-7.2.5
	Polymeric materials — Inspectors and Inspector’s Delegates qualification	MJ-9.6.2
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	Corrosion testing	MM-8.2
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Examination	Pressure vessels and tanks — Examination requirements	MJ-7.3.1(a)
	Visual examination acceptance criteria for welds on metallic pressure vessels and tanks	Table MJ-8.2-1
Inspection	Pressure vessels and tanks — Inspection requirements	MJ-7.3.1(b)
Testing	Pressure vessels and tanks — Testing requirements	MJ-7.3.1(c)

Type	Topic	Paragraph/Figure/Table
	Piping	
Examination	Piping examination requirements	MJ-7.3.2(a)
	Visual examination acceptance criteria for welds on metallic pipe	Table MJ-8.3-1
Inspection	Piping inspection requirements	MJ-7.3.2(b)
Testing	Piping testing requirements	MJ-7.3.2(c)
	Metallic Tubing and Components	
Examination	Acceptable and unacceptable weld profiles for groove welds on metallic tube-to-tube butt joints	Figure MJ-8.4-1
	Discoloration acceptance criteria for welds and heat-affected zones on mechanically polished UNS S31603 tubing	Figure MJ-8.4-3
	Acceptable and unacceptable metallic weld bead width and meander on non-process contact surfaces of groove welds on tube-to-tube butt joints	Figure MJ-8.4-4
	Acceptable weld profiles for metallic tube-attachment fillet welds	Figure MJ-8.5-1
	Discoloration acceptance criteria for welds and heat-affected zones on electropolished UNS S31603 tubing	Figure MJ-8.4-2
	Tubing examination requirements	MJ-7.3.3(a)
	Tube attachments examination requirements	MJ-7.3.4(a)
	Visual examination acceptance criteria for groove welds on metallic tube-to-tube butt joints	Table MJ-8.4-1
	Visual examination acceptance criteria for metallic tube-attachment welds	Table MJ-8.5-1
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	Fitting testing	DT-6
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Examination	Polymeric materials examination requirements	MJ-9.6.3.1
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Type	Topic	Paragraph/Figure/Table
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	Fluid requirements for leak testing	SD-7.3

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NONMANDATORY APPENDIX DD
CONDUCTIVITY SENSOR SELECTION GUIDE

DD-1 PURPOSE

The purpose of this Appendix is to provide guidance for the selection of conductivity sensor technologies based on general process conditions and owner/user requirements. This document is a supplement to the applicable suctions of Part PI.

DD-2 GENERAL CONSIDERATIONS OF COMMON SENSE TECHNOLOGIES

The following are important aspects for the owner/user to consider when selecting a conductivity sensor.

DD-2.1 Measurement Range and Accuracy

DD-2.1.1 Range. Identifying the range of measurement helps the owner/user select the proper type of sensor technology and appropriate cell constant, when applicable. See Figure DD-2.1.1-1 for general applications and where they might fall on a conductivity scale.

(a) Two-electrode technologies are well suited for low conductivity applications with a narrow conductivity range.

(b) Multielectrode technologies are well suited for medium conductivity applications with relatively wide conductivity ranges.

(c) Electrodeless technologies are well suited for medium to high conductivity applications with wide conductivity ranges.

DD-2.1.2 Accuracy. The accuracy of the conductivity measurement is dependent on the combined accuracy of the individual components in the measurement loop.

DD-2.2 Temperature Compensation

DD-2.2.1 Sensors. Most conductivity sensors incorporate an integrated temperature sensor to compensate for the effects of temperature on raw conductivity measurements. Integrated temperature sensors are not intended to replace external temperature sensors dedicated to process monitoring.

DD-2.2.2 Response Times. Integrated temperature sensor response times may lag changes in the process fluid temperature, resulting in longer conductivity stabilization times. To improve temperature sensor response time, an external temperature sensor can be used as long as the external temperature sensor is compatible with the conductivity sensor electronics.

DD-2.3 Installation

DD-2.3.1 Interferences. Consideration should be given to reducing or eliminating installation-induced interferences. Installation-induced interferences include but are not limited to siphoning, cavitation, flashing, nonfull process line, field effects (i.e., proximity to pipe or vessel walls), incomplete mixing, and EMI and RFI interference. Installation-induced interferences may result in measurement errors.

DD-2.3.2 Other Considerations. Other installation considerations include ensuring that the sensor is accessible and does not interfere with other process components.

DD-2.4 Process

Consideration should be given to reducing or eliminating process-induced interferences. Process-induced interferences include but are not limited to entrained air and rapid temperature changes. Process-induced interferences may result in measurement errors.

DD-2.5 Maintenance

Ensure that sensor maintenance requirements can be performed (e.g., gasket or seal replacement).

DD-2.6 Pros vs. Cons of Common Sensor Technologies

See Table DD-2.6-1.

Figure DD-2.1.1-1 Conductivity Scale

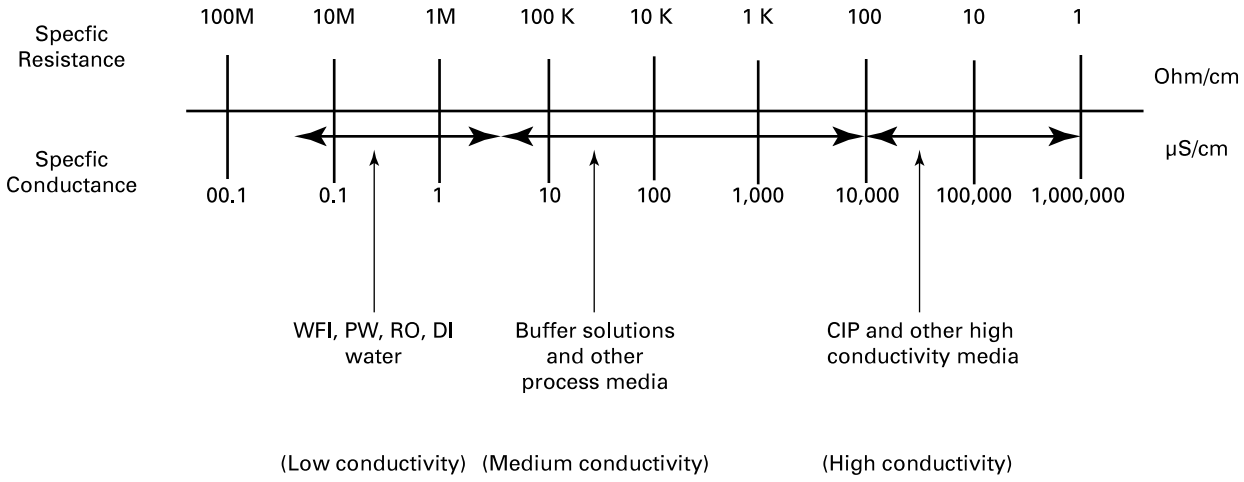

Table DD-2.6-1 Technological Considerations

Sensor Type	Pros	Cons
Two-electrode	1. Works well in low conductivity applications [Note (1)].	1. Limited resistance to electrode coating, fouling, or chemical attack may result in measurement error or drift.
	2. Sensor technology is not sensitive to pipe/tube wall proximity when properly installed. On-line calibration is not necessary to account for conductivity field distortions.	2. Narrow turndown ratio. Best applied to applications with a narrow conductivity range (e.g., compendial water).
Multielectrode	1. Works well in low, medium, or high conductivity applications, but generally selected for medium conductivity applications.	1. Electrodes mounted too close to the tube wall may cause conductivity field distortions. In such cases, on-line calibration would be required [Note (2)].
	2. High turndown ratio.	2. Certain chemicals and sensor materials may not be compatible.
	3. Ability to reduce the potential for measurement error or drift by compensating for some types of process-induced electrode coating, fouling, or chemical attack.	3. Generally open (uncontrolled) geometry sensor designs require on-line calibration.
	4. Coating, fouling, or chemical attack alarms/diagnostics usually available.	4. Coating, fouling, or chemical attack alarms/diagnostics usually not available.
	5. Controlled geometry technologies are available.	5. Using a sensor without a controlled geometry may create measurement errors based on proximity to pipe/tube wall and material of pipe/tube wall construction.
Electrodeless (Toroidal)	1. Works well in medium to high conductivity applications. Best suited for the measurement of high conductivity process fluids.	1. Low conductivity designs are available, but may require larger pipe/tube sizes due to sensitivity concerns.
	2. High turndown ratio.	2. Certain chemicals may not be compatible with certain sensor materials.
	3. Ability to reduce the potential for measurement error or drift by compensating for some types of process-induced electrode coating, fouling, or chemical attack.	3. Generally open (uncontrolled) geometry sensor designs require on-line calibration.

NOTES:

(1) Can also be used for medium to high conductivity applications, although multielectrode or electrodeless sensors are generally preferred because of greater resistance to electrode coating, fouling, and chemical attack.

(2) In some low conductivity applications, calibration verification can be difficult because low conductivity standards are easily contaminated.

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