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Standard Practice for the Collection of Samples of Filterable and Nonfilterable Matter in Water¹

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1. Scope

1.1 This practice is applicable for sampling condensed steam or water, such as boiler feedwater, for the collection of filterable and (optional) nonfilterable matter using 0.45- μ m membrane filter (filterable matter) and ion exchange media (nonfilterable matter). As the major filterable containment found in most boiler feedwaters is some form of corrosion product from the preboiler system, the device used for this practice is commonly called a corrosion product sampler.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

¹ This practice is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-formed Deposits, Surveillance Analysis of Water; for Power Generation and Flow Measurement of Process Use, On-Line Water Samples, Analysis, and Surveillance of Water.

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- D 1066 Practice for Sampling Steam²
- D 1129 Terminology Relating to Water²
- D 1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits²
- D 1193 Specification for Reagent Water²
- D 1971 Practices for Digestion of Water Samples for Determination of Metals by Flame Atomic Absorption, Graphite Furnace Atomic Absorption, Plasma Emission Spectroscopy, or Plasma Mass Spectrometry²
- D 2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence³
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water²
- D 3370 Practice for Sampling Water from Closed Conduits²
- D 3864 Guide for Continual On-Line Monitoring Systems for Water Analysis²

3. Terminology

3.1 *Definitions*— For definitions of terms used in this practice, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *corrosion product sampler, n*—a device used to collect integrated samples of filterable and (as an option) nonfilterable matter. It consists of a flow totalizer that accurately measures the amount of sample passing through the device and a 0.45- μm pore size membrane filter. Adding a second filter—holder for ion exchange resin impregnated membranes allows for collecting nonfilterable matter.

3.2.2 *filterable matter, n*—includes all matter that is removed by a 0.45- μm pore size filter.

3.2.3 *nonfilterable matter, n*—includes all matter that will pass through a 0.45- μm pore size filter and may be captured on anion, or cation ion exchange membranes, or both.

4. Summary of Practice

4.1 A typical sampling apparatus, ~~commonly called a~~ or corrosion product sampler, is used to obtain integrated, representative samples of filterable and nonfilterable matter using a 0.45- μm membrane filter and ion exchange membranes. The sampling is accomplished at system operating pressure or after pressure reduction, and sample temperature of $\leq 50^\circ\text{C}$. The practice utilizes a modified stainless steel high pressure filter housing to accommodate a 47-mm diameter filter (for filterable matter) and if desired, ion exchange membranes (for nonfilterable matter). The sample collection system (corrosion product sampler) is designed and operated specifically for quantitative ~~determination~~ collection of filterable and nonfilterable matter. An important feature of the sampler is the flow totalizer, which accurately determines the total volume of sample ~~water which~~ that has passed through the sampler, regardless of changes in flowrate or pressure during the collection period. Control and pressure reducing valves and metering devices are downstream of the filter housing to eliminate the possible contribution of filterable and nonfilterable matter from these components to the sample stream. Additional flow may bypass the filter housing, so that flows within the sample lines are maintained within required range (see Guide D 3864). If a single sampling point is not representative due to lack of homogeneity in the process fluid (the water being sampled), multiple point sampling may be required.

5. Significance and Use

5.1 The transport of ~~suspended and~~ any filterable matter or corrosion products from the preboiler cycle has been shown to be detrimental to all types of steam generating equipment. Corrosion product transport as low as 10 ppb can have significant impact on steam generators performance.

5.2 Deposited corrosion products on PWR steam generator tubes can reduce heat transfer, and, if the deposit is sufficiently thick, can provide a local area for impurities in the bulk water to concentrate, resulting in a corrosive environment. In BWR plants, the transport of corrosion products can cause fuel failure, out of core radiation problems from activation reactions, and other material related problems.

5.3 In fossil plants, the transport of corrosion products can reduce heat transfer in the boilers leading to tube failures from overheating. The removal of these corrosion products by chemical cleaning is expensive and potentially harmful to the boiler tubes.

5.4 Normally, grab samples are not sensitive enough to detect changes in the level of corrosion product transport. Also, system transients may be missed by only taking grab samples. An integrated sample over time will increase the sensitivity for detecting the corrosion products and provide a better understanding of the total corrosion product transport to steam generators.

6. Interferences

6.1 The ion exchange capacity may be exceeded if an excessive volume of sample is passed through the ion exchange media.

6.2 The removal efficiency of the ion exchange media is flowrate and matrix dependent and could show variations from lot to lot.

6.3 Sample temperature greater than 50°C may have deleterious effects on the ion exchange media.

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 11.02.

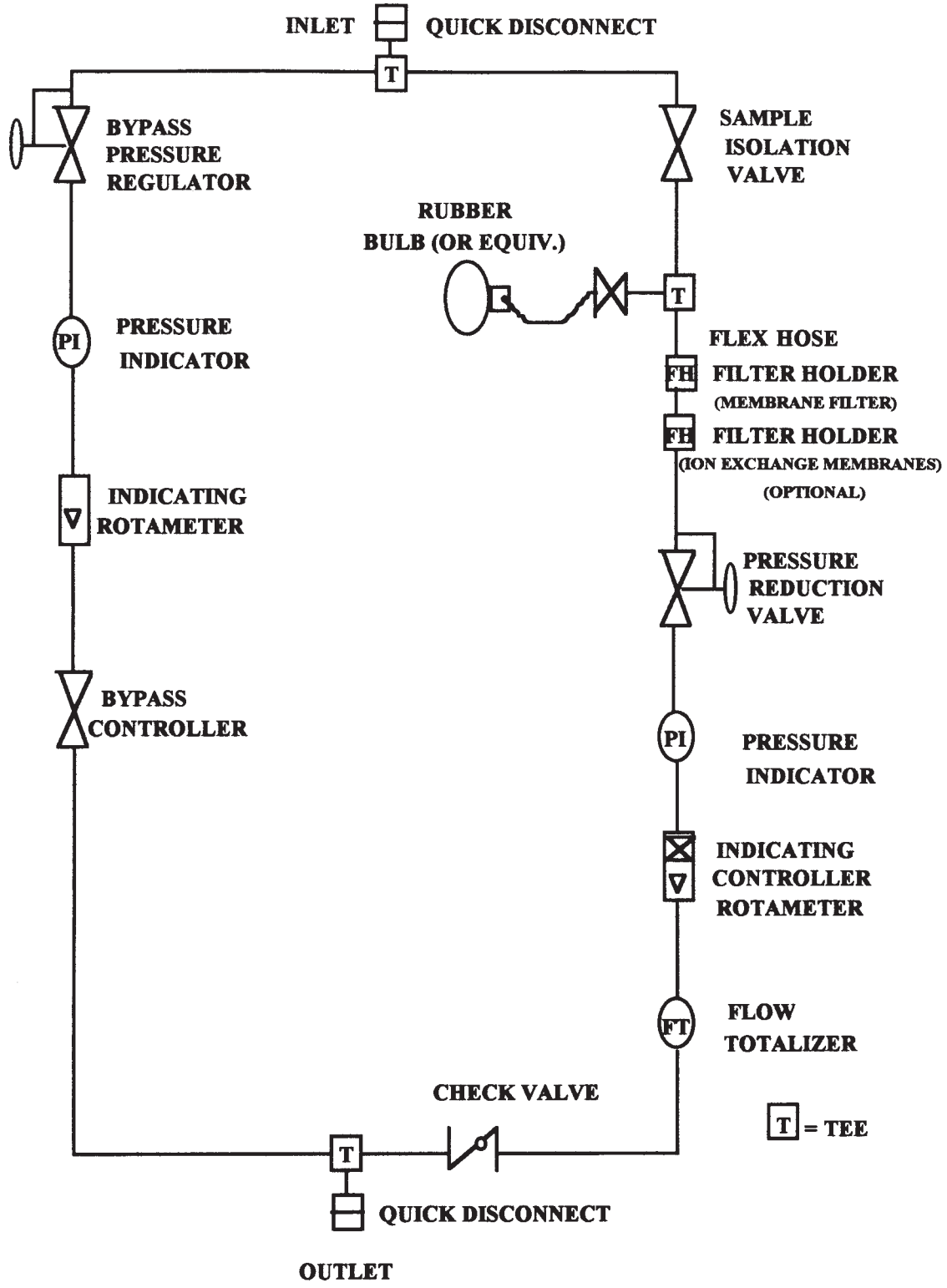


FIG. 1 Simplified Flow Diagram for Corrosion Product Sampler

6.4 The corrosion products collected on the 0.45- μm filter may be loose so care should be taken to prevent loss of sample.

6.5 Due to settling, or deposition, or both, in sampling lines with low velocity, flow in sampling lines must be turbulent and maintained at a velocity of 1.5 – 2.1 m/s (5–7 ft/s) (see also Guide D 1192).

7. Apparatus

7.1 *Sample heat exchanger*, made of such material that full system pressure can be maintained within the coil, and of such capacity that the water being sampled will be cooled to less than 50°C when the sampling flow rate is established (see Guide D 1192).

7.2 *Corrosion Product Sampler*—See Fig. 1, Simplified Flow Diagram.

7.3 *Flow Totalizer*—Water meter that will maintain $\pm 5\%$ accuracy over full range.

8. Reagents and Materials

8.1 *Reagent Water*—Reference to reagent water shall be understood to mean Type I reagent water conforming to Guide D 1193.

8.2 Anion resin impregnated membranes (47-mm diameter), optional.

8.3 Cation resin impregnated membranes (47-mm diameter), optional.

8.4 *Membrane Filters*, (47-mm diameter), 0.45- μm pore size, without grid.

8.5 *Petri Dishes*, large enough to hold the 47-mm filters.

9. Calibration

9.1 Calibrate the flow totalizer following the manufacturer's recommendation.

10. Procedure

10.1 If subsequent chemical analysis of collected filterable/nonfilterable material is desired, record the lot numbers of the ion exchange membranes. Prepare sample blanks from same lot.

10.2 Install filter and optional ion exchange membranes in filter holder so that the sample goes through the filter first, taking care to ensure that they are centered. If necessary, use a few drops of water to wet the membranes to help hold them in place.

NOTE 1—If two filter holders are used, the filter membrane should precede the ion exchange membranes in the second holder.

10.3 Install top of the filter housing, taking care not to disturb membranes.

10.4 With the sample “indicating controller rotameter” closed, slowly open the sample isolation valve. Take the initial flow totalizer reading.

10.5 Slowly increase flow through filter holder to the desired settings. Select the flow rate not to exceed the capacity of the ion exchange papers, if used (the normal flow range is 80 to 200 mL/min).

10.6 Collect the sample using Practices D 1066 or D 3370. Maintain flow constant throughout the incoming line and through the filter holder.

10.7 Slowly isolate and depressurize the corrosion product sampler at the end of the collection period. Record the final totalizer reading.

10.8 Remove excess water from the filter housing by draining it or by purging it with a small amount of air (see Fig. 1, rubber bulb).

10.9 Remove the top of the filter housing, taking care not to disturb collected material.

10.10 Remove membranes from filter housing. Use Petri dishes to store membranes. Membrane filters, anion ion exchange membranes, and cation ion exchange membranes should be stored in separate Petri dishes.

10.11 Analyses of the membrane filters or of the ion exchange membranes may be accomplished by a variety of methods following appropriate sample preparation technique, depending on the analytes of interest and the quantity of the specific analyte collected on the 0.45- μm pore size filter or on the resin impregnated membranes. Preparation should be consistent with the method of analysis, and is beyond the scope of this practice. analysis. Refer to the specific method of analysis for specific guidance and information on preparation, sample storage, etc. For the usual corrosion products of interest (iron, copper, and zinc collected as either filterable or nonfilterable matter), refer to Practices D 1971. For elements above atomic weight 11, particularly chlorine and sulfur, contained in anions collected as nonfilterable matter and concentrated above 0.1%, Practice D 2332 might be used.

11. Calculation

11.1 A calculation will be required to make the result of the analysis of the 0.45- μm filter or ion exchange membrane relate to the flow totalizer reading and express a meaningful result.

11.2 For example, to express ppb results:

$$\text{ppb of analyte in sample stream} = \frac{\mu\text{g of analyte on filter}}{\text{total flow in liters}}$$

12. Quality Control

12.1 The accuracy of the flowmeter and agreement with the totalizer should be checked by timing a measured quantity of sample through the sampler. This procedure is repeated periodically to check on the operation of the totalizer.

12.2 Initially, a blank will be determined on the type of 0.45 µm filter or the type of ion exchange membrane used, or both, that has gone through the complete handling, installation, and removal procedures, minus the sample flow, for each analyte of interest.

12.3 Any additional QC practices required by the method of analysis, such as Practices D 1971 or Practice D 2332 referenced above, if used will be followed.

13. Precision and Bias

13.1 Practice D 2777 is not applicable to this Practice because it includes methodology involving continuous sampling.

13.2 The precision and bias of the analysis of the filters or ion exchange membranes will be governed by those procedures used for the individual analytes of interest.

14. Keywords

14.1 corrosion products; corrosion product sampler; filtration; integrated sample; ion exchange membranes

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