Designation: D6301 – 13

Standard Practice for
Collection of On-Line Composite Samples of Suspended Solids and Ionic Solids in Process Water[1]

This standard is issued under the fixed designation D6301; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is applicable for sampling condensed steam or water, such as boiler feedwater, for the collection of suspended solids and (optional) ionic solids using a 0.45-µm membrane filter (suspended solids) and ion exchange media (ionic solids). As the major suspended component 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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:*[2]

D1066 Practice for Sampling Steam

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1971 Practices for Digestion of Water Samples for Determination of Metals by Flame Atomic Absorption, Graphite Furnace Atomic Absorption, Plasma Emission Spectroscopy, or Plasma Mass Spectrometry

D2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3370 Practices for Sampling Water from Closed Conduits

D3864 Guide for On-Line Monitoring Systems for Water Analysis

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *corrosion product sampler, n*—a device used to collect integrated samples of suspended solids and (as an option) ionic solids.

3.2.1.1 *Discussion*—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 for ion exchange resin impregnated membranes allows for collecting ionic solids.

3.2.2 *ionic solids, 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.

3.2.3 *suspended solids, n*—includes all matter that is removed by a 0.45-µm pore size filter.

4. Summary of Practice

4.1 A typical sampling apparatus, or corrosion product sampler, is used to obtain integrated, representative samples of suspended solids and ionic solids 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 ≤50°C. The practice utilizes a modified stainless steel high pressure filter housing to accommodate a 47-mm diameter filter (for suspended solids) and if desired, ion exchange membranes (for ionic solids). The sample collection system (corrosion product sampler) is designed and operated specifically for quantitative collection of suspended solids and ionic solids. An important feature of the sampler is the flow totalizer, which accurately determines the total volume of sample 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 suspended solids and ionic solids from these components to the sample stream.

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[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

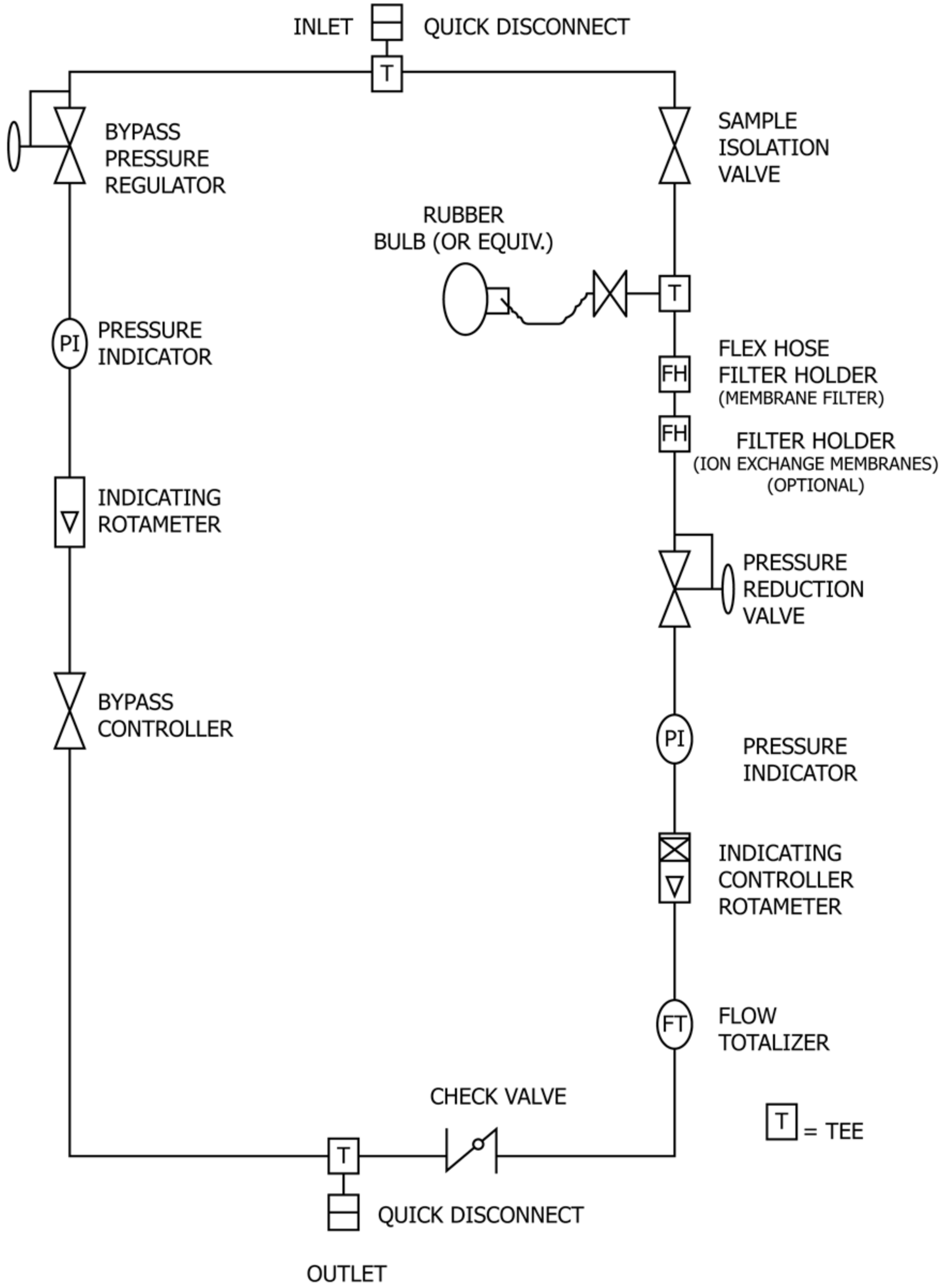

FIG. 1 Simplified Flow Diagram for Corrosion Product Sampler

Additional flow may bypass the filter housing, so that flows within the sample lines are maintained within required range (see Guide D3864). 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 any suspended solids 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.

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 1.8 m/s (6 ft/s) (see also Practices D3370).

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 Practices D3370).

7.2 *Corrosion Product Sampler*—See Fig. 1.

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

8. Reagents and Materials

8.1 *Reagent Water*—References to water shall be understood to mean water that meets or exceeds the quantitative specifications for Type III reagent water conforming to Specification D1193, Section 1.1.

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 suspended solids/ionic solids 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 D1066 or D3370. 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. 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 suspended solids or ionic solids), refer to Practices D1971. For elements above atomic weight 11, particularly chlorine and sulfur, contained in anions collected as ionic solids and concentrated above 0.1 %, Practice D2332 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:

$$\text{ppb of analyte in sample stream} = \frac{\mu\text{g of analyte on filter}}{\text{total flow in litres}} \quad (1)$$

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 quality control (QC) practices required by the method of analysis, such as Practices D1971 or Practice D2332 referenced above, if used will be followed.

13. Precision and Bias

13.1 Practice D2777 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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