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Standard Practices for Sampling Water-Formed Deposits¹

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1. Scope

1.1 These practices cover the sampling of water-formed deposits for chemical, physical, biological, or radiological analysis. The practices cover both field and laboratory sampling. It also defines the various types of deposits. The following practices are included:

	Sections
Practice A—Sampling Water-Formed Deposits From Tubing of Steam Generators and Heat Exchangers	8 to 10
Practice B—Sampling Water-Formed Deposits From Steam Turbines	11 to 14

1.2 The general procedures of selection and removal of deposits given here can be applied to a variety of surfaces that are subject to water-formed deposits. However, the investigator must resort to his individual experience and judgment in applying these procedures to his specific problem.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *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. See Section 7, 9.8, 9.8.4.6, and 9.14 for specific hazards statements.*

2. Referenced Documents

2.1 ASTM Standards:²

- D512 Test Methods for Chloride Ion In Water
- D934 Practices for Identification of Crystalline Compounds in Water-Formed Deposits By X-Ray Diffraction
- D1129 Terminology Relating to Water

¹ These practices are under the jurisdiction of ASTM Committee D19 on Water and are 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.

- D1193 Specification for Reagent Water
- D1245 Practice for Examination of Water-Formed Deposits by Chemical Microscopy
- D1293 Test Methods for pH of Water
- D2331 Practices for Preparation and Preliminary Testing of Water-Formed Deposits
- D2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence
- D3483 Test Methods for Accumulated Deposition in a Steam Generator Tube
- D4412 Test Methods for Sulfate-Reducing Bacteria in Water and Water-Formed Deposits

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *biological deposits*—water-formed deposits of organisms or the products of their life processes.

3.1.1.1 The biological deposits may be composed of microscopic organisms, as in slimes, or of macroscopic types such as barnacles or mussels. Slimes are usually composed of deposits of a gelatinous or filamentous nature.

3.1.2 *corrosion products*—a result of chemical or electrochemical reaction between a metal and its environment.

3.1.2.1 A corrosion deposit resulting from the action of water, such as rust, usually consists of insoluble material deposited on or near the corroded area; corrosion products may, however, be deposited a considerable distance from the point at which the metal is undergoing attack.

3.1.3 *scale*—a deposit formed from solution directly in place upon a surface.

3.1.3.1 Scale is a deposit that usually will retain its physical shape when mechanical means are used to remove it from the surface on which it is deposited. Scale, which may or may not adhere to the underlying surface, is usually crystalline and dense, frequently laminated, and occasionally columnar in structure.

3.1.4 *sludge* —a water-formed sedimentary deposit.

3.1.4.1 The water-formed sedimentary deposits may include all suspended solids carried by the water and trace elements which were in solution in the water. Sludge usually does not cohere sufficiently to retain its physical shape when mechanical means are used to remove it from the surface on which it deposits, but it may be baked in place and be hard and adherent.

3.1.5 *water-formed deposits*—any accumulation of insoluble material derived from water or formed by the reaction of water upon surfaces in contact with the water.

3.1.5.1 Deposits formed from or by water in all its phases may be further classified as scale, sludge, corrosion products, or biological deposit. The overall composition of a deposit or some part of a deposit may be determined by chemical or spectrographic analysis; the constituents actually present as chemical substances may be identified by microscope or x-ray diffraction studies. Organisms may be identified by microscopic or biological methods.

3.2 *Definitions*—For definitions of other terms used in these practices, refer to Definitions **D1129**.

4. Summary of Practices

4.1 These practices describe the procedures to be used for sampling water-formed deposits in both the field and laboratory from boiler tubes and turbine components. They give guidelines on selecting tube and deposit samples for removal and specify the procedures for removing, handling, and shipping of samples.

5. Significance and Use

5.1 The goal of sampling is to obtain for analysis a portion of the whole that is representative. The most critical factors are the selection of sampling areas and number of samples, the method used for sampling, and the maintenance of the integrity of the sample prior to analysis. Analysis of water-formed deposits should give valuable information concerning cycle system chemistry, component corrosion, erosion, the failure mechanism, the need for chemical cleaning, the method of chemical cleaning, localized cycle corrosion, boiler carryover, flow patterns in a turbine, and the rate of radiation build-up. Some sources of water-formed deposits are cycle corrosion products, make-up water contaminants, and condenser cooling water contaminants.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all cases. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of analysis.

6.1.1 *Purity of 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.

6.2 *Materials*:

6.2.1 The highest purity material available should be used for removing the deposit samples.

6.2.2 *Filter Paper* may contain water leachable contaminants (chloride, fluoride, and sulfur) which can be removed by pretreatment prior to sampling.

6.2.3 *Polyester Tape* may contain impurities of antimony and cadmium which must be considered during analysis.

7. Hazards

7.1 *Warnings*:

7.1.1 Special safety precautions are necessary in using acetone on a wipe material for removing water-formed deposits (see **9.8.4.6**).

7.1.2 Special handling precautions may be required for working with water-formed deposits containing radioactive nuclides (see **9.14**).

7.2 *Cautions*:

7.2.1 Extreme care must be taken not to damage the underlying surface when removing water-formed deposit samples from equipment in the field (see **9.8**).

7.2.2 The selection of samples necessarily depends on the experience and judgment of the investigator. The intended use of the sample, the accessibility and type of the deposit, and the problem to be solved will influence the selection of the samples and the sampling method.

7.2.3 The most desirable amount of deposit to be submitted as a sample is not specific. The amount of deposit should be consistent with the type of analysis to be performed.

7.2.4 The samples must be collected, packed, shipped, and manipulated prior to analysis in a manner that safeguards against change in the particular constituents or properties to be examined.

7.2.5 The selection of sampling areas and number of samples is best guided by a thorough investigation of the problem. Very often the removal of a number of samples will result in more informative analytical data than would be obtained from one composite sample representing the entire mass of deposit. A typical example is the sampling of deposits from a steam turbine. Conversely, in the case of a tube failure in a steam generator, a single sample from the affected area may suffice.

7.2.6 Most deposits are sampled at least twice before being submitted to chemical or physical tests. The gross sample is first collected from its point of formation in the field and then this sample is prepared for final examination in the laboratory.

7.2.7 A representative sample is not an absolute prerequisite. The quantity of deposit that can be removed is often limited. In such cases, it is better to submit a single mixed sample (composite) and to describe how the sample was obtained. For radiological analysis all samples should be checked for activity levels before preparing a composite since wide variations in radioactive content may occur in samples of similar appearance and chemical composition.

7.2.8 It is good practice for deposits to be taken and analyzed every time a turbine is opened for repairs or inspection. Deposit history can then supplement chemical records of a unit, and deposit chemistry of units with and without corrosion and other problems can be compared. Enough

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Selection of Samples

	Tube 1		Tube 2
	Principal Area	Adjacent Area	Adjacent Tube 1 or Related Tube
Preferred selection procedure	X	X	X
Alternative selection procedure 1	X	X	
Alternative selection procedure 2	X		
Alternative selection procedure 3		X	
Alternative selection procedure 4			X

information on deposits has been published (1,2)⁴ that a comparison between different types of boilers and different water treatments, as well as an assessment of deposit corrosiveness, are possible. It has been a general experience that about 0.2 % of a corrosive impurity, such as chloride, in a deposit, is a division between corrosive and noncorrosive deposits.

7.2.9 Deposits taken after a turbine is open do not exactly represent chemical composition of deposits in an operating turbine. Chemical thermodynamic data on steam additives and impurities, such as vapor pressures of solutions, ionization, and volatility data are needed to reconstruct chemistry of environment during operation.

7.2.10 Typical changes which occur after the hot turbine is shut down and air is admitted are: (1) reactions with oxygen and carbon dioxide, (2) drying of some deposits and water absorption by others, (3) leaching and recrystallization where moisture is allowed to condense, and (4) formation of iron hydroxide and hematite.

PRACTICE A—SAMPLING WATER-FORMED DEPOSITS FROM TUBING OF STEAM GENERATORS AND HEAT EXCHANGERS

8. Scope

8.1 This practice covers the sampling of water-formed deposits from tubing of steam generators and heat exchangers. It covers both field and laboratory sampling of water-formed deposits. It gives guidelines on selecting tube samples for removal and specifies the procedure for removing tube samples from the unit.

9. Field Sampling

9.1 *Selection of Tube Samples*—Whenever feasible, remove the tube containing the water-formed deposit. The length of tubing removed depends on the amount of deposits present and the type of analyses to be performed. As a guideline, 3 ft (0.9 m) of tubing is suggested. Table 1 contains a summary of the various procedures for selection of samples in the order of preference.

9.1.1 *Preferred Selection Procedure*—Select one or more separate tube samples containing the area of failure, heaviest deposition, or principle concern (primary area) and include any adjacent or closely related areas of these tube samples that

might contain deposits significantly different from the primary area. Also, one or more tube samples is selected from adjacent rows or other related areas that might contain deposits significantly different from the primary area.

9.1.2 *Alternative Selection Procedure 1*—Select one or more separate tube samples containing the area of failure, heaviest deposition, or principle concern (primary area) and include any adjacent or closely related areas of these tube samples that might contain deposits significantly different from the primary area. Use this procedure when it is impractical to remove the samples from adjacent rows or other related areas or when it is improbable that the information gained by such sampling will justify the additional work involved.

9.1.3 *Alternative Selection Procedure 2*—Select one or more separate tube samples containing the area of failure, heaviest deposition, or principle concern (primary area). Use this procedure when only the tube section containing the primary area can be removed or when it is impractical to remove adjacent or closely related areas, or tube samples from adjacent rows or other related areas, or when it is improbable that the information gained by such sampling will justify the additional work involved.

9.1.4 *Alternative Selection Procedure 3*—Select one or more tube samples containing an area adjacent or closely related to the primary area. Use this procedure only when it is not possible to obtain a tube section containing the primary area.

9.1.5 *Alternative Selection Procedure 4*—Select one or more separate tube samples from adjacent rows or other related areas. Use this procedure only when it is not possible to remove a tube section from the primary area, adjacent to the primary area, or closely related to the primary area.

9.2 *Taking the Tube Sample*—Mark the tube that is to be removed (sampled) with a crayon. A long arrow can be used to show: (1) the ligament that is facing into the furnace, and (2) which end of the tube is up. Mark the tube before it is removed. The marking should not involve the use of a hammer and die or paint.

9.2.1 Whenever possible, remove the tube samples by sawing. The tube should be dry-cut (no oil). Grinding wheels and cutting torches can be used to obtain tube samples which cannot be sawed. Grinding wheels and cutting torches can produce sufficient heat to alter the composition of the deposit near the cutting point. If a grinding wheel or cutting torch is used, make the cut a minimum of 6 in. (152 mm) from the area of concern.

9.2.2 Usually it is impractical and inconvenient to remove short sections of tubes from a water-cooled furnace wall in order to obtain deposits from the waterside surfaces. Several convenient ways can be used to remove such surfaces. Avoid torch burning whenever possible, since original sample environment is often destroyed and a valid metallographic examination cannot be made of the specimen because of the effects of burning.

9.2.3 If a tube cannot be removed, trepan (hole-saw cutting) above and below the affected area harboring the deposits to remove “window section” from the tube. The window section is removed by connecting the holes with longitudinal cuts using an abrasive wheel. In most cases, the original deposit can

⁴ The boldface numbers in parentheses refer to the references at the end of these practices.

be retained or collected from the affected area on the waterside surfaces of the tube. Also, a metallographic examination can be made because the original area is not altered or destroyed.

9.2.4 Penetrate the tube on the casing side or cold side of the tube if entrance on the furnace side is impractical from a time and cost standpoint. The deposit can be removed readily and a local examination of the waterside surfaces of the tube can be made.

9.2.5 Remove “window sections” only from tubes which have not failed. It is difficult to remove “window sections” from small diameter, thick-walled tubes.

9.3 *Sealing the Sample*—After removing the tube sample, allow the ends to cool. Dry the tube sample as soon as possible. Seal the tube ends with rubber stoppers or cardboard and secure the seal with tape.

9.4 *Sample Label*—Affix a label or a cardboard or linen tag to the sample.

9.4.1 Note the following information on the label or tag as soon as it becomes available. If this information is too voluminous for inclusion on the tag, it can be forwarded in a separate letter or report, properly identified with the samples concerned.

9.4.1.1 Name of organization supplying sample.

9.4.1.2 Name and location of plant.

9.4.1.3 Name and other designation of unit from which sample was removed.

9.4.1.4 Number of sample.

9.4.1.5 Date and time of sampling.

9.4.1.6 Precise location occupied in service.

9.4.1.7 Appearance of sample (note failures, bulges, pits, cracks, etc.).

9.4.1.8 Type of deposit (whether scale, sludge, or corrosion products).

9.4.1.9 Appearance of deposit (note the color, uniformity, texture, odor, and oily matter).

9.4.1.10 Exact procedure that was used in removing the sample and notes concerning any contamination that might have occurred during the process.

9.4.1.11 Identification of opposite walls of the tube sample that might contain different deposits.

9.4.1.12 Statement of whether liquid or vapor was present in the tube sample during operation.

9.4.1.13 Operating temperature, pressure, and rate of flow of liquid or vapor in the tube sample.

9.4.1.14 Type of treatment applied to the water and a chemical analysis of the water that formed the deposit or furnished steam to the affected zone.

9.4.1.15 Description of why the sample was taken; that is, the problem involved.

9.4.1.16 Description of discrepancies in operating condition that could have contributed to the problem.

9.4.1.17 Results of field tests made on the sample or related equipment.

9.4.1.18 Type of analysis necessary to solve the problem.

9.4.1.19 Signature of sampler.

9.4.2 Provide other background information that may be necessary for the specific problem. A few of these are date of

unit startup, dates of chemical cleanings, type and location of water purification equipment, and direction of flow of fluid in the sample.

9.4.3 When numerous samples are collected, forward a diagram of the unit or affected zone to show the precise location of where the samples were removed.

9.5 *Sample Shipping Container*—Use a clean wooden box having a separate compartment for each sample as the shipping container. Line the compartment with corrugated paper, an elastic packing material, or other suitable material. Place each sample in a plastic bag or wrap in heavy paper and place in a compartment in the container. Seal the container properly to protect the samples during transit.

9.6 *Shipping Label*—Print the addresses of consignee and consignor plainly upon two sides of the outer container, or attach firmly thereon by cards or labels. Attach warning and descriptive labels to the outer container.

9.7 *Selection of Deposit Samples*—When it is not possible to remove the tube containing the water-formed deposit, remove the deposit directly from the tube surface in the field. The selection of sampling points will be somewhat limited and depend mainly on accessibility. Representative samples of water-formed deposits on tubes usually can be obtained near steam and mud drums, handholes, and manways. These samples can provide useful information upon analysis, depending on the specific problem involved. A photograph of the area, before and after removal of the deposit, could be a valuable aid in studying the problem and planning the analysis of the sample. The composition next to the underlying surface may be different from that which was in contact with the water. If possible, separation of these two different surfaces should be made to ascertain this possibility. At times, the quantity of deposit that can be removed is limited. In such cases, it is better to submit a single mixed sample (composite) and to describe how the sample was obtained, than to collect no sample. If the deposit weight per unit area is to be determined, Test Methods **D3483** should be reviewed prior to removal of the deposit.

9.8 *Taking the Deposit Sample*—The method of deposit removal will depend on accessibility and the type and amount of deposit present. (**Warning**—in all cases, extreme care must be taken to minimize damage to the underlying surface or contaminate the sample. To avoid chloride and sodium contamination, deposits shall not be contacted with the hands.)

9.8.1 *Tightly Adherent Deposits*—Remove hard, adherent deposits by using a sharp penknife, steel scraper, or scalpel. A vibrating mechanical power device may be required for extremely tenacious deposits.

9.8.2 *Loosely Adherent Deposits*—Remove deposits that adhere loosely to the surface with a knife, spatula, or spoon.

9.8.3 *Thin Hard Films*—Remove thin film deposits on rough or irregular surfaces by using a stainless steel brush, knife, or spatula.

9.8.4 *Thin Soft Films*—Remove thin, soft film deposits by using polyester tape or wiping with filter paper or lint-free cloth. Scotch tape or a comparable polyester tape has been found to be suitable for this application. When using filter paper or lint-free cloth, use the following procedure.