INTERNATIONAL

Designation: D887 − 13

Standard Practices for Sampling Water-Formed Deposits[1]

This standard is issued under the fixed designation D887; 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 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
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, n*—water-formed deposits of organisms or the products of their life processes.

3.1.1.1 Discussion- 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, n*—a result of chemical or electrochemical reaction between a metal and its environment.

3.1.2.1 Discussion- 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, n*—a deposit formed from solution directly in place upon a surface.

3.1.3.1 Discussion- 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, n* —a water-formed sedimentary deposit.

3.1.4.1 Discussion- 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.

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[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.

Current edition approved Jan. 1, 2013. Published February 2013. Originally approved in 1946. Last previous edition approved in 2008 as D887 – 08. DOI: 10.1520/D0887-13.

[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.

3.1.5 *water-formed deposits, n*—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 Discussion- 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*— Reference to water that is used for reagent preparation, rinsing or dilution shall be understood to mean water that conforms to the quantitative specifications of Type III reagent water of SpecificationD1193.

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

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[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

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[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.

9.8.4.1 Wet a filter paper or lint-free cloth with water. (Whatman No. 40 or comparable filter paper, 12.5 cm has been found to be a suitable wiping material.) Wear rubber gloves to reduce the possibility of contaminating the wipe material and the sample.

9.8.4.2 Fold the wipe material to make a compact wad.

9.8.4.3 Wipe the surface to remove the deposit.

9.8.4.4 Continue wiping until it appears that no additional deposit is being removed. Unfold, turn, and refold the wipe material, if necessary. Keep the wipe material moist. Use more than one wipe material if required, but do not use more than is necessary.

9.8.4.5 Measure and record the area wiped.

9.8.4.6 Wet another wipe material with acetone and repeat 9.8.4.2, 9.8.4.3, and 9.8.4.4 using the same area. (**Warning—**Acetone is an extremely flammable liquid and precautions are necessary when it is used. Methods for shipping, handling, storage, and disposal of acetone have been published by the National Safety Council (**3**).)

9.8.4.7 Place the wipe materials (water and acetone wipes) containing the sample onto a sample container. Allow the acetone soaked wipe materials to air dry before placing into the sample container.

9.8.4.8 Select six clean filter papers or three clean cloths from the same lot as was used for removing the deposit and wet with water.

9.8.4.9 Select another six clean filter papers or three clean cloths from the same lot as was used for removing the deposit and wet with acetone. Allow to air dry.

9.8.4.10 Place the clean wipe materials (water and acetone soaked) into a sample container. These will serve as "blanks."

9.8.5 *Heterogeneous Layered Deposits* —Remove these deposits as near to the underlying surface as possible. The separation of the individual layers for examination is best done in the laboratory.

9.8.6 *Deposits in Pits*— Remove deposits in pits with a sharp penknife, steel scraper, scalpel, icepick, or sturdy needle-like probe.

9.9 *Quantity of Samples*—See 7.2.3 and 7.2.7.

9.10 *Sample Container*— Place each sample, as soon as possible after removal from the surface, in a container that will protect it from contamination or chemical change. Sample containers shall be made of materials that will not contaminate the sample and shall be clean and dry. A wide-mouth plastic bottle, small plastic vial, plastic bag, or noncorrodible metal container are suitable containers. Do not use corrodible metal containers for wet samples. Glass bottles and vials are also suitable, but are fragile and subject to breakage. Paper containers are not recommended. Fix the closures for the sample containers in place by wire, tape, or cord to prevent loss of the sample in transit if they are to be shipped.

9.11 *Sample Labels*— See 9.4.

9.12 *Sample Shipping Container*—Use a wooden box, cardboard box, or envelope as the shipping container, depending on the number and size of the samples. Line the shipping container with corrugated paper, an elastic packing material, or other suitable material and properly seal.

9.13 *Shipping Labels*— See 9.6.

9.14 *Radioactive Material*—In sampling water-formed deposits for the determination of its radioactivity, apply the normal principles of sampling. However, it may be impossible to remove deposit samples by the procedures given in 9.8. In these cases, chemical removal of the deposit may be the only alternative. (**Warning—**Because of the potential hazards related to working with water-formed deposits containing radioactive nuclides, special handling precautions may be required. Such handling will depend on the amount and type of radioactive substances contained in the water-formed deposits and whether determination is to be made for gross radioactivity, for a specific nuclide, or for nonradioactive constituents by established chemical or physical tests.) (**Warning—**The radiation hazard associated with deposit samples may increase significantly when the sample is removed from a system which shields it or when suspended solids are filtered from water.)

9.14.1 *Handling Precautions*—When sampling water-formed deposits where radiation levels or radioactive contamination may be high, such as in nuclear steam systems, follow the applicable health physics regulations. Anyone who is not experienced in the handling of radioactive materials should consult a health physicist or other person experienced in such activities. Personnel monitoring devices may be necessary where external radiation levels present a potential hazard. Where the level of radioactivity in the sampling area is sufficiently great to introduce a radiation hazard, shielding may be required to minimize exposure to radiation. Also, the use of suitable protective clothing may be required and precautionary measures should be exercised to prevent contamination.

9.14.2 For information on radiological hazards and recommendations on radiation protection, refer to the publications by the National Committee on Radiation Protection (**4, 5**) . The recommendations of the Federal Radiation Council on radiation protection guides have been adopted by all federal agencies by executive order of the President (**6**). The existing standards for protection against radiation are set fourth in the Code of Federal Regulations (**7**). Special methods and precautionary measures for handling radioactive samples are described in the literature (**8, 9**).

9.14.3 *Sample Containers*—Select the sample container by considering the type of sample involved and its level of radioactivity. Plastic containers usually are preferable when sampling for radioactivity determinations. The level of radioactivity in the sampling area and of the sample itself must be considered. A special apparatus may be required for shielding sample containers.

9.14.4 *Time Interval Between Collection and Analysis of Samples*—When sampling for radioactivity determinations, note the exact time of sample collection. If short-lived activity is of interest, the analysis should be made as rapidly as practical to minimize loss of activity by radioactive decay. If only long-lived radioactivity is of interest, measurement sometimes can be simplified by allowing sufficient time for the decay of the short-lived radionuclides before analysis.

9.14.5 *Shipping Containers*—Shield and pack the samples to comply with regulations for shipping radioactive materials (**10, 11**) .

10. Laboratory Sampling

10.1 *Preliminary Examination of Tube Samples*—Carefully unpack the tube sample and remove seals and tape from the tube end. Burrs and sharp edges can be removed with a small file. Excessive handling of the sample, which can dislodge or contaminate the deposits, should be avoided. A photograph of the deposit in its original state and in different stages of removal can be taken to provide a valuable record of its appearance. The sample should be examined visually and its length and outside diameter plus any failures, cracks, or pits on the tube noted and recorded. Also, the characteristics of the water-formed deposit on the tube should be noted. The history of the sample should be reviewed and a definite plan for examination and analysis of the sample determined. If a metallurgical analysis (destructive or microchemical, or both) is to be performed, the examining metallurgist should remove specimens prior to removal of the water-formed deposit. The procedure of deposit removal and the amount of deposit removed must be consistent with the type of examination and analyses planned.

10.2 *Removal of Deposits from a Concave Surface (Interior Surface):*

10.2.1 *Preferred Procedure*—Apply this procedure to remove water-formed deposits from a specific location on a tube sample. The procedure can be used when the underlying metal surface is required for metallurgical examination. A cylindrical section that contains adequate deposits for the planned analyses is removed from each tube (or portion thereof) to be sampled. The section is split longitudinally with a shaper or dry saw (no oil). If opposite walls of the section might contain different types or amounts of deposits, as in a furnace wall boiler tube, the longitudinal split is made so as to separate the dissimilar portions. Care should be taken not to contaminate the sample with deposits from the exterior (convex) surface of the section. The deposits are separated from the tube by a suitable mechanical means that will not score the tube metal or otherwise contaminate the sample. See 9.8. If possible, deposits near the tube ends where saw filings and slag may have contaminated the deposit are avoided. If the tube sample was removed with a cutting torch in the field, the deposit near the cutting point should be avoided. The exact location of the deposit removed is noted. The surface area from which the deposit was removed is measured and recorded.

10.2.2 *Alternative Removal Procedure 1*—Apply this procedure when only a bulk, random sample of deposit from the tube surface is desired. Do not use this procedure when the underlying metal is required for metallurgical tests. Not all types of deposits can be dislodged by this method. A cylindrical section is removed from each tube and sectioned longitudinally as in 9.2.1. The deposit is dislodged by squeezing the freshly cut, open edges of the two resulting hemicylindrical sections in a vise.

10.2.3 *Alternative Removal Procedure 2*—When it is not permissible to destroy the tube sample by sectioning, remove superficial deposits by brushing the interior surface and dislodge the principal deposit with a turbine, vibrating head, or rotary cutter. The action of the cutter shall be vigorous enough to remove the deposit, instead of simply polishing it, but shall not be so severe that it causes excessive abrasion of the underlying surface and contamination of the sample.

10.2.4 *Alternative Removal Procedure 3*—Use inhibited chemical solvents when it is desirable to strip the tightly adherent water-formed deposits or protective film, or both from the tube surface after the overlaying loose deposits have been removed. Extreme care is necessary to ensure that only the desired deposits are removed and contamination from other surfaces of the tube sample is avoided.

10.3 *Removal of Deposits from a Convex Surface (Exterior Surface):*

10.3.1 *Preferred Removal Procedure* —See 10.2.1.

10.3.2 *Alternative Removal Procedure 4*—See 10.2.2.

10.3.3 *Alternative Removal Procedure 5*—See 10.2.4.

10.4 *Storage of Samples*—See 9.10.

10.5 *Sample Label*— See 9.4.

10.6 The final preparation of water-formed deposit samples received from the field or obtained from tube samples in the laboratory is a part of the specific analysis to be performed. Various physical and chemical treatments may be given to portions of the sample undergoing identification. Care must be exercised in selecting a representative sample for analysis, since the value of an analysis is related to the sampling technique employed. The following ASTM standards should be reviewed for specific instructions in selecting and preparing samples: Practices D934, Test Methods D4412, Practices D1245, Practices D2331, Practice D2332, and Test Methods D3483.

10.7 Radioactive Material—See 9.14.

PRACTICE B—SAMPLING WATER-FORMED DEPOSITS FROM STEAM TURBINES

11. Scope

11.1 This practice covers the sampling of water-formed deposits from steam turbines. It covers both field and laboratory sampling of water-formed deposits. It gives guidelines for removal of turbine components.

12. Field Sampling

12.1 Field sampling immediately after a turbine is open is preferred since the deposits are easily contaminated by airborne impurities, lubricants and solvents used in disassembly, and by nondestructive test (NDT) fluids. Most of these fluids contain chloride and sulfur as contaminants, some are chlorinated hydrocarbons.

12.2 *Selection of Deposit Samples* —Significance of a sample location differs for different types of turbine cycles (superheated, wet) and depends upon the reasons for deposit analysis (routine, corrosion failure analysis, component malfunction due to deposits, loss of thermodynamic efficiency due to deposits).

12.2.1 For a routine analysis in turbines using superheated steam, deposits from rotating blades at the inlet and exit of each turbine of a turbine set (high, intermediate and low pressure) as well as from the blades at the dry-wet transition region (Wilson line) should be sampled. Both, convex and concave surfaces and the whole length of the blades should be scraped. If there is anything indicating a flow or washing pattern or localized deposition anywhere on the rotating and stationary blades, disks, seals, pipes, valves, etc., a picture or sketch and a localized deposit should be taken.

12.2.2 For a corrosion failure analysis and any time there is a possibility of corrosion influence, deposits from the failed or cracked components and its vicinity should be taken in addition to all the deposits identified in 12.2.3. Frequently, there is a problem with deposit collection from cracked turbine components because they are cleaned (usually by alumina blasting) before the cracks are discovered by NDT. For cases like that, deposits from adjacent stationary blades and inlet pipes should be taken.

12.2.3 When corrosion and corrosion cracking (stress corrosion, corrosion fatigue) is found before component cleaning, a part of the surface should be masked by taping a plastic pad over a deposited surface to allow later microscopic analysis of the corroded surface.

12.2.4 Valve and seal malfunction due to buildup of deposits has been experienced. When solvents are needed in these cases for a disassembly, their chemistry should be known and accounted for in the deposit analysis.

12.2.5 Where deposition of rotating blades results in loss of thermodynamic efficiency, flow pressure changes and increase of thrust load, a deposit profile along all the blade rows is useful. In these cases, deposits may be taken from every rotating and stationary row and localized deposition of individual chemicals, thickness and pH profile established.

12.3 *Taking Deposit Samples*—Refer to 9.8.

12.4 Deposits within pits, cracks, and crevices on components which cannot be brought into a laboratory can be stripped by an acetate replicating tape used in fractography. Such a deposit is suitable for a microscopic, energy dispersive X-ray (EDX) and X-ray diffraction analysis.

12.5 *Quantity of Samples*—See 7.2.3 and 7.2.7.

12.6 *Sample Container*— See 9.10.

12.7 *Sample Label*— See 9.4.

12.8 *Sample Shipping Container*—See 9.12.

12.9 *Shipping Label*— See 9.6.

12.10 *Radioactive Material*—See 9.14.

13. Removal of Turbine Components for Analysis

13.1 Turbine components are usually removed from a disassembled turbine for replacement or laboratory failure analysis. They are rarely removed for analysis of deposits. In analysis of a corrosion problem, analysis of surface morphology and deposition is important.

TABLE 2 Procedures for Analysis of Turbine Deposits

ASTM Designation	Measurement
Practices D2331	phosphates, sulfates, and carbonates
Test Methods D512	chloride
Practices D934	crystalline compounds
Test Methods D1293	pH
Semiquantitative spectroscopy	Fe, Si, Ni, Cr, Mo, Cu, Mn, Al, Co, Ti, V, Ca, Cb, Pb, Mg, Zr
Secondary ion mass spectroscopy (SIMS)	compounds in thin layers
Energy dispersive x-ray (EDX)	elements with atomic number 11 and higher
Liquid ion chromatography **(12, 13)**	Na^{+}, $NH_4^{\ +}$, K^{+}, F^{-}, $Cl^{\ -}$, NO_2^{-}, $NO^{3\ -}$, $PO_4^{-\ 3}$, Br^{-}, $SO_4^{-\ 2}$
Inductively coupled plasma (ICP) **(14)**	metals

13.2 *Removal of a Component*—Critical parts of a turbine component destined for further analysis should be protected against contamination during removal, handling, and shipment. Use of solvents and other fluids should be avoided or their chemistry should be known and accounted for.

13.3 *Shipping*—The whole component or a part of its surface should be reliably protected against rain and airborne contamination, particularly by sea salt and road salts. For labeling and containers, refer to 9.4, 9.6, 9.10, 9.12, and 9.14.

14. Laboratory Sampling

14.1 Laboratory sampling of deposits from turbine components is done similar to the field sampling on a section of a component. Dry cutting of such a section is recommended to avoid deposit washing and contamination.

14.2 Such samples are suitable for microscopic and EDX analysis, studies of deposits within cracks and pits, and for secondary ion mass spectroscopy (SIMS) analysis. Since the quantity of a deposit is usually low, highly sensitive analytical techniques such as liquid ion chromatography and inductively coupled plasma emission of washed deposits are very useful.

14.3 Turbine deposits are usually analyzed semi-quantitatively (by spectroscopy or EDX); quantitatively for anions, sodium, total carbon and total sulfur; and by X-ray diffraction.

14.4 The analyses considered most important are: chloride, sulfate, total sulfur, phosphate, carbonate, hydroxide, silicon, copper, total carbon, iron, pH, and X-ray diffraction identification of chemical compounds. In special cases, additional analyses, such as for chromium, manganese, molybdenum, calcium, potassium, aluminum, lead, fluoride, etc., may be needed. Deposit thickness should be measured or estimated. A list of commonly used procedures is shown in Table 2.

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