Designation: D3483 – 14

Standard Test Methods for Accumulated Deposition in a Steam Generator Tube[1]

1. Scope

1.1 These test methods cover the determination of the weight per unit area of waterside deposits on heat-transfer surfaces of steam generator tubes. The following test methods are included:

	Sections
Test Method A—Mechanical removal by scraper or vibrating tool-removed deposit weight method	7 to 16
Test Method B—Chemical removal by solvent-tube weight loss method	17 to 27
Test Method C—Mechanical removal by glass-bead blasting-tube weight loss method	28 to 37

1.2 Test Method A is a procedure generally applicable to deposits ranging from 1 to 100 g/ft^2. The precision was determined in the collaborative study over the range of 16 to 76 g/ft^2. This procedure allows the discretionary selection of the area on the tube to be sampled. The removed deposit allows for further chemical analysis.

1.3 Test Method B is a procedure generally applicable to deposits ranging from 1 to 100 g/ft^2. The precision was determined in a collaborative study over the range of 28 to 73 g/ft^2. The procedure averages out the heavier and lighter deposited areas. The solvent solution produced allows for further chemical analysis.

1.4 Test Method C is a procedure generally applicable to deposits ranging from 1 to 100 g/ft^2. The precision was determined in a collaborative study over the range of 17 to 88 g/ft^2. The procedure averages out the heavier and lighter deposited areas. The removed deposit does not allow for further chemical analysis.

1.5 These test methods have been generally evaluated on the types of waterside deposits generally found on heat-transfer surfaces of steam generator tubes. It is the user's responsibility to ensure the validity of these test methods for other types of deposits or high temperature scale.

1.6 These methods are sometimes used for accumulated deposition in rifled steam generator tubes. Experience has shown that there is a significant difference in the deposition in the grooves and on the lands on some rifled steam generator tubes. The grooves have been shown to hold more deposit. Test Method B and Test Method C will average out this difference. In Method A the choice exists, either to choose to remove the deposition from the groove if it is visually determined to be more heavily deposited, or to remove equally over the grooves and lands. It is important that it be understood what choices were made and that the report reflect the choices made when using Test Method A on rifled steam generator tubes.

1.7 There are some steam generator tubes where it is apparent that half of the tube is exposed to the flame from the external appearance, this side is typically called the fireside or hot side. The other half of the tube is not exposed to the flame from the external appearance is typically called the casing side or cold side. These test methods also require that the tube be split in half, so the tube is generally split along these lines. On these tubes it is generally found that more internal deposition exists on the fireside or hot side. Some users of these methods will determine the deposition only on side where it appears visually that more deposition exists. Some users will determine the deposition on both sides and report the results separately and some will average the two results. It is important that the user of the data be aware of the choices made and that the report of the results be specific.

1.8 The values stated in either SI or inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.9 *This standard does not purport to address 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]

D887 Practices for Sampling Water-Formed Deposits

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[1] These test methods 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.

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

2.2 *Other Documents:*

NACE Standard TM0199-99, Item No. 21236, Standard Test Method for Measuring Deposit Mass Loading ("Deposit Weight Density") Values for Boiler Tubes by the Glass-Bead-Blasting Technique
NACE International Publication 7H100, Item No. 24206 Evaluation of Boiler Tube Deposit Mass Loading (Deposit Weight Density) Methodology

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D1129.

4. Significance and Use

4.1 The weight per unit area measurement is an indication of the relative cleanliness or dirtiness of the boiler. It is used to determine the effectiveness of the boiler chemical treatment program and to determine the need for chemically cleaning the boiler systems. Allowing the internal deposition to accumulate unchecked will likely lead to boiler tube failures by mechanisms of under deposit corrosion and tube metal overheating.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all regents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[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 the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type 4.

6. Sampling

6.1 Select a tube section likely to have the heaviest deposit. Experience has shown that deposit accumulation is usually heaviest on tube surfaces that receive the highest heat transfer. Representative areas of especially high heat transfer are:

6.1.1 The center of the division wall at the top burner elevation in a boiler with a division panel wall where firing occurs on opposite sides.

6.1.2 The side wall near the top burner elevation, at about ⅓ furnace depth from the burner wall, in a boiler without a division wall.

6.1.3 Other high heat transfer areas in a more complex boiler design as indicated by the boiler manufacturer.

6.2 Areas in the boiler where impaired circulation is suspected may also be sampled.

6.3 The optimal tube removal method is by dry cutting. No oil or water is to be used in the tube cutting process. The length of sample should be 24 to 36 in., but allow an extra 12 in. on each end if the sample is cut out by torch.

6.4 After the tube sample is removed, provide suitable identification, showing location in the boiler, orientation of tube, and the hot and cold sides of the tube in accordance with Practices D887.

TEST METHOD A—MECHANICAL REMOVAL BY SCRAPER OR VIBRATING TOOL

7. Scope

7.1 This test method covers the determination of accumulated deposition in a steam generator tube by the mechanical removal of the deposit by scraper or vibrating tool, the collection and weighing of the dry deposit. This collected deposit is then available for further chemical analysis, if desired. The method also allows for discretionary removal of the deposit from the tube in areas of the most interest.

8. Summary of Test Method

8.1 The tube is split and the area to be tested is determined after examination; usually the area judged to be most heavily deposited. The deposits are removed mechanically by scraping or vibrating from a measured area. The collected deposit is weighed and the result is usually reported as grams of deposit per square foot of boiler tube surface.

9. Interferences

9.1 There are no interferences, only errors in collecting the deposit, removing sufficient deposit, not removing base metal, determination of the sample weight and the sampled area.

10. Apparatus

10.1 *Cutting Tool or Torch,* for removing a suitable portion of the boiler tube.

10.2 *Tube Cutter.*

10.3 *Tube End Sealers,* to protect the sample until the determination can be made.

10.4 *Milling Machine or Band Saw,* to separate the tube into halves by longitudinal sectioning (dry cut).

10.5 *Magnet,* to remove cutting metal chips from the deposited material.

10.6 *Scraping Tool,* for removal of less adherent deposits. Scraper material of construction needs to be of sufficient hardness as not to contaminate the deposit sample.

10.7 *Vise,* sometimes is used to crimp tube to remove brittle deposits.

10.8 *Vibrating Tool,* to remove more adherent deposits. A small head should be available for use within pits.

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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.

10.9 *Oven,* for drying deposits.

10.10 *Analytical Balance,* for weighing deposits.

11. Procedure

11.1 Split the tube sample taken in Section 6 in halves longitudinally, separating the hot side from the cold side if the distinction is apparent from the external surface or from labeling. Be careful to disturb the internal deposit as little as possible. Carefully remove all cutting fragments from the deposit with the magnet.

11.2 Examine and photograph the internal surfaces, if desired. Determine the area to be tested and mark off the boundaries. This will usually be the area of heaviest deposit and will be on the hot side section of the tube. A similar area can be marked off and tested on the cold side section for comparison, if desired.

11.3 Carefully, scrape the surface to dislodge and individually collect the more easily removable deposits from the selected area. Complete the deposit removal by brushing or applying an electric vibrating tool, or both. Dry the removed material in an oven at 105°C for 1 hour if it appears to be hydrated. It is optional to grind the removed deposit sufficiently to pass through a No. 325 (45-µm) stainless steel sieve and weigh the screened portion. Record the weight in milligrams.

11.4 Determine the area from which the deposit was removed, measuring each dimension to the nearest 1 mm. If the area has a regular shape, direct measurement can be used. If the area has an irregular shape, one technique used to determine the area is to trim paper to the pattern of the actual surface that had deposit removed. Then determine the area by comparing the weight of the irregular paper pattern to the weight of a paper of known area.

12. Calculation

12.1 Determine the weight of accumulated deposits per unit area, by dividing the removed deposit weight by the measured area.

13. Report

13.1 The results are usually reported as g/ft^2.

14. Precision and Bias

14.1 Practice D2777 is not applicable to this test method because a known and repeatable standard reference deposit contained within a steam generator tube cannot be produced.

14.2 Factors that produce errors in mass loss measurement include improper balance calibration, loss of removed deposition prior to weighing, incomplete deposition removal, and removal of base metal. Generally, modern analytical balances if calibrated and used correctly are not considered to be a significant source of error. The other factors are considered more significant.

14.3 The determination of the sample area is likely the least precise step with this method, particularly for riffled tubes.

14.4 This method was tested by five laboratories. These collaborative test data were obtained on sections of boiler tube samples from two specific boilers. For other boiler tube samples, these data may not apply.

14.4.1 *Precision*—An example of the precision obtained on these specific boiler tubes is shown in Fig. 1.

14.4.2 *Bias*—Bias data could not be determined because of the lack of a boiler tube containing a known and consistent deposit.

14.5 Data for this estimated procedure variance is from NACE International Publication 7H100, Item No. 24206.

15. Quality Control (QC)

15.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test:

15.2 A method has been shown to determine the optimum length of time or other variable factors concerning the cleaning procedure, which indicates the optimum point where deposition has been removed but base metal has not been removed, as shown in Practice G1.

15.3 *Calibration and Calibration Verification:*

15.3.1 *Analytical Balance*—Follow the balance manufacturer's recommendations for calibration procedure and frequency. Check the balance with a class S weight in the range of the expected deposit sample weight on the day of use. Recalibrate, as needed.

16. Keywords

16.1 boiler; deposit; deposition; rifled; steam generator tube

TEST METHOD B—CHEMICAL REMOVAL BY SOLVENT

17. Scope

17.1 This test method covers the determination of accumulated deposition in a steam generator tube by chemical removal of the deposit and measuring the weight change in the deposit. This solvent containing the deposit is then available for further chemical analysis, if desired. The method does not allow for discretionary removal of the deposit from the tube in areas of the most interest.

18. Summary of Test Method

18.1 The deposit that has accumulated in the selected boiler tube specimen is determined by measuring the weight loss of the tube sample after deposit removal with inhibited hydrochloric acid. In the event copper plates out on the tube sample during the cleaning operation, an ammonium persulfate solution is used to remove the copper prior to final weighing. The required machining of the outside surface of the selected tube sample to a wall thickness of approximately 1 mm (1⁄16 in.) will increase the sensitivity of the measurement and eliminate interference from external deposits.

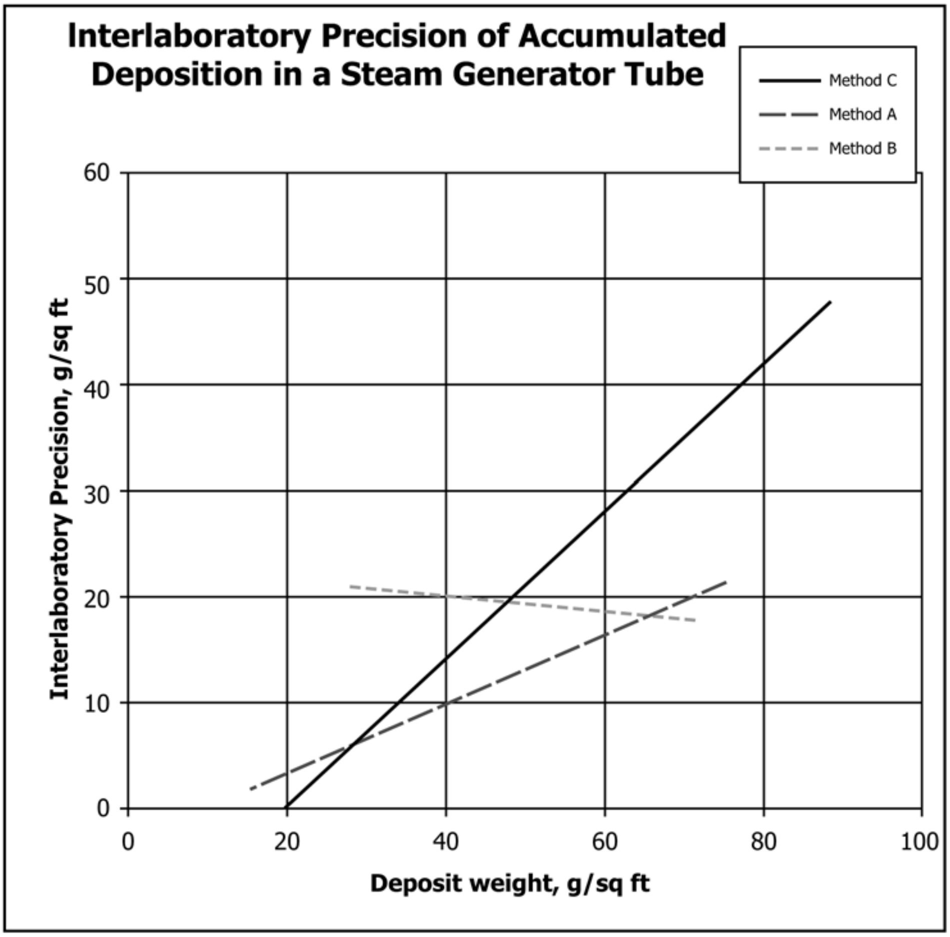

FIG. 1 Interlaboratory Precision of Accumulated Deposition in a Steam Generator Tube

19. Interferences

19.1 Loss of base metal by the solvent, incomplete cleaning of external surface followed by loss of external material, incomplete removal of deposit.

20. Apparatus

20.1 *Cutting Tool or Torch,* removing a suitable portion of boiler tube and a vise for crimping.

20.2 *Tube Cutter.*

20.3 *Tube End Sealers,* to protect the sample if the determination is to be made elsewhere than on the site.

20.4 *Milling Machine (Preferred) or Band Saw,* to separate the fireside half of the tube from the shielded half by longitudinal sectioning (dry cut).

20.5 *Magnet,* to remove metal chips from the deposited material, especially if a band saw is used.

20.6 *Oven,* for drying the deposits.

20.7 *Analytical Balance.*

20.8 *Machine Lathe,* to turn down the outer surface of the tube sample.

20.9 *Hot Plate with Magnetic Stirrer.*

20.10 *Plastic Coated Stirring Bar.*

20.11 *Heat Resistant Glass or Plastic Beaker,* 1000-mL.

21. Reagents

21.1 *Acetone.*

21.2 *Ammonium Persulfate Solution (10 g/L)*—Dissolve 1 g of ammonium persulfate ($(NH_4)_2S_2O_8$) in 50 mL of water and add 50 mL of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90).

21.3 *Inhibited Hydrochloric Acid Solution (1 + 9)*—Add 100 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 900 mL of water. Add 2 mL of appropriate inhibitor and mix well.

21.4 *Sodium Carbonate Solution (10 g/L)*—Dissolve 10 g of sodium carbonate (Na_2CO_3) in 1 L of water.

22. Procedure

22.1 Visually select the section that appears to be most heavily encrusted, assisted by suitable illumination. Select the 50-mm (2-in.) specimen from this section on which the weight of accumulated deposit per unit area will be determined.

22.2 Using the tubing cutter, isolate an approximately 150-mm (6-in.) length that will include the 50-mm (2-in.) specimen. Center the short piece in a lathe and, after marking the end with a file to show the center of the fireside, decrease the outside diameter of the tube over a length that includes the 50-mm (approximately 2-in.) specimen. When the wall thickness has been decreased to about 1.5 mm (1⁄16 in.), remark the fireside, if necessary, and separate this 50-mm ring or shell by applying a suitable parting tool. Then by using a milling machine (preferred) or a band saw, isolate the fireside half of this shell from the casing half and carefully remove any adhering chips. Dry the tube specimen in an oven at 105°C for 1 h, if necessary.

22.3 Weigh the tube sample to the nearest 0.1 mg.

22.4 Remove the deposit in inhibited acid solution.

22.5 Immerse the specimen in a 1000-mL beaker containing 750 mL of inhibited hydrochloric acid solution (1 + 9) which has previously been brought to 70°C (160°F) on a hot plate with a plastic-coated magnetic stirrer. Keep the specimen immersed until the deposit is removed as evaluated visually. Maintain the circulation of the solvent solution and keep the solvent temperature within ±2°C throughout the test.

22.6 Remove, rinse, dry, and weigh the specimen in the following manner: Remove the specimen from the inhibited acid cleaning solution, wash in warm water, neutralize in the Na_2CO_3 solution and immerse in the $(NH_4)_2S_2O_8$ solution if copper plating is present until the plating is removed. Rinse the specimen in hot water for a length of time sufficient to heat it; then dry with acetone. Weigh the dry and cleaned specimen.

22.7 Subject the clean specimen to a second 10-min treatment in the same cleaning solution, and again remove, rinse, dry, and weigh. The sample weight found should be identical within 2 mg of that recorded in 16.6, if the inhibitor is effective. If the difference is greater, it may be applied as a rough factor to correct for base metal corrosion (added to the first clean and dry weight). Base metal corrosion is generally less than 0.1 mg/cm^2.

22.8 Determine the internal surface area of each specimen (use a paper pattern of the cleaned surface, if necessary) and record the area in square millimeters.

23. Calculation

23.1 Determine the weight of accumulated deposits per unit area, by dividing the removed deposit weight by the measured area.

24. Report

24.1 The results are usually reported as g/ft^2.

25. Precision and Bias

25.1 Practice D2777 is not applicable to this test method because a known and repeatable standard reference deposit contained within a steam generator tube cannot be produced.

25.2 Factors that produce errors in mass loss measurement include improper balance calibration, incomplete cleaning of the tube exterior, incomplete deposition removal, and removal of base metal. Generally, modern analytical balances if calibrated and used correctly are not considered to be a significant source of error. The other factors are considered more significant.

25.3 The determination of the sample area is likely the least precise step with this method, particularly for riffled tubes.

25.4 This method was tested by seven laboratories. These collaborative test data were obtained on sections of boiler tube samples from two specific boilers. For other boiler tube samples, these data may not apply.

25.4.1 *Precision*—An example of the precision obtained on these specific boiler tubes is shown in Fig. 1.

25.4.2 *Bias*—Bias data could not be determined because of the lack of a boiler tube containing a known and consistent deposit.

25.5 Data for this estimated procedure variance is from NACE International Publication 7H100, Item No. 24206.

26. Quality Control (QC)

26.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test:

26.2 A method has been shown to determine the optimum length of time or other variable factors concerning the cleaning procedure, which indicates the optimum point where deposition has been removed but base metal has not been removed, as shown in Practice G1.

26.3 *Calibration and Calibration Verification:*

26.3.1 *Analytical Balance*—Follow the balance manufacturer's recommendations for calibration procedure and frequency. Check the balance with a class S weight in the range of the expected deposit sample weight on the day of use. Recalibrate, as needed.

27. Keywords

27.1 boiler; deposit; deposition; rifled; steam generator tube

TEST METHOD C—MECHANICAL REMOVAL BY GLASS BEAD BLASTING

28. Scope

28.1 This test method covers the determination of accumulated deposition in a steam generator tube by the mechanical removal of the deposit by glass-bead blasting. The method does not provide a sample of the deposit suitable for chemical analysis after the removal process. The method does not allow for discretion in selecting the area to be cleaned.

29. Summary of Test Method

29.1 The tube is split and the internal deposits are removed by glass-bead blasting. The weight change of the tube specimen is determined before and after the deposit is removed. The cleaned internal surface area is determined and the result is reported as grams of deposit per square foot of boiler tube surface.

30. Interferences

30.1 Removal of weld spatter or cutting debris from the tube removal, cutting or sectioning process.

30.2 Loss of external material from the tube due to not cleaning the external surface.

30.3 Continued reuse of glass beads can cause them to be contaminated with scale particles, which can make a more aggressive abrasive. Alternatively, the glass beads eventually wear down to a dust. The beads need to be inspected and changed as needed.

30.4 Tenacious scale such as silica based scale or high temperature metal oxides formed due to tube overheating will require longer blasting times and have risk of base metal loss. An alternative removal procedure is best for tenacious scales.

31. Apparatus

31.1 *Commercial Glass-Bead Blasting Cabinet,* shall be used. Typical operating requirements are compressed air at 690 kPa (100 psig) with a minimum of 0.34 cubic meters/min (12 cubic feet/min) flow rate.

31.2 *Medium Size Glass Beads,* 150 to 250 micron diameter range (60 to 100 mesh size).

31.3 *Cutting Tool or Torch,* for removing a suitable portion of the boiler tube.

31.4 *Tube Cutter.*

31.5 *Tube End Sealers,* to protect the sample until the determination can be made.

31.6 *Milling Machine or Band Saw,* to separate the tube into halves by longitudinal sectioning (dry cut).

31.7 *Analytical Balance,* accurate to a minimum of 0.01 grams in the range of 200-600 gram or larger capacity.

31.8 *Small Flexible Ruler or Measuring Tape,* with graduations of 1.0 mm.

32. Procedure

32.1 Cut a section, typically a 3-in. long specimen of interest, out of the tube sample.

32.2 For tubes with only one side facing the heat zone, identify the hot and cold side. Split the tube along this line. The hot side will typically have the higher deposit. If no hot side can be identified but one side appears to have a heavier internal deposit, split to include this area. Select the heaviest deposited section to be tested.

32.3 If the external surface of the tube section to be tested has loose deposit, clean the external surface first using the glass-bead blaster.

32.4 Weigh the tube specimen to be tested to the nearest 0.01 grams.

32.5 The internal surface is then cleaned completely with the glass-bead blaster. The typical pressure setting is 550 kPa (80 psig) while using the appropriate gun nozzle tip giving sufficient airflow. The blasting duration is typically about 30 seconds. The surface is blasted clean until completely free of deposit, down to a shiny metal condition. Blasting times exceeding one minute should not be necessary.

32.6 Tests on clean tube sections can be performed to judge the equipment setup and bead condition for a given cleaning time to determine the extent of base metal loss.

32.7 After blasting of the specimen is complete, reweigh to the nearest 0.01 grams.

32.8 Determine the area from which the deposit was removed, measuring each dimension to the nearest 1 mm. If the area has a regular shape, direct measurement can be used. If the area has an irregular shape, one technique used to determine the area is to trim paper to the pattern of the actual surface that had deposit removed. Then determine the area by comparing the weight of the irregular paper pattern to the weight of a paper of known area.

33. Calculation

33.1 Determine the weight of accumulated deposits per unit area, by dividing the removed deposit weight by the measured area.

34. Report

34.1 The results are usually reported as g/ft^2.

35. Precision and Bias

35.1 Practice D2777 is not applicable to this test method because a known and repeatable standard reference deposit contained within a steam generator tube cannot be produced.

35.2 Factors that produce errors in mass loss measurement include improper balance calibration, incomplete deposition removal, and removal of base metal. Generally, modern analytical balances if calibrated and used correctly are not considered to be a significant source of error. The other factors are considered more significant.

35.3 The determination of the sample area is likely the least precise step with this method, particularly for riffled tubes.

35.4 Inconsistencies in the bead blast duration, nozzle tip distance, and angle can produce errors.

35.5 This method was tested by thirteen laboratories. These collaborative test data were obtained on sections of boiler tube samples from two specific boilers. For other boiler tube samples, these data may not apply.

35.5.1 *Precision*—An example of the precision obtained on these specific boiler tubes is shown in Fig. 1.

35.5.2 *Bias*—Bias data could not be determined because of the lack of a boiler tube containing a known and consistent deposit.

35.6 Data for this estimated procedure variance is from NACE International Publication 7H100, Item No. 24206.

36. Quality Control (QC)

36.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test:

36.2 A method has been shown to determine the optimum length of time or other variable factors concerning the cleaning procedure, which indicates the optimum point where deposition has been removed but base metal has not been removed, as shown in Practice G1.

36.3 *Calibration and Calibration Verification:*

36.3.1 *Analytical Balance*—Follow the balance manufacturer's recommendations for calibration procedure and frequency. Check the balance with a class S weight in the range of the expected deposit sample weight on the day of use. Recalibrate, as needed.

37. Keywords

37.1 boiler; deposit; deposition; glass bead; rifled; steam generator tube

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