Designation: D 4743 – 92 (Reapproved 1996)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Method for Efficacy of Solvent Systems for Dissolving Water-Formed Deposits[1]

This standard is issued under the fixed designation D 4743; 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 test method covers the determination of the efficacy of solvent systems for dissolving either water-formed deposits that have been removed from the underlying material or synthetically prepared deposits.

1.2 The nature of this test method is such that statements of precision and bias as determined by round robin tests could mislead users of the test method (see Sections 11 and 12). Therefore, no precision and bias statements are made.

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:*

D 887 Practices for Sampling Water-Formed Deposits[2]
D 1129 Terminology Relating to Water[3]
D 1193 Specification for Reagent Water[3]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[3]
D 2790 Methods of Analysis of Solvent Systems Used for Removal of Water-Formed Deposits[4]
D 3263 Test Methods for Corrosivity of Solvent Systems for Removing Water-Formed Deposits[2]
D 3483 Test Methods for Accumulated Deposition in a Steam Generator Tube[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *water-formed deposits (Note 1)*—any accumulation of insoluble material derived from water or formed by the reaction of water upon the surface in contact with the water.

NOTE 1—Deposits formed from or by water in all its phases may be further classified as scale, sludge, corrosion products, or biological deposits. 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 microscopical or biological methods.

3.1.2 For definitions of other terms used in this test method, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *solvent system*—specified chemicals or combinations of chemicals that may include corrosion inhibitors, formulated to react with and remove deposits.

3.2.2 *single and multiple solvent systems*—a single solvent system is a one-solution treatment. A multiple solvent system is a treatment using two or more solutions in sequence.

4. Summary of Test Method

4.1 This test method determines the ability of solvent systems to dissolve deposits.

4.2 This test method consists of exposing weighed amounts of deposits to solvent systems and determining the weight loss of the exposed deposit, thereby determining the efficacy of the solvent system.

5. Significance and Use

5.1 This test method is useful because the choice of a solvent system for removing water-formed deposits depends primarily upon the ability of the solvent system to dissolve the deposit. It is used for evaluating solvent systems on water-formed deposits that have been removed from the underlying material.

6. Apparatus

6.1 *Balance*—Capable of weighing to the nearest 0.1 mg.

6.2 *Constant Temperature Bath*—Thermostatically controlled to ±1°C (±2°F) and provided with suitable openings and supports for the test containers.

6.3 *Test Containers*—Tall form, 220-mL, lipless, polypropylene beakers. They shall be fitted with tight covers. A small slit (approximately 13 mm (½ in.) in length) shall be made in each cover.

6.4 *Glass Filter Crucibles*—Gooch 30-mL low form coarse fritted disk crucibles.

6.5 *Vacuum Oven*, for drying residual deposits.

6.6 *Desiccator*.

6.7 *Mild Steel Coupon*—A clean, mild steel coupon, nominally 25 by 50 by 1.5 mm (1 by 2 by 1⁄16 in.) used for

---

[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Surveillance of Water, and Flow Measurement of Water.

Current edition approved Oct. 15, 1992. Published December 1992. Originally published as D 4743 – 87. Last previous edition D 4743 – 87.

[2] *Annual Book of ASTM Standards*, Vol 11.02.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] Discontinued; see 1993 *Annual Book of ASTM Standards*, Vol 11.02.

chelant-based solvent studies.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All solvent materials such as acids, inhibitors, and other additives shall be of the grade normally employed in chemical cleaning practices for the removal of water-formed deposits. Unless otherwise indicated, it is intended that all chemicals shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] 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.

7.2 *Purity of Water*—Unless otherwise indicated references to water shall be understood to mean Type II reagent water conforming to Specification D 1193.

8. Sampling

8.1 Collect and preserve the sample in accordance with Practices D 887.

NOTE 2—If Test Methods D 3483 are being used, the scale removed in that test can be used in this test method.

9. Procedure

9.1 Prepare a water solution of the solvent systems to be tested using a weight percentage basis for the acid or other active material including any additives. Only inhibitors supplied in liquid form shall be added on a volume basis as prescribed by the manufacturer.

9.2 *Preparation of Sample*:

9.2.1 *Preliminary Examination*—Examine the sample as collected, using a microscope if available, for structure, color, odor, oil matter, appearance of mother liquor if any, and other characteristics of note (for example, attraction to magnet). Record results for future reference.

9.2.1.1 Filtration and other steps in the preparation of the sample may frequently be bypassed; for example, a moist sample that contains no separated water or an apparent dry sample shall be started in accordance with 9.2.3.1, and a dry sample shall be started in accordance with 9.2.4.

9.2.2 *Filtration of Sample*—If the sample includes an appreciable quantity of separated water, remove the solid material by filtration. Save the filtrate, undiluted, pending decision as to whether or not its chemical examination is required. Transfer all of the solid portion to the filter, using the filtrate to rinse the sample container if necessary. Air-drying or partial air-drying of the filter is frequently helpful toward effecting a clean separation of the deposit.

9.2.3 *Sample Drying*—Remove the drained solid sample from the filter, being careful to avoid gross contamination with filter paper. Spread the entire quantity of sample in a thin layer on a nonreactive, impervious surface.

9.2.3.1 *Oven Drying*—Dry the sample using a vacuum oven at a temperature that will not degrade the deposit (Note 3). Place the sample in a desiccator and allow it to cool to room temperature. Weigh the sample to the nearest 0.01 g. Repeat until a constant weight is obtained. A record of the weight loss during drying is often used.

NOTE 3—A common choice is 52°C (125°F) and 34 kPa (absolute) (20 in. of mercury).

9.2.3.2 *Air Drying*—Air dry the entire quantity of solid, spread it in a thin layer on a nonreactive impervious surface. A record of the loss of weight during air drying is often used.

9.2.4 Choose a representative portion of the dried sample for testing.

9.3 The weight of sample to be tested and the volume of solvent to be used shall be chosen based on the best estimate of actual ratio of deposit weight to volume in the equipment to be cleaned.

NOTE 4—A common choice is 1 g of deposit and 100 mL of solvent.

9.3.1 Some solvent systems, primarily chelant based, perform better when in contact with a steel surface. For those solvent systems it is necessary to add a mild steel coupon (8.7).

9.4 *Single Solvent Systems*:

9.4.1 Pour the chosen volume (9.3) of the test solvent into a test container (6.3) and place it in the constant temperature bath (6.2) that has been preheated to the desired test temperature. Cover the containers to prevent excessive evaporation and loss of volatile solvent components.

9.4.1.1 If a mild steel coupon (9.3.1) is necessary for the test, add it to the solvent before adding the deposit (9.4.3).

9.4.2 Take a portion (9.3) of the deposit, weigh it to the nearest 0.01 g, and record an original deposit weight.

9.4.3 When the solvent temperature has reached that of the bath, add the weighed portion (9.4.2) of the deposit. Let the sample stand undisturbed.

9.4.4 Weigh a clean, dry filter crucible (6.4) to the nearest 0.01 g. Record the weight as original crucible weight.

9.4.5 At the conclusion of the exposure period, as determined by visual examination (usually 6 h), filter the solvent-deposit mixture through the weighed filter crucible (9.4.4). Vacuum filtration increases the filtration rate.

NOTE 5—Analysis of the spent solvent using Methods D 2790 can yield information about the performance of the solvent system.

9.4.6 Rinse the filter crucible and residual deposit with water.

9.4.7 Dry the crucible and residual deposit in the vacuum oven. Choose a temperature and vacuum that will not degrade the residual deposit.

NOTE 6—A common choice is 52°C (125°F) and 34 kPa (absolute) (20 in. of mercury).

9.4.8 Place the dried crucible and residue in the desiccator and allow them to cool to room temperature.

9.4.9 Weigh the crucible plus residue to the nearest 0.01 g.

9.4.10 Repeat 9.4.7 to 9.4.9 until a constant weight is obtained. Record this weight as the final crucible weight.

9.5 *Multiple Solvent Systems*:

9.5.1 Perform the first solvent exposure by following the

---

[5] *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.

procedures given in 9.4.1 through 9.4.4.

9.5.2 Heat the chosen amount of the next solvent to the desired test temperature.

NOTE 7—A common choice is to use water heated to the temperature of the subsequent step but not exceeding 66°C (150°F).

9.5.3 Decant or siphon the previous solvent from the test container, leaving the residual deposit.

9.5.3.1 Rinse the residual deposit with water decanting or siphoning off the water leaving the original deposit.

9.5.4 Pour the preheated solvent into the test container and place in the constant temperature bath that has been preheated to the desired test temperature.

9.5.5 If another solvent exposure is required, then at the conclusion of the exposure period repeat 9.5.2 through 9.5.5. Otherwise, complete the test by performing 9.4.5 through 9.4.10.

10. Calculation

10.1 Determine the amount of deposit that was not dissolved by subtracting the original crucible weight (9.4.4) from the final crucible weight (9.4.10).

10.2 Determine the amount of deposit that was dissolved by subtracting the amount not dissolved (10.1) from the original deposit weight (9.4.2).

10.3 The efficacy of the solvent system is expressed as the percent deposit dissolved, which is determined by dividing the amount dissolved by the original amount and converting the resultant decimal to a percentage.

10.4 Calculate solvent efficacy as follows:

$$SE = \frac{OD - (FC - OC)}{OD} \times 100$$

where:

SE = solvent efficacy,

OD = original deposit weight,

FC = final crucible weight, and

OC = original crucible weight.

11. Interpretation of Results

11.1 The results of this test method can be used to determine if a solvent should be effective in removing a deposit.

11.2 The actual choice of a solvent system to remove the deposit depends not only on the efficacy (11.1) but on many other factors as well. Some of these factors are:

11.2.1 The compatibility of the solvent with the alloys contained in the system (see Test Methods D 3263),

11.2.2 The water requirements of the solvent system,

11.2.3 The temperature requirements of the solvent system,

11.2.4 The disposal restrictions for the solvent system,

11.2.5 The mechanical requirements to apply the solvent system, and

11.2.6 Other considerations such as relative cost.

12. Precision and Bias

12.1 Because of the many variables involved in the selection of a solvent system (see 11.2), a statement regarding precision and bias would be meaningless and may even be misleading. The user is cautioned to select test conditions as close as possible to the actual system conditions.

12.2 The Results Advisor and Technical Operation Section of the Committee D-19 Executive Subcommittee concur that this precision statement meets the requirements of Practice D 2777.

13. Keywords

13.1 deposit; efficacy; removal; scale; solvent; static; systems; waterformed