



# Standard Test Method for Relative Efficacy of Dynamic Solvent Systems for Dissolving Water-Formed Deposits<sup>1</sup>

This standard is issued under the fixed designation D5256; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of the relative efficacy of dynamic solvent systems for dissolving water-formed deposits that have been removed from the underlying material or deposits attached to the underlying material.

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 this test method (see Sections 11 and 12). Therefore, no precision and bias statements are made.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound 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.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[D887 Practices for Sampling Water-Formed Deposits](#)

[D1129 Terminology Relating to Water](#)

[D1193 Specification for Reagent Water](#)

[D2331 Practices for Preparation and Preliminary Testing of Water-Formed Deposits](#)

[D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)

[D3263 Test Methods for Corrosivity of Solvent Systems for](#)

[Removing Water-Formed Deposits \(Withdrawn 2008\)](#)<sup>3</sup>  
[D3483 Test Methods for Accumulated Deposition in a Steam Generator Tube](#)

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of other terms used in this test method, refer to Terminology [D1129](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dynamic solvent system, n*—any closed loop system in which the solvent is in motion across the deposit surface.

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

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

3.2.4 *water-formed deposits, n*—any accumulation of insoluble material derived from water or formed by the reaction of water upon the surface in contact with the water.

3.2.4.1 *Discussion*—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.

## 4. Summary of Test Method

4.1 This test method consists of determining the ability of a dynamic solvent system to dissolve deposits.

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

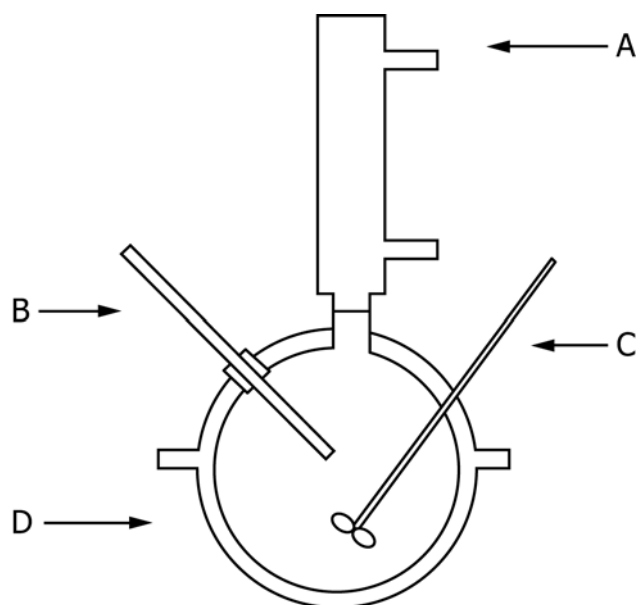
4.3 For the attached deposits, the amount and time required to yield a constant amount of certain loss of interest and the

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee [D19](#) on Water and is the direct responsibility of Subcommittee [D19.03](#) on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

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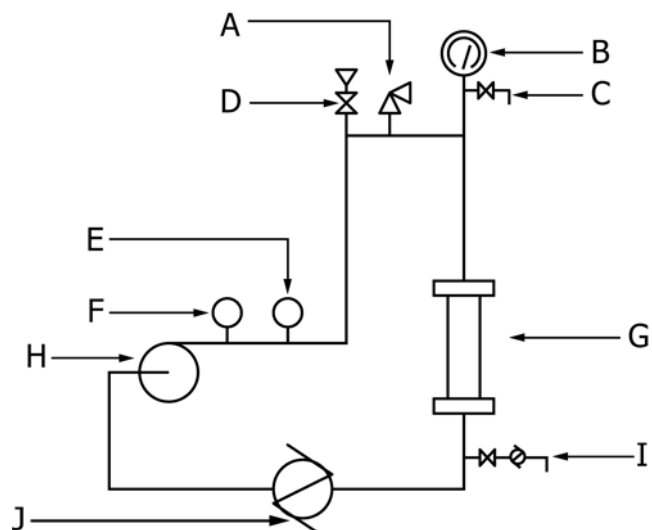
<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).



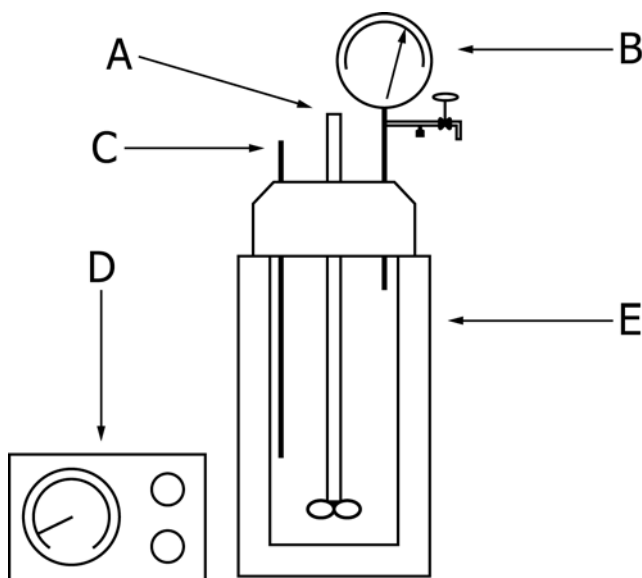
A: Condenser, B: Temperature Measurement, C: Stirrer, D: Heated Reaction Flask

FIG. 1 Low Temperature Test Equipment



A: High Point Pressure Relief, B: Pressure Gauge, C: Vent, D: Solvent Addition Valve, E: Temperature Measurement, F: Flow Measurement, G: Tube Sample Holder, H: Variable Speed Pump, I: Sample Valve With Integral Cooler, J: Regulated Heat Source

FIG. 3 Dynamic Test Equipment



A: Stirrer, B: Pressure Control (Gauge, Vent and Relief), C: Temperature Measurement, D: Temperature Control, E: Heated Autoclave

FIG. 2 High Temperature Test Equipment

amount of deposit not removed determines the relative efficacy of the dynamic 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 upon the ability of the dynamic solvent system to dissolve both unattached and attached deposits.

## 6. Apparatus

### 6.1 Common Equipment:

NOTE 1—The equipment listed in this section is basic and serves the function of this test method. This basic test procedure could be modified to meet the specific needs of a particular investigation. The test apparatus, however, must be identified and reported with the results. For comparative type tests, as described in the procedure, it is important that all tests be run in an identical manner.

### 6.2 Unattached Deposit Removal (see Fig. 1 and Fig. 2):

- 6.2.1 Balance, capable of weighing to the nearest 0.1 mg.
- 6.2.2 Heating Bath, thermostatically controlled to  $\pm 1^\circ\text{C}$ .
- 6.2.3 Stirrer, controlled agitation.
- 6.2.4 Temperature Indicator.

6.2.5 Reaction Flask, stirred tests are best accomplished in round bottom flasks.

### 6.2.6 Condenser.

6.2.7 Glass Filter Crucibles, Gooch 30-mL low form coarse fritted disc crucibles.

### 6.2.8 Vacuum Oven, for drying residual deposits.

### 6.2.9 Desiccator.

### 6.3 Attached Deposit Removal (see Fig. 3):

- 6.3.1 Solvent Heater/Solvent Reservoir.
- 6.3.2 Sample Holder.
- 6.3.3 Flow Controller.
- 6.3.4 Sample System (usually with cooler).
- 6.3.5 Pressure Relief.
- 6.3.6 Pump.
- 6.3.7 Flow Meter.
- 6.3.8 Temperature Controller.

## 7. Reagents

7.1 Purity of Reagents—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,<sup>4</sup> where such specifications are available.

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

## 8. Sampling

8.1 Collect and preserve the sample in accordance with Practices **D887** (see **Note 2**).

NOTE 2—If Method A of Test Methods **D3483** is being used, the deposit removed in that test can be used in this test method.

8.2 Collect and preserve the sample(s) obtained during the cleaning of attached deposit sample.

## 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. Add only inhibitors supplied in liquid form on a volume basis as prescribed by the manufacturer.

### 9.2 *Unattached Deposits:*

#### 9.2.1 *Preparation of Sample:*

9.2.1.1 *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 the results for future reference. Further information about examination and documentation of the nature of deposits can be found in Practices **D2331**.

(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 **9.2.1.3(I)**, and a dry sample shall be started in accordance with **9.2.1.4**.

9.2.1.2 *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 sample 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.

9.2.1.3 *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.

(1) *Oven Drying*—Dry the sample using a vacuum oven at a temperature that will not degrade the deposit. A common choice is 52°C (125°F) and 34 kPa (absolute) (20 in. of

mercury). 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 constant weight is obtained. A record of the weight loss during drying is often used.

(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.

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

9.2.2 Pour a measured volume of the test solvent (see **9.1**) into the chosen test apparatus (see Section **6**).

9.2.2.1 The weighed deposit can be added to the solvent prior to heating the solvent; however, a controlled and reported temperature must be maintained so that the results between tests can be readily compared.

9.2.3 Heat the solvent to the test temperature while stirring at the specified RPM (see **Note 3**).

NOTE 3—A stir rate of 60 to 90 RPM is commonly used.

9.2.4 Take a portion of the deposit (see **9.2.1**), weigh to the nearest 0.01 g and record an original deposit weight. The deposit weight should meet a solvent volume/deposit weight ratio for this specified application (see **Note 4**).

NOTE 4—A solvent volume/deposit weight ratio of 100 mL solvent/1 g deposit is commonly used.

9.2.5 After the solvent has stabilized at the test temperature, draw a sample for analysis, then add the deposit to the solvent. This is considered as “time zero.”

9.2.6 If required, samples can be drawn at periodic time intervals. Maintain strict control of all sample weights (to nearest 0.01 g).

9.2.7 At the termination of the test, cool and filter the solvent.

9.2.7.1 Weigh a clean, dry filter crucible to the nearest 0.01 g. Record the weight as original crucible weight.

9.2.7.2 Filter the solvent-deposit mixture through the weighed filter crucible. Vacuum filtration increases the filtration rate.

9.2.7.3 Rinse the filter crucible and residual deposit with water.

9.2.7.4 Dry the crucible and residual deposit in the vacuum oven. Choose a temperature and vacuum that will not degrade the residual deposit. A common choice is 52°C (125°F) and 34 kPa (absolute) (20 in. of mercury).

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

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

9.2.7.7 Repeat **9.2.7.4** to **9.2.7.6** until a constant weight is obtained. Record this weight as the final crucible weight.

### 9.3 *Attached Deposit (Comparative Tests):*

9.3.1 Establish accumulated deposition in accordance with Test Methods **D3483** on a portion of the sample adjacent to and representative of the section to be tested below. Further information about examination and documentation of the nature of deposits can be found in Practices **D2331**.

9.3.2 The ratio of the weight of attached deposit on the test specimen to test apparatus volume must be recorded and should be appropriate for the application (see **Note 5**).

<sup>4</sup> *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 *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

NOTE 5—Due to the constraints imposed by the solvent circulation system, the solvent volume/deposit weight ratio on attached deposits is often larger than desired. This excess capacity can be partially compensated by reducing the concentration of the active components of the solvent. However, in all cases the actual cleaning operation should be designed and controlled according to the accepted practice for the particular solvent system being applied and not according to the values generated by this test method.

9.3.3 Use a measured volume of test solvent to fill the test apparatus.

9.3.4 Establish solvent flow in the test apparatus. The solvent flow should bypass the test specimen until the proper flow rate, system pressure, and solvent temperature is reached.

9.3.4.1 Establish and record the solvent flow rate based on the cross section area of the test specimen.

9.3.5 Heat the solvent to the appropriate test temperature. After the solvent temperature and flow rate have stabilized, draw a sample for analysis.

9.3.6 Establish flow through the test specimen. This is considered as “time zero.”

9.3.7 Monitor and record flow rate, pressure and temperature during the test interval.

9.3.8 Draw samples at periodic time intervals and analyze for ions of choices.

9.3.9 If necessary, use intermediate visual examinations to determine cleaning efficacy.

9.3.10 At the completion of the test period, determined by 9.3.8 and 9.3.9, determine the remaining deposit accumulation according to Test Methods D3483 (see Note 6). This value as well as the time required to achieve the level of cleanliness is used as the relative measure of cleaning efficacy for attached deposit.

NOTE 6—Since the remaining deposit may be acid insoluble, the preferred method is Method A of Test Methods D3483.

## 10. Calculation

### 10.1 Unattached Deposits:

10.1.1 Determine the amount of deposit that was dissolved by subtracting the amount not dissolved (see 9.2.7) from the original deposit weight (see 9.2.4).

10.1.2 Express the efficacy of the solvent system as the percent deposit dissolved, by dividing the amount dissolved (see 10.1.1) by the original amount (see 9.2.4) and converting the resultant decimal to a percentage.

10.1.3 Use the following formula to calculate solvent efficacy:

$$SE = \frac{OS - (FS)}{OS} \times 100 \quad (1)$$

where:

*SE* = solvent efficacy,  
*OS* = original deposit weight, g, and  
*FS* = final deposit residue weight, g.

### 10.2 For Attached Deposits:

10.2.1 Determine the amount of deposit that was removed by subtracting the final deposit accumulation (see 9.3.10) from the original deposit accumulation (see 9.3.1).

10.2.2 Express the efficacy of the solvent system as the percent of accumulated deposit removed, by dividing the amount removed (see 10.2.1) by the original amount (see 9.3.1) and converting the resultant decimal to a percentage.

10.2.3 Use the following formula to calculate solvent efficacy:

$$SE = \frac{ODA - (FDA)}{ODA} \times 100 \quad (2)$$

where:

*SE* = solvent efficacy,  
*ODA* = original deposit accumulation, mg/cm<sup>2</sup>, and  
*FDA* = residual deposit accumulation, mg/cm<sup>2</sup>.

## 11. Interpretation of Results

11.1 Use the results of the procedure in this test method to determine if a solvent is be effective in removing the deposit.

11.2 The actual choice of a solvent system to remove the deposit depends not only on the efficacy (see 11.1) but on many other factors as well. Some of these factors are: the compatibility of the solvent with the alloys contained in the system (see Test Methods D3263); the water requirements of the solvent system; the temperature requirements of the solvent system; the disposal restrictions of the solvent system; the mechanical requirements to apply the solvent system; and other considerations such as relative cost.

11.3 The results obtained by these test methods should include the type and method of test, the types and concentrations of the solvents tested, the physical parameters involved in the tests, the calculation of the efficacy of the solvents tested and any observations that might be pertinent to choice of a solvent system. To aid in reporting these parameters a guide to reporting is shown in Fig. 4.

## 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 his actual system conditions.

12.2 The Results Advisor and Technical Operation Section of the Executive Subcommittee concur that the above precision statement meets the requirements of Practice D2777.

## 13. Keywords

13.1 attached; deposit; dynamic; efficacy; removal; scale; solvent; systems; unattached; water formed

Name of Company Performing The Test \_\_\_\_\_

Solvent System \_\_\_\_\_

Deposit ID \_\_\_\_\_

Duration of Test \_\_\_\_\_ (Hrs)

Temperature \_\_\_\_\_ °C \_\_\_\_\_ °F

Surface or Deposit Weight to Solvent Ratios \_\_\_\_\_

Velocity or Stirring Rate \_\_\_\_\_

Efficiency Calculations \_\_\_\_\_

Visual Observations \_\_\_\_\_

\_\_\_\_\_

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**Description of Apparatus**

\_\_\_\_\_

\_\_\_\_\_

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**FIG. 4 Guide to Reporting**

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