



Standard Test Method

Laboratory Screening Test to Determine the Ability of Scale Inhibitors to Prevent the Precipitation of Barium Sulfate or Strontium Sulfate, or Both, from Solution (for Oil and Gas Production Systems)

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NACE International
1440 South Creek Dr.
Houston, Texas 77084-4906
+1 281-228-6200

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Foreword

Mineral scale may be defined as an adherent deposit of predominantly inorganic compounds. A common process leading to scale formation is the precipitation of sparingly soluble salts from oilfield brines. Some oilfield brines contain sufficient sulfate ion in the presence of barium ions or strontium ions, or both, that the potential for forming barium sulfate (BaSO_4) scale or strontium sulfate (SrSO_4) scale, or both, exists. Often the formation of scale results in reduced production and increased maintenance costs. In some locations, naturally occurring radioactive materials have been found to incorporate themselves into the scale. This complication may result in significant health, safety, and liability concerns and increased scale disposal costs.

Removal of scale after it has formed is particularly difficult when BaSO_4 and SrSO_4 are involved. Therefore, oil and gas producers most often use treatment chemicals to inhibit formation of these scales and reduce their tendency to adhere to surfaces. The choice of the best scale inhibitor for a given application often follows a lengthy testing program. The program typically begins with the collection of potentially useful products that are then put through a screening process in the laboratory to determine whether specific products or classes of products perform better than others.

NACE Standard TM0374¹ addresses only the screening of CaSO_4 and CaCO_3 scale inhibitors. By contrast, this standard test method is intended to provide the user with a relative and quantitative measure of the ability of scale inhibitors to prevent (1) the formation and (2) the precipitation of solid BaSO_4 or SrSO_4 , or both, which are necessary and critical stages in scale deposition. This standard is intended for use by skilled laboratory personnel who have previously performed similar tests. The laboratory screening procedure described in this standard may not allow for the simulation of all oilfield system variables. It must be regarded only as a starting point in the evaluation of scale inhibitors. The procedure standardizes the collection of screening test results to facilitate discussion of the results by interested parties. No attempt has been made to define the test brine composition, test temperature, or test duration. Users of this standard must agree on these and other critical parameters to facilitate comparison of test results.

This standard was originally prepared in 1997 by Work Group T-1D-36a, a subgroup of Task Group (TG) T-1D-36, "Scale Inhibitor Evaluation in Oil and Gas Production," a component of Unit Committee T-1D, "Corrosion Monitoring and Control of Corrosion Environments in Petroleum Operations." It was reaffirmed in 2002 by Specific Technology Group (STG) 31, "Oil and Gas Production—Corrosion and Scale Inhibition," and it was revised in 2010 by TG 383, "Scale Inhibitor Evaluation in Oil and Gas Production." This standard is issued by NACE International under the auspices of STG 31.

In NACE standards, the terms *shall*, *must*, *should*, and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual*. The terms *shall* and *must* are used to state a requirement, and are considered mandatory. The term *should* is used to state something good and is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

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Section 1: General

1.1 This standard describes a test method to screen scale inhibitors for their ability to prevent precipitation of BaSO₄ or SrSO₄, or both, from oilfield brines. The test method does not fix such critical variables as test brine composition, test temperature, and test duration. It is the responsibility of the user of this standard to determine the most appropriate choices for these parameters. For example, Pritchard, et al.² address static tests using North Sea brines.

1.2 This standard is intended only for ranking the performance of different scale inhibitors under laboratory conditions. It is not intended to establish field treatment rates. CAUTION: Unique field conditions encountered may alter the performance ranking of the scale inhibitors.

1.3 Factors such as reaction kinetics, fluid velocity, transient composition, temperature and pressure changes, scale adherence, and solids dispersion can significantly affect scale deposition under field conditions. Consideration of these parameters is outside the scope of this standard. However, field conditions, field brine composition, and other variables noted above should be considered at some point in the scale inhibitor evaluation prior to final selection for field use.

Section 2: Test Equipment

2.1 Constant-temperature water bath or forced-air oven capable of maintaining the specified test temperature.

2.2 Analytical balance capable of weighing mineral salts and other reagents to one-hundredth of the quantity specified.

2.3 Clean, dust-free clear and colorless glass test bottles with a capacity of 100 to 125 mL. Caps must be capable of a positive seal (i.e., they must not deform so as to release pressure generated within the bottle under the test conditions).

2.3.1 The best practice is to use clean, previously unused bottles for each test.

2.3.2 Test bottles may be reused after cleaning if an adequate and consistent procedure is demonstrated to remove sparingly soluble salts and adsorbed scale inhibitors from the glass surfaces.

2.4 Flasks fitted with dispensers capable of reproducibly delivering 50.0 mL of the test solutions.

2.5 Assorted clear, colorless glass containers for deionized/distilled water, buffer solutions, unfiltered brines, scale inhibitor stock solutions, post-test analytical samples, etc.

2.6 Filter assembly(ies) capable of filtering brines using 0.45 µm cellulose acetate membrane filters.

2.7 A pH meter with an appropriate probe.

2.8 Syringes and 0.2 µm cellulose acetate syringe filters for collecting post-test analytical samples.

Section 3: Reagents

- 3.1 ACS⁽¹⁾ reagent grade³ chemicals (mineral salts and organic buffering agents) to prepare test solutions.
- 3.2 Deionized or distilled water.
- 3.3 Concentrated solutions of the scale inhibitors to be evaluated.

Section 4: Preparation of Test Solutions

4.1 Choose an appropriate composite test brine, and specify the concentrations (mg/L) for all species in the composite brine.

4.1.1 If the purpose of performing the screening test relates to a specific field application, the user should choose a composite brine that simulates the scaling brine expected to occur in the field.

4.1.2 Alternatively, a composite test brine that corresponds to a brine supersaturated in BaSO₄ or SrSO₄, or both, that results from the mixing of a pair of incompatible brines at a chosen mixing ratio and yields the highest supersaturation index or the highest amount of precipitation may be chosen. Supersaturation indexes and potential precipitation amount may be determined from scaling prediction calculations, such as those used by Patton.⁴

4.2 Define two new component brines which, when mixed in equal volumes, result in a composite brine with concentrations equivalent to those specified in Paragraph 4.1. One component brine, Brine A, shall contain all of the polyvalent cations, such as Ba²⁺ and Sr²⁺, and the other component brine, Brine B, shall contain all of the scaling anions, such as SO₄²⁻. The remaining ions, such as Na⁺ and Cl⁻, shall be allotted such that the ionic concentrations in the two component brines are comparable if not equal. REMINDER: When equal volumes of the two component brines are mixed to form the composite test brine, the resulting concentration of some ions (including the scaling ions) in the composite brine is one-half of the concentration in Brine A or Brine B.

4.3 Define another component brine, Brine C, which differs from Brine B only in that NaCl is used to substitute for the salts containing the scaling anions. The NaCl substitution shall result in a Brine C ionic strength equivalent to that of Brine B.⁽²⁾

4.4 CAUTION: Some inhibitors may be incompatible with highly saline brines or with high concentrations of specific cations. A resulting deposit may be mistaken for BaSO₄/SrSO₄ and may remove the inhibitor from the solution. The user of this test method may investigate inhibitor-brine incompatibility by treating the theoretically nonscaling mixtures of Brine A and Brine C with the highest concentration of the inhibitor(s) being evaluated.

4.5 On the day of the test⁽³⁾ prepare sufficient quantities of Brine A, Brine B, and Brine C by weighing out the salts and dissolving them in deionized/distilled water to provide the required ionic concentrations.

⁽¹⁾ American Chemical Society (ACS), 1155 Sixteenth St. NW, Washington, DC 20036.

⁽²⁾ Because only salts containing the scaling anions are being replaced, only their contributions to the solution's ionic strength should be calculated. The milliequivalents (meq) of divalent anions and cations will be replaced with twice as many meq of chloride and sodium, respectively. For example, 200 meq. of potassium and of sulfate (17.43 g K₂SO₄) have approximately the same contribution to solution ionic strength as 200 meq. of sodium and 400 meq. of chloride. Therefore, 18.78 g NaCl would be substituted in Brine C for the 17.43 g K₂SO₄ used in the preparation of Brine B.

⁽³⁾ The component brines may be prepared (Paragraphs 4.5 through 4.7) the previous day for convenience. If this approach is used, overnight refrigeration of the brines should be used to retard the loss of dissolved CO₂. It is the responsibility of the user of this variation to ensure that satisfactory results are obtained.

4.6 Mix sufficient buffer into Brine A so that the composite brine mixtures (A+B, A+C) have the required pH at the test temperatures.⁽⁴⁾ The required quantity of buffer should be determined in advance by mixing, at test temperature, appropriate aliquots of Brine A and Brine B and quickly but carefully adding buffer.

4.7 Filter each component brine through a 0.45 µm cellulose acetate membrane filter.

4.8 Transfer the component brines to labeled flasks fitted with dispensers capable of reproducibly delivering 50.0 mL to the test bottles. Place the flasks into a constant-temperature bath/oven set at the test temperature.

4.9 Prepare a stock solution of each scale inhibitor to be evaluated. Measure the inhibitor quantity gravimetrically. One-percent solutions (mass/volume) are normally suitable.

Section 5: Test Procedure

5.1 Deliver 50.0 mL of component brines into labeled test bottles. Perform all tests at least in duplicate.

5.1.1 Label the test bottles containing Brine C with the designation "Control."

5.1.2 Label the test bottles containing Brine B with no inhibitor dosed with the designation "Blank."

5.2 Dose a range of aliquots of each scale inhibitor to be evaluated into test bottles containing Brine B. The range chosen should be wide enough to demonstrate undertreatment (scale precipitation) and overtreatment (no precipitation) for the scale inhibitors being evaluated.

5.3 Place the test bottles into a constant-temperature bath/oven set at the test temperature. The test temperature chosen should normally be associated with a specific field application.

5.4 When the brines have reached the test temperature, mix Brine A with Brine B or Brine C by pouring Brine A into test bottles containing Brine B or Brine C. Secure the cap tightly, shake the test bottle (10 up-and-down cycles), immediately place the test bottle back into the constant-temperature bath/oven stabilized at the test temperature and start the stop clock ($t = 0$).

5.5 Expose all test bottles at the test temperature for a test duration that reflects the likely residence time of the scale inhibitor in the field application being modeled.

5.5.1 Test durations should not be less than one hour or more than 24 hours.

5.5.2 Some users choose to perform a sufficient number of duplicate tests, including controls and blanks, such that some of the test bottles may be removed from the test early (after, for example, two hours) to obtain a preliminary evaluation, while the remainder continue to be exposed for the full test duration (for example, 24 hours).

5.6 At the end of the specified test duration, remove each test bottle, in turn, from the bath/oven and immediately take a sample of the brine for analysis. Filter each analytical sample without delay through a 0.2 µm syringe filter into a clean glass container and then stabilize the sample in preparation for cation analysis. Some methods of sample stabilization are listed in Appendix A (nonmandatory).

5.6.1 If the sample analysis is performed immediately, sample stabilization may not be necessary.

⁽⁴⁾ Partially neutralized 1.0 molar acetic acid, formic acid, or imidazole may be used to adjust the composite test brine pH to desired values: [acetic acid] = [acetate ion] at pH ≈ 4.8; [formic acid] = [formate ion] at pH ≈ 3.8; [imidazole] = [imidazole-HCl] at pH ≈ 7.0. Other buffers or pH adjustment agents may be used if they are compatible with the composite test brine.

5.6.2 It is the user's responsibility to establish that stabilization is not necessary.

5.7 Analyze all samples to establish the levels of the sparingly soluble cations of interest (Ba²⁺, Sr²⁺, etc.); avoid unnecessary delay in completing the analysis.

5.7.1 Some analytical methods that may be considered are listed in Appendix A.

5.7.2 Analysis of the blanks establishes the "zero inhibition" concentration of the Ba²⁺ ions or Sr²⁺ ions, or both, for the test. Analysis of the controls establishes the maximum possible (100% inhibition) Ba²⁺ values or Sr²⁺ values, or both, for the test. The concentration values obtained for Ba²⁺ or Sr²⁺, or both, are required for the calculation of test results in Section 6.

Section 6: Calculation and Reporting of Test Results

6.1 Calculation of Test Results

For each scale inhibitor concentration, the test results shall be calculated as either percentage inhibition of BaSO₄ or SrSO₄ or percentage retention of soluble Ba²⁺ or Sr²⁺ (M²⁺) as shown in Equation (1) and Equation (2), respectively. The standard deviation shall also be calculated.

$$\% \text{ Inhibition of BaSO}_4 \text{ or SrSO}_4 = \frac{[M^{2+}]_{\text{sample}} - [M^{2+}]_{\text{blank}}}{[M^{2+}]_{\text{control}} - [M^{2+}]_{\text{blank}}} \times 100 \quad (1)$$

$$\% \text{ Retention of Ba}^{2+} \text{ or Sr}^{2+} = \frac{[M^{2+}]_{\text{sample}}}{[M^{2+}]_{\text{control}}} \times 100 \quad (2)$$

6.2 Reporting of Test Results

6.2.1 Test results shall be expressed as percentage inhibition or percentage retention as calculated in Paragraph 6.1, along with scale inhibitor type and concentration.

6.2.2 Ba²⁺ or Sr²⁺, or both, retained in solution shall also be reported in mg/L.

6.2.3 The test results report must include information on test variables—composite test brine composition, test temperature, pH, buffer system and amount of buffer added, test duration, and specific method of M²⁺ analysis used.

References

1. NACE Standard TM0374 (latest revision), "Laboratory Screening Tests to Determine the Ability of Scale Inhibitors to Prevent the Precipitation of Calcium Sulfate and Calcium Carbonate from Solution (for Oil and Gas Production Systems)" (Houston, TX: NACE).
2. A.M. Pritchard, W. Barbour, L. Cowie, A. Hunton, D. Wilson, J.R. Goulding, M. Anstey, "An Evaluation of the Jar Test for BaSO₄ and SrSO₄ Inhibitors," CORROSION/92, paper no. 92018 (Houston, TX: NACE, 1992).
3. *Reagent Chemicals*, ACS Specifications, 10th ed. (Washington, DC: ACS, 2006).
4. C.C. Patton, *Applied Water Technology*, 3rd ed. (Norman, OK: John M. Campbell & Co., 2007), pp. 65-89.

5. A.E. Eaton, L.S. Clasceri, E.W. Rice, A.E. Greenberg, eds., *Standard Methods for the Examination of Water and Wastewater*, 21st ed. (Washington, DC: American Public Health Association, 2005).
6. ASTM⁽⁵⁾ D3651 (latest revision), "Standard Test Method for Barium in Brackish Water, Seawater, and Brines" (West Conshohocken, PA: ASTM).
7. ASTM D3986 (latest revision) "Standard Test Method for Barium in Brines, Seawater, and Brackish Water by Direct-Current Argon Plasma Atomic Emission Spectroscopy" (West Conshohocken, PA: ASTM).
8. ASTM D3352 (latest revision) "Standard Test Method for Strontium Ion in Brackish Water, Seawater, and Brines" (West Conshohocken, PA: ASTM).

Appendix A Sample Stabilization and Analysis (Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

A1 Sample Stabilization

The purpose of sample stabilization is to stop the precipitation of Ba²⁺ and Sr²⁺ sulfates in the analytical samples. Test solutions should not be allowed to cool before stabilization. The following possible stabilization methods have been suggested for consideration by the user, without any representation of the suitability of these methods for stabilizing the user's test brine.

A1.1 Chelant solutions (e.g., tetrapotassium EDTA) have been used for stabilization. The use of chelant may limit the choice of acceptable analytical methods. Stabilized test solutions should contain sufficient alkaline chelant to exhibit a pH > 10. If a chelant is used, it should be confirmed, by spiking experiments or other means, that the chelant does not interfere with the accurate measurement of Ba²⁺ and Sr²⁺.

A1.2 A dilution of test samples with a solution of scale inhibitor has been used to stabilize the test solutions. For this solution, 1,000 to 5,000 mg/L of polyacrylate or sulfonated copolymer inhibitor may be used. Calcium tolerance of the inhibitor at the concentration used should be confirmed.

A1.3 A high degree of dilution with deionized/distilled water may be sufficient to stabilize some test solutions. This technique may be impractical for solutions high in barium ion. The suitability of this method should be thoroughly evaluated before use.

A2 Sample Analysis

The following analytical methods and references are offered for informational purposes without any recommendation of their suitability for analysis of specific brines.

A2.1 *Standard Methods for the Examination of Water and Wastewater*.⁵

A2.2 ASTM D3651.⁶ This method uses atomic absorption with a multielement hollow cathode lamp and an acetylene-nitrous oxide flame. The wavelength chosen is 553.6 nm and a secondary wavelength of 455.4 nm is given for the purpose of minimizing interference from calcium.

⁽⁵⁾ ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

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A2.3 ASTM D3986.⁷ This method uses plasma emission at 455.4 nm. Calcium is noted as a likely interference. Matrix interferences are minimized by buffering samples and standards with lithium ion.

A2.4 ASTM D3352.⁸ This method uses atomic absorption with a multielement hollow cathode lamp and an air-acetylene flame at the 460.7 nm wavelength and addition of lanthanum ion to control interference.