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Item No. 21208

## Standard Test Method

# 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)

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NACE International  
1440 South Creek Drive  
Houston, Texas 77084-4906  
+1 (281) 228-6200

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## Foreword

Scale is an adherent deposit of inorganic compounds precipitated from water onto surfaces. Most oilfield waters are brines containing large amounts of calcium salts. When calcium is deposited as calcium sulfate or calcium carbonate scale, a loss of production and increased maintenance expenses can result; therefore, effective scale inhibition is of primary importance to the oil producer.

Scale inhibitors can be used in many circumstances to control scale formation, thereby reducing production difficulties. Scale inhibitors are commercially available and are widely used in oil and gas production systems. The test methods in this standard are designed to provide a relative and quantitative measure of the abilities of scale inhibitors to prevent the precipitation of solids, a necessary and critical stage in the formation of scale. The laboratory screening tests described in this standard cannot and do not allow for the wide variation in water chemistry and system properties seen in field operations. As such they must only be regarded as a starting point in the evaluation of scale inhibitors. The existence and use of these methods allow for a uniform mode of collection of screening test results and facilitate discussion of the results by interested parties.

The test methods in this standard have been selected as a means of comparing, under the specified laboratory conditions, the effectiveness of scale inhibitors in preventing precipitation of calcium sulfate and calcium carbonate from solution. Because the prices of scale inhibitors change with time and may be unknown to the tester, no attempt has been made to dilute the scale inhibitor to a common cost base.

This standard was originally prepared in 1974 by Task Group T-1D-9 and was revised in 1990 by Task Group T-1D-31, a component of Unit Committee T-1D on Corrosion Monitoring and Control of Corrosion Environments in Petroleum Production Operations. It was reviewed and reaffirmed in 1995 by members of T-1D, and in 2001 and 2007 by members of Specific Technology Group (STG) 31 on Oil and Gas Production—Corrosion and Scale Inhibition. It is issued by NACE International under the auspices of STG 31.

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Standard  
Test Method**

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Inhibitors to Prevent the Precipitation of Calcium Sulfate and  
Calcium Carbonate from Solution (for Oil and Gas Production  
Systems)**

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## Section 1: General

1.1 The test methods described in this standard are static laboratory screening tests designed to give a measure of the ability of scale inhibitors to prevent the precipitation of calcium sulfate and calcium carbonate from solution at 71°C (160°F).

1.2 These test methods are recommended only for ranking the performance of different scale inhibitors under laboratory conditions set by these methods. They are not intended to provide actual field treating rates.

1.3 Many factors, such as reaction kinetics, fluid velocity and composition, variable temperatures and pressures, scale adherence, and solids dispersion can significantly affect actual scale deposition under field conditions. Detailed consideration of these parameters is deemed to be outside the scope of this standard. However, field

conditions, field brine composition, and other variables noted above should be considered at some point in scale inhibitor evaluation prior to final scale inhibitor selection for field use.

1.4 Tests should be conducted at various scale inhibitor concentrations in order to obtain a better understanding of performance under laboratory conditions set by these methods. The scale inhibitor concentration required for a field application is likely to be different from that determined under these laboratory conditions.

1.5 This standard lists the necessary apparatus, reagents, and procedures for conducting these laboratory screening tests.

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## Section 2: Calcium Sulfate Precipitation Test

2.1 This section lists the apparatus, solutions, and procedure for conducting the calcium sulfate precipitation screening test.

### 2.2 Apparatus and Solutions

2.2.1 Constant-temperature water bath or forced-draft oven with the capability of maintaining the specified temperature within  $\pm 1^\circ\text{C}$  ( $\pm 2^\circ\text{F}$ ).

2.2.2 Clean and dust-free glass test cells (approximately 125-mL [4-oz] bottles with positive seals).

2.2.3 Synthetic brines prepared with distilled or deionized water, as follows:

2.2.3.1 Calcium-containing brine: 7.50 g/L NaCl (ACS<sup>(1)</sup> reagent grade); 11.10 g/L  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  (ACS reagent grade).

2.2.3.2 Sulfate-containing brine: 7.50 g/L NaCl (ACS reagent grade); 10.66 g/L  $\text{Na}_2\text{SO}_4$  (ACS reagent grade).

2.2.3.3 Note: Very small quantities of insoluble materials may remain after the specified reagents have completely dissolved. For consistency of

results, solutions shall be filtered through a 0.45- $\mu\text{m}$  filter.

2.2.4 Apparatus for reproducibly delivering 50  $\pm$ 0.5 mL (e.g., graduated cylinders or volumetric pipets).

2.2.5 One percent by weight (1 wt%) and 0.1 wt% dilutions of the as-received scale inhibitors to be tested, prepared with deionized water.

2.2.6 Graduated measuring pipets in the following sizes: 0.1, 0.5, and 1.0 mL.

2.2.7 Standard reagents and apparatus for determination of calcium concentration in accordance with ASTM<sup>(2)</sup> D 511,<sup>1</sup> ASTM D 1126,<sup>2</sup> APHA<sup>(3)</sup> *Standard Methods for the Examination of Water and Wastewater* (Part 300),<sup>4</sup> or another accepted test method.

### 2.3 Test Procedure

2.3.1 Pipet the desired amount of scale inhibitor into each test cell using the 1 wt% and 0.1 wt% dilutions. The 0.1 wt% dilution shall be used for tests in which scale inhibitor loadings are less than 10 mg/L. Run duplicates of each concentration.

<sup>(1)</sup> American Chemical Society (ACS), 1155 16<sup>th</sup> St. NW, Washington, DC 20036.

<sup>(2)</sup> ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428.

<sup>(3)</sup> American Public Health Association (APHA), 800 I St. NW, Washington, DC 20001-3710.

2.3.2 Prepare duplicate blanks as follows:

2.3.2.1 Set aside two samples of the calcium-containing brine (50 mL each). Determine the calcium ion concentration of the blanks before precipitation in accordance with Paragraph 2.3.8 and divide each value by 2.

2.3.2.2 Prepare and handle the blanks after precipitation as described in Paragraphs 2.3.3 through 2.3.8, but do not include a scale inhibitor.

2.3.3 Add 50 mL of sulfate-containing brine to the test cell and mix well. Add 50 mL of calcium-containing brine to the test cell.

2.3.4 Cap the test cell immediately and agitate to mix the brines and the scale inhibitor thoroughly.

2.3.5 Place all test cells and blanks in a forced-draft oven or immerse to 75% of their lengths in a water bath at 71 ±1°C (160 ±2°F) for 24 hours.

2.3.6 Remove the test cells after the 24-hour exposure and avoid agitation. Allow the test cells to cool to 25 ±5°C (77 ±9°F) for a time not to exceed two hours.

2.3.7 Pipet 1 mL of the test brine to a suitable vessel, avoiding the transfer of calcium sulfate crystals, and dilute with distilled water, deionized water, or as otherwise specified in the calcium determination method to be used.

2.3.8 Determine the calcium ion concentration by procedures given in ASTM D 511, ASTM D 1126, APHA *Standard Methods for the Examination of Water and Wastewater* (Part 300), or another accepted test method. NOTE: Calcium ion concentration values for duplicate test samples often differ by 2% or more. A 5% difference is considered unacceptable and shall be cause for rerunning the test.

2.3.9 Report the average of the duplicate calcium ion concentration values as mg/L calcium sulfate retained in solution for each inhibitor test concentration and the blank.

2.3.10 Percent inhibition may be calculated in accordance with Paragraph 4.1 and reported.

2.3.11 Representative data from the evaluation of three scale inhibitors are given in Table 1. These data are examples only and do not reflect experimental precision. These data indicate that scale inhibitor A is most effective. Note: Costs of the scale inhibitors have not been considered.

**TABLE 1—Calcium Sulfate Retained in Solution (as Calcium Sulfate, mg/L)**

Scale Inhibitor	1 mg/L	3 mg/L	5 mg/L	10 mg/L	20 mg/L
A	5,140	5,140	5,140	5,140	5,140
B	4,080	4,352	4,896	5,068	5,140
C	4,896	5,103	5,140	5,140	5,140

Blank (after precipitation): 3,808 mg/L  
 Blank (before precipitation): 5,140 mg/L

**Section 3: Calcium Carbonate Precipitation Test**

3.1 This section lists the apparatus, solutions, and procedure for conducting the calcium carbonate precipitation screening test.

3.2 Apparatus and Solutions

3.2.1 A pressure-regulated source of carbon dioxide (CO<sub>2</sub>). All recognized grades of CO<sub>2</sub> are suitable for this test.

3.2.2 Constant-temperature water bath or forced-draft oven with the capability of maintaining the specified temperature within ±1°C (±2°F).

3.2.3 Clean and dust-free glass test cells (approximately 125-mL [4-oz] bottles with positive seals). Caution: The amount of vapor space above the test solutions in Paragraph 3.3.5 will affect the test results. To maximize the validity and reproducibility of test results, choose test cells that vary in capacity (volume) when sealed by 5% or less; that is,  $V_r = V \pm 0.025 V$ , where  $V_r$  equals the desired range of test cell capacities and  $V$  equals the mean test capacity.

3.2.4 Fritted-glass gas dispersion tube(s) (medium or coarse porosity rating).

3.2.5 Synthetic brines prepared with distilled or deionized water, as follows:

3.2.5.1 Calcium-containing brine: 12.15 g/L  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  (ACS reagent grade); 3.68 g/L  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  (ACS reagent grade); 33.0 g/L NaCl (ACS reagent grade).

3.2.5.2 Bicarbonate-containing brine: 7.36 g/L  $\text{NaHCO}_3$  (ACS reagent grade); 33.0 g/L NaCl (ACS reagent grade).

3.2.5.3 Note: Very small quantities of insoluble materials may remain after the specified reagents have completely dissolved. For consistency of results, the solutions shall be filtered through a 0.45- $\mu\text{m}$  filter.

3.2.6 Apparatus for reproducibly delivering 50  $\pm$ 0.5 mL (e.g., graduated cylinders or volumetric pipets).

3.2.7 One percent by weight (1 wt%) and 0.1 wt% dilutions of the as-received scale inhibitors to be tested, prepared with deionized water.

3.2.8 Graduated measuring pipets in the following sizes: 0.1, 0.5, and 1.0 mL.

3.2.9 Standard reagents and apparatus for determination of calcium concentration in accordance with ASTM D 511, ASTM D 1126, *APHA Standard Methods for the Examination of Water and Wastewater* (Part 300), or another accepted test method.

### 3.3 Test Procedure

3.3.1 Pipet the desired amount of inhibitor into each test cell using the 1 wt% and 0.1 wt% dilutions. The 0.1 wt% dilution shall be used for tests in which scale inhibitor loadings are less than 10 mg/L. Run duplicates of each concentration.

3.3.2 Prepare duplicate blanks as follows:

3.3.2.1 Set aside two samples of the calcium-containing brine (50 mL each). Determine the calcium ion concentration of the blanks before precipitation in accordance with Paragraph 3.3.9 and divide each value by 2.

3.3.2.2 Prepare and handle the blanks after precipitation according to Paragraphs 3.3.3 through 3.3.9, but do not include a scale inhibitor.

3.3.3 Saturate both the calcium- and bicarbonate-containing brines with  $\text{CO}_2$  immediately before using. Accomplish saturation at room temperature by bubbling  $\text{CO}_2$  through a fritted-glass gas dispersion tube immersed to the bottom of the container. A rate of 250

mL/min of  $\text{CO}_2$  for 30 minutes will be sufficient to saturate up to 1 L of each brine simultaneously. A tee may be used to split the gas flow for this purpose.

3.3.4 Add 50 mL of bicarbonate-containing brine to the test cell and mix well. Add 50 mL of calcium-containing brine to the test cell.

3.3.5 Cap the test cell immediately and agitate to mix the brines and the scale inhibitor thoroughly. The test cells must be capped tightly to avoid loss of  $\text{CO}_2$ . Note: Pressure will build in the test cells as the  $\text{CO}_2$ -saturated test brine approaches and reaches the test temperature. Rupture of the test cells has not been reported, yet it is a potential hazard associated with this test procedure. Safety precautions must be used to address this potential hazard. Also, an improperly sealed test cell may lead to pressure release, a resulting test brine compositional change, and an invalid test result.

3.3.6 Place all test cells and blanks in a forced-draft oven or immerse to 75% of their lengths in a water bath at 71  $\pm$ 1°C (160  $\pm$ 2°F) for 24 hours.

3.3.7 Remove the test cells after the 24-hour exposure and avoid agitation. Allow the test cells to cool to 25  $\pm$ 5°C (77  $\pm$ 9°F) for a time not to exceed two hours.

3.3.8 Pipet 1 mL of the test brine to a suitable vessel, avoiding the transfer of calcium carbonate crystals, and dilute with distilled water, deionized water, or as otherwise specified in the calcium determination method to be used.

3.3.9 Determine the calcium ion concentration by procedures given in ASTM D 511, ASTM D 1126, *APHA Standard Methods for the Examination of Water and Wastewater* (Part 300), or another accepted test method. NOTE: Calcium ion concentration values for duplicate test samples often differ by 2% or more. A 5% difference is considered unacceptable and shall be cause for rerunning the test.

3.3.10 Report the average of the duplicate calcium concentration values as mg/L calcium carbonate retained in solution for each inhibitor test concentration and the blank.

3.3.11 Percent inhibition may be calculated in accordance with Paragraph 4.1 and reported.

3.3.12 Representative data from the evaluation of three scale inhibitors are given in Table 2. These data are examples only and do not reflect experimental precision. These data indicate that scale inhibitor C is most effective. Note: Costs of the scale inhibitors have not been considered.

**TABLE 2—Calcium Carbonate Retained in Solution (as Calcium Carbonate, mg/L)**

Scale Inhibitor	1 mg/L	3 mg/L	5 mg/L	10 mg/L	20 mg/L
A	3,000	3,400	3,800	4,000	4,140
B	3,500	4,000	4,100	4,100	4,100
C	3,600	4,140	4,140	4,140	4,140

Blank (after precipitation): 2,600 mg/L  
 Blank (before precipitation): 4,140 mg/L

**Section 4: Percent Inhibition Calculation**

4.1 Percent inhibition values may be calculated as shown in Equation (1):

$$\% \text{ Inhibition} = \frac{C_a - C_b}{C_c - C_b} \times 100 \quad (1)$$

Where:

$C_a$  =  $Ca^{2+}$  concentration in the treated sample after precipitation  
 $C_b$  =  $Ca^{2+}$  concentration in the blank after precipitation  
 $C_c$  =  $Ca^{2+}$  concentration in the blank before precipitation

4.2 Caution: The percent inhibition calculation is for comparative purposes only. It is not intended to reflect the ability of a particular scale inhibitor to prevent scaling in a field application.

**References**

1. ASTM D 511 (latest revision), "Standard Test Methods for Calcium and Magnesium in Water" (West Conshohocken, PA: ASTM).
2. ASTM D 1126 (latest revision), "Standard Test Method for Hardness in Water" (West Conshohocken, PA: ASTM).
3. APHA, Standard Methods for the Examination of Water and Wastewater (Part 300) (Washington, DC: APHA).