Recommended Practice for Laboratory Testing of Drilling Fluids

API RECOMMENDED PRACTICE 13I EDITION, xxxx 201X
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Suggested revisions are invited and should be submitted to the Standards Department, API, 1220 L Street NW, Washington, DC 20005, standards@api.org.
Introduction

This Standard, which establishes testing methodologies for drilling fluid materials, is based on API RP 13I, seventh edition. This Standard was developed in response to a demand for more exacting testing methodologies. The tests contained herein were developed over several years by a group of industry experts and were identified as being those which can yield reproducible and accurate results. The tests are anticipated to be performed in a laboratory setting, but can be applicable in a field situation with more rigorous apparatus and conditions than normally found in a drilling fluid field-test kit.

These tests are designed to assist in the evaluation of certain parameters for drilling fluids, with these properties not necessarily used for the maintenance of a drilling fluid in field use. The tests provide either more precision or different properties than those given in the field-testing standards API RP 13B-1 and API RP 13B-2.

It is necessary that users of this Standard be aware that further or differing requirements can be needed for individual applications. This Standard is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for individual applications. This may be particularly appropriate where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this Standard and provide details.

As with any laboratory procedure requiring the use of potentially hazardous chemicals, the user is expected to have received proper knowledge and training in the use and disposal of these chemicals. The user is responsible for compliance with all applicable local, regional, and national regulations for worker and local health, safety and environmental liability.

This Standard contains footnotes giving examples of apparatus, reagents and sometimes the supplier(s) of those materials that are available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by API of the products named. Equivalent products may be used if they can be shown to lead to the same results.
1 Scope

This Standard provides procedures for the laboratory testing of both drilling fluid materials and drilling fluid physical, chemical and performance properties. It is applicable to both water-based and non-aqueous drilling fluids, as well as the base or “make-up” fluid.

It is not applicable as a detailed manual on drilling fluid control procedures. Recommendations regarding agitation and testing temperature are presented because the agitation history and temperature have a profound effect on drilling fluid properties.

NOTE For procedures related to testing barite for mercury, cadmium, and arsenic see API 13K.

NOTE For procedures related to testing barite, see API 13A.

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

API 13A, Specification for Drilling Fluids Materials
API RP 13B-1, Recommended Practice for Field Testing Water-Based Drilling Fluids
API RP 13B-2, Recommended Practice for Field Testing Oil-Based Drilling Fluids
ASTM D93, 5.12 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
ASTM D97, Petroleum products — Determination of pour point
ASTM E100, Standard Specification for ASTM Hydrometers
ASTM D611:1997, Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point
ASTM D1141, Standard Practice for the Preparation of Substitute Ocean Water
ASTM D4052, Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter
ASTM D5186, Standard Test Method for Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography
IP 200, Guidelines for the use of the Petroleum Measurement Tables
IP 391, Petroleum products — Determination of aromatic hydrocarbon types in middle distillates — High performance liquid chromatography method with refractive index detection
ISO 91-1:1992, Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 °C and 60 °F
ISO 3007, Petroleum products and crude petroleum — Determination of vapour pressure — Reid method
ISO 3104, Petroleum products — Transparent and opaque liquids—Determination of kinematic viscosity and calculation of dynamic viscosity

3 Terms, Symbols, and Abbreviations

3.1 Terms and Definitions

3.1.1 ACS reagent grade
chemical which meets purity standards as specified by the American Chemical Society (ACS)

3.1.2 autoclave

xxxxxxxxxxxxx
3.1.3 **base oil**
solids- and water-free hydrocarbon oil, commonly used in the drilling fluid industry for preparation and/or dilution of an oil-based drilling or completion fluid

NOTE Commonly used base oils are often termed “mineral oils”, “solvent oils” or “absorber oils” and also include the “diesel oils”.

3.1.4 **Carboy**
A rigid container with a typical capacity of 20-60 L. Used for transporting liquids.

3.1.5 **darcy**, (d)
Permeability (k) is a measure of the ability of a porous media to allow a fluid to flow through it. A porous media with a permeability of 1 darcy permits the laminar flow of 1 cm³/s of a fluid with a viscosity of 1 cP, under a pressure gradient of 1 atm/cm, across an area of 1 cm².

NOTE 1 cP = 1 mPa's

NOTE The darcy is not an SI unit. Permeability in SI unit is expressed in square meters and, 1 d = 0.9869 x 10⁻¹² m².

3.1.6 **flash side**
side containing residue (“flash”) from stamping and with concave indentations

3.1.7 **quarter**, verb
mix and divide into four equal specimens to assure homogeneity of specimens

3.1.8 **spurt loss**
volume of fluid that passes through the filtration medium before a filter cake is formed

3.1.9 **tube sampling**
sampling method comprising withdrawal of powdered sample from bag or bulk via a cylindrical device pushed into the sample, locked shut and withdrawn

3.1.10 **White spirit**

3.2 **Abbreviations**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>AA</td>
<td>atomic absorption spectroscopy</td>
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<tr>
<td>ACS</td>
<td>American Chemical Society</td>
</tr>
<tr>
<td>API</td>
<td>American Petroleum Institute</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
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<tr>
<td>BB</td>
<td>ball bearings</td>
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<tr>
<td>CAS</td>
<td>Chemical Abstracts Service, a division of ACS</td>
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<tr>
<td>DCP</td>
<td>direct current plasma</td>
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<tr>
<td>DS</td>
<td>drill solids</td>
</tr>
<tr>
<td>EDTA</td>
<td>ethylenediaminetetraacetic acid</td>
</tr>
<tr>
<td>HTHP</td>
<td>high-temperature, high-pressure</td>
</tr>
</tbody>
</table>
ICP  inductively coupled plasma
IP  standards issued by Energy Institute (formerly, Institute of Petroleum)
ISA  ionic strength adjuster
ISE  ion-selective electrode
LGS  low-gravity solids
MBT  methylene blue test
NAF  non-aqueous drilling fluid
NABF  non-aqueous base fluid
PAC-HV  high-viscosity polyanionic cellulose
PAC-LV  low-viscosity polyanionic cellulose
PI  performance index
PPA  permeability plugging apparatus
PPT  permeability plugging test
PTFE  polytetrafluoroethylene
YP  yield point

3.3 Symbols

$C$  concentration
$C_{B,M}$  concentration of bentonite, in kilograms per cubic meter;
$C_{B,US}$  concentration of bentonite, in pounds per barrel;
$C_{DS,M}$  concentration of drilled solids, in kilograms per cubic meter
$C_{DS,US}$  concentration of drilled solids, in pounds per barrel
$C_{LG,M}$  concentration of low gravity solids, in kilograms per cubic meter;
$C_{LG,US}$  concentration of low gravity solids, in pounds per barrel;
$C_{MBT-AVE}$  average methylene blue capacity for all low-gravity solids, in milliequivalents per hundred grams (meq/100 g)
$C_{MBT-B}$  methylene blue capacity of commercial bentonite, in milliequivalents per hundred grams (meq/100 g)
$C_{MBT-DS}$  methylene blue capacity of drill solids, in milliequivalents per hundred grams (meq/100 g)
$d$  inner diameter
$D$  outer diameter
$E_{BE,M}$  bentonite equivalent, expressed in kilograms per cubic meter
$E_{BE,US}$  bentonite equivalent, expressed in pounds per barrel;
$e$  thinner efficiency, in percent
$F_{PI}$  the performance index (mathematical symbol)
$H_{c}$  corrected hydrometer reading (the hydrometer reading minus composite correction)
a abrasion, in milligrams per minute

$G_{10\, s}$ 10 s gel reading

$G_{10\, \text{min}}$ 10 min gel reading

$L$ effective depth of hydrometer, in centimeters (see Table 2)

$m_{\text{As}}$ mass of arsenic in the digested sample, in micrograms

$m_b$ initial blade mass, in milligrams

$m_B$ mass of a commercial bentonite sample, in grams

$m_h$ mass of water required for solids adjustment, in grams

$m_L$ specified mass of suspension, in grams

$m_d$ mass of a dry sample, in grams

$m_{\text{DS}}$ mass of drill solids sample, in grams

$m_f$ final blade mass, in milligrams

$m_{\text{Hg}}$ mass of mercury in the digested sample, in micrograms

$m_o$ mass of original sample, in grams

$m_r$ mass of residue, in grams

$m_s$ mass of sample, in grams

$P_{\text{df}}$ phenolphthalein alkalinity of the drilling fluid

$p_r$ performance of reference thinner, for example yield point or gel strength as determined in accordance with API RP13B-1

$p_s$ performance of test sample, for example yield point or gel strength as determined in accordance with API RP13B-1

$t_1$ time at initial reading, in minutes

$t_2$ time at final reading, in minutes

$w_{\text{As}}$ mass fraction of arsenic in the sample, in micrograms per gram

$w_{\text{Cd}}$ mass fraction of cadmium in the sample, in micrograms per gram

$w_d$ mass fraction of sample in suspension, in percent

$w_h$ moisture content, as percent mass fraction

$w_s$ mass fraction of solids, in percent

$w_{\text{Hg}}$ mass fraction of mercury in the sample, in micrograms per gram

$w_f$ part of material finer than the sieve, in percent (mass fraction)

$w_{\text{Pb}}$ mass fraction of lead in the sample, in micrograms per gram

$w_r$ mass recovery (mass residue), expressed as a mass fraction in percent

$t$ time, in minutes
4.1 Principle

Drilling fluid weighting materials can vary considerably in relative abrasivity. This laboratory test is designed to measure and evaluate this relative abrasivity.

4.1.2 The test is performed using an abrasion test blade (see 4.2.8) attached to a high-speed mixer to mix a base drilling fluid containing the weighting material. The mass loss of the blade is used to calculate the abrasivity of the weighting material in milligrams per minute (mg/min).

4.1.3 This test is intended to give only a relative index of wear for weighting materials. It should not be used to infer whether or not these materials can cause abrasion problems in a field drilling fluid system.

4.2 Reagents and Apparatus

4.2.1 Calibration bentonite.

Calibration bentonite can be acquired from various suppliers. Accuracy of results from the use of this material is strictly the responsibility of the laboratory using it. See API Spec 13A for a description of the required properties of calibration bentonite and for details on API’s Reference Material program.

4.2.2 Detergent.

4.2.3 Distilled / de-ionized water, in accordance with ISO 3696:1987, or prepared by passing distilled water through a series of cation and anion exchange resins.

Use distilled-deionized water for preparation of all reagents and calibration standards and as dilution water.
4.2.4 **Mixer**, capable of operation at 11,500 r/min ±300 r/min under load, with single, corrugated impeller. The impeller shall be replaced with an abrasion test blade in accordance with 4.2.8.

4.2.5 **Mixing Container**, approximately 7 1/8 in. (180 mm) deep, \( d = 3 \frac{3}{4} \) in. (70 mm) at bottom.

4.2.6 **Balance**, accuracy ±0.0001 g.

4.2.7 **Balance**, of capacity 500 g and accuracy ±0.1 g.

4.2.8 **Blade**, for abrasion test, steel, of diameter 1.4 in. (36.5 mm); flat side of thickness 0.05 in. (1.4 mm); Rockwell scale hardness of 16; four waves of depth 0.25 in. (6.4 mm) and width 0.6 in. (15.3 mm); center hole of diameter 0.28 in. (7.1 mm), and a G-90 coating with a mass of approximately 12 g.

Each blade may be used up to a cumulative mass loss of 200 mg (approximately four runs).

4.2.9 **Screw**, 0.5 in. (13 mm), size 10, round-head machine screw, 32 threads/in., to fasten the abrasion test blade securely onto the shaft of the abrasion mixer.

4.2.10 **Tachometer**, capable of reading 11,000 r/min ±50 r/min.

4.2.11 **Cylinder**, graduated, TD 500 mL ±2.5 mL.

4.2.12 **Viscometer**, direct indicating, as described in API RP 13B-1.

4.2.13 **Timer**, mechanical or electrical, accurate to ±0.1 min over the test interval.

4.2.14 **Small brush**.

4.3 **Determination of Abrasivity of Weighting Materials**

4.3.1 Prepare a base suspension by adding 15.0 g ±0.1 g reference bentonite to 350 mL ±2.5 mL distilled water in a container while stirring on the base suspension mixer. At least two of these suspensions are needed for the test.

4.3.2 After about 5 min stirring time on the mixer, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container. Be sure that all clay clinging to the spatula is incorporated into the suspension.

4.3.3 Replace the container on the mixer and continue stirring for about 15 min. The container should be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls at 5 min intervals. Total stirring time after adding the clay shall be 20 min.

4.3.4 Pour the base suspension into the cup provided with the direct-indicating viscometer. Note the dial reading at the 600 r/min rotor-speed setting when it reaches a constant value. The 600 r/min dial reading shall not exceed 10 at a test temperature of 77 °F ±3 °F (25 °C ±1 °C). If necessary, the 600 r/min dial reading of the base suspension can be lowered by adding a small amount of water.

4.3.5 Pour 300 mL ±2.5 mL of base suspension into the mixing container (use 280 mL ±2.5 mL when testing barite).

4.3.6 Add 300 g ±1 g of weighting materials into the base suspension in the test container.

4.3.7 Immediately prior to use, clean the blade by washing with detergent and a small brush. Rinse thoroughly and dry. Weigh a freshly cleaned and dried abrasion test blade to the nearest 0.1 mg. Record as \( m_b \), in milligrams.

Only non-corroded blades should be used.

4.3.8 Disconnect the mixer power cord to avoid accidental operation during blade installation. Center and fasten the abrasion test blade, with the waves in the downward position, onto the abrasion mixer spindle with the lock washer and screw.
4.3.9 With the mixer off, place the mixing container into position on the mixer, such that the rim engages with the trip switch. Start the mixer using a series of quick “on-off” flips of the switch to bring the spindle up to speed gently.

NOTE An abrupt start can sling some suspension out of the cup.

Blades should be polished after manufacture to remove rough edges and the blades should be installed on the mixer with the waves in the downward position.

4.3.10 Run the test for 20 min ±0.1 min.

4.3.11 Turn the mixer off, disconnect the power cord, remove the mixing container and the abrasion test blade.

4.3.12 Clean and dry the abrasion test blade and weigh to the nearest 0.1 mg. Record as \( m_f \), in milligrams.

4.4 Calculation of Determination of Abrasivity of Weighting Materials

4.4.1 Calculate, \( a \), the abrasion, expressed in milligrams per minute, as given in Equation (1):

\[
a = \frac{m_b - m_f}{t}
\]

where

- \( m_b \) is the initial blade mass, expressed in milligrams;
- \( m_f \) is the final blade mass, expressed in milligrams;
- \( t \) is the time, expressed in minutes (20 min in this procedure).

4.4.2 The test precision for this procedure has been determined to be as follows:

\( r = \pm 0.45 \)

\( R = \pm 0.78 \)

where

- \( r \) is the within-laboratory repeatability, the maximum expected difference between two test results on the same sample by the same lab at the 95% confidence level;
- \( R \) is the between-laboratories reproducibility; the maximum expected difference between test results by two labs on the same sample at the 95% confidence level.

Because this test is not an absolute measurement, test results should be evaluated cautiously. Best evaluations are obtained by comparing test results of a sample to test results of a weighting material of proven performance, rather than using the test-result value obtained as an absolute.

5 Filtration-control agents

5.1 Principle

5.1.1 Organic filtration-control agents can vary greatly in chemical composition and performance characteristics in different drilling fluid environments. Judgments are made by comparing the performance of test filtration control materials to that of known materials that have been accepted for field use.

5.1.2 In specific cases, comparisons may be made in the actual field drilling fluid in which the material will be used, but in general, evaluations are performed in laboratory-prepared drilling fluids.

5.1.3 It is recommended that filtration-control materials considered for broad applications be screened in different drilling fluid systems. While filtration control is the property being evaluated, the effect of the test material on rheological properties, pH or other properties can be of concern.

5.2 Reagents and apparatus
5.2.1 Calcium chloride, CaCl₂ (CAS number 10043-52-4), ACS reagent grade.

5.2.2 Magnesium chloride, MgCl₂ (CAS number 7786-30-3), ACS reagent grade.

5.2.3 Potassium chloride, KCl (CAS number 7447-40-7), ACS reagent grade.

5.2.4 Xanthan gum, drilling fluid grade dry powder. Refer to API 13A for material specifications.

5.2.5 Attapulgite, refer to API 13A for material specifications.

5.2.6 Simulated drilled solids, for example Standard Evaluation Base Clay. Refer to API 13A for material specifications.

5.2.7 Reference bentonite. If fluid loss properties are not obtained within the specified range, replace the Reference Bentonite. See API 13A for material specifications.

5.2.8 Salt, stock solution No. 1, in accordance with ASTM D 1141, 555.6 g magnesium chloride hexahydrate [MgCl₂·6H₂O (CAS number 7791-18-6)], 57.9 g anhydrous calcium chloride [CaCl₂ (CAS number 10043-52-4)] and 21 g strontium chloride hexahydrate [SrCl₂·6H₂O (CAS number 10025-70-4)] diluted to 1 L with distilled or de-ionized water.

5.2.9 Salt, stock solution No. 2, in accordance with ASTM D 1141, 69.5 g potassium chloride [KCl (CAS number 7447-40-7)], 20.1 g sodium bicarbonate [NaHCO₃ (CAS number 144-55-8)], 10.0 g potassium bromide [KBr (CAS number 7758-02-3)], 2.7 g boric acid [H₃BO₃ (CAS number 10043-35-3)] and 0.3 g sodium chloride [KCl (CAS number 7681-49-4)], diluted to 1 L with distilled or de-ionized water.

5.2.10 Sodium hydroxide, (NaOH) i.e. caustic soda (CAS number 1310-73-2), standardized solution, cNaOH = 10 N and cNaOH = 0.1 N.

5.2.11 Lignosulfonate (chrome or ferrochrome), drilling fluid grade.

5.2.12 Lime, ACS reagent grade or equivalent.

5.2.13 Sodium chloride (CAS number 7647-14-5), ACS reagent grade.

5.2.14 Sodium sulfate, anhydrous (CAS number 7757-82-6), ACS reagent grade.

5.2.15 Deionized or distilled water.

5.2.16 Defoamer

NOTE For example octyl alcohol.

5.2.17 Balance, accuracy ±0.01 g.

5.2.18 Mixer, capable of operation at 11,500 r/min ±300 r/min under load, with a single, corrugated impeller approximately 1.0 in. (25.4 mm) in diameter.

The impeller shall be replaced when a mass loss of approximately 10% occurs. The original blade mass is about 5.5 g and should have been recorded when the impeller was placed in service.

All mixing referenced in Section 5 shall be conducted at 11,500 r/min ±300 r/min

5.2.19 Mixing Container, 7 1/8 in. (180 mm) deep, d = 3 3/4 in. (97 mm) at top and 2 3/4 in. (70 mm) at bottom. A standard mix cup generally conforms to these specifications.

5.2.20 Clock or timer, direct-reading counter with an accuracy of 0.1 s over the test interval.

5.2.21 Sealable container, appropriate to volume of fluid to be prepared.

5.2.22 Spatula.

5.2.23 Filter press, low-temperature/low-pressure, in accordance with API 13B-1, having a filter area of 7.00-7.20 in.² (diameter of 2.98-3.03 in.) [filter area of 4520-4640 mm² (diameter of 75.86-76.86 mm)].

The filter-press gasket is the determining factor for the filter area. It is recommended that the filter-press gasket used be tested by a conical gauge having the maximum [3.02 in. (76.86 mm)] and the minimum [2.98 in. (75.86 mm)] allowed diameter dimensions marked on it. Any filter press gasket diameter measuring outside this range should be discarded.

5.2.24 Viscometer, direct-reading, in accordance with API 13B-1.
5.2.25 **Roller oven**, regulated to maintain a temperature of 150 °F ±5 °F (65 °C ±3 °C).

5.2.26 **pH meter**, in accordance with API 13B-1.

5.2.27 **Jar**, small, covered, e.g. 0.5 L glass jar with screw-type lid.

5.2.28 **Oven**, regulated to maintain a temperature of 220 °F ±5 °F (105 °C ±3 °C).

5.2.29 **Aging cells**, in accordance with Clause 14.3 of this document.

5.2.30 **Filter cell**, high-temperature/high-pressure, in accordance with API 13B-1.

5.2.31 **Syringes**, disposable, 3 mL capacity.

5.3 **General Instructions for Preparation of Base Drilling Fluids**

5.3.1 Prepare a sufficient quantity of base drilling fluid for a series of tests. This can be achieved by preparing a large batch or by combining small batches for uniformity prior to aging.

5.3.2 Composition, order of addition, mixing time, shear, and aging time shall be consistent from batch to batch.

5.3.3 Stir all base drilling fluids prior to testing to ensure uniformity.

5.3.4 Test a sample of the untreated base drilling fluid each time a new series of tests is begun. Subject a sample of the untreated base drilling fluid to the same amount of stirring, heat aging and testing as the treated drilling fluids.

All quantities given are for the preparation of a single lab barrel.

5.4 **Salt-saturated Drilling Fluid Preparation for Filtration Testing**

5.4.1 Prepare saturated salt water by adding 400 g of sodium chloride to sufficient deionized water to yield 1 L (this will exceed the solubility of sodium chloride in water at room temperature).

5.4.2 Mix for 30 min, age overnight (16 ±2 h) at room temperature and decant into a large covered carboy (approximately 4 L).

5.4.3 Prepare a suspension of attapulgite clay and simulated drilled solids using 8 g of attapulgite and 30 g of simulated drilled solids in 338 mL (401 g) saturated salt water in a container. Sift the clay and solids into the water while stirring with the mixer. This yields 1 lab barrel.

5.4.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.4.5 Replace on the mixer and continue stirring for an additional 25 min (for a total mixing time of 30 min).

5.4.6 Store the suspension for 24 h in a sealed container at room temperature.

5.4.7 Stir the suspension for 5 min on the mixer.

5.4.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 90-105 mL. If not, prepare a new sample for testing.

5.4.9 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 439 g per 350 mL.

5.5 **High-hardness, Salt-saturated Drilling Fluid Preparation for Filtration Testing**

5.5.1 Prepare a high hardness brine by adding 125 g calcium chloride and 22 g magnesium chloride to sufficient saturated salt solution, as prepared in 5.4.1 to yield 1 L total volume.

5.5.2 Mix for 30 min, age overnight (16 ±2 h) at room temperature (75 ±5 °F) and decant into a large covered carboy.

5.5.3 Prepare a suspension of attapulgite clay and simulated drilled solids using 8 g of attapulgite and 30 g of simulated drilled solids in 338 mL (429 g) of high-hardness, salt-saturated drilling fluid in a container. Sift the clay and solids into the water while stirring with the mixer.
5.5.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.5.5 Replace on the mixer and continue stirring for an additional 25 min (for a total mixing time of 30 min).

5.5.6 Store the suspension for 24 h in a sealed container at room temperature.

5.5.7 Stir the suspension for 5 min on the mixer.

5.5.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 90-105 mL. If not, prepare a new sample for testing.

5.5.9 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 467 g per 350 mL.

5.6 10 % Potassium Chloride (KCl) Drilling Fluid Preparation for Filtration Testing

5.6.1 Prepare a 10 % potassium chloride (KCl) solution by adding 111 g of KCl to a container and diluting with water to 1 L.

5.6.2 Sift 1.0 g of Xanthan gum slowly into 360 g (340 mL) of the 10 % KCl solution while stirring on a mixer.

5.6.3 After 5 min, remove the container from the mixer and scrape the sides to dislodge any polymer adhering to the container.

5.6.4 Replace on the mixer and continue stirring for an additional 10 min (total mixing time 15 min).

5.6.5 Add 30 g of simulated drilled solids while continuing to stir with the mixer.

5.6.6 After 5 min, remove the container from the mixer and scrape the sides to dislodge any solids adhering to the container.

5.6.7 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 30 min).

5.6.8 Measure the rheological properties using the rheometer, in accordance with API 13B-1. If properties are as follows, continue to the next step in the testing procedure. If they are not, prepare a new sample for testing:

- plastic viscosity 3-7 cP (3-7 mPa·s);
- yield point 6-10 lbs/100 ft² (2.9-4.8 Pa);
- 10 s gel 2-4 lbs/100 ft² (0.96-1.92 Pa);
- 10 min gel 3-5 lbs/100 ft² (1.44-2.39 Pa).

5.6.9 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 30-50 mL, if not, prepare a new sample for testing.

5.6.10 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 391 g per 350 mL.

5.7 Pre-hydrated Bentonite Slurry Preparation for Filtration Testing

5.7.1 For procedures requiring pre-hydrated bentonite in a base drilling fluid, prepare and age the bentonite in advance.

5.7.2 Prepare a suspension of the bentonite using 26 g bentonite (as received) in 350 mL water (6.67 % mass fraction solids) in a container for each sample being tested. Sift the clay into the water while stirring with the mixer.

5.7.3 After 5 min, remove the container from the mixer and scrape the sides to dislodge any bentonite adhering to the container.
5.7.4 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).
5.7.5 Store the suspension overnight (16 ±2 h) in a sealed container at room temperature.
5.7.6 Stir the suspension for 5 min on a mixer before using.

5.8 Modified Seawater Drilling Fluid Preparation for Filtration Testing

5.8.1 Prepare simulated seawater by adding 24.5 g sodium chloride and 4.1 g anhydrous sodium sulfate to a 1 L flask. Dilute to 800 mL with distilled or deionized water. Pour 20.0 mL stock salt solution No. 1 and 10.0 mL of stock salt solution No. 2 into the flask and dilute to 1 L with deionized water while stirring. Adjust pH to 8.2 with 0.1 N sodium hydroxide solution.

5.8.2 Prepare a suspension of bentonite clay using 150 g (144 mL) of pre-hydrated bentonite slurry in a container, as prepared in 5.7. Add 193 mL of simulated seawater, as prepared in 5.8.1, while stirring on the mixer.

5.8.3 Continue stirring while adding 2.5 mL of 10 N NaOH and sifting in 30 g of simulated drilled solids.

5.8.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.8.5 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

5.8.6 Store the suspension for 24 h in a sealed container at room temperature.

5.8.7 Stir the suspension for 5 min on the mixer.

5.8.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 60-70 mL. If not, prepare a new sample for testing.

5.8.9 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 382 g per 350 mL fluid.

5.9 Low-salinity Drilling Fluid Preparation for Filtration Testing

5.9.1 Prepare a low-salinity solution by weighing 42 g sodium chloride into a flask and diluting to 1 L with water, resulting in 4 % sodium chloride brine.

5.9.2 Prepare a suspension of bentonite clay using 150 g (144 mL) of pre-hydrated bentonite slurry in a container, as prepared in 5.7. Add 193 mL of low-salinity water to the suspension of bentonite while stirring with the mixer.

5.9.3 Continue stirring while adding 2.5 mL of 10 N NaOH and sifting in 30 g of simulated drilled solids.

5.9.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.9.5 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

5.9.6 Store the suspension for 24 h in a sealed container at room temperature.

5.9.7 Stir the suspension for 5 min on the mixer.

5.9.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 35-45 mL. If not, prepare a new sample for testing.

5.9.9 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 382 g per 350 mL fluid.

5.10 Lime-treated Drilling Fluid Preparation for Filtration Testing
5.10.1 Prepare a suspension of bentonite clay using 260 g (250 mL) of pre-hydrated bentonite slurry in a container, as prepared in 5.7. Add 73 mL of water, 4 g of chrome lignosulfonate and 2.5 mL of 10 N NaOH while stirring on the mixer.

5.10.2 Stir for 30 min.

5.10.3 Continue stirring and sift in 3 g of lime and 30 g of simulated drilled solids.

5.10.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.10.5 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 45 min).

5.10.6 Roll the suspension for 16 h in a sealed container in an oven set at 150 °F (65 °C).

5.10.7 Cool and stir the suspension for 5 min on the mixer.

5.10.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 12-20 mL. If not, prepare a new sample for testing.

5.10.9 Measure the phenolphthalein alkalinity, $P_{df}$, of the drilling fluid using the procedure in API 13B-1. The $P_{df}$ of the drilling fluid should be 10-20 mL; if less than 10 mL, adjust with lime, if greater than 20 mL, prepare a new sample for testing.

5.10.10 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 373 g per 350 mL fluid.

5.11 Low Solids, Non-dispersed Drilling Fluid Preparation for Filtration Testing

5.11.1 Prepare a suspension of bentonite clay using 150 g (144 mL) of pre-hydrated bentonite slurry in a container, as prepared in 5.7. Add 200 mL of water and 15 g of simulated drilled solids while stirring with the mixer.

5.11.2 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.11.3 Replace the container on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

5.11.4 Store the suspension for 24 h in a sealed container at room temperature.

5.11.5 Stir the suspension for 5 min on the mixer.

5.11.6 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 20-30 mL. If not, prepare a new sample for testing.

5.11.7 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 365 g per 350 mL fluid.

5.12 Freshwater Lignosulfonate Drilling Fluid Preparation for Filtration Testing

5.12.1 Prepare a suspension of bentonite clay using 225 g (216 mL) of pre-hydrated bentonite slurry in a container, as prepared in 5.7. Add 127 mL of water, 15 g of simulated drilled solids, 1 g of chrome lignosulfonate and 1.2 mL of 10 N NaOH while stirring with the mixer.

5.12.2 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

5.12.3 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

5.12.4 Store the suspension for 24 h in a sealed container at room temperature.

5.12.5 Stir the suspension for 5 min on the mixer.
5.12.6 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with API 13B-1. The filtrate volume should be 9-15 mL. If not, prepare a new sample for testing.

5.12.7 Measure the pH of the suspension using a pH-meter, in accordance with API 13B-1. The pH should be 9.5-10.5. Adjust if necessary with NaOH if below 9.5; if above 10.5, prepare a new sample for testing.

5.12.8 For the purpose of testing (Clause 5.12), the mass equivalent for this sample is 369 g per 350 mL fluid.

5.13 Initial Performance Filtration Test

In order to perform a Performance Filtration Test on a sample of prepared drilling fluid from Section 5, the following procedure shall be used:

5.13.1 Select the concentrations of filtration control agents to cover a wide range (e.g. 0.1 g/350 mL, 0.5 g/350 mL, 1.0 g/350 mL, 3.0 g/350 mL and 5.0 g/350 mL). Include an untreated sample of the base drilling fluid.

If comparison to a known field-accepted material is being made, performance tests should be run on an equivalent range of known field-accepted material concentrations.

5.13.2 Weigh into each of several mixing containers the mass equivalent of 350 mL of the desired base drilling fluid. Place the containers on the mixer and mix to uniformity. Measure the pH of the base drilling fluid and record.

5.13.3 Weigh out the desired amounts of filtration-control agents and add uniformly over a period of 30 s while stirring on the mixer.

5.13.4 Mix for 30 min, removing the containers twice during mixing to scrape the sides to dislodge any clay and solids adhering to the container. If foaming occurs while stirring and remains when the sample is removed from the mixer, attempt to remove the entrained air by stirring slowly with a spatula. Add a defoamer if necessary; however, additions of a defoamer can change filtration results. Record the amount of defoamer added.

5.13.5 Measure the pH of the suspensions using the procedure described in API 13B-1. If the pH is lower than the untreated test drilling fluid by more than 0.5 pH units, adjust the pH to that of the untreated test drilling fluid with 10 N NaOH. If the pH is higher than the untreated test drilling fluid, record the pH, but do not make any adjustment.

5.13.6 Determine the low-temperature/low-pressure filtrate volume, in accordance with API 13B-1. Record the filtrate volume, cake thickness and cake consistency.

The filtrate volume should be determined immediately after preparation.

5.14 Low Temperature Filtration Performance Testing after Heat Aging

5.14.1 Dynamically age the samples prepared and tested in 5.12 by rolling or rotating in sealed jars for at least 16 h in a roller oven set at 150 °F (65 °C).

5.14.2 Cool to room temperature.

5.14.3 Measure the pH of the suspensions and adjust the pH with 10 N NaOH to the initial pH as described in 5.13.5.

5.14.4 Stir for 10 min on the mixer.

5.14.5 Determine the low-temperature/low-pressure filtrate volume. Record the filtrate volume, cake thickness and cake consistency.

5.15 High Temperature/High Pressure Performance Testing after Heat Aging

5.15.1 If an indication of the high-temperature stability is desired, age the suspensions either statically or dynamically at the higher temperature in a suitable test container. See Clause 14.3 for further details on metal aging cells.
5.15.2 Cool to room temperature. Stir for 5 min on the mixer.

5.15.3 Determine the high-temperature/high-pressure filtrate volume by the procedure described in API 13B-1. Record the filtrate volume, cake thickness and cake consistency.

6 Methylene Blue Test for Drilled Solids and Commercial Bentonite

6.1 Methylene Blue Capacity of Drill Solids

6.1.1 Principle

6.1.1.1 The methylene blue capacity of drill solids can be determined by a procedure that is similar to that used for the drilling fluid. The methylene blue capacity is an estimate of the cation-exchange capacity of the drill solids. The true cation-exchange capacity of the drill solids will probably be somewhat higher than the methylene blue capacity.

6.1.1.2 Methylene blue solution is used to titrate a sample of drill solids that has been dried, ground to a powder and treated with a strong dispersant and an oxidizing agent. The dispersed drill solids sample is titrated with methylene blue solution until a faint blue ring or "halo" appears around a drop of dyed drill solids on filter paper.

6.1.2 Reagents and Apparatus

6.1.2.1 Methylene blue solution, 3.20 g reagent grade methylene blue (C_{16}H_{18}N_{3}SCl) diluted to 1 L (1 mL = 0.01 milliequivalent) (CAS number 61-73-4).

If the solution is prepared in the laboratory, the moisture content of reagent grade methylene blue shall be determined each time the solution is prepared. Dry a 1.00 g portion of methylene blue to constant mass at 200 °F ±5 °F (93 °C ±3 °C). Make the appropriate correction in the mass, \( m_0 \), of methylene blue powder taken to prepare the solution as follows:

\[
m_0 = \frac{3.20}{m_d}
\]

where \( m_d \) is the mass of dry methylene blue powder sample, in grams.

6.1.2.2 Tetrasodium pyrophosphate, 2 % solution [20 g of tetrasodium pyrophosphate diluted to 1 L (CAS number 7722-88-5)].

6.1.2.3 Hydrogen peroxide, 3 % solution (CAS number 7722-84-1).

6.1.2.4 Sulfuric acid, dilute, \( c = 5 \) N.

6.1.2.5 Erlenmeyer flask, of capacity 250 mL.

6.1.2.6 Burette, 10 mL; micropipette 0.5 mL; or graduated pipette 1 mL.

6.1.2.7 Graduated cylinder, 50 mL (TD).

6.1.2.8 Stirring rod.

6.1.2.9 Hot plate.

6.1.2.10 Magnetic stirrer, with coated stirring bar.

6.1.2.11 Filter paper, hardened, low-ash grade.

6.1.2.12 Drying oven, capable of being maintained at 220 °F ±5 °F (105 °C ±3 °C).

6.1.2.13 Mortar and pestle.

6.1.2.14 Sieve, 75 µm (200 mesh).

6.1.2.15 Balance, accuracy ±0.05 g.

6.1.3 Procedure for Methylene Blue Capacity Testing of Drill Solids
6.1.3.1 Clean all traces of drilling fluid from the drill solids sample and dry at 220 °F ±5 °F (105 °C ±3 °C) for 2 h.

6.1.3.2 Grind the drill solids to a fine powder using the mortar and pestle, and pass through the 75 µm (200 mesh) sieve.

6.1.3.3 Weigh 1.0 g of the drill solids of size less than 75 µm, and add to the Erlenmeyer flask containing 25 mL of 2% tetrasodium pyrophosphate.

6.1.3.4 Mix well and boil gently for 10 min. Do not boil to dryness.

6.1.3.5 Add 15 mL hydrogen peroxide and 1 mL sulfuric acid. Boil gently for an additional 10 min. Cool to room temperature and dilute with water to a volume greater than 50 mL.

6.1.3.6 Add methylene blue solution to the flask in increments of 0.5 mL while stirring on the magnetic stirrer. If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous tests, somewhat larger increments (1-2 mL) can be used at the beginning of the titration. After each addition of the methylene blue solution, swirl the contents of the flask for 30 s. While the solids are still suspended, remove one drop of suspension with the stirring rod and place on the filter paper. The initial end point of the titration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids, as shown in Figure 2, key item 4, for a 6 mL addition.

6.1.3.7 When the initial end point is detected, shake the flask for an additional 2 min and place another drop on the test paper. If the blue ring is again evident, the final endpoint has been reached, as shown in Figure 2, key item 8. If the faint blue ring or halo does not appear, continue as in 6.1.3.6 until the second drop taken after 2 min shows the faint blue tint or halo, as shown in key items 8 and 9. This is the 2 min endpoint. Free dye detected immediately after adding methylene blue solution, which is adsorbed after 2 min, indicates that the endpoint has not quite been reached. See key items 4 and 9 in Figure 2.

6.1.3.8 Continue stirring an additional 10 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the 10 min endpoint has been reached. If the faint blue ring does not appear, continue as in 6.1.3.6 until the second drop taken after stirring 2 min shows the faint blue ring or halo.
Key

1. volume of methylene blue solution added
2. moisture
3. dyed drilling fluid solids (no free, unadsorbed dye present)
4. free dye (faint blue ring or halo), visible immediately after adding 6 mL, is adsorbed after 2 min and indicates that the end point has not quite been reached
5. dyed drilling fluid solids
6. free, unadsorbed dye
7. moisture
8. endpoint, free dye is not adsorbed after stirring an additional 2 min
9. appearance of spot after additional 2 min agitation

Figure 1—Spot Tests for Endpoint of Methylene Blue Titration

6.1.3.9 The methylene blue capacity of the drill solids, \( C_{MBT-DS} \), expressed in milliequivalents per 100 g (meq/100 g), is given by Equation (3).

\[
C_{MBT-DS} = \frac{V}{m_{DS}}
\]  

(3)

where

- \( V \) is the volume of methylene blue solution used in titration, in milliliters;
- \( m_{DS} \) is the mass of drill solids sample, in grams.
6.2 Methylene Blue Capacity of Commercial Bentonite

6.2.1 Principle

The methylene blue capacity of commercial bentonite can be determined using a procedure similar to that used for the drill solids. The methylene blue capacity is an estimate of the cation exchange capacity of the bentonite. The true cation exchange capacity of the bentonite will probably be somewhat higher than the methylene blue capacity. A well dispersed sample of the bentonite is titrated with methylene blue solution until a blue or turquoise “halo” appears around a drop of dyed bentonite solids on filter paper.

6.2.2 Reagents and apparatus

6.2.2.1 Methylene blue solution, 1 mL = 0.01 milliequivalent (see 6.1.2.1).

6.2.2.2 Tetrasodium pyrophosphate, 2 % solution (see 6.1.2.2).

6.2.2.3 Erlenmeyer flask, of capacity 250 mL.

6.2.2.4 Burette, of capacity 50 mL.

6.2.2.5 Graduated cylinder, 50 mL (TD).

6.2.2.6 Stirring rod.

6.2.2.7 Hot plate.

6.2.2.8 Magnetic stirrer and stirring bar (coated).

6.2.2.9 Filter paper, hardened, low-ash grade.

6.2.2.10 Balance, accuracy ±0.05 g.

6.2.3 Procedure for Methylene Blue Capacity Testing of Commercial Bentonite

6.2.3.1 Weigh 0.5 g bentonite and add to the Erlenmeyer flask containing 50 mL of 2 % tetrasodium pyrophosphate solution.

6.2.3.2 Mix well and boil gently for 10 min. Do not boil to dryness.

6.2.3.3 Cool to room temperature and bring volume to about 50 mL with water.

6.2.3.4 Add methylene blue solution to the flask with the burette in 1 mL increments while stirring on the magnetic stirrer. Somewhat larger increments can be used at the beginning of the titration (10-15 mL) as the endpoint should only be reached after 25 mL or more have been added. After each addition, swirl the contents of the flask for 30 s and remove one drop of the suspension with the stirring rod and place on the filter paper. The initial endpoint of the titration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids as shown in Figure 2, key item 4.

For best accuracy, it is recommended that increments no larger than 1 mL be used.

6.2.3.5 When the initial endpoint is detected, stir the flask for an additional 2 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the 2 min endpoint has been reached, as shown in Figure 2, key items 8 and 9. If the ring does not appear, continue as in 6.2.3.4 until a drop taken after stirring an additional 2 min shows the faint blue tint or halo.

6.2.3.6 Continue stirring an additional 10 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the 10 min endpoint has been reached. If the faint blue ring does not appear, continue as in 6.2.3.4 until a drop taken after stirring 2 min shows the faint blue ring or halo.

6.2.3.7 The methylene blue capacity of commercial bentonite, $C_{MBT-B}$, expressed in milliequivalents per hundred grams (meq/100 g), is calculated from Equation (4).

$$C_{MBT-B} = \frac{V}{m_B}$$

(4)
where

\( V \) is the volume of methylene blue solution used in titration, in milliliters;

\( m_B \) is the mass of a commercial bentonite sample, in grams.

### 6.3 Equations to Calculate Solids content from MBT results

#### 6.3.1 Principle

The equations in 6.3.2 can be used to give a rough estimate of the amount of drill solids and commercial bentonite in a drilling fluid using the concentration of low-gravity solids, \( C_{lg,M} \), expressed in kilograms per cubic meter, or \( C_{lg,US} \), expressed in pounds per barrel calculated in API 13B-1, plus the bentonite equivalent, \( E_{BE,US} \), expressed in pounds per barrel or \( E_{BE,M} \), expressed in kilograms per cubic meter, and the methylene blue capacity of the drill solids (6.1.3.9) and commercial bentonite (6.2.3.7).

#### 6.3.2 Calculation

The concentration of each type of low-gravity solid can be calculated from Equations (5) to (10):

When calculating in USC units the following equations shall be used:

\[
C_{MBT-AVG} = \frac{70 \times E_{BE,US}}{C_{LG,US}} \tag{5}
\]

\[
C_{B,US} = \frac{C_{MBT-AVG} - C_{MBT-DS}}{C_{MBT-B} - C_{MBT-DS}} \times C_{LG,US} \tag{6}
\]

\[
C_{DS,US} = C_{LG,US} - C_{B,US} \tag{7}
\]

where

- \( C_{MBT-AVG} \) is the average methylene blue capacity of for all low gravity solids, expressed in milliequivalents per hundred grams;
- \( C_{MBT-DS} \) is the methylene blue capacity of drilled solids, expressed in milliequivalents per hundred grams;
- \( C_{MBT-B} \) is the methylene blue capacity of commercial bentonite, expressed in milliequivalents per hundred grams;
- \( E_{BE,US} \) is the bentonite equivalent, expressed in pounds per barrel (API 13B-1);
- \( C_{LG,US} \) is the concentration of low gravity solids, expressed in pounds per barrel (API 13B-1);
- \( C_{DS,US} \) is the concentration of drilled solids, expressed in pounds per barrel

When calculating in metric units, the following equations shall be used:

\[
C_{MBT-AVG} = \frac{70 \times E_{BE,M}}{C_{LG,M}} \tag{8}
\]

\[
C_{B,M} = \frac{C_{MBT-AVG} - C_{MBT-DS}}{C_{MBT-B} - C_{MBT-DS}} \times C_{LG,M} \tag{9}
\]

\[
C_{DS,B} = C_{LG,B} - C_{B,B} \tag{10}
\]

where
7 Deflocculation Test for Thinner Evaluation

7.1 Principle

7.1.1 The following laboratory test is designed to evaluate the relative effectiveness of a thinner in a high-solids environment. The data obtained using this procedure should be compared to data using a reference thinner in the same series of tests, and should not be interpreted as thinner performance in actual drilling fluid formulations.

7.1.2 A 28.0 % (mass fraction) suspension of neutralized calcium bentonite is prepared with deionized or distilled water and aged at room temperature for 1 week or more. A laboratory barrel portion (350 mL) of the suspension is treated with 5 lb/bbl (14.3 kg/m³) of chrome or ferrochrome lignosulfonate for the uncontaminated tests and with 8 lb/bbl (22.8 kg/m³) of the reference thinner for contaminated [5 lb/bbl (14.3 kg/m³) gypsum and 4 lb/bbl (11.4 kg/m³) sodium chloride] tests and the pH adjusted to 9.5 with sodium hydroxide. Viscometric properties are measured initially and after hot-rolling for 16 h at 150 °F (65 °C). Filtration volume is measured after hot-rolling. After completing the measurements on the hot-rolled samples, the suspension’s pH is adjusted to 9.5 and aged for 16 h in a stainless steel cell at 350 °F (175 °C). The cell is pressurized to 500 psi (3450 kPa) with nitrogen prior to heating. Viscometric properties of the suspension are measured after high-temperature aging as removed from the cell. The pH of the suspension is readjusted to 9.5, and viscometric properties are re-measured and the filtration volume is determined.

7.1.3 The effectiveness of the test sample is evaluated by comparing the results with the performance of a reference sample of lignosulfonate or other thinner.

7.2 Reagents and apparatus

7.2.1 Calcium bentonite (CAS 67479-91-8)

7.2.2 Sodium chloride (CAS number 7647-14-5), 99 % minimum purity.
7.2.3  **Gypsum** (CAS number 7778-18-9), 92 % minimum Ca\(_2\)SO\(_4\).2H\(_2\)O, 85 % through 100 mesh.

7.2.4  **Sodium hydroxide** (CAS number 1310-73-2) CP, used to make a solution such that 1 mL contains 0.25 g NaOH.

7.2.5  **Deionized or distilled water**.

7.2.6  **Standard or reference thinner**.

7.2.7  **Mixer**, capable of operation at 11,500 r/min ±300 r/min under load, with a single, corrugated impeller approximately 1.0 in. (25.4 mm) in diameter.

The impeller shall be replaced when a mass loss of approximately 10 % occurs. The original blade mass is about 5.5 g and should have been recorded when the impeller was placed in service.

7.2.8  **Container**, for mixing, with air-tight cover.

7.2.9  **Balance**, 3 kg capacity to accuracy ±0.01 g.

7.2.10  **Drilling fluid balance**.

7.2.11  **Viscometer**, in accordance with API RP 13B-1.

7.2.12  **Transformer**, variable speed, type 116.

7.2.13  **pH meter**, accurate to 0.1 pH unit.

7.2.14  **Minute timer**, accurate to 0.1 min over the test interval.

7.2.15  **Filter press**, in accordance with API RP 13B-1.

7.2.16  **Filter paper**, hardened, low-ash grade.

7.2.17  **Spatula**.

7.2.18  **Aging cells**, high-temperature, stainless steel with pressure connector, in accordance with Clause 14.3.

7.2.19  **Drying oven**, regulated to 220 °F ±5 °F (105 °C ±3 °C).

7.2.20  **Jar**, 500 mL, glass canning.

7.2.21  **Burette**, graduated to 0.1 mL.

7.2.22  **Nitrogen source**, at a pressure of 500 psi (3450 kPa) or greater.

7.2.23  **Syringe**, 10 mL, graduated to 0.2 mL.

7.2.24  **Mixer**, capable of operating at 7500 r/min ±500 r/min under load and designed so that the bearings are not exposed to the test fluid.

7.2.25  **Roller oven**, regulated to 150 °F ±5 °F (65 °C ±3 °C), preferably with gravity convention of air (forced-air circulation).

7.2.26  **Aging oven**, static, capable of maintaining a temperature of 350 °F ±5 °F (175 °C ±3 °C).

7.3  **Procedure for Moisture Content Test of Calcium Bentonite**

7.3.1  Determine the moisture content of the calcium bentonite as given in 7.3.2 and 7.4.

7.3.2  Weigh 10 g ±0.01 g of clay and dry to a constant mass at a temperature of 220 °F ±5 °F (105 °C ±3 °C). Cool the sample in a desiccator and weigh.

7.4  **Calculation of Moisture Content of Calcium Bentonite**

Calculate the moisture content, \(w_h\), expressed as a percent mass fraction, from Equation (11):

\[
w_h = 100 \frac{m_o - m_d}{m_o} \tag{11}
\]

where
is the mass of original sample, expressed in grams;

is the mass of dry sample, expressed in grams.

**7.5 Preparation of Drilling Fluid Base for Calcium Bentonite Test**

7.5.1 Prepare a 500 g suspension by dispersing 140 g of calcium bentonite (dry basis) in deionized or distilled water. The amount of water required is 360 g minus the amount of water in the clay as determined in 7.4. The clay should be added to the water slowly, while stirring the suspension with the mixer.

7.5.2 Shear the suspension for 2.5 h, seal the container airtight so that no moisture can escape and age overnight (16 ±2 h) at room temperature. On the following day, stir for 30 min and determine the solids content of the suspension as in 7.5.3.

7.5.3 Weigh 10 g ±0.01 g of suspension and dry at a temperature of 220 °F ±5 °F (105 °C ±3 °C). Cool the sample in a desiccator and weigh. Repeat this test in until the final two results agree within ±0.1 % by mass.

7.5.4 The solids content should be 28.0 % ±0.1 % by mass. If the solids content is too low, let the suspension stand overnight (16 ±2 h) exposed to the atmosphere so that some of the moisture can evaporate. Then determine the solids content again as in 7.5.3. Repeat until the solids content is 28.0 % ±0.1 % by mass. If the solids content is too high, see 7.6.2.

**7.6 Calculation of Solids Content of Calcium Bentonite**

7.6.1 Calculate the solids content, \( w_s \), expressed as a percent mass fraction of the solids, from Equation (12):

\[
w_s = 100 \frac{m_d}{m_o}
\]

where

is the mass of dry sample, expressed in grams;

is the mass of original sample, expressed in grams.

7.6.2 If the solids content is too high, add deionized water. Calculate the additional mass of water, \( m_h \), expressed in grams, required for solids adjustment from Equation (13):

\[
m_h = m_l - 100 \frac{m_s}{w_s}
\]

where

is the specified mass of water required to correct the solids content

is the specified mass of the suspension, expressed in grams (500 g in this procedure);

is the total mass of solids added, expressed in grams [140 g calcium bentonite (dry basis) in this procedure];

is the mass fraction of the solids, expressed in percent.

**EXAMPLE** Solids content of the sample is determined as equal to 29.1 %, which is too high.

\[
m_h = 500 - 100 \frac{140}{29.1}
\]

\[
m_h = 500 - 481.1
\]
\[ m_h = 18.9 \text{ g water required to correct solids content.} \]

### 7.7 Determination of Rheological Properties of Calcium Bentonite sample

#### 7.7.1
To 350 mL of the calcium bentonite suspension, add 1.75 g sodium chloride and stir for 3 min at 11,500 r/min ±300 r/min with a mixer.

#### 7.7.2
Add 5 g of the thinner being tested. Stir the sample for 1 min.

#### 7.7.3
Add sufficient caustic soda (1 mL = 0.25 g NaOH) so that the pH measures 9.5 ±0.1 after 20 min total stirring time.

#### 7.7.4
Scrape down the sides of the jar at least once to ensure proper mixing. The mixing speed shall be kept at 7500 r/min ±500 r/min. After 20 min total stirring time, remove the drilling fluid from the mixer. Check to see that the pH is 9.5 ±0.1.

#### 7.7.5
Stir for 1 min and measure the flow properties with a viscometer. Record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings, and the values for initial gel, pH and total NaOH used.

#### 7.7.6
Record the values for initial gel, pH and total volume of NaOH used in 7.7.3 and 7.7.4.

#### 7.7.7
Cap the jar tightly and hot-roll for 16 h at 150 °F (65 °C).

#### 7.7.8
Cool the hot-rolled drilling fluid to room temperature. Readjust the pH.

#### 7.7.9
Again take the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings, and the readings for the initial gel, pH, 10 min gel and filtrate.

#### 7.7.10
Place the drilling fluid in a stainless steel high-temperature aging cell. Pressurize the aging cell to 500 psi (3450 kPa) with nitrogen and test for leaks by immersing in water and examining for bubbles. Place the cell in a 350 °F (175 °C) oven for 16 h.

#### 7.7.11
Remove the cell, cool to room temperature, and transfer the drilling fluid to a pint jar.

#### 7.7.12
Measure and record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings. Readjust the pH to 9.5 ±0.1 and re-measure the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min properties and the filtrate volume.

#### 7.7.13
To 350 mL of the base drilling fluid, add 5 g gypsum, 4 g sodium chloride, and 8 g of the thinner being evaluated. Stir into the drilling fluid for 20-30 s by raising, lowering and turning the jar by hand while the mixer is running at 11,500 r/min ±300 r/min.

#### 7.7.14
Place the jar into the jar holder. Set the timer for 20 min stirring time. After an elapsed time of 1 min, add a sufficient volume of NaOH solution (1 mL = 0.25 g NaOH) rapidly with a syringe to give a pH of 9.5 ±0.1 at the end of the 20 min stirring time. The quantity of NaOH varies with each type of thinner used and should be determined by trial and error.

#### 7.7.15
After stirring 5 min, remove the sample from the mixer and scrape down the sides of the jar.

#### 7.7.16
Replace the sample on the mixer and continue stirring for a total elapsed time of 20 min at 11,500 r/min ±300 r/min.

#### 7.7.17
At the end of 20 min elapsed time, remove the drilling fluid from the mixer and measure the pH; if the pH is 9.5 ±0.1, stir for 1 min at 11,500 r/min ±300 r/min and measure the flow properties with the viscometer. If the pH is not 9.5 ±0.1, discard the sample and repeat the test.

#### 7.7.18
Measure and record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings, and the values for initial gel, pH and the total NaOH used.

#### 7.7.19
Cap the jar tightly and hot-roll for 16 h at 150 °F (65 °C).

#### 7.7.20
Cool the hot-rolled drilling fluid to room temperature and stir for 5 min at 11,500 r/min ±300 r/min.

#### 7.7.21
Record the 600 r/min, 300 r/min, 200 r/min, 100 r/min and 6 r/min readings, the values for the initial gel, the pH and the filtrate.
7.7.22 Adjust the pH to 9.5 with NaOH and place in a stainless steel high-temperature aging cell. Pressure the aging cell to 500 psi (3450 kPa) with nitrogen and test for leaks by immersing in water. Place the cell in an oven at 350 °F (175 °C) for 16 h.

7.7.23 Remove the cell, cool and transfer the drilling fluid to a pint jar. Stir in mixer for 5 min at 11,500 r/min ±300 r/min.

7.7.24 Measure and record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings following the procedure outlined in API 13B-1. Record the pH.

7.7.25 Then adjust the pH to 9.5 with NaOH and re-measure the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min properties and the filtrate volume. If the pH is above 9.5, do not adjust and record pH.

7.8 Calculation of Thinner Efficiency

Calculate the thinner efficiency, \( e \), expressed as a percent of the performance of the test sample relative to the performance of a reference sample of lignosulfonate or other thinner, as given in Equation (14).

\[
e = 100 \frac{p_s}{p_r}
\]  

(14)

where

- \( p_s \) is the performance, for example yield point or gel strength as determined in accordance with API RP 13B-1, of the test sample;
- \( p_r \) is the performance, for example yield point or gel strength as determined in accordance with API RP 13B-1, of the reference thinner.

8 Potassium Ion Content—Ion-selective Electrode Method

8.1 Principle

8.1.1 Potassium compounds, primarily the chloride and, to a lesser degree, the hydroxide compounds, are used in drilling fluids to aid in the stabilization of the formation of clays and/or shales. The ion-selective electrode (ISE) method can provide accurate, quantitative data on filtrate samples having a range of potassium ion concentration from a few milligrams per liter to saturation.

8.1.2 The measurement of potassium ion concentration by the ISE method is analogous to a pH measurement on a filtrate sample and can be performed with almost the same ease and simplicity. Both procedures rely on the measurement of the difference in potential between a sensing and a reference electrode. Depending on the instrumentation used, this signal is presented as either the actual potential, measured in millivolts, or in some form of direct readout of concentration.

8.1.3 For monovalent ions such as hydrogen and potassium, the change in potential between a sensing and a reference electrode is theoretically 59 mV for a ten-fold change in the activity of the ion being measured. The actual change is normally somewhat lower, usually about 56 mV ±2 mV for the potassium electrode system. This value is defined as the slope of the electrode system.

8.1.4 It should be noted that the actual property being measured is activity, rather than concentration. Since the activity of any ionic species is strongly influenced by the total ionic strength of the solution, it is feasible to use the two terms interchangeably so long as the total ionic strength is held constant. This procedure provides for a constant background total ionic strength by the addition of sodium chloride solution to all standards and samples.

8.1.5 Under the conditions of this test method, none of the materials normally present in drilling fluid filtrate samples has been found to interfere. Errors arising from sodium contamination are eliminated by providing a high constant background level of sodium through the use of sodium chloride as the ISA solution. The relatively high dilution reduces all other potential interference to a negligible level.
8.2 Reagents and Apparatus

8.2.1 Potassium Chloride, standard solution, $\rho = 1$ g/L.

This solution is readily available from most laboratory supply firms, or can be prepared by dissolving 1907 g reagent grade KCl in deionized or distilled water and diluting to 1 L.

When preparing the solution, first dry the KCl in an oven at 220 °F ± 5 °F (105 °C ± 3 °C) until constant mass is achieved and store in a desiccator prior to preparation of the standard solution.

8.2.2 Sodium Chloride, solution, $c_{NaCl} = 5$ N.

Dissolve 292 g reagent grade sodium chloride in deionized or distilled water and dilute to 1 L. This solution is used as an ISA and to provide a high, constant background sodium concentration.

8.2.3 Sodium Chloride, solution, $c_{NaCl} = 0.1$ N, saturated with the silver ion.

Dilute 2 mL of the 5 N sodium chloride solution to 100 mL and add silver nitrate solution dropwise until a cloudiness persists. This solution is used as the internal filling solution for the single-junction reference electrode.

8.2.4 Specific-ion meter or pH meter.

Any of a wide variety of commercially available instruments is suitable, so long as it can provide a millivolt readout with a resolution of at least 0.1 mV.

Either analog or digital readout is acceptable, but the preferred type of instrument is a model with a digital readout and controls that permit direct indication of concentration after proper calibration.

8.2.5 Potassium ISE, potassium electrode.

A two-part electrode, with a reusable body and a disposable, screw-on sensing module. Only the module, not the entire electrode, requires replacement. Average service life of each module is six months.

8.2.6 Reference electrode, a single-junction reference electrode.

NOTE Both the sensing and reference electrodes are available with durable epoxy bodies.

8.2.7 Magnetic stirrer, with coated stirring bars.

8.2.8 Beakers, 150 mL, of borosilicate glass or plastic.

8.2.9 Pipettes, volumetric, various sizes for preparing dilutions of samples and standards.

The use of microliter pipettes with disposable plastic tips is particularly convenient for diluting filtrate samples where the quantity of filtrate available is limited or extremely high dilutions are required.

8.2.10 Graph paper, 3-cycle or 4-cycle semi-logarithmic, for preparing calibration curves.

8.2.11 Flasks, volumetric, various sizes for preparing calibration standards and dilutions of filtrate samples.

8.2.12 Oven.

8.3 Preparation of Electrodes

8.3.1 If the two-part electrode is used, remove the sensing module from the vial and screw it firmly into the electrode body, taking care to ensure that the rubber washer is in place.

8.3.2 Shake down the electrode in the manner of a mercury-filled medical thermometer.

8.3.3 Soak the electrode in deionized or distilled water for at least 10 min, then store in a diluted potassium solution containing 10 mg/L to 100 mg/L potassium until ready for use.

8.3.4 By means of either an eye-dropper or syringe, fill the single-junction reference electrode with the 0.1 N sodium chloride (saturated with silver ion) solution (see 8.2.4).

8.3.5 If other types of sensing or reference electrodes are used, follow the instructions of the manufacturer when preparing them for use.
8.4 Operational Check of Electrode System

8.4.1 The two properties of the electrode system having the greatest influence on accuracy and reproducibility are the slope and the stability. The slope is defined as the change in potential observed when the concentration changes by a factor of 10. The stability of the readings is an indication of the amount of noise (rapidly, continuously changing readings) and drift (readings slowly changing in one direction).

8.4.2 When performing the initial calibration with the standard solutions according to the instructions in either Error! Reference source not found. or Error! Reference source not found., record the actual measured potential for the 10 mg/L and the 100 mg/L standard solutions. For a new potassium electrode (or sensing module), the difference between these two readings shall be between 54 mV and 59 mV. The slope normally decreases with the age of the equipment. Replace the electrode (or module) when the slope reaches a value of approximately 48 mV.

8.4.3 If, when performing the initial calibration, there is an indication of noise or drift, refer to the troubleshooting clause of the electrode manual. There are a number of possible causes for these effects with any electrode system, and the cause is normally found only by a process of elimination. If the analyst is unable to achieve a stability level that meets the manufacturer’s specifications, replace the sensing electrode (or module).

8.5 Measurements using a Meter with Direct Concentration Readout Capability

8.5.1 Prepare a 100 mg/L potassium calibration solution and a 10 mg/L potassium calibration solution by serial dilution of the 1 g/L standard solution.

8.5.2 Add the ISA solution (5 N sodium chloride) to each calibration solution at the rate of 2 mL of ISA per 100 mL of standard.

8.5.3 Transfer the calibration solutions to 150 mL beakers. Place the 10 mg/L calibration solution on the magnetic stirrer and stir at a moderate rate. Immerse the electrodes.

8.5.4 Wait for a stable reading (normally 1-2 min), then adjust the controls to display the potassium concentration of the calibration solution following the procedure described in the meter instruction manual.

8.5.5 Remove the electrodes, rinse with distilled water, blot dry, and place in the 100 mg/L calibration solution. Stir at a moderate rate.

8.5.6 Wait for a stable reading, then adjust the controls to display the potassium concentration of the calibration solution. The meter and electrode system are now calibrated.

8.5.7 Prepare a 1:1000 dilution of each drilling fluid filtrate sample.

Drilling fluid concentrations are frequently much greater than that measureable by potassium electrode monitors. High dilution performed in this method allows these devices to reliably measure potassium concentrations in the samples, which can then be extracted to the drilling fluid being tested.

8.5.8 Transfer 100 mL of diluted sample to a 150 mL beaker, add 2 mL ISA and stir at a moderate rate.

8.5.9 Rinse the electrodes with distilled water, blot dry, immerse in the sample, and wait for a stable reading. The value of the reading displayed on the meter is the diluted sample’s potassium concentration. This value, multiplied by the dilution factor (1000 in this case), gives the original sample’s potassium concentration.

NOTE Concentration values that are above or below the concentrations of the standard solutions for calibration are still reliable, since the 10 mg/L and 100 mg/L standard solutions are near the midpoint of the region of linear response of the potassium electrode.

8.6 Measurements with Instruments that provide either a Digital or an Analog Readout in Millivolts
8.6.1 Prepare 100 mg/L, 10 mg/L, and 1 mg/L potassium calibration solutions by serial dilution of the 100 mg/L standard solution.

8.6.2 Add the ISA solution (5 N sodium chloride) to each standard solution at the rate of 2 mL of ISA per 100 mL of calibration solution.

8.6.3 Transfer the calibration solutions to 150 mL beakers. Place the 1 mg/L calibration solution on the magnetic stirrer and stir at a moderate rate. Place the electrodes in the standard solution.

8.6.4 Adjust the controls of the instrument so that the readout is in millivolts; wait until the reading is stable, and record the reading.

8.6.5 Remove electrodes, rinse, blot dry and place in the 10 mg/L calibration solution. When the reading is stable, record the reading.

8.6.6 Repeat step 8.6.5 with the 100 mg/L calibration solution.

8.6.7 Plot the millivolt readings on the linear axis and potassium concentration, in milligrams per liter, on the logarithmic axis of standard 3-cycle or 4-cycle semi-logarithmic paper. The instrument is now calibrated.

8.6.8 Transfer 100 mL of the filtrate sample diluted 1:1000 to a 150 mL beaker, add 2 mL of the ISA solution and stir at a moderate rate. Place the electrodes in the solution.

8.6.9 Record the reading when stable and determine the concentration of the diluted sample by use of the calibration curve. This value, multiplied by the dilution factor, gives the potassium content of the original sample.

All measurements should be at ambient temperature. Some magnetic stirrers generate considerable heat, and it may be necessary to place a layer of insulating material (plastic or cardboard) between the stirrer and beaker. Between measurements, rinse electrodes and blot dry with tissue. After immersion in the sample or standard solutions, check the electrode end for air bubbles and remove if present. Always read and carefully follow the manufacturer’s instructions regarding the care, maintenance, storage, servicing, and troubleshooting of the instrument and electrodes.

9 Calcium Ion Content—Ion-selective Electrode Method

9.1 Principle

9.1.1 Calcium is present to some extent in the liquid phase of all water-based drilling fluids. The concentration level can vary from several percent by mass to a few milligrams per liter. It may be intentionally added in the form of lime, gypsum, or calcium chloride, or it may be introduced through the make-up or dilution water, as an impurity in various products or from drilled formations. Regardless of the concentration level or the origin of the calcium, it is usually necessary or desirable to monitor the calcium content on a quantitative basis.

9.1.2 The ion-selective electrode (ISE) method is a rapid, simple, and accurate method for the measurement of calcium concentrations in filtrate samples. Unlike the EDTA titrimetric method, no sample treatment is required for highly colored or turbid samples. None of the materials normally present in drilling fluids have been found to interfere with this test method. The combination of relatively high dilution, ionic-strength adjustment through addition of KCl solution, and adjustment of the pH to 5.0 ensures maximum conversion of calcium to ionic form and effectively eliminates or minimizes interference.

9.1.3 This method is intended for the analysis of solution samples only and is not suitable for the analysis of whole drilling fluid samples.

9.2 Reagents and Apparatus

9.2.1 Calcium Carbonate, standard solution, $\rho = 1$ g/L.
Weigh 2.497 g of reagent grade calcium carbonate on an analytical balance, add 50 mL water and dissolve by the dropwise addition of concentrated hydrochloric acid (HCl) (approximately 10 mL is required). When dissolution is complete, dilute to 1 L in a volumetric flask.

NOTE This solution is also readily available from laboratory supply houses.

9.2.2 **Potassium Chloride**, solution, \( c_{KCl} = 4 \ N \).

Dissolve 298 g of reagent grade KCl in deionized water and dilute to 1 L. This solution is used as an ISA solution.

9.2.3 **Acetate Buffer Solution**, pH 5.0.

Dissolve 136 g of sodium acetate trihydrate (\( \text{NaC}_2\text{H}_3\text{O}_2\cdot3\text{H}_2\text{O} \)) in about 500 mL of deionized water. Add 57.5 mL of glacial acetic acid (\( \text{HC}_2\text{H}_3\text{O}_2 \)) and dilute to 1 L. This solution is used to adjust the pH of all standard and sample solutions to a pH of 4.8 to 5.2.

9.2.4 **Potassium Chloride**, solution, \( c_{KCl} = 4 \ N \), saturated with silver.

Transfer about 50 mL of the 4 N KCl (see 9.2.2) to a 2 oz plastic bottle and add silver nitrate solution dropwise until a cloudiness persists. This solution is used as the filling solution for the reference electrode.

NOTE This solution is also readily available from chemical supply houses.

9.2.5 **pH Meter**, capable of providing a millivolt readout with a resolution of at least 0.1 mV.

There are many instruments available, ranging from battery-operated, hand-held portable units for field use to sophisticated and versatile “research” instruments programmed to provide readouts in concentration units. Readouts may be either analog or digital. The preferred type for laboratory use is a research model with a digital readout and controls that provide direct indication of concentration after proper calibration.

9.2.6 **Calcium ISE**, calcium electrode.

A two-part electrode, with a reusable epoxy body and a disposable, screw-on sensing module.

9.2.7 **Reference electrode**, single-junction

9.2.8 **Magnetic stirrer**, with PTFE-coated stirring bars, approximately 3/8 in. (10 mm) in diameter and 1.0 in. (25 mm) long.

9.2.9 **Beaker**, 150 mL, of borosilicate glass or inert plastic.

9.2.10 **Pipettes**, volumetric, Class A, various sizes are required for preparing dilutions of standards and samples.

The use of a good-quality microliter pipette with disposable tips is particularly convenient for quantitative transfer of volumes of 1 mL and smaller.

9.2.11 **Flasks**, volumetric, Class A, various sizes for preparing calibration standards and dilutions of filtrate samples.

9.2.12 **Graph paper**, 3-cycle or 4-cycle semi-logarithmic, for preparing calibration curves.

9.3 **Preparation of Electrodes**

9.3.1 Remove the electrode’s sensing module from the vial and screw it firmly into the electrode body, taking care to ensure that the rubber washer is in place.

9.3.2 Shake down the electrode in the manner of a mercury-filled medical thermometer.

9.3.3 Soak the electrode in deionized or distilled water for at least 15 min, then store in a dilute calcium solution, containing 10 mg/L to 100 mg/L calcium, until ready for use.
9.3.4 By means of either an eye dropper or syringe, fill the single-junction reference electrode with the 4 \textit{N} potassium chloride solution (saturated with silver ion).

9.3.5 If other types of sensing or reference electrode are used, follow the instructions of the manufacturer when preparing them for use.

9.4 Operational Check of Electrode System

9.4.1 The two properties of the electrode system that have the greatest influence on accuracy and reproducibility are the slope and the stability. The slope is defined as the change in potential observed when the concentration changes by a factor of 10. The stability of the readings is an indication of the amount of noise (rapidly, continuously changing readings) and drift (readings slowly changing in one direction).

9.4.2 When performing the initial calibration with the standard solutions according to the instructions in either 9.5 or 9.6, record the actual measured potential for the 1 mg/L and the 10 mg/L standard solutions. For a new calcium electrode (or sensing module), the difference between these two readings shall be between 25 mV and 29 mV. The slope normally decreases with age of the equipment. Replace the module when the slope reaches a value of approximately 23 mV.

9.4.3 If there is an indication of noise or drift when performing the initial calibration, refer to the troubleshooting clause of the electrode manual. There are a number of possible causes for these effects with any electrode system, and only a process of elimination normally finds the cause. Replace the sensing electrode (or module) if the analyst is unable to achieve a stability level that meets the manufacturer’s specifications.

9.5 Measurements using a Meter with Direct Concentration Readout Capability

9.5.1 Prepare a 10 mg/L and a 1 mg/L calcium calibration solution by serial dilution of the 1 g/L standard calcium solution (9.2.1).

9.5.2 Add the ISA solution [4 \textit{N} potassium chloride (9.2.2)] to each calibration solution at the rate of 2 mL of ISA solution per 100 mL. Add 1 mL of the acetate buffer to each.

9.5.3 Transfer the calibration solutions to 150 mL beakers. Place the 1 mg/L calibration solution on the magnetic stirrer and stir the solution at a moderate rate. Place the electrodes in the solution.

9.5.4 Wait for a stable reading (normally 1-2 min), then adjust the controls to display the calcium concentration of the calibration solution following the procedure described in the meter instruction manual.

9.5.5 Remove the electrodes, rinse with distilled water, blot dry and place in the 10 mg/L calibration solution. Stir at a moderate rate and repeat 9.5.4. The electrode is now calibrated.

9.5.6 Prepare a 1:100 dilution of each drilling fluid filtrate sample.

9.5.7 Transfer 100 mL of diluted sample to a 150 mL beaker, add 1 mL of the acetate buffer, solution, add 2 mL ISA solution, and stir at a moderate rate.

9.5.8 Rinse electrodes with distilled water, blot dry, immerse in the sample and wait for a stable reading. The value displayed on the meter is the calcium concentration in the diluted sample. This value, multiplied by the dilution factor (100 in this case), gives the calcium concentration in the original sample.

NOTE Concentration values above or below the concentrations of the calcium standards used for calibration are still reliable, since the response of the calcium electrode is linear from about 0.2 mg/L to above 1 g/L.

9.6 Measurements with Instruments that provide either a Digital or an Analog Readout in Millivolts

9.6.1 Prepare 100 mg/L, 10 mg/L, and 1 mg/L calcium calibration solutions by serial dilution of the 1 g/L standard calcium solution (9.2.1).
9.6.2 Add the ISA solution [4 N potassium chloride (9.2.2)] to each calibration solution at the rate of 2 mL of ISA solution per 100 mL. Add 1 mL of the acetate buffer solution to each calibration solution.

9.6.3 Transfer the calibration solutions to 150 mL beakers. Place the 1 mg/L calibration solution on the magnetic stirrer and stir the solution at a moderate rate. Place the electrodes in the calibration solution.

9.6.4 Adjust the controls of the instrument so that the readout is in millivolts, wait until the reading is stable and record the reading.

9.6.5 Remove electrodes, rinse, blot dry and place in the 10 mg/L standard solution. When the reading is stable, record the reading.

9.6.6 Repeat step 9.6.5 with the 100 mg/L calibration solution.

9.6.7 Plot the millivolt readings on the linear axis and concentration, in milligrams per liter, on the logarithmic axis of standard 3-cycle or 4-cycle semi-logarithmic paper. The instrument is now calibrated.

9.6.8 Transfer 100 mL of the sample diluted 1:100 to a 150 mL beaker, add 2 mL of the ISA solution, add 1 mL of the acetate buffer, place the electrodes in the solution and stir at a moderate rate.

9.6.9 Record the reading when stable, and determine the concentration of the diluted sample by use of the calibration curve. This value multiplied by the dilution factor gives the calcium content of the original sample.

All measurements should be at ambient temperature. Some magnetic stirrers generate considerable heat, and it may be necessary to place a layer of insulating material (plastic or cardboard) between the stirrer and beaker. Between measurements, rinse electrodes and blot dry with tissue. After immersion in sample or standard solutions, check the electrode end for air bubbles and remove if present. Always read and carefully follow the manufacturer’s instructions regarding the care, maintenance, storage, servicing, and troubleshooting of the instrument and electrodes.

10 Sodium Ion Content—Ion-selective Electrode Method

10.1 Principle

10.1.1 This procedure provides an accurate, quantitative method for determining the sodium content in water-based drilling fluids.

10.1.2 Sodium is present to some extent in all water-based fluid systems. Concentrations can range from less than 100 mg/L to over 100 g/L in saturated salt systems. This method is applicable to all concentrations from less than 100 mg/L to over 100 g/L in saturated salt systems. This method is applicable to all concentrations. None of the other materials normally present in drilling fluids have been found to interfere under the conditions of this test. The combination of dilution and treatment with ISA solution effectively eliminates interference.

10.1.3 The sodium electrode, because of its glass membrane construction, is not as sturdy as liquid membrane electrodes such as those used for potassium and calcium. Strict adherence to the specific practices described in the manufacturer’s instruction manual is necessary to obtain reliable data.

10.1.4 The electrode should only be stored in the designated storage solution. Never expose the glass sensing membrane of the electrode to fresh water. Always rinse with the rinse solution designated in the applicable electrode manual. Never wipe or blot electrodes dry with a cloth or tissue. Let the rinse solution drain, then shake off as much as possible of the remainder.

10.1.5 Verify calibration at least every 2 h during use, using a freshly prepared calibration solution for each recalibration.

10.1.6 Minimize the use of glass containers and apparatus and, when glass is used, ensure that it is a chemically resistant glass. Store standards and reagents in plastic containers when possible.

10.1.7 Recondition the electrode when the response reading is sluggish or a noticeable drift is detected. Be sure to follow instructions in the applicable electrode manual when reconditioning.
10.2 Reagents and Apparatus

10.2.1 Sodium chloride (CAS number 7647-14-5), standard solutions: 1 g/L sodium, 100 mg/L sodium, 10 mg/L sodium and 1 mg/L sodium; the 1 mg/L solution should be prepared immediately before use by serial dilution of the 10 mg/L standard or the 100 mg/L standard.

These standards are prepared by dissolving sodium chloride, NaCl, in deionized water.

10.2.2 Sodium ISA solution, \( c = 4 \, N \) of both ammonium chloride, \( \text{NH}_4\text{Cl} \), (CAS number 12125-02-9), and ammonium hydroxide, \( \text{NH}_4\text{OH} \) (CAS number 1336-21-6).

10.2.3 Electrode rinse solution, add 10 mL of ISA solution to water and dilute to 1 L.

10.2.4 Ammonium bifluoride, \( (\text{NH}_4\text{HF}_2) \) (CAS number 1341-49-7), for the sodium electrode reconditioning solution, 0.1 \( N \).

10.2.5 Sodium electrode storage solution, \( c = 5 \, N \) in sodium chloride, \( \text{NaCl} \) (CAS number 7647-14-5), 0.08 \( N \) in ammonium chloride, \( \text{NH}_4\text{Cl} \) (CAS number 12125-02-9), and 0.08 \( N \) in ammonium hydroxide, \( \text{NH}_4\text{OH} \) (CAS number 1336-21-6).

10.2.6 Reference electrode filling solution, \( c = 2 \, N \) ammonium chloride, \( \text{NH}_4\text{Cl} \) (CAS number 12125-02-9), used for filling the reference electrode.

Do not use the potassium chloride filling solutions normally supplied with the reference electrode.

10.2.7 pH meter, capable of providing a millivolt readout with a resolution of at least 0.1 mV, or a combination pH/ISE meter with provisions for direct concentration readout.

Use of the combination instrument is recommended.

10.2.8 Sodium ion-selective electrode.

10.2.9 Reference electrode, reference electrode.

10.2.10 Magnetic stirrer, with plastic-coated stirring bars.

10.2.11 Beakers, 150 mL, made of HDPE or LDPE plastic or chemically-resistant glass (plastic is preferred).

10.2.12 Pipettes, volumetric, Class A, various sizes for preparing dilutions of standards and samples.

The use of microliter pipettes with disposable plastic tips is recommended for measuring and transferring small volumes (1.0 mL or less).

10.2.13 Flasks, volumetric, Class A, various sizes.

10.2.14 Graph paper, 3-cycle or 4-cycle semi-logarithmic paper for preparing calibration curves when using a meter that provides readouts in millivolts only.

10.3 Preparation and Operational Check of the Electrode System

10.3.1 Remove the sodium electrode from the shipping container and rinse thoroughly with the electrode rinse solution.

10.3.2 Soak the electrode for at least 2 h, preferably overnight, in the sodium electrode storage solution.

10.3.3 Fill the reference electrode using the 2 \( N \) ammonium chloride solution.

10.3.4 Connect electrodes to the meter and rinse with the electrode rinse solution.

10.3.5 Transfer 100 mL of the 10 mg/L sodium standard solution to a 150 mL beaker, stir solution on the magnetic stirrer at a moderate rate while adding 10 mL of the ISA solution, and then immerse the electrodes.

10.3.6 Operate the meter in the millivolts mode. When a stable reading is obtained, record the electrode potential in millivolts.
10.3.7 Remove electrodes and rinse with the electrode rinse solution. Do not wipe or blot the electrode dry. Place 100 mL of the 100 mg/L sodium standard in a 150 mL beaker, add a stirring bar and stir at a moderate rate. Add 10 mL of the ISA solution to the standard sodium solution while stirring.

10.3.8 Immerse electrodes and record the potential, in millivolts, once a stable reading is obtained.

10.3.9 The difference between the two readings is the slope of the electrode. This difference should be in the range of 54-59 mV. If it is less than 54 mV, recondition the sodium electrode according to the procedure detailed in the electrode instruction manual.

10.4 Measurements using a Meter with a Direct Concentration-readout Capability

10.4.1 Transfer 100 mL each of the 100 mg/L sodium standard (10.2.1) and the 10 mg/L sodium standard (10.2.1) to 150 mL beakers.

10.4.2 Add 10 mL of the ISA solution (10.2.2) to each standard solution.

10.4.3 Place the 10 mg/L calibration solution on the magnetic stirrer and stir at a moderate rate. Immerse electrodes and wait for a stable reading (normally 1-2 min), then adjust the controls on the instrument to display the sodium concentration of the standard.

10.4.4 Remove electrodes, rinse, place in the 100 mg/L calibration solution and, when a stable reading is obtained, adjust the controls to display the sodium concentration of the standard.

10.4.5 Prepare either a 1:100 or a 1:1000 dilution with deionized or distilled water of each filtrate sample, depending on the expected sodium concentration. The sodium concentration in the diluted sample in which the measurement is made shall be between 10 mg/L and 100 mg/L. The meter is now calibrated.

10.4.6 Transfer 100 mL of the diluted sample to a 150 mL beaker, add 10 mL of ISA solution, immerse electrodes and wait for a stable reading. The value displayed is the sodium concentration of the diluted sample. This value multiplied by the dilution factor gives the sodium concentration in the original filtrate.

If the concentration of sodium in the diluted sample is greater than 100 mg/L, make an additional dilution in order to provide a concentration between 10 mg/L and 100 mg/L. If it is between 1 mg/L and 10 mg/L, the value obtained is expected to be reliable since the response of the electrodes is normally linear to sodium concentrations less than 1 mg/L.

10.5 Measurements using a Meter with readout in millivolts

10.5.1 Transfer 100 mL each of the 100 mg/L, 10 mg/L, and 1 mg/L sodium standard solutions to a separate 150 mL beaker.

10.5.2 Add 10 mL of ISA solution to each standard.

10.5.3 Place the 1 mg/L calibration solution on the magnetic stirrer and stir at a moderate rate. Immerse electrodes, and record the reading in millivolts when stable.

10.5.4 Repeat step 10.5.3 with the 10 mg/L calibration, then the 100 mg/L calibration solution. Be sure to rinse electrodes thoroughly with water when moving from one calibration solution to another.

10.5.5 Plot the millivolt readings on the linear axis and the sodium concentration, in milligrams per liter, on the logarithmic axis of 3-cycle or 4-cycle semi-logarithmic paper. The meter is now calibrated.

10.5.6 Transfer 100 mL of sample diluted 1:100 or 1:1000 to a separate 150 mL beaker and add 10 mL of ISA solution to each.

10.5.7 Stir at a moderate rate, immerse electrodes, and record reading when stable. Determine the sodium concentration in the diluted sample by use of the calibration curve prepared in 10.5.5. This value, multiplied by the dilution factor, gives the sodium concentration of the original sample.

10.5.8 If the calculated concentration of sodium in the diluted sample is outside the 1 mg/L to 100 mg/L range, prepare another sample, using a dilution that provides a concentration within this range.

11 Preparation and Evaluation of Non-Aqueous Fluids (NAFs)
11.1 Principle

11.1.1 Occasionally, evaluation and comparison of NAFs are required. The tests described are designed to provide a standardized method for preparing and evaluating the slurries under the same conditions. Significant changes in rheological properties, electrical stability, or HTHP fluid loss can indicate instability of the test drilling fluid. It is recommended that the test apparatus and procedures be agreed upon between vendor and client prior to initiating testing.

11.1.2 The test methods provide specific mixing and heat-aging procedures in an attempt to minimize inter-laboratory variations.

11.1.3 Where possible, the test methods and apparatus used are in accordance with API RP 13B-2.

11.1.4 The test protocol is deliberately simple, designed only to provide a means of comparing the fluids' response to heat aging and to the incorporation of reasonable levels of various contaminants that can be expected during drilling, such as drill solids, seawater and formation brines.

11.1.5 Other methods exist for evaluating NAFs.

11.2 Reagents and Apparatus

11.2.1 Sea salt, mixed according to the manufacturer's instructions. The standard seawater mix shall conform to ASTM D 1141, or equivalent.

11.2.2 Standard Evaluation base clay. See API 13A for material specifications.

11.2.3 Thermometer, metal contact, with a range 32-220 °F (0 °C-05 °C).

11.2.4 Balance, accuracy ±0.01 g.

11.2.5 Mixer, high shear, heavy-duty laboratory model with a square-hole, high-shear impeller screen. The screen shall be replaced when a mass loss of approximately 10 % has occurred through wear. The bushing shall be replaced when any appreciable play can be felt between it and the shaft. Similarly, the shaft shall be replaced when the diameter at the bushing has decreased by approximately 1 %.

11.2.6 Mixer, capable of operating at 10,000 r/min ±100 r/min under load, with a single sine-wave impeller system, approximately 1.0 in. (2.5 cm) in diameter at low gear/speed setting. The impeller shall be replaced when a mass loss of approximately 11 % to 13 % has occurred through wear.

11.2.7 Container, conical, stainless steel, approximately 2 L capacity, with base internal diameter of 3.0 in. (76 mm) and top internal diameter of 6.0 in. (152 mm).

11.2.8 Mixing Container, approximately 7 1/8 in. (180 mm) deep, d = 3 ¾ in. (97 mm) at the top and 2 ¾ in. (70 mm) at the bottom.

11.2.9 Spatula.

11.2.10 Tachometer, optical, magnetic induction or equivalent.

11.2.11 Mud balance, in accordance with API RP 13B-2.

11.2.12 Viscometer, direct-indicating, in accordance with API RP 13B-2.

11.2.13 Cup, viscometer, thermostatically controlled, in accordance with API RP 13B-2.

11.2.14 Filter press, high-temperature/high-pressure, in accordance with API RP 13B-2.

11.2.15 Electrical stability meter, in accordance with API RP 13B-2.

11.2.16 Oven, for heat-aging, capable of maintaining a stable temperature and equipped with a roller system, or equivalent, to keep the aging cells in motion.

11.2.17 Cell, heat-aging, preferably stainless steel, capacity 350-500 mL, capable of pressurization by means of a valve system, if necessary.

11.2.18 Water bath.

11.3 Mixing of the Initial Drilling Fluid
11.3.1 Prepare four laboratory barrels (one laboratory barrel ≈ 350 mL) of fluid to the required formulation with the following parameters specified:

a) drilling fluid density;

b) oil-water ratio;

c) mass fraction of salts in the brine phase, expressed in percent;

d) rheological properties (ranges are generally specified);

e) HTHP filtrate volume (a range is generally specified);

f) HTHP filtrate testing temperature;

11.3.2 Mix the fluid for at least 60 min or until homogenous with a high-shear mixer, fitted with a high-shear, square-hole screen surrounding the impeller.

11.3.3 Starting at ambient temperature, mix the sample in the conical mixing vessel and control the shaft speed during mixing to 6000 r/min ±300 r/min. Use the optical tachometer or magnetic induction tachometer to monitor rotational speed. Measure the rotational speed under actual mixing load and monitor during the period of mixing. Make adjustments as necessary, particularly during and after the addition of the weighting material.

11.3.4 The fluid temperature can be expected to rise because of shear, but do not allow it to exceed a maximum of 150 °F (65 °C) as measured by a metal contact thermometer. Thereafter, control it as necessary by means of a water bath to between 140 °F - 150 °F (60 °C - 65 °C).

11.3.5 If the program of testing requires more than four laboratory barrels, mix any additional volume of drilling fluid in batches of four laboratory barrels using the procedure outlined above.

11.3.6 Blend all batches together to ensure uniformity before commencing the test regime. Blending should be carried out by means of a large spatula or by pouring from one container to another several times.

Blending should not be done by means of a mechanical mixer. The pouring or gentle stirring technique is necessary to ensure that each laboratory barrel is subjected to the same shear per unit time per unit volume.

11.4 Testing the Properties of the Initial Drilling Fluid

11.4.1 The fluid shall be tested after mixing using the techniques in accordance with API RP 13B-2. Tests on the initial fluid shall cover all properties listed below:

a) density at ambient temperature of approximately 68 °F (20 °C);

b) rheological properties at speeds of 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min, and 10 s gel and 10 min gel with the temperature maintained at 120 °F (50 °C);

c) electrical stability at 120 °F (50 °C);

d) HTHP filtrate volume at a temperature of 250 °F (120 °C) and a differential pressure of 500 psi (3450 kPa), or other specified temperature and pressure. Consult API RP 13B-2 for recommended temperature/pressure values.

If there is a requirement for HTHP filtrate volume testing, especially above 250 °F (120 °C), there can be variance in results with different test apparatus and test procedures. It is recommended that the test apparatus and procedures be agreed between vendor and client prior to initiation of testing.

11.4.2 One 350 mL sample (one laboratory barrel) of initial drilling fluid shall be submitted to hot rolling and be used as a blank in all further testing.

11.5 Preparation of the Sample Contaminated by Seawater

11.5.1 To one laboratory barrel of drilling fluid, add 10 % by volume of the standard synthetic seawater mix.
11.5.2 Mix the drilling fluid and synthetic seawater under ambient conditions on a mixer. Add all the seawater over 1 min and carry out a further 4 min of mixing for a total mixing time of 5 min.

11.5.3 Age the sample under the conditions agreed upon by the interested parties.

11.6 Preparation of the Sample Contaminated by Base Evaluation Clay

11.6.1 To one laboratory barrel of drilling fluid, add base evaluation clay on a mixer, stirring at the low-speed setting.

Additions of base evaluation clay should be based on the expected density of the fluid, as follows:
- for a density below 15.0 lbs/gal (1800 kg/m$^3$), add 35 g of base evaluation clay;
- for a density above 15.0 lbs/gal (1800 kg/m$^3$), add 20 g of base evaluation clay.

11.6.2 Mix the drilling fluid and base evaluation clay under ambient conditions on a mixer. Add the clay over a period of 1 min and carry out a further 4 min of mixing for a total mixing time of 5 min.

11.6.3 Age the sample under the conditions agreed upon by the interested parties.

11.7 Preparation of the Sample Contaminated by Mixed-salt Brine

11.7.1 This is an additional contamination sample recommended for those wells where it is considered relevant (e.g. where Zechstein salt is expected). The type of mixed-salt brine to be used in the test will already have been determined by discussion between the interested parties.

11.7.2 A typical mixed-salt brine is Carnallite brine, but any simulated brine can be used. To prepare a Carnallite brine, add 125.0 g magnesium chloride hexahydrate (CAS number 7786-30-3), 14.5 g potassium chloride (CAS number 7447-40-7) and 5.0 g sodium chloride (CAS number 7647-14-5) to 100 g water.

11.7.3 Mix the brine thoroughly on a mixer and extract the volume to be used as a contaminant with a syringe during mixing. This ensures that any undissolved salts are incorporated into the contaminant.

11.7.4 To one laboratory barrel of drilling fluid, add 10% mixed-salt brine by volume on a mixer, stirring at the low-speed setting. Add the brine to the drilling fluid over a period of 1 min, with an additional 4 min mixing. Carry out the mixing at ambient temperature.

11.7.5 Age the sample under the conditions agreed upon by the interested parties.

11.8 Procedure for Hot-rolling

11.8.1 Purge and pressurize the hot-roll cell with nitrogen.

11.8.2 Samples of the initial formulation and of the contaminated drilling fluid shall be hot-rolled for 16 h or other specified time, at 250 °F (120 °C), or other specified temperature, as agreed upon by the interested parties, in a preheated hot-rolling oven.

11.8.3 After hot-rolling, switch off the oven heat and open the oven door.

11.8.4 Roll the samples during the cooling period for at least 30 min prior to removal.

11.8.5 Subsequently, cool in ambient air for an additional 30 min. Then cool the cell to ambient temperature by partially immersing the cell in water.

11.8.6 Stir all aged samples on a mixer for 5 min immediately prior to testing.

11.9 Procedure for Static Aging

11.9.1 Purge and pressurize the aging cell with nitrogen.

11.9.2 Samples of the initial formulation and of the contaminated drilling fluid shall be statically aged for 16 h at 250 °F (120 °C), or other specified temperature, in a preheated aging oven.

11.9.3 After static aging, switch off the oven heat and open the oven door.

11.9.4 Cool the samples for at least 30 min prior to removal.
11.9.5 Subsequently, cool in ambient air for an additional 30 min. Then cool the cell to ambient temperature by partially immersing the cell in water.

11.9.6 Stir all aged samples on a mixer for 5 min immediately prior to testing.

11.10 Procedure for Testing after Heat Aging

The fluid shall be tested after mixing using the techniques in accordance with API RP 13B-2. The procedure for testing the initial fluid shall cover all properties listed below:

a) density at ambient temperature of 68 °F (20 °C);

b) rheological properties at speeds of 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min, and 10 s gel and 10 min gel with temperature maintained at 120 °F (50°C);

c) electrical stability at 120 °F (50 °C);

d) HTHP filtrate volume at a temperature of 250 °F (120 °C) and a differential pressure of 500 psi (3450 kPa), or other specified temperature and pressure. Consult API RP 13B-2 for recommended temperature/pressure values for HTHP testing.

If there is a requirement for HTHP filtrate volume testing, especially above 250 °F (120 °C), note that there can be variance in results with different test apparatus and test procedures. It is recommended that the test apparatus and procedures be agreed upon between vendor and client prior to initiation of testing.

12 Testing Base Fluid Properties used in Non-Aqueous Fluids (NAF)

12.1 General

This clause gives a brief summary of the ASTM testing methods that API Subcommittee 13 on Drilling Fluids Materials has reviewed and found to be acceptable for the testing of base oils commonly used in drilling and completion NAFs.

12.2 Reagents and Apparatus

The reagents and apparatus as specified in the appropriate Standard, ASTM, or IP procedure bulletin shall be used to test a particular base fluid. These reagents and test apparatus are mandatory to perform the standard tests correctly and safely, and to achieve results which meet API requirements. They are described in this Standard in general terms only.

12.3 Density, Relative Density (Specific Gravity), or API Gravity-hydrometer Method (see ASTM D 287 REV B, ASTM D 1298 REV B, and API MPMS Chapter 9.1)

12.3.1 Principle

This method covers the laboratory determination, using a glass hydrometer, of density, relative density (specific gravity), or API gravity of crude petroleum, petroleum products, or mixtures of petroleum and non-petroleum products normally handled as liquids, and having a Reid vapor pressure (see ASTM D 323) of 180 kPa (1.8 bar; 26 psi) or less. Values are measured on a hydrometer at convenient temperatures, readings of density being reduced to 60 °F (16 °C) and readings of relative density (specific gravity) and API gravity to 60 °F (16 °C), in accordance with the petroleum reference tables given in ASTM D 1250 and IP 200. By means of these tables, values determined in any one of the three systems of measurement can be converted to equivalent values in either of the other two, so that measurements may be made in the units of local convenience. Generally, the drilling fluids industry prefers to use the relative density (specific gravity) units.

12.3.2 Summary of the Method

The sample is brought to the specified temperature and transferred to a cylinder that is at approximately the same temperature. The appropriate hydrometer is lowered into the sample. After temperature equilibrium has been reached and the hydrometer is still, the hydrometer scale is read and the temperature of the sample is noted. If necessary, the cylinder and its contents may be placed in a constant-temperature bath to avoid excessive temperature variation during the test.
12.4  Density and Relative Density of Liquids using a Digital Density Meter (see ASTM D 4052)

12.4.1  Principle
This method covers the determination of the density or relative density of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at test temperatures between 60 °F and 95 °F (16 °C and 35 °C). Its application is restricted to liquids with vapor pressures below 11 psi (80 kPa) and viscosities below about 0.015 m²/s at the temperature of test. It should not be applied to samples so dark in color that the absence of air bubbles in the sample cell cannot be established with certainty.

12.4.2  Summary of Method
Approximately 0.7 mL of liquid sample is introduced into the oscillating sample tube of a digital density meter, and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

12.5  Kinematic Viscosity of Transparent and Opaque Oils—Calibrated Capillary Tube Method (see ASTM D445)

12.5.1  Principle
This test method covers the determination of the kinematic viscosity (a measure of the resistance to flow under gravity) of liquid petroleum products, both transparent and opaque, by measuring the time for a given volume of the liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid.

12.5.2  Summary of Method
The time, in seconds, is measured for a fixed volume of the liquid sample to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer. Testing temperatures usually selected are 104 °F and 158 °F (40 °C and 70 °C) for base oils, unless these temperatures can constitute a hazard of fire or flash explosion during the testing.

12.6  Distillation (see ASTM D86)

12.6.1  Principle
This method covers the distillation of motor gasolines, aviation gasolines, aviation turbine fuels, special boiling-point spirits, naphthas, white spirits, kerosenes, gas oils, distillate fuel oils, and similar petroleum products.

12.6.2  Summary of Method
A 100 mL sample is distilled under specified conditions that are appropriate to its nature as shown in ASTM D86 (16th Edition, 2016), Table 1. Systematic observation of the thermometer readings and volumes of condensate are made and, from these data, the results of the tests are calculated and reported.

Important temperature values found are
a)  initial boiling point, when the first drop is condensed,
b)  final boiling point, when maximum temperature is reached, and
c)  dry point, the temperature just as the last drop evaporates from the lowest point in the flask.

12.7  Aniline Point and Mixed Aniline Point (see ASTM D 611)

Caution—Application of ASTM D 611 can involve the use of hazardous materials, operations and apparatus. ASTM D 611 does not purport to address all of the safety problems associated with its
use. It is the responsibility of the user of the method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

12.7.1 Principle
These test methods cover the determination of the aniline point of petroleum products and hydrocarbon solvents. Method 1 (ASTM D 611, Annex A1) is suitable for transparent samples and to samples not darker than No. 6.5 ASTM color as determined by Test Method D 1500 having initial boiling points well above the expected aniline point. Method B (ASTM D 611, Annex A2), a thin-film method, is suitable for samples too dark for testing by method 1. Methods C (ASTM D 611, Annex A3) and D (ASTM D 611, Annex A4) are for samples that can vaporize appreciably at the aniline point.

12.8 Pour Point (see ASTM D 97)
12.8.1 Principle
The test for pour point is intended for use on any petroleum oil. A procedure suitable for black oils, cylinder stock and non-distillate fuel oil is also described in ASTM D 97.

12.8.2 Summary of Method
After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 5 °F (3 °C) for flow characteristics. The lowest temperature at which movement of the oil is observed is recorded as the pour point.

12.9 Flash Point by Pensky-Martens Closed Tester (see ASTM D93)
12.9.1 Principle
This method covers the determination of the flash point by the Pensky-Martens closed-cup tester of fuel oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids. This method is not recommended for testing emulsions of water and oil, such as drilling fluids.

12.9.2 Summary of Method
The oil sample is heated in a cup at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

12.10 Aromatics Content (see IP 391 or ASTM D 5186 or EPA 1654)
12.10.1 Principle
12.10.1.1 General
These methods measure the aromatic content of drilling fluid oils, petroleum distillates, and diesel fuels. The user should realize that analyses of a complex oil by the two methods might not give the same values for aromatic content. The reason for variation is that the two instruments separate aromatic components differently and their detectors function by different principles. Instruments for the IP method are more widely available and less expensive than those required for the ASTM method. The ASTM supercritical fluid chromatography method uses a column and detector system that gives a better representation of the types of aromatics found in complex oils, but it does not measure low concentrations of aromatics. The EPA method uses high performance liquid chromatography (HPLC) with a Ultraviolet (UV) absorption detector.

12.10.2 IP 391
The IP 391 method is suitable for determining monocyclic, bicyclic and tricyclic aromatic fractions in drilling fluid oils and petroleum distillates that boil in the range of 285-680 °F (140-360 °C) with bromine numbers (determined in accordance with ISO 3839) of less than 1.0. This method is appropriate for concentrations from 0.05 % volume fraction to 25 % volume fraction monocyclic aromatics and 0.05 % volume fraction to 5 % volume fraction bicyclic and tricyclic aromatics. Higher concentrations require appropriate dilutions.
NOTE Compounds containing oxygen or nitrogen can cause erroneous results for the IP method.

12.10.2.1 **Summary of the IP 391 Method**

A 5-10 µL sample of oil is injected into a liquid chromatograph that is fitted with a polar separation column with hexane as the mobile phase. The column separates the aromatic components into mono-, bi- and tricyclic fractions that appear as peaks on the chromatogram. The fractions are identified by a differential refractive-index detector as they exit the column. Percentages are determined by integrating the areas under the peaks based on prior calibration with known concentrations of an aromatic compound.

12.10.3 ASTM D 5186

The ASTM D 5186 method is suitable for determining aromatic compounds in fuels with concentrations from 5 % mass fraction to 75 % mass fraction of aromatics.

12.10.3.1 **Summary of the ASTM D 5186 Method**

A 0.05-0.2 µL oil sample is injected into a packed silica column using supercritical carbon dioxide as the mobile phase. Aromatic components in the sample are separated from non-aromatic components by adsorption in the silica column. Components are measured by a flame ionization detector as they exit the column. Concentrations are calculated from peak areas based on prior calibration using known concentrations of an aromatic compound.

12.10.4 EPA Method 1654

The EPA method is suitable for determining aromatic compounds in very low concentrations.

12.10.4.1 **Summary of EPA Method**

An oil sample is diluted in acetonite and a 20 µL aliquot is injected into the HPLC. The PAHs are partially separated by HPLC and detected with the UV detector. Identification of PAH (qualitative analysis) is performed by comparing the response of the UV detector to the response during the retention-time range characteristic of the PAH in diesel oil. PAH is present when a response occurs during this retention-time range. Quantitative analysis is performed by calibrating the HPLC with phenanthrene using an external standard technique, and using the calibration factor to determine the concentration of PAH in the sample.

13 Testing Particle Size Distribution in a Non-aqueous Drilling Fluid using a Laser Diffraction Instrument

13.1 **Testing Summary**

This procedure is an API recommended practice for measuring the particle size distribution of non-aqueous drilling fluids using laser diffraction based instruments. Measurement will involve a recommended sampling protocol followed by dilution of the mud sample in an appropriate carrier fluid, sonication of the sample and data collection.

13.2 **Reagents & Apparatus**

13.2.1 **Isopropanol (IPA)** (technical grade or better)

13.2.2 **Hexane** (technical grade or better)

13.2.3 **Base Fluid** (if IPA or hexane is not available)

13.2.4 **Transfer pipettes** (minimum opening of 3 mm)

13.2.5 **Magnetic stir bar**, 1 in.

13.2.6 **Magnetic stirrer**

13.2.7 **Overhead mixer with single sinewave impeller** (10,000 to 15,000 rpm)

13.2.8 **Glass beaker**, 25 mL

13.3 **Instrument Settings**
13.3.1 If necessary, verify the instrument gives the expected values on the reference material per manufacturer's recommendation.

13.3.2 Set the refractive index of the solids to be measured to 1.6.

13.3.3 If prompted, set the absorption value of the particles to an appropriate number (should be either 0 or 0.01 depending on the instrument. Contact instrument supplier for the recommended value).

13.3.4 If prompted, choose the “General Purpose” calculation model.

13.3.5 Set sonication power to 100%.

13.3.6 If prompted, choose “Irregular particle” as the particle shape to be measured.

13.3.7 Set the measurement duration to 30 s. Perform measurement three times.

13.4 Procedure for Measuring Particle Size Distribution in a Non-aqueous Drilling Fluid using a Laser Diffraction Instrument

13.4.1 Qualitative Sample Dispersion Test prior to Bulk Mixing

13.4.1.1 All PSD measurements are done using IPA as the carrier fluid. If IPA is not available, it is acceptable to use base oil as the carrier fluid.

13.4.1.2 Check the miscibility of mud with IPA. Add a few drops of mud using a transfer pipette to glass beaker/vial containing IPA and stir or shake by hand. Check for any visible mud clumps or mud settling to the bottom. If none is observed, the mud is miscible with IPA; otherwise, the mud should be miscible with hexane.

13.4.1.3 For muds that are miscible with IPA, all sample PSD runs will be carried out in IPA.

13.4.1.4 For muds that are immiscible with IPA, all sample PSD runs will be still carried out in IPA. However, pre-dispersion of mud in hexane is required. Hexane is used to remove base oil from the solids so that the solids can be dispersed in IPA.

13.4.2 Bulk Mixing of Mud sample Preparation and Sampling

13.4.2.1 Remix the mud in the container to obtain best representative sample.

13.4.2.2 Homogenize approximately 400 mL of mud with an overhead mixer for 2 minutes.

13.4.2.3 Fill particle sizing distribution (PSD) instrument with IPA (use base oil when IPA is not available).

13.4.2.4 Adjust circulation speed to 75 % of full speed.

13.4.2.5 Record the PSD background profile of carrier fluid before adding mud when the background is stable.

13.4.2.6 For muds that are miscible with IPA, add homogenized mud drop-wise and slowly to the PSD instrument until laser signal obscuration of 10 ±2 % is observed. Laser signal reading is not instantaneous and mud should be added slowly.

13.4.2.7 For muds that are immiscible with IPA, pre-dispersion of mud in hexane is required. Add three to five drops of mud to a beaker/vial containing 10 mL of hexane and swirl or stir until the sample is fully dispersed. Then add entire hexane solution to circulating reservoir of the PSD instrument, Rinse the beaker/vial well with IPA (< 10 mL) and add solution to circulating reservoir to make sure the entire sample aliquot is transferred. Make sure the obscuration is 8-15 %.

13.4.2.8 Allow sample to circulate inside the PSD reservoir for 1 min.

13.4.2.9 Sonicate sample at 100 % power for 60 s. Sonication typically causes a slight increase in obscuration, this is to be expected, and this increase in obscuration has no impact on the measurement results.

13.4.2.10 If necessary, allow sample to circulate until all evidence of thermal effects from the sonication has disappeared from the PSD curve (this typically takes between 1-5 min depending on the instrument.)
13.4.2.11 Collect three repeat measurements, 30 s duration each, 60 s between each measurement, to ensure the measurement is repeatable.

14 Aging of Water-based Drilling Fluids

14.1 Principle

Drilling fluid aging is the process of allowing a drilling fluid sample that contains all required ingredients and that has been subject to a period of shear to more fully develop its rheological and filtration properties through additional time for the hydration, etc. of its components. The time period needed to more fully develop properties varies from as little as several hours (more commonly overnight (16 ± 2 h) or 16 h) to as much as several days. The aging can be carried out at either ambient or elevated temperatures, depending upon the system under investigation.

It is strongly recommended that this clause be read completely before any elevated-temperature aging of drilling fluid samples is carried out.

The purposes of this clause are:

a) to inform those unfamiliar with drilling fluid formulation and testing of the techniques and apparatus commonly used in a laboratory setting to mix and age drilling fluid systems, and

b) to remind those already familiar with drilling fluid formulation and testing of some basic guidelines and safety considerations.

Users of this clause should familiarize themselves with the types of tests, testing methods and properties measured presented in API RP 13B-1, as well as the methodologies given in this Standard.

This clause is not intended to cover aging technology appropriate to either oil-based drilling fluids or brines used for completion, packer, or workover fluids. Aging of oil-based fluids is discussed in clause 16 of this document.

14.2 Practices Common to Preparation, Handling, and Testing over all Temperature Ranges

14.2.1 Water-based Drilling Fluids and Components

Most drilling fluid formulations contain a base liquid and drilling fluid materials that are dissolved or mechanically dispersed into the liquid to form a homogeneous fluid. The resulting fluid may contain one or more of the following: water-dispersible (“soluble”) polymers or resins, clays or other insoluble-but-dispersible fine solids and soluble salts. The fluids are mixed or sheared for a time sufficient to achieve a homogeneous mixture and are then set aside to “age”. Aging is done under conditions that vary from static to dynamic and from ambient to highly elevated temperatures.

14.2.2 Mixing, Blending, and/or Shearing Devices

14.2.2.1 Drilling fluid formulations are commonly mixed with various shearing devices that may be either fixed speed or variable speed. The motor turns a mixing shaft with rounded “propellers”, sharp blades, wave-form shapes or others. Single-shaft or multiple-shaft shearing devices are used. Nozzle shear devices are also used to prepare some formulations.

14.2.2.2 These shearing devices vary widely in the amount of shear imparted. This means that long shearing times can be required for low-shear devices to achieve complete dissolution/hydration of fluid components. High-shear devices can produce nearly completely homogenized drilling fluid blends in a few minutes. Aging of drilling fluid samples tends to minimize differences in properties that can result from shearing treatment.

14.2.3 Hydration of bentonite and other clays is a function of ionic composition of the base fluid, particle size, shear, and time.

14.2.3 Property Ranges of Drilling Fluids or Base Fluids, emphasizing pH Values
14.2.3.1 The pH of drilling fluid formulations containing bentonite clays usually does not fall below 8.5 unless acidic materials are added. Except for some drilling fluid systems viscosified with certain water-soluble polymers, the pH of these formulations is usually raised above pH 8.5 with alkalinity-control agents, such as sodium or potassium hydroxide (caustic soda or caustic potash), calcium hydroxide (lime), or magnesium oxide.

**CAUTION**—It is strongly recommended to wear eye protection whenever drilling fluids and chemicals are formulated, handled or tested, in order to avoid injury.

14.2.3.2 Fluid alkalinity is lowered by the reaction of hydroxide groups with aluminosilicates (clays), gradually at ambient temperature and rapidly at elevated temperature. Some drilling fluid additives require that alkalinity be maintained within a certain narrow and at times, elevated ranges in order to function at optimum levels. Therefore, pH levels are often raised after aging if there has been a substantial pH drop.

14.2.4 Drilling Fluid Sample Storage, Disposal

14.2.4.1 Drilling fluid formulations or base fluid (bentonite slurries) are frequently kept until used up or until properties move outside acceptable ranges. Some laboratories routinely keep unused portions of test drilling fluids at ambient temperatures for an arbitrary time, such as 1 week or 1 month. Prolonged storage is best done in refrigerated units with a maximum storage time of up to 3 months at 40 °F (4 °C).

14.2.4.2 Drilling fluid samples that contain certain organic materials or polymer products subject to fermentation (i.e. starches, biopolymers, etc.) should either have a preservative added or be discarded after a suitable time that precedes the expected onset of biological degradation.

14.2.4.3 Discard unused samples in an environmentally appropriate manner, based upon known ingredients. Clays, most polymers derived from cellulosics and starches, and lignites pose no special threat to living organisms and may be sent to normal landfill sites.

14.2.4.4 Dispose of drilling fluids containing toxic materials as appropriate for chemical waste.

14.3 Metal Aging Cells

14.3.1 General

Metal aging cells and caps should have serial numbers provided by the manufacturer which can be used to verify specifications. Cells and caps containing the same serial number shall be used together.

**Caution**—Aluminum bronze cells are not recommended for drilling fluids at any temperature.

14.3.2 Characteristics of metal aging cells

14.3.2.1 Since aging temperatures are often selected to be near the estimated or anticipated bottom hole temperatures, the aging cells chosen shall be designed and constructed in such a way as to exceed the pressure and temperature requirements of the aging tests.

14.3.2.2 Beyond the issue of being able to meet the temperature and pressure requirements of specific aging conditions, the choice of the proper metal is complex. It depends upon both the ultimate temperature to which the sample and cell are exposed and the salinity or corrosivity of the fluid sample.

14.3.2.3 The largest percentage of aging between 150 °F and 400 °F (65 °C and 205 °C) is done in cells constructed from various grades of stainless steel. Sometimes high-carbon-content steel cells are used if there is a strong desire to simulate field conditions where “mild steel” pipe is in use and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e. they have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

14.3.2.4 For prolonged exposure to elevated salinity at high temperatures [e.g. 20 g/L chlorides at 400 °F (205 °C)], cells constructed from premium metals or having a teflon insert can be desirable.

14.3.2.5 A key consideration in the use of metal aging cells at elevated temperatures is that care shall be taken to ensure that the cells are not overfilled. When enclosed liquids expand with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as the liquid hydraulically
loads the cell cap. Leaving gaps of 1.5-2 in. (40-50 mm) between the top of the liquid and the cell cap, or not filling the cell to more than 85-90% of its volume appear to be adequate precautions for safe operation during most aging conditions between 150 °F and 400 °F (65 °C and 205 °C).

Warning—Overfilling can result in catastrophic cell failure and injury

14.3.3 Additional Safety Considerations for Metal Aging Cells

14.3.3.1 It is necessary to emphasize that metal aging cells shall be used to age only drilling fluids containing the usual types or classes of drilling fluid additives. Experimental material, when high-temperature compatibility with drilling fluid chemicals/minerals is in doubt, shall not be aged at elevated temperatures in metal cells. Such compatibility should be tested in an apparatus such as an autoclave designed for extreme-pressure service.

WARNING—Testing unknown compositions of chemical additives in a heating cell can result in catastrophic failure and injury

14.3.3.2 The only known catastrophic failures (explosions) of stainless steel aging cells resulted either from the cells being used inappropriately as chemical-reaction autoclaves (instead of for drilling fluid aging) or from the cells being overfilled.

14.3.3.3 The reported types of failure of metal aging cells, either from inadequate inspection and maintenance or from prolonged use for aging of drilling fluids, were leaks which developed in the cells. The drilling fluids leaked into the aging ovens but caused no danger to laboratory personnel.

14.3.3.4 Rupture disks may be placed in the cell caps if there is concern about cell failure. Since the use of rupture disks lowers the pressure rating of the metal aging cell, conduct tests at temperatures that keep the pressure lower.

14.3.4 Maintenance of Metal Aging Cells

14.3.4.1 Cell bodies and cell caps should have serial numbers recorded whenever they are used along with the cell contents. Further, it is suggested that separate sets of metal aging cells be maintained for testing fresh-water fluids, high-chloride fluids, and oil-based fluids. Cells exposed only to fresh water should have the longest useful life.

14.3.4.2 Stainless steel aging cells tend to experience chloride stress corrosion cracking or pitting in environments where there is prolonged exposure at temperatures over 140 °F (60 °C) to fluid with substantially elevated chloride levels (over several thousand milligrams per liter). Such concerns are legitimate, but there are a number of mitigating circumstances existing in the technology of drilling fluid aging. These tend to lessen the stress corrosion.

14.3.4.3 One drilling fluids aging practice that mitigates accelerated corrosion is the washing and visual inspection of metal aging cells after each use to see if they have been affected by the exposure. The onset of pitting and cracking corrosion is often detected at such a time. Affected stainless steel cells are then usually sand-blasted to remove corrosion spots. The cells can be then returned to useful service.

14.3.4.4 Even when no obvious corrosion in a metal aging cell is detected, historical experience has shown that periodic grit-blasting of the cells removes developing problem sites, keeps the surface passive and significantly increases the useful life of the cell.

14.3.4.5 Other mitigating circumstances include the following.

a) The cells are used intermittently and cleaned, and are not used in continuous-process exposures.

b) The cells used in drilling fluids testing are made from metal bar stock and are not welded. The absence of the stress point that is present in welded or cast cell bodies mitigates against the likelihood or severity of corrosion that can be experienced under certain aggressive conditions of exposure;

c) The drilling fluid formulations have a high pH and, typically, a low oxygen content.

d) Using teflon liners in stainless steel cells, or premium metal cells.

14.3.4.6 Inertness and chemical compatibility in High-temperature aging cells
14.3.5 Chemical compatibility of Materials with Metal aging cells

14.3.5.1 As mentioned in 14.3.3, do not subject materials of unknown or suspicious reactivity to high-temperature aging in cells containing drilling fluids.

14.3.5.2 Do not include materials that are known to produce, or suspected of initiating or taking part in, highly exothermic reactions in heat-aged drilling fluid formulations.

14.3.5.3 Exclude materials that are known or suspected strong redox (oxidation-reduction) reagents from heat-aged drilling fluid formulations.

14.3.6 Inertness of Metal Aging Cells to Chemicals

14.3.6.1 Metal aging cells of certain nickel-based alloys provide the greatest degree of inertness to potentially corrosive conditions. Verify with the cell manufacturer to determine inertness.

14.3.6.2 Iron can be leached from the various stainless steels by high-chloride fluids at high temperatures. This removal of iron causes pits and cracks after stainless steel cells are exposed to severe temperature/chloride conditions.

14.3.7 Use of Lining Materials in Metal Aging Cells

14.3.7.1 Some investigators have found that test results from high-chloride drilling fluids containing water-soluble polymers might not be reproducible when stainless steel vessels are used. Iron appears to be leached from the cell bodies causing these anomalies.

14.3.7.2 In order to use the more economical stainless steel cells under aggressive fluid aging conditions, lining materials should be inserted in the aging cells. Materials that have received widespread acceptance as liners include PTFE, glass, and ceramic.

14.3.7.3 Many PTFE liners can be safely used up to 400 °F (205 °C) and offer a high degree of inertness to a wide variety of drilling fluid formulations. Verify material specifications with the manufacturer.

14.3.7.4 Glass liners can be attacked at the high pH levels used in many drilling fluid formulations. Glass liners are also susceptible to attack under certain highly saline conditions at elevated temperatures.

14.3.8 Considerations Regarding Metal Plating to Enhance Contamination Resistance of Aging Cells

14.3.8.1 Since drilling fluid samples aged at elevated temperatures often become viscous or even hard, it is sometimes necessary to scrape solidified sample material from the cells. Even when taking great care, there is a risk that scratches will occur when removing solidified drilling fluids. Therefore, plated cells are not recommended for aging drilling fluids at elevated temperatures.

14.4 Drilling Fluid Sample Preparation and Aging at Ambient Temperature

14.4.1 Sample Preparation

Drilling fluids may be mixed at ambient temperatures in a variety of open containers made from metal, plastic, or glass. Little or no incompatibility exists between normal, water-based drilling fluid and these container materials under ambient temperature conditions.

14.4.2 Apparatus

14.4.2.1 Containers

Plastic containers and lids may be made from polyethylene, polypropylene, or other suitably inert, mechanically strong and durable materials. A wide variety of metal containers may be used at ambient temperatures. Commonly used metal includes various grades of stainless steel or mild carbon steel. More inert metal formulations may be used but these are expensive for ambient temperature use.

Metal containers/cells such as aluminum bronze are not recommended for any drilling fluid formulations. Reactions between the bronze aging cells and numerous drilling fluid systems or drilling fluid materials have been observed.
a) crack-resistant glass, such as home canning jars or equivalent jars or bottles;
b) Plastic, (buckets are commonly used for large batches)
c) Metal

Caution—Aluminum bronze cells are not recommended for drilling fluids at any temperature.

14.4.2 Coverings or lids for the glass vessels, commonly plastic or metal, often lined with plastic, rubber, enamel, or other relatively “inert” material.
14.4.3 Mixer, in accordance with 14.2.2.
14.4.4 Balance, accuracy ±0.01 g.

14.4.3 Procedure for Aging Water-based Drilling Fluids at Ambient Temperatures

14.4.3.1 After the initial shearing/blending stages of drilling fluid base or full sample preparation, most additional aging at ambient temperatures is done statically in metal, plastic, or glass containers, capped to prevent loss of moisture.
14.4.3.2 Prepared samples are left overnight (16 h ±2h) or for as long as necessary to reach stable or desirable properties.
14.4.3.3 Rolling or tumbling (rotating) the prepared samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.
14.4.3.4 The fluid properties can continue to change until the components are fully hydrated, and some components react further in the aqueous suspension due to secondary reactions between various components or due to bacterial attack on sensitive materials. Biocides may be added to prolong shelf-life of these sensitive formulations.

14.5 Drilling Fluid Sample Preparation and Aging at Moderate Temperatures [Ambient to 150 °F (65 °C)]

14.5.1 Sample Preparation

Drilling fluid samples to be aged at moderate temperatures are mixed at ambient temperatures in accordance with 14.4.3. Additional materials may be added to slurries that have already been aged at ambient or elevated temperatures.

14.5.2 Apparatus

14.5.2.1 Containers, glass, plastic or metal.

Most of the acceptable containers used for ambient aging (14.4.2.1) have been found acceptable for static or dynamic aging at temperatures up to and including 150 °F (65 °C).

Caution—Aluminum bronze cells are not recommended for drilling fluids at any temperature.

14.5.2.2 Covers, to seal the container.
14.5.2.3 Oven, regulated to 150 °F (65 °C), or other suitable temperature.
14.5.2.4 Mixer, in accordance with 14.2.2.

14.5.3 Procedure for Water-based Drilling Fluid Aging at Moderate Temperatures

14.5.3.1 Aging at moderate temperature is usually done for one of the following reasons:
a) to hasten the equilibrium hydration level of clays and/or polymers in the fluid system;
b) to expose the fluid to thermal conditions similar to field conditions.

14.5.3.2 For exposure to moderate temperature, drilling fluid samples are placed in one of a wide variety of commercially available or custom-built ovens. These ovens range from being tabletop, portable models capable of holding only a few one-pint samples, to large floor-standing units having large capacities. These ovens also vary from static units to those equipped with rollers or pulley systems to roll the containers or to tumble (rotate) strapped-in cells.
14.5.3.3 Preliminary studies indicate that the methods of heating and cooling the drilling fluid samples (i.e. preheating the oven versus no preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequate air circulation), and the exact heating exposure time affect the fluid properties. To optimize repeatability between test runs, use the same heating and cooling method and heat-exposure time for all samples in a series of comparative tests.

14.5.3.4 During aging at temperatures through 150 °F (65 °C), some glass and plastic vessels containing drilling fluids, especially those with elevated salinity (e.g. seawater with a salinity of approximately 19 g chlorides per liter) develop modest pressures in the air overlying the drilling fluid. After 15-30 min of heating, these vessels are removed from the oven and the lids carefully loosened to allow excess pressure to escape (“burping”), the lids restored tightly and the containers replaced in the oven to continue heating to the desired temperature. Sometimes a second check for pressure build-up is advisable.

14.5.4 Maintaining Fluid Properties at Moderate Temperatures

As mentioned in 14.2.3.2, alkalinity levels drop with time when exposed to clay (or other drilling fluid additives that exhibit weakly acidic behavior). The reaction between clays and hydroxyl ions accelerates with increasing temperature. For drilling fluids that are exposed to elevated temperatures, it is necessary that alkalinites be more closely monitored and, if desired, more frequently adjusted than for those at lower temperatures. Purging and blanketing samples with nitrogen gas appears to impart strong benefits in reducing oxidative degradation of polymer-treated samples. The use of nitrogen leads to better reproducibility in the testing of polymer drilling fluids.

14.6 Water-based Drilling Fluid Sample Preparation and Aging at Elevated Temperatures [over 150 °F (65 °C)]

14.6.1 Sample Preparation

Drilling fluid samples for aging at elevated temperatures are also blended at ambient temperatures in accordance with 14.4.3. Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperature may be added to slurries that have already been aged at ambient or elevated temperatures.

14.6.2 Apparatus

14.6.2.1 Oven, capable of maintaining a temperature of 350 °F ±5 °F (175 °C ±3 °C) or greater.

14.6.2.2 Aging cells, constructed from metals suitable for exposure to drilling fluids at elevated temperatures.

Caution—Aluminum bronze cells are not recommended for drilling fluids at any temperature.

14.6.3 Procedure for Aging Water-based Drilling Fluids at Elevated Temperatures

14.6.3.1 If the correct match of cell metallurgy and drilling fluid is established for a given temperature regime, then the samples may be statically or dynamically aged in a suitable oven, in accordance with 14.5.

14.6.3.2 The time and temperature for hot rolling should be selected based on anticipated well conditions.

14.6.3.3 For high-temperature wells over 300 °F (149 °C), the usual 16 h aging interval is a reasonable simulation of the time a drilling fluid is left in the hole during a bit trip. For longer operations, such as extended electric well-logging runs, a 48-72 h aging period may be more appropriate.

14.6.3.4 In choosing a test temperature, it should be noted that fluid left in the hole can take many hours to even approach the actual bottomhole temperature, since the circulating fluid cools the wellbore. Therefore, a test temperature below the bottomhole temperature can be a more realistic simulation of downhole conditions for bit-trip periods.
14.6.3.5 Dynamic aging should be performed in rolling or rotating ovens capable of achieving and safely maintaining temperatures of 150-400 °F (65-205 °C). A 16 h minimum aging time is recommended for such exposures.

14.6.3.6 For static aging, any qualified oven or a dynamic oven, with the rollers/rotating mechanisms switched off, may be used. Static aging at elevated temperatures simulates the conditions of a drilling fluid that is left downhole during regular rig operations.

NOTE A meaningful test to use in evaluating long-term gelation of heat-aged drilling fluids is the shear strength test found in API RP 13B-1.

14.6.4 Contrast between Drilling Fluid Material Performance in Laboratory Testing and Field Environments

Be cautious about interpreting results from laboratory aging studies. Field results can be at variance with the results obtained from aging tests conducted under the conditions in the laboratory environment. Laboratory testing is to be used as a guideline and not an absolute predictor of drilling fluid performance.

15 Aging of Non-Aqueous Drilling Fluids

15.1 Principle

This clause is written to inform those unfamiliar with formulating and testing drilling fluids about the techniques and apparatus commonly used in a laboratory to mix and age NAF systems. It is also a reminder of the basic guidelines and safety considerations in working with aging practices. It is strongly recommended that this clause be read in its entirety before any aging of NAF drilling fluid samples at elevated temperatures is attempted.

Drilling fluid aging is the process in which a drilling fluid sample, previously extensively sheared, is allowed to more fully develop rheological and filtration properties. The time period to develop properties may vary at either ambient or elevated temperatures.

Users of this clause should familiarize themselves with the types of tests, testing methods and properties found in API RP 13B-2, and the methodologies described in this Standard.

This clause is not intended to cover aging technology of water-based drilling fluids or brines used for completion, packer, or workover fluids.

15.2 Apparatus

15.2.1 Shearing Device, used to blend drilling fluid formulations.

Shearing devices may be either fixed speed or variable speed. The motor turns a mixing shaft with rounded "propellers", corrugated impellers, sharp blades or wave-form shapes, among others. Either single-shaft or multiple-shaft devices are used. Nozzle shearing devices are preferred by some users.

Shearing devices can vary widely in the amount of shear that they impart. Aging of drilling fluid samples tends to minimize differences in properties that can result from being prepared by different shearing devices.

15.2.2 Glass Jars and Bottles, used to prepare batches up to 4 L in volume.

Crack-resistant glass containers, such as jars supplied for home canning purposes, or equivalent types of jars/bottles that are equipped with metal screw-on caps, commercially available straight-sided round jars, and wide-mouth round jars are suitable. The latter types are commonly used in rolling ovens up to about 150 °F (65 °C). All types may be securely fastened in tumbling ovens operating below 150 °F (65 °C).

Heat-resistant containers made of Pyrex or equivalent materials are also used to temperatures of up to 150 °F (65 °C).

Coverings/lids for glass vessels are made of plastic or metal. Inert seals or liners (e.g. those made of PTFE) are recommended for the best long-term use, regardless of the composition of the lid.
15.2.3 Plastic Jars and Containers.

Acceptable plastic materials for containers and lids include polyethylene, polypropylene, or other suitably inert, mechanically strong and durable materials. These materials might not be compatible with some oil-based fluids. Most oil-based drilling fluids may be mixed and stored in thick-walled polyethylene commercial buckets.

Thin-walled plastic containers, such as milk, soft drink or juice bottles, are not recommended.

15.3 Metal Aging Cells

15.3.1 General

Metal aging cells and caps should have serial numbers provided by the manufacturer which can be used to verify specifications. Cells and caps containing the same serial number shall be used together.

Caution—Aluminum bronze cells are not recommended for drilling fluids at any temperature.

15.3.2 Characteristics of metal aging cells

15.3.2.1 Since aging temperatures are often selected to be near the estimated or anticipated bottom hole temperatures, the aging cells chosen shall be designed and constructed in such a way as to exceed the pressure and temperature requirements of the aging tests.

15.3.2.2 Beyond the issue of being able to meet the temperature and pressure requirements of specific aging conditions, the choice of the proper metal is complex. It depends upon both the ultimate temperature to which the sample and cell are exposed and the salinity or corrosivity of the fluid sample.

15.3.2.3 The largest percentage of aging between 150 °F and 400 °F (65 °C and 205 °C) is done in cells constructed from various grades of stainless steel. Sometimes high-carbon-content steel cells are used if there is a strong desire to simulate field conditions where “mild steel” pipe is in use and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e. they have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

15.3.2.4 For prolonged exposure to elevated salinity at high temperatures [e.g. 20 g/L chlorides at 400 °F (205 °C)], cells constructed from premium metals or having a teflon insert can be desirable.

15.3.2.5 A key consideration in the use of metal aging cells at elevated temperatures is that care shall be taken to ensure that the cells are not overfilled. When enclosed liquids expand with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as the liquid hydraulically loads the cell cap. Leaving gaps of 1.5-2 in. (40-50 mm) between the top of the liquid and the cell cap, or not filling the cell to more than 85-90 % of its volume appear to be adequate precautions for safe operation during most aging conditions between 150 °F and 400 °F (65 °C and 205 °C).

Warning—Overfilling can result in catastrophic cell failure and injury

15.3.3 Additional Safety Considerations for Metal Aging Cells

15.3.3.1 It is necessary to emphasize that metal aging cells shall be used to age only drilling fluids containing the usual types or classes of drilling fluid additives. Experimental material, when high-temperature compatibility with drilling fluid chemicals/minerals is in doubt, shall not be aged at elevated temperatures in metal cells. Such compatibility should be tested in an apparatus such as an autoclave designed for extreme-pressure service.

WARNING—Testing unknown compositions of chemical additives in a heating cell can result in catastrophic failure and injury

15.3.3.2 The only known catastrophic failures (explosions) of stainless steel aging cells resulted either from the cells being used inappropriately as chemical-reaction autoclaves (instead of for drilling fluid aging) or from the cells being overfilled.

15.3.3.3 The reported types of failure of metal aging cells, either from inadequate inspection and maintenance or from prolonged use for aging of drilling fluids, were leaks which developed in the cells. The drilling fluids leaked into the aging ovens but caused no danger to laboratory personnel.
15.3.3.4 Rupture disks may be placed in the cell caps if there is concern about cell failure. Since the use of rupture disks lowers the pressure rating of the metal aging cell, conduct tests at temperatures that keep the pressure lower.

15.3.4 Maintenance of Metal Aging Cells

15.3.4.1 Cell bodies and cell caps should have serial numbers recorded whenever they are used along with the cell contents. Further, it is suggested that separate sets of metal aging cells be maintained for testing fresh-water fluids, high-chloride fluids, and oil-based fluids. Cells exposed only to fresh water should have the longest useful life.

15.3.4.2 Stainless steel aging cells tend to experience chloride stress corrosion cracking or pitting in environments where there is prolonged exposure at temperatures over 140 °F (60 °C) to fluid with substantially elevated chloride levels (over several thousand milligrams per liter). Such concerns are legitimate, but there are a number of mitigating circumstances existing in the technology of drilling fluid aging. These tend to lessen the stress corrosion.

15.3.4.3 One drilling fluids aging practice that mitigates accelerated corrosion is the washing and visual inspection of metal aging cells after each use to see if they have been affected by the exposure. The onset of pitting and cracking corrosion is often detected at such a time. Affected stainless steel cells are then usually sand-blasted to remove corrosion spots. The cells can be then returned to useful service.

15.3.4.4 Even when no obvious corrosion in a metal aging cell is detected, historical experience has shown that periodic grit-blasting of the cells removes developing problem sites, keeps the surface passive and significantly increases the useful life of the cell.

15.3.4.5 Other mitigating circumstances include the following.

a) The cells are used intermittently and cleaned, and are not used in continuous-process exposures.

b) The cells used in drilling fluids testing are made from metal bar stock and are not welded. The absence of the stress point that is present in welded or cast cell bodies mitigates against the likelihood or severity of corrosion that can be experienced under certain aggressive conditions of exposure;

c) The drilling fluid formulations have a high pH and, typically, a low oxygen content.

d) Using teflon liners in stainless steel cells, or premium metal cells.

15.3.4.6 Inertness and chemical compatibility in High-temperature aging cells

15.3.5 Chemical compatibility of Materials with Metal aging cells

15.3.5.1 As mentioned in 14.3.3, do not subject materials of unknown or suspicious reactivity to high-temperature aging in cells containing drilling fluids.

15.3.5.2 Do not include materials that are known to produce, or suspected of initiating or taking part in, highly exothermic reactions in heat-aged drilling fluid formulations.

15.3.5.3 Exclude materials that are known or suspected strong redox (oxidation-reduction) reagents from heat-aged drilling fluid formulations.

15.3.6 Inertness of Metal Aging Cells to Chemicals

15.3.6.1 Metal aging cells of certain nickel-based alloys provide the greatest degree of inertness to potentially corrosive conditions. Verify with the cell manufacturer to determine inertness.

15.3.6.2 Iron can be leached from the various stainless steels by high-chloride fluids at high temperatures. This removal of iron causes pits and cracks after stainless steel cells are exposed to severe temperature/chloride conditions.

15.3.7 Use of Lining Materials in Metal Aging Cells

15.3.7.1 Some investigators have found that test results from high-chloride drilling fluids containing water-soluble polymers might not be reproducible when stainless steel vessels are used. Iron appears to be leached from the cell bodies causing these anomalies.
15.3.7.2 In order to use the more economical stainless steel cells under aggressive fluid aging conditions, lining materials should be inserted in the aging cells. Materials that have received widespread acceptance as liners include PTFE, glass, and ceramic.

15.3.7.3 Many PTFE liners can be safely used up to 400 °F (205 °C) and offer a high degree of inertness to a wide variety of drilling fluid formulations. Verify material specifications with the manufacturer.

15.3.7.4 Glass liners can be attacked at the high pH levels used in many drilling fluid formulations. Glass liners are also susceptible to attack under certain highly saline conditions at elevated temperatures.

15.3.8 Considerations Regarding Metal Plating to Enhance Contamination Resistance of Aging Cells

15.3.8.1 Since drilling fluid samples aged at elevated temperatures often become viscous or even hard, it is sometimes necessary to scrape solidified sample material from the cells. Even when taking great care, there is a risk that scratches will occur when removing solidified drilling fluids. Therefore, plated cells are not recommended for aging drilling fluids at elevated temperatures.

15.3.9 Ovens

Ovens may be static or dynamic [equipped with rollers or pulley systems which provide rolling of jars or cells or tumbling (rotating) of strapped-in cells]. Static ovens range from portable (tabletop) models that can hold only a few sample jars or cells, to larger (floor-standing) multi-shelf models that can hold dozens of samples.

It is expected that ovens be rated to operate at temperatures from 150-500 °F (65-260 °C).

Adequate air circulation within ovens to ensure uniform temperature distribution is essential to the validity of aging.

CAUTION—Ovens used with oil-based fluids should not have exposed heating elements that can cause ignition of combustible materials in the event of sample leakage.

15.4 Practices Common to Preparation, Handling, and Testing over all Temperature Ranges

15.4.1 NAF Drilling Fluids and their Components

15.4.1.1 NAF drilling fluids contain one or more base liquids and drilling fluid materials dissolved or mechanically dispersed into the liquid to form a homogeneous fluid. The resulting fluid may contain oil-dispersible, soluble polymers or resins, and organophilic clays or other insoluble but dispersible fine solids.

15.4.1.2 The NAF drilling fluids may be “all-oil” drilling fluids, i.e. formulated without an aqueous phase, or “invert” emulsions where the oil is the continuous phase and the emulsified water phase is the dispersed phase. Typically the water phase contains sodium chloride (NaCl) or calcium chloride (CaCl₂).

15.4.1.3 Fluids are mixed or sheared to achieve a homogeneous mixture of the components and then set aside to “age”. Aging may be done under static or dynamic conditions and from ambient to elevated temperatures.

15.4.2 Mixing/blending/shearing Devices

Shearing devices vary widely in the amount of shear that they impart. Long shearing times can be required for low-shear devices to achieve fairly complete dissolution or solvation of fluid components. High-shear devices can produce nearly completely dispersed blends in a few minutes.

Aging of drilling fluids samples tends to minimize property differences that can result from using different shearing devices.

15.4.3 Property Ranges of NAF Drilling Fluids or their Base Fluids

15.4.3.1 Base oils vary widely in their inherent viscosities. These viscosities also vary differently with temperature for different base fluids. Therefore, the drilling fluid properties vary widely.
15.4.3.2 Drilling properties can also be affected by the presence of different emulsifier packages (or the lack of them for all-oil fluids), and/or organophilic clays and/or the oil-soluble viscosifying polymers used to provide suspending characteristics.

15.4.3.3 Some emulsifiers or wetting agents require the presence of lime to be activated into calcium soaps. Sometimes lime is also added to control acid gases, such as hydrogen sulfide or carbon dioxide, which can be encountered in drilling operations or from the high-temperature breakdown of some drilling fluid materials.

15.4.4 Sample Storage and Disposal
15.4.4.1 NAF drilling fluid samples are usually not subject to rapid deterioration. It is considered good practice to limit testing reliance on samples that are over 1 month old. Some laboratories routinely keep unused portions of test drilling fluids at ambient temperatures for an arbitrary time, such as 1 week or 1 month. Prolonged storage is best done in refrigerated units with a maximum storage time of up to 3 months at 40 °F (4 °C).

15.4.4.2 Fluid samples should be monitored to ensure that acceptable properties are maintained.

15.4.4.3 Unused samples should be discarded in accordance with local statutory regulations and best practices based upon their known ingredients.

15.5 Non-aqueous Drilling Fluid Aging at Ambient Temperatures

15.5.1 Containers for Preparation and Storage
The drilling fluids may be mixed at ambient temperature in a variety of open containers made from metal, plastic or glass, in accordance with 15.2. Incompatibility can exist between oil-based drilling fluids and some plastic materials. There are not likely to be problems with metal or glass containers.

15.5.2 Procedure for Aging at Ambient Temperatures
15.5.2.1 After the initial shearing/blending stages of drilling fluid base or full sample preparation, most additional aging at ambient temperatures is done statically.

15.5.2.2 Prepared samples are left overnight (16 h ±2 h) or for as long as necessary, to reach stable or desirable properties.

15.5.2.3 Rolling or tumbling (rotating) of the prepared samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.

15.5.2.4 Oil-based drilling fluids are routinely aged at ambient temperatures in metal or glass containers that are capped to prevent loss of organic vapors. The fluid properties are expected to change little from this point on.

15.6 Non-aqueous Drilling Fluid Aging at Moderate Temperatures [Ambient to 150 °F (65 °C)]

15.6.1 Sample Preparation

15.6.1.1 Oil-based drilling fluid samples to be aged at moderate temperatures are usually blended at ambient temperatures, in accordance with 15.5.

15.6.1.2 Additional materials may be added to slurries that have already been aged.

15.6.1.3 Many glass and metal containers used for ambient-temperature aging (see 15.2) have been found to be acceptable for static or dynamic aging up to 150 °F (65 °C).

15.6.2 Procedure for Aging at Moderate Temperatures
15.6.2.1 Aging of drilling fluids at moderate temperatures is usually done for one of the following reasons:

a) to speed up the chemical equilibrium of the system;

b) to activate chemical additives;

c) to expose the fluid to thermal conditions similar to the field conditions.
15.6.2.2 For exposure to moderate temperatures, drilling fluid samples are placed in any one of a variety of commercially available or custom-built ovens, in accordance with 15.3.9.

15.6.2.3 The methods of heating and cooling samples (i.e. preheating the oven versus not preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequacy of air circulation) and exact heat exposure time can affect the fluid properties. To optimize repeatability between test runs, the same heating and cooling methods and heat exposure times shall be employed for all samples in a series of comparative tests.

15.6.3 Maintaining Fluid Properties at Moderate Temperatures

Purging and blanketing samples with nitrogen gas appears to have strong benefits in reducing oxidative degradation of polymer-treated samples. This use of nitrogen leads to better reproducibility.

15.7 Non-aqueous Drilling Fluid Aging at Elevated Temperatures [over 150 °F (65 °C)]

15.7.1 Sample preparation

15.7.1.1 Drilling fluid samples for aging at elevated temperatures are usually blended at ambient temperatures in accordance with 15.5.

15.7.1.2 Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperatures may be added to slurries that have already been aged.

15.7.1.3 Only aging cells constructed from suitable metals shall be used at temperatures over 150 °F (65 °C), as stated in 15.3.

15.7.2 Contrast between Drilling Fluid Material Performance in Laboratory Testing and Field Environments

15.7.2.1 Be cautious about interpreting results from laboratory aging studies. Field results can be at variance with the results obtained from aging tests conducted under the conditions in the laboratory environment. Laboratory testing is to be used as a guideline and not an absolute predictor of drilling fluid performance.

15.7.2.2 Be cautious about interpreting results from laboratory aging studies using cells made from inert metal or containing inert material liners. In field applications, the drilling fluids are exposed to the steel in drill pipe, which is usually made from mild carbon steel.

15.7.2.3 Field results can differ from the laboratory results obtained under more ideal conditions. While the amount of steel surface exposed to drilling fluids is much less in steel aging cells than in drill pipe, it is still more than can be expected within inert aging cells.

16 Shale-particle Disintegration Test by Hot Rolling

16.1 Principle

16.1.1 This procedure involves grinding, sieving, and placing a known mass of shale particles into a drilling fluid and aging the fluid with shale particles, either statically or dynamically. Retrieving, cleaning, and reweighing the particles to determine mass loss, indicates the tendency of the fluid to prevent shale dispersion. This test is only a relative measure and should be included as part of a comprehensive testing program.

16.1.2 The ability of a drilling fluid to inhibit the disintegration of shale cuttings is important. The laboratory method presented in this clause can be used for evaluating the properties of drilling fluids with regard to inhibiting shale-particle disintegration. This test is intended as a comparison of various whole drilling fluid compositions.

16.1.3 There are many laboratory techniques used to assess the degree to which a drilling fluid formulation can cause shale to weaken and disintegrate when immersed in a fluid. The present method, although commonly used, is not the only test that indicates the ability of drilling fluids to inhibit shale disintegration. This test can give a wide variability of results. Strict control and reporting of the condition of the test shale, the rheological properties of the test fluid and the washing of shale are all necessary.

NOTE ASTM 39E presents test comparison procedures which may be of interest to the user.
16.1.4 Shale selection and condition (e.g. water content), while extremely important, are neither specified nor limited by this procedure. Properly preserved shale samples should be used, when available. It is strongly recommended that the shale be as near to its in situ moisture content as possible, and not having been previously air-dried or oven-dried. The way the shale was preserved prior to its use in the test should be recorded with the results.

16.1.5 Drilling fluid rheological properties are variables that have proven difficult to control from test to test. As rheological properties can have a mechanical influence on shale recovery independent from the chemical effects of the drilling fluid, it is recommended that the rheological properties be measured at the test temperature and be recorded with the results. This enables consideration of the effect of the rheological properties along with the other variables. Modest changes in the properties from one fluid to another can strongly influence shale dispersion final results.

16.1.6 It should be noted that the results do not necessarily imply wellbore stability. This test might not be appropriate for evaluating the inhibiting properties of certain drilling fluid materials.

16.2 Reagents and Apparatus

16.2.1 Wash water, inhibitive, that is either
a) a synthetic version of the drilling fluid base brine, or
b) a fluid known to inhibit the disintegration of the shale used [e.g. 15 lb/bbl (42.75 kg/m^3) potassium chloride].

NOTE Wash water of salinity much greater than the drilling fluid base brine can cause sieve blocking due to drilling fluid coagulation.

16.2.2 Supply, for inhibitive wash water, at a flow rate of 2 qt/min ±0.2 qt/min (approximately 2 L/min ±0.2 L/min) with an outlet, d, of approximately 1/3 in. (7.5 mm).

16.2.3 Balance, accuracy ±0.01 g.

16.2.4 Oven, roller, capable of achieving, typically, 17-35 r/min.

For best results, all tests should be conducted with the same roller oven to ensure consistent test conditions.

16.2.5 Metal Aging cells, as described in 14.3

WARNING—DO NOT USE ALUMINUM BRONZE METAL AGING CELLS

16.2.6 Clock or timer, accurate to ±0.1 min over the test interval.

16.2.7 Weighing boats.

16.2.8 Spatula.

16.2.9 Wash bath or sink, at least 0.5 gal (approximately 2 L) in size.

16.2.10 Sieves, 4 in. (10 cm) in diameter with mesh sizes of:
- 0.02 in. (0.5 mm) (U.S. Sieve number 35),
- 0.08 in. (2 mm) (U.S. Sieve number 10),
- and 0.16 in. (4 mm) (U.S. Sieve number 5).

16.2.11 Sieve base, 4 in. (10 cm) diameter.

16.2.12 Drying oven, regulated to 220 °F ±5 °F (105 °C ±3 °C).

16.2.13 Desiccator.

16.3 Procedure

16.3.1 Obtain shale pieces with moisture content as near to that in situ as possible. Sieve shale pieces just prior to use to fractions of less than 0.16 in. (4 mm) and greater than 0.08 in. (2 mm). The moisture content of bulk shale supply shall be established at the time of sampling by the method for bentonite in accordance with API 13A and recorded.
16.3.2 Cuttings should not be dried in an oven prior to use. When cuttings are sized for this test, enough extra cuttings must be available with which to measure the moisture content via oven-drying.

16.3.3 Pre-screen the drilling fluid through a 0.02 in. (0.5 mm) sieve to remove any oversize particles that can be incorrectly reported as recovered shale.

16.3.4 Add 350 mL of drilling fluid to each aging cell, measured either by volume or by mass.

16.3.5 Add 20.0 g of shale pieces (screened prior to use) to the fluid in each aging cell, seal and quickly agitate gently to wet and separate the particles.

NOTE With high-viscosity drilling fluids, it can be advantageous to add the drilling fluid and the shale simultaneously to achieve separate wetting of particles or gently stir the drilling fluid with a spatula while adding shale.

16.3.6 Place the aging cells in a roller oven preheated to the desired temperature and roll for a predetermined period of time.

The rolling time should be the same for all fluids being evaluated for a particular shale. The rolling time is dependent on the shale and some shales require more rolling time than others for sufficient disintegration to occur to permit discrimination among fluids.

16.3.7 After the rolling period is complete and the aging cells have cooled to a safe handling temperature, transfer to a static position to cool further to a temperature that is constant for all sequential recovery operations (e.g. from a cold-water bath to room temperature).

16.3.8 Pour the contents of each aging cell onto a numbered pre-weighed 0.02 in. (0.5 mm) mesh sieve while applying inhibitive wash water to the screen to prevent viscous blocking. Also displace the residual contents of the aging cell onto the sieve with wash water (the use of a wash bottle is convenient).

16.3.9 Wash the drilling fluid from the remaining shale pieces using wash-water, systematically passing across the full area of the screen many times for a period of approximately 1 min or until the shale and screen are visually free of drilling fluid residue.

16.3.10 Transfer the sieve to a bath containing tap water and quickly but gently submerge the sieve (at an angle to avoid an air trap) and remove it so that the sieve and shale have been rinsed of wash water.

16.3.11 Place the sieve onto a pre-weighed sieve base and dry in the drying oven to a constant mass. Cool in a desiccator and reweigh to 0.01 g. This is the final dry mass.

16.4 Calculation

16.4.1 The mass fraction of shale recovered (mass residue), \( w_r \), expressed in percent, is given by Equation (15):

\[
 w_r = \frac{w_2}{w_1 - ((w_1 \times M) / 100)} \times 100
\]

(15)

Where,

\( w_1 \) is the initial weight of cuttings used

\( w_2 \) is the final dry sample weight after hot rolling

\( M \) is the initial % moisture content of the sample
16.4.2 Record conditions used (shale preservation technique, history prior to test, drilling fluid rheological properties, test temperature and rolling time) and the shale mass recovered. Running the test in duplicate or triplicate is recommended. Differences between test values are typically less than 4%.

17 Measurement of Shear Strength Using Shearometer Tube

17.1 Principle

17.1.1 Experience has shown that some drilling fluids tend to develop excessive shear strength under static conditions, especially at elevated temperatures. Excessive shear strength results in high pump pressures to “break circulation”, and can result in loss of returns. High shear strength can also cause difficulties in logging, perforating, and other downhole operations.

17.1.2 The technique described in this annex can be used to determine this tendency and to estimate the extent to which the drilling fluid will develop excessive shear strength. This shear strength measurement is normally made on a static, heat-aged drilling fluid sample. Aging temperatures are therefore selected to be near the estimated bottom-hole temperature of the well. Aging cells or vessels meeting the pressure and temperature requirements for the test are required. See section XXXX for a complete description of metal aging cells.

17.2 Apparatus

17.2.1 Stainless steel shearometer tube, having the following characteristics:
- length 3.5 in. (89 mm)
- outside diameter 1.4 in. (36 mm)
- wall thickness 0.008 in. (0.2 mm)

NOTE A slight outside taper on the bottom of the shear tube has been found to improve reproducibility of the test results.

17.2.2 Platform for weights.

17.2.3 Set of weights, in gram increments.

17.2.4 Ruler, graduated in inches (millimeters).

17.3 Procedure

17.3.1 Place and balance the shear tube carefully on the surface of the aged sample that has cooled to room temperature. It may be necessary to shift the weights on the platform to ensure that the tube's initial penetration into the drilling fluid is vertical. If a crust develops on the heat-aged sample, this crust should be gently broken before setting the shear tube in place for the test. If base fluid is present on the surface of the heat-aged sample, it should be carefully removed and its volume measured, before setting the shear tube in place for the test.

17.3.2 Carefully place sufficient weights on the platform to start the downward movement of the shear tube. Unless too much mass is added, the tube will stop its downward travel at the point where the shear strength of the aged drilling fluid against the surface of the tube is sufficient to support the applied mass. It is desirable to submerge at least one-half of the length of the tube.

17.3.3 Record the total mass, in grams, of the platform and weights. Measure the length of the tube submerged in the fluid. The length of the tube submerged can be most accurately determined by measuring the length of the non-submerged portion while the tube is at its maximum penetration depth. A small ruler held at the drilling fluid surface and alongside the tube will facilitate this measurement. The length of the tube minus the exposed length equals the submerged portion.

17.4 Calculation

17.4.1 Shear strength in USC units is calculated as follows in equation (16):

\[
\gamma_A = \frac{3.61 \times (m_{st} + m_{tot})}{l_A} - 0.256\rho
\]  

(16)
where
\( \gamma_{US} \) is the shear strength of the drilling fluid, expressed in pounds-force per one hundred square feet;
\( m_{st} \) is the mass of the shear tube, expressed in grams;
\( m_{tot} \) is the total shear mass (sum of platform and weights), expressed in grams;
\( l_{US} \) is the submerged length of shear tube, expressed in inches;
\( \rho_{US} \) is the drilling fluid density, expressed in pounds per gallon.

17.4.2 Shear strength in SI units is calculated as follows in equation (17):

\[
\gamma_{M} = \frac{4.40 \times (m_{st} + m_{tot})}{l} - 1.02 \rho_{S}
\]  

(17)

where
\( \gamma_{M} \) is the shear strength of the drilling fluid, expressed in pascals;
\( m_{st} \) is the mass of shear tube, expressed in grams;
\( m_{tot} \) is the total shear mass (sum of platform and weights), expressed in grams;
\( L_{M} \) is the submerged length of shear tube, expressed in centimeters;
\( \rho_{M} \) is the drilling fluid density, expressed in grams per milliliter.
Annex A
(informative)

High-temperature/High-pressure Filtration Testing of Drilling Fluids using the Permeability-plugging Apparatus (PPA)

A.1 Principle

A.1.1 Measurements of the filtration behavior and wall-building characteristics of drilling fluid are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water or emulsion concentration.

A.1.2 The PPA can be operated at pressure differentials and temperatures approximating those prevailing down-hole and it permits the use of filtration media chosen to simulate exposed sands. The PPA utilizes an inverted fluid cell with the pressure applied from the bottom, the filter medium on top and the filtrate collected from the top. A hydraulic pump applies pressure to the cell, which is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

The PPA utilizes ceramic disks as filtration media. Ceramic disks are available with mean pore throat sizes ranging from 10-120 µm. The PPA uses media more representative of sands which allows evaluation of filtration / bridging agents in drilling fluids.

A.1.3 These characteristics are affected by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The permeability plugging apparatus (PPA) is a modified high-temperature, high-pressure filter press used to evaluate these interactions through ceramic-disk filter media at pressures up to 5000 psi (34,474 kPa) and temperatures from ambient to as high as 500 °F (260 °C) (refer to manufacturer's specifications for temperature and pressure limits). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

A.2 Safety Considerations

A.2.1 The pressure limitation in the use of the PPA depends upon the cell in use. For safety, it is imperative that the operator know the maximum operating pressure of the test apparatus with certainty and that this pressure not be exceeded. If in doubt, contact the manufacturer or use the lowest of the possible limits.

A.2.2 Cell damage can be categorized as follows: end-cap bending, end-cap compression, cylinder shear, cylinder stress, and the use of non-hardened set screws. End-cap bending can be detected visually or by measurement. End-cap compression can be detected by the distortion of the set-screw holes or seats, which become oval rather than round. Caps and/or manufacturer's recommended hardened set screws showing signs of damage shall not be used. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set-screw holes, shall not be used.

A.2.3 Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the apparatus. Improper assembly, incorrect operation, or using defective parts, creates the possibility of cell leakage or failure, which could result in serious injury or apparatus damage.
A.2.4 The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching hot parts of the apparatus during normal operation.

A.2.5 These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury and apparatus damage. These instruments should be used only on grounded circuits.

A.2.6 For safe operation ensure that the hydraulic pressure has been released before:

a) attempting to disconnect pressure hose from cell at quick coupler
b) attempting to remove cell from heating jacket
c) moving the PPA
d) refilling the hydraulic pump
e) performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly

Caution—After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

A.2.7 For safe pneumatic pressurization of backpressure receiver, consider the following.

A.2.7.1 Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it shall be supplied in an approved nitrogen gas cylinder or the nitrogen supply system. CO$_2$ is normally supplied in small cartridges pressurized to about 900 psi (6200 kPa).

Warning—Do not allow CO$_2$ cartridges to be heated or exposed to fire. They can explode if overheated.

Warning—Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas-train carbonate analysis.

A.2.7.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

A.2.7.3 When pressurizing the backpressure assembly, ensure that the regulator T-screw has been rotated counter-clockwise and turns freely. This ensures that no pressure is going into the regulator. Always open the supply pressure first then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the manufacturer’s recommended rating or relief valve settings. When relieving backpressure, shut the supply pressure, loosen the T-screw on the regulator and then bleed the pressure from the system.

Caution—Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings and hoses should be kept in good condition and leaks should be found and corrected. Periodically test the pressure-relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

A.2.8 For safe heating, consider the following.

A.2.8.1 Caution should be exercised to avoid injury while operating the PPA, which becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended.

A.2.8.2 The practice of removing the cell and cooling it with water is hazardous and shall be avoided. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell or by accidentally dropping the cell.

A.2.9 For safe electrical operation, consider the following.
A.2.9.1 Make sure that the electrical source is fused and grounded (earthed). Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

A.2.9.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

A.2.10 For test-cell maintenance, the user should be aware that the filtration cell is a pressure vessel and should be considered a source of potential hazard. The safety precautions listed below shall be followed to ensure safe operation.

A.2.10.1 Cell material shall be compatible with the test samples.

A.2.10.2 Do not use cells that show signs of severe pitting or stress cracking.

A.2.10.3 Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect threads carefully for signs of damage.

A.2.10.4 For cells that use set screws, use only hardened steel set screws as supplied by the manufacturer. Deformed set screws are unsafe.

The operating manual and this procedure should be read by anyone who is unfamiliar with the apparatus, before using it.

A.3 Apparatus—Permeability-plugging Apparatus (PPA)

A.3.1 PPA cell

A.3.1.1 There are different ratings for cells. Refer to manufacturer's specifications for temperature and pressure limits.

Warning—There are still a number of older cells in use which are stamped “2500 psi”. In 1996, the rating on these cells was reduced to “1800 psi” (12,420 kPa). Unless the user can absolutely verify that the cell used is rated at 2000 psi (13,800 kPa), the “1800 psi” (12,420 kPa) pressure limit should be observed.

Warning—Follow the manufacturer's recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

A.3.1.2 The PPA utilizes an inverted fluid cell with the pressure applied from the bottom, the filter medium on top and the filtrate collected from the top. A hydraulic pump applies pressure to the cell, which is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

A.3.1.3 The PPA utilizes ceramic disks as filtration media. Ceramic disks are available in various mean pore throat sizes (see Table A.1).

A.3.1.4 For temperatures above 200 °F (93 °C), the backpressure receiver shall be pressurized to prevent boiling of the filtrate. The standard backpressure receiver uses a CO2 pressurizing source to provide the backpressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO2 when desired.

A.3.1.5 The PPA cell is encased in a thermostatically controlled heating chamber. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 500 °F (260 °C) (Check with manufacturer's specifications). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel.

A.3.2 Filtration media.

A.3.2.1 Standard ceramic disk thickness is 0.25 in. (6.5 mm). Thicker disks can be used with proper manufacturer's cells. A new disk is required for each test. For water-based drilling fluid samples, the disk
shall be soaked in base fluid (water, brine, etc.) for at least 30 min; for non-aqueous drilling fluid the disk shall be soaked in base oil. When possible, vacuum saturate for at least 5-30 min prior to use is preferred.

These disks are sold with a mean pore throat diameter as determined by mercury injection porosimetry. Table A.1 lists the commonly available ceramic disks and the mean pore throat diameter.

<table>
<thead>
<tr>
<th>API Designation</th>
<th>Hg Injection Mean Pore Throat Diameter μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>API 10</td>
<td>10</td>
</tr>
<tr>
<td>API 12</td>
<td>12</td>
</tr>
<tr>
<td>API 20</td>
<td>20</td>
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<tr>
<td>API 40</td>
<td>40</td>
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<td>API 50</td>
<td>50</td>
</tr>
<tr>
<td>API 55</td>
<td>55</td>
</tr>
<tr>
<td>API 120</td>
<td>120</td>
</tr>
</tbody>
</table>

**NOTE** Mercury injection porosimetry is not a viable technique for measuring pore throat sizes exceeding 120 μm. Therefore, there is no API designation for previously offered 150 μm and 190 μm ceramic disks.

A.3.2.2 Ceramic disks were adopted for use in the Particle Plugging Apparatus (PPA) as a method to test plugging/bridging materials in drilling fluids. Ceramic disks do not exhibit the normal relationship between pore throat size and permeability as naturally occurring sandstone. Work conducted to assess bridging characteristics should be based on pore throat size.

A.3.3 **Timer**, accurate to ±0.1 min over the test interval.

A.3.4 **Thermometer**, with scale up to 500 °F (260 °C).

A.3.5 **Graduated cylinder**, 25 mL (TC) or 50 mL (TC).

A.3.6 **High-speed mixer**.

A.4 **Procedure for the Permeability Plugging Apparatus**:

A.4.1 **Assembling the filtration cell**

**NOTE** If the cell is completely disassembled and the floating piston has been removed follow clause A.4.1.1 through A.4.1.10. If the piston is in the cell with hydraulic fluid and with the inlet nipple and quick connect attached, then proceed to clause A.4.1.7

**Caution**—The filtration cell is a pressure vessel.

A.4.1.1 Remove the end caps from the cell. A cell cap removal tool is available from certain manufacturers for some cells.

A.4.1.2 Check the O-rings on the valve stems, the piston, the cell body and the end caps, and replace any that are damaged or brittle [O-rings should be replaced after each test at temperatures above 300 °F (150 °C)]. Apply a thin coating of stopcock grease completely around the O-rings, being careful to ensure that those on the piston and cell caps are well lubricated.

A.4.1.3 Screw the floating piston onto the T-bar wrench and install the piston into the inlet end of the cell, working it up and down to ensure that it moves freely (the inlet end, has a shorter recess than the
outlet end). Position the piston so that it is at the lip of the O-ring groove in the cell body. Leave the piston T-bar wrench in place.

A.4.1.4 Fill the inlet space with hydraulic oil.
A.4.1.5 Install the hydraulic inlet cap onto the cell.

Some oil should flow from the hole in the end cap, indicating that no air is trapped between the piston and the end cap.

A.4.1.6 Install and tighten the inlet nipple and quick connect.

NOTE Steps A.4.1.7 to A.4.1.10 should be performed with the cell placed in an unheated jacket.

A.4.1.7 Invert the cell so the outlet end is upwards and place in an unheated jacket.
A.4.1.8 Connect the cell inlet to the pump quick connect and follow the manufacturer’s recommended procedure to purge all air from the system.
A.4.1.9 Apply pressure to the cell using the hydraulic pump. Using the piston T-handle as a guide, stroke the pump enough so the piston rises 2 in. inside the cell. Remove the piston T-handle.
A.4.1.10 Disconnect the inlet quick connect and remove cell from heating jacket. Place on a suitable stand.

A.4.2 Preheating the heating jacket

A.4.2.1 Connect the power cord to the proper voltage as indicated on the nameplate.
A.4.2.2 Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light illuminates when the heating-jacket temperature reaches the thermostat setting.
A.4.2.3 Readjust the thermostat to 10 °F (6 °C) over the desired test temperature.

A.4.3 Loading Test Fluid into the cell.

NOTE For improved consistency in test results, test fluid must be stirred for 5 min immediately before loading the cell.

Caution—Thermal expansion of the cell contents and of the hydraulic fluid causes the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the fluid periodically.

A.4.3.1 With the cell on a suitable stand, fill the cell to the bottom of the outlet O-ring groove with the test fluid. If the floating piston has been elevated at least 2 in. above the inlet cap face, then any fluid expansion due to heat will force the piston toward the inlet cap, and the ceramic disk will maintain pre-saturation.
A.4.3.2 Install the outlet cell O-ring and set the selected pre-saturated ceramic disk or other filtration media on top of it.
A.4.3.3 Position the outlet end cap in the cell, tighten the cell cap and then install and close the ball valve on the outlet end cap.
A.4.3.4 Install the cell in the heating jacket. Lower the cell fully into the heating jacket. Rotate the cell so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.
A.4.3.5 Connect the cell inlet to the quick connect.
A.4.3.6 Transfer the thermometer from the heating jacket and place into the cell body. Monitor the cell temperature with the thermometer in the cell wall, not in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature.

A.4.4 Pressurization
A.4.4.1 Apply pressure to the cell using the hydraulic pump immediately after transferring the thermometer to the cell body. This pressure should equal the pressure that will be applied to the back pressure receiver. (Refer to Table A.2 for the pressure corresponding to the test temperature).

Filtration at temperatures above the boiling point of the fluid sample requires the use of pressure to the cell while heating and of the back pressure receiver to prevent vaporization of the filtrate.

A.4.4.2 Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. The fluid should not be heated more than one hour prior to filtrate collection. If the test fluid cell temperature has not reached the test temperature after one hour the heater may be defective and the validity of the test is questionable.

A.4.4.3 While the cell is heating, use the following procedure to prepare the receiver.

A.4.4.4 Check to ensure that the regulator T-screw has been rotated counter-clockwise. The regulator T-screw should turn freely when no pressure is introduced into the regulator.

A.4.4.5 Verify that the pressure-relief valve on the pressure assembly and the filtrate drain valve on the receiver are closed.

A.4.4.6 Mount the back pressure receiver on the outlet valve assembly. Secure the receiver with a retaining pin. Install the pressurizing unit on top of the receiver. Lock the pressurizing unit in place with the other retaining pin. Do not adjust the regulator at this time.

NOTE A single CO₂ cartridge may not reach the maximum pressures in the table below. If necessary, loosen the regulator T-screw, remove the empty cartridge, and replace with a new one. Tighten the barrel enough to puncture the cartridge.

NOTE If a Nitrogen manifold capable of 700 psi (4850 kPa) is available it may be used

<table>
<thead>
<tr>
<th>Temperature range</th>
<th>Heating pressure or backpressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C</td>
<td>°F</td>
</tr>
<tr>
<td>Less than 95</td>
<td>200</td>
</tr>
<tr>
<td>95-150</td>
<td>200-300</td>
</tr>
<tr>
<td>151-175</td>
<td>301-350</td>
</tr>
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<tr>
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<td>219-232</td>
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<tr>
<td>233-246</td>
<td>451-475</td>
</tr>
<tr>
<td>247-260</td>
<td>476-500</td>
</tr>
</tbody>
</table>

A.4.4.7 If a drain hose is used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space between the filtration medium and the outlet ball valve should be filled with the base fluid before starting the test. This ensures that the fluid passing through the
filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

A.4.4.8 See Table A.2 to determine the appropriate pressure for the backpressure receiver and apply it by turning the T-screw on the pressure regulator clockwise until the desired pressure is reached.

A.4.4.9 After the test fluid has heated for one hour, actuate the pump to raise the cell pressure to the desired level and then open the valve between the cell and the backpressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the inlet pressure applied to the cell and that maintained on the backpressure receiver.

A.4.5 Conducting the filtration test

A.4.5.1 A laboratory timer shall be used and filtrate shall be collected initially after approximately 15 s to ensure the fluid has good filtration properties (excessive filtrate). Collect filtrate and record the volume at 1 min, 7.5 min, and 30 min. Plotting these data against the square root of time provides a useful description of the spurt loss. If desired, samples can be collected more frequently. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

A.4.5.2 Begin the filtration by opening the valve between the cell and the backpressure receiver. Verify that both the cell pressure, as indicated on the pump gauge, and the backpressure are at desired levels. Adjust them as necessary throughout the test.

A.4.5.3 Cell pressure can decrease slightly as filtration and any leakage at the pump reduce the cell contents. The pump should be used as necessary to maintain the pressure at the desired level.

A.4.5.4 After each interval, the filtrate shall be drained into the graduated cylinder from the backpressure receiver and the time and cumulative volume shall be recorded.

It is recommended to recover the filtrate directly from the backpressure receiver and not from a drain hose attached to it. However, if a hose is used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

A.4.5.5 After 30 min, close the valve between the cell and the backpressure receiver and drain any remaining filtrate from the backpressure receiver into the graduated cylinder by opening the receiver outlet valve.

A.5 Test conclusion and disassembly

A.5.1 Disconnect the heating jacket from the power source.

The pressurized cell assembly shall be allowed to cool in the heating jacket.

Warning—Extreme care should be exercised with cooling hot cells.

A.5.2 Turn the T-screw on the back pressure receiver regulator counter-clockwise until it turns freely.

A.5.3 Bleed the pressure from the backpressure receiver by opening the pressure relief valve.

A.5.4 Open the drain valve on the backpressure receiver and collect the last few drops of filtrate in the graduated cylinder. After removing the locking pin, remove the backpressure assembly from the cell.

A.5.5 Because the back pressure receiver is used with the flat end downward, some filtrate may be trapped inside the receiver body. Place the receiver with the flat end downward and the inlet hole over the graduated cylinder. Gently rock the receiver side to side to remove any remaining liquid.

A.5.6 The total volume of the filtrate in the graduated cylinder shall be recorded.

A.5.7 The temperature of the sample in the cell should be reduced to below 100 °F (38 °C) to ensure that the cell can safely be opened. Once the cell has cooled, release the pressure on the cell by opening the hydraulic pump valve. Disconnect the quick coupler between the pump and the bottom of the cell. The cell may be raised in the heating jacket and placed on the cell support or laid on a bench while it is being opened.
A.5.8 The cell should be opened only when the user is confident that the contents are no longer under pressure. If pressure is suspected to remain in the cell consult manufacturers’ procedure for pressure release process. Trapped pressure is usually indicated by difficulty in removing end cap.

A.5.9 The end cap should be removed with the cell in the vertical position and the filtration end facing upwards.

A.5.10 To remove the filter disk pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with base fluid (water, brine, oil, etc.), then measure and record its thickness and remark on its composition and texture.

A.5.11 Pour the remaining fluid from the cell. Wash the inside of the cell with base fluid. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 300 °F (150 °C) or higher.

If testing was conducted at temperatures above 300 °F (150 °C), the O-rings should be replaced which will require removal of the inlet cell cap and floating piston.

A.5.12 Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the inlet end cap using the manufacturer’s procedure.

b) Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. The floating piston can be removed through the top end without the removal of the bottom end cap. Remove and dispose of the O-rings on the piston and the cap.

c) Clean the parts for reuse.

A.6 Test reports

A.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, in milliliters, collected through each of the selected time periods.

A.6.2 Spurt loss

The spurt loss can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume [doubled to correct for filtration area when using 3.5 in.$^2$ (22.6 cm$^2$) filtration media] is plotted along the y-axis. Alternatively, an approximate value can be calculated using Equation (A.2).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with A.4.5.1.

A.6.3 Calculation

Set the permeability-plugging test volume, $V_{PPT}$, expressed in milliliters, equal to two times the $V_{30}$, the filtrate volume after 30 min, expressed in milliliters, as given in Equation (A.1):

$$V_{PPT} = 2V_{30} \quad (A.1)$$

Calculate the spurt loss, $V_1$, expressed in milliliters, as given in Equation (A.2):

$$V_1 = 2\left[V_{7.5} - (V_{30} - V_{7.5})\right] = 2(2V_{7.5} - V_{30}) \quad (A.2)$$

where $V_{7.5}$ is the filtrate volume after 7.5 min, expressed in milliliters.

Calculate the static filtration rate (velocity of flow), $v_{sf}$, expressed in milliliters per square root of the minutes, as given in Equation (A.3):
\[ v_{sf} = \frac{2(V_{30} - V_{7.5})}{\sqrt{t_2} - \sqrt{t_1}} = \frac{2(V_{30} - V_{7.5})}{2.739} \]  

(A.3)

where

\( t_1 \) is the time at initial reading, expressed in minutes;

\( t_2 \) is the time at final reading, expressed in minutes.

Observe that these parameters, \( V_{PPT}, V_1 \) and \( v_{sf} \), are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard low-pressure filtration test. Doubling the filtrate volume compensates for this area difference.

A.6.4 Test report for filter cake

Measure and record the filter cake thickness to the nearest 1/32 in. (0.76 mm). Include a description, such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgments, they can convey important information.
Annex B
(informative)

Testing Particle Size Distribution in a Non-aqueous Drilling Fluid using a Laser Diffraction Instrument

B.1 Summary
This procedure is an API recommended practice for measuring the particle size distribution of non-aqueous drilling fluids using laser diffraction based instruments. Measurement will involve a recommended sampling protocol followed by dilution of the mud sample in an appropriate carrier fluid, sonication of the sample and data collection.

B.2 Reagents & Apparatus
B.2.1 Isopropanol (IPA) (technical grade or better)
B.2.2 Hexane (technical grade or better)
B.2.3 Base oil (if IPA or hexane is not available)
B.2.4 Transfer pipettes (minimum opening of 3 mm)
B.2.5 Magnetic stir bar, 1 in.
B.2.6 Magnetic stirrer
B.2.7 Overhead mixer with single sinewave impeller (10,000 to 15,000 rpm)
B.2.8 Glass beaker, 25 mL

B.3 Instrument Settings
B.3.1 If necessary, verify the instrument gives the expected values on the reference material per manufacturer’s recommendation.
B.3.2 Set the refractive index of the solids to be measured to 1.6. (Appendix 1)
B.3.3 If prompted, set the absorption value of the particles to an appropriate number (should be either 0 or 0.01 depending on the instrument. Contact instrument supplier for the recommended value).
B.3.4 If prompted, choose the “General Purpose” calculation model.
B.3.5 Set sonication power to 100%.
B.3.6 If prompted, choose “Irregular particle” as the particle shape to be measured.
B.3.7 Set the measurement duration to 30 s. Perform measurement three times.

B.4 Procedure for Measuring Particle Size Distribution in a Non-aqueous Drilling Fluid using a Laser Diffraction Instrument
B.4.1 Qualitative Sample Dispersion Test prior to bulk mixing of mud in step
B.4.1.1 All PSD measurements are done using IPA as the carrier fluid. If IPA is not available, it is acceptable to use base oil as the carrier fluid.
B.4.1.2 Check the miscibility of mud with IPA. Add a few drops of mud using a transfer pipette to glass beaker/vial containing IPA and stir or shake by hand. Check for any visible mud clumps or mud settling to the bottom. If none is observed, the mud is miscible with IPA; otherwise, the mud should be miscible with hexane (Appendix 2).
B.4.1.3 For muds that are miscible with IPA, all sample PSD runs will be carried out in IPA.
B.4.1.4 For muds that are immiscible with IPA, all sample PSD runs will be still carried out in IPA. However, pre-dispersion of mud in hexane is required. Hexane is used to remove base oil from the solids so that the solids can be dispersed in IPA.

B.4.2 Bulk mixing of mud sample Preparation and Sampling

B.4.2.1 Remix the mud in the container to obtain best representative sample.

B.4.2.2 Homogenize approximately 400 mL of mud with an overhead mixer for 2 minutes.

B.4.2.3 Fill particle sizing distribution (PSD) instrument with IPA (use base oil when IPA is not available).

B.4.2.4 Adjust circulation speed to 75 % of full speed (Appendix 3).

B.4.2.5 Record the PSD background profile of carrier fluid before adding mud when the background is stable.

B.4.2.6 For muds that are miscible with IPA, add homogenized mud drop-wise and slowly to the PSD instrument until laser signal obscuration of 10 ±2 % is observed (Note: laser signal reading is not instantaneous – add mud slowly).

B.4.2.7 For muds that are immiscible with IPA, pre-dispersion of mud in hexane is required. Add three to five drops of mud to a beaker/vial containing 10 mL of hexane and swirl or stir until the sample is fully dispersed. Then, add entire hexane solution to circulating reservoir of the PSD instrument. Rinse the beaker/vial well with IPA (< 10 mL) and add solution to circulating reservoir to make sure the entire sample aliquot is transferred. Make sure the obscuration is 8 to 15%.

B.4.2.8 Allow sample to circulate inside the PSD reservoir for 1 min.

B.4.2.9 Sonicate sample at 100 % power for 60 s (refer to Appendix 4 to see the effect of sonication time). Sonication typically causes a slight increase in obscuration, this is to be expected, and this increase in obscuration has no impact on the measurement results.

B.4.2.10 If necessary, allow sample to circulate until all evidence of thermal effects from the sonication has disappeared from the PSD curve (this typically takes between 1-5 min depending on the instrument.) (Appendix 5)

B.4.2.11 Collect three repeat measurements, 30 s duration each, 60 s between each measurement, to ensure the measurement is repeatable.

B.5 Refractive Index value used in the procedure

The solids in most non-aqueous drilling fluids are primarily composed of barite, calcite, quartz and bentonite. The proportion of these minerals may vary in different drilling fluids, and hence, ideally the refractive index used in the PSD measurement should be determined by averaging the refractive index values of these components in the volume basis for every drilling fluid being analyzed. However, it is not practical in terms of time and cost to acquire the abundance of these components for each PSD measurement. Moreover, the refractive index values of minerals may also vary according to the optical axis of a given mineral, for example, calcite ranges from 1.486 to 1.658. Consequently, the actual refractive index value of a certain mineral is also related to the particle shape (granular, elongated, flaky, etc.) as well as how these particles move and rotate in the carrier fluid during the measurement. Such complexity makes it difficult to determine the true refractive index of each drilling fluid given the volumetric variation of mineral components as well as the variation of refractive index of each mineral. An average refractive index value of 1.6 is a good representation of barite muds. However, this value should be changed accordingly when other weighting materials are used.
Table B.1—Average refractive index value of minerals used in drilling fluids

<table>
<thead>
<tr>
<th>Minerals</th>
<th>Average Refractive Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>1.54</td>
</tr>
<tr>
<td>Calcite</td>
<td>1.65</td>
</tr>
<tr>
<td>Dolomite</td>
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</tr>
<tr>
<td>Bentonite</td>
<td>1.56</td>
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<tr>
<td>Montmorillonite</td>
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<td>Illite</td>
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<tr>
<td>Kaolinite</td>
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</tr>
<tr>
<td>Plagioclase Feldspar</td>
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<tr>
<td>Potassium Feldspar</td>
<td>1.52</td>
</tr>
<tr>
<td>Barite</td>
<td>1.64</td>
</tr>
</tbody>
</table>
B.6 Visual Inspection of Miscibility of Mud in IPA

Figure B.1—Mud Clumps form when Alkane based Mud is Dispersed in IPA.

Figure B.2—Olefin based mud is fully dispersed in IPA.
B.7 Flow Rate Effect

The flow rate of the carrier fluid has great influence on the PSD data quality including sampling statistics and measurement reproducibility. During the PSD measurement, the particles being analyzed are circulated through the measurement window at a certain speed. In order to achieve a representative PSD result, good sampling statistics are required, more specifically; all/a majority of the particles in the circulation reservoir of particle sizer should pass through the measurement window. A low flow rate affects the carrying capacity of the fluid to incorporate larger particles. In addition, the fluid system may take longer to reach thermal equilibrium with the operating environment when refilling carrier fluid, loading sample, or applying sonication when the flow rate is low.

The experiments with a 70/30 (olefin/water) mud in IPA subjected to 4 different flow rates: 25%, 50%, 75% and 100% respectively, showed that the flow dynamics alone can help break down particle aggregates and well disperse the sample (Figure 3). A higher flow rate can break down particle aggregates better. Hence, the maximum flow rate (100%) is preferred when considering the effect of flow dynamics on breaking down particle aggregates. However, the flow rate at 100% can possibly introduce bubbles or/and thermal drift of the carrier fluid, leading to a long tail at the coarse end of PSD curve as shown in Figure 3.

When combined with sonication treatment, these four different flow rates yielded the PSD curves showed in Figure 4. The fluid didn’t circulate efficiently at 25% flow rate, resulting in a thermal artifact peak at the coarse end of PSD curve even though the measurement took place 10 minutes after the sonication. It appears that the combination of 50% flow rate and 1 minute sonication gives the smallest D50 value followed by the combination of 75% flow rate and 1 minute sonication. However, further experiments showed that the D50 value increased from 11.9 μm at 50% flow rate to 15.5 μm at 75% flow rate after sonication (Figure 5). Such an increase in the D50 value clearly indicated that 50% flow rate was insufficient to keep the largest particles in the sample suspended in the carrier fluid, as large and dense particles have increased tendency to settle and not contribute as much to the measured PSD curve resulting in a slightly smaller D50 value.

![PSD curves of 70/30 (olefin/water) mud at different flow rates.](image)
Figure B.4—PSD curves of 70/30 (olefin/water) mud measured at different flow rates after 1 minute sonication.

The PSD measured at 50% flow rate after 1 minute sonication gave the D50 value of 11.9 μm, then the flow rate was increased to 75% and the measured PSD yielded the D50 value of 15.5 μm. After an additional 1 minute sonication at 75% flow the D50 decreased to 14.8 μm.

Figure B.5—PSD curves of 70/30 (olefin/water) mud at 50% and 75% flow rate after sonication.
B.8 The Effect of Sonication

It has been shown that the application of sonication can effectively break down particle aggregates. Sonication can even break up single particles that are loosely held together, for example some clay minerals, with appropriate output power and duration of sonication. The sonication power typically varies across different PSD instruments, making it impractical to define sonication power used in this procedure. Instead, full power (100%) is recommended. A series of PSD measurements were carried out on a 70/30 (olefin/water) mud with various duration of sonication treatment and the resultant PSD curves are plotted in Figure 6. The PSD curves shift to the fine end as the sonication duration increases from 0 to 60 seconds, in accordance with the decrease of D50 values from 22.6 μm to 14.1 μm. The PSD curve measured after 2 min of sonication didn’t show significant difference compared with the one measured after 60 second sonication although the D50 value slightly increases. It appears that sonication for 60 seconds is optimum.

![PSD curves of 70/30 (olefin/water) mud with different duration of sonication at 100% power and the corresponding D50 values.](image)

Figure B.6—PSD curves of 70/30 (olefin/water) mud with different duration of sonication at 100% power and the corresponding D50 values.
B.9 Sonication Induced Thermal Effect

The heat generated from sonication causes an increase in temperature of the carrier fluid. Correspondingly, it changes the refractive index of the carrier fluid which is typically temperature dependent. Consequently, the scattering signal profile of the carrier fluid drifts from the initial background profile, resulting in artifact peaks at the coarse end of the distribution. Depending on the size of particles being analyzed, these artifact peaks may appear as disconnected or partially-disconnected from the primary peak, or even overlap with the primary peak. Such sonication induced thermal effect is more significant in volatile carrier fluids such as IPA. As the solvent gradually reaches thermal equilibrium with the operating environment, these artifact peaks become smaller and smaller until they disappear (Figure 7). To eliminate these artifact peaks from the PSD curve, longer recirculation time is required before the measurement takes place.

![Artifact peaks](image)

Figure B.6—Artifact peaks induced by sonication disappear over time.

Bibliography

[1] API RP 13K, Recommended Practice for Chemical Analysis of Barite