

# Drilling Fluids Materials

API SPECIFICATION 13A  
NINETEENTH EDITION, OCTOBER 2019

API MONOGRAM PROGRAM EFFECTIVE DATE: OCTOBER 1, 2020

ADDENDUM 1, APRIL 2020



AMERICAN PETROLEUM INSTITUTE

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## Introduction

This Standard covers materials that are in common usage in petroleum and natural-gas drilling fluids. These materials are used in bulk quantities, can be purchased from multiple sources and are available as commodity products. No single-source or limited-source products are included, nor are specialty products.

Standards are published to facilitate communication between purchasers and manufacturers, to provide interchangeability between similar equipment and materials purchased from different manufacturers and/or at different times and to provide an adequate level of safety when the equipment or materials are utilized in the manner and for the purposes intended. This Standard provides minimum requirements and is not intended to inhibit anyone from purchasing or producing materials to other standards.

The purpose of this Standard is to provide product specifications for materials manufactured for use in oil- and gas-well drilling fluids. The materials covered are barite, hematite, bentonite, non-treated bentonite, Oil Companies Material Association (OCMA)-grade bentonite, attapulgite, sepiolite, technical grade low-viscosity carboxymethyl cellulose (CMC-LVT), technical grade high-viscosity carboxymethyl cellulose (CMC-HVT), starch, low-viscosity polyanionic cellulose (PAC-LV), high-viscosity polyanionic cellulose (PAC-HV), and drilling-grade xanthan gum.

A survey of the industry found that only the American Petroleum Institute (API) issued testing procedures and specification standards for these materials.

Annex A (informative) discusses the use of the API Monogram by Licensees, Annex B provides the test precision, Annex C (informative) details examples of calculations, and Annex D discusses the API Reference Material Program.

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

In this standard, quantities expressed in the international System of Units (SI) are also, where practical, expressed in U.S. customary units (USC) in parentheses for information. The units do not necessarily represent a direct conversion of SI units to USC units, or USC units to SI units. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one degree increments, thus temperature values have been rounded to the nearest degree.

Calibrating an instrument refers to ensuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility, of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate but not precise, precise but not accurate, neither accurate nor precise, or both accurate and precise. A result is valid if it is both accurate and precise.

This document uses a format for numbers which follows the examples given in *API Document Format and Style Manual*, First edition, June 2017 (Editorial Revision, November 2017). This numbering format is different than that used in API 13A 18<sup>th</sup> Edition. In this document the decimal mark is a period and separates the whole part from the fractional part of a number. No spaces are used in the numbering format. The thousands separator is a comma and is only used for numbers greater than 10,000 (i.e. 5000 items, 12,500 bags).

# Drilling Fluids Materials

## 1 Scope

This specification covers physical properties and test procedures for materials manufactured for use in oil- and gas-well drilling fluids. The materials covered are barite; hematite; bentonite; non-treated bentonite; OCMA-grade bentonite; attapulgite; sepiolite; technical grade, low-viscosity carboxymethyl cellulose (CMC-LVT); technical grade, high-viscosity carboxymethyl cellulose (CMC-HVT); starch; low-viscosity polyanionic cellulose (PAC-LV); high-viscosity polyanionic cellulose (PAC-HV); and drilling-grade xanthan gum. This specification is intended for the use of manufacturers, distributors, and end users of named products. Annex A (informative) contains information on the API Monogram Program and requirements for the approved use of the API Monogram by licensees.

## 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 Recommended Practice 13B-1, *Recommended Practice for Field Testing Water-based Drilling Fluids*

ASTM D422-63 <sup>1</sup>, *Standard Test Method for Particle-size Analysis of Soils*

ASTM E11, *Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves*

ASTM E77, *Standard Test Method for Inspection and Verification of Thermometers*

ASTM E161, *Standard Specification for Electroformed Material and Test Sieves*

ASTM E617, *Standard Specification for Laboratory Weights and Precision Mass Standards*

ISO 386 <sup>2</sup>, *Liquid-in glass laboratory thermometers—Principles of design, construction and use*

NIST National Bureau of Standards Monograph 150 <sup>3</sup>, *Liquid-In-Glass Thermometry*

## 3 Terms, Definitions, Symbols, and Abbreviations

### 3.1 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

#### 3.1.1

##### **ACS reagent grade**

Chemicals that meet purity standards as specified by the ACS <sup>4</sup>.

#### 3.1.2

##### **flash side**

Side containing residue (“flash”) from stamping, or the side with concave indentation.

---

<sup>1</sup> ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, [www.astm.org](http://www.astm.org).

<sup>2</sup> International Organization for Standardization, Chemin de Blandonnet, 8, CP401, 1214 Vernier Geneve, Switzerland, [www.iso.org](http://www.iso.org).

<sup>3</sup> National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899, [www.nist.gov](http://www.nist.gov).

<sup>4</sup> American Chemical Society, 1155 Sixteenth Street NW, Washington, DC 20036, [www.acs.org](http://www.acs.org).

**3.1.3****mineral spirits**

Distilled petroleum product.

NOTE Available in many varieties.

**3.2 Symbols**

$B_c$	hydrometer correction line at initial value ( $\theta_1$ ), dimensionless (ASTM 151H hydrometer)
$b$	yield point-plastic viscosity ratio, expressed in lbf/(100 ft <sup>2</sup> •cP)
$C_{Ca,c}$	corrected concentration of soluble alkaline earth metals as calcium, expressed in milligrams per kilogram
$C_c$	ethylenediaminetetraacetic acid (EDTA) solution calcium calibration correction, expressed in milligrams per kilogram
$C_m$	measured calcium concentration in the standard calcium chloride solution, expressed in milligrams per kilogram
$C_s$	sample calcium test value, expressed in milligrams per kilogram
$C_{sc}$	sample corrected calcium test value, expressed in milligrams per kilogram
$c(\text{CaCl}_2)$	concentration of calcium chloride, expressed in moles per liter
$D_e$	equivalent spherical diameter, expressed in micrometers
$D_1$	equivalent particle diameter immediately greater than 6 $\mu\text{m}$ , expressed in micrometers
$D_2$	equivalent particle diameter immediately less than 6 $\mu\text{m}$ , expressed in micrometers
$d$	diameter, expressed in millimeters (inches)
$K_s$	sample constant, expressed in percent per gram
$L$	hydrometer effective depth, expressed in centimeters
$M_c$	hydrometer correction line slope (negative), expressed in reciprocal degrees Celsius ( $^{\circ}\text{C}^{-1}$ ) [degrees Fahrenheit ( $^{\circ}\text{F}^{-1}$ )]
$m$	sample mass, expressed in grams
$m_1$	dried sample mass, expressed in grams
$m_2$	residue mass retained by 75 $\mu\text{m}$ sieve, expressed in grams
$m_3$	residue mass retained by 45 $\mu\text{m}$ sieve, expressed in grams
$m_4$	mass of the 425 $\mu\text{m}$ sieve and sample retained, expressed in grams

$m_5$	mass of 425 $\mu\text{m}$ sieve, expressed in grams
$m_6$	mass of the bottom receiver, expressed in grams
$m_7$	mass of the bottom receiver and sample content, expressed in grams
$R$	hydrometer relative density (specific gravity) reading, dimensionless
$R_{0.2-3}$	viscometer (F0.2 spring) dial reading at 3 r/min, expressed in degrees of deflection
$R_{0.2-6}$	viscometer (F0.2 spring) dial reading at 6 r/min, expressed in degrees of deflection
$R_{0.2-100}$	viscometer (F0.2 spring) dial reading at 100 r/min, expressed in degrees of deflection
$R_{0.2-200}$	viscometer (F0.2 spring) dial reading at 200 r/min, expressed in degrees of deflection
$R_{0.2-300}$	viscometer (F0.2 spring) dial reading at 300 r/min, expressed in degrees of deflection
$R_{0.2-600}$	viscometer (F0.2 spring) dial reading at 600 r/min, expressed in degrees of deflection
$R_1$	average hydrometer relative density reading at lower temperature $\theta_1$
$R_{1.5}$	spindle-type viscometer reading at 1.5 r/min, expressed in centipoises
$R_2$	average hydrometer relative density reading at higher temperature $\theta_2$
$R_{3-M}$	viscometer (F0.2 spring) dial reading at 3 r/min of the xanthan gum solution with borate, expressed in degrees of deflection
$R_{3-P}$	viscometer (F0.2 spring) dial reading at 3 r/min of the xanthan gum solution, expressed in degrees of deflection
$R_{6-M}$	viscometer (F0.2 spring) dial reading at 6 r/min of the xanthan gum solution with borate, expressed in degrees of deflection
$R_{6-P}$	viscometer (F0.2 spring) dial reading at 6 r/min of the xanthan gum solution, expressed in degrees of deflection
$R_{300}$	viscometer dial reading at 300 r/min, expressed in degrees of deflection
$R_{300-M}$	viscometer (F0.2 spring) dial reading at 300 r/min of the xanthan gum solution with borate, expressed in degrees of deflection
$R_{300-P}$	viscometer (F0.2 spring) dial reading at 300 r/min of the xanthan gum solution, expressed in degrees of deflection
$R_{600}$	viscometer dial reading at 600 r/min, expressed in degrees of deflection
$t$	time, expressed in minutes
$V$	total filtrate volume, expressed in milliliters

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$V_c$	filtrate volume collected between 7.5 min and 30 min, expressed in milliliters
$V_1$	initial volume, expressed in milliliters
$V_2$	final volume, expressed in milliliters
$V_3$	volume EDTA used, expressed in milliliters
$w_a$	cumulative percent finer, expressed in percent
$w_1$	mass fraction residue of particles greater than 75 $\mu\text{m}$ , expressed in percent
$w_2$	cumulative percent for point immediately greater than 6 $\mu\text{m}$ , expressed in percent
$w_3$	cumulative percent for point immediately less than 6 $\mu\text{m}$ , expressed in percent
$w_4$	cumulative percent less than 6 $\mu\text{m}$ , expressed in percent
$w_5$	mass fraction residue of particles greater than 45 $\mu\text{m}$ , expressed in percent
$w_6$	mass fraction moisture, expressed in percent
$w_{75}$	mass fraction of sample passing through a 75 $\mu\text{m}$ sieve, expressed in percent
$w_{425}$	mass fraction passing through a 425 $\mu\text{m}$ sieve, expressed in percent
$Y_P$	yield point, expressed in pounds-force 100 $\text{ft}^2$
$\theta$	temperature, expressed in degrees Celsius
$\theta_1$	average temperature reading at lower temperature, expressed in degrees Celsius (degrees Fahrenheit)
$\theta_2$	average temperature reading at higher temperature, expressed in degrees Celsius (degrees Fahrenheit)
$\rho$	sample density, expressed in grams per milliliters
$\eta$	viscosity of water, expressed in millipascal•seconds (centipoises)
$\eta_{AV}$	apparent viscosity, expressed in millipascal•seconds (centipoises)
$\eta_{oil}$	viscosity of calibrating fluid expressed in millipascal•seconds (centipoises)
$\eta_{20}$	1.002 mPa•s (cP) is the viscosity of water at 20 °C (68 °F)
$\eta_\theta$	viscosity at desired temperature, expressed in millipascal•seconds (centipoises)

### 3.3 Abbreviations

CAS	Chemical Abstracts Service
CMC-HVT	carboxymethyl cellulose—technical grade, high viscosity
CMC-LVT	carboxymethyl cellulose—technical grade, low viscosity
CP	chemical industry pallet
EDTA	ethylenediaminetetraacetic acid
LVNAF	low-viscosity, non-aqueous fluid
NaCMC	sodium carboxymethyl cellulose
OCMA	Oil Companies Materials Association
PAC-LV	polyanionic cellulose—low viscosity
PAC-HV	polyanionic cellulose—high viscosity
SEBC	standard evaluation base clay
TC	to contain
TD	to deliver

## 4 Requirements

### 4.1 Quality Control Instructions

All quality control work shall be controlled by manufacturer's documented instructions, which include appropriate methodology and quantitative or qualitative acceptance criteria.

### 4.2 Use of Test Standard Materials in Checking Testing Procedures

**4.2.1** Test calibration barite, reference (calibration) bentonite, and standard evaluation base clay (SEBC) can be obtained by contacting drilling fluid testing equipment supply companies.

**4.2.2** The supplier of calibration material shall furnish a certificate of analysis for each sample.

**4.2.3** For calibration requirements of test calibration materials and their required periodic use, refer to 5.2.11 and 5.3.11.

**4.2.4** The API Standard Reference Material program qualifies test calibration barite and reference (calibration) bentonite, which can be used for testing and calibration. See Annex D for additional details.

### 4.3 Records Retention

All records specified in this standard shall be maintained for a minimum of 5 years from the date of preparation.

## **5 Calibration**

### **5.1 Coverage**

**5.1.1** Section 5 covers calibration procedures and calibration intervals for laboratory equipment and reagents specified.

**5.1.2** The manufacturer shall control, calibrate, verify, and maintain the laboratory equipment and reagents used in this standard for measuring product conformance to the standard's requirements.

**5.1.3** The manufacturer shall maintain and use laboratory equipment and reagents in a manner such that measurement uncertainty is known and meets required measurement capability.

**5.1.4** The manufacturer shall document and maintain calibration procedures, including details of laboratory equipment and reagent type, identification number, frequency of checks, acceptance criteria, and corrective action that shall be taken when results are unsatisfactory.

**5.1.5** The manufacturer shall establish and document responsibility for administration of the calibration program and responsibility for corrective action.

**5.1.6** The manufacturer shall document and maintain calibration records for laboratory equipment and reagents; shall review these records for trends, sudden shifts, or other signals of approaching malfunction; and shall identify each item with an indicator or approved identification record to show calibration status.

### **5.2 Equipment Requiring Calibration**

#### **5.2.1 Volumetric Glassware**

Manufacturers of products to this standard shall document evidence of glassware calibration prior to use. Supplier certification is acceptable. Periodic recalibration is not required.

#### **5.2.2 Laboratory Thermometers**

##### **5.2.2.1 General**

The manufacturer shall calibrate all laboratory thermometers used in measuring product conformance to standards against a secondary reference thermometer. The secondary reference thermometer shall show evidence of calibration as performed against master instruments certified and traceable to a national/international body such as NIST, ISO, or equivalent, in accordance with the procedures specified by ASTM E77, ISO 386, or NIST National Bureau of Standards Monograph 150.

##### **5.2.2.2 Calibration—Thermometers**

The following procedure shall be used to calibrate a thermometer.

- a) Place the thermometer being calibrated side by side with a secondary reference thermometer into a constant-temperature water bath [or suitable container of 1 gal (4 L) or more, filled with water, on a counter in a constant-temperature room] and allow to equilibrate for at least 1 h.
- b) Read both thermometers and record readings.
- c) Repeat readings throughout at least a 5 min interval to obtain a minimum of four readings.
- d) Calculate the average and the range of readings for each thermometer. The difference between the range of readings for each thermometer shall not exceed  $\pm 0.1^\circ\text{C}$  ( $\pm 0.2^\circ\text{F}$ ) or the smallest scale division on the thermometer being calibrated.



- e) Calculate the average deviation of the thermometer reading from the secondary reference thermometer reading. Calculate and document the correction for the thermometer being calibrated.

### 5.2.3 Laboratory Balances

**5.2.3.1** Laboratory balances shall be periodically calibrated in the range of use with ASTM E617 Class 4, or better, weights.

**5.2.3.2** The laboratory balances shall be serviced and adjusted whenever instrument falls outside calibration range.

### 5.2.4 Sieves

Sieves shall be in accordance with ASTM E11 or ASTM E161. A supplier's verification that a sieve conforms to ASTM E11 or ASTM E161 is satisfactory evidence of compliance.

### 5.2.5 Hydrometer

#### 5.2.5.1 General

Hydrometers shall be calibrated with a prepared dispersant solution to determine the hydrometer reading correction line coefficients.

#### 5.2.5.2 Calibration—Hydrometer Correction

The following procedure shall be used to calibrate a hydrometer so that it can be used for measuring particles less than 6  $\mu\text{m}$  for barite and hematite in opaque solutions using the sedimentation method detailed in 7.12 and 8.12 at temperatures other than 20 °C (68 °F). This calibration and a hydrometer reading correction are needed because the ASTM 151H hydrometer is graduated to read relative density of water at the bottom of the meniscus at 20 °C (68 °F), yet in opaque solutions the top meniscus must be used, the test procedure uses a dispersant solution not water, and test temperatures are at values other than 20 °C (68 °F). The result of the following calibration is to determine the numerical values for the correction line slope,  $M_c$ , using Equation (1) and initial value of the correction line,  $B_c$ , at the lower temperature,  $\theta_1$ , using Equation (2), for the range of test temperatures to be used. This correction line can be used to determine a numerical correction for the observed hydrometer value for any test temperature. However, Equation (10) uses these two values to directly calculate the cumulative percent finer value from the non-corrected hydrometer reading and the test temperature.

- a) Calibrate each hydrometer using the same concentration dispersant solution as is used in the test, at temperatures spanning the anticipated test temperatures, and by reading the top rather than the bottom of the meniscus.
- b) Prepare 1 L of dispersant solution, as follows.
- c) Place 125 mL  $\pm$  2 mL (127 g  $\pm$  2 g) of dispersant solution from test procedure (7.11.2 or 8.11.2) into a 1 L volumetric flask.
- d) Dilute to the 1000 mL mark with deionized or distilled water. Mix thoroughly.
- e) Place the dispersant solution in a sedimentation cylinder. Then place the cylinder in a constant-temperature bath. Set bath temperature to the lowest expected temperature for any actual test. Allow to reach equilibrium  $\pm$  0.2 °C (  $\pm$  0.4 °F). Insert the hydrometer being calibrated and wait at least 5 min for the hydrometer and solution to reach bath temperature.
- f) To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1.010 relative density reading before releasing. After the hydrometer stabilizes, read the top of the meniscus at the dispersant liquid contact at the stem of the hydrometer to the nearest 0.001 relative density graduation.

Repeat readings at least 5 min apart to obtain a minimum of four readings each. Carefully remove the hydrometer, rinse with deionized water, and dry after each reading.

- g) Calculate the average hydrometer relative density reading and designate as  $R_1$ . Calculate the average temperature reading and designate as  $\theta_1$ .
- h) Set the bath temperature to highest expected test temperature and repeat Steps e) and f). Calculate the average hydrometer and temperature readings and designate these readings as  $R_2$  and  $\theta_2$ .
- i) Calculate the hydrometer correction line slope,  $M_c$ , expressed in reciprocal degrees ( $^{\circ}\text{C}^{-1}$  or  $^{\circ}\text{F}^{-1}$ ), as given in Equation (1):

$$M_c = 1000 \frac{(R_1 - R_2)}{(\theta_2 - \theta_1)} \quad (1)$$

where

$R_1$  is the average hydrometer reading at lower temperature;

$R_2$  is the average hydrometer reading at higher temperature;

$\theta_1$  is the average temperature reading ( $^{\circ}\text{C}$  or  $^{\circ}\text{F}$ ) at lower temperature;

$\theta_2$  is the average temperature reading ( $^{\circ}\text{C}$  or  $^{\circ}\text{F}$ ) at higher temperature.

NOTE The temperature may be measured in either degrees Celsius or degrees Fahrenheit. However, all measurements and calculations must be made using consistent units (including subsequent use of the hydrometer in routine test situations).

- j) Calculate the hydrometer correction line initial value,  $B_c$ , at the lower temperature,  $\theta_1$ , dimensionless, as given in Equation (2):

$$B_c = (M_c \times \theta_1) + [1000 \times (R_1 - 1)] \quad (2)$$

where

$M_c$  is the hydrometer correction line slope, expressed in gram per reciprocal degrees Celsius ( $^{\circ}\text{C}^{-1}$ ) or Fahrenheit ( $^{\circ}\text{F}^{-1}$ );

$\theta_1$  is the average thermometer reading ( $^{\circ}\text{C}$  or  $^{\circ}\text{F}$ ) at the lower temperature;

$R_1$  is the average hydrometer reading at the lower temperature.

- k) Record  $M_c$ ,  $B_c$ , and the hydrometer serial number in a permanent calibration record, as shown on Table C.1, and on the datasheet used in the calculations in 7.13 and 8.13, as shown in C.2.1 and C.3.1, respectively.

NOTE For hydrometer calibration example data and calculations of hydrometer correction line coefficients, see C.1.

### 5.2.6 Motor-driven, Direct-indicating Viscometer

**5.2.6.1** The specifications for a direct-indicating viscometer are given in API 13B-1 and included here for reference:

- a) rotor sleeve—R1:
  - inside diameter: 36.83 mm (1.450 in.),
  - total length: 87.0 mm (3.425 in.),
  - scribed line: 58.4 mm (2.30 in.) above the bottom of sleeve, with two rows of 3.18 mm (0.125 in.) holes spaced 120° (2.09 rad) apart, around rotor sleeve just below scribed line;
- b) bob—B1, closed, with flat base and tapered top:
  - diameter: 34.49 mm (1.358 in.),
  - cylinder length: 38.0 mm (1.496 in.);
- c) torsion spring F1.0, constant:
  - torsional stiffness: 10.54 N•m/rad  $\equiv$  0.184 N•m/degree (386 dyne•cm/degree),
  - shear stress constant: 29.3 Pa/rad  $\equiv$  0.511 Pa/degree (1.067 lbf/100•ft<sup>2</sup> per degree);
- d) rotor sleeve speeds:
  - high speed: 600 r/min,
  - low speed: 300 r/min.

NOTE Other rotor speeds are available in viscometers from various manufacturers.

**5.2.6.2** The manufacturer shall calibrate each viscometer with 20 mPa•s (cP) and 50 mPa•s (cP) certified standard silicone fluids.

NOTE One centipoise (cP) is equivalent to 1 millipascal•second (mPa•s).

### 5.2.6.3 Apparatus and Materials—Calibrating a Motor-driven, Direct-indicating Viscometer

**5.2.6.3.1 Standard thermometer**, with an accuracy of  $\pm 0.1$  °C ( $\pm 0.2$  °F), e.g. ASTM 90C or 91C grade.

**5.2.6.3.2 Certified calibration fluid**, of viscosity 20 mPa•s (cP), with chart (viscosity vs temperature).

**5.2.6.3.3 Certified calibration fluid**, of viscosity 50 mPa•s (cP), with chart (viscosity vs temperature).

### 5.2.6.4 Procedure—Calibration a Motor-driven, Direct-indicating Viscometer

The following procedure shall be applied to calibrate a motor-driven, direct-indicating viscometer (R1-B1, F1.0 configuration).

- a) Allow the viscometer and the calibration fluids to achieve temperature equilibrium before testing.
- b) Operate viscometer without fluid a minimum of 2 min to loosen bearing and gears.

- c) Clean and dry viscometer cup. Fill the viscometer cup to the scribed line with 20 cP calibration fluid and place on meter stage. Raise stage until the fluid level reaches the inscribed line on rotor sleeve.
- d) Place thermometer into the fluid and hold or tape to the side of viscometer to prevent breakage.
- e) Operate viscometer at 100 r/min setting until thermometer reading is stable to within  $\pm 0.1$  °C ( $\pm 0.2$  °F). Record the temperature reading.
- f) Take dial readings at 300 r/min and 600 r/min settings. Estimate readings to nearest 0.5 dial unit and record.
- g) Compare 300 r/min dial reading to certified viscosity at test temperature from fluid calibration chart supplied with the calibration fluid. Record readings and deviation from certified calibration fluid viscosity as furnished by supplier. Divide 600 r/min reading by 1.98 to obtain viscosity value at 600 r/min. Compare this value to the certified fluid.
- h) Repeat Steps a) through g) using the 50 mPa•s (cP) fluid.
- i) Compare the deviations to the values in Table 1. Tolerances shall not exceed values in Table 1.

**Table 1—Dial Reading Tolerances with Various Calibration Fluids, F-1 Spring (or Equivalent) in Motor-driven, Direct-indicating Viscometer**

Calibration Fluid	Acceptable Tolerance mPa•s (cP)	
	300 r/min	[600 r/min]/1.98
20 mPa•s (cP)	$\pm 1.5$	$\pm 1.5$
50 mPa•s (cP)	$\pm 1.5$	$\pm 1.5$

## 5.2.7 Laboratory Pressure-measuring Devices

### 5.2.7.1 General

The manufacturer shall document evidence of the laboratory pressure-measuring device calibration prior to use.

### 5.2.7.2 Calibration—Laboratory Pressure-measuring Device

**5.2.7.2.1** Regarding type and accuracy, the dial markings on the pressure-measuring devices shall be readable to at least 2.5 % of full-scale range.

**5.2.7.2.2** Pressure-measuring devices shall be calibrated to maintain  $\pm 2.5$  % accuracy of full-scale range.

**5.2.7.2.3** The pressure measurements for calibration shall be made at not less than 25 % nor more than 75 % of the full-pressure span of the pressure gauge.

**5.2.7.2.4** Pressure-measuring devices shall be calibrated annually with a master reference pressure-measuring device or a dead-weight tester. At least three equidistant points between 25 % and 75 % of full scale shall be used (excluding zero and full scale as required points of calibration).

## 5.2.8 Mixer

The user shall verify that all spindles rotate at 11,500 r/min  $\pm 300$  r/min under no load with one spindle operating. Each spindle shall be fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter mounted flash side up. New impellers shall be weighed prior to installation, with mass and date recorded.

## 5.2.9 Timers

Digital or mechanical timers shall be accurate to within  $\pm 5$  s/h and shall be verified over a period no less than 12 min. Calibration may use the time signal from the NIST or similar websites or radio stations. If not within required accuracy, the timer shall be adjusted or rejected.

## 5.2.10 Chemicals and Solutions

### 5.2.10.1 General

Chemicals shall meet ACS or international equivalent reagent grade.

### 5.2.10.2 Deionized or Distilled Water

The user shall develop, document, and implement a method to determine hardness of water. The water shall not be used if hardness is indicated.

### 5.2.10.3 Chemicals and Reagents—Calibration of EDTA Solution

EDTA solution prepared as per 7.5.2 (or 8.5.2) shall require the following reagents.

**5.2.10.3.1 Hardness indicator solution**, see 7.5.4 (or 8.5.4).

**5.2.10.3.2 Hardness buffer solution**, see 7.5.3 (or 8.5.3).

**5.2.10.3.3 Standard calcium chloride solution**,  $c_{(\text{CaCl}_2)} = 0.0100 \text{ mol/L} \pm 0.0001 \text{ mol/L}$ .

### 5.2.10.4 Procedure—Calibration of EDTA Solution

The following procedure shall be applied for calibrating an EDTA solution.

- a) To a suitable flask, add 50.00 mL  $\pm 0.05$  mL of deionized water and 50.00 mL  $\pm 0.05$  mL of standard calcium chloride solution.
- b) Proceed as in 7.6 a) through 7.6 e) or 8.6 a) through 8.6 e), but without adding barite or additional water. Use the 100 mL solution prepared above in place of the 100 mL  $\pm 1$  mL deionized water specified in 7.6 a) or 8.6.a).
- c) Calculate the EDTA solution calibration correction for calcium,  $C_c$ , expressed in milligram per kilogram, as given in Equation (3):

$$C_c = C_m - 200 \quad (3)$$

where

$C_m$  is the measured calcium concentration in the standard calcium chloride solution expressed in mg/kg;

and  $C_m$  equals 40 times the EDTA volume used in step b), expressed in milliliters.

NOTE The EDTA solution calcium calibration correction, as determined by this procedure, results in a number that is subtracted from the barite sample calcium test value,  $C_s$ .

- d) Calculate the corrected EDTA solution calcium test concentration,  $C_{sc}$ , expressed in milligrams per kilogram as given in Equation (4).

$$C_{sc} = C_s - C_c \quad (4)$$

where

$C_s$  is the calcium test value for the sample, expressed in milligrams per kilograms;

$C_c$  is the EDTA solution calibration correction, expressed in milligrams per kilograms.

#### EXAMPLE

Step 1 Calibration correction for EDTA solution determination:

EDTA volume for the standard  $\text{CaCl}_2$  solution is equal to 4.8 mL:

$$C_m = 40 \times 4.8 = 192 \text{ mg/kg}$$

Equation (3):  $C_c = 192 - 200 \text{ mg/kg} = -8 \text{ mg/k}$

Step 2 Corrected calcium test concentration for barite:

EDTA for the barite sample is equal to 6.1 mL:

Test value for calcium of the sample:

$$C_s = 40 \times 6.1 = 244 \text{ mg/kg}$$

Step 3 Corrected test value for calcium of the sample,

Equations (4), (6), or (14):

$$C_{sc} = C_s - C_c = 244 - (-8) = 252 \text{ mg/kg}$$

### 5.2.11 Test Calibration Materials

The user shall perform in-house verification of calibration barite and/or (where applicable) reference bentonite for properties listed with the material's certificates of analysis, as required by this standard. These results shall be within the "reproducibility limit (interlab)" listed in Table B.1 and Table B.3.

## 5.3 Calibration Intervals

### 5.3.1 General

Any instrument subjected to conditions that can affect its calibration shall be recalibrated prior to use.

### 5.3.2 Thermometers

Each thermometer shall be calibrated before its first use. After calibration, mark each thermometer with an identifying number that ties it to its corresponding correction chart. Calibration shall be checked annually against the secondary reference thermometer.

### 5.3.3 Laboratory Balances

Each balance shall be calibrated prior to its first use. Calibration shall be checked at least monthly for 6 months, then at least once every 6 months if required measurement accuracy is being maintained. If not, service and recalibrate, then check at least monthly until the required measurement accuracy is maintained for 6 months, then once per 6 months.

### **5.3.4 Sieves**

No calibration of sieves is required. A supplier's verification that a sieve shall conform to ASTM E11 or ASTM E161 is a satisfactory evidence of compliance. See 5.3.11 for periodic measurement requirements using standard reference materials.

### **5.3.5 Hydrometers**

Each hydrometer prior to its first use shall be calibrated. After calibration, note and record each hydrometer identifying number that ties it to its correction chart. Periodic recalibration is not required.

### **5.3.6 Motor-driven, Direct-indicating Viscometer**

Each viscometer shall be calibrated prior to its first use. Calibration shall be checked at least once per week for 3 months, then at least once per month if required measurement capability is being maintained.

### **5.3.7 Laboratory Pressure-measuring Devices**

The user shall document evidence of laboratory pressure-measuring device calibration prior to its being placed into first use by the manufacturer, then annually thereafter.

### **5.3.8 Mixer**

Mixer spindle speed shall be checked at least once every month to ensure that the operation falls within the prescribed range, using a photo-tachometer or similar device. Remove, clean, dry, and weigh each impeller blade in use at least once every 90 days. Record masses and replace blades when the mass drops below 90 % of its original value.

### **5.3.9 Timers**

All timers shall be checked before its first use, and then check calibration annually.

### **5.3.10 Deionized or Distilled Water**

The user shall determine the hardness of the water whenever a new batch of water is prepared or purchased or whenever the deionizing cartridges are replaced.

### **5.3.11 Test Calibration Materials**

The user shall test the applicable test calibration material(s) at least once per 40 tests. The API Standard Reference Material program qualifies test calibration barite and reference (calibration) bentonite, which can be used for this purpose (see Annex D).

## **6 Packaged Material**

### **6.1 Description**

**6.1.1** Packaging of palletized goods safeguards the safe handling, transport, storage, and identification and minimizes damage and spillage. Packed material should be inside the dimensions of the pallet although some overhang is allowed.

**6.1.2** This procedure applies to products covered by this standard. The main intention is to improve the possibility of recycling of all packaging materials for components used in drilling fluids, completion fluids, and oil well cements, including dry, powdered, or granular materials not covered under this standard.

## 6.2 Apparatus—Pallets

**6.2.1** The preferred pallet design and construction should be in accordance with ISO 6780.

**6.2.2** Preferred sizes for wooden chemical industry pallets (CP) include the following:

- a) 120 cm × 100 cm (47 in. × 39 in.) CP6;
- b) 114 cm × 114 cm (45 in. × 45 in.) CP8/CP9/CP3;
- c) 122 cm × 122 cm (48 in. × 48 in.);
- d) 112 cm × 1300 cm (44 in. × 52 in.);
- e) 110 cm × 130 cm (42 in. × 52 in.), equivalent to CP4/CP7;
- f) 102 mm × 1219 mm (40 in. × 48 in.).

NOTE CP number is the size of a chemical industry pallet as defined in ISO 6780.

**6.2.3** Other pallet sizes and details concerning design and construction should be agreed upon by the manufacturer and the customer.

**6.2.4** The maximum outside dimensions of the total package shall be in accordance with the applicable pallet size plus a maximum overhang of 30 mm (1.2 in.). The overall height shall not exceed 2.0 m (80 in.).

**6.2.5** The maximum net mass shall not exceed 2000 kg (4400 lb) unless agreed upon by the manufacturer and the customer.

## 6.3 Apparatus—Bags

**6.3.1** When filling the bag, reasonable steps should be taken to ensure that the bag construction is capable of safe handling, transport, and storage.

**6.3.2** Reasonable steps should be taken to select bags that minimize waste and provide the possibility for recycling of the packaging material.

**6.3.3** Humidity-barrier capabilities of the bags relative to the needs of the particular product should be considered when selecting bags.

## 6.4 Marking—Palletized Material

Markings should include the following where applicable and as specified by individual contracts:

- a) product name;
- b) gross/net mass, in pounds (kilograms);
- c) other information as required, such as manufacturer's name, gross allowable mass, and disposal options.

## 6.5 Marking—Bags

Markings shall include the following, where applicable and as specified by individual contracts:

- a) name of the material in print script at least 13 mm (0.5 in.) high;



- b) mass, which shall be denominated in pounds (kilograms), of the material in letters, or numbers and letters, at least 6 mm (0.25 in.) high;
- c) lot/batch number in print script and/or numbers at least 3 mm (0.125 in.) high, traceable to the manufacturer's country of origin;
- d) safety information;
- e) identification as recyclable.

## **6.6 Pallet Covers**

**6.6.1** Each unit of palletized material should have a cover appropriate to its intended use and expected conditions. Examples of materials commonly used include:

- a) polyethylene (PE) shrink or wrapped film;
- b) PE bonnet type;
- c) polypropylene (PP) bonnet type.

**6.6.2** All plastics in a pallet cover should be UV stabilized, unless otherwise requested. Cardboard, carton, or wood covers may be used in place of the above. If appropriate, a bottom layer of cardboard, PE sheet, or plywood may be connected to the cover to unitize the overall package.

## **6.7 Package Mass**

Each sack shall contain a specified net mass  $\pm 5\%$ . The average mass of 5 % of all sacks in a shipment, taken at random, shall not be less than the specified mass.

## **6.8 Storage**

The supplier shall advise on product storage.

## **6.9 Recycling**

If appropriate, recycling of the remaining materials should be in accordance with local instructions and in compliance with the local regulatory administration concerned.

**NOTE** When handling chemicals, reduction in the volume of packaging materials is possible by using dedicated non-disposable, refillable containers.

# **7 Barite**

## **7.1 Principle**

**7.1.1** Drilling-grade barite is produced from commercial barium sulfate containing ores. The manufacturer shall retain certificates of analysis or similar documentation on these commercial barium sulfate ores. It may be produced from a single ore or a blend of ores and may be a straight-mined product or processed by beneficiation methods, i.e. washing, tabling, jigging, or flotation. It may contain minerals in addition to the barium sulfate ( $\text{BaSO}_4$ ) mineral. Common barite impurities include silicates such as quartz and chert, carbonate compounds such as siderite and dolomite, metallic oxide, and sulfide compounds. Because of mineral impurities, commercial barite can vary in color from off-white to grey to red or brown. Although these impurities are normally insoluble under certain conditions, they can react with other components in some drilling fluids and cause adverse changes in drilling fluid properties. (See API 13K for more details on barite testing.)

**7.1.2** Drilling-grade barite shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the chemical and physical specifications of Table 2, represents the product produced, and is controlled by the manufacturer. This test procedure is to be calibrated using test calibration barite at least once per 40 tests, as described in 5.3.11.

NOTE API monogrammed barite is currently available in 4.1 relative density and 4.2 relative density grades that otherwise meet identical test specifications. 4.1 Barite grade may not be appropriate for higher mud weights.

**Table 2—Barite Physical and Chemical Requirements**

Requirement	Standard	
Density	4.10 g/mL minimum	4.2 g/mL minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum	
Residue greater than 75 $\mu\text{m}$	maximum mass fraction 3.0 %	
Particles less than 6 $\mu\text{m}$ in equivalent spherical diameter	maximum mass fraction 30 %	

## **7.2 Reagents and Apparatus—Measuring Barite Density by the Le Chatelier Flask Method**

**7.2.1 Low-viscosity, non-aqueous fluid (LVNAF)**, including but not limited to kerosene, mineral spirits, or mineral oils.

**7.2.2 Oven**, regulated to 105 °C  $\pm$ 2.5 °C (220 °F  $\pm$ 5 °F).

**7.2.3 Desiccator**, with calcium sulfate [Chemical Abstracts Service (CAS) No. 7778-18-9] desiccant or equivalent.

**7.2.4 Le Chatelier flask**, with graduations of 0.1 mL, clamped or weighted to prevent flotation in water bath.

**7.2.5 Constant-temperature bath**, transparent, at 32 °C  $\pm$ 0.5 °C regulated to  $\pm$ 0.1 °C (90 °F  $\pm$ 1 °F regulated to  $\pm$ 0.2 °F), e.g. an approximately 40 L (10 gal) aquarium (fish tank) with heater/circulator attachment, or functional equivalent.

**7.2.6 Balance**, with accuracy of  $\pm$ 0.01 g.

**7.2.7 Pipette**, volumetric, 10 mL.

**7.2.8 Magnifying glass**.

**7.2.9 Dowel**, wooden, approximately 8 mm (0.33 in.) in diameter and 30 cm (12 in.) in length, or a functional equivalent.

**7.2.10 Tissue paper**, absorbent.

NOTE Laboratory-grade tissues are non-absorbent and unsuitable for use in this test procedure.

**7.2.11 Weighing dish**, low form, with spout, approximately 100 mL capacity, or a functional equivalent.

**7.2.12 Brush**, small, fine bristle.

## **7.3 Procedure—Measuring Barite Density by Le Chatelier Flask Method**

The following procedure shall be applied to measure barite density using the Le Chatelier flask method.

- a) Take approximately 100 g of powdered barite that has been oven dried for at least 2 h to a constant mass and cooled to room temperature in a desiccator.
- b) Fill a clean Le Chatelier flask to approximately 22 mm (0.8 in.) below the zero mark with LVNAF.
- c) Place the flask upright in the constant-temperature bath. The level of water in the bath shall be higher than the 24 mL graduation of the flask but below the stopper level. Assure that the flask is stabilized using clamps or weights.
- d) Allow the flask and contents to equilibrate for a minimum of 1 h. Using the magnifying glass with care to keep eyes at meniscus level, read the volume at the lowest portion of the curved interface, and record the initial volume, in milliliters, to the nearest 0.05 mL without removing the flask from the constant-temperature bath. Record as  $V_1$ .

If the LVNAF level is outside the  $-0.2$  mL to  $+1.2$  mL volume range after equilibrating, use the 10 mL pipette to add or remove LVNAF to bring it within this range. Allow the flask to equilibrate for at least 1 h and record the initial volume,  $V_1$ , in milliliters, as in Step d).

- e) Remove the Le Chatelier flask from the bath, wipe dry, and remove the stopper. Roll several lengths of tissue paper diagonally along the length of the dowel and use this assembly as a swab to dry the inside neck of the flask. Do not allow the swab to come into contact with the LVNAF in the flask.
- f) Weigh  $80.00 \text{ g} \pm 0.05 \text{ g}$  of dried barite into the weighing dish and carefully transfer it to the Le Chatelier flask. Take care to avoid splashing the LVNAF or plugging the flask with barite at the bulb. This is a slow process, requiring repeated transfers of small amounts of barite. Use a brush to transfer any residual barite into the flask, and then replace the stopper. Record the mass, in grams, as  $m$ .
- g) If necessary, carefully tap the neck of the flask with the wooden dowel, or agitate carefully side to side, to dislodge any barite clinging to the walls. Do not allow LVNAF to come into contact with the ground glass stopper joint of the flask.
- h) Gently roll the flask along a smooth surface at no more than  $45^\circ$  from vertical or twirl the upright flask at the neck vigorously between the palms of both hands to remove entrained air from the barite sample. Repeat this procedure until no more bubbles can be seen rising from the barite.
- i) Return the flask to the bath and let stand for at least 30 min.
- j) Remove the flask from the bath and repeat Step h) to remove any remaining air from the barite sample.
- k) Immerse the flask in the bath again for at least 1 h.
- l) Record the final volume, in milliliters, in the same manner as described in Step d). Record the volume as  $V_2$ .

#### 7.4 Calculation—Barite Density by Le Chatelier Flask Method

Calculate the barite density,  $\rho$ , in grams per milliliter, according to Equation (5):

$$\rho = \frac{m}{(V_2 - V_1)} \quad (5)$$

where

$m$  is the sample mass, expressed in grams;

$V_1$  is the initial volume, expressed in milliliters;

$V_2$  is the final volume, expressed in milliliters.

Record the calculated density.

## 7.5 Reagents and Apparatus—Measuring Water-soluble Alkaline Earths as Calcium in Barite

**7.5.1** Chemicals shall meet ACS or international equivalent reagent grade.

**7.5.2 Aqueous EDTA solution**, composed of 3.72 g  $\pm$ 0.01 g of disodium salt of EDTA dihydrate (disodium salt of ethylenedinitrilo-tetraacetic acid dihydrate) (CAS No. 6381-92-6) diluted to a final volume of 1000 mL with deionized water in a volumetric flask.

**7.5.3 Hardness buffer solution**, comprising 67.50 g  $\pm$ 0.01 g of ammonium chloride (CAS No. 12125-02-9) and 570 mL  $\pm$ 1 mL of 15 mol/L ammonium hydroxide (CAS No. 1336-21-6) solution diluted to a final volume of 1000 mL with deionized water in a volumetric flask.

**7.5.4 Hardness indicator solution**, comprising 1.00 g  $\pm$ 0.01 g of [1-(1-hydroxy-4-methylphenylazo)-2-naphthol-4-sulfonic acid] or equivalent such as calmagite (CAS No. 3147-14-6), diluted to a final volume of 1000 mL with deionized water in a volumetric flask.

**7.5.5** Deionized or distilled water.

**7.5.6 Balance**, of capacity exceeding 100 g with an accuracy  $\pm$ 0.01 g.

**7.5.7 Conical flask** (Erlenmeyer), 250 mL nominal capacity, equipped with a tight-fitting stopper.

**7.5.8 Graduated cylinder**, 100 mL to 150 mL to contain (TC) with 1 mL graduations.

**7.5.9 Titration vessel**, e.g. beaker, 100 mL to 150 mL capacity.

**7.5.10 Serological pipettes or burette**, with graduations of 0.1 mL.

**7.5.11 Volumetric pipettes** to deliver (TD), of capacity 10 mL, or equivalent.

**7.5.12 Filter press**, low pressure/low temperature, in accordance with API 13B-1, or filtration funnel.

**7.5.13 Filter paper**<sup>5</sup>, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1.

**7.5.14 Glass container**, small.

**7.5.15 Magnetic stirrer and stir bar**.

**7.5.16 Wrist-action shaker**, optional.

**7.5.17 Volumetric flask**, 1000 mL.

**7.5.18 Stirring rod**.

<sup>5</sup> This grade 50 filter paper is a thin, highly glazed, qualitative, hardened low-ash filter paper made from high-quality cotton linters, designed for slow filtration and the retention of very fine crystalline precipitates. It should have a lint-free surface and be highly resistant to acid and alkaline solutions. The filter paper has the following typical properties that may vary by manufacturer. Slow filtration rate: 2685 s/100 mL (herzbergs); particle retention in liquid: 2  $\mu$ m to 5  $\mu$ m (at 98 % efficiency); ash content: <0.015 % by weight; basis weight: 96 g/m<sup>2</sup> (0.02 lb/ft<sup>2</sup>); and thickness: 0.115 mm (0.0045 in.).

## 7.6 Procedure—Measuring Water-soluble Alkaline Earth Metals as Calcium in Barite

To measure water-soluble alkaline earth metals as calcium in a barite sample, the following procedure shall be applied.

- a) Weigh 100.00 g  $\pm$  0.05 g of barite. Transfer to the Erlenmeyer flask and add 100 mL  $\pm$  1 mL of deionized water. Stopper the flask and shake by hand for 1 min at 5 min intervals during an approximate 1 h period or use a mechanical shaking apparatus at least 20 min or stirring fast enough to form a vortex using a magnetic stirrer and stir bar for 1 h.
- b) After shaking or stirring, filter the suspension through the low-pressure filter cell or funnel using two sheets of filter paper and collect filtrate in a suitable glass container.
- c) Add 50 mL  $\pm$  1 mL of deionized water to the titration vessel. Add 2 mL of hardness buffer and add hardness indicator until a distinct blue color is achieved. Swirl to mix.

A solution with color other than distinct blue at this point indicates contamination of equipment and/or water. Find and eliminate the source of contamination and rerun the test.

- d) Using the volumetric pipette, measure 10 mL of the filtrate into the titrating vessel. Swirl to mix. A blue color indicates no calcium hardness and the test is complete. A wine-red color develops if calcium and/or magnesium are present, indicating hardness.
- e) If hardness is present, begin stirring and titrate with EDTA solution to the blue endpoint. The endpoint of the titration is the point at which additional EDTA produces no further red to blue change. The EDTA volume used to produce the blue endpoint is used in the calculation in 7.7. Record as  $V_3$  in milliliters.

If endpoint is unclear or unobtainable, other tests shall be performed. Results and methodology of these tests shall be recorded.

## 7.7 Calculation—Measuring Water-soluble Alkaline Earths as Calcium in Barite

Calculate the concentration (corrected) of soluble alkaline earth metals as calcium,  $C_{Ca,c}$ , in barite, expressed in milligrams per kilogram, according to Equation (6):

$$C_{Ca,c} = (40 \times V_3) - C_c \quad (6)$$

where

$V_3$  is the volume EDTA used, expressed in milliliters;

$C_c$  is the EDTA calibration correction determined as per 5.2.10.4, expressed in milligrams per kilogram.

Record the calculated corrected soluble alkaline earth metals value of the barite sample.

## 7.8 Reagents and Apparatus—Measuring Barite Residue Greater than 75 $\mu$ m

**7.8.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution, 10 %  $\pm$  0.5 % by mass.

**7.8.2 Oven**, regulated to 105 °C  $\pm$  2.5 °C (220 °F  $\pm$  5 °F).

**7.8.3 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

**7.8.4 Balance**, with an accuracy of  $\pm$  0.01 g.

**7.8.5 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**7.8.6 Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**7.8.7 Sieve**, 75  $\mu\text{m}$  (0.0029 in., U.S. No. 200), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**7.8.8 Spray nozzle** with 6.35 mm ( $1/4$  in.) inlet connection, rated for 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.<sup>2</sup>) with approximately 45° spray angle attached to a water line with a 90° elbow.

**7.8.9 Water-pressure regulator**, capable of regulation to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>).

**7.8.10 Evaporating dish** or functional equivalent.

**7.8.11 Wash bottle**.

## 7.9 Procedure—Measuring Barite Residue Greater than 75 $\mu\text{m}$

When measuring barite residue of diameter greater than 75  $\mu\text{m}$  in a sample, the following procedure shall be applied.

- Take approximately 60 g of powdered barite that has been oven dried for at least 2 h to a constant mass and cooled to room temperature in a desiccator.
- Weigh 50.00 g  $\pm$  0.01 g of dried barite. Record the mass, in grams, as  $m$ . Add the weighed sample to 350 mL  $\pm$  3 mL of water containing 0.20 g  $\pm$  0.05 g of sodium hexametaphosphate dispersant in the container. Sodium hexametaphosphate dispersant may be added as 0.20 g  $\pm$  0.05 g of sodium hexametaphosphate powder or by adding 2.0 mL  $\pm$  0.1 mL of sodium hexametaphosphate 10 %  $\pm$  0.5 % by mass solution. Add weighed barite sample while stirring on the mixer. Stir with the mixer for 5 min  $\pm$  10 s.
- Transfer the sample to the 75  $\mu\text{m}$  sieve. Use a wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>) from a spray nozzle for 2 min  $\pm$  15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
- Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.
- Dry the residue in the oven to a constant mass. Record the residue mass, in grams, as  $m_2$  and total drying time.

## 7.10 Calculation—Barite Residue Greater than 75 $\mu\text{m}$

Calculate the mass fraction of barite residue particles greater than 75  $\mu\text{m}$ ,  $w_1$ , in percent, according to Equation (7):

$$w_1 = 100 \left( \frac{m_2}{m} \right) \quad (7)$$

where

$m$  is the sample mass, expressed in grams;

$m_2$  is the residue mass retained by 75  $\mu\text{m}$  sieve, expressed in grams.

Record the calculated value.

## 7.11 Reagents and Apparatus—Measuring Barite Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter by Sedimentation Method

7.11.1 Chemicals shall meet ACS or international equivalent reagent grade.

7.11.2 **Dispersant solution**, comprised of 40.0 g  $\pm$  0.1 g of sodium hexametaphosphate (CAS No. 10124-56-8) and 3.6 g  $\pm$  0.1 g of anhydrous sodium carbonate (CAS No. 497-19-8) per 1000 mL of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9.0.

7.11.3 **Oven**, regulated to 105 °C  $\pm$  3 °C (220 °F  $\pm$  5 °F).

7.11.4 **Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

7.11.5 **Balance**, with an accuracy of  $\pm$  0.01 g.

7.11.6 **Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

7.11.7 **Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

7.11.8 **Sedimentation cylinder**, glass, approximately 457 mm (18 in.) high and 63 mm (2.5 in.) in diameter, marked for a volume of 1000 mL (in accordance with ASTM D422-63).

7.11.9 **Rubber stopper**, size No. 13.

7.11.10 **Water bath** or **constant-temperature room**, capable of maintaining a constant temperature of 24 °C  $\pm$  5 °C (75 °F  $\pm$  10 °F).

7.11.11 **Thermometer**, including the range 16 °C to 32 °C (60 °F to 90 °F) readable to 0.5 °C (1 °F).

7.11.12 **Hydrometer**, ASTM 151H, graduated to read the relative density (specific gravity) of the suspension, range 0.995 to 1.038, graduation as 0.001 relative density, temperature 20 °C/20 °C (68 °F /68 °F).

7.11.13 **Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

7.11.14 **Spatula**.

## 7.12 Procedure—Measuring Barite Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter by Sedimentation Method

To measure particles less than 6  $\mu\text{m}$  in equivalent spherical diameter by sedimentation method in a barite sample, the following procedure in accordance with ASTM D422-63 shall be applied.

- Weigh 80.00 g  $\pm$  0.01 g of the dry barite [see 7.3 a) or 7.9 a)] and place in container. Record the mass, in grams, as  $m$ .
- Add 125 mL  $\pm$  2 mL (127 g  $\pm$  2 g) of dispersant solution (7.11.2). Dilute to approximately 400 mL with deionized water. Rinse all adhering particles from the spatula into the suspension.
- Stir for 5 min  $\pm$  6 s on a mixer.
- Transfer the suspension to the sedimentation cylinder. Rinse container with deionized water to assure that all sample particles are transferred to the sedimentation cylinder.

- e) Add deionized water to the 1000 mL mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s while holding a No. 13 rubber stopper in the top of the cylinder.

This is a critical step. The suspension shall be homogeneous at the start of sedimentation. This is difficult to obtain because of the high density of barite.

- f) Set the cylinder into the water bath (or on the counter-top of a constant-temperature room) and simultaneously start the timer. Hang the thermometer in the water bath.
- g) Take hydrometer readings at intervals of 10 min  $\pm$  6 s, 20 min  $\pm$  6 s, 30 min  $\pm$  6 s, and 40 min  $\pm$  6 s (or until the first point below the 6  $\mu$ m value is reached). To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1.020 relative density reading before releasing. After the hydrometer stabilizes, read the top of the meniscus at the prescribed time. Carefully and slowly remove the hydrometer, rinse with deionized water, and dry after each reading. The hydrometer shall be removed immediately after each reading to eliminate particle buildup on the shoulders, which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve the suspension-settling equilibrium.
- h) Record the time,  $t$ , expressed in minutes, the temperature,  $\theta$ , expressed in degrees Celsius (degrees Fahrenheit) and the hydrometer reading,  $R$ , on the datasheet.

NOTE The temperature may be measured in either degrees Celsius or degrees Fahrenheit. However, all measurements and calculations must be made using consistent units including hydrometer calibration (or subsequent use of the hydrometer in routine test situations).

- i) For each time interval, determine the water viscosity,  $\eta$ , and the effective hydrometer depth,  $L$ , from Table 3 [or Equation (12)] and Table 4. Record on the datasheet.

### 7.13 Calculation—Barite Particles Less than 6 $\mu$ m in Equivalent Spherical Diameter by Sedimentation Method

**7.13.1** From the hydrometer calibration (5.2.5), enter the hydrometer correction line slope,  $M_c$ , and the hydrometer correction line initial value,  $B_c$ , onto the datasheet.

NOTE Annex C contains example calculations for hydrometer calibration values, hydrometer correction line coefficient calculations, and a complete example calculation for particles less than 6  $\mu$ m in equivalent spherical diameter by sedimentation method.

**7.13.2** Calculate the sample constant,  $K_s$ , expressed in percent per gram as given in Equation (8) or determine from Table 5 and enter into datasheet:

$$K_s = 100 \frac{\rho}{m(\rho - 1)} \quad (8)$$

where

$\rho$  is the sample density, expressed in grams per milliliter;

$m$  is the sample mass, expressed in grams.



**7.13.3** Calculate and enter onto the datasheet the equivalent spherical diameter,  $D_e$ , in micrometers, for each time interval as given in Equation (9):

$$D_e = 17.5 \sqrt{\frac{\eta L}{(\rho - 1) t}} \quad (9)$$

where

$\eta$  is the viscosity of water, expressed in centipoises millipascal•seconds (centipoises), from Table 3 or Equation (12);

$\rho$  is the sample density, expressed in grams per milliliter;

$t$  is the time, expressed in minutes;

$L$  is the effective hydrometer depth (see Table 4), expressed in centimeters.

**7.13.4** For each time interval, calculate and enter onto the datasheet the cumulative percent finer,  $w_a$ , for the equivalent particle diameter,  $D_e$ , expressed in micrometers as given in Equation (10):

$$w_a = K_s \left[ (M_c \times \theta) - B_c + (R - 1) \times 1000 \right] \quad (10)$$

where

$K_s$  is the sample constant, as per Equation (8) or Table 5, expressed in percent per gram;

$M_c$  is the hydrometer correction line slope, determined in Equation (1), expressed in reciprocal degrees Celsius ( $^{\circ}\text{C}^{-1}$ ) [degrees Fahrenheit ( $^{\circ}\text{F}^{-1}$ )];

$\theta$  is the suspension temperature, expressed in degrees Celsius (degrees Fahrenheit);

$B_c$  is the hydrometer correction line initial value at  $\theta_1$ , dimensionless, as determined in Equation (2);

$R$  is the hydrometer relative density (specific gravity) reading at the top of the meniscus (not corrected).

Using this procedure and Equation (10), the cumulative percent finer,  $w_2$ , for particle diameter,  $D_1$ , immediately greater than 6  $\mu\text{m}$  and the cumulative percent finer,  $w_3$ , for particle diameter,  $D_2$ , immediately less than 6  $\mu\text{m}$  data are determined.

**7.13.5** Calculate and enter onto the datasheet the cumulative percent for barite particles less than 6 µm in equivalent spherical diameter,  $w_4$ , as given in Equation (11):

$$w_4 = \left[ \left( \frac{w_2 - w_3}{D_1 - D_2} \right) \times (6 - D_2) \right] + w_3 \quad (11)$$

where

$w_2$  is the cumulative percent for the point immediately greater than 6 µm;

$w_3$  is the cumulative percent for the point immediately less than 6 µm;

$D_1$  is the equivalent particle diameter immediately greater than 6 µm, determined in Equation (10);

$D_2$  is the equivalent particle diameter immediately less than 6 µm, determined in Equation (10).

For an example of the calculation for barite particles less than 6 µm in equivalent spherical diameter, see C.2.

**7.13.6** The viscosity of water at any temperature between 20 °C (68 °F) and 150 °C (302 °F) can be calculated<sup>[13]</sup> using Equation (12):

$$\eta_{\theta} = \frac{1.002}{10^{\left( \frac{1.370\,23 \times (\theta - 20) + 8.36 \times 10^{-4} \times (\theta - 20)^2}{(109 + \theta)} \right)}} \quad (12)$$

where

$\eta_{\theta}$  is the water viscosity at desired temperature, expressed in pascal•seconds (centipoises) (see Table 3);

$\theta$  is the temperature, in degrees Celsius (degrees Fahrenheit).

NOTE 1 1.002 is the viscosity of water at 20 °C (68 °F) expressed in pascal•seconds (centipoises).

NOTE 2 To convert temperature expressed in degrees Fahrenheit to degrees Celsius:  $^{\circ}\text{C} = \frac{5}{9}(^{\circ}\text{F} - 32)$ .

**Table 3—Viscosity of Water at Various Temperatures**

Temperature $\theta$ °C	Viscosity $\eta$ mPa•s	Temperature $\theta$ °F	Viscosity $\eta$ cP
15.0	1.1375	59.0	1.1375
15.5	1.1227	60.0	1.1211
16.0	1.1082	61.0	1.1050
16.5	1.0940	62.0	1.0893
17.0	1.0800	63.0	1.0739
17.5	1.0664	64.0	1.0589
18.0	1.0530	65.0	1.0442
18.5	1.0399	66.0	1.0298
19.0	1.0270	67.0	1.0158
19.5	1.0144	68.0	1.0020
20.0	1.0020	69.0	0.9885
20.5	0.9899	70.0	0.9753
21.0	0.9780	71.0	0.9624
21.5	0.9663	72.0	0.9498
22.0	0.9548	73.0	0.9374
22.5	0.9436	74.0	0.9253
23.0	0.9325	75.0	0.9134
23.5	0.9217	76.0	0.9018
24.0	0.9111	77.0	0.8904
24.5	0.9006	78.0	0.8792
25.0	0.8904	79.0	0.8683
25.5	0.8803	80.0	0.8576
26.0	0.8705	81.0	0.8470
26.5	0.8607	82.0	0.8367
27.0	0.8512	83.0	0.8266
27.5	0.8419	84.0	0.8167
28.0	0.8327	85.0	0.8070
28.5	0.8236	86.0	0.7974
29.0	0.8147	87.0	0.7881
29.5	0.8060	88.0	0.7789
30.0	0.7974	89.0	0.7699
30.5	0.7890	90.0	0.7610
31.0	0.7807	91.0	0.7523
NOTE 1 Water viscosity values are calculated using Equation (12).			
NOTE 2 1 cP = 1 mPa•s.			

**Table 4—Values of Effective Depth Based on Readings on Hydrometer ASTM 151H  
Used in Specific Sedimentation Cylinder**

Uncorrected Hydrometer Reading <i>R</i>	Effective Depth <i>L</i> cm	Uncorrected Hydrometer Reading <i>R</i>	Effective Depth <i>L</i> cm
1.000	16.3	1.020	11.0
1.001	16.0	1.021	10.7
1.002	15.8	1.022	10.5
1.003	15.5	1.023	10.2
1.004	15.2	1.024	10.0
1.005	15.0	1.025	9.7
1.006	14.7	1.026	9.4
1.007	14.4	1.027	9.2
1.008	14.2	1.028	8.9
1.009	13.9	1.029	8.6
1.010	13.7	1.030	8.4
1.011	13.4	1.031	8.1
1.012	13.1	1.032	7.8
1.013	12.9	1.033	7.6
1.014	12.6	1.034	7.3
1.015	12.3	1.035	7.0
1.016	12.1	1.036	6.8
1.017	11.8	1.037	6.5
1.018	11.5	1.038	6.2
1.019	11.3	—	—

**Table 5—Sample Constant,  $K_s$ , for Barite (80.0 g Sample)**

Sample Density $\rho$ g/mL	Sample Constant $K_s$ % / g	Sample Density $\rho$ g/mL	Sample Constant $K_s$ % / g
4.05	1.6598	4.23	1.6370
4.06	1.6585	4.24	1.6358
4.07	1.6572	4.25	1.6346
4.08	1.6558	4.26	1.6334
4.09	1.6545	4.27	1.6323
4.10	1.6532	4.28	1.6311
4.11	1.6519	4.29	1.6299
4.12	1.6506	4.30	1.6288
4.13	1.6494	4.31	1.6276
4.14	1.6481	4.32	1.6265
4.15	1.6468	4.33	1.6254
4.16	1.6456	4.34	1.6243
4.17	1.6443	4.35	1.6231
4.18	1.6431	4.36	1.6220
4.19	1.6418	4.37	1.6209
4.20	1.6406	4.38	1.6198
4.21	1.6394	4.39	1.6187
4.22	1.6382	4.40	1.6176

## 8 Hematite (Haematite)

### 8.1 Principle

**8.1.1** Drilling-grade hematite is produced from commercial ores and may be a single ore or blends of hematite ores composed of iron oxid. Hematite ores may be a mined product or processed material. Common impurities, including minor amounts of silicon oxide, aluminum oxide, calcium oxide, and magnesium oxide, may be present.

**8.1.2** Drilling-grade hematite shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the chemical and physical specifications of Table 6, represents the product produced, and is controlled by the manufacturer.

**Table 6—Hematite Chemical and Physical Specifications**

Requirement	Standard
Density	5.05 g/mL, minimum
Water-soluble alkaline earth metals, as calcium	100 mg/kg, maximum
Residue greater than 75 $\mu\text{m}$	maximum mass fraction 1.5 %
Residue greater than 45 $\mu\text{m}$	maximum mass fraction 15 %
Particles less than 6 $\mu\text{m}$ in equivalent spherical diameter	maximum mass fraction 15 %

## 8.2 Reagent and Apparatus—Measuring Hematite Density by Le Chatelier Flask Method

**8.2.1 Low-viscosity non-aqueous fluids (LVNAF)**, including but not limited to kerosene, mineral spirits, or mineral oils.

**8.2.2 Oven**, regulated to  $105\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$  ( $220\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ).

**8.2.3 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

**8.2.4 Le Chatelier flask**, clamped or weighted to prevent flotation in water bath.

**8.2.5 Constant-temperature bath**, transparent, at  $32\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$  regulated to  $\pm 0.1\text{ }^{\circ}\text{C}$  ( $90\text{ }^{\circ}\text{F} \pm 1.0\text{ }^{\circ}\text{F}$  regulated to  $\pm 0.2\text{ }^{\circ}\text{F}$ ), e.g. an approximately 40 L (10 gal) aquarium (fish tank) with heater/circulator attachment, or functional equivalent.

**8.2.6 Balance**, with accuracy of  $\pm 0.01\text{ g}$ .

**8.2.7 Pipette**, volumetric, 10 mL.

**8.2.8 Magnifying glass**.

**8.2.9 Dowel**, wooden, approximately 8 mm (0.33 in.) in diameter and 30 cm (12 in.) in length, or a functional equivalent.

**8.2.10 Tissue paper**, absorbent.

NOTE Laboratory-grade tissues are non-absorbent and unsuitable for use in this test procedure.

**8.2.11 Weighing dish**, low form, with spout, approximately 100 mL capacity, or a functional equivalent.

**8.2.12 Brush**, small, fine bristle.

## 8.3 Procedure—Measuring Hematite Density by Le Chatelier Flask Method

To measure the density of a hematite sample with a Le Chatelier flask, the following procedure shall be applied.

- a) Take approximately 120 g of powdered hematite that has been oven dried for at least 2 h to a constant mass and cooled to room temperature in a desiccator.
- b) Fill a clean Le Chatelier flask to approximately 22 mm (0.8 in.) below the zero mark with LVNAF.
- c) Place the flask upright in the constant-temperature bath. The level of water in the bath shall be higher than the 24 mL graduation of the flask but below the stopper level. Assure that the flask is stabilized using clamps or weights.
- d) Allow the flask and contents to equilibrate for a minimum of 1 h. Using the magnifying glass with care to keep eyes at meniscus level, read the volume at the lowest portion of the curved interface and record the initial volume, in milliliters, to the nearest 0.05 mL without removing the flask from the constant-temperature bath. Record as  $V_1$ .

If LVNAF level is outside the  $-0.2\text{ mL}$  to  $+1.2\text{ mL}$  volume range after equilibrating, use the 10 mL pipette to add or remove LVNAF to bring it within this range. Allow the flask to equilibrate for at least 1 h and record the initial volume,  $V_1$ , in milliliters, as in Step d).

- e) Remove the Le Chatelier flask from the bath, wipe dry, and remove the stopper. Roll several lengths of tissue paper diagonally along the length of the dowel and use this assembly as a swab to dry the inside neck of the flask. Do not allow the swab to come into contact with the LVNAF in the flask.
- f) Weigh 100.00 g  $\pm$ 0.05 g of the dried hematite into the weighing dish and carefully transfer to the Le Chatelier flask. Take care to avoid splashing the LVNAF or plugging the flask with hematite at the bulb. This is a slow process, requiring repeated transfers of small amounts of hematite. Use a brush to transfer any residual hematite into the flask, then replace the stopper. Record the mass, in grams, as  $m$ .
- g) If necessary, carefully tap the neck of the flask with the wooden dowel or agitate carefully side to side to dislodge any hematite clinging to the walls. Do not allow the LVNAF to come into contact with the ground-glass stopper joint of the flask.
- h) Gently roll the flask along a smooth surface at no more than 45° from vertical or twirl the upright flask at the neck vigorously between the palms of both hands to remove entrained air from the hematite sample. Repeat this procedure until no more bubbles can be seen rising from the hematite.
- i) Return the flask to the bath and let stand for at least 30 min.
- j) Remove the flask from the bath and repeat Step h) to remove any remaining air from the hematite sample.
- k) Immerse the flask in the bath again for at least 1 h.
- l) Record the final volume,  $V_2$ , in the same manner as described in Step d).

#### 8.4 Calculation—Hematite Density by Le Chatelier Flask Method

Calculate the hematite density,  $\rho$ , in grams per milliliter, according to Equation (13):

$$\rho = \frac{m}{(V_2 - V_1)} \quad (13)$$

where

$m$  is the sample mass, in grams;

$V_1$  is the initial volume, in milliliters;

$V_2$  is the final volume, in milliliters.

Record the calculated density.

#### 8.5 Reagents and Apparatus—Measuring Water-soluble Alkaline Earth Metals as Calcium in Hematite

**8.5.1** Chemicals shall meet ACS or international equivalent reagent grade.

**8.5.2 Aqueous EDTA solution**, comprised of 3.72 g  $\pm$ 0.01 g of disodium salt of EDTA dihydrate (disodium salt of ethylenedinitrilo-tetraacetic acid dehydrate) (CAS No. 6381-92-6) diluted to a final volume of 1000 mL with deionized water in a volumetric flask.

**8.5.3 Hardness buffer solution**, comprised of 67.50 g  $\pm$ 0.01 g of ammonium chloride (CAS No. 12125-02-9) and 570 mL  $\pm$ 1 of 15mol/L ammonium hydroxide solution (CAS No. 1336-21-6), diluted to a final volume of 1000 mL with deionized water in a volumetric flask.

**8.5.4 Hardness indicator solution**, 1.00 g  $\pm$ 0.01 g of [1-(1-hydroxy-4-methylphenylazo)-2-naphthol-4-sulfonic acid] or equivalent such as calmagite (CAS No. 3147-14-6), diluted to a final volume of 1000 mL with deionized water in a volumetric flask.

**8.5.5 Deionized or distilled water.**

**8.5.6 Balance**, of capacity exceeding 100 g with an accuracy of  $\pm$ 0.01 g.

**8.5.7 Conical flask** (Erlenmeyer), 250 mL nominal capacity, equipped with a tight-fitting stopper.

**8.5.8 Graduated cylinder**, 100 mL (TC) with 1 mL graduations.

**8.5.9 Titration vessel**, e.g. beaker, 100 mL to 150 mL.

**8.5.10 Serological pipettes or burette**, graduated to 0.1 mL.

**8.5.11 Volumetric pipettes**, 10 mL (TD), or equivalent.

**8.5.12 Filter press**, low pressure/low temperature, in accordance with API 13B-1, or filtration funnel.

**8.5.13 Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**8.5.14 Glass container**, small.

**8.5.15 Magnetic stirrer and stir bar.**

**8.5.16 Wrist-action shaker**, optional.

**8.5.17 Volumetric flask**, 1000 mL.

**8.5.18 Stirring rod.**

## **8.6 Procedure—Measuring Water-soluble Alkaline Earth Metals as Calcium in Hematite**

To measure water soluble alkaline earth metals as calcium in a sample of hematite, the following procedure shall be applied.

- a) Weigh 100.00 g  $\pm$ 0.05 g of hematite. Transfer to the Erlenmeyer flask and add 100 mL  $\pm$ 1 mL of deionized water. Stopper the flask and shake by hand for 1 min at 5 min intervals during an approximate 1 h period or use a mechanical shaking apparatus at least 20 min or stirring fast enough to form a vortex using a magnetic stirrer and stir bar for 1 h.
- b) After shaking or stirring, filter the suspension through the low-pressure filter cell or funnel using two sheets of filter paper and collect filtrate into suitable glass container.
- c) Add 50 mL  $\pm$ 1 mL of deionized water to the titration vessel. Add about 2 mL of hardness buffer and sufficient hardness indicator to achieve a distinct blue color. Swirl to mix.

A solution with color other than a distinct blue at this point indicates contamination of equipment and/or water. Find and eliminate the source of contamination and rerun the test.

- d) Using the volumetric pipette, measure 10 mL of the filtrate into the titrating vessel. Swirl to mix. A blue color indicates no calcium hardness and the test is complete. A wine-red color develops if calcium and/or magnesium is/are present.



- e) If hardness is present, begin stirring and titrate with EDTA solution to the blue endpoint. The endpoint of the titration is best described as the point at which additional EDTA produces no further red to blue change. The EDTA volume used to produce the blue endpoint is used in the calculation in 8.7. Record as  $V_3$  in milliliters.

If endpoint is unclear or unobtainable, other tests shall be performed. Results and methodology of these tests shall be recorded.

### 8.7 Calculation—Water-soluble Alkaline Earth Metals as Calcium in Hematite

Calculate the concentration (corrected) of soluble alkaline earth metals as calcium,  $C_{Ca,c}$ , in the hematite sample in milligrams per kilogram, according to Equation (14):

$$C_{Ca,c} = (40 \times V_3) - C_c \quad (14)$$

where

$V_3$  is the volume EDTA used, expressed in milliliters;

$C_c$  is the EDTA calibration correction determined as per 5.2.10.4, expressed in milligrams per kilogram.

Record the calculated corrected soluble alkaline earth metals value of the hematite sample.

### 8.8 Reagents and Apparatus—Measuring Hematite Residue Greater than 75 $\mu\text{m}$ and 45 $\mu\text{m}$

**8.8.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution, 10 %  $\pm$  0.5 % by mass.

**8.8.2 Oven**, regulated to 105 °C  $\pm$  3 °C (220 °F  $\pm$  5 °F).

**8.8.3 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

**8.8.4 Balance**, with an accuracy of  $\pm$  0.01 g.

**8.8.5 Mixer** having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**8.8.6 Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**8.8.7 Sieve**, 75  $\mu\text{m}$  (0.0029 in., U.S. No. 200), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**8.8.8 Sieve**, 45  $\mu\text{m}$  (0.0017 in., U.S. No. 325), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**8.8.9 Spray nozzle**, with 6.35 mm ( $1/4$  in.) inlet connection, rated for 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.<sup>2</sup>) with approximately 45° spray angle attached to a water line with a 90° elbow.

**8.8.10 Water-pressure regulator**, capable of regulation to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>).

**8.8.11 Evaporating dish**, or functional equivalent.

**8.8.12 Wash bottle**.

## 8.9 Procedure—Measuring Hematite Residue Greater than 75 $\mu\text{m}$ and 45 $\mu\text{m}$

To measure the residue of diameters greater than 75  $\mu\text{m}$  and 45  $\mu\text{m}$  in a sample of hematite, the following procedure shall be applied.

- Take approximately 120 g of dried hematite been oven dried for at least 2 h at a constant mass and cooled to room temperature in the desiccator.
- Weigh 50.00 g  $\pm 0.01$  g of dried hematite. Record the mass, in grams, as  $m$ . Add the weighed sample to 350 mL  $\pm 3$  mL of water containing 0.20 g  $\pm 0.05$  g of sodium hexametaphosphate dispersant in the container. Sodium hexametaphosphate dispersant may be added as 0.20 g  $\pm 0.05$  g of sodium hexametaphosphate powder or by adding 2.0 mL  $\pm 0.1$  mL of sodium hexametaphosphate 10 %  $\pm 0.5$  % by mass solution. Add weighed hematite while stirring on the mixer. Stir with the mixer for 5 min  $\pm 10$  s.
- Transfer the sample to the 75  $\mu\text{m}$  sieve. Use a wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 70 kPa  $\pm 6$  kPa (10 lbf/in.<sup>2</sup>  $\pm 1$  lbf/in.<sup>2</sup>) from a spray nozzle for 2 min  $\pm 15$  s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
- Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.
- Dry the residue in the oven to a constant mass. Record the residue mass on the 75  $\mu\text{m}$  sieve, in grams, as  $m_2$  and total drying time.
- Repeat Steps b) through e) using the 45  $\mu\text{m}$  sieve. Record the residue mass on the 45  $\mu\text{m}$  sieve, in grams, as  $m_3$  and total drying time.

## 8.10 Calculation—Hematite Residue Greater than 75 $\mu\text{m}$ and 45 $\mu\text{m}$

Calculate the mass fraction hematite residue of particles greater than 75  $\mu\text{m}$ ,  $w_1$ , expressed in percent, according to Equation (15) and the mass fraction residue of particles greater than 45  $\mu\text{m}$ ,  $w_5$ , expressed in percent, according to Equation (16):

$$w_1 = 100 \left( \frac{m_2}{m} \right) \quad (15)$$

where

$m$  is the sample mass, expressed in grams;

$m_2$  is the residue mass on 75  $\mu\text{m}$  sieve, expressed in grams.

Record the calculated value.

$$w_5 = 100 \left( \frac{m_3}{m} \right) \quad (16)$$

where

$m$  is the sample mass, expressed in grams;

$m_3$  is the residue mass on 45  $\mu\text{m}$  sieve, expressed in grams.

Record the calculated value.

## 8.11 Reagents and Apparatus—Measuring Hematite Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter by the Sedimentation Method

8.11.1 Chemicals shall meet ACS or international equivalent reagent grade.

8.11.2 **Dispersant solution**, comprised of 40.0 g  $\pm$  0.1 g of sodium hexametaphosphate (CAS No. 10124-56-8) and 3.6 g  $\pm$  0.1 g of anhydrous sodium carbonate (CAS No. 497-19-8) per 1000 mL of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9.0.

8.11.3 **Oven**, regulated to 105 °C  $\pm$  3 °C (220 °F  $\pm$  5 °F).

8.11.4 **Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

8.11.5 **Balance**, with an accuracy of  $\pm$  0.01 g.

8.11.6 **Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

8.11.7 **Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

8.11.8 **Sedimentation cylinder**, glass, approximately 457 mm (18.0 in.) high and 63 mm (2.5 in.) in diameter, marked for a volume of 1000 mL (in accordance with ASTM D422-63).

8.11.9 **Rubber stopper**, size No. 13.

8.11.10 **Water bath** or **constant-temperature room**, capable of maintaining a constant temperature of 24 °C  $\pm$  5 °C (75 °F  $\pm$  10 °F).

8.11.11 **Thermometer**, including the range 16 °C to 32 °C (60 °F to 90 °F) readable to 0.5 °C (1 °F).

8.11.12 **Hydrometer**, ASTM 151H, graduated to read the relative density (specific gravity) of the suspension, range 0.995 to 1.038, graduation as 0.001 relative density, temperature 20 °C/20 °C (68 °F/68 °F).

8.11.13 **Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

8.11.14 **Spatula**.

## 8.12 Procedure—Measuring Hematite Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter by the Sedimentation Method

To measure particles less than 6  $\mu\text{m}$  in equivalent spherical diameter by the sedimentation method in a hematite sample, the following procedure in accordance with ASTM D422-63 shall be applied.

- Weigh 80.00 g  $\pm$  0.01 g of dry hematite [see 8.3 a) or 8.9 a)] and place in the container. Record the mass, in grams, as  $m$ .
- Add 125 mL  $\pm$  2 mL (127 g  $\pm$  2 g) of dispersant solution (8.11.2). Dilute to approximately 400 mL with deionized water. Rinse all adhering particles from the spatula into the suspension.
- Stir for 5 min  $\pm$  6 s on the mixer.
- Transfer the suspension to the sedimentation cylinder. Rinse mixing container with deionized water to assure that all sample particles are transferred to the sedimentation cylinder.

- e) Add deionized water to the 1000 mL mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s while holding a No. 13 rubber stopper in the top of the cylinder.

This is a critical step. Suspensions shall be homogeneous at the start of sedimentation. This is difficult to obtain because of hematite's high density.

- f) Set the cylinder into the water bath (or counter-top of the constant-temperature room) and simultaneously start the timer. Hang the thermometer in the water bath.
- g) Take hydrometer readings at intervals of 10 min  $\pm 6$  s, 20 min  $\pm 6$  s, 30 min  $\pm 6$  s, and 40 min  $\pm 6$  s (or until the first point below the 6  $\mu\text{m}$  value is reached). To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1.020 relative density reading before releasing. After the hydrometer stabilizes, read the top of the meniscus at the prescribed time. Carefully and slowly remove the hydrometer, rinse with deionized water, and dry after each reading. Remove the hydrometer immediately after each reading to eliminate particle buildup on the shoulders, which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve the suspension-settling equilibrium.
- h) Record time,  $t$ , in minutes, the temperature,  $\theta$ , in degrees Celsius (degrees Fahrenheit), and the hydrometer relative density reading,  $R$ , on the datasheet.

NOTE The temperature may be measured in either degrees Celsius or degrees Fahrenheit. However, all measurements and calculations must be made using consistent units including hydrometer calibration (or subsequent use of the hydrometer in routine test situations).

- i) For each time interval, determine water viscosity,  $\eta$ , and effective hydrometer depth,  $L$ , from Table 3 and Table 4. Record on the datasheet.

### 8.13 Calculation—Hematite Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter by the Sedimentation Method

**8.13.1** From hydrometer calibration (5.2.5), enter the hydrometer correction slope,  $M_C$ , and the hydrometer correction line initial value,  $B_C$ , onto the datasheet.

NOTE Annex C contains example calculations for hydrometer correction and particles less than 6  $\mu\text{m}$  in equivalent spherical diameter by sedimentation method.

**8.13.2** Calculate the sample constant,  $K_s$ , as given in Equation (17) (or determine from Table 7) and enter onto the datasheet:

$$K_s = 100 \frac{\rho}{m(\rho - 1)} \quad (17)$$

where

$\rho$  is the density of sample, expressed in grams per milliliter;

$m$  is the mass of sample, in grams.

**8.13.3** Calculate and enter onto the datasheet the equivalent spherical diameter,  $D_e$ , expressed in micrometers, for each time interval as given in Equation (18):

$$D_e = 17.5 \sqrt{\frac{\eta L}{(\rho - 1) t}} \quad (18)$$

where

$\eta$  is the viscosity of water, expressed in millipascal•seconds (centipoises), from Table 3 or Equation (12);

$\rho$  is the sample density, expressed in grams per milliliter;

$t$  is the time, expressed in minutes;

$L$  is the effective depth from Table 4, expressed in centimeters.

**8.13.4** For each time interval, calculate and enter onto the datasheet the cumulative percent finer,  $w_a$ , for the equivalent particle diameter,  $D_e$ , expressed in micrometers, as given in Equation (19):

$$w_a = K_s \left[ (M_c \times \theta) - B_c + (R - 1) \times 1000 \right] \quad (19)$$

where

$K_s$  is the sample constant, from Table 7 or Equation (17), expressed in percent per gram;

$M_c$  is the hydrometer correction line slope as per Equation (1) expressed in reciprocal degrees Celsius ( $^{\circ}\text{C}^{-1}$ ) [degrees Fahrenheit ( $^{\circ}\text{F}^{-1}$ )];

$\theta$  is the suspension temperature, expressed in degrees Celsius (degrees Fahrenheit);

$B_c$  is the hydrometer correction line initial value at  $\theta_1$ , as per Equation (2), dimensionless;

$R$  is the hydrometer relative density (specific gravity) reading at the top of the meniscus (not corrected).

Using this procedure and Equation (19), the cumulative percent finer,  $w_2$ , for particle diameter,  $D_1$ , immediately greater than 6  $\mu\text{m}$ , and the cumulative percent finer,  $w_3$ , for particle diameter,  $D_2$ , immediately less than 6  $\mu\text{m}$  data are determined

**8.13.5** Calculate and enter onto the datasheet the cumulative percent for hematite particles less than 6  $\mu\text{m}$  in equivalent spherical diameter expressed in micrometers,  $w_4$ , as given in Equation (20):

$$w_4 = \left[ \left( \frac{w_2 - w_3}{D_1 - D_2} \right) \times (6 - D_2) \right] + w_3 \quad (20)$$

where

$w_2$  is the cumulative percent for point immediately greater than 6  $\mu\text{m}$ ;

$w_3$  is the cumulative percent for point immediately less than 6  $\mu\text{m}$ ;

$D_1$  is the equivalent particle diameter immediately greater than 6  $\mu\text{m}$ , determined in Equation (18);

$D_2$  is the equivalent particle diameter immediately less than 6  $\mu\text{m}$ , determined in Equation (18).

For an example of the calculation for hematite particles less than 6  $\mu\text{m}$  in equivalent spherical diameter, see 0.

**Table 7—Sample Constant,  $K_s$ , for Hematite (80.0 g sample)**

Sample Density $\rho$ g/mL	Sample Constant $K_s$ % / g
5.00	1.5625
5.01	1.5617
5.02	1.5609
5.03	1.5602
5.04	1.5594
5.05	1.5586
5.06	1.5579
5.07	1.5571
5.08	1.5564
5.09	1.5556
5.10	1.5549
5.11	1.5541
5.12	1.5534
5.13	1.5527
5.14	1.5519
5.15	1.5512
5.16	1.5505
5.17	1.5498
5.18	1.5490
5.19	1.5483
5.20	1.5476
5.21	1.5469
5.22	1.5462
5.23	1.5455
5.24	1.5448
5.25	1.5441
5.26	1.5434
5.27	1.5427
5.28	1.5421
5.29	1.5414
5.30	1.5407

## 9 Bentonite

### 9.1 Principle

**9.1.1** Drilling-grade bentonite is a naturally occurring clay containing the clay minerals of smectite. It can also contain quartz, mica, feldspar, and calcite and other trace minerals.

**9.1.2** Drilling-grade bentonite shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 8, represents the product produced, and is controlled by the manufacturer. This test procedure is to be calibrated using reference (calibration) bentonite at least once per 40 tests, as described in 5.3.11.

**Table 8—Bentonite Physical Specifications**

Requirement	Standard
Suspension properties	
Viscometer dial reading at 600 r/min	minimum 30
Yield point-plastic viscosity ratio	maximum 3 lbf/(100 ft <sup>2</sup> •cP)
Filtrate volume	maximum 15.0 mL
Residue of diameter greater than 75 µm	maximum mass fraction 4.0 %

### 9.2 Reagents and Apparatus—Measuring Properties of a Bentonite Suspension

**9.2.1 Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the range specified in this procedure.

**9.2.2 Balance**, with an accuracy of  $\pm 0.01$  g.

**9.2.3 Mixer**, having a spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**9.2.4 Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**9.2.5 Spatula**.

**9.2.6 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**9.2.7 Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**9.2.8 Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**9.2.9 Container**, glass or plastic, at least 50 mL, to capture 7.5 min filtrate.

**9.2.10 Graduated cylinders**, two, 500 mL  $\pm 5$  mL (TC) and 10 mL  $\pm 0.1$  mL (TC).

**9.2.11 Deionized or distilled water**.

**9.2.12 Storage container**, with lid, capacity about 500 mL.

**9.2.13 Timers**, two, digital or mechanical, see 5.2.9 and 5.3.9.

### 9.3 Procedure—Measuring Rheological Properties of a Bentonite Suspension

To measure rheological properties of a bentonite suspension, the following procedure shall be applied.

- Prepare a suspension of the bentonite in the mixing container. Add 22.50 g  $\pm$  0.01 g of bentonite/clay (as received) to 350 mL  $\pm$  3 mL of deionized water while stirring on the mixer.
- After stirring 5 min  $\pm$  6 s, remove the container from the mixer and scrape its sides with the spatula to dislodge any bentonite adhering to the container walls. Be sure that all bentonite clinging to the spatula is incorporated into the suspension.
- Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min  $\pm$  1 min.
- Age the bentonite suspension at least 16 h in a sealed or covered storage container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.
- After aging the bentonite suspension, shake well and then pour the suspension into the mixing container. Stir the suspension on the mixer for 5 min  $\pm$  6 s.
- Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial readings at 600 r/min,  $R_{600}$ , and 300 r/min,  $R_{300}$ , rotor speed settings of the viscometer shall be recorded when a constant value for each rotational velocity is reached. Readings shall be taken at a suspension test temperature of 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F).

### 9.4 Calculation—Measuring Rheological Properties of a Bentonite Suspension

Calculate the plastic viscosity,  $\eta_{PV}$ , expressed in centipoises, according to Equation (21), the yield point,  $Y_P$ , in lbf/100 ft<sup>2</sup>, according to Equation (22), and the yield point-plastic viscosity ratio,  $b$ , expressed in lbf/(100 ft<sup>2</sup>•cP) according to Equation (23):

$$\eta_{PV} = R_{600} - R_{300} \quad (21)$$

$$Y_P = R_{300} - \eta_{PV} \quad (22)$$

$$b = \frac{Y_P}{\eta_{PV}} \quad (23)$$

where

$R_{600}$  is the viscometer dial reading at 600 r/min;

$R_{300}$  is the viscometer dial reading at 300 r/min.

Record the calculated values for plastic viscosity, yield point, and yield point-plastic viscosity ratio.

NOTE 1 Plastic viscosity in SI units, millipascal•seconds are numerically equivalent to centipoises. Yield point values reported in lbf/100 ft<sup>2</sup> units are not exact. A direct conversion from lbf/100 ft<sup>2</sup> to pascals would be to multiply the value 0.479. However, each 1 degree of deflection is exactly equal to 0.511 Pa in SI units, so the more exact conversion is to multiply the result from Equation (22) by 0.511 to obtain pascals.

NOTE 2 Abbreviations for plastic viscosity and yield point in the industry, PV and YP, are commonly used. Then the ratio of yield point-plastic viscosity is given as YP/PV. When expressed in SI units, this ratio is numerically equivalent to reciprocal milliseconds or 1000 s<sup>-1</sup>.



## 9.5 Procedure—Measuring the Filtrate Volume of a Bentonite Suspension

To measure the filtrate volume of a bentonite suspension, the following procedure shall be applied.

- a) Recombine all of the suspension, as prepared and tested in 9.3, and stir in the mixing container for 1 min  $\pm$  6 s on the mixer. Adjust the suspension temperature to 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F).
- b) Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. Pour the bentonite suspension into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- c) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$  30 kPa (100 lbf/in.<sup>2</sup>  $\pm$  5 lbf/in.<sup>2</sup>). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- d) At 7.5 min  $\pm$  6 s on the first timer, remove the container and any liquid adhering to the drain tube, and discard. Place the dry 10 mL graduated cylinder under the drain tube and continue collecting filtrate until the end of the second timer set at 30 min. At 30 min  $\pm$  6 s, remove the graduated cylinder and record the volume of filtrate collected,  $V_c$ .

## 9.6 Calculation—Filtrate Volume of a Bentonite Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the bentonite suspension as given in Equation (24):

$$V = 2V_c \quad (24)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

## 9.7 Reagents and Apparatus—Measuring Bentonite Residue of Greater than 75 $\mu$ m

**9.7.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution, 10 %  $\pm$  0.5 % by mass.

**9.7.2 Oven**, regulated to 105 °C  $\pm$  3 °C (220 °F  $\pm$  5 °F).

**9.7.3 Balance**, with an accuracy of  $\pm$  0.01 g.

**9.7.4 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**9.7.5 Mixing container**, approximate dimensions: depth 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**9.7.6 Sieve**, 75  $\mu$ m (0.0029 in., U.S. No. 200), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**9.7.7 Spray nozzle**, with 6.35 mm ( $1/4$  in.) inlet connection, rated for 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.<sup>2</sup>) with approximately 45° spray angle attached to a water line with a 90° elbow.

**9.7.8 Water-pressure regulator**, capable of regulation to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>).

**9.7.9 Evaporating dish** or functional equivalent.

**9.7.10 Wash bottle.**

## **9.8 Procedure—Measuring Bentonite Residue Greater than 75 µm**

The following procedure shall be applied when measuring bentonite residue greater than 75 µm.

- a) Weigh 10.00 g  $\pm$ 0.01 g of bentonite. Record the mass in grams, as  $m$ .
- b) Add the weighed bentonite sample to approximately 350 mL  $\pm$ 3 mL of water containing 0.20 g  $\pm$ 0.05 g of sodium hexametaphosphate dispersant in the container. Sodium hexametaphosphate dispersant may be added as 0.20 g  $\pm$ 0.05 g of sodium hexametaphosphate powder or by adding 2.0 mL  $\pm$ 0.1 mL of sodium hexametaphosphate 10 %  $\pm$ 0.5 % by mass solution. Add weighed bentonite while stirring on the mixer.
- c) Stir suspension on the mixer for 30 min  $\pm$ 10 s.
- d) Transfer the sample to the sieve. Use the wash bottle to transfer all material from the mixing container to the sieve. Wash the material on the sieve with water controlled to 70 kPa  $\pm$ 6 kPa (10 lbf/in.<sup>2</sup>  $\pm$ 1 lbf/in.<sup>2</sup>) from the spray nozzle for 2 min  $\pm$ 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
- e) Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.
- f) Dry the residue in the oven to a constant mass. Record the residue mass,  $m_2$ , and total drying time.

## **9.9 Calculation—Bentonite Residue Greater than 75 µm**

Calculate the mass fraction residue of bentonite particles greater than 75 µm,  $w_1$ , in percent, as given in Equation (25):

$$w_1 = 100 \left( \frac{m_2}{m} \right) \quad (25)$$

where

$m$  is the sample mass, in grams;

$m_2$  is the residue mass retained by 75 µm sieve, in grams.

Record the calculated value.

## **10 Non-treated Bentonite**

### **10.1 Principle**

**10.1.1** Drilling-grade non-treated bentonite clay, composed principally of the mineral smectite is dried and ground but not chemically treated. It may also contain quartz, mica, feldspar, calcite, and other natural contaminants.

**10.1.2** Drilling-grade non-treated bentonite shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 9, represents the product produced, and is controlled by the manufacturer. This test procedure is to be calibrated using reference (calibration) bentonite at least once per 40 tests, as described in 5.3.11.

**Table 9—Non-treated Bentonite Physical Specifications**

Requirement	Standard
Suspension properties	
Yield point-plastic viscosity ratio	maximum 1.5 lbf/(100 ft <sup>2</sup> •cP)
Dispersed plastic viscosity	minimum 10 mPa•s (cP)
Dispersed filtrate volume	maximum 12.5 mL

## 10.2 Reagents and Apparatus—Measuring Properties of a Non-treated Bentonite Solution

**10.2.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution, 10 %  $\pm$ 0.5 % by mass.

**10.2.2 Deionized or distilled water.**

**10.2.3 Balance**, with an accuracy of  $\pm$ 0.01 g.

**10.2.4 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**10.2.5 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**10.2.6 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**10.2.7 Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**10.2.8 Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**10.2.9 Spatula.**

**10.2.10 Thermometer**, accurate to  $\pm$ 0.5 °C ( $\pm$ 1.0 °F) over the range specified in this procedure.

**10.2.11 Graduated cylinders**, two, 500 mL  $\pm$ 5 mL (TC) and 10 mL  $\pm$ 0.1 mL (TC).

**10.2.12 Storage container**, with lid, capacity about 500 mL.

**10.2.13 Syringe or pipette**, 5 mL  $\pm$ 0.1 mL.

**10.2.14 Timers**, two, digital or mechanical, see 5.2.9 and 5.3.9.

## 10.3 Procedure—Measuring Yield Point-Plastic Viscosity Ratio of a Non-treated Bentonite Suspension

To measure the yield point-plastic viscosity ratio of a non-treated bentonite suspension, the following procedure shall be applied.

- Prepare a suspension of non-treated bentonite. Add 25.00 g  $\pm$ 0.01 g of clay (as received) to 350 mL  $\pm$ 3 mL of deionized water while stirring on the mixer.
- After stirring 5 min  $\pm$ 6 s, remove the mixing container from the mixer and scrape its sides with the spatula to dislodge any bentonite adhering to the container walls. Be sure that all bentonite clinging to the spatula is incorporated into the suspension.

- c) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min  $\pm$  1 min.
- d) Age the bentonite suspension at least 16 h in a sealed storage container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.
- e) After aging the bentonite suspension, shake well and then pour the suspension into the mixing container. Stir the suspension on the mixer for 5 min  $\pm$  6 s.
- f) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial readings at 600 r/min,  $R_{600}$ , and at 300 r/min,  $R_{300}$ , rotor speed settings of the viscometer shall be recorded when a constant value for each rotational velocity is reached. Readings shall be taken at a suspension test temperature of 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F).

#### 10.4 Calculation—Yield Point-Plastic Viscosity Ratio of a Non-treated Bentonite Suspension

Calculate the plastic viscosity,  $\eta_{PV}$ , in centipoises, according to Equation (26), the yield point,  $Y_P$ , in lbf/100 ft<sup>2</sup>, according to Equation (27), and the yield point-plastic viscosity ratio,  $b$ , expressed in lbf/(100 ft<sup>2</sup>•cP) according to Equation (28):

$$\eta_{PV} = R_{600} - R_{300} \quad (26)$$

$$Y_P = R_{300} - \eta_{PV} \quad (27)$$

$$b = \frac{Y_P}{\eta_{PV}} \quad (28)$$

where

$R_{600}$  is the viscometer dial reading at 600 r/min;

$R_{300}$  is the viscometer dial reading at 300 r/min.

Record the calculated values for plastic viscosity, yield point, and yield point-plastic viscosity ratio.

NOTE 1 Plastic viscosity in SI units, millipascal•seconds are numerically equivalent to centipoises. Yield point values reported in lbf/100 ft<sup>2</sup> units are not exact. A direct conversion from lbf/100 ft<sup>2</sup> to pascals would be to multiply the value 0.479. However, each 1 degree of deflection is exactly equal to 0.511 Pa in SI units, so the more exact conversion is to multiply the result from Equation (27) by 0.511 to obtain pascals.

NOTE 2 Abbreviations for plastic viscosity and yield point in the industry, PV and YP, are commonly used. Then the ratio of yield point-plastic viscosity is given as YP/PV. When expressed in SI units this ratio is numerically equivalent to reciprocal milliseconds or 1000 s<sup>-1</sup>.

#### 10.5 Procedure—Measuring the Plastic Viscosity of a Dispersed Non-treated Bentonite Suspension

To measure the plastic viscosity of a dispersed non-treated bentonite suspension, the following procedure shall be applied.

- a) Recombine all of the bentonite suspension, as prepared and tested in 10.3, and stir in the mixing container for 1 min  $\pm$  6 s on the mixer.

- b) Add 0.50 g  $\pm$ 0.05 g of sodium hexametaphosphate dispersant to the suspension and stir for 3 min  $\pm$ 30 s on the mixer. Sodium hexametaphosphate dispersant may be added as 0.50 g  $\pm$ 0.05 g of sodium hexametaphosphate powder or by adding 5.0 mL  $\pm$ 0.1 mL of 10 %  $\pm$ 0.5 % by mass solution.
- c) Add 5 mL  $\pm$ 0.1 mL of a 10 % solution of sodium hexametaphosphate to the suspension and stir for 3 min  $\pm$ 6 s on the mixer.
- d) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial readings at 600 r/min and 300 r/min rotor speed settings of the viscometer shall be recorded when a constant value for each rotational velocity is reached. Readings shall be taken at a suspension test temperature of 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F).
- e) Calculate and record the dispersed plastic viscosity as per Equation (26).

### 10.6 Procedure—Measuring the Filtrate Volume of a Dispersed Non-treated Bentonite Suspension

To measure the filtrate volume of a dispersed non-treated bentonite suspension, the following procedure shall be applied.

- a) Recombine all of the suspension as prepared and tested in 10.5 and stir in the container for 1 min  $\pm$  6 s on the mixer. Adjust the suspension temperature to 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F).
- b) Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. Pour the non-treated bentonite suspension into the filter press to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- c) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$ 30 kPa (100 lbf/in.<sup>2</sup>  $\pm$ 5 lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- d) At 7.5 min  $\pm$ 6 s on the first timer, remove the container and any liquid adhering on the drain tube and discard. Place the dry 10 mL graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 min. At 30 min  $\pm$ 6 s remove the graduated cylinder and record the volume of filtrate collected,  $V_c$ .

### 10.7 Calculation—Filtrate Volume of a Dispersed Non-treated Bentonite Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the clay suspension as given in Equation (29):

$$V = 2V_c \quad (29)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

## 11 Attapulgite

### 11.1 Principle

**11.1.1** Drilling-grade attapulgite clay is a naturally occurring clay mineral. It may also contain quartz, mica, feldspar, calcite, and other natural contaminants.

**11.1.2** Drilling-grade attapulgite shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 10, represents the product produced, and is controlled by the manufacturer.

**Table 10—Attapulgite Physical Specifications**

Requirement	Standard
Suspension properties	
Viscometer dial reading at 600 r/min	minimum 30
Residue of diameter greater than 75 $\mu\text{m}$	maximum mass fraction 8.0 %
Moisture mass fraction	maximum mass fraction 16.0 %

### 11.2 Reagents and Apparatus—Measuring Properties of an Attapulgite Suspension

**11.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**11.2.2 Sodium chloride** (CAS No. 7647-14-5).

**11.2.3 Deionized or distilled water.**

**11.2.4 Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the range specified in this procedure.

**11.2.5 Balance**, with an accuracy of  $\pm 0.01$  g.

**11.2.6 Storage container**, with lid, capacity about 500 mL.

**11.2.7 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**11.2.8 Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**11.2.9 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**11.2.10 Spatula.**

**11.2.11 Graduated cylinders**, two, 500 mL  $\pm 5$  mL (TC) and 100 mL  $\pm 1$  mL (TC).

**11.2.12 Defoamer.**

**11.2.13 Storage container**, glass or plastic, with stopper or lid, for salt solution.

**11.2.14 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

### 11.3 Procedure—Measuring 600 r/min Dial Reading of an Attapulgite Suspension

To measure the 600 r/min dial reading of an attapulgite suspension, the following procedure shall be applied.

- a) Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 45.0 g  $\pm$  0.1 g of sodium chloride per 100 mL  $\pm$  1 mL of deionized water. Allow the solution to stand for approximately 1 h to ensure saturation. Carefully decant the solution or filter into a storage container.
- b) Prepare a suspension of attapulgite in the mixing container. Add 20.00 g  $\pm$  0.01 g of attapulgite (as received) to 350 mL  $\pm$  3 mL of saturated salt solution while stirring on the mixer.
- c) After stirring 5 min  $\pm$  6 s, remove the mixing container from the mixer and scrape its sides with the spatula to dislodge any attapulgite adhering to the container walls. Be sure that all attapulgite clinging to the spatula is incorporated into the suspension.
- d) Replace the mixing container on the mixer and continue to stir. If necessary, the mixing container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min  $\pm$  1 min.
- e) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. Add 2 to 3 drops of defoamer and stir in with the spatula to break the surface froth. Place the viscometer cup on the direct-indicating viscometer. The dial reading,  $R_{600}$ , at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value for 600 r/min is reached. Readings shall be taken at a suspension test temperature of 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F).

#### 11.4 Reagent and Apparatus—Measuring Attapulgite Residue Greater than 75 $\mu$ m

**11.4.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution, 10 %  $\pm$  0.5 % by mass.

**11.4.2 Oven**, regulated to 105 °C  $\pm$  3 °C (220 °F  $\pm$  5 °F).

**11.4.3 Balance**, with an accuracy of  $\pm$  0.01 g.

**11.4.4 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**11.4.5 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**11.4.6 Sieve**, 75  $\mu$ m (0.0029 in., U.S. No. 200), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**11.4.7 Spray nozzle**, with 6.35 mm ( $1/4$  in.) inlet connection, rated for 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.<sup>2</sup>) with approximately 45° spray angle attached to a water line with a 90° elbow.

**11.4.8 Water-pressure regulator**, capable of regulation to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>).

**11.4.9 Evaporating dish** or functional equivalent.

**11.4.10 Wash bottle**.

#### 11.5 Procedure—Measuring Attapulgite Residue Greater than 75 $\mu$ m

The following procedure shall be applied when measuring attapulgite residue greater than 75  $\mu$ m.

- a) Weigh 10.00 g  $\pm$  0.01 g of attapulgite. Record the mass in grams, as  $m$ .

- b) Add the weighed attapulgite sample to 350 mL  $\pm$ 3 mL of water containing 0.20 g  $\pm$ 0.05 g of sodium hexametaphosphate dispersant in the container. Sodium hexametaphosphate dispersant may be added as 0.20 g  $\pm$ 0.05 g of sodium hexametaphosphate powder or by adding 2.0 mL  $\pm$ 0.1 mL of sodium hexametaphosphate 10 %  $\pm$ 0.5 % by mass solution. Add weighed attapulgite to the mixing container while stirring on the mixer.
- c) Stir the suspension on the mixer for 30 min  $\pm$ 10 s.
- d) Transfer the sample to the sieve. Use the wash bottle to transfer all material from the mixing container to the sieve. Wash the material on the sieve with water controlled to 70 kPa  $\pm$ 6 kPa (10 lbf/in.<sup>2</sup>  $\pm$ 1 lbf/in.<sup>2</sup>) from the spray nozzle for 2 min  $\pm$ 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
- e) Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.
- f) Dry the residue in the oven to a constant mass. Record the residue mass as  $m_2$  and total drying time.

### 11.6 Calculation—Residue of Attapulgite Greater than 75 $\mu$ m

Calculate the mass fraction residue of particles greater than 75  $\mu$ m,  $w_1$ , in percent, as given in Equation (30):

$$w_1 = 100 \left( \frac{m_2}{m} \right) \quad (30)$$

where

$m$  is the sample mass, in grams;

$m_2$  is the residue mass retained by 75  $\mu$ m sieve, in grams.

Record the calculated value.

### 11.7 Reagent and Apparatus—Measuring Moisture Mass Fraction of Attapulgite

**11.7.1 Oven**, regulated to 105 °C  $\pm$ 3 °C (220 °F  $\pm$ 5 °F).

**11.7.2 Balance**, with an accuracy of  $\pm$ 0.01 g.

**11.7.3 Evaporating dish** or functional equivalent.

**11.7.4 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

### 11.8 Procedure—Measuring Moisture Mass Fraction of Attapulgite

To measure the moisture fraction in a sample of attapulgite, the following procedure shall be applied.

- a) Weigh 10.0 g  $\pm$ 0.01 g of attapulgite sample into a tared evaporating dish. Record the mass as  $m$ .
- b) Dry the sample in the oven for at least 2 h to a constant mass.
- c) Cool to room temperature in desiccator.
- d) Reweigh the evaporating dish containing the dried attapulgite. Record the residue mass as  $m_1$ .



## 11.9 Calculation—Moisture Mass Fraction of Attapulgite

Calculate, the attapulgite sample moisture mass fraction,  $w_6$ , in percent, as given in Equation (31):

$$w_6 = 100 \left( \frac{m - m_1}{m} \right) \quad (31)$$

where

$m$  is the sample mass, expressed in grams;

$m_1$  is the residue mass, expressed in grams.

Record the calculated value.

## 12 Sepiolite

### 12.1 Principle

**12.1.1** Drilling-grade sepiolite is a naturally occurring clay mineral. It may also contain quartz, mica, feldspar, calcite, and other natural contaminants.

**12.1.2** Drilling-grade sepiolite shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 11, represents the product produced, and is controlled by the manufacturer.

**Table 11—Sepiolite Physical Specifications**

Requirement	Standard
Suspension properties	
Viscometer dial reading at 600 r/min	minimum 30
Residue of diameter greater than 75 $\mu\text{m}$	maximum mass fraction 8.0 %
Moisture mass fraction	maximum mass fraction 16.0 %

### 12.2 Reagents and Apparatus—Measuring Properties of a Sepiolite Suspension

**12.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**12.2.2 Sodium chloride** (CAS No. 7647-14-5).

**12.2.3 Deionized or distilled water.**

**12.2.4 Defoamer.**

**12.2.5 Thermometer**, accurate to  $\pm 0.5^\circ\text{C}$  ( $\pm 1.0^\circ\text{F}$ ) over the range specified in this procedure.

**12.2.6 Balance**, with an accuracy of  $\pm 0.01$  g.

**12.2.7 Storage container**, with lid, capacity about 500 mL.

**12.2.8 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**12.2.9 Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**12.2.10 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**12.2.11 Spatula**.

**12.2.12 Graduated cylinders**, two, 500 mL  $\pm 5$  mL (TC) and 100 mL  $\pm 1$  mL (TC).

**12.2.13 Container**, with glass or plastic stopper or lid, for salt solution.

**12.2.14 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

### 12.3 Procedure—Measuring 600 r/min Dial Reading of a Sepiolite Suspension

To measure 600 r/min dial reading of a sepiolite solution, the following procedure shall be applied.

- Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 45.0 g  $\pm 0.1$  g of sodium chloride per 100 mL  $\pm 1$  mL of deionized water. Allow solution to stand for approximately 1 h to ensure saturation. Carefully decant solution or filter it into a storage container.
- Prepare a suspension of sepiolite. Add 20.00 g  $\pm 0.01$  g (as received) to 350 mL  $\pm 3$  mL of saturated salt solution to the mixing container while stirring on the mixer.
- After stirring for 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape its sides with the spatula to remove or dislodge any sepiolite adhering to the container walls. Be sure that all sepiolite clinging to the spatula is incorporated into the suspension.
- Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min  $\pm 1$  min.
- Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. Add 2 to 3 drops of defoamer and stir in with the spatula to break the surface froth. Place the viscometer cup on direct-indicating viscometer. The dial reading,  $R_{600}$ , at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value for 600 r/min is reached. Readings shall be taken at a suspension test temperature of 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F).

### 12.4 Reagents and Apparatus—Measuring Sepiolite Residue Greater than 75 $\mu$ m

**12.4.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution, 10 %  $\pm 0.5$  % by mass.

**12.4.2 Oven**, regulated to 105 °C  $\pm 3$  °C (220 °F  $\pm 5$  °F).

**12.4.3 Balance**, with an accuracy of  $\pm 0.01$  g.

**12.4.4 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**12.4.5 Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**12.4.6 Sieve**, 75  $\mu$ m (0.0029 in., U.S. No. 200), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**12.4.7 Spray nozzle**, with 6.35 mm (1/4 in.) inlet connection, rated to 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.<sup>2</sup>) with approximatively 45° spray angle, attached to a water line with a 90° elbow.

**12.4.8 Water-pressure regulator**, capable of regulation to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>).

**12.4.9 Evaporating dish** or functional equivalent.

**12.4.10 Wash bottle**.

## 12.5 Procedure—Measuring Sepiolite Residue Greater than 75 $\mu$ m

The following procedure shall be applied when measuring sepiolite residue greater than 75  $\mu$ m.

- a) Weigh 10.0 g  $\pm$  0.01 g of sepiolite. Record the mass in grams, as  $m$ .
- b) Add the weighed sepiolite sample to 350 mL  $\pm$  3 mL of water containing 0.20 g  $\pm$  0.05 g of sodium hexametaphosphate dispersant in the container. Sodium hexametaphosphate dispersant may be added as 0.20 g  $\pm$  0.05 g of sodium hexametaphosphate powder or by adding 2.0 mL  $\pm$  0.1 mL of sodium hexametaphosphate 10 %  $\pm$  0.5 % by mass solution. Add weighed sepiolite sample while stirring on the mixer.
- c) Stir the suspension on the mixer for 30 min  $\pm$  10 s.
- d) Transfer the sample to the sieve. Use the wash bottle to transfer all material from the container to the sieve. Wash the material on the sieve with water controlled to 70 kPa  $\pm$  6 kPa (10 lbf/in.<sup>2</sup>  $\pm$  1 lbf/in.<sup>2</sup>) from the spray nozzle for 2 min  $\pm$  15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
- e) Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.
- f) Dry the residue in the oven to a constant mass. Record the residue mass as  $m_2$ .

## 12.6 Calculation—Sepiolite Residue Greater than 75 $\mu$ m

Calculate the mass fraction sepiolite residue of particles greater than 75  $\mu$ m,  $w_1$ , in percent, as given in Equation (32):

$$w_1 = 100 \left( \frac{m_2}{m} \right) \quad (32)$$

where

$m$  is the sample mass, expressed in grams;

$m_2$  is the residue mass retained by 75  $\mu$ m sieve, expressed in grams.

Record the calculated value.

## 12.7 Reagents and Apparatus—Measuring Moisture Mass Fraction of Sepiolite

**12.7.1 Oven**, regulated to 105 °C  $\pm$  3 °C (220 °F  $\pm$  5 °F).

**12.7.2 Balance**, with an accuracy of  $\pm$  0.01 g.

**12.7.3 Evaporating dish** or functional equivalent.

**12.7.4 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

## 12.8 Procedure—Measuring Moisture Mass Fraction of Sepiolite

To measure the moisture fraction in a sample of sepiolite, the following procedure shall be applied.

- Weigh 10.0 g  $\pm$ 0.01 g of sepiolite sample into a tared evaporating dish. Record as  $m$ .
- Dry the sample in the oven for at least 2 h to a constant mass.
- Cool to room temperature in a desiccator.
- Reweigh the evaporating dish containing the dry sepiolite. Record the residue mass as  $m_1$ .

## 12.9 Calculation—Moisture Mass Fraction of Sepiolite

Calculate the sepiolite sample moisture mass fraction,  $w_6$ , in percent, as given in Equation (33):

$$w_6 = 100 \left( \frac{m - m_1}{m} \right) \quad (33)$$

where

$m$  is the sample mass, expressed in grams;

$m_1$  is the dried sample mass, expressed in grams.

Record the calculated value.

## 13 Technical-grade, Low-viscosity Carboxymethyl Cellulose (CMC-LVT)

### 13.1 Principle

**13.1.1** The CMC-LVT is an alkali-metal salt of carboxymethyl cellulose. The manufacturer should maintain documentation of the analysis of the cellulosic raw material.

**13.1.2** The product is a free-flowing or granulated powder usually containing reaction byproducts.

**13.1.3** The CMC-LVT shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 12, represents the product produced, and is controlled by the manufacturer.

**13.1.4** The CMC-LVT shall be free of any starch or starch derivatives. A qualitative starch determination shall be performed before proceeding with the CMC-LVT performance testing. If starch is found, no further testing should be performed, and the sample shall be rejected.

**Table 12—CMC-LVT Physical Specifications**

Requirement	Standard
Starch or starch derivatives presence	no
Solution properties	
Viscometer dial reading at 600 r/min	maximum 90
Suspension properties	
Filtrate volume	maximum 10 mL

## 13.2 Qualitative Determination of Starch or Starch Derivatives in a CMC-LVT Sample

### 13.2.1 Description

**13.2.1.1** This test identifies the presence of starch or starch derivatives in water-soluble polymers, like CMC-LVT, supplied in powder or granular form.

**13.2.1.2** An iodine/iodide solution is mixed with a solution of the polymer being analyzed. In the presence of amylose (linear fraction of starch), a colored complex is formed.

### 13.2.2 Reagents and Apparatus—Qualitative Determination of Starch or Starch Derivatives in a CMC-LVT Sample

**13.2.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**13.2.2.2** **Deionized or distilled water.**

**13.2.2.3** **Iodine solution** (CAS No. 7553-56-2), 0.05 mol/L (0.1 N).

**13.2.2.4** **Potassium iodide** (CAS No. 7681-11-0).

**13.2.2.5** **Sodium hydroxide** (CAS No. 1310-73-2), dilute solution, 0.1 % to 0.5 %.

**13.2.2.6** **Mixer**, having each spindle fitted with a single-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**13.2.2.7** **Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**13.2.2.8** **Storage container**, with lid, capacity about 500 mL.

**13.2.2.9** **Spatula.**

**13.2.2.10** **Balance**, with accuracy of  $\pm 0.01$  g.

**13.2.2.11** **Volumetric flask**, 100 mL  $\pm 0.1$  mL.

**13.2.2.12** **Pasteur pipette or drop bottle**, plastic.

**13.2.2.13** **Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

**13.2.2.14** **pH meter with pH electrode**, accuracy of 0.01 pH.

**13.2.2.15** **Test tubes**, four.

### 13.2.3 Procedure—Preparation of the Iodine/Iodide Solution

The iodine/iodide solution shall be prepared as per the following procedure.

- a) Prepare the iodine/iodide solution using a 100 mL  $\pm 0.1$  mL volumetric flask. Add 10 mL  $\pm 0.1$  mL of the 0.05 mol/L iodine solution.
- b) Add 0.60 g  $\pm 0.01$  g of the potassium iodide and dissolve by gently swirling the volumetric flask.
- c) Bring to the 100 mL mark with deionized water and mix thoroughly. Record the day of preparation.
- d) The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place. It may be used for up to 3 months. After the date of expiry, discard the solution and prepare again.

### 13.2.4 Procedure—Preparation of the CMC-LVT Solution and Qualitative Determination of Starch or Starch Derivatives

To conduct a test for the presence of starch and starch derivatives in water-soluble polymers, like CMC-LVT, supplied in powder or granular form in a polymer solution, the following procedure shall be applied.

- a) Prepare a 5 % solution of the water-soluble polymer under examination (CMC-LVT) by adding 380 g  $\pm 0.1$  g of deionized water to the mixing container. Add 20.00 g  $\pm 0.01$  g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.
- b) After stirring for 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container walls. Be sure that all of the polymer clinging to the spatula is incorporated into the solution.
- c) Measure the pH. If the pH value is less than 10.01, raise the pH to  $>10.01$  by adding, dropwise, the dilute sodium hydroxide solution.
- d) Replace the mixing container on the mixer and continue to stir. Total mixing time shall equal 20 min  $\pm 1$  min.
- e) Put 2 mL  $\pm 0.1$  mL of the polymer solution in a test tube and add, dropwise, in portions of 3 drops at a time, up to 30 drops of the iodine/iodide solution.
- f) Prepare three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution for comparison.
- g) After every 3-drop addition, swirl the test tube gently and compare the color of the solution under examination with the blank tests. A color comparison made against a white background allows for ease of comparison.

### 13.2.5 Interpretation—Determination of Starch or Starch Derivatives in a CMC-LVT Sample

**13.2.5.1** The sample under examination gives a yellow color comparable to one of the blank tests [see 13.2.4 f)] if, and only if, the sample does not contain any starch or starch derivatives.

**13.2.5.2** The development of a light green to dark blue color, either in solution or as a precipitate, is an indication of the presence of starch (amylose fraction).

**13.2.5.3** The development of a light pink to reddish-brown color is an indication of the presence of a highly substituted starch, dextrine, or starches with a high amylopectin content.

**13.2.5.4** The development of any other color is a strong indication of the presence of starch or starch derivatives.

**13.2.5.5** Instant de-colorizing indicates the presence of a reducing agent. In this case, continue the dropwise addition of the iodine/iodide solution and compare the color obtained with cases 13.2.5.1 through 13.2.5.4.

**13.2.5.6** The presence of starch or starch derivatives is contrary to the product definition in Table 12; therefore, further testing shall be abandoned.

### **13.3 Reagents and Apparatus—Measuring Properties of CMC-LVT Water-soluble Polymers**

**13.3.1** Chemicals shall meet ACS or international equivalent reagent grade.

**13.3.2** **Standard evaluation base clay (SEBC)**, see 4.2 and Annex D.

**13.3.3** **Sodium chloride** (CAS No. 7647-14-5).

**13.3.4** **Sodium bicarbonate** (CAS No. 144-55-8).

**13.3.5** **Deionized or distilled water**.

**13.3.6** **Thermometer**, accurate to  $\pm 0.5\text{ }^{\circ}\text{C}$  ( $\pm 1.0\text{ }^{\circ}\text{F}$ ) over the temperature range specified in the procedure.

**13.3.7** **Balance**, with an accuracy of  $\pm 0.01\text{ g}$ .

**13.3.8** **Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**13.3.9** **Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**13.3.10** **Storage container**, with lid, capacity about 500 mL.

**13.3.11** **Container**, glass or plastic, at least 50 mL, to capture 7.5 min filtrate.

**13.3.12** **Spatula**.

**13.3.13** **Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**13.3.14** **Timers**, two, digital or mechanical, see 5.2.9 or 5.3.9.

**13.3.15** **Graduated cylinders**, three TC, 10 mL  $\pm 0.1\text{ mL}$ , 100 mL  $\pm 1\text{ mL}$ , and 500 mL  $\pm 5\text{ mL}$ .

**13.3.16** **Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**13.3.17** **Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**13.3.18** **Constant-temperature device** (e.g. water bath) set at  $20\text{ }^{\circ}\text{C}$  to  $25\text{ }^{\circ}\text{C}$  ( $68\text{ }^{\circ}\text{F}$  to  $77\text{ }^{\circ}\text{F}$ ), necessary if room temperature is not in the range of  $20\text{ }^{\circ}\text{C}$  to  $25\text{ }^{\circ}\text{C}$  ( $68\text{ }^{\circ}\text{F}$  to  $77\text{ }^{\circ}\text{F}$ ).

### **13.4 Procedure—Measuring 600 r/min Dial Reading of CMC-LVT Deionized Water Solution**

To measure 600 r/min dial reading of a CMC-LVT deionized water solution, the following procedure shall be applied.

- a) Prepare a solution of CMC-LVT in the mixing container. Add  $10.50\text{ g} \pm 0.01\text{ g}$  of CMC-LVT to  $350\text{ mL} \pm 3\text{ mL}$  of deionized water at a uniform rate over a time interval of  $60\text{ s} \pm 6\text{ s}$  while stirring on the mixer. The CMC-LVT shall be added away from impeller shaft to minimize dusting.

NOTE This is equivalent to 30.00 g/L  $\pm$ 0.03 g/L.

- b) After stirring 5 min  $\pm$ 6 s, remove the mixing container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-LVT adhering to the container walls. Be sure that all CMC-LVT clinging to the spatula is incorporated into the solution.
- c) Replace the mixing container on the mixer and continue to stir. If necessary, the mixing container may be removed from the mixer and the sides scraped to dislodge any CMC-LVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min  $\pm$ 1 min.
- d) Age the solution for 2 h  $\pm$ 5 min in a sealed or covered container at room temperature or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F).
- e) After aging, stir the solution on the mixer for 5 min  $\pm$ 6 s.
- f) Pour the solution into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value at 600 r/min is reached. The reading shall be taken at a solution test temperature of 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F).

### 13.5 Procedure—Measuring Filtrate Volume of CMC-LVT Clay Suspension

To measure filtrate volume of a CMC-LVT clay suspension, the following procedure shall be applied.

- a) Prepare an ample volume of saturated salt solution by thoroughly mixing a suitable container 45.0 g  $\pm$ 0.1 g of sodium chloride per 100 mL  $\pm$ 1 mL of deionized water. Allow the solution to stand for approximately 1 h to ensure saturation. Carefully decant the solution or filter it into a storage container.
- b) Prepare a clay-based suspension by adding 350 mL  $\pm$ 3 mL of the saturated salt solution to the mixer container. Add 1.0 g  $\pm$ 0.1 g of sodium bicarbonate and stir on the mixer for 60 s  $\pm$ 6 s.
- c) Slowly add 35.0 g  $\pm$ 0.1 g of SEBC while stirring on the mixer.
- d) After stirring 5 min  $\pm$ 6 s, remove the mixing container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.
- e) Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min  $\pm$ 1 min.
- f) Add 3.15 g  $\pm$ 0.01 g of CMC-LVT to the suspension while stirring on the mixer, adding at a uniform rate over 60 s  $\pm$ 6 s. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.

NOTE This is equivalent to 9.01 g/L  $\pm$ 0.03 g/L.

- g) After stirring 5 min  $\pm$ 6 s, remove the mixing container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-LVT adhering to the container walls. Be sure that all material clinging to the spatula is incorporated into the suspension.
- h) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-LVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min  $\pm$ 1 min.
- i) Age the suspension for 2 h  $\pm$ 5 min in a sealed or covered container at room temperature or in a constant-temperature device. Record the storage temperature.



- j) After aging, stir the suspension on the mixer for 5 min  $\pm$  6 s.
- k) Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. The temperature of the suspension shall be 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F). Immediately pour the CMC-LVT treated suspension into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- l) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$  30 kPa (100 lbf/in.<sup>2</sup>  $\pm$  5 lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- m) At 7.5 min  $\pm$  6 s on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 mL graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. At 30 min  $\pm$  6 s remove the graduated cylinder and record the volume of filtrate,  $V_c$ , collected.

### 13.6 Calculation—Filtrate Volume of a CMC-LVT Clay Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the clay suspension as given in Equation (34):

$$V = 2V_c \quad (34)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

## 14 Technical-grade, High-viscosity Carboxymethyl Cellulose (CMC-HVT)

### 14.1 Principle

**14.1.1** The CMC-HVT is an alkali-metal salt of carboxymethyl cellulose. The manufacturer shall maintain documentation of the analysis of the cellulosic raw material used.

**14.1.2** The product is a free-flowing or granulated powder usually containing reaction byproducts.

**14.1.3** The CMC-HVT shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 13, represents the product produced, and is controlled by the manufacturer.

**14.1.4** The CMC-HVT shall be free from any starch or starch derivatives. Therefore, a qualitative starch determination shall be performed before proceeding with the CMC-HVT performance testing. If starch is found, no further testing should be performed, and the sample shall be rejected.

**Table 13—CMC-HVT Physical Specifications**

Requirement	Standard
Starch or starch derivatives presence	no
Solution properties	
Viscometer dial reading at 600 r/min	
— in deionized water	minimum 30
— in 40 g/L salt solution	minimum 30
— in saturated salt water	minimum 30
Suspension properties	
Filtrate volume	maximum 10 mL

## 14.2 Qualitative Determination of Starch or Starch Derivatives in a CMC-HVT Sample

### 14.2.1 Description

**14.2.1.1** This test identifies the presence of starch or starch derivatives in water-soluble polymers, like CMC-HVT, supplied in powder or granular form.

**14.2.1.2** An iodine/iodide solution is mixed with a solution of the polymer being analyzed. In the presence of amylose (linear fraction of starch), a colored complex is formed.

### 14.2.2 Reagents and Apparatus—Qualitative Determination of Starch or Starch Derivatives in a CMC-HVT Sample

**14.2.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**14.2.2.2** **Deionized or distilled water.**

**14.2.2.3** **Iodine solution** (CAS No. 7553-56-2), 0.05 mol/L (0.1 N).

**14.2.2.4** **Potassium iodide** (CAS No. 7681-11-0).

**14.2.2.5** **Sodium hydroxide** (CAS No. 1310-73-2), dilute solution, 0.1 % to 0.5 %.

**14.2.2.6** **Mixer**, having each spindle fitted with a single-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**14.2.2.7** **Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**14.2.2.8** **Spatula.**

**14.2.2.9** **Balance**, with accuracy of  $\pm 0.01$  g.

**14.2.2.10** **Volumetric flask**, 100 mL  $\pm 0.1$  mL.

**14.2.2.11** **Pasteur pipette or drop bottle**, plastic.

**14.2.2.12 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

**14.2.2.13 pH meter with pH electrode**, accuracy of 0.01 pH.

**14.2.2.14 Test tubes**, four.

### **14.2.3 Procedure—Preparation of the Iodine/Iodide Solution**

The iodine/iodide solution shall be prepared as per the following procedure.

- a) Prepare the iodine/iodide solution using a 100 mL  $\pm 0.1$  mL volumetric flask. Add 10 mL  $\pm 0.1$  mL of the 0.05 mol/L iodine solution.
- b) Add 0.60 g  $\pm 0.01$  g of potassium iodide and dissolve by gently swirling the volumetric flask.
- c) Bring to the 100 mL mark with deionized water and mix thoroughly. Record the day of preparation.
- d) The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place. It may be used for up to 3 months. After the date of expiry, discard the solution and prepare again.

### **14.2.4 Procedure—Preparation of the CMC-HVT Solution and Qualitative Determination of Starch or Starch Derivatives**

To conduct a test for the presence of starch and starch derivatives in water-soluble polymers, like CMC-HVT, supplied in powder or granular form in a polymer solution, the following procedure shall be applied.

- a) Prepare a 1 % solution of the water-soluble polymer under examination (CMC-HVT) by adding 396.0 g  $\pm 0.1$  g of deionized water to the container. Add 4.00 g  $\pm 0.01$  g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.
- b) After stirring 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container walls. Be sure that all of the polymer clinging to the spatula is incorporated into the solution.
- c) Measure the pH. If the pH value is less than 10.01, raise the pH to  $>10.01$  by adding, dropwise, the dilute sodium hydroxide solution.
- d) Replace the mixing container on the mixer and continue to stir. Total mixing time shall equal 20 min  $\pm 1$  min.
- e) Put 2 mL  $\pm 0.1$  mL of the polymer solution in a test tube and add, dropwise, in portions of 3 drops at a time, up to 30 drops of iodine/iodide solution.
- f) Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution shall be prepared for comparison.
- g) After every 3-drop addition, swirl the test tube gently and compare the color of the solution under examination with the blank tests. A color comparison made against a white background allows for ease of comparison.

### **14.2.5 Interpretation—Qualitative Determination of Starch or Starch Derivatives in a CM-HVT Sample**

**14.2.5.1** The sample under examination gives a yellow color comparable to one of the blank tests [see 14.2.4 f)] if, and only if, the sample does not contain any starch or starch derivatives.

**14.2.5.2** The development of a light green to dark blue color, either in solution or as a precipitate, is an indication of the presence of starch (amylose fraction).

**14.2.5.3** The development of a light pink to reddish-brown color is an indication of the presence of a highly substituted starch, dextrine, or starches with a high amylopectin content. The development of any other color is a strong indication of the presence of starch or starch derivatives.

**14.2.5.4** Instant decolorizing indicates the presence of a reducing agent. In this case, continue the dropwise addition of the iodine/iodide solution and compare the color obtained with cases 14.2.5.1 through 14.2.5.3.

**14.2.5.5** The presence of starch or starch derivatives is contrary to the product definition in Table 13; therefore, further testing is abandoned.

### **14.3 Reagents and Apparatus—Measuring Properties of CMC-HVT Water-soluble Polymers**

**14.3.1** Chemicals shall meet ACS or international equivalent reagent grade.

**14.3.2 Standard evaluation base clay (SEBC)**, see 4.2 and Annex D.

**14.3.3 Sodium chloride** (CAS No. 7647-14-5).

**14.3.4 Sodium bicarbonate** (CAS No. 144-55-8).

**14.3.5 Deionized or distilled water.**

**14.3.6 Defoamer.**

**14.3.7 Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the range specified in this procedure.

**14.3.8 Balance**, with an accuracy of  $\pm 0.01$  g.

**14.3.9 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**14.3.10 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**14.3.11 Spatula.**

**14.3.12 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**14.3.13 Container**, glass or plastic, at least 50 mL, to capture 7.5 min filtrate.

**14.3.14 Storage container**, capacity approximately 500 mL, with lid.

**14.3.15 Volumetric flask**, 1000 mL.

**14.3.16 Timers**, two, digital or mechanical, see 5.2.9 and 5.3.9.

**14.3.17 Graduated cylinders**, three TC, 10 mL  $\pm 0.1$  mL, 100 mL  $\pm 1$  mL, and 500 mL  $\pm 5$  mL.

**14.3.18 Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**14.3.19 Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**14.3.20 Constant-temperature device** (e.g. water bath) set at 20 °C to 25 °C (68 °F to 77 °F), necessary if the room temperature is not in the range of 20 °C to 25 °C (68 °F to 77 °F).

#### 14.4 Procedure—Measuring 600 r/min Dial Reading of CMC-HVT Deionized Water Solution

To measure 600 r/min dial reading of a CMC-HVT deionized water solution, the following procedure shall be applied.

- a) Prepare a solution of CMC-HVT. Add  $2.20 \text{ g} \pm 0.01 \text{ g}$  of CMC-HVT to  $350 \text{ mL} \pm 3 \text{ mL}$  of deionized water to the mixing container at a uniform rate over a time interval of  $60 \text{ s} \pm 6 \text{ s}$  while stirring on the mixer. CMC-HVT shall be added away from impeller shaft to minimize dusting.

NOTE This is equivalent to  $6.29 \text{ g/L} \pm 0.03 \text{ g/L}$ .

- b) After stirring  $5 \text{ min} \pm 6 \text{ s}$ , remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all CMC-HVT clinging to the spatula is incorporated into the suspension.
- c) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal  $20 \text{ min} \pm 1 \text{ min}$ .
- d) Age the solution for up to 16 h in a sealed or covered container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.
- e) After aging, stir the solution on the mixer for  $5 \text{ min} \pm 6 \text{ s}$ .
- f) Pour the solution into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value at 600 r/min is reached. The reading shall be taken at a solution test temperature of  $25 \text{ }^{\circ}\text{C} \pm 1 \text{ }^{\circ}\text{C}$  ( $77 \text{ }^{\circ}\text{F} \pm 2 \text{ }^{\circ}\text{F}$ ).

#### 14.5 Procedure—Measuring 600 r/min Dial Reading of a CMC-HVT 40 g/L Saltwater Solution

To measure 600 r/min dial reading of a CMC-HVT 40 g/L saltwater solution, the following procedure shall be applied.

- a) Prepare a 40 g/L salt solution by adding  $40.0 \text{ g} \pm 0.1 \text{ g}$  of sodium chloride to a 1000 mL volumetric flask and diluting with deionized water to the inscribed mark on the flask. Mix thoroughly.
- b) Prepare a solution of CMC-HVT. Add  $2.70 \text{ g} \pm 0.01 \text{ g}$  of CMC-HVT to  $350 \text{ mL} \pm 3 \text{ mL}$  of the 40 g/L salt solution at a uniform rate over a time interval of 60 s while stirring on the mixer. Add defoamer if necessary.
- c) After stirring  $5 \text{ min} \pm 6 \text{ s}$ , remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all CMC-HVT clinging to the spatula is incorporated into the suspension.
- d) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal  $20 \text{ min} \pm 1 \text{ min}$ .
- e) Age the suspension for up to 16 h in a sealed or covered storage container at room temperature or in a constant-temperature device regulated to  $20 \text{ }^{\circ}\text{C}$  to  $25 \text{ }^{\circ}\text{C}$  ( $68 \text{ }^{\circ}\text{F}$  to  $77 \text{ }^{\circ}\text{F}$ ). Record the storage temperature and storage duration.
- f) After aging, stir the solution on the mixer for  $5 \text{ min} \pm 6 \text{ s}$ .
- g) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at 600 r/min shall be recorded when a constant value at 600 r/min is reached. The reading shall be taken at a solution test temperature of  $25 \text{ }^{\circ}\text{C} \pm 1 \text{ }^{\circ}\text{C}$  ( $77 \text{ }^{\circ}\text{F} \pm 2 \text{ }^{\circ}\text{F}$ ).

#### 14.6 Procedure—Measuring 600 r/min Dial Reading of a CMC-HVT Saturated Saltwater Solution

To measure 600 r/min dial reading of a CMC-HVT saturated saltwater solution, the following procedure shall be applied.

- a) Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 45.0 g  $\pm$  0.1 g of sodium chloride per 100 mL  $\pm$  1 mL of deionized water. Allow the solution to stand for approximately 1 h to ensure saturation. Carefully decant solution or filter it into a storage container.
- b) Prepare a suspension of CMC-HVT. Add 2.50 g  $\pm$  0.01 g of CMC-HVT to 350 mL  $\pm$  3 mL of saturated salt water at a uniform rate over a time interval of 60 s while stirring on the mixer. Add defoamer if necessary.
- c) After stirring 5 min  $\pm$  6 s, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all CMC-HVT clinging to the spatula is incorporated into the suspension.
- d) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min  $\pm$  1 min.
- e) Age the suspension for up to 16 h in a sealed or covered storage container at room temperature or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F). Record the storage temperature and storage duration.
- f) After aging, stir the suspension on the mixer for 5 min  $\pm$  6 s.
- g) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at 600 r/min shall be recorded when a constant value at 600 r/min is reached. The reading shall be taken at a suspension test temperature of 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F).

#### 14.7 Procedure—Measuring Filtrate Volume of a CMC-HVT Clay Suspension

To measure filtrate volume of a CMC-HVT clay suspension, the following procedure shall be applied.

- a) Prepare an ample volume of saturated salt solution by thoroughly mixing a suitable container 45.0 g  $\pm$  0.1 g of sodium chloride per 100 mL  $\pm$  1 mL of deionized water. Allow solution to stand for approximately 1 h to ensure saturation. Carefully decant solution or filter it into a storage container.
- b) Prepare a clay-based suspension by adding 350 mL  $\pm$  3 mL of the saturated salt solution to the mixing container. Add 1.0 g  $\pm$  0.1 g of sodium bicarbonate and stir on the mixer for 60 s  $\pm$  6 s.
- c) Slowly add 35.0 g  $\pm$  0.1 g of SEBC while stirring on the mixer.
- d) After stirring 5 min  $\pm$  6 s, remove the mixing container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.
- e) Replace the mixing container on the mixer and continue to stir. If necessary, the mixing container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min  $\pm$  1 min.
- f) Add 3.15 g  $\pm$  0.01 g of CMC-HVT to the suspension while stirring on the mixer, adding at a uniform rate over 60 s  $\pm$  6 s. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.

- g) After stirring 5 min  $\pm$  6 s, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all material clinging to the spatula is incorporated into the suspension.
- h) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min  $\pm$  1 min.
- i) Age the suspension for 2 h  $\pm$  5 min in a sealed or covered storage container at room temperature or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F). Record the storage temperature.
- j) After aging, stir the suspension on the mixer for 5 min  $\pm$  6 s.
- k) Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. The temperature of the suspension shall be 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F). Immediately pour the CMC-HVT treated suspension into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- l) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust pressure on the cell to 700 kPa  $\pm$  35 kPa (100 lbf/in.<sup>2</sup>  $\pm$  5 lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- m) At 7.5 min  $\pm$  6 s on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 mL graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. At 30 min  $\pm$  6 s remove the graduated cylinder and record the volume of filtrate collected,  $V_c$ .

#### 14.8 Calculation—Filtrate Volume of a CMC-HVT Clay Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the CMC-HVT clay suspension as given in Equation (35):

$$V = 2V_c \quad (35)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

### 15 Starch

#### 15.1 Principle

**15.1.1** Drilling-grade starch can be manufactured from several kinds of native starches. The starch shall be made cold-water hydratable (pre-gelatinized) and can be treated further in such a way that it is suitable for use as a filtrate-reducing agent in water-base drilling fluids.

**15.1.2** Drilling-grade starch shall be free-flowing and free from lumps. Drilling-grade starch shall be deemed to meet the requirements of this standard if a composite sample representing no more than 1 day's production conforms to the physical specifications of Table 14, represents the product produced, and is controlled by the manufacturer.

**Table 14—Starch Physical Specifications**

Requirement	Standard
Suspension properties	
Viscometer dial reading at 600 r/min	
— in 40 g/L salt water	maximum 18
— in saturated salt water	maximum 20
Filtrate volume	
— in 40 g/L salt water	maximum 10 mL
— in saturated salt water	maximum 10 mL
Residue greater than 2000 $\mu\text{m}$	no residue

## 15.2 Reagents and Apparatus—Measuring Properties of a Starch Suspension

**15.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**15.2.2** **Standard evaluation base clay (SEBC)**, see 4.2 and Annex D.

**15.2.3** **Sodium chloride** (CAS No. 7647-14-5).

**15.2.4** **Sodium bicarbonate** (CAS No. 144-55-8).

**15.2.5** **Deionized** or **distilled water**.

**15.2.6** **Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the range specified in the procedure.

**15.2.7** **Balance**, with an accuracy of  $\pm 0.01$  g.

**15.2.8** **Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**15.2.9** **Mixing container**, of approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**15.2.10** **Storage container**, glass, capacity approximately 500 mL, with lid.

**15.2.11** **Container**, glass or plastic, at least 50 mL, to capture 7.5 min filtrate.

**15.2.12** **Spatula**.

**15.2.13** **Volumetric flask**, 1000 mL.

**15.2.14** **Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**15.2.15** **Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**15.2.16** **Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**15.2.17** **Timers**, two, digital or mechanical, see 5.2.9 and 5.3.9.

**15.2.18** **Graduated cylinders**, TC, three, 10 mL  $\pm 0.1$  mL, 100 mL  $\pm 1$  mL, and 500 mL  $\pm 5$  mL.



**15.2.19 Constant-temperature device** (e.g. water bath) set at 20 °C to 25 °C (68 °F to 77 °F), necessary if room temperature is not in the range of 20 °C to 25 °C (68 °F to 77 °F).

### 15.3 Procedure—Measuring 600 r/min Dial Reading of a Starch 40 g/L Saltwater Suspension

To measure 600 r/min dial reading of a starch 40 g/L saltwater suspension, the following procedure shall be applied.

- a) Prepare a 40 g/L saltwater solution by adding 40.0 g  $\pm 0.1$  g of sodium chloride to a 1000 mL volumetric flask and diluting with deionized water to the mark inscribed on the flask. Mix thoroughly.
- b) Prepare a clay-based suspension by adding 350 mL  $\pm 3$  mL of the 40 g/L saltwater solution to the mixing container. Add 1.0 g  $\pm 0.1$  g of sodium bicarbonate, and stir on the mixer for 60 s  $\pm 6$  s.
- c) Slowly add 35.0 g  $\pm 0.1$  g of SEBC while stirring on the mixer.
- d) After stirring 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape its sides with the spatula to dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.
- e) Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the clay shall equal 20 min  $\pm 1$  min.
- f) Add 3.50 g  $\pm 0.01$  g of starch to the suspension while stirring on the mixer, adding at a uniform rate over 60 s  $\pm 6$ .
- g) After stirring 5 min  $\pm 6$  s, remove the container from the mixer and scrape its sides with the spatula to dislodge any starch adhering to the container walls. Be sure that all starch clinging to the spatula is incorporated into the suspension.
- h) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any starch clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the starch shall equal 20 min  $\pm 1$  min.
- i) Age the suspension up to 24 h in a sealed or covered container at room temperature or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F). Record the storage temperature and storage duration.
- j) After aging, stir the suspension on the mixer for 5 min  $\pm 6$  s.
- k) Immediately pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at the 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value for 600 r/min is reached. The 600 r/min dial reading shall be taken at a suspension test temperature of 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F). Record the 600 r/min dial reading.

### 15.4 Procedure—Measuring Filtrate Volume of a Starch 40 g/L Saltwater Suspension

To measure filtrate volume of a starch 40 g/L saltwater suspension, the following procedure shall be applied.

- a) Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. The temperature of the suspension shall be 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F). Pour the starch saltwater suspension from 15.3 into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

- b) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$ 30 kPa (100 lbf/in.<sup>2</sup>  $\pm$ 5 lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- c) At 7.5 min  $\pm$ 6 s on the first timer, remove the container and any liquid adhering to the drain tube and discard the filtrate. Place a dry 10 mL graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. At 30 min  $\pm$ 6 s, remove the graduated cylinder and record the volume of filtrate collected,  $V_c$ .

### 15.5 Calculation—Filtrate Volume of a Starch 40 g/L Saltwater Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the clay suspension as given in Equation (36):

$$V = 2V_c \quad (36)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

### 15.6 Procedure—Measuring 600 r/min Dial Reading of a Starch Saturated Salt Suspension

To measure 600 r/min dial reading of a starch saturated salt suspension, the following procedure shall be applied.

- a) Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 45.0 g  $\pm$ 0.1 g of sodium chloride per 100 mL  $\pm$ 1 mL of deionized water. Allow the solution to stand for approximately 1 h to ensure saturation. Carefully decant the solution or filter it into a storage container.
- b) Prepare a clay-based suspension by adding 350 mL  $\pm$ 3 mL of the saturated salt solution to a container. Add 1.0 g  $\pm$ 0.1 g of sodium bicarbonate and stir on the mixer for 60 s  $\pm$ 6 s.
- c) Slowly add 35.0 g  $\pm$ 0.1 g of SEBC to the container while stirring on the mixer.
- d) After stirring 5 min  $\pm$ 6 s, remove the container from the mixer and scrape its sides with the spatula to dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.
- e) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the clay shall equal 20 min  $\pm$ 1 min.
- f) Add 3.50 g  $\pm$ 0.01 g of starch to the suspension while stirring on the mixer, adding at a uniform rate over 60 s  $\pm$ 6 s.
- g) After stirring 5 min  $\pm$ 6 s, remove the container from the mixer and scrape its sides with the spatula to dislodge any starch adhering to the container walls. Be sure that all starch clinging to the spatula is incorporated into the suspension.
- h) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any starch clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the starch shall equal 20 min  $\pm$ 1 min.

- i) Age the suspension up to 24 h in a sealed or covered storage container at room temperature or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F). Record the storage temperature and storage duration.
- j) After aging, stir the suspension on the mixer for 5 min  $\pm$  6 s.
- k) Immediately pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at the 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value at 600 r/min is reached. The 600 r/min dial reading shall be taken at a suspension test temperature of 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F). Record the 600 r/min dial reading.

### 15.7 Procedure—Measuring Filtrate Volume of a Starch in Saturated Salt Suspension

To measure filtrate volume of a starch in saturated salt suspension, the following procedure shall be applied.

- a) Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. The temperature of the suspension shall be 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F). Pour the starch saturated salt suspension from 15.6 into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- b) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$  30 kPa (100 lbf/in.<sup>2</sup>  $\pm$  5 lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- c) At 7.5 min  $\pm$  6 s on the first timer, remove the container and any liquid adhering to the drain tube and discard the filtrate. Place a dry 10 mL graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. At 30 min  $\pm$  6 s, remove the graduated cylinder and record the volume of filtrate,  $V_c$ , collected.

### 15.8 Calculation—Filtrate Volume of a Starch in Saturated Salt Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the starch in salt saturated clay suspension as given in Equation (37):

$$V = 2V_c \quad (37)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

### 15.9 Apparatus—Measuring Starch Residue Greater than 2000 $\mu$ m

**15.9.1 Sieve**, 2000  $\mu$ m (0.7874 in., U.S. No. 10), in accordance with ASTM E11 or ASTM E161, approximate diameter of 203 mm (8.0 in.).

**15.9.2 Balance**, with an accuracy of  $\pm$ 0.01 g.

### 15.10 Procedure—Measuring Starch Residue Greater than 2000 $\mu$ m

The following procedure shall be applied when measuring starch residue greater than 2000  $\mu$ m.

- a) Weigh 25.0 g  $\pm$  0.01 g starch and transfer to the 2000  $\mu$ m sieve.

- b) Shake for a maximum of 5 min.
- c) Record the presence or absence of residue.

## 16 Low-viscosity Polyanionic Cellulose (PAC-LV)

### 16.1 Principle

**16.1.1** PAC-LV is a water-soluble polymer produced only from cellulose chemically reacted with carboxymethyl (anionic) groups. The product obtained is further purified to significantly increase the active polymer content. It shall not contain any other polysaccharides such as starch, guar, or other naturally occurring polymers or their derivatives. The product is a free-flowing and/or granular powder. See Table 15 for physical requirements.

**16.1.2** The PAC-LV is widely used in water-based drilling fluids for a variety of applications, such as filtration control, viscosity and shale inhibition. Although field use can vary, this procedure focuses on filtration control and viscosity characteristics.

**16.1.3** The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-LV. Specification parameters have been developed for this material.

**16.1.4** The PAC-LV shall be free of any foreign polymer. Therefore, a qualitative starch determination shall be performed. If starch or starch derivatives is found, no further testing should be performed and the sample shall be rejected.

**NOTE** It is recommended that the purity of PAC-LV be greater than 96 % sodium carboxymethyl cellulose (NaCMC) on a dry-weight basis.

**16.1.5** A synthetic-seawater drilling fluid is used for determining the viscosity and filtration control of PAC-LV.

**NOTE** Corrected fluid loss is the filtrate volume collected between 7.5 min and 30.0 min, expressed in milliliters, multiplied by 2. It eliminates the spurt loss seen in many fluid-loss tests.

**16.1.6** The PAC-LV is a polyanionic cellulosic polymer that answers all the requirements specified in 16.1.1 through 16.1.5 and, when testing according to the procedure in 16.2, has a viscosity of less than 40 mPa•s (cP), and has a low-pressure, low-temperature fluid loss of less than 16 mL (corrected).

**NOTE** To obtain the best handling in the field, it is recommended that the particle size for PAC-LV powder be under 0.8 mm [e.g. 0.0315 in., passing through 20 U.S. mesh sieve (850 µm)].

**Table 15—PAC-LV Physical Requirements**

Requirement	Standard
Starch or starch derivatives presence	no
Moisture mass fraction	maximum 10 %
Apparent viscosity	maximum 40 mPa•s (cP)
Filtrate volume	maximum 16 mL

### 16.2 Qualitative Determination of Starch or Starch Derivatives in a PAC-LV Sample

#### 16.2.1 Description

**16.2.1.1** The purpose of this test is to identify the presence of starch or starch derivatives in water-soluble polymers like PAC-LV, supplied in powder or granular form.

**16.2.1.2** An iodine/iodide solution is mixed with a solution of the polymer being analyzed. In the presence of amylose (linear fraction of starch), a colored complex is formed.

**16.2.2 Reagents and Apparatus—Qualitative Determination of Starch or Starch Derivatives in a PAC-LV Sample**

**16.2.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**16.2.2.2** Deionized or distilled water.

**16.2.2.3** Iodine solution (CAS No. 7553-56-2), 0.05 mol/L (0.1 N).

**16.2.2.4** Potassium iodide (CAS No. 7681-11-0).

**16.2.2.5** Sodium hydroxide (CAS No. 1310-73-2), dilute solution, 0.1 % to 0.5 %.

**16.2.2.6** Mixer, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**16.2.2.7** Mixing container, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.). An alternative container option is a 600 mL glass jar.

**16.2.2.8** Laboratory spoon.

**16.2.2.9** Spatula and powder spatula (recommended).

**16.2.2.10** Balance, accuracy of  $\pm 0.01$  g.

**16.2.2.11** Volumetric flask, 100 mL  $\pm 0.1$  mL.

**16.2.2.12** Pasteur pipette or dropper.

**16.2.2.13** Timer, digital or mechanical, see 5.2.9 and 5.3.9.

**16.2.2.14** pH meter with pH electrode, accuracy of 0.01 pH.

**16.2.2.15** Test tubes, four.

**16.2.3 Procedure—Preparation of the Iodine/Iodide Solution**

To prepare the iodine/iodide solution the following procedure shall be applied.

- a) Prepare the iodine/iodide solution using a 100 mL volumetric flask. Add 10 mL  $\pm 0.1$  mL of a 0.05 mol/L iodine solution.
- b) Add 0.60 g  $\pm 0.01$  g of potassium iodide and dissolve by gently swirling the volumetric flask.
- c) Fill to the 100 mL mark with deionized water and mix thoroughly. Record the day of preparation.
- d) The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place and can be used for up to 3 months. After the date of expiration, the solution shall be discarded and prepared again.

#### **16.2.4 Procedure—Preparation of the PAC-LV Solution and Qualitative Determination of Starch or Starch Derivatives**

To conduct a test for the presence of starch and starch derivatives in a PAC-LV water-soluble polymers, supplied in powder or granular form in a polymer solution, the following procedure shall be applied.

- a) Prepare a 5 % solution of the water-soluble polymer under examination. Add 380 g  $\pm$ 0.1 g of deionized water to the mixing container and add 20.00 g  $\pm$ 0.01 g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s while mixing. The PAC-LV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- b) After stirring 5 min  $\pm$ 6 s, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container wall. Be sure that all polymer clinging to the spatula is incorporated into the solution.
- c) Measure the pH; if the pH value is less than 10.01, raise the pH to  $>10.01$  by adding, dropwise, the dilute sodium hydroxide solution.
- d) Replace the mixing container on the mixer and continue to stir. Total mixing time shall equal 20 min  $\pm$ 1 min.
- e) Put 2 mL of the polymer solution in a test tube and add, dropwise, in portions of 3 drops at a time, up to 30 drops of iodine/iodide solution.
- f) Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution should be prepared for comparison.
- g) After every 3-drop addition, swirl the test tube gently and compare the color of the solution under examination with the blank tests. A color comparison made against a white background allows for ease of comparison.

#### **16.2.5 Interpretation—Qualitative Determination of Starch or Starch Derivatives in a PAC-LV Sample**

**16.2.5.1** Instant discoloration indicates the presence of a reducing agent; in this case, continue the dropwise addition of the iodine/iodide solution. Compare the color obtained with one of the blank tests; see 16.2.4 f).

**16.2.5.2** If the sample under examination gives a yellow color comparable to one of the blank tests, the sample does not contain any starch or starch derivatives.

**16.2.5.3** The development of any other color is a strong indication of the presence of starch or starch derivatives. If any color reaction other than that mentioned under 16.2.5.2 is detected, starch or starch derivatives is indicated, and further testing is abandoned.

### **16.3 Measuring Moisture Mass Fraction of a PAC-LV Sample**

#### **16.3.1 Apparatus—Measuring PAC-LV Moisture Mass Fraction**

**16.3.1.1 Oven**, regulated to 105 °C  $\pm$ 3 °C (220 °F  $\pm$ 5 °F).

**16.3.1.2 Balance**, accuracy of  $\pm$ 0.01 g.

**16.3.1.3 Evaporating dish** or functional equivalent.

**16.3.1.4 Desiccator**, with calcium sulfate (CAS number 7778-18-9) desiccant or equivalent.

#### **16.3.2 Procedure—Measuring PAC-LV Moisture Mass Fraction**

To measure the moisture fraction in a sample of PAC-LV, the following procedure shall be applied

- a) Weigh 10.0 g  $\pm 0.01$  g of the PAC-LV sample into a tared evaporating dish. Record the mass as  $m$ .
- b) Dry the sample in the oven for at least 2 h and a constant mass.
- c) Cool the sample to room temperature in a desiccator.
- d) Reweigh the evaporating dish containing the dried PAC-LV. Record the mass as  $m_1$ .

### 16.3.3 Calculation—PAC-LV Moisture Content

Calculate the moisture mass fraction of the PAC-LV sample,  $w_6$ , in percent, as given in Equation (38):

$$w_6 = 100 \frac{(m - m_1)}{m} \quad (38)$$

where

$m$  is the sample mass, expressed in grams;

$m_1$  is the dried sample mass, expressed in grams.

Record the calculated value.

## 16.4 Reagents and Apparatus—Measuring Apparent Viscosity of a PAC-LV Sea Salt Solution

**16.4.1 Sea salt**, ASTM D1141 or equivalent.

**16.4.2 Deionized or distilled water**.

**16.4.3 Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the temperature range specified in the procedure.

**16.4.4 Balance**, accuracy of  $\pm 0.01$  g.

**16.4.5 Mixer**, each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**16.4.6 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**16.4.7 Spatula and powder spatula** (recommended).

**16.4.8 Storage container**, glass or plastic, capacity approximately 500 mL, with lid.

**16.4.9 Constant-temperature device** (e.g. water bath) set at 20 °C to 25 °C (68 °F to 77 °F), necessary if room temperature is not in the range of 20 °C to 25 °C (68 °F to 77 °F).

**16.4.10 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**16.4.11 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

## 16.5 Procedure—Measuring Apparent Viscosity of a PAC-LV Sea Salt Solution

To measure apparent viscosity of a PAC-LV sea salt solution, the following procedure shall be applied.

- a) Add 42.0 g  $\pm 0.1$  g sea salt to 1000 mL  $\pm 2$  mL of deionized water.

- b) To 358 g  $\pm$ 3 g of the sea salt solution in a mixing container, add 35.00 g  $\pm$ 0.01 g of potassium chloride.
- c) Weigh 5.00 g  $\pm$ 0.01 g of the PAC-LV. Add the PAC-LV slowly at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The PAC-LV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- d) After stirring 5 min  $\pm$ 6 s, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.
- e) Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-LV after another 5 min and 10 min. Total mixing time elapsed from beginning of the PAC-LV addition shall equal 20 min  $\pm$ 1 min.
- f) Statically age the suspension for 16 h  $\pm$ 30 min in a sealed or covered storage container in a constant-temperature device at 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F). Record the storage temperature and storage duration.
- g) Stir the suspension on the mixer for 5 min  $\pm$ 6 s.
- h) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at the 600 r/min rotor speed setting of the viscometer shall be taken at a suspension test temperature of 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F).

## 16.6 Calculation—Apparent Viscosity of a PAC-LV Sea Salt Solution

Calculate the test fluid apparent viscosity,  $\eta_{AV}$ , expressed in millipascal•seconds (centipoises), from Equation (39):

$$\eta_{AV} = \frac{R_{600}}{2} \quad (39)$$

where

$R_{600}$  is the viscometer dial reading at 600 r/min.

Record the calculated value.

## 16.7 Reagents and Apparatus—Measuring Filtrate Volume of a PAC-LV Suspension

**16.7.1** Chemicals shall meet ACS or international equivalent reagent grade.

**16.7.2** **Sea salt**, ASTM D1141 or equivalent.

**16.7.3** **Standard evaluation base clay (SEBC)**, see 4.2 and Annex D.

**16.7.4** **Potassium chloride** (CAS No. 7447-40-7).

**16.7.5** **Sodium bicarbonate** (CAS No. 144-55-8).

**16.7.6** **Deionized or distilled water**.

**16.7.7** **Thermometer**, accurate to  $\pm$ 0.5 °C ( $\pm$ 1.0 °F) over the range specified in the procedure.

**16.7.8** **Balance**, accurate to  $\pm$ 0.01 g.



**16.7.9 Mixer**, each spindle is fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**16.7.10 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.).

**16.7.11 Storage container**, glass or plastic, capacity approximately 500 mL, with lid.

**16.7.12 Constant-temperature device** (e.g. water bath) set at 20 °C to 25 °C (68 °F to 77 °F), necessary if room temperature is not in the range of 20 °C to 25 °C (68 °F to 77 °F).

**16.7.13 Container**, glass or plastic, at least 50, to capture 7.5 min filtrate.

**16.7.14 Spatula and powder spatula** (recommended).

**16.7.15 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**16.7.16 Timers**, two, digital or mechanical, see 5.2.9 and 5.39.

**16.7.17 Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**16.7.18 Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**16.7.19 Graduated cylinders**, two, 10 mL  $\pm 0.1$  mL and 500 mL  $\pm 5$  mL.

## **16.8 Procedure—Measuring Filtrate Volume of a PAC-LV Suspension**

To measure filtrate volume of a PAC-LV suspension, the following procedure shall be applied.

- a) Add 42.0 g  $\pm 0.1$  g sea salt to 1000 mL  $\pm 2$  mL of deionized water.
- b) To 358 g  $\pm 3$  g of the sea salt solution in a mixing container, add 35.00 g  $\pm 0.01$  g potassium chloride.
- c) After stirring 3 min  $\pm 6$  s, add 1.00 g  $\pm 0.01$  g of sodium bicarbonate.
- d) After stirring 3 min  $\pm 6$  s, add 28.00 g  $\pm 0.01$  g of SEBC.
- e) After stirring 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.
- f) Replace the container on the mixer and continue to stir an additional 5 min  $\pm 6$  s.
- g) Weigh 2.00 g  $\pm 0.01$  g of PAC-LV.
- h) Add the PAC-LV slowly at a uniform rate over a time interval of 60 s  $\pm 6$  s while stirring on the mixer. The PAC-LV should be added away from the impeller shaft but in the vortex to minimize dusting, preferably with a laboratory powder spatula.
- i) After stirring 5 min  $\pm 6$  s, remove the container from the mixer and scrape its side with the spatula to dislodge any PAC-LV adhering to the container walls. Be sure that all PAC-LV clinging to the spatula is incorporated into the suspension.
- j) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-LV after another 5 min and 10 min. Total mixing time elapsed from the beginning of PAC-LV addition shall equal 20 min  $\pm 1$  min.

- k) Age the suspension for 16 h  $\pm$ 30 min in a sealed or covered storage container in a constant-temperature device at 77 °F  $\pm$ 2 °F (25 °C  $\pm$ 1 °C). Record the storage temperature and storage duration.
- l) After aging, stir the suspension on the mixer for 5 min  $\pm$ 6 s.
- m) Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. The temperature of the suspension shall be 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F). Pour the PAC-LV suspension into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- n) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$ 30 kPa (100 lbf/in.<sup>2</sup>  $\pm$ 5 lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium
- o) After 7.5 min  $\pm$ 6 s on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place a dry 10 mL graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. After 30 min  $\pm$ 6 s, remove the graduated cylinder and record the volume of filtrate,  $V_c$ , collected.

## 16.9 Calculation—Filtrate Volume of PAC-LV Suspension

Calculate  $V$ , the total filtrate volume of the PAC-LV suspension, expressed in milliliters, as given in Equation (40):

$$V = 2V_c \quad (40)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

## 17 High-viscosity Polyanionic Cellulose (PAC-HV)

### 17.1 Principle

**17.1.1** PAC-HV, also referred to as API PAC regular-grade, is a water-soluble polymer produced only from cellulose chemically reacted with carboxymethyl (anionic) groups. The product obtained is further purified to significantly increase the active polymer content. It shall not contain any other polysaccharides, such as starch, guar, or other naturally occurring polymers or their derivatives. The product is a free-flowing and/or granular powder. See Table 16 for physical requirements.

**NOTE** It is recommended that the purity of the PAC-HV be greater than 96 % NaCMC on a dry-weight basis.

**17.1.2** The PAC-HV is widely used in water-based drilling fluids for a variety of applications, such as filtration control, viscosity, and shale inhibition. Although field use can vary, this procedure focuses on filtration-control and viscosity-control characteristics.

**17.1.3** The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-HV. Specification parameters are developed for this material.

**17.1.4** The PAC-HV shall be free of any foreign polymer. Therefore, a qualitative starch determination shall be performed before the performance tests. If starch or starch derivatives is found, no further testing should be performed, and the sample shall be rejected.

**17.1.5** A synthetic-seawater drilling fluid is used for determining the filtration control and apparent viscosity of the PAC-HV.

NOTE Corrected fluid loss is the filtrate volume collected between 7.5 min and 30.0 min, in milliliters, multiplied by 2. It eliminates the spurt loss seen in many fluid-loss tests.

**17.1.6** The PAC-HV is a polyanionic cellulosic polymer that answers all the requirements in 17.1.1 through 17.1.5 and, when tested in accordance with 17.2, has an apparent viscosity above 50 cP (mPa•s) and a low-pressure/low-temperature fluid loss of less than 23 mL (corrected).

NOTE To obtain the best handling in the field, it is recommended that the particle size for the PAC-HV powder be under 800  $\mu\text{m}$  [e.g. 0.0315 in., passing through 20 mesh sieve (850  $\mu\text{m}$ )].

**Table 16—PAC-HV Physical Requirements**

Requirement	Standard
Starch or starch derivatives presence	no
Moisture mass fraction	maximum 10 %
Apparent viscosity	minimum 50 mPa•s (cP)
Filtrate volume	maximum 23 mL

## **17.2 Qualitative Determination of Starch or Starch Derivatives in PAC-HV Sample**

### **17.2.1 Description**

**17.2.1.1** The purpose of the test is to identify the presence of starch or starch derivatives in water-soluble polymers like PAC-HV, supplied in powder or granular form.

**17.2.1.2** An iodine/iodide solution is mixed with a solution of the polymer being analyzed. In the presence of amylose (linear fraction of starch), a colored complex is formed.

### **17.2.2 Reagents and Apparatus—Qualitative Determination of Starch or Starch Derivatives in a PAC-HV Sample**

**17.2.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**17.2.2.2** Deionized or distilled water.

**17.2.2.3** Iodine solution (CAS No. 7553-56-2), 0.05 mol/L (0.1N).

**17.2.2.4** Potassium iodide (CAS No. 7681-11-0).

**17.2.2.5** Sodium hydroxide (CAS no. 1310-73-2), dilute, 0.1 % to 0.5 %.

**17.2.2.6** Mixer, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**17.2.2.7** Mixing container, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.). An alternative container option is a glass jar of 600 mL.

**17.2.2.8** Laboratory spoon.

**17.2.2.9** Spatula and powder spatula (recommended).

**17.2.2.10** Balance, accurate to  $\pm 0.01$  g.

**17.2.2.11** Volumetric flask, 100 mL  $\pm 0.1$  mL.

**17.2.2.12 Pasteur pipette**, or **drop bottle**, plastic.

**17.2.2.13 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

**17.2.2.14 pH meter** with **pH electrode**, accuracy of 0.01 pH.

**17.2.2.15 Test tubes**, four.

### **17.2.3 Procedure—Preparation of the Iodine/Iodide Solution**

To prepare the iodine/iodide solution the following procedure shall be applied.

- a) Prepare the iodine/iodide solution using a 100 mL volumetric flask. Add 10 mL  $\pm 0.1$  mL of a 0.05 mol/L iodine solution.
- b) Add 0.60 g  $\pm 0.01$  g of potassium iodide and dissolve by gently swirling the volumetric flask.
- c) Fill to the 100 mL mark with deionized water and mix thoroughly. Record the day of preparation.
- d) The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place and can be used for up to 3 months. After the date of expiration, the solution should be discarded and prepared again.

### **17.2.4 Procedure—Preparation of the PAC-HV Solution and Qualitative Determination of Starch or Starch Derivatives**

To conduct a test for the presence of starch and starch derivatives in water-soluble polymers, like PAC-HV, supplied in powder or granular form in a polymer solution, the following procedure shall be applied.

- a) Prepare a 1 % solution of the water-soluble polymer under examination. Add 396.0 g  $\pm 0.1$  g of deionized water to the mixing container and add 4.00 g  $\pm 0.01$  g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s. The PAC-HV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- b) After stirring 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container wall. Be sure that all the polymer clinging to the spatula is incorporated into the solution.
- c) Measure the pH. If the pH value is less than 10.01, raise the pH to  $>10.01$  by adding, dropwise, dilute sodium hydroxide solution.
- d) Replace the container on the mixer and continue to stir. Total mixing time shall equal 20 min  $\pm 1$  min.
- e) Put 2 mL of the polymer solution in a test tube and add, dropwise, in portions of 3 drops at a time, up to 30 drops of iodine/iodide solution.
- f) Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution shall be prepared for comparison.
- g) After every 3-drop addition, swirl the test tube gently and compare the color of the solution under examination with the blank tests. A color comparison made against a white background allows for ease of comparison.

### **17.2.5 Interpretation—Qualitative Determination of Starch or Starch Derivatives in a PAC-HV Sample**

**17.2.5.1** Instant discoloration indicates the presence of a reducing agent; in this case, continue the dropwise addition of the iodine/iodide solution. Compare the color obtained with one of the blank tests; see 16.2.4 f).

**17.2.5.2** If the sample under examination gives a yellow color comparable to one of the blank tests, the sample does not contain any starch or starch derivatives.

**17.2.5.3** The development of any other color is a strong indication of the presence of starch or starch derivatives. If any other color develops other than that mentioned under 16.2.5.2, starch or starch derivatives is indicated, and further testing shall be abandoned.

### **17.3 Measuring Moisture Mass Fraction of a PAC-HV Sample**

#### **17.3.1 Reagents Apparatus—Moisture Mass Fraction of a PAC-HV Sample**

**17.3.1.1 Oven**, regulated to 105 °C ±3 °C (220 °F ±5 °F).

**17.3.1.2 Balance**, accuracy of ±0.01 g.

**17.3.1.3 Evaporating dish** or functional equivalent.

**17.3.1.4 Spatula** and **powder spatula** (recommended).

**17.3.1.5 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

#### **17.3.2 Procedure—Moisture Mass Fraction of a PAC-HV Sample**

To measure the moisture fraction in a sample of PAC-LV, the following procedure shall be as applied.

- a) Weigh 10.0 g ±0.01 g of the PAC-HV sample into a tared evaporating dish. Record the mass as  $m$ .
- b) Dry the sample in the oven for at least 2 h to a constant mass.
- c) Cool the sample to room temperature in the desiccator.
- d) Reweigh the evaporating dish containing the PAC-HV. Record the mass as  $m_1$ .

#### **17.3.3 Calculation—Moisture Mass Fraction of a PAC-HV Sample**

Calculate the mass fraction moisture,  $w_6$ , in percent, as given in Equation (41):

$$w_6 = 100 \frac{(m - m_1)}{m} \quad (41)$$

where

$m$  is the sample mass, expressed in grams;

$m_1$  is the dried sample mass, expressed in grams.

Record the calculated value.

### **17.4 Reagents and Apparatus—Measuring Apparent Viscosity of a PAC-HV Sea Salt Solution**

**17.4.1 Sea salt**, ASTM D1141 or equivalent.

**17.4.2 Deionized or distilled water**.

**17.4.3 Thermometer**, accurate to ±0.5 °C (±1.0 °F) over the temperature range specified in the procedure.

**17.4.4 Balance**, accuracy of  $\pm 0.01$  g.

**17.4.5 Mixer**, each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**17.4.6 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**17.4.7 Spatula and powder spatula** (recommended).

**17.4.8 Storage container**, glass or plastic, capacity approximately 500 mL, with lid.

**17.4.9 Constant-temperature device** (e.g. water bath) set at 20 °C to 25 °C (68 °F to 77 °F), necessary if room temperature is not in the range of 20 °C to 25 °C (68 °F to 77 °F).

**17.4.10 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**17.4.11 Timers**, digital or mechanical, see 5.2.9 and 5.3.9.

## **17.5 Procedure—Measuring Apparent Viscosity of a PAC-HV Sea Salt Solution**

To measure apparent viscosity of a PAC-HV sea salt solution, the following procedure shall be applied.

- a) Add 42.0 g  $\pm 0.1$  g of sea salt to 1000 mL  $\pm 2$  mL of deionized water.
- b) To 358 g  $\pm 3$  g of the sea salt solution, add 35.00 g  $\pm 0.01$  g of potassium chloride.
- c) Weigh 3.00 g  $\pm 0.01$  g of PAC-HV. Add the PAC-HV slowly at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The PAC-HV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- d) After stirring 5 min  $\pm 6$  s, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.
- e) Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-HV after another 5 min and 10 min. Total mixing time elapsed from beginning of the PAC-HV addition shall equal 20 min  $\pm 1$  min.
- f) Age the suspension for 16 h  $\pm 30$  min in a sealed or covered storage container in a constant-temperature device at 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F). Record the storage temperature and storage duration.
- g) Stir the suspension on the mixer for 5 min  $\pm 6$  s.
- h) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading,  $R_{600}$ , at the 600 r/min rotor speed setting of the viscometer shall be taken at a suspension test temperature of 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F).

## 17.6 Calculation—Apparent Viscosity of a PAC-HV Sea Salt Solution

Calculate the PAC-HV sea salt solution apparent viscosity,  $\eta_{AV}$ , expressed in millipascal•seconds (centipoises), from Equation (42):

$$\eta_{AV} = \frac{R_{600}}{2} \quad (42)$$

where

$R_{600}$  is the viscometer dial reading at 600 r/min.

Record the calculated value.

## 17.7 Reagents and Apparatus—Measuring Filtrate Volume of a PAC-HV Suspension

**17.7.1** Chemicals shall meet ACS or international equivalent reagent grade.

**17.7.2** **Sea salt**, ASTM D1141 or equivalent.

**17.7.3** **Standard evaluation base clay (SEBC)**, see 4.2 and Annex D.

**17.7.4** **Potassium chloride** (CAS No. 7447-40-7).

**17.7.5** **Sodium bicarbonate** (CAS No. 144-55-8).

**17.7.6** **Deionized or distilled water**.

**17.7.7** **Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the temperature range specified in the procedure.

**17.7.8** **Balance**, accuracy of  $\pm 0.01$  g.

**17.7.9** **Mixer**, each spindle is fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**17.7.10** **Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**17.7.11** **Spatula** and **powder spatula** (recommended).

**17.7.12** **Storage container**, glass or plastic, capacity approximately 500 mL, with lid.

**17.7.13** **Container**, glass or plastic, at least 50 mL, to capture 7.5 min filtrate.

**17.7.14** **Constant-temperature device** (e.g. water bath) set at 20 °C to 25 °C (68 °F to 77 °F), necessary if room temperature is not in the range of 20 °C to 25 °C (68 °F to 77 °F).

**17.7.15** **Viscometer**, motor driven, direct indicating, in accordance with API 13B-1.

**17.7.16** **Timers**, two, digital or mechanical, see 5.2.9 and 5.3.9.

**17.7.17** **Filter press**, low pressure/low temperature, in accordance with API 13B-1.

**17.7.18** **Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13).

**17.7.19 Graduated cylinders**, two, 10 mL  $\pm 0.1$  mL and 500 mL  $\pm 5$  mL.

## **17.8 Procedure—Measuring the Filtrate Volume of a PAC-HV Suspension**

To measure the filtrate volume of a PAC-HV suspension, the following procedure shall be applied.

- a) Add 42.0 g  $\pm 0.1$  g of sea salt to 1000 mL  $\pm 2$  mL of deionized water.
- b) To 358 g  $\pm 3$  g of the sea salt solution, add 35.00 g  $\pm 0.01$  g of potassium chloride.
- c) After stirring 3 min  $\pm 6$  s, add 1.00 g  $\pm 0.01$  g of sodium bicarbonate.
- d) After stirring 3 min  $\pm 6$  s, add 28.00 g  $\pm 0.01$  g of SEBC.
- e) After stirring 5 min  $\pm 6$  s, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.
- f) Replace the container on the mixer and continue to stir an additional 5 min  $\pm 6$  s.
- g) Weigh 1.00 g  $\pm 0.01$  g of PAC-HV.
- h) Add the PAC-HV slowly at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The PAC-HV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- i) After stirring 5 min  $\pm 6$  s, remove the container from the mixer and scrape its side with the spatula to dislodge any PAC-HV adhering to the container walls. Be sure that all PAC-HV clinging to the spatula is incorporated into the suspension.
- j) Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-HV after another 5 min and 10 min. Total mixing time elapsed from the beginning of PAC-HV addition shall equal 20 min  $\pm 1$  min.
- k) Age the suspension for 16 h  $\pm 30$  min in a sealed or covered container in a constant-temperature device at 77 °F  $\pm 2$  °F (25 °C  $\pm 1$  °C). Record the storage temperature and storage duration.
- l) After aging, stir the suspension on the mixer for 5 min  $\pm 6$  s.
- m) Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with API 13B-1. The temperature of the suspension shall be 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F). Pour the PAC-HV suspension into the filter press cell to about 13 mm (0.5 in.) from the top of the cell. Complete the assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- n) Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm 30$  kPa (100 lbf/in.<sup>2</sup>  $\pm 5$  lbf/in.<sup>2</sup>). Both steps shall be completed in less than 15 s. The pressure shall be supplied by compressed air, nitrogen, or helium.
- o) After 7.5 min  $\pm 6$  s on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place a dry 10 mL graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. At 30 min  $\pm 6$  s remove the graduated cylinder and record the volume of the filtrate collected,  $V_C$ .



## 17.9 Calculation—Filtrate Volume of PAC-HV Suspension

Calculate the filtrate volume of the PAC-HV clay Suspension,  $V$ , expressed in milliliters, from Equation (43).

$$V = 2 V_c \quad (43)$$

where

$V_c$  is the filtrate volume collected, expressed in milliliters, between 7.5 min and 30 min.

## 18 Drilling-grade Xanthan Gum

### 18.1 Principle

**18.1.1** Xanthan gum is a water-soluble polysaccharide, commercially produced by a distinct fermentation process of *Xanthomonas campestris*, and shall not contain any other polysaccharide, such as starch, guar gum, or other naturally occurring polymers or their derivatives. The product may contain up to 3 % of a material added specifically to enhance dispersibility or solubility of the product. Xanthan gum is an off-white, free-flowing granular powder.

**18.1.2** Xanthan gum is used in a wide variety of water-based drilling, workover, or completion fluids as a rheology modifier to develop low-shear-rate viscosity and enhance suspension properties.

**18.1.3** This procedure is a simple and reproducible method for assessing the performance properties of xanthan gum used in drilling operations. Other specifications may be established for pay-zone applications, including stimulation, workover, or completion operations.

**18.1.4** Xanthan gum shall be free of any foreign polymer, including cellulosic, starch, or guar gum. Qualitative tests should be performed to detect the presence of guar or starch or their derivatives, which are known to have synergistic effects on the viscosity of xanthan gum solutions.

**18.1.5** A solution of xanthan gum in synthetic seawater is used to determine the viscosity characteristics of xanthan gum.

**18.1.6** Xanthan gum shall conform to the specifications of Table 17.

**NOTE** For optimum handling and mixing of the polymer in the field, it is recommended that the particle size of xanthan gum powder be less than 425  $\mu\text{m}$  (0.0167 in., passing through 40 U.S. mesh sieve).

**Table 17—Xanthan Gum Physical Requirements**

Requirement	Standard
Starch, guar, or their derivatives presence	no
Moisture mass fraction	maximum 13 %
Screen analysis	
Less than 425 $\mu\text{m}$ (0.167 in.)	minimum 95 %
Less than 75 $\mu\text{m}$ (0.003 in.)	maximum 50 %
Viscosity <sup>a</sup>	
Rotational viscometer, 300 r/min	minimum 11 mPa•s (cP) (minimum 55 dial reading)
Rotational viscometer, 6 r/min	minimum 180 mPa•s (cP) (minimum 18 dial reading)
Rotational viscometer, 3 r/min	minimum 320 mPa•s (cP) (minimum 16 dial reading)
Low-shear-rate viscometer, 1.5 r/min <sup>b</sup>	minimum 1950 mPa•s (cP)
<sup>a</sup> For rotational viscometer, equipped with F0.2 torsion spring, R1/B1 configuration: a) 300 r/min, millipascal•seconds (cP) equals the dial reading times 0.2; b) 6 r/min, millipascal•seconds (cP) equals the dial reading times 10.0; c) 3 r/min, millipascal•seconds (cP) equals the dial reading times 20.0. <sup>b</sup> For the low-viscosity viscometer, see 18.10.2.1.	

## 18.2 Qualitative Determination of Starch or Starch Derivatives in a Xanthan Gum Sample

### 18.2.1 Description

**18.2.1.1** The purpose of this test is to identify the presence of starch or starch derivatives in xanthan gum, supplied in powder or granular form. If starch or starch derivatives is found, no further testing should be performed, and the sample shall be rejected.

**18.2.1.2** An iodine/iodide solution is mixed with a solution of the xanthan gum being analyzed. In the presence of amylose (linear fraction of starch), a colored complex is formed.

### 18.2.2 Reagents and Apparatus—Qualitative Determination of Starch or Starch Derivatives in a Xanthan Gum Sample

**18.2.2.1** Chemicals shall meet ACS or international equivalent reagent grade.

**18.2.2.2 Iodine solution** (CAS No. 7553-56-2), 0.05 mol/L (0.1N).

**18.2.2.3 Potassium iodide** (CAS No. 7681-11-0).

**18.2.2.4 Sodium hydroxide**, e.g. caustic soda (CAS No. 1310-73-2), dilute 0.1 % to 0.5 %.

**18.2.2.5 Deionized or distilled water.**

**18.2.2.6 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**18.2.2.7 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); d top, 97 mm (3.8 in.); d bottom, 70 mm (2.75 in.).

**18.2.2.8 Laboratory spoon.**

**18.2.2.9 Spatula and powder spatula** (recommended).

**18.2.2.10 Balance**, accurate to  $\pm 0.01$  g.

**18.2.2.11 Volumetric flask**, 100 mL  $\pm 0.1$  mL.

**18.2.2.12 Pasteur pipette or dropper**.

**18.2.2.13 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

**18.2.2.14 pH meter with pH electrode**, accuracy of 0.01 pH.

**18.2.2.15 Test tubes**, four.

### **18.2.3 Procedure—Preparation of the Iodine/Iodide Solution**

To prepare the iodine/iodide solution the following procedure shall be applied.

- a) Prepare the iodine/iodide solution using the 100 mL volumetric flask. Add 10 mL  $\pm 0.1$  mL of a 0.05 mol/L iodine solution.
- b) Add 0.60 g  $\pm 0.01$  g of potassium iodide and dissolve by gently swirling the volumetric flask.
- c) Fill to the 100 mL mark with deionized water and mix thoroughly. Record the day of preparation.
- d) The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place and can be used for up to 3 months. After the date of expiration, the solution should be discarded and prepared again.

### **18.2.4 Procedure—Qualitative Determination of Starch or Starch Derivatives in a Xanthan Gum Sample**

To conduct a test for the presence of starch and starch derivatives in water-soluble polymers, like xanthan gum, supplied in powder or granular form in a polymer solution, the following procedure shall be applied.

- a) Prepare a 1 % solution of the xanthan gum under examination. Add 396.0 g  $\pm 0.1$  g of deionized water to the mixing container and add 4.00 g  $\pm 0.01$  g of xanthan gum at a uniform rate over a time interval of 60 s to 120 s. The xanthan gum polymer shall be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- b) After stirring 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any xanthan gum adhering to the container wall. Be sure that all xanthan gum clinging to the spatula is incorporated into the solution.
- c) Measure the pH. If the pH value is less than 10.01, raise the pH to  $>10.01$  by adding, dropwise, dilute sodium hydroxide solution.
- d) Replace the mixing container on the mixer and continue to stir. Total mixing time shall be 20 min  $\pm 1$  min.
- e) Put 2 mL of the xanthan gum solution in a test tube and add, dropwise, in portions of 3 drops at a time, up to 30 drops of the iodine/iodide solution.
- f) Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution shall be prepared for comparison.
- g) After every 3-drop addition, gently swirl the test tube and compare the color of the solution under examination with the blank test. A color comparison made against a white background allows for ease of comparison.

### 18.2.5 Interpretation—Qualitative Determination of Starch or Starch Derivatives in a Xanthan Gum Sample

**18.2.5.1** Instant discoloration indicates the presence of a reducing agent. In this case, continue the dropwise addition of the iodine/iodide solution.

**18.2.5.2** If the sample under examination gives a yellow color comparable to one of the blank tests [see 18.2.4 f)], the sample does not contain any starch or starch derivatives.

**18.2.5.3** The development of a light green to dark blue color, either in solution or as a precipitate, indicates the presence of starch (amylose fraction).

**18.2.5.4** The development of a light pink to reddish-brown color is an indication of the presence of a highly substituted starch, dextrin, or starches with a high amylopectin content.

**18.2.5.5** The development of any other color is a strong indication of the presence of starch or starch derivatives.

**18.2.5.6** If any color reaction is detected other than that mentioned under 18.2.5.2 to 18.2.5.5, the testing is abandoned.

### 18.3 Qualitative Determination of Guar in a Xanthan Gum Sample

#### 18.3.1 Principle

**18.3.1.1** The presence of guar additives to xanthan gum can be detected if guar is present in the minimum range of 7.5 % to 10.0 % by mass.

**18.3.1.2** Sodium borate (borax) is used to crosslink the guar fraction, which causes an increase in the viscosity of the xanthan gum solution.

#### 18.3.2 Reagents and Apparatus—Qualitative Determination of Guar in a Xanthan Gum Sample

**18.3.2.1 Water** (CAS No.7732-18-5), tap.

**18.3.2.2 Sodium borate decahydrate** (CAS No. 1303-06-4), e.g. borax.

**18.3.2.3 Water**, warm, tap, 32 °C  $\pm$  2 °C (90 °F  $\pm$  3 °F), for borate solution.

**18.3.2.4 Balance**, accurate to 0.01 g.

**18.3.2.5 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**18.3.2.6 Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.); *d* top, 97 mm (3.8 in.); *d* bottom, 70 mm (2.75 in.). An alternative container option is a 600 mL glass jar.

**18.3.2.7 Spatula and powder spatula** (recommended).

**18.3.2.8 Thermometer**, accurate to  $\pm$ 0.5 °C ( $\pm$ 1.0 °F) over the range specified in this procedure.

**18.3.2.9 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1, with an R1-B1 configuration and equipped with a F0.2 torsion spring. See 18.11 for calibration.

NOTE F0.2 spring: torsional stiffness 77.2 dyne•centimeters per degree of deflection, shear stress constant equals to 0.102 pascals per degree of deflection (0.213 lbf/100•ft<sup>2</sup> per degree of deflection).

**18.3.2.10 Beaker**, tall form, 400 mL, borosilicate glass.

**18.3.2.11 Graduated cylinder**, one 50 mL  $\pm 1.0$  mL.

**18.3.2.12 Weighing paper**.

**18.3.2.13 Water bath**, maintained at 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F).

**18.3.2.14 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

### **18.3.3 Procedure—Preparation of the Freshwater Xanthan Gum Solution**

Freshwater xanthan gum solution shall be prepared with the following procedure.

- a) Weigh 350 g  $\pm 1$  g of water into the mixing container.
- b) Weigh 1.50 g  $\pm 0.01$  g of xanthan gum onto weighing paper.
- c) Place the container onto the mixer and switch on the mixer.
- d) Add the xanthan gum at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The xanthan gum should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.
- e) After stirring 5 min  $\pm 6$  s, remove the mixing container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any xanthan gum adhering to the container wall. Be sure that all xanthan gum clinging to the spatula is incorporated into the solution.
- f) Replace the mixing container on the mixer and continue to stir. Total mixing time shall equal 15 min  $\pm 1$  min.
- g) Place the mixing container in chilled water bath for not longer than 30 min to lower the fluid temperature to 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F).

### **18.3.4 Procedure—Preparation of the Sodium Borate Solution**

Sodium borate solution shall be prepared with the following procedure.

- a) Weigh 10.0 g  $\pm 0.1$  g of sodium borate onto weighing paper.
- b) Weigh 90 g  $\pm 1$  g of warm water into the beaker.
- c) Add the sodium borate to water and stir with a spatula for approximately 1 min to dissolve the sodium borate.

### **18.3.5 Procedure—Viscosity Measurement of the Xanthan Gum Solution**

To measure viscosity of the xanthan gum solution, the following procedure shall be applied.

- a) Pour the freshwater xanthan gum solution (18.3.3) into the beaker to the 250 mL mark.
- b) Place the beaker on the viscometer stage and raise the stage so that the fluid level is equal to the inscribed line on the rotor sleeve. The reading shall be taken at a solution test temperature of 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F).
- c) With the viscometer set at 300 r/min, switch on the viscometer and wait 15 s to 20 s for the reading to stabilize. Read to the nearest 0.5 dial reading after stabilization. Record the dial reading as  $R_{300-P}$ .

- d) Change the viscometer setting to 6 r/min, wait 20 s to 30 s for the dial to stabilize, and read to the nearest 0.5 dial reading after stabilization. Record the dial reading as  $R_{6-P}$ .
- e) Change the viscometer setting to 3 r/min, wait 20 s to 30 s for the dial to stabilize, and read to the nearest 0.5 dial reading after stabilization. Record the dial reading as  $R_{3-P}$ .

### 18.3.6 Procedure—Viscosity Measurement of the Xanthan Gum Solution Treated with the Sodium Borate Solution

To measure viscosity of the xanthan gum solution treated with the sodium borate solution, the following procedure shall be applied.

- a) Following the completion of 18.3.5, pour the xanthan gum solution into the original mixing container.
- b) Measure 30 mL  $\pm$ 2.0 mL of sodium borate solution (18.3.4) using the graduated cylinder and add to the xanthan gum solution.
- c) Hand stir with the spatula for 20 s to 30 s. Allow mixture to stand for 5 min  $\pm$ 15 s.
- d) Pour the mixture into the beaker to the 250 mL mark.
- e) Place the beaker on the viscometer stage and raise the stage so that the fluid level is equal to the inscribed line on the rotor sleeve. The reading shall be taken at a solution test temperature of 25 °C  $\pm$ 1 °C (77 °F  $\pm$ 2 °F).
- f) With the viscometer set at 300 r/min, switch on the viscometer and wait 20 s to 30 s for the reading to stabilize. Read to the nearest 0.5 dial reading after stabilization. Record the dial reading as  $R_{300-M}$ .
- g) Change the viscometer setting to 6 r/min, wait 20 s to 30 s for the dial to stabilize and read to the nearest 0.5 dial reading after stabilization. Record the dial reading as  $R_{6-M}$ .
- h) Change the viscometer setting to 3 r/min, wait 20 s to 30 s for the dial to stabilize and read to the nearest 0.5 dial reading after stabilization. Record the dial reading as  $R_{3-M}$ .

### 18.3.7 Interpretation—Qualitative Determination of Guar in a Xanthan Gum Sample

**18.3.7.1** Compare the readings for the xanthan gum solution ( $R_{300-P}$ ,  $R_{6-P}$ , and  $R_{3-P}$ ) and the mixture of xanthan gum and sodium borate solutions ( $R_{300-M}$ ,  $R_{6-M}$ , and  $R_{3-M}$ ).

**18.3.7.2** Any increase between mixture readings and xanthan gum solution readings indicates the presence of guar in the xanthan gum sample, and further testing shall be abandoned.

## 18.4 Measuring Moisture Mass Fraction of a Xanthan Gum Sample

### 18.4.1 Apparatus—Measuring Moisture Mass Fraction of a Xanthan Gum

**18.4.1.1 Oven**, regulated to 105 °C  $\pm$ 3 °C (220 °F  $\pm$ 5 °F).

**18.4.1.2 Balance**, accuracy of  $\pm$ 0.01 g.

**18.4.1.3 Metal weighing pan**.

**18.4.1.4 Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant or equivalent.

### 18.4.2 Procedure—Measuring Moisture Mass Fraction of a Xanthan Gum

To measure the moisture fraction in a sample of xanthan gum, the following procedure shall be as applied.

- Weigh 4.00 g  $\pm 0.01$  g xanthan gum in a tared metal weighing pan and record the mass,  $m$ .
- Dry sample in the oven for at least 2 h to a constant mass.
- Cool to room temperature in desiccator.
- Reweigh the metal weighing pan containing the dry xanthan gum. Record the residue mass as  $m_1$ .

### 18.4.3 Calculation—Measuring Moisture Mass Fraction of a Xanthan Gum

Calculate the mass fraction moisture of the xanthan gum sample,  $w_6$ , in percent, as given in Equation (44):

$$w_6 = 100 \frac{(m - m_1)}{m} \quad (44)$$

where

$m$  is the sample mass, expressed in grams;

$m_1$  is the dried sample mass, expressed in grams.

Record the calculated value.

## 18.5 Measuring Particle Size of a Xanthan Gum Sample

### 18.5.1 Apparatus—Measuring Particle Size of a Xanthan Gum

**18.5.1.1 Mechanical sieve shaker**, capable of simultaneous horizontal and vertical motion.

**18.5.1.2 Sieves**, in accordance with ASTM E11 or ASTM E161, one 425  $\mu\text{m}$  (0.0166 in., U.S. No. 40), and one 75  $\mu\text{m}$  (0.0029 in., U.S. No. 200), each approximately 250 mm (8 in.) diameter.

**18.5.1.3 Top lid and bottom receiver**, to fit standard sieves.

**18.5.1.4 Balance**, accurate to  $\pm 0.01$  g.

**18.5.1.5 Weighing pan or weighing paper**.

**18.5.1.6 Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

### 18.5.2 Procedure—Measuring Particle Size of a Xanthan Gum

The following procedure shall be applied when measuring particle size of a xanthan gum sample.

- Visually inspect each sieve making certain that each is free of damage or blinding.
- Weigh the 425  $\mu\text{m}$  sieve and the bottom receiver. Record the masses as  $m_5$  for the 425  $\mu\text{m}$  sieve and  $m_6$  for the bottom receiver.
- Thoroughly mix dry xanthan gum sample and weigh 50.00 g  $\pm 0.01$  g of the sample into a weighing pan or onto weighing paper. Record the mass sample in grams, as  $m$ .

- d) Assemble the sieves with bottom receiver, then the 75  $\mu\text{m}$  sieve followed by the 425  $\mu\text{m}$  sieve.
- e) Pour the sample onto the 425  $\mu\text{m}$  sieve, cover with the top lid, and place entire assembly on the sieve shaker.
- f) Secure the assembled sieves on the sieve shaker with the tie-down apparatus.
- g) Set the timer for 5 min and activate and switch on the shaker.
- h) After 5 min  $\pm 6$  s, stop the shaker, remove the tie-downs, and remove the top lid.
- i) Place the top lid on the balance and “tare” the lid. This lid supports each sieve when weighed.
- j) Carefully separate the 425  $\mu\text{m}$  sieve from the assembled sieves and place on the tared lid on the balance. Record the mass of the sieve and sample retained, as  $m_4$ . Remove the 425  $\mu\text{m}$  sieve.
- k) Carefully separate the 75  $\mu\text{m}$  sieve from the bottom receiver and set aside.
- l) Remove the lid from the balance and “zero” the balance. Weigh the bottom receiver and its sample contents. Record the mass as  $m_7$ .

### 18.5.3 Calculation—Particle Size of a Xanthan Gum Sample

**18.5.3.1** The mass fraction of xanthan gum sample passing through a 425  $\mu\text{m}$  sieve,  $w_{425}$ , expressed in percent, can be calculated by subtracting the percent residue from 100 % or by using Equation (45):

$$w_{425} = 100 \left[ 1 - \frac{(m_4 - m_5)}{m} \right] \quad (45)$$

where

$m_4$  is the mass of 425  $\mu\text{m}$  sieve and sample retained, expressed in grams;

$m_5$  is the mass of the 425  $\mu\text{m}$  sieve, expressed in grams;

$m$  is the mass of the sample, expressed in grams.

**18.5.3.2** The mass fraction of xanthan gum sample passing through a 75  $\mu\text{m}$  sieve,  $w_{75}$ , expressed in percent, can be calculated from the percentage residue remaining in the bottom receiver, as given in Equation (46):

$$w_{75} = 100 \left[ \frac{m_7 - m_6}{m} \right] \quad (46)$$

where

$m_7$  is the mass of the bottom receiver and sample retained, expressed in grams;

$m_6$  is the mass of the bottom receiver, expressed in grams;

$m$  is the mass of the sample, expressed in grams.



## 18.6 Reagents and Apparatus—Measuring the Viscosity of a Xanthan Gum Solution

**18.6.1** Chemicals shall meet ACS or international equivalent reagent grade.

**18.6.2** **Sea salt**, ASTM D1141 or equivalent.

**18.6.3** **Deionized** or **distilled water**.

**18.6.4** **Sodium hydroxide** (CAS No. 1310-73-2) solution, 0.1 mol/L (0.1 N).

**18.6.5** **Hydrochloric acid** (CAS No. 7646-01-0) solution, 0.1 mol/L (0.1 N).

**18.6.6** **Defoamer**, octanol (CAS No. 111-87-5) or equivalent.

**Warning**—Octanol has a hazard rating of 2, is mildly toxic when ingested, and is a skin irritant.

**18.6.7** **Thermometer**, accurate to  $\pm 0.5\text{ }^{\circ}\text{C}$  ( $\pm 1.0\text{ }^{\circ}\text{F}$ ) over the temperature range specified in the procedure.

**18.6.8** **Balance**, accuracy of  $\pm 0.001\text{ g}$ .

**18.6.9** **Mixer**, each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**18.6.10** **Mixing container**, approximate dimensions: depth, 180 mm (7.0 in.);  $d$  top, 97 mm (3.8 in.);  $d$  bottom, 70 mm (2.75 in.).

**18.6.11** **Spatula** and **powder spatula** (recommended).

**18.6.12** **Viscometer**, motor driven, direct indicating, in accordance with API 13B-1 with an R1-B1 configuration and equipped with a F 0.2 torsion spring. See 18.11 for calibration.

**NOTE** F0.2 spring: torsional stiffness 77.2 dyne•centimeters per degree of deflection, shear stress constant equals to 0.102 pascals per degree of deflection (0.213 lbf/100•ft<sup>2</sup> per degree of deflection).

**18.6.13** **Beakers**, 400 mL, borosilicate glass, tall form; one, 2000 mL.

**18.6.14** **Overhead stirrer**, capable of rotating at less than 1000 r/min.

**18.6.15** **Water bath**, maintained at  $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  ( $77\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$ ).

**18.6.16** **Timer**, digital or mechanical, see 5.2.9 and 5.3.9.

**18.6.17** **pH meter** with **pH electrode**, accuracy of 0.01 pH.

## 18.7 Procedure—Preparation of the Synthetic Seawater

Synthetic seawater shall be prepared as per the following procedure.

- a) For a total volume of 1000 mL, weigh out  $41.95\text{ g} \pm 0.01\text{ g}$  of sea salt into the 2000 mL beaker.
- b) Add  $983.0\text{ g} \pm 0.1\text{ g}$  of deionized or distilled water.
- c) Mix for 15 min  $\pm 1$  min with the overhead stirrer set at a low speed.
- d) Measure the pH and adjust to 8.2 to 8.5 with 0.1 mol/L (0.1 N) sodium hydroxide or hydrochloric acid.

## 18.8 Procedure—Preparation of the Xanthan Gum Polymer Solution

Solution of xanthan gum polymer shall be prepared as per the following procedure.

- a) Prepare a solution of xanthan gum polymer by adding  $1.0 \text{ g} \pm 0.001 \text{ g}$  of polymer to  $358 \text{ g} \pm 3 \text{ g}$  of synthetic seawater at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The xanthan gum should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a laboratory powder spatula.

NOTE This is equivalent to  $2.85 \text{ g/L} \pm 0.01 \text{ g/L}$ .

- b) After stirring  $5 \text{ min} \pm 6 \text{ s}$ , remove the mixing container from the mixer and scrape its sides with the spatula to dislodge any particles adhering to the container walls. Be sure all particles clinging to the spatula are incorporated into the suspension.
- c) Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any particles clinging to the container walls at 10 min intervals. Total stirring time after adding the xanthan gum polymer shall equal  $30 \text{ min} \pm 1 \text{ min}$ .
- d) Remove the mixing container and add 2 to 3 drops of defoamer to the fluid. Stir 10 s to 15 s with the spatula to assist in the removal of entrapped air or foam.
- e) Place the mixing container in chilled water bath for not longer than 30 min to lower the fluid temperature to  $77^\circ\text{F} \pm 2^\circ\text{F}$  ( $25^\circ\text{C} \pm 1^\circ\text{C}$ ).

NOTE To achieve a more uniform temperature adjustment, immerse a thermometer in the fluid while stirring with the spatula to assure that the thermometer is not in contact with the mixing container.

## 18.9 Procedure—Measuring the Viscosity of a Xanthan Gum Solution

To measure viscosity of a xanthan gum polymer solution, the following procedure shall be applied.

- a) Pour the solution into the viscometer cup provided with the direct-indicating viscometer or the 400 mL tall-form beaker to about the 250 mL level. The solution level shall be exactly equal to the inscribed line on the direct-indicating viscometer rotor sleeve.
- b) The dial reading at the 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value at 600 r/min is reached. The 600 r/min dial reading shall be taken at a solution test temperature of  $25^\circ\text{C} \pm 1^\circ\text{C}$  ( $77^\circ\text{F} \pm 2^\circ\text{F}$ ). Record  $R_{0.2-600}$ , the 600 r/min dial reading.
- c) Continue to test the solution at 300 r/min, 200 r/min, 100 r/min, 6 r/min, and 3 r/min, waiting for the dial to reach a constant value at each rotational velocity. These subsequent readings shall be taken at a solution test temperature of  $25^\circ\text{C} \pm 1^\circ\text{C}$  ( $77^\circ\text{F} \pm 2^\circ\text{F}$ ). Record  $R_{0.2-300}$ ,  $R_{0.2-200}$ ,  $R_{0.2-100}$ ,  $R_{0.2-6}$ , and  $R_{0.2-3}$ , respectively, the 300 r/min, 200 r/min, 100 r/min, 6 r/min, and 3 r/min dial readings.

## 18.10 Measuring Low-shear-rate Viscosity of a Xanthan Gum Solution

### 18.10.1 Principle

**18.10.1.1** This procedure shall follow within 5 min of the test viscosity on the direct-reading viscometer, see 18.9.

**18.10.1.2** The suspending capabilities of xanthan gum are determined by measuring viscosity at low-shear rates at less than  $1.0 \text{ s}^{-1}$ , with a digital viscometer.

### 18.10.2 Apparatus—Measuring Low-shear-rate Viscosity of a Xanthan Gum Solution

**18.10.2.1 Digital viscometer**, with capable viscosity range of 15 mPa•s (cP) to 20,000 mPa•s (cP) and equipped with a cylindrical spindle having approximate dimensions: cylinder diameter, 18.84 mm (0.7418 in.); cylinder height, 65.1 mm (2.563 in.); immersion depth, 80.97 mm (3.188 in.).

**18.10.2.2 Beaker**, tall form, 400 mL, borosilicate glass.

**18.10.2.3 Lab jack**, adjustable, with 15 cm × 15 cm (6 in. × 6 in.) stage.

**18.10.2.4 Thermometer**, accurate to  $\pm 0.5\text{ }^{\circ}\text{C}$  ( $\pm 1.0\text{ }^{\circ}\text{F}$ ) over the temperature range specified in the procedure.

### 18.10.3 Procedure—Measuring Low-shear-rate Viscosity of a Xanthan Gum Solution

To measure low-shear-rate viscosity for a xanthan gum solution, the following procedure shall be applied.

- Pour the xanthan gum solution (18.8) into the beaker. Make certain that the temperature of the solution is  $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  ( $77\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$ ).
- Place the beaker on the lab jack and raise the stage so that the cylindrical spindle is immersed in the fluid to the point where the fluid level is equal to the inscribed line on the spindle shaft.
- Set the viscometer speed to 1.5 r/min and wait 30 s. Record the reading as  $R_{1.5}$ .

## 18.11 Calibration of Direct-indicating Viscometer (R1-B1 Geometry, F0.2 Spring)

### 18.11.1 Apparatus

**18.11.1.1 Viscometer**, motor driven, direct indicating, in accordance with API 13B-1, with an R1-B1 configuration and equipped with F0.2 torsion spring.

**18.11.1.2 Calibration fluid**, 200 mPa•s (cP), with accompanying chart showing viscosity versus temperature data.

**18.11.1.3 Thermometer**, digital, precision grade, with accuracy of  $[\pm 0.1\text{ }^{\circ}\text{C} (\pm 0.2\text{ }^{\circ}\text{F})]$ .

**18.11.1.4 Beaker**, tall form, 400 mL, borosilicate glass.

### 18.11.2 Procedure—Calibrating Direct-indicating Viscometer (R1-B1 Geometry, F0.2 Spring)

To calibrate a direct-indicating viscometer, the following procedure shall be applied.

- Clean and dry the viscometer bob and rotor.
- Pour approximately 250 mL of the calibration fluid into the 400 mL beaker.
- Place the beaker on the viscometer stage and raise the stage to the point where fluid level is equal to the inscribed line on the rotor sleeve.
- Place the thermometer into the fluid.
- Set the viscometer to 6 r/min and switch on power. Allow the viscometer to run for approximately 3 min to allow the fluid to stabilize to room temperature within a thermometer reading of  $\pm 0.1\text{ }^{\circ}\text{C}$  ( $\pm 0.2\text{ }^{\circ}\text{F}$ ). Record the dial reading  $R_{0.2-6}$ , with 0.5 units and temperature reading.

- f) Set the viscometer to 3 r/min and wait 2 min while the dial stabilizes. Record the dial reading  $R_{0.2-3}$ , within 0.5 units.

NOTE Excessive fluctuations in dial readings (greater than 1.0) can indicate the need to replace the viscometer bearings.

- g) Compare  $R_{0.2-6}$  and  $R_{0.2-3}$ , the 6 r/min and 3 r/min readings with the calibration oil viscosity at the recorded temperature using the supplied calibration fluid chart.

### 18.11.3 Calculation—Viscometer Accuracy

The accuracy of the viscometer readings is calculated as given in Equations (47) and (48) and shall equal  $\pm 15$  cP:

$$(R_{0.2-6} \times 10) - \eta_{oil} = \pm 15 \text{ mPa}\cdot\text{s (cP)} \quad (47)$$

$$(R_{0.2-3} \times 20) - \eta_{oil} = \pm 15 \text{ mPa}\cdot\text{s (cP)} \quad (48)$$

where

$\eta_{oil}$  is the viscosity of the calibration fluid at the measured temperature, expressed in millipascal-seconds (centipoises);

$R_{0.2-6}$  is the oil viscometer reading at 6 r/min (spring F0.2), expressed in degrees of deflection;

$R_{0.2-3}$  is the oil viscometer reading at 3 r/min (spring F0.2), expressed in degrees of deflection.

If the viscometer readings are beyond the limits, adjust torsion spring as per manufacturer's procedure or return the viscometer to the manufacturer for calibration.

## 19 OCMA-grade Bentonite

### 19.1 Principle

**19.1.1** OCMA-grade bentonite is a naturally occurring clay containing the clay minerals of smectite, which, by nature of its source, cannot meet all aspects of Clause 9. This bentonite may have been treated with soda ash, polymer, or other chemicals to improve suspension property performance. It can also contain quartz, mica, feldspar, and calcite, and other trace minerals.

**19.1.2** OCMA-grade bentonite shall be deemed to meet the requirements of this standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 18, represents the product produced, and is controlled by the manufacturer. This test procedure is to be calibrated using reference (calibration) bentonite at least once per 40 tests, as described in 5.3.11.

**Table 18—OCMA-grade Bentonite Physical Specifications**

Requirement	Standard
Suspension properties:	
Viscometer dial reading at 600 r/min	minimum 30
Yield point-plastic viscosity ratio	maximum 6 lbf/(100ft <sup>2</sup> •CP)
Filtrate volume	maximum 16.0 mL
Residue of diameter greater than 75 $\mu\text{m}$	maximum mass fraction 2.5 %

## 19.2 Reagents and Apparatus—Measuring Properties of an OCMA-grade Bentonite Suspension

- 19.2.1 **Thermometer**, accurate to  $\pm 0.5$  °C ( $\pm 1.0$  °F) over the range specified in this procedure.
- 19.2.2 **Balance**, with an accuracy of 0.01 g.
- 19.2.3 **Mixer** having a spindle fitted with a single sine-wave impeller approximately 1 in. (25 mm) in diameter, mounted flash side up.
- 19.2.4 **Mixing container**, of approximate dimensions: depth, 180 mm (7.1 in.);  $d$  top, 97 mm ( $3 \frac{5}{8}$  in.);  $d$  bottom, 70 mm (2.75 in.).
- 19.2.5 **Spatula**.
- 19.2.6 **Filter press**, low-pressure/low-temperature, in accordance with API R13B-1.
- 19.2.7 **Filter paper**, qualitative, hardened low ash, slow filtration rate, grade 50 in accordance with API 13B-1 (see 7.5.13)
- 19.2.8 **Container**, glass or plastic, at least 50 mL, to capture 7.5 min filtrate.
- 19.2.9 **Graduated cylinders**, two, 500 mL  $\pm 5$  mL (TC) and 10 mL  $\pm 0.1$  mL (TC).
- 19.2.10 Deionized or distilled water.
- 19.2.11 **Storage container**, with lid, capacity about 500 mL.
- 19.2.12 **Timers**, two, digital or mechanical (see 5.2.9 and 5.3.9).

## 19.3 Procedure—Measuring Rheological Properties of an OCMA-grade Bentonite Suspension

To measure rheological properties of a bentonite suspension, the following procedure shall be applied.

- a) Prepare a suspension of the OCMA-grade bentonite in the mixing container. Add 22.50 g  $\pm 0.01$  g of bentonite/clay (as received) to 350 mL  $\pm 5$  mL of deionized water while stirring on the mixer.
- b) After stirring 5 min  $\pm 6$  s, remove the container from the mixer and scrape its sides with the spatula to dislodge any bentonite adhering to the container walls. Be sure that all bentonite clinging to the spatula is incorporated into the suspension.
- c) Replace the mixing container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min  $\pm 1$  min.
- d) Age the bentonite suspension at least 16 h in a sealed or covered storage container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.
- e) After aging the bentonite suspension, shake well and then pour the suspension into the mixing container. Stir the suspension on the mixer for 5 min  $\pm 6$  s.
- f) Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial readings at 600 r/min,  $R_{600}$ , and 300 r/min,  $R_{300}$ , rotor speed settings of the viscometer shall be recorded when a constant value for each rotational velocity is reached. Readings shall be taken at a suspension test temperature of 25 °C  $\pm 1$  °C (77 °F  $\pm 2$  °F).

## 19.4 Calculation—Rheological Properties of an OCMA-grade Bentonite Suspension

Calculate the plastic viscosity,  $\eta_{PV}$ , expressed in centipoises, according to Equation (49), the yield point,  $Y_P$ , in pounds-force per 100 ft<sup>2</sup>, according to Equation (50), and the yield point-plastic viscosity ratio,  $b$ , expressed in lbf/(100 ft<sup>2</sup>•cP), according to Equation (51):

$$\eta_{PV} = R_{600} - R_{300} \quad (49)$$

$$Y_P = R_{300} - \eta_{PV} \quad (50)$$

$$b = \frac{Y_P}{\eta_{PV}} \quad (51)$$

where

$R_{600}$  is the viscometer dial reading at 600 r/min;

$R_{300}$  is the viscometer dial reading at 300 r/min.

Record the calculated values for plastic viscosity, yield point, and yield point/plastic viscosity ratio.

NOTE 1 Plastic viscosity in SI units, millipascal•seconds are numerically equivalent to centipoises. Yield point values reported in lbf/100 ft<sup>2</sup> units are not exact. A direct conversion from lbf/100 ft<sup>2</sup> to pascals would be to multiply the value 0.479. However, each 1 degree of deflection is exactly equal to 0.511 Pa in SI units, so the more exact conversion is to multiply the result from Equation (50) by 0.511 to obtain pascals.

NOTE 2 Abbreviations for plastic viscosity and yield point in the industry PV and YP are commonly used. Then, the ratio yield point-plastic viscosity is given as YP/PV. When expressed in SI units, this ratio is numerically equivalent to reciprocal milliseconds or 1000 s<sup>-1</sup>.

## 19.5 Procedure—Measuring the Filtrate Volume of an OCMA-grade Bentonite Suspension

To measure the filtrate volume of a bentonite suspension, the following procedure shall be applied.

- Recombine all of the suspension, as prepared and tested in 19.3, and stir in the mixing container for 1 min  $\pm$  6 s on the mixer. Adjust the suspension temperature to 25 °C  $\pm$  1 °C (77 °F  $\pm$  2 °F).
- Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn in accordance with API 13 B-1. Pour the suspension into the filter press cell to within about 13 mm (0.5 in.) of the top of the cell. Complete assembly of the filter press cell using one sheet of filter paper. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.
- Set one timer for 7.5 min and the second timer for 30 min. Start both timers and adjust the pressure on the cell to 700 kPa  $\pm$  30 kPa (100 psi  $\pm$  5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen, or helium.
- At 7.5 min  $\pm$  6 s on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 mL graduated cylinder under the drain tube and continue collecting filtrate until the end of the second timer set at 30 min. At 30 min  $\pm$  6 s, remove the graduated cylinder and record the volume of filtrate collected,  $V_C$ .

## 19.6 Calculation—Filtrate Volume of an OCMA-grade Bentonite Suspension

Calculate the filtrate volume,  $V$ , in milliliters, of the OCMA-grade bentonite suspension as given in Equation (52):

$$V = 2 V_c \quad (52)$$

where

$V_c$  is the filtrate volume, expressed in milliliters, collected between 7.5 min and 30 min.

Record the calculated filtrate volume.

## 19.7 Reagents and Apparatus—Measuring OCMA-grade Bentonite Residue of Greater than 75 $\mu\text{m}$

**19.7.1 Sodium hexametaphosphate** (CAS No. 10124-56-8), ACS reagent grade. Anhydrous powder or aqueous solution 10 %  $\pm 0.5$  % by mass.

**19.7.2 Oven**, regulated to 105 °C  $\pm 2.5$  °C (220 °F  $\pm 5$  °F).

**19.7.3 Balance**, with an accuracy of  $\pm 0.01$  g.

**19.7.4 Mixer**, having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in.) in diameter, mounted flash side up.

**19.7.5 Mixing container**, approximate dimensions: depth, 180 mm (7.1 in.);  $d$  top, 97 mm (3  $\frac{5}{8}$  in.);  $d$  bottom, 70 mm (2.75 in.).

**19.7.6 Sieve**, 75  $\mu\text{m}$  (0.0029 in., US No.200), in accordance with ASTM E11 or ASTM E161, approximate dimensions: 76 mm (3.0 in.) in diameter and 69 mm (2.75 in.) from top of frame to wire cloth or electroformed sieve.

**19.7.7 Spray Nozzle**, with 6.35 mm ( $\frac{1}{4}$  in.) inlet connection, rated for 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.<sup>2</sup>) with approximately 45° spray angle attached to a water line with a 90° elbow.

**19.7.8 Water-pressure regulator**, capable of regulation to (70 kPa  $\pm 6$  kPa (10 lbf/in.<sup>2</sup>  $\pm 1$  lbf/in.<sup>2</sup>)).

**19.7.9** Evaporating Dish.

**19.7.10** Wash Bottle.

## 19.8 Procedure—Measuring OCMA-grade Bentonite Residue of Greater than 75 $\mu\text{m}$

The following procedure shall be followed when measuring barite residue of greater than 75  $\mu\text{m}$ .

- a) Weigh 10.00 g  $\pm 0.01$  g of bentonite.
- b) Add the weighed bentonite sample to approximately 350 mL of water containing 0.20 g  $\pm 0.05$  g of sodium hexametaphosphate in the mixing container while stirring on the mixer. Sodium hexametaphosphate dispersant may be added as 0.20 g  $\pm 0.05$  g of sodium hexametaphosphate powder or by adding 2.0 mL  $\pm 0.1$  mL of sodium hexametaphosphate 10 %  $\pm 0.5$  % by mass solution.
- c) Stir suspension on the mixer for 30 min  $\pm 10$  s.
- d) Transfer the sample to the sieve. Use the wash bottle to transfer all material from the mixing container to the sieve. Wash the material on the sieve with water controlled to 70 kPa  $\pm 6$  kPa (10 psi  $\pm 1$  lbf/in.<sup>2</sup>) from the spray nozzle for 2 min  $\pm 15$  s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.
- e) Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

f) Dry the residue in the oven to a constant mass. Record the residue mass,  $m_2$ , and total drying time.

### 19.9 Calculation—OCMA-grade Bentonite Residue of Greater than 75 $\mu\text{m}$

Calculate the mass fraction residue of OCMA-grade bentonite particles of greater than 75  $\mu\text{m}$ ,  $w_1$ , in percent, as given in Equation (53):

$$w_1 = 100 \left( \frac{m_2}{m} \right) \quad (53)$$

where

$m$  is the sample mass, in grams;

$m_2$  is the residue mass, in grams.

Record the calculated value.



## **Annex A**

### **(informative)**

## **API Monogram Program**

### **Use of the API Monogram by Licensees**

#### **A.1 Scope**

The API Monogram® is a registered certification mark owned by the American Petroleum Institute (API) and authorized for licensing by the API Board of Directors. Through the API Monogram Program, API licenses product manufacturers to apply the API Monogram to new products which comply with product specifications and have been manufactured under a quality management system that meets the requirements of API Spec Q1. API maintains a complete, searchable list of all Monogram licensees on the API Composite List website (<http://compositelist.api.org>).

The application of the API Monogram and license number on products constitutes a representation and warranty by the licensee to API and to purchasers of the products that, as of the date indicated, the products were manufactured under a quality management system conforming to the requirements of API Spec Q1 and that the product conforms in every detail with the applicable standard(s) or product specification(s). API Monogram Program licenses are issued only after on-site audits have verified that an organization has implemented and continually maintained a quality management system that meets the requirements of API Spec Q1 and that the resulting products satisfy the requirements of the applicable API product specification(s) and/or standard(s). Although any manufacturer may claim that its products meet API product requirements without monogramming them, only manufacturers with a license from API can apply the API Monogram to their products.

Together with the requirements of the API Monogram license agreement, this annex establishes the requirements for those organizations who wish to voluntarily obtain an API license to provide API monogrammed products that satisfy the requirements of the applicable API product specification(s) and/or standard(s) and API Monogram Program requirements.

For information on becoming an API Monogram Licensee, please contact API, Certification Programs, 200 Massachusetts Ave NW, Suite 1100, Washington, DC 20001 at [Certification@api.org](mailto:Certification@api.org).

#### **A.2 Normative References**

For Licensees under the Monogram Program, the latest version of this document shall be used. The requirements identified therein are mandatory.

#### **A.3 Terms and Definitions**

For purposes of this annex, the following terms and definitions apply.

##### **A.3.1**

##### **API monogrammmable product**

Product that has been newly manufactured by an API Licensee utilizing a fully implemented API Spec Q1 compliant quality management system and that meets all the API-specified requirements of the applicable API product specification(s) and/or standard(s).

##### **A.3.2**

##### **API product specification**

Prescribed set of rules, conditions, or requirements attributed to a specified product that address the definition of terms; classification of components; delineation of procedures; specified dimensions; manufacturing

criteria; material requirements; performance testing; design of activities; and the measurement of quality and quantity with respect to materials, products, processes, services, and/or practices.

### **A.3.3**

#### **API-specified requirements**

Requirements, including performance and Licensee-specified requirements, set forth in API Spec Q1 and the applicable API product specification(s) and/or standard(s).

NOTE Licensee-specified requirements include those activities necessary to satisfy API-specified requirements.

### **A.3.4**

#### **design package**

Records and documents required to provide evidence that the applicable product has been designed in accordance with API Spec Q1 and the requirements of the applicable product specification(s) and/or standard(s).

### **A.3.5**

#### **licensee**

Organization that has successfully completed the application and audit process and has been issued a license by API to use the API Monogram Mark.

## **A.4 Quality Management System Requirements**

An organization applying the API Monogram to products shall develop, maintain, and operate at all times a quality management system conforming to API Specification Q1.

## **A.5 Control of the Application and Removal of the API Monogram**

Each licensee shall control the application and removal of the API Monogram in accordance with the following.

- a) Products that do not conform to API specified requirements shall not bear the API Monogram.
- b) Each licensee shall develop and maintain an API Monogram marking procedure that documents the marking/monogramming requirements specified by this annex and any applicable API product specification(s) and/or standard(s). The marking procedure shall:
  - 1) define the authority responsible for application and removal of the API Monogram and license number;
  - 2) define the method(s) used to apply the Monogram and license number;
  - 3) identify the location on the product where the API Monogram and license number are to be applied;
  - 4) require the application of the date of manufacture of the product in conjunction with the use of the API Monogram and license number;
  - 5) require that the date of manufacture, at a minimum, be two digits representing the month and two digits representing the year (e.g. 05-12 for May 2012) unless otherwise stipulated in the applicable API product specification(s) or standard(s); and
  - 6) define the application of all other required API product specification(s) and/or standard(s) marking requirements.
- c) Only an API licensee shall apply the API Monogram and its designated license number to API monogramable products.

- d) The API Monogram and license number, when issued, are site-specific and subsequently the API Monogram shall only be applied at that site specific licensed facility location.
- e) The API Monogram may be applied at any time appropriate during the production process but shall be removed in accordance with the licensee's API Monogram marking procedure if the product is subsequently found to be out of conformance with any of the requirements of the applicable API product specification(s) and/or standard(s) and API Monogram Program.

For certain manufacturing processes or types of products, alternative API Monogram marking procedures may be acceptable. Requirements for alternative API Monogram marking are detailed in the API Alternative Marking Agreement (AMA), which is available on the API Monogram Program website at: [https://www.api.org/~media/Files/Certification/Monogram-APIQR/0\\_API-Monogram-APIQR/Resources/API-Monogram-Alt-Marking-Agreement\\_Rev-8\\_FM-011\\_Modified-20180601.pdf](https://www.api.org/~media/Files/Certification/Monogram-APIQR/0_API-Monogram-APIQR/Resources/API-Monogram-Alt-Marking-Agreement_Rev-8_FM-011_Modified-20180601.pdf).

## A.6 Design Package Requirements

Each licensee and/or applicant for licensing shall maintain a current design package for all of the applicable products that fall under the scope of each Monogram license. The design package information shall provide objective evidence that the product design meets the requirements of the applicable and most current API product specification(s) and/or standard(s). The design package(s) shall be made available during API audits of the facility.

In specific instances, the exclusion of design activities is allowed under the Monogram Program, as detailed in Advisory # 6, available on the API Monogram Program website at <https://www.api.org/products-and-services/api-monogram-and-apiqr/advisories-updates>.

## A.7 Manufacturing Capability

The API Monogram Program is designed to identify facilities that have demonstrated the ability to manufacture equipment that conforms to API specifications and/or standards. API may refuse initial licensing or suspend current licensing based on a facility's level of manufacturing capability. If API determines that additional review is warranted, API may perform additional audits (at the organization's expense) of any primary subcontractors to ensure their compliance with applicable specifications.

Facilities with capabilities that are limited to the processes or activities defined below do not meet the manufacturing capability requirements to produce new products, and therefore, shall not be licensed or be the basis for licensing under the API Monogram Program:

- capabilities that are limited to performing final inspection and testing of the product, except for testing agencies as specified in API Spec 14A and/or API Spec 6AV;
- buying, selling, and/or distributing finished products and materials;
- design and development activities;
- tearing down and/or re-assembling of products/components; and,
- repairing or remanufacturing of existing, used, worn, or damaged products.

In all instances where requirements for manufacturing or manufacturing facilities are explicitly identified within the API product specification, those requirements shall take precedence over this advisory.

## **A.8 Product Marking Requirements**

### **A.8.1 General**

These marking requirements shall apply only to those API Licensees wishing to mark applicable products in conjunction with the requirements of the API Monogram Program.

### **A.8.2 Product Specification Identification**

Manufacturers shall mark products as specified by the applicable API specifications or standards. Marking shall include reference to the applicable API specification and/or standard. Unless otherwise specified, reference to the API specifications and/or standards shall be, as a minimum, “API [Document Number]” (e.g. API 6A, or API 600). Unless otherwise specified, when space allows, the marking may include use of “Spec” or “Std,” as applicable (e.g. API Spec 6A or API Std 600).

### **A.8.3 Units**

Products shall be marked with units as specified in the API specification and/or standard. If not specified, equipment shall be marked with U.S. customary (USC) units. Use of dual units [USC units and metric (SI) units] may be acceptable, if such units are allowed by the applicable product specification and/or standard.

### **A.8.4 Nameplates**

Nameplates, when applicable, shall be made of a corrosion-resistant material unless otherwise specified by the API specification and/or standard. Nameplate shall be located as specified by the API specification and/or standard. If the location is not specified, then the licensee shall develop and maintain a procedure detailing the location to which the nameplate shall be applied. Nameplates may be attached at any time during the manufacturing process.

The API Monogram and license number shall be marked on the nameplate, in addition to the other product marking requirements specified by the applicable product specification and/or standard.

### **A.8.5 License Number**

The API Monogram license number shall not be used unless it is marked in conjunction with the API Monogram. The license number shall be used in close proximity to the API Monogram.

## **A.9 API Monogram Program: Nonconformance Reporting**

API solicits information on products that are found to be nonconforming with API specified requirements, as well as field failures (or malfunctions), which are judged to be caused by either specification and/or standard deficiencies or nonconformities against API specified requirements. Customers are requested to report to API all problems with API monogrammed products. A nonconformance may be reported using the API Nonconformance Reporting System available at <http://ncr.api.org/ncr.aspx>.

## **Annex B** **(informative)**

### **Test Precision**

#### **B.1 Description**

##### **B.1.1 General**

Precision statements for this standard's tests are presented in this annex for the following materials:

- a) barite;
- b) hematite;
- c) bentonite;
- d) non-treated bentonite;
- e) OCMA-grade bentonite;
- f) attapulgite;
- g) sepiolite;
- h) CMC-LVT;
- i) CMC-HVT;
- j) starch.

The precision values in Annex B do not represent what can be expected from any random laboratory. Rather, they reflect the precision obtained by personnel who are trained and experienced in running the tests using equipment that is in good working order and in calibration.

Any laboratory that wishes to test materials for conformance to this standard shall demonstrate at least the degree of test precision given in this annex. Calibration barite and test calibration bentonite are available (see Annex D) for use by laboratories to determine their test precision.

##### **B.1.2 Considerations for Manufacturers and Users**

The precision data in Tables B.1 through B.11 reflect the fact that the standard test methods are subject to normal test variability. This variability can lead to conflicts between a manufacturer and a user based on their respective test results on the same sample of material.

The precision data indicate the maximum expected difference between two test results on the same sample, both within and between laboratories, at the 95 % confidence level.

#### **B.2 Basis**

The limits in Tables B.1, B.3, B.4, B.5, B.6, B.9, B.10, and B.11 were determined from interlaboratory studies conducted from 1988 to 1991. The data analysis is given in API Reports 88-30, 89-30, and 90-30. The studies were designed and conducted by an independent consulting laboratory under contract to the API. The project

was overseen by a Technical Advisory Committee of API Committee C3/SC13. The limits in Tables B.2, B.7, and B.8 were determined from interlaboratory studies conducted in 1992.

The experimental design and data analysis is patterned after ASTM E691. Extra effort was made to assure that each participating laboratory used trained and experienced personnel, maintained equipment in calibration, and followed the procedures in every detail.

### B.3 Definitions

For B.4, the procedures used for the statistical evaluation of sample differences shall be in accordance with ASTM E177.

The term “repeatability limit” is used as described in ASTM E177, i.e. the maximum expected difference between two test results on samples of the same material by the same laboratory at the 95 % confidence level.

The term “reproducibility limit” is used as described in ASTM E177, i.e. the maximum expected difference between test results by two laboratories on samples of the same material at the 95 % confidence level.

The respective standard deviations at the 95 % confidence level can be obtained by dividing the limits by 2.8.

### B.4 Test Precision Tables

**Table B.1—Test Precision for Barite**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Density	0.022 g/mL	0.030 g/mL
Water soluble alkaline earth metals, as calcium	9.2 mg/kg	23.0 mg/kg
Particles of diameter less than 6 $\mu\text{m}$	1.7 %	2.5 %

The values in Table B.1 were determined from a study conducted in 1988 and 1989. Each of the 12 laboratories tested each of the 3 samples once each on each of 3 separate days. Each test result is the result of a single determination.

**Table B.2—Test Precision for Hematite**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Density	0.026 g/mL	0.050 g/mL
Water soluble alkaline earth metals, as calcium	9.2 mg/kg	23.0 mg/kg
Particles of diameter less than 6 $\mu\text{m}$	0.9 %	3.4 %

The values in Table B.2 were determined from a study conducted in 1992. Each of the 7 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each test result is the result of a single determination.

**Table B.3—Test Precision for Bentonite**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	2.9	6.3
Yield point-plastic viscosity ratio	0.16	0.25
Filtrate volume	0.8 mL	1.3 mL

The values in Table B.3 were determined from a study conducted in 1989. Each of the 8 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each test result is the result of a single determination.

**Table B.4—Test Precision for Non-treated Bentonite**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Dispersed plastic viscosity	2.1 cP (mPa•s)	3.1 cP (mPa•s)
Dispersed filtrate volume	0.9 mL	1.5 mL

The values in Table B.4 were determined from a study conducted in 1989. Each of the 8 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each test result is the result of a single determination.

**Table B.5—Test Precision for OCMA-grade Bentonite**

Test	Repeatability limit (intralab)	Reproducibility limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	4.9	9.3
Yield point-plastic viscosity ratio	1.15	1.16
Filtrate volume	0.8 mL	1.6 mL

The values in Table B.5 were determined from a study conducted in 1991. Each of seven laboratories tested each of two samples once each on each of three separate days. Each test result is the result of a single determination.

**Table B.6—Test Precision for Reference (Calibration) Bentonite**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	4.9	9.3
Yield point-plastic viscosity ratio	1.15	1.16
Filtrate volume	0.8 mL	1.6 mL

The values in Table B.6 were determined from a study conducted in 1991. Each of the 7 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each test result is the result of one determination.

**Table B.7—Test Precision for Attapulgate**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	3.2	6.8
Moisture mass fraction	1.5 %	2.1 %

The values in Table B.7 were determined from a study conducted in 1992. Each of the 5 laboratories tested each of the 2 samples once each on each of the 3 separate days for all properties except moisture content. Only 4 laboratories participated in the moisture test. Each test result is the result of a single determination.

**Table B.8—Test Precision for Sepiolite**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	4.5	5.6
Moisture mass fraction	1.6 %	1.6 %

The values in Table B.8 were determined from a study conducted in 1992. Each of the 4 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each test result is the result of single determination.

**Table B.9—Test Precision for CMC-LVT**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	2.6	5.1
Filtrate volume	0.9 mL	1.6 mL

The values in Table B.9 were determined from a study conducted in 1991. For the viscometer dial reading at 600 r/min test, each of the 8 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each viscometer 600 r/min test result was the result of a single determination. For the filtrate volume test, each of the 6 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each filtrate volume test result is an average of 2 determinations.



**Table B.10—Test Precision for CMC-HVT**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min		
— in distilled water	2.4	4.8
— in 40 g/L salt water	2.1	5.8
— in saturated salt water	3.0	5.6
Filtrate volume	0.6 mL	1.9 mL

The values in Table B.10 were determined from a study conducted in 1991. For the viscosity reading at 600 r/min tests, each of the 9 laboratories tested each of the 2 samples once each on each of the 3 separate days. Each viscometer result was the result of a single test. For the filtrate volume test, each of the 7 laboratories tested each of 2 samples once each on each of the 3 separate days. Each filtrate test result is an average of 2 determinations.

**Table B.11—Test Precision Starch**

Test	Repeatability Limit (intralab)	Reproducibility Limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min		
— in 40 g/L salt water	2.0	7.1
— in saturated salt water	1.5	4.9
Filtrate volume		
— in 40 g/L salt water	0.7 mL	2.3 mL
— in saturated salt water	0.6 mL	1.5 mL

The values in Table B.11 were determined from a study conducted in 1991. For the 40 g/L saltwater viscometer dial reading at 600 r/min test, each of the 8 laboratories tested each of the 2 samples once each on each of the 3 separate days. For the saturated saltwater tests, each of the 7 laboratories tested each of the 2 samples once each on each of the 3 separate days. For the 40 g/L saltwater filtrate volume test, each of the 7 laboratories tested each of the 2 samples once each on each of the 3 separate days. For the saturated saltwater tests, each test result is an average of 2 determinations.

## Annex C (informative)

### Examples of Calculations

*The following examples are merely for illustration purposes only. They are not to be considered exclusive or exhaustive in nature. API makes no warranties, express or implied, for reliance on or any omissions from the information contained in this document.*

#### C.1 Example Hydrometer Calibration Sheet (ASTM 151H)

NOTE 1 The temperature units for these calculations may be measured in either degrees Celsius or degrees Fahrenheit. However, all measurements and calculations must be made using consistent units. Temperature values in degrees Celsius are used for these examples.

NOTE 2 The calculations shown below use the equations or tables from this document and the intermediate calculated values as shown. The results shown below may differ slightly from a spreadsheet calculation where more exact values are carried throughout the calculations.

**Table C.1—Example Hydrometer Calibration Sheet**

Hydrometer Serial Number	Average Temperature $\theta$ °C	Average Relative Density Reading $R$	Correction Curve Slope $M_c$ °C <sup>-1</sup>	Correction Curve Initial Value at $\theta_1$ $B_c$
Xxxxx	$\theta_1 = 22.4$	$R_1 = 1.006$	0.408	15.14
	$\theta_2 = 32.2$	$R_2 = 1.002$		

Certified by: \_\_\_\_\_

Date: \_\_\_\_\_

Using Equation (1) calculate the hydrometer correction line slope,  $M_c$ :

$$M_c = 1000 \frac{(R_1 - R_2)}{(\theta_2 - \theta_1)}$$

$$M_c = 1000 \frac{(1.006 - 1.002)}{(32.2 - 22.4)} = 0.408 \text{ °C}^{-1}$$

Using Equation (2), calculate the hydrometer correction line initial value at  $\theta_1$ ,  $B_c$ :

$$B_c = (M_c \times \theta_1) + [(R_1 - 1) \times 1000] = (0.408 \times 22.4) + [(1.006 - 1) \times 1000] = 15.14$$

## C.2 Barite—Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter

### C.2.1 Example of Datasheet

Hydrometer serial No. XXXXX:

- $M_C$ ,  $0.408\text{ }^\circ\text{C}^{-1}$ ;
- $B_C$ , 15.14;
- sample density,  $\rho$ , 4.30 g/mL;
- sample mass,  $m$ , 80.0 g.

**Table C.2—Example Hydrometer Datasheet (Barite)**

Time $T$ min	Temperature $\theta$ $^\circ\text{C}$	Hydrometer Relative Density Reading $R$	Water Viscosity $\eta$ mPa·s (cP)	Effective Depth $L$ cm	Equivalent Spherical Diameter $D_e$ $\mu\text{m}$	Cumulative % Finer $w_a$	% Finer <6 $\mu\text{m}$ $w_4$
10	26.0	1.028	0.8705	8.9	8.5	38.2	
20	26.0	1.021	0.8705	10.7	6.6 ( $D_1$ )	26.8 ( $w_2$ )	
30	26.0	1.017	0.8705	11.8	5.6 ( $D_2$ )	20.3 ( $w_3$ )	
40	26.0	1.014	0.8705	12.6	5.0	15.4	
							22.9
NOTE Temperature values in degrees Celsius are used for this example. Water viscosity value is from Table 3 for $\theta$ in $^\circ\text{C}$ ; it may also be calculated from Equation (12).							

**C.2.2** Sample constant,  $K_s$ , is from Table 5 or can be calculated, as shown below, from Equation (8):

$$K_s = 100 \frac{\rho}{m(\rho - 1)}$$

$$K_s = 100 \frac{4.30}{80 \times (4.30 - 1)} = 1.6288$$

**C.2.3** For each 10 min time interval, enter the time,  $t$ , the temperature,  $\theta$ , the uncorrected hydrometer reading,  $R$ , the water viscosity,  $\eta$ , and the effective depth,  $L$ . The effective depth,  $L$ , is from Table 4.

**C.2.4** For each 10 min time interval, calculate the equivalent spherical diameter using Equation (9). Calculation for the equivalent spherical diameter,  $D_e$ , for the 20 min data is:

$$D_e = 17.5 \sqrt{\frac{\eta L}{(\rho - 1) t}}$$

$$D_e = 17.5 \sqrt{\frac{0.8705 \times 10.7}{(4.30 - 1) \times 20}} = 6.6 \mu\text{m}$$

For each 10 min time interval, calculate the cumulative percent finer,  $w_a$ , from Equation (10). For the 20 min data:

$$w_a = K_s[(M_c \times \theta) - B_c + (R - 1) \times 1000]$$

$$w_a = 1.6288[(0.408 \times 26.0) - 15.14 + (1.021 - 1) \times 1000]$$

$$w_a = 1.6288 \times [10.608 - 15.14 + 21] = 26.8 \%$$

Once the first equivalent spherical diameter,  $D_e$ , immediately below  $6 \mu\text{m}$  is determined (at 30 min in Table C.2), use the cumulative percent finer as  $w_3$  and the equivalent spherical particle diameter as  $D_2$ . From the previous interval (20 min in Table C.2) where the data is immediately greater than  $6 \mu\text{m}$ , use the cumulative percent finer as  $w_2$  and the equivalent spherical particle diameter as  $D_1$ .

**C.2.5** Calculate the percent of particles less than  $6 \mu\text{m}$ ,  $w_4$ , from Equation (11):

$$w_4 = \left[ \left( \frac{w_2 - w_3}{D_1 - D_2} \right) \times (6 - D_2) \right] + w_3$$

For

$$w_2 = 26.8 \%$$

$$w_3 = 20.3 \%$$

$$D_1 = 6.6 \mu\text{m}$$

$$D_2 = 5.6 \mu\text{m}$$

$$w_4 = \left[ \left( \frac{26.8 - 20.3}{6.6 - 5.6} \right) \times (6 - 5.6) \right] + 20.3 = 22.9\%$$

### C.3 Hematite—Particles Less than 6 $\mu\text{m}$ in Equivalent Spherical Diameter

#### C.3.1 Example Datasheet

Hydrometer serial No. XXXXX:

- $M_C$ ,  $0.408\text{ }^\circ\text{C}^{-1}$ ;
- $B_C$ , 15.14;
- sample density,  $\rho$ , 5.05 g/mL;
- sample mass,  $m$ , 80.0 g.

**Table C.3—Example Hydrometer Datasheet (Hematite)**

Time $t$ min	Temperature $\theta$ $^\circ\text{C}$	Hydrometer Reading $R$	Water Viscosity $\eta$ mPa·s (cP)	Effective Depth $L$ cm	Equivalent Spherical Diameter $D_e$ $\mu\text{m}$	Cumulative % Finer $w_a$	% Finer <6 $\mu\text{m}$ $w_4$
10	25.5	1.013	0.8803	12.9	9.3	12.9	
20	25.0	1.010	0.8904	13.7	6.8 ( $D_1$ )	7.9 ( $w_2$ )	
30	25.0	1.008	0.8904	14.2	5.6 ( $D_2$ )	4.8 ( $w_3$ )	
							5.8

NOTE Temperature values in degrees Celsius are used for this example. Water viscosity value is from Table 3 for  $\theta$  in  $^\circ\text{C}$ ; it may also be calculated from Equation (12).

**C.3.2** Sample constant,  $K_S$ , is from Table 5 or can be calculated, as shown below, from Equation (8)

$$K_S = 100 \frac{\rho}{m(\rho - 1)}$$

$$K_S = 100 \frac{5.05}{80 \times (5.05 - 1)} = 1.5586$$

**C.3.3** For each 10 min time interval, enter the time,  $t$ , the temperature,  $\theta$ , the uncorrected hydrometer reading,  $R$ , the water viscosity,  $\eta$ , and the effective depth,  $L$ . The effective depth,  $L$ , is from Table 4.

**C.3.4** For each 10 min time interval, calculate the equivalent spherical diameter using Equation (9). Calculation for the equivalent spherical diameter,  $D_e$ , for 20 min data is:

$$D_e = 17.5 \sqrt{\frac{\eta L}{(\rho - 1) t}}$$

$$D_e = 17.5 \sqrt{\frac{0.8904 \times 13.7}{(5.05 - 1) \times 20}} = 6.8 \mu\text{m}$$

For each 10 min time interval, calculate the cumulative percent finer,  $w_a$ , from Equation (10). For the 20 min data:

$$w_a = K_s[(M_c \times \theta) - B_c + (R - 1) \times 1000]$$

$$w_a = 1.5586[(0.408 \times 25.0) - 15.14 + (1.010 - 1) \times 1000]$$

$$w_a = 1.5586 \times [10.2 - 15.14 + 10.0] = 7.9 \%$$

Once the first equivalent spherical diameter,  $D_e$ , immediately below 6  $\mu\text{m}$  is determined (at 30 min in Table C.3), use the cumulative percent finer as  $w_3$  and the equivalent spherical particle diameter as  $D_2$ . From the previous interval (20 min in Table C.3) where the data is immediately greater than 6  $\mu\text{m}$ , use the cumulative percent finer as  $w_2$  and the equivalent spherical particle diameter as  $D_1$ .

**C.3.5** Calculation the percent of particles less than 6  $\mu\text{m}$ ,  $w_4$ , from Equation (11):

$$w_4 = \left[ \left( \frac{w_2 - w_3}{D_1 - D_2} \right) \times (6 - D_2) \right] + w_3$$

For

$$w_2 = 7.9 \%$$

$$w_3 = 4.8 \%$$

$$D_1 = 6.8 \mu\text{m}$$

$$D_2 = 5.6 \mu\text{m}$$

$$w_4 = \left[ \left( \frac{7.9 - 4.8}{6.8 - 5.6} \right) \times (6 - 5.6) \right] + 4.8 = 5.8 \%$$

## **Annex D**

### **(informative)**

## **API Reference Material Program**

### **D.1 Overview**

**D.1.1** The API operates a Standard Reference Material program to qualify test calibration barite, reference (calibration) bentonite, and standard evaluation base clay (SEBC) for API 13A and API 13I testing. The intent of this program is to guarantee that disparate laboratories have access to identical testing material to minimize the differences between interlaboratory and intralaboratory testing and to satisfy the requirements of API 13A and the test methods in API 13I.

**D.1.2** For calibration requirements of test calibration materials and their required periodic use, refer to 5.2.11 and 5.3.11.

### **D.2 Custodian**

**D.2.1** The API contracts with a designated custodian, who is responsible for obtaining, coordinating round robin qualification testing, and packaging these reference materials. The custodian is responsible for ensuring a supply of uniform reference material for the industry.

**D.2.2** The custodian shall furnish a certificate of analysis and safety datasheet for each sample.

**D.2.3** The custodian shall clearly label each sample with the custodian's contact information, date packaged, and the API qualified material batch number.

### **D.3 Standard Reference Material Information and Qualification Criteria**

#### **D.3.1 General**

The following are the requirements for calibration materials necessary to comply with this standard.

#### **D.3.2 Test Calibration Barite**

Test calibration barite meets the Table 2 barite physical and chemical specifications for 4.2 g/mL barite using Section 7 testing methods. This material is sourced from a single supplier, pretested, blended, repackaged, and then qualified by round robin testing using at least 3 different labs operated by difference companies. Acceptance is at the discretion of API Subcommittee 13 Task Group 8 using a comparison of the current and previous round robin testing results and a comparison with the repeatability and reproducibility values listed in Table B.1.

#### **D.3.3 Reference (Calibration) Bentonite**

Reference (calibration) bentonite meets the Table 9 non-treated bentonite specifications using the Section 10 testing methods. This material is sourced from a single supplier, pretested, blended, repackaged, and then qualified by round robin testing using at least 3 different labs operated by difference companies. Acceptance is at the discretion of API Subcommittee 13 Task Group 8 using a comparison of the current and previous round robin testing results and a comparison with the repeatability and reproducibility values listed in Table B.4.

**NOTE** Reference (calibration) bentonite has previously been referred to as "reference bentonite" in previous editions of API 13I and "test calibration bentonite" in previous editions of API 13A.

### D.3.4 SEBC

API SEBC is a select ball clay, a sedimentary clay that is composed of both expandable and non-expandable clay particles and quartz particles. It contains highly disordered kaolinite, illite/mixed layer clays, and quartz. It is used to test the performance specifications of a number of materials in API 13A and to simulate “drill solids” in certain laboratory tests in API 13I.

The specification in Table D.1 is for the product currently sourced as SEBC. The data in Table D.2, Table D.3, and Table D.4 are informative only and do not represent actual values of any single lot of SEBC.

**Table D.1—Product Specification for SEBC**

Chemical Composition	Range	
	Minimum	Maximum
SiO <sub>2</sub> , mass fraction	52 %	56 %
Fe <sub>2</sub> O <sub>3</sub> , mass fraction	1.1 %	1.6 %
TiO <sub>2</sub> , mass fraction	0.9 %	1.4 %
K <sub>2</sub> O, mass fraction	2.8 %	3.4 %
Surface area (BET *)	36 m <sup>2</sup> /g	46 m <sup>2</sup> /g
* Brunauer, Emmett, and Teller gas adsorption measurement.		

**Table D.2—Typical Chemical Analysis for SEBC**

Typical Analysis Mass Fraction %	Value
SiO <sub>2</sub>	54
Al <sub>2</sub> O <sub>3</sub>	30
Fe <sub>2</sub> O <sub>3</sub>	1.4
TiO <sub>2</sub>	1.2
CaO	0.3
MgO	0.4
K <sub>2</sub> O	3.1
Na <sub>2</sub> O	0.5
Soluble salts	0.1
Loss on ignition	8.8



**Table D.3—Typical Particle Analysis for SEBC**

Typical Particle Size	Value %
>125 $\mu\text{m}$	0.1
>53 $\mu\text{m}$	0.3
>5 $\mu\text{m}$	96
<22 $\mu\text{m}$	88
<11 $\mu\text{m}$	79
<0.5 $\mu\text{m}$	67

**Table D.4—SEBC Mineralogy from X-ray Diffraction**

Mineral	Average Value %
Quartz	16.7
Plagioclase	trace
K-felspar	trace
Illite	59.2
Mixed layer	13.8
Kaolinite	10.5

SEBC mineralogy given in Table D.4 was from X-ray diffraction performed on 11 samples of the material sourced as SEBC (these values should be considered “semi-quantitative” as this type of analysis is only accurate to  $\pm 5\%$ ).

Recently qualified SEBC lots have had an API 13I methylene blue capacity in the range of 16–20 (meq/100 g) and an oilfield “barrel yield” of 27 barrels/ton to 32 barrels/ton (4.7 m<sup>3</sup>/t to 5.6 m<sup>3</sup>/t).

API SEBC is sourced from a single supplier, pretested, blended, repackaged, and then qualified by round robin testing using at least 3 different labs operated by different companies. Acceptance is at the discretion of API Subcommittee 13 Task Group 8 using a comparison of the current and previous round robin testing results for API 13A materials.

## **D.4 Contact Information**

**D.4.1** API Reference Materials are commonly available through drilling fluid testing supply companies.

**D.4.2** For further information on the API Reference Material Program, please contact the Standards Department, API, 200 Massachusetts Ave NW, Suite 1100, Washington, DC 20001, [standards@api.org](mailto:standards@api.org).

## Bibliography

- [1] API Recommended Practice 13I, *Recommended Practice for Laboratory Testing of Drilling Fluids*
- [2] API Recommended Practice 13K, *Recommended Practice for Chemical Analysis of Barite*
- [3] API Report 88-30, *Specification Parameters Determination Drilling Fluids Materials*, 1989
- [4] API Report 89-30, *Specification Parameters Determination Drilling Fluids Materials*, 1990
- [5] API Report 90-30, *Specification Parameters Determination Drilling Fluids Materials*, 1992
- [6] ASTM E177 <sup>6</sup>, *Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods*
- [7] ASTM D1141, *Standard Practice for the Preparation of Substitute Ocean Water*
- [8] ASTM E691, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*
- [9] ISO 3310-1 <sup>7</sup>, *Test sieves—Technical requirements and testing—Part 1: Test sieves of metal wire cloth*
- [10] ISO 10241, *International terminology standards—Preparation and layout*
- [11] ISO 6780, *Flat pallets for intercontinental materials handling—Principal dimensions and tolerances*
- [12] ISO 80000 (all parts), *Quantities and units*
- [13] Korosi, A. and Fabuss, B.M. (1968), Viscosity of Liquid Water from 25 °C to 150 °C, *Anal. Chem.*, 40(1), pp. 157–162.

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<sup>6</sup> ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, [www.astm.org](http://www.astm.org).

<sup>7</sup> International Organization for Standardization, Chemin de Blandonnet, 8, CP401, 1214 Vernier Geneve, Switzerland, [www.iso.org](http://www.iso.org).





AMERICAN PETROLEUM INSTITUTE

200 Massachusetts Avenue, NW  
Suite 1100  
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