

Drilling Fluids Materials

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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) concerning use of the API Monogram by Licensees is now purposely blank; refer to Annex A of API Specification Q1. 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 18th 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) on the API Monogram Program is now purposely blank; refer to Annex A of API Specification Q1.

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 ¹, *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 ², *Liquid-in glass laboratory thermometers—Principles of design, construction and use*

NIST National Bureau of Standards Monograph 150 ³, *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 ⁴.

3.1.2

flash side

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

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, www.astm.org.

² International Organization for Standardization, Chemin de Blandonnet, 8, CP401, 1214 Vernier Geneve, Switzerland, www.iso.org.

³ National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899, www.nist.gov.

⁴ American Chemical Society, 1155 Sixteenth Street NW, Washington, DC 20036, 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 (θ_1), dimensionless (ASTM 151H hydrometer)
b	yield point-plastic viscosity ratio, expressed in lbf/(100 ft ² •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
$c_{(CaCl_2)}$	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 µm, expressed in micrometers
D_2	equivalent particle diameter immediately less than 6 µm, 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 (°C ⁻¹) [degrees Fahrenheit (°F ⁻¹)]
m	sample mass, expressed in grams
m_1	dried sample mass, expressed in grams
m_2	residue mass retained by 75 µm sieve, expressed in grams
m_3	residue mass retained by 45 µm sieve, expressed in grams
m_4	mass of the 425 µm sieve and sample retained, expressed in grams

m_5	mass of 425 μm 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 θ_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 θ_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

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 μm , expressed in percent
w_2	cumulative percent for point immediately greater than 6 μm , expressed in percent
w_3	cumulative percent for point immediately less than 6 μm , expressed in percent
w_4	cumulative percent less than 6 μm , expressed in percent
w_5	mass fraction residue of particles greater than 45 μm , expressed in percent
w_6	mass fraction moisture, expressed in percent
w_{75}	mass fraction of sample passing through a 75 μm sieve, expressed in percent
w_{425}	mass fraction passing through a 425 μm sieve, expressed in percent
Y_p	yield point, expressed in pounds-force 100 ft^2
θ	temperature, expressed in degrees Celsius
θ_1	average temperature reading at lower temperature, expressed in degrees Celsius (degrees Fahrenheit)
θ_2	average temperature reading at higher temperature, expressed in degrees Celsius (degrees Fahrenheit)
ρ	sample density, expressed in grams per milliliters
η	viscosity of water, expressed in millipascal•seconds (centipoises)
η_{AV}	apparent viscosity, expressed in millipascal•seconds (centipoises)
η_{oil}	viscosity of calibrating fluid expressed in millipascal•seconds (centipoises)
η_{20}	1.002 mPa•s (cP) is the viscosity of water at 20 °C (68 °F)
η_θ	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 ± 0.1 °C (± 0.2 °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 μm 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, θ_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 θ_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 θ_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;

θ_1 is the average temperature reading ($^{\circ}\text{C}$ or $^{\circ}\text{F}$) at lower temperature;

θ_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, θ_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 ($^{\circ}\text{F}^{-1}$);

θ_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•ft² 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 ± 0.1 °C (± 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 ± 0.1 °C (± 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)	± 1.5	± 1.5
50 mPa·s (cP)	± 1.5	± 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 ± 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 ± 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 ± 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.

- To a suitable flask, add 50.00 mL ± 0.05 mL of deionized water and 50.00 mL ± 0.05 mL of standard calcium chloride solution.
- 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 ± 1 mL deionized water specified in 7.6 a) or 8.6.a).
- 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 .

- 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 CaCl_2 solution is equal to 4.8 mL:

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

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

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 the reference material program samples of test calibration barite and/or (where applicable) reference (calibration) bentonite for properties listed on the reference material's certificates of analysis or lot label, as required by this standard.

These results shall be within the "reproducibility limit (interlab)" listed in Table B.1 and Table B.3 for the value listed on the certificate of analysis or lot label. An example calculation is provided in Annex B for test calibration barite and reference (calibration) bentonite.

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 Packaging and Marking—Bagged and Bulk Material

6.1 Description

The manufacturer shall develop and maintain a procedure for packaging and marking of drilling fluid products that conform to this specification.

Packaging should preserve products used in drilling fluids, completion fluids, and oil well cements, including dry, powdered, or granular products not covered under this standard. Marking shall identify the product in each package or bulk container.

Packaging of palletized goods should safeguard the safe handling, transport, storage, and identification and minimizes damage and spillage.

NOTE Drilling fluid products may be supplied in unpackaged pneumatically conveyed bulk, bulk bags, multi-wallpaper sacks, or cardboard boxes.

6.2 Apparatus—Pallets

The preferred pallet design and construction shall conform to ISO 6780.

Unless otherwise agreed between the manufacturer and purchaser, the sizes for wooden chemical industry pallets (CP) shall be one of 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 × 130 cm (44 in. × 52 in.);
- e) 110 cm × 130 cm (42 in. × 52 in.), equivalent to CP4/CP7;
- f) 102 mm × 122 mm (40 in. × 48 in.).

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

When practicable, packed material shall be inside the dimensions of the pallet. 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.).

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

6.3 Apparatus—Super Sacks and Bulk Bags

Super sacks and bulk bags [flexible intermediate bulk container (FIBC) commonly named “big bag”] shall be resistant to moisture and damage in order to provide safe handling, transport, and storage of the product.

Super sacks and bulk bags should be selected with consideration for humidity-barrier capabilities relative to the needs of the specific product.

NOTE Drilling fluid product sacks are typically constructed with multiple layers of paper and plastic (usually polyethylene or polypropylene) between paper layers.

6.4 Marking—Palletized Material

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

- a) Manufacturer or customer trade name, or for barite as per API grade (Barite 4.x g/mL);
- b) gross/net mass, in kilograms (pounds);
- c) other information as specified by the manufacturer or in the contract, such as generic product's name, gross allowable mass, lot/batch number, date of manufacture, regulatory, transportation, and health, environmental, and safety information.

6.5 Marking—Super Sacks and Bulk Bags

Marking for super sacks and bulk bags shall include the following:

- a) manufacturer or customer trade name, or for barite as per API grade (Barite 4.x g/mL);
- b) net mass, in kilograms (pounds);
- c) lot/batch number traceable to the manufacturer's facility and country of origin.

6.6 Marking—Bulk Products

Marking requirements for bulk products shall be marked on or attached to the bill of lading/delivery ticket and shall include the following:

- a) manufacturer's product name, or for barite as per API grade (Barite 4.x g/mL);
- b) net mass, in kilograms (pounds);
- c) lot/batch number traceable to the manufacturer's country of origin.

6.7 Pallet Covers

When required by customer orders or environmental conditions, each unit of palletized material shall include a cover to further protect the material from external conditions that can result in damage to the product.

Examples of materials commonly used in pallet covers include:

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

Plastic used in a pallet cover shall be UV stabilized.

NOTE Cardboard, carton, or wood covers may be used in place of the above. If needed, a bottom layer of cardboard, PE sheet, or plywood may be connected to the cover to unitize the overall package.

6.8 Super Sack and Bulk Bag Mass

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

6.9 Storage

The manufacturer shall provide to the customer specific product storage requirements that can prevent damage to the product.

6.10 Recycling

When practicable, recycling of the remaining product shall conform to manufacturer or customer instructions.

Super sacks and bulk bags shall be selected to allow for recycling of the packaging material.

NOTE When handling chemicals, reduction in the volume of packaging materials is possible by using dedicated reusable, refillable containers such as intermediate bulk container (IBC) totes for liquids, bulk bags, and hoppers, or pneumatic transfer silos and associated equipment.

7 Barite

7.1 Principle

7.1.1 Drilling-grade barite is produced from commercial barium sulfate containing ores. Specifications are provided in Table 2 for two density grades of barite: Barite 4.1 g/mL and Barite 4.2 g/mL. 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 (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 specifications for barite are for two density grades: Barite 4.1 g/mL and Barite 4.2 g/mL. Other than density, Barite 4.1 g/mL and Barite 4.2 g/mL have identical test specifications. Barite 4.1 g/mL grade may not be appropriate for higher mud weights and a performance evaluation with Barite 4.2 g/mL should be made to determine suitability.

Table 2—Barite Physical and Chemical Requirements

Requirement	Standard	
	Barite 4.1 g/mL	Barite 4.2 g/mL
Density	4.10 g/mL minimum	4.20 g/mL minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum	
Residue greater than 75 μm	maximum mass fraction 3.0 %	
Particles less than 6 μm 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 $105\text{ }^{\circ}\text{C} \pm 2.5\text{ }^{\circ}\text{C}$ ($220\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$).

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 $32\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ regulated to $\pm 0.1\text{ }^{\circ}\text{C}$ ($90\text{ }^{\circ}\text{F} \pm 1\text{ }^{\circ}\text{F}$ 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.

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

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° 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, ρ , 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 ± 0.01 g of disodium salt of ETDA 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 ± 0.01 g of ammonium chloride (CAS No. 12125-02-9) and 570 mL ± 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 ± 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 ± 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 paper⁵, 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.

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.

- 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.
- 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.
- 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.

- 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.
- 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):

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

⁵ 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 μ m to 5 μ m (at 98 % efficiency); ash content: <0.015 % by weight; basis weight: 96 g/m² (0.02 lb/ft²); and thickness: 0.115 mm (0.0045 in.).

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 μm

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 μm (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 ($\frac{1}{4}$ in.) inlet connection, rated for 2.5 L/min at 70 kPa (0.65 gal/min at 10 lbf/in.²) 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.² $\pm$ 1 lbf/in.²).

7.8.10 Evaporating dish or functional equivalent.

7.8.11 Wash bottle.

7.9 Procedure—Measuring Barite Residue Greater than 75 μm

When measuring barite residue of diameter greater than 75 μm 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 μm 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.² $\pm$ 1 lbf/in.²) 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.

- e) 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 μm

Calculate the mass fraction of barite residue particles greater than 75 μm , 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 μm sieve, expressed in grams.

Record the calculated value.

7.11 Reagents and Apparatus—Measuring Barite Particles Less than 6 μm 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 $^{\circ}\text{C} \pm 3^{\circ}\text{C}$ (220 $^{\circ}\text{F} \pm 5^{\circ}\text{F}$).

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

7.11.5 **Balance**, with an accuracy of ± 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 $^{\circ}\text{C} \pm 5^{\circ}\text{C}$ (75 $^{\circ}\text{F} \pm 10^{\circ}\text{F}$).

7.11.11 **Thermometer**, including the range 16 $^{\circ}\text{C}$ to 32 $^{\circ}\text{C}$ (60 $^{\circ}\text{F}$ to 90 $^{\circ}\text{F}$) readable to 0.5 $^{\circ}\text{C}$ (1 $^{\circ}\text{F}$).

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 $^{\circ}\text{C}/20^{\circ}\text{C}$ (68 $^{\circ}\text{F}/68^{\circ}\text{F}$).

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 μm in Equivalent Spherical Diameter by Sedimentation Method

To measure particles less than 6 μm 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 ± 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 ± 2 mL (127 g ± 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 ± 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.
- 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.

- 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.
- Take hydrometer readings at intervals of 10 min ± 6 s, 20 min ± 6 s, 30 min ± 6 s, and 40 min ± 6 s (or until the first point below the 6 μm 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.
- Record the time, t , expressed in minutes, the temperature, θ , 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).

- For each time interval, determine the water viscosity, η , 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 μm 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 μm 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

ρ 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

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

ρ 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 ($^{\circ}\text{F}^{-1}$)];

θ is the suspension temperature, expressed in degrees Celsius (degrees Fahrenheit);

B_c is the hydrometer correction line initial value at θ_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 μm and the cumulative percent finer, w_3 , for particle diameter, D_2 , immediately less than 6 μm 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^[13] using Equation (12):

$$\eta_{\theta} = \frac{1.002}{10^{\left(\frac{1.37023 \times (\theta - 20) + 8.36 \times 10^{-4} \times (\theta - 20)^2}{(109 + \theta)} \right)}} \quad (12)$$

where

η_{θ} is the water viscosity at desired temperature, expressed in pascal•seconds (centipoises) (see Table 3);

θ 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 θ °C	Viscosity η mPa•s	Temperature θ °F	Viscosity η 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 ρ g/mL	Sample Constant K_s % / g	Sample Density ρ 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 oxide. 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 μm	maximum mass fraction 1.5 %
Residue greater than 45 μm	maximum mass fraction 15 %
Particles less than 6 μm in equivalent spherical diameter	maximum mass fraction 15 %