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Addendum 1

Table of Contents: The following entries shall be added:

Table of Contents (Tables section): The following entries shall be added:

Introduction: The third paragraph shall be amended to read:

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, <mark>Oil Companies Material Association (OCMA)–grade bentonite</mark>, 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.

Section 1: The second sentence shall be amended to read:

The materials covered are barite; hematite; bentonite; non-treated bentonite; <mark>OCMA–grade bentonite</mark>; 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.

Section 3.3: The following abbreviation shall be added to the list:

OCMA   Oil Companies Materials Association

Section 19: The following new section shall be added:

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/(100ft²•CP)
Filtrate volume	maximum 16.0 mL
Residue of diameter greater than 75 µm	maximum mass fraction 2.5 %

19.2 Reagents and Apparatus—Measuring Properties of an OCMA-grade Bentonite Suspension

19.2.1 **Thermometer**, accurate to ±0.5 °C (±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 $^5/_6$ 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 ±5 mL (TC) and 10 mL ±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 ±0.01 g of bentonite/clay (as received) to 350 mL ±5 mL of deionized water while stirring on the mixer.

b) After stirring 5 min ±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 ±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 ±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 ±1 °C (77 °F ±2 °F).

19.4 Calculation—Rheological Properties of an OCMA-grade Bentonite Suspension

Calculate the plastic viscosity, η_{PV}, expressed in centipoises, according to Equation (49), the yield point, Y_P, in pounds-force per 100 ft^2, according to Equation (50), and the yield point-plastic viscosity ratio, b, expressed in lbf/(100 ft^2•cP), according to Equation (51):

$$\eta_{PV} = R_{600} - R_{300} \tag{49}$$

$$Y_P = R_{300} - \eta_{PV} \tag{50}$$

$$b = \frac{Y_P}{\eta_{PV}} \tag{51}$$

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 ft² units are not exact. A direct conversion from lbf/100 ft² 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^{-1}.

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.

a) Recombine all of the suspension, as prepared and tested in 19.3, and stir in the mixing container for 1 min ±6 s on the mixer. Adjust the suspension temperature to 25 °C ±1 °C (77 °F ±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 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.

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 ±30 kPa (100 psi ±5 psi). 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 ±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 ±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):

$$V = 2\,V_c \tag{52}$$

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 µm

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

19.7.2 **Oven**, regulated to 105 °C ±2.5 °C (220 °F ±5 °F).

19.7.3 **Balance**, with an accuracy of ±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 $^5/_6$ in.); d bottom, 70 mm (2.75 in.).

19.7.6 **Sieve**, 75 µm (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 (¼ 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.

19.7.8 **Water-pressure regulator**, capable of regulation to (70 kPa ±6 kPa (10 lbf/in.² ±1 lbf/in.²).

19.7.9 Evaporating Dish.

19.7.10 Wash Bottle.

19.8 Procedure—Measuring OCMA-grade Bentonite Residue of Greater than 75 µm

The following procedure shall be followed when measuring barite residue of greater than 75 µm.

a) Weigh 10.00 g ±0.01 g of bentonite.

b) Add the weighed bentonite sample to approximately 350 mL of water containing 0.20 g ±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 ±0.05 g of sodium hexametaphosphate powder or by adding 2.0 mL ±0.1 mL of sodium hexametaphosphate 10 % ±0.5 % by mass solution.

c) Stir suspension on the mixer for 30 min ±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 ±6 kPa (10 psi ±1 lbf/in.²) from the spray nozzle for 2 min ±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 μm

Calculate the mass fraction residue of OCMA-grade bentonite particles of greater than 75 μm, w_1, in percent, as given in Equation (53):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{53}$$

where

m is the sample mass, in grams;

m_2 is the residue mass, in grams.

Record the calculated value.

Annex B.1.1: The following list shall be amended to read:

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.

Annex B.1.2: The first sentence shall be amended to read:

The precision data in Tables B.1 through B.11 reflect the fact that the standard test methods are subject to normal test variability.

Annex B.2: The first sentence of the first paragraph shall be amended to read:

The limits in Tables B.1, B.3, B.4, B.5, B.6, B.9, B.10, and B.11 were determined by interlaboratory studies conducted inform 1988 to 1991.

Annex B.2: The last sentence of the first paragraph shall be amended to read:

The limits in Tables B.2, B.7, and B.8 were determined by interlaboratory studies conducted in 1992.

Table B.5: The following table and associated paragraph shall be added after Table B.4:

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.