Addendum 1
June 1999

EFFECTIVE DATE: DECEMBER 1999

Drilling Fluid Materials

API SPECIFICATION 13A
FIFTEENTH EDITION, MAY 1993

American
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Addendum 1 to Drilling Fluid Materials
(Also contains items from September 1993 Errata)

Front Cover

Revise Effective Date of Specification to:

November 1993

Page 2

Statement beginning "Attention Users:" concerning bar notations should be deleted.

Page 2

Revise note to read as follows:

Note: This edition supercedes the Fourteenth Edition, dated August 1, 1991. It includes revisions adopted at the 1992 Standardization Conference as reported in CIRC PS-1986 and subsequently approved by letter ballot.

Page 2

Change the word:

Director to general manager

Pages 2, 3, and 5

Revise references to the address of API to read as follows:

API
Upstream Department
1220 L Street, NW
Washington, DC 20005
Phone: 202-682-8000

Page 5, Section 1.5

Add the following reference documents:

ISO 6780

APME 1993

BS 6382 Part 1: 1983

BS 6939 Parts 1 & 3: 1987

European Flexible Intermediate Bulk Container Association (EFIBCA), 1985: EFIBCA Standard 001, First Edition,
March 1985.

European Flexible Intermediate Bulk Container Association (EFIBCA), 1985: ICHCA, Safety Panel Briefing Pamphlet No. 9, March 1996.

Page 6, Section 2.2.j
Page 12, Section 3.2.j

Add the following:

Note: Laboratory grade tissues are nonabsorbent and thus unsuitable for use in this test procedure.

Page 8, Section 2.10, Equation (a)
Page 14, Section 3.10 Equations (a) and (b)
Page 18, Section 4.9, Equation

Change in these equations:

"$- C_c$" to "$+ C_c$"

Page 11, Figure 2.2

The pressure gauge shown in the picture is not correct.

Page 15, Section 13.3.c, Equation (b)

The constant should read:

"17.5".

Page 17, Table 4.1
Page 21, Table 6.1

Delete the Moisture requirement and specification from both tables.

Page 18, Sections 4.10, 4.11, and 4.12
Pages 22 and 23, Sections 6.10, 6.11, and 6.12

Delete these sections.

Page 17, Section 4.2.a
Page 19, Section 5.2.g
Page 21, Section 6.2.a
Page 24, Section 7.2.a
Page 26, Section 8.2.a
Page 28, Section 9.2.a
Page 30, Section 10.2.a
Page 33, Section 11.2.a

Thermometers with a range of 32 – 220 ± 1°F (0 – 105 ± 0.5°C) are not generally available. Delete reference to these thermometers and add the following text:

Use thermometer with an accuracy specified in the procedure that covers the operating range of the test.

Page 17, Section 4.3.d
Page 19, Section 5.3.d
Page 21, Section 6.3.d

To the end of the first sentence add the words:

"or in a constant temperature device."

Page 37, Section 12.4.e

Change the word:

"primary" to "secondary"

Page 40, Section 12.9

Change the title of this section to:

"Standardization of EDTA Solution"

Page 42, Section 13

Replace with the following:

SECTION 13
PACKAGING, MARKING, AND STORAGE

13.1 PACKAGED MATERIAL

13.1.1 Marking

Each package of drilling fluid material conforming to the appropriate section of this specification shall be marked so as to include the following information:

a. The name of the material as it appears in the appropriate section of this specification, in block letters at least $^1/_2$ in. (13 mm) in height.
b. The weight of the material in numbers and letters at least $^1/_4$ in. (6 mm) in height. The weight will be listed in pounds and kilograms.
c. The date of manufacture, in block letters and/or numbers at least $^1/_8$ in. (3 mm) in height.

13.1.2 API Monogram

The API monogram, if applicable, will be applied in a place of prominence.

13.1.3 Package Weight

Each package shall contain the product weight marked thereon, ± 5%. The average weight shall not be less than the weight as marked. The average weight is determined by averaging the total measured weight of a minimum of 2% of the packaging run, selected at random, of the material and dividing the total weight by the number of packages actually weighed. This information will be documented each 24 hours.

13.1.4 Storage

The manufacturer shall develop, document, and implement methods for the proper preservation of packaged drilling fluid material.

13.2 BULK MATERIAL—API MONOGRAM

The API monogram, if applied, will be placed on the delivery ticket accompanying each bulk shipment in a position of prominence.

If the licensee receives material not under his control, and this material conforms to the chemical and physical requirements of the material specification and all other requirements under the API Specification Q1, the API monogram can be applied.

Following Page 47, add the following Appendix D to the end of the document:

APPENDIX D—RECOMMENDED PRACTICE FOR PACKAGING MATERIALS

D.1 GENERAL

D.1.1 These recommendations apply to products covered by this standard and sold in bagged/palleted quantities and in flexible intermediate bulk containers (FIBCs), i.e., barite, hematite, bentonite, nontreated bentonite, OCMA Grade bentonite, attapulgite, sepiolite, technical grade low viscosity CMC, technical grade high viscosity CMC, and starch.

D.1.2 Packaging of palletized goods should safeguard the means of safe handling, transport, storage, and identification, and minimize damage and spillage. Packed material should be inside the dimensions of the pallet although some overhang is allowed.

D.1.3 Packaging of FIBCs, i.e., Big Bags or Super Sacks, should safeguard the means of safe handling, transport, storage, and identification, and minimize damage and spillage.

D.1.4 A potential benefit of these recommendations for packaged materials is the improvement of possible recycling of all packaging materials, including that for dry powdered or granular materials not covered under the standard, and used in drilling fluids, completion fluids, and oil well cements.

D.2 EQUIPMENT—PALLETS

D.2.1 The preferred pallet design and construction should be in accordance with ISO 6780 or APME 1993.

D.2.2 Preferred sizes for wooden pallets include:

a. 1200 × 1000 mm (47 × 39 in.) CP6.
b. 1140 × 1140 mm (45 × 45 in.) CP8/CP9/CP3.
c. 1219 × 1219 mm (48 × 48 in.).
d. 1118 × 1321 mm (44 × 52 in.).
e. 1067 × 1321 mm (42 × 52 in.) equivalent to CP4/CP7.
f. 1016 × 1219 mm (40 × 48 in.).

Note: CP = sizes according to ISO 6780.

D.2.3 Other pallet sizes and details concerning design and construction shall be agreed upon by the manufacturer and the customer.

D.2.4 The maximum outside dimensions of the total package will be in accordance with the applicable pallet size plus a maximum overhang of 3 cm (1.2 in.). The overall height shall not exceed 203.2 cm (80 in.).

D.2.5 The maximum net weight should not exceed 2000 kg (4409 lb).

D.2.6 Each pallet may have a cover made of at least one of the following:

a. Polyethylene (PE) shrink or wrapped film.
b. PE bonnet type.
c. Polypropylene (PP) bonnet type.

All plastics must be UV stabilized when required by contract. Cardboard, carton, or wood covers may be used in place of the above covers. If appropriate, a bottom layer of cardboard, PE sheet, or plywood may be connected to the cover to unitize the overall package.

D.3 EQUIPMENT—BAGS

D.3.1 The manufacturer filling the bag shall take reasonable steps to ensure bag construction capable of safe handling, transport and storage.

D.3.2 The manufacturer shall take reasonable steps to select bags that will minimize waste and provide recycling possibilities of the packaging material.

D.3.3 The manufacturer shall consider humidity barrier capabilities of the bags against the needs of the particular product when selecting bags.

D.4 EQUIPMENT—FIBCs

D.4.1 FIBCs have bodies made of flexible material such as woven fabric, plastic film or paper and are designed to be in contact with the contents, either directly or indirectly, through an inner liner and are collapsible when empty.

It should be realized when considering their use that FIBCs:

- cannot be handled manually when filled.
- are designed to be lifted from above by means of integral, permanently attached, or detachable devices.
- are limited to maximum capacity of 3 cubic meters (9106 cubic feet).

D.4.2 Following are classifications of FIBCs:

a. **Heavy-duty reusable FIBC**—FIBC designed and intended to be used for many filling and discharge cycles. This class is both field and factory repairable.
b. **Standard-duty reusable FIBC**—FIBC designed and intended to be used for a limited number of filling and discharge cycles where permitted. This class should not be reused if damaged and is not repairable.
c. **Single-trip FIBC**—FIBC designed and intended to be used for one fill and discharge cycle only. This FIBC should

not be re-used. Neither replacement of inner liner nor repair of the FIBC is relevant to this category.

D.4.3 When handling, if more than one loop is provided the load should be distributed evenly by all loops. Lifting devices should be free from sharp edges or burrs that may cause damage to lifting loops.

D.4.4 Fill devices should be suitable for the manufacturer to properly fill and close the FIBC to prevent spillage, waste, and contamination of the packaged product.

D.4.5 Discharge devices should be chosen considering the nature of the product and customer requirements.

D.5 MARKING—PALLETS

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

a. Product name.
b. Gross/net weight in kg or lb.

D.6 MARKING—BAGS

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

a. The name of the material in print script at least 13 mm (0.5 in.) in height.
b. The weight of the material in letters, or numbers and letters, at least 6 mm (0.25 in.) in height. The weight will be listed in kilograms or pounds.
c. c. The lot/batch number in print script and/or numbers at least 3 mm (0.125 in.) in height, traceable to manufacturer's country of origin, and date of manufacture.
d. Identification as recyclable.
e. Safety information.

D.7 MARKING—FIBCs

D.7.1 The FIBC Manufacturing Label/Tag information shall indicate FIBC performance information pertaining to one or more of the following standards:

a. BS6382 Part 1: 1983.
b. BS 6939 Parts 1 & 3: 1987.
c. EFIBCA STANDARD 001, March 1985.

D.7.2 The following product information shall be included, where applicable and as specified by contract:

a. Name of material in print script.
b. Weight of material in letters and/or numbers. The weight shall be listed in kilograms and/or pounds.
c. The Lot/Batch Number in letters and/or numbers.
d. Identification as recyclable.
e. Safety information.

Note: Type size shall be in accordance with local transportation and safety regulations regarding the identification of goods.

D.8 STORAGE

The manufacturer will advise on storage requirements upon request or contract requirements.

D.9 RECYCLING

If appropriate, recycling of the remaining materials after using the contents may be done in accordance with the following guidelines:

a. For pallets, general recovery and recycling, provided that pallet description is in accordance with ISO 6780 or APME 1993.
b. For pallet covers, selection for PE, PP or carton and recycle accordingly.
c. For bags, using high performance paper quality results in less packaging materials and less waste for recycling. After separation of the various components, recycle accordingly.
d. For FIBCs, only those mentioned in D.4.2.a and D.4.2.b are designed for recycling, resulting in less waste.

All recycling should be done in accordance with local regulations and instructions agreed upon by the parties involved.

Note: When handling chemicals, reduction of the packaging materials volume can be obtained by application of containers in a dedicated container scheme.

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Specification for Drilling-Fluid Materials

API SPECIFICATION 13A (SPEC 13A)
FIFTEENTH EDITION, MAY 1, 1993

American Petroleum Institute
1220 L Street, Northwest
Washington, DC 20005

Issued by
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Production Department

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THIS PUBLICATION CONTACT THE API PRODUCTION DEPARTMENT,
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FOREWORD

a. This specification is under the jurisdiction of the API Committee on Standardization of Drilling Fluid Materials.

b. The purpose of this specification is to provide standards for materials used in oil well drilling fluids. The materials covered are barite, hematite, bentonite, nontreated bentonite, OCMA grade bentonite, attapulgite, sepiolite, technical-grade low viscosity carboxymethylcellulose (CMC), technical-grade high viscosity carboxymethylcellulose (CMC), and starch.

c. Reference is made herein to API RP 13B-1: Standard Procedure for Field Testing of Water-Based Drilling Fluids, which is also under the jurisdiction of the Committee on Standardization of Drilling Fluid Materials.

d. Additional publications under jurisdiction of this committee are:

RP 13B-1	Recommended Practice for Standard Procedure for Field Testing Water-Based Drilling Fluids
RP 13B-2	Recommended Practice for Standard Procedure for Field Testing Oil-Based Drilling Fluids
Bul 13C	Bulletin on Drilling Fluids Processing Equipment
Bul 13D	Bulletin on the Rheology of Oil Well Drilling Fluids
RP 13E	Recommended Practice for Shale Shaker Screen Cloth Designation
RP 13G	Recommended Practice for Drilling Mud Report Form
RP 13I	Recommended Practice for Laboratory Testing of Drilling Fluids
RP 13J	Recommended Practice for Testing Heavy Brines
RP 13K	Recommended Practice for Chemical Analysis of Barite

e. Manufactures interested in obtaining a monogram license for these materials should either write the API Dallas office at 2535 One Main Place, Dallas, Texas 75202 for an application package or purchase a copy of the *Composite List of Manufacturers Licensed for Use of the API Monogram,* Order No. 811-00005.

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SEE ALSO, OSHA PERMISSIBLE EXPOSURE LIMITS (PELS) FOR BARIUM AND OTHER MATERIALS REFERENCED: 29 CFR 1910.1000 AND AMERICAN CONFERENCE OF GOVERNMENTAL INDUSTRIAL HYGIENISTS, THRESHOLD LIMIT VALUES® (TLV's).**

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SECTION 1
GENERAL INFORMATION

1.1 Coverage

This specification covers physical properties and test procedures for materials manufactured for use in oil well drilling fluids. The materials covered are barite, hematite, bentonite, nontreated bentonite, attapulgite, sepiolite, OCMA grade bentonite, technical-grade low-viscosity carboxymethylcellulose (CMC-LVT), technical-grade high-viscosity carboxymethylcellulose (CMC-HVT), and starch. This document is intended for the use of manufacturers of named products.

1.2 Definitions

a. ACS reagent grade: American Chemical Society (ACS) grade chemicals meet purity standards as specified by the ACS.

b. TC: To contain

c. TD: To deliver

d. Flash side: Side containing residue ("flash") from stamping; also, the side with concave indentations

e. CAS: Chemical Abstract Service

1.3 Quality Control Procedures

a. 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.

b. Use of Test Calibration Materials in Checking Testing Procedures:

1. Test Calibration Barite Lot 001* and Lot 002*, and Test Calibration Bentonite may be obtained by contacting the API Production Department Office, 1201 Main Street, Suite 2535, Dallas, TX 75202-3994. The calibration test materials are shipped in a 2-gallon (7.6 liter) plastic container.

   *NOTE: *Test Calibration Barite 001 will be routinely supplied until quantities are exhausted at which time Test Calibration Barite 002 will take its place.*

2. The API office will forward the request to the designated custodian for further handling. The test calibration products will be furnished with a certificate of calibration giving the established values for each property and the confidence limits within which a laboratory's results should fall.
3. The custodian shall furnish a certificate of analysis for each sample.
4. For calibration requirements of API Test Calibration materials, refer to Par. 12.2k and Par. 12.3j.

1.4 API Standard Evaluation Base Clay (Formerly OCMA Base Clay — Not OCMA Grade Bentonite)

Stocks of API Standard Evaluation Base Clay have been set aside and can be ordered through the API Production Department Office, 1201 Main Street, Suite 2535, Dallas, TX 75202-3994.

1.5 Reference Document

API RP 13B-1, 1st Edition, June, 1990.

1.6 API Standards

API specifications are published to facilitate communications between purchasers and manufacturers, to provide interchangeability between like 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 specification provides minimum requirements and is not intended to inhibit anyone from purchasing or producing materials to other specifications. This specification is subject to periodic review and may be revised or withdrawn at such time as deemed appropriate.

The status of this specification can be ascertained from the API Production Department (Tel. 214-748-3841). For further information regarding API Policy, please refer to the Policy Statement on Page 4 of this specification.

1.7 Records Retention

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

SECTION 2
BARITE

2.1 Description

a. 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 accessory minerals other than the barium sulfate ($BaSO_4$) mineral. Because of mineral impurities, commercial barite may vary in color from off-white to grey to red or brown. Common accessory minerals are silicates such as quartz and chert, carbonate compounds such as siderite and dolomite, and metallic oxide and sulfide compounds. Although these minerals are normally insoluble, they can, under certain conditions, react with other components in some types of drilling fluids and cause adverse changes in the drilling fluid properties. (See Spec. 13A, Appendix B for more details.)

b. Drilling grade barite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the chemical and physical requirements of Table 2.1, represents the product produced, and is controlled by the manufacturer.

TABLE 2.1
BARITE PHYSICAL AND CHEMICAL REQUIREMENTS

Requirement	Specification
Density	4.20 g/cm^3, minimum
Water Soluble Alkaline Earth Metals as Calcium	250 mg/kg, maximum
Residue greater than 75 micrometers	3.0 wt. percent, maximum
Particles less than 6 micrometers in equivalent spherical diameter	30 wt. percent, maximum

DENSITY

2.2 Equipment — Le Chatelier Flask Method

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

c. Le Chatelier flask: clamped or weighted to prevent flotation in water bath

d. Kerosene or mineral spirits

e. Transparent constant temperature bath: 90 ±1°F (32 ±0.5°C) regulated to ±0.1°C (e.g., 10-gallon aquarium with heater/circulator attachment or functional equivalent)

f. Balance: precision of 0.01 g

g. Volumetric pipette: one 10-cm^3

h. Magnifying glass

i. Wooden dowel: approximately 5/16 inch (8mm) in diameter and 12 inches (30 cm) in length, or a functional equivalent

j. Tissue paper

k. Low-form weighing dish with spout: approximately 100-cm^3 capacity, or a functional equivalent

l. Fine bristle brush

2.3 Procedure

a. If required, equilibrate approximately 100 g dried barite to room temperature in the desiccator.

b. Fill a clean Le Chatelier flask to approximately 7/8 inch (22mm) below the zero mark with kerosene.

c. Place the flask upright in the constant temperature bath. The level of water in the bath should be higher than the 24-cm^3 graduation of the flask, but below the stopper level. Assure flask is stabilized by use of clamps or weights.

d. Allow the flask and contents to equilibrate for a minimum of one hour. Using the magnifying glass with care to keep eyes at meniscus level, record the initial volume to the nearest 0.05 cm^3 (doubtful digit) without removing the flask from the constant temperature bath.

NOTE: *If kerosene level is above or below the -0.2 to +1.2 cm^3 volume range after equilibrating, use the 10-cm^3 pipette to add or remove kerosene in order for it to come within this range. Allow the flask to equilibrate at least one hour and record initial volume as in Par. 2.3d.*

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 kerosene in the flask.*

f. Weigh 80 ±0.05 g dried barite into the weighing dish and carefully transfer to the Le Chatelier flask. Take care to avoid splashing of the kerosene or plugging of 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, then replace the stopper.

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 kerosene 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 degrees 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 one-half hour.

j. Remove the flask from the bath and repeat Par. 2.3h to remove any remaining air from the barite sample.

k. Immerse the flask in the bath again for at least one hour.

l. Record the final volume in the same manner as described in Par. 2.3d.

2.4 Calculation

$$\text{Density, g/cm}^3 = \frac{\text{Weight sample, g}}{\text{Final vol., cm}^3 - \text{initial vol., cm}^3} \qquad \text{(a)}$$

Record calculated density.

WATER SOLUBLE ALKALINE EARTH METALS AS CALCIUM

2.5 Equipment

a. Balance: capacity exceeding 100 g with a precision of 0.01 g

b. Erlenmeyer flask: 250-cm^3 nominal capacity, equipped with a tight fitting stopper

c. Graduated cylinder (TD): 100-cm^3 capacity with 1-cm^3 graduations

d. Aqueous EDTA solution: 3.72 ±0.01 g of disodium salt of ethylenediaminetetraacetic acid dihydrate [disodium salt of (ethylenedinitrilo) tetraacetic acid dihydrate] (CAS #6381-92-6) diluted to a final volume of 1000 cm^3 with deionized water in a volumetric flask

e. Buffer solution: 67.5 ±0.01 g ammonium chloride (CAS #12125-02-9) and 570 ±1 cm^3 15N ammonium hydroxide (CAS #1336-21-6) solution diluted to a final volume of 1000 cm^3 with deionized water in a volumetric flask

f. Hardness indicator solution: 1 ±0.01 g Calmagite (CAS #3147-14-6), or equivalent [1-(1-hydroxy-4-methyl-phenylazo)-2-naphthol-4-sulfonic acid] diluted to a final volume of 1000 cm^3 with deionized water in a volumetric flask

g. Titration vessel: beaker of 100 to 150-cm^3 capacity

h. Graduated serological pipettes or burette. Graduations should be 0.1 cm^3

i. Volumetric pipettes (TD): 10-cm^3 or equivalent.

j. Deionized (or distilled) water

k. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, Paragraph 3.2, or filtration funnel

l. Filter paper: Whatman 50, or equivalent

m. Small glass container

n. Wrist action shaker, optional

o. Volumetric flask: one 1000-cm^3

p. Stirring rod

2.6 Procedure

a. Weigh 100 ±0.05 g of barite. Transfer to the Erlenmeyer flask and add 100 ±1 cm^3 of deionized water. Stopper the flask and shake for at least five minutes during an approximate one hour interval or by an optional mechanical shaking apparatus for 20 to 30 minutes.

b. After shaking, 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 ±1 cm^3 deionized water to the titration vessel. Add about 2 cm^3 of hardness buffer and sufficient hardness indicator to achieve a distinct blue color. Swirl to mix.

NOTE: *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 cm^3 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 will develop if calcium and/or magnesium are present.

e. If hardness is present, begin stirring and titrate with EDTA solution to the blue end point. The end point of the titration is best described as the point at which additional EDTA produces no further red to blue change.

NOTE: *If end point is unclear or unobtainable, other tests shall be performed. Results and methodology of these tests will be recorded.*

2.7 Calculation

$$\begin{array}{c}\text{Soluble Alkaline} \\ \text{Earth Metals} \\ \text{as Calcium,} \\ \text{mg/kg}\end{array} = 400\left(\frac{\text{EDTA vol., cm}^3}{\text{Sample vol., cm}^3}\right) - C_c{*} \qquad \text{(a)}$$

*where C_c = calibration correction from Par. 12.9

Record calculated value.

RESIDUE GREATER THAN 75 MICROMETERS

2.8 Equipment

a. Oven regulated to 220 ±5°F (105 ±3°C)

b. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant or equivalent

c. Balance: precision of ±0.01 g

d. Sodium hexametaphosphate (CAS #10124-56-8)

e. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

f. Container: approximate dimensions — 7 inches (180mm) deep, 3-13/16 inch (97mm) ID top, 2-3/4 inch (70mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D or equivalent)

g. A 75-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth.

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

h. Spray nozzle: 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body, or equivalent) attached to water line with 90 degree elbow as shown in Fig. 2.2

i. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)

j. Evaporating dish or functional equivalent

k. Wash bottle

2.9 Procedure

a. If required, equilibrate approximately 60 g of dried barite in a desiccator.

b. Weigh 50 ±0.01 g of dried barite. Add the weighed sample to approximately 350 cm^3 of water containing about 0.2 g of sodium hexametaphosphate. Stir on the mixer for 5 ±1 minutes.

c. Transfer the sample to the 75-micrometer sieve. Use wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ±7 kPa) from a spray nozzle for two minutes ±15 seconds. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve — move the spray of water repeatedly over the sample.

d. 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 weight. Record residue weight and total drying time.

2.10 Calculation

$$\begin{array}{l}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{75 micrometers}\end{array} = 100\left(\frac{\text{Weight residue, g}}{\text{Weight sample, g}}\right) - C_c \ast \quad \text{(a)}$$

*where: C_c = correction factor as calculated from Par. 12.5

Record calculated value.

PARTICLES LESS THAN 6 MICROMETERS IN EQUIVALENT SPHERICAL DIAMETER

2.11 Equipment — Sedimentation Method

a. Oven regulated to 220 ±5°F (105 ±3°C)

b. Desiccator, with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

c. Balance: precision of 0.01 g

d. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

e. Container: 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

f. Dispersant solution: 40 ±0.1 g sodium hexametaphosphate and 3.60 ±0.1g anhydrous sodium carbonate (CAS #497-19-8) per 1000 cm3 of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9.0.

g. Glass sedimentation cylinder: 18 inches (457 mm) in height and 2-1/2 inches (63 mm) in diameter, marked for a volume of 1000 cm^3 (ASTM D-422-63)

h. Rubber stopper: Number 13

i. Water bath or constant temperature room: capable of maintaining a convenient constant temperature at 75 ±10°F (24 ±5°C)

j. Thermometer: 60-90 ±1°F (16-32 ±0.5°C)

k. Hydrometer: ASTM 151H, graduated to read specific gravity of suspension

l. Timer: interval, mechanical or electrical, precision of 0.1 minutes

2.12 Procedure — Sedimentation Method

a. Weigh 80 ±0.1 g of dry barite and place in mixing container.

b. Add 125 ±2 cm^3 of dispersant solution (from Par. 2.11f). Dilute to approximately 400 cm^3 with deionized water. Rinse all adhering particles from spatula into suspension.

c. Stir 5 ±0.5 minutes on mixer.

d. 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-cm^3 mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 ±5 seconds while holding a No. 13 rubber stopper in the top of the cylinder.

NOTE: *This is a critical step. Suspension must be homogeneous at start of sedimentation. This is difficult to obtain because of the high density of barite.*

f. Set the cylinder into the water bath (or counter top of constant temperature room) and simultaneously start the timer. Hang the thermometer in the suspension.

g. Take hydrometer readings at intervals of 10, 20, 30, and 40 ±0.1 minutes (or until the first point below the 6 micrometer value is reached). To take a hydrometer reading, *carefully and slowly* lower the hydrometer to approximately the 1.020 reading before releasing. After hydrometer stabilizes, read the top of the meniscus at the prescribed time. *Carefully and* *slowly* remove hydrometer, rinse with deionized water, and dry after each reading. The hydrometer must be removed immediately after each reading to eliminate particle build-up on the shoulders which causes erroneous results. All hydrometer readings must be done with a minimum of fluid disturbance to preserve suspension settling equilibrium.

h. Record time, minutes (T), Temperature, °F or °C* (t), and hydrometer reading (H) on data sheet.

*NOTE: *Temperature may be measured in either °F or °C as long as all measurements and calculations are consistent in units, including hydrometer calibration.*

i. For each time interval, determine effective hydrometer depth (L) and water viscosity (v) from Table 2.3 and Table 2.2. Record on data sheet.

2.13 Calculation — Sedimentation procedure.

a. From hydrometer calibration (Par. 12.6) enter hydrometer correction slope (Mc) and hydrometer correction intercept (Bc) into data sheet.

b. Calculate sample constant (Ks) as follows (or determine from Table 2.4) and enter into data sheet:

$$Ks = \frac{100 \times D}{W\,(D-1)} \qquad \text{(a)}$$

where: Ks = Sample Constant

D = Sample density, g/cm^3

W = Sample weight, g

c. Calculate and enter onto data sheet the equivalent spherical diameter (de) for each time interval as follows:

$$de = 17.5\sqrt{\frac{vL}{(D-1)\,T}} \qquad \text{(b)}$$

where: de = Equivalent spherical diameter, micrometers

v = Viscosity of water, cP

D = Density of sample, g/cm^3

T = Time, minutes

L = Effective depth, cm

d. Calculate and enter onto data sheet the cumulative percent finer for the equivalent particle diameter (de) immediately greater than 6 micrometers (SH) and the equivalent particle diameter (de) immediately less than 6 micrometers (SL) as follows:

$$S = Ks \times [(Mc \times t) - Bc + (H-1) \times 1000] \qquad \text{(c)}$$

where: S = Cumulative percent finer than size

Ks = Sample constant

Mc = Hydrometer correction slope

t = Suspension temperature, °F or °C (See note, Par. 2.12h)

Bc = Hydrometer correction intercept

H = Hydrometer reading.

e. Calculate and enter onto data sheet cumulative percent less than 6 micrometers (S6) as follows:

$$S6 = \left[\left(\frac{SH - SL}{dH - dL}\right)(6 - dL)\right] + SL \qquad \text{(d)}$$

where: S6 = Cumulative percent less than 6 micrometers

SH = Cumulative percent for point immediately greater than 6 micrometers

SL = Cumulative percent for point immediately less than 6 micrometers

dH = Equivalent particle diameter immediately greater than 6 micrometers

dL = Equivalent particle diameter immediately less than 6 micrometers

Example Data Sheet

Hydrometer Serial #	XXXXX
Mc	0.144
Bc	13.08
Ks	1.629
Sample Density (D)	4.30 g/cm^3
Sample Weight (W)	80 g

Time (T)	Temp (°F) (t)	Hyd. Rdg. (H)	Water Vis. (v)	Effective Depth (L)	Particle Diameter (de)	%Finer (S)	%Finer (S6)
10	78	1.0280	.8792	8.9	8.5	42.6	
20	78	1.0210	.8792	10.7	6.6	31.2	
30	78	1.0170	.8792	11.8	5.7	24.7	
40	78	1.0140	.8792	12.6	5.1	9.8	
							26.9%

Example Calculation:

Sample Constant, Ks from (a)

$$Ks = \frac{100 \times 4.30}{80 \times (4.30-1)} = 1.629$$

Example Calculation for 20 minute reading:

a. Equivalent diameter, de, from (b)

$$de = 17.5\sqrt{\frac{0.8792 \times 10.7}{(4.30-1) \times 20}} = 6.6 \textit{ micrometers}$$

b. Percent finer, S, from (c)

$$S = 1.629 \times [(.1444 \times 78) - 13.08 + (1.021-1) \times 1000]$$

$$^*S = 1.629 \times (11.26 - 13.08 + 21.0) = 31.2\ \%$$

Example Calculation for percent less than 6 micrometers, S6, from (d)

SH = 31.2
SL = 24.7
dH = 6.6
dL = 5.7

$$S6 = \frac{(31.2-24.7)}{(6.6-5.7)}(6.0-5.7) + 24.7 = 26.9\%$$

TABLE 2.2
VISCOSITY OF WATER AT VARIOUS TEMPERATURES

Temperature °F	(°C)	Viscosity cP or mPa·S	Temperature °F	(°C)	Viscosity cP or mPa.S
60	15.6	1.1211	72	22.2	0.9498
61	16.1	1.1050	73	22.8	0.9374
62	16.7	1.0893	74	23.3	0.9253
63	17.2	1.0739	75	23.9	0.9134
64	17.8	1.0589	76	24.4	0.9018
65	18.3	1.0442	77	25.0	0.8904
66	18.9	1.0298	78	25.6	0.8792
67	19.4	1.0158	79	26.1	0.8683
68	20.0	1.0020	80	26.7	0.8576
69	20.6	0.9885	81	27.2	0.8470
70	21.1	0.9753	82	27.8	0.8367
71	21.7	0.9624	83	28.3	0.8266

$$^*\mathrm{LOG}(V20/Vt) = \frac{[1.37023(t-20)+0.000836(t-20)^2]}{(109+t)}$$

where: t = TEMP IN DEGREES CELSIUS(C)
V20 = 1.002
Vt = VIS. AT DESIRED TEMPERATURE

TABLE 2.3
VALUES OF EFFECTIVE DEPTH BASED ON READINGS ON HYDROMETER NO. 151H USED IN SPECIFIC SEDIMENTATION CYLINDER

Uncorrected Hydrometer Reading	Effective Depth L, cm	Uncorrected Hydrometer Reading	Effective Depth L, 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 2.4
SAMPLE CONSTANT (Ks) FOR 80.0g SAMPLE WT.

Sample Density	Sample Constant
4.20	1.641
4.21	1.640
4.22	1.639
4.23	1.637
4.24	1.636
4.25	1.635
4.26	1.634
4.27	1.633
4.28	1.631
4.29	1.630
4.30	1.629
4.31	1.628
4.32	1.627
4.33	1.625
4.34	1.624
4.35	1.623
4.36	1.622
4.37	1.621
4.38	1.620
4.39	1.619
4.40	1.618

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*Korosi, A. and Fabuss, B., Analytical Chemistry Vol 40 No. 1, Jan 1968 "Viscosity of Liquid Water from 25C to 150C", p. 162.

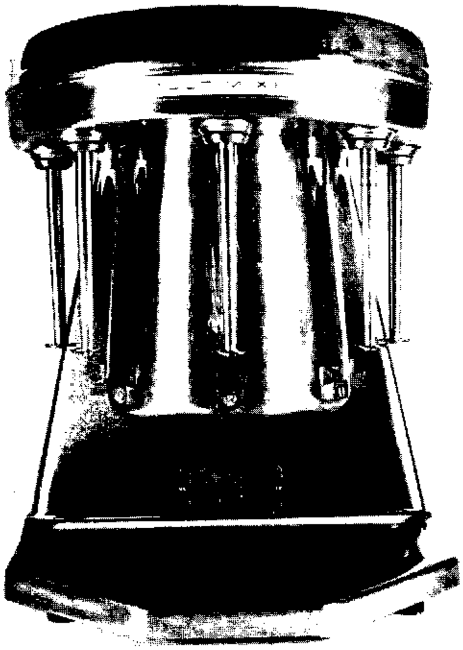

FIG. 2.1
MULTIMIXER MODEL 9B WITH
9B29X IMPELLER

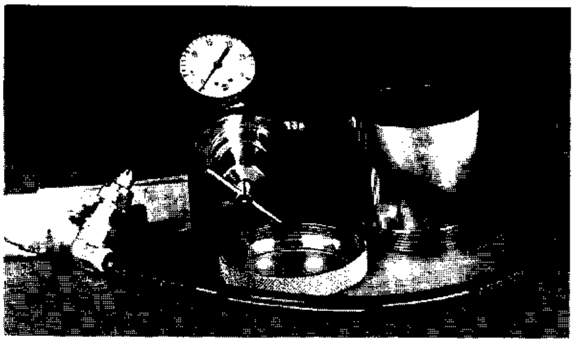

FIG. 2.2
SPRAY NOZZLE — TG 6.5 TIP
WITH 1/4 TT BODY

SECTION 3
HEMATITE

3.1 Description

a. Drilling grade hematite is produced from commercial ores, and may be a single ore or blends of hematite ores. The ores may be a straight mined product or processed material. Minor amounts of common accessory materials, other than the iron oxide (Fe_2O_3) mineral, include silicon oxide, aluminum oxide, calcium oxide, and magnesium oxide.

b. Drilling grade hematite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the chemical and physical requirements of Table 3.1, represents the product produced, and is controlled by the manufacturer.

TABLE 3.1
HEMATITE PHYSICAL AND CHEMICAL REQUIREMENTS

Requirement	Specification
Density	5.05 g/cm^3, minimum
Water Soluble Alkaline Earth Metals as Calcium	100 mg/kg, maximum
Residue greater than 75 micrometers	1.5 wt. percent, maximum
Residue greater than 45 micrometers	15 wt. percent, maximum
Particles less than 6 micrometers in equivalent spherical diameter	15 wt. percent, maximum

DENSITY

3.2 Equipment — Le Chatelier Flask Method

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

c. Le Chatelier flask: clamped or weighted to prevent flotation in water bath

d. Kerosene or mineral spirits

e. Transparent constant temperature bath: 90 ±1°F (32 ±0.5°C) regulated to ±0.1°C (e.g., 10-gallon aquarium with heater/circulator attachment, or functional equivalent)

f. Balance: precision of 0.01 g

g. Volumetric pipette: one 10-cm^3

h. Magnifying glass

i. Wooden dowel: approximately 5/16 inch (8 mm) in diameter and 12 inches (30 cm) in length, or a functional equivalent

j. Tissue paper

k. Low form weighing dish with spout: approximately 100-cm^3 capacity, or a functional equivalent

l. Fine bristle brush

3.3 Procedure

a. If required, equilibrate approximately 120 g of dried hematite to room temperature in the desiccator.

b. Fill a clean Le Chatelier flask to approximately 7/8 inch (22 mm) below the zero mark with kerosene.

c. Place the flask upright in the constant temperature bath. The level of water in the bath should be higher than the 24-cm^3 graduation of the flask, but below the stopper level. Assure flask is stabilized by use of clamps or weights.

d. Allow the flask and contents to equilibrate for a minimum of one hour. Using the magnifying glass with care to keep eyes at meniscus level, record the initial volume to the nearest 0.05 cm^3 (doubtful digit) without removing the flask from the constant temperature bath.

NOTE: *If kerosene level is above or below the -0.2 to +1.2 cm^3-volume range after equilibrating, use the 10-cm^3 pipette to add or remove kerosene in order for it to come within this range. Allow the flask to equilibrate at least one hour and record initial volume as in Par. 3.3d.*

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 kerosene in the flask.*

f. Weigh 100 ±0.01 g dried hematite into the weighing dish and carefully transfer to the Le Chatelier flask. Take care to avoid splashing of the kerosene or plugging of 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.

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 kerosene 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 degrees 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 one-half hour.

j. Remove the flask from the bath and repeat Par. 3.3h to remove any remaining air from the hematite sample.

k. Immerse the flask in the bath again for at least one hour.

l. Record the final volume in the same manner as described in Par. 3.3d.

3.4 Calculation

$$\text{Density, g/cm}^3 = \frac{\text{Weight sample, g}}{\text{Final vol., cm}^3 - \text{initial vol., cm}^3} \quad \text{(a)}$$

Record calculated density.

WATER SOLUBLE ALKALINE EARTH METALS AS CALCIUM

3.5 Equipment

a. Balance: capacity exceeding 100 g with a precision of 0.01 g

b. Erlenmeyer flask: 250-cm^3 nominal capacity, equipped with a tight fitting stopper

c. Graduated cylinder (TD): 100-cm^3 capacity with 1-cm^3 graduations

d. Aqueous EDTA solution: 3.72 ±0.01 g of disodium salt of ethylenediaminetetraacetic acid dihydrate [disodium salt of (ethylenedinitrilo) tetraacetic acid dihydrate] CAS #6381-92-6 diluted to a final volume of 1000 cm^3 with deionized water in a volumetric flask

e. Buffer solution: 67.5 ±0.1 g ammonium chloride (CAS #12125-02-9) and 570 ±1 cm^3 15N ammonium hydroxide (CAS #1336-21-6) solution diluted to a final volume of 1000 cm^3 with deionized water in a volumetric flask

f. Hardness indicator solution: 1 ±0.01 g Calmagite (CAS #3147-14-6) or equivalent [1-(1-hydroxy-4-methyl-phenylazo)-2-naphthol-4-sulfonic acid] diluted to a final volume of 1000 cm^3 with deionized water in a volumetric flask

g. Titration vessel: beaker of 100 to 150-cm^3 capacity

h. Graduated serological pipettes or burette. Graduations should be 0.1 cm^3.

i. Volumetric pipettes (TD), 10-cm^3 or equivalent

j. Deionized (or distilled) water

k. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, section 3.2, or filtration funnel

l. Filter paper: Whatman 50, or equivalent

m. Small glass container

n. Wrist action shaker, optional

o. Volumetric flask: one 1000-cm^3

p. Stirring rod

3.6 Procedure

a. Weigh 100 ±0.05 g of hematite. Transfer to the Erlenmeyer flask and add 100 ±1 cm^3 of deionized water. Stopper the flask and shake for at least five minutes during an approximate one hour interval or by an optional mechanical shaking apparatus for 20 to 30 minutes.

b. After shaking, 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 ±1 cm^3 deionized water to the titration vessel. Add about 2 cm^3 of hardness buffer and sufficient hardness indicator to achieve a distinct blue color. Swirl to mix.

NOTE: *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 cm^3 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 will develop if calcium and/or magnesium are present.

e. If hardness is present, begin stirring and titrate with EDTA solution to the blue end point. The end point of the titration is best described as the point at which additional EDTA produces no further red to blue change.

NOTE: *If end point is unclear or unobtainable, other tests shall be performed. Results and methodology of these tests will be recorded.*

3.7 Calculation

$$\text{Soluble Alkaline Earth Metals as Calcium, mg/kg} = 400\frac{\text{EDTA vol., cm}^3}{\text{Sample vol., cm}^3} - C_c{}^* \quad \text{(a)}$$

*where: C_c = calibration correction (Par. 12.9)

Record calculated value.

RESIDUE GREATER THAN 75 AND 45 MICROMETERS

3.8 Equipment

a. Oven regulated to 220 ±5°F (105 ±3°C)

b. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant or equivalent

c. Balance: precision of 0.01 g

d. Sodium hexametaphosphate (CAS #10124-56-8)

e. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

f. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D or equivalent)

g. A 75-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth.

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

h. A 45-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth.

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

i. Spray nozzle: 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body or equivalent) attached to water line with 90 degree elbow as shown in Fig. 2.2

j. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)

k. Evaporating dish, or functional equivalent

l. Wash bottle

3.9 Procedure

a. If required, equilibrate approximately 120 g of dried hematite to room temperature in the desiccator.

b. Weigh 50 ±0.01 g of dried hematite. Add the weighed sample to approximately 350 cm^3 of water containing about 0.2 g of sodium hexametaphosphate. Stir on the mixer for 5 ±1 minutes.

c. Transfer the sample to the 75-micrometer sieve. Use wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ± 7 kPa) from a spray nozzle for two minutes ±15 seconds. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve — move the spray of water repeatedly over the sample.

d. 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 weight. Record residue weight and total drying time.

f. Repeat steps Par. 3.9b through Par. 3.9e using the 45-micrometer sieve.

3.10 Calculation

$$\begin{array}{l}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{75 micrometers}\end{array} = 100\left(\frac{\text{Weight residue, g}}{\text{Weight sample, g}}\right) - C_c{}^{*} \qquad \text{(a)}$$

*where: C_c = calibration correction (Par. 12.5)

$$\begin{array}{l}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{45 micrometers}\end{array} = 100\left(\frac{\text{Weight residue, g}}{\text{Weight sample, g}}\right) - C_c{}^{*} \qquad \text{(b)}$$

*where: C_c = calibration correction (Par. 12.10)

Record calculated values for % greater than 45 and 75 micrometers.

PARTICLES LESS THAN 6 MICROMETERS IN EQUIVALENT SPHERICAL DIAMETER

3.11 Equipment — Sedimentation Method

a. Oven regulated to 220 ±5°F (105 ±3°C)

b. Desiccator, with calcium sulfate (CAS #7778-18-9) desiccant or equivalent

c. Balance: precision of 0.01 g

d. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

e. Container: Approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

f. Dispersant solution: 40 ±0.1 g sodium hexametaphosphate and 3.60 ±0.1 g anhydrous sodium carbonate (CAS #497-19-8) per 1000 cm^3 of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9.0.

g. Glass sedimentation cylinder: 18 inches (457 mm) in height and 2-1/2 inches (63 mm) in diameter, marked for a volume of 1000 cm^3 (ASTM D-422-63)

h. Rubber stopper: Number 13

i. Water bath or constant temperature room: capable of maintaining a convenient constant temperature at 75 ±10°F (24 ±5°C)

j. Thermometer: 60-90 ±0.1°F (16-32 ±0.5°C)

k. Hydrometer: ASTM 151H, graduated to read specific gravity of suspension

l. Timer: interval, mechanical or electrical, precision of 0.1 minutes

3.12 Procedure — Sedimentation Method

a. Weigh 80 ±0.1 g of dry hematite and place in mixing container.

b. Add 125 ±2 cm^3 of dispersant solution (from Par. 3.11f). Dilute to approximately 400 cm^3 with deionized water. Rinse all adhering particles from spatula into suspension.

c. Stir five ±0.5 minutes on mixer.

d. 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-cm^3 mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 ±5 seconds while holding a No. 13 rubber stopper in the top of the cylinder.

NOTE: *This is a critical step. Suspension must be homogeneous at start of sedimentation. This is difficult to obtain because of the high density of hematite.*

f. Set the cylinder into the water bath (or counter top of constant temperature room) and simultaneously start the timer. Hang the thermometer in the suspension.

g. Take hydrometer readings at intervals of 10, 20, 30, and 40 ±0.1 minutes (or until the first point below the 6 micrometer value is reached). To take a hydrometer reading, *carefully and slowly* lower the hydrometer to approximately the 1.020 reading before releasing. After hydrometer stabilizes, read the top of the meniscus at the prescribed time. *Carefully and slowly* remove hydrometer, rinse with deionized water, and dry after each reading. The hydrometer must be removed immediately after each reading to eliminate particle build-up on the shoulders which causes erroneous results. All hydrometer readings must be done with a minimum of fluid disturbance to preserve suspension settling equilibrium.

h. Record time, minutes (T), Temperature, °F or °C* (t) and hydrometer reading (H) on data sheet.

*NOTE: *Temperature may be measured in either °F or °C as long as all measurements and calculations are consistent in units, including hydrometer calibration.*

i. For each time interval, determine effective hydrometer depth (L) and water viscosity (v) from Table 3.3 and Table 3.2. Record on data sheet.

3.13 Calculation — Sedimentation procedure.

a. From hydrometer calibration (Par. 12.6) enter hydrometer correction slope (Mc) and hydrometer correction intercept (Bc) into data sheet.

b. Calculate sample constant (Ks) as follows (or determine from Table 3.4) and enter into data sheet:

$$Ks = \frac{100 \times D}{W\ (D-1)} \qquad \text{(a)}$$

where: Ks = Sample Constant

D = Sample density, g/cm^3

W = Sample weight, g

c. Calculate and enter onto data sheet the equivalent spherical diameter (de) for each time interval as follows:

$$de = 1.75 \sqrt{\frac{vL}{(D-1)T}} \qquad \text{(b)}$$

where: de = Equivalent spherical diameter, micrometers

v = Viscosity of water, cP

D = Density of sample, g/cm^3

T = Time, minutes

L = Effective depth, cm

d. Calculate and enter onto data sheet the cumulative percent finer for the equivalent particle diameter (de) immediately greater than 6 micrometers (SH) and the equivalent particle diameter (de) immediately less than 6 micrometers (SL) as follows:

$$S = Ks \times [(Mc \times t) - Bc + (H - 1) \times 1000] \qquad \text{(c)}$$

where: S = Cumulative percent finer than size

Ks = Sample constant

Mc = Hydrometer correction slope

*t = Suspension temperature, °F or °C (See note, Par. 3.12h)

Bc = Hydrometer correction intercept

H = Hydrometer reading.

e. Calculate and enter onto data sheet cumulative percent less than 6 micrometers (S6) as follows:

$$S6 = [\frac{(SH - SL)}{(dH - -dL)}(6 - dL)] + SL \qquad \text{(d)}$$

where: S6 = Cumulative percent less than 6 micrometers

SH = Cumulative percent for point immediately greater than 6 micrometers

SL = Cumulative percent for point immediately less than 6 micrometers

dH = Equivalent particle diameter immediately greater than 6 micrometers

dL = Equivalent particle diameter immediately less than 6 micrometers

Example Data Sheet

Hydrometer Serial #	XXXXX
Mc	0.144
Bc	13.08
Ks	1.559
Sample Density (D)	5.05 g/cm^3
Sample Weight (W)	80 g

Time (T)	Temp (°F) (t)	Hyd. Rdg. (H)	Water Vis. (v)	Effective Depth (L)	Particle Diameter (de)	%Finer (S)	%Finer (S6)
10	78	1.0130	.8792	12.9	9.3	17.3	
20	77	1.0100	.8904	13.7	6.8	*13.0	
30	77	1.0080	.8904	14.2	5.7	9.3	
							**10.3%

Example Calculation:

Sample Constant, Ks from (a)

$$Ks = \frac{(100 \text{ X } 5.05)}{(80 \text{ X } (5.05-1))} = 1.559$$

Example Calculation for 20 minute reading:

a. Equivalent diameter, de, from (b)

$$de = 17.5 \sqrt{\frac{0.8904 \text{ x } 13.7}{(5.05-1) \text{ x } 20}} = 6.8 \text{ micrometers}$$

b. Percent finer, S, from (c)

$$* S = 1.559 \text{ X } [(0.1444 \text{ X } 77) - 13.08 + (1.010-1 \text{ X } 1000)]$$

$$* S = 1.559 \text{ X } (-13.08 + 10.0) = 13.0\%$$

Example Calculation for percent less than 6 micrometers, S6, from (d)

SH = 13.0
SL = 9.3
dH = 6.8
dL = 5.7

$$** \; S6 = [\frac{(13.0-9.3)}{(6.8-5.7)} \text{ x } (6.0-5.7)] + 9.3 = 10.3\%$$

TABLE 3.2
VISCOSITY OF WATER AT VARIOUS TEMPERATURES

Temperature °F	(°C)	Viscosity cP or mPa·S	Temperature °F	(°C)	Viscosity cP or mPa.S
60	15.6	1.1211	72	22.2	0.9498
61	16.1	1.1050	73	22.8	0.9374
62	16.7	1.0893	74	23.3	0.9253
63	17.2	1.0739	75	23.9	0.9134
64	17.8	1.0589	76	24.4	0.9018
65	18.3	1.0442	77	25.0	0.8904
66	18.9	1.0298	78	25.6	0.8792
67	19.4	1.0158	79	26.1	0.8683
68	20.0	1.0020	80	26.7	0.8576
69	20.6	0.9885	81	27.2	0.8470
70	21.1	0.9753	82	27.8	0.8367
71	21.7	0.9624	83	28.3	0.8266

$$*\text{LOG}(V20/Vt) = \frac{[1.37023(t-20)+0.000836(t-20)^2]}{(109+t)}$$

where: t = TEMP IN DEGREES CELSIUS(C)
V20 = 1.002
Vt = VIS. AT DESIRED TEMPERATURE

*Korosi, A. and Fabuss, B., Analytical Chemistry Vol 40 No. 1, Jan 1968 "Viscosity of Liquid Water from 25C to 150C", p. 162.

TABLE 3.3
VALUES OF EFFECTIVE DEPTH BASED ON READINGS ON HYDROMETER NO. 151H USED IN SPECIFIC SEDIMENTATION CYLINDER

Uncorrected Hydrometer Reading	Effective Depth L, cm	Uncorrected Hydrometer Reading	Effective Depth L, 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 3.4
SAMPLE CONSTANT (Ks) FOR 80.0g SAMPLE WT.

Sample Density	Sample Constant
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

SECTION 4
BENTONITE

4.1 Description

a. Drilling grade bentonite is a naturally occurring clay containing the clay mineral smectite. It may also contain accessory minerals such as quartz, mica, feldspar, and calcite.

b. Drilling grade bentonite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 4.1, represents the product produced, and is controlled by the manufacturer.

TABLE 4.1
BENTONITE PHYSICAL REQUIREMENTS

Requirement	Specification
Suspension Properties	
Viscometer Dial Reading at 600 rpm	30, minimum
Yield Point/Plastic Viscosity Ratio	3, maximum
Filtrate Volume	15.0 cm^3, maximum
Residue greater than 75 micrometers	4.0 wt. percent, maximum
Moisture	10.0 wt. percent, maximum

SUSPENSION PROPERTIES

4.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of ±0.01 g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

e. Spatula

f. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

g. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, section 3.2

h. Graduated cylinder (TD): 500 ±5 cm^3

i. Deionized or distilled water

j. Container: about 500-cm^3 with lid

k. Timers: two interval, mechanical or electrical, precision of ±0.1 minute

l. Graduated cylinder (TC): 10 ±0.1 cm^3

4.3 Procedure — 600 rpm Reading, Plastic Viscosity, Yield Point

a. Prepare a suspension of the bentonite. Add 22.5 ±0.01 g of clay (as received) to 350 ±5 cm^3 of deionized water while stirring on the mixer.

b. After stirring five ±0.5 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any bentonite adhering to container walls. Be sure all bentonite clinging to the spatula is incorporated into the suspension.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

d. Age the bentonite suspension up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

e. After aging bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 ±0.5 minutes.

f. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C).

4.4 Calculation — Plastic Viscosity and Yield Point

Plastic Viscosity, cP =
[600 rpm reading] - [300 rpm reading] (a)

Yield Point, lb/100 ft^2 =
[300 rpm reading] - Plastic Viscosity (b)

Yield Point/Plastic viscosity ratio =
Yield point [Eq. 4.4(b)] ÷
Plastic viscosity [Eq. 4.4(a)] (c)

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

4.5 Procedure — Filtrate Volume

a. Recombine all of the suspension as prepared and tested in Par. 4.3 and stir in container for 1 ±0.5 minute on the mixer. Adjust suspension temperature to 77 ±2°F (25 ±1°C).

b. Pour the suspension into filter press cell. Before adding suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. Pour the mud to within about 1/2 inch (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place filter cell in frame and close relief valve. Place a container under the drain tube.

c. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on cell at 100 ±5 psi (690 ±35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen, or helium.

d. At 7.5 ±0.1 minutes on the first timer remove the container and any adhering liquid on the drain tube and discard. Place the dry 10-cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

4.6 Calculation — Filtrate Volume

Calculate the filtrate volume of the clay suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c \qquad \text{(a)}$$

where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated filtrate volume.

RESIDUE GREATER THAN 75 MICROMETERS

4.7 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Sodium hexametaphosphate (CAS #10124-56-8)

c. Balance: precision of ±0.01 g

d. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

e. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

f. Spatula

g. A 75-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth.

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

h. Spray nozzle: 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body or equivalent) attached to water line with 90 degree elbow as shown in Fig. 2.2

i. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)

j. Evaporating dish

k. Wash bottle

4.8 Procedure

a. Weigh 10 ±0.01 g of bentonite.

b. Add the weighed bentonite sample to approximately 350 cm^3 of water containing about 0.2 g of sodium hexameta-phosphate while stirring on the mixer.

c. Stir suspension on the mixer for 30 ±1 minutes.

d. Transfer the sample to the sieve. Use the wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ±7 kPa) from the spray nozzle for two minutes ±15 seconds. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve — 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 weight. Record residue weight and total drying time.

4.9 Calculation

$$\begin{array}{l}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{75 micrometers}\end{array} = 100\frac{\text{(Weight residue, g)}}{\text{(Weight sample, g)}} - C_c{}^*$$

*where: C_c = calibration correction (Par. 12.5)

Record calculated value.

MOISTURE

4.10 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Balance: precision of ±0.01 g

c. Evaporating dish

d. Spatula

e. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

4.11 Procedure

a. Weigh 10 ±0.01 g of bentonite sample into a tared evaporating dish.

b. Dry sample in the oven to a constant weight. Record total drying time.

c. Cool to room temperature in desiccator.

d. Reweigh the evaporating dish containing the dry bentonite. Record weight.

4.12 Calculation

$$M = 100\,\frac{(W_1 - W_2)}{W_1} \qquad \text{(a)}$$

where: M = moisture, wt. percent

W_1 = original sample weight, g

W_2 = dry sample weight, g

Record calculated value.

SECTION 5
NONTREATED BENTONITE

5.1 Description

a. Drilling grade nontreated bentonite is dried and ground, but not chemically-treated clay, composed principally of the mineral smectite. It may also contain accessory minerals such as quartz, mica, feldspar, and calcite.

b. Drilling grade nontreated bentonite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 5.1, represents the product produced, and is controlled by the manufacturer.

TABLE 5.1
NONTREATED BENTONITE
PHYSICAL REQUIREMENT

Requirement	Specification
Suspension Properties	
Yield Point/Plastic Viscosity Ratio	1.5, maximum
Dispersed Plastic Viscosity	10 cP, minimum
Dispersed Filtrate Volume	12.5 cm^3, maximum

SUSPENSION PROPERTIES

5.2 Equipment

a. Balance: precision of 0.01 g

b. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

c. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

d. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

e. Filter press: as referenced in API RP 13B-1, 1st edition, June 1, 1990, Par. 3.2

f. Spatula

g. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

h. Deionized or distilled water

i. Graduated cylinder (TD): 500 ±5 cm^3

j. Container: about 500-cm^3 with lid

k. Syringe or pipette: 5 ±0.01 cm^3

l. Sodium hexametaphosphate (CAS #10124-56-8) solution: 10 ±0.5% by wt

m. Timers: two interval, mechanical or electrical, precision of 0.1 minute

n. Graduated cylinder (TC): 10 ±0.01 cm^3

5.3 Procedure — Yield Point/Plastic Viscosity Ratio

a. Prepare a suspension of nontreated bentonite. Add 25 ±0.01 g of clay (as received) to 350 ±5 cm^3 deionized water while stirring on the mixer.

b. After stirring 5 ±0.5 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any bentonite adhering to container walls. Be sure all bentonite clinging to the spatula is incorporated into the suspension.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

d. Age the bentonite suspension up to 16 hours in a sealed container at room temperature. Record storage temperature and storage duration.

e. After aging bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 ±0.5 minutes.

f. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C).

5.4 Calculation — Plastic Viscosity and Yield Point

Plastic Viscosity, cP =
[600 rpm reading] -[300 rpm reading] (a)

Yield Point, lb/100 ft^2 = (b)
[300 rpm reading] - Plastic Viscosity

Yield Point/Plastic viscosity ratio =
Yield point [Eq. 5.4(b)] ÷
Plastic viscosity [Eq. 5.4(a)] (c)

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

5.5 Procedure — Dispersed Plastic Viscosity

a. Recombine all of the bentonite suspension as prepared and tested in Par. 5.3 and stir in container for 1 ±0.5 minute on the mixer.

b. Add 5 ±0.1 cm^3 of a 10% solution of sodium hexameta-phosphate to the suspension and stir for 3 ±0.5 minutes on the mixer.

c. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C).

d. Calculate and record the dispersed plastic viscosity as per Par. 5.4a.

5.6 Procedure — Dispersed Filtrate Volume

a. Recombine all of the suspension as prepared and tested in Par. 5.5 and stir in container for 1 ±0.5 minute on the mixer. Adjust suspension temperature to 77 ±2°F (25 ±1°C).

b. Pour the suspension into filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. Pour the suspension to within about 1/2 inch (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place filter cell in frame and close relief valve. Place a container under the drain tube.

c. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on cell at 100 ±5 psi (690 ±35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen, or helium.

d. At 7.5 ±0.1 minutes on the first timer remove the container and any adhering liquid on the drain tube and discard. Place dry 10-cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

5.7 Calculation — Dispersed Filtrate Volume

Calculate the dispersed filtrate volume of the clay suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c \qquad \text{(a)}$$

where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated filtrate volume.

SECTION 6
OCMA GRADE BENTONITE

6.1 Description

a. OCMA grade bentonite is a montmorillonite-based clay which by nature of its source cannot meet all aspects of API Specification 13A, Section 4. This bentonite may have been treated with soda ash, polymer, or other chemicals to improve suspension property performance.

b. OCMA grade bentonite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 6.1, represents the product produced, and is controlled by the manufacturer.

c. Manufacturers and licensees shall provide appropriate markings on the container in block letters at least 1/4 inch (6mm) in height below the name of the material, the type of treatment of the bentonite with polymer, soda ash or other material.

TABLE 6.1
OCMA GRADE BENTONITE
PHYSICAL REQUIREMENTS

Requirements	Specification
Suspension Properties	
Viscometer Dial Reading at 600 rpm	30, minimum
Yield Point/Plastic Viscosity Ratio	6, maximum
Filtrate Volume	16.0 cm^3, maximum
Residue greater than 75 micrometers	2.5 wt. percent, maximum
Moisture	13.0 wt. percent, maximum

SUSPENSION PROPERTIES

6.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of 0.01 g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

e. Spatula

f. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

g. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 3.2

h. Graduated cylinder (TD): 500 ±5 cm^3

i. Deionized (or distilled) water

j. Sealed container: about 500-cm^3 with lid

k. Timers: two interval, mechanical or electrical, precision of 0.1 minute

l. Graduated cylinder (TD): 10 ±0.1 cm^3

6.3 Procedure — 600 rpm Dial Reading, Plastic Viscosity, Yield Point

a. Prepare a suspension of OCMA grade bentonite. Add 22.5 ±0.01 g clay (as received) to 350 ±5 cm^3 of deionized water while stirring on the mixer.

b. After stirring five ±0.5 minutes, remove container from mixer and scrape its sides with the spatula to dislodge any bentonite adhering to container walls. Be sure all bentonite clinging to the spatula is incorporated into the suspension.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

d. Age the bentonite suspension up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

e. After aging bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 ±0.5 minutes.

f. Pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial readings at 600 and 300 rpm rotor speed settings of the viscometer shall be recorded when a constant value for each rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C).

6.4 Calculation — Plastic Viscosity and Yield Point

$$\text{Plastic Viscosity, cP} = [600\text{ rpm reading}] - [300\text{ rpm reading}] \quad \text{(a)}$$

$$\text{Yield Point, lb/100 ft}^2 = [300\text{ rpm reading}] - \text{Plastic viscosity} \quad \text{(b)}$$

$$\text{Yield Point/Plastic Viscosity ratio} = \text{Yield point [eq. 6.4(b)]} \div \text{Plastic viscosity [Eq. 6.4(a)]} \quad \text{(c)}$$

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

6.5 Procedure — Filtrate Volume

a. Recombine all of the suspension as prepared and tested in Par. 6.3 and stir in container for 1 ±0.5 minute on the mixer. Adjust suspension temperature to 77 ±2°F (25 ±1°C).

b. Pour the suspension into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. Pour the suspension to within about 1/2 inch (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place filter cell in frame and close relief valve. Place a container under the drain tube.

c. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on cell at 100 ±5 psi (690 ±35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen, or helium.

d. At 7.5 ±0.1 minutes on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place a dry 10-cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

6.6 Calculation — Filtrate Volume

Calculate the filtrate volume of the clay suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c \qquad \text{(a)}$$

where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated filtrate volume.

RESIDUE GREATER THAN 75 MICROMETERS

6.7 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Sodium hexametaphosphate (CAS #10124-56-8)

c. Balance: precision of 0.01 g

d. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

e. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

f. Spatula

g. A 75-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth.

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

h. Spray nozzle: 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body or equivalent) attached to water line with 90 degree elbow as shown in Fig. 2.2

i. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)

j. Evaporating dish

k. Wash bottle

6.8 Procedure

a. Weigh 10 ±0.01 g of bentonite.

b. Add the weighed sample to approximately 350 cm^3 of water containing about 0.2 g of sodium hexametaphosphate while stirring on the mixer.

c. Stir suspension on the mixer for 30 ±1 minutes.

d. Transfer the sample to sieve. Use the wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ±7 kPa) from a spray nozzle for two minutes ±15 seconds. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve — 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 weight. Record residue weight and total drying time.

6.9 Calculation

$$\begin{array}{c}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{75 micrometers}\end{array} = 100\,\frac{\text{Weight residue, g}}{\text{Weight sample, g}} - C_c{}^*$$

*where: C_c = calibration correction from Par. 12.5

Record calculated value.

MOISTURE

6.10 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Balance: precision of ±0.01 g

c. Evaporating dish

d. Spatula

e. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

6.11 Procedure

a. Weigh 10 ±0.01 g of bentonite sample into a tared evaporating dish.

b. Dry sample in the oven to a constant weight. Record total drying time.

c. Cool to room temperature in a desiccator.

d. Reweigh the evaporating dish containing the dry bentonite. Record weight.

6.12 Calculation

$$M = 100\frac{(W_1 - W_2)}{W_1} \qquad \text{(a)}$$

where: M = Moisture, wt. percent

W_1 = Original sample weight, g

W_2 = Dry sample weight, g

Record calculated value.

SECTION 7
ATTAPULGITE

7.1 Description

a. Drilling grade attapulgite clay is a naturally occurring clay mineral. Accessory minerals include quartz, feldspar, and calcite.

b. Drilling grade attapulgite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 7.1, represents the product produced, and is controlled by the manufacturer.

TABLE 7.1
ATTAPULGITE PHYSICAL REQUIREMENTS

Requirement	Specification
Suspension Properties	
Viscometer Dial Reading at 600 rpm	30, minimum
Residue greater than 75 micrometers	8.0 wt. percent, maximum
Moisture	16.0 wt. percent, maximum

SUSPENSION PROPERTIES

7.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of 0.01 g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

e. Sodium chloride (CAS #7647-14-5)

f. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

g. Spatula

h. Graduated cylinder (TD): 500 ±5 cm^3

i. Deionized or distilled water

j. Defoamer

k. Container: glass or plastic with stopper or lid for salt solution

l. Graduated cylinder (TD): 100 ±1 cm^3

m. Filter paper

n. Timer: interval, mechanical or electrical, precision of 0.1 minute

o. Funnel

7.3 Procedure — 600 rpm Dial Reading

a. Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 40 to 45 g of sodium chloride per 100 ±1 cm^3 of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter into a storage container.

b. Prepare a suspension of attapulgite. Add 20 ±0.01 g (as received) to 350 ±5 cm^3 saturated salt solution while stirring on the mixer.

c. After stirring 5 ±0.5 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any attapulgite adhering to container walls. Be sure all attapulgite clinging to the spatula is incorporated into the suspension.

d. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

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 spatula to break surface froth. Place the viscometer cup on direct indicating viscometer. The dial reading at 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value for 600 rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F. (25 ±1°C).

RESIDUE GREATER THAN 75 MICROMETERS

7.4 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Sodium hexametaphosphate (CAS #10124-56-8)

c. Balance: precision of ±0.01 g

d. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

e. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

f. Spatula

g. A 75-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

h. Spray nozzle: 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body or equivalent) attached to water line with 90 degree elbow as shown in Fig. 2.2

i. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)

j. Evaporating dish

k. Wash bottle

7.5 Procedure

a. Weigh 10 ±0.01 g of attapulgite.

b. Add the weighed attapulgite sample to approximately 350 cm^3 of water containing about 0.2 g of sodium hexameta-phosphate.

c. Stir suspension on the mixer for 30 ±1 minutes.

d. Transfer the sample to the sieve. Use the wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ±7 kPa) from the spray nozzle for two minutes ±15 seconds. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve — 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 until weight repeats within 0.01 g. Record weight.

7.6 Calculation

$$\begin{array}{l}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{75 micrometers}\end{array} = 100\,\frac{\text{Weight of residue, g}}{\text{Weight of sample, g}}$$

Record calculated value.

MOISTURE

7.7 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Balance: precision of ±0.01 g

c. Evaporating dish

d. Spatula

e. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

7.8 Procedure

a. Weigh 10 ±0.01 g of attapulgite sample into a tared evaporating dish.

b. Dry sample in the oven to a constant weight. Record total drying time.

c. Cool to room temperature in desiccator.

d. Reweigh the evaporating dish containing the dry attapulgite. Record weight.

7.9 Calculation

$$M = 100\frac{(W_1 - W_2)}{W_1} \qquad \text{(a)}$$

where: M = Moisture, wt. percent

W_1 = Original sample weight, g

W_2 = Dry sample weight, g

Record calculated value.

SECTION 8
SEPIOLITE

8.1 Description

a. Drilling grade sepiolite is a naturally occurring clay mineral. Accessory minerals include quartz, feldspar, and calcite.

b. Drilling grade sepiolite shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 8.1, represents the product produced, and is controlled by the manufacturer.

TABLE 8.1
SEPIOLITE PHYSICAL REQUIREMENTS

Requirement	Specification
Suspension Properties	
Viscometer Dial Reading at 600 rpm	30, minimum
Residue greater than 75 micrometers	8.0 wt. percent, maximum
Moisture	16.0 wt. percent, maximum

SUSPENSION PROPERTIES

8.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of 0.01 g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

e. Sodium chloride (CAS #7647-14-5)

f. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, section 2.4

g. Spatula

h. Graduated cylinder (TD): 500 ±5 cm^3

i. Deionized or distilled water

j. Defoamer

k. Container: glass or plastic stopper or lid for salt solution

l. Graduated cylinder (TD): 100 ±1 cm^3

m. Filter paper

n. Timer: interval, mechanical or electrical, precision of ±0.1 minute

o. Funnel

8.3 Procedure — 600 rpm Dial Reading

a. Prepare an ample volume saturated salt solution by thoroughly mixing in a suitable container 40 to 45 g of sodium chloride per 100 ±1 cm^3 of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

b. Prepare a suspension of sepiolite. Add 20 ±0.01 g (as received) to 350 ±5 cm^3 of saturated salt solution while stirring on the mixer.

c. After stirring 5 ±0.5 minutes, remove the container from mixer and scrape its sides with the spatula to remove or dislodge any sepiolite adhering to container walls. Be sure all sepiolite clinging to the spatula is incorporated into the suspension.

d. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

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 spatula to break surface froth. Place the viscometer cup on direct indicating viscometer. The dial reading at 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value for 600 rpm is reached. Readings shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C).

RESIDUE GREATER THAN 75 MICROMETERS

8.4 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Sodium hexametaphosphate (CAS #10124-56-8)

c. Balance: precision of ±0.01 g

d. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

e. Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

f. Spatula

g. A 75-micrometer sieve conforming* to the requirements of ASTM E11 Standard Specifications for Wire-Cloth Sieves for Testing Purposes, latest edition. Approximate dimensions are 3 in. (76mm) diameter, and 2.5 in. (69mm) from top of frame to wire cloth

*NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

h. Spray nozzle: 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with 1/4 TT body or equivalent) attached to water line with 90 degree elbow as shown in Fig. 2.2

i. Water pressure regulator capable of regulation to 10 ±1 psi (69 ±7 kPa)

j. Evaporating dish

k. Wash bottle

8.5 Procedure

a. Weigh 10 ±0.01 g of sepiolite.

b. Add the weighed sepiolite sample to approximately 350 cm^3 of water containing about 0.2 g of sodium hexametaphosphate.

c. Stir suspension on the mixer for 30 ±1 minutes.

d. Transfer the sample to the sieve. Use the wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 10 ±1 psi (69 ±7 kPa) from the spray nozzle for two minutes ±15 seconds. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve — 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 until weight repeats within 0.01 g. Record weight.

8.6 Calculation

$$\begin{array}{l}\text{Residue, wt.}\\ \text{\% greater than}\\ \text{75 micrometers}\end{array} = 100\left[\frac{\text{Weight of residue, g}}{\text{Weight of sample, g}}\right]$$

Record calculated value.

MOISTURE

8.7 Equipment

a. Oven: regulated to 220 ±5°F (105 ±3°C)

b. Balance: precision of ±0.01 g

c. Evaporating dish

d. Spatula

e. Desiccator with calcium sulfate (CAS #7778-18-9) desiccant, or equivalent

8.8 Procedure

a. Weigh 10 ±0.01 g of sepiolite sample into a tared evaporating dish.

b. Dry sample in the oven to a constant weight. Record total drying time.

c. Cool to room temperature in desiccator.

d. Reweigh the evaporating dish containing the dry sepiolite. Record weight.

8.9 Calculation

$$M = 100\,\frac{(W_1 - W_2)}{W_1} \qquad \text{(a)}$$

where: M = Moisture, wt. percent

W_1 = Original sample weight, g

W_2 = Dry sample weight, g

Record calculated value.

SECTION 9
TECHNICAL-GRADE LOW-VISCOSITY CMC (CMC-LTV)

9.1 Description

a. Technical-grade low-viscosity carboxymethylcellulose (CMC-LVT), an alkali metal salt of carboxymethylcellulose that is chemically modified to obtain a water soluble polymer. The manufacturer shall maintain documentation of analysis of the cellulosic raw material used.

b. The product obtained is a free-flowing or granulated powder and is not normally purified of byproducts formed in the reaction. It is known as CMC-LVT.

c. CMC-LVT shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 9.1, represents the product produced, and is controlled by the manufacturer.

TABLE 9.1
CMC-LVT
PHYSICAL REQUIREMENTS

Requirement	Specification
Solution Properties	
Viscometer Dial Reading at 600 rpm	90, maximum
Filtrate Volume	10 cm^3, maximum

SOLUTION PROPERTIES

9.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of 0.01 g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Mixer Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

e. Spatula

f. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

g. Deionized (or distilled) water

h. Timers: two interval, mechanical or electrical, precision of 0.1 minute

i. Sealed container: about 500-cm^3 with lid

j. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 3.2

k. Graduated cylinders: one 10 ±0.1 cm^3 (TC), one 100 ±1 cm^3 (TD), and one 500 ±5 cm^3 (TD)

l. pH meter (optional): precision of 0.1 pH unit

m. Container, glass or plastic, with stopper or lid for salt solutions

n. API Standard Evaluation Base Clay (see Par. 1.4)

o. Sodium chloride: (CAS #7647-14-5)

p. Sodium bicarbonate: (CAS #144-55-8)

9.3 Procedure — 600 rpm Dial Reading

a. Prepare a solution of CMC-LVT. Add 10.5 ±0.01 g CMC-LVT to 350 ±5 cm^3 of deionized water at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

NOTE: *CMC-LVT shall be added away from impeller shaft to minimize dusting.*

b. After stirring 5 ±0.1 minutes, remove container from mixer and scrape its sides with the spatula to remove or dislodge any CMC-LVT adhering to container walls. Be sure all CMC-LVT clinging to the spatula is incorporated into the solution.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-LVT clinging to container walls after another 5 and 10 minutes. Total mixing time shall equal 20 ±1 minutes.

d. Age the solution for 2 hours ±5 minutes in a sealed or covered container at room temperature. Record storage temperature.

e. After aging, stir the solution on the mixer for 5 ±0.1 minutes.

f. Pour the solution into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value at 600 rpm is reached. The reading shall be taken at a solution test temperature of 77 ±2°F (25 ±1°C).

9.4 Procedure — Filtrate Volume

a. Prepare an ample volume of saturated salt solution by thoroughly mixing in a suitable container 40 to 45 g of sodium chloride per 100 ±1 cm^3 of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

b. Prepare a clay-base suspension by adding 350 ±5 cm^3 of the saturated salt solution to the mixer container. Add 1.0 ±0.1 g of sodium bicarbonate and stir on the mixer for about 1 minute.

c. Slowly add 35.0 ±0.1 g of API Standard Evaluation Base Clay while stirring on the mixer.

d. After stirring 5 ±0.1 minutes, remove container from mixer and scrape its sides with the spatula to dislodge any clay adhering to container walls. Be sure all clay clinging to the spatula is incorporated into the suspension.

e. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the clay shall equal 20 ±1 minutes.

f. Add 3.15 ±0.01 g (9.01 ±0.03 g/L) of CMC-LVT to the suspension while stirring on the mixer, adding at a uniform rate over about 60 seconds.

g. After stirring 5 ±0.1 minutes, remove the container from the mixer and scrape its sides with the spatula to dislodge any CMC-LVT adhering to container walls. Be sure all material clinging to the spatula is incorporated into the suspension.

h. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-LVT clinging to the container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

i. Age the suspension for 2 hours ±5 minutes in a sealed or covered container at room temperature. Record storage temperature.

j. After aging, stir the suspension on the mixer for 5 ±0.1 minutes.

k. Immediately pour CMC-LVT-treated suspension into a filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. The temperature of the suspension shall be 77 ±2°F (25 ±1°C). Pour the suspension to within about 1/2 (13 mm) of the top of the cell. Complete 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.

l. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on the cell to 100 ±5 psi (690 ±35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen or helium.

m. At 7.5 ±0.1 minutes on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place a dry 10-cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

9.5 Calculation — Filtrate Volume

Calculate the filtrate volume of the CMC-LVT-treated suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c \qquad \text{(a)}$$

where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated value.

SECTION 10
TECHNICAL-GRADE HIGH-VISCOSITY CMC (CMC-HVT)

10.1 Description

a. Technical-grade high-viscosity carboxymethyl cellulose (CMC-HVT) (an alkali metal salt of carboxymethylcellulose) is cellulose that is modified chemically to obtain a water-soluble polymer. The manufacturer shall maintain documentation of analysis of the cellulosic raw material used.

b. The product obtained is a free-flowing or granulated powder and is not normally purified of byproducts formed in the reaction. It is known as CMC-HVT.

c. CMC-HVT shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 10.1, represents the product produced, and is controlled by the manufacturer.

TABLE 10.1
CMC-HVT PHYSICAL REQUIREMENTS

Requirement	Specification
Solution Properties	
Viscometer Dial Reading at 600 rpm	
in Deionized Water	30, minimum
in 40 g/L Salt Water	30, minimum
in Saturated Salt Water	30, minimum
Filtrate Volume	10 cm^3, maximum

SOLUTION PROPERTIES

10.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of 0.01g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Mixer Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)

e. Spatula

f. Container: glass or plastic with stopper or lid for salt solutions

g. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

h. Deionized (or distilled) water

i. Sealed container: about 500-cm^3 with lid

j. Sodium chloride: (CAS #7647-14-5)

k. 1000-cm^3 volumetric flask

l. Defoamer

m. Timers: two interval, mechanical or electrical, precision to 0.1 minute

n. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 3.2

o. Graduated cylinders (TD): one 10 ±0.1 cm^3, one 100 ±1 cm^3, and one 500 ±5 cm^3

p. pH meter: precision of 0.1 pH unit

q. API Standard Evaluation Base Clay (see Par. 1.4)

r. Sodium bicarbonate: (CAS #144-55-8)

10.3 Procedure — Deionized Water Test

a. Prepare a solution of CMC-HVT. Add 2.20 ±0.01 g (6.29 ±0.03 g/L) of CMC-HVT to 350 ±5 cm^3 of deionized water at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

NOTE: *CMC-HVT shall be added away from impeller shaft to minimize dusting.*

b. After stirring 5 ±0.1 minutes, remove 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 all CMC-HVT clinging to the spatula is incorporated into the solution.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT after another 5 and 10 minutes. Total mixing time shall equal 20 ±1 minutes.

d. Age the solution for up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

e. After aging, stir the solution on the mixer for 5 ±0.1 minutes.

f. Pour the solution into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value at 600 rpm is reached. The 600 rpm dial reading shall be taken at a solution test temperature of 77 ±2°F (25 ±1°C).

10.4 Procedure — 40 g/L Salt Water Test

a. Prepare a 40 g/L salt solution by adding 40 ±0.1 g of sodium chloride to a 1000-cm^3 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 ±0.01 g (7.72 ±0.03 g/L) of CMC-HVT to 350 ±5 cm^3 of the 40 g/L salt solution at a uniform rate over a time interval of about 60 seconds while stirring on the mixer. Add defoamer if necessary.

c. After stirring 5 ±0.1 minutes, remove container from the mixer and scrape its sides with the spatula to dislodge any CMC-HVT adhering to the container walls. Be sure all CMC-HVT clinging to the spatula is incorporated into the solution.

d. Replace the container on mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT after another 5 and 10 minutes. Total mixing time shall equal 20 ±1 minutes.

e. Age the solution for up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

f. After aging, stir the solution on the mixer for 5 ±0.1 minutes.

g. Pour the solution into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value at 600 rpm is reached. The 600 rpm dial reading shall be taken at a solution test temperature of 77 ±2°F (25 ±1°C).

10.5 Procedure — Saturated Salt Water Test

a. Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container to 45 g of sodium chloride per 100 ± 1 cm^3 of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

b. Prepare a solution of CMC-HVT.* Add 2.50 ±0.01 g (7.15 ±0.03 g/L) CMC-HVT to 350 ±5 cm^3 of saturated salt water at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

c. After stirring 5 ±0.1 minutes, remove container from mixer and scrape its sides with the spatula to dislodge any CMC-HVT adhering to the container walls. Be sure any CMC-HVT clinging to the spatula is incorporated into the solution.

d. Replace the container on mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT after another 5 and 10 minutes. Total mixing time shall equal 20 ±1 minutes.

e. Age the solution for up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

f. After aging, stir the solution on the mixer for 5 ±0.1 minutes.

g. Immediately pour the solution into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value for 600 rpm is reached. The 600 rpm dial reading shall be taken at a solution test temperature of 77 ±2°F (25 ±1°C).

10.6 Procedure — Filtrate Volume

a. Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 40 to 45 g sodium chloride per 100 ±1 cm^3 of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

b. Prepare a clay-base suspension by adding 350 ±5 cm^3 of the saturated salt solution to a mixer container. Add 1.0 ±0.1 g of sodium bicarbonate and stir on the mixer for about one minute.

c. Slowly add 35.0 ±0.1 g of API Standard Evaluation Base Clay while stirring on the mixer.

d. After stirring 5 ±0.1 minutes, remove container from mixer and scrape its sides with the spatula to dislodge any clay adhering to container walls. Be sure all clay clinging to the spatula is incorporated into the suspension.

e. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the clay shall equal 20 ±1 minutes.

f. Add 3.15 ±0.01 g (9.01 ±0.03 g/L) of CMC-HVT to the suspension while stirring on the mixer, adding at a uniform rate over about 60 seconds.

g. After stirring 5 ±0.1 minutes, remove the container from the mixer and scrape its sides with the spatula to dislodge any CMC-HVT adhering to container walls. Be sure all material clinging to the spatula is incorporated into the suspension.

h. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ±1 minutes.

i. Age the suspension for 2 hours ±5 minutes in a sealed or covered container at room temperature. Record storage temperature.

j. After aging, stir the suspension on the mixer for 5 ±0.1 minutes.

k. Immediately pour the CMC-HVT-treated suspension into a filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. The temperature of the suspension shall be 77 ±2°F (25 ±1°C). Pour the suspension to within about 1/2 inch (13 mm) of the top of the cell. Complete 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.

l. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on the cell to 100 ±5 psi (690 ±35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen or helium.

m. At 7.5 ±0.1 minutes on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place a dry 10 cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

10.7 Calculation — Filtrate Volume

Calculate the filtrate volume of the CMC-HVT-treated suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c{}^* \qquad \text{(a)}$$

*where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated value.

SECTION 11
STARCH

11.1 Description

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

b. Drilling grade starch should be free-flowing and free from lumps. Drilling grade starch shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 11.1, represents the product produced, and is controlled by the manufacturer.

TABLE 11.1
STARCH PHYSICAL REQUIREMENTS

Requirement	Specification
Suspension Properties	
Viscometer Dial Reading at 600 rpm	
in 40 g/L Salt Water	18, maximum
in Saturated Salt Water	20, maximum
Filtrate Volume	
in 40 g/L Salt Water	10 cm^3, maximum
in Saturated Salt Water	10 cm^3, maximum
Residue Greater than 2000 micrometers	No residue

SUSPENSION PROPERTIES

11.2 Equipment

a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)

b. Balance: precision of 0.01 g

c. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up.

d. Mixer Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D)

e. Sealed glass container: about 500-cm^3 with lid

f. Container: glass or plastic with stopper or lid for salt solutions

g. Spatula

h. 1000-cm^3 volumetric flask

i. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4

j. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 3.2

k. Timers: two interval, mechanical or electrical, precision of 0.1 minutes

l. Graduated cylinders (TD): one 10 ±0.1 cm^3, one 100 ±1 cm^3, and one 500 ±5 cm^3

m. pH meter: precision of 0.1 pH units

n. Deionized (or distilled) water

o. API Standard Evaluation Base Clay (see Par. 1.4)

p. Sodium chloride: (CAS #7647-14-5)

q. Sodium bicarbonate: (CAS #144-55-8)

11.3 Procedure — 40 g/L Salt Water Test

a. Prepare a 40 g/L salt water solution by adding 40 ±0.1 g of sodium chloride to a 1000-cm^3 volumetric flask and diluting with deionized water to the inscribed mark on the flask. Mix thoroughly.

b. Prepare a clay-base suspension by adding 350 ±5 cm^3 of the 40 g/L salt water solution to the mixer container. Add 1.0 ±0.1 g of sodium bicarbonate and stir on the mixer for about 1 minute.

c. Slowly add 35.0 ±0.1 g API Standard Evaluation Base Clay while stirring on the mixer.

d. After stirring 5 ±0.1 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any clay adhering to container walls. Be sure all clay clinging to the spatula is incorporated into the suspension.

e. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the clay shall equal 20 ±1 minutes.

f. Add 3.50 ±.01 g (10.0 ±0.03 g/L) of starch to the suspension while stirring on the mixer, adding at a uniform rate over about 60 seconds.

g. After stirring 5 ±0.1 minutes, remove the container from the mixer and scrape its sides with the spatula to dislodge any starch adhering to container walls. Be sure all starch clinging to the spatula is incorporated into the suspension.

h. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any starch clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the starch shall equal 20 ±1 minutes.

i. Age the suspension up to 24 hours in a sealed or covered container at room temperature. Record the storage temperature and storage duration.

j. After aging, stir the suspension on the mixer for 5 ±0.1 minutes.

k. Immediately pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value for 600 rpm is reached. The 600 rpm dial reading shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C). Record 600 rpm dial reading.

l. Pour the suspension into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. The temperature of the suspension shall be 77 ±2°F (25 ±1°C). Pour the suspension to within about 1/2 inch (13 mm) of the top of the cell. Complete 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.

m. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on the cell at 100 ±5 psi (690 ±35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen or helium.

n. At 7.5 ±0.1 minutes on the first timer, remove the container and any adhering liquid on the drain tube and discard the filtrate. Place a dry 10-cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

11.4 Calculation — Filtrate Volume

Calculate the filtrate volume of the starch-treated suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c* \qquad \text{(a)}$$

*where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated value.

11.5 Procedure — Saturated Salt Water Test

a. Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container 40 to 45 g of sodium chloride per 100 ±1 cm^3 of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

b. Prepare a clay-base suspension by adding 350 ±2.5 cm^3 of the saturated salt solution to a mixer container. Add 1.0 ±0.1 g of sodium bicarbonate and stir on the mixer for about one minute.

c. Slowly add 35.0 ±0.1 g API Standard Evaluation Base Clay to each container while stirring on the mixer.

d. After stirring 5 ±0.1 minutes, remove the container from mixer and scrape its sides with the spatula to dislodge any clay adhering to the container walls. Be sure all clay clinging to the spatula is incorporated into the suspension.

e. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the clay shall equal 20 ±1 minutes.

f. Add 3.50 ±01 g (10.0 ±0.03 g/L) of starch to the suspension while stirring on the mixer, adding at a uniform rate over about 60 seconds.

g. After stirring 5 ±0.1 minutes, remove the container from the mixer and scrape its sides with the spatula to dislodge any starch adhering to container walls. Be sure all starch clinging to the spatula is incorporated into the suspension.

h. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any starch clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the starch shall equal 20 ±1 minutes.

i. Age the suspension up to 24 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

j. After aging, stir the suspension on the mixer for 5 ±0.1 minutes.

k. Immediately pour the suspension into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value at 600 rpm is reached. The 600 rpm dial reading shall be taken at a suspension test temperature of 77 ±2°F (25 ±1°C). Record 600 rpm dial reading.

l. Pour the suspension into a filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. The temperature of the suspension shall be 77 ±2°F (25 ±1°C). Pour the suspension to within about 1/2 inch (13 mm) of the top of the cell. Complete 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.

m. Set one timer for 7.5 ±0.1 minutes and the second timer for 30 ±0.1 minutes. Start both timers and adjust pressure on the cell at 100 ±5 psi (690 + 35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen or helium.

n. At 7.5 ±0.1 minutes on the first timer, remove the container and any adhering liquid on the drain tube and discard the filtrate. Place a dry 10-cm^3 graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

11.6 Calculation — Filtrate Volume

Calculate the filtrate volume of the starch-treated suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c \qquad \text{(a)}$$

where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated value.

RESIDUE GREATER THAN 2000 MICROMETERS

11.7 Equipment

2000-micrometer sieve: approximate dimensions — 8 inches (203 mm) diameter, meeting ASTM Specification E-11, latest edition.

NOTE: *Supplier's verification that sieve conforms to ASTM E11 is satisfactory evidence of compliance.*

11.8 Procedure

a. Weigh 25 ±0.1 g of starch and transfer to the 2000 micrometer sieve.

b. Shake for a maximum of 5 minutes.

c. Record the presence or absence of residue.

SECTION 12
CALIBRATION

12.1 Coverage

This section covers calibration procedures and calibration intervals for laboratory equipment and reagents specified. For laboratory items not listed, the manufacturer should develop procedures where deemed appropriate.

a. The manufacturer shall control, calibrate, verify, and maintain the laboratory equipment and reagents used in this specification for measuring product conformance to specification requirements.

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

c. 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 to be taken when results are unsatisfactory.

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

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

12.2 Laboratory Equipment and Reagents

a. Volumetric glassware: Laboratory volumetric glassware used for final acceptance, including Le Chatelier flasks, pipettes, and burettes, are usually calibrated by the supplier. Manufacturers of products to this specification shall document evidence of glassware calibration prior to use. Supplier certification is acceptable. Calibration may be checked gravimetrically. Periodic recalibration is not required.

b. Laboratory Thermometers: The manufacturer shall calibrate all laboratory thermometers used in measuring product conformance to specifications against a secondary reference thermometer. The secondary reference thermometer must show evidence of calibration as performed against NIST certified master instruments in accordance with the procedures outlined by ASTM E77-89 and NBS (NIST) Monograph 150.

c. Laboratory Balances: The manufacturer shall calibrate laboratory balances periodically in the range of use with NIST Class P, Grade 3, or better weights; and shall service and adjust balances whenever calibration indicates a problem.

d. Sieves: Barite (section 2), bentonite (sections 4 and 6), and hematite (section 3) manufacturers shall calibrate 75 micrometer (U.S. #200) sieves using API Test Calibration Barite with established values for residue retained. Hematite (section 3) manufacturers shall calibrate 45 micrometer (U.S. #325) sieves using a suitable quantity of uniform hematite.

e. Hydrometer: The manufacturer shall calibrate each hydrometer with the dispersant solution used in the sedimentation procedure.

f. Motor-Driven Direct Indicating Viscometer: The manufacturer shall calibrate each meter with 20 cP and 50 cP, certified standard silicone fluids.

g. Laboratory Pressure Measuring Device: The manufacturer shall document evidence of laboratory pressure measuring device calibration prior to use.

h. Mixer (e.g., Multimixer Model 9B with 9B29X impeller blades or equivalent as shown in Fig. 2.1, mounted flash side up): The manufacturer shall verify that all spindles rotate at 11,500 ±300 rpm under no load with one spindle operating. Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flash side up. New impellers shall be weighed prior to installation, with weight and date recorded.

i. Chemicals and Solutions: Must meet ACS reagent grade if available.

j. Deionized (or Distilled) Water: Manufacturer shall develop, document, and implement a method to determine hardness of water. The water shall not be used if hardness is indicated.

k. API Test Calibration Materials: Manufacturer shall test API Test Calibration Barite and/or (where applicable) API Test Calibration Bentonite for properties listed with their Certificates of Analysis.

12.3 Calibration Intervals

a. Any instrument subjected to movement which may affect its calibration shall be recalibrated prior to use.

b. Thermometers: Calibrate each thermometer before being put into service. After calibration, mark each thermometer with an identifying number that ties it to its corresponding correction chart. Check calibration annually against the secondary reference thermometer.

c. Laboratory Balances: Calibrate each balance prior to being put into service. Check calibration at least once per month for six months, then at least once per six months *if* required measurements capability is being maintained. If not, service and adjust, then check at least once per month until required measurement capability is maintained for six months, then once per six months.

d. Sieves: Calibrate each sieve (where required: reference Par. 12.2d) prior to being put into service. Check calibration at least once per 40 tests. After calibration, mark each sieve with an identifying number that ties it to its correction record. Since sieve calibration will change with use, maintain an up-to-date correction record.

e. Hydrometer: Calibrate each hydrometer prior to being put into service. After calibration, note and record each hydrometer identifying number that ties it to its correction chart. Periodic recalibration is not required.

f. Motor-Driven Direct Indicating Viscometers: Calibrate each viscometer prior to being put into service. Check calibration at least once per week for three months, then at least once per month *if* required measurement capability is being maintained.

g. Mixer (e.g., Multimixer Model 9B with 9B29X impellers or equivalent as shown in Fig. 2.1): Check and record mixer spindle speed at least once every 90 days to ensure operation within the prescribed rpm range, using a phototachometer or similar device. Remove, clean, dry, and weigh each impeller blade in service at least once every 90 days. Record weights, and replace blades when weight drops below 90% of its original value.

h. Deionized (or Distilled) Water: Manufacturer shall determine hardness of water whenever a new batch of water is prepared or purchased, or whenever deionizing cartridges are replaced.

i. Laboratory Pressure Measuring Devices: Manufacturer shall document evidence of laboratory pressure measuring device calibration prior to placing into service and annually thereafter.

j. API Test Calibration Materials: Manufacturer shall test the applicable API Test Calibration Material(s) at least once per three months.

12.4 Calibration Procedure — Thermometers

a. Place thermometer to be calibrated side by side with secondary reference thermometer into a constant temperature water bath (or suitable container of one gallon or more, filled with water, on a counter in a constant temperature room) and allow to equilibrate for at least one hour.

b. Read both thermometers and record readings.

c. Repeat readings throughout at least a one hour 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 0.2°F (0.1°C), or the smallest scale division on the thermometer being calibrated.

e. Calculate average deviation of thermometer reading from primary reference thermometer reading. Calculate and document correction for each thermometer.

12.5 Calibration Procedure — Sieve, 75 micrometers [Reference Par. 12.2d] for Barite, Hematite and Bentonite

a. Obtain a 75 micrometer (μm) sieve with a *certified centerline value*.

b. Take at least three samples of approximately 50 g dry API Test Calibration Barite (TCB).

c. Test each of the samples per Par. 2.9 using the certified sieve described in Par. 12.5a.

d. Calculate residue for each sample by:

$$\%\ \text{Residue, R} = 100\ \frac{(\text{Weight of residue, g})}{(\text{Weight of sample, g})}$$

e. Calculate average % residue of test calibration barite:

$$S = \frac{R_1 + R_2 + R_3 + \ldots}{N}$$

where: $R_1+R_2+R_3 \ldots =$ each individual test result

$N =$ number of samples tested result

NOTE: *Individual sample values must agree within ±0.2 of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat.*

f. From the intersection of % retained and certified sieve opening size on the Sieve Calibration Graph (Fig. 12.1), determine the calibration line (A, B, C, etc.) for use with the specific container of test calibration barite. Record this and identify the container with this value.

g. Repeat Par. 12.5b through Par. 12.5d except substitute the working sieve to be calibrated for the certified sieve.

h. Calculate average % residue of test calibration barite on working sieve:

$$R_a = \frac{R_1 + R_2 + R_3 + \ldots}{N}$$

NOTE: *Individual sample values must agree within ±0.2 of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat beginning at Par. 12.5g.*

i. From Sieve Calibration Graph (Fig. 12.1) determine the working sieve opening size to the nearest whole value from intersection of % retained and the calibration line from Par. 12.5f above. Record the working sieve opening size and identify the calibrated sieve and test calibration barite container.

j. Determine correction value (C) for working sieve from Table 12.1. Record this value and identify it with the calibrated sieve and specified test calibration barite container.

NOTE: *The sieve correction value obtained from Table 12.1 as specified is a number to be added to the residue value obtained on a test sample. (Negative values are subtracted).*

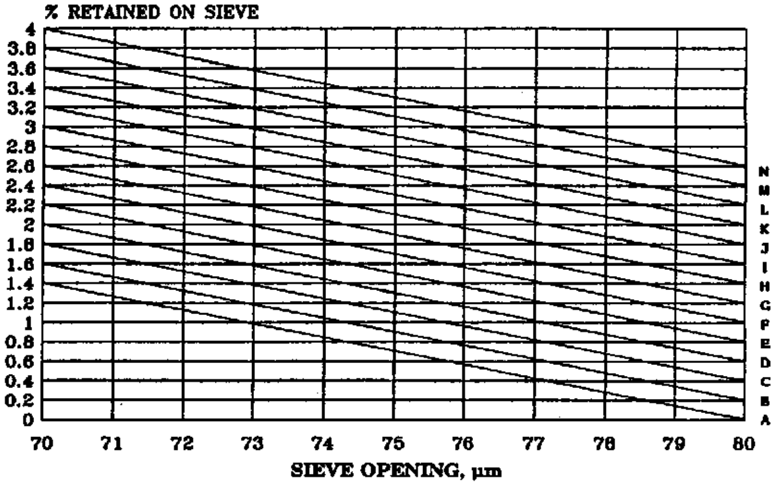

FIG. 12.1
SIEVE CALIBRATION CURVE —
75 MICROMETER SIEVE

TABLE 12.1
CORRECTION VALUES FOR 75 MICROMETER* (µm) SIEVES

Working Sieve Size Average Opening***, µm	Correction Size**: Barite/ Hematite	Bentonite
70	-0.7	-0.3
71	-0.6	-0.2
72	-0.4	0
73	-0.3	0
74	-0.1	0
75	0	0
76	+0.1	0
77	+0.3	0
78	+0.4	0
79	+0.6	+0.2
80	+0.7	+0.3

These correction values are valid between 0% to 4% retained on sieve.

*ASTM Sieve specifications allow ± 5 µm variation.

**Value to be added to test result of sample tested on sieve to convert results to equivalent 75 µm (NOTE: Negative values are subtracted).

***Determined from Calibration Graph (Fig. 12.1).

NOTE: *Correction values are rounded to the nearest 0.1.*

(1) Example of Barite Sieve Correction Value Determination:

Certified sieve size = 73 µm

Test Calibration Barite Average % Residue on Certified Sieve, S = 2.0%

Calibration Line = F

Test Calibration Barite Average % Residue on Working Sieve, Ra = 1.3%

Working Sieve size = 78 µm

Correction Value (from Table 12.1), C = +0.4%

(2) Example of Sieve Correction Application:

Sieve Correction Value, C = +0.4%

Test Sample Residue, Rs = 2.8%

Corrected Test Sample Residue, Rc = 2.8 + 0.4 = 3.2%

12.6 Calibration Procedure — Hydrometers

a. Calibrate each hydrometer to be used 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. Calibrate *each hydrometer* using the procedure below.

b. Prepare one liter of dispersant solution as follows:

(1) Place 125 ±2 cm^3 (127 ±1 g) of dispersant solution from test procedure (see Par. 2.11f) into a 1 liter volumetric flask.

(2) Dilute to the 1000-cm^3 mark with deionized water. Mix thoroughly.

c. 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 ±0.5°F (0.2°C). Insert the hydrometer to be calibrated and wait at least five minutes for the hydrometers and solution to reach bath temperature.

d. Take a hydrometer reading at the top of the meniscus formed by the stem, and take a thermometer reading. Repeat readings at least 5 minutes apart so as to obtain a minimum of four readings each.

e. Calculate the average hydrometer reading and designate as R1. Calculate the average temperature reading and designate as t1.

f. Repeat Par. 12.6c and Par. 12.6d except set bath temperature to highest expected test temperature, calculate average hydrometer and temperature readings, and designate these readings as R2 and t2.

g. Calculate the hydrometer correction curve slope (Mc) as follows:

$$Mc = 1000\,\frac{(R1-R2)}{(t2-t1)} \qquad \text{(a)}$$

where: R1 = average hydrometer reading at lower temperature

R2 = average hydrometer reading at higher temperature

t1 = average temperature reading at lower temperature

t2 = average temperature reading at higher temperature

NOTE: *Temperature may be measured in either °C or °F so long as all measurements and calculations are consistent in units.*

h. Calculate the hydrometer correction curve intercept (Bc) as follows:

$$Bc = (Mc \times t1) + [(R1-1) \times 1000] \qquad \text{(b)}$$

where: Mc = hydrometer correction curve slope

t1 = thermometer reading at lower temperature

R1 = hydrometer reading at lower temperature

i. Record Mc, Bc and hydrometer serial number in permanent calibration record and on test data sheet used in test Par. 2.13 and Par. 3.13.

Hydrometer Calibration
Example Data Sheet and Calculation

Hydrometer Serial No.	Temperature, °F (t)	Reading (R)	Corr. Curve Slope (Mc)	Corr. curve Intercept (Bc)
XXXXX	t1 = 78.8	R1 = 1.0017	0.1444	13.08
	t2 = 87.8	R2 = 1.0004		

Certified by: ______________________________ Date: ______________

Using (a):

$$Mc = \frac{1.0017 - 1.0004}{87.8 - 78.8} \times 1000 = 0.1444$$

Using (b):

$$Bc = (0.1444 \times 78.8) + (1.0017-1)(1000) = 13.08$$

12.7 Calibration — Motor-Driven Direct Indicating Viscometers

a. Equipment and Materials

(1) Standard thermometer, ±0.1°C, e.g., ASTM 90c or 91c

(2) Certified calibration fluid, 20 cP, with chart (viscosity vs. temperature).

(3) Certified calibration fluid, 50 cP, with chart (viscosity vs. temperature).

(4) Magnifying glass, approximately 3x magnification

b. Procedure

(1) Allow the viscometer and the calibration fluids to stand on counter top a minimum of two hours to approach temperature equilibrium.

(2) Operate viscometer with no fluid a minimum of two minutes to loosen bearing and gears.

(3) Clean and dry viscometer cup. Fill the viscometer cup to scribed line with 20 cP calibration fluid and place on meter stage. Raise stage until fluid level is to inscribed line on rotor sleeve.

(4) Place thermometer into the fluid and hold or tape to the side of viscometer to prevent breakage.

(5) Operate viscometer at 100 rpm setting until thermometer reading is stable to within ±0.1°C. Record temperature reading.

(6) Using magnifying glass, take dial readings at 300 and 600 rpm settings. Estimate readings to nearest 0.5 dial unit and record.

(7) Compare 300 rpm dial reading to certified viscosity at test temperature from fluid calibration chart. Record readings and deviation from certified calibration fluid viscosity as furnished by supplier. Divide 600 rpm reading by 1.98 to obtain viscosity value at 600 rpm. Compare this value to certified fluid.

(8) Repeat Par. 12.7b, steps 3-7 using 50 cP fluid.

(9) Compare deviations to values in Table 12.2. Tolerances should not exceed values in Table 12.2.

TABLE 12.2
DIAL READING TOLERANCES WITH VARIOUS CALIBRATION FLUIDS F-1 SPRING IN MOTOR DRIVEN VISCOMETER

	Acceptable Tolerance	
Fluid	**300 rpm**	**600 rpm/1.98**
20 cP	±1.5	±2
50 cP	±1.5	±2

12.8 Calibration — Laboratory Pressure Measuring Devices

a. Type and Accuracy: Pressure measuring devices shall be readable to at least 2.5% of full scale range.

b. Pressure measuring devices shall be calibrated to maintain ±2.5% accuracy of full scale range.

c. Usable Range: Pressure measurements shall be made at not less than 25% nor more than 75% of the full pressure span of pressure gauges.

d. Pressure measuring devices shall be recalibrated with a master pressure measuring device or a dead weight tester at 25%, 50%, and 75% of scale.

12.9 Calibration — Standard Calcium Chloride Solution

a. Standard Reagent

Standard Calcium Chloride Solution (0.0100 ±0.0001 molar) such that 1 cm^3 = 400 mg/kg Ca^{++}.

b. Procedure

1. To a suitable flask, add 50 ±0.05 cm^3 deionized water and 50 ±0.05 cm^3 of standard $CaCl_2$ solution.
2. Proceed as in Par. 2.6a — 2.6e, but without adding barite or additional water. (Use the 100 cm^3 solution prepared above in place of the 100 cm^3 deionized water specified in Par. 2.6a.)
3. Calculate calibration correction (C_c) as follows:

$$C_c = Cm - 200$$

where: Cm = 40 (EDTA volume, cm^3)

NOTE: *The calibration correction as determined by this procedure results in a number to be subtracted from the sample test value.*

Example of calibration correction determination:

EDTA for $CaCl_2$ solution = 4.8 cm^3

$Cm = 40 \times 4.8 = 192$

$C_c = 192 - 200$

$C_c = -8$

Example of calibration correction use:

EDTA for sample = 6.1 cm^3

Test value for sample = Ss = 244 mg/kg

Corrected test value = Ss-C_c = 244-(-8) = 252 mg/kg

12.10 Calibration Procedure — Sieve, 45 micrometers [Reference Par. 12.2d] for Hematite

a. Obtain a 45 micrometer (µm) sieve with a *certified centerline value.*

b. Obtain a suitable quantity of uniform hematite sufficient to last 6 months or longer. Mix thoroughly and store in a closed container. Identify this as "uniform hematite for 45 micrometer sieve calibration". Take at least three samples of approximately 50 g dry hematite for testing.

c. Test each of the samples per Par. 3.9 using the certified sieve described in Par. 12.10a.

d. Calculate residue for each sample by:

$$\%\ \text{Residue, R} = 100\ \frac{(\text{Weight of residue, g})}{(\text{Weight of sample, g})}$$

e. Calculate average % residue of uniform hematite:

$$S = \frac{R_1 + R_2 + R_3 + ..}{N}$$

where: R_1+R_2+R_3...= each individual test result

N = number of samples tested

NOTE: *Individual sample values must agree within ±0.5 of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat.*

f. From the intersection of % retained and certified sieve opening size on the Sieve Calibration Graph (Fig. 12.2), determine the calibration line (A, B, C, etc.) for use with the specific container of uniform hematite. Record this and identify the container with this value.

g. Repeat Par. 12.10b through Par. 12.10d except substitute the working sieve to be calibrated for the certified sieve.

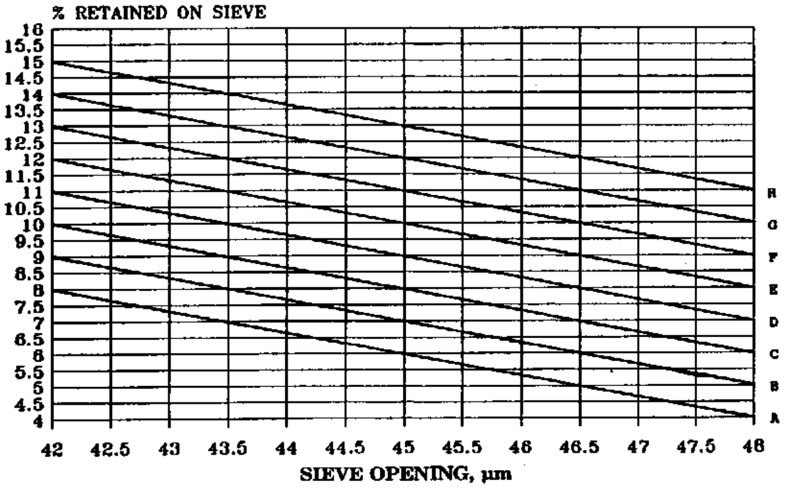

FIG.12.2
SIEVE CALIBRATION CURVE
45 MICROMETER SIEVE

h. Calculate average % residue of test calibration material on work sieve:

$$R_a = \frac{R_1 + R_2 + R_3 + . . .}{N}$$

NOTE: *Individual sample values must agree within ±0.2 of their average. If not, review test procedure technique and equipment operation for sources of error. Make corrections where needed and repeat beginning at Par. 12.10g.*

i. From Sieve Calibration Graph (Fig. 12.2) determine the working sieve opening size to the nearest 0.5 micrometer from the intersection of % retained and the calibration line from Par. 12.5f above. Record the working sieve opening size and identify the calibrated sieve and uniform hematite container.

j. Determine correction value (C) for working sieve from Table 12.3. Record this value and identify it with the calibrated sieve and specified test calibration material container.

NOTE: *The sieve correction value obtained from Table 12.3 as specified is a number to be added to the residue value obtained on a test sample. (Negative values are subtracted).*

(1) Example of Barite Sieve Correction Value Determination:

Certified sieve size = 46.5 µm

Hematite Average % Residue on Certified Sieve, S = 7.0%

Calibration Line = C

Hematite Average % Residue on Working Sieve, Ra = 9.7%

Working Sieve size = 42.5 µm

Correction Value (from Table 12.3), C = -1.7%

(2) Example of Sieve Correction Application:

Sieve Correction Value, C = -1.7%

Test Sample Residue, Rs = 8.8%

Corrected Test Sample Residue, Rc = 8.8 + (-1.7) = 7.1%

TABLE 12.3
CORRECTION VALUES FOR 45 MICROMETER* (µm) SIEVES

Working Sieve Size Average Opening***, µm	Correction Size**: Hematite
42	-2.0
42.5	-1.7
43	-1.3
43.5	-1.0
44.5	-0.3
45	0.0
45.5	+0.3
46	+0.7
46.5	+1.0
47	+1.3
47.5	+1.7
48	+2.0

*ASTM Sieve specifications allow ± 3 µm variation.
**Value to be added to test result of sample tested on sieve to convert results to equivalent 45 µm (NOTE: *Negative values are subtracted*).
***Determined from Calibration Graph (Fig. 12.2).

NOTE: *Correction values are rounded to the nearest 0.1.*

SECTION 13
PACKAGING, MARKING, AND STORAGE

13.1 Packaged Material

a. Each package of drilling fluid material conforming to the appropriate section of this specification shall be marked so as to include the following information:

- **(1)** The name of the material as it appears in the appropriate section of this specification, in block letters at least 1/2 inches (13 mm) in height.
- **(2)** The weight of the material in numbers and letters at least 1/4 inches (6 mm) in height. The weight will be listed in pounds and kilograms.
- **(3)** The date of manufacture, in block letters and/or numbers at least 1/8 inches (3 mm) in height.

b. The API monogram, if applicable, will be applied in a place of prominence.

See Bulletin S1 (Policy and Procedures for Standardization of Oilfield Equipment and Materials) for licensing requirements.

c. Each package shall contain the product weight marked thereon, ±5 percent. The average weight shall not be less than the weight as marked. The average weight is determined by averaging the total measured weight of a minimum of eighty (80) pack-ages, selected at random, of the material and dividing the total weight by the number of packages actually weighed. This information will be documented each 24 hours.

d. The manufacturer shall develop, document, and implement methods for the proper preservation of packaged drilling fluid material.

13.2 Bulk Material

a. The API monogram, if applied, will be placed on the delivery ticket accompanying each bulk shipment in a position of prominence.

See Bulletin S1 (Policy and Procedures for Standardization of Oilfield Equipment and Materials) for licensing requirements.

b. If the licensee receives material not under his control, and this material conforms to the chemical and physical requirements of the material specification and all other requirements under the Q-1 specification, the monogram can be applied.

APPENDIX A
METRIC "SI" UNIT CONVERSION TABLE

PROPERTY	TRADITIONAL UNIT	RECOMMENDED SI UNIT	SYMBOL	CONV. FACTOR	EXAMPLE
				Multiply By:	
Mechanical Properties					
Depth	feet	metre	m	0.3048	10,000 ft = 3048 m
Hole Diameter	inch	millimetre	mm	25.4	12¼ in. = 311 mm
Pipe Diameter	inch	millimetre	mm	25.4	4½ in. = 114 mm
Bit Size	inch	millimetre	mm	25.4	12¼ in. = 311 mm
Weight on Bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary Table Speed	rpm	*revolutions per minute	r/min	1	45 rpm = 45 r/min
Nozzle Size	1/32 inch	millimetre	mm	0.79	10/32 in = 7.9 mm
Nozzle Velocity	feet/sec	metre/sec	m/s	0.3048	400 ft/sec = 122 m/s
Drilling Rate	feet/hour	*metre/hour	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels	cubic metre	m^3	0.159	3000 bbl = 477 m^3
Liner Size	inch	millimetre	mm	25.4	6½ in. = 165 mm
Rod Diameter	inch	millimetre	mm	25.4	2¼ in. = 57 mm
Stroke length	inch	millimetre	mm	25.4	16 in = 406 mm
Pump Output	barrel/minute	*cubic metre/min	m^3/min	0.159	8.5 bbl/min = 1.35 m^3/min
	gallon/minute	*cubic metre/min	m^3/min	.00378	357 gpm = 1.35 m^3/min
Pump Pressure	pound/sq inch	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular Velocity	feet/min	*metre/minute	m/min	0.3048	200 ft/min = 61 m/min
Slip Velocity	feet/min	*metre/minute	m/min	0.3048	20 ft/min = 6.1 m/min
Temperature	degree Fahrenheit	degree Celsius	°C	$\frac{(°F-32)}{1.8}$	80°F = 27°C
	degree centigrade	degree Celsius	°C	1.0	
Funnel Viscosity	seconds/quart	seconds/litre	s/L	Units cannot normally be converted	
	seconds/1000 cm^3	seconds/litre	s/L	1.0	
Mud Weight	pounds/gallon	kilogram/cubic metre	kg/m^3	120	10 lb/gal = 1200 kg/m^3
	pounds/ft^3	kilogram/cubic metre	kg/m^3	16	74.8 lb/ft^3 = 1200 kg/m^3
Pressure Gradient	psi/ft	kilopascal/metre	kPa/m	22.6	0.52 psi/ft = 11.8 kPa/m
Hydrostatic Head	psi	kilopascal	kPa	6.9	4000 psi = 27,600 kPa
Shear Stress	lbf/100 ft^2	pascal	Pa	0.48	20 lb/100 ft^2 = 960 Pa
	dynes/cm^2	pascal	Pa	0.100	10 dynes/cm^2 = 1.0 Pa
Shear Rate	reciprocal second	reciprocal second	s^{-1}	1.0	
Apparent, Plastic & Effective Viscosity	centipoise	*centipoise	cP		
Yield Point	lbf/100 ft^2	pascal	Pa	0.48	15 lb/100 ft^2 = 7.2 Pa
Gel Strength	lbf/100 ft^2	pascal	Pa	0.48	3 lb/100 ft^3 = 1.44 Pa
Dial Reading Standard V-G Meter	$\frac{\text{lbf/100 ft}^2}{1.065}$	pascal	Pa	0.51	dial reading - 10 = 5.1 Pa
Power Law Constants					
n	dimensionless				
K	dynes s^n/cm^2	millipascal seconds^n/square centimetre	mPa·s^n/cm^2	100	10 dynes s^n/cm^2 = 100 mPa·s^n/cm^2
	lbf s^n/100 ft^2	millipascal seconds^n/square centimetre	mPa·s^n/cm^2	479	1.2 lb s^n/100 ft^2 = 575 mPa·s^n/cm^2
API Filtrate	cubic centimetre/30 min	*cubic centimetre/30 min	cm^3/30 min		
Filter Cake	1/32 inch	millimetre	mm	0.8	3/32 in.= 2.4 mm
Sand Content) Solids Content) Oil Content) Water Content)	Volume percent	cubic metre/cubic metre	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Particle Size	micron	micrometre	um	1.0	
Chemical Properties					
Ionic Concentration	grains/gallon	*milligram/litre	mg/L	17.1	500 grains/gal = 8600 mg/L
	parts/million	*milligram/litre	mg/L	x specific gravity	100,000 ppm of NaCl x 1.0707 = 107,070 mg/L
Alkalinity P_f, M_f, P_1, P_2	cubic centimetre/cubic centimetre	cubic centimetre/cubic centimetre	cm^3/cm^3		
MBT Methylene Blue Content	cubic centimetre/cubic centimetre	cubic centimetre/cubic centimetre	cm^3/cm^3		
Miscellaneous					
Additive Concentration	pound/barrel	kilogram/cubic metre	kg/m^3	2.85	10 lb/bbl = 28.5 kg/m^3
Corrosion Rate	lb/ft^2/yr	kilogram/square metre/year	kg/m^2/yr	4.9	0.9 lb/ft^2/year = 4.3 kg/m^2/year
	mils/year	millimetre/year	mm/a	.0254	200 mils/year = 5.08 mm/a
Bentonite Yield	barrels/US ton	*cubic metre/metric tonne	m^3/t	0.175	100 bbl/ton = 17.5 m^3/t
Hydraulic HP	horsepower	kilowatt	kw	0.746	600 hhp = 450 kw
Screen Size					
Mesh	openings/inch	openings/centimetre	openings/cm	0.254	100 mesh = 25.4 openings/cm
Aperature	micron	micrometer	um	1	
Open Area	area percent	square metre/square metre	m^2/m^2	0.01	30% = 0.3 m^2/m^2
Weight of Drill Pipe	pound/foot	kilogram/metre	kg/m	1.49	19.5 lb/ft = 29.1 kg/m
Oil Muds					
Oil Content) Water Content)	volume percent	cubic metre/cubic metre	m^3/m^3	0.01	10% = 0.1 m^3/m^3
Emulsion Stability	volts	volts	V		
Activity	Dimensionless				
Salinity	ppm	*milligrams/litre	mg/L	specific gravity	250,000 ppm of $CaCl_2$ x 1.24 = 310,000 mg/L
Aniline Point	degree Fahrenheit	degree Celsius	°C	$\frac{(°F-32)}{1.8}$	150°F = 66°C

*Denotes acceptable deviation from API 2564 preferred unit

APPENDIX B
MINERAL IMPURITIES IN BARITE

The presence of certain accessory materials in a barite can produce undesirable performance in some drilling fluids. These accessory materials include naturally occurring minerals such as gypsum, siderite, dolomite, and pyrrhotite.

Gypsum is somewhat soluble in water and releases calcium ion, one of the alkaline earth metals which are covered in API Specification 13A. Other minerals such as siderite, dolomite, and pyrrhotite are not covered in Spec 13A but can cause undesirable performance in some drilling fluids. These minerals, though not soluble in water to a significant degree, can release their anions in the presence of hydroxyl ions. Carbonate ions are released from siderite and dolomite in the presence of hydroxyl ions and sulfide ions are released from pyrrhotite at high temperature in combination with high pH.

Because of the conditions under which these minerals are solubilized, not all drilling fluids will be detrimentally affected by their presence. Consequently, API specifications limiting their concentrations in barite have not been developed. When it is suspected that one or more are affecting mud performance, procedures for determining their concentration in the barite may be found in API RP 13K, "Chemical Analysis of Barite."

APPENDIX C
TEST PRECISION

C.1 Description

a. Scope

Precision statements for the specification tests are presented in this appendix for the following materials:

1. Barite
2. Bentonite
3. Nontreated Bentonite
4. OCMA Grade Bentonite
5. Technical-Grade Low-Viscosity CMC
6. Technical-Grade High-Viscosity CMC
7. Starch
8. Hematite
9. Attapulgite
10. Sepiolite

The precision values in this appendix do not represent what might 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 Specification 13A should demonstrate at least the degree of test precision given in this appendix. Test calibration barite and test calibration bentonite are available through the API Production Department Office (see Par. 1.12) for use by laboratories to determine their test precision.

b. Considerations for Manufacturers and Users

The precision data in Tables C.1 through C.10 reflect the fact that the specification test methods are subject to normal test variability. This variability may 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.

C.2 Basis

The limits in Tables C.1 through C.10 were determined from interlaboratory studies conducted from 1988 to 1992. The data analysis is given in API Research Reports 88-30, 89-30, 90-30 and 92-30. 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 13.

The experimental design and data analysis was patterned after ASTM Practice E 691. 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.

C.3 Definitions

The term repeatability limit is used as described in ASTM Practice E177-90a, 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 Practice E177-90a, 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.

C.4 Test Precision Tables

TABLE C.1
TEST PRECISION
BARITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
Density	0.022 g/cm^3	0.030 g/m^3
Water Soluble Alkaline Earth Metals as Calcium	9.2 mg/kg	23.0 mg/kg
Residue greater than 75 micrometers, %	0.22 %	0.50 %
Particles less than 6 micrometers in equivalent spherical diameter	1.7 %	2.5 %

These values were determined from an interlaboratory study conducted in 1988 and 1989. Each of twelve laboratories tested each of three samples once each on each of three separate days. Each test result was the result of a single determination.

TABLE C.2
TEST PRECISION
BENTONITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
<u>Suspension Properties</u>		
Viscometer Dial Reading at 600 rpm	2.9	6.3
Yield Point/Plastic Viscosity Ratio	0.16	0.25
Filtrate Volume	0.8 cm^3	1.3 cm^3
Residue greater than 75 micrometers	0.7 %	1.3 %
Moisture	0.8 %	1.4 %

These values were determined from an interlaboratory study conducted in 1989. Each of eight laboratories tested each of two samples once each on each of three separate days. Each test result was the result of a single determination.

TABLE C.3
TEST PRECISION
NONTREATED BENTONITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
<u>Suspension Properties</u>		
Dispersed Plastic Viscosity	2.1 cP	3.1 cP
Dispersed Filtrate Volume	0.9 cm^3	1.5 cm^3

These values were determined from an interlaboratory study conducted in 1989. Each of eight laboratories tested each of two samples once each on each of three separate days. Each test result was the result of a single determination.

TABLE C.4
TEST PRECISION
OCMA BENTONITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
<u>Suspension Properties</u>		
Viscometer Dial Reading at 600 rpm	4.9	9.3
Yield Point/Plastic Viscosity Ratio	1.15	1.16
Filtrate Volume	0.8 cm^3	1.6 cm^3
Residue greater than 75 micrometers	0.5 %	1.1 %
Moisture	1.2 %	1.9 %

These values were determined from an interlaboratory study conducted in 1991. Each of seven laboratories tested each of two samples once each on each of three separate days. Each test result was the result of one determination.

TABLE C.5
TEST PRECISION
TECHNICAL-GRADE LOW-VISCOSITY CMC

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
<u>Solution Properties</u>		
Viscometer Dial Reading at 600 rpm	2.6	5.1
Filtrate Volume	0.9 cm^3	1.6 cm^3

These values were determined from an interlaboratory study conducted in 1991. For the viscometer dial reading at 600 test, each of eight laboratories tested each of two samples once each on each of three separate days. Each viscometer 600 rpm test result was the result of a single determination. For the filtrate volume test, each of six laboratories tested each of two samples once each on each of three separate days. Each filtrate volume test result was an average of two determinations.

TABLE C.6
TEST PRECISION
TECHNICAL-GRADE HIGH-VISCOSITY CMC

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
<u>Solution Properties</u>		
Viscometer Dial Reading at 600 rpm		
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 cm^3	1.9 cm^3

These values were determined from an interlaboratory study conducted in 1991. For the viscometer dial reading at 600 rpm tests, each of nine laboratories tested each of two samples once each on each of three separate days. Each viscometer result was the result of a single test. For the filtrate volume test, each of seven laboratories tested each of two samples once each on each of three separate days. Each filtrate test result was an average of two determinations.

TABLE C.7
TEST PRECISION
STARCH

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
<u>Suspension Properties</u>		
Viscometer Dial Reading at 600 rpm		
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 cm^3	2.3 cm^3
in Saturated Salt Water	0.6 cm^3	1.5 cm^3

These values were determined from an interlaboratory study conducted in 1991. For the 40 g/L salt water viscometer dial reading at 600 rpm test, each of eight laboratories tested each of two samples once each on each of three separate days. For the saturated salt water tests, each of seven laboratories tested each of two samples once each on each of three separate days. For the 40 g/L salt water filtrate volume test, each of seven laboratories tested each of two samples once each on each of three separate days. Each test result was an average of two determinations.

TABLE C.8
TEST PRECISION
HEMATITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
Density	0.026 g/cm^3	0.050 g/cm^3
Water soluble Alkaline Earth Metals, as Calcium	9.2 mg/kg	23.0 mg/kg
Residue greater than:		
75 micrometers, %	0.16 %	0.36 %
45 micrometers, %	0.6 %	3.2 %
Particles less than 6 micrometers, %	0.9%	3.4 %

These values were determined from an interlaboratory study conducted in 1992. Each of seven laboratories tested each of two samples once each on each of three separate days. Each test result was the result of a single determination.

TABLE C.9
TEST PRECISION
ATTAPULGITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
Suspension Properties		
Viscometer Dial Reading at 600 rpm	3.2	6.8
Residue greater than 75 micrometers, %	0.6 %	1.0 %
Moisture, %	1.5 %	2.1 %

These values were determined from an interlaboratory study conducted in 1992. Each of five laboratories tested each of two samples once each on each of three separate days for all properties except moisture content. Only four laboratories participated in the moisture test. Each test result was the result of a single determination.

TABLE C.10
TEST PRECISION
SEPIOLITE

Test	Repeatability Limit (Within-lab)	Reproducibility Limit (Between-lab)
Suspension Properties		
Viscometer Dial Reading at 600 rpm	4.5	5.6
Residue greater than 75 micrometers, %	0.3 %	0.8 %
Moisture, %	1.6 %	1.6 %

These values were determined from an interlaboratory study conducted in 1992. Each of four laboratories tested each of two samples once each on each of three separate days. Each test result was the result of a single determination.

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