Specification for Drilling Fluids Materials

ANSI/API SPECIFICATION 13A
EIGHTEENTH EDITION, FEBRUARY 2010

EFFECTIVE DATE: AUGUST 1, 2010

CONTAINS API MONOGRAM ANNEX AS PART OF U.S. NATIONAL ADOPTION

ISO 13500:2009 (Identical), Petroleum and natural gas industries—Drilling Fluids—Specifications and testing

AMERICAN PETROLEUM INSTITUTE

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Upstream Segment

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Shall: As used in a standard, “shall” denotes a minimum requirement in order to conform to the specification.

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Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. A one-time extension of up to two years may be added to this review cycle. Status of the publication can be ascertained from the API Standards Department, telephone (202) 682-8000. A catalog of API publications and materials is published annually by API, 1220 L Street, NW, Washington, DC 20005.

Suggested revisions are invited and should be submitted to the Standards Department, API, 1220 L Street, NW, Washington, DC 20005, standards@api.org.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13500 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

This third edition cancels and replaces the second edition (ISO 13500:2006), subclauses 7.1.2/Table 2, 7.3.1, 8.5.2, 8.6.5, 8.13.4, 10.2.5, 11.4, 14.4.3, and 15.4.3 of which have been technically revised. Clause 17 on low-viscosity polyanionic cellulose, Clause 18 on high-viscosity polyanionic cellulose, and Clause 19 on drilling-grade xanthan gum have been added. Clause 20 is added for a new grade of barite.

Introduction

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

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

This International Standard is substantially based on API Spec 13A, 16th Edition, February 1, 2004. The purpose of this International Standard is to provide product specifications for barite, haematite, bentonite, nontreated bentonite, Oil Companies' Materials Association (OCMA) grade bentonite, attapulgite, sepiolite, technical-grade low-viscosity carboxymethylcellulose (CMC-LVT), technical-grade high-viscosity carboxymethylcellulose (CMC-HVT), starch, low-viscosity polyanionic cellulose, high-viscosity polyanionic cellulose, drilling-grade Xanthum gum, and barite 4,1.

The intent of the document is to incorporate all International Standards for drilling fluid materials into an ISO-formatted document. A survey of the industry found that only the American Petroleum Institute (API) issued testing procedures and specification standards for these materials.

Reference to OCMA materials has been included in API work, as the OCMA and subsequent holding committees were declared defunct, and all specifications were submitted to API in 1983.

Annex A (informative) lists the mineral impurities in barite, Annex B (informative) provides the test precision and Annex C (informative) details examples of calculations.

Petroleum and natural gas industries — Drilling fluids — Specifications and testing

1 Scope

This International Standard covers physical properties and test procedures for materials manufactured for use in oil- and gas-well drilling fluids. The materials covered are barite, haematite, bentonite, nontreated bentonite, OCMA-grade bentonite, attapulgite, sepiolite, technical-grade low-viscosity carboxymethylcellulose (CMC-LVT), technical-grade high-viscosity carboxymethylcellulose (CMC-HVT), starch, low-viscosity polyanionic cellulose (PAC-LV), high-viscosity polyanionic cellulose (PAC-HV), drilling-grade Xanthan gum, and barite 4,1. This International Standard is intended for the use of manufacturers of named products.

2 Normative references

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

ISO 6780, *Flat pallets for intercontinental materials handling — Principal dimensions and tolerances*

ISO 10414-1:2008, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ASTM D422, *Standard Test Method for Particle-Size Analysis of Soils*

ASTM E11, *Standard Specification for Wire Cloth and Sieves for Testing Purposes*

ASTM E161, *Standard Specification for Precision Electroformed Sieves*

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

ASTM E177, *Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods*

NIST (NBS) Monograph 150, *Liquid-In-Glass Thermometry*

3 Terms, definitions, symbols and abbreviations

3.1 Terms and definitions

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

3.1.1
ACS reagent grade
chemicals that meet purity standards as specified by the American Chemical Society (ACS)

3.1.2

flash side

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

3.2 Symbols and abbreviations

ACS	American Chemical Society
API	American Petroleum Institute
APME	Association of Plastic Manufacturers in Europe
ASTM	American Society for Testing and Materials
EDTA	Ethylenediaminetetraacetic acid
CAS	Chemical Abstracts Service
CMC-HVT	Carboxymethylcellulose — High-viscosity, technical-grade
CMC-LVT	Carboxymethylcellulose — Low-viscosity, technical-grade
OCMA	Oil Companies' Materials Association
NBS	National Bureau of Standards
NIST	National Institute of Standards and Technology
TC	to contain
TD	to deliver
B_c	hydrometer correction curve intercept
b	yield point/plastic viscosity ratio
D_1	equivalent particle diameter immediately greater than 6 µm, determined in Equation (9)
D_2	equivalent particle diameter immediately less than 6 µm, determined in Equation (9)
D_e	equivalent spherical diameter, expressed in micrometres
C_c	calibration correction
C_m	40 times the EDTA volume, expressed in millilitres
K_S	sample constant
L	effective depth, expressed in centimetres
$\log(\eta_{20}/\eta_\theta)$	correction for temperature variance
M_c	hydrometer correction curve slope
m	sample mass, expressed in grams
m_1	mass of water-soluble calcium, in milligrams per kilogram

m_2	residue mass, expressed in grams
m_3	mass of the 425 µm sieve, expressed in grams
m_4	mass of 425 µm sieve and sample retained, expressed in grams
m_5	mass passing through a 425 µm sieve, expressed in grams
m_6	mass of the bottom receiver, expressed in grams
m_7	mass of the bottom receiver and sample content, expressed in grams
m_8	mass of sample passing through a 75 µm sieve, expressed in grams
R	hydrometer reading
R_1	average hydrometer reading at lower temperature
R_2	average hydrometer reading at higher temperature
R_{600}	viscometer dial reading at 600 r/min
R_{300}	viscometer dial reading at 300 r/min
S_s	sample test value
t	time, expressed in minutes
V	total filtrate volume, expressed in millilitres
V_c	filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min
V_1	initial volume, expressed in millilitres
V_2	final volume, expressed in millilitres
V_3	volume EDTA used, expressed in millilitres
V_4	volume of filtrate used, expressed in millilitres
w_1	mass fraction residue of particles greater than 75 µm, expressed in percent
w_2	cumulative percent for point immediately greater than 6 µm
w_3	cumulative percent for point immediately less than 6 µm
w_4	cumulative percent less than 6 µm
w_5	mass fraction residue of particles greater than 45 µm, expressed in percent (see 8.9.6)
w_6	mass fraction moisture, expressed in percent
w_a	cumulative percent finer
w_{75}	mass fraction of sample passing through a 75 µm sieve, expressed in percent
w_{425}	mass fraction passing through a 425 µm sieve, expressed in percent

ρ	sample density, expressed in grams per millilitre
θ	temperature, expressed in degrees Celsius or degrees Fahrenheit
θ_1	average temperature reading at lower temperature
θ_2	average temperature reading at higher temperature
η_A	apparent viscosity, expressed in centipoise
η	viscosity of water, expressed in millipascal seconds
η_{20}	1,002, is the viscosity of water at 20 °C (68 °F)
η_θ	viscosity at desired temperature (see Table 3)
η_P	plastic viscosity, expressed in millipascal·seconds
η_Y	yield point, expressed in pounds per 100 ft^2

4 Requirements

4.1 Quality control instructions

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

4.2 Use of test calibration materials in checking testing procedures

4.2.1 Test calibration barite and test calibration bentonite can be obtained by contacting the API1). The calibration test materials are shipped in a 7,6 l (2 gal) plastic container.

4.2.2 The API office forwards the request to the designated custodian for further handling. The test calibration products are furnished with a certificate of calibration giving the established values for each property and the confidence limits within which a laboratory's results shall fall.

4.2.3 The custodian shall furnish a certificate of analysis for each sample.

4.2.4 For calibration requirements of API test calibration materials, refer to 5.2.11 and 5.3.10.

4.2.5 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.

4.3 Records retention

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

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1) American Petroleum Institute, 1220 L Street NW, Washington, D.C. 20005-4070, USA.

5 Calibration

5.1 Coverage

5.1.1 Clause 5 covers calibration procedures and calibration intervals for laboratory equipment and reagents specified. For laboratory items not listed, the manufacturer shall develop procedures where deemed appropriate.

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

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

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

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

5.1.6 The manufacturer shall document and maintain calibration records for laboratory equipment and reagents; shall 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.

5.2 Equipment requiring calibration

5.2.1 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 International Standard shall document evidence of glassware calibration prior to use. Supplier certification is acceptable. Calibration may be checked gravimetrically. Periodic recalibration is not required.

5.2.2 Laboratory thermometers

5.2.2.1 The manufacturer shall calibrate all laboratory thermometers used in measuring product conformance to standards against a secondary reference thermometer. The secondary reference thermometer shall show evidence of calibration as performed against NIST-certified master instruments, in accordance with the procedures specified by ASTM E77 and NIST (NBS) Monograph 150.

5.2.2.2 Calibration — Thermometers

5.2.2.2.1 Place the thermometer being calibrated side by side with a secondary reference thermometer into a constant-temperature water bath (or suitable container of 4 l or more, filled with water, on a counter in a constant-temperature room) and allow to equilibrate for at least 1 h.

5.2.2.2.2 Read both thermometers and record readings.

5.2.2.2.3 Repeat readings throughout at least a 1 h interval to obtain a minimum of four readings.

5.2.2.2.4 Calculate the average and the range of readings for each thermometer. The difference between the range of readings for each thermometer shall not exceed ± 0,1 °C (± 0,2 °F), or the smallest scale division on the thermometer being calibrated.

5.2.2.2.5 Calculate the average deviation of the thermometer reading from the secondary reference thermometer reading. Calculate and document the correction for each thermometer.

5.2.3 Laboratory balances

5.2.3.1 The manufacturer shall calibrate the laboratory balances periodically in the range of use with NIST class P, grade 3, or better weights.

5.2.3.2 The manufacturer shall service and adjust balances whenever calibration indicates a problem.

5.2.4 Sieves

Sieves shall be in accordance with ASTM E11 and ASTM E161 and have approximate dimensions of 76 mm (3 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

5.2.5 Hydrometer

5.2.5.1 The manufacturer shall calibrate each hydrometer with the dispersant solution used in the sedimentation procedure.

5.2.5.2 Calibration — Hydrometer

5.2.5.2.1 Calibrate each hydrometer using the same concentration dispersant solution as is used in the test, at temperatures spanning the anticipated test temperatures, and by reading the top rather than the bottom of the meniscus. Calibrate each hydrometer using the procedure in 5.2.5.2.2 to 5.2.5.2.9.

5.2.5.2.2 Prepare 1 l of dispersant solution, as follows.

a) Place 125 ml ± 2 ml (127 g ± 2 g) of dispersant solution from test procedure (7.11.1 and 7.12.2) into a 1 l volumetric flask.

b) Dilute to the 1 000 ml mark with deionized water. Mix thoroughly.

5.2.5.2.3 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 ± 0,2 °C (± 0,4 °F). Insert the hydrometer being calibrated and wait at least 5 min for the hydrometer and solution to reach bath temperature.

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

5.2.5.2.5 Calculate the average hydrometer reading and designate as R_1. Calculate the average temperature reading and designate as θ_1.

5.2.5.2.6 Repeat 5.2.5.2.3 and 5.2.5.2.4, except set bath temperature to highest expected test temperature. Calculate the average hydrometer and temperature readings and designate these readings as R_2 and θ_2.

5.2.5.2.7 Calculate the hydrometer correction curve slope, M_c, as given in Equation (1):

$$M_\mathrm{c} = 1\,000 \frac{(R_1 - R_2)}{(\theta_2 - \theta_1)} \tag{1}$$

where

R_1 is the average hydrometer reading at lower temperature;

R_2 is the average hydrometer reading at higher temperature;

θ_1 is the average temperature reading at lower temperature;

θ_2 is the average temperature reading at higher temperature.

The temperature may be measured in either degrees Celsius or degrees Fahrenheit, so long as all measurements and calculations are consistent in units (including subsequent use of the hydrometer in routine test situations).

5.2.5.2.8 Calculate the hydrometer correction curve intercept, B_c, as given in Equation (2):

$$B_\mathrm{c} = \left(M_\mathrm{c} \times \theta_1\right) + \left[\left(R_1 - 1\right) \times 1\,000\right] \tag{2}$$

where

M_c is the hydrometer correction curve slope;

θ_1 is the average thermometer reading at the lower temperature;

R_1 is the average hydrometer reading at the lower temperature.

5.2.5.2.9 Record M_c, B_c and the hydrometer serial number in a permanent calibration record and on the data sheet used in the calculations in 7.13 and 8.13.

For hydrometer calibration, example data sheet and calculation, see Clause C.1.

5.2.6 Motor-driven, direct-indicating viscometer

5.2.6.1 The specifications for a direct-indicating viscometer are given in ISO 10414-1 and reproduced here for reference:

a) rotor sleeve:

- inside diameter: 36,83 mm (1,450 in),
- total length: 87,0 mm (3,425 in),
- scribed line: 58,4 mm (2,30 in) above the bottom of sleeve, with two rows of 3,18 mm (0,125 in) holes spaced 120° (2,09 rad) apart, around rotor sleeve just below scribed line;

b) bob, closed, with flat base and tapered top:

- diameter: 34,49 mm (1,358 in),
- cylinder length: 38,0 mm (1,496 in);

c) torsion-spring constant:

- 386 dyne-cm/degree deflection;

d) rotor sleeve speeds:

- high speed: 600 r/min,
- low speed: 300 r/min.

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

5.2.6.2 The manufacturer shall calibrate each meter with 20 mPa·s and 50 mPa·s, certified standard silicone fluids.

5.2.6.3 Apparatus and materials.

5.2.6.3.1 Standard thermometer, with an accuracy of ± 0,1 °C (± 0,2 °F), e.g. ASTM 90c or 91c grade.

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

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

5.2.6.3.4 Magnifying glass, approximately ×3 magnification.

5.2.6.4 Procedure.

5.2.6.4.1 Allow the viscometer and the calibration fluids to stand on counter-top a minimum of 2 h to approach temperature equilibrium.

5.2.6.4.2 Operate viscometer without fluid a minimum of 2 min to loosen bearing and gears.

5.2.6.4.3 Clean and dry viscometer cup. Fill the viscometer cup to scribed line with 20 mPa·s calibration fluid and place on meter stage. Raise stage until fluid level reaches the inscribed line on rotor sleeve.

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

5.2.6.4.5 Operate viscometer at 100 r/min setting until thermometer reading is stable to within ± 0,1 °C (± 0,2 °F). Record the temperature reading.

5.2.6.4.6 Using magnifying glass, take dial readings at 300 r/min and 600 r/min settings. Estimate readings to nearest 0,5 dial unit and record.

5.2.6.4.7 Compare 300 r/min 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 r/min reading by 1,98 to obtain viscosity value at 600 r/min. Compare this value to the certified fluid.

5.2.6.4.8 Repeat 5.2.6.4.1 through 5.2.6.4.7 using the 50 mPa·s fluid.

5.2.6.4.9 Compare the deviations to the values in Table 1. Tolerances shall not exceed values in Table 1.

Table 1 — Dial reading tolerances with various calibration fluids, F-1 spring (or equivalent) in motor-driven, viscometer

Calibration fluid	Acceptable tolerance	
	300 r/min	600 r/min/1,98
20 mPa·s	± 1,5	± 1,5
50 mPa·s	± 1,5	± 1,5

5.2.7 Laboratory pressure-measuring device

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

5.2.7.2 Calibration — Laboratory pressure-measuring device

5.2.7.2.1 Regarding type and accuracy, the pressure-measuring devices shall be readable to at least 2,5 % of full-scale range.

5.2.7.2.2 Pressure-measuring devices shall be calibrated to maintain ± 2,5 % accuracy of full-scale range.

5.2.7.2.3 Regarding usable range, the pressure measurements shall be made at not less than 25 % nor more than 75 % of the full-pressure span of pressure gauges.

5.2.7.2.4 Pressure-measuring devices shall be calibrated annually with a master pressure-measuring device or a dead-weight tester at at least three equidistant points of full scale (excluding zero and full scale as required points of calibration).

5.2.8 Mixer

EXAMPLE Multimixer® Model 9B [2] with 9B29X impeller blades, or equivalent, mounted flash side up.

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

5.2.9 Chemicals and solutions

5.2.9.1 These shall meet ACS or international equivalent reagent grade, if available.

5.2.9.2 Calibration — EDTA solution

5.2.9.2.1 Reagent

5.2.9.2.1.1 Standard calcium chloride solution, $c(\mathrm{CaCl_2}) = (0{,}010\,0 \pm 0{,}000\,1)$ mol/l.

5.2.9.2.2 Procedure

a) To a suitable flask, add 50 ml ± 0,05 ml of deionized water and 50 ml ± 0,05 ml of standard $\mathrm{CaCl_2}$ solution.

b) Proceed as in 7.6.1 through 7.6.5, but without adding barite or additional water. (Use the 100 ml solution prepared above in place of the 100 ml deionized water specified in 7.6.1.)

c) Calculate the calibration correction, C_c, as given in Equation (3):

$$C_\mathrm{c} = C_\mathrm{m} - 200 \tag{3}$$

where C_m is 40 times the EDTA volume, expressed in millilitres.

NOTE The calibration correction, as determined by this procedure, results in a number that is subtracted from the sample test value, S_s.

EXAMPLE 1 Calibration correction determination:

EDTA volume for the $\mathrm{CaCl_2}$ solution is equal to 4,8 ml:

$C_\mathrm{m} = 40 \times 4{,}8 = 192$

$C_\mathrm{c} = 192 - 200$

$C_\mathrm{c} = -8$

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[2] Multimixer® Model 9B is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

EXAMPLE 2 Calibration correction:

EDTA for the sample is equal to 6,1 ml:

Test value for the sample, $S_s = 244$ mg/kg

Corrected test value, $S_c = S_s - C_c = 244 - (-8) = 252$ mg/kg.

5.2.10 Deionized (or distilled) water

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

5.2.11 API test calibration materials

The manufacturer shall perform in-house verification of API calibration barite and/or (where applicable) API test calibration bentonite for properties listed with their certificates of analysis, as required by this International Standard.

5.3 Calibration intervals

5.3.1 General

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

5.3.2 Thermometers

Calibrate each thermometer before its first use by the manufacturer. After calibration, mark each thermometer with an identifying number that ties it to its corresponding correction chart. Check the calibration annually against the secondary reference thermometer.

5.3.3 Laboratory balances

Calibrate each balance prior to its first use by the manufacturer. Check calibration at least once per month for six months, then at least once per six months if required measurement capability is being maintained. If not, service and recalibrate, then check at least once per month until the required measurement capability is maintained for six months, then once per six months.

5.3.4 Sieves

No calibration of sieves is required. See 5.2.11 for periodic measurement requirements using standard reference materials.

5.3.5 Hydrometer

Calibrate each hydrometer prior to its first use by the manufacturer. After calibration, note and record each hydrometer identifying number that ties it to its correction chart. Periodic recalibration is not required.

5.3.6 Motor-driven, direct-indicating viscometers

Calibrate each viscometer prior to its first use by the manufacturer. Check the calibration at least once per week for three months, then at least once per month if required measurement capability is being maintained.

5.3.7 Mixer

EXAMPLE Multimixer® Model 9B with 9B29X impeller blades, or equivalent, mounted flash side up.

Check and record the mixer spindle speed at least once every 90 days to ensure that the operation falls within the prescribed range, using a phototachometer or similar device. Remove, clean, dry and weigh each impeller blade in use at least once every 90 days. Record masses and replace blades when the mass drops below 90 % of its original value.

5.3.8 Deionized (or distilled) water

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

5.3.9 Laboratory pressure-measuring devices

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

5.3.10 API test calibration materials

The manufacturer shall test the applicable API test calibration material(s) at least once per 40 tests. Sieve calibration requirements have been removed.

6 Packaged material

6.1 Description

6.1.1 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.

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

6.2 Apparatus — Pallets

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

6.2.2 Preferred sizes for wooden pallets include the following:

a) 1 200 mm × 1 000 mm (47 in × 39 in) CP6;

b) 1 140 mm × 1 140 mm (45 in × 45 in) CP8/CP9/CP3;

c) 1 219 mm × 1 219 mm (48 in × 48 in);

d) 1 118 mm × 1 321 mm (44 in × 52 in);

e) 1 067 mm × 1 321 mm (42 in × 52 in), equivalent to CP4/CP7;

f) 1 016 mm × 1 219 mm (40 in × 48 in).

NOTE CP is the size as defined in ISO 6780.

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

6.2.4 The maximum outside dimensions of the total package shall be in accordance with the applicable pallet size plus a maximum overhang of 3 cm (1,2 in). The overall height shall not exceed 2,0 m (80 in).

6.2.5 The maximum net mass should not exceed 2 000 kg (4 409 lb).

6.3 Apparatus — Bags

6.3.1 The manufacturer filling the bag should take reasonable steps to ensure that the bag construction is capable of safe handling, transport and storage.

6.3.2 The manufacturer should take reasonable steps to select bags that minimize waste and provide the possibility for recycling of the packaging material.

6.3.3 The manufacturer should consider the humidity-barrier capabilities of the bags relative to the needs of the particular product when selecting bags.

6.4 Marking — Pallets

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

a) product name;

b) gross/net mass, in kilograms (pounds);

c) other information as required, such as manufacturer's name, gross allowable mass, disposal options.

6.5 Marking — Bags

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

a) name of the material in print script at least 13 mm (0,5 in) high;

b) mass, which shall be denominated in kilograms, of the material in letters, or numbers and letters, at least 6 mm (0,25 in) high;

c) lot/batch number in print script and/or numbers at least 3 mm (0,125 in) high, traceable to manufacturer’s country of origin;

d) identification as recyclable;

e) safety information.

6.6 Pallet covers

6.6.1 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.

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

6.7 Package mass

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

6.8 Storage

The manufacturer shall advise on storage upon request.

6.9 Recycling

6.9.1 General

If appropriate, recycling of the remaining materials after using the contents may be done in accordance with the guidelines given in 6.9.2 to 6.9.4. All recycling should be done in accordance with local instructions and in compliance with the local regulatory administration concerned.

6.9.2 Pallets

General recovery and recycling, provided that pallet description is in accordance with ISO 6780.

6.9.3 Cover

Identify PE, PP or carton, and recycle accordingly.

6.9.4 Bags

Use of quality, high-performance paper results in less packaging materials and less waste for recycling. After separation of the various components, recycle accordingly.

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

7 Barite

7.1 Principle

7.1.1 Drilling-grade barite is produced from commercial barium sulfate-containing ores. The manufacturer shall retain certificates of analysis or similar documentation on these commercial barium sulfate ores. It may be produced from a single ore or a blend of ores and may be a straight-mined product or processed by beneficiation methods, i.e. washing, tabling, jigging or flotation. It may contain accessory minerals in addition to the barium sulfate ($BaSO_4$) mineral. Because of mineral impurities, commercial barite can vary in colour 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 Annex A for more details.)

7.1.2 Drilling-grade barite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the chemical and physical specifications of Table 2, represents the product produced and is controlled by the manufacturer.

Table 2 — Barite physical and chemical requirements

Requirement	Standard
Density	4,20 g/ml, minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum
Residue greater than 75 µm	Maximum mass fraction 3,0 %
Particles less than 6 µm in equivalent spherical diameter	Maximum mass fraction 30 %

7.2 Reagents and apparatus — Density by Le Chatelier flask

7.2.1 Kerosene or mineral spirits.

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

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

7.2.4 Le Chatelier flask, with graduations of 0,1 ml, clamped or weighted to prevent flotation in water bath.

7.2.5 Constant-temperature bath, transparent, at 32 °C ± 0,5 °C regulated to ± 0,1 °C (90 °F ± 1,0 °F regulated to ± 0,2 °F), e.g. an approximately 40-l aquarium (fish tank) with heater/circulator attachment, or functional equivalent.

7.2.6 Balance, with accuracy of 0,01 g.

7.2.7 Pipette, volumetric, 10 ml.

7.2.8 Magnifying glass.

7.2.9 Dowel, wooden, approximately 8 mm (0,33 in) in diameter and 30 cm (12 in) in length, or a functional equivalent.

7.2.10 Tissue paper, absorbent.

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

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

7.2.12 Brush, small, fine-bristle.

7.3 Procedure — Density by Le Chatelier flask

7.3.1 Take approximately 100 g of barite that has been oven dried for at least two hours and cooled to room temperature in a desiccator.

7.3.2 Fill a clean Le Chatelier flask to approximately 22 mm (0,8 in) below the zero mark with kerosene.

7.3.3 Place the flask upright in the constant-temperature bath. The level of water in the bath shall be higher than the 24 ml graduation of the flask but below the stopper level. Assure that the flask is stabilized by the use of clamps or weights.

7.3.4 Allow the flask and contents to equilibrate for a minimum of 1 h. Using the magnifying glass with care to keep eyes at meniscus level, read the volume at the lowest portion of the curved interface and record the initial volume to the nearest 0,05 ml without removing the flask from the constant-temperature bath. Record as V_1.

If the kerosene level is outside the −0,2 ml to +1,2 ml volume range after equilibrating, use the 10 ml pipette to add or remove kerosene in order to bring it within this range. Allow the flask to equilibrate for at least 1 h and record the initial volume as in 7.3.4.

7.3.5 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.

7.3.6 Weigh 80 g ± 0,05 g of dried barite into the weighing dish and carefully transfer it to the Le Chatelier flask. Take care to avoid splashing the kerosene or plugging the flask with barite at the bulb. This is a slow process, requiring repeated transfers of small amounts of barite. Use a brush to transfer any residual barite into the flask, then replace the stopper. Record the mass as m.

7.3.7 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.

7.3.8 Gently roll the flask along a smooth surface at no more than 45° from vertical, or twirl the upright flask at the neck vigorously between the palms of both hands, to remove entrained air from the barite sample. Repeat this procedure until no more bubbles can be seen rising from the barite.

7.3.9 Return the flask to the bath and let stand for at least 0,5 h.

7.3.10 Remove the flask from the bath and repeat 7.3.8 to remove any remaining air from the barite sample.

7.3.11 Immerse the flask in the bath again for at least 1 h.

7.3.12 Record the final volume in the same manner as described in 7.3.4. Record the volume as V_2.

7.4 Calculation — Density by Le Chatelier flask

Calculate the density, ρ, in grams per millilitre, according to Equation (4):

$$\rho = \frac{m}{(V_2 - V_1)} \tag{4}$$

where

m is the sample mass, expressed in grams;

V_1 is the initial volume, expressed in millilitres;

V_2 is the final volume, expressed in millilitres.

Record the calculated density.

7.5 Reagents and apparatus — Water-soluble alkaline earths as calcium

7.5.1 **Aqueous EDTA solution**, composed of 3,72 g ± 0,01 g of the disodium salt of ethylenediaminetetraacetic acid dihydrate [disodium salt of (ethylenedinitrilo)tetraacetic acid dihydrate] (CAS No. 6381-92-6) diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

7.5.2 **Buffer solution**, comprising 67,5 g ± 0,01 g of ammonium chloride (CAS No. 12125-02-9) and 570 ml ± 1 ml of 15 mol/l ammonium hydroxide (CAS No. 1336-21-6) solution diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

7.5.3 **Hardness indicator solution**, comprising 1 g ± 0,01 g Calmagite (CAS No. 3147-14-6), or equivalent [1-(1-hydroxy-4-methylphenylazo)-2-naphthol-4-sulfonic acid] diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

7.5.4 **Deionized** (or **distilled**) **water**.

7.5.5 **Balance**, of capacity exceeding 100 g with an accuracy of 0,01 g.

7.5.6 **Erlenmeyer flask**, 250 ml nominal capacity, equipped with a tight-fitting stopper.

7.5.7 **Graduated cylinder**, 100 ml to 150 ml (TC) with 1 ml graduations.

7.5.8 **Titration vessel**, e.g. beaker, 100 ml to 150 ml capacity.

7.5.9 **Serological pipettes** or **burette**, with graduations of 0,1 ml.

7.5.10 **Volumetric pipettes (TD)**, of capacity 10 ml, or equivalent.

7.5.11 **Filter press,** low-pressure/low-temperature, in accordance with API 13B-1/ISO 10414-1:2008, Clause 7, or filtration funnel.

7.5.12 **Filter paper**, Whatman 50, or equivalent**.**

7.5.13 **Glass container**, small.

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

7.5.15 **Volumetric flask**, 1 000 ml.

7.5.16 **Stirring rod**.

7.6 Procedure — Water-soluble alkaline earth metals as calcium

7.6.1 Weigh 100 g ± 0,05 g of barite. Transfer to the Erlenmeyer flask and add 100 ml ± 1 ml of deionized water. Stopper the flask and shake for at least 5 min during an approximate 1-h interval or by an optional mechanical shaking apparatus for 20 min to 30 min.

7.6.2 After shaking, filter the suspension through the low-pressure filter cell or funnel using two sheets of filter paper and collect filtrate in a suitable glass container.

7.6.3 Add 50 ml ± 1 ml of deionized water to the titration vessel. Add about 2 ml of hardness buffer and sufficient hardness indicator to achieve a distinct blue colour. Swirl to mix.

A solution with colour 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.

7.6.4 Using the volumetric pipette, measure 10 ml of the filtrate into the titrating vessel. Swirl to mix. A blue colour indicates no calcium hardness and the test is complete. A wine-red colour develops if calcium and/or magnesium are present. Record as V_4.

7.6.5 If hardness is present, begin stirring and titrate with EDTA solution to the blue endpoint. The endpoint of the titration is best described as the point at which additional EDTA produces no further red to blue change. The EDTA volume used to produce the blue endpoint is used in the calculation in 7.7. Record as V_3.

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

7.7 Calculation — Water-soluble alkaline earths as calcium

Calculate the soluble alkaline earth metals as calcium, m_1, in milligrams per kilogram, according to Equation (5):

$$m_1 = 400\left(\frac{V_3}{V_4}\right) \tag{5}$$

where

m_1 mass of water-soluble calcium, in milligrams per kilogram;

V_3 is the volume EDTA used, expressed in millilitres;

V_4 is the volume of filtrate used, expressed in millilitres.

Record the calculated value.

7.8 Reagents and materials — Residue of diameter greater than 75 µm

7.8.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

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

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

7.8.4 Balance, with an accuracy of 0,01 g.

7.8.5 Mixer (e.g. Multimixer® Model 9B with 9B29X 2) impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

7.8.6 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D 3), or equivalent).

7.8.7 Sieve, 75 µm, in accordance with ASTM E161, approximate dimensions: 76 mm (3,0 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E161 is satisfactory evidence of compliance.

7.8.8 Spray nozzle with 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body 4)), or equivalent), attached to a water line with a 90° elbow.

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

7.8.10 Evaporating dish or functional equivalent.

7.8.11 Wash bottle.

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3) Hamilton Beach® mixer cup No. M110-D is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

4) Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body, is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

7.9 Procedure — Residue of diameter greater than 75 µm

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

7.9.2 Weigh 50 g ± 0,01 g of dried barite. Record the mass as m. Add the weighed sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate. Stir on the mixer for 5 min ± 1 min.

7.9.3 Transfer the sample to the 75 µm sieve. Use a wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from a spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

7.9.4 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

7.9.5 Dry the residue in the oven to a constant mass. Record the residue mass as m_2 and total drying time.

7.10 Calculation — Residue of diameter greater than 75 µm

Calculate the mass fraction residue of particles greater than 75 µm, w_1, in percent, according to Equation (6):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{6}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

7.11 Reagents and apparatus — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

7.11.1 Dispersant solution, comprised of 40 g ± 0,1 g of sodium hexametaphosphate and 3,60 g ± 0,1 g of anhydrous sodium carbonate (CAS No. 497-19-8) per 1 000 ml of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9,0.

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

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

7.11.4 Balance, with an accuracy of 0,01 g.

7.11.5 Mixer [e.g. Multimixer Model 9B with 9B29X 2) impellers, or equivalent], having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

7.11.6 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

7.11.7 Sedimentation cylinder, glass, approximately 457 mm (18 in) high and 63 mm (2,5 in) in diameter, marked for a volume of 1 000 ml (in accordance with ASTM D422).

7.11.8 Rubber stopper, Number 13.

7.11.9 Water bath or **constant-temperature room**, capable of maintaining a constant temperature of 24 °C ± 5 °C (75 °F ± 7°F).

7.11.10 Thermometer, including the range 16 °C ± 0,5 °C to 32 °C ± 0,5 °C (60 °F ± 1,0 °F to 90 °F ± 1,0 °F).

7.11.11 Hydrometer, ASTM 151H, graduated to read the specific gravity of the suspension.

7.11.12 Timer, mechanical or electrical, with an accuracy of 0,1 min over the test period.

7.12 Procedure — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

7.12.1 Weigh 80 g ± 0,1 g of the dry barite and place in container. Record the mass as m.

7.12.2 Add 125 ml ± 2 ml (127 g ± 2 g) of dispersant solution (7.11.1). Dilute to approximately 400 ml with deionized water. Rinse all adhering particles from the spatula into the suspension.

7.12.3 Stir for 5 min ± 0,5 min on a mixer.

7.12.4 Transfer the suspension to the sedimentation cylinder. Rinse container with deionized water to assure that all sample particles are transferred to the sedimentation cylinder.

7.12.5 Add deionized water to the 1 000 ml mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s ± 5 s while holding a No. 13 rubber stopper in the top of the cylinder.

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

7.12.6 Set the cylinder into the water bath (or on the counter-top of a constant-temperature room) and simultaneously start the timer. Hang the thermometer in the water bath.

7.12.7 Take hydrometer readings at intervals of 10 min ± 0,1 min, 20 min ± 0,1 min, 30 min ± 0,1 min and 40 min ± 0,1 min (or until the first point below the 6 µm value is reached). To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1,020 reading before releasing. After the hydrometer stabilizes, read the top of the meniscus at the prescribed time. Carefully and slowly remove the hydrometer, rinse with deionized water and dry after each reading. The hydrometer shall be removed immediately after each reading to eliminate particle build-up on the shoulders, which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve the suspension-settling equilibrium.

7.12.8 Record the time, t, expressed in minutes, the temperature, θ, expressed in degrees Celsius (degrees Fahrenheit) and the hydrometer reading, R, on the data sheet.

Temperature may be measured in either degrees Celsius or degrees Fahrenheit, as long as all measurements and calculations are consistent in units, including hydrometer calibration.

7.12.9 For each time interval, determine the water viscosity, η, and the effective hydrometer depth, L, from Tables 3 and 4. Record on the data sheet.

7.13 Calculation — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

7.13.1 From the hydrometer calibration (5.2.5.2), enter the hydrometer correction slope, M_{c}, and the hydrometer correction intercept, B_{c}, onto the data sheet.

7.13.2 Calculate the sample constant, K_s, as given in Equation (7) (or determine from Table 5) and enter into data sheet:

$$K_s = 100 \frac{\rho}{m(\rho - 1)} \tag{7}$$

where

ρ is the sample density, expressed in grams per millilitre;

m is the sample mass, expressed in grams.

7.13.3 Calculate and enter onto the data sheet the equivalent spherical diameter, D_e, in micrometres, for each time interval as given in Equation (8):

$$D_e = 17{,}5\sqrt{\frac{\eta L}{(\rho - 1)t}} \tag{8}$$

where

η is the viscosity of water, expressed in millipascal seconds;

ρ is the sample density, expressed in grams per millilitre;

t is the time, expressed in minutes;

L is the effective depth (see Table 4), expressed in centimetres.

7.13.4 Calculate and enter onto the data sheet the cumulative percent finer, w_a, for the equivalent particle diameter, D_e, immediately greater than 6 µm, w_2, and the equivalent particle diameter, D_e, immediately less than 6 µm, w_3, as given in Equation (9):

$$w_a = K_s\left[(M_c \cdot \theta) - B_c + (R - 1)1\,000\right] \tag{9}$$

where

K_s is the sample constant;

M_c is the hydrometer correction slope, determined in Equation (1);

θ is the suspension temperature, expressed in degrees Celsius [degrees Fahrenheit (see 7.12.8)];

B_c is the hydrometer correction intercept, as determined in Equation (2);

R is the hydrometer reading.

7.13.5 Calculate and enter onto the data sheet the cumulative percent less than 6 µm, w_4, as given in Equation (10):

$$w_4 = \left[\left(\frac{w_2 - w_3}{D_1 - D_2} \right) (6 - D_2) \right] + w_3 \tag{10}$$

where

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

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

D_1 is the equivalent particle diameter immediately greater than 6 µm, determined in Equation (8);

D_2 is the equivalent particle diameter immediately less than 6 µm, determined in Equation (8).

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

7.13.6 A correction, $\log(\eta_{20}/\eta_\theta)$, for water not at the reference temperature of 20 °C (68 °F), can be calculated according to an established equation [10] as given in Equation (11).

$$\log(\eta_{20}/\eta_\theta) = [1{,}370\,23\,(\theta - 20) + 0{,}000\,836\,(\theta - 20)^2]/(109 + \theta) \tag{11}$$

where

θ is the temperature, in degrees Celsius;

η_{20} 1,002, is the viscosity of water at 20 °C (68 °F);

η_θ is the viscosity at desired temperature (see Table 3).

Table 3 — Viscosity of water at various temperatures

Temperature θ °C (°F)	Viscosity η mPa·s	Temperature θ °C (°F)	Viscosity η mPa·s
15,6 (60)	1,121 1	22,2 (72)	0,949 8
16,1 (61)	1,105 0	22,8 (73)	0,937 4
16,7 (62)	1,089 3	23,3 (74)	0,925 3
17,2 (63)	1,073 9	23,9 (75)	0,913 4
17,8 (64)	1,058 9	24,4 (76)	0,901 8
18,3 (65)	1,044 2	25,0 (77)	0,890 4
18,9 (66)	1,029 8	25,6 (78)	0,879 2
19,4 (67)	1,015 8	26,1 (79)	0,868 3
20,0 (68)	1,002 0	26,7 (80)	0,857 6
20,6 (69)	0,988 5	27,2 (81)	0,847 0
21,1 (70)	0,975 3	27,8 (82)	0,836 7
21,7 (71)	0,962 4	28,3 (83)	0,826 6

Table 4 — Values of effective depth based on readings on hydrometer ASTM 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 5 — Sample constant, K_s, for barite (80,0 g sample)

Sample density g/ml	**Sample constant** K_s
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

8 Haematite (hematite)

8.1 Principle

8.1.1 Drilling-grade haematite is produced from commercial ores, and may be a single ore or blends of haematite ores. The haematite 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, aluminium oxide, calcium oxide, and magnesium oxide.

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

Table 6 — Haematite chemical and physical specifications

Requirement	**Standard**
Density	5,05 g/ml, minimum
Water-soluble alkaline earth metals, as calcium	100 mg/kg, maximum
Residue greater than 75 µm	maximum mass fraction 1,5 %
Residue greater than 45 µm	maximum mass fraction 15 %
Particles less than 6 µm in equivalent spherical diameter	maximum mass fraction 15 %

8.2 Reagent and apparatus — Density by Le Chatelier flask

8.2.1 Kerosene or mineral spirits.

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

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

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

8.2.5 Constant-temperature bath, transparent, at 32 °C ± 0,5 °C regulated to ± 0,1 °C (90 °F ± 1,0 °F regulated to ± 0,2 °F), e.g. an approximately 40 l aquarium (fish tank) with heater/circulator attachment, or functional equivalent.

8.2.6 Balance, with accuracy of 0,01 g.

8.2.7 Pipette, volumetric, 10 ml.

8.2.8 Magnifying glass.

8.2.9 Dowel, wooden, approximately 8 mm (0,33 in) in diameter and 30 cm (12 in) in length, or a functional equivalent.

8.2.10 Tissue paper, absorbent.

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

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

8.2.12 Brush, small, fine-bristle.

8.3 Procedure — Density by Le Chatelier flask

8.3.1 Take approximately 120 g of haematite that has been oven dried for at least two hours and cooled to room temperature in a desiccator.

8.3.2 Fill a clean Le Chatelier flask to approximately 22 mm (0,8 in) below the zero mark with kerosene.

8.3.3 Place the flask upright in the constant-temperature bath. The level of water in the bath shall be higher than the 24 ml graduation of the flask but below the stopper level. Assure that the flask is stabilized by the use of clamps or weights.

8.3.4 Allow the flask and contents to equilibrate for a minimum of 1 h. Using the magnifying glass with care to keep the eyes at the meniscus level, read the volume at the lowest portion of the curved interface and record the initial volume to the nearest 0,05 ml without removing the flask from the constant-temperature bath.

If kerosene level is outside the – 0,2 ml to + 1,2 ml volume range after equilibrating, use the 10 ml pipette to add or remove kerosene in order to bring it within this range. Allow the flask to equilibrate for at least 1 h and record the initial volume as in 8.3.4.

8.3.5 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.

8.3.6 Weigh 100 g ± 0,01 g of the dried haematite into the weighing dish and carefully transfer to the Le Chatelier flask. Take care to avoid splashing the kerosene or plugging of the flask with haematite at the bulb.

This is a slow process, requiring repeated transfers of small amounts of haematite. Use a brush to transfer any residual haematite into the flask, then replace the stopper.

8.3.7 If necessary, carefully tap the neck of the flask with the wooden dowel or agitate carefully side to side to dislodge any haematite clinging to the walls. Do not allow the kerosene to come into contact with the ground-glass stopper joint of the flask.

8.3.8 Gently roll the flask along a smooth surface at no more than 45° from vertical or twirl the upright flask at the neck vigorously between the palms of both hands to remove entrained air from the haematite sample. Repeat this procedure until no more bubbles can be seen rising from the haematite.

8.3.9 Return the flask to the bath and let stand for at least 0,5 h.

8.3.10 Remove the flask from the bath and repeat 8.3.8 to remove any remaining air from the haematite sample.

8.3.11 Immerse the flask in the bath again for at least 1 h.

8.3.12 Record the final volume in the same manner as described in 8.3.4.

8.4 Calculation — Density by Le Chatelier flask

Calculate the density, ρ, in grams per millilitre, according to Equation (12):

$$\rho = \frac{m}{\left(V_2 - V_1\right)} \tag{12}$$

where

m is the sample mass, in grams;

V_1 is the initial volume, in millilitres;

V_2 is the final volume, in millilitres.

Record the calculated density.

8.5 Reagents and apparatus — Water-soluble alkaline earth metals as calcium

8.5.1 Aqueous EDTA solution, comprised of 3,72 g ± 0,01 g of disodium salt of ethylenediaminetetraacetic acid dihydrate [disodium salt of (ethylenedinitrilo)tetraacetic acid dihydrate] (CAS No. 6381-92-6) diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

8.5.2 Buffer solution, comprised of 67,5 g ± 0,01 g of ammonium chloride (CAS No. 12125-02-9) and 570 ml ± 1 ml of ammonium hydroxide (CAS No. 1336-21-6) solution, $c(\mathrm{NH_4OH}) = 15$ mol/l, diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

8.5.3 Hardness indicator solution, 1 g ± 0,01 g of Calmagite (CAS No. 3147-14-6), or equivalent [1-(1-hydroxy-4-methylphenylazo)-2-naphthol-4-sulfonic acid], diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

8.5.4 Deionized (or **distilled**) **water**.

8.5.5 Balance, of capacity exceeding 100 g with a precision of 0,01 g.

8.5.6 Erlenmeyer flask, 250 ml nominal capacity, equipped with a tight-fitting stopper.

8.5.7 **Graduated cylinder**, 100 ml (TC) with 1 ml graduations.

8.5.8 **Titration vessel**, e.g. beaker, 100 ml to 150 ml.

8.5.9 **Serological pipettes** or **burette**, graduated to 0,1 ml.

8.5.10 **Volumetric pipettes**, 10 ml (TD), or equivalent.

8.5.11 **Filter press**, low pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7, or filtration funnel.

8.5.12 **Filter paper**, Whatman 50, or equivalent.

8.5.13 **Glass container**, small.

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

8.5.15 **Volumetric flask**, 1 000 ml.

8.5.16 **Stirring rod**.

8.6 Procedure — Water-soluble alkaline earth metals as calcium

8.6.1 Weigh 100 g ± 0,05 g of haematite. Transfer to the Erlenmeyer flask and add 100 ml ± 1 ml of deionized water. Stopper the flask and shake for at least 5 min during an approximate 1-h interval or by an optional mechanical shaking apparatus for 20 min to 30 min.

8.6.2 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.

8.6.3 Add 50 ml ± 1 ml of deionized water to the titration vessel. Add about 2 ml of hardness buffer and sufficient hardness indicator to achieve a distinct blue colour. Swirl to mix.

A solution with colour 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.

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

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

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

8.7 Calculation — Water-soluble alkaline earth metals as calcium

Calculate the soluble alkaline earth metals as calcium, m_1, in milligrams per kilogram, according to Equation (13):

$$m_1 = 400\left(\frac{V_3}{V_4}\right) \tag{13}$$

where

m_1 is the mass of water-soluble calcium, in milligrams per kilogram;

V_3 is the volume EDTA used, expressed in millilitres;

V_4 is the volume of filtrate used, expressed in millilitres.

Record the calculated value.

8.8 Reagents and apparatus — Residues greater than 75 µm and less than 45 µm

8.8.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

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

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

8.8.4 Balance, with an accuracy of 0,01 g.

8.8.5 Mixer (e.g. Multimixer® Model 9B, with 9B29X impellers, or equivalent) having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

8.8.6 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton® Beach mixer cup No. M110-D), or equivalent.

8.8.7 Sieve, 75 µm, in accordance with ASTM E 161-96, approximate dimensions: 76 mm (3,0 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E 161-96 is satisfactory evidence of compliance.

8.8.8 Sieve, 45 µm, in accordance with ASTM E 161-96, approximate dimensions: 76 mm (3,0 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E 161-96 is satisfactory evidence of compliance.

8.8.9 Spray nozzle, 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with a 1/4 TT body [5]), or equivalent), attached to a water line with a 90° elbow.

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

8.8.11 Evaporating dish, or functional equivalent.

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5) This item is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

8.8.12 Wash bottle.

8.9 Procedure — Residues of diameter greater than 75 µm and 45 µm

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

8.9.2 Weigh 50 g ± 0,01 g of dried haematite. Add the weighed sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate. Stir on the mixer for 5 min ± 1 min.

8.9.3 Transfer the sample to the 75 µm sieve. Use a wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from a spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

8.9.4 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

8.9.5 Dry the residue in the oven to a constant mass. Record the residue mass and total drying time.

8.9.6 Repeat steps 8.9.2 through 8.9.5 using the 45 µm sieve.

8.10 Calculation — Residues of diameter greater than 75 µm and 45 µm

Calculate the mass fraction residue of particles greater than 75 µm, w_1, expressed in percent, according to Equation (14) and the mass fraction residue of particles greater than 45 µm, w_5, expressed in percent, according to Equation (15) (see 8.9.6):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{14}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

$$w_5 = 100\left(\frac{m_2}{m}\right) \tag{15}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

8.11 Reagents and apparatus — Particles less than 6 µm in equivalent spherical diameter by the sedimentation method

8.11.1 Dispersant solution, comprised of 40 g ± 0,1 g of sodium hexametaphosphate and 3,60 g ± 0,1 g of anhydrous sodium carbonate (CAS No. 497-19-8) per 1 000 ml of solution.

The sodium carbonate is used to adjust the pH of the solution to approximately 9,0.

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

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

8.11.4 Balance, with an accuracy of 0,01 g.

8.11.5 Mixer (e.g. Multimixer Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

8.11.6 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

8.11.7 Sedimentation cylinder, glass, approximately 457 mm (18,0 in) high and 63 mm (2,5 in) in diameter, marked for a volume of 1 000 ml (in accordance with ASTM D422).

8.11.8 Rubber stopper, size No. 13.

8.11.9 Water bath or **constant-temperature room**, capable of maintaining a constant temperature of 24 °C ± 5 °C (75 °F ± 9 °F).

8.11.10 Thermometer, including the range 16 °C ± 0,5 °C (60 °F ± 1 °F) to 32 °C ± 0,5 °C (90 °F ± 1 °F).

8.11.11 Hydrometer, ASTM 151H, graduated to read the specific gravity of the suspension.

8.11.12 Timer, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

8.12 Procedure — Particles less than 6 µm in equivalent spherical diameter by the sedimentation method

8.12.1 Weigh 80 g ± 0,1 g of dry haematite and place in the container.

8.12.2 Add 125 ml ± 2 ml (127 g ± 2 g) of dispersant solution (8.11.1). Dilute to approximately 400 ml with deionized water. Rinse all adhering particles from the spatula into the suspension.

8.12.3 Stir for 5 min ± 0,5 min on the mixer.

8.12.4 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.

8.12.5 Add deionized water to the 1 000 ml mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s ± 5 s while holding a No. 13 rubber stopper in the top of the cylinder.

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

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

8.12.7 Take hydrometer readings at intervals of 10 min ± 0,1 min, 20 min ± 0,1 min, 30 min ± 0,1 min and 40 min ± 0,1 min (or until the first point below the 6 µm value is reached). To take a hydrometer reading, carefully and slowly lower the hydrometer to approximately the 1,020 reading before releasing. After the hydrometer stabilizes, read the top of the meniscus at the prescribed time. Carefully and slowly remove the hydrometer, rinse with deionized water and dry after each reading. Remove the hydrometer immediately after each reading to eliminate particle build-up on the shoulders, which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve the suspension-settling equilibrium.

8.12.8 Record time, t, in minutes, the temperature, θ, in degrees Celsius (degrees Fahrenheit) and the hydrometer reading, R, on the data sheet.

Temperature may be measured in either degrees Celsius or degrees Fahrenheit as long as all measurements and calculations are consistent in units, including hydrometer calibration.

8.12.9 For each time interval, determine water viscosity, η, and effective hydrometer depth, L, from Tables 3 and 4. Record on the data sheet.

8.13 Calculation — Particles less than 6 µm in equivalent spherical diameter by the sedimentation method

8.13.1 From hydrometer calibration (5.2.5), enter the hydrometer correction slope, M_{c}, and the hydrometer correction intercept, B_{c}, onto the data sheet.

8.13.2 Calculate the sample constant, K_{s}, as given in Equation (16) (or determine from Table 7) and enter onto the data sheet:

$$K_{\mathrm{s}} = 100 \frac{\rho}{m(\rho - 1)} \tag{16}$$

where

ρ is the density of sample, expressed in grams per millilitre;

m is the mass of sample, in grams.

8.13.3 Calculate and enter onto the data sheet the equivalent spherical diameter, D_{e}, expressed in micrometres, for each time interval as given in Equation (17):

$$D_{\mathrm{e}} = 17{,}5 \sqrt{\frac{\eta L}{(\rho - 1)t}} \tag{17}$$

where

η is the viscosity of water, expressed in millipascal·seconds;

ρ is the sample density, expressed in grams per millilitre;

t is the time, expressed in minutes;

L is the effective depth, expressed in centimetres.

8.13.4 Calculate and enter onto the data sheet the cumulative percent finer, w_a, for the equivalent particle diameter, D_e, immediately greater than 6 µm, w_2, and the equivalent particle diameter, D_e, immediately less than 6 µm, w_3, as given in Equation (18):

$$w_a = K_s\left[\left(M_c \cdot \theta\right) - B_c + \left(R - 1\right)1\,000\right] \quad (18)$$

where

K_s is the sample constant;

M_c is the hydrometer correction slope;

θ is the suspension temperature, expressed in degrees Celsius or degrees Fahrenheit (see 8.12.8);

B_c is the hydrometer correction intercept;

R is the hydrometer reading.

8.13.5 Calculate and enter onto the data sheet the cumulative percent less than 6 µm, w_4, as given in Equation (19):

$$w_4 = \left[\left(\frac{w_2 - w_3}{D_1 - D_2}\right)\left(6 - D_2\right)\right] + w_3 \quad (19)$$

where

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

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

D_1 is the equivalent particle diameter immediately greater than 6 µm;

D_2 is the equivalent particle diameter immediately less than 6 µm.

For an example of the calculation for particles less than 6 µm in equivalent spherical diameter, see Clause C.3.

9 Bentonite

9.1 Principle

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

9.1.2 Drilling-grade bentonite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 8, represents the product produced and is controlled by the manufacturer.

Table 7 — Sample constant, K_s, for haematite (80,0 g sample)

Sample density g/ml	**Sample constant** K_s
5,00	1,562 5
5,01	1,561 7
5,02	1,560 9
5,03	1,560 2
5,04	1,559 4
5,05	1,558 6
5,06	1,557 9
5,07	1,557 1
5,08	1,556 4
5,09	1,555 6
5,10	1,554 9
5,11	1,554 1
5,12	1,553 4
5,13	1,552 7
5,14	1,551 9
5,15	1,551 2
5,16	1,550 5
5,17	1,549 8
5,18	1,549 0
5,19	1,548 3
5,20	1,547 6
5,21	1,546 9
5,22	1,546 2
5,23	1,545 5
5,24	1,544 8
5,25	1,544 1
5,26	1,543 4
5,27	1,542 7
5,28	1,542 1
5,29	1,541 4
5,30	1,540 7

Table 8 — Bentonite physical specifications

Requirement	**Standard**
Suspension properties:	
Viscometer dial reading at 600 r/min	minimum 30
Yield point/plastic viscosity ratio	Maximum 3
Filtrate volume	maximum 15,0 ml
Residue of diameter greater than 75 µm	maximum mass fraction 4,0 %

9.2 Reagents and apparatus — Suspension properties

9.2.1 Thermometer, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

9.2.2 Balance, with an accuracy of 0,01 g.

9.2.3 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having a spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

9.2.4 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton® Beach mixer cup No. M110-D, or equivalent).

9.2.5 Spatula.

9.2.6 Viscometer, Motor-driven, direct-indicating, in accordance with ISO 10414-1.

9.2.7 Filter press, low-pressure/low-temperature, in accordance with IS0 10414-1:2008, Clause 7.

9.2.8 Graduated cylinders, 500 ml ± 5 ml (TC) and 10 ml ± 0,1 ml (TC).

9.2.9 Deionized (or **distilled**) **water**.

9.2.10 Container, with lid, capacity about 500 ml.

9.2.11 Timers, two, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

9.3 Procedure — Rheology of suspension

9.3.1 Prepare a suspension of the bentonite. Add 22,5 g ± 0,01 g of clay (as received) to 350 ml ± 5 ml of deionized water while stirring on the mixer.

9.3.2 After stirring 5 min ± 0,5 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any bentonite adhering to the container walls. Be sure that all bentonite clinging to the spatula is incorporated into the suspension.

9.3.3 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min ± 1 min.

9.3.4 Age the bentonite suspension up to 16 h in a sealed or covered container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.

9.3.5 After ageing the bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 min ± 0,5 min.

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

9.4 Calculation — Rheology of suspension

Calculate the plastic viscosity, η_P, expressed in millipascal·seconds, according to Equation (20), the yield point, η_Y, in pounds per 100 ft^2, according to Equation (21), and the yield point/plastic viscosity ratio, b, according to Equation (22):

$$\eta_\mathrm{P} = R_{600} - R_{300} \tag{20}$$

$$\eta_\mathrm{Y} = R_{300} - \eta_\mathrm{P} \tag{21}$$

$$b = \frac{\eta_\mathrm{Y}}{\eta_\mathrm{P}} \tag{22}$$

where

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

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

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

9.5 Procedure — Filtrate volume of suspension

9.5.1 Recombine all of the suspension, as prepared and tested in 9.3, and stir in the container for 1 min ± 0,5 min on the mixer. Adjust the suspension temperature to 25 °C ± 1 °C (77 °F ± 2 °F).

9.5.2 Pour the suspension into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn. Pour the suspension to within about 13 mm (0,5 in) 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.

9.5.3 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

9.5.4 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 ml graduated cylinder under the drain tube and continue collecting filtrate until the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

9.6 Calculation — Filtrate volume of the suspension

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (23):

$$V = 2 \cdot V_\mathrm{c} \tag{23}$$

where V_c is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

9.7 Reagents and apparatus — Residue of diameter greater than 75 µm

9.7.1 **Sodium hexametaphosphate** (CAS No. 10124-56-8).

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

9.7.3 **Balance**, with an accuracy of 0,01 g.

9.7.4 Mixer (e.g. Multimixer Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

9.7.5 Container, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

9.7.6 Spatula.

9.7.7 Sieve, 75 µm, in accordance with ASTM E161, approximate dimensions: 76 mm (3,0 in) in diameter, and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that the sieve conforms to ASTM E161 is satisfactory evidence of compliance.

9.7.8 Spray nozzle, 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with a 1/4 TT body, or equivalent) attached to a water line with a 90° elbow.

9.7.9 Low-pressure regulator, capable of regulation to 69 kPa ± 7 kPa (10 psi ± 1 psi).

9.7.10 Evaporating dish.

9.7.11 Wash bottle.

9.8 Procedure — Residue of diameter greater than 75 µm

9.8.1 Weigh 10 g ± 0,01 g of bentonite.

9.8.2 Add the weighed bentonite sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate while stirring on the mixer.

9.8.3 Stir suspension on the mixer for 30 min ± 1 min.

9.8.4 Transfer the sample to the sieve. Use the wash bottle to transfer all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from the spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

9.8.5 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

9.8.6 Dry the residue in the oven to a constant mass. Record the residue mass and total drying time.

9.9 Calculation — Residue of diameter greater than 75 µm

Calculate w_1, the mass fraction residue of particles greater than 75 µm, in percent, as given in Equation (24):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{24}$$

where

m is the sample mass, in grams;

m_2 is the residue mass, in grams.

Record the calculated value.

10 Non-treated bentonite

10.1 Principle

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

10.1.2 Drilling-grade non-treated bentonite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 9, represents the product produced and is controlled by the manufacturer.

Table 9 — Non-treated bentonite physical specifications

Requirement	Standard
Suspension properties:	
Yield point/plastic viscosity ratio	maximum 1,5
Dispersed plastic viscosity, millipascal-seconds	minimum 10
Dispersed filtrate volume, millilitres	maximum 12,5

10.2 Reagents and apparatus — Suspension properties

10.2.1 **Sodium hexametaphosphate** (CAS No. 10124-56-8) solution, 10 % ± 0,5 % by mass.

10.2.2 **Deionized** (or **distilled**) **water**.

10.2.3 **Balance**, with an accuracy of 0,01 g.

10.2.4 **Mixer** (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

10.2.5 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm; (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

10.2.6 **Viscometer**, motor-driven, direct-indicating, in accordance with ISO 10414-1.

10.2.7 **Filter press**, low-pressure/low temperature, in accordance with ISO 10414-1:2008, Clause 7.

10.2.8 **Spatula**.

10.2.9 **Thermometer**, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

10.2.10 **Two graduated cylinders**, 500 ml ± 5 ml (TC) and 10 ml ± 0,1 ml (TC).

10.2.11 **Container**, with lid, capacity about 500 ml.

10.2.12 **Syringe** or **pipette**, 5 ml ± 0,1 ml.

10.2.13 **Two timers**, mechanical or electrical, with accuracy of 0,1 min over the test interval.

10.3 Procedure — Rheology of the suspension

10.3.1 Prepare a suspension of nontreated bentonite. Add 25 g ± 0,01 g of clay (as received) to 350 ml ± 5 ml of deionized water while stirring on the mixer.

10.3.2 After stirring 5 min ± 0,5 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any bentonite adhering to the container walls. Be sure that all bentonite clinging to the spatula is incorporated into the suspension.

10.3.3 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min ± 1 min.

10.3.4 Age the bentonite suspension up to 16 h in a sealed container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.

10.3.5 After ageing the bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 min ± 0,5 min.

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

10.4 Calculation — Rheology of the suspension

Calculate the plastic viscosity, η_P, in millipascal·seconds, according to Equation (25), the yield pointy, η_Y, in pounds per 100 ft^2, according to Equation (26), and the yield point/plastic viscosity ratio, b, according to Equation (27):

$$\eta_\mathrm{P} = R_{600} - R_{300} \tag{25}$$

$$\eta_\mathrm{Y} = R_{300} - \eta_\mathrm{P} \tag{26}$$

$$b = \frac{\eta_\mathrm{Y}}{\eta_\mathrm{P}} \tag{27}$$

where

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

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

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

10.5 Procedure — Dispersed plastic viscosity of the suspension

10.5.1 Recombine all of the bentonite suspension, as prepared and tested in 10.3, and stir in the container for 1 min ± 0,5 min on the mixer.

10.5.2 Add 5 ml ± 0,1 ml of a 10 % solution of sodium hexametaphosphate to the suspension and stir for 3 min ± 0,5 min on the mixer.

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

10.5.4 Calculate and record the dispersed plastic viscosity as per Equation (25).

10.6 Procedure — Dispersed filtrate volume of the suspension

10.6.1 Recombine all of the suspension as prepared and tested in 10.5 and stir in the container for 1 min ± 0,5 min on the mixer. Adjust the suspension temperature to 25 °C ± 1 °C (77 °F ± 2 °F).

10.6.2 Pour the suspension into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn. Pour the suspension to within about 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

10.6.3 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

10.6.4 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering on the drain tube and discard. Place the dry 10 ml graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

10.7 Calculation — Dispersed filtrate volume of the suspension

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (28):

$$V = 2 \cdot V_{\mathrm{c}} \tag{28}$$

where V_{c} is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

11 OCMA grade bentonite

11.1 Principle

11.1.1 OCMA-grade bentonite is a montmorillonite-based clay which, by nature of its source, cannot meet all aspects of Clause 9. This bentonite may have been treated with soda ash, polymer or other chemicals to improve suspension property performance.

11.1.2 OCMA-grade bentonite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 10, represents the product produced and is controlled by the manufacturer.

11.1.3 Manufacturers and licensees shall provide appropriate markings on the container in block letters at least 6 mm (0,25 in) high below the name of the material, the type of treatment of the bentonite with polymer, soda ash or other material.

Table 10 — OCMA grade bentonite physical specifications

Requirement	Standard
Suspension properties:	
Viscometer dial reading at 600 r/min	minimum 30
Yield point/plastic viscosity ratio	maximum 6
Filtrate volume, millilitres	maximum 16,0
Residue of diameter greater than 75 µm	maximum 2,5 % mass fraction

11.2 Reagents and apparatus — Suspension properties

11.2.1 Thermometer, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

11.2.2 Balance, with an accuracy of 0,01 g.

11.2.3 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

11.2.4 Container, of approximate dimensions: depth, 180 mm (7,1 in); *d* top, 97 mm (3-5/6 in); *d* bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

11.2.5 Spatula.

11.2.6 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

11.2.7 Filter press, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7.

11.2.8 Two graduated cylinders, 500 ml ± 5 ml (TC) and 10 ml ± 0,1 ml (TC).

11.2.9 Deionized (or **distilled**) **water**.

11.2.10 Container, capacity approximately 500 ml, with lid.

11.2.11 Two timers, mechanical or electrical, with accuracy of 0,1 min over the test interval.

11.3 Procedure — Rheology of the suspension

11.3.1 Prepare a suspension of OCMA-grade bentonite. Add 22,5 g ± 0,01 g of clay (as received) to 350 ml ± 5 ml of deionized water while stirring on the mixer.

11.3.2 After stirring 5 min ± 0,5 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any bentonite adhering to the container walls. Be sure that all bentonite clinging to the spatula is incorporated into the suspension.

11.3.3 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min ± 1 min.

11.3.4 Age the bentonite suspension up to 16 h in a sealed or covered container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.

11.3.5 After ageing the bentonite suspension, shake well and then pour the suspension into the mixer container. Stir the suspension on the mixer for 5 min ± 0,5 min.

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

11.4 Calculation — Rheology of the suspension

Calculate the plastic viscosity, η_{P}, in millipascal·seconds, according to Equation (29), the yield point, η_{Y}, in pounds per 100 ft^2, according to Equation (30), and the yield point/plastic viscosity ratio, b, according to Equation (31):

$$\eta_{\mathrm{P}} = R_{600} - R_{300} \tag{29}$$

$$\eta_{\mathrm{Y}} = R_{300} - \eta_{\mathrm{P}} \tag{30}$$

$$b = \frac{\eta_{\mathrm{Y}}}{\eta_{\mathrm{P}}} \tag{31}$$

where

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

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

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

11.5 Procedure — Filtrate volume of the suspension

11.5.1 Recombine all of the suspension, as prepared and tested in 11.3, and stir in the container for 1 min ± 0,5 min on the mixer. Adjust the suspension temperature to 25 °C ± 1 °C (77 °F ± 2 °F).

11.5.2 Pour the suspension into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn. Pour the suspension to within about 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

11.5.3 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

11.5.4 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 ml graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of the filtrate collected.

11.6 Calculation — Filtrate volume of the suspension

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (32):

$$V = 2 \cdot V_{\mathrm{c}} \tag{32}$$

where V_{c} is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

11.7 Reagents and apparatus — Residue of diameter greater than 75 µm

11.7.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

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

11.7.3 Balance, with an accuracy of 0,01 g.

11.7.4 Mixer (e.g. Multimixer Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

11.7.5 Container, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

11.7.6 Spatula.

11.7.7 Sieve, 75 µm, in accordance with ASTM E161, approximate dimensions: 76 mm (3,0 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E161 is satisfactory evidence of compliance.

11.7.8 Spray nozzle, 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with a 1/4 TT body, or equivalent), attached to water line with 90° elbow.

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

11.7.10 Evaporating dish.

11.7.11 Wash bottle.

11.8 Procedure — Residue of diameter greater than 75 µm

11.8.1 Weigh 10 g ± 0,01 g of bentonite.

11.8.2 Add the weighed sample to approximately 350 ml of water, containing about 0,2 g of sodium hexametaphosphate while stirring on the mixer.

11.8.3 Stir suspension on the mixer for 30 min ± 1 min.

11.8.4 Transfer the sample to the sieve. Use the wash bottle to transfer all the material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from a spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

11.8.5 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

11.8.6 Dry the residue in the oven to a constant mass. Record the residue mass and total drying time as m_2.

11.9 Calculation — Residue of diameter greater than 75 µm

Calculate w_1, the mass fraction residue of particles greater than 75 µm, in percent, as given in Equation (33):

$$w_1 = 100\left(\frac{m_2}{m}\right) \qquad (33)$$

where

m is the sample mass, in grams;

m_2 is the residue mass, in grams.

Record the calculated value.

12 Attapulgite

12.1 Principle

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

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

Table 11 — Attapulgite physical specifications

Requirement	Standard
Suspension properties:	
Viscometer dial reading at 600 r/min	minimum 30
Residue of diameter greater than 75 µm	maximum mass fraction 8,0 %
Moisture, percent	maximum mass fraction 16,0 %

12.2 Reagents and apparatus — Suspension properties

12.2.1 Sodium chloride (CAS No. 7647-14-5).

12.2.2 Deionized (or **distilled**) **water**.

12.2.3 Thermometer, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

12.2.4 Balance, with an accuracy of 0,01 g.

12.2.5 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

12.2.6 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

12.2.7 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

12.2.8 Spatula.

12.2.9 Graduated cylinders, two, 500 ml ± 5 ml (TC) and 100 ml ± 1 ml (TC).

12.2.10 Defoamer.

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

12.2.12 Filter paper.

12.2.13 Timer, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

12.2.14 Funnel.

12.3 Procedure — 600 r/min dial reading of the suspension

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

12.3.2 Prepare a suspension of attapulgite. Add 20 g ± 0,01 g (as received) to 350 ml ± 5 ml of saturated salt solution while stirring on the mixer.

12.3.3 After stirring 5 min ± 0,5 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any attapulgite adhering to the container walls. Be sure that all attapulgite clinging to the spatula is incorporated into the suspension.

12.3.4 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min ± 1 min.

12.3.5 Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. Add 2 drops to 3 drops of defoamer and stir in with the spatula to break the surface froth. Place the viscometer cup on the direct-indicating viscometer. The dial reading at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value for 600 r/min is reached. Readings shall be taken at a suspension test temperature of 25 °C ± 1 °C (77 °F ± 2 °F).

12.4 Reagent and apparatus — Residue of diameter greater than 75 µm

12.4.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

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

12.4.3 Balance, with an accuracy of 0,01 g.

12.4.4 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

12.4.5 Container, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

12.4.6 Spatula.

12.4.7 Sieve, 75 µm, in accordance with ASTM E161, approximate dimensions: 76 mm (3,0 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that the sieve conforms to ASTM E161 is satisfactory evidence of compliance.

12.4.8 Spray nozzle, 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with a 1/4 TT body, or equivalent) attached to a water line with a 90° elbow.

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

12.4.10 Evaporating dish.

12.4.11 Wash bottle.

12.5 Procedure — Residue of diameter greater than 75 µm

12.5.1 Weigh 10 g ± 0,01 g of attapulgite.

12.5.2 Add the weighed attapulgite sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate.

12.5.3 Stir the suspension on the mixer for 30 min ± 1 min.

12.5.4 Transfer the sample to the sieve. Use the wash bottle to transfer all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from the spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

12.5.5 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

12.5.6 Dry the residue in the oven to a constant mass. Record the residue mass as m_2 and total drying time.

12.6 Calculation — Residue of diameter greater than 75 µm

Calculate w_1, the mass fraction residue of particles greater than 75 µm, in percent, as given in Equation (34):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{34}$$

where

m is the sample mass, in grams;

m_2 is the residue mass, in grams.

Record the calculated value.

12.7 Reagent and apparatus — Moisture

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

12.7.2 Balance, with an accuracy of 0,01 g.

12.7.3 Evaporating dish.

12.7.4 Spatula.

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

12.8 Procedure — Moisture

12.8.1 Weigh 10 g ± 0,01 g of attapulgite sample into a tared evaporating dish. Record the mass as m.

12.8.2 Dry the sample in the oven to a constant mass. Record the total drying time.

12.8.3 Cool to room temperature in desiccator.

12.8.4 Reweigh the evaporating dish containing the dry attapulgite. Record the residue mass as m_2.

12.9 Calculation — Moisture

Calculate w_6, the mass fraction moisture, in percent, as given in Equation (34):

$$w_6 = 100 \frac{(m - m_2)}{m} \tag{35}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

13 Sepiolite

13.1 Principle

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

13.1.2 Drilling-grade sepiolite shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 12, represents the product produced and is controlled by the manufacturer.

Table 12 — Sepiolite physical specifications

Requirement	Standard
Suspension properties:	
Viscometer dial reading at 600 r/min	minimum 30
Residue of diameter greater than 75 µm	maximum mass fraction 8,0 %
Moisture, percent	maximum mass fraction 16,0 %

13.2 Reagents and apparatus — Suspension properties

13.2.1 Sodium chloride (CAS No. 7647-14-5).

13.2.2 Deionized (or **distilled**) **water**.

13.2.3 Defoamer.

13.2.4 Thermometer, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

13.2.5 Balance, with an accuracy of 0,01 g.

13.2.6 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

13.2.7 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

13.2.8 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

13.2.9 Spatula.

13.2.10 Graduated cylinders, two, 500 ml ± 5 ml (TC) and 100 ml ± 1 ml (TC).

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

13.2.12 Filter paper.

13.2.13 Timer, mechanical or electrical, with an accuracy of ± 1 min over test interval.

13.2.14 Funnel.

13.3 Procedure — 600 r/min dial reading of suspension

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

13.3.2 Prepare a suspension of sepiolite. Add 20 g ± 0,01 g (as received) to 350 ml ± 5 ml of saturated salt solution while stirring on the mixer.

13.3.3 After stirring for 5 min ± 0,5 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any sepiolite adhering to the container walls. Be sure that all sepiolite clinging to the spatula is incorporated into the suspension.

13.3.4 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total stirring time shall equal 20 min ± 1 min.

13.3.5 Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. Add 2 drops to 3 drops of defoamer and stir in with the spatula to break the surface froth. Place the viscometer cup on direct-indicating viscometer. The dial reading at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value for 600 r/min is reached. Readings shall be taken at a suspension test temperature of 25 °C ± 1 °C (77 °F ± 2 °F).

13.4 Reagents and apparatus — Residue of diameter greater than 75 µm

13.4.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

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

13.4.3 Balance, with an accuracy of 0,01 g.

13.4.4 Mixer (e.g. Multimixer Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

13.4.5 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

13.4.6 Spatula.

13.4.7 Sieve, 75 µm, in accordance with ASTM E161, approximate dimensions: 76 mm (3,0 in) in diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that the sieve conforms to ASTM E161 is satisfactory evidence of compliance.

13.4.8 Spray nozzle, 1/4 TT body (Spraying Systems Company, No. TG 6.5 tip with a 1/4 TT body, or equivalent), attached to a water line with a 90 degree elbow.

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

13.4.10 Evaporating dish.

13.4.11 Wash bottle.

13.5 Procedure — Residue of diameter greater than 75 µm

13.5.1 Weigh 10 g ± 0,01 g of sepiolite.

13.5.2 Add the weighed sepiolite sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate.

13.5.3 Stir the suspension on the mixer for 30 min ± 1 min.

13.5.4 Transfer the sample to the sieve. Use the wash bottle to transfer all material from the container to the sieve. Wash the material on the sieve with water controlled to 69 kPa ± 7 kPa (10 psi ± 1 psi) from the spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

13.5.5 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

13.5.6 Dry the residue in the oven to a constant mass. Record the residue mass as m_2 and total drying time.

13.6 Calculation — Residue of diameter greater than 75 µm

Calculate w_1, the mass fraction residue of particles greater than 75 µm, in percent, as given in Equation (36):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{36}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

13.7 Reagents and apparatus — Moisture

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

13.7.2 Balance, with an accuracy of 0,01 g.

13.7.3 Evaporating dish.

13.7.4 Spatula.

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

13.8 Procedure — Moisture

13.8.1 Weigh 10 g ± 0,01 g of sepiolite sample into a tared evaporating dish. Record as m.

13.8.2 Dry the sample in the oven to a constant mass. Record the total drying time.

13.8.3 Cool to room temperature in a desiccator.

13.8.4 Reweigh the evaporating dish containing the dry sepiolite. Record the residue mass as m_2.

13.9 Calculation — Moisture

Calculate w_6, the mass fraction moisture, in percent, as given in Equation (37):

$$w_6 = 100 \frac{(m - m_2)}{m} \tag{37}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

14 Technical-grade low-viscosity CMC (CMC-LVT)

14.1 Principle

14.1.1 Technical-grade low-viscosity carboxymethylcellulose (CMC-LVT) is an alkali metal salt of carboxymethylcellulose. The manufacturer shall maintain documentation of the analysis of the cellulosic raw material used.

14.1.2 The product is a free-flowing or granulated powder and is not normally purified of the by-products formed in the reaction.

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

Table 13 — CMC-LVT physical specifications

Requirement	Standard
Starch or starch derivates presence	No
Solution properties	
Viscometer dial reading at 600 r/min	maximum 90
Filtrate volume, millilitres	maximum 10

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

14.2 Reagents and apparatus — Determination of starch and starch derivatives

14.2.1 Distilled (or **deionized**) **water**.

14.2.2 Iodine solution (CAS No. 7553-56-2), 0,1 N.

14.2.3 Potassium iodide (CAS No. 7681-11-0).

14.2.4 Sodium hydroxide (NaOH) (CAS No. 1310-73-2), dilute solution, 0,1 % to 0,5 %.

14.2.5 Mixer (e.g. Multimixer® model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

14.2.6 Container, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

14.2.7 Spatula.

14.2.8 Balance, with accuracy of 0,01 g.

14.2.9 Volumetric flask, 100 ml.

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

14.2.11 Timer, mechanical or electric, with an accuracy of 0,1 min over the test interval.

14.2.12 pH meter.

14.2.13 Test tubes, four.

14.3 Procedure — Determination of starch and starch derivatives

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

14.3.2 An iodine/iodide solution is mixed with a solution of the polymer being analysed. In the presence of amylose (linear fraction of starch), a coloured complex is formed.

14.3.3 Prepare the iodine/iodide solution using a 100 ml ± 0,1 ml volumetric flask. Add 10 ml ± 0,1 ml of the 0,1 N iodine solution. Add 0,60 g ± 0,01 g of the potassium iodide (KI) and dissolve by gently swirling the volumetric flask. Bring to the 100 ml mark with deionized water and mix thoroughly. Record the day of preparation.

Store the prepared iodine/iodide solution in a sealed container, in a dark, cool, dry place. It may be used for up to three months. After the date of expiry, discard the solution and prepare again.

14.3.4 Prepare a 5 % solution of the water-soluble polymer under examination (CMC-LVT) by adding 380 g ± 0,1 g of deionized water to the container. Add 20 g ± 0,1 g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.

14.3.5 After stirring for 5 min ± 0,1 min, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container walls. Be sure that all of the polymer clinging to the spatula is incorporated into the solution.

14.3.6 Measure the pH. If the pH value is less than 10, raise the pH to 10 by adding, drop-wise, the dilute NaOH solution.

14.3.7 Replace the container on the mixer and continue to stir. Total mixing time shall equal 20 min ± 1 min.

14.3.8 Put 2 ml ± 0,1 ml of the polymer solution in a test tube and add, drop-wise, in portions of 3 drops at a time, up to 30 drops of the iodine/iodide solution.

14.3.9 Prepare three blank tests using only deionized water with 3 drops, 9 drops and 30 drops, respectively, of iodine/iodide solution for comparison.

14.3.10 After every 3 drop addition, swirl the test tube gently and compare the colour of the solution under examination with the blank tests. The colour comparison should be made against a white background.

14.4 Interpretation — Determination of starch and starch derivatives

14.4.1 The sample under examination gives a yellow colour comparable to one of the blank tests, if and only if, the sample does not contain any starch or starch derivatives.

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

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

14.4.4 The development of any other colour is a strong indication of the presence of starch or starch derivatives.

14.4.5 Instant de-colourizing indicates the presence of a reducing agent. In this case, continue the drop-wise addition of the iodine/iodide solution and compare the colour obtained with cases 14.4.1 through 14.4.4.

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

14.5 Reagents and apparatus — Solution properties of water-soluble polymers

14.5.1 **API standard evaluation base clay** (see 4.2.5).

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

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

14.5.4 **Deionized** (or **distilled**) **water**.

14.5.5 **Thermometer**, accurate ± 0,5 °C (± 1,0 °F) over the temperature range specified in the procedure.

14.5.6 **Balance**, with an accuracy of 0,01 g.

14.5.7 **Mixer** (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

14.5.8 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

14.5.9 Spatula.

14.5.10 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

14.5.11 Timers, two, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

14.5.12 Graduated cylinders, three, 10 ml ± 0,1 ml (TC), 100 ml ± 1 ml (TC), and 500 ml ± 5 ml (TC).

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

14.5.14 Filter press, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7.

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

14.6 Procedure — Viscometer reading in deionized water

14.6.1 Prepare a solution of CMC-LVT. Add 10,5 g ± 0,01 g of CMC-LVT to 350 ml ± 5 ml of deionized water at a uniform rate over a time interval of about 60 s while stirring on the mixer. The CMC-LVT shall be added away from impeller shaft to minimize dusting.

NOTE This is equivalent to 30,0 g/l ± 0,03 g/l.

14.6.2 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-LVT adhering to the container walls. Be sure that all CMC-LVT clinging to the spatula is incorporated into the solution.

14.6.3 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-LVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

14.6.4 Age the solution for 2 h ± 5 min in a sealed or covered container at room temperature, or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F).

14.6.5 After ageing, stir the solution on the mixer for 5 min ± 0,1 min.

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

14.7 Procedure — Filtrate volume of suspension

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

14.7.2 Prepare a clay-based suspension by adding 350 ml ± 5 ml of the saturated salt solution to the mixer container. Add 1,0 g ± 0,1 g of sodium bicarbonate and stir on the mixer for about 1 min.

14.7.3 Slowly add 35,0 g ± 0,1 g of API standard evaluation base clay while stirring on the mixer.

14.7.4 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.

14.7.5 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

14.7.6 Add 3,15 g ± 0,01 g of CMC-LVT to the suspension while stirring on the mixer, adding at a uniform rate over about 60 s. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.

NOTE This is equivalent to 9,01 g/l ± 0,03 g/l.

14.7.7 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-LVT adhering to the container walls. Be sure that all material clinging to the spatula is incorporated into the suspension.

14.7.8 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-LVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

14.7.9 Age the suspension for 2 h ± 5 min in a sealed or covered container at room temperature, or in a constant-temperature device. Record the storage temperature.

14.7.10 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

14.7.11 Immediately pour the CMC-LVT-treated suspension into a filter press cell. Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 2 °F). Pour the suspension to within 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

14.7.12 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

14.7.13 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

14.8 Calculation — Filtrate volume of the suspension

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (38):

$$V = 2 \cdot V_\mathrm{c} \qquad (38)$$

where V_c is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

15 Technical-grade high-viscosity CMC (CMC-HVT)

15.1 Principle

15.1.1 Technical-grade high-viscosity carboxymethylcellulose (CMC-HVT) is an alkali metal salt of carboxymethylcellulose. The manufacturer shall maintain documentation of the analysis of the cellulosic raw material used.

15.1.2 The product is a free-flowing or granulated powder and is not normally purified of by-products formed in the reaction.

15.1.3 CMC-HVT shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 14, represents the product produced and is controlled by the manufacturer.

Table 14 — CMC-HVT physical specifications

Requirement	Standard
Starch or starch derivates presence	No
Solution properties:	
Viscometer dial reading at 600 r/min	
— in deionized water	minimum 30
— in 40 g/l salt solution	minimum 30
— in saturated salt water	minimum 30
Filtrate volume, millilitres	maximum 10,0

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

15.2 Reagents and apparatus — Determination of starch and starch derivatives

15.2.1 Distilled (or **deionized**) **water**.

15.2.2 Iodine solution (CAS No. 7553-56-2), 0,1 N.

15.2.3 Potassium iodide (CAS No. 7681-11-0).

15.2.4 Sodium hydroxide (NaOH) (CAS No. 1310-73-2), dilute solution, 0,1 % to 0,5 %.

15.2.5 Mixer (e.g. Multimixer® model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

15.2.6 Container, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

15.2.7 Spatula.

15.2.8 Balance, with accuracy of 0,01 g.

15.2.9 Volumetric flask, 100 ml.

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

15.2.11 Timer, mechanical or electric, with an accuracy of 0,1 min over the test interval.

15.2.12 pH meter.

15.2.13 Test tubes, four.

15.3 Procedure — Determination of starch and starch derivatives

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

15.3.2 An iodine/iodide solution is mixed with a solution of the polymer being analysed. In the presence of amylose (linear fraction of starch), a coloured complex is formed.

15.3.3 Prepare the iodine/iodide solution using a 100 ml ± 0,1 ml volumetric flask. Add 10 ml ± 0,1 ml of the 0,1 N iodine solution. Add 0,60 g ± 0,01 g of potassium iodide (KI) and dissolve by gently swirling the volumetric flask. Bring to the 100 ml mark with deionized water and mix thoroughly. Record the day of preparation.

Store the prepared iodine/iodide solution in a sealed container, in a dark, cool, dry place. It may be used for up to three months. After the date of expiry, discard the solution and prepare again.

15.3.4 Prepare a 1 % solution of the water-soluble polymer under examination (CMC-HVT) by adding 396 g ± 0,1 g of deionized water to the container. Add 4 g ± 0,1 g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s while stirring on the mixer. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.

15.3.5 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container walls. Be sure that all of the polymer clinging to the spatula is incorporated into the solution.

15.3.6 Measure the pH. If the pH value is less than 10, raise the pH to 10 by adding, drop-wise, the dilute NaOH solution.

15.3.7 Replace the container on the mixer and continue to stir. Total mixing time shall equal 20 min ± 1 min.

15.3.8 Put 2 ml ± 0,1 ml of the polymer solution in a test tube and add, drop-wise, in portions of 3 drops at a time, up to 30 drops of iodine/iodide solution.

15.3.9 Three blank tests using only deionized water with 3 drops, 9 drops and 30 drops, respectively, of iodine/iodide solution shall be prepared for comparison.

15.3.10 After every 3 drop addition, swirl the test tube gently and compare the colour of the solution under examination with the blank tests. The colour comparison should be made against a white background.

15.4 Interpretation — Determination of starch and starch derivatives

15.4.1 The sample under examination gives a yellow colour comparable to one of the blank tests, if and only if, the sample does not contain any starch or starch derivatives.

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

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

15.4.4 The development of any other colour is a strong indication of the presence of starch or starch derivatives.

15.4.5 Instant decolourizing indicates the presence of a reducing agent. In this case, continue the drop-wise addition of the iodine/iodide solution and compare the colour obtained with cases 15.4.1 through 15.4.4.

15.4.6 The presence of starch or starch derivatives is contrary to the product definition in Table 14, therefore, further testing is abandoned.

15.5 Reagents and apparatus — Solution properties of water-soluble polymers

15.5.1 API standard evaluation base clay (see 4.2.5).

15.5.2 Sodium chloride (CAS No. 7647-14-5).

15.5.3 Sodium bicarbonate (CAS No. 144-55-8).

15.5.4 Deionized (or **distilled**) **water**.

15.5.5 Defoamer.

15.5.6 Thermometer, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

15.5.7 Balance, with an accuracy of 0,01 g.

15.5.8 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 In) in diameter, mounted flash side up.

15.5.9 Container, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

15.5.10 Spatula.

15.5.11 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

15.5.12 Containers, glass or plastic, with stoppers or lids, for salt solutions.

15.5.13 Container, capacity approximately 500 ml, with lid.

15.5.14 Volumetric flask, 1 000 ml.

15.5.15 Timers, two, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

15.5.16 Graduated cylinders, three, 10 ml ± 0,1 ml (TC), 100 ml ± 1 ml (TC), and 500 ml ± 5 ml (TC).

15.5.17 Filter press, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7.

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

15.6 Procedure — Viscometer reading in deionized water

15.6.1 Prepare a suspension of CMC-HVT. Add 2,20 g ± 0,01 g of CMC-HVT to 350 ml ± 5 ml of deionized water at a uniform rate over a time interval of about 60 s while stirring on the mixer. CMC-HVT shall be added away from impeller shaft to minimize dusting.

NOTE This is equivalent to 6,29 g/l ± 0,03 g/l.

15.6.2 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all CMC-HVT clinging to the spatula is incorporated into the suspension.

15.6.3 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

15.6.4 Age the solution for up to 16 h in a sealed or covered container at room temperature, or in a constant-temperature device [20 °C to 25 °C (68 °F to 77 °F)]. Record the storage temperature and storage duration.

15.6.5 After ageing, stir the solution on the mixer for 5 min ± 0,1 min.

15.6.6 Pour the solution into the viscometer cup provided with the direct-indicating viscometer. The dial reading at 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value at 600 r/min is reached. The reading shall be taken at a solution test temperature of 25 °C ± 1 °C.

15.7 Procedure — Viscometer reading in 40 g/l salt water

15.7.1 Prepare a 40 g/l salt solution by adding 40 g ± 0,1 g of sodium chloride to a 1 000 ml volumetric flask and diluting with deionized water to the inscribed mark on the flask. Mix thoroughly.

15.7.2 Prepare a solution of CMC-HVT. Add 2,70 g ± 0,01 g of CMC-HVT to 350 ml ± 5 ml of the 40 g/l salt solution at a uniform rate over a time interval of 60 s while stirring on the mixer. Add defoamer if necessary.

NOTE This is equivalent to 7,72 g/l ± 0,03 g/l.

15.7.3 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all CMC-HVT clinging to the spatula is incorporated into the suspension.

15.7.4 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

15.7.5 Age the suspension for up to 16 h in a sealed or covered container at room temperature, or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F). Record the storage temperature and storage duration.

15.7.6 After ageing, stir the solution on the mixer for 5 min ± 0,1 min.

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

15.8 Procedure — Viscometer reading in saturated salt water

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

15.8.2 Prepare a suspension of CMC-HVT. Add 2,50 g ± 0,01 g of CMC-HVT to 350 ml ± 5 ml of saturated salt water at a uniform rate over a time interval of 60 s while stirring on the mixer. Add defoamer if necessary.

15.8.3 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all CMC-HVT clinging to the spatula is incorporated into the suspension.

15.8.4 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

15.8.5 Age the suspension for up to 16 h in a sealed or covered container at room temperature, or in a constant-temperature device regulated to 20 °C to 25 °C (68 °F to 77 °F). Record the storage temperature and storage duration.

15.8.6 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

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

15.9 Procedure — Filtrate volume of the suspension

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

15.9.2 Prepare a clay-based suspension by adding 350 ml ± 5 ml of the saturated salt solution to the mixer container. Add 1,0 g ± 0,1 g of sodium bicarbonate and stir on the mixer for about 1 min.

15.9.3 Slowly add 35,0 g ± 0,1 g of API standard evaluation base clay while stirring on the mixer.

15.9.4 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.

15.9.5 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

15.9.6 Add 3,15 g ± 0,01 g of CMC-HVT to the suspension while stirring on the mixer, adding at a uniform rate over about 60 s. The water-soluble polymer shall be added into the vortex away from the impeller shaft to minimize dusting.

NOTE This is equivalent to 9,01 g/l ± 0,03 g/l.

15.9.7 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure that all material clinging to the spatula is incorporated into the suspension.

15.9.8 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 min and after 10 min. Total mixing time shall equal 20 min ± 1 min.

15.9.9 Age the suspension for 2 h ± 5 min in a sealed or covered container at room temperature, or in a constant-temperature device. Record the storage temperature.

15.9.10 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

15.9.11 Immediately pour the CMC-HVT-treated suspension into a filter press cell. Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 2 °F). Pour the suspension to within 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

15.9.12 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust pressure on the cell to 690 kPa ± 1 kPa (100 psi ± 0,5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

15.9.13 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place the dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

15.10 Calculation — Filtrate volume of the suspension

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (39):

$$V = 2 \cdot V_c \tag{39}$$

where V_c is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

16 Starch

16.1 Principle

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

16.1.2 Drilling-grade starch shall be free-flowing and free from lumps. Drilling-grade starch shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the physical specifications of Table 15, represents the product produced and is controlled by the manufacturer.

Table 15 — Starch physical specifications

Requirement	Standard
Suspension properties:	
Viscometer dial reading at 600 r/min	
— in 40 g/l salt water	maximum 18
— in saturated salt water	maximum 20
Filtrate volume	
— in 40 g/l salt water, millilitres	maximum 10
— in saturated salt water, millilitres	maximum 10
Residue greater than 2 000 µm	no residue

16.2 Reagents and apparatus — Suspension properties

16.2.1 API standard evaluation base clay (see 4.2.5).

16.2.2 Sodium chloride (CAS No. 7647-14-5).

16.2.3 Sodium bicarbonate (CAS No. 144-55-8).

16.2.4 Deionized (or **distilled**) **water**.

16.2.5 Thermometer, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in the procedure.

16.2.6 Balance, with an accuracy of 0,01 g.

16.2.7 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

16.2.8 Container, of approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach® mixer cup No. M110-D).

16.2.9 Container, glass, capacity approximately 500 ml, with lid.

16.2.10 Container, glass or plastics, with stopper or lid, for salt solutions.

16.2.11 Spatula.

16.2.12 Volumetric flask, 1 000 ml.

16.2.13 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

16.2.14 Filter press, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7.

16.2.15 Timers, two, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

16.2.16 Graduated cylinders, three, 10 ml ± 0,1 ml (TC), 100 ml ± 1 ml (TC) and 500 ml ± 5 ml (TC).

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

16.3 Procedure — Viscometer reading in 40 g/l salt water

16.3.1 Prepare a 40 g/l salt-water solution by adding 40 g ± 0,1 g of sodium chloride to a 1 000 ml volumetric flask and diluting with deionized water to the mark inscribed on the flask. Mix thoroughly.

16.3.2 Prepare a clay-based suspension by adding 350 ml ± 5 ml of the 40 g/l salt-water solution to the container. Add 1,0 g ± 0,1 g of sodium bicarbonate and stir on the mixer for about 1 min.

16.3.3 Slowly add 35,0 g ± 0,1 g of API standard evaluation base clay while stirring on the mixer.

16.3.4 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.

16.3.5 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the clay shall equal 20 min ± 1 min.

16.3.6 Add 3,50 g ± 0,01 g of starch to the suspension while stirring on the mixer, adding at a uniform rate over about 60 s.

NOTE This is equivalent to 10,0 g/l ± 0,03 g/l.

16.3.7 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any starch adhering to the container walls. Be sure that all starch clinging to the spatula is incorporated into the suspension.

16.3.8 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any starch clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the starch shall equal 20 min ± 1 min.

16.3.9 Age the suspension up to 24 h in a sealed or covered container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.

16.3.10 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

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

16.4 Procedure — Filtrate volume of 40 g/l salt solution

16.4.1 Pour the suspension from 16.3 into the filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 2 °F). Pour the suspension to within about 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

16.4.2 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

16.4.3 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard the filtrate. Place a dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

16.5 Calculation — Filtrate volume of the 40 g/l salt solution

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (40):

$$V = 2 \cdot V_{\mathrm{c}} \tag{40}$$

where V_{c} is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

16.6 Procedure — Viscometer reading in the saturated salt solution

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

16.6.2 Prepare a clay-based suspension by adding 350 ml ± 5 ml of the saturated salt solution to a container. Add 1,0 g ± 0,1 g of sodium bicarbonate and stir on the mixer for about 1 min.

16.6.3 Slowly add 35,0 g ± 0,1 g of API standard evaluation base clay to the container while stirring on the mixer.

16.6.4 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any clay adhering to the container walls. Be sure that all clay clinging to the spatula is incorporated into the suspension.

16.6.5 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the clay shall equal 20 min ± 1 min.

16.6.6 Add 3,50 g ± 0,01 g of starch to the suspension while stirring on the mixer, adding at a uniform rate over about 60 s.

NOTE This is equivalent to 10,0 g/l ± 0,03 g/l.

16.6.7 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any starch adhering to the container walls. Be sure that all starch clinging to the spatula is incorporated into the suspension.

16.6.8 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any starch clinging to the container walls after another 5 min and another 10 min. Total stirring time after adding the starch shall equal 20 min ± 1 min.

16.6.9 Age the suspension up to 24 h in a sealed or covered container at room temperature or in a constant-temperature device. Record the storage temperature and storage duration.

16.6.10 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

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

16.7 Procedure — Filtrate volume of the saturated salt solution

16.7.1 Pour the suspension from 16.6 into a filter press cell. Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 2 °F). Pour the suspension to within about 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

16.7.2 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Both of these steps shall be completed in less than 15 s. Pressure shall be supplied by compressed air, nitrogen or helium.

16.7.3 At 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard the filtrate. Place a dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

16.8 Calculation — Filtrate volume of the saturated salt solution

Calculate the filtrate volume, V, in millilitres, of the clay suspension as given in Equation (41):

$$V = 2 \cdot V_{\mathrm{c}} \tag{41}$$

where V_{c} is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated filtrate volume.

16.9 Reagents and apparatus — Residue greater than 2 000 µm

Sieve, 2 000 µm, approximate diameter of 203 mm (8,0 in), in accordance with ASTM E11.

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

16.10 Procedure — Residue greater than 2 000 µm

16.10.1 Weigh 25 g ± 0,1 g starch and transfer to the 2 000-µm sieve.

16.10.2 Shake for a maximum of 5 min.

16.10.3 Record the presence or absence of residue.

17 Low-viscosity polyanionic cellulose (PAC-LV)

17.1 Principle

17.1.1 PAC-LV, also referred to as PAC low-viscosity grade, is a water-soluble polymer produced only from cellulose chemically reacted with carboxy-methyl (anionic) groups. The product obtained is further purified to significantly increase the active polymer content. It shall not contain any other polysaccharides such as starch, guar or other naturally occurring polymers or their derivatives. The product is a free-flowing and/or granular powder. See Table 16 for physical requirements.

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

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

NOTE It is recommended that the purity of PAC-LV be greater than 96 % NaCMC on a dry-weight basis.

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

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

17.1.6 PAC-LV is a polyanionic cellulosic polymer that answers all the requirements specified in 17.1.1 through 17.1.5 and, when testing according to the procedure in 17.2, has a viscosity of less than 40 cP 6) and a low-pressure, low-temperature fluid loss of less than 16 ml (corrected).

NOTE Corrected fluid loss is the filtrate volume collected between 7,5 min and 30,0 min, expressed in millilitres, multiplied by 2. It eliminates the spurt loss seen in many fluid-loss tests.

17.1.7 To obtain the best handling in the field, it is recommended that the particle size for PAC LV powder be u 0,8 mm (u 20 mesh).

Table 16 — PAC-LV physical requirements

Requirement	Standard
Presence of starch or starch derivatives	Absent
Moisture content	Maximum 10 %
Apparent viscosity	Maximum 40 cP
Filtrate volume	Maximum 16 ml

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6) 1 cP = 1 mPa·s.

17.2 Qualitative starch determination in water-soluble polymers

17.2.1 Description

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

17.2.1.2 An iodine/iodide solution is mixed with a solution of the polymer being analysed. In the presence of amylose (linear fraction of starch), a coloured complex is formed.

17.2.2 Reagents and materials

17.2.2.1 **Deionized** (or **distilled**) **water**.

17.2.2.2 **Iodine solution**, e.g. Merck 1.09.099.1000 (CAS No. 7553-56-2) 7), 0,05 mol/l.

17.2.2.3 **Potassium iodide**, Merck 1.05043.0250 PA (CAS No. 7681-11-0) 8).

17.2.2.4 **Sodium hydroxide** (NaOH) (CAS no. 1310-73-2), dilute solution, 0,1% to 0,5 %.

17.2.3 Apparatus

17.2.3.1 **Mixer** (e.g. Multimixer® model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

17.2.3.2 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton® Beach mixer cup No. M110-D, or equivalent). (An alternative container option is a 600 ml glass jar.)

17.2.3.3 **Laboratory spoon**.

17.2.3.4 **Spatula**.

17.2.3.5 **Balance,** accuracy of 0,01 g.

17.2.3.6 **Volumetric flask**, 100 ml.

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

17.2.3.8 **Timer,** mechanical or electric, accuracy to 0,1 min.

17.2.3.9 **pH-meter with pH-electrode** (e.g. Thermo Russell type KDCW11) 9).

17.2.3.10 **Polymer dosing device** [Fann® 10) or OFI® 11)].

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7) Merck No. 1.09.099.1000 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

8) Merck 1.05043.0250 PA is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

9) Thermo Russell type KDCW11 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

10) Fann is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

17.2.3.11 Test tube.

17.2.4 Procedure — Preparation of the iodine/iodide solution

17.2.4.1 To a 100 ml ± 0 1 ml volumetric flask, add 10 ml ± 0,1 ml of a 0,05 mol/l iodine solution.

17.2.4.2 Add 0,60 g ± 0,01 g of potassium iodide (KI) and dissolve by gently swirling the volumetric flask.

17.2.4.3 Fill to the 100 ml mark with deionized water and mix thoroughly. Record the day of preparation.

17.2.4.4 The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place and can be used for up to three months. After the date of expiration, the solution should be discarded and prepared again.

17.2.5 Procedure — Preparation of the PAC-LV solution and starch determination

17.2.5.1 Prepare a 5 % solution of the water-soluble polymer under examination. Add 380 g ± 0,1 g of deionized water to the container and add 20 g ± 0,1 g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s. The PAC-LV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a polymer-dosing device (17.2.3.10).

17.2.5.2 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container wall. Be sure that all polymer clinging to the spatula is incorporated into the solution.

17.2.5.3 Measure the pH; if the pH value is less than 10, raise the pH to 10 by adding, drop-wise, the dilute NaOH solution.

17.2.5.4 Replace the container on the mixer and continue to stir. Total mixing time shall equal 20 min ± 1 min.

17.2.5.5 Put 2 ml of the polymer solution in a test tube and add, drop-wise, in portions of 3 drops at a time, up to 30 drops of iodine/iodide solution.

17.2.5.6 Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution should be prepared for comparison.

17.2.5.7 After every 3 drop addition, swirl the test tube gently and compare the colour of the solution under examination with the blank tests. The colour comparison should be made against a white background.

17.2.6 Results — Starch test for PAC-LV

17.2.6.1 If the sample under examination gives a yellow colour comparable to one of the blank tests, the sample does not contain any starch or starch derivatives.

17.2.6.2 The development of any other colour is a strong indication of the presence of starch or starch derivatives.

17.2.6.3 Instant discolouration indicates the presence of a reducing agent; in this case, continue the drop-wise addition of the iodine/iodide solution. Compare the colour obtained with one of the blank tests; see 17.2.6.1.

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11) OFI is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

17.2.6.4 If any colour reaction other than that mentioned under 17.2.6.1 is detected, further testing is abandoned.

17.3 Moisture content

17.3.1 Apparatus

17.3.1.1 **Oven,** regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

17.3.1.2 **Balance,** accuracy of ± 0,01 g.

17.3.1.3 **Evaporating dish**, capacity 150 ml.

17.3.1.4 **Spatula**.

17.3.1.5 **Desiccator**, with calcium sulfate (CAS number 7778-18-9) desiccant, or equivalent.

17.3.2 Procedure

17.3.2.1 Weigh 10 g ± 0,1 g of the PAC-LV sample into a tared evaporating dish. Record the mass as m.

17.3.2.2 Dry the sample in the oven for 4 h.

17.3.2.3 Cool the sample to room temperature in a desiccator.

17.3.2.4 Reweigh the evaporating dish containing the dried PAC-LV. Record the mass as m_2.

17.3.3 Calculation

Calculate w_6, the mass fraction moisture, in percent, as given in Equation (42):

$$w_6 = 100 \frac{(m - m_2)}{m} \tag{42}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

17.4 Filtrate volume

17.4.1 Reagents and materials

17.4.1.1 **Sea salt**, ASTM D1141-98 (2003) 12).

17.4.1.2 **API standard evaluation base clay,** such as provided by API 13).

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12) Sea salt, ASTM D1141-98 (2003), is available from Lake Products Company, Inc., P.O. Box 2248, Maryland Heights 63043 Missouri, USA, and is a commercially available product. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

17.4.1.3 **Potassium chloride** (CAS Number 7447-40-7).

17.4.1.4 **Sodium bicarbonate** (CAS Number 144-55-8).

17.4.1.5 **Deionized** (or **distilled**) **water**.

17.4.2 Apparatus

17.4.2.1 **Thermometer**, range 0 °C to 60 °C, with accuracy ± 0,5 °C (range 32 °F to 140 °F, with an accuracy of ± 1,0 °F).

17.4.2.2 **Balance**, accurate to ± 0,01 g.

17.4.2.3 **Mixer** (e.g. Multimixer Model 9B with 9B29X impellers); each spindle is fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

17.4.2.4 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D).

17.4.2.5 **Spatula**.

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

17.4.2.7 **Viscometer, motor-driven, direct-indicating**, in accordance with ISO 10414-1.

17.4.2.8 **Timers**, two, mechanical or electrical, accurate to ± 0,1 min over the test interval.

17.4.2.9 **Filter press**, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7.

17.4.2.10 **Graduated cylinders**, one 10 ml ± 0,1 ml and one 500 ml ± 5 ml.

17.4.2.11 **Polymer dosing device** (Fann or OFI).

17.4.3 Procedure — Filtrate volume of the PAC-LV

17.4.3.1 Add 42 g ± 0,01 g sea salt to 1 l ± 2 ml of deionized water.

17.4.3.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g potassium chloride (KCl).

17.4.3.3 After stirring 3 min ± 0,1 min, add 1,0 g ± 0,01 g of sodium bicarbonate.

17.4.3.4 After stirring 3 min ± 0,1 min, add 28,0 g ± 0,01 g of API standard evaluation base clay.

17.4.3.5 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.

17.4.3.6 Replace the container on the mixer and continue to stir an additional 5 min ± 0,1 min.

17.4.3.7 Weigh 2,0 g ± 0,01 g of PAC-LV.

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13) API standard evaluation base clay is an example of a suitable product available commercially. Requests for clay should be directed to the API which will forward the request to a supplier for further handling. This information is given for the convenience of users of this Internal Standard and does not constitute an endorsement by ISO of this product.

17.4.3.8 Add the PAC-LV slowly at a uniform rate over a time interval of about 60 s while stirring on the mixer. The PAC-LV should be added away from the impeller shaft but in the vortex to minimize dusting, preferably by means of a polymer-dosing device (17.4.2.11).

17.4.3.9 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its side with the spatula to dislodge any PAC-LV adhering to the container walls. Be sure that all PAC-LV clinging to the spatula is incorporated into the suspension.

17.4.3.10 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-LV after another 5 min and 10 min. Total mixing time elapsed from the beginning of PAC-LV addition shall equal 20 min ± 1 min.

17.4.3.11 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 2 °F). Record the storage temperature and storage duration.

17.4.3.12 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

17.4.3.13 Pour the PAC-LV suspension into a filter press cell. Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with ISO 10414-1. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 2 °F). Pour the suspension to within about 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

17.4.3.14 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Pressure shall be supplied by compressed air, nitrogen or helium and applied within 15 s.

17.4.3.15 After 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place a dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of filtrate collected.

17.4.4 Calculation — Filtrate volume of PAC-LV

Calculate V, the total filtrate volume, expressed in millilitres, as given in Equation (43):

$$V = 2 \cdot V_{\mathrm{C}} \tag{43}$$

where V_{C} is the filtrate volume, expressed in millilitres, collected between 7,5 min and 30 min.

17.5 Fluid apparent viscosity

17.5.1 Procedure — Fluid apparent viscosity

17.5.1.1 Add 42 g ± 0,01 g sea salt to 1 l ± 2 ml of deionized water.

17.5.1.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g of potassium chloride (KCl).

17.5.1.3 Weigh 5,0 g ± 0,01 g of the PAC-LV. Add the PAC-LV slowly at a uniform rate over a time interval of about 1 min while stirring on the mixer. The PAC-LV should be added away from the impeller shaft but in the vortex to minimize dusting.

17.5.1.4 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.

17.5.1.5 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-LV after another 5 min and 10 min. Total mixing time elapsed from beginning of the PAC-LV addition shall equal 20 min ± 1 min.

17.5.1.6 Statically age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 2 °F). Record the storage temperature and storage duration.

17.5.1.7 Stir the suspension on the mixer for 5 min ± 0,1 min.

17.5.1.8 Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading at the 600 r/min rotor speed setting of the viscometer shall be taken at a suspension test temperature of 25 °C ± 1 °C (77 °F ± 2 °F).

17.5.2 Calculation — Fluid apparent viscosity

Calculate the test fluid viscosity, V_A, expressed in centipoise, from Equation (44):

$$V_\mathrm{A} = \frac{R_{600}}{2} \tag{44}$$

where R_{600} is the viscometer dial reading at 600 r/min.

Record the calculated value.

18 High-viscosity polyanionic cellulose (PAC-HV)

18.1 Principle

18.1.1 PAC-HV, also referred to as API PAC regular grade, is a water-soluble polymer produced only from cellulose chemically reacted with carboxy-methyl (anionic) groups. The product obtained is further purified to significantly increase the active polymer content. It shall not contain any other polysaccharides, such as starch, guar or other naturally occurring polymers or their derivatives. The product is a free-flowing and/or granular powder. See Table 17 for physical requirements.

NOTE It is recommended that the purity of the PAC-HV be greater than 96 % NaCMC on a dry-weight basis.

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

18.1.3 The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-HV. Specification parameters are developed for this material.

18.1.4 PAC-HV shall be free of any foreign polymer. Therefore, a qualitative starch determination shall be performed before the performance tests. If starch is found, no further testing should be performed and the sample shall be rejected.

18.1.5 A synthetic-seawater drilling fluid is used for determining the filtration control and apparent viscosity of the PAC-HV.

18.1.6 PAC-HV is a polyanionic cellulosic polymer that answers all the requirements in 18.1.1 through 18.1.5 and, when tested in accordance with 18.2, has an API viscosity above 50 cP 6) and a low-pressure/low-temperature fluid loss of less than 23 ml (corrected).

NOTE Corrected fluid loss is the filtrate volume collected between 7,5 min and 30,0 min, in millilitres, multiplied by 2. It eliminates the spurt loss seen in many fluid-loss tests.

18.1.7 To obtain the best handling in the field, it is recommended that the particle size for the PAC-HV powder be u 0,8 mm (u 20 mesh).

Table 17 — PAC-HV physical requirements

Requirement	Standard
Presence of starch or starch derivatives	Absent
Moisture content	Maximum 10 %
Apparent viscosity	Minimum 50 cP
API filtrate volume	Maximum 23 ml

18.2 Qualitative starch determination in water soluble polymers

18.2.1 Principle

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

18.2.1.2 An iodine/iodide solution is mixed with a solution of the polymer being analysed. In the presence of amylose (linear fraction of starch), a coloured complex is formed.

18.2.2 Reagents and materials

18.2.2.1 **Deionized** (or **distilled**) **water**.

18.2.2.2 **Iodine solution**, e.g. Merck 1.09.099.1000 (CAS No. 7553-56-2), 0,05 mol/l.

18.2.2.3 **Potassium iodide**, Merck 1.05043.0250 PA (CAS No. 7681-11-0).

18.2.2.4 **Sodium hydroxide** (NaOH) (CAS no. 1310-73-2), dilute, 0,1 % to 0,5 %.

18.2.3 Apparatus

18.2.3.1 **Mixer** (e.g. Multimixer® model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

18.2.3.2 **Container**, approximate dimensions: depth, 180 mm (7,1 in); *d* top, 97 mm (3-5/6 in); *d* bottom, 70 mm (2,75 in) (e.g. Hamilton® Beach mixer cup No. M110-D, or equivalent). For the alternative mixer option is a glass jar of 600 ml.

18.2.3.3 **Laboratory spoon**.

18.2.3.4 **Spatula**.

18.2.3.5 **Balance,** accurate to 0,01 g.

18.2.3.6 **Volumetric flask**, 100 ml.

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

18.2.3.8 **Timer**, mechanical or electric, accurate to 0,1 min.

18.2.3.9 **pH-meter with pH-electrode** (e.g. Thermo Russell type KDCW11).

18.2.3.10 Polymer-dosing device (Fann or OFI).

18.2.3.11 Test tube.

18.2.4 Procedure — Preparation of the iodine/iodide solution

18.2.4.1 To a 100 ml ± 0,1 ml volumetric flask, add 10 ml ± 0,1 ml of a 0,05 mol/l iodine solution.

18.2.4.2 Add 0,60 g ± 0,01 g of potassium iodide (KI) and dissolve by gently swirling the volumetric flask.

18.2.4.3 Fill to the 100 ml mark with deionized water and mix thoroughly. Record the day of preparation.

18.2.4.4 The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place and can be used for up to three months. After the date of expiration, the solution should be discarded and prepared again.

18.2.5 Procedure — Preparation of the PAC-HV solution and starch determination

18.2.5.1 Prepare a 1 % solution of the water-soluble polymer under examination. Add 396 g ± 0,1 g of deionized water to the container and add 4 g ± 0,1 g of the water-soluble polymer under examination at a uniform rate over a time interval of 60 s to 120 s. The PAC-HV should be added into the vortex away from the impeller shaft to minimize dusting, preferably with a polymer-dosing device (18.2.3.10).

18.2.5.2 After stirring about 5 min ± 0,1 min, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any polymer adhering to the container wall. Be sure that all the polymer clinging to the spatula is incorporated into the solution.

18.2.5.3 Measure the pH; if the pH value is less than 10, raise the pH to 10 by adding, drop-wise, dilute NaOH solution.

18.2.5.4 Replace the container on the mixer and continue to stir. Total mixing time shall equal 20 min ± 1 min.

18.2.5.5 Put 2 ml of the polymer solution in a test tube and add, drop-wise, in portions of 3 drops at a time, up to 30 drops of iodine/iodide solution.

18.2.5.6 Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution should be prepared for comparison.

18.2.5.7 After every 3 drop addition, swirl the test tube gently and compare the colour of the solution under examination with the blank tests. The colour comparison should be made against a white background.

18.2.6 Results — Starch test for PAC-HV

18.2.6.1 If the sample under examination gives a yellow colour comparable to one of the blank tests, the sample does not contain any starch or starch derivatives.

18.2.6.2 The development of any other colour is a strong indication of the presence of starch or starch derivatives.

18.2.6.3 Instant discolouration indicates the presence of a reducing agent; in this case, continue the drop-wise addition of the iodine/iodide solution.

18.2.6.4 If any colour-reaction is detected other than that mentioned under 18.2.1, the further testing shall be abandoned.

18.3 Moisture content

18.3.1 Apparatus

18.3.1.1 **Oven**, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

18.3.1.2 **Balance**, accurate to ± 0,01 g.

18.3.1.3 **Evaporating dish**.

18.3.1.4 **Spatula**.

18.3.1.5 **Desiccator**, with calcium sulfate (CAS Number 7778-18-9) desiccant, or equivalent.

18.3.1.6 **Polymer-dosing device** (Fann or OFI).

18.3.2 Procedure

18.3.2.1 Weigh 10 g ± 0,1 g of the PAC-HV sample into a tared evaporating dish. Record the mass as m.

18.3.2.2 Dry the sample in the oven for 4 h.

18.3.2.3 Cool the sample to room temperature in the desiccator.

18.3.2.4 Reweigh the evaporating dish containing the PAC-HV. Record the mass as m_2.

18.3.3 Calculation

Calculate the mass fraction moisture, w_6, in percent, as given in Equation (45):

$$w_6 = 100 \frac{(m - m_2)}{m} \tag{45}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

18.4 Filtrate volume

18.4.1 Reagents and materials

18.4.1.1 **Sea salt**, ASTM D1141-98 (2003).

18.4.1.2 **API standard evaluation base clay**, such as provided by API.

18.4.1.3 **Potassium chloride** (CAS Number 7447-40-7).

18.4.1.4 **Sodium bicarbonate** (CAS Number 144-55-8).

18.4.1.5 **Deionized** (or **distilled**) **water**.

18.4.2 Apparatus

18.4.2.1 **Thermometer**, range 0 °C to 60 °C, ± 0,5 °C (range 32 °F to 140 °F, ± 1,0 °F).

18.4.2.2 **Balance**, accurate to ± 0,01 g.

18.4.2.3 **Mixer** (e.g. Multimixer Model 9B with 9B29X impellers); each spindle is fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

18.4.2.4 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D).

18.4.2.5 **Spatula**.

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

18.4.2.7 **Viscometer, motor-driven, direct-indicating**, in accordance with ISO 10414-1.

18.4.2.8 **Timers**, two, mechanical or electrical, accurate to ± 0,1 min over the test interval.

18.4.2.9 **Filter press**, low-pressure/low-temperature, in accordance with ISO 10414-1:2008, Clause 7.

18.4.2.10 **Graduated cylinders**, one 10 ml ± 0,1 ml and one 500 ml ± 5 ml.

18.4.2.11 **Polymer-dosing device** (Fann or OFI).

18.4.3 Procedure — Filtrate volume of the PAC-HV

18.4.3.1 Add 42 g ± 0,01 g of sea salt to 1 l ± 2 ml of deionized water.

18.4.3.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g of potassium chloride (KCl).

18.4.3.3 After stirring 3 min ± 0,1 min, add 1,0 g ± 0,01 g of sodium bicarbonate.

18.4.3.4 After stirring 3 min ± 0,1 min, add 28,0 g ± 0,01 g of the API standard evaluation base clay.

18.4.3.5 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.

18.4.3.6 Replace the container on the mixer and continue to stir an additional 5 min ± 0,1 min.

18.4.3.7 Weigh 1,0 g ± 0,01 g of PAC-HV.

18.4.3.8 Add the PAC-HV slowly at a uniform rate over a time interval of about 60 s while stirring on the mixer. The PAC-HV should be added away from the impeller shaft but in the vortex to minimize dusting, preferably by means of a polymer-dosing device (18.4.2.11).

18.4.3.9 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its side with the spatula to dislodge any PAC-HV adhering to the container walls. Be sure that all PAC-HV clinging to the spatula is incorporated into the suspension.

18.4.3.10 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-HV after another 5 min and 10 min. Total mixing time elapsed from the beginning of PAC-HV addition shall equal 20 min ± 1 min.

18.4.3.11 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 2 °F). Record the storage temperature and storage duration.

18.4.3.12 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

18.4.3.13 Pour the PAC-HV suspension into a filter press cell. Before adding the suspension, be sure that each part of the filter cell is dry and that none of the gaskets is distorted or worn, in accordance with ISO 10414-1. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 2 °F). Pour the suspension to within about 13 mm (0,5 in) of the top of the cell. Complete the assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

18.4.3.14 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). The pressure shall be supplied by compressed air, nitrogen or helium and applied within 15 s.

18.4.3.15 After 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering to the drain tube and discard. Place a dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate to the end of the second timer set at 30 min. Remove the graduated cylinder and record the volume of the filtrate collected.

18.4.4 Calculation — Filtrate volume of the PAC-HV

Calculate the filtrate volume, V, expressed in millilitres, from Equation (46):

$$V = 2 \cdot V_{\mathrm{c}} \tag{46}$$

where V_{c} is the filtrate volume collected, expressed in millilitres, between 7,5 min and 30 min.

18.5 Fluid apparent viscosity

18.5.1 Procedure — Fluid apparent viscosity

18.5.1.1 Add 42 g ± 0,01 g of sea salt to 1 l ± 2 ml of deionized water.

18.5.1.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g of potassium chloride (KCl).

18.5.1.3 Weigh 3,0 g ± 0,01 g of PAC-HV. Add the PAC-HV slowly at a uniform rate over a time interval of about 1 min while stirring on the mixer. The PAC-HV should be added away from the impeller shaft but in the vortex to minimize dusting.

18.5.1.4 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its side with the spatula to dislodge any material adhering to the container wall. Be sure that all material clinging to the spatula is incorporated into the suspension.

18.5.1.5 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any adhering PAC-HV after another 5 min and 10 min. Total mixing time elapsed from beginning of the PAC-HV addition shall equal 20 min ± 1 min.

18.5.1.6 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 2 °F). Record the storage temperature and storage duration.

18.5.1.7 Stir the suspension on the mixer for 5 min ± 0,1 min.

18.5.1.8 Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading at the 600 r/min rotor speed setting of the viscometer shall be taken at a suspension test temperature of 25 °C ± 1 °C (77 °F ± 2 °F).

18.5.2 Calculation — Fluid apparent viscosity

Calculate the test fluid viscosity, V_A, expressed in centipoise from Equation (47):

$$V_A = \frac{R_{600}}{2} \qquad (47)$$

where R_{600} is the viscometer dial reading at 600 r/min.

Record the calculated value.

19 Drilling-grade xanthan gum

19.1 Principle

19.1.1 Xanthan gum is a water-soluble polysaccharide, commercially produced by a distinct fermentation process of *Xanthomonas campestris*, and shall not contain any other polysaccharide, such as starch, guar gum or other naturally occurring polymers or their derivatives. The product may contain up to 3 % of a material added specifically to enhance dispersibility or solubility of the product. Xanthan gum is an off-white, free-flowing granular powder.

19.1.2. Xanthan gum is used in a wide variety of water-based drilling, workover or completion fluids as a rheology modifier to develop low-shear-rate viscosity and enhance suspension properties.

19.1.3 This procedure is a simple and reproducible method for assessing the performance properties of xanthan gum used in drilling operations. Other specifications may be established for pay-zone applications, including stimulation, workover or completion operations.

19.1.4 Xanthan gum shall be free of any foreign polymer including cellulosics, starch or guar gum. Qualitative tests should be performed to detect the presence of guar or starch, which are known to have synergistic effects on the viscosity of xanthan gum solutions.

19.1.5 A solution of xanthan gum in synthetic seawater is used to determine the viscosity characteristics of xanthan gum.

19.1.6 For optimum handling and mixing of the polymer in the field, it is recommended that the particle size of xanthan gum powder be less than 0,4 mm (0,17 in; 425 µm). Xanthan gum shall conform to the specifications of Table 18.

19.2 Qualitative starch determination in xanthan gum

19.2.1 Principle

19.2.1.1 The purpose of this test is to identify the presence of guar, starch and starch derivatives in xanthan gum, supplied in powder or granular form. If starch is found, no further testing should be performed and the sample shall be rejected.

19.2.1.2 An iodine/iodide solution is mixed with a solution of the xanthan gum being analysed. In the presence of amylose (linear fraction of starch), a coloured complex is formed.

19.2.2 Reagents

19.2.2.1 **Deionized** (or **distilled**) **water**.

Table 18 — Xanthan gum physical requirements

Requirement	Standard
Starch, guar, or their derivatives	Absent
Moisture content	Maximum 13 %
Screen analysis	
Less than 425 µm (11/64 in)	Minimum 95 %
Less than 75 µm	Maximum 50 %
Viscosity [a]	
Rotational viscometer, 300 r/min	Minimum 11 cP 6) (minimum 55 dial reading)
Rotational viscometer, 6 r/min	Minimum 180 cP (minimum 18 dial reading)
Rotational viscometer, 3 r/min	Minimum 320 cP (minimum 16 dial reading)
Brookfield LV, 1,5 r/min	Minimum 1 950 cP

[a] For rotational viscometer, equipped with f0.2 torsion spring, R1/B1 configuration:

d) 300 r/min, cP equals the dial reading times 0,2;
e) 6 r/min, cP equals the dial reading times 10,0;
f) 3 r/min, cP equals the dial reading times 20,0.

19.2.2.2 **Iodine solution**, e.g. Merck 1.09.099.1000 (CAS No. 7553-56-2), 0,05 mol/l.

19.2.2.3 **Potassium iodide**, Merck 1.05043.0250 PA (CAS No. 7681-11-0).

19.2.2.4 **Sodium hydroxide** (NaOH) (CAS no. 1310-73-2), dilute, 0,1 % to 0,5 %.

19.2.3 Apparatus

19.2.3.1 **Mixer** (e.g. Multimixer® model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

19.2.3.2 **Container**, approximate dimensions: depth, 180 mm (7,1 in); *d* top, 97 mm (3-5/6 in); *d* bottom, 70 mm (2,75 in) (e.g. Hamilton® Beach mixer cup No. M110-D, or equivalent). An alternative container option is a 600 ml glass jar.

19.2.3.3 **Laboratory spoon**.

19.2.3.4 **Spatula**.

19.2.3.5 **Balance,** accurate to 0,01 g.

19.2.3.6 **Volumetric flask**, 100 ml.

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

19.2.3.8 **Timer**, mechanical or electric, accurate to 0,1 min.

19.2.3.9 **pH-meter with pH-electrode** (e.g. Thermo Russell type KDCW11).

19.2.3.10 **Polymer-dosing device** (Fann or OFI).

19.2.3.11 Test tube.

19.2.4 Procedure — Preparation of the iodine/iodide solution for qualitative starch determination

19.2.4.1 To a 100 ml ± 0 1 ml volumetric flask, add 10 ml ± 0,1 ml of a 0,05 mol/l iodine solution.

19.2.4.2 Add 0,60 g ± 0,01 g of potassium iodide (KI) and dissolve by gently swirling the volumetric flask.

19.2.4.3 Fill to the 100 ml mark with deionized water and mix thoroughly. Record the day of preparation.

19.2.4.4 The prepared iodine/iodide solution shall be stored in a sealed container, in a dark, cool, dry place and can be used for up to three months. After the date of expiration, the solution should be discarded and prepared again.

19.2.5 Procedure — Preparation of xanthan gum solution for qualitative starch determination

19.2.5.1 Prepare a 1 % solution of the xanthan gum under examination. Add 396 g ± 0,1 g of deionized water to the container and add 4,0 g ± 0,1 g of xanthan gum at a uniform rate over a time interval of 60 s to 120 s. The xanthan gum polymer shall be added into the vortex away from the impeller shaft to minimize dusting, preferably with a polymer-dosing device as mentioned in 19.2.3.10.

19.2.5.2 After stirring about 5 min ± 0,1 min, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any xanthan gum adhering to the container wall. Be sure that all xanthan gum clinging to the spatula is incorporated into the solution.

19.2.5.3 Measure the pH. If the pH value is less than 10, raise the pH to 10 by adding, dropwise, dilute NaOH solution.

19.2.5.4 Replace the container on the mixer and continue to stir. Total mixing time shall be 20 min ± 1 min.

19.2.5.5 Put 2 ml of the xanthan gum solution in a test tube and add, dropwise, in portions of 3 drops at a time, up to 30 drops of the iodine/iodide solution.

19.2.5.6 Three blank tests using only deionized water with 3 drops, 9 drops, and 30 drops, respectively, of iodine/iodide solution should be prepared for comparison.

19.2.5.7 After every 3 drop addition, gently swirl the test tube and compare the colour of the solution under examination with the blank test. The colour comparison should be made against a white background.

19.2.6 Results

19.2.6.1 If the sample under examination gives a yellow colour comparable to one of the blank test, the sample does not contain any starch or starch derivatives.

19.2.6.2 The development of a light green to dark blue colour, either in solution or as a precipitate, indicates the presence of starch (amylose fraction).

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

19.2.6.4 The development of any other colour is a strong indication of the presence of starch or starch derivatives.

19.2.6.5 Instant discolouration indicates the presence of a reducing agent, in this case, continue the dropwise addition of the iodine/iodide solution. Compare the colour obtained with those of 19.2.6.1 to 19.2.6.4.

19.2.6.6 If any colour reaction is detected other than that mentioned under 19.2.5, the testing is abandoned.

19.3 Qualitative guar determination in xanthan gum

19.3.1 Principle

19.3.1.1 The presence of guar additives to xanthan gum can be detected if guar is present in the minimum range of 7,5 % to 10,0 % by mass.

19.3.1.2 Sodium borate (borax) is used to crosslink the guar fraction, which causes an increase in the viscosity of the xanthan gum solution.

19.3.2 Reagents and materials

19.3.2.1 **Water** (CAS 7732-18-5), tap.

19.3.2.2 **Sodium borate decahydrate** (CAS 1303-06-4), e.g. borax.

19.3.2.3 **Water**, warm, tap, 32 °C ± 2 °C (90 °F ± 3°F).

19.3.3 Apparatus

19.3.3.1 **Balance**, accurate to 0,01 g.

19.3.3.2 **Mixer** (e.g. Multimixer® model 9B with 9B29X impellers, or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

19.3.3.3 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton® Beach mixer cup No. M110-D, or equivalent). An alternative container option is a 600 ml glass jar.

19.3.3.4 **Spatula**.

19.3.3.5 **Thermometer**, accurate to ± 0,5 °C (± 1,0 °F) over the range specified in this procedure.

19.3.3.6 **Viscometer, motor-driven, direct-indicating**, in accordance with ISO 10414-1, for example, Fann model 35 or equivalent, with an R1B1 configuration and equipped with a f0.2 torsion spring. See 19.8 for calibration.

19.3.3.7 **Beaker**, one 400 ml, borosilicate glass, e.g. Pyrex, tall form (model 1060).

19.3.3.8 **Graduated cylinder**, one 50 ml ± 1,0 ml.

19.3.3.9 **Weighing paper**.

19.3.3.10 **Water bath**, maintained below 25 °C ± 1 °C (77 °F ± 2 °F).

19.3.3.11 **Timer**, mechanical or electric, accurate to 0,1 min.

19.3.4 Procedure — Preparation of the fresh water xanthan gum solution

19.3.4.1 Weigh 350 g ± 1,0 g of water into the container.

19.3.4.2 Weigh 1,5 g ± 0,01 g of xanthan gum onto weighing paper.

19.3.4.3 Place the container onto the mixer and switch on the mixer.

19.3.4.4 Add the xanthan gum at a uniform rate over a time interval of 30 s. The xanthan gum should be added into the vortex away from the impeller shaft to minimize dusting.

19.3.4.5 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape the sides of the mixing container with the spatula to remove or dislodge any xanthan gum adhering to the container wall. Be sure that all xanthan gum clinging to the spatula is incorporated into the solution.

19.3.4.6 Replace the container on the mixer and continue to stir. Total mixing time shall equal 15 min ± 1 min.

19.3.4.7 Place the mixing cup in chilled water for not longer than 30 min to lower the fluid temperature to 25 °C ± 1 °C (77 °F ± 2 °F).

19.3.5 Procedure — Preparation of the sodium borate solution

19.3.5.1 Weigh 10 g ± 0,1 g of sodium borate onto weighing paper.

19.3.5.2 Weigh 90 g ± 1,0 g of warm water into the beaker.

19.3.5.3 Add the sodium borate to water and stir with a spatula for approximately 1 min to dissolve the sodium borate.

19.3.6 Procedure — Viscosity measurement of the xanthan gum solution

19.3.6.1 Pour the fresh water xanthan gum solution (19.3.4) into the beaker to the 250 ml mark.

19.3.6.2 Place the beaker on the viscometer stage and raise the stage so that the fluid level is equal to the inscribed line on the rotor sleeve. The reading shall be taken at a solution test temperature of 25 °C ± 1 °C (77 °F ± 2 °F).

19.3.6.3 With the viscometer set at 300 r/min, switch on the viscometer and wait 15 s to 20 s for reading to stabilize. Read to the nearest 0,5 dial reading after stabilization. Record the dial reading as $R_{300\text{-P}}$.

19.3.6.4 Change the viscometer setting to 6 r/min, wait 20 s to 30 s for the dial to stabilize, and read to the nearest 0,5 dial reading after stabilization. Record the dial reading as $R_{6\text{-P}}$.

19.3.6.5 Change the viscometer setting to 3 r/min, wait 20 s to 30 s for the dial to stabilize, and read to the nearest 0,5 dial reading after stabilization. Record the dial reading as $R_{3\text{-P}}$.

19.3.7 Procedure — Viscosity measurement of the xanthan gum solution treated with the sodium borate solution

19.3.7.1 Following the completion of 19.3.6, pour the xanthan gum solution into the original container.

19.3.7.2 Measure 30 ml ± 2,0 ml of sodium borate solution (19.3.5) using the graduated cylinder and add to the xanthan gum solution.

19.3.7.3 Hand stir with the spatula for 20 s to 30 s. Allow mixture to stand for 5 min.

19.3.7.4 Pour the mixture into the beaker to the 250 ml mark.

19.3.7.5 Place the beaker on the viscometer stage and raise the stage so that the fluid level is equal to the inscribed line on the rotor sleeve. The reading shall be taken at a solution test temperature of 25 °C ± 1 °C (77 °F ± 2 °F).

19.3.7.6 With the viscometer set at 300 r/min, switch on the viscometer and wait 20 s to 30 s for the reading to stabilize. Read to the nearest 0,5 dial reading after stabilization. Record the dial reading as $R_{300\text{-M}}$.

19.3.7.7 Change the viscometer setting to 6 r/min, wait 20 s to 30 s for the dial to stabilize and read to the nearest 0,5 dial reading after stabilization. Record the dial reading as $R_{6\text{-M}}$.

19.3.7.8 Change the viscometer setting to 3 r/min, wait 20 s to 30 s for the dial to stabilize and read to the nearest 0,5 dial reading after stabilization. Record the dial reading as $R_{3\text{-M}}$.

19.3.8 Results

19.3.8.1 Compare the readings for the polymer solution ($R_{300\text{-P}}$, $R_{6\text{-P}}$ and $R_{3\text{-P}}$) and the mixture of polymer and sodium borate ($R_{300\text{-M}}$, $R_{6\text{-M}}$ and $R_{3\text{-M}}$).

19.3.8.2 Any increase between mixture readings and polymer solution readings indicates the presence of guar in the xanthan gum.

19.4 Moisture content

19.4.1 Apparatus

19.4.1.1 **Oven**, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

19.4.1.2 **Balance**, accuracy of ± 0,01 g.

19.4.1.3 **Metal weighing pan**.

19.4.1.4 **Spatula**.

19.4.1.5 **Desiccator**, with calcium sulfate (CAS number 7778-18-9) desiccant, or equivalent.

19.4.2 Procedure

19.4.2.1 Weigh 4 g ± 0,01 g xanthan gum in a tared metal weighing pan and record the mass, m.

19.4.2.2 Dry sample in the oven for 2 h.

19.4.2.3 Cool to room temperature in desiccator.

19.4.2.4 Reweigh the metal weighing pan containing the dry xanthan gum. Record the residue mass as m_2.

19.4.3 Calculation

Calculate w_6, the mass fraction moisture, in percent, as given in Equation (48):

$$w_6 = 100 \frac{(m - m_2)}{m} \tag{48}$$

where

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record the calculated value.

19.5 Particle size

19.5.1 Apparatus

19.5.1.1 **Cenco-Meinzer® sieve shaker**, or equivalent 14).

19.5.1.2 **U.S. Standard sieves**, one 425 µm (35 mesh, U.S. Standard No. 40) and one 75 µm (200 mesh, U.S. Standard No. 200).

19.5.1.3 **Top lid and bottom receiver**, to fit standard sieves.

19.5.1.4 **Balance**, accurate to ± 0,01 g.

19.5.1.5 **Weighing pan** or **weighing paper**.

19.5.1.6 **Timer**, mechanical or electrical, with an accuracy of 0,1 min over the test interval.

19.5.2 Procedure

19.5.2.1 Visually inspect each sieve making certain that each is free of damage or blinding.

19.5.2.2 Weigh the 425 µm sieve and the bottom receiver. Record the masses as m_3 for the 425 µm sieve and m_6 for the bottom receiver.

19.5.2.3 Thoroughly mix dry sample and weigh 50,0 g ± 0,1 g of the sample into a weighing pan or onto weighing paper.

19.5.2.4 Assemble the sieves with bottom receiver, then the 75 µm sieve followed by the 425 µm sieve.

19.5.2.5 Pour the sample onto the 425 µm sieve, cover with the top lid and place entire assembly on the sieve shaker.

19.5.2.6 Secure the assembled sieves on the sieve shaker with the tie-down apparatus.

19.5.2.7 Set the timer for 5 min, activate and switch on the shaker.

19.5.2.8 After 5 min, stop the shaker, remove the tie-downs and remove the top lid.

19.5.2.9 Place the top lid on the balance and “tare” the lid. This lid supports each sieve when weighed.

19.5.2.10 Carefully separate the 425 µm sieve from the assembled sieves and place on the tared lid on the balance. Record the mass of the sieve and sample retained, as m_4. Remove the 425 µm sieve.

19.5.2.11 Carefully separate the 75 µm sieve from the bottom receiver and set aside.

19.5.2.12 Remove the lid from the balance and “zero” the balance. Weigh the bottom receiver and its sample contents. Record the mass as m_7.

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14) Cenco-Meinzer is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

19.5.3 Calculation

19.5.3.1 The mass fraction passing through a 425 µm sieve, w_{425}, expressed in percent, can be calculated by subtracting the percent residue from 100 % or by using Equation (49):

$$w_{425} = 100 \left[1 - \frac{(m_4 - m_3)}{50} \right] \tag{49}$$

where

m_4 is the mass of 425 µm sieve and sample retained, expressed in grams;

m_3 is the mass of the 425 µm sieve, expressed in grams.

19.5.3.2 The mass fraction of sample passing through a 75 µm sieve, w_{75}, expressed in percent, can be calculated from the percentage residue remaining in the bottom receiver, as given in Equation (50):

$$w_{75} = 100 \left[\frac{m_7 - m_6}{50} \right] \tag{50}$$

where

m_7 is the mass of the bottom receiver and sample retained, expressed in grams;

m_6 is the mass of the bottom receiver, expressed in grams.

19.6 Fluid viscosity

19.6.1 Reagents

19.6.1.1 **Sea salt**, ASTM D1141-98 (2003), or equivalent.

19.6.1.2 **Deionized** (or **distilled**) **water**.

19.6.1.3 **Sodium hydroxide** (NaOH) (CAS 1310-73-2) solution, 0,1 mol/l (0,1 N).

19.6.1.4 **Hydrochloric acid** (HCl) (CAS 7646-01-0) solution, 0,1 mol/l (0,1 N).

19.6.1.5 **Defoamer**, octanol (CAS 111-87-5), or equivalent.

CAUTION — Octanol has a hazard rating of 2, is mildly toxic when ingested and is a skin irritant.

19.6.2 Apparatus

19.6.2.1 **Thermometer**, accurate to ± 0,5 °C (± 1,0 °F) over the temperature range specified in the procedure.

19.6.2.2 **Balance**, accuracy of ± 0,001 g.

19.6.2.3 **Mixer** (e.g. Multimixer Model 9B with 9B29X impellers); each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter, mounted flash side up.

19.6.2.4 **Container**, approximate dimensions: depth, 180 mm (7,1 in); d top, 97 mm (3-5/6 in); d bottom, 70 mm (2,75 in) (e.g. Hamilton Beach mixer cup No. M110-D).

19.6.2.5 **Spatula**.

19.6.2.6 **Viscometer, motor-driven, direct-indicating**, in accordance with ISO 10414-1, for example, Fann model 35 or equivalent, with an R1B1 configuration and equipped with a f0.2 torsion spring. See 19.8 for calibration.

19.6.2.7 **Beaker**, one, 400 ml, borosilicate glass, tall form; one, 2 000 ml.

19.6.2.8 **Overhead stirrer**, capable of rotating at less than 1 000 r/min.

19.6.2.9 **Water bath**, maintained below 25 °C ± 1 °C (77 °F ± 2 °F).

19.6.2.10 **Timers**, mechanical or electrical, accurate to ± 0,1 min over the test interval.

19.6.2.11 **pH-meter with pH-electrode** (e.g. Thermo Russell type KDCW11).

19.6.3 Procedure — Preparation of the synthetic seawater

19.6.3.1 For a total volume of 1 000 ml, weigh out 41,95 g of sea salt into the 2 000 ml beaker.

19.6.3.2 Add 983 g of deionized or distilled water.

19.6.3.3 Mix 15 min with the overhead stirrer set at a low speed.

19.6.3.4 Measure the pH and adjust to 8,2 to 8,5 with 0,1 M (0,1 N) NaOH or HCl.

19.6.4 Procedure — Preparation of the xanthan gum polymer solution

19.6.4.1 Prepare a solution of xanthan gum polymer by adding 1,0 g ± 0,001 g of polymer to 358 g of synthetic seawater at a uniform rate over a time interval of about 30 s while stirring on the mixer. The xanthan gum polymer shall be added away from the impeller shaft to minimize dusting.

NOTE This is equivalent to 2,85 g/l ± 0,01 g/l.

19.6.4.2 After stirring 5 min ± 0,1 min, remove the container from the mixer and scrape its sides with the spatula to dislodge any particles adhering to the container walls. Be sure all particles clinging to the spatula are incorporated into the suspension.

19.6.4.3 Replace the container on the mixer and continue to stir. If necessary, the container may be removed from the mixer and the sides scraped to dislodge any particles clinging to the container walls at 10 min intervals. Total stirring time after adding the xanthan gum polymer shall equal 30 min ± 1 min.

19.6.4.4 Remove the mixing cup and add 2 drops to 3 drops of defoamer to the fluid. Stir 10 s to 15 s with the spatula to assist in the removal of entrapped air or foam.

19.6.4.5 Place the mixing cup in chilled water for not longer than 30 min to lower the fluid temperature to 25 °C ± 1 °C (77 °F ± 2 °F).

NOTE To achieve a more uniform temperature adjustment, immerse a thermometer in the fluid while stirring with the spatula to assure that the thermometer is not in contact with the container.

19.6.5 Procedure — Measurement of the viscosity

19.6.5.1 Pour the solution into the viscometer cup provided with the direct-indicating viscometer or the 400 ml tall-form beaker to about the 250 ml level. The solution level shall be exactly equal to the inscribed line on the direct-indicating viscometer rotor sleeve.

19.6.5.2 The dial reading at the 600 r/min rotor speed setting of the viscometer shall be recorded when a constant value at 600 r/min is reached. The 600 r/min dial reading shall be taken at a solution test temperature of 25 °C ± 1 °C (77 °F ± 2 °F). Record the 600 r/min dial reading.

19.6.5.3 Continue to test the solution at 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min, waiting for the dial to reach a constant value at each rotational velocity. These subsequent readings shall be taken at a solution test temperature of 25 °C ± 1 °C (77 °F ± 2 °F). Record the 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min dial readings.

19.7 Low-shear-rate viscosity

19.7.1 Principle

19.7.1.1 This procedure should follow within 5 min of the test viscosity on the direct-reading viscometer. See 19.6.5.

19.7.1.2 The suspending capabilities of xanthan gum are determined by measuring viscosity at low-shear rates at less than 1,0 s^{-1}, with a Brookfield® LV series viscometer, or equivalent.15)

19.7.2 Apparatus

19.7.2.1 **Digital viscometer**, Brookfield LV series or equivalent, equipped with a #1 cylindrical spindle and guard leg.

19.7.2.2 **Beaker**, one, 400 ml, borosilicate glass, e.g. Pyrex model 1060, tall form, or equivalent.

19.7.2.3 **Lab jack,** adjustable, with 15 cm × 15 cm (6 in × 6 in) stage.

19.7.2.4 **Thermometer**, accurate to ± 0,5 °C (± 1,0 °F) over the temperature range specified in the procedure.

19.7.3 Procedure

19.7.3.1 Pour the xanthan gum solution (19.6.5) into the beaker. Make certain that the temperature of the solution is 25 °C ± 1 °C (77 °F ± 2 °F).

19.7.3.2 Place the beaker on the lab jack and raise the stage so that the cylindrical spindle is immersed in the fluid to the point where the fluid level is equal to the inscribed line on the spindle shaft.

19.7.3.3 Set the viscometer speed to 1,5 r/min and wait 30 s. Record the reading as $R_{1,5}$.

19.8 Calibration of direct-indicating viscometer

19.8.1 Apparatus

19.8.1.1 **Viscometer, motor-driven, direct-indicating**, in accordance with ISO 10414-1, for example, Fann model 35 or equivalent, with an R1B1 configuration and equipped with f0.2 torsion spring.

19.8.1.2 **Calibration fluid**, 200 cP 6), with accompanying chart showing viscosity versus temperature data.

19.8.1.3 **Thermometer,** digital, precision grade, with accuracy of ± 0,1 °C (± 0,2 °F).

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15) Brookfield LV series is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

19.8.1.4 **Beaker**, one, 400 ml, borosilicate glass, e.g. Pyrex® model 1060, or equivalent.

19.8.2 Procedure

19.8.2.1 Clean and dry the viscometer bob and rotor.

19.8.2.2 Pour approximately 250 ml of the calibration fluid into the 400 ml beaker.

19.8.2.3 Place the beaker on the viscometer stage and raise the stage to the point where fluid level is equal to the inscribed line on the rotor sleeve.

19.8.2.4 Place the thermometer into the fluid.

19.8.2.5 Set the viscometer to 6 r/min and switch on power. Allow the viscometer to run for approximately 3 min to allow the fluid to stabilize to room temperature within a thermometer reading of ± 0,1 °C (± 0,2 °F). Record the dial reading with 0,5 units and temperature reading.

19.8.2.6 Set the viscometer to 3 r/min and wait 2 min while the dial stabilizes. Record the dial reading within 0,5 units.

NOTE Excessive fluctuations in dial readings (greater than 1,0) can indicate the need to replace the viscometer bearings.

19.8.2.7 Compare the 6 r/min and 3 r/min readings with the calibration oil viscosity at the recorded temperature using the supplied calibration fluid chart.

19.8.3 Calculation

The accuracy of the viscometer readings is calculated as given in Equations (51) and (52), and shall equal ± 15 cP $^{6)}$:

$$(R_{0,6} \times 10) - \eta_{\text{oil}} = \pm 15 \text{ cP} \tag{51}$$

$$(R_{0,3} \times 20) - \eta_{\text{oil}} = \pm 15 \text{ cP} \tag{52}$$

where η_{oil} is the viscosity of the calibration oil.

If the viscometer readings are beyond the limits, adjust torsion spring as per manufacturer's procedure or return the viscometer to the manufacturer for calibration.

20 Barite 4,1

20.1 Principle

20.1.1 Drilling grade barite is produced from commercial barium sulfate-containing ores. The manufacturer shall retain certificates of analysis or similar documentation on these commercial barium sulfate ores. It may be produced from a single ore or a blend of ores and may be a straight-mined product or processed by beneficiation methods, i.e. washing, tabling, jigging, or flotation. It may contain accessory minerals other than the barium sulfate ($BaSO_4$) mineral. Because of mineral impurities, commercial barite may vary in colour 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 Annex A for more details.)

20.1.2 Drilling grade barium sulfate meeting the Barite 4,1 physical and chemical specifications listed in Table 19 is not intended to meet the requirements of performance tests described in ISO/API Standard publications.

Additionally, it is not intended to be used as a reference or calibration material as specified in ISO/API Standard publications.

20.1.3 End users should be aware that the material described in this clause has a lower density than ISO 13500/API 13A, Clause 7 Barite. Drilling fluid formulations should be developed to accommodate this lower density material. Maximum practical drilling fluid density for specific formulations may be lower than attainable with Clause 7 Barite.

20.1.4 End users should review fluids engineering calculations to determine where density differences between Clause 20 Barite 4,1 and ISO/API Clause 7 Barite will require revisions to these calculations.

20.1.5 Barite 4,1 shall be deemed to meet the requirements of this International Standard if a composite sample representing no more than one day's production conforms to the chemical and physical specifications of Table 19, represents the product produced, and is controlled by the manufacturer.

Table 19 – Barite 4,10 physical and chemical requirements

Requirement	Standard
Density	4,10 g/ml, minimum
Water-soluble alkaline earth metals, as calcium	250 mg/kg, maximum
Residue greater than 75 µm	Maximum mass fraction, 3,0%
Particles less than 6 µm in equivalent spherical diameter	Maximum mass fraction, 30%

20.2 Reagents and apparatus — Density by Le Chatelier flask

20.2.1 Kerosene or mineral spirits.

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

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

20.2.4 Le Chatelier flask, with graduations of 0,1 ml, clamped or weighted to prevent flotation in water bath.

20.2.5 Constant-temperature bath, transparent at 32 °C ± 0,5 °C regulated to ± 0,1 °C (90 °F ± 1,0 °F regulated to ± 0,2 °F) [e.g. approximately 40-l aquarium (fish tank) with heater/circulator attachment, or functional equivalent].

20.2.6 Balance, with accuracy of 0,01 g.

20.2.7 Pipette, volumetric, of capacity 10 ml.

20.2.8 Magnifying glass.

20.2.9 Dowel, wooden, approximately 8 mm (1/3 in) in diameter and 30 cm (12 in) in length, or a functional equivalent.

20.2.10 Tissue paper, absorbent.

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

20.2.11 Weighing dish, low-form, with spout, of approximately 100-ml capacity, or a functional equivalent.

20.2.12 Brush, small, fine-bristle.

20.3 Procedure — Density by Le Chatelier flask

20.3.1 Take approximately 100 g of Barite 4,1 which has been oven dried for at least two hours and cooled to room temperature in a desiccator.

20.3.2 Fill a clean Le Chatelier flask to approximately 22 mm (0,8 in) below the zero mark with kerosene.

20.3.3 Place the flask upright in the constant-temperature bath. The level of water in the bath shall be higher than the 24-ml graduation of the flask, but below the stopper level. Assure flask is stabilized by use of clamps or weights.

20.3.4 Allow the flask and contents to equilibrate for a minimum of 1 h. Using the magnifying glass with care to keep eyes at meniscus level, read the volume at the lowest portion of the curved interface, and record the initial volume to the nearest 0,05 ml) without removing the flask from the constant-temperature bath. Record as V_1.

If the kerosene level is above or below the – 0,2 ml to + 1,2 ml volume range after equilibrating, use the 10-ml pipette to add or remove kerosene in order for it to come within this range. Allow the flask to equilibrate at least 1 h and record initial volume as in 20.3.4.

20.3.5 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.

20.3.6 Weigh 80 g ± 0,05 g dried Barite 4,1 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 4,1 at the bulb. This is a slow process, requiring repeated transfers of small amounts of Barite 4,1. Use a brush to transfer any residual Barite 4,1 into the flask, then replace the stopper. Record mass as m.

20.3.7 If necessary, carefully tap the neck of the flask with the wooden dowel, or agitate carefully side to side, to dislodge any Barite 4,1 clinging to the walls. Do not allow kerosene to come into contact with the ground glass stopper joint of the flask.

20.3.8 Gently roll the flask along a smooth surface at no more than 45° from vertical, or twirl the upright flask at the neck vigorously between the palms of both hands, to remove entrained air from the Barite 4,1 sample. Repeat this procedure until no more bubbles can be seen rising from the Barite 4,1.

20.3.9 Return the flask to the bath and let stand for at least 0,5 h.

20.3.10 Remove the flask from the bath and repeat 20.3.8 to remove any remaining air from the barite 4.1 sample.

20.3.11 Immerse the flask in the bath again for at least 1 h.

20.3.12 Record the final volume in the same manner as described in 20.3.4. Record volume as V_2.

20.4 Calculation — Density by Le Chatelier flask

Calculate the density, ρ, in grams per millilitre, according to Equation (53):

$$\rho = \frac{m}{(V_2 - V_1)} \tag{53}$$

where

ρ is the sample density, expressed in grams per millilitre;

m is the sample mass, expressed in grams;

V_1 is the initial volume, expressed in millilitres;

V_2 is the final volume, expressed in millilitres.

Record calculated density.

20.5 Reagents and apparatus — Water-soluble alkaline earths as calcium

20.5.1 Aqueous EDTA solution, composed of 3,72 g ± 0,01 g of the disodium salt of ethylenediaminetetraacetic acid dihydrate [disodium salt of (ethylenedinitrilo) tetraacetic acid dihydrate] (CAS No. 6381-92-6) diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

20.5.2 Buffer solution, comprising 67,5 g ± 0,01 g ammonium chloride (CAS No. 12125-02-9) and 570 ml ± 1 ml of 15 mol/l ammonium hydroxide (CAS No. 1336-21-6) solution diluted to a final volume of 1 000 ml with deionized water in a volumetric flask.

20.5.3 Hardness indicator solution, comprising 1,00 g ± 0,01 g Calmagite (CAS No. 3147-14-6), or equivalent [1-(1-hydroxy-4-methylphenylazo)-2-naphthol-4-sulfonic acid] diluted to a final volume of 1000 ml with deionized water in a volumetric flask.

20.5.4 Deionized (or distilled) water.

20.5.5 Balance, of capacity exceeding 100 g with an accuracy of 0,01 g.

20.5.6 Erlenmeyer flask, of 250 ml nominal capacity, equipped with a tight-fitting stopper.

20.5.7 Graduated cylinder, 100 ml to 150 ml capacity (TC) with 1 ml graduations.

20.5.8 Titration vessel, e.g. beaker, of 100 ml to 150 ml capacity.

20.5.9 Serological pipettes or burette with graduations of 0,1 ml·

20.5.10 Volumetric pipettes (TD), of capacity 10 ml or equivalent.

20.5.11 Filter press, low-pressure/low-temperature, in accordance with API 13B-1/ISO 10414-1, Clause 7, or filtration funnel.

20.5.12 Filter paper, Whatman 50, or equivalent**.**

20.5.13 Glass container, small.

20.5.14 Wrist-action shaker, optional.

20.5.15 Volumetric flask, of capacity 1 000 ml.

20.5.16 Stirring rod.

20.6 Procedure — Water-soluble alkaline earth metals as calcium

20.6.1 Weigh 100 g ± 0,05 g of Barite 4,1. Transfer to the Erlenmeyer flask and add 100 ml ± 1 ml of deionized water. Stopper the flask and shake for at least 5 min during an approximate 1-h interval or by an optional mechanical shaking apparatus for 20 min to 30 min.

20.6.2 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.

20.6.3 Add 50 ml ± 1 ml deionized water to the titration vessel. Add about 2 ml of hardness buffer and sufficient hardness indicator to achieve a distinct blue colour. Swirl to mix.

A solution with colour 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.

20.6.4 Using the volumetric pipette, measure 10 ml of the filtrate into the titrating vessel. Swirl to mix. A blue colour indicates no calcium hardness and the test is complete. A wine-red colour will develop if calcium and/or magnesium are present. Record as V_4.

20.6.5 If hardness is present, begin stirring and titrate with EDTA solution to the blue endpoint. The endpoint of the titration is best described as the point at which additional EDTA produces no further red to blue change. The EDTA volume used to produce the blue endpoint will be used in the calculation in 20.7. Record as V_3.

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

20.7 Calculation — Water-soluble alkaline earths as calcium

Calculate the soluble alkaline earth metals as calcium, m_1, in milligrams per kilogram, according to Equation (54):

$$m_1 = 400\left(\frac{V_3}{V_4}\right) \tag{54}$$

where

m_1 is the soluble alkaline earth metals as calcium, expressed in milligrams per kilogram;

V_3 is the volume of EDTA used, expressed in millilitres;

V_4 is the volume of filtrate used, expressed in millilitres.

Record calculated value.

20.8 Reagents and materials — Residue of diameter greater than 75 µm

20.8.1 Sodium hexametaphosphate (CAS No. 10124-56-8).

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

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

20.8.4 Balance, with an accuracy of 0,01 g.

20.8.5 Mixer (e.g. Multimixer® Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller, approximately 25 mm (1 in) in diameter, mounted flash side up.

20.8.6 Container, of approximate dimensions 180 mm (7,1 in) deep, 97 mm (3-5/6 in) d top, 70 mm (2,75 in) d bottom (e.g. Hamilton Beach® mixer cup No. M110-D, or equivalent).

20.8.7 Sieve, 75 µm, conforming to the requirements of ASTM E161, of approximate dimensions 76 mm (3,0 in) diameter and 69 mm (2,75 in) from top of frame to wire cloth.

NOTE Supplier's verification that sieve conforms to ASTM E161 is satisfactory evidence of compliance.

20.8.8 Spray nozzle with 1/4 TT body (Spraying Systems Co., No. TG 6.5 tip with 1/4 TT body, or equivalent), attached to water line with 90° elbow.

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

20.8.10 Evaporating dish or functional equivalent.

20.8.11 Wash bottle.

20.9 Procedure — Residue of diameter greater than 75 µm

20.9.1 If required, equilibrate approximately 60 g of dried Barite 4,1 in a desiccator.

20.9.2 Weigh 50 g ± 0,01 g of dried barite 4.1. Record weight as m. Add the weighed sample to approximately 350 ml of water containing about 0,2 g of sodium hexametaphosphate. Stir on the mixer for 5 min ± 1 min.

20.9.3 Transfer the sample to the 75 µm sieve. Use wash bottle to remove all material from the container to the sieve. Wash the material on the sieve with water controlled to 690 kPa ± 35 kPa (100 psi ± 7 psi) from a spray nozzle for 2 min ± 15 s. While washing, hold the tip of the spray nozzle approximately in the plane of the top of sieve and move the spray of water repeatedly over the sample.

20.9.4 Wash the residue from the sieve into a tared evaporating dish and decant excess clear water.

20.9.5 Dry the residue in the oven to a constant mass. Record residue mass as m_2 and total drying time.

20.10 Calculation — Residue of diameter greater than 75 µm

Calculate the mass fraction residue of particles greater than 75 µm, w_1, in percent, according to Equation (55):

$$w_1 = 100\left(\frac{m_2}{m}\right) \tag{55}$$

where

w_1 is the mass fraction residue of particles greater than 75 µm, expressed in percent;

m is the sample mass, expressed in grams;

m_2 is the residue mass, expressed in grams.

Record calculated value.

20.11 Reagents and apparatus — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

20.11.1 Dispersant solution, comprising 40 g ± 0,1 g sodium hexametaphosphate and 3,60 g ± 0,1 g anhydrous sodium carbonate (CAS No. 497-19-8) per 1 000 ml of solution. The sodium carbonate is used to adjust the pH of the solution to approximately 9,0.

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

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

20.11.4 Balance, with an accuracy of 0,01 g.

20.11.5 Mixer (e.g. Multimixer Model 9B with 9B29X impellers or equivalent), having each spindle fitted with a single sine-wave impeller approximately 25 mm (1 in) in diameter mounted flash side up.

20.11.6 Container, of approximate dimensions 180 mm (7,1 in) deep, 97 mm (3-5/6 in) d top, 70 mm (2,75 in) d bottom (e.g. Hamilton Beach mixer cup No. M110-D, or equivalent).

20.11.7Sedimentation cylinder, glass, approximately 457 mm (18 in) in height and 63 mm (2,5 in) in diameter, marked for a volume of 1 000 ml (ASTM D422).

20.11.8 Rubber stopper, Number 13.

20.11.9 Water bath or **constant-temperature room**, capable of maintaining a convenient constant temperature at 24 °C ± 5 °C (75 °F ± 7°F).

20.11.10Thermometer, including the range 16 °C ± 0,5 °C to 32 °C ± 0,5 °C (60 °F ± 1,0 °F to 90 °F ± 1,0 °F).

20.11.11Hydrometer, ASTM 151H, graduated to read specific gravity of suspension.

20.11.12Timer, mechanical or electrical, with an accuracy of 0,1 min over the test period.

20.12 Procedure — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

20.12.1 Weigh 80 g ± 0,1 g of dry Barite 4,1 and place in container. Record mass as m.

20.12.2 Add 125 ml ± 2 ml (127 g ± 2 g) of dispersant solution [from 20.11.1]. Dilute to approximately 400 ml with deionized water. Rinse all adhering particles from spatula into suspension.

20.12.3 Stir 5 min ± 0,5 min on mixer.

20.12.4 Transfer the suspension to the sedimentation cylinder. Rinse container with deionized water to assure that all sample particles are transferred to the sedimentation cylinder.

20.12.5 Add deionized water to the 1 000-ml mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s ± 5 s while holding a No. 13 rubber stopper in the top of the cylinder.

This is a critical step. Suspension shall be homogeneous at start of sedimentation. This is difficult to obtain because of the high density of Barite 4,1.

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

20.12.7 Take hydrometer readings at intervals of 10 min ± 0,1 min, 20 min ± 0,1 min, 30 min ± 0,1 min, and 40 min ± 0,1 min (or until the first point below the 6 µm value is reached). To take a hydrometer reading, *carefully and slowly* lower the hydrometer to approximately the 1,020 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 shall be removed immediately after each reading to eliminate particle build-up on the shoulders which causes erroneous results. All hydrometer readings shall be done with a minimum of fluid disturbance to preserve suspension-settling equilibrium.

20.12.8 Record time, t, in minutes, the temperature, θ, in degrees Celsius or degrees Fahrenheit, and the hydrometer reading, R, on data sheet.

Temperature may be measured in either degrees Celsius or degrees Fahrenheit, as long as all measurements and calculations are consistent in units, including hydrometer calibration.

20.12.9 For each time interval, determine water viscosity, η, and effective hydrometer depth, L, from Tables 3 and 4. Record on data sheet.

20.13 Calculation — Particles less than 6 µm in equivalent spherical diameter by sedimentation method

20.13.1 From hydrometer calibration (5.2.5.2) enter hydrometer correction slope, M_c, and hydrometer correction intercept, B_c, into data sheet. Clause 5 is the Calibration specifications from ISO 13500:2008.

20.13.2 Calculate sample constant, K_s, as given in Equation (56) (or determine from Table 22) and enter into data sheet:

$$K_\text{s} = 100\frac{\rho}{m(\rho - 1)} \tag{56}$$

where

K_S is the sample constant;

ρ is the sample density, in grams per millilitre;

m is the sample mass, expressed in grams.

20.13.3 Calculate and enter onto data sheet the equivalent spherical diameter, D_e, in micrometres, for each time interval as given in Equation (57):

$$D_\text{e} = 17{,}5\sqrt{\frac{\eta L}{(\rho - 1)t}} \tag{57}$$

where

D_e is the equivalent spherical diameter, expressed in micrometres;

η is the viscosity of water, expressed in millipascal seconds;

ρ is the sample density, expressed in grams per millilitre;

t is the time, expressed in minutes;

L is the effective depth, expressed in centimetres. See Table 21.

20.13.4 Calculate and enter onto data sheet the cumulative percent finer value, w_a, for the equivalent particle diameter, D_e, immediately greater than 6 µm, w_2, using D_1, and for the equivalent particle diameter, D_e, immediately less than 6 µm, w_3 , using D_2, as given in Equation (58):

$$w_a = K_s\left[\left(M_c \cdot \theta\right) - B_c + \left(R - 1\right)1\,000\right] \tag{58}$$

where

w_a is the cumulative percent finer;

K_s is the sample constant;

M_c is the hydrometer correction slope, determined in Equation (1), as given in subclause 5.2.5.7;

θ is the suspension temperature, expressed in degrees Celsius or degrees Fahrenheit (see 20.12.8);

B_c is the hydrometer correction intercept, as determined in subclause 5.2.5.2.8, Equation (2);

R is the hydrometer reading.

20.13.5 Calculate and enter onto data sheet cumulative percent less than 6 µm (w_4) as given in Equation (59):

$$w_4 = \left[\left(\frac{w_2 - w_3}{D_1 - D_2}\right)\left(6 - D_2\right)\right] + w_3 \tag{59}$$

where

w_4 is the cumulative percent less than 6 µm;

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

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

D_1 is the equivalent particle diameter immediately greater than 6 µm, determined in Equation (57);

D_2 is the equivalent particle diameter immediately less than 6 µm, determined in Equation (57).

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

20.13.6 A correction for water not at the reference temperature of 20 °C (68 °F), can be calculated according to an established equation, see Reference [10].

$$\log(\eta_{20}/\eta_{\theta}) = [1{,}370\,23\ (\theta - 20) + 0{,}000\,836\ (\theta - 20)^2]/(109 + \theta) \tag{60}$$

where

$log\ (\eta_{20}/\eta_{\theta})$ is the correction for temperature variance;

θ is the temperature, in degrees Celsius;

η_{20} 1,002, is the viscosity of water at 20 °C (68 °F);

η_{θ} viscosity at desired temperature (see Table 20).

Table 20 — Viscosity of water at various temperatures

Temperature θ °C (°F)	Viscosity η mPa·s	Temperature θ °C (°F)	Viscosity η mPa·s
15,6 (60)	1,121 1	22,2 (72)	0,949 8
16,1 (61)	1,105 0	22,8 (73)	0,937 4
16,7 (62)	1,089 3	23,3 (74)	0,925 3
17,2 (63)	1,073 9	23,9 (75)	0,913 4
17,8 (64)	1,058 9	24,4 (76)	0,901 8
18,3 (65)	1,044 2	25,0 (77)	0,890 4
18,9 (66)	1,029 8	25,6 (78)	0,879 2
19,4 (67)	1,015 8	26,1 (79)	0,868 3
20,0 (68)	1,002 0	26,7 (80)	0,857 6
20,6 (69)	0,988 5	27,2 (81)	0,847 0
21,1 (70)	0,975 3	27,8 (82)	0,836 7
21,7 (71)	0,962 4	28,3 (83)	0,826 6

Table 21— Values of effective depth based on readings on hydrometer ASTM 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 22 — Sample constant, K_s, for Barite 4,1 (80,0 g sample)

Sample density g/ml	**Sample constant, K_s**
4,10	1,653
4,11	1,652
4,12	1,651
4,13	1,649
4,14	1,648
4,15	1,647
4,16	1,646
4,17	1,644
4,18	1,643
4,19	1,642
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

Annex A
(informative)

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 ions of calcium, one of the alkaline earth metals that are covered in this International Standard. Other minerals such as siderite, dolomite and pyrrhotite are not covered in this International Standard, 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 are detrimentally affected by their presence. Consequently, International Standards limiting their concentrations in barite have not been developed. When it is suspected that one or more is/are affecting mud performance, procedures for determining their concentration in the barite can be found in API RP 13K.

Annex B
(informative)

Test precision

B.1 Description

B.1.1 General

Precision statements for this International Standard's tests are presented in this annex for the following materials:

g) barite;

h) haematite;

i) bentonite;

j) non-treated bentonite;

k) OCMA-grade Bentonite;

l) attapulgite;

m) sepiolite;

n) technical-grade low-viscosity CMC;

o) technical-grade high-viscosity CMC;

p) starch.

The precision values in this annex do not represent what can be expected from any random laboratory. Rather, they reflect the precision obtained by personnel who are trained and experienced in running the tests using equipment that is in good working order and in calibration.

Any laboratory that wishes to test materials for conformance to this International Standard shall demonstrate at least the degree of test precision given in this annex. Calibration barite and test calibration bentonite are available through the API offices (see 4.2.1) for use by laboratories to determine their test precision.

B.1.2 Considerations for manufacturers and users

The precision data in Tables B.1 through B.10 reflect the fact that the standard test methods are subject to normal test variability. This variability can lead to conflicts between a manufacturer and a user based on their respective test results on the same sample of material.

The precision data indicate the maximum expected difference between two test results on the same sample, both within and between laboratories, at the 95 % confidence level.

B.2 Basis

The limits in Tables B.1, B.3, B.4, B.5, B.8, B.9 and B.10 were determined from interlaboratory studies conducted from 1988 to 1991. The data analysis is given in API Research Reports 88-30, 89-30 and 90-30. The studies were designed and conducted by an independent consulting laboratory under contract to the API. The project was overseen by a Technical Advisory Committee of API Committee C3/SC13. The limits in Tables B.2, B.6 and B.7 were determined from interlaboratory studies conducted in 1992.

The experimental design and data analysis is patterned after ASTM Practice E691. Extra effort was made to assure that each participating laboratory used trained and experienced personnel, maintained equipment in calibration and followed the procedures in every detail.

B.3 Definitions

For Clause B.4, the procedures used for the statistical evaluation of sample differences shall be in accordance with ASTM E177.

The term “repeatability limit” is used as described in ASTM Practice E177, i.e. the maximum expected difference between two test results on samples of the same material by the same laboratory at the 95 % confidence level.

The term “reproducibility limit” is used as described in ASTM Practice E177, i.e. the maximum expected difference between test results by two laboratories on samples of the same material at the 95 % confidence level.

The respective standard deviations at the 95 % confidence level can be obtained by dividing the limits by 2,8.

B.4 Test precision tables

Table B.1 — Test precision for barite

Test	**Repeatability limit** (intralab)	**Reproducibility limit** (interlab)
Density, grams per millilitre	0,022	0,030
Water soluble alkaline earth metals, as calcium, milligrams per kilogram	9,2	23,0
Particles of diameter less than 6 µm, %	1,7	2,5

The values in Table B.1 were determined from a 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 is the result of a single determination.

Table B.2 — Test precision for haematite

Test	**Repeatability limit** (intralab)	**Reproducibility limit** (interlab)
Density, grams per millilitres	0,026	0,050
Water soluble alkaline earth metals, as calcium, milligrams per kilogram	9,2	23,0
Particles of diameter less than 6 µm, %	0,9	3,4

The values in Table B.2 were determined from a study conducted in 1992. Each of seven laboratories tested each of two samples once each on each of three separate days. Each test result is the result of a single determination.

Table B.3 — Test precision for bentonite

Test	**Repeatability limit** (intralab)	**Reproducibility limit** (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	2,9	6,3
Yield point/plastic viscosity ratio	0,16	0,25
Filtrate volume, millilitres	0,8	1,3

The values in Table B.3 were determined from a study conducted in 1989. Each of eight laboratories tested each of two samples once each on each of three separate days. Each test result is the result of a single determination.

Table B.4 — Test precision for non-treated bentonite

Test	**Repeatability limit** (intralab)	**Reproducibility limit** (interlab)
Suspension properties		
Dispersed plastic viscosity, millipascal-seconds	2,1	3,1
Dispersed filtrate volume, millilitres	0,9	1,5

The values in Table B.4 were determined from a study conducted in 1989. Each of eight laboratories tested each of two samples once each on each of three separate days. Each test result is the result of a single determination.

Table B.5 — Test precision for OCMA bentonite

Test	**Repeatability limit** (intralab)	**Reproducibility limit** (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	4,9	9,3
Yield point/plastic viscosity ratio	1,15	1,16
Filtrate volume, millilitres	0,8	1,6

The values in Table B.5 were determined from a study conducted in 1991. Each of seven laboratories tested each of two samples once each on each of three separate days. Each test result is the result of one determination.

Table B.6 — Test precision for attapulgite

Test	Repeatability limit (intralab)	Reproducibility limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	3,2	6,8
Moisture, percent	1,5	2,1

The values in Table B.6 were determined from a 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 is the result of a single determination.

Table B.7 — Test precision for sepiolite

Test	Repeatability limit (intralab)	Reproducibility limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	4,5	5,6
Moisture, percent	1,6	1,6

The values in Table B.7 were determined from a study conducted in 1992. Each of four laboratories tested each of two samples once each on each of three separate days. Each test result is the result of single determination.

Table B.8 — Test precision for technical-grade low-viscosity CMC

Test	Repeatability limit (intralab)	Reproducibility limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min	2,6	5,1
Filtrate volume, millilitres	0,9	1,6

The values in Table B.8 were determined from a study conducted in 1991. For the viscometer dial reading at 600 r/min test, each of eight laboratories tested each of two samples once each on each of three separate days. Each viscometer 600 r/min 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 is an average of two determinations.

Table B.9 — Test precision for technical-grade high-viscosity CMC

Test	Repeatability limit (intralab)	Reproducibility limit (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min		
— in distilled water	2,4	4,8
— in 40 g/l salt water	2,1	5,8
— in saturated salt water	3,0	5,6
Filtrate volume, millilitres	0,6	1,9

The values in Table B.9 were determined from a study conducted in 1991. For the viscosity reading at 600 r/min 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 is an average of two determinations.

Table B.10 — Test precision starch

Test	**Repeatability limit** (intralab)	**Reproducibility limit** (interlab)
Suspension properties		
Viscometer dial reading at 600 r/min		
— in 40 g/l salt water	2,0	7,1
— in saturated salt water	1,5	4,9
Filtrate volume, millilitres		
— in 40 g/l salt water	0,7	2,3
— in saturated salt water	0,6	1,5

The values in Table B.10 were determined from a study conducted in 1991. For the 40 g/l salt water viscometer dial reading at 600 r/min 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. For the saturated salt water tests, each test result is an average of two determinations.

Annex C
(informative)

Examples of calculations

C.1 Hydrometer calibration

Hydrometer serial number	**Temperature** θ °C	**Reading** R	**Corrected curve slope** M_c	**Corrected curve intercept** B_c
Xxxxx	$\theta_1 = 26{,}0$	$R_1 = 1{,}001\ 7$	0,260	8,46
	$\theta_2 = 31{,}0$	$R_2 = 1{,}000\ 4$		

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Date: ________________________________

Using Equation (1):

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

$$M_c = \frac{1{,}0017 - 1{,}000\ 4}{31{,}0 - 26{,}0} \times 1000 = 0{,}260$$

Using Equation (2):

$$B_c = (M_c \times \theta_1) + [(R_1 - 1) \times 1\ 000] = (0{,}260 \times 26{,}00) + [(1{,}001\ 7 - 1) \times 1\ 000] = 8{,}46$$

C.2 Barite — Particles less than 6 µm in equivalent spherical diameter

C.2.1 Example of data sheet

Hydrometer serial No. XXXXX:

— M_c 0,260;

— B_c 8,46;

— K_s 1,629;

— Sample density, ρ 4,30 g/ml;

— Sample mass, m 80 g.

Time t min	Temperature θ °C (°F)	Hydrometer reading R	Water viscosity η mPa·s	Effective depth L cm	Particle diameter D_e µm	% Finer W	% Finer w_a
10	26 (79)	1,028 0	0,879 2	8,9	8,5	42,6	
20	26 (79)	1,021 0	0,879 2	10,7	6,6	31,4	
30	26 (79)	1,017 0	0,879 2	11,8	5,7	24,7	
40	26 (79)	1,014 0	0,879 2	12,6	5,1	9,8	
							26,9

C.2.2 Sample constant, K_s, from Equation (7)

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

$$K_s = \frac{100 \times 4{,}30}{80 \times (4{,}30 - 1)} = 1{,}629$$

C.2.3 Calculation of D_e for 20 min reading

Equivalent diameter, D_e, from Equation (8):

$$D_e = 17{,}5 \sqrt{\frac{\eta L}{(\rho - 1)\, t}}$$

$$D_e = 17{,}5 \sqrt{\frac{0{,}879\,2 \text{ x } 10{,}7}{(4{,}30 - 1) \text{ x } 20}} = 6{,}6\ \mu\text{m}$$

Percent finer, w_a, from Equation (9):

$$w_a = K_s \left[(M_c \cdot \theta) - B_c + (R - 1) 1\,000 \right]$$

$$w_a = 1{,}629 \times [(0{,}260 \times 26) - 8{,}46 + (1{,}021 - 1) \times 1\,000]$$

$$w_a = 1{,}629 \times (6{,}76 - 8{,}46 + 21{,}0) = 31{,}4\ \%$$

C.2.4 Calculation for percent of diameter less than 6 µm

Percent less than 6 µm, w_4, from Equation (10):

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

For

$w_2 = 31,4$

$w_3 = 24,7$

$D_1 = 6,6$

$D_2 = 5,7$

$$w_4 = \left[\left(\frac{31,4 - 24,7}{6,6 - 5,7} \right) (6 - 5,7) \right] + 24,7 = 26,9\ \%$$

C.3 Haematite — Particles less than 6 µm in equivalent spherical diameter

C.3.1 Example data sheet

Hydrometer serial No. XXXXX

- M_c 0,260;
- B_c 8,460;
- K_s 1,559;
- Sample density, ρ 5,05 g/ml;
- Sample mass, m 80 g.

Time t min	**Temperature** θ °C (°F)	**Hydrometer reading** R	**Water viscosity** η mPa·s	**Effective depth** L Cm	**Particle diameter** D_e µm	**% Finer** W	**% Finer** w_a
10	25,6 (78)	1,013 0	0,879 2	12,9	9,3	17,4	
20	25,0 (77)	1,010 0	0,890 4	13,7	6,8	12,5	
30	25,0 (77)	1,008 0	0,890 4	14,2	5,6	9,4	
							10,4

C.3.2 Sample constant, K_s, from Equation (7)

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

$$K_s = \frac{(100 \times 5,05)}{80 \times (5,05 - 1)} = 1,559$$

C.3.3 Calculation of D_e for 20 min reading

Equivalent diameter, D_e, from Equation (8):

$$D_e = 17{,}5\sqrt{\frac{\eta L}{(\rho - 1)\,t}}$$

$$D_e = 17{,}5\sqrt{\frac{0{,}890\,4 \times 13{,}7}{(5{,}05 - 1) \times 20}} = 6{,}8\ \mu\text{m}$$

Percent finer, w_a, from Equation (9):

$$w_a = K_s\left[(M_c \cdot \theta) - B_c + (R - 1)1\,000\right]$$

$$w_a = 1{,}559 \times \left[(0{,}260 \times 25{,}0) - 8{,}46 + (1{,}010 - 1) \times 1000\right]$$

$$w_a = 1{,}559 \times (-1{,}96 + 10{,}0) = 12{,}5\ \%$$

C.3.4 Calculation for percent of diameter less than 6 µm

Percent less than 6 µm, w_4, from Equation (10):

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

For

$w_2 = 12{,}5$

$w_3 = 9{,}4$

$D_1 = 6{,}8$

$D_2 = 5{,}6$

$$w_4 = \left[\left[\frac{12{,}5 - 9{,}4}{6{,}8 - 5{,}6}\right] \times (6 - 5{,}6)\right] + 9{,}4 = 10{,}4\ \%$$

Annex D
(informative)

Use of the API Monogram by Licensees

D.1 Scope

The API Monogram Program allows an API Licensee to apply the API Monogram to products. The API Monogram Program delivers significant value to the international oil and gas industry by linking the verification of an organization's quality management system with the demonstrated ability to meet specific product specification requirements. The use of the Monogram on products constitutes a representation and warranty by the Licensee to purchasers of the products that, on the date indicated, the products were produced in accordance with a verified quality management system and in accordance with an API product specification.

When used in conjunction with the requirements of the API License Agreement, API Q1, in its entirety, defines the requirements for those organizations who wish to voluntarily obtain an API license to provide API monogrammed products in accordance with an API product specification.

API Monogram Program licenses are issued only after an on-site audit has verified that the Licensee conforms to the requirements described in API Q1 in total, and the requirements of an API product specification. Customers/users are requested to report to API all problems with API monogrammed products. The effectiveness of the API Monogram Program can be strengthened by customers/users reporting problems encountered with API monogrammed products. A nonconformance may be reported using the API Nonconformance Reporting System available at http://compositelist.api.org/ncr.asp. API solicits information on new product that is found to be nonconforming with API specified requirements, as well as field failures (or malfunctions), which are judged to be caused by either specification deficiencies or nonconformities with API specified requirements.

This annex sets forth the API Monogram Program requirements necessary for a supplier to consistently produce products in accordance with API specified requirements. For information on becoming an API Monogram Licensee, please contact API, Certification Programs, 1220 L Street, N.W., Washington, D.C. 20005 or call 202-962-4791 or by email at certification@api.org.

D.2 References

In addition to the referenced standards listed earlier in this document, this annex references the following standard:

API Specification Q1.

For Licensees under the Monogram Program, the latest version of this document shall be used. The requirements identified therein are mandatory.

D.3 API Monogram Program: Licensee Responsibilities

D.3.1 Maintaining a License to Use the API Monogram

For all organizations desiring to acquire and maintain a license to use the API Monogram, conformance with the following shall be required at all times:

a) the quality management system requirements of API Q1;

b) the API Monogram Program requirements of API Q1, Annex A;

c) the requirements contained in the API product specification(s) for which the organization desires to be licensed;

d) the requirements contained in the API Monogram Program License Agreement.

D.3.2 Monogrammed Product—Conformance with API Q1

When an API-licensed organization is providing an API monogrammed product, conformance with API specified requirements, described in API Q1, including Annex A, is required.

D.3.3 Application of the API Monogram

Each Licensee shall control the application of the API Monogram in accordance with the following.

a) Each Licensee shall develop and maintain an API Monogram marking procedure that documents the marking/monogramming requirements specified by the API product specification to be used for application of the API Monogram by the Licensee. The marking procedure shall define the location(s) where the Licensee shall apply the API Monogram and require that the Licensee's license number and date of manufacture be marked on monogrammed products in conjunction with the API Monogram. At a minimum, the date of manufacture shall be two digits representing the month and two digits representing the year (e.g. 05-07 for May 2007) unless otherwise stipulated in the applicable API product specification. Where there are no API product specification marking requirements, the Licensee shall define the location(s) where this information is applied.

b) The API Monogram may be applied at any time appropriate during the production process but shall be removed in accordance with the Licensee’s API Monogram marking procedure if the product is subsequently found to be nonconforming with API specified requirements. Products that do not conform to API specified requirements shall not bear the API Monogram.

c) Only an API Licensee may apply the API Monogram and its license number to API monogrammable products. For certain manufacturing processes or types of products, alternative API Monogram marking procedures may be acceptable. The current API requirements for Monogram marking are detailed in the API Policy Document, *Monogram Marking Requirements*, available on the API Monogram Program website at http://www.api.org/certifications/monogram/.

d) The API Monogram shall be applied at the licensed facility.

e) The authority responsible for applying and removing the API Monogram shall be defined in the Licensee’s API Monogram marking procedure.

D.3.4 Records

Records required by API product specifications shall be retained for a minimum of five years or for the period of time specified within the product specification if greater than five years. Records specified to demonstrate achievement of the effective operation of the quality system shall be maintained for a minimum of five years.

D.3.5 Quality Program Changes

Any proposed change to the Licensee’s quality program to a degree requiring changes to the quality manual shall be submitted to API for acceptance prior to incorporation into the Licensee's quality program.

D.3.6 Use of the API Monogram in Advertising

Licensee shall not use the API Monogram on letterheads or in any advertising (including company-sponsored web sites) without an express statement of fact describing the scope of Licensee’s authorization (license number). The Licensee should contact API for guidance on the use of the API Monogram other than on products.

D.4 Marking Requirements for Products

These marking requirements apply only to those API Licensees wishing to mark their products with the API Monogram.

D.4.1 Product Specification Identification

Manufacturers shall mark the product with the information identified in 6.4 and 6.5, as a minimum, including "API Spec 13A."

D.4.2 Bulk Material

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

The complete API monogram marking consists of the following:

- the letters "Spec 13A,"
- the manufacturer's API license number,
- the API monogram,
- the date of manufacture (defined as the month and year when the monogram is applied by the manufacturer).

The name of the product shall match its designation in the standard.

D.4.3 Units

As a minimum, equipment should be marked with U.S. customary (USC) units. Use of dual units [metric (SI) units and USC units] is acceptable.

D.4.4 License Number

The API Monogram license number shall not be used unless it is marked in conjunction with the API Monogram.

D.5 API Monogram Program: API Responsibilities

The API shall maintain records of reported problems encountered with API monogrammed products. Documented cases of nonconformity with API specified requirements may be reason for an audit of the Licensee involved, (also known as audit for "cause").

Documented cases of specification deficiencies shall be reported, without reference to Licensees, customers or users, to API Subcommittee 18 (Quality) and to the applicable API Standards Subcommittee for corrective actions.

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