

Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests

The European Standard EN ISO 13500:2006 has the status of a
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National foreword

This British Standard is the official English language version of EN ISO 13500:2006. It is identical with ISO 13500:2006. It supersedes BS EN ISO 13500:1999 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee PSE/17, Materials and equipment for petroleum, petrochemical and natural gas industries, to Subcommittee PSE/17/-/3, Drilling and completion fluids and well cements, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii to vii, a blank page, pages 1 to 74, an inside back cover and a back cover.

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Petroleum and natural gas industries - Drilling fluid materials -
Specifications and tests (ISO 13500:2006)

Industries du pétrole et du gaz naturel - Produits pour
fluides de forage - Spécifications et essais (ISO
13500:2006)

Erdöl- und Erdgasindustrie - Bohrspülungen -
Spezifikationen und Prüfungen (ISO 13500:2006)

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Foreword

This document (EN ISO 13500:2006) has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2006, and conflicting national standards shall be withdrawn at the latest by August 2006.

This document supersedes EN ISO 13500:1998.

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Endorsement notice

The text of ISO 13500:2006 has been approved by CEN as EN ISO 13500:2006 without any modifications.

INTERNATIONAL STANDARD

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13500

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Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests

*Industries du pétrole et du gaz naturel — Produits pour fluides de
forage — Spécifications et essais*



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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 second edition cancels and replaces the first edition (ISO 13500:1998), which has been technically revised.

Introduction

This International Standard covers materials which 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 utilised 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, December 1, 2003. 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), and starch.

The intent of the document was 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 fluid materials — Specifications and tests

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), and starch. 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, *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 which 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	point/plastic viscosity ratio;
B_c	hydrometer correction intercept;
C_c	calibration correction;
C_m	40 times the EDTA volume, expressed in centimetres;
d	inner diameter;
D_e	equivalent spherical diameter, expressed in micrometres;
D_2	equivalent particle diameter immediately greater than 6 μm ;
D_3	equivalent particle diameter immediately less than 6 μm ;
K_s	sample constant;
L	effective hydrometer depth, in centimetres (see Table 4);
m	sample mass, expressed in grams;
m_1	soluble alkaline earth metals as calcium, expressed in milligrams per kilogram;
m_2	residue mass, expressed in grams;
M_c	hydrometer correction curve slope;
R	average hydrometer reading;
R_1	average hydrometer reading at lower temperature;

R_2	average hydrometer reading at higher temperature;
R_3	hydrometer reading;
S_c	corrected test value;
S_s	sample test value;
t	time;
V_c	filtrate volume, in cubic centimetres, collected between 7,5 min and 30 min;
V_1	initial volume, expressed in cubic centimetres;
V_2	final volume, expressed in cubic centimetres;
V_3	volume EDTA used, expressed in cubic centimetres;
V_4	volume of filtrate used, expressed in cubic centimetres;
w_a	cumulative percent finer than size;
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 of particles less than 6 μm ;
w_5	mass fraction of residue of particles greater than 45 μm , expressed in percent;
w_6	cumulative percent less than 6 μm ;
w_7	mass fraction of moisture, expressed in percent;
η	water viscosity, expressed in millipascals-seconds;
η_P	plastic viscosity, in millipascal-seconds;
η_Y	yield point, Pa (lb/100 ft ²);
θ	temperature reading;
θ_1	average temperature reading at lower temperature;
θ_2	average temperature reading at higher temperature;
ρ	density, expressed in grams per cubic centimetre.

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 will forward the request to the designated custodian for further handling. The test calibration products is 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.

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 to 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.

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

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 outlined by ASTM E77 and NBS (NIST) Monograph 150.

5.2.2.2 Calibration — Thermometers

5.2.2.2.1 Place thermometer to be calibrated side by side with 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 readings for each thermometer shall not exceed 0,1 °C, or the smallest scale division on the thermometer being calibrated.

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

5.2.3 Laboratory balances

5.2.3.1 The manufacturer shall calibrate 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 conforming to ASTM E11 and ASTM E161

Approximate dimensions are 76 mm diameter and 69 mm 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 to be used using the same concentration dispersant solution as is used in the test, at temperatures spanning the anticipated test temperatures, and by reading the top rather than the bottom of the meniscus. Calibrate *each* hydrometer using the procedure below.

5.2.5.2.2 Prepare 1 l of dispersant solution, as follows.

- a) Place $125 \text{ cm}^3 \pm 2 \text{ cm}^3$ ($125 \text{ g} \pm 2 \text{ g}$) of dispersant solution from test procedure [7.11 a)] into a 1-l volumetric flask.
- b) Dilute to the 1 000-cm³ 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 $\pm 0,2 \text{ }^\circ\text{C}$. Insert the hydrometer to be 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 through 5.2.5.2.4 except set bath temperature to highest expected test temperature, calculate 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):

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

where

R_1 is the average hydrometer reading at lower temperature;

R_2 is the average hydrometer reading at higher temperature;

θ_1 is the average temperature reading at lower temperature;

θ_2 is the average temperature reading at higher temperature.

Temperature may be measured in either $^\circ\text{C}$ or $^\circ\text{F}$, so long as all measurements and calculations are consistent in units (including subsequent use of hydrometer in routine test situations).

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

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

where

M_c is the hydrometer correction curve slope;

θ_1 is the average thermometer reading at lower temperature;

R_1 is the average hydrometer reading at lower temperature.

5.2.5.2.9 Record M_c , B_c and hydrometer serial number in 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 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 given 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.

- a) **Standard thermometer**, with an accuracy of $\pm 0,1$ °C, e.g. ASTM 90c or 91c grade.
- b) **Certified calibration fluid**, of viscosity 20 mPa·s, with chart (viscosity vs. temperature).
- c) **Certified calibration fluid**, of viscosity 50 mPa·s, with chart (viscosity vs. temperature).
- d) **Magnifying glass**, approximately $\times 3$ 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 is to 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 $\pm 0,1$ °C. Record 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 certified fluid.

5.2.6.4.8 Repeat 5.2.6.4.1, 5.2.6.4.2 through 5.2.6.4.7 using 50 mPa·s fluid.

5.2.6.4.9 Compare deviations to 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	$\pm 1,5$	$\pm 1,5$
50 mPa·s	$\pm 1,5$	$\pm 1,5$

5.2.7 Laboratory pressure-measuring device

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

5.2.7.2 Calibration — Laboratory pressure-measuring device

5.2.7.2.1 Type and accuracy: Pressure-measuring devices shall be readable to at least 2,5 % of full-scale range.

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

5.2.7.2.3 Usable range: 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 annually calibrated with a master pressure-measuring device or a dead-weight tester to 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 $\pm$ 300 r/min under no load with one spindle operating. Each spindle will be fitted with a single sine-wave impeller approximately 25 mm in diameter mounted flash side up. New impellers shall be weighed prior to installation, with mass and date recorded.

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.

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

Standard calcium chloride solution, concentration $c(\text{CaCl}_2) = (0,010\ 0 \pm 0,000\ 1) \text{ mol/l}$.

5.2.9.2.2 Procedure

- To a suitable flask, add $50 \text{ cm}^3 \pm 0,05 \text{ cm}^3$ deionized water and $50 \text{ cm}^3 \pm 0,05 \text{ cm}^3$ of standard CaCl_2 solution.
- Proceed as in 7.6.1 through 7.6.5, but without adding barite or additional water. (Use the 100 cm^3 solution prepared above in place of the 100 cm^3 deionized water specified in 7.6.1.)
- Calculate calibration correction, C_c , as given in Equation (3):

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

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

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

EXAMPLE 1 Calibration correction determination:

EDTA volume for CaCl_2 solution equal to $4,8 \text{ cm}^3$:

$$C_m = 40 \times 4,8 = 192$$

$$C_c = 192 - 200$$

$$C_c = -8$$

EXAMPLE 2 Calibration correction:

EDTA for sample equal to $6,1 \text{ cm}^3$:

Test value for sample, $S_s = 244 \text{ mg/kg}$

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

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 which can affect its calibration shall be recalibrated prior to use.

5.3.2 Thermometers

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

5.3.3 Laboratory balances

Calibrate each balance prior to 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 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 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 mixer spindle speed at least once every 90 days to ensure operation 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 mass drops below 90 % of its original value.

5.3.8 Deionized (or distilled) water

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

5.3.9 Laboratory pressure-measuring devices

Manufacturer shall document evidence of laboratory pressure-measuring device calibration prior to placing into first use by the manufacturer and annually thereafter.

5.3.10 API Test calibration materials

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 possible recycling of all packaging materials, including dry powdered or granular materials, not covered under this International Standard, used in drilling fluids, completion fluids and oil well cements.

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:

- 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 in accordance with 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 bag construction capable of safe handling, transport and storage.

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

6.3.3 The manufacturer should consider humidity-barrier capabilities of the bags against 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 or 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 in height;
- b) mass of the material in letters, or numbers and letters, at least 6 mm in height; the mass shall be listed in kilograms;
- c) lot/batch number in print script and/or numbers at least 3 mm in height, 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 weight

Each sack shall contain a specified net mass $\pm 2\%$. 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 below. 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 or APME 1993.

6.9.3 Cover

Identify PE, PP or carton, and recycle accordingly.

6.9.4 Bags

Use of high-performance paper quality 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 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.)

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 specifications

Test parameter	Specification
Density	4,20 g/cm ³ , minimum
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

- Kerosene** or **mineral spirits**.
- Oven**, regulated to 105 °C ± 3 °C.
- Desiccator**, with calcium sulfate (CAS No. 7778-18-9) desiccant, or equivalent.
- Le Chatelier flask**, clamped or weighted to prevent flotation in water bath.
- Constant-temperature bath**, transparent at 32 °C ± 0,5 °C regulated to ± 0,1 °C [e.g. approximately 40 l aquarium (fish tank) with heater/circulator attachment, or functional equivalent].
- Balance**, with accuracy of 0,01 g.

- g) **Pipette**, volumetric, of capacity 10 cm³.
- h) **Magnifying glass**.
- i) **Dowel**, wooden, approximately 8 mm in diameter and 30 cm in length, or a functional equivalent.
- j) **Tissue paper**, absorbent.

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

- k) **Weighing dish**, low-form, with spout, of approximately 100 cm³ capacity, or a functional equivalent.
- l) **Brush**, small, fine-bristle.

7.3 Procedure — Density by Le Chatelier flask

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

7.3.2 Fill a clean Le Chatelier flask to approximately 22 mm 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 cm³ graduation of the flask, but below the stopper level. Assure flask is stabilized by 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 cm³ (doubtful digit) without removing the flask from the constant-temperature bath. Record as V_1 .

If the kerosene level is above or below the – 0,2 cm³ to + 1,2 cm³ volume range after equilibrating, use the 10 cm³ 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 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 dried barite into the weighing dish and carefully transfer to the Le Chatelier flask. Take care to avoid splashing of the kerosene or plugging of the flask with barite at the bulb. This is a slow process, requiring repeated transfers of small amounts of barite. Use a brush to transfer any residual barite into the flask, then replace the stopper. Record 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 volume as V_2 .

7.4 Calculation — Density by Le Chatelier flask

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

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

where

m is the sample mass, in grams;

V_1 is the initial volume, in cubic centimetres;

V_2 is the final volume, in cubic centimetres.

Record calculated density.

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

- a) **Aqueous EDTA solution**, composed of $3,72 \text{ g} \pm 0,01 \text{ g}$ 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 $1\,000 \text{ cm}^3$ with deionized water in a volumetric flask.
- b) **Buffer solution**, comprising $67,5 \text{ g} \pm 0,01 \text{ g}$ ammonium chloride (CAS No. 12125-02-9) and $570 \text{ cm}^3 \pm 1 \text{ cm}^3$ of 15 mol/l ammonium hydroxide (CAS No. 1336-21-6) solution diluted to a final volume of $1\,000 \text{ cm}^3$ with deionized water in a volumetric flask.
- c) **Hardness indicator solution**, comprising $1 \text{ g} \pm 0,01 \text{ g}$ 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 cm^3 with deionized water in a volumetric flask.
- d) **Deionized (or distilled) water**.
- e) **Balance**, of capacity exceeding 100 g with an accuracy of $0,01 \text{ g}$.
- f) **Erlenmeyer flask**, of 250 cm^3 nominal capacity, equipped with a tight-fitting stopper.
- g) **Graduated cylinder**, of 100 cm^3 capacity (TC) with 1 cm^3 graduations.
- h) **Titration vessel**, e.g. beaker, of 100 cm^3 to 150 cm^3 capacity.
- i) **Serological pipettes or burette** with graduations of $0,1 \text{ cm}^3$.
- j) **Volumetric pipettes (TD)**, of capacity 10 cm^3 or equivalent.
- k) **Filter press**, in accordance with ISO 10414-1, or **filtration funnel**.
- l) **Filter paper**, Whatman 50, or equivalent.
- m) **Glass container**, small.
- n) **Wrist-action shaker**, optional.
- o) **Volumetric flask**, of capacity $1\,000 \text{ cm}^3$.
- p) **Stirring rod**.