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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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.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 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 hygrometer 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 milllipascal·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 API[1]. 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.

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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 $cm^3 \pm 2$ cm^3 (125 g ± 2 g) of dispersant solution from test procedure [7.11 a)] into a 1-l volumetric flask.

b) Dilute to the 1 000-cm^3 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. 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 °C or °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 ± 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 ×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.