INTERNATIONAL STANDARD

ISO 13500

Third edition
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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 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.

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 and drilling-grade *Xanthomaonas campestris*.

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 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), starch, low-viscosity polyanionic cellulose (PAC-LV), high-viscosity polyanionic cellulose (PAC-HV) and drilling-grade *Xanthomonas campestris* (Xanthan gum). 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_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_{AEM}	soluble alkaline earth metals as calcium, expressed in milligrams per kilogram
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 API[1]. 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.

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.

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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 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):

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

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.

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):

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

where

M_c is the hydrometer correction curve slope;

θ_1 is the average thermometer reading at 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.

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

5.2.9.2.1.1 Standard calcium chloride solution, $c(CaCl_2) = (0{,}010\ 0 \pm 0{,}000\ 1)$ 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 $CaCl_2$ 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):

$$C_c = C_m - 200 \tag{3}$$

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 $CaCl_2$ solution is equal to 4,8 ml:

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

$C_c = 192 - 200$

$C_c = -8$

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.