INTERNATIONAL STANDARD

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Petroleum and natural gas industries — Drilling fluids — Laboratory testing

Industries du pétrole et du gaz naturel — Fluides de forage — Essais en laboratoire

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10416 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

Introduction

This International Standard, which establishes testing methodologies for drilling fluid materials, is based on API RP 13I, fifth edition, June 1, 1995 [1]. This International Standard was developed in response to demand for more exacting testing methodologies. The tests contained herein were developed over several years by a group of industry experts and were identified as being those which would yield reproducible and accurate results. The tests are anticipated to be performed in a laboratory setting, but could be applicable in a field situation with more rigorous apparatus and conditions than normally found in a drilling fluid field-test kit.

These tests are designed to assist in the evaluation of certain parameters for drilling fluids, with these properties not necessarily used for the maintenance of a drilling fluid in field use. The tests provide either more precision or different properties than those given in the field-testing standards ISO 10414-1 and ISO 10414-2.

Users of this International Standard should be aware that further or differing requirements may be needed for individual applications. This International Standard is not intended to inhibit a vendor from offering, or the purchaser from accepting alternative equipment or engineering solutions for the individual application. This may be particularly appropriate where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this International Standard and provide details.

As with any laboratory procedure requiring the use of potentially hazardous chemicals, the user is expected to have received proper knowledge and training in the use and disposal of these chemicals. The user is responsible for compliance with all applicable local, regional, and national regulations for worker and local health, safety and environmental liability.

This International Standard contains footnotes giving examples of apparatus and reagents, and sometimes the supplier(s) of those materials which are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the products named. Equivalent products may be used if they can be shown to lead to the same results.

Petroleum and natural gas industries — Drilling fluids — Laboratory testing

1 Scope

This International Standard provides procedures for the laboratory testing of both drilling fluid materials and drilling fluid physical, chemical and performance properties. It is applicable to both water-based and oil-based drilling fluids, as well as the base or “make-up” fluid.

It is not intended as a detailed manual on drilling fluid control procedures. Recommendations regarding agitation and testing temperature are presented because the agitation history and temperature have a profound effect on drilling fluid properties.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2719, *Determination of flash point — Pensky-Martens closed cup method*

ISO 2977, *Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point*

ISO 3007, *Petroleum products and crude petroleum — Determination of vapour pressure — Reid method*

ISO 3016, *Petroleum products — Determination of pour point*

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic visocisty*

ISO 3405:2000, *Petroleum products — Determination of distillation characteristics at atmospheric pressure*

ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method*

ISO 3839, *Petroleum products — Determination of bromine number of distillates and aliphatic olefins — Electrometric method*

ISO 10414-1:2001, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ISO 10414-2:2002, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 2: Oil-based fluids*

ISO 13500:1998, *Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests*

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

ASTM D1141, *Standard Practice for Substitute Ocean Water*

ASTM D4052, *Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter*

ASTM D5186, *Standard Test Method for Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography*

ASTM E100, *Standard Specification for ASTM Hydrometers*

IP 391, *Test Method for Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography*

3 Terms, definitions and abbreviated terms

For the purposes of this International Standard, the following terms, definitions and abbreviated terms apply.

3.1 Terms and definitions

3.1.1
ACS reagent grade
chemical which meets purity standards as specified by the American Chemical Society (ACS)

3.1.2
flash side
side containing residue ("flash") from stamping and with concave indentations

3.1.3
darcy
k
permeability of a porous medium, where one darcy is the flow of a single-phase fluid of 1 cP viscosity that completely fills the voids of the porous medium, flowing through the medium under conditions of viscous flow at a rate of 1 $cm^3 \cdot s^{-1} \cdot cm^{-2}$ cross-sectional area, and under a pressure or equivalent hydraulic gradient of 1 $atm \cdot cm^{-1}$

NOTE 1 cP = 1 mPa·s.

3.1.4
quarter, verb
mix and divide into four specimens to assure homogeneity of specimens

3.1.5
spurt loss
volume of fluid that passes through the filtration medium before a filter cake is formed

3.1.6
tube sampling
sampling method comprising withdrawal of powdered sample from bag or bulk via a cylindrical device pushed into the sample, locked shut and withdrawn

3.2 Symbols and abbreviated terms

d	inner diameter
D	outer diameter
AA	atomic absorption spectroscopy
ACS	American Chemical Society
API	American Petroleum Institute

ASTM	American Society for Testing and Materials
BB	ball bearings
CAS	Chemical Abstracts Service
DCP	direct current plasma
DS	drill solids
EDTA	ethylenediaminetetraacetic acid
HTHP	high temperature, high pressure
ICP	inductively coupled plasma
ISA	ionic strength adjuster
ISE	ion-selective electrode
LGS	low gravity solids
MBT	methylene blue test
PAC-HV	high viscosity polyanionic cellulose
PAC-LV	low viscosity polyanionic cellulose
PPA	permeability plugging apparatus
PPT	permeability plugging test
PTFE	polytetrafluoroethylene

4 Particle size analysis for fines in barite

4.1 Principle

Fines are the particles in the range of 2 µm to 10 µm equivalent spherical diameter and are considered to be detrimental to drilling fluids at high concentrations. Both sieve analysis and sedimentation methods for determining fines concentration are described below.

4.2 Reagents and apparatus

4.2.1 Dispersant solution.

Prepare a solution of 40 g sodium hexametaphosphate and approximately 3,6 g sodium carbonate diluted to 1 l with deionized or distilled water. The sodium carbonate is used to adjust the pH of the solution to 9,0 or slightly less. After the initial pH adjustment, check the pH each day the solution is used. When the pH falls below 8,0, discard the solution.

4.2.2 Oven, capable of maintaining a temperature of 105 °C ± 3 °C.

4.2.3 Mixer, capable of operation at 11 500 r/min ± 300 r/min under load, with single corrugated impeller approximately 25,4 mm in diameter[1)].

4.2.4 Container for mixing, 180 mm deep, d = 97 mm at top and 70 mm at bottom[2)].

1) Multimixer Model 9B with B29 impeller 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.

2) 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.

4.2.5 Sieves, of mesh sizes 75 µm, 45 µm and 30 µm, having a diameter of 76 mm and a depth of 64 mm from top of the frame to the wire cloth.

4.2.6 Stopwatch, with direct-reading counter and an accuracy of 0 min to 25 min over the test interval.

4.2.7 Rubber stopper, size 13 (diameters 68 mm top and 58 mm bottom).

4.2.8 Wash bottles, one containing 125 ml dispersant solution diluted to 1 l with deionized water, and one with deionized water.

4.2.9 Laboratory balance, of sensitivity 0,01 g.

4.2.10 Thermometer, with scale reading including 16 °C to 32 °C, with an accuracy of 0,5 °C.

4.2.11 Beaker, of capacity 250 ml.

4.2.12 Water bath or constant temperature room, capable of maintaining a convenient constant temperature at or near 20 °C.

4.2.13 Glass sedimentation cylinder, 457 mm high and 63,5 mm diameter, and marked for a volume of 1 l (see ASTM D422).

4.2.14 Hydrometer, ASTM No. 151H, conforming to ASTM E100, graduated to read specific gravity of the suspension.

4.2.15 Evaporating dishes.

4.2.16 Laboratory spatulas, of assorted sizes.

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

4.2.18 Spray nozzle[3].

4.3 Sampling

Obtain four samples of approximately 10 g and one sample of approximately 80 g of the barite by tube sampling and quartering.

4.4 Calculation of moisture content

4.4.1 Weigh 10 g ± 0,01 g of barite obtained in 4.3.

4.4.2 Dry to constant mass at a temperature of 105 °C ± 3 °C.

4.4.3 Cool the sample in a desiccator and weigh.

4.4.4 Calculate the moisture content from equation (1), as a percent (mass fraction)

$$w_h = 100\,\frac{m_o - m_d}{m_o} \qquad (1)$$

3) Spraying Systems Company No. TG 6.5 tip with 1/4 TT body is the trade name of a suitable product supplied by Spraying Systems. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

where

w_h is the moisture content, in percent (mass fraction);

m_o is the mass of original sample, in grams;

m_d is the mass of dry sample, in grams.

4.5 Sieve analysis

4.5.1 Weigh 10 g ± 0,01 g of barite obtained in 4.3, and place in a mixing container. Add 44 ml of dispersant solution. Hand-stir the sample and dilute to approximately 350 ml with deionized water. Stir 5 min on mixer.

4.5.2 Wash the sample with the diluted dispersant solution onto a 75 µm mesh sieve. Continue to wash with approximately 400 ml of the dilute dispersant solution using a wash bottle. Then wash the material on the screen using tap water from a spray nozzle at 70 kPa for 2 min. While washing, allow the elbow bend of the nozzle to rest on the rim of the sieve and move the spray of water repeatedly over the surface of the screen. After tap-water washing, wash the sample at least twice with deionized water; then transfer the residue from the screen to a tared evaporating dish, using deionized water to remove the residue from the screen.

4.5.3 Dry the residue in the oven to constant mass, cool in a desiccator. Weigh to ± 0,01 g.

4.5.4 Repeat 4.5.1, 4.5.2, and 4.5.3 using 45 µm and 30 µm mesh sieves with separate barite samples.

4.5.5 Calculate m_d, w_r and w_f from equations (2), (3) and (4).

$$m_d = m_o \frac{100 - w_h}{100} \quad (2)$$

$$w_r = 100 \frac{m_r}{m_d} \quad (3)$$

$$w_f = 100 - w_r \quad (4)$$

where

w_h is the moisture content, in percent (mass fraction), as determined in 4.4.4;

m_o is the mass of original sample, in grams;

m_d is the mass of dry sample, in grams;

m_r is the mass of residue, in grams;

w_r is the residue remaining on the sieve, in percent (mass fraction);

w_f is the part of material finer than the sieve, in percent (mass fraction).

4.6 Sedimentation analysis

4.6.1 Weigh 80 g ± 0,1 g of barite obtained in 4.3, and place in a mixing container. Add 125 ml ± 2 ml of dispersant solution, hand-stir the sample and dilute to approximately 400 ml with deionized water. Stir 5 min on mixer.

4.6.2 Transfer the mixture to a 1 l sedimentation cylinder, washing completely the sample into the cylinder and adding deionized water to the 1 l mark. Mix the contents thoroughly by constantly changing the cylinder from the upright to the inverted position and back for 60 s while holding a rubber stopper in the top of the cylinder.

4.6.3 When the cylinder is set on the countertop, start the timer immediately. Hang the thermometer in the sample suspension.

4.6.4 Take hydrometer and thermometer readings after 5 min, 10 min, 20 min, 40 min, 90 min, 180 min and 360 min. (This should give particle sizes ranging from less than 2 µm to over 10 µm).

When taking a hydrometer reading, carefully and slowly insert the hydrometer, about 20 s to 25 s before the reading is due, to approximately the depth at which the reading is taken. As soon as the reading has been taken, carefully and slowly remove the hydrometer. Clean hydrometer in deionized or distilled water, and dry.

4.6.5 Calculate the amount (w_p) and particle diameter (D) of the fines material from equations (5) and (6) (see example data sheet and calculation below).

$$w_d = \frac{100\ 000\rho}{m_d(\rho-1)} \times (H_c - 1) \quad (5)$$

where

w_d is the mass fraction of sample in suspension, in percent;

m_d is the mass of dried sample, in grams;

ρ is the density of the barite sample, in grams per millilitre (determined according to ISO 13500:1998, clause 7);

H_c is the corrected hydrometer reading [the hydrometer reading minus composite correction (see 4.6.7 and 4.6.8)];

D is the particle diameter (equivalent spherical diameter), in micrometres;

$$D = 100\sqrt{\frac{30\eta \cdot l}{980(\rho-1)t}} = 17{,}5\sqrt{\frac{\eta \cdot l}{(\rho-1)t}} \quad (6)$$

where

η is the viscosity of water at test temperature, in centipoise (cP) (see Table 1);

l is the effective depth of hydrometer, in centimetres (see Table 2);

t is the time interval from start of sedimentation to taking of reading, in minutes.

Table 1 — Viscosity of water at various temperatures

Temperature θ °C	Viscosity η cP	Temperature θ °C	Viscosity η cP
15,6	1,1225	22,2	0,9533
16,1	1,1061	22,8	0,9399
16,7	1,0911	23,3	0,9291
17,2	1,0773	23,9	0,9163
17,8	1,0611	24,4	0,9058
18,3	1,0479	25,0	0,8937
18,9	1,0324	25,6	0,8815
19,4	1,0197	26,1	0,8717
20,0	1,0050	26,7	0,8601
20,6	0,9904	27,2	0,8507
21,1	0,9785	27,8	0,8396
21,7	0,9646	28,3	0,8305

NOTE 1 1 cP = 1 mPa·s.

NOTE 2 Values were calculated by the formula:

$$\eta = \frac{1}{0{,}021482\left[(\theta - 8{,}435) + \sqrt{8078{,}4 + (\theta - 8{,}435)^2}\right] - 1{,}2}$$

where

η is the viscosity, in centipoise;

θ is the temperature, in degrees Celsius.

NOTE 3 See references [3] and [4].

EXAMPLE A typical data sheet for barite and calculation is given below.

Data sheet for barite, SG 4,30				
Time t min	**Hydrometer reading** ρ g/ml	**Temperature** θ °C	**Hydrometer correction from curve**	**Corrected hydrometer reading** H_c
5	1,035 0	25,5	− 0,001 9	1,033 1
10	1,028 0	25,5	− 0,001 9	1,026 1
20	1,021 0	25,5	− 0,001 9	1,019 1
40	1,014 0	25,5	− 0,001 9	1,012 1
90	1,012 0	25,5	− 0,001 9	1,010 1
180	1,008 5	25,0	− 0,002 0	1,006 5
360	1,007 0	25,0	− 0,002 0	1,005 0

$$w_d = \frac{100\,000(4{,}30)(1{,}0261 - 1)}{80(4{,}30 - 1)} = 42{,}5\ \%$$

$$D = 17{,}5\sqrt{\frac{\eta l}{(\rho - 1)t}} = 17{,}5\sqrt{\frac{(0{,}8837)\ 8{,}9}{(4{,}30 - 1)10}} = 8{,}5$$