INTERNATIONAL STANDARD

ISO 10416

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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 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 10416 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 10416:2002), which has been technically revised.

Introduction

This International Standard, which establishes testing methodologies for drilling fluid materials, is based on API RP 13I, seventh edition/ISO 10416:2002 [2]. This International Standard was developed in response to a 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 can yield reproducible and accurate results. The tests are anticipated to be performed in a laboratory setting, but can 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.

It is necessary that users of this International Standard be aware that further or differing requirements can 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, reagents and sometimes the supplier(s) of those materials that 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 applicable 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 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 91-1:1992, *Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 °C and 60 °F*

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

ISO 2977:1997, *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 viscosity*

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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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

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

ISO 10414-2:—[1)], *Petroleum and natural gas industries — Field testing of drilling fluids — Part 2: Oil-based fluids*

1) To be published. (Revision of ISO 10414-2:2002)

ISO 13500:—[2], *Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests*

ASTM D 1141, *Standard Practice for the Preparation of Substitute Ocean Water*

ASTM D 4052, *Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter*

ASTM D 5186, *Standard Test Method for Determination of Aromatic Content and Polynuclear Aromatic Content of Diesel Fuels and Aviation Turbine Fuels by Supercritical Fluid Chromatography*

ASTM E 100, *Standard Specification for ASTM Hydrometers*

IP 391, *Petroleum products — Determination of aromatic hydrocarbon types in middle distillates — High performance liquid chromatography method with refractive index detection*

3 Terms and definitions

3.1
ACS reagent grade
chemical which meets purity standards as specified by the American Chemical Society (ACS)

3.2
base oil
solids- and water-free hydrocarbon oil, commonly used in the drilling fluid industry for preparation and/or dilution of an oil-based drilling or completion fluid

NOTE 1 Commonly used base oils are often termed "mineral oils", "solvent oils" or "absorber oils" and also include the "diesel oils".

NOTE 2 See Clause 15.

3.3
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 $cm^3 \cdot s^{-1} \bullet cm^{-2}$ cross-sectional area, and under a pressure or equivalent hydraulic gradient of 1 $atm \bullet cm^{-1}$

NOTE 1 cP = 1 mPa·s.

3.4
flash side
side containing residue ("flash") from stamping and with concave indentations

3.5
quarter, verb
mix and divide into four specimens to assure homogeneity of specimens

3.6
spurt loss
volume of fluid that passes through the filtration medium before a filter cake is formed

3.7
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

2) To be published. (Revision of ISO 13500:2006)

4 Symbols and abbreviations

AA	atomic absorption spectroscopy
ACS	American Chemical Society
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
BB	ball bearings
C	concentration
$C_{B,A}$	concentration of bentonite, in kilograms per cubic metre;
$C_{B,B}$	concentration of bentonite, in pounds per barrel;
$C_{DS,A}$	is the concentration of drilled solids, in kilograms per cubic metre
$C_{DS,B}$	is the concentration of drilled solids, in pounds per barrel
$C_{LG,A}$	concentration of low gravity solids, in kilograms per cubic metre;
$C_{LG,B}$	concentration of low gravity solids, in pounds per barrel;
$C_{MBT-AVE}$	average methylene blue capacity for all low-gravity solids, in milliequivalents per hundred grams (meq/100 g)
C_{MBT-B}	methylene blue capacity of commercial bentonite, in milliequivalents per hundred grams (meq/100 g)
C_{MBT-DS}	methylene blue capacity of drill solids, in milliequivalents per hundred grams (meq/100 g)
CAS	Chemical Abstracts Service, a division of ACS
d	inner diameter
D	outer diameter
DCP	direct current plasma
DS	drill solids
$E_{BE,A}$	bentonite equivalent, expressed in kilograms per cubic metre
$E_{BE,B}$	bentonite equivalent, expressed in pounds per barrel;
e	thinner efficiency, in percent
EDTA	ethylenediaminetetraacetic acid
F_{PI}	the performance index (mathematical symbol)
H_c	corrected hydrometer reading (the hydrometer reading minus composite correction)
HTHP	high-temperature, high-pressure
ICP	inductively coupled plasma

IP	standards issued by Energy Institute (formerly, Institute of Petroleum)
ISA	ionic strength adjuster
ISE	ion-selective electrode
LGS	low-gravity solids
MBT	methylene blue test
a	abrasion, in milligrams per minute
$G_{10\ s}$	10 s gel reading
$G_{10\ min}$	10 min gel reading
l	effective depth of hydrometer, in centimetres (see Table 2)
m_{As}	mass of arsenic in the digested sample, in micrograms
m_b	initial blade mass, in milligrams
m_B	mass of a commercial bentonite sample, in grams
m_h	mass of water required for solids adjustment, in grams
m_l	specified mass of suspension, in grams
m_d	mass of a dry sample, in grams
m_{DS}	mass of drill solids sample, in grams
m_f	final blade mass, in milligrams
m_{Hg}	mass of mercury in the digested sample, in micrograms
m_o	mass of original sample, in grams
m_r	mass of residue, in grams
m_s	mass of sample, in grams
PAC-HV	high-viscosity polyanionic cellulose
PAC-LV	low-viscosity polyanionic cellulose
PI	performance index
P_{df}	phenolphthalein alkalinity of the drilling fluid
PPA	permeability plugging apparatus
PPT	permeability plugging test
p_r	performance of reference thinner, for example yield point or gel strength as determined in accordance with ISO 10414-1
p_s	performance of test sample, for example yield point or gel strength as determined in accordance with ISO 10414-1

PTFE	polytetrafluoroethylene
t_1	time at initial reading, in minutes
t_2	time at final reading, in minutes
w_{As}	mass fraction of arsenic in the sample, in micrograms per gram
w_{Cd}	mass fraction of cadmium in the sample, in micrograms per gram
w_d	mass fraction of sample in suspension, in percent
w_h	moisture content, as percent mass fraction
w_s	mass fraction of solids, in percent
w_{Hg}	mass fraction of mercury in the sample, in micrograms per gram
w_f	part of material finer than the sieve, in percent (mass fraction)
w_{Pb}	mass fraction of lead in the sample, in micrograms per gram
w_r	mass recovery (mass residue), expressed as a mass fraction in percent
t	time, in minutes
V	volume of methylene blue solution used in titration, in millilitres
V_c	volume of filtrate collected between 7,5 min and 30 min, in millilitres
V_f	corrected volume of filtrate, in millilitres
V_o	volume of solution, in millilitres
V_s	volume of sample, in millilitres
V_{PPT}	PPT volume, in millilitres
V_1	spurt loss, in millilitres
$V_{7,5}$	filtrate volume after 7,5 min, in millilitres
V_{30}	filtrate volume after 30 min, in millilitres
v_f	static filtration rate (velocity of flow), in millilitres per minute
Y_P	yield point, in pascals
ρ	density, in grams per millilitre
ρ_{Cd}	density of cadmium in the digested sample, in micrograms per millilitre
ρ_{Pb}	density of lead in the digested sample, in micrograms per millilitre
η	viscosity of water at test temperature, in centipoise (cP) (see Table 1)
η_A	apparent viscosity
η_P	plastic viscosity, in centipoise
η_{600}	viscosity reading at 600 r/min, in millipascal seconds

5 Barite

5.1 Principle

Fines are the particles of 2 µm to 10 µm equivalent spherical diameter and are considered detrimental to drilling fluids at high concentrations. Both sieve analysis and sedimentation methods for determining fines concentration are described in 5.2 to 5.6.

5.2 Reagents and apparatus

5.2.1 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. 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.

5.2.2 Oven, capable of maintaining a temperature of 105 °C ± 3 °C (220 °F ± 5 °F).

5.2.3 Mixer, capable of operation at 11 500 r/min ± 300 r/min under load, with single corrugated impeller approximately 25,4 mm (1 in) in diameter [3)].

5.2.4 Container, for mixing, 180 mm (7-1/8 in) deep, d = 97 mm (3-3/4 in) at top and 70 mm (2-3/4 in) at bottom [4)].

5.2.5 Sieves, of mesh sizes 75 µm, 45 µm and 30 µm, having a diameter of 76 mm (3,0 in) and a depth of 64 mm (2,5 in) from the top of the frame to the wire cloth.

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

5.2.7 Stopper, rubber, size 13 [diameters 68 mm (2-2/3 in) top and 58 mm (2-1/4 in) bottom].

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

5.2.9 Balance, accuracy ± 0,01 g.

5.2.10 Thermometer, with a scale reading 16 °C to 32 °C (60 °F to 90 °F), accurate to 0,5 °C (1 °F).

5.2.11 Beaker, 250 ml.

5.2.12 Water bath or **constant-temperature room**, capable of maintaining a convenient constant temperature at or near 20 °C (68 °F).

5.2.13 Cylinder, glass sedimentation, 457 mm (18,0 in) high and 63,5 mm (2,5 in) in diameter, and marked for a volume of 1 l (see ASTM D 422).

5.2.14 Hydrometer, ASTM No. 151H, conforming to ASTM E 100, graduated to read the specific gravity of the suspension.

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3) 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.

4) 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.

5.2.15 **Dishes**, evaporating.

5.2.16 **Spatulas**, laboratory, of assorted sizes.

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

5.2.18 **Spray nozzle** [5].

5.3 Sampling

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

5.4 Calculation of moisture content

5.4.1 Weigh 10 g ± 0,01 g of the barite obtained in 5.3.

5.4.2 Dry to constant mass at a temperature of 105 °C ± 3 °C (220 °F ± 5 °F).

5.4.3 Cool the sample in a desiccator and weigh.

5.4.4 Calculate w_h, the moisture content, expressed as a percent (mass fraction), from Equation (1):

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

where

m_o is the mass of original sample, expressed in grams;

m_d is the mass of dry sample, expressed in grams.

5.5 Sieve analysis

5.5.1 Weigh 10 g ± 0,01 g of the barite obtained in 5.3, and place it 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 the mixer.

5.5.2 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 (10 psi) 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.

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

5.5.4 Repeat 5.5.1, 5.5.2, and 5.5.3 using 45 µm and 30 µm mesh sieves with separate barite samples.

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5) 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.