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

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Foreword

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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.

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This second edition cancels and replaces the first edition (ISO 10416:2002), which has been technically revised.

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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

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.

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: Waterbased 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

Commonly used base oils are often termed "mineral oils", "solvent oils" or "absorber oils" and also include the "diesel oils". (standards.iteh.ai)

NOTE 2 See Clause 15.

ISO 10416:2008

3.3 darcy

https://standards.iteh.ai/catalog/standards/sist/864f4d7d-5ab0-49dc-be1e-801152e7ab1a/iso-10416-2008

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 cm³·s⁻¹•cm⁻² cross-sectional area, and under a pressure or equivalent hydraulic gradient of 1 atm•cm⁻¹

NOTE 1 cP = 1 mPa·s.

3.4

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

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

Cconcentration

 $C_{\mathsf{B},\mathsf{A}}$ concentration of bentonite, in kilograms per cubic metre;

concentration of bentonite, in pounds per barrel; $C_{\mathsf{B},\mathsf{B}}$

is the concentration of drilled solids, in kilograms per cubic metre $C_{\mathsf{DS},\mathsf{A}}$

is the concentration of drilled solids, in pounds per barrel $C_{\mathsf{DS},\mathsf{B}}$

 $C_{\mathsf{LG.A}}$ concentration of low gravity solids, in kilograms per cubic metre;

concentration of low gravity solids, in pounds per barrel; $C_{\mathsf{LG.B}}$

average methylene blue capacity for all low-gravity solids, in milliequivalents per hundred grams $C_{\mathsf{MBT-AVE}}$

(meq/100 g) (standards.iteh.ai)

methylene blue capacity of commercial bentonite, in milliequivalents per hundred grams $C_{\mathsf{MBT-B}}$

(meq/100 g) https://standards.iteh.ai/catalog/standards/sist/864f4d7d-5ab0-49dc-be1e-

methylene blue capacity of driff solids, in milliequivalents per hundred grams (meq/100 g) $C_{\mathsf{MBT-DS}}$

Chemical Abstracts Service, a division of ACS CAS

d inner diameter

Douter diameter

DCP direct current plasma

DS drill solids

bentonite equivalent, expressed in kilograms per cubic metre E_{BF} A

bentonite equivalent, expressed in pounds per barrel; $E_{\mathsf{BE},\mathsf{B}}$

thinner efficiency, in percent

EDTA ethylenediaminetetraacetic acid

 F_{PI} the performance index (mathematical symbol)

corrected hydrometer reading (the hydrometer reading minus composite correction) H_{c}

HTHP high-temperature, high-pressure

ICP inductively coupled plasma

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ISO 10416:2008(E)

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

 $G_{10 \text{ s}}$ 10 s gel reading

 $G_{10 \text{ min}}$ 10 min gel reading

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

 $m_{\rm R}$ mass of a commercial bentonite sample, in grams

 m_{h} mass of water required for solids adjustment, in grams

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 m_{\parallel} specified mass of suspension, in grams

(ständards.iteh.ai)

 $m_{\rm d}$ mass of a dry sample, in grams

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 $m_{\rm DS}$ mass of drill solids sample in grams alog/standards/sist/864f4d7d-5ab0-49dc-be1e-

801152e7ab1a/iso-10416-2008

 $m_{\rm f}$ final blade mass, in milligrams

 $m_{\rm Hg}$ mass of mercury in the digested sample, in micrograms

 $m_{\rm O}$ mass of original sample, in grams

 $m_{\rm r}$ mass of residue, in grams

 $m_{\rm s}$ mass of sample, in grams

PAC-HV high-viscosity polyanionic cellulose

PAC-LV low-viscosity polyanionic cellulose

PI performance index

 $P_{
m df}$ phenolphthalein alkalinity of the drilling fluid

PPA permeability plugging apparatus

PPT permeability plugging test

 $p_{\rm r}$ 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
<i>t</i> ₁	time at initial reading, in minutes
t_2	time at final reading, in minutes
[₩] As	mass fraction of arsenic in the sample, in micrograms per gram
$^{\mathcal{W}}$ 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
$^{\mathcal{W}}$ 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 millilitres6:2008
V_{o}	https://standards.iteh.ai/catalog/standards/sist/864f4d7d-5ab0-49dc-be1e-volume of solution, in millilitrese7ab1a/iso-10416-2008
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
$ ho_{Cd}$	density of cadmium in the digested sample, in micrograms per millilitre
$ ho_{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

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5 Barite

5.1 Principle

Fines are the particles of $2 \, \mu m$ to $10 \, \mu m$ 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 \pm 3 °C (220 °F \pm 5 °F).
- **5.2.3 Mixer**, capable of operation at 11 500 r/min \pm 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: (3,1 teh. 21)
- **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 \pm 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.

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 \pm 0,01 g of the barite obtained in 5.3.
- **5.4.2** Dry to constant mass at a temperature of 105 °C \pm 3 °C (220 °F \pm 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):

$$w_{h} = 100 \frac{m_{o} - m_{d}}{m_{o}} \text{ iTeh STANDARD PREVIEW}$$
(standards.iteh.ai)

where

 $m_{\rm O}$ is the mass of original sample, expressed in grams; https://standards.iteh.ai/catalog/standards/sist/864f4d7d-5ab0-49dc-bele- $m_{\rm cl}$ is the mass of dry sample, expressed in grams.416-2008

5.5 Sieve analysis

- **5.5.1** Weigh 10 g \pm 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 μ m 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 \pm 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.