BRITISH STANDARD

BS EN ISO 10416:2009

Petroleum and natural gas industries — Drilling fluids — Laboratory testing

ICS 75.180.10

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National foreword

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The UK participation in its preparation was entrusted by Technical Committee PSE/17, Materials and equipment for petroleum, petrochemical and natural gas industries, to Subcommittee PSE/17/-/3, Drilling and completion fluids and well cements.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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Contents

Page

Introduction vii

1 Scope 1

2 Normative references 1

3 Terms and definitions 2

4 Symbols and abbreviations 3

5 Barite 6
5.1 Principle 6
5.2 Reagents and apparatus 6
5.3 Sampling 7
5.4 Calculation of moisture content 7
5.5 Sieve analysis 7
5.6 Sedimentation analysis 8

6 Barite performance 12
6.1 Principle 12
6.2 Reagents and apparatus 12
6.3 Base drilling fluid preparation 13
6.4 Rheology test 13
6.5 Calculation 14

7 Abrasiveness of weighting materials 14
7.1 Principle 14
7.2 Reagents and apparatus 15
7.3 Determination of abrasion 15

8 Mercury in drilling fluid barite 17
8.1 Principle 17
8.2 Reagents and apparatus 17
8.3 Preparation of standards 19
8.4 Sample digestion 19
8.5 Check for recovery of Hg during digestion 20
8.6 Analysis of standards and samples 20
8.7 Calculation 20

9 Cadmium and lead in drilling fluid barite 21
9.1 Principle 21
9.2 Reagents and apparatus 21
9.3 Preparation of combined cadmium and lead standards 22
9.4 Sample digestion 22
9.5 Analysis of standards and samples 22
9.6 Calculation 23

10 Arsenic in drilling fluid barite 23
10.1 Principle 23
10.2 Reagents and apparatus 24
10.3 Preparation of standards 25
10.4 Sample digestion 25
10.5 Analysis of standards and samples 26
10.6 Calculation 26

11 Bridging materials for regaining circulation 26

Documents
Document Sharing Hub

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Full document details: BS EN ISO 10416:2009

11.1 Principle ... 26
11.2 Apparatus ... 27
11.3 Preparation of test drilling fluid ... 27
11.4 Static slot test ... 27
11.5 Dynamic slot test ... 28
11.6 Static marble bed test ... 28
11.7 Dynamic marble bed test ... 28
11.8 Static ball bearings (BB shot) bed test ... 29
11.9 Dynamic ball bearings (BB shot) bed test ... 29

12 Filtration-control agents ... 29
12.1 Principle ... 29
12.2 Reagents and apparatus ... 29
12.3 General instructions for preparation of base drilling fluids ... 31
12.4 Salt-saturated drilling fluid ... 31
12.5 High-hardness, salt-saturated drilling fluid ... 32
12.6 10 % potassium chloride (KCl) drilling fluid ... 32
12.7 Pre-hydrated bentonite slurry ... 33
12.8 Modified seawater drilling fluid ... 33
12.9 Low-salinity drilling fluid ... 33
12.10 Lime-treated drilling fluid ... 34
12.11 Low solids, non-dispersed drilling fluid ... 34
12.12 Freshwater lignosulfonate drilling fluid ... 35
12.13 Initial performance test ... 35
12.14 Performance after heat ageing ... 36

13 Methylene blue test for drilled solids and commerical bentonite ... 36
13.1 Methylene blue capacity of drill solids ... 36
13.2 Methylene blue capacity of commercial bentonite ... 39
13.3 Solids content ... 40

14 Deflocculation test for thinner evaluation ... 41
14.1 Principle ... 41
14.2 Reagents and apparatus ... 42
14.3 Procedure for moisture content ... 43
14.4 Calculation of moisture content ... 43
14.5 Preparation of drilling fluid base ... 43
14.6 Calculation ... 44
14.7 Determination of rheological properties ... 44
14.8 Calculation of thinner efficiency ... 46

15 Testing base oils used in drilling fluids ... 46
15.1 General ... 46
15.2 Reagents and apparatus ... 46
15.3 Density, relative density (specific gravity), or API gravity-hydrometer method (see ISO 3675) ... 46
15.4 Density and relative density of liquids using a digital density meter (see ASTM D 4052) ... 47
15.5 Kinematic viscosity of transparent and opaque oils — Calibrated capillary tube method (see ISO 3104) ... 47
15.6 Distillation (see ISO 3405) ... 47
15.7 Aniline point and mixed aniline point (see ISO 2977:1997) ... 48
15.8 Pour point (see ISO 3016) ... 48
15.9 Flash point by Pensky-Martens closed tester (see ISO 2719) ... 49
15.10 Aromatics content (see IP 391 or ASTM D 5186) ... 49

16 Potassium ion content — Ion-selective electrode method ... 50
16.1 Principle ... 50
16.2 Reagents and apparatus ... 50
16.3 Preparation of electrodes ... 51
16.4 Operational check of electrode system ... 51
16.5 Measurements using a meter with direct concentration readout capability ... 52

© ISO 2010

Documents
Document Sharing Hub
Full document details: BS EN ISO 10416:2009

16.6 Measurements with instruments that provide either a digital or an analogue readout in millivolts 52

17 Calcium ion content — Ion-selective electrode method 53
17.1 Principle 53
17.2 Reagents and apparatus 53
17.3 Preparation of electrodes 54
17.4 Operational check of electrode system 55
17.5 Measurements using a meter with direct concentration readout capability 55
17.6 Measurements with instruments that provide either a digital or an analogue readout in millivolts 55

18 Sodium ion content — Ion-selective electrode method 56
18.1 Principle 56
18.2 Reagents and apparatus 57
18.3 Preparation and operational check of the electrode system 57
18.4 Measurements using a meter with a direct concentration-readout capability 58
18.5 Measurements using a meter with readout in millivolts 58

19 Density of solids — Stereopycnometer method 59
19.1 Principle 59
19.2 Apparatus 59
19.3 Procedure — Stereopycnometer method 59
19.4 Calculation — Stereopycnometer method 60

20 Density of solids — Air comparison pycnometer method 61
20.1 Principle 61
20.2 Apparatus 61
20.3 Procedure — Air comparison pycnometer method 61
20.4 Calculation — Air comparison pycnometer method 61

21 Ageing of water-based drilling fluids 62
21.1 Principle 62
21.2 Practices common to preparation, handling and testing over all temperature ranges 62
21.3 Drilling fluid sample preparation and ageing at ambient temperature 63
21.4 Drilling fluid ageing at moderate temperatures [ambient to 65 °C (150 °F)] 64
21.5 Drilling fluid ageing at substantially elevated temperatures [over 65 °C (150 °F)] 66
21.6 Inertness and chemical compatibility in high-temperature ageing cells 68
21.7 Obtaining supplies and services for the ageing of drilling fluid samples 69

22 Ageing of oil-based drilling fluids 69
22.1 Principle 69
22.2 Apparatus 70
22.3 Practices common to preparation, handling and testing over all temperature ranges 71
22.4 Drilling fluid ageing at ambient temperatures 72
22.5 Drilling fluid ageing at moderate temperatures [ambient to 65 °C (150 °F)] 73
22.6 Drilling fluid ageing at substantially elevated temperatures [over 65 °C (150 °F)] 74
22.7 Inertness and chemical compatibility in high-temperature ageing cells 75
22.8 Obtaining supplies and services for the ageing of drilling fluid samples 76

23 Shale-particle disintegration test by hot rolling 76
23.1 Principle 76
23.2 Reagents and apparatus 77
23.3 Procedure 77
23.4 Calculation 78

24 Drilling fluid materials — High-viscosity polyanionic cellulose (PAC-HV) (regular) 79
24.1 Principle 79
24.2 Determination of moisture content 79
24.3 Procedures with test fluid containing PAC-HV 80

25 Drilling fluid materials — Low-viscosity polyanionic cellulose (PAC-LV) 82
25.1 Principle 82
25.2 Determination of moisture content 83

Documents
Document Sharing Hub

© ISO 2010

Full document details: BS EN ISO 10416:2009

25.3 Procedures with test fluid containing PAC-LV ... 83

26 Preparation and evaluation of invert-emulsion drilling fluids ... 86
26.1 Principle ... 86
26.2 Reagents and apparatus ... 86
26.3 Mixing of the initial drilling fluid ... 87
26.4 Testing the properties of the initial drilling fluid ... 88
26.5 Preparation of the sample contaminated by seawater ... 88
26.6 Preparation of the sample contaminated by base evaluation clay ... 89
26.7 Preparation of the sample contaminated by mixed-salt brine ... 89
26.8 Procedure for hot-rolling ... 89
26.9 Procedure for static ageing ... 89
26.10 Procedure for testing after heat ageing ... 90

27 High-temperature/high-pressure filtration testing of drilling fluids using the permeability plugging apparatus and cells with set-screw-secured end caps ... 90
27.1 Principle ... 90
27.2 Safety considerations ... 90
27.3 Apparatus — Permeability-plugging apparatus (PPA) with set-screw-secured end caps ... 92
27.4 Procedure for high-temperature/high-pressure (HTHP) filtration ... 94
27.5 Test conclusion and disassembly ... 97
27.6 Data reporting ... 99

28 High-temperature/high-pressure filtration testing of drilling fluids using the permeability-plugging apparatus and cells with threaded end caps ... 100
28.1 Principle ... 100
28.2 Safety considerations ... 100
28.3 Apparatus — Permeability-plugging apparatus (PPA) with threaded end caps ... 102
28.4 Procedure for high-temperature/high-pressure (HTHP) filtration ... 104
28.5 Test conclusion and disassembly ... 106
28.6 Data reporting ... 108

Bibliography ... 109

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Documents
Document Sharing Hub
Full document details: BS EN ISO 10416:2009

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.

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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*

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

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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)

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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;
$C_{MBT\text{-}AVE}$	average methylene blue capacity for all low-gravity solids, in milliequivalents per hundred grams (meq/100 g)
$C_{MBT\text{-}B}$	methylene blue capacity of commercial bentonite, in milliequivalents per hundred grams (meq/100 g)
$C_{MBT\text{-}DS}$	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

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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

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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

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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.

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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.

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5.5.5 Calculate m_d, the mass of dry sample, expressed in grams; m_r, the mass of residue, expressed in grams; and w_f, the part of material finer than the sieve, in percent (mass fraction), from Equations (2), (3) and (4), respectively.

$$m_d = m_o \frac{100 - w_h}{100} \tag{2}$$

$$w_r = 100 \frac{m_r}{m_d} \tag{3}$$

$$w_f = 100 - w_r \tag{4}$$

where

w_h is the moisture content, expressed in percent (mass fraction), as determined in 5.4.4;

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

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

5.6 Sedimentation analysis

5.6.1 Weigh the 80 g ± 0,1 g of barite obtained in 5.3, and place it 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 the mixer.

5.6.2 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.

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

5.6.4 Take hydrometer and thermometer readings after 5 min, 10 min, 20 min, 40 min, 90 min, 180 min and 360 min. (This is expected to 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 the hydrometer in deionized or distilled water and dry.

5.6.5 Calculate w_d, the mass fraction of sample in suspension, expressed as a percentage, from Equation (5) and D, the particle diameter (equivalent spherical diameter), expressed in micrometres, from Equation (6) (see the subsequent example data sheet and calculation):

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

where

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

ρ is the density of the barite sample, expressed in grams per millilitre (determined in accordance with ISO 13500:—, Clause 7);

H_c is the corrected hydrometer reading [the hydrometer reading minus composite correction (see 5.6.7 and 5.6.8)];

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

where

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

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

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

Table 1 — Viscosity of water at various temperatures

Temperature θ		Viscosity [a, b] η	Temperature θ		Viscosity [a, b] η
°C	°F	cP	°C	°F	cP
15,6	60	1,122 5	22,2	72	0,953 3
16,1	61	1,106 1	22,8	73	0,939 9
16,7	62	1,091 1	23,3	74	0,929 1
17,2	63	1,077 3	23,9	75	0,916 3
17,8	64	1,061 1	24,4	76	0,905 8
18,3	65	1,047 9	25,0	77	0,893 7
18,9	66	1,032 4	25,6	78	0,881 5
19,4	67	1,019 7	26,1	79	0,871 7
20,0	68	1,005 0	26,7	80	0,860 1
20,6	69	0,990 4	27,2	81	0,850 7
21,1	70	0,978 5	27,8	82	0,839 6
21,7	71	0,964 6	28,3	83	0,830 5

a 1 cP = 1 mPa·s.

b Values are calculated as given in the following equation:

$$\eta = \frac{1}{0{,}021\,482\left[(\theta - 8{,}435) + \sqrt{8\,078{,}4 + (\theta - 8{,}435)^2}\right] - 1{,}2}$$

where

η is the viscosity, expressed in centipoise;
θ is the temperature, expressed in degrees Celsius.

NOTE See References [3] and [4].

EXAMPLE A typical data sheet for barite; the calculation follows.

Data sheet for barite with a specific gravity of 4,30

Time t min	Hydrometer reading ρ g/ml	Temperature θ °C	°F	Hydrometer correction from curve g/ml	Corrected hydrometer reading H_c g/ml
5	1,035 0	25,5	78	–0,001 9	1,033 1
10	1,028 0	25,5	78	–0,001 9	1,026 1
20	1,021 0	25,5	78	–0,001 9	1,019 1
40	1,014 0	25,5	78	–0,001 9	1,012 1
90	1,012 0	25,5	78	–0,001 9	1,010 1
180	1,008 5	25,0	77	–0,002 0	1,006 5
360	1,007 0	25,0	77	–0,002 0	1,005 0

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

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

Table 2 — Values of effective depth

Uncorrected hydrometer reading [a]	Effective depth l		Uncorrected hydrometer reading [a]	Effective depth l	
	cm	in		cm	in
1,000	16,3	6,42	1,020	11,0	4,33
1,001	16,0	6,30	1,021	10,7	4,21
1,002	15,8	6,22	1,022	10,5	4,13
1,003	15,5	6,10	1,023	10,2	4,02
1,004	15,2	5.98	1,024	10,0	3,93
1,005	15,0	5,91	1,025	9,7	3,81
1,006	14,7	5,79	1,026	9,4	3,70
1,007	14,4	5,67	1,027	9,2	3,62
1,008	14,2	5,59	1,028	8,9	3,50
1,009	13,9	5,47	1,029	8,6	3,39
1,010	13,7	5,39	1,030	8,4	3,20
1,011	13,4	5,28	1,031	8,1	3,31
1,012	13,1	5,16	1,032	7,8	3,07
1,013	12,9	5,08	1,033	7,6	2,99
1,014	12,6	4,96	1,034	7,3	2,87
1,015	12,3	4,84	1,035	7,0	2,76
1,016	12,1	4,76	1,036	6,8	2,68
1,017	11,8	4,65	1,037	6,5	2,56
1,018	11,5	4,53	1,038	6,2	2,44
1,019	11,3	4,45	—	—	—

[a] Based on readings using Hydrometer No. 151H, conforming to ASTM E 100, graduated to read relative density (specific gravity) of the suspension.

5.6.6 Plot w_d, the percent (mass fraction) of sample in suspension, on an arithmetic scale as the ordinate (labelled “Cumulative percent finer”) and D, the particle diameter, on a logarithmic scale as the abscissa.

5.6.7 The composite correction is read from a curve drawn for each hydrometer. (Notice that this is a negative number.) The correction is necessary for the following reasons.

a) Equations for percentages of barite remaining in suspension in 5.6.5 are based on the use of deionized or distilled water. A dispersing agent is used in the water, however, and the specific gravity of the resulting solution is greater than that of water.

b) Hydrometers are calibrated at 20 °C (68 °F), and deviations from this standard temperature produce inaccuracies in the actual hydrometer reading. The amount of inaccuracy increases as the deviation from standard temperature increases.

c) Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of barite suspensions at the bottom of the meniscus, readings should be taken at the top and a correction applied.

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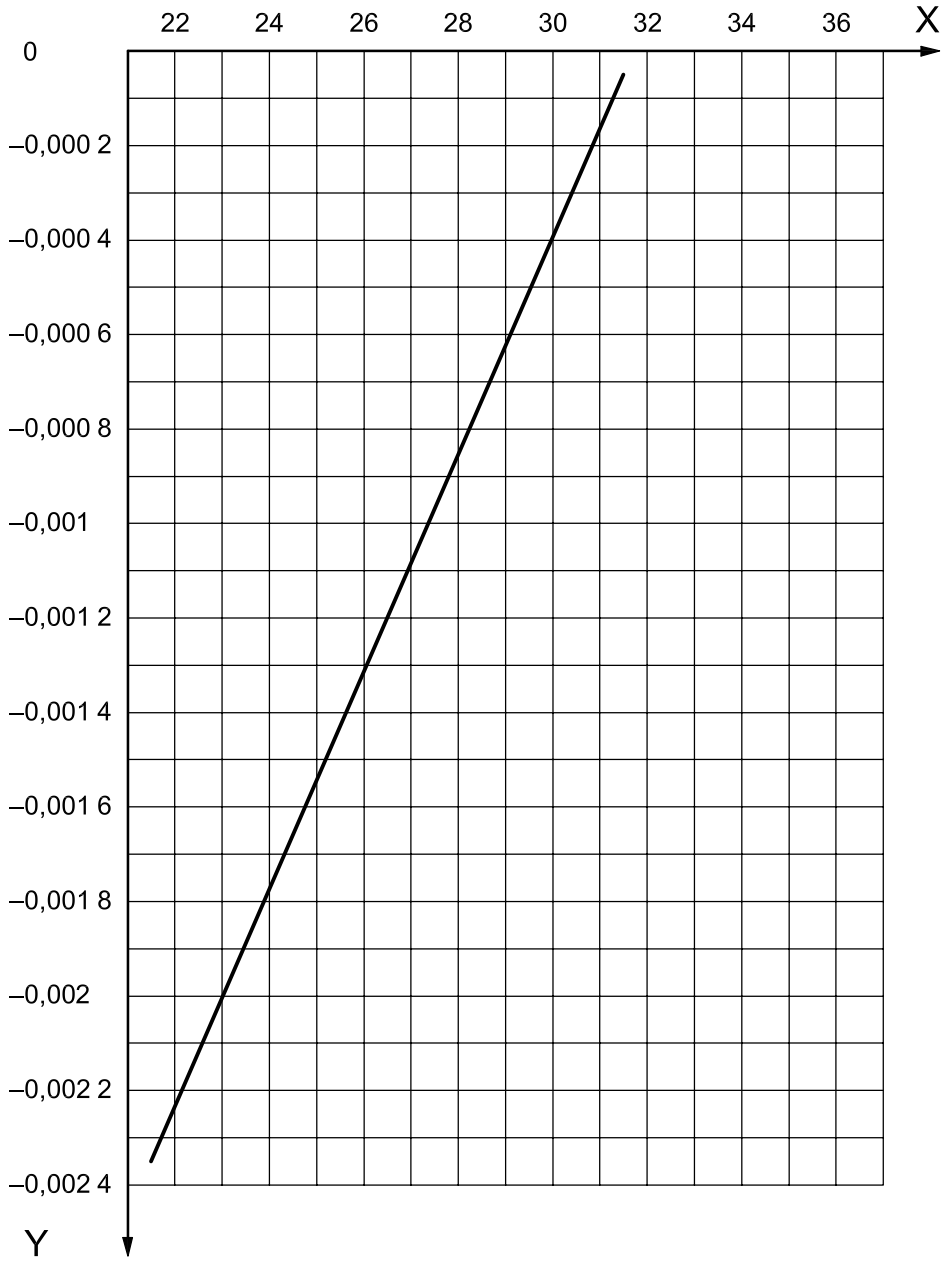
5.6.8 The sum of the corrections for the three items listed is designated as the composite correction, and may be determined experimentally as follows.

a) For convenience, a graph or table of composite corrections for a series of one-degree temperature differences for the range of expected test temperature may be prepared and used as needed. Measurement of the composite corrections may be made at several temperatures spanning the range of expected test temperatures and corrections for the test temperatures calculated assuming a straight-line relationship for the observed values (see Figure 1).

b) Prepare 1 l of liquid composed of deionized water and dispersing agent in the same proportion as is used in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature bath set for the first of the two selected temperatures. When the temperature of the liquid becomes constant, carefully and slowly insert the hydrometer and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H, the composite correction is equal to one minus this reading. Bring the liquid and the hydrometer to the second selected temperature and read the composite correction as before.



Key

X test temperature, expressed in degrees Celsius

Y correction to observed hygrometer reading at test temperature, expressed in grams per litre

NOTE See 5.6.7.

Figure 1 — Example of a graph of composite corrections for a hydrometer

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6 Barite performance

6.1 Principle

6.1.1 The rheological properties of a weighted drilling fluid vary depending upon the density, impurities and fines in the barite. This test compares the rheological properties of a drilling fluid using a barite to a similar system using an API test calibration barite (ISO 13500:—, 4.2) or a barite of known satisfactory performance.

6.1.2 The drilling fluid is a fresh-water, pre-hydrated bentonite/lignosulfonate slurry. A good indicator of undesirable performance due to excessive impurities and/or fines can be when the test sample has significantly higher rheological properties than the comparison barite.

6.1.3 This test does not reveal all undesirable impurities and should be used in conjunction with the barite specification tests in ISO 13500 and the recommended practice for chemical analysis of barite in API RP 13K [3].

6.2 Reagents and apparatus

6.2.1 Reagents

6.2.1.1 **Test calibration barite**.

6.2.1.2 **Reference bentonite**.

Stocks of test calibration barite and reference bentonite have been set aside by API. Requests for this material should be directed to the API, which will forward them to the supplier for all further handling. Accuracy of results for use of this calibration material is strictly the responsibility of the laboratories using it.

For locations without ready access to the test calibration barite and reference bentonite from API, tests using local material are possible by adjusting the amount of bentonite to give a proper base drilling fluid viscosity and using a reference barite of proven performance. When non-API test calibration materials are used, comparisons of data among laboratories can be very difficult or impossible.

6.2.1.3 **Sodium hydroxide** (CAS number 1310-73-2), $c_{NaOH} = 5$ mol/l NaOH solution.

6.2.1.4 **Chrome** or **ferrochrome lignosulfonate**, for between-laboratory comparisons.

A common sample of the same high-quality lignosulfonate should be divided among laboratories for all testing.

6.2.2 Apparatus

6.2.2.1 **Oven**, roller type, capable of maintaining a temperature of 105 °C ± 3 °C (220 °F ± 5 °F).

6.2.2.2 **pH meter**.

6.2.2.3 **Ageing cells**, two or more, of capacity 500 ml and made of stainless steel.

6.2.2.4 **Viscometer**, capable of operation at 3 r/min, 300 r/min and 600 r/min, in accordance with ISO 10414-1:2008, Clause 6.

6.2.2.5 **Mixer** 6), high-shear, equipped with a duplex blade.

6.2.2.6 **Speed control**, variable-transformer, for high-shear mixer.

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6) Dispersator® high shear mixer 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.

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6.2.2.7 **Beaker**, 1,5 l to 2 l.

6.2.2.8 **Flask**, volumetric, 1 l.

6.2.2.9 **Container**, 10 l.

6.2.2.10 **Balance**, of capacity 0 g to 3 000 g, accuracy ± 1 g.

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

The impeller shall be replaced when a mass loss of approximately 10 % occurs. Original blade mass is about 5,5 g.

6.2.2.12 **Mixer cups**, two or more depending on the number of samples for analysis, 180 mm (7-1/8 in) deep, d = 97 mm (3-3/4 in) at top and 70 mm (2-3/4 in) [5)].

6.3 Base drilling fluid preparation

6.3.1 Prepare approximately 3,5 l equivalents of bentonite slurry having a pH of 11,8 to 11,9 and a 600 r/min dial reading on the viscometer of 20 to 24 at 27 °C ± 3 °C (80 °F ± 5 °F).

Larger or smaller batches may be prepared. Use a suitably sized container and proportional quantities of bentonite, water, lignosulfonate and sodium hydroxide solution for a 59,9 kg/m^3 bentonite slurry containing 11,4 kg/m^3 lignosulfonate and 3,42 kg/m^3 NaOH.

6.3.2 To make a 3,5 l equivalent, add 3,5 l deionized or distilled water into a 10 l container. While stirring with a high-shear mixer at 8 500 r/min ± 1 000 r/min, slowly add 210 g of bentonite. Position the high-shear mixer shaft well off-centre in the container for maximum mixing shear. Continue stirring for 30 min.

6.3.3 Age for a minimum of 16 h in a sealed container at room temperature.

6.3.4 After ageing, re-stir for 5 min with the high-shear mixer at 8 500 r/min ± 1 000 r/min. Slowly add 40 g of lignosulfonate, then add 55 ml ± 10 ml of 5 mol/l NaOH. Stir for 30 min after the addition of the NaOH.

6.3.5 Age for a minimum of 4 h in a sealed container at room temperature.

Although the base drilling fluid can be used after only 4 h, ageing for 16 h is recommended.

6.3.6 Stir the base drilling fluid for 5 min at 8 500 r/min ± 1 000 r/min. Measure the pH of the drilling fluid and adjust within the range of 11,8 to 11,9 using 5 mol/l NaOH.

6.3.7 Read the dial on the viscometer for the base drilling fluid at 600 r/min at 27 °C ± 3 °C (80 °F ± 5 °F). The 600 r/min dial reading shall be 20 to 24. If the reading is 18 to 19, additional high-shear stirring will usually increase the reading to the acceptable range. If a second trial fails to give the proper dial reading, check materials and techniques used. If the reading is in the 24 to 28 range, readjust by adding deionized or distilled water in approximately 50 ml increments. After each addition, stir for 10 min and check the 600 r/min dial reading. If it is still greater than 24, add more water but do not exceed 250 ml. When the 600 r/min dial reading is within the specified range, measure and record the pH and proceed to 6.4.

6.4 Rheology test

6.4.1 Measure 240 ml of base drilling fluid into two or more mixer cups (one cup for each barite being tested and one cup for the test calibration barite).

6.4.2 While stirring on the mixer, slowly add 480 g of the sample being tested to the base drilling fluid. Stir for 5 min. Scrape the sides of the container to ensure that all the barite is well mixed. Stir for an additional 10 min.

6.4.3 Repeat 6.4.2 using the test calibration barite.

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6.4.4 Transfer each of the weighted slurries to an ageing cell. Seal and weigh the cells. Record the mass to the nearest gram.

6.4.5 Age the samples while hot-rolling for 16 h at 105 °C ± 3 °C (220 °F ± 5 °F).

6.4.6 Cool the cells to 25 °C ± 3 °C (77 °F ± 5 °F) and weigh. If the mass change exceeds 5 g compared to the result in 6.4.4, discard and repeat the test.

6.4.7 Open ageing cells and stir the aged drilling fluid for 5 min on the mixer.

6.4.8 Within 30 s after stirring and with the sample temperature at 27 °C ± 3 °C (80 °F ± 5 °F), measure the rheological properties. Determine and record the plastic viscosity, yield point, 10 s gel and 10 min gel and pH in accordance with ISO 10414-1:2008, Clauses 6 and 11. Also record the sample temperature.

6.5 Calculation

6.5.1 Significant differences between the rheological properties of the API test calibration barite slurry and the sample barite slurries are the best indicators of undesirable performance effects. A barite with fewer impurities and fines normally has a lower plastic viscosity, yield point and gels than the API test calibration barite.

6.5.2 One means of comparison is to determine F_{PI}, the performance index, of the various slurries using Equation (7):

$$F_{\mathrm{PI}} = \eta_{\mathrm{P}} + Y_{\mathrm{P}} + 3\left(G_{10\ \mathrm{s}} + G_{10\ \mathrm{min}}\right) \tag{7}$$

where

η_{P} is the plastic viscosity, expressed in centipoise;

Y_{P} is the yield point, expressed in pascals;

$G_{10\ \mathrm{s}}$ is the 10 s gel reading;

$G_{10\ \mathrm{min}}$ is the 10 min gel reading.

NOTE 1 1 cP = 1 mPa·s.

NOTE 2 In most cases, improved performance can be expected if the performance index of the test sample is equivalent to or less than the API test calibration barite.

EXAMPLE If the F_{PI} for the test calibration barite is equal to 150, an F_{PI} of 150 or less indicates a better performing barite. In the same test sequence, a test barite with an F_{PI} equal to 200 can add excessive fines and give high viscosities in a field drilling fluid.

7 Abrasiveness of weighting materials

7.1 Principle

7.1.1 Drilling fluid weighting materials can vary considerably in relative abrasivity. This laboratory test is designed to measure and evaluate this relative abrasiveness.

7.1.2 The test is performed using a standard test blade attached to a high-speed mixer to mix a base drilling fluid containing the weighting material. The mass loss of the blade is used to calculate the abrasiveness of the weighting material in milligrams per minute (mg/min).

7.1.3 This test is intended to give only a relative index of wear for weighting materials. It should not be used to infer whether or not these materials can cause abrasion problems in a field drilling fluid system.

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7.2 Reagents and apparatus

7.2.1 API reference bentonite.

Stocks of API reference bentonite for the test have been set aside by API. Requests for this material should be directed to the API, which will forward the request to the supplier for all further handling. Accuracy of results from the use of this material is strictly the responsibility of the laboratory using it.

7.2.2 Detergent.

7.2.3 Distilled-deionized water, in accordance with ISO 3696:1987, grade 3, or prepared by passing distilled water through a series of cation and anion exchange resins.

Use distilled-deionized water for preparation of all reagents and calibration standards and as dilution water.

7.2.4 Mixer, capable of operation at 11 500 r/min ± 300 r/min under load, with single, corrugated impeller [4)].

The impeller shall be replaced with an abrasion test blade in accordance with 7.2.8.

7.2.5 Container, approximately 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 [5)].

7.2.6 Balance, accuracy ± 0,000 1 g.

7.2.7 Balance, of capacity 500 g and accuracy ± 0,1 g.

7.2.8 Blade, for abrasion test, steel, of diameter 36,5 mm (1,4 in); flat side of thickness 1,4 mm (0,05 in); Rockwell scale hardness of 16; four waves of depth 6,4 mm (0,25 in) and width 15,3 mm (0,6 in); centre hole of diameter 7,1 mm (0,28 in), and a G-90 coating with a mass of approximately 12 g.

Each blade may be used up to a cumulative mass loss of 200 mg (approximately four runs).

7.2.9 Screw, 13 mm (1/2 in), size 10, round-head machine screw, 32 threads/in, to fasten the abrasion test blade securely onto the shaft of the abrasion mixer.

7.2.10 Tachometer, capable of reading 11 000 r/min ± 50 r/min.

7.2.11 Cylinder, graduated, TD 500 ml ± 2,5 ml.

7.2.12 Viscometer, direct indicating, as described in ISO 10414-1:2008, Clause 6.

7.2.13 Timer, mechanical or electrical, accurate to ± 0,1 min over the test interval.

7.2.14 Small brush.

7.3 Determination of abrasion

7.3.1 Prepare a base suspension by adding 15,0 g ± 0,1 g API reference bentonite to 350 ml ± 2,5 ml distilled water in a container while stirring on the base suspension mixer. At least two of these suspensions are needed for the test.

7.3.2 After about 5 min stirring time on the mixer, remove the container from the mixer and scrape its sides with the spatula to remove or dislodge any clay adhering to the container. Be sure that all clay clinging to the spatula is incorporated into the suspension.

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7.3.3 Replace the container on the mixer and continue stirring for about 15 min. The container should be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls at 5 min intervals. Total stirring time after adding the clay shall be 20 min.

7.3.4 Pour the base suspension into the cup provided with the direct-indicating viscometer. Note the dial reading at the 600 r/min rotor-speed setting when it reaches a constant value. The 600 r/min dial reading shall not exceed 10 at a test temperature of 25 °C ± 1 °C (77 °F ± 3 °F). If necessary, the 600 r/min dial reading of the base suspension can be lowered by adding a small amount of water.

7.3.5 Pour 300 ml ± 2,5 ml of base suspension into the mixing container (use 280 ml ± 2,5 ml when testing barite).

7.3.6 Add 300 g ± 1 g of weighting materials into the base suspension in the test container.

7.3.7 Immediately prior to use, clean the blade by washing with detergent and a small brush. Rinse thoroughly and dry. Weigh a freshly cleaned and dried abrasion test blade to the nearest 0,1 mg. Record as m_b, in milligrams.

Only non-corroded blades should be used.

7.3.8 Disconnect the mixer power cord to avoid accidental operation during blade installation. Centre and fasten the abrasion test blade, with the waves in the downward position, onto the abrasion mixer spindle with the lock washer and screw.

7.3.9 With the mixer off, place the mixing container into position on the mixer, such that the rim engages with the trip switch. Start the mixer using a series of quick “on-off” flips of the switch to bring the spindle up to speed gently.

NOTE An abrupt start slings some suspension out of the cup.

Blades should be polished after manufacture to remove rough edges and the blades should be installed on the mixer with the waves in the downward position.

7.3.10 Run the test for 20 min ± 0,1 min.

7.3.11 Turn the mixer off, disconnect the power cord, remove the mixing container and the abrasion test blade.

7.3.12 Clean and dry the abrasion test blade and weigh to the nearest 0,1 mg. Record as m_f, in milligrams.

7.3.13 Calculate, a, the abrasion, expressed in milligrams per minute, as given in Equation (8):

$$a = \frac{m_b - m_f}{t} \tag{8}$$

where

m_b is the initial blade mass, expressed in milligrams;

m_f is the final blade mass, expressed in milligrams;

t is the time, expressed in minutes (20 min in this procedure).

7.3.14 The test precision for this procedure has been determined to be as follows:

$r = \pm 0{,}45$

$R = \pm 0{,}78$

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where

r is the within-laboratory repeatability, the maximum expected difference between two test results on the same sample by the same lab at the 95 % confidence level;

R is the between-laboratories reproducibility, the maximum expected difference between test results by two labs on the same sample at the 95 % confidence level.

Because this test is not an absolute measurement, test results should be evaluated cautiously. Best evaluations are obtained by comparing test results of a sample to test results of a weighting material of proven performance, rather than using the test-result value obtained as an absolute.

8 Mercury in drilling fluid barite

8.1 Principle

8.1.1 This method covers the determination of mercury (Hg) in drilling fluid barite. The cold-vapour atomic absorption technique is used for the analysis, following sample digestion and oxidation to ensure that most of the Hg in the sample is dissolved in the aqueous medium and converted to the mercuric ion.

8.1.2 The cold-vapour atomic absorption technique is based on the absorption of light energy at 253,7 nm by Hg vapour. Hg is reduced to the elemental state and purged from solution in a closed system. The Hg vapour passes through a cell positioned in the light path of an Hg source lamp. Absorbance (peak height) is measured as a function of Hg content.

8.2 Reagents and apparatus

8.2.1 **Distilled-deionized water**, in accordance with ISO 3696:1987, grade 2, or prepared by passing distilled water through a series of cation and anion exchange resins.

Use distilled-deionized water for preparation of all reagents and calibration standards and as dilution water.

NOTE Special reagents low in mercury are available for the chemicals described in 8.2.2 to 8.2.7.

8.2.2 **Hydrochloric acid** (HCl) (CAS number 7647-01-0), concentrated.

8.2.3 **Nitric acid** (HNO_3) (CAS number 7697-37-2), concentrated.

8.2.4 **Aqua regia**.

Prepare immediately before use by carefully adding three volumes of concentrated hydrochloric acid (HCl) to one volume of concentrated nitric acid (HNO_3).

8.2.5 **Hydrochloric acid**, c_{HCl} = 1,2 mol/l.

Add 100 ml of concentrated hydrochloric acid (HCl) to 500 ml of water in a 1 l volumetric flask. Dilute to 1 l with water.

8.2.6 **Stannous chloride** (CAS number 7772-99-8), solution.

Add 10 g stannous chloride ($SnCl_2$) to 50 ml of 1,2 mol/l hydrochloric acid (HCl) (8.2.5) in a 100 ml volumetric flask. Dilute to volume with 1,2 mol/l hydrochloric acid.

8.2.7 **Hydroxylamine hydrochloride** (CAS number 5470-11-1), solution.

Dissolve 12 g of hydroxylamine hydrochloride in water and dilute to 100 ml with distilled-deionized water.

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8.2.8 Potassium permanganate (CAS number 7722-64-7), 5 % solution.

Dissolve 5 g of potassium permanganate ($KMnO_4$) in 100 ml of water.

8.2.9 Potassium persulfate (CAS number 7727-21-1), solution.

Dissolve 50 g potassium persulfate ($K_2S_2O_8$) in water and dilute to 1 l with hot (60 °C) (140 °F) water.

8.2.10 Mercury, stock solution (1 ml solution = 1 mg Hg, which is equivalent to a 1 g/l Hg standard).

Dissolve 0,135 4 g of mercuric chloride in 75 ml of water. Add 10 ml of concentrated HNO_3 and adjust the volume to 100 ml.

NOTE Commercial Hg stock solutions are available and can be used as an alternative to preparing the stock solution.

8.2.11 Mercury, intermediate solution (1 ml solution = 10 µg Hg, which is equivalent to a 10 µg/ml or 10 mg/l Hg standard).

Pipette 1 ml Hg stock solution into a 100 ml volumetric flask and bring to volume with water containing 10 ml concentrated HNO_3 per litre. Solution is expected to be stable for several weeks.

Lower-concentration Hg standards (< 10 µg/ml) should be stored in glass to avoid losses/gains of Hg by exchange with the atmosphere.

8.2.12 Mercury, working solution (1 ml solution = 0,1 µg Hg, which is equivalent to 0,1 µg/ml or 0,1 mg/l Hg standard).

Pipette 1 ml of the Hg intermediate solution into a 100 ml volumetric flask and bring to volume with water containing 10 ml concentrated HNO_3 per litre.

8.2.13 Atomic absorption spectrophotometer.

Any atomic absorption unit equipped with background compensation and having an open sample presentation area in which to mount the absorption cell is suitable. Recommendations for instrument settings by the particular manufacturer should be followed.

Instruments designed specifically for the measurement of mercury using the cold-vapour technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

8.2.14 Mercury hollow-cathode or **electrode-discharge lamp**.

8.2.15 Recorder.

Any multi-range, variable-speed recorder compatible with the ultraviolet detection system is suitable.

8.2.16 Absorption cell.

Standard spectrophotometer cells 100 mm (4,0 in) long having quartz end windows may be used. The cell is attached to a burner for support and aligned in the light beam to give maximum transmittance.

NOTE 1 Suitable cells can be constructed from glass tubing with dimensions approximately D = 25 mm (1,0 in) × 100 mm (4,0 in) length with quartz windows 25 mm (1,0 in) in diameter × 1,6 mm (1/16 in) thickness cemented in place at the ends. Gas inlet and outlet ports [also of glass but D = 8 mm (3/16 in)] are attached approximately 13 mm (5/8 in) from each end.

NOTE 2 Longer cells [e.g. 300 mm (12,0 in)] are common to separable Hg systems and provide lower detection limits.

8.2.17 Gas source, nitrogen or argon.

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8.2.18 **Flow meter**, capable of measuring a gas flow rate of approximately 1 l/min.

8.2.19 **Aerator**, comprised of a straight glass frit having a coarse porosity.

Clear flexible plastic tubing is used for passage of the Hg vapour from the sample bottle to the absorption cell and return.

8.2.20 **Drying tube**, of diameter 150 mm (6 in) × 20 mm (3/4 in), containing 20 g of magnesium perchlorate with glass wool packed at each end.

8.2.21 **Reaction bottle**, 250 ml to 300 ml glass container, fitted with ground glass joint.

A gas-washing bottle best serves as a reaction bottle.

8.2.22 **Digestion vessel**, 250 ml flask with a ground-glass joint fitted with a water-cooled condenser.

8.2.23 **Filtration cell**, any apparatus capable of filtering the digested sample through filter paper number 40 or number 42 [7)].

8.3 Preparation of standards

NOTE Some mercury test apparatus requires standards with concentrations ten times higher.

8.3.1 **Hg standard**, 0,005 µg/ml: Place 5 ml of Hg working solution (8.2.12) in a 100 ml volumetric flask and dilute to the mark with 1,2 mol/l hydrochloric acid (HCl).

8.3.2 **Hg standard**, 0,010 µg/ml: Place 10 ml of Hg working solution in a 100 ml volumetric flask and dilute as in 8.3.1.

8.3.3 **Hg standard**, 0,020 µg/ml: Place 20 ml of Hg working solution in a 100 ml volumetric flask and dilute as in 8.3.1.

8.3.4 **Hg standard**, 0,050 µg/ml: Place 50 ml of Hg working solution in a 100 ml volumetric flask and dilute as in 8.3.1.

8.4 Sample digestion

8.4.1 Weigh a 2,0 g sample of drilling fluid barite and place in the 250 ml flask.

8.4.2 Add 40 ml aqua regia, 15 ml $KMnO_4$, and 8 ml $K_2S_2O_8$ to the flask and reflux for 1 h using a water-cooled condenser in a fume hood. Cool.

IMPORTANT — Take extreme care to prevent loss of mercury during the digestion step.

8.4.3 Add 6 ml of the hydroxylamine hydrochloride solution to reduce excess permanganate as evidenced by a loss of colour.

8.4.4 Allow solids to settle and filter through number 40 or number 42 filter paper into a 100 ml volumetric flask. Wash the digestion flask and residue several times onto the filter with water. Dilute to the mark with water.

8.4.5 Prepare a procedural blank by carrying out 8.4.2 through 8.4.4 without a sample.

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7) Whatman® filter paper number 40 or number 42 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.

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8.5 Check for recovery of Hg during digestion

8.5.1 Transfer a 10 ml aliquot of the working solution (8.2.12), containing 1,0 µg Hg, to one of the 250 ml digestion flasks.

8.5.2 Treat as in 8.4.2 through 8.4.4.

8.6 Analysis of standards and samples

CAUTION — Because of the toxic nature of mercury vapour, precautions should be taken to avoid its inhalation. Include a trap in the system to pass the vapour through an absorbing medium, such as equal volumes of 0,1 mol/l $KMnO_4$ and 10 % H_2SO_4.

8.6.1 Switch on the Hg system, adjust airflow and zero instrument according to manufacturer's specifications.

NOTE Each Hg analysis system has a slightly different physical arrangement and methodology. The system discussed below serves as one useful example of a single-pass arrangement.

8.6.2 Place 5 ml of stannous chloride solution in a reaction bottle containing 100 ml water and allow purge gas to pass through the aeration apparatus and the absorption cell until no absorbance signal is observed.

8.6.3 Divert gas flow with a two-way valve and place 1 ml of the 0,005 µg/ml Hg standard (8.3.1) in the reaction bottle, wait 1 min, and then let purge gas run until a peak is observed on the recorder and the signal subsequently returns to zero. Rinse flask with 1,2 mol/l hydrochloric acid and then water between analyses. Repeat with each standard prepared in 8.3.2 through 8.3.4.

NOTE With some Hg systems, adjust airflow and zero instrument according to the manufacturer's specification.

8.6.4 Repeat 8.6.2 and 8.6.3 for each sample, using 0,5 ml to 5 ml aliquots (from the 100 ml total).

NOTE With some Hg systems, the standard curve can use 0,05 µg, 0,1 µg, 0,2 µg, 0,5 µg and 1,0 µg Hg values.

8.6.5 Also analyse 1 ml of the sample prepared for Hg recovery check in 8.5. Absorbance for that sample shall be at least 95 % of that for the 0,010 µg/ml Hg standard (8.3.2).

8.7 Calculation

8.7.1 Following analysis of the standards, construct a standard curve by plotting peak height versus micrograms of mercury (0 µg, 0,005 µg, 0,01 µg, 0,02 µg, 0,05 µg, 0,10 µg).

NOTE With some Hg systems, the standard curve can use 0,05 µg, 0,1 µg, 0,2 µg, 0,5 µg and 1,0 µg Hg values.

8.7.2 Measure the peak height of the test sample and read its mercury value from the standard curve.

8.7.3 Calculate, w_{Hg}, the mercury mass fraction, expressed in micrograms per gram sample, using Equation (9).

$$w_{Hg} = \frac{m_{Hg} V_o}{m_o V_s} \tag{9}$$

where

m_{Hg} is the mass of mercury in the digested sample, expressed in micrograms;

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

V_o is the volume of digested solution, expressed in millilitres (100 ml in this procedure);

V_s is the sample aliquot, expressed in millilitres.

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9 Cadmium and lead in drilling fluid barite

9.1 Principle

9.1.1 This method covers the determination of cadmium (Cd) and lead (Pb) in drilling fluid barite. Atomic absorbtion (AA) is used for analysis, following sample digestion, to ensure that most of the Cd and Pb in the sample dissolves in the aqueous medium.

9.1.2 The aqueous sample containing the dissolved Cd and Pb ions is atomized and aspirated into a flame. During separate analyses, light beams of wavelength 228,8 nm for Cd and 283,3 nm for Pb are passed through the flame into a monochromator and onto a detector that measures the amount of light absorbed. The light energy absorbed in the flame is a measure of the concentrations of Cd and Pb in the sample.

9.2 Reagents and apparatus

9.2.1 **Distilled-deionized water**, in accordance with ISO 3696:1987, grade 2, or prepared by passing distilled water through a series of cation and anion exchange resins.

Use distilled-deionized water for the preparation of all reagents and calibration standards and as dilution water.

9.2.2 **Hydrochloric acid solution**.

Add one volume concentrated hydrochloric acid (HCl) (CAS number 7647-01-0) to one volume water (1:1).

9.2.3 **Nitric acid** (HNO_3) (CAS number 7697-37-2), concentrated, reagent grade.

9.2.4 **Cadmium nitrate** (CAS number 10022-68-1), for Cd stock solution (1 ml solution = 0,1 mg Cd, which is equivalent to a 100 µg/ml or 100 mg/l standard).

Weigh 0,274 4 g of cadmium nitrate [$Cd(NO_3)_2 \cdot 4H_2O$, analytical reagent grade], dissolve in 200 ml water in a 1 l volumetric flask. Add 20 ml hydrochloric acid solution (9.2.2) and dilute to mark with water.

9.2.5 **Lead nitrate** (CAS number 10099-74-8), for Pb stock solution (1 ml solution = 1 mg Pb, which is equivalent to a 1 mg/ml or 1 g/l standard).

Weigh 1,599 g [$Pb(NO_3)_2$, analytical reagent grade], dissolve in 200 ml of water, add 10 ml HNO_3 (concentrated) and dilute to 1 l with water.

NOTE Commercial Cd and Pb stock solutions are available and recommended as an alternative to preparing the stock solution. For Cd, the 100 µg/ml stock solution (9.2.4) can be prepared by placing 100 ml of the 1 µg/l standard in a 1 l volumetric flask, adding 20 ml of hydrochloric acid (9.2.2) and diluting to the mark with water.

9.2.6 **Atomic absorption spectrophotometer**.

Any commercial atomic absorption unit having an energy source, an atomizer burner system, a monochromator, a detector and background compensation is suitable. Recommendations for instrument settings by the particular manufacturer should be followed.

9.2.7 **Cd and Pb hollow-cathode or electrodeless discharge lamps**.

9.2.8 **Fuel**.

Commercial grade acetylene is generally acceptable.

9.2.9 **Oxidation air**, supplied from a compressed-air line, a laboratory compressor or from a cylinder of compressed air.

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9.2.10 **Digestion flask**, of capacity 250 ml, with a ground-glass joint, fitted with a water-cooled condenser.

9.2.11 **Filtration cell**.

Any apparatus capable of filtering the digested sample through number 40 or number 42 filter paper [8)] is suitable.

9.3 Preparation of combined cadmium and lead standards

9.3.1 **Standard**, 0,1 µg/ml Cd plus 1 µg/ml Pb: Transfer 1 ml aliquots each of the Cd and the Pb stock solutions to a 1 l volumetric flask, fill about half full with distilled-deionized water, add 10 ml hydrochloric acid (9.2.2) and dilute with water to 1 l. Store in acid-washed plastic bottles.

9.3.2 **Standard**, 0,2 µg/ml Cd plus 2 µg/ml Pb: Use 2 ml aliquots of each stock solution and repeat dilution as in 9.3.1.

9.3.3 **Standard**, 0,5 µg/ml Cd plus 5 µg/ml Pb: Use 5 ml aliquots of each stock solution and repeat dilution as in 9.3.1.

9.3.4 **Standard**, 1 µg/ml Cd plus 10 µg/ml Pb: Use 10 ml aliquots of each stock solution and repeat dilution as in 9.3.1.

9.3.5 **Standard**, 2 µg/ml Cd plus 20 µg/ml Pb: Use 20 ml aliquots of each stock solution and repeat dilution as in 9.3.1.

All standards should be stored in polyethylene bottles and it is expected that they are stable for several months.

9.3.6 Prepare an acid blank by the above procedure without adding Cd or Pb stock solution.

9.4 Sample digestion

9.4.1 Weigh out a 10 g or smaller sample of drilling fluid barite and place in the 250 ml flask.

9.4.2 Add 50 ml hydrochloric acid (9.2.2) to the flask and reflux for 1 h using the water-cooled condenser.

9.4.3 Allow flask and contents to cool.

9.4.4 Let solids settle and decant through a number 40 or number 42 filter, into a 100 ml volumetric flask. Wash residue and digestion flask with water, let settle and decant through filter. Dilute to the mark with water. Aliquots of the well-mixed, filtered digest solution may be stored in smaller plastic containers for several weeks.

9.4.5 Prepare a procedural blank by performing 9.4.1 through 9.4.4 without sample.

NOTE No losses of Cd or Pb were found during acid digestion.

9.5 Analysis of standards and samples

NOTE 1 One alternative procedure for determining Cd or Pb in the extract is the use of a graphite furnace, or flameless atomic absorption spectrophotometry. In this process, a heated graphite tube rapidly volatilizes the Cd- or Pb-containing solution; the vapour thus generated is analysed for the Cd or Pb content using an atomic absorption spectrophotometer.

NOTE 2 A second alternative method for determining Cd or Pb in the digest solution is the use of a plasma spectrophotometer, available as either DCP or ICP. These instruments aspirate the extract into a plasma where the solution is volatilized. The Cd or Pb atoms thus generated are excited to a high energy level by the plasma and then emit radiation that is characteristic of the atomic structure of Cd or Pb. This radiation is then separated from other radiation emitted by a monochromator and is measured quantitatively by a photomultiplier.

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9.5.1 Switch on the atomic absorption instrument and configure it for flame atomization for Cd at 228,8 nm according to the manufacturer's instructions.

9.5.2 Aspirate each of the Cd/Pb standards (9.3.1 through 9.3.5) into the instrument and record the absorbance and Cd concentration of each standard. Repeat until each of the standards gives a uniform absorbance. Aspirate water between the analyses of each different standard.

9.5.3 Proceed with the aspiration of acid (see 9.3.6), procedural blanks (see 9.4.5) and samples, recording the absorbances and again aspirating water between analyses of each sample.

9.5.4 Re-run the standards after every 6 to 10 samples and at the conclusion of the sample set.

9.5.5 Configure the instrument for flame atomization for Pb at 228,8 nm according to the manufacturer's instructions and repeat 9.5.2 through 9.5.4 for analysis of Pb.

9.6 Calculation

9.6.1 Prepare a separate calibration curve for Cd and for Pb by plotting the absorbance versus concentration for each standard.

9.6.2 Using the sample absorbance and the Cd and Pb calibration curves from 9.6.1, separately determine the digest solution Cd and Pb concentrations, subtracting any procedural blank (which is expected to be essentially zero).

9.6.3 Calculate the Cd and Pb mass fractions, w_{Cd} and w_{Pb}, respectively, expressed in micrograms per gram sample, from Equations (10) and (11).

$$w_{Cd} = \frac{\rho_{Cd} \cdot V_o}{m_o} \tag{10}$$

$$w_{Pb} = \frac{\rho_{Pb} \cdot V_o}{m_o} \tag{11}$$

where

ρ_{Cd} is the density of cadmium in the digested sample, expressed in micrograms per millilitre;

ρ_{Pb} is the density of lead in the digested sample, expressed in micrograms per millilitre;

V_o is the volume of solution, expressed in millilitres (100 ml in this procedure);

m_o is the sample mass, expressed in grams.

10 Arsenic in drilling fluid barite

10.1 Principle

10.1.1 This method covers the determination of arsenic (As) in drilling fluid barite. The gaseous hydride atomic absorption method is used for analysis, following sample digestion and reduction to ensure that most of the As in the sample is dissolved in the aqueous medium and converted to the trivalent form.

10.1.2 The gaseous hydride method is based on the absorption of light energy at 193,7 nm by As. Arsenic in the trivalent form is converted to gaseous arsine (AsH_3) using sodium borohydride ($NaBH_4$) in an acid medium. The gaseous hydride is swept through a heated quartz tube or into the argon/hydrogen flame of an atomic absorption spectrophotometer. The As concentration is determined as a function of the measured absorbance (peak height).

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NOTE An alternative procedure for determining As in the extract is the use of flameless atomic absorption spectrophotometry. In this process, a heated graphite tube rapidly volatilizes the As-containing solution; the vapour thus generated is measured for As using an atomic absorption spectrophotometer. This method of analysis eliminates the need for the arsine generator and is also more sensitive to As. Graphite furnaces are commercially available from most manufacturers of atomic absorption instruments. The manufacturer's recommendations for instrument settings for As should be followed. However, slight modifications in these settings can be needed to optimize the sensitivity and reproducibility of the As analysis.

10.2 Reagents and apparatus

10.2.1 Distilled-deionized water, in accordance with ISO 3696:1987, grade 2, or prepared by passing distilled water through a series of cation and anion exchange resins.

Use distilled-deionized water for the preparation of all reagents and calibration standards and as dilution water.

10.2.2 Nitric acid (HNO_3) (CAS number 7697-37-2), concentrated, redistilled, ρ = 1,42.

Use analytical grade with an arsenic content not greater than 10 µg/l As.

10.2.3 Nitric acid, c_{HNO3} = 3,2 mol/l.

Dilute 200 ml of concentrated HNO_3 to 1 l with water.

10.2.4 Potassium iodide (KI) (CAS number 7681-11-0), 150 g/l solution.

Dissolve 15 g of potassium iodide in 100 ml of water. Store in an amber bottle.

10.2.5 Potassium thiocyanate (KSCN) (CAS number 333-20-0), 50 g/l solution.

Dissolve 5 g of potassium thiocyanate in 100 ml of water.

10.2.6 Sodium borohydride (CAS number 16940-66-2), 0,8 cm (0,3 in) pellets weighing about 0,25 g each.

10.2.7 Sodium borohydride, solution.

Dissolve 30 g $NaBH_4$ in a 1 % NaOH solution and dilute to 1 litre in a volumetric flask.

10.2.8 Arsenic(III) oxide (As_2O_3) (CAS number 1327-53-3), for As stock solution (1 ml solution = 1 mg As, which is equivalent to 1 mg/ml or 1 g/l As).

Dissolve 1,320 g of arsenic(III) oxide in 100 ml water containing 4 g NaOH and dilute to 1 litre with water.

Commercial As stock solutions are available and recommended as an alternative to preparing the stock solution.

10.2.9 Arsenic(III) oxide (10.2.8), for As intermediate solution (1 ml solution = 10 µg As, which is equal to 10 µg/ml or 10 mg/l As).

Pipette 1 ml of the arsenic stock solution into a 100 ml volumetric flask and bring to volume with 3,2 mol/l HNO_3.

10.2.10 Arsenic(III) oxide (10.2.8), for As working solution (1 ml solution = 1 µg As, which is equivalent to 1 µg/ml As).

Pipette 10 ml intermediate arsenic solution into a 100 ml volumetric flask and bring to volume with 3,2 mol/l HNO_3.

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10.2.11 Atomic absorption spectrophotometer.

Any commercial atomic absorption unit having an energy source, a heated quartz tube (preferred) or an atomizer burner system, a monochromator, a detector and background compensation is suitable. The manufacturer's recommendations for instrument settings for As should be followed.

NOTE With the heated quartz tube hydride generation system, experimental results indicate that a background correction is not required.

10.2.12 Arsenic hollow-cathode or electrodeless discharge lamp.

10.2.13 Arsine generator, comprised of a reaction flask of about 125 ml capacity fitted with inlet-outlet tubes for argon flow through the solution and a device for adding sodium borohydride in a closed system.

NOTE Arsine generators are available commercially from instrument manufacturers and can have configurations slightly different from that described above.

10.2.14 Digestion vessel, e.g. a PTFE-lined digestion bomb of capacity 25 ml.

Such bombs, which are commercially available from several manufacturers, shall be leakproof and airtight when sealed, and shall be capable of withstanding operating temperatures of at least 110 °C (230 °F) and pressures of at least 1 380 kPa (200 psi). Typical capacities are in the range of 21 ml to 25 ml.

10.2.15 Heating apparatus, e.g. a **water bath**, controllable to 100 °C ± 1 °C (212 °F ± 2 °F).

10.2.16 Filtration cell.

Any apparatus capable of filtering the digested sample through number 40 or number 42 filter paper [8)].

10.3 Preparation of standards

10.3.1 As standard, 0,025 µg/ml: Transfer 2,5 ml of the As working solution (10.2.10) to a 100 ml volumetric flask and bring to volume with 3,2 mol/l HNO_3.

10.3.2 As standard, 0,05 µg/ml: Use 5 ml of As working solution and dilute as in 10.3.1.

10.3.3 As standard, 0,075 µg/ml: Use 7,5 ml of As working solution and dilute as in 10.3.1.

10.3.4 As standard, 0,10 µg/ml: Use 10 ml of As working solution and dilute as in 10.3.1.

10.4 Sample digestion

10.4.1 Weigh a 250 mg sample of drilling fluid barite and place in the 25 ml PTFE bomb. Add 10 ml concentrated HNO_3, tightly stopper the bomb, and heat for 1,5 h in the water bath at 80 °C (175 °F).

10.4.2 Cool for 1 h at ambient temperature.

10.4.3 Reheat for 1,5 h at 80 °C (175 °F).

10.4.4 Cool to ambient temperature.

10.4.5 Remove the cover carefully and decant into a 50 ml beaker.

10.4.6 Rinse the bomb and lid several times with water and add rinse to the beaker.

10.4.7 Add water to the beaker to bring volume to about 40 ml.

10.4.8 Filter through a number 40 or number 42 filter paper into a 50 ml volumetric flask. Wash the beaker with small portions of water into the filter. Bring flask to volume with water.

10.4.9 To obtain a procedural blank, repeat 10.4.1 to 10.4.8 without a sample.

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10.5 Analysis of standards and samples

10.5.1 Switch on the atomic absorption instrument and configure according to the manufacturer's directions.

10.5.2 For each analysis, add 1 ml aliquots of the standard solutions (see 10.3) and blanks (see 10.4.9) to the arsine generator. Add 2 ml of the potassium thiocyanate (KSCN) solution, 2 ml of the KI solution and 15 ml of 3,2 mol/l HNO_3.

NOTE Aliquots larger than 1 ml can be required with some systems.

10.5.3 Allow 10 min for the As to be reduced to the trivalent state.

10.5.4 Connect the generator and add 1 $NaBH_4$ pellet or pump $NaBH_4$ solution until the maximum signal is obtained according to the manufacturer's specifications. Record the peak height. When the recorder returns to the baseline, disconnect the generator.

10.5.5 Prepare a standard curve by plotting peak height versus micrograms As for each standard.

10.5.6 Run sample solutions using 0,5 ml to 5 ml or more (from the total of 100 ml) in the same manner as for the standards (see 10.5.2 through 10.5.4).

NOTE To minimize As absorption losses on glassware, run the analysis on the standards and sample immediately upon preparation.

10.6 Calculation

10.6.1 Determine the mass of As in the digested sample from the calibration curve prepared in 10.5.5.

10.6.2 Calculate, w_{As}, the mass fraction of As, expressed in micrograms per gram sample, from Equation (12):

$$w_{As} = \frac{m_{As} V_o}{m_o V_s} \tag{12}$$

where

m_{As} is the mass of arsenic in the digested sample, expressed in micrograms;

V_o is the volume of solution, expressed in millilitres (50 ml in this procedure);

m_o is the sample mass, expressed in grams;

V_s is the sample volume added to the generator, expressed in millilitres.

11 Bridging materials for regaining circulation

11.1 Principle

The ability of a material to seal a lost-circulation zone is of importance in the determination of the proper material. The size of the opening being sealed or bridged varies with formation, and it is necessary that the particles of sealing material be sized to adequately bridge that opening. The objective of this testing is to determine the necessary size and concentration of drilling fluid materials, commonly referred to as lost-circulation materials, that can bridge the permeable slots or beds described in 11.4 to 11.9 and prevent further loss of drilling fluid from the cell. Depending on the physical characteristics of the formation drilled, test results can suggest that one or more of the lost-circulation materials tested can reduce losses to the drilled formation. Cells for testing bridging materials are available from suppliers of drilling fluid testing equipment, and the manufacturer's instructions for assembly and testing should be followed. General guidelines for testing can be found in 11.4 to 11.9.

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11.2 Apparatus

11.2.1 **Cell for testing bridging materials**, fitted with the following:

11.2.1.1 **Stainless steel disks**, 6,4 mm (0,25 in) thick and 47,5 mm (1-7/8 in) in diameter.

The disks each have one square-edged slot, 35,05 mm (1-3/8 in) in length and 1 mm (0,04 in), 2 mm (0,08 in), 3 mm (0,12 in), 4 mm (0,16 in) or 5 mm (0,20 in) in width.

11.2.1.2 **Sleeve**, 73 mm (2-7/8 in) in diameter and 57 mm (2-1/4 in) high, with a perforated baseplate containing approximately thirty-two 6,4 mm (1/4 in) holes.

11.2.1.3 **Brass** or **stainless-steel marbles**, minimum number of 95, 14,3 mm (9/16 in) in diameter (enough to just fill a bed volume).

11.2.1.4 **Brass-clad** or **stainless-steel ball bearings (BB shot)**, 1 200 g, 4,4 mm (0,17 in) in diameter.

11.2.1.5 **Stainless steel screen**, 2 000 µm mesh, 73 mm (2-3/4 in) in diameter.

11.2.2 **Nitrogen**, pressure-regulated.

11.2.3 **Plastic container**, 3,5 l capacity, fitted with an inlet and an outlet suitable to accommodate the sudden discharge of drilling fluid from the cell.

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

The impeller shall be replaced when a mass loss of approximately 10 % occurs. The original blade mass is about 5,5 g.

11.2.5 **Container**, for mixing, 180 mm (7-1/8 in) deep, d = 97 mm (3-3/4 in) at the top and 70 mm (2-3/4 in) at the bottom [5)].

11.2.6 **Base drilling fluid**, consisting of 5 % to 8 % mass fraction Wyoming bentonite, aged for a minimum of 72 h and adjusted to an apparent viscosity of 0,025 Pa·s ± 0,002 Pa·s after stirring for 10 min on a mixer.

11.3 Preparation of test drilling fluid

To 3,5 l (10 laboratory barrels) of base drilling fluid (11.2.6), add a weighed amount of the material being tested. The concentration of the test material in the drilling fluid is expressed in kilograms per cubic metre (pounds per barrel).

11.4 Static slot test

11.4.1 Remove the perforated plate and sleeve used to support the BB or marble beds from the cell; select a disk, preferably having a small slot, and place it in the valve outlet half-union.

11.4.2 Open the cylinder bleed valve and place the graduated container under the outlet.

11.4.3 Pour the drilling fluid containing the material being tested into the cell with cell outlet valve open. Record the volume of drilling fluid which flows out.

11.4.4 Screw the cap onto the cell. The free piston may be placed on the drilling fluid in the cell, if desired.

11.4.5 Start the timer and apply pressure at a rate of 13,8 kPa/s (2 psi/s), until a pressure of 690 kPa (100 psi) is reached. Record the volume of drilling fluid discharged. The minimum pressure at which a seal occurs might or might not be observed. If observed, it shall be recorded.

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