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Petroleum and natural gas industries — Drilling fluids — Laboratory testing

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

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

Introduction

This International Standard, which establishes testing methodologies for drilling fluid materials, is based on API RP 13I, seventh edition/ISO 10416:2002 [2]. This International Standard was developed in response to a demand for more exacting testing methodologies. The tests contained herein were developed over several years by a group of industry experts and were identified as being those which can yield reproducible and accurate results. The tests are anticipated to be performed in a laboratory setting, but can be applicable in a field situation with more rigorous apparatus and conditions than normally found in a drilling fluid field-test kit.

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

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

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

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

Petroleum and natural gas industries — Drilling fluids — Laboratory testing

1 Scope

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

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

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 91-1:1992, *Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 °C and 60 °F*

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

ISO 2977:1997, *Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

ASTM E 100, *Standard Specification for ASTM Hydrometers*

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

3 Terms and definitions

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

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

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

NOTE 2 See Clause 15.

3.3
darcy
k
permeability of a porous medium, where one darcy is the flow of a single-phase fluid of 1 cP viscosity that completely fills the voids of the porous medium, flowing through the medium under conditions of viscous flow at a rate of 1 $\text{cm}^3 \cdot \text{s}^{-1} \bullet \text{cm}^{-2}$ cross-sectional area, and under a pressure or equivalent hydraulic gradient of 1 $\text{atm} \bullet \text{cm}^{-1}$

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

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

4 Symbols and abbreviations

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

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

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

5 Barite

5.1 Principle

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

5.2 Reagents and apparatus

5.2.1 Dispersant solution.

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

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

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

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

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

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

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

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

5.2.9 Balance, accuracy ± 0,01 g.

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

5.2.11 Beaker, 250 ml.

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

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

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

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3) Multimixer Model 9B with B29 impeller is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

4) Hamilton Beach Mixer Cup No. M110-D is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

5.2.15 **Dishes**, evaporating.

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

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

5.2.18 **Spray nozzle** [5].

5.3 Sampling

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

5.4 Calculation of moisture content

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

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

5.4.3 Cool the sample in a desiccator and weigh.

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

$$w_\mathrm{h} = 100 \frac{m_\mathrm{o} - m_\mathrm{d}}{m_\mathrm{o}} \tag{1}$$

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.

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)];

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

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:

$$\eta = \frac{1}{0{,}021482\left[\left(\theta - 8{,}435\right) + \sqrt{8\,078{,}4 + (\theta - 8{,}435)^2}\right] - 1{,}2}$$

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

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

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

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.

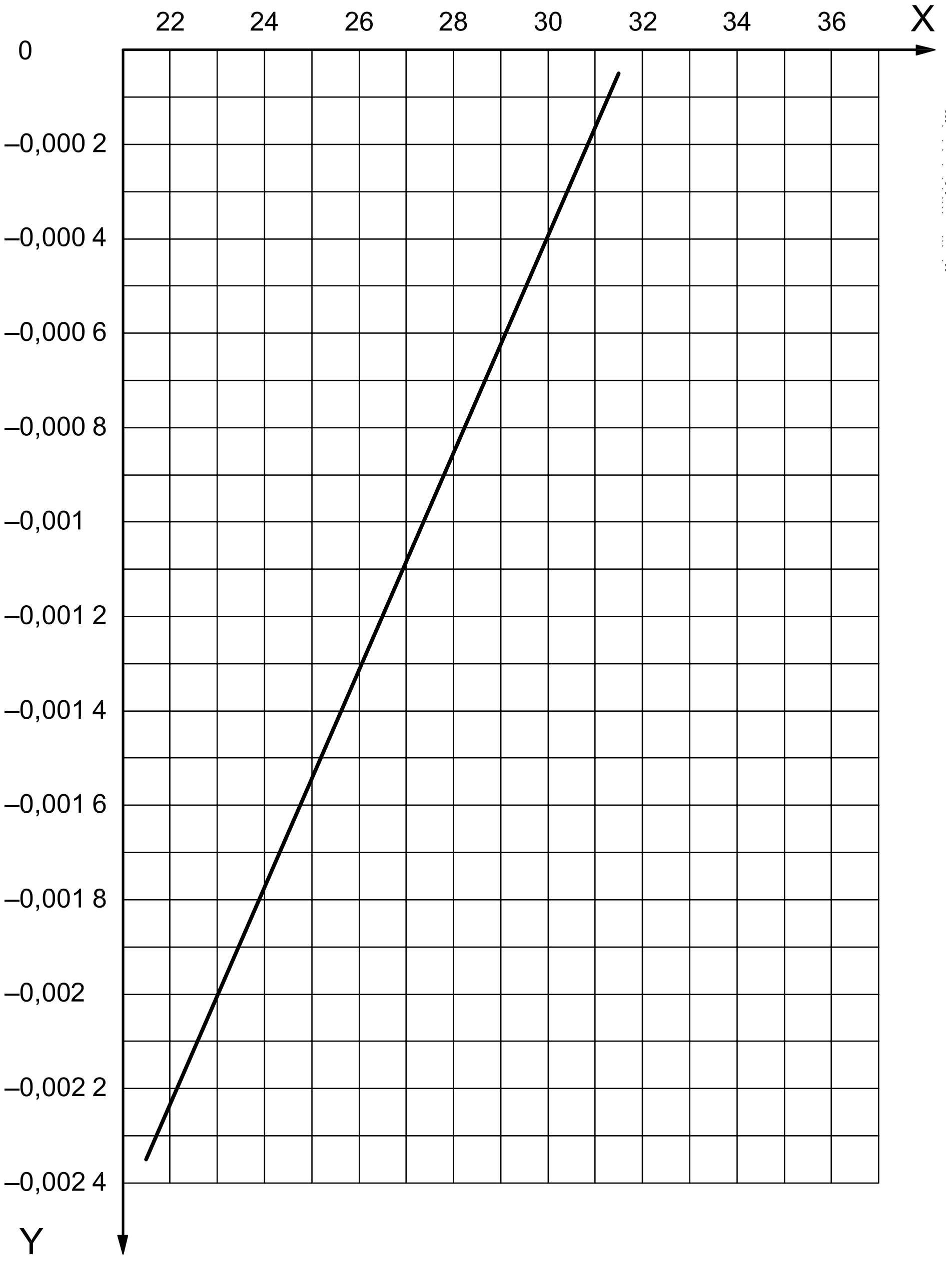
b) Hydrometers are calibrated at 20 °C (68 °F), and deviations from this standard temperature produce inaccuracies in the actual hydrometer reading. The amount of inaccuracy increases as the deviation from standard temperature increases.

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.

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

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.

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 diameter4).

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.

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):

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

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.

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.

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):

$$a = \frac{m_\mathrm{b} - m_\mathrm{f}}{t} \tag{8}$$

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

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.

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.

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.

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).

$$w_{\text{Hg}} = \frac{m_{\text{Hg}} V_{\text{o}}}{m_{\text{o}} V_{\text{s}}} \tag{9}$$

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.

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.

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.

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).

$$w_{\text{Cd}} = \frac{\rho_{\text{Cd}} \cdot V_{\text{o}}}{m_{\text{o}}} \tag{10}$$

$$w_{\text{Pb}} = \frac{\rho_{\text{Pb}} \cdot V_{\text{o}}}{m_{\text{o}}} \tag{11}$$

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).

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, $\rho = 1{,}42$.

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

10.2.3 Nitric acid, $c_{\text{HNO3}} = 3{,}2$ 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.

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.

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):

$$w_{\mathrm{As}} = \frac{m_{\mathrm{As}} V_{\mathrm{o}}}{m_{\mathrm{o}} V_{\mathrm{s}}} \tag{12}$$

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.

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.

11.4.6 Increase pressure at a rate of 69 kPa/s (10 psi/s) to 6 900 kPa (1 000 psi), or until the seal fails and the cylinder empties. Record the volume of drilling fluid discharged or the maximum pressure obtained. If a seal is obtained, maintain the pressure for 10 min and record the final volume of drilling fluid discharged.

11.4.7 Repeat the test using disks with increasing sizes of slot until no permanent seal is achieved at 6 900 kPa (1 000 psi).

11.5 Dynamic slot test

11.5.1 Prepare the test drilling fluid in accordance with 11.3 and the apparatus as outlined in 11.4.1.

11.5.2 With the cell outlet valve closed, pour the test drilling fluid into the cell. The free piston may be placed on top of the drilling fluid.

11.5.3 Close the cap and set the gas regulator to deliver nitrogen at the test pressure of 690 kPa (100 psi).

11.5.4 Open the cell outlet valve and start the timer. Record the volume of drilling fluid that flows through the slot until it is sealed, and the time required to seal.

11.5.5 Increase pressure to 6 900 kPa (1 000 psi) at the rate of 69 kPa/s (10 psi/s) and maintain for 10 min, as in 11.4.6.

11.5.6 Repeat the test using disks with increasing sizes of slot until no permanent seal is achieved.

11.6 Static marble bed test

11.6.1 Remount the perforated plate and prepare a marble bed by pouring the 14,3 mm (9/16 in) marbles into the sleeve so as to form a bed 57 mm (2-1/4 in) thick above the perforated plate (just to the top of the container). Place the full-bore ring in the slot groove.

11.6.2 With the cell outlet valve open and the graduated container under the outlet, pour the test drilling fluid into the cell. Record the volume of fluid passing through the bed under the hydrostatic head.

11.6.3 Place the free piston on top of the drilling fluid and the cap on the cell. Close the cylinder bleed valve and start the timer. Apply pressure and record the results as in 11.4.5 and 11.4.6.

11.6.4 On completion of the test, release the pressure. Remove the marble bed and examine the appearance of the seal and the depth of penetration of the sealing material.

11.7 Dynamic marble bed test

11.7.1 Prepare the marble bed as in 11.6.1.

11.7.2 With the cell outlet valve closed, pour the test drilling fluid into the cell to fill the void spaces under and within the marble bed until the top of the test drilling fluid is level with the top of the sleeve.

11.7.3 Carefully pour the test drilling fluid into the cell so as not to disturb the drilling fluid in the bed. The free piston may be placed on top of the drilling fluid.

11.7.4 Close the cell and apply pressure with the gas regulator adjusted to 6 900 kPa (1 000 psi). Open the cell outlet valve and start the timer. Record the volume of drilling fluid that flows through the bed and the time to obtain a seal.

11.7.5 Continue the test as in 11.4.6. After 10 min at 6 900 kPa (1 000 psi) or after failure, inspect the bed as in 11.6.4.

11.8 Static ball bearings (BB shot) bed test

11.8.1 Prepare the BB shot bed by placing the 2 000 µm (10 mesh) stainless-steel screen on the perforated plate and pouring the brass-clad or stainless-steel BB shot into the sleeve so as to form a bed above the screen (just to the top of sleeve). The thickness of the shot bed can be varied from 25 mm to 57 mm (1,0 in to 2-1/4 in). If less than 57 mm (2-1/4 in), record the thickness.

11.8.2 Place the full-bore ring in the slot groove. With the cell outlet valve open and the graduated cylinder under the outlet, pour the test drilling fluid into the cell.

11.8.3 Proceed with the test according to 11.6.2 through 11.6.4.

11.9 Dynamic ball bearings (BB shot) bed test

11.9.1 Prepare the BB shot bed as in 11.8.1.

11.9.2 With the cell outlet valve closed, pour the test drilling fluid into the cell to fill the void spaces under and within the shot bed, until the top of the test drilling fluid is level with the top of the sleeve.

11.9.3 Proceed with the test according to 11.7.3 through 11.7.5.

12 Filtration-control agents

12.1 Principle

12.1.1 Organic filtration-control agents can vary greatly in chemical composition and performance characteristics in different drilling fluid environments. Judgements are made by comparing the performance of test filtration control materials to that of known materials that have been accepted for field use.

12.1.2 In specific cases, comparisons may be made in the actual field drilling fluid in which the material will be used, but in general, evaluations are performed in laboratory-prepared drilling fluids.

12.1.3 It is recommended that filtration-control materials considered for broad applications be screened in different drilling fluid systems. While filtration control is the property being evaluated, the effect of the test material on rheology, pH or other properties can be of concern.

12.2 Reagents and apparatus

12.2.1 Reagents

12.2.1.1 **Calcium chloride** (CAS number 10043-52-4), ACS reagent grade.

12.2.1.2 **Magnesium chloride** (CAS number 7786 30-3), ACS reagent grade.

12.2.1.3 **Potassium chloride** (CAS number 7447-40-7), ACS reagent grade.

12.2.1.4 **Xanthan gum**, drilling fluid grade dry powder.

12.2.1.5 **Attapulgite**, ISO 13500 specification material.

12.2.1.6 Simulated drilled solids, for example Rev-Dust 8), ball clay 9) or clay dust 10).

12.2.1.7 API reference bentonite. If fluid loss properties are not obtained within the specified range, replace the reference bentonite.

12.2.1.8 Salt, stock solution No. 1, in accordance with ASTM D 1141, 555,6 g magnesium chloride hexahydrate (CAS number 7791-18-6), 57,9 g anhydrous calcium chloride (CAS number 10043-52-4) and 2,1 g strontium chloride hexahydrate (CAS number 10025-70-4) diluted to 1 l with distilled or deionized water.

12.2.1.9 Salt, stock solution No. 2, in accordance with ASTM D 1141, 69,5 g potassium chloride (CAS number 7447-40-7), 20,1 g sodium bicarbonate (CAS number 144-55-8), 10,0 g potassium bromide (CAS number 7758-02-3), 2,7 g boric acid (CAS number 10043-35-3) and 0,3 g sodium fluoride (CAS number 7681-49-4), diluted to 1 l with distilled or deionized water.

12.2.1.10 Sodium hydroxide (CAS number 1310-73-2), standardized solution, $c_{\text{NaOH}} = 10$ mol/l.

12.2.1.11 Lignosulfonate (chrome or **ferrochrome)**, drilling fluid grade.

12.2.1.12 Lime, ACS reagent grade or equivalent.

12.2.1.13 Sodium chloride (CAS number 7647-14-5), ACS reagent grade.

12.2.1.14 Sodium sulfate, anhydrous (CAS number 7757-82-6), ACS reagent grade.

12.2.1.15 Deionized or **distilled water**.

12.2.1.16 Defoamer.

12.2.2 Apparatus.

12.2.2.1 Balance, accuracy ± 0,01 g.

12.2.2.2 Mixer, capable of operation at 11 500 r/min ± 300 r/min under load, with a single, corrugated impeller approximately 25,4 mm (1,0 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.

12.2.2.3 Container, 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).

12.2.2.4 Clock or **timer**, direct-reading counter with an accuracy of 0,1 min over the test interval.

12.2.2.5 Carboy, covered, approximately 4 l capacity, with wide-mouth screw-on lid.

12.2.2.6 Spatula.

12.2.2.7 Filter press, low-temperature/low-pressure, in accordance with ISO 10414-1:2008, Clause 7, having a filter area of 4 520 mm^2 to 4 640 mm^2 (diameter of 75,86 mm to 76,86 mm) [filter area of 7,00 in^2 to 7,20 in^2 (diameter of 2,98 in to 3,03 in)].

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8) Rev-Dust is an example(s) of a suitable product available commercially from The Milwhite Company, 7050 Portwest, Houston, TX 77024, U.S.A. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

9) Martin's number 5 Ball Clay is an example of a suitable product(s) available commercially from Kentucky-Tennessee Clay Company, Box 449, Mayfield, Tennessee 42066, U.S.A. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

10) Standard base evaluation clay dust is an example of a suitable product available commercially. Requests for clay should be directed to the API which will forward the request to a supplier for all further handling. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

The filter-press gasket is the determining factor for the filter area. It is recommended that the filter-press gasket used be tested by a conical gauge having the maximum [76,86 mm (3,02 in)] and the minimum [75,86 mm (2,98 in)] allowed diameter dimensions marked on it. Any filter press gasket diameter measuring outside this range should be discarded.

12.2.2.8 Viscometer, direct-reading, in accordance with ISO 10414-1:2008, Clause 6.

12.2.2.9 Roller oven, regulated to maintain a temperature of 65 °C ± 3 °C (150 °F ± 5 °F).

12.2.2.10 pH meter, in accordance with ISO 10414-1:2008, Clause 11.

12.2.2.11 Jar, small, covered, e.g. 0,5 l glass jar with screw-type lid.

12.2.2.12 Oven, regulated to maintain a temperature of 105 °C ± 3 °C (220 °F ± 5 °F).

12.2.2.13 Ageing cells, in accordance with Clause 21.

12.2.2.14 Filter cell, high-temperature/high-pressure, in accordance with ISO 10414-1:2008, Clause 7.

12.2.2.15 Syringes, disposable, 3 ml capacity.

12.3 General instructions for preparation of base drilling fluids

12.3.1 Prepare a sufficient quantity of base drilling fluid for a series of tests. This can be achieved by preparing a large batch or by combining small batches for uniformity prior to ageing.

12.3.2 Composition, order of addition, mixing time, shear and ageing time shall be consistent from batch to batch.

12.3.3 Stir all base drilling fluids prior to testing to insure uniformity.

12.3.4 Test a sample of the untreated base drilling fluid each time a new series of tests is begun. Subject a sample of the untreated base drilling fluid to the same amount of stirring, heat ageing and testing as the treated drilling fluids.

12.4 Salt-saturated drilling fluid

12.4.1 Prepare saturated salt water by adding 400 g of sodium chloride and diluting to 1 l with water (to exceed the solubility of sodium chloride in water at room temperature).

12.4.2 Mix for 30 min, age overnight at room temperature and decant into a large covered carboy (approximately 4 l).

12.4.3 Prepare a suspension of attapulgite clay and simulated drilled solids using 8 g of attapulgite and 30 g of simulated drilled solids in 338 ml (401 g) saturated salt water in a container. Sift the clay and solids into the water while stirring with the mixer set at high shear.

12.4.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.4.5 Replace on the mixer and continue stirring for an additional 25 min (for a total mixing time of 30 min).

12.4.6 Store the suspension for 24 h in a sealed carboy at room temperature.

12.4.7 Stir the suspension for 5 min on the mixer.

12.4.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 90 ml to 105 ml.

12.4.9 For the purpose of testing, the mass equivalent is 439 g per 350 ml.

12.5 High-hardness, salt-saturated drilling fluid

12.5.1 Prepare a brine with a high hardness by adding 125 g calcium chloride and 22 g magnesium chloride and diluting to 1 l with the saturated salt solution, as prepared in 12.4.1.

12.5.2 Mix for 30 min, age overnight at room temperature and decant into a large covered carboy.

12.5.3 Prepare a suspension of attapulgite clay and simulated drilled solids using 8 g of attapulgite and 30 g of simulated drilled solids in 338 ml (429 g) of high-hardness, salt-saturated drilling fluid in a container. Sift the clay and solids into the water while stirring with the mixer set at high shear.

12.5.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.5.5 Replace on the mixer and continue stirring for an additional 25 min (for a total mixing time of 30 min).

12.5.6 Store the suspension for 24 h in a sealed carboy at room temperature.

12.5.7 Stir the suspension for 5 min on the mixer.

12.5.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 90 ml to 105 ml.

12.5.9 For the purposes of testing, the mass equivalent is 467 g per 350 ml.

12.6 10 % potassium chloride (KCl) drilling fluid

12.6.1 Prepare a 10 % potassium chloride (KCl) solution by adding 111 g of KCl to a container and diluting with water to 1 l.

12.6.2 Sift 1,0 g of Xanthan gum slowly into 360 g (340 ml) of the 10 % KCl solution while stirring on a mixer set at high shear.

12.6.3 After 5 min, remove the container from the mixer and scrape the sides to dislodge any polymer adhering to the container.

12.6.4 Replace on the mixer and continue stirring for an additional 10 min (total mixing time 15 min).

12.6.5 Add 30 g of simulated drilled solids while continuing to stir with the mixer set at high shear.

12.6.6 After 5 min, remove the container from the mixer and scrape the sides to dislodge any solids adhering to the container.

12.6.7 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 30 min).

12.6.8 Measure the rheological properties using the rheometer, in accordance with ISO 10414-1:2008, Clause 6.

Properties shall be as follows:

- plastic viscosity 0,003 Pa•s to 0,007 Pa•s (3 cP to 7 cP);
- yield point 2,9 Pa to 4,8 Pa (6 lbs/100 ft^2 to 10 lbs/100 ft^2);
- 10 s gel 0,96 Pa to 1,92 P (2 lbs/100 ft^2 to 4 lbs/100 ft^2);
- 10 min gel 1,44 Pa to 2,39 Pa (3 lbs/100 ft^2 to 5 lbs/100 ft^2).

12.6.9 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 30 ml to 50 ml.

12.6.10 For the purpose of testing, the mass equivalent is 391 g per 350 ml.

12.7 Pre-hydrated bentonite slurry

12.7.1 For procedures requiring pre-hydrated bentonite in a base drilling fluid, prepare and age the bentonite in advance.

12.7.2 Prepare a suspension of the bentonite using 26 g bentonite (as received) in 350 ml water (6,67 % mass fraction solids) in a container for each sample being tested. Sift the clay into the water while stirring with the mixer at high speed.

12.7.3 After 5 min, remove the container from the mixer and scrape the sides to dislodge any bentonite adhering to the container.

12.7.4 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

12.7.5 Store the suspension overnight in a sealed carboy at room temperature.

12.7.6 Stir the suspension for 5 min on a mixer before using.

12.8 Modified seawater drilling fluid

12.8.1 Prepare simulated seawater by adding 24,53 g sodium chloride and 4,09 g anhydrous sodium sulfate to a 1 l flask. Dilute to 800 ml with distilled or deionized water. Pour 20,0 ml stock salt solution No. 1 and 10,0 ml of stock salt solution No. 2 into the flask and dilute to 1 l with water while stirring. Adjust pH to 8,2 with 0,1 mol/l sodium hydroxide solution.

12.8.2 Prepare a suspension of bentonite clay using 150 g (144 ml) of pre-hydrated bentonite slurry in a container, as prepared in 12.7. Add 193 ml of simulated seawater, as prepared in 12.8.1, while stirring on the mixer.

12.8.3 Continue stirring while adding 2,5 ml of 10 mol/l NaOH and sifting in 30 g of simulated drilled solids.

12.8.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.8.5 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

12.8.6 Store the suspension for 24 h in a sealed carboy at room temperature.

12.8.7 Stir the suspension for 5 min on the mixer.

12.8.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 60 ml to 70 ml.

12.8.9 For the purpose of testing, the mass equivalent is 382 g per 350 ml fluid.

12.9 Low-salinity drilling fluid

12.9.1 Prepare a low-salinity solution by weighing 42 g sodium chloride into a flask and diluting to 1 l with water, resulting in a 4 % sodium chloride brine.

12.9.2 Prepare a suspension of bentonite clay using 150 g (144 ml) of pre-hydrated bentonite slurry in a container, as prepared in 12.7. Add 193 ml of low-salinity water to the suspension of bentonite while stirring with the mixer set on high shear.

12.9.3 Continue stirring while adding 2,5 ml of 10 mol/l NaOH and sifting in 30 g of simulated drilled solids.

12.9.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.9.5 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

12.9.6 Store the suspension for 24 h in a sealed carboy at room temperature.

12.9.7 Stir the suspension for 5 min on the mixer.

12.9.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 35 ml to 45 ml.

12.9.9 For the purpose of testing, the mass equivalent is 382 g per 350 ml fluid.

12.10 Lime-treated drilling fluid

12.10.1 Prepare a suspension of bentonite clay using 260 g (250 ml) of pre-hydrated bentonite slurry in a container, as prepared in 12.7. Add 73 ml of water, 4 g of chrome lignosulfonate and 2,5 ml of 10 mol/l NaOH while stirring on the mixer set at high speed.

12.10.2 Stir for 30 min.

12.10.3 Continue stirring and sift in 3 g of lime and 30 g of simulated drilled solids.

12.10.4 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.10.5 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 45 min).

12.10.6 Roll the suspension for 16 h in a sealed container in an oven set at 65 °C (150 °F).

12.10.7 Cool and stir the suspension for 5 min on the mixer.

12.10.8 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 12 ml to 20 ml.

12.10.9 Measure the phenolphthalein alkalinity, P_{df}, of the drilling fluid using the procedure in ISO 10414-1:2008, Clause 12. The P_{df} of the drilling fluid shall be 10 ml to 20 ml; adjust with lime.

12.10.10 For the purpose of testing, the mass equivalent is 373 g per 350 ml fluid.

12.11 Low solids, non-dispersed drilling fluid

12.11.1 Prepare a suspension of bentonite clay using 150 g (144 ml) of pre-hydrated bentonite slurry in a container, as prepared in 12.7. Add 200 ml of water and 15 g of simulated drilled solids while stirring with the mixer set at high shear.

12.11.2 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.11.3 Replace the container on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

12.11.4 Store the suspension for 24 h in a sealed carboy at room temperature.

12.11.5 Stir the suspension for 5 min on the mixer.

12.11.6 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 20 ml to 30 ml.

12.11.7 For the purpose of testing, the mass equivalent is 365 g per 350 ml fluid.

12.12 Freshwater lignosulfonate drilling fluid

12.12.1 Prepare a suspension of bentonite clay using 225 g (216 ml) of pre-hydrated bentonite slurry in a container, as prepared in 12.7. Add 127 ml of water, 15 g of simulated drilled solids, 1 g of chrome lignosulfonate and 1,2 ml of 10 mol/l NaOH while stirring with the mixer set at high speed.

12.12.2 After 5 min, remove the container from the mixer and scrape the sides to dislodge any clay and solids adhering to the container.

12.12.3 Replace on the mixer and continue stirring for an additional 10 min (for a total mixing time of 15 min).

12.12.4 Store the suspension for 24 h in a sealed carboy at room temperature.

12.12.5 Stir the suspension for 5 min on the mixer.

12.12.6 Measure the filtrate volume using a low-temperature/low-pressure filtrate cell, in accordance with ISO 10414-1:2008, Clause 7. The filtrate volume shall be 9 ml to 15 ml.

12.12.7 Measure the pH of the suspension using a pH-meter or strips, in accordance with ISO 10414-1:2008, Clause 11. The pH shall be 9,5 to 10,5 — adjust with NaOH.

12.12.8 For the purpose of testing, the mass equivalent is 369 g per 350 ml fluid.

12.13 Initial performance test

12.13.1 Select the concentrations of filtration control agent to cover a wide range (e.g. 0,1 g/350 ml, 0,5 g/350 ml, 1,0 g/350 ml, 3,0 g/350 ml and 5,0 g/350 ml). Include an untreated sample of the base drilling fluid.

If comparison to a known field-accepted material is being made, performance tests should be run on an equivalent range of known field-accepted material concentrations.

12.13.2 Weigh into each of several mixing containers the mass equivalent of 350 ml of the desired base drilling fluid. Place the containers on the mixer set at high speed.

12.13.3 Weigh out the desired amounts of filtration-control agents and add uniformly over a period of 30 s while stirring on the mixer.

12.13.4 Mix for 30 min, removing the containers twice to scrape the sides to dislodge any clay and solids adhering to the container. If foaming occurs while stirring and remains when the sample is removed from the mixer, attempt to remove the entrained air by stirring slowly with a spatula. Add a defoamer if necessary; however, additions of a defoamer can change filtration results. Record the amount of defoamer added.

12.13.5 Measure the pH of the suspensions using the procedure described in ISO 10414-1:2008, Clause 7. If the pH is lower than the untreated test drilling fluid by more than 0,5 pH units, adjust the pH to that of the untreated test drilling fluid with 10 mol/l NaOH. If the pH is higher than the untreated test drilling fluid, record the pH, but do not make any adjustment.

12.13.6 Determine the low-temperature/low-pressure filtrate volume, in accordance with ISO 10414-1:2008, Clause 7. Record the filtrate volume, cake thickness and cake consistency.

The filtrate volume should be determined immediately after preparation.

12.14 Performance after heat ageing

12.14.1 Dynamically age the samples prepared and tested in 12.13 by rolling or rotating in sealed jars for at least 16 h in an oven set at 65 °C (150 °F).

12.14.2 Cool to room temperature.

12.14.3 Measure the pH of the suspensions and adjust the pH with 10 mol/l NaOH to the initial pH as described in 12.13.5.

12.14.4 Stir for 10 min on the mixer set at high shear.

12.14.5 Determine the low-temperature/low-pressure filtrate volume. Record the filtrate volume, cake thickness and cake consistency.

12.14.6 If an indication of the high-temperature stability is desired, age the suspensions either statically or dynamically at the higher temperature in a suitable test container. See Clauses 21 and 22 for guidelines.

12.14.7 Cool to room temperature. Stir for 5 min on the mixer set at high shear.

12.14.8 Determine the high-temperature/high-pressure filtrate volume by the procedure described in ISO 10414-1:2008, Clause 7. Record the filtrate volume, cake thickness and cake consistency.

13 Methylene blue test for drilled solids and commerical bentonite

13.1 Methylene blue capacity of drill solids

13.1.1 Principle

13.1.1.1 The methylene blue capacity of drill solids can be determined by a procedure that is similar to that used for the drilling fluid. The methylene blue capacity is an estimate of the cation-exchange capacity of the drill solids. The true cation-exchange capacity of the drill solids will probably be somewhat higher than the methylene blue capacity.

13.1.1.2 Methylene blue solution is used to titrate a sample of drill solids that has been dried, ground to a powder and treated with a strong dispersant and an oxidizing agent. The dispersed drill solids sample is titrated with methylene blue solution until a faint blue ring or "halo" appears around a drop of dyed drill solids on filter paper.

13.1.2 Reagents and apparatus

13.1.2.1 Methylene blue solution, 3,20 g reagent grade methylene blue ($C_{16}H_{18}N_3SCl$) diluted to 1 l (1 ml = 0,01 milliequivalent) (CAS number 61-73-4).

If the solution is prepared in the laboratory, the moisture content of reagent grade methylene blue shall be determined each time the solution is prepared. Dry a 1,000 g portion of methylene blue to constant mass at 93 °C ± 3 °C (200 °F ± 5 °F). Make the appropriate correction in the mass, m_o, of methylene blue powder taken to prepare the solution as follows:

$$m_o = \frac{3,20}{m_d} \tag{13}$$

where m_d is the mass of dry methylene blue powder sample, in grams.

13.1.2.2 Tetrasodium pyrophosphate, 2 % solution [20 g of tetrasodium pyrophosphate diluted to 1 l (CAS number 7722-88-5)].

13.1.2.3 Hydrogen peroxide, 3 % solution (CAS number 7722-84-1).

13.1.2.4 Sulfuric acid, dilute, c = 2,5 mol/l (5 N).

13.1.2.5 Erlenmeyer flask, of capacity 250 ml.

13.1.2.6 Burette, 10 ml; **micropipette** 0,5 ml; or **graduated pipette** 1 ml.

13.1.2.7 Graduated cylinder, 50 ml (TD).

13.1.2.8 Stirring rod.

13.1.2.9 Hot plate.

13.1.2.10 Magnetic stirrer, with coated stirring bar.

13.1.2.11 Filter paper, Whatman number 1 11).

13.1.2.12 Drying oven, capable of being maintained at 105 °C ± 3 °C (220 °F ± 5 °F).

13.1.2.13 Mortar and pestle.

13.1.2.14 Sieve, 75 µm (200 mesh).

13.1.2.15 Balance, accuracy ± 0,05 g.

13.1.3 Procedure

13.1.3.1 Clean all traces of drilling fluid from the drill solids sample and dry at 105 °C ± 3 °C (220 °F ± 5 °F) for 2 h.

13.1.3.2 Grind the drill solids to a fine powder using the mortar and pestle, and pass through the 75 µm (200 mesh) sieve.

13.1.3.3 Weigh 1,0 g of the drill solids of size less than 75 µm, and add to the Erlenmeyer flask containing 25 ml of 2 % tetrasodium pyrophosphate.

13.1.3.4 Mix well and boil gently for 10 min. Do not boil to dryness.

13.1.3.5 Add 15 ml hydrogen peroxide and 1 ml sulfuric acid. Boil gently for an additional 10 min. Cool to room temperature and dilute with water to a volume greater than 50 ml.

13.1.3.6 Add methylene blue solution to the flask in increments of 0,5 ml while stirring on the magnetic stirrer. If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous tests, somewhat larger increments (1 ml to 2 ml) can be used at the beginning of the titration. After each addition of the methylene blue solution, swirl the contents of the flask for 30 s. While the solids are still suspended, remove one drop of suspension with the stirring rod and place on the filter paper The initial end point of the titration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids, as shown in Figure 2, key item 4 for a 6 ml addition.

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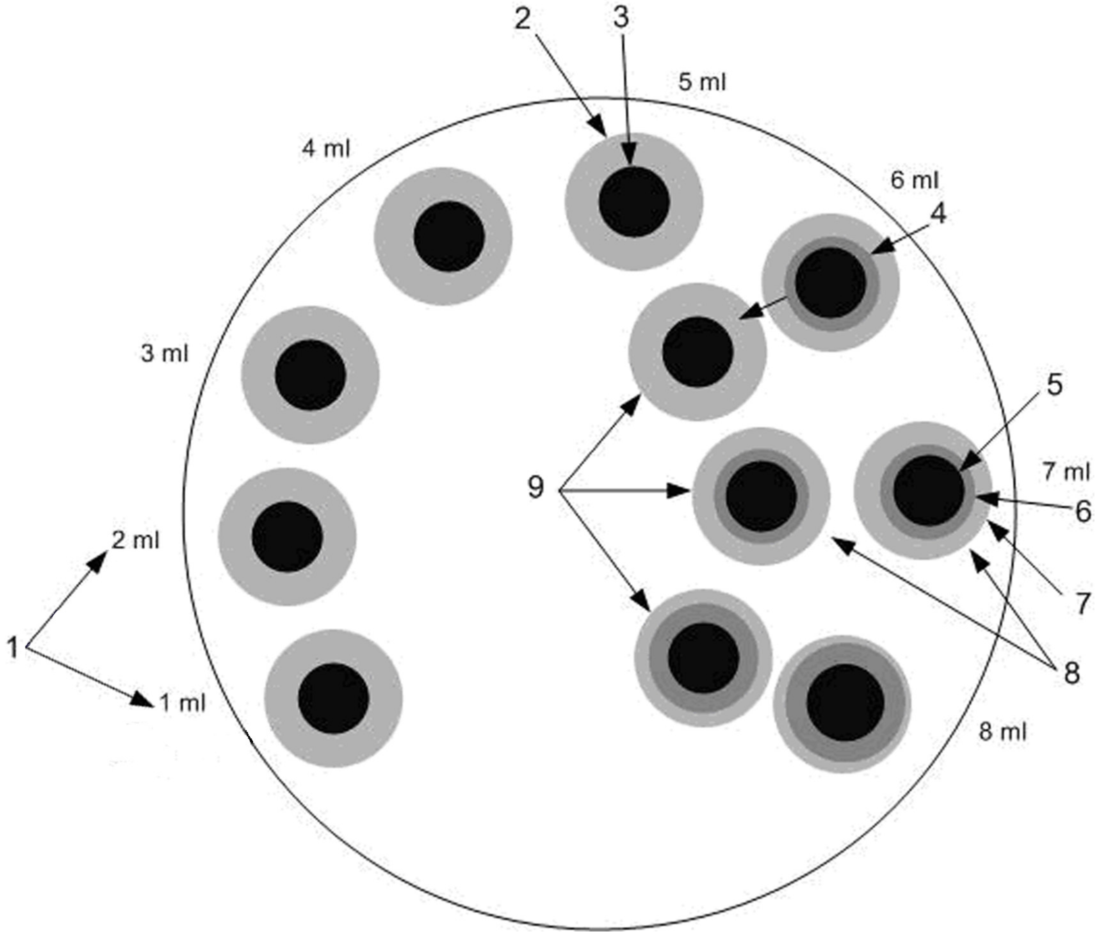
11) Whatman No. 1 filter paper is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

13.1.3.7 When the initial end point is detected, shake the flask for an additional 2 min and place another drop on the test paper. If the blue ring is again evident, the final endpoint has been reached, as shown in Figure 2, key item 8. If the faint blue ring or halo does not appear, continue as in 13.1.3.6 until the second drop taken after 2 min shows the faint blue tint or halo, as shown in key items 8 and 9. This is the 2 min endpoint. Free dye detected immediately after adding methylene blue solution, which is adsorbed after 2 min, indicates that the endpoint has not quite been reached. See key items 4 and 9.

13.1.3.8 Continue stirring an additional 10 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the 10 min endpoint has been reached. If the faint blue ring does not appear, continue as in 13.1.3.6 until the second drop taken after stirring 2 min shows the faint blue ring or halo.



Key

1	volume of methylene blue solution added
2	moisture
3	dyed drilling fluid solids (no free, unadsorbed dye present)
4	free dye (faint blue ring or halo), visible immediately after adding 6 ml, is adsorbed after 2 min and indicates that the end point has not quite been reached
5	dyed drilling fluid solids
6	free, unadsorbed dye
7	moisture
8	endpoint, free dye is not adsorbed after stirring an additional 2 min
9	appearance of spot after additional 2 min agitation

Figure 2 — Spot tests for endpoint of methylene blue titration

13.1.3.9 The methylene blue capacity of the drill solids, $c_{\text{MBT-DS}}$, expressed in milliequivalents per 100 g (meq/100 g), is given by Equation (14).

$$c_{\text{MBT-DS}} = \frac{V}{m_{\text{DS}}} \tag{14}$$

where

V is the volume of methylene blue solution used in titration, in millilitres;

m_{DS} is the mass of drill solids sample, in grams.

13.2 Methylene blue capacity of commercial bentonite

13.2.1 Principle

The methylene blue capacity of commercial bentonite can be determined using a procedure similar to that used for the drill solids. The methylene blue capacity is an estimate of the cation exchange capacity of the bentonite. The true cation exchange capacity of the bentonite will probably be somewhat higher than the methylene blue capacity. A well dispersed sample of the bentonite is titrated with methylene blue solution until a blue or turquoise “halo” appears around a drop of dyed bentonite solids on filter paper.

13.2.2 Reagents and apparatus

13.2.2.1 Methylene blue solution, 1 ml = 0,01 milliequivalent (see 13.1.2.1).

13.2.2.2 Tetrasodium pyrophosphate, 2 % solution (see 13.1.2.2).

13.2.2.3 Erlenmeyer flask, of capacity 250 ml.

13.2.2.4 Burette, of capacity 50 ml.

13.2.2.5 Graduated cylinder, 50 ml (TD).

13.2.2.6 Stirring rod.

13.2.2.7 Hot plate.

13.2.2.8 Magnetic stirrer and stirring bar (coated).

13.2.2.9 Filter paper, Whatman No. 1 [9].

13.2.2.10 Balance, accuracy ± 0,05 g.

13.2.3 Procedure

13.2.3.1 Weigh 0,5 g bentonite and add to the Erlenmeyer flask containing 50 ml of 2 % tetrasodium pyrophosphate solution.

13.2.3.2 Mix well and boil gently for 10 min. Do not boil to dryness.

13.2.3.3 Cool to room temperature and bring volume to about 50 ml with water.

13.2.3.4 Add methylene blue solution to the flask with the burette in 1 ml increments while stirring on the magnetic stirrer. Somewhat larger increments can be used at the beginning of the titration (10 ml to 15 ml) as the endpoint should only be reached after 25 ml or more have been added. After each addition, swirl the contents of the flask for 30 s and remove one drop of the suspension with the stirring rod and place on the filter paper. The initial endpoint of the titration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids as shown in Figure 2, key item 4.

For best accuracy, it is recommended that increments no larger than 1 ml be used.

13.2.3.5 When the initial endpoint is detected, stir the flask for an additional 2 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the 2 min endpoint has been reached, as shown in Figure 2, key items 8 and 9. If the ring does not appear, continue as in 13.2.3.4 until a drop taken after stirring an additional 2 min shows the faint blue tint or halo.

13.2.3.6 Continue stirring an additional 10 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the 10 min endpoint has been reached. If the faint blue ring does not appear, continue as in 13.2.3.4 until a drop taken after stirring 2 min shows the faint blue ring or halo.

13.2.3.7 The methylene blue capacity of commercial bentonite, $c_{\text{MBT-D}}$, expressed in milliequivalents per hundred grams (meq/100 g), is calculated from Equation (15).

$$c_{\text{MBT-B}} = \frac{V}{m_{\text{B}}} \tag{15}$$

where

V is the volume of methylene blue solution used in titration, in millilitres;

m_{B} is the mass of a commercial bentonite sample, in grams.

13.3 Solids content

13.3.1 Principle

The procedure in 13.3.2 can be used to give a rough estimate of the amount of drill solids and commercial bentonite in a drilling fluid using the concentration of low-gravity solids, $c_{\text{lg,A}}$, expressed in kilograms per cubic metre, or $c_{\text{lg,B}}$, expressed in pounds per barrel calculated in ISO 10414-1:2008, Clause 8, plus the bentonite equivalent, $E_{\text{BE,A}}$, expressed in kilograms per cubic metre, or $E_{\text{BE,B}}$, expressed in pounds per barrel (ISO 10414-1:2008, Clause 10), and the methylene blue capacity of the drill solids (13.1.3.9) and commercial bentonite (13.2.3.7).

13.3.2 Calculation

The concentration of each type of low-gravity solid can be calculated from Equations (16) to (21):

$$c_{\text{MBT-AVG}} = \frac{70 \times E_{\text{BE,A}}}{c_{\text{LG,A}}} \tag{16}$$

$$c_{\text{MBT-AVG}} = \frac{70 \times E_{\text{BE,B}}}{c_{\text{LG,B}}} \tag{17}$$

$$c_{\text{B,A}} = \frac{c_{\text{MBT-AVG}} - c_{\text{MBT-DS}}}{c_{\text{MBT-B}} - c_{\text{MBT-DS}}} \times c_{\text{LG,A}} \tag{18}$$

$$c_{\text{B,B}} = \frac{c_{\text{MBT-AVG}} - c_{\text{MBT-DS}}}{c_{\text{MBT-B}} - c_{\text{MBT-DS}}} \times c_{\text{LG,B}} \tag{19}$$

$$c_{\text{DS,A}} = c_{\text{LG,A}} - c_{\text{B,A}} \tag{20}$$

$$c_{\text{DS,B}} = c_{\text{LG,B}} - c_{\text{B,B}} \tag{21}$$

where

$C_{\text{MBT-AVG}}$	is the average methylene blue capacity of for all low gravity solids, expressed in milliequivalents per hundred grams;
$C_{\text{MBT-DS}}$	is the methylene blue capacity of drilled solids, expressed in milliequivalents per hundred grams;
$C_{\text{MBT-B}}$	is the methylene blue capacity of commercial bentonite, expressed in milliequivalents per hundred grams;
$E_{\text{BE,A}}$	is the bentonite equivalent, expressed in kilograms per cubic metre (ISO 10414-1:2008, Clause 10);
$E_{\text{BE,B}}$	is the bentonite equivalent, expressed in pounds per barrel (ISO 10414-1:2008, Clause 10);
$C_{\text{LG,A}}$	is the concentration of low gravity solids, expressed in kilograms per cubic metre (ISO 10414-1:2008, Clause 8);
$C_{\text{LG,B}}$	is the concentration of low gravity solids, expressed in pounds per barrel (ISO 10414-1:2008, Clause 8);
$C_{\text{B,A}}$	is the concentration of bentonite, expressed in kilograms per cubic metre;
$C_{\text{B,B}}$	is the concentration of bentonite, expressed in pounds per barrel;
$C_{\text{MBT-DS}}$	is the methylene blue capacity of drilled solids, expressed in milliequivalents per hundred grams;
$C_{\text{DS,A}}$	is the concentration of drilled solids, expressed in kilograms per cubic metre
$C_{\text{DS,B}}$	is the concentration of drilled solids, expressed in pounds per barrel

If the $C_{\text{MBT-B}}$ for bentonite is not known, use a value of 70 meq/100 g, as is assumed in the calculation of the bentonite equivalent of the drilling fluid (ISO 10414-8:2001, Clause 8) and for the calculations of the average MBT, $C_{\text{MBT-AVG}}$, of all low gravity solids above. The solids composition calculated assuming a $C_{\text{MBT-AVE}}$ of 70 meq/100 g for bentonite is not as accurate as if the full procedure above is used, but the calculated amounts of bentonite and drill solids can still be useful as a monitoring tool.

14 Deflocculation test for thinner evaluation

14.1 Principle

14.1.1 The following laboratory test is designed to evaluate the relative effectiveness of a thinner in a high-solids environment. The data obtained using this procedure should be compared to data using a reference thinner in the same series of tests, and should not be interpreted as thinner performance in actual drilling fluid formulations.

14.1.2 A 28,0 % (mass fraction) suspension of Neutral Panther Creek [13)] bentonite is prepared with deionized or distilled water and aged at room temperature for 1 week or more. A laboratory barrel portion (350 ml) of the suspension is treated with 14,3 kg/m^3 of chrome or ferrochrome lignosulfonate for the uncontaminated tests and with 22,8 kg/m^3 of the reference thinner for contaminated [14,3 kg/m^3 (5 lbs/bbl) gypsum and 11,4 kg/m^3 (4 lbs/bbl) sodium chloride] tests and the pH adjusted to 9,5 with sodium hydroxide. Viscometric properties are measured initially and after hot-rolling for 16 h at 65 °C (150 °F). Filtration volume is measured after hot-rolling. After completing the measurements on the hot-rolled samples, the pH of the suspension is adjusted to 9,5 and aged for 16 h in a stainless steel cell at 175 °C (350 °F). The cell is pressurized to 3 450 kPa (500 psi) with nitrogen prior to heating. Viscometric properties of the suspension are measured after high-temperature ageing as removed from the cell. The pH of the suspension is readjusted to 9,5, and viscometric properties are remeasured and the filtration volume is determined.

14.1.3 The effectiveness of the test sample is evaluated by comparing the results with the performance of a reference sample of lignosulfonate or other thinner.

14.2 Reagents and apparatus

14.2.1 **Neutral Panther Creek bentonite** (air-floated) 12).

14.2.2 **Sodium chloride** (CAS number 7647-14-5), 99 % minimum purity.

14.2.3 **Gypsum** (CAS number 7778-18-9), 92 % minimum $Ca_2SO_4 \cdot 2H_2O$, 85 % through 100 mesh.

14.2.4 **Sodium hydroxide** (CAS number 1310-73-2) CP, used to make a solution such that 1 ml contains 0,25 g NaOH.

14.2.5 **Deionized** or **distilled water**.

14.2.6 **Standard** or **reference thinner**.

14.2.7 **Mixer** 13), capable of operating at 10 000 r/min ± 100 r/min under load and designed so that the bearings are not exposed to the test fluid.

14.2.8 **Container**, for mixing, with air-tight cover.

14.2.9 **Balance**, 3 kg capacity.

14.2.10 **Drilling fluid balance**.

14.2.11 **Viscometer** 14), in accordance with ISO 10414-1:2008, Clause 6.

14.2.12 **Transformer**, variable speed, e.g. Powerstat® 15), type 116.

14.2.13 **pH meter**, accurate to 0,1 pH unit.

14.2.14 **Minute timer**, accurate to 0,1 min over the test interval.

14.2.15 **Filter press**, in accordance with ISO 10414-1:2008, Clause 7.

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12) Neutral Panther Creek bentonite is the trade name of a product supplied by the American Colloid Company, Skokie, Illinois, U.S.A. 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.

13) Hamilton Beach Mixer Model 936 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.

14) Fann VG Meter Model 35 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.

15) Powerstat, type 116 is an example of a suitable product available commercially. This information is given for the convenicence of users of this International Standard and does not constitute an endorsement by ISO of this product.

14.2.16 Filter paper, Whatman No. 50 [16].

14.2.17 Spatula.

14.2.18 Ageing cells, high-temperature, stainless steel with pressure connector, in accordance with Clause 21.

14.2.19 Drying oven, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

14.2.20 Jar, 500 ml, glass canning.

14.2.21 Burette, graduated to 0,1 ml.

14.2.22 Nitrogen source, at a pressure of 3 450 kPa (500 psi) or greater.

14.2.23 Syringe, 10 ml, graduated to 0,2 ml.

14.2.24 Mixer, capable of operating at 7 500 r/min ± 500 r/min under load and designed so that the bearings are not exposed to the test fluid [17].

14.2.25 Roller oven, regulated to 65 °C ± 3 °C (150 °F ± 5 °F), preferably with gravity convention of air (forced-air circulation)

14.2.26 Ageing oven, static, capable of maintaining a temperature of 175 °C ± 3 °C (350 °F ± 5 °F).

14.3 Procedure for moisture content

14.3.1 Determine the moisture content of the Neutral Panther Creek bentonite as given in 14.3.2 and 14.4.

14.3.2 Weigh 10 g ± 0,01 g of clay and dry to a constant mass at a temperature of 105 °C ± 3 °C (220 °F ± 5 °F). Cool the sample in a desiccator and weigh.

14.4 Calculation of moisture content

Calculate the moisture content, w_h, expressed as a percent mass fraction, from Equation (22):

$$w_\mathrm{h} = 100\,\frac{m_\mathrm{o} - m_\mathrm{d}}{m_\mathrm{o}} \tag{22}$$

where

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

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

14.5 Preparation of drilling fluid base

14.5.1 Prepare a 500 g suspension by dispersing 140 g of Neutral Panther Creek bentonite (dry basis) in deionized or distilled water. The amount of water required is 350 g minus the amount of water in the clay as determined in 14.4. The clay should be added to the water slowly, while stirring the suspension with the mixer.

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[16] Whatman No. 50 filter paper 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.

[17] Hamilton Beach Model 30 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

14.5.2 Shear the suspension for 2,5 h, seal the container airtight so that no moisture can escape and age overnight at room temperature. On the following day, stir for 30 min and determine the solids content of the suspension as in 14.5.3.

14.5.3 Weigh 10 g ± 0,01 g of suspension and dry at a temperature of 105 °C ± 3 °C (220 °F ± 5 °F). Cool the sample in a desiccator and weigh. Perform this test in duplicate until results agree within ± 0,1 % by mass.

14.5.4 The solids content shall be 28,0 % ± 0,1 % by mass. If the solids content is too low, let the suspension stand overnight exposed to the atmosphere so that some of the moisture can evaporate. Then again determine the solids content as in 14.5.3. Repeat until the solids content is correct. If the solids content is too high, see 14.6.2.

14.6 Calculation

14.6.1 Calculate the solids content, w_s, expressed as a percent mass fraction of the solids, from Equation (23):

$$w_\mathrm{s} = 100 \frac{m_\mathrm{d}}{m_\mathrm{o}} \tag{23}$$

where

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

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

14.6.2 If the solids content is too high, add deionized water. Calculate the additional mass of water, m_h, expressed in grams, required for solids adjustment from Equation (24):

$$m_\mathrm{h} = m_\mathrm{l} - 100 \frac{m_\mathrm{s}}{w_\mathrm{s}} \tag{24}$$

where

m_l is the specified mass of the suspension, expressed in grams (500 g in this procedure);

m_s is the total mass of solids added, expressed in grams [140 g Neutral Panther Creek bentonite (dry basis) in this procedure];

w_s is the mass fraction of the solids, expressed in percent.

EXAMPLE Solids content, determined as equal to 29,1 %, is too high.

$$m_\mathrm{h} = 500 - 100 \frac{140}{29{,}1}$$

$$m_\mathrm{h} = 500 - 481{,}1$$

$m_\mathrm{h} = 18{,}9$ g water required to correct solids content.

14.7 Determination of rheological properties

14.7.1 To 350 ml of the Neutral Panther Creek bentonite suspension, add 1,75 g salt and stir for 3 min at 750 r/min ± 50 r/min with a mixer.

14.7.2 Add 5 g of the thinner being tested. Stir the sample for 1 min.

14.7.3 Add sufficient caustic soda (1 ml = 0,25 g NaOH) so that the pH measures 9,5 ± 0,1 after 20 min total stirring time.

14.7.4 Scrape down the sides of the jar at least once to ensure proper mixing. The mixing speed shall be kept at 7 500 r/min ± 500 r/min. After 20 min total stirring time, remove the drilling fluid from the mixer. Check to see that the pH is 9,5 ± 0,1.

14.7.5 Stir for 1 min and measure the flow properties with a viscometer. Record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings.

14.7.6 Record the values for the initial gel, pH and total volume of NaOH used in 14.7.3.and 14.7.4.

14.7.7 Cap the jar tightly and hot-roll for 16 h at 65 °C (150 °F).

14.7.8 Cool the hot-rolled drilling fluid to room temperature. Readjust the pH.

14.7.9 Again take the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings, and the readings for the initial gel, pH,10 min gel and filtrate.

14.7.10 Place the drilling fluid in a stainless steel high-temperature ageing cell. Pressurize the ageing cell to 3 450 kPa (500 psi) with nitrogen and test for leaks by immersing in water and examining for bubbles. Place the cell in a 175 °C (350 °F) oven for 16 h.

14.7.11 Remove the cell, cool and transfer the drilling fluid to a pint jar.

14.7.12 Measure and record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings. Readjust the pH to 9,5 ± 0,1 and remeasure the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min properties and the filtrate volume.

14.7.13 To 350 ml of the base drilling fluid, add 5 g gypsum, 4 g sodium chloride, and 8 g of the thinner being evaluated. Stir into the drilling fluid for 20 s to 30 s by raising, lowering and turning the jar by hand while the mixer is running at 7 000 r/min to 8 000 r/min with the toggle switch in the high-speed position.

14.7.14 Place the jar into the jar holder. Set the timer for 20 min stirring time. After an elapsed time of 1 min, add a sufficient volume of NaOH solution (1 ml = 0,25 g NaOH) rapidly with a syringe to give a pH of 9,5 ± 0,1 at the end of the 20 min stirring time. The quantity of NaOH varies with each type of thinner used and should be determined by trial and error.

14.7.15 After 5 min stirring, remove the sample from the mixer and scrape down the sides of the jar.

14.7.16 Replace the sample on the mixer and continue stirring for a total elapsed time of 20 min at 7 000 r/min to 8 000 r/min.

14.7.17 At the end of 20 min elapsed time, remove the drilling fluid from the mixer and measure the pH; if the pH is 9,5 ± 0,1, stir for 1 min at 7 000 r/min to 8 000 r/min and measure the flow properties with the viscometer. If the pH is not 9,5 ± 0,1, discard the sample and repeat the test.

14.7.18 Measure and record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings, and the values for initial gel, pH and the total NaOH used.

14.7.19 Cap the jar tightly and hot-roll for 16 h at 65 °C (150 °F).

14.7.20 Cool the hot-rolled drilling fluid to room temperature and stir for 5 min at 7 000 r/min to 8 000 r/min.

14.7.21 Record the 600 r/min, 300 r/min, 200 r/min, 100 r/min and 6 r/min readings, the values for the initial gel, the pH and the filtrate.

14.7.22 Adjust the pH to 9,5 with NaOH and place in a stainless steel high-temperature ageing cell. Pressure the ageing cell to 3 450 kPa (500 psi) with nitrogen and test for leaks by immersing in water. Place the cell in an oven at 175 °C (350 °F) for 16 h.

14.7.23 Remove the cell, cool and transfer the drilling fluid to a pint jar. Stir for 5 min at 7 000 r/min to 8 000 r/min on a Hamilton Beach mixer®.

14.7.24 Measure and record the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min viscometer readings. Record the pH.

14.7.25 Then adjust the pH to 9,5 with NaOH and remeasure the 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min properties and the filtrate volume.

14.8 Calculation of thinner efficiency

Calculate the thinner efficiency, e, expressed as a percent of the performance of the test sample relative to the performance of a reference sample of lignosulfonate or other thinner, as given in Equation (25).

$$e = 100 \frac{p_\mathrm{s}}{p_\mathrm{r}} \tag{25}$$

where

p_s is the performance, for example yield point or gel strength as determined in accordance with ISO 10414-1, of the test sample;

p_r is the performance, for example yield point or gel strength as determined in accordance with ISO 10414-1, of the reference thinner.

15 Testing base oils used in drilling fluids

15.1 General

This clause gives a brief summary of the ASTM testing methods that API Subcommittee 13 on Drilling Fluids Materials has reviewed and found to be acceptable for the testing of base oils commonly used in oil-based drilling and completion fluids. Where a procedure is also described in an International Standard, as is most often the case, the International Standard is given in preference to the national standard, such as ASTM or IP. The national standard (ASTM, IP) is given when this is the sole procedure.

15.2 Reagents and apparatus

The reagents and apparatus as specified in the appropriate International Standard, or ASTM or IP procedure bulletin shall be used. These reagents and test apparatus are mandatory to perform the standard tests correctly and safely, and to achieve results which meet ISO requirements. They are described in this International Standard in general terms only.

15.3 Density, relative density (specific gravity), or API gravity-hydrometer method (see ISO 3675)

15.3.1 Principle

This method covers the laboratory determination, using a glass hydrometer, of density, relative density (specific gravity), or API gravity of crude petroleum, petroleum products, or mixtures of petroleum and non-petroleum products normally handled as liquids, and having a Reid vapour pressure (see ISO 3007) of 180 kPa (1,8 bar; 26 psi) or less. Values are measured on a hydrometer at convenient temperatures, readings of density being reduced to 16 °C (60 °F) and readings of relative density (specific gravity) and API gravity to

16 °C (60 °F), in accordance with the petroleum reference tables given in ISO 91-1. By means of these tables, values determined in any one of the three systems of measurement can be converted to equivalent values in either of the other two, so that measurements may be made in the units of local convenience. Generally, the drilling fluids industry prefers to use the relative density (specific gravity) units.

15.3.2 Summary of the method

The sample is brought to the specified temperature and transferred to a cylinder that is at approximately the same temperature. The appropriate hydrometer is lowered into the sample. After temperature equilibrium has been reached and the hydrometer is still, the hydrometer scale is read and the temperature of the sample is noted. If necessary, the cylinder and its contents may be placed in a constant-temperature bath to avoid excessive temperature variation during the test.

15.4 Density and relative density of liquids using a digital density meter (see ASTM D 4052)

15.4.1 Principle

This method covers the determination of the density or relative density of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at test temperatures between 16 °C and 35 °C (60 °F and 95 °F). Its application is restricted to liquids with vapour pressures below 80 kPa (11 psi) and viscosities below about 0,015 m^2/s at the temperature of test. It should not be applied to samples so dark in colour that the absence of air bubbles in the sample cell cannot be established with certainty.

15.4.2 Summary of method

Approximately 0,7 ml of liquid sample is introduced into the oscillating sample tube of a digital density meter, and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

15.5 Kinematic viscosity of transparent and opaque oils — Calibrated capillary tube method (see ISO 3104)

15.5.1 Principle

This test method covers the determination of the kinematic viscosity (a measure of the resistance to flow under gravity) of liquid petroleum products, both transparent and opaque, by measuring the time for a given volume of the liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid.

15.5.2 Summary of method

The time, in seconds, is measured for a fixed volume of the liquid sample to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer. Testing temperatures usually selected are 40 °C and 70 °C (105 °F and 160 °F) for base oils, unless these temperatures can constitute a hazard of fire or flash explosion during the testing.

15.6 Distillation (see ISO 3405)

15.6.1 Principle

This method covers the distillation of motor gasolines, aviation gasolines, aviation turbine fuels, special boiling-point spirits, naphthas, white spirit, kerosenes, gas oils, distillate fuel oils and similar petroleum products.

15.6.2 Summary of method

A 100 ml sample is distilled under specified conditions that are appropriate to its nature as shown in ISO 3405:2000, Table 1. Systematic observation of the thermometer readings and volumes of condensate are made and, from these data, the results of the tests are calculated and reported.

Important temperature values found are

a) initial boiling point, when the first drop is condensed,

b) final boiling point, when maximum temperature is reached, and

c) dry point, the temperature just as the last drop evaporates from the lowest point in the flask.

15.7 Aniline point and mixed aniline point (see ISO 2977:1997)

CAUTION — Application of ISO 2977 can involve the use of hazardous materials, operations and apparatus. ISO 2977 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the International Standard to consult and establish appropriate safety and heath practices and determine the applicability of regulatory limitations prior to use.

15.7.1 Principle

These test methods cover the determination of the aniline point of petroleum products and hydrocarbon solvents. Method 1 (ISO 2977:1997, Annex A) is suitable for transparent samples with an initial boiling point above room temperature and when the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture. Method 2 (ISO 2977:1997, Annex B), a thin-film method, is suitable for samples too dark for testing by method 1. Methods 3 (ISO 2977:1997, Annex C) and 4 (ISO 2977:1997, Annex D) are for samples that can vapourize appreciably at the aniline point. Method 4 is particularly suitable when only small samples are available. Method 5 (ISO 2977:1997, Annex E) describes a procedure using an automatic apparatus suitable for the range of liquids covered by methods 1 and 2. These test methods also cover the determination of the mixed aniline point of petroleum products and hydrocarbon solvents having aniline points below the temperature at which aniline crystallizes from the aniline-sample mixture.

15.7.2 Summary of methods

Method 1, and perhaps method 2, are the most likely to be applicable to drilling fluid industry use for testing base oils. The specified volumes of aniline and oil sample (or aniline and sample and *n*-heptane) are placed in a tube and mechanically mixed. The mixture is heated at a controlled rate until the two phases become miscible. The mixture is then cooled at a controlled rate and the temperature at which two phases begin to separate and cloudiness appears is recorded as the aniline point (or mixed aniline point if *n*-heptane is used as a solvent).

15.8 Pour point (see ISO 3016)

15.8.1 Principle

The test for pour point is intended for use on any petroleum oil. A procedure suitable for black oils, cylinder stock and non-distillate fuel oil is also described in ISO 3016.

15.8.2 Summary of method

After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3 °C (5 °F) for flow characteristics. The lowest temperature at which movement of the oil is observed is recorded as the pour point.

15.9 Flash point by Pensky-Martens closed tester (see ISO 2719)

15.9.1 Principle

This method covers the determination of the flash point by the Pensky-Martens closed-cup tester of fuel oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids.

This method is not recommended for testing emulsions of water and oil, such as drilling fluids.

15.9.2 Summary of method

The oil sample is heated in a cup at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapour above the sample to ignite.

15.10 Aromatics content (see IP 391 or ASTM D 5186)

15.10.1 Principle

15.10.1.1 General

Both the ASTM and the IP method measures the “aromatic content” of drilling fluid oils, petroleum distillates and diesel fuels. The user should realize that analyses of a complex oil by the two methods might not give the same values for aromatic content. The reason for variation is that the two instruments separate aromatic components differently and their detectors function by different principles. Instruments for the IP method are more widely available and less expensive than those required for the ASTM method. The ASTM method uses a column and detector system that gives a better representation of the types of aromatics found in complex oils, but it does not measure low concentrations of aromatics.

15.10.1.2 IP 391

The IP 391 method is suitable for determining monocyclic, bicyclic and tricyclic aromatic fractions in drilling fluid oils and petroleum distillates that boil in the range of 140 °C to 360 °C (285 °F and 680 °F) with bromine numbers (determined in accordance with ISO 3839) of less than 1,0. This method is appropriate for concentrations from 0,05 % volume fraction to 25 % volume fraction monocyclic aromatics and 0,05 % volume fraction to 5 % volume fraction bicyclic and tricyclic aromatics. Higher concentrations require appropriate dilutions.

NOTE Compounds containing oxygen or nitrogen can cause erroneous results for the IP method.

15.10.1.3 ASTM D 5186

The ASTM D 5186 method is suitable for determining aromatic compounds in fuels with concentrations from 5 % mass fraction to 75 % mass fraction of aromatics.

15.10.2 Summary of the IP 391 method

A 5 µl to 10 µl sample of oil is injected into a liquid chromatograph that is fitted with a polar separation column with hexane as the mobile phase. The column separates the aromatic components into mono-, bi- and tricyclic fractions that appear as peaks on the chromatogram. The fractions are identified by a differential refractive-index detector as they exit the column. Percentages are determined by integrating the areas under the peaks based on prior calibration with known concentrations of an aromatic compound.

15.10.3 Summary of the ASTM D 5186 method

A 0,05 µl to 0,2 µl oil sample is injected into a packed silica column using supercritical carbon dioxide as the mobile phase. Aromatic components in the sample are separated from non-aromatic components by adsorption in the silica column. Components are measured by a flame ionization detector as they exit the column. Concentrations are calculated from peak areas based on prior calibration using known concentrations of an aromatic compound.

16 Potassium ion content — Ion-selective electrode method

16.1 Principle

16.1.1 Potassium compounds, primarily the chloride and, to a lesser degree, the hydroxide compounds, are used in drilling fluids to aid in the stabilization of the formation of clays and/or shales. The ion-selective electrode (ISE) method can provide accurate, quantitative data on samples having a range of potassium ion concentration from a few milligrams per litre to saturation.

16.1.2 The measurement of potassium ion concentration by the ISE method is analogous to a pH measurement and can be performed with almost the same ease and simplicity. Both procedures rely on the measurement of the difference in potential between a sensing and a reference electrode. Depending on the instrumentation used, this signal is presented as either the actual potential, measured in millivolts, or in some form of direct readout of concentration.

16.1.3 For monovalent ions such as hydrogen and potassium, the change in potential between a sensing and a reference electrode is theoretically 59 mV for a ten-fold change in the activity of the ion being measured. The actual change is normally somewhat lower, usually about 56 mV ± 2 mV for the potassium electrode system. This value is defined as the slope of the electrode system.

16.1.4 It should be noted that the actual property being measured is activity, rather than concentration. However, since the activity of any ionic species is strongly influenced by the total ionic strength of the solution, it is feasible to use the two terms interchangeably so long as the total ionic strength is held constant. This procedure provides for a constant background total ionic strength by the addition of sodium chloride solution to all standards and samples.

16.1.5 Under the conditions of this test method, none of the materials normally present in drilling fluid filtrate samples has been found to interfere. Errors arising from sodium contamination are eliminated by providing a high constant background level of sodium through the use of sodium chloride as the ISA solution. The relatively high dilution reduces all other potential interference to a negligable level.

16.2 Reagents and apparatus

16.2.1 **Potassium chloride**, standard solution, $\rho = 1$ g/l.

This solution is readily available from most laboratory supply firms, or can be prepared by dissolving 1,907 g reagent grade KCl in deionized or distilled water and diluting to 1 l. Dry the KCl in an oven at 105 °C ± 3 °C (220 °F ± 5 °F) for 2 h and store in a desiccator prior to preparation of the standard solution.

16.2.2 **Sodium chloride**, solution, $c_{\text{NaCl}} = 5$ mol/l.

Dissolve 292 g reagent grade sodium chloride in deionized or distilled water and dilute to 1 l. This solution is used as an ISA and to provide a high, constant background sodium concentration.

16.2.3 **Sodium chloride**, solution, $c_{\text{NaCl}} = 0{,}1$ mol/l, saturated with the silver ion.

Dilute 2 ml of the 5 mol sodium chloride solution to 100 ml and add silver nitrate solution dropwise until a cloudiness persists. This solution is used as the internal filling solution for the single-junction reference electrode.

16.2.4 **Specific-ion meter** or **pH meter**.

Any of a wide variety of commercially available instruments is suitable, so long as it can provide a millivolt readout with a resolution of at least 0,1 mV.

Either analogue or digital readout is acceptable, but the preferred type of instrument is one of the “research” models with a digital readout and controls that permit direct indication of concentration after proper calibration.

16.2.5 Potassium ISE, potassium electrode.

The Orion Model 93-19 [18] is a two-part electrode, with a reusable body and a disposable, screw-on sensing module. Only the module, not the entire electrode, requires replacement. Average service life of each module is six months.

16.2.6 Reference electrode, a single-junction reference electrode [19].

NOTE Both the sensing and reference electrodes are available with durable epoxy bodies.

16.2.7 Magnetic stirrer, with coated stirring bars.

16.2.8 Beakers, 150 ml, of borosilicate glass (Pyrex®) or plastic.

16.2.9 Pipettes, volumetric, various sizes for preparing dilutions of samples and standards.

The use of microlitre pipettes with disposable plastic tips [20] is particularly convenient for diluting filtrate samples where the quantity of filtrate available is limited or extremely high dilutions are required.

16.2.10 Graph paper, 3-cycle or 4-cycle semilogarithmic, for preparing calibration curves.

16.2.11 Flasks, volumetric, various sizes for preparing calibration standards and dilutions of filtrate samples.

16.3 Preparation of electrodes

16.3.1 If the Orion 93-19 electrode is used, remove the sensing module from the vial and screw it firmly into the electrode body, taking care to ensure that the rubber washer is in place.

16.3.2 Shake down the electrode in the manner of a mercury-filled medical thermometer.

16.3.3 Soak the electrode in deionized or distilled water for at least 10 min, then store in a diluted potassium solution containing 10 mg/l to 100 mg/l potassium until ready for use.

16.3.4 By means of either an eye-dropper or syringe, fill the single-junction reference electrode with the 0,1 mol/l sodium chloride (saturated with silver ion) solution (see 16.2.3).

16.3.5 If other types of sensing or reference electrodes are used, follow the instructions of the manufacturer when preparing them for use.

16.4 Operational check of electrode system

16.4.1 The two properties of the electrode system having the greatest influence on accuracy and reproducibility are the slope and the stability. The slope is defined as the change in potential observed when the concentration changes by a factor of 10. The stability of the readings is an indication of the amount of noise (rapidly, continuously changing readings) and drift (readings slowly changing in one direction).

16.4.2 When performing the initial calibration with the standard solutions according to the instructions in either 16.5 or 16.6, record the actual measured potential for the 10 mg/l and the 100 mg/l standard solutions. For a new potassium electrode (or sensing module), the difference between these two readings shall be between 54 mV and 59 mV. The slope normally decreases with time. Replace the electrode (or module) when the slope reaches a value of approximately 48 mV.

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[18] Orion Model 93-19 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

[19] Orion Model 90-01 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

[20] Eppendorf and Oxford are examples of suitable products available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of these products.

16.4.3 If, when performing the initial calibration, there is an indication of noise or drift, refer to the troubleshooting clause of the electrode manual. There are a number of possible causes for these effects with any electrode system, and the cause is normally found only by a process of elimination. If the analyst is unable to achieve a stability level that meets the manufacturer's specifications, replace the sensing electrode (or module).

16.5 Measurements using a meter with direct concentration readout capability

16.5.1 Prepare a 100 mg/l potassium standard and a 10 mg/l potassium standard by serial dilution of the 1 g/l standard solution.

16.5.2 Add the ISA solution (5 mol/l sodium chloride) to each standard solution at the rate of 2 ml of ISA per 100 ml of standard.

16.5.3 Transfer the standards to 150 ml beakers. Place the 10 mg/l standard on the magnetic stirrer and stir at a moderate rate. Immerse the electrodes.

16.5.4 Wait for a stable reading (normally 1 min to 2 min), then adjust the controls to display the potassium concentration of the standard following the procedure described in the meter instruction manual.

16.5.5 Remove the electrodes, rinse with distilled water, blot dry, and place in the 100 mg/l standard. Stir at a moderate rate.

16.5.6 Wait for a stable reading, then adjust the controls to display the potassium concentration of the standard. The meter and electrode system are now calibrated.

16.5.7 Prepare a 1:1 000 dilution of each drilling fluid filtrate sample.

16.5.8 Transfer 100 ml of diluted sample to a 150 ml beaker, add 2 ml ISA and stir at a moderate rate.

16.5.9 Rinse the electrodes with distilled water, blot dry, immerse in the sample and wait for a stable reading. The value of the reading displayed on the meter is the potassium concentration in the diluted sample. This value, multiplied by the dilution factor (1 000 in this case), gives the potassium concentration in the original sample.

NOTE Concentration values that are above or below the concentrations of the standards for calibration are still reliable, since the 10 mg/l and 100 mg/l standards are near the midpoint of the region of linear response of the potassium electrode.

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

16.6.1 Prepare 100 mg/l, 10 mg/l and 1 mg/l potassium standards by serial dilution of the 100 mg/l standard solution.

16.6.2 Add the ISA solution (5 mol/l sodium chloride) to each standard solution at the rate of 2 ml of ISA per 100 ml of standard.

16.6.3 Transfer the standards to 150 ml beakers. Place the 1 mg/l standard on the magnetic stirrer and stir the solution at a moderate rate. Place the electrodes in the standard solution.

16.6.4 Adjust the controls of the instrument so that the readout is in millivolts; wait until the reading is stable, and record the reading.

16.6.5 Remove electrodes, rinse, blot dry and place in the 10 mg/l standard. When the reading is stable, record the reading.

16.6.6 Repeat step 16.6.5 with the 100 mg/l standard.

16.6.7 Plot the millivolt readings on the linear axis and potassium concentration, in milligrams per litre, on the logarithmic axis of standard 3-cycle or 4-cycle semilogarithmic paper.

16.6.8 Transfer 100 ml of the sample diluted 1:1 000 to a 150 ml beaker, add 2 ml of the ISA solution and stir at a moderate rate. Place the electrodes in the solution.

16.6.9 Record the reading when stable and determine the concentration of the diluted sample by use of the calibration curve. This value, multiplied by the dilution factor, gives the potassium content of the original sample.

For accurate measurements, all standards and samples should be at ambient temperature. Some magnetic stirrers generate considerable heat, and it can be necessary to place a layer of insulating material (plastic or cardboard) between the stirrer and beaker. Between measurements, rinse electrodes and blot dry with tissue. After immersion in the sample or standard solutions, check the electrode end for air bubbles and remove if present. Always read and carefully follow the manufacturer’s instructions regarding the care, maintenance, storage, servicing and troubleshooting of the instrument and electrodes.

17 Calcium ion content — Ion-selective electrode method

17.1 Principle

17.1.1 Calcium is present to some extent in the liquid phase of all water-based drilling fluids. The concentration level can vary from several percent by mass to a few milligrams per litre. It may be intentionally added in the form of lime, gypsum, or calcium chloride, or it may be introduced through the make-up or dilution water, as an impurity in various products or from drilled formations. Regardless of the concentration level or the origin of the calcium, it is usually necessary or desirable to monitor the calcium content on a quantitative basis.

17.1.2 The ion-selective electrode (ISE) method is a rapid, simple and accurate method for the determination of calcium at any concentration in the filtrate samples. Unlike the EDTA titrimetric method, no sample treatment is required for highly coloured or turbid samples. Under the conditions of this test method, none of the materials normally present in drilling fluids has been found to interfere. The combination of relatively high dilution, ionic-strength adjustment through addition of KCl solution and adjustment of the pH to 5,0 ensures maximum conversion of calcium to ionic form and effectively eliminates or minimizes interference.

17.1.3 This method is intended for the analysis of solution samples only and is not suitable for the analysis of whole drilling fluid samples.

17.2 Reagents and apparatus

17.2.1 **Calcium carbonate**, standard solution, $\rho = 1$ g/l.

Weigh 2,497 2 g of reagent grade calcium carbonate on an analytical balance, add about 50 ml water and dissolve by the dropwise addition of concentrated hydrochloric acid (HCl) (about 10 ml is required). When dissolution is complete, dilute to 1 l in a volumetric flask.

NOTE This solution is also readily available from laboratory supply houses.

17.2.2 **Potassium chloride**, solution, $c_{\text{KCl}} = 4$ mol/l.

Dissolve 298 g of reagent grade KCl in deionized water and dilute to 1 l. This solution is used as an ISA solution.

17.2.3 **Buffer solution**, pH 5,0.

Dissolve 136 g of sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3H_2O$) in about 500 ml of deionized water. Add 57,5 ml of glacial acetic acid ($HC_2H_3O_2$) and dilute to 1 l. This solution is used to adjust the pH of all standard and sample solutions to a pH of 4,8 to 5,2.

17.2.4 Potassium chloride, solution, $c_{\mathrm{KCl}} = 4$ mol/l, saturated with silver.

Transfer about 50 ml of the 4 mol/l KCl (see 17.2.2) to a 2 ounce plastic bottle and add silver nitrate solution dropwise until a cloudiness persists. This solution is used as the filling solution for the reference electrode.

NOTE This solution is also readily available from chemical supply houses.

17.2.5 pH meter, capable of providing a millivolt readout with a resolution of at least 0,1 mV.

There are many instruments available, ranging from battery-operated, hand-held portable units for field use to sophisticated and versatile “research” instruments programmed to provide readouts in concentration units. Readouts may be either analogue or digital. The preferred type for laboratory use is a research model with a digital readout and controls that provide direct indication of concentration after proper calibration.

17.2.6 Calcium ISE, calcium electrode.

The Orion Model 93-20 (21) is a two-part electrode, with a reusable epoxy body and a disposable, screw-on sensing module. Average service life of the module is six months.

17.2.7 Reference electrode, single-junction (20).

17.2.8 Magnetic stirrer, with PTFE-coated stirring bars, approximately 10 mm (3/8 in) in diameter and 25 mm (1,0 in) long.

17.2.9 Beaker, 150 ml, of Pyrex or inert plastic.

17.2.10 Pipettes, volumetric, Class A, various sizes are required for preparing dilutions of standards and samples.

The use of a good-quality microlitre pipette with disposable tips is particularly convenient for quantitative transfer of volumes of 1 ml and smaller.

17.2.11 Flasks, volumetric, Class A, various sizes for preparing calibration standards and dilutions of filtrate samples.

17.2.12 Graph paper, 3-cycle or 4-cycle semilogarithmic, for preparing calibration curves.

17.3 Preparation of electrodes

17.3.1 If the Orion 93-20 electrode is used, remove the sensing module from the vial and screw it firmly into the electrode body, taking care to ensure that the rubber washer is in place.

17.3.2 Shake down the electrode in the manner of a mercury-filled medical thermometer.

17.3.3 Soak the electrode in deionized or distilled water for at least 15 min, then store in a dilute calcium solution, containing 10 mg/l to 100 mg/l calcium, until ready for use.

17.3.4 By means of either an eye dropper or syringe, fill the single-junction reference electrode with the 4 mol/l potassium chloride solution (saturated with silver ion).

17.3.5 If other types of sensing or reference electrode are used, follow the instructions of the manufacturer when preparing them for use.

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21) Orion Model 93-20 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

17.4 Operational check of electrode system

17.4.1 The two properties of the electrode system that have the greatest influence on accuracy and reproducibility are the slope and the stability. The slope is defined as the change in potential observed when the concentration changes by a factor of 10. The stability of the readings is an indication of the amount of noise (rapidly, continuously changing readings) and drift (readings slowly changing in one direction).

17.4.2 When performing the initial calibration with the standard solutions according to the instructions in either 17.5 or 17.6, record the actual measured potential for the 1 mg/l and the 10 mg/l standard solutions. For a new calcium electrode (or sensing module), the difference between these two readings shall be between 25 mV and 29 mV. The slope normally decreases with time. Replace the module when the slope reaches a value of approximately 23 mV.

17.4.3 If, when performing the initial calibration, there is an indication of noise or drift, refer to the troubleshooting clause of the electrode manual. There are a number of possible causes for these effects with any electrode system, and only a process of elimination normally finds the cause. Replace the sensing electrode (or module) if the analyst is unable to achieve a stability level that meets the manufacturer's specifications.

17.5 Measurements using a meter with direct concentration readout capability

17.5.1 Prepare a 10 mg/l calcium standard and a 1 mg/l calcium standard by serial dilution of the 1 g/l standard calcium solution (17.2.1).

17.5.2 Add the ISA solution [4 mol/l potassium chloride (17.2.2)] to each standard at the rate of 2 ml of ISA solution per 100 ml of standard. Add 1 ml of the acetate buffer to each standard.

17.5.3 Transfer the standards to 150 ml beakers. Place the 1 mg/l standard on the magnetic stirrer and stir the solution at a moderate rate. Place the electrodes in the standard solution.

17.5.4 Wait for a stable reading (normally 1 min to 2 min), then adjust the controls to display the calcium concentration of the standard following the procedure described in the meter instruction manual.

17.5.5 Remove the electrodes, rinse with distilled water, blot dry and place in the 10 mg/l standard solution. Stir at a moderate rate.

17.5.6 Wait for a stable reading, then adjust the controls to display the calcium concentration of the standard. The meter and electrode system are now calibrated.

17.5.7 Prepare a 1:100 dilution of each drilling fluid filtrate sample.

17.5.8 Transfer 100 ml of diluted sample to a 150 ml beaker, add 1 ml of the acetate buffer solution, add 2 ml ISA solution and stir at a moderate rate.

17.5.9 Rinse electrodes with distilled water, blot dry, immerse in the sample and wait for a stable reading. The value displayed on the meter is the calcium concentration in the diluted sample. This value, multiplied by the dilution factor (100 in this case), gives the calcium concentration in the original sample.

NOTE Concentration values above or below the concentrations of the calcium standards used for calibration are still reliable, since the response of the calcium electrode is linear from about 0,2 mg/l to above 1 g/l.

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

17.6.1 Prepare 100 mg/l, 10 mg/l and 1 mg/l calcium standards by serial dilution of the 1 g/l standard calcium solution (17.2.1).

17.6.2 Add the ISA solution [4 mol/l potassium chloride (17.2.2)] to each standard at the rate of 2 ml of ISA solution per 100 ml of standard solution. Add 1 ml of the acetate buffer solution to each standard.

17.6.3 Transfer the standards to 150 ml beakers. Place the 1 mg/l standard solution on the magnetic stirrer and stir the solution at a moderate rate. Place the electrodes in the standard solution.

17.6.4 Adjust the controls of the instrument so that the readout is in millivolts, wait until the reading is stable and record the reading.

17.6.5 Remove electrodes, rinse, blot dry and place in the 10 mg/l standard solution. When the reading is stable, record the reading.

17.6.6 Repeat step 17.6.5 with the 100 mg/l standard solution.

17.6.7 Plot the millivolt readings on the linear axis and concentration, in milligrams per litre, on the logarithmic axis of standard 3-cycle or 4-cycle semilogarithmic paper.

17.6.8 Transfer 100 ml of the sample diluted 1:100 to a 150 ml beaker, add 2 ml of the ISA solution, add 1 ml of the acetate buffer, place the electrodes in the solution and stir at a moderate rate.

17.6.9 Record the reading when stable, and determine the concentration of the diluted sample by use of the calibration curve. This value multiplied by the dilution factor gives the calcium content of the original sample.

For accurate measurements, all standards and samples should be at ambient temperature. Some magnetic stirrers generate considerable heat, and it can be necessary to place a layer of insulating material (plastic or cardboard) between the stirrer and beaker. Between measurements, rinse electrodes and blot dry with tissue. After immersion in sample or standard solutions, check the electrode end for air bubbles and remove if present. Always read and carefully follow the manufacturer's instructions regarding the care, maintenance, storage, servicing and troubleshooting of the instrument and electrodes.

18 Sodium ion content — Ion-selective electrode method

18.1 Principle

18.1.1 This procedure provides an accurate, quantitative method for determining the sodium content in water-based drilling fluids. At the present time there is no other approved field method for performing this analysis.

18.1.2 Sodium is present to some extent in all water-based fluid systems. Concentrations can range from less than 100 mg/l to over 100 g/l in saturated salt systems. This method is applicable to all concentrations. None of the other materials normally present in drilling fluids has been found to interfere under the conditions of this test. The combination of dilution and treatment with ISA solution effectively eliminates interference.

18.1.3 The sodium electrode, because of the glass membrane construction, is not as sturdy as the liquid membrane electrodes such as those used for potassium and calcium. Strict adherence to the specific practices described in the instruction manual is necessary in order to obtain reliable data.

18.1.4 The electrode should be stored in the designated storage solution only. Never expose the glass sensing membrane of the electrode to fresh water . Always rinse with the rinse solution designated in the applicable electrode manual. Never wipe or blot electrodes dry with a cloth or tissue. Let the rinse solution drain, then shake off as much as possible of the remainder.

18.1.5 Verify calibration at least every 2 h during use, using a freshly prepared standard for each recalibration.

18.1.6 Minimize the use of glass containers and apparatus and, when glass is used, ensure that it is a chemically resistant glass. Store standards and reagents in plastic containers when possible.

18.1.7 Recondition the electrode when the response reading is sluggish or a noticeable drift is detected. Be sure to follow instructions in the applicable electrode manual when reconditioning.

18.2 Reagents and apparatus

18.2.1 Sodium chloride (CAS number 7647-14-5), standard solutions[22]: 1 g/l sodium, 100 mg/l sodium, 10 mg/l sodium and 1 mg/l sodium; prepared immediately before use by serial dilution of the 10 mg/l standard or the 100 mg/l standard.

These standards are prepared by dissolving sodium chloride, NaCl, in deionized water.

18.2.2 Sodium ISA solution, $c = 4$ mol/l of both ammonium chloride, NH_4Cl, (CAS number 12125-02-9), and ammonium hydroxide, NH_4OH (CAS number 1336-21-6).

18.2.3 Electrode rinse solution, add 10 ml of ISA solution to water and dilute to 1 l.

18.2.4 Ammonium bifluoride (NH_4HF_2) (CAS number 1341-49-7), for the sodium electrode reconditioning solution, 0,1 mol/l.

18.2.5 Sodium electrode storage solution, $c = 5$ mol/l in sodium chloride, NaCl (CAS number 7647-14-5), 0,08 mol/l in ammonium chloride, NH_4Cl (CAS number 12125-02-9), and 0,08 mol/l in ammonium hydroxide, NH_4OH (CAS number 1336-21-6).

18.2.6 Reference electrode filling solution, $c = 2$ mol/l ammonium chloride, NH_4Cl (CAS number 12125-02-9), used for filling the reference electrode.

Do not use the potassium chloride filling solutions normally supplied with the reference electrode.

18.2.7 pH meter, capable of providing a millivolt readout with a resolution of at least 0,1 mV, or a combination pH/ISE meter with provisions for direct concentration readout.

Use of the combination instrument is recommended.

18.2.8 Sodium ion-selective electrode, Orion Model 84-11[23].

18.2.9 Reference electrode, Orion Model 80-03 reference electrode.

18.2.10 Magnetic stirrer, with plastic-coated stirring bars.

18.2.11 Beakers, 150 ml, made of HDPE or LDPE plastic or chemically-resistant glass (plastic is preferred).

18.2.12 Pipettes, volumetric, Class A, various sizes for preparing dilutions of standards and samples.

The use of microlitre pipettes with disposable plastic tips is recommended for measuring and transferring small volumes (1,0 ml or less).

18.2.13 Flasks, volumetric, Class A, various sizes.

18.2.14 Graph paper, 3-cycle or 4-cycle semilogarithmic paper for preparing calibration curves when using a meter that provides readouts in millivolts only.

18.3 Preparation and operational check of the electrode system

18.3.1 Remove the sodium electrode from the shipping container and rinse thoroughly with the electrode rinse solution.

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[22] Orion sodium solutions (Orion 841108, Orion 941107 and Orion 941105) are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

[23] Orion Model 84-11 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

18.3.2 Soak the electrode for at least 2 h, preferably overnight, in the sodium electrode storage solution.

18.3.3 Fill the reference electrode using the 2 mol/l ammonium chloride solution.

18.3.4 Connect electrodes to the meter and rinse with the electrode rinse solution.

18.3.5 Transfer 100 ml of the 10 mg/l sodium standard solution to a 150 ml beaker, stir solution on the magnetic stirrer at a moderate rate while adding 10 ml of the ISA solution, and then immerse the electrodes.

18.3.6 Operate the meter in the millivolts mode. When a stable reading is obtained, record the electrode potential in millivolts.

18.3.7 Remove electrodes and rinse with the electrode rinse solution. Do not wipe or blot the electrode dry. Place 100 ml of the 100 mg/l sodium standard in a 150 ml beaker, add a stirring bar and stir at a moderate rate. Add 10 ml of the ISA solution to the standard sodium solution while stirring.

18.3.8 Immerse electrodes and record the potential, in millivolts, once a stable reading is obtained.

18.3.9 The difference between the two readings is the slope of the electrode. This difference shall be in the range of 54 mV to 59 mV. If it is less than 54 mV, recondition the sodium electrode according to the procedure detailed in the electrode instruction manual.

18.4 Measurements using a meter with a direct concentration-readout capability

18.4.1 Transfer 100 ml each of the 100 mg/l sodium standard (18.2.1) and the 10 mg/l sodium standard (18.2.1) to 150 ml beakers.

18.4.2 Add 10 ml of the ISA solution (18.2.2) to each standard solution.

18.4.3 Place the 10 mg/l standard solution on the magnetic stirrer and stir at a moderate rate. Immerse electrodes and wait for a stable reading (normally 1 min to 2 min), then adjust the controls on the instrument to display the sodium concentration of the standard.

18.4.4 Remove electrodes, rinse, place in the 100 mg/l standard solution and, when a stable reading is obtained, adjust the controls to display the sodium concentration of the standard.

18.4.5 Prepare either a 1:100 or a 1:1 000 dilution with deionized or distilled water of each filtrate sample, depending on the expected sodium concentration. The sodium concentration in the diluted sample in which the measurement is made shall be between 10 mg/l and 100 mg/l.

18.4.6 Transfer 100 ml of the diluted sample to a 150 ml beaker, add 10 ml of ISA solution, immerse electrodes and wait for a stable reading. The value displayed is the sodium concentration of the diluted sample. This value multiplied by the dilution factor gives the sodium concentration in the original filtrate.

If the concentration of sodium in the diluted sample is a greater than 100 mg/l, make an additional dilution in order to provide a concentration between 10 mg/l and 100 mg/l. If it is between 1 mg/l and 10 mg/l, the value obtained is expected to be reliable since the response of the electrodes is normally linear to sodium concentrations less than 1 mg/l.

18.5 Measurements using a meter with readout in millivolts

18.5.1 Transfer 100 ml each of the 100 mg/l, 10 mg/l, and 1 mg/l sodium standard solutions to a separate 150 ml beaker.

18.5.2 Add 10 ml of ISA solution to each standard.

18.5.3 Place the 1 mg/l standard solution on the magnetic stirrer and stir at a moderate rate. Immerse electrodes, and record the reading in millivolts when stable.

18.5.4 Repeat step 18.5.3 with the 10 mg/l standard, then the 100 mg/l standard. Be sure to rinse electrodes thoroughly with water when moving from one standard solution to another.

18.5.5 Plot the millivolt readings on the linear axis and the sodium concentration, in milligrams per litre, on the logarithmic axis of 3-cycle or 4-cycle semilogarithmic paper.

18.5.6 Transfer 100 ml of sample diluted 1:100 or 1:1 000 to a separate 150 ml beaker and add 10 ml of ISA solution to each.

18.5.7 Stir at a moderate rate, immerse electrodes and record reading when stable. Determine the sodium concentration in the diluted sample by use of the calibration curve prepared in 18.5.5. This value, multiplied by the dilution factor, gives the sodium concentration of the original sample.

18.5.8 If the calculated concentration of sodium in the diluted sample is outside the 1 mg/l to 100 mg/l range, prepare another sample, using a dilution that provides a concentration within this range.

19 Density of solids — Stereopycnometer method

19.1 Principle

19.1.1 This method covers the determination of the true volume of dry powders and granular solids. When an accurately weighed sample of the solid is used, the density (mass per unit volume, normally expressed as grams per millilitre) can be calculated.

19.1.2 The method employs the Archimedes principle of fluid displacement for the volume measurement. The fluid in this case is actually a gas, preferably high-purity helium. The method is used primarily for the determination of the density of weighting materials, such as barite, but it can also be used for determining the density of drill solids and commercial clays.

19.2 Apparatus

19.2.1 **Oven**, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

19.2.2 **Desiccator**.

19.2.3 **Balance**, accuracy ± 0,05 g.

19.2.4 **Helium bottle**.

19.2.5 **Stereopycnometer** [24], calibrated and operated according to manufacturer's instructions.

19.3 Procedure — Stereopycnometer method

19.3.1 The stereopycnometer method may be used as an alternative method for determining the specific gravity of barite and haematite. In case of dispute, the results from the Le Chatelier flask method shall prevail (see ISO 13500).

19.3.2 Turn the instrument on and allow 10 min to 15 min for the pressure transducer to warm up and stabilize.

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[24] Quantachrome Stereopycnometer model SPY-2 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

19.3.3 Remove the sample cell by rotating the plastic cover on top of the sample cell holder clockwise and insert the U-shaped bayonet connector into the slots near the top of the cell to lift out the sample cell.

19.3.4 To the tared sample cell, add 150 g ± 0,05 g solid material that has been oven-dried for at least 2 h at 105 °C ± 3 °C (220 °F ± 5 °F) and cooled to room temperature in a desiccator. Record the sample mass as m_s, expressed in grams.

19.3.5 Insert sample cell in the compartment. Replace the sample cell-holder cover and rotate clockwise until metal-to-metal contact is made with the cell holder.

19.3.6 Purge the system and sample with helium by closing the “FLOW” toggle valve and opening the “CELL VENT” toggle valve. Turn selector valve to “$\mathrm{V_A}$ IN”.

19.3.7 Open “CELL, VENT CONTROL” fully in a counter-clockwise direction.

19.3.8 Close the “FLOW CONTROL” needle valve fully in a clockwise direction. Open the “FLOW” toggle valve. The “FLOW CONTROL” needle valve should be adjusted to give a slow rate of gas bubbling through water in a beaker. Remove the tubing from water.

19.3.9 After 20 min of flow, close both the “FLOW” toggle valve and the “FLOW CONTROL” needle valve. With the “CELL VENT” and “CELL VENT CONTROL” open, zero the digital display. Wait for a stable zero reading.

19.3.10 Set the selector valve to “$\mathrm{V_A}$ OUT” and close the “CELL VENT” valve slowly; otherwise the reading on the digital display can deviate from zero.

19.3.11 Open the “FLOW” toggle valve and pressurize to slightly less than 134,4 kPa (19,5 psi). Use the “FLOW CONTROL” needle valve to control the rate of pressurization. Stop the flow with the “FLOW” toggle valve.

Do not exceed a pressure of 137,9 kPa (20,0 psi). If this happens, a flashing “20” appears on the digital display and it is necessary to repeat the measurement.

19.3.12 Record display reading when stabilized.

19.3.13 Turn the selector valve to “$\mathrm{V_A}$ IN”.

19.3.14 Record the display reading when stabilized.

19.3.15 Vent pressure slowly to prevent blowing powder out of the cell by opening “CELL VENT” with the “CELL VENT CONTROL” cracked open.

19.4 Calculation — Stereopycnometer method

19.4.1 Calculate the volume, V_s, of the solid material using the equation supplied with the instrument. V_s is the calculated volume of solid material, expressed in millilitres.

19.4.2 Calculate the density, ρ, expressed in grams per millilitre, of the solid material from Equation (26):

$$\rho = \frac{m_\mathrm{s}}{V_\mathrm{s}} \tag{26}$$

where m_s is the mass of sample, expressed in grams.

20 Density of solids — Air comparison pycnometer method

20.1 Principle

20.1.1 The density of a drilling fluid weighting material is determined by the Le Chatelier flask method when it is necessary to determine conformance to ISO 13500 requirements.

20.1.2 The air comparison pycnometer may be used as an alternate method for determining density.

20.1.3 The air comparison pycnometer compares the density relative to air, expressed in g/ml.

20.1.4 In case of dispute, the results from the Le Chatelier flask method prevail.

20.2 Apparatus

20.2.1 **Oven**, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

20.2.2 **Desiccator**.

20.2.3 **Balance**, accuracy ± 0,01 g.

20.2.4 **Pycnometer** [25], air comparison, calibrated and operated according to the manufacturer's instructions.

20.3 Procedure — Air comparison pycnometer method

20.3.1 Open coupling valve and remove sample cup. Rotate both reference and measuring handwheels counter-clockwise to rest against stops.

20.3.2 Turn measuring handwheel clockwise until starting number for the instrument is set on counter.

20.3.3 To the sample cup, add 80 g ± 0,05 g of barite that has been oven-dried for at least 2 h and cooled to room temperature in a desiccator. Record the sample mass, m_s, expressed in grams.

20.3.4 Insert cup in the compartment. Lock sample cup in place by pressing the handle down firmly.

20.3.5 Wait 15 s. Close the coupling valve.

20.3.6 Turn both handwheels simultaneously or alternately until the reference handwheel rests against the stop. Keep pointer on the scale during this process.

20.3.7 Wait 10 s. Bring the pointer to "0" with the measuring handwheel.

20.3.8 Open the coupling valve. Record the counter reading to give the sample volume, V_s. Correct if necessary.

20.4 Calculation — Air comparison pycnometer method

Calculate the density, ρ, expressed in grams per millilitre, as given in Equation (27):

$$\rho = \frac{m_\mathrm{s}}{V_\mathrm{s}} \tag{27}$$

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[25] Beckman Model 930 is an example of a suitable product available commercially. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of this product.

where

m_s is the mass of sample, expressed in grams;

V_s is the volume of sample, expressed in millilitres.

21 Ageing of water-based drilling fluids

21.1 Principle

Drilling fluid ageing is the process of allowing a drilling fluid sample that contains all required ingredients and that has been subject to a period of shear to more fully develop its rheological and filtration properties through additional time for the hydration, etc. of its components. The time period needed to more fully develop properties varies from as little as several hours (more commonly overnight or 16 h) to as much as several days. The ageing can be carried out at either ambient or elevated temperatures, depending upon the system under investigation.

It is strongly recommended that this clause be read completely before any elevated-temperature ageing of drilling fluid samples is carried out.

The purposes of this clause are

a) to inform those unfamiliar with drilling fluid formulation and testing of the techniques and apparatus commonly used in a laboratory setting to mix and age drilling fluid systems, and

b) to remind those already familiar with drilling fluid formulation and testing of some basic guidelines and safety considerations.

Users of this clause should familiarize themselves with the types of tests, testing methods and properties measured presented in ISO 10414-1, as well as the methodologies given in this International Standard.

This clause is not intended to cover ageing technology appropriate to either oil-based drilling fluids or brines used for completion, packer or workover fluids.

21.2 Practices common to preparation, handling and testing over all temperature ranges

21.2.1 Water-based drilling fluids and components

Most drilling fluid formulations contain a base liquid and drilling fluid materials that are dissolved or mechanically dispersed into the liquid to form a homogeneous fluid. The resulting fluid may contain one or more of the following: water-dispersible ("soluble") polymers or resins, clays or other insoluble-but-dispersible fine solids and soluble salts. The fluids are mixed or sheared for a time sufficient to achieve a homogeneous mixture and are then set aside to "age". Ageing is done under conditions that vary from static to dynamic and from ambient to highly elevated temperatures.

21.2.2 Mixing, blending and/or shearing devices

21.2.2.1 Drilling fluid formulations are commonly mixed with various shearing devices that may be either fixed speed or variable speed. The motor turns a mixing shaft with rounded "propellers", sharp blades, wave-form shapes or others. Single-shaft or multiple-shaft shearing devices are used 26). Nozzle shear devices are also used to prepare some formulations.

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26) Examples of more widely used shearing devices available commercially are Hamilton Beach Model 936, Dispersator, Waring Blendor®, Multimixer Model 9B with 9B29 impeller, Silverson® L4R mixer, and Oliani® mixer. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

21.2.2.2 These shearing devices vary widely in the amount of shear imparted. This means that long shearing times can be required for low-shear devices to achieve complete dissolution/hydration of fluid components. High-shear devices can produce nearly completely homogenized and hydrated drilling fluid blends in a few minutes. Ageing of drilling fluid samples tends to minimize differences in properties that can result from shearing treatment.

21.2.3 Property ranges of drilling fluids or base fluids, emphasizing pH values

21.2.3.1 The pH of drilling fluid formulations containing bentonite clays usually does not fall below a value of 8,5 unless acidic materials are added to these base fluids. Except for some drilling fluid systems viscosified with certain water-soluble polymers, the pH of these formulations is usually raised above pH 8,5 with alkalinity-control agents, such as sodium or potassium hydroxide (caustic soda or caustic potash) or calcium hydroxide (lime).

CAUTION — It is strongly recommended to wear eye protection whenever drilling fluids and chemicals are formulated, handled or tested, in order to avoid injury.

21.2.3.2 Fluid alkalinity is lowered by the reaction of hydroxide groups with aluminosilicates (clays), gradually at ambient temperature and rapidly at elevated temperature. Some drilling fluid additives require that alkalinities be maintained within a certain narrow, but elevated, range in order to function at optimum levels. Therefore, pH levels are often raised after ageing if there has been a substantial pH drop.

21.2.4 Drilling fluid sample storage, disposal and test methods

21.2.4.1 Drilling fluid formulations or base fluid (bentonite slurries) are kept until used up or until properties move outside acceptable ranges. Some laboratories do not routinely keep unused portions of test drilling fluids at ambient temperatures longer than some arbitrary time, such as 1 week or 1 month. Prolonged storage is best done in refrigerated units with a maximum storage time of up to 1 year at 4 °C (40 °F).

21.2.4.2 Drilling fluid samples that contain certain organic materials or polymer products subject to fermentation (i.e. starches, biopolymers, etc.) should either have a preservative added or be discarded after a suitable time that precedes the expected onset of biological degradation.

21.2.4.3 Discard unused samples in an environmentally appropriate manner, based upon known ingredients. For example, clays, most polymers derived from celluloses and starches, and lignites pose no special threat to living organisms and may be sent to normal landfill sites.

21.2.4.4 Dispose of drilling fluids containing toxic materials as appropriate for chemical waste. The recommended test apparatus and methods for water-based drilling fluids are found in this International Standard and in ISO 10414-1.

21.3 Drilling fluid sample preparation and ageing at ambient temperature

21.3.1 Sample preparation

Drilling fluids may be mixed at ambient temperatures in a variety of open containers made from metal, plastic or glass. Little or no incompatibility exists between normal, water-based drilling fluid and these container materials under ambient temperature conditions.

21.3.2 Apparatus

21.3.2.1 Glass containers of

a) crack-resistant glass, such as home canning jars or equivalent jars or bottles;

b) heat-resistant containers or equivalent materials.

21.3.2.2 **Coverings** or **lids** for the glass vessels, commonly plastic or metal, often lined with plastic, rubber, enamel or other relatively “inert” material.

Plastic containers and lids may be made from polyethylene, polypropylene, or other suitably inert, mechanically strong and durable materials. A wide variety of metal containers may be used at ambient temperatures. Commonly used metal includes various grades of stainless steel or mild carbon steel. More inert metal formulations may be used but these are expensive for ambient temperature use.

Metal containers/cells such as aluminium bronze are not recommended for any drilling fluid formulations. Reactions between the bronze ageing cells and numerous drilling fluid systems or drilling fluid materials have been observed.

21.3.2.3 **Mixer**, in accordance with in 21.2.2.

21.3.2.4 **Balance**, accuracy ± 0,01 g.

21.3.3 Procedure for ageing at ambient temperatures

21.3.3.1 After the initial shearing/blending stages of drilling fluid base or full sample preparation, most additional ageing at ambient temperatures is done statically.

21.3.3.2 Prepared samples are left overnight (16 h) or for days, if necessary, to reach stable or desirable properties.

21.3.3.3 Rolling or tumbling (rotating) the prepared samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.

21.3.3.4 Drilling fluids are routinely aged at ambient temperatures in metal, plastic or glass containers, capped to prevent loss of moisture.

21.3.3.5 The fluid properties can continue to change until the components are fully hydrated, and some components react further in the aqueous suspension due to secondary reactions between various components or due to bacterial attack on sensitive materials. Biocides may be added to prolong shelf-life of these sensitive formulations.

21.3.3.6 Drilling fluid formulations that are kept for extended periods are usually stored under refrigeration at 4 °C (40 °F). However, drilling fluids stored at ambient temperatures are often discarded after several days and are seldom kept for longer than a month. Formulations are monitored to ensure that the properties remain within acceptable ranges.

21.4 Drilling fluid ageing at moderate temperatures [ambient to 65 °C (150 °F)]

21.4.1 Sample preparation

Drilling fluid samples to be aged at elevated temperatures are mixed at ambient temperatures in accordance with 21.2.2 and 21.3.1. Additional materials may be added to slurries that have already been aged at ambient or elevated temperatures.

21.4.2 Apparatus

21.4.2.1 **Containers**, glass, plastic or metal.

Most of the acceptable containers used for ambient ageing (21.3.1) have been found acceptable for static or dynamic ageing at temperatures up to and including 65 °C (150 °F).

21.4.2.2 **Covers**, to seal the container.

21.4.2.3 **Oven**, regulated to 65 °C (150 °F), or other suitable temperature.

21.4.2.4 Mixer, in accordance with 21.2.2.

21.4.3 Procedure for ageing at moderate temperatures

21.4.3.1 Ageing at elevated temperature is usually done for one of the following reasons:

a) to hasten the equilibrium hydration level of clays and/or polymers in the fluid system;

b) to expose the fluid to thermal conditions similar to field conditions.

21.4.3.2 For exposure to elevated temperature, drilling fluid samples are placed in one of a wide variety of commercially available or custom-built ovens. These ovens range from being tabletop, portable models capable of holding only a few one-pint samples, to large floor-standing units having very large capacities. These ovens also vary from static units to those equipped with rollers or pulley systems to roll the containers or to tumble (rotate) strapped-in cells.

21.4.3.3 Preliminary studies indicate that the methods of heating and cooling the drilling fluid samples (i.e. preheating the oven versus no preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequate air circulation), and the exact heating exposure time affect the data values measured. To optimize data repeatability between test runs, use the same heating and cooling method and heat-exposure time for all samples in a series of comparative tests.

21.4.3.4 During ageing at temperatures through 65 °C (150 °F), some glass and plastic vessels containing drilling fluids, especially those with elevated salinity (e.g. seawater with a salinity of approximately 19 g chlorides per litre) develop modest pressures in the air overlying the liquid drilling fluid. After 15 min to 30 min of heating, these vessels are removed from the oven and the lids carefully loosened to allow excess pressure to escape (“burping”), the lids restored tightly and the containers replaced in the oven to continue heating to the desired temperature. Sometimes a second check for pressure build-up is advisable.

21.4.4 Maintaining fluid properties at moderate temperatures

As mentioned in 21.2.3.2, alkalinity levels drop with time when exposed to clay (or other drilling fluid additives that exhibit weakly acidic behaviour). The reaction between clays and hydroxyl ions is accelerated with increasing temperature. Therefore, for drilling fluids that are exposed to elevated temperature, it is necessary that alkalinities be more closely monitored and, if desired, more frequently adjusted than for those at lower temperatures. Purging and blanketing samples with nitrogen gas appears to impart strong benefits in reducing oxidative degradation of polymer-treated samples. The use of nitrogen leads to better reproducibility in the testing of polymer drilling fluids.

21.4.5 Storage and testing of samples aged at moderate temperature

Even for samples aged at elevated temperature for time periods ranging from hours to days, prolonged storage of such samples, if desired, is usually done at ambient temperatures or under refrigeration, depending upon the length of time anticipated for storage.

In addition to the rheology- and filtration-measuring devices mentioned elsewhere in this International Standard and in ISO 13500 and ISO 10414-1, ambient-to-elevated-temperature dynamic-filtration and elevated-temperature rheometers are commercially available for use [27].

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[27] The Fann Model 50 Rheometer, the Fann Consistometer, the Huxley Bertram Rheometer and the Haake Rotoviscometer are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

21.5 Drilling fluid ageing at substantially elevated temperatures [over 65 °C (150 °F)]

21.5.1 Sample preparation

Drilling fluid samples for ageing at substantially elevated temperatures are also blended at ambient temperatures in accordance with 21.2.2 and 21.3.1. Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperature may be added to slurries that have already been aged at ambient or elevated temperatures.

21.5.2 Apparatus

21.5.2.1 **Oven**, capable of maintaining a temperature of 175 °C ± 3 °C (350 °F ± 5 °F) or greater.

21.5.2.2 **Ageing cells**, constructed from metals suitable for exposure to drilling fluids at elevated temperatures.

Aluminium bronze cells are not recommended for drilling fluids at any temperature.

21.5.3 Characteristics of metal ageing cells

21.5.3.1 Since ageing temperatures are often selected to be near the estimated or anticipated bottom hole temperatures, the ageing cells chosen shall be designed and constructed in such a way as to meet (or exceed) the pressure and temperature requirements of the ageing tests.

21.5.3.2 Beyond the issue of being able to meet the temperature and pressure requirements of specific ageing conditions, the choice of the proper metal is complex. It depends upon both the ultimate temperature to which the sample and cell are exposed and the salinity of the fluid sample.

21.5.3.3 The largest percentage of ageing between 65 °C and 205 °C (150 °F and 400 °F) is done in cells constructed from various grades of stainless steel. Sometimes high-carbon-content steel cells are used if there is a strong desire to simulate field conditions where “mild steel” pipe is in use and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e. they have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

21.5.3.4 For prolonged exposure to elevated salinity at high temperatures [e.g. 20 g/l chlorides at 205 °C (400 °F)], cells constructed from premium metals [28] can be desirable.

21.5.3.5 A key consideration in the use of metal ageing cells at elevated temperatures is that care shall be taken to ensure that the cells are not overfilled. When enclosed liquids expand with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as the liquid hydraulically loads the cell cap. Leaving gaps of 40 mm to 50 mm (1-9/16 in to 1-15/16 in) between the top of the liquid and the cell cap, or not filling the cell to more than 85 % to 90 % of its volume appear to be adequate precautions for safe operation during most ageing conditions between 65 °C and 205 °C (150 °F and 400 °F).

21.5.4 Additional safety considerations for metal ageing cells

21.5.4.1 It is necessary to emphasize that metal ageing cells shall be used to age only drilling fluids containing the usual types or classes of drilling fluid additives. Experimental material, when high-temperature compatibility with drilling fluid chemicals/minerals is in doubt, shall not be aged at elevated temperatures in metal cells. Such compatibility should be tested in an apparatus such as an autoclave designed for extreme-pressure service.

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[28] Hastalloy®, Inconel® 600 and Incolloy® 825 are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

21.5.4.2 The only known catastrophic failures (explosions) of stainless steel ageing cells resulted either from the cells being used inappropriately as chemical-reaction autoclaves (instead of for drilling fluid ageing) or from the cells being overfilled.

21.5.4.3 The reported types of failure of metal ageing cells, either from inadequate inspection and maintenance or from prolonged use for ageing of drilling fluids, were simply leaks that developed in the cells. The drilling fluids leaked into the ageing ovens but caused no danger to laboratory personnel.

21.5.4.4 Rupture disks may be placed in the cell caps if there is concern about cell failure. Since the use of rupture disks lowers the pressure rating of the metal ageing cell, conduct tests at temperatures that keep the pressure lower.

21.5.5 Maintenance of metal ageing cells

21.5.5.1 Cell bodies and cell caps should have serial numbers recorded whenever they are used along with the cell contents. Further, it is suggested that separate sets of metal ageing cells be maintained for testing fresh-water fluids, high-chloride fluids and oil-based fluids. The cells exposed to fresh water should have the longest useful life.

21.5.5.2 Stainless steel ageing cells tend to experience chloride stress corrosion cracking or pitting in environments where there is prolonged exposure at temperatures over 60 °C (140 °F) to fluid with substantially elevated chloride levels (over several thousand milligrams per litre). Such concerns are legitimate, but there are a number of mitigating circumstances existing in the technology of drilling fluid ageing. These tend to lessen the severity of the effects experienced.

21.5.5.3 One drilling fluids ageing practice that mitigates accelerated corrosion is the washing and visual inspection of metal ageing cells after each use to see if they have been affected by the exposure. The onset of pitting and cracking corrosion is often detected at such a time. Affected stainless steel cells are then usually sand-blasted to remove corrosion spots. The cells can be then returned to useful service.

21.5.5.4 Even when no obvious corrosion in a metal ageing cell is detected, historical experience has shown that periodic grit-blasting of the cells removes developing problem sites, keeps the surface passive and significantly increases the useful life of the cell.

21.5.5.5 Other mitigating circumstances include the following.

a) The cells are used intermittently and cleaned, and are not used in continuous-process exposures.

b) The cells used in drilling fluids testing are made from metal bar stock and are not welded. The absence of the stress point that is present in welded or cast cell bodies mitigates against the likelihood or severity of corrosion that can be experienced under certain aggressive conditions of exposure;

c) The drilling fluid formulations have a high pH and, typically, a low oxygen content.

21.5.6 Procedure for ageing at elevated temperatures

21.5.6.1 If the correct match of cell metallurgy and drilling fluid is established for a given temperature regime, then the samples may be statically or dynamically aged in a suitable oven, in accordance with 21.4.3.

21.5.6.2 Dynamic ageing should be performed in rolling or rotating ovens capable of achieving and safely maintaining temperatures of 65 °C to 205 °C (150 °F and 400 °F). A 16 h minimum ageing time is recommended for such exposures. Depending upon the temperature stability of the product(s) being evaluated, the time and temperature can need to be adjusted.

21.5.6.3 For static ageing, any qualified oven or a dynamic oven, with the rollers/rotating mechanisms switched off, may be used. Static ageing at elevated temperatures simulates the conditions of a drilling fluid that is left quiescent downhole during regular rig operations.

21.5.6.4 For high-temperature wells over 150 °C (302 °F), the usual 16 h ageing interval is a reasonable simulation of the time a drilling fluid is left in the hole during a bit trip. For longer operations, such as extended electric well-logging runs, a 48 h to 72 h ageing period is more appropriate.

21.5.6.5 In choosing a test temperature, it should be noted that fluid left in the hole can take many hours to even approach the actual bottomhole temperature, since the circulating fluid cools the wellbore. Therefore, a test temperature below the bottomhole temperature can be a more realistic simulation of downhole conditions for bit-trip periods.

NOTE A meaningful test to use in evaluating long-term gelation of heat-aged drilling fluids is the shear strength test found in ISO 10414-1.

21.6 Inertness and chemical compatibility in high-temperature ageing cells

21.6.1 Chemical compatibility of materials with metal ageing cells

21.6.1.1 As mentioned in 21.5.4, do not subject materials of unknown or suspicious reactivity to high-temperature ageing in cells containing drilling fluids.

21.6.1.2 Do not include materials that are known to produce, or suspected of initiating or taking part in, highly exothermic reactions in heat-aged drilling fluid formulations.

21.6.1.3 Exclude materials that are known or suspected strong redox (oxidation-reduction) reagents from heat-aged drilling fluid formulations.

21.6.2 Inertness of metal ageing cells to chemicals

21.6.2.1 Metal ageing cells of nickel-based alloys[29] provide the greatest degree of inertness to potentially corrosive conditions.

21.6.2.2 Iron can be leached from the various stainless steels by high-chloride fluids at high temperatures. This removal of iron causes pits and cracks after stainless steel cells are exposed to severe temperature/chloride conditions.

21.6.2.3 Various materials can be used as more “inert” liners within standard stainless steel cells. These materials are listed in 21.6.3.

21.6.3 Use of lining materials in metal ageing cells

21.6.3.1 Some investigators have found that test results from high-chloride drilling fluids containing water-soluble polymers might not be reproducible when stainless steel vessels are used. Iron appears to be leached from the cell bodies causing these anomalies.

21.6.3.2 In order to use the more economical stainless steel cells under aggressive fluid ageing conditions, lining materials may be applied in the ageing cells. Materials that have received widespread acceptance as liners include PTFE, glass and ceramic.

21.6.3.3 It is generally agreed that PTFE liners can be safely used to at least 205 °C (400 °F) and offer the highest degree of inertness to a wide variety of drilling fluid formulations.

21.6.3.4 Glass liners can be attacked at the high pH levels used in many drilling fluid formulations. Glass liners are also susceptible to attack under certain highly saline conditions at elevated temperatures.

21.6.4 Considerations regarding metal plating to enhance contamination resistance of ageing cells

21.6.4.1 Plating of stainless steel cells with inert or noble metals, such as gold, has been suggested to avoid the much greater expense of fabricating new cells from premium metal alloys.

21.6.4.2 The otherwise attractive consideration in 21.6.4.1 has one serious drawback: if the plated cell is scratched, the exposed steel experiences severe localized corrosion at accelerated rates above those of unplated steel. Therefore, the scratching of plated cells leads to a likelihood of earlier cell leaks and failures.

21.6.4.3 Since drilling fluid samples aged at elevated temperatures often become very viscous or even hard, it is sometimes necessary to scrape solidified sample material from the cells. Even when taking great care, there is a risk that scratches will occur when removing solidified drilling fluids. Therefore, gold-plated cells are not recommended for ageing drilling fluids at elevated temperatures.

21.6.5 Contrast between drilling fluid material performance in inert and real work environments

21.6.5.1 Be cautious about interpreting results from laboratory ageing studies using cells that contain inert metals or inert material liners. Drilling fluids in field applications are exposed to drill pipe, usually made from mild carbon steel.

21.6.5.2 While the amount of steel surface exposed to drilling fluids is less in steel ageing cells than that in drill pipe, it is still more than can be experienced within inert ageing cells.

21.6.5.3 Therefore, field results can be at variance with the results obtained from ageing tests conducted under the more ideal, inert conditions in the laboratory environment.

21.7 Obtaining supplies and services for the ageing of drilling fluid samples

21.7.1 Apparatus suppliers

21.7.1.1 Glass and plastic jars and bottles are widely available from laboratory supply houses, container suppliers and many retail and wholesale outlets.

21.7.1.2 Metal ageing cells, ovens suitable for ageing drilling fluids at elevated temperatures and/or drilling fluid testing apparatus and ancillary supplies can be obtained from the recognized oilfield testing equipment supply houses.

21.7.2 Metallurgical consultants

For best advice on acquiring the proper metal ageing cells required for severe service (elevated chlorides at high temperatures), contact a reputable consulting metallurgist or firm that specializes in metallurgical consulting.

21.7.3 Pressure-vessel consultants

For advice on pressure-vessel design for a situation not adequately addressed by commercially available or special-order metal ageing cells, contact a firm or individual with established credentials in pressure-vessel design.

22 Ageing of oil-based drilling fluids

22.1 Principle

This clause is written to inform those unfamiliar with formulating and testing drilling fluids about the techniques and apparatus commonly used in a laboratory to mix and age oil-based drilling fluid systems. It is also a reminder of the basic guidelines and safety considerations in working with ageing practices. It is strongly recommended that this clause be read in its entirety before any ageing of drilling fluid samples at elevated temperatures is attempted.

Drilling fluid ageing is the process in which a drilling fluid sample, previously extensively sheared, is allowed to more fully develop rheological and filtration properties. The time period to develop properties may be from 16 h to as much as several days, at either ambient or elevated temperatures.

Users of this clause should familiarize themselves with the types of tests, testing methods and properties found in ISO 10414-2, and the methodologies described in this International Standard.

This clause is not intended to cover ageing technology of water-based drilling fluids or brines used for completion, packer or workover fluids.

22.2 Apparatus

22.2.1 Shearing device[27], used to blend drilling fluid formulations.

Shearing devices may be either fixed speed or variable speed. The motor turns a mixing shaft with rounded "propellers", corrugated impellers, sharp blades or wave-form shapes, among others. Either single-shaft or multiple-shaft devices are used. Nozzle shearing devices are preferred by some users.

Shearing devices can vary widely in the amount of shear that they impart. Ageing of drilling fluid samples tends to minimize differences in properties that can result from being prepared by different shearing devices.

22.2.2 Glass jars and bottles, used to prepare batches up to 4 l in volume.

Crack-resistant glass containers, such as jars supplied for home canning purposes[29], or equivalent types of jars/bottles that are equipped with metal screw-on caps, commercially available straight-sided round jars, and wide-mouth round jars are suitable. The latter types are commonly used in rolling ovens up to about 65 °C (150 °F). All types may be securely fastened in tumbling ovens operating below 65 °C (150 °F).

Heat-resistant containers made of Pyrex or equivalent materials are also used to temperatures of up to 65 °C (150 °F).

Coverings/lids for glass vessels are made of plastic or metal. Inert seals or liners (e.g. those made of PTFE) are recommended for the best long-term use, regardless of the composition of the lid.

22.2.3 Plastic jars and containers.

Acceptable plastic materials for containers and lids include polyethylene, polypropylene or other suitably inert, mechanically strong and durable materials. These materials might not be compatible with some oil-based fluids. Most oil-based drilling fluids may be mixed and stored in thick-walled polyethylene commercial buckets.

Thin-walled plastic containers, such as milk, soft drink or juice bottles, are not recommended.

22.2.4 Metal containers.

A wide variety of metal containers may be used at ambient temperatures. The most commonly used metals are various grades of stainless steel or mild carbon steel. More inert metal formulations may be used, but these are expensive for ambient-temperature use.

Aluminium bronze cells are not recommended for any drilling fluid formulations. Electrochemical reactions between bronze ageing cells and numerous drilling fluid systems or drilling fluid materials have been observed.

Ageing-cell bodies and cell caps should have serial numbers that are logged, whenever they are used, along with the cell contents.

A concern regarding the use of stainless steel ageing cells is their tendency to experience chloride-stress corrosion cracking or pitting in environments where there has been prolonged exposure at temperatures over 60 °C (140 °F) to fluids with substantially elevated chloride levels (over several thousand milligrams per litre). This should not be a concern when testing oil-based drilling fluids in which the water-in-oil emulsion is maintained throughout the ageing process.

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29) Mason, Kerr and Ball glass containers are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Such concerns are legitimate, but there are a number of mitigating circumstances existing in the technology of drilling fluid ageing, such as the following.

a) One drilling fluid ageing practice that mitigates accelerated corrosion is the washing and visual inspection of metal ageing cells after each use to see if they have been affected by the exposure. The onset of pitting and cracking corrosion is often detected at such a time. Stainless steel cells, when the beginnings of corrosion are observed, are usually grit-blasted clean to remove corrosion spots. The cells can then be returned to useful, frequent service for as long as 25 years.

b) Even when no obvious corrosion in a metal ageing cell is detected, experience has shown that periodic grit-blasting of the cell removes developing anionic anode sites, keeps the surface passive and increases the useful life of the cell.

c) Metal ageing cells used in drilling fluids testing are made from metal bar stock and are not welded. The absence of welded stress points reduces the potential of corrosion initiation points under severe exposure conditions.

d) Many oil-based drilling fluids have a high pH in the internal water phase and a low oxygen content due to the oil phase; these are factors that reduce corrosion.

22.2.5 Liners for metal cells.

When concerns exist about reaction at elevated temperatures between a particular type of metal static-ageing cell and the contained drilling fluid, liners of material believed to be more inert to such fluids may be used.

Suitable liner materials include glass, ceramic and PTFE or equivalent. Liners of each type may be commercially obtained.

22.2.6 Ovens.

Ovens may be static or dynamic [equipped with rollers or pulley systems which provide rolling of jars or cells or tumbling (rotating) of strapped-in cells]. Static ovens range from portable (tabletop) models that can hold only a few sample jars or cells, to larger (floor-standing) multi-shelf models that can hold dozens of samples.

It is expected that ovens be rated to operate at temperatures from 65 °C to 260 °C (150 °F to 500 °F).

Adequate air circulation within ovens to ensure uniform temperature distribution is essential to the validity of ageing.

CAUTION — Ovens used with oil-based fluids should not have exposed heating elements that can cause ignition of combustible materials in the event of sample leakage.

22.3 Practices common to preparation, handling and testing over all temperature ranges

22.3.1 Oil-based drilling fluids and their components

22.3.1.1 Oil-based drilling fluids contain one or more base liquids and drilling fluid materials dissolved or mechanically dispersed into the liquid to form a homogeneous fluid. The resulting fluid may contain oil-dispersible, soluble polymers or resins, and organophilic clays or other insoluble but dispersible fine solids.

22.3.1.2 The oil-based drilling fluids may be “all-oil” drilling fluids, i.e. formulated without an aqueous phase, or “invert” oil-based emulsion where the oil is the continuous phase and the emulsified water phase is the dispersed phase. Typically the water phase contains sodium chloride (NaCl) or calcium chloride ($CaCl_2$).

22.3.1.3 Fluids are mixed or sheared to achieve a homogeneous mixture of the components and then set aside to “age”. Ageing may be done under static or dynamic conditions and from ambient to elevated temperatures.

22.3.2 Mixing/blending/shearing devices

Shearing devices vary widely in the amount of shear that they impart. Long shearing times can be required for low-shear devices to achieve fairly complete dissolution or solvation of fluid components. High-shear devices can produce nearly completely dispersed blends in a few minutes.

Ageing of drilling fluids samples tends to minimize property differences that can result from using different shearing devices.

22.3.3 Property ranges of oil-based drilling fluids or their base fluids

22.3.3.1 The base oils vary widely in their inherent viscosities. These viscosities also vary differently with temperature for different base fluids. Therefore, the drilling fluid properties vary widely.

22.3.3.2 Drilling properties can also be affected by the presence of different emulsifier packages (or the lack of them for all-oil fluids), and/or organophilic clays and/or the oil-soluble viscosifying polymers used to provide suspending characteristics.

22.3.3.3 Some emulsifiers or wetting agents require the presence of lime to be activated into calcium soaps. Sometimes lime is also added to control acid gases, such as hydrogen sulfide or carbon dioxide, which can be encountered in drilling operations or from the high-temperature breakdown of some drilling fluid materials.

22.3.4 Sample storage and disposal

22.3.4.1 Oil-based drilling fluid samples are usually not subject to deterioration with time. However, it is considered good practice to limit testing reliance on samples that are over 1 month old, unless the sample has been refrigerated at 4 °C (40 °F).

22.3.4.2 Unused samples should be discarded in accordance with local statutory regulations based upon their known ingredients.

22.4 Drilling fluid ageing at ambient temperatures

22.4.1 Containers for preparation and storage

The drilling fluids may be mixed at ambient temperature in a variety of open containers made from metal, plastic or glass, in accordance with 22.2.2, 22.2.3 and 22.2.4. Incompatibility can exist between oil-based drilling fluids and some plastic materials. There are not likely to be problems with metal or glass containers.

22.4.2 Procedure for ageing at ambient temperatures

22.4.2.1 After the initial shearing/blending stages of drilling fluid base or full sample preparation, most additional ageing at ambient temperatures is done statically.

22.4.2.2 Prepared samples are left overnight (16 h) or for days, if necessary, to reach stable or desirable properties.

22.4.2.3 Rolling or tumbling (rotating) of the prepared samples may be used to combat settling of solid components or segregation of liquid layers, but this is seldom done at ambient temperatures.

22.4.2.4 Oil-based drilling fluids are routinely aged at ambient temperatures in metal or glass containers that are capped to prevent loss of organic vapours. The fluid properties are expected to change very little from this point on.

22.4.3 Storage

22.4.3.1 Drilling fluids to be kept for extended periods are usually stored under refrigeration at 4 °C (40 °F).

22.4.3.2 Drilling fluids stored at ambient temperatures are often discarded after several days and are seldom kept for longer than a month.

22.4.3.3 Fluid samples are monitored to ensure that acceptable properties are maintained.

22.5 Drilling fluid ageing at moderate temperatures [ambient to 65 °C (150 °F)]

22.5.1 Sample preparation

22.5.1.1 Oil-based drilling fluid samples to be aged at elevated temperatures are usually blended at ambient temperatures, in accordance with 22.3.2 and 22.4.

22.5.1.2 Additional materials may be added to slurries that have already been aged at ambient or elevated temperatures.

22.5.1.3 Most of the acceptable glass and metal containers used for ambient-temperature ageing (see 22.3.2 and 22.4) have been found to be acceptable for static or dynamic ageing up to 65 °C (150 °F).

22.5.2 Procedure for ageing at moderate temperatures

22.5.2.1 Ageing of drilling fluids at temperatures above ambient is usually done for one of the following reasons:

a) to speed up the chemical equilibrium of the system;

b) to expose the fluid to thermal conditions similar to the field conditions.

22.5.2.2 For exposure to temperatures above ambient, drilling fluid samples are placed in any one of a variety of commercially available or custom-built ovens, in accordance with 22.2.6.

22.5.2.3 The methods of heating and cooling samples (i.e. preheating the oven versus not preheating, cooling samples in open or closed ovens or in water), uniformity of temperature throughout the oven (due to the degree of adequacy of air circulation) and exact heat exposure time can affect the data measured.

22.5.2.4 To optimize data repeatability between test runs, the same heating and cooling methods and heat exposure times shall be employed for all samples in a series of comparative tests.

22.5.3 Maintaining fluid properties at moderate temperatures

Purging and blanketing samples with nitrogen gas appears to have strong benefits in reducing oxidative degradation of polymer-treated samples. This use of nitrogen leads to better reproducibility.

22.5.4 Storage and testing of samples aged at moderate temperatures

Storage of samples is usually done at ambient temperatures or under refrigeration, depending upon the length of time anticipated for storage, even though the samples have been aged at elevated temperatures for time periods ranging from hours to days.

In addition to the rheology- and filtration-measuring devices mentioned in ISO 13500, ISO 10414-2 and this International Standard, ambient-to-elevated-temperature dynamic-filtration rheometers and elevated-temperature rheometers are commercially available for use [30].

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[30] Fann® Model 50C Viscometer, Fann Model 5STDL Consistometer, Fann Model 70 viscometer, EG&G Chandler® Engineering Model 7400 Rheometer, and Haake® RV20/D100 Viscometer are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

22.6 Drilling fluid ageing at substantially elevated temperatures [over 65 °C (150 °F)]

22.6.1 Sample preparation

22.6.1.1 Drilling fluid samples for ageing at substantially elevated temperatures are usually blended at ambient temperatures in accordance with 22.3.2 and 22.4.

22.6.1.2 Additional quantities of pre-existing components or new materials designed to increase stability at elevated temperatures may be added to slurries that have already been aged at ambient or elevated temperatures.

22.6.1.3 Only ageing cells constructed from suitable metals shall be used at temperatures over 65 °C (150 °F), as stated in 22.6.2.

22.6.2 Choice of ageing cell and temperature

22.6.2.1 Ageing temperatures are often selected to approximate the anticipated bottomhole temperature and for applications where specific systems or drilling fluid materials are used. Ageing cells shall be constructed to meet or exceed the pressure and temperature requirements of the ageing test parameters. In addition, the choice of the proper metal depends on the ultimate temperature to which the sample and cell are exposed and the chloride content of the aqueous phase in the oil-based fluid sample.

22.6.2.2 By far, the ageing of most drilling fluids between 65 °C and 190 °C (150 °F and 375 °F) is done in cells constructed from various grades of stainless steel. Sometimes high-carbon-content steel cells are used when the intent is to simulate field conditions where “mild steel” pipe is in use and to which drilling fluids are naturally exposed. These stainless or mild steel cells, when properly used, have never experienced catastrophic failure, i.e. they have not exploded upon failure. When these cells have failed, they have simply sprung leaks.

22.6.2.3 For prolonged exposure of ageing cells to elevated salinity at high temperatures, cells constructed from premium metals 29) can be desirable.

22.6.2.4 A key consideration in the use of metal ageing cells at elevated temperatures is that care should be taken to not overfill the cells. When enclosed liquid expands with increasing temperature, an inadequate head space of gas (air) can lead to a piston effect as the liquid hydraulically loads the cell cap. Leaving a gap of 40 mm to 50 mm (1-9/16 in to 1-15/16 in) between the liquid and the cell cap (or not filling the cell to over 85 % to 90 % of its volume) appears to be adequate precaution for safe operation under ageing conditions between 65 °C and 205 °C (150 °F and 400 °F).

22.6.3 Procedure for ageing at elevated temperatures

22.6.3.1 If the correct match of cell metallurgy and contained drilling fluid has been established for a given elevated temperature, the sample may be statically or dynamically aged in an oven in accordance with 22.5.2.

22.6.3.2 Dynamic ageing shall be performed in a rolling or rotating oven capable of achieving and safely maintaining temperatures of 65 °C to 205 °C (150 °F and 400 °F). A 16 h minimum ageing time is usually recommended. Depending upon the temperature stability of the product(s) being evaluated, the time and temperature may be adjusted.

22.6.3.3 For static ageing, any qualified oven or a dynamic oven with the rollers/rotating mechanisms switched off may be used. Static ageing at elevated temperatures simulates the conditions of a drilling fluid that is left quiescent downhole during regular rig operations.

22.6.3.4 For high-temperature wells over 135 °C (275 °F), the usual 16 h ageing interval is a reasonable simulation of the time a drilling fluid is left in the hole during a bit trip. For longer operations, such as extended electric well-logging runs, a 48 h to 72 h ageing period is more appropriate.

22.6.3.5 In choosing a test temperature, it should be noted that fluid left in the hole can take many hours to approach the actual bottomhole temperature, since wellbores are cooled by the circulating fluid. Therefore, a test temperature below the bottomhole temperature may be a more realistic simulation of downhole conditions for bit-trip periods.

NOTE A meaningful test to use in evaluating long-term gelation of heat-aged drilling fluids is the shear strength test found in ISO 10414-2:—, Annex B. This is especially useful for oil-based drilling fluids considered for use as packer fluids.

22.6.4 Safety considerations for metal ageing cells

22.6.4.1 Metal ageing cells shall be used only to age drilling fluids containing known, common drilling fluid materials.

22.6.4.2 Experimental material, where high-temperature compatibility with the usual chemicals/minerals is in doubt, shall not be aged at elevated temperatures in metal cells. Such compatibility should be tested in an apparatus such as an autoclave designed for extreme-pressure service.

22.6.4.3 The only known catastrophic failures (explosions) of stainless steel ageing cells resulted either from the cells being used inappropriately as chemical-reaction autoclaves (instead of for drilling fluid ageing) or from the cells being overfilled.

22.6.4.4 The types of failures of metal ageing cells resulting from inadequate inspection and maintenance are simply leaks in the cells.

22.6.4.5 Rupture disks may be placed in the cell caps if there is a concern about cell failure. Since the use of rupture disks essentially lowers the pressure rating of the ageing cell, conduct ageing at temperatures that keep the pressure lower.

CAUTION — Ovens used with oil-based fluids shall not have exposed heating elements which could cause ignition of combustible materials in the event of sample leakage. This is especially critical if cells with rupture disks are used.

22.7 Inertness and chemical compatibility in high-temperature ageing cells

22.7.1 Chemical compatibility of materials with metal ageing cells

Do not include materials that are known to produce, or suspected of initiating or taking part in, highly exothermic reactions in heat-aged drilling fluid formulations.

22.7.2 Inertness of metal ageing cells to chemicals

22.7.2.1 Metal ageing cells of nickel-based alloys[29] provide the greatest degree of inertness to potentially corrosive environments.

22.7.2.2 Iron can be leached from the various stainless steels by high-chloride fluids at high temperatures. This removal of iron leads to the pits and cracks seen after exposing stainless steel cells to severe temperature/chloride conditions.

22.7.2.3 Various materials can be used as more “inert” liners in standard stainless steel cells. These are listed in 22.7.3 below.

22.7.3 Considerations regarding metal plating to enhance contamination resistance of ageing cells

22.7.3.1 Gold-plated cells are not recommended for ageing drilling fluids at elevated temperatures. Plating stainless steel cells with inert or noble metals, such as gold, has been suggested as a means of avoiding the greater expense of fabricating new cells from the premium metal alloys. This attractive consideration has one serious drawback: if the plating is scratched, the exposed steel is prone to accelerated, more severe localized corrosion rates than unplated steel experiences. Scratching the plating can lead to earlier cell leaks and failures.

22.7.3.2 Since drilling fluid samples aged at elevated temperatures often become very viscous or even hard, scraping solidified samples from the cells is often required. Even when great care is taken, there is a risk that scratches will occur when removing solidified drilling fluids from ageing cells.

22.7.4 Contrast between drilling fluid material performance in inert and real work environments

22.7.4.1 Be cautious about interpreting results from laboratory ageing studies using cells made from inert metal or containing inert material liners. In field applications, the drilling fluids are exposed to the steel in drill pipe, which is usually made from mild carbon steel.

22.7.4.2 Field results can differ from the laboratory results obtained under more ideal conditions. While the amount of steel surface exposed to drilling fluids is much less in steel ageing cells than in drill pipe, it is still more than can be expected within inert ageing cells.

22.8 Obtaining supplies and services for the ageing of drilling fluid samples

22.8.1 Apparatus suppliers

22.8.1.1 Glass and plastic jars and bottles are widely available from laboratory supply houses, container suppliers, and many retail and wholesale outlets.

22.8.1.2 Metal ageing cells, ovens suitable for ageing drilling fluids at elevated temperatures and/or drilling fluids testing apparatus and ancillary supplies may be obtained from companies who specialise in drilling fluid testing apparatus.

22.8.2 Metallurgical consultants

For the best advice on acquiring the proper metal ageing cells required for severe service (elevated chloride content at high temperatures), contact a reputable metallurgist or firm that specializes in metallurgical consulting.

22.8.3 Pressure-vessel consultants

For advice on pressure-vessel design, if a situation is expected that might not be adequately addressed by commercially available or special-order metal ageing cells, contact a firm or individual with established credentials in pressure-vessel design.

23 Shale-particle disintegration test by hot rolling

23.1 Principle

23.1.1 This procedure involves grinding, sieving and placing a known mass of shale particles into a drilling fluid and ageing, either statically or dynamically with hot rolling, the fluid with shale particles. Retrieving, cleaning and reweighing the particles, to determine mass loss, indicates the tendency of the fluid to prevent dispersion of the shale into the fluid. This test is only a relative measure and should be included as part of a comprehensive testing programme.

23.1.2 The ability of a drilling fluid to inhibit the disintegration of shale cuttings is important. The laboratory method presented in this clause can be used for evaluating the properties of drilling fluids with regard to inhibiting shale-particle disintegration. This test is intended as a comparison of various whole drilling fluid compositions.

23.1.3 There are many laboratory techniques used to assess the degree to which a drilling fluid formulation can cause a shale to weaken and disintegrate when immersed in a fluid. The present method, although commonly used, is not the only test that indicates the ability of drilling fluids to inhibit shale disintegration. This test can give a wide variability of results. Strict control and reporting of the condition of the test shale, the rheology of the test fluid and the washing of shale are all necessary.

23.1.4 Shale selection and condition (e.g. water content), while extremely important, are neither specified nor limited by the procedure. Properly preserved shale samples should be used. It is strongly recommended that the shale be as near to its *in situ* moisture content as possible, and not having been previously air-dried or oven-dried. The way the shale was preserved prior to its use in the test should be recorded with the results.

23.1.5 Drilling fluid rheology is one variable that has proven difficult to control from test to test. As this can have an independent influence on shale recovery from the drilling fluid, it is recommended that the rheological properties be measured at the test temperature and be recorded with the results. This enables consideration of the effect of the rheology along with the other variables. Modest changes in the rheology from one fluid to another can strongly influence shale dispersion final results.

23.1.6 It should be noted that the results do not necessarily imply wellbore stability. This test might not be appropriate for evaluating the inhibiting properties of certain drilling fluid materials.

23.2 Reagents and apparatus

23.2.1 **Wash water**, inhibitive, that is either

a) a synthetic version of the drilling fluid base brine, or

b) a fluid known to inhibit the disintegration of the shale used [e.g. 42,75 kg/m^3 potassium chloride].

NOTE Wash water of salinity much greater than the drilling fluid base brine can cause sieve blocking due to drilling fluid coagulation.

23.2.2 **Supply**, for inhibitive wash water, at a flow rate of 2 l/min ± 0,2 l/min with an outlet, d, of approximately 7,5 mm (1/3 in).

23.2.3 **Balance**, accuracy ± 0,01 g.

23.2.4 **Oven**, roller, capable of achieving, typically, 17 r/min to 35 r/min.

For best results, all tests should be conducted with the same roller oven to ensure constant test conditions.

23.2.5 **Ageing cells**, not made from aluminium bronze.

23.2.6 **Clock** or **timer**, accurate to 0,1 min over the test interval.

23.2.7 **Weighing boats**.

23.2.8 **Spatula**.

23.2.9 **Wash bath** or **sink**, at least 2 l in size.

23.2.10 **Sieves**, 10 cm (4 in) in diameter with mesh sizes of 0,5 mm (0,02 in) (U.S. Sieve number 35), 4 mm (0,16 in) (U.S. Sieve number 5) and 2 mm (0,08 in) (U.S. Sieve number 10).

23.2.11 **Sieve base**, 10 cm (4 in) diameter.

23.2.12 **Drying oven**, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

23.2.13 **Desiccator**.

23.3 Procedure

23.3.1 Obtain shale pieces with moisture content as near to that *in situ* as possible. Sieve shale pieces just prior to use to fractions of less than 4 mm (0,16 in) and greater than 2 mm (0,08 in). The moisture content of bulk shale supply shall be established at the time of sampling by the method for bentonite in accordance with ISO 13500:—, Clause 9, and recorded.

23.3.2 Pre-screen the drilling fluid through a 0,5 mm (0,02 in) sieve to remove any oversize particles that can be incorrectly reported as recovered shale.

23.3.3 Add 350 ml of drilling fluid to each ageing cell, measured either by volume or by mass.

23.3.4 Add 20,0 g of shale pieces (screened prior to use) to the fluid in each ageing cell, seal and quickly agitate gently to wet and separate the particles.

NOTE With high-viscosity drilling fluids, it can be advantageous to add the drilling fluid and the shale simultaneously to achieve separate wetting of particles or gently stir the drilling fluid with a spatula while adding shale.

23.3.5 Place the ageing cells in a roller oven preheated to the desired temperature and roll for a predetermined period of time.

The rolling time should be the same for all fluids being evaluated for a given shale. The rolling time is dependent on the shale and some shales require more rolling time than others for sufficient disintegration to occur to permit discrimination among fluids.

23.3.6 After the rolling period is complete and the ageing cells have cooled to a safe handling temperature, transfer to a static position to cool further to a temperature that is constant for all sequential recovery operations (e.g. from a cold-water bath to room temperature).

23.3.7 Pour the contents of each ageing cell onto a numbered pre-weighed 0,5 mm (0,02 in) mesh sieve while applying inhibitive wash water (23.2.1) to the screen to prevent viscous blocking. Also displace the residual contents of the ageing cell onto the sieve with wash water (the use of a wash bottle is convenient).

NOTE Viscous blocking, if it occurs, can be alleviated by applying a slight pressure with a stream of wash water above the sieve.

23.3.8 Wash the drilling fluid from the remaining shale pieces using a wash-water flow rate of 2 l/min ± 0,2 l/min through a hose with a circular outlet approximately 7,5 mm (0,3 in) in diameter, systematically passing across the full area of the screen many times for a period of approximately 1 min or until the shale and screen are visually free of drilling fluid residue.

23.3.9 Transfer the sieve to a 2 l bath containing tap water and quickly but gently submerge the sieve (at an angle to avoid an air trap) and remove it so that the sieve and shale have been rinsed of wash water.

23.3.10 Place the sieve onto a pre-weighed sieve base and dry in the drying oven to a constant mass. Cool in a desiccator and reweigh to 0,01 g. This is the final dry mass.

23.4 Calculation

23.4.1 The mass fraction of shale recovered (mass residue), w_r, expressed in percent, is given by Equation (28):

$$w_\mathrm{r} = 100 \frac{m_\mathrm{d}}{m_\mathrm{o}} \tag{28}$$

where

m_d is the mass of dry recovered shale, in grams;

m_o is the mass of the initial shale sample, corrected for moisture content, in grams.

In this example, which uses an initial shale sample of 20,0 g, the mass of dry recovered shale is calculated as given in Equation (29):

$$m_\mathrm{d} = 20\ (100 - w_\mathrm{h})/100 \tag{29}$$

where w_h is the moisture content of initial shale, expressed as a mass fraction, in percent.

23.4.2 Record conditions used (shale preservation technique, history prior to test, initial moisture content, drilling fluid rheology, test temperature and rolling time) and the shale mass recovered. Replication of the test is recommended. Differences between test values are typically less than 4 %.

24 Drilling fluid materials — High-viscosity polyanionic cellulose (PAC-HV) (regular)

24.1 Principle

23.1.1 PAC-HV is a water-soluble polymer produced only from cellulose chemically reacted with carboxymethyl (anionic) groups and does not contain any other polysaccharides, such as starch, guar or other naturally occurring polymers or their derivatives. The material is a free-flowing or granular powder.

24.1.2 PAC-HV is widely used in water-based drilling fluids for a variety of applications, such as filtration control, viscosity control and inhibition. Although field use can vary, this procedure focuses on viscosity and filtration-control characteristics.

24.1.3 The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-HV. Specification parameters have not yet been developed for this material.

24.1.4 A synthetic-seawater drilling fluid is used for determining the filtration control and apparent viscosity of PAC-HV.

24.2 Determination of moisture content

24.2.1 Apparatus

24.2.1.1 **Oven**, regulated to 105 °C ± 3 °C (220 °F ± 5 °F).

24.2.1.2 **Balance**, accuracy ± 0,01 g.

24.2.1.3 **Dish**, evaporating.

24.2.1.4 **Spatula**.

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

24.2.2 Procedure

24.2.2.1 Weigh 10 g ± 0,1 g PAC-HV sample into a tared evaporating dish.

24.2.2.2 Dry the sample in the oven (24.2.1.1) for 4 h. Cool to room temperature in a desiccator.

24.2.2.3 Reweigh the evaporating dish containing the dried sample. Record the mass, m_d, of the dry sample.

24.2.3 Calculation of PAC-HV moisture content

Calculate the mass fraction moisture, w_h, expressed in percent, of the PAC-HV sample as given in Equation (30):

$$w_h = 100 \frac{m_o - m_d}{m_o} \qquad (30)$$

where

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

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

24.3 Procedures with test fluid containing PAC-HV

24.3.1 Reagents and apparatus

24.3.1.1 Stock salt solution No. 1, in accordance with ASTM D 1141: 555,6 g magnesium chloride hexahydrate (CAS number 7791-18-6), 57,9 g anhydrous calcium chloride (CAS number 10043-52-4) and 2,1 g strontium chloride hexahydrate (CAS number 10025-70-4) diluted to 1 l with distilled or deionized water.

24.3.1.2 Stock salt solution No. 2, in accordance with ASTM D 1141: 69,5 g potassium chloride (CAS number 7447-40-7), 20,1 g sodium bicarbonate (CAS number 144-55-8), 10,0 g potassium bromide (CAS number 7758-02-3), 2,7 g boric acid (CAS number 10043-35-3) and 0,3 g sodium fluoride (CAS number 7681-49-4) diluted to 1 l with distilled or deionized water.

24.3.1.3 Sodium chloride (CAS number 7647-14-5), ACS reagent grade.

24.3.1.4 Sodium sulfate, andydrous (CAS number 7757-82-6), ACS reagent grade.

24.3.1.5 Standard evaluation base clay, such as provided by API or equivalent.

24.3.1.6 Potassium chloride (CAS number 7447-40-7).

24.3.1.7 Sodium bicarbonate (CAS number 144-55-8).

24.3.1.8 Deionized or **distilled water**.

24.3.1.9 PAC-HV, for testing, of known moisture content (see 24.2).

24.3.1.10 Thermometer, with a scale of 0 °C to 60 °C, accurate to ± 1 °C (32 °F to 150 °F, accurate to ± 2 °F).

24.3.1.11 Balance, accuracy ± 0,01 g.

24.3.1.12 Mixer, capable of maintaining 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 approximately 10 % mass loss occurs. Original blade mass is about 5,5 g. Each spindle shall be fitted with a single sine-wave impeller mounted flash-side up.

24.3.1.13 Container, for mixing, approximately 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 bottom 5).

24.3.1.14 Spatula.

24.3.1.15 Container, glass or plastic, with stopper or lid for salt solutions.

24.3.1.16 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

24.3.1.17 Timers, two, mechanical or electrical, accurate to 0,1 min over the test interval.

24.3.1.18 Filter press, in accordance with ISO 10414-1.

24.3.1.19 Cylinders, graduated, one 10 ml ± 0,1 ml and one 500 ml ± 5 ml.

24.3.2 Measurement of test fluid filtrate volume

24.3.2.1 Prepare simulated seawater by adding 24,53 g sodium chloride and 4,09 g anhydrous sodium sulfate to a 1 l flask. Dilute to 800 ml with distilled or deionized water. Pour 20,0 ml stock salt solution No. 1 and 10,0 ml of stock salt solution No. 2 into a 1 l flask and dilute to 1 l with water while stirring. Adjust the pH to 8,2 with a 0,1 mol/l sodium hydroxide solution.

24.3.2.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g potassium chloride (KCl).

24.3.2.3 After stirring for 3 min ± 0,1 min, add 1,0 g ± 0,01 g sodium bicarbonate.

24.3.2.4 After stirring for 3 min ± 0,1 min, add 28,0 g ± 0,01 g of standard evaluation base clay.

24.3.2.5 After stirring for 5 min ± 0,1 min, remove the container from the mixer and scrape the sides with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.

24.3.2.6 Replace the container on the mixer and continue to stir for an additional 5 min ± 0,1 min.

24.3.2.7 Weigh 1,0 g ± 0,01 g PAC-HV (24.3.1.9). Add the PAC-HV slowly at a uniform rate over a time interval of about 60 s while stirring the suspension on the mixer. Add the PAC-HV away from the impeller shaft, but in the vortex, to minimize dusting.

24.3.2.8 After stirring for 5 min ± 0,1 min, remove container from the mixer and scrape the sides with the spatula to dislodge any PAC-HV adhering to the container walls. Be sure all PAC-HV clinging to the spatula is incorporated into the suspension.

24.3.2.9 Replace the container on the mixer and continue to stir. It can be necessary to remove the container from the mixer and scrape the sides to dislodge any adhering PAC-HV after another 5 min or 10 min. Total mixing time elapsed from the beginning of PAC-HV addition shall equal 20 min ± 1 min.

24.3.2.10 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 3 °F). Record storage temperature and storage duration.

24.3.2.11 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

24.3.2.12 Pour the PAC-HV suspension into a filter-press cell. Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 3 °F). Pour the suspension to within about 13 mm (1/2 in) of the top of the cell. Complete the assembly of the filter-press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

24.3.2.13 Set one timer for 7,5 min ± 0,1 min, and the second timer for 30 min ± 0,1 min. Start both timers and adjust the pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Pressure shall be supplied by compressed air, nitrogen or helium, and applied within 15 s.

24.3.2.14 After 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering on the drain tube and discard. Place a dry 10 ml graduated cylinder under the drain tube and continue collecting filtrate until the end of the 30 min period as measured by the second timer. Remove the graduated cylinder and record the volume of filtrate collected. This filtrate volume represents the volume collected in the time interval from 7,5 min to 30 min.

24.3.3 Calculation of corrected test fluid filtrate volume

Calculate V_f, the corrected volume of filtrate, expressed in millilitres, using Equation (31):

$$V_f = 2 V_c \tag{31}$$

where V_c is the volume of filtrate, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated value.

24.3.4 Measurement of test fluid viscosity

24.3.4.1 Add 42 g ± 0,01 g of sea salt to 1 l ± 2 ml of deionized water.

24.3.4.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g of potassium chloride (KCl).

24.3.4.3 Weigh 3,0 g ± 0,01 g of PAC-HV. Add the PAC-HV slowly at a uniform rate over a time interval of about 60 s while stirring on the mixer. Add the PAC-HV away from the impeller shaft, but in the vortex to minimize dusting.

24.3.4.4 After stirring for 5 min ± 0,1 min, remove the container from the mixer and scrape the sides with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.

24.3.4.5 Replace the container on the mixer and continue to stir. It can be necessary to remove the container from the mixer and scrape the sides to dislodge any adhering PAC-HV after another 5 min or 10 min. Total mixing time elapsed from beginning of the PAC-HV addition shall equal 20 min ± 1 min.

24.3.4.6 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 3 °F). Record storage temperature and storage duration.

24.3.4.7 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

24.3.4.8 Pour the suspension into the viscometer cup provided with the direct-indicating viscometer. The dial reading at the 600 r/min rotor speed setting of the viscometer shall be taken at a suspension test temperature of 25 °C ± 1 °C (77 °F ± 3 °F). Record the 600 r/min dial reading.

24.3.5 Calculation of test-fluid apparent viscosity

Calculate η_A, the apparent viscosity, using Equation (32).

$$\eta_A = \frac{\eta_{600}}{2} \tag{32}$$

where η_{600} is the viscosity reading at 600 r/min, expressed in millipascal seconds.

NOTE 1 cP = 1 mPa·s.

Record the calculated value.

25 Drilling fluid materials — Low-viscosity polyanionic cellulose (PAC-LV)

25.1 Principle

25.1.1 PAC-LV is a water-soluble polymer produced only from cellulose chemically reacted with anionic groups and does not contain any other polysaccharides, such as starch, guar or other naturally occurring polymers or their derivatives. The material is a free-flowing or granular powder.

25.1.2 PAC-LV is widely used in water-based drilling fluids for a variety of applications, such as filtration control, viscosity control and inhibition. Although field use can vary, this procedure focuses on viscosity- and filtration-control characteristics.

25.1.3 The intention of this procedure is to present a simple and reproducible method for assessing the performance properties of PAC-LV. Specification parameters have not yet been developed for this material.

25.1.4 A synthetic-seawater drilling fluid is used for determining the viscosity and filtration control properties of PAC-LV.

25.2 Determination of moisture content

25.2.1 Apparatus

25.2.1.1 **Oven**, regulated to 25 °C ± 1 °C (77 °F ± 3 °F).

25.2.1.2 **Balance**, accuracy ± 0,01 g.

25.2.1.3 **Dish**, evaporating.

25.2.1.4 **Spatula**.

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

25.2.2 Procedure

25.2.2.1 Weigh 10 g ± 0,1 g of the PAC-LV sample into a tared evaporating dish.

25.2.2.2 Dry the sample in the oven (25.2.1.1) for 4 h. Cool to room temperature in the desiccator.

25.2.2.3 Re-weigh the evaporating dish containing the dried sample. Record the mass, m_{d}, of the dry sample.

25.2.3 Calculation of moisture content

Calculate the mass fraction moisture, w_{h}, expressed in percent, of the PAC-LV sample, using Equation (33):

$$w_{\mathrm{h}} = 100\frac{m_{\mathrm{o}} - m_{\mathrm{d}}}{m_{\mathrm{o}}} \tag{33}$$

where

m_{o} is the mass of original sample, expressed in grams;

m_{d} is the mass of dry sample, expressed in grams.

25.3 Procedures with test fluid containing PAC-LV

25.3.1 Reagents and apparatus

25.3.1.1 **Stock salt solution No. 1**, in accordance with ASTM D 1141: 555,6 g magnesium chloride hexahydrate (CAS number 7791-18-6), 57,9 g anhydrous calcium chloride (CAS number 10043-52-4) and 2,1 g strontium chloride hexahydrate (CAS number 10025-70-4) diluted to 1 l with distilled or deionized water.

25.3.1.2 **Stock salt solution No. 2**, in accordance with ASTM D 1141: 69,5 g potassium chloride (CAS number 7447-40-7), 20,1 g sodium bicarbonate (CAS number 144-55-8), 10,0 g potassium bromide (CAS number 7758-02-3), 2,7 g boric acid (CAS number 10043-35-3) and 0,3 g sodium fluoride (CAS number 7681-49-4) diluted to 1 l with distilled or deionized water.

25.3.1.3 **Sodium chloride** (CAS number 7647-14-5), ACS reagent grade.

25.3.1.4 **Sodium sulfate**, andydrous (CAS number 7757-82-6), ACS reagent grade.

25.3.1.5 Standard evaluation base clay, such as provided by API or equivalent.

25.3.1.6 Potassium chloride (CAS number 7447-40-7).

25.3.1.7 Sodium bicarbonate (CAS number 144-55-8).

25.3.1.8 Distilled-deionized water.

25.3.1.9 PAC-LV, for testing, of known moisture content (see 25.2).

25.3.1.10 Thermometer, with a scale reading 0 °C to 60 °C, accurate to ± 3 °C (32 °F to 150 °F, accurate to ± 5 °F).

25.3.1.11 Balance, accuracy ± 0,01 g.

25.3.1.12 Mixer, capable of maintaining 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 mass of the blade is about 5,5 g. Each spindle shall be fitted with a single sine-wave impeller, mounted flash-side up.

25.3.1.13 Container, for mixing, approximately 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).

25.3.1.14 Spatula.

25.3.1.15 Container, glass or plastic, with a stopper or lid, for salt solutions.

25.3.1.16 Viscometer, motor-driven, direct-indicating, in accordance with ISO 10414-1.

25.3.1.17 Timers, two, mechanical or electrical, accurate to 0,1 min over the test interval.

25.3.1.18 Filter press, in accordance with ISO 10414-1.

25.3.1.19 Cylinders, graduated, one 10 ml ± 0,1 ml and one 500 ml ± 5 ml.

25.3.2 Measurement of test-fluid filtrate volume

25.3.2.1 Prepare simulated seawater by adding 24,53 g sodium chloride and 4,09 g anhydrous sodium sulfate to a 1 l flask. Dilute to 800 ml with distilled or deionized water. Pour 20,0 ml stock salt solution No. 1 and 10,0 ml of stock salt solution No. 2 into a flask and dilute to 1 l with water while stirring. Adjust the pH to 8,2 with a 0,1 mol/l sodium hydroxide solution.

25.3.2.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g potassium chloride (KCl).

25.3.2.3 After stirring for 3 min ± 0,1 min, add 1,0 g ± 0,01 g sodium bicarbonate.

25.3.2.4 After stirring for 3 min ± 0,1 min, add 28,0 g ± 0,01 g standard evaluation base clay.

25.3.2.5 After stirring for 5 min ± 0,1 min, remove the container from the mixer and scrape the sides with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.

25.3.2.6 Replace the container on the mixer and continue to stir for an additional 5 min ± 0,1 min.

25.3.2.7 Weigh 2,0 g ± 0,01 g of PAC-LV (25.3.1.9). Add the PAC-LV slowly at a uniform rate over a time interval of about 60 s while stirring on the mixer.

The PAC-LV should be added away from the impeller shaft, but in the vortex, to minimize dusting.

25.3.2.8 After stirring for 5 min ± 0,1 min, remove container from the mixer and scrape the sides with the spatula to dislodge any PAC-LV adhering to the container walls. Be sure all PAC-LV clinging to the spatula is incorporated into the suspension.

25.3.2.9 Replace the container on the mixer and continue to stir. It can be necessary to remove the container from the mixer and scrape the sides to dislodge any adhering PAC-LV after another 5 min or 10 min. Total mixing time elapsed from the beginning of PAC-LV addition shall equal 20 min ± 1 min.

25.3.2.10 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 3 °F). Record storage temperature and storage duration.

25.3.2.11 After ageing, stir the suspension on the mixer for 5 min ± 0,1 min.

25.3.2.12 Pour the PAC-LV suspension into a filter-press cell. Before adding the suspension, be sure each part of the filter cell is dry and that none of the gaskets is distorted or worn. The temperature of the suspension shall be 25 °C ± 1 °C (77 °F ± 3 °F). Pour the suspension to within about 13 mm (1/2 in) of the top of the cell. Complete the assembly of the filter-press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

25.3.2.13 Set one timer for 7,5 min ± 0,1 min and the second timer for 30 min ± 0,1 min. Start both timers and adjust pressure on the cell to 690 kPa ± 35 kPa (100 psi ± 5 psi). Pressure shall be supplied by compressed air, nitrogen or helium and applied within 15 s.

25.3.2.14 After 7,5 min ± 0,1 min on the first timer, remove the container and any liquid adhering on the drain tube and discard. Place a dry 10 ml graduated cylinder under the drain tube and continue collecting the filtrate until the end of the 30 min period as measured by the second timer. Remove the graduated cylinder and record the volume of filtrate collected. This filtrate volume represents the volume collected in the time interval from 7,5 min to 30 min.

25.3.3 Calculation of corrected test-fluid filtrate volume

Calculate V_f, the corrected volume of filtrate, expressed in millilitres, using Equation (34):

$$V_\mathrm{f} = 2V_\mathrm{c} \tag{34}$$

where V_c is the volume of filtrate, expressed in millilitres, collected between 7,5 min and 30 min.

Record the calculated value.

25.3.4 Measurement of test-fluid viscosity

25.3.4.1 Add 42 g ± 0,01 g of sea salt to 1 l ± 2 ml of deionized water.

25.3.4.2 To 358 g of the sea salt solution, add 35,0 g ± 0,01 g of potassium chloride (KCl).

25.3.4.3 Weigh 5,0 g ± 0,01 g of PAC-LV (25.3.1.9). Add the PAC-LV slowly at a uniform rate over a time interval of about 60 s while stirring on the mixer.

The PAC-LV should be added away from the impeller shaft, but in the vortex, to minimize dusting.

25.3.4.4 After stirring for 5 min ± 0,1 min, remove the container from the mixer and scrape the sides with the spatula to dislodge any material adhering to the container wall. Be sure all material clinging to the spatula is incorporated into the suspension.

25.3.4.5 Replace the container on the mixer and continue to stir. It can be necessary to remove the container from the mixer and scrape the sides to dislodge any adhering PAC-LV after another 5 min or 10 min. Total mixing time elapsed from beginning of the PAC-LV addition shall equal 20 min ± 1 min.

25.3.4.6 Age the suspension for 16 h ± 0,5 h in a sealed or covered container at 25 °C ± 1 °C (77 °F ± 3 °F). Record storage temperature and storage duration.

25.3.4.7 Stir the suspension on the mixer for 5 min ± 0,1 min.

25.3.4.8 Pour the suspension into a viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 r/min rotor speed setting of the viscometer shall be taken at a suspension test temperature of 25 °C ± 1 °C (77 °F ± 3 °F).

25.3.5 Calculation of test-fluid apparent viscosity

Calculate η_A, the apparent viscosity, using Equation (35):

$$\eta_A = \frac{\eta_{600}}{2} \tag{35}$$

where η_{600} is the viscosity reading at 600 r/min, expressed in millipascal seconds.

NOTE 1 cP = 1 mPa·s.

Record the calculated value.

26 Preparation and evaluation of invert-emulsion drilling fluids

26.1 Principle

26.1.1 Occasionally, evaluation and comparison of invert-emulsion drilling fluids are required. The tests described are designed to provide a standardized method for preparing and evaluating the slurries under the same conditions. Significant changes in rheological properties, electrical stability or HTHP fluid loss can indicate instability of the test drilling fluid. It is recommended that the test apparatus and procedures be agreed upon between vendor and client prior to initiation of testing.

26.1.2 The test methods provide specific mixing and heat-ageing procedures in an attempt to minimize interlaboratory variations.

26.1.3 Where possible, the test methods and apparatus used are in accordance with ISO 10414-2.

26.1.4 The test protocol is deliberately simple, designed only to provide a means of comparing the fluids' response to heat ageing and to the incorporation of reasonable levels of various contaminants that can be expected during drilling, such as drill solids, seawater and formation brines.

26.1.5 Other methods exist for evaluating invert-emulsion drilling fluids.

26.2 Reagents and apparatus

26.2.1 **Sea salt**, obtained from the local laboratory supply houses [31] and mixed according to the manufacturer's instructions. The standard seawater mix shall conform to ASTM D 1141, or equivalent.

26.2.2 **Evaluation base clay** [32].

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[31] Merck 33153 3P-Seawater Corrosion Test Mixture 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.

[32] API evaluation base clay, OCMA base evaluation clay or Hymod Prima clay are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

26.2.3 **Thermometer**, metal contact, with a range 0°C to 105 °C (32 °F to 220 °F).

26.2.4 **Balance**, accuracy ± 0,01 g.

26.2.5 **Mixer** 33), high shear, heavy-duty laboratory model with a square-hole, high-shear impeller screen.

The screen shall be replaced when a mass loss of approximately 10 % has occurred through wear. The bushing shall be replaced when any appreciable play can be felt between it and the shaft. Similarly, the shaft shall be replaced when the diameter at the bushing has decreased by approximately 1 %.

26.2.6 **Mixer**, capable of operating at 10 000 r/min ± 100 r/min under load, with a single sine-wave impeller system, approximately 2,5 cm (1 in) in diameter (e.g. Hamilton Beach Mixer 14) at low gear/speed setting).

The impeller shall be replaced when a mass loss of approximately 11 % to 13 % has occurred through wear.

26.2.7 **Container**, conical, stainless steel, approximately 2 l capacity, with base internal diameter of 76 mm (3,0 in) and top internal diameter of 152 mm (6,0 in).

26.2.8 **Container** 5), for mixing, approximately 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.

26.2.9 **Spatula**.

26.2.10 **Tachometer**, optical, magnetic induction or equivalent.

26.2.11 **Mud balance**, in accordance with ISO 10414-2.

26.2.12 **Viscometer**, direct-indicating, in accordance with ISO 10414-2.

26.2.13 **Cup**, viscometer, thermostatically controlled, in accordance with ISO 10414-2.

26.2.14 **Filter press**, high-temperature/high-pressure, in accordance with ISO 10414-2.

26.2.15 **Electrical stability meter**, in accordance with ISO 10414-2.

26.2.16 **Oven**, for heat-ageing, capable of maintaining a stable temperature and equipped with a roller system, or equivalent, to keep the ageing cells in motion.

26.2.17 **Cell**, heat-ageing, preferably stainless steel, capacity 350 ml to 500 ml, capable of pressurization by means of a valve system, if necessary.

26.2.18 **Water bath**.

26.3 Mixing of the initial drilling fluid

26.3.1 Prepare four laboratory barrels (one laboratory barrel ≈ 350 ml) of fluid to the required formulation with the following parameters specified:

a) drilling fluid mass;

b) oil-water ratio;

c) mass fraction of the brine phase, expressed in percent;

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33) Silverson® L2R or L4R are trade names of mixers supplied by Silverson Machines. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

d) HTHP filtrate volume (a range is generally specified);

e) HTHP filtrate testing temperature;

f) rheology requirements (a range is generally specified).

26.3.2 Mix the fluid for a total of 60 min with a high-shear mixer, fitted with a high-shear, square-hole screen surrounding the impeller.

26.3.3 Starting at ambient temperature, mix the sample in a 2 l conical mixing vessel and control the shaft speed during mixing to 6 000 r/min ± 300 r/min. Use the optical tachometer or magnetic induction tachometer to monitor rotational speed. Measure the rotational speed under actual mixing load and monitor during the period of mixing. Make adjustments as necessary, particularly during and after the addition of the weighting material.

26.3.4 The fluid temperature can be expected to rise because of shear, but do not allow it to exceed a maximum of 65 °C (150 °F) as measured by a metal contact thermometer. Thereafter, control it as necessary by means of a water bath to between 60 °C and 65 °C (140 °F and 150 °F).

26.3.5 If the programme of testing requires more than four laboratory barrels, mix any additional volume of drilling fluid in batches of four laboratory barrels using the procedure outlined above.

26.3.6 Blend all batches together to ensure uniformity before commencing the test regime. Blending should be carried out by means of a large spatula or by pouring from one container to another several times.

Blending should not be done by means of a mechanical mixer. The pouring or gentle stirring technique is necessary to ensure that each laboratory barrel is subjected to the same shear per unit time per unit volume.

26.4 Testing the properties of the initial drilling fluid

26.4.1 The fluid shall be tested after mixing using the techniques in accordance with ISO 10414-2. Tests on the initial fluid shall cover all properties listed below:

a) density at ambient temperature of 20 °C (68 °F);

b) rheological properties at speeds of 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min, and 10 s gel and 10 min gel with the temperature maintained at 50 °C (300 °F);

c) electrical stability at 50 °C (300 °F);

d) HTHP filtrate volume at a temperature of 120 °C (250 °F) and a differential pressure of 3 450 kPa (500 psi), or other specified temperature and pressure. Consult ISO 10414-2 for recommended temperature/pressure values.

If there is a requirement for HTHP filtrate volume testing, especially above 120 °C (250 °F), there can be variance in results with different test apparatus and test procedures. It is recommended that the test apparatus and procedures be agreed between vendor and client prior to initiation of testing.

26.4.2 One 350 ml sample (one laboratory barrel) of initial drilling fluid shall be submitted to hot rolling and be used as a blank in all further testing.

26.5 Preparation of the sample contaminated by seawater

26.5.1 To one laboratory barrel of drilling fluid, add 10 % by volume of the standard synthetic seawater mix.

26.5.2 Mix the drilling fluid and synthetic seawater under ambient conditions on a mixer. Add all the seawater over 1 min and carry out a further 4 min of mixing for a total mixing time of 5 min.

26.5.3 Hot-roll the sample at the temperature agreed upon by the interested parties.

26.6 Preparation of the sample contaminated by base evaluation clay

26.6.1 To one laboratory barrel of drilling fluid, add base evaluation clay on a mixer, stirring at the low-speed setting.

Additions of base evaluation clay should be based on the expected density of the fluid, as follows:

— for a density below 1 800 kg/m^3, add 35 g of base evaluation clay;

— for a density above 1 800 kg/m^3, add 20 g of base evaluation clay.

26.6.2 Mix the drilling fluid and base evaluation clay under ambient conditions on a mixer. Add all the clay over a period of 1 min and carry out a further 4 min of mixing for a total mixing time of 5 min.

26.6.3 Hot-roll the sample at the temperature agreed upon by the interested parties.

26.7 Preparation of the sample contaminated by mixed-salt brine

26.7.1 This is an additional contamination sample recommended for those wells where it is considered relevant (e.g. where Zechstein salt is expected). The type of mixed-salt brine to be used in the test will already have been determined by discussion between the interested parties.

26.7.2 A typical mixed-salt brine is Carnallite brine, but any simulated brine can be used. To prepare a Carnallite brine, add 125,0 g of magnesium chloride hexahydrate (CAS number 7786-30-3), 14,5 g of potassium chloride (CAS number 7447-40-7) and 5,0 g of sodium chloride (CAS number 7647-14-5) to 100 g of water.

26.7.3 Mix the brine thoroughly on a mixer and extract the volume to be used as a contaminant with a syringe during mixing. This ensures that any undissolved salts are incorporated into the contaminant.

26.7.4 To one laboratory barrel of drilling fluid, add 10 % mixed-salt brine by volume on a mixer, stirring at the low-speed setting. Add the brine to the drilling fluid over a period of 1 min, with an additional 4 min mixing. Carry out the procedure at ambient temperature.

26.7.5 Hot-roll the sample at the temperature agreed upon by the interested parties.

26.8 Procedure for hot-rolling

26.8.1 Samples of the initial formulation and of the contaminated drilling fluid shall be hot-rolled for 16 h at 120 °C (250 °F), or other specified temperature, in a preheated hot-rolling oven.

Pressurization of the hot-roll cell with nitrogen should be at the discretion of the company formulating the drilling fluid.

26.8.2 After 16 h of hot-rolling, switch off the oven heat and open the oven door.

26.8.3 Roll the samples during the cooling period for at least 30 min prior to removal.

26.8.4 Subsequently, cool in ambient air for an additional 30 min. Then cool the cell to ambient temperature by partially immersing the cell in water.

26.9 Procedure for static ageing

26.9.1 Samples of the initial formulation and of the contaminated drilling fluid shall be statically aged for 16 h at 120 °C (250 °F), or other specified temperature, in a preheated ageing oven.

Pressurization of the ageing cell with nitrogen should be at the discretion of the company formulating the drilling fluid.

26.9.2 After 16 h of static ageing, switch off the oven heat and open the oven door.

26.9.3 Cool the samples for at least 30 min prior to removal.

26.9.4 Subsequently, cool in ambient air for an additional 30 min. Then cool the cell to ambient temperature by partially immersing the cell in water.

26.9.5 Stir all aged samples on a mixer for 5 min immediately prior to testing.

26.10 Procedure for testing after heat ageing

The fluid shall be tested after mixing using the techniques in accordance with ISO 10414-2. The procedure for testing the initial fluid shall cover all properties listed below:

a) density at ambient temperature of 20 °C (68 °F);

b) rheological properties at speeds of 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min, and 10 s gel and 10 min gel with temperature maintained at 50°C (120 °F);

c) electrical stability at 50 °C (120 °F);

d) HTHP filtrate volume at a temperature of 120 °C (250 °F) and a differential pressure of 3 450 kPa (500 psi), or other specified temperature and pressure. Consult ISO 10414-2 for recommended temperature/pressure values.

If there is a requirement for HTHP filtrate volume testing, especially above 120 °C (250 °F), note that there can be variance in results with different test apparatus and test procedures. It is recommended that the test apparatus and procedures be agreed upon between vendor and client prior to initiation of testing.

27 High-temperature/high-pressure filtration testing of drilling fluids using the permeability plugging apparatus and cells with set-screw-secured end caps

27.1 Principle

27.1.1 Measurements of the filtration behaviour and wall-building characteristics of drilling fluid are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water or emulsion concentration.

27.1.2 These characteristics are affected by the types and quantities of solids in the fluid and by their physical and chemical interactions. The permeability plugging apparatus (PPA) is a modified high-temperature, high-pressure filter press used to evaluate these interactions through various types of filter media at pressures of up to 13 800 kPa (2 000 psi) and temperatures from ambient to as high as 260 °C (500 °F). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

27.2 Safety considerations

27.2.1 The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cell available: those with threaded end caps and those with set-screw-secured end caps. Among these cells is a total of five different pressure ratings. For safety, it is imperative that the operator know the maximum operating pressure of the test apparatus with certainty and that this pressure not be exceeded. If in doubt, contact the manufacturer or use the lowest of the possible limits.

27.2.2 Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the apparatus. Improper assembly, incorrect operation or the use of defective parts creates the possibility of cell leakage or failure, which can result in serious injury or apparatus damage.

27.2.3 The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching the hot parts of the apparatus during normal operation.

27.2.4 These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury and apparatus damage. These instruments should be used only on grounded circuits.

27.2.5 For safe operation of the hydraulic pressurization system, follow the instructions in 27.2.5.1 to 27.2.5.3.

27.2.5.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero before

a) attempting to disconnect the pressure hose from cell at the quick coupler,

b) attempting to remove the cell from the heating jacket,

c) moving the PPA,

d) refilling the hydraulic pump,

e) performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings or cell assembly.

27.2.5.2 After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

27.2.5.3 Make sure, when assembling the cell, that the end-cap set screws are properly aligned and tightened.

27.2.6 For safe pneumatic pressurization of the backpressure receiver, follow the instructions in 27.2.6.1 to 27.2.6.4.

27.2.6.1 Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it shall be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system should be built into the laboratory. Nitrogen cylinders should be secured to meet safety standards. CO_2 is normally supplied in small cartridges pressurized to about 6 200 kPa (900 psi). They are primarily used for field operations.

CAUTION 1 — Do not allow CO_2 cartridges to be heated or exposed to fire. They can explode if overheated.

CAUTION 2 — Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas-train carbonate analysis.

27.2.6.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

27.2.6.3 Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings and hoses should be kept in good condition, and leaks should be identified and corrected. Periodically test the pressure-relief valve on the hydraulic pump to verify that it will function properly if excessive pressure develops. Never plug or bypass this safety valve.

27.2.6.4 When pressurizing the backpressure assembly, always open the supply pressure first, then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the apparatus rating or relief-valve settings. When relieving backpressure, shut the supply pressure, bleed the pressure from the system, and then back out the regulator T-screw.

27.2.7 For safe heating, follow the instructions in 27.2.7.1 and 27.2.7.2.

27.2.7.1 Caution should be exercised to avoid injury while operating the PPA, which becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

27.2.7.2 The practice of removing the cell and cooling it with water is dangerous and should be avoided. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell or by accidentally dropping the cell.

27.2.8 For safe electrical operation, follow the instructions in 27.2.8.1 and 27.2.8.2.

27.2.8.1 Make sure that the electrical source is fused and grounded (earthed). Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

27.2.8.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

27.2.9 For test cell maintenance, the user must be aware that the filtration cell is a pressure vessel and is to be considered a source of potential danger. The safety precautions listed in 27.2.9.1 to 27.2.9.4 shall be followed to ensure safe operation.

27.2.9.1 Cell material shall be compatible with the test samples.

27.2.9.2 Do not use cells that show signs of severe pitting or stress cracking.

27.2.9.3 Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

27.2.9.4 Use only undamaged, hardened steel set-screws. Others are unsafe.

27.3 Apparatus — Permeability-plugging apparatus (PPA) with set-screw-secured end caps

27.3.1 PPA cell [34].

27.3.1.1 There are three different types of cell available that use set-screw-secured end caps. Those of current and recent manufacture are rated at either 13 800 kPa (2 000 psi) or 12 420 kPa (1 800 psi).

There are still a number of older cells in use which are stamped “2 500 psi”. In 1996, the rating on these cells was reduced to “1 800 psi” (12 420 kPa). Unless the user can absolutely verify that the cell being used is rated at 13 800 kPa (2000 psi), the “1 800 psi” (12 420 kPa) pressure limit should be observed.

The operating manual, or this International Standard, should be attached to the apparatus and should be read by anyone who is unfamiliar with the apparatus before using it.

CAUTION — Follow the manufacturer’s recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

27.3.1.2 The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top and the filtrate collected from the top. A small hydraulic hand pump applies the cell pressure. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

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[34] OFITE Model 171-90 is an example of a suitable product available commercially. This information is for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

27.3.1.3 The PPA can use any one of a number of filtration media, including porous ceramic or sintered metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from 100 mD to 100 D. The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provides the user with a greatly improved picture of what is happening downhole.

To improve the uniformity of test conditions and the repeatability of results, the disks can be classified utilizing the user's own flow test procedure or that which is outlined in 27.3.2.6.

27.3.1.4 Test pressures are limited by the safety limits of the cell as specified by the manufacturer: usually either 12 420 kPa (1 800 psi) or 13 800 kPa (2 000 psi) at some defined temperature. If backpressure is used in the test, it can be necessary to reduce the test pressure to avoid exceeding the pressure limit of the cell and thus damaging the cell.

27.3.1.5 Cell damage caused by excessive pressure can be categorized as follows:

- end-cap bending, which can be detected visually or by measurement;
- end-cap compression, which can de detected by the distortion of the set-screw holes or seats that become oval rather than round;
- cylinder shear;
- cylinder stress.

Caps showing signs of damage should not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set-screw holes, should not be used.

27.3.1.6 For temperatures above 93 °C (200 °F), the backpressure receiver shall be pressurized to prevent boiling of the filtrate. The standard backpressure receiver uses a CO_2 pressurizing source to provide the backpressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

27.3.1.7 The PPA cell is encased in a thermostatically controlled aluminium heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 W.

27.3.2 **Filter medium**, disks of any porous material, such as ceramic, sintered metal or resin-coated sand, graded sands or core samples.

27.3.2.1 Standard disk thickness is 6,5 mm (0,25 in) but, with adapters, thicker disks can be used. A new disk is required for each test. For water-based drilling fluid samples, the disks shall be soaked in fresh water or brine until saturated, at least 5 min to 30 min prior to use. For oil-based drilling fluids, the disk shall be soaked for 5 min to 10 min in a sample of the base oil before use. Vacuum saturation shall be used for filter media with low porosity and permeability.

There is unavoidable variability in the pore throat sizes of the ceramic disks normally used in these tests. Consequently, when running comparative tests, it is recommended that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with fresh water to further classify the disk.

27.3.2.2 Other disk types are available, including Berea sand cores of different porosities and permeabilities. The user should note that these cores can have some variability in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 6,5 mm (0,25 in) thick. With modification of the cylinder, 25,4 mm (1 in) cores can also be used.

27.3.2.3 Resin-coated sand can be made into a solid disk, selecting the sand grain size to provide the desired permeability. The sand is heated at 150 °C (300 °F) for 1 h to 3 h in moulds slightly larger than the normal disk size, and either 6,5 mm (0,25 in) or 25,4 mm (1 in) thick. The moulds shall be coated with silicone grease prior to heating. Resin-coated sand disks can be manufactured to provide a substantial variation in pore throat size and permeability by varying the mesh sizes of the sands. Coarser sands can be used to provide a filter medium for testing lost circulation material, for use in controlling seepage losses in severe fluid-loss environments.

27.3.2.4 Sintered metal disks or slotted metal disks can be used to simulate fractures or high-permeability formations. In the evaluation of seepage loss material needed to seal off a specific formation, the disk pore throat size should be matched with that of the formation.

27.3.2.5 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed, and then weigh the amount of sand necessary to obtain that height. The sand bed shall be soaked with the base fluid prior to the test. If the user desires to run the test in the standard manner, with the filter medium at the top of the cell, the resin-coated sand can be placed in the cell, heated for 1 h to 3 h at 150 °C (300 °F), cooled, and then inverted for the test.

27.3.2.6 Procedure for ceramic-disk comparison: Install the disk in a PPA cell and fill the cell with water. Using the air-permeability apparatus, with the upper cell valve closed, adjust the pressure on the 200 kPa (30 psi) test gauge to 28 kPa to 31 kPa (4,0 psi to 4,5 psi). Open the valve on top of the cell and adjust the pressure to 14 kPa ± 0,7 kPa (2 psi ± 0,1 psi). After opening the valve at the bottom of the cell, readjust the pressure with the upper valve to 14 kPa ± 0,7 kPa (2 psi ± 0,1 psi). Measure the time for 300 ml to pass through using a 500 ml graduated cylinder, timing the flow from the 100 ml mark to the 400 ml mark. If it is intended that the PPT be used for comparison purposes, run several disks, classify the disks and use those of similar values.

27.3.3 **Timer**, accurate to 0,1 min over the test interval.

27.3.4 **Thermometer**, with a scale up to 260 °C (500 °F).

27.3.5 **Graduated cylinder**, 25 ml or 50 ml (TC).

27.3.6 **High-speed mixer** 4).

27.4 Procedure for high-temperature/high-pressure (HTHP) filtration

27.4.1 Preheating the heating jacket

27.4.1.1 Connect the power cord to the proper voltage as indicated on the nameplate.

27.4.1.2 Turn the thermostat to the mid-scale and place a metal-stem dial thermometer in the thermometer well of the heating jacket. The pilot light illuminates when the heating-jacket temperature has reached the thermostat setting.

27.4.1.3 Readjust the thermostat to 6 °C (10 °F) over the desired test temperature.

27.4.2 Loading the filtration cell

27.4.2.1 The filtration cell is a pressure vessel. The following safety precautions shall be followed to ensure safe operation.

a) The cell material shall be resistant to the test sample.

b) Cell bodies that show signs of stress-cracking or severe pitting should not be used.

c) Use only undamaged, hardened steel set-screws. The use of damaged, or common, mild steel set-screws is hazardous.

27.4.2.2 As-received from the manufacturer, the PPA is equipped with valves that are rated to 260 °C (500 °F). If it becomes necessary to change any valves during the life of this apparatus, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

27.4.2.3 Loosen the set-screws securing the end caps, then remove the caps from the cell by pulling them straight out, using the nipples and connected hardware as handles. If the cap is stuck and cannot be freed by rocking it gently, screw the cell-cap-removal tool into the seat for the nipple and press downward on the handle to remove it. Then unscrew the nipples from the caps and remove the piston from the cell.

27.4.2.4 Check the O-rings on the nipples, the floating piston, the cell body and the end caps and replace any that are damaged or brittle [all O-rings should be replaced routinely after tests at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease completely around all of the O-rings, being especially careful to ensure that those on the piston are well lubricated.

27.4.2.5 Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely (the bottom of the cell, the inlet end, has a shorter recess than the top). Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

27.4.2.6 Fill the space above the piston with hydraulic oil to just above the end face.

27.4.2.7 Install the hydraulic end cap onto the bottom of the cell by pushing in on the backpressure ball on the nipple of the end cap on the pressure-inlet end of the cell to relieve the pressure, and allow the cap to slide into the cell more easily. Install and tighten the set-screws.

Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

27.4.2.8 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

The steps in 27.4.2.9 to 27.4.2.13 can be accomplished in the jacket that is being preheated, in an unheated jacket if one is available or in a specially constructed stand.

27.4.2.9 Turn the cell upright and fill it with approximately 275 ml of the drilling fluid. This allows for expansion while heating. Do not exceed this amount.

For improved consistency in test results, stir the drilling fluid for 5 min immediately before loading the cell.

27.4.2.10 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

27.4.2.11 Install the O-ring and set the selected ceramic disk or other filtering medium on top of it.

27.4.2.12 Position the top end cap in the cell, tighten the set screws snugly, and close the valve on the top end cap.

Thermal expansion of the cell contents and of the hydraulic fluid cause the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid to prevent overpressurization. During heating, the pressure in the cell should be controlled by bleeding off the excess fluid periodically.

27.4.2.13 Install the cell in the heating jacket. Make sure that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

27.4.3 Pressurizing the cell

27.4.3.1 Refer to Table 3 for the pressure corresponding to the test temperature, and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it should always be operated at about one stroke per second.

Filtration at temperatures above the boiling point of the fluid sample requires the use of the backpressure receiver to prevent vaporization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling.

It is necessary to modify nitrogen manifolds having a 4 100 kPa (600 psi) gauge for back-pressure to attain a pressure of 4 850 kPa (700 psi). However, tests are possible within the limits of the manifolds.

Table 3 — Starting cell pressures and backpressures for various test temperatures

Temperature range θ		Heating pressure or backpressure p	
°C	°F	kPa	psi
Less than 95	Less than 200	0	0
95 to 150	200 to 300	690	100
151 to 175	301 to 350	1 050	150
176 to 190	351 to 375	1 400	200
191 to 205	376 to 400	1 725	250
206 to 218	401 to 425	2 420	350
219 to 232	426 to 450	3 100	560
233 to 246	451 to 475	3 800	550
247 to 260	476 to 500	4 850	700

27.4.3.2 While the cell is heating, use the following procedure to prepare the backpressure receiver.

a) Check to ensure that the regulator T-screw has been rotated counter-clockwise enough to release all pressure. When the pressure has been released, the screw turns freely.

b) Open the pressure-relief valve to relieve any remaining pressure and remove the CO_2 cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time (see 27.4.3.6).

c) Verify that the pressure-relief valve on the CO_2 assembly and the filtrate drain valve are closed.

d) Set the backpressure assembly aside. Instructions for its installation are given in 27.4.3.4.

27.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as 1 h.

27.4.3.4 When the cell is at the desired temperature and the cell pressure stabilized, mount the backpressure receiver on the upper valve adapter. Secure the receiver with a retaining pin. Install the CO_2 pressurizing unit on top of the receiver. Lock the CO_2 pressurizing unit in place with the other retaining pin.

27.4.3.5 If a drain hose is used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space from the filtration medium to the backpressure receiver outlet and the receiver valve should be filled with the base fluid before starting the test. This ensures that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this procedure can introduce significant error.

27.4.3.6 See Table 3 to determine the appropriate pressure for the backpressure receiver and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

27.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the backpressure receiver to start the test.

The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the backpressure receiver.

27.4.4 Conducting the filtration test

27.4.4.1 A laboratory timer shall be used and filtrate shall be collected and its volume recorded at 1 min, 7,5 min and 30 min. Plotting these data against the square root of time provides a useful description of the spurt loss. If desired, samples can be taken more frequently but none should be taken before 1 min. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

For improved definition of the spurt loss, collect filtrate at 1 min, 5 min, 7,5 min, 15 min, 25 min and 30 min, and plot the cumulative filtrate volumes versus the square root of time.

27.4.4.2 Begin the filtration by opening the valve between the cell and the backpressure receiver. Verify that both the cell pressure, as indicated on the pump gauge, and the backpressure are at desired levels. Adjust them as necessary throughout the test.

27.4.4.3 Cell pressure can be expected to decrease slightly as filtration and any leakage at the pump reduce the cell contents. The pump should be used as necessary to maintain the pressure at the desired level. If a manually operated pump is used, actuate it at about one stroke per second.

27.4.4.4 After each interval, the filtrate shall be drained into the graduated cylinder from the backpressure receiver and the time and cumulative volume shall be recorded.

It is recommended to recover the filtrate directly from the backpressure receiver not from a drain hose attached to it. If a hose is used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

27.4.4.5 After 30 min, close the filtrate valve and drain any remaining filtrate from the backpressure receiver into the graduated cylinder. The total volume of the filtrate in the graduated cylinder shall be recorded.

27.5 Test conclusion and disassembly

27.5.1 Disconnect the heating jacket from the power source.

The temperature of the sample in the cell should be reduced to below 38 °C (100 °F) to ensure that the cell can safely be opened.

27.5.2 The pressurized cell assembly shall be allowed to cool in the heating jacket. When these tests are run with sufficient frequency to justify it, a cooling stand, station or bath can be provided to expedite the cooling process. A cell-handling tool is available, which should be used any time a hot cell is to be handled.

Extreme care should be exercized in cooling hot cells.

This procedure, as recommended, makes it difficult to perform more than one test in an 8 h work day with a single PPA. In the interest of improving productivity, it can be useful to the users to design their own cell cooling procedures and apparatus. Safety should be the primary consideration in these designs.

27.5.3 Close the valve between the cell and the backpressure receiver.

27.5.4 Release the pressure on the pump and the cell by opening the pump valve, then disconnect the quick coupler between the pump and the nipple adapter on the bottom of the cell.

27.5.5 Release the backpressure by turning the T-screw on the regulator counter-clockwise until it turns freely.

27.5.6 Bleed the pressure from the backpressure receiver by opening the pressure-relief valve on the CO_2 unit. Open the drain valve on the backpressure receiver and collect the last few drops of filtrate in the graduated cylinder. After removing the locking pin securing it, remove the CO_2 assembly from the top nipple adapter. After removing its locking pin, remove the backpressure receiver.

27.5.7 The cell may be opened after its contents have been allowed to cool. The cell should be opened only when the user is confident that the contents are no longer under pressure.

27.5.7.1 If is suspected that there is still pressure in the cell and the lower end cap does not include a screen, the following procedure can be used to determine the position of the floating piston.

- Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom.
- If the piston is not at the bottom, there is no pressure. If the piston is at the bottom, there can be pressure remaining in the cell.
- Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the force required to move the piston.

27.5.7.2 If there are indications that the cell is still pressurized, completely remove the filtration valve assembly from the cell and insert a small drill bit or wire into the cell cap to remove the obstruction. The drill or wire will stop when it contacts the filter disk. Make sure that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

27.5.8 The cell may be raised in the heating jacket and placed on the cell support or laid on a bench while it is being opened.

27.5.9 Loosen the set-screws securing the end cap and, using the valve and nipple adapter as a handle, pull the cap from the cell. If it is stuck, a rocking motion of the valve and nipple adapter might free it. If it cannot be freed in this manner, unscrew the valve and nipple adapter, install the cap removal tool in its place and use it to remove the cap.

27.5.10 The end cap should be removed with the cell in the vertical position and the filtration end facing upwards.

27.5.11 Remove the filter disk. Use a small knife, small screwdriver or similar thin-bladed tool to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh water, or base oil if the sample is oil-based, then measure and record its thickness and record any remarks concerning its composition and texture.

27.5.12 Pour the remaining fluid from the cell. Wash the inside of the cell with fresh water or a suitable solvent if the sample is oil-based. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 150 °C (300 °F) or higher.

When testing is conducted at temperatures above 150 °C (300 °F), the O-rings should be replaced.

27.5.13 Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the bottom end cap using the procedure outlined in 27.5.9 and 27.5.10.

b) Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. Note that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all of the O-rings on the piston and the cap.

c) Clean the parts for reuse.

27.6 Data reporting

27.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, in millilitres, collected during each of the selected time periods.

27.6.2 Spurt loss

The spurt loss can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume [doubled to correct for filtration area when using 22,6 cm^2 (3,5 in^2) filtration media] is plotted along the y-axis. Alternatively, an approximate value can be calculated using Equation (37).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with 27.4.4.1, second paragraph.

27.6.3 Calculation

Calculate V_{PPT}, the permeability plugging test volume, expressed in millilitres, using Equation (36):

$$V_{\text{PPT}} = 2\,V_{30} \tag{36}$$

where V_{30} is the filtrate volume after 30 min, expressed in millilitres.

Calculate V_1, the spurt loss, expressed in millilitres, using Equation (37):

$$V_1 = 2\left[V_{7,5} - \left(V_{30} - V_{7,5}\right)\right] = 2\left(2V_{7,5} - V_{30}\right) \tag{37}$$

where $V_{7,5}$ is the filtrate volume after 7,5 min, expressed in millilitres.

Calculate v_{f}, the static filtration rate (velocity of flow), expressed in millilitres per minute, using Equation (38):

$$v_{\text{f}} = \frac{2\left(V_{30} - V_{7,5}\right)}{\sqrt{t_2} - \sqrt{t_1}} = \frac{2\left(V_{30} - V_{7,5}\right)}{2,739} \tag{38}$$

where

t_1 is the time at initial reading, expressed in minutes;

t_2 is the time at final reading, expressed in minutes.

Observe that all three of these parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of those used in the standard low-pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant (2 in this case) can be modified as necessary to accommodate tests made utilizing other filtration areas.

27.6.4 Filter cake reporting

Measure and record the filter cake thickness to the nearest 1,0 mm (1/32 in). Include a description, such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgements, they can convey important information.

28 High-temperature/high-pressure filtration testing of drilling fluids using the permeability-plugging apparatus and cells with threaded end caps

28.1 Principle

28.1.1 Measurements of the filtration behaviour and wall-building characteristics of drilling fluid are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water or emulsion concentration.

28.1.2 These characteristics are affected by the types and quantities of solids in the fluid and by their physical and chemical interactions. The PPA is a modified HTHP filter press used to evaluate these interactions through various types of filter media at pressures of up to 34 500 kPa (5 000 psi) and temperatures from ambient to as high as 260 °C (500 °F). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

28.2 Safety considerations

28.2.1 The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cell available: those with threaded end caps and those with set-screw-secured end caps. Among these cells is a total of five different pressure ratings. For safety, it is imperative that the operator know the maximum operating pressure of the test apparatus with certainty and that this pressure not be exceeded. If in doubt, contact the manufacturer or use the lowest of the possible limits.

28.2.2 Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the apparatus. Improper assembly, incorrect operation, or the use of defective parts creates the possibility of cell leakage or failure, which can result in serious injury or apparatus damage.

28.2.3 The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching parts of the apparatus during normal operation.

28.2.4 These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury and apparatus damage. These devices should be used only on grounded circuits.

28.2.5 For safe operation of the hydraulic pressurization system, follow the instructions in 28.2.5.1 to 28.2.5.3.

28.2.5.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero before

a) attempting to disconnect pressure hose from cell at the quick-coupler,

b) attempting to remove the cell from the heating jacket,

c) moving the PPA,

d) refilling the hydraulic pump,

e) performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

28.2.5.2 After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on floors is hazardous and accumulations of spilled oil near the PPA are fire hazards.

28.2.5.3 Make sure that the O-rings in the end caps are properly seated when assembling the cell.

28.2.6 For safe pneumatic pressurization of backpressure receiver, follow the instructions in 28.2.6.1 to 28.2.6.4.

28.2.6.1 Always use either nitrogen or carbon dioxide to pressurize the backpressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it should be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system should be built into the laboratory. Nitrogen cylinders should be secured to meet safety standards. CO_2 is normally supplied in small cartridges pressurized to about 6 200 kPa (460 psi). They are primarily used for field operations.

CAUTION — Do not allow CO_2 cartridges to be heated or exposed to fire. They can explode if overheated.

CAUTION — Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous material. Nitrous oxide cartridges shall be used only for Garrett gas-train carbonate analysis.

28.2.6.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

28.2.6.3 Repair or replace leaking hydraulic or pneumatic pressurization systems. Gauges, fittings and hoses should be kept in good condition, and leaks should be found and corrected. Periodically test the pressure-relief valve on the hydraulic pump to verify that it will function properly if excessive pressure develops. Never plug or bypass this safety valve.

28.2.6.4 Always open the supply pressure first when pressurizing the backpressure assembly. Then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the apparatus rating or relief-valve settings. When relieving backpressure, shut the supply pressure, bleed the pressure from the system and back out the regulator T-screw.

28.2.7 For safe heating, follow the instructions in 28.2.7.1 and 28.2.7.2.

28.2.7.1 Exercise caution to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

28.2.7.2 Avoid the practice of removing the cell and cooling it with water. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell or by accidentally dropping the cell.

28.2.8 For safe electrical operation, follow the instructions in 28.2.8.1 and 28.2.8.2.

28.2.8.1 Make sure that the electrical source is fused and grounded. Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

28.2.8.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

28.2.9 For safe test cell maintenance, it is necessary that the user be aware that the filtration cell is a pressure vessel and is considered to be a source of potential danger. The safety precautions listed in 28.2.9.1 to 28.2.9.3 shall be followed to ensure safe operation.

28.2.9.1 Cell material shall be compatible with the test samples.

28.2.9.2 Do not use cells that show signs of severe pitting or stress cracking.

28.2.9.3 Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

28.3 Apparatus — Permeability-plugging apparatus (PPA) with threaded end caps

28.3.1 PPA cell[35].

28.3.1.1 There are two manufacturers of PPAs. Each supply threaded end caps for the cells used for tests run at pressures in excess of 13 800 kPa (2 000 psi). There are threaded caps with three different pressure ratings available: 20 700 kPa (3 000 psi), 27 600 kPa (4 000 psi), and 34 500 kPa (5 000 psi). The operating manual, or this International Standard, should be attached to the apparatus and should be read by anyone who is unfamiliar with the apparatus, before using it. If the user is unable to determine the operating limits with certainty, the lowest pressure limit should be assumed to be applicable.

CAUTION — Follow the manufacturer's recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

28.3.1.2 As received from the manufacturer, the PPA is equipped with valves that are rated to 260 °C (500 °F). If it becomes necessary to change any valves during the life of this apparatus, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

28.3.1.3 The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top and the filtrate collected from the top. A small hydraulic hand-pump applies the cell pressure. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent mixing of the hydraulic oil with the sample.

28.3.1.4 The PPA can use any one of a number of filtration media, including porous ceramic or sintered metal disks, core samples and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from $9{,}87 \times 10^{-16}$ m^2 to $9{,}87 \times 10^{-11}$ m^2 (100 mD to 100 D). The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provides the user with a greatly improved picture of what is happening downhole.

To improve the uniformity of test conditions and the repeatability of results, the disks can be classified utilizing the user's own flow test procedure or that which is outlined in 28.3.2.7.

28.3.1.5 Test pressures are limited by the safety limits of the cell as specified by the manufacturer: usually 20 700 kPa (3 000 psi), 27 600 kPa (4 000 psi), and 34 500 kPa (5 000 psi) at 260 °C (500 °F). The backpressure receiver may be used at pressures as high as 5 170 kPa (750 psi). If backpressure is used in the test, it can be necessary to reduce the maximum test pressure to avoid exceeding the pressure limit of the cell.

Cell caps showing signs of damage should not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting should not be used.

28.3.1.6 For temperatures above 93 °C (200 °F), the backpressure receiver shall be pressurized to prevent boiling of the filtrate. The standard backpressure receiver uses a CO_2 pressurizing source to provide the backpressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

28.3.1.7 The PPA cell is encased in a thermostatically controlled aluminium heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 W.

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[35] Fann Model 170-72, Fann Model 17015, and OFITE Model 171-84 are examples of suitable products available commercially. This information is for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

28.3.1.8 The PPA can be used in the field or in the laboratory. A stainless steel carrying case with a fold-down workshelf is available for use in the field.

28.3.2 Filter medium, disks of any porous material such as ceramic, sintered metal, or resin-coated sand, graded sands, or core samples.

28.3.2.1 Standard disk thickness is 6,5 mm (0,25 in) but, with adapters, thicker disks can be used. A new disk is required for each test. For water-based drilling fluid samples, the disks shall be soaked in fresh water or brine until saturated, at least 5 min to 30 min prior to use. For oil-based drilling fluids, the disk shall be soaked for 5 min to 10 min in a sample of the base oil before use. Vacuum saturation shall be used for filter media with low porosity and permeability.

There is unavoidable variability in the pore throat sizes of the ceramic disks normally used in these tests. Consequently, when running comparative tests, it is recommended that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with fresh water to further classify the disk.

28.3.2.2 Other disk types are available, including Berea sand cores of different porosities and permeabilities. The user should note that these cores have some variability in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 6,5 mm (0,25 in) thick. With modification of the cylinder, 25,4 mm (1 in) cores can also be used.

28.3.2.3 Resin-coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The sand is heated at 150 °C (300 °F) for 1 h to 3 h in moulds slightly larger than the normal disk size, and either 6,5 mm (0,25 in) or 25,4 mm (1 in) thick. The moulds shall be coated with silicone grease prior to heating.

28.3.2.4 Resin-coated sand disks can be manufactured to provide a substantial variation in pore throat size and permeability by varying the mesh sizes of the sands. Coarser sands can be used to provide a filter medium for testing lost circulation material, for use in controlling seepage losses to severe fluid loss environments.

28.3.2.5 Sintered metal disks or slotted metal disks can be used to simulate fractures or high-permeability formations. In the evaluation of seepage-loss material needed to seal off a specific formation, the disk pore throat size should be matched with that of the formation.

28.3.2.6 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed and then weigh the amount of sand necessary to obtain that height. The sand bed shall be saturated with the base fluid before the test. If the user desires to run the test in the standard manner with the filter medium at the top of the cell, the resin-coated sand can be placed in the cell, heated for 1 h to 3 h at 150 °C (300 °F), cooled and then inverted for the test.

28.3.2.7 Procedure for ceramic disk comparison: Install the disk in a PPA cell and fill the cell with water. Using the air-permeability apparatus, with the upper cell valve closed, adjust the pressure on the 200 kPa (30 psi) test gauge to 28 kPa to 31 kPa (4,0 psi to 4,5 psi). Open the valve on top of the cell and adjust pressure to 14 kPa ± 0,7 kPa (2 psi ± 0,1 psi). After opening the valve at the bottom of the cell, readjust the pressure with the upper valve to 14 kPa ± 0,7 kPa (2 psi ± 0,1 psi). Measure the time for 300 ml to pass through using a 500 ml graduated cylinder, timing from the 100 ml mark to the 400 ml mark. If it is intended that the PPT be used for comparison purposes, run several disks, classify the disks and use those of similar values.

28.3.3 Timer, accurate to 0,1 min over the test interval.

28.3.4 Thermometer, with a scale reading up to 260 °C (500 °F).

28.3.5 Cylinder, graduated, 25 ml or 50 ml (TC).

28.3.6 High-speed mixer[4].

28.4 Procedure for high-temperature/high-pressure (HTHP) filtration

28.4.1 Preheating the heating jacket

28.4.1.1 Connect the power cord to the proper voltage as indicated on the nameplate.

28.4.1.2 Turn the thermostat to the mid-scale setting and place a metal-stem dial thermometer in the thermometer well of the heating jacket. The pilot light illuminates when the heating jacket temperature has reached the thermostat setting.

28.4.1.3 Readjust the thermostat to 6 °C (10 °F) above the desired test temperature.

28.4.2 Loading the filtration cell

28.4.2.1 The filtration cell is a pressure vessel. Cell bodies that show signs of stress cracking or severe pitting should not be used. The procedure in 28.4.2.2 to 28.4.2.15 shall be followed to ensure safe operation.

28.4.2.2 Use the spanner wrench to remove the end caps. Then unscrew the nipples from the caps and remove the piston from the cell.

28.4.2.3 Check the O-rings on the nipples, the floating piston, the cell body, and the end caps, and replace any that are damaged or brittle [all O-rings should be replaced routinely after tests at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease completely around all of the O-rings, being especially careful to ensure that those on the piston are well lubricated. Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely (the bottom of the cell, the inlet end, has a shorter recess than the top). Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

28.4.2.4 Fill the space above the piston with hydraulic oil to just above the end face.

28.4.2.5 Lubricate the end face of the cell bore, the horizontal area at the end of the bore, with an anti-seizing compound and fill the space above the piston with hydraulic oil to just above the end face.

28.4.2.6 Lubricate the threads with high-temperature-resistant grease and then screw the end cap into place, tightening it moderately with the two-pin spanner wrench. Over-tightening does not improve the seal and makes the cap difficult to remove.

28.4.2.7 Install the hydraulic end cap onto the bottom of the cell: Push in on the backpressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to be screwed into the cell more easily.

Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

28.4.2.8 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

The steps in 28.4.2.9 to 28.4.2.14 can be accomplished in the jacket that is being preheated, in an unheated jacket if one is available or in a specially constructed stand.

28.4.2.9 Turn the cell upright and fill with approximately 275 ml of drilling fluid. For improved consistency in test results, stir drilling fluid for 5 min immediately before loading the cell. This allows for fluid expansion while heating. Do not exceed this amount.

28.4.2.10 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

28.4.2.11 Install the O-ring and set the selected ceramic disk or other filtering medium on top of it.

28.4.2.12 Install the top end cap in the cell.

28.4.2.13 Lubricate the threads and the bottom of the retainer ring and screw the ring into the top of the cell. Tighten it, using the single pin spanner wrench if necessary, until the outer knurled flange of the retainer ring is flush against the top of the cell body. Attempting to tighten it further does not improve the seal and makes the cap more difficult to remove.

This step applies only to cells that utilize retainer rings for the top end caps.

28.4.2.14 Install the cell in the heating jacket. Make sure that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

28.4.2.15 Thermal expansion of cell contents and of the hydraulic fluid causes the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the excess periodically.

28.4.3 Pressurizing the cell

28.4.3.1 Filtration at temperatures above the boiling point of the fluid sample requires the use of the backpressure receiver to prevent vapourization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling. Refer to Table 3 for the pressure corresponding to the test temperature and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it shall always be operated at about one stroke per second.

28.4.3.2 While the cell is heating, use the following procedure to prepare the backpressure receiver.

a) Check to ensure that the regulator T-screw has rotated counter-clockwise enough to release all pressure. When the pressure has been released, the screw turns freely.

b) Open the pressure-release valve to relieve any remaining pressure and remove the CO_2 cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time (see 28.4.3.6).

c) Verify that the pressure release valve on the CO_2 assembly and the filtrate drain valve are closed.

d) Set the backpressure assembly aside. Instructions for its installation are given in 28.4.3.4.

28.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as 1 h.

28.4.3.4 When the cell is at the desired temperature and the cell pressure stabilized, mount the backpressure receiver on the upper valve adapter. Secure the receiver with a retaining pin. Install the CO_2 pressurizing unit on top of the receiver. Lock the CO_2 pressurizing unit in place with a retaining pin.

28.4.3.5 If a drain hose is used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space from the filtration medium to the backpressure receiver outlet and the receiver valve should be filled with the base fluid before starting the test. This ensures that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

28.4.3.6 Refer to Table 3 to determine the appropriate pressure for the backpressure receiver and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

28.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the backpressure receiver to start the test.

The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the backpressure receiver.

28.4.4 Conducting the filtration test

28.4.4.1 Verify that the backpressure as read on the pressure regulator gauge is correct. Adjust if required.

28.4.4.2 Set the timer for the desired filtration test times. Filtrate shall be collected at 1 min, 7,5 min and 30 min and the volumes recorded. Data can be collected at additional times, if desired; however, the first sample should not be taken prior to 1 min. Precisely recorded test times and filtrate measurements are necessary for accurate calculation of the filtration parameters.

For improved definition of the spurt loss, collect filtrate at 1 min, 5 min, 7,5 min, 15 min, 25 min and 30 min, and plot cumulative filtrate volumes versus the square root of time.

28.4.4.3 Open the filtration valve to start the test. The cell pressure, as read on the pump gauge, will drop initially. Operate the pump to maintain it as close to test pressure as possible. If a manually actuated pump is used, it shall be operated at about one stroke per second.

28.4.4.4 At the desired times, use the drain valve to bleed the filtrate from the backpressure receiver into the graduated cylinder and record the time and cumulative volume received.

28.4.4.5 The pressure may slowly decrease as the test continues, due to the volume lost through filtration. Additional pressure shall be applied to the cell in order to maintain a constant pressure. Hold the desired pressure on the cell and on the backpressure receiver for the duration of the test.

It is recommended to recover the filtrate directly from the backpressure receiver, not from a drain hose attached to it. If a hose is used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

28.4.4.6 After 30 min, close the filtrate valve and drain any remaining filtrate from the backpressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder shall be recorded.

28.5 Test conclusion and disassembly

28.5.1 Disconnect the heating jacket from the power source. The temperature of the sample in the cell should be reduced to below 38 °C (100 °F) before the cell can safely be opened.

28.5.2 Allow the pressurized cell assembly to cool in the heating jacket. When these tests are run with sufficient frequency to justify it, a cooling stand, station or bath can be provided to expedite the cooling process. There is a cell-handling tool available that should be used any time a hot cell is to be handled.

Extreme care should be exercised in cooling hot cells.

This procedure, as recommended, makes it difficult to perform more than one test in an 8 h work day with a single PPA. In the interest of improving productivity, it can be useful to the users to design their own cell-cooling procedures and apparatus. Safety should be the primary consideration in these designs.

28.5.3 Isolate the backpressure assembly from its pressure source by turning the T-screw on the backpressure regulator counter-clockwise until it turns freely.

28.5.4 Open the bleed valve on the CO_2 unit to release the pressure in the backpressure receiver.

28.5.5 After removing the locking pin securing it, remove the CO_2 assembly from the top nipple adapter.

28.5.6 After removing its locking pin, remove the backpressure receiver.

28.5.7 Open the valve on the hydraulic pump to release cell pressure, then disconnect the hydraulic quick-coupler.

28.5.8 Open the filtration valve to relieve any pressure remaining between the cell filter and the backpressure receiver.

28.5.9 If there are indications that the cell is still pressurized and the screened end cap is not in the lower position, the following procedure can be used to verify the position of the floating piston.

- Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom.
- If the piston is not at the bottom, there is no pressure. If the piston is at the bottom, there can be pressure remaining in the cell.
- Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the force required to move the piston.

28.5.10 If there are indications are that the cell is still pressurized, completely remove the filtration valve assembly from the cell and insert a small drill bit or wire into the cell cap to remove the obstruction. The drill or wire will stop when it contacts the filter disk. Make sure that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

The cell should be opened only when the operator is fully confident that the contents are no longer under pressure.

28.5.11 Raise or remove the cell assembly. If desired, the cell may be raised in the heating jacket either by lifting it by the filter valve assembly or using the optional cell-handling tool. Attach this tool to the backpressure inlet nipple just above the filtrate valve where the backpressure receiver is normally attached. Secure it using the valve-stem locking pin. The cell can be supported on the cell support or lifted out of the heating well and laid on a bench while the cell is being opened.

28.5.12 Remove the threaded caps using a spanner wrench. It can be necessary to tap on the wrench to get it started. Opening difficulty is an indication of insufficient lubrication, over-tightening or insufficient cleaning. It can be necessary to use a suitable holding tool, such as a soft-jaw vice, chain wrench, strap wrench or similar device, to secure the cell while the cap is unscrewed.

28.5.13 Reposition the cell so that the filter end is up and unscrew the top cap.

28.5.14 Remove the filter disk. Use a small knife, small screwdriver or similar thin blade to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh water or the base oil if the sample is oil-based, then measure and record its thickness and record any remarks concerning its composition.

28.5.15 Pour the remaining fluid from the cell and wash the inside of the cell with fresh water or a suitable solvent if the sample is oil-based. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 150 °C (300 °F) or higher.

When testing at temperatures above 150 °C (300 °F), the O-rings should be replaced.

28.5.16 Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the bottom end cap using the procedure outlined in 28.5.12 and 28.5.13, except that the cell position is reversed and a two-pin spanner wrench is used.

b) Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. Note that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all of the O-rings on the piston and the cap.

c) Clean the parts for reuse.

28.6 Data reporting

28.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, expressed in millilitres, collected through each of the selected time periods.

28.6.2 Spurt loss

The spurt loss (3.6) can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of the filtration time is plotted along the x-axis, and the filtrate volume [doubled to correct for filtration area when using 22,6 cm^2 (3,5 in^2) filtration media] is plotted along the y-axis. Alternatively, an approximate value can be calculated using Equation (37).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with 27.4.4.1, second paragraph.

28.6.3 Calculation

Calculate the permeability-plugging test volume, spurt loss and static filtration rate using Equations (36), (37) and (38), respectively.

28.6.4 Filter cake reporting

Measure and record the filter cake thickness to the nearest 1,0 mm (1/32 in). Include a description such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgements, they can convey important information.

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