
**Petroleum and natural gas industries —
Field testing of drilling fluids**

**Part 1:
Water-based fluids**

Industries du pétrole et du gaz naturel — Essais in situ des fluides de forage

Partie 1: Fluides aqueux



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Contents

Page

Foreword.....	v
Introduction	vi
1 Scope	1
2 Terms and definitions.....	2
3 Symbols and abbreviated terms	2
3.1 Symbols	2
3.2 Abbreviations	6
4 Drilling fluid density (mud weight).....	7
4.1 Principle	7
4.2 Apparatus	7
4.3 Procedure	7
4.4 Calculation.....	8
5 Alternative drilling fluid density method.....	9
5.1 Principle	9
5.2 Apparatus	10
5.3 Procedure	10
5.4 Calculation.....	10
6 Viscosity and gel strength	11
6.1 Principle	11
6.2 Determination of viscosity using the Marsh funnel	11
6.3 Determination of viscosity and/or gel strength using a direct-indicating viscometer	11
7 Filtration.....	14
7.1 Principle	14
7.2 Low-temperature/low-pressure test.....	14
7.3 High-temperature/high-pressure (HTHP) test	15
8 Water, oil and solids contents.....	18
8.1 Principle	18
8.2 Apparatus	18
8.3 Procedure	19
8.4 Calculation.....	20
9 Sand content	22
9.1 Principle	22
9.2 Apparatus	22
9.3 Procedure	22
10 Methylene blue capacity	23
10.1 Principle	23
10.2 Reagents and apparatus	23
10.3 Procedure	24
10.4 Calculation.....	26
11 pH	26
11.1 Principle	26
11.2 Reagents and apparatus	27
11.3 Procedure for pH measurement	28
11.4 Care of electrode.....	29
12 Alkalinity and lime content	29
12.1 Principle	29

12.2	Reagents and apparatus	30
12.3	Procedure — Phenolphthalein and methyl orange filtrate alkalinities	30
12.4	Procedure — Phenolphthalein drilling fluid alkalinity	31
12.5	Calculation of ion concentrations from P_f and M_f	31
12.6	Estimation of lime content	31
13	Chloride ion content	32
13.1	Principle	32
13.2	Reagents and apparatus	32
13.3	Procedure.....	32
13.4	Calculation	32
14	Total hardness as calcium	33
14.1	Principle	33
14.2	Reagents and apparatus	33
14.3	Procedure.....	34
14.4	Calculation	35
Annex A	(informative) Chemical analysis of water-based drilling fluids	36
Annex B	(informative) Shear strength measurement using shearometer tube	52
Annex C	(informative) Resistivity.....	54
Annex D	(informative) Removal of air or gas prior to testing	56
Annex E	(informative) Drill pipe corrosion ring coupon.....	57
Annex F	(informative) Sampling, inspection and rejection	61
Annex G	(informative) Rig-site sampling	63
Annex H	(informative) Calibration and verification of glassware, thermometers, viscometers, retort-kit cup and drilling fluid balances	66
Annex I	(normative) High-temperature/high-pressure filtration testing of water-based drilling fluids using the permeability-plugging apparatus and cells equipped with set-screw- secured end caps	71
Annex J	(normative) High-temperature/high-pressure filtration testing of water-based drilling fluids using the permeability-plugging apparatus and cells equipped with threaded end caps	81
Annex K	(informative) Water-based drilling fluids report form.....	90
Bibliography		91

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 10414-1 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 10414-1:2001), to which Annexes I, J and K have been added and other minor changes made to the sentence structure, grammar and other non-technical editing.

ISO 10414 consists of the following parts, under the general title *Petroleum and natural gas industries — Field testing of drilling fluids*:

- *Part 1: Water-based fluids*
- *Part 2: Oil-based fluids*

Introduction

This part of ISO 10414 is based on API RP 13B-1, third edition, December 2003^[2] and ISO 10414 (all parts)^[6].

Annexes A to H and K of this part of ISO 10414 are for information only. Annexes I and J are normative.

In this part of ISO 10414, where practical, U.S. Customary (USC) units are included in brackets for information.

Petroleum and natural gas industries — Field testing of drilling fluids

Part 1: Water-based fluids

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

1 Scope

This part of ISO 10414 provides standard procedures for determining the following characteristics of water-based drilling fluids:

- a) drilling fluid density (mud weight);
- b) viscosity and gel strength;
- c) filtration;
- d) water, oil and solids contents;
- e) sand content;
- f) methylene blue capacity;
- g) pH;
- h) alkalinity and lime content;
- i) chloride content;
- j) total hardness as calcium.

Annexes A through K provide additional test methods which may be used for

- chemical analysis for calcium, magnesium, calcium sulfate, sulfide, carbonate and potassium;
- determination of shear strength;
- determination of resistivity;
- removal of air;
- drill-pipe corrosion monitoring;
- sampling, inspection and rejection;

- rig-site sampling;
- calibration and verification of glassware, thermometers, viscometers, retort-kit cup and drilling-fluid balances;
- permeability-plugging testing at high temperature and high pressure for two types of equipment;
- example of a report form for water-based drilling fluid.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1

ACS reagent grade

chemical meeting the purity standards specified by the American Chemical Society (ACS)

2.2

darcy

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

NOTE 1 cP = 1 mPa.s.

2.3

quarter

⟨verb⟩ mix and divide into four specimens to ensure homogeneity of specimens

2.4

spurt loss

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

2.5

tube sampling

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

3 Symbols and abbreviated terms

3.1 Symbols

NOTE Subscript "A" to symbol denotes metric units. Subscript "B" to symbol denotes U.S. customary units.

A_A	area, in square centimetres
A_B	area, in square inches
$c_{b,A}$	concentration of weighting material, in kilograms per cubic metre
$c_{b,B}$	concentration of weighting material, in pounds per barrel
$c_{Ca,A}$	concentration of calcium ion, in milligrams per litre
$c_{Ca,B}$	concentration of calcium ion, in parts per million by mass (USC)

$c_{\text{Ca+Mg,A}}$	concentration of calcium and magnesium ion (total hardness), in milligrams per litre
$c_{\text{Ca+Mg,B}}$	concentration of calcium and magnesium ion (total hardness), in parts per million (USC)
$c_{\text{CaSO}_4\text{,A}}$	concentration of calcium sulfate, in milligrams per litre
$c_{\text{CaSO}_4\text{,B}}$	concentration of calcium sulfate, in parts per million by mass (USC)
$c_{\text{CO}_2+\text{CO}_3+\text{HCO}_3\text{,A}}$	concentration of total soluble carbonates, in milligrams per litre
$c_{\text{CO}_2+\text{CO}_3+\text{HCO}_3\text{,B}}$	concentration of total soluble carbonates, in parts per million by mass (USC)
$c_{\text{Cl,A}}$	concentration of chloride ion, in milligrams per litre
$c_{\text{Cl,B}}$	concentration of chloride ion, in parts per million by mass (USC)
$c_{\text{ex-CaSO}_4\text{,A}}$	concentration of excess, undissolved calcium sulfate, in milligrams per litre
$c_{\text{ex-CaSO}_4\text{,B}}$	concentration of excess, undissolved calcium sulfate, in parts per million by mass (USC)
$c_{\text{f,KCl,A}}$	concentration of potassium chloride in filtrate, in milligrams per litre
$c_{\text{f,KCl,B}}$	concentration of potassium chloride in filtrate, in parts per million by mass (USC)
$c_{\text{K,A}}$	concentration of potassium ion, in milligrams per litre
$c_{\text{K,B}}$	concentration of potassium ion, in parts per million by mass (USC)
$c_{\text{KCl,A}}$	concentration of potassium chloride, in milligrams per litre
$c_{\text{KCl,B}}$	concentration of potassium chloride, in parts per million by mass (USC)
$c_{\text{lg,A}}$	concentration of low-gravity solids, in kilograms per cubic metre
$c_{\text{lg,B}}$	concentration of low-gravity solids, in pounds per barrel
$c_{\text{lime,A}}$	lime content of the drilling fluid, in kilograms per cubic metre
$c_{\text{lime,B}}$	lime content of the drilling fluid, in pounds per barrel
$c_{\text{NaCl,A}}$	concentration of sodium chloride, in milligrams per litre
$c_{\text{NaCl,B}}$	concentration of sodium chloride, in parts per million by mass (USC)
$c_{\text{S,A}}$	concentration of sulphide ion, in milligrams per litre
$c_{\text{S,B}}$	concentration of sulphide ion, in parts per million by mass (USC)
$c_{\text{SS,A}}$	suspended solids concentration, in kilograms per cubic metre
$c_{\text{SS,B}}$	suspended solids concentration in pounds per barrel
c_{MBT}	methylene blue capacity
c_{th}	thermometer correction to be added to the working thermometer reading
D	outer diameter
$E_{\text{BE,A}}$	bentonite equivalent, expressed in kilograms per cubic metre

$E_{BE,B}$	bentonite equivalent, expressed in pounds per barrel
f	tube factor from either Table A.1 or Table A.2, for sulfide or carbonate
F_W	fraction (volume fraction) of water
k_{cor}	correction factor
K	cell constant, in metres squared per metre
l_A	submerged length of shear tube, in centimetres
l_B	submerged length of shear tube, in inches
l_{st}	Dräger tube stain length
m_{ds}	mass of the dried sample, in grams
m_s	mass of methylene blue, in grams
m_{st}	mass of shear tube, in grams
m_{tot}	total shear mass, in grams (sum of platform and weights)
m_W	mass of water, in grams
Δm	mass loss, in milligrams
M_f	methyl orange alkalinity of the filtrate
P_{df}	phenolphthalein alkalinity of the drilling fluid
P_f	phenolphthalein alkalinity of the filtrate
q_A	corrosion rate, kilograms per squared metre per year
q_B	corrosion rate, pounds per squared foot per year
r_{df}	drilling fluid resistivity, in ohm metres
r_f	filtrate resistivity, in ohm metres
$R_{QAS/STPB}$	ratio of the concentration of QAS to that of STPB
R_r	resistivity meter reading, in ohms
R_1	average reading for the standard thermometer
R_2	average reading for the working thermometer
$R_{2,cor}$	corrected reading for the working thermometer
R_{300}	viscometer dial reading at 300 r/min
R_{600}	viscometer dial reading at 600 r/min
t	exposure time, in hours
$t_{7,5}$	initial reading taken at 7,5 min

t_{30}	final reading taken at 30 min
V_{df}	volume of drilling fluid sample, in millilitres
V_{EDTA}	volume of EDTA solution, in millilitres
$V_{EDTA,df}$	EDTA volume of whole drilling fluid
$V_{EDTA,f}$	EDTA volume of the drilling fluid filtrate
V_f	volume of the filtrate, in millilitres
V_{mb}	volume of methylene blue solution, in millilitres
V_o	volume of oil, in millilitres
V_{PPT}	PPT volume, in millilitres
V_{RC}	retort cup volume, expressed in millilitres
V_s	volume of the sample, in millilitres
V_{sn}	volume of silver nitrate solution, in millilitres
V_W	volume of water, 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_{st}	static filtration rate (velocity of flow), millilitres per square root of the minutes, in millilitres per minute
$Y_{P,A}$	yield point, in pascals
$Y_{P,B}$	yield point, in pounds per one hundred square feet
γ_A	shear strength, expressed in pascals
γ_B	shear strength, expressed in pounds per hundred square feet
$\Gamma_{DFG,A}$	drilling fluid gradient, expressed in kilopascals per metre
$\Gamma_{DFG,B}$	drilling fluid gradient, in pounds per square inch per foot
η_a	apparent viscosity, in millipascal seconds
$\eta_{p,A}$	plastic viscosity, in millipascal seconds
$\eta_{p,B}$	plastic viscosity, in pounds per one-hundred square feet
θ	temperature
ρ	density, expressed in g/ml when compared to distilled water
ρ_A	density, expressed in kilograms per cubic metre
ρ_{B1}	density, expressed in pounds per gallon

ρ_{B2}	density, expressed in pounds per cubic foot
ρ_b	density of weighting material, in grams per millilitre
ρ_{df}	drilling fluid density, in grams per millilitre
ρ_f	density of filtrate, in grams per millilitre
ρ_g	density of low-gravity solids, in grams per millilitre (use 2,6 if unknown)
ρ_o	density of oil, in grams per millilitre (use 0,8 if unknown)
ρ_w	water density, in grams per millilitre, at test temperature
φ_b	volume fraction of weighting material, in percent
φ_g	volume fraction of low-gravity solids, in percent
φ_o	volume fraction of oil, in percent
φ_s	volume fraction of retort solids, in percent
φ_{ss}	volume fraction of suspended solids, in percent
φ_w	volume fraction of water, in percent

3.2 Abbreviations

AA	atomic absorption spectroscopy
ACS	American Chemical Society
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
BE	bentonite equivalent
CAS	Chemical Abstracts Service
DFG	drilling fluid gradient
DS	drill solids
EDTA	ethylenediaminetetraacetic acid
HTHP	high-temperature, high-pressure
LGS	low-gravity solids
MBT	methylene blue test/capacity
meq	milliequivalents
OCMA	Oil Companies Materials Association (originally, Middle East companies)
PPA	permeability plugging apparatus
PPT	permeability plugging test

PTFE	polytetrafluoroethylene
PV	plastic viscosity, in common oilfield terminology
QAS	quaternary ammonium salt
TC	to contain
TD	to deliver
USC	U.S. Customary units, commonly used in U.S.-based testing

4 Drilling fluid density (mud weight)

4.1 Principle

This test procedure is a method for determining the mass of a given volume of liquid (equivalent to density). Drilling-fluid density is expressed as grams per cubic centimetre or kilograms per cubic metre (pounds per gallon or pounds per cubic foot).

4.2 Apparatus

4.2.1 Density-measuring instrument, of accuracy to within 0,01 g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

The mud balance is the instrument generally used for drilling-fluid density determinations. The mud balance is designed such that the drilling-fluid holding cup, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. Attachments for extending the range of the balance may be used when necessary.

The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m³ (8,33 lb/gal or 62,3 lb/ft³) at 21 °C (70 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

4.2.2 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F).

4.3 Procedure

4.3.1 The instrument base should be set on a flat, level surface.

4.3.2 Measure and record the temperature of the drilling fluid.

4.3.3 Fill the clean, dry cup with drilling fluid to be tested; put the cap on the filled drilling-fluid holding cup and rotate the cap until it is firmly seated. Ensure that some of the drilling fluid is expelled through the hole in the cap in order to free any trapped air or gas (see Annex D for information on air or gas removal).

4.3.4 Holding the cap firmly on the drilling-fluid holding cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

4.3.5 Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the centreline.

4.3.6 Read the drilling fluid density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of g/ml, lb/gal, and lb/ft³, or as a drilling fluid gradient in psi/1 000 ft.

4.4 Calculation

4.4.1 Report the drilling fluid density to the nearest 0,01 g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

4.4.2 Equations (1) to (3) are used to convert the density, ρ , expressed in grams per cubic centimetre to other units:

$$\rho_A = 1\,000 \times \rho \quad (1)$$

where ρ_A is the density, expressed in kilograms per cubic metre.

$$\rho_{B1} = 8,33 \times \rho \quad (2)$$

where ρ_{B1} is the density, expressed in pounds per gallon.

$$\rho_{B2} = 62,3 \times \rho \quad (3)$$

where ρ_{B2} is the density, expressed in pounds per cubic foot.

Table 2 provides the multiplication factor for conversion from one density unit to another.

Equations (4) to (7) are used to convert the density to the drilling fluid gradient, I_{DFG} , expressed in kilopascals per metre (pounds per square inch per foot):

$$I_{DFG,A} = 9,81 \times \rho \quad (4)$$

$$I_{DFG,A} = 0,022\,6 \times \rho \quad (5)$$

$$I_{DFG,B} = 0,052\,0 \times \rho \quad (6)$$

$$I_{DFG,B} = 0,006\,94 \times \rho \quad (7)$$

where

$I_{DFG,A}$ is the drilling fluid gradient, expressed in kilopascals per metre;

$I_{DFG,B}$ is the drilling fluid gradient, expressed in pounds per square inch per foot.

A list of density conversions is given in Table 1.

Table 1 — Density conversion

Grams per cubic centimetre ^a g/ml	Kilograms per cubic metre kg/m ³	Pounds per US gallon (lb/US gal)	Pounds per cubic foot (lb/ft ³)
0,70	700	5,8	43,6
0,80	800	6,7	49,8
0,90	900	7,5	56,1
1,00	1 000	8,345 ^b	62,3
1,10	1 100	9,2	68,5
1,20	1 200	10,0	74,7
1,30	1 300	10,9	81,0

Table 1 (continued)

Grams per cubic centimetre ^a g/ml	Kilograms per cubic metre kg/m ³	Pounds per US gallon (lb/US gal)	Pounds per cubic foot (lb/ft ³)
1,40	1 400	11,7	87,2
1,50	1 500	12,5	93,5
1,60	1 600	13,4	99,7
1,70	1 700	14,2	105,9
1,80	1 800	15,0	112,1
1,90	1 900	15,9	118,4
2,00	2 000	16,7	124,6
2,10	2 100	17,5	130,8
2,20	2 200	18,4	137,1
2,30	2 300	19,2	143,3
2,40	2 400	20,0	149,5
2,50	2 500	20,9	155,8
2,60	2 600	21,7	162,0
2,70	2 700	22,5	168,2
2,80	2 800	23,4	174,4
2,90	2 900	24,2	180,7
^a Same value as relative density.			
^b Accurate conversion factor.			

Table 2 — Conversion of density units

Measured in	Multiply to get			
	g/ml	kg/m ³	lb/gal	lb/ft ³
g/ml	1	1 000	8,33	62,3
kg/m ³	0,001	1	0,008 3	16,026
lb/gal	0,120	120	1	7,49
lb/ft ³	0,016 0	16,03	0,133 5	1

5 Alternative drilling fluid density method

5.1 Principle

The density of a drilling fluid containing entrained air or gas can be determined more accurately by using the pressurized mud balance. The pressurized mud balance is similar in operation to the conventional mud balance, the difference being that the slurry sample can be placed in a fixed-volume sample cup under pressure.

The purpose of placing the sample under pressure is to minimize the effect of entrained air or gas upon slurry density measurements. By pressurizing the sample cup, any entrained air or gas is decreased to a negligible volume, thus providing a slurry density measurement more closely in agreement with that which is realized under downhole conditions.

5.2 Apparatus

5.2.1 Density-measuring instrument, of accuracy to within 0,01 g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

The pressurized mud balance is the instrument generally used for pressurized drilling-fluid density determinations. The pressurized mud balance is designed such that the drilling-fluid holding cup and screw-on lid, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing.

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m³ (8,33 lb/gal or 62,3 lb/ft³) at 21 °C (70 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

5.2.2 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F).

5.3 Procedure

5.3.1 Measure and record the temperature of the drilling fluid.

5.3.2 Fill the sample cup to a level slightly below the upper edge of the cup [approximately 6,5 mm (0,25 in)].

5.3.3 Place the lid on the cup with the attached check-valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Any excess slurry is expelled through the check-valve. When the lid has been placed on the cup, pull the check-valve up into the closed position, rinse off the cup and threads with water and screw the threaded cap on the cup.

5.3.4 The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submerging its end in the slurry with the piston rod completely inside. Then draw the piston rod upward, thereby filling the cylinder with slurry. This volume should be expelled with the plunger action and refilled with fresh slurry sample to ensure that this plunger volume is not diluted with liquid remaining from the last clean-up of the plunger mechanism.

5.3.5 Push the nose of the plunger onto the mating O-ring surface of the cap valve. Pressurize the sample cup by maintaining a downward force on the cylinder housing in order to hold the check-valve down (open) and at the same time to force the piston rod inside. A force of approximately 225 N (50 lbf) or greater should be maintained on the piston rod.

5.3.6 The check-valve in the lid is pressure-actuated; when the inside of the cup is pressurized, the check-valve is pushed upward into the closed position. To close the valve, gradually ease up on the cylinder housing while maintaining pressure on the piston rod. When the check-valve closes, release pressure on the piston rod before disconnecting the plunger.

5.3.7 The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place the instrument on the knife edge. Move the sliding weight to the right or left until the beam is balanced. The beam is balanced when the attached bubble is centred between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of g/ml, lb/gal and lb/ft³, or as a drilling fluid gradient in psi/1 000 ft.

5.3.8 To release the pressure inside the cup, reconnect the empty plunger assembly and push downward on the cylinder housing.

5.3.9 Clean the cup and rinse thoroughly with water. For best operation in water-based slurries, the valve should be greased frequently with waterproof grease.

5.4 Calculation

Report the drilling fluid density to the nearest 0,01 g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

For conversions, use the equations given in 4.4.2.

6 Viscosity and gel strength

6.1 Principle

Viscosity and gel strength are measurements that relate to the flow properties (rheology) of drilling fluids. The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

- a) Marsh funnel — a simple device for indicating viscosity on a routine basis;
- b) direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

NOTE Information on the rheology of drilling fluids can be found in Reference [3].

6.2 Determination of viscosity using the Marsh funnel

6.2.1 Apparatus

6.2.1.1 Marsh funnel, calibrated to out-flow 946 ml (1 quart) of fresh water at a temperature of $(21 \pm 3) ^\circ\text{C}$ [$(70 \pm 5) ^\circ\text{F}$] in $(26 \pm 0,5)$ s, with a graduated cup as a receiver.

6.2.1.1.1 Funnel cone, of length 305 mm (12,0 in), diameter 152 mm (6,0 in) and a capacity to bottom of screen of 1 500 ml (1,6 quarts).

6.2.1.1.2 Orifice, of length 50,8 mm (2,0 in) and inside diameter 4,7 mm (0,188 in = 3/16 in).

6.2.1.1.3 Screen, with 1,6 mm (0,063 in = 1/16 in) openings (12 mesh); fixed at 19,0 mm (0,75 in = 3/4 in) below top of funnel.

6.2.1.2 Graduated cup, with capacity at least 946 ml (1 quart).

6.2.1.3 Stopwatch.

6.2.1.4 Thermometer, with a range of $0 ^\circ\text{C}$ to $105 ^\circ\text{C}$ ($32 ^\circ\text{F}$ to $220 ^\circ\text{F}$).

6.2.2 Procedure

6.2.2.1 Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.

6.2.2.2 Remove finger and start stopwatch. Measure the time for drilling fluid to fill to 946 ml (1 quart) mark of the cup.

6.2.2.3 Measure temperature of the fluid, in degrees Celsius (degrees Fahrenheit).

6.2.2.4 Report the time (6.2.2.2), to the nearest second, as the Marsh funnel viscosity. Report the temperature (6.2.2.3) of fluid to the nearest degree Celsius (degree Fahrenheit).

6.3 Determination of viscosity and/or gel strength using a direct-indicating viscometer

6.3.1 Apparatus

6.3.1.1 Direct-indicating viscometer.

This type of viscometer is a rotational instrument powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity. The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates

displacement of the bob. Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 r/min and 600 r/min.

A direct-indicating viscometer shall meet the following specifications:

a) rotor sleeve:

- inside diameter 36,83 mm (1,450 in);
- total length 87,0 mm (3,425 in);
- scribed line 58,4 mm (2,30 in) above the bottom of sleeve, with two rows of 3,18 mm (0,125 in) holes spaced 120° (2,09 radians) apart, around rotor sleeve just below scribed line;

b) bob, closed, with flat base and tapered top:

- diameter 34,49 mm (1,358 in);
- cylinder length 38,0 mm (1,496 in);

c) torsion spring constant:

- 386 dyne-cm/degree deflection;

d) rotor sleeve speed:

- high speed 600 r/min;
- low speed 300 r/min.

NOTE Other rotor speeds are available in viscometers from various manufacturers.

6.3.1.2 Stopwatch.

6.3.1.3 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F).

6.3.1.4 Suitable container, e.g., the cup provided with the viscometer.

6.3.2 Procedure

6.3.2.1 Place the sample in a container and immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay (within 5 min, if possible) and at a temperature as near as practical to that of the drilling fluid at the place of sampling, but not differing by more than 6 °C (10 °F). The place of sampling should be stated on the test report.

DANGER — Maximum recommended operating temperature is 90 °C (200 °F). If it is necessary to test fluids above this temperature, a solid metal bob or a hollow metal bob with a completely dry interior should be used. Liquid trapped inside a hollow bob can vaporize when immersed in a high-temperature fluid and cause the bob to explode.

6.3.2.2 Record the temperature of the sample.

6.3.2.3 With the sleeve rotating at 600 r/min, wait for viscometer dial reading to reach a steady value (the time required is dependent on the drilling-fluid characteristics). Record the dial reading for 600 r/min.

6.3.2.4 Reduce the rotor speed to 300 r/min and wait for the viscometer dial reading to reach a steady value. Record the dial reading for 300 r/min.

6.3.2.5 Stir drilling fluid sample for 10 s at 600 r/min. Stop the rotor.

6.3.2.6 Allow drilling fluid sample to stand undisturbed for 10 s. Slowly and steadily turn the hand-wheel in the appropriate direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a speed of 3 r/min, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength (10-second gel) in pounds per 100 ft².

NOTE To convert the dial reading to pascals: 1 Pa = 0,511 lb/100 ft².

6.3.2.7 Re-stir the drilling fluid sample at 600 r/min for 10 s, stop the rotor and allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 6.3.2.6 and report the maximum reading as the 10-minute gel in pounds per 100 ft².

NOTE To convert the dial reading to pascals: 1 Pa = 0,511 lb/100 ft².

6.3.3 Calculation

The calculation for the plastic viscosity, η_P , expressed in millipascal seconds (centipoise), is given in Equation (8):

$$\eta_P = R_{600} - R_{300} \quad (8)$$

where

R_{600} is the dial reading at 600 r/min;

R_{300} is the dial reading at 300 r/min.

NOTE 1 The plastic viscosity is commonly known in the industry by the abbreviation PV.

NOTE 2 1 cP = 1 mPa·s.

The calculation for the yield point, $Y_{P,A}$, expressed in pascals, is given in Equation (9):

$$Y_{P,A} = 0,48 \times (R_{300} - \eta_P) \quad (9)$$

When calculating values in USC units, the yield point (expressed in pounds per one hundred square feet) is calculated as follows:

$$Y_{P,B} = R_{300} - \eta_P \quad (10)$$

NOTE 3 The yield point, expressed in pounds per one hundred square feet, is commonly known in the industry by the abbreviation YP.

The calculation for apparent viscosity, η_a , expressed in millipascal seconds (centipoise), is given in Equation (11):

$$\eta_a = \frac{R_{600}}{2} \quad (11)$$

NOTE 4 The apparent viscosity, expressed in millipascal seconds (centipoise), is commonly known in the industry by the abbreviation AV.

7 Filtration

7.1 Principle

Measurement of the filtration behaviour and filter cake-building characteristics of a drilling fluid are fundamental to drilling-fluid control and treatment, as are the characteristics of the filtrate such as oil, water or emulsion content. These characteristics are affected by the types and quantities of solids in the fluid and their physical and chemical interactions which, in turn, are affected by temperature and pressure. Therefore, tests are run at both low pressure/low temperature and high pressure/high temperature, and each requires different equipment and techniques.

7.2 Low-temperature/low-pressure test

7.2.1 Apparatus

7.2.1.1 Filter press, consisting mainly of a cylindrical drilling-fluid cell having an inside diameter of 76,2 mm (3 in) and a height of at least 64,0 mm (2,5 in).

This cell is made of materials resistant to strongly alkaline solutions and is so fitted that a pressure medium can be conveniently admitted into, and bled from, the top. It shall also be fitted such that a sheet of 90 mm (3,54 in) diameter filter paper can be placed in the bottom of the cell just above a suitable support. The filtration area is $(45,8 \pm 0,6) \text{ cm}^2$ $[(7,1 \pm 0,1) \text{ in}^2]$. Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets and the entire assembly supported by a stand. Pressure can be applied with any non-hazardous fluid medium. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midjet pressure cartridges or means for utilizing hydraulic pressure. To obtain correlative results, one thickness of the proper 90 mm diameter filter paper (e.g. Whatman No. 50, S&S No. 576¹⁾ or equivalent) shall be used.

The low-temperature/low-pressure filter press should have a filter area of $45,2 \text{ cm}^2$ to $46,4 \text{ cm}^2$ ($7,0 \text{ in}^2$ to $7,2 \text{ in}^2$) which corresponds to a diameter of 75,86 mm to 76,86 mm (2,987 in to 3,026 in). The filter press gasket is the determining factor of the filter area. It is recommended that a filter press gasket used be tested by a conical gauge that has the maximum 76,86 mm (3,026 in) and the minimum 75,86 mm (2,987 in) diameters marked on it. Any filter-press gasket found out of these ranges (either larger or smaller than the markings) shall be discarded.

NOTE Results obtained from the use of a filter press with different filter area do not directly correlate with the results obtained when using the standard-sized press.

7.2.1.2 Timer, with at least a 30 min interval.

7.2.1.3 Graduated cylinder, with a volume of 10 ml (TC) or 25 ml (TC).

7.2.2 Procedure

7.2.2.1 Be sure each part of the cell, particularly the screen, is clean and dry and that the gaskets are not distorted or worn. Pour the drilling fluid sample into the cell to within 1 cm to 1,5 cm (0,4 in to 0,6 in) of the top (to minimize CO₂ contamination of filtrate), and complete the assembly with the filter paper in place.

7.2.2.2 Place a dry graduated cylinder under the drain tube to collect the filtrate. Close the relief valve and adjust the regulator so that a pressure of $690 \text{ kPa} \pm 35 \text{ kPa}$ ($100 \text{ psi} \pm 5 \text{ psi}$) is applied within 30 s or less. The test period begins at the time of pressure application.

1) Whatman No. 50 and S&S No. 576 are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.

7.2.2.3 At the end of 30 min, measure the volume of filtrate collected. Shut off the flow through the pressure regulator and open the relief valve carefully. The time interval, if other than 30 min, shall be reported.

7.2.2.4 Report the volume of filtrate in millilitres (to the nearest 0,1 ml) and the initial drilling fluid temperature in degrees Celsius (degrees Fahrenheit). Save the filtrate for chemical analysis.

7.2.2.5 Remove the cell from the frame, first making certain that all pressure has been relieved. Carefully save the filter paper with a minimum of disturbance to the cake, disassemble the cell and discard the drilling fluid. Wash the filter cake on the paper with a gentle stream of water.

7.2.2.6 Measure and report the thickness of the filter cake, to the nearest millimetre (1/32 in).

7.2.2.7 Although cake descriptions are subjective, such notations as hard, soft, tough, rubbery, firm, etc., can convey important information of cake quality.

7.3 High-temperature/high-pressure (HTHP) test

7.3.1 Apparatus

7.3.1.1 HT/HP filter press, consisting of a controlled pressure source (CO₂ or nitrogen), regulators, a drilling-fluid cell able to contain working pressures from 4 000 kPa to 8 900 kPa (600 psi to 1 300 psi), a system for heating the cell, a pressurized collection cell able to maintain proper backpressure (see Table 3) in order to prevent flashing or evaporation of the filtrate, and a suitable stand. The drilling-fluid cell has a thermometer well, oil-resistant gaskets, a support for the filter medium and a valve on the filtrate delivery tube to control flow from the cell. It can be necessary to replace the gaskets frequently.

Features such as an overheating safety fuse for the heating jacket and a pressure indicating device on the filtration cell are examples of safety features that are available. Devices are available that aid in cell disassembly when trapped pressures are suspected.

DANGER — Rigid adherence to manufacturers' recommendations as to sample volumes, equipment temperatures and pressures is essential. Failure to do so can result in serious injury.

DANGER — Do not use nitrous oxide cartridges as pressure sources for HT/HP 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.

7.3.1.2 Filter media²⁾.

7.3.1.2.1 Filter paper, Whatman No. 50 or equivalent, for temperatures to 190 °C (375 °F).

7.3.1.2.2 Porous disc, Dynalloy X-5 or equivalent, for temperatures above 200 °C (400 °F). A new disc is required for each test.

7.3.1.3 Timer, with at least a 30 min interval.

7.3.1.4 Thermometer, with a range up to 260 °C (500 °F).

7.3.1.5 Graduated cylinder, with a volume of 25 ml (TC) or 50 ml (TC).

7.3.1.6 High-speed mixer.

2) Whatman No. 50 and Dynalloy X-5 discs are examples of suitable products available commercially. Dynalloy is a trade name of a product supplied by Memtec America Corporation. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.

7.3.2 Procedure for temperatures to 150 °C (300 °F)

7.3.2.1 Place the thermometer in the well in the jacket and preheat to 6 °C (10 °F) above the desired temperature. Adjust the thermostat to maintain the desired temperature.

7.3.2.2 Stir drilling fluid sample for 10 min with a high speed mixer. Close the valve on the drilling-fluid cell and pour the drilling fluid sample into the drilling fluid cell, being careful not to fill closer than 1,5 cm (0,6 in) from the top to allow for expansion. Install the filter paper.

7.3.2.3 Complete the assembly of the cell and, with both top and bottom valves closed, place it in the heating jacket. Transfer the thermometer to the well in the drilling fluid cell.

7.3.2.4 Connect the high-pressure collection cell to the bottom valve and lock in place.

7.3.2.5 Connect a regulated pressure source to the top valve and collection cell, and lock in place.

7.3.2.6 Keeping the valves closed, adjust top and bottom regulators to 690 kPa (100 psi). Open the top valve, applying 690 kPa (100 psi) to the drilling fluid. Maintain this pressure for 1 h. If the drilling-fluid cell temperature has not reached the test temperature after 1 h, the test should be terminated and the equipment repaired.

NOTE An API-funded study has found that some equipment used for high-temperature filtration testing does not adequately heat drilling fluid to the specified test temperature. Modifications of the drilling-fluid cell can be made with the addition of an internal heat sink and insulation to correct this problem [7]. Accurate temperature measurement of the drilling fluid during the heating portion of the test can be ensured by installing a thermocouple to directly measure the drilling fluid temperature in the cell.

7.3.2.7 After 1 h, increase the pressure of the top pressure unit to 4 140 kPa (600 psi) and open the bottom valve to start filtration. Collect the filtrate for 30 min, maintaining the selected temperature within ± 3 °C (± 5 °F). If backpressure rises above 690 kPa (100 psi) during the test, cautiously reduce the pressure by drawing off a portion of the filtrate. Record the total volume collected, the temperature, pressure and time.

7.3.2.8 Correct the filtrate volume to a filter area of 45,8 cm² (7,1 in²). For example, if the filter area is 22,6 cm² (3,5 in²), double the filtrate volume reported.

7.3.2.9 At the end of test, close top and bottom valves on the drilling fluid cell. Bleed pressure from the regulators.

DANGER — Pressure in the drilling fluid cell is still approximately 4 140 kPa (600 psi). To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembling. Difficulty in removing the set screws from the cell can be indicative of pressure remaining in the cell. Cells with pressure-indicating devices that can provide an extra margin of safety are available. Devices are available that aid in cell disassembly when trapped pressures are suspected.

7.3.2.10 Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from cell contents and open. Discard drilling fluid, and retrieve filter cake. Wash filter cake on the paper with a gentle stream of water.

7.3.2.11 Measure and report the thickness of the filter cake, to the nearest millimetre (1/32 in).

7.3.2.12 Although cake descriptions are subjective, such notations as hard, soft, tough, rubbery, firm, etc., can convey important information on cake quality.

7.3.3 Procedure for temperatures above 150 °C (300 °F)

7.3.3.1 Place the thermometer in the well in the jacket and preheat to 6 °C (10 °F) above the desired temperature. Adjust the thermostat to maintain the correct temperature.

7.3.3.2 Stir drilling fluid sample for 10 min with a high speed mixer. Close the valve on the drilling-fluid cell and pour the drilling fluid sample into the drilling fluid cell, being careful not to fill the cell closer than 4 cm (1,5 in) from the top to allow for expansion. Install the proper filter medium (see 7.3.1.2).

DANGER — Not all manufacturers' equipment can be used above 150 °C (300 °F). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions.

All pressure cells should be equipped with manual relief valves. Heating jackets should be equipped with both an overheat safety fuse and thermostatic cut-off. Vapour pressure of the liquid phase of drilling fluids becomes an increasingly critical design factor as test temperatures are raised. Water-vapour pressures at various temperatures are shown in Table 3.

7.3.3.3 Complete the assembly of the cell, and with top and bottom valves closed, place the drilling fluid cell in the heating jacket. Transfer the thermometer to the well in the drilling fluid cell.

7.3.3.4 Connect the high-pressure collection cell to the bottom valve and lock in place.

7.3.3.5 Connect the regulated pressure source to the top valve and the collection cell and lock in place.

7.3.3.6 With top and bottom valves closed, apply the recommended backpressure (see Table 3) for the test temperature to both top and bottom. Open the top valve, applying the same pressure to the drilling fluid while heating. Maintain this pressure for 1 h. If the drilling-fluid cell temperature has not reached the test temperature after 1 h, the test should be terminated and the equipment repaired.

NOTE An API-funded study has found that some equipment used for high-temperature filtration testing does not adequately heat drilling fluid to the specified test temperature. Modifications of the drilling fluid cell can be made with the addition of an internal heat sink and insulation to correct this problem [7]. Accurate temperature measurement of the drilling fluid during the heating portion of the test can be ensured by installing a thermocouple to directly measure the drilling-fluid temperature in the cell.

7.3.3.7 After 1 h, increase the pressure on the top by 3 450 kPa (500 psi) over the backpressure being held and open the bottom valve to begin filtration. Collect the filtrate for 30 min, holding the test temperature within ± 3 °C (± 5 °F) and maintaining the proper backpressure. If the backpressure should begin to rise, it can be reduced by cautiously drawing off a small portion of the filtrate. The sample in the filter cell should never be heated for a period exceeding a total of 1 h.

7.3.3.8 After the test period, close both top and bottom valves on the pressure cell and bleed pressure from the regulators. Allow a minimum of 5 min for the filtrate to cool to avoid vaporizing, then cautiously drain and record the total volume. Also record the temperature, pressures and time.

DANGER — Pressure inside the filter cell can be as high as 6 500 kPa (950 psi). To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembly. Difficulty in removing the set screws from the cell can be indicative of pressure remaining in the cell. Cells with pressure-indicating devices that can provide an extra margin of safety are available. Devices are available that aid in cell disassembly when trapped pressures are suspected.

7.3.3.9 At the end of test, close top and bottom valves on the drilling-fluid cell. Bleed pressure from the regulators.

7.3.3.10 Correct the filtrate volume to a filter area of 45,8 cm² (7,1 in²). For example, if the filter area is 22,6 cm² (3,5 in²), double the filtrate volume reported.

7.3.3.11 Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from cell contents and open. Discard drilling fluid and retrieve filter cake. Wash filter cake on the paper with a gentle stream of water.

7.3.3.12 Measure and report the thickness of the filter cake, to the nearest millimetre (1/32 in).

7.3.3.13 Although cake descriptions are subjective, such notations as hard, soft, tough, rubbery, firm, etc., can convey important information of cake quality.

Table 3 — Recommended minimum backpressure

Test temperature		Vapour pressure		Minimum backpressure	
°C	°F	kPa	psi	kPa	psi
100	212	101	14,7	690	100
120	250	207	30	690	100
150	300	462	67	690	100
Limit of “normal” field testing					
175	350	932	135	1 104	160
200	400	1 704	247	1 898	275
230	450	2 912	422	3 105	450

8 Water, oil and solids contents

8.1 Principle

The retort instrument provides a means for separating and measuring the volumes of water, oil and solids contained in a sample of water-based drilling fluid. In the retort, a known volume of a whole drilling-fluid sample is heated to vaporize the liquid components that are then condensed and collected in a graduated receiver. Liquid volumes are determined directly from reading the oil and water phases in the receiver. The total volume of solids (suspended and dissolved) is obtained by difference (total sample volume minus liquid volume). Calculations are necessary to determine the volume of suspended solids, since any dissolved solids are retained in the retort. The relative volumes of low-gravity solids and weighting material can also be calculated. Knowledge of the solids concentration and composition is considered basic to viscosity and filtration control in water-based drilling fluids.

8.2 Apparatus

8.2.1 Retort instrument.

Retorts of three sizes (10 ml, 20 ml and 50 ml) are commonly available. Specifications for these retorts are given below.

8.2.1.1 Sample cup, in a standard size of 10 ml (precision $\pm 0,05$ ml), 20 ml (precision $\pm 0,10$ ml) or 50 ml (precision $\pm 0,25$ ml).

NOTE Other sample cup sizes are available from manufacturers of this equipment.

8.2.1.2 Liquid condenser, of sufficient mass to cool the oil and water vapours below their vaporization temperature prior to leaving the condenser.

8.2.1.3 Heating element, of sufficient power to raise the temperature of the sample above the vaporization point of the liquid components within 15 min without causing solids boil-over.

8.2.1.4 Temperature control (optional), capable of limiting the temperature of the retort to $500\text{ }^{\circ}\text{C} \pm 40\text{ }^{\circ}\text{C}$ ($930\text{ }^{\circ}\text{F} \pm 70\text{ }^{\circ}\text{F}$).

8.2.2 Liquid receiver (TC), specially designed cylindrical glassware with a rounded bottom to facilitate cleaning and a funnel-shaped top to catch falling drops, meeting the following specifications:

Total volume, ml:	10	20	50
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Precision (0 to 100 %), ml:	$\pm 0,05$	$\pm 0,05$	$\pm 0,25$
Frequency of graduation marks (0 to 100 %), ml:	0,10	0,10	0,50
Calibration: To contain (TC) at 20 °C (68 °F)			
Scale: ml or volume fraction (as percent)			
Material: Transparent and inert to oil, water and salt solutions at temperatures up to 32 °C (90 °F).			

The receiver volume should be verified gravimetrically. The procedure and calculations are provided in Annex H for the 10 ml, 20 ml and 50 ml liquid receivers.

8.2.3 Fine steel wool, oil-free.

“Liquid steel wool” or similar products should not be used for this application.

8.2.4 High-temperature silicone grease, to be used as a thread seal and a lubricant.

8.2.5 Pipe cleaners.

8.2.6 Putty knife or spatula, with blade shaped to fit the inside dimensions of the sample cup of the retort.

8.2.7 Marsh funnel.

8.2.8 Defoaming agent.

8.2.9 Corkscrew.

8.3 Procedure

8.3.1 Be sure that the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use. The inside of the sample cup and lid shall be thoroughly cleaned with a putty knife or spatula prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool. The condenser passage should also be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser can decrease condensation efficiency and cause erroneous liquid readings in a test.

NOTE Procedures vary slightly depending on type of retort used. See manufacturers' instructions for complete procedure.

8.3.2 Collect a representative sample of water-based drilling fluid and allow it to cool to approximately 27 °C (80 °F). Screen the sample through the 1,68 mm (0,066 in) (12 mesh) screen on the Marsh funnel to remove lost-circulation material, large cuttings or debris.

8.3.3 If drilling fluid sample contains gas or air, add two to three drops of defoaming agent to about 300 ml of drilling fluid and stir slowly for 2 min to 3 min to release gases.

8.3.4 Lubricate the threads on the sample cup and condenser tube with a light coating of silicone grease. This prevents vapour loss through the threads and also facilitates disassembly of the equipment and cleaning at the end of the test.

8.3.5 Lightly pack a ring of steel wool into the chamber above the sample cup. Use only enough steel wool to prevent boil-over of solids into the liquid receiver.

NOTE This is determined from experience.

8.3.6 Fill the retort sample cup with degassed water-based drilling fluid, see 8.3.3. See Annex D for information on air or gas removal.

8.3.7 Carefully place the lid on the sample cup and allow an overflow of the sample through the hole in the lid to ensure that the correct volume of sample is in the cup.

8.3.8 With the lid held tightly in place, wipe the overflow from the sample cup and lid. Be sure that the sample cup threads are still covered with silicone grease after wiping, and that the hole in the lid is not plugged.

8.3.9 Screw the retort cup onto the retort chamber with its condenser.

8.3.10 Place a clean, dry, liquid receiver under the condenser discharge tube.

8.3.11 Heat the retort and observe the liquid falling from the condenser. Continue heating for 10 min after the last condensate is collected.

8.3.12 Remove the liquid receiver from the retort. Note whether solids are in the liquid that was recovered. If so, the whole drilling fluid has boiled over from the sample cup and the test shall be repeated from 8.3.6.

8.3.13 Read the volumes of water and oil in the liquid receiver after it has cooled to ambient temperature. Record the volumes (or volume percentages) of water and oil collected.

8.3.14 Cool the retort, remove the steel wool with corkscrew and clean the sample cup with a putty knife or spatula.

8.4 Calculation

8.4.1 Using the measured volumes of oil and water and the volume of the original whole drilling fluid sample (10 ml, 20 ml, or 50 ml), calculate as percentages the volume fractions of water, oil and total solids in the drilling fluid.

a) volume fraction water:

The volume fraction water, φ_W , expressed as a percentage of the total sample volume, is calculated as given in Equation (12):

$$\varphi_W = 100 \times \frac{V_W}{V_{df}} \quad (12)$$

where

V_W is the volume of water, expressed in millilitres;

V_{df} is the volume of the drilling fluid sample, expressed in millilitres.

b) volume fraction oil:

The volume fraction oil, φ_O , expressed as a percentage of the total sample volume, is calculated as given in Equation (13):

$$\varphi_O = 100 \times \frac{V_O}{V_{df}} \quad (13)$$

where

V_O is the volume of oil, expressed in millilitres;

V_{df} is the volume of the drilling fluid sample, expressed in millilitres.

c) volume fraction retort solids:

The volume fraction retort solids, φ_S , expressed as a percentage of the total sample volume, is calculated as given in Equation (14):

$$\varphi_S = 100 - (\varphi_W + \varphi_O) \quad (14)$$

NOTE The percentage (volume fraction) retort solids in Equation (14) is only the difference between water plus oil, and the total sample volume (10 ml, 20 ml or 50 ml). This difference is both suspended solids (weighting material and low-gravity) and dissolved materials (e.g. salt). This percentage (volume fraction) retort solids is the suspended solids only if the drilling fluid is an untreated, fresh-water drilling fluid.

8.4.2 Additional calculations are required to find the percentage (volume fraction) suspended solids and relate them to the relative volumes of low-gravity solids and weighting material. To make these calculations, an accurate drilling fluid density and chloride concentration are needed. The percentage (volume fraction) suspended solids, φ_{SS} , expressed as a percentage of the total sample volume, is calculated as given in Equation (15):

$$\varphi_{SS} = \varphi_S - \left(\frac{\varphi_W \times c_{Cl}}{1680\,000 - 1,21\,c_{Cl}} \right) \quad (15)$$

where

c_{Cl} is the chloride concentration, in milligrams per litre;

φ_W is the percentage (volume fraction) of water;

φ_S is the percentage (volume fraction) of solids.

8.4.3 The volume fraction of low-gravity solids, φ_{lg} , expressed as a percentage of the total sample volume, is calculated as given in Equation (16) for SI units and Equation (17) for USC units:

$$\varphi_{lg} = \frac{1}{(\rho_b - \rho_{lg})} \left[100\,\rho_f + (\rho_b - \rho_f) \times \varphi_{SS} - 100\rho_{df,A} - (\rho_f - \rho_o) \times \varphi_o \right] \quad (16)$$

$$\varphi_{lg} = \frac{1}{(\rho_b - \rho_{lg})} \left[100\,\rho_f + (\rho_b - \rho_f) \times \varphi_{SS} - 12\rho_{df,B} - (\rho_f - \rho_o) \times \varphi_o \right] \quad (17)$$

where

$\rho_{df,A}$ is the drilling fluid density, expressed in grams per cubic centimetre;

$\rho_{df,B}$ is the drilling fluid density, expressed in pounds per gallon;

ρ_f is the density of filtrate, expressed in grams per cubic centimetre, as given by Equation (18):

$$\rho_f = 1 + 0,000\,001\,09 \times c_{Cl} \quad (18)$$

ρ_b is the density of weighting material, expressed in grams per cubic centimetre;

ρ_{lg} is the density of low-gravity solids, expressed in grams per cubic centimetre (use 2,6 if unknown);

ρ_o is the density of oil, expressed in grams per cubic centimetre (use 0,8 if unknown);

NOTE The ρ_f density calculation Equation (18) is based on the sodium chloride concentration.

8.4.4 The volume fraction of the weighting material, φ_b , expressed as a percentage, is calculated as given in Equation (19):

$$\varphi_b = \varphi_{SS} - \varphi_{lg} \quad (19)$$

8.4.5 Concentration, expressed in kilograms per cubic metre, of low-gravity solids, $c_{lg,A}$, weighting material, $c_{b,A}$, and suspended solids, $c_{SS,A}$, can be calculated as given in Equations (20), (21) and (22), respectively:

$$c_{lg,A} = 10\rho_{lg} \times \varphi_{lg} \quad (20)$$

$$c_{b,A} = 10\rho_b \times \varphi_b \quad (21)$$

$$c_{SS,A} = c_{lg,A} + c_{b,A} \quad (22)$$

where

φ_{lg} is the volume fraction of low-gravity solids, expressed as a percentage;

φ_b is the volume fraction of barite, expressed as a percentage.

Concentration, expressed in pounds per barrel, of low-gravity solids, $c_{lg,B}$, weighting material, c_b , and suspended solids, $c_{SS,B}$, can be calculated as given in Equations (23), (24), and (25), respectively:

$$c_{lg,B} = 3,49 \rho_{lg} \times \varphi_{lg} \quad (23)$$

$$c_{b,B} = 3,49 \rho_b \times \varphi_b \quad (24)$$

$$c_{SS,B} = c_{lg,B} + c_{b,B} \quad (25)$$

where

φ_{lg} is the volume fraction of low-gravity solids, expressed as a percentage;

φ_b is the volume fraction of barite, expressed as a percentage.

9 Sand content

9.1 Principle

The sand content of drilling fluid is the percentage (volume fraction) of particles of diameter larger than 74 μm . It is measured by a sand-screen set.

9.2 Apparatus

9.2.1 Sieve, 74 μm (200 mesh) and 63,5 mm (2,5 in) in diameter.

9.2.2 Funnel, to fit sieve.

9.2.3 Glass measuring tube, marked for the volume of drilling fluid to be added and graduated from 0 % to 20 % in order to read the percentage of sand directly.

9.3 Procedure

9.3.1 Fill the glass measuring tube with drilling fluid to the “drilling fluid” mark. Add water to the next mark. Close the mouth of the tube and shake vigorously.

9.3.2 Pour the mixture onto the clean, wet screen. Discard the liquid passing through the screen. Add more water to the tube, shake and again pour onto the screen. Repeat until the tube is clean. Wash the sand retained on the screen to free it of any remaining drilling fluid.

9.3.3 Put the funnel upside down over the top of the sieve. Slowly invert the assembly and insert the tip of the funnel into the mouth of the glass tube. Wash the sand into the tube by playing a fine spray of water through the screen. Allow the sand to settle. From the graduations on the tube, read the volume percent of the sand.

9.3.4 Report the sand content of the drilling fluid as a percentage (volume fraction). Report the source of the drilling fluid sample, i.e. above shaker, suction pit, etc. Coarse solids other than sand (e.g. lost circulation material) are retained on the screen and the presence of such solids should be noted.

10 Methylene blue capacity

10.1 Principle

10.1.1 The methylene blue capacity of drilling fluid is an indication of the amount of reactive clays (bentonite and/or drill solids) present as determined by the methylene blue test. The methylene blue capacity provides an estimate of the total cation-exchange capacity of the drilling fluid solids. Methylene blue capacity and cation-exchange capacity are not necessarily equivalent, the former normally being somewhat less than the actual cation-exchange capacity.

10.1.2 Methylene blue solution is added to a sample of drilling fluid (which has been treated with hydrogen peroxide and acidified) until saturation is noted by formation of a dye “halo” around a drop of solids suspension placed on filter paper. Variations of the procedure used on the drilling fluid can be performed on drill solids and commercial bentonite to allow an estimate of the amount of each type of solid present in the fluid (see ISO 10416^[4] or API RP 13^[5]).

10.1.3 Drilling fluids frequently contain substances in addition to reactive clays that absorb methylene blue. Pretreatment with hydrogen peroxide (see 10.3.2) is intended to remove the effect of organic materials such as lignosulfonates, lignites, cellulosic polymers, polyacrylates, etc.

10.2 Reagents and apparatus

10.2.1 Methylene blue solution, reagent grade methylene blue (CAS No. 61-73-4), 3,20 g/l (1 ml = 0,01 meq).

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 a constant mass at $93\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ ($200\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$). Make the appropriate correction in the mass, m_s , of methylene blue, expressed in grams, to be taken to prepare the solution as given in Equation (26):

$$m_s = \frac{3,2}{m_{ds}} \quad (26)$$

where m_{ds} is the mass of the dried sample, expressed in grams.

10.2.2 Hydrogen peroxide (CAS No. 7722-88-5), 3 % solution.

DANGER — H_2O_2 is a strong oxidizer. Contact with skin should be avoided.

10.2.3 Sulfuric acid (CAS No. 7664-93-9), dilute, approximately 2,5 mol/l (5 N).

DANGER — H_2SO_2 is a strong and toxic acid.

NOTE Normality (N) is not a recognized term, although many drilling fluids equipment manufacturers label the titration, indicator, and buffer solutions with normality.

10.2.4 Syringe, 2,5 ml (TD) or 3 ml (TD).

10.2.5 Erlenmeyer flask, capacity 250 ml.

10.2.6 Burette, 10 ml (TD); **micropipette**, 0,5 ml (TD); or **graduated pipette**, 1 ml (TD).

10.2.7 Graduated cylinder, 50 ml (TD).

10.2.8 Stirring rod.

10.2.9 Hot plate.

10.2.10 Filter paper, Whatman No. 1³⁾ or equivalent.

10.3 Procedure

10.3.1 Add 2,0 ml of drilling fluid (or suitable volume of drilling fluid to require from 2 ml to 10 ml of methylene blue solution) to 10 ml of water in the Erlenmeyer flask. The syringe used should have a capacity of more than 2 ml, generally 2,5 ml or 3 ml. By using a larger syringe, it is not necessary to remove the air trapped in the syringe. To assure that exactly 2,0 ml of drilling fluid is being added, use the following procedure.

- a) Remove the air or gas entrained in the drilling fluid (see Annex D for information on air or gas removal). Stir the drilling fluid to break the gel and quickly draw the drilling fluid into the syringe. Then slowly discharge the syringe back into the drilling fluid, keeping the tip submerged.
- b) Again draw the drilling fluid into the syringe until the end of the plunger is at the last graduation on the syringe (e.g. at the 3-ml line on a 3-ml syringe).
- c) Deliver 2,0 ml of drilling fluid by pushing the plunger until the end of the plunger is exactly 2 ml from the last graduation on the syringe. This, in a 3-ml syringe, is at the 1-ml line.

10.3.2 Add 15 ml of 3 % hydrogen peroxide and 0,5 ml of sulfuric acid. Boil gently for 10 min, but do not allow to boil to dryness. Dilute to about 50 ml with water.

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

10.3.4 When the blue tint spreading from the spot is detected, shake the flask for an additional 2 min and place another drop on the filter paper. If the blue ring is again evident, the final endpoint has been reached, as shown in key item 8 in Figure 1. If the blue ring does not appear as shown in items 4 and 9, continue as before (see 10.3.3) until a drop taken after 2 min shows the blue tint, as shown. Free dye detected immediately after adding 6 ml methylene blue solution is adsorbed after 2 min, and indicates that endpoint has not quite been reached.

3) Whatman No. 1 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.