

BS EN ISO 10414-2:2011



BSI Standards Publication

Petroleum and natural gas industries — Field testing of drilling fluids

Part 2: Oil-based fluids (ISO 10414-2:2011)

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National foreword

This British Standard is the UK implementation of EN ISO 10414-2:2011. It supersedes BS ISO 10414-2:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PSE/17/-/3, Drilling and completion fluids and well cements.

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

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Foreword

This document (EN ISO 10414-2:2011) has been prepared by Technical Committee ISO/TC 67 “Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries” in collaboration with Technical Committee CEN/TC 12 “Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries” the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

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

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Endorsement notice

The text of ISO 10414-2:2011 has been approved by CEN as a EN ISO 10414-2:2011 without any modification.

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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 10414-2 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for the 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 10414-2:2002), which has been technically revised.

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-2:2005, *Recommended practice for field testing of oil-based drilling fluids*.

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

In this part of ISO 10414, quantities expressed in the International System (SI) of units are also, where practical, expressed in United States Customary (USC) units in parentheses for information. The units do not necessarily represent a direct conversion of SI units to USC units, or USC units to SI units. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one degree increments, thus temperature values have been rounded to the nearest degree.

Calibrating an instrument refers to ensuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility, of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate but not precise, precise but not accurate, neither accurate nor precise, or both accurate and precise. A result is valid if it is both accurate and precise.

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

Part 2: Oil-based fluids

1 Scope

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

- a) drilling fluid density (mud weight);
- b) viscosity and gel strength;
- c) filtration;
- d) oil, water and solids concentrations;
- e) alkalinity, chloride concentration and calcium concentration;
- f) electrical stability;
- g) lime and calcium concentrations, calcium chloride and sodium chloride concentrations;
- h) low-gravity solids and weighting material concentrations.

The annexes provide additional test methods or examples that can optionally be used for the determination of:

- shear strength (Annex A);
- oil and water concentrations from cuttings (Annex B);
- drilling fluid activity (Annex C);
- aniline point (Annex D);
- lime, salinity and solids concentration (Annex E);
- sampling, inspection and rejection (Annex F);
- rig-site sampling (Annex G);
- cuttings activity (Annex H);
- active sulphides (Annex I);
- calibration and verification of glassware, thermometers, viscometers, retort kit cups and drilling fluid balances (Annex J);

- permeability plugging apparatus with set-screw secured end cap (Annex K);
- permeability plugging apparatus with threaded end cap (Annex L);
- elastomer compatibility (Annex M);
- sand content of oil-based fluid (Annex N);
- identification and monitoring of weight-material sag (Annex O);
- oil-based drilling fluid test report form (Annex P).

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 10414-1:2008¹⁾, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

ISO 13501²⁾, *Petroleum and natural gas industries — Drilling fluids — Processing equipment evaluation*

API RP 13D:2010, *Recommended practice on the rheology and hydraulics of oil-well drilling fluids*

3 Terms and definitions

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

3.1
ACS reagent grade
grade of chemical meeting the purity standards specified by the American Chemical Society (ACS) and listed in the Chemical Abstracting Service (CAS)

3.2
density of water
density of 1 g/ml (8,334 lb/gal) for deionized or distilled water and 1 g/ml (8,345 lb/gal) for clean tap water

NOTE Deionized or distilled water is used for all equipment calibration. The volume of 1 kg of water is 1 l for the purposes of this part of ISO 10414, and the volume of water is numerically equivalent to the volume of the water measured in cubic centimetres or millilitres, i.e. 1 g = 1 ml.

3.3
spurt loss
volume of fluid that passes through the filtration medium before a filter cake is formed

3.4
pound
U.S. customary unit used to indicate pound-mass (weight), as opposed to pound-force (shear stress)

1) For the purposes of this part of ISO 10414, API RP 13B-1:2009, *Recommended practice for field testing water-based drilling fluids*, is equivalent.

2) For the purposes of this part of ISO 10414, API RP 13C, *Recommended practice on drilling fluids processing systems evaluation*, is equivalent.

3.5

volumic mass

dimensionless ratio of the mass of a volume of an object substance to the mass of the same volume of a reference substance, i.e. the ratio of their respective mass densities

NOTE 1 Generally speaking, the reference substance is pure water.

NOTE 2 Volumic mass is commonly known as specific gravity.

4 Symbols and abbreviated terms

4.1 Symbols

a_{DF}	measure of the chemical potential or reaction availability of drilling fluid
a_W	measure of the chemical potential or reaction availability of water solutions of standard salts
a_C	measure of the chemical potential or reaction availability of drilled cuttings
b	slope of the annular velocity and shear stress at the wall in laminar flow, as defined in O.7.2.8
B_{VSST}	amount of weight-material sag, expressed in pounds-mass per gallon ³⁾
C	correction value to add to thermometer reading
$c_{Ca^{+2},DF}$	whole-drilling-fluid calcium concentration, expressed in milligrams per litre
c_{Ca^{+2},H_2O}	aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per litre
$c_{CaCl_2,AQ}$	aqueous-phase calcium chloride concentration, expressed in milligrams per litre
$c_{CaCl_2,DF,A}$	whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre
$c_{CaCl_2,DF,B}$	whole-drilling-fluid calcium chloride concentration, expressed in pounds per barrel
$c_{CaCl_2,DF,C}$	whole-drilling-fluid calcium chloride concentration, expressed in kilograms per cubic metre
$c_{Ca(OH)_2,\%}$	lime assay value, expressed as a weight fraction
$c_{Ca(OH)_2,DF,B}$	whole-drilling-fluid total lime concentration, expressed in pounds per barrel
$c_{Ca(OH)_2,DF,C}$	whole-drilling-fluid total lime concentration, expressed in kilograms per cubic metre
$c_{Ca(OH)_2,F}$	lime concentration of field lime, expressed in kilograms per cubic metre or pounds per barrel

3) Gallon as used throughout this part of ISO 10414 refers to the U.S. gallon of 3,785 4 litres.

$c_{\text{Cl}^-, \text{DF}}$	whole-drilling-fluid chloride concentration, expressed in milligrams per litre
$c_{\text{Cl}^-(\text{CaCl}_2), \text{DF}}$	whole-drilling-fluid chloride concentration as calcium chloride, expressed in milligrams per litre
$c_{\text{Cl}^-(\text{NaCl}), \text{DF}}$	whole-drilling-fluid chloride concentration from sodium chloride, expressed in milligrams per litre
$c_{\text{NaCl}, \text{AQ}}$	aqueous-phase sodium chloride concentration, expressed in milligrams per litre
$c_{\text{LG}, \text{B}}$	low-gravity solids concentration, expressed in pounds per barrel
$c_{\text{LG}, \text{C}}$	low-gravity solids concentration, expressed in kilograms per cubic metre
$c_{\text{NaCl}, \text{DF}, \text{A}}$	whole-drilling-fluid sodium chloride concentration, expressed in milligrams per litre
$c_{\text{NaCl}, \text{DF}, \text{B}}$	whole-drilling-fluid sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl}, \text{DF}, \text{C}}$	whole-drilling-fluid sodium chloride concentration, expressed in kilograms per cubic metre
$c_{\text{NaCl}, \text{DF}, \text{INSOL}, \text{A}}$	whole-drilling fluid insoluble sodium chloride concentration, expressed in milligrams per litre
$c_{\text{NaCl}, \text{DF}, \text{INSOL}, \text{B}}$	whole-drilling-fluid insoluble sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl}, \text{DF}, \text{SOL}, \text{A}}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in milligrams per litre
$c_{\text{NaCl}, \text{DF}, \text{SOL}, \text{B}}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl}, \text{DF}, \text{SOL}, \text{C}}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in kilograms per cubic metre
$c_{\text{S}-2}$	concentration of active sulfides, expressed in milligrams per litre
$c_{\text{WM}, \text{B}}$	weighting material concentration, expressed in pounds per barrel
$c_{\text{WM}, \text{C}}$	weighting material concentration, expressed in kilograms per cubic metre
d_1	distance from the outer wall, expressed in inches
D	outer pipe diameter or inner diameter of open hole, expressed in inches
d	inner pipe diameter, expressed in inches
D_{TVD}	true vertical depth, expressed in feet
E	pump efficiency, expressed as percentage
f	tube factor, taken from Table I.2
G'	storage modulus, expressed in Newtons per square metre
G''	loss modulus, expressed in Newtons per square metre
k_{C}	consistency factor, expressed in pounds-force per second

L	length of the hydraulic section, expressed in feet
L_A	length, expressed in feet
l	submerged length of shear tube, expressed in centimetres
l_A	submerged length of shear tube, expressed in inches
l_D	Dräger tube darkened length (stained length), marked in units on the tube
m_1	mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams
m_2	mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams
m_3	mass of the empty, dry liquid receiver, expressed in grams
m_4	mass of the cooled liquid receiver with condensed liquids, expressed in grams
m_5	mass of the cooled retort assembly (body packed with steel wool), expressed in grams;
m_d	mass of the dried retort cuttings, expressed in grams
m_{F1}	mass of drilling fluid following shear at 100 r/min, expressed in grams
m_{F2}	mass of drilling fluid taken from Sag Shoe following shear at 100 r/min, expressed in grams
m_{F3}	mass of drilling fluid taken from Sag Shoe following shear at 600 r/min, expressed in grams
m_L	mass of the liquid condensed (oil and water), expressed in grams
m_O	mass of the oil, expressed in grams
m_S	mass of the liquid drilling fluid sample, expressed in grams
m_{st}	mass of shear tube, expressed in grams
m_{tot}	total shear mass (sum of platform and weights), expressed in grams
m_{WC}	mass of the wet cuttings, expressed in grams
m_W	mass of water, expressed in grams
P	measured pressure, expressed in pounds-gauge per square inch
ΔP	anticipated pressure increase, expressed in pounds-gauge per square inch
$\frac{\Delta P}{\Delta L_A}$	pressure gradient, expressed in pounds-gauge per square inch per foot
Q	pump rate, expressed in gallons per minute
R_1	average reading for the standard thermometer, expressed in degrees

R_2	average reading for the working thermometer, expressed in degrees
R_{600}	dial reading at 600 revolutions per minute, expressed in degrees deflection
R_{300}	dial reading at 300 revolutions per minute, expressed in degrees deflection
R_{BPU}	calculated bed pickup measurement ratio, expressed as a percentage
R_{O}	ratio of the volume fraction of oil to the sum of the volume fractions of oil and pure water from the retort analysis, expressed as a percentage
R_{B}	ratio of the volume fraction of oil to the sum of the volume fractions of oil and brine, expressed as a percentage
R_{W}	ratio of the volume fraction of water to the sum of the volume fractions of oil and pure water from the retort analysis, expressed as a percentage
ROC	retained oil on cuttings, expressed in grams per kilogram of cuttings (either wet or dry)
S	Sag Register
t	time, expressed in minutes
V_1	spurt loss, expressed in millilitres
$V_{7,5}$	filtrate volume after 7,5 min, expressed in millilitres
V_{30}	filtrate volume after 30 min, expressed in millilitres
V_{A}	annular volume, expressed in barrels
V_{AgNO_3}	volume of 0,282 mol/l (0,282 N) silver nitrate reagent, expressed in millilitres
V_{B}	base alkalinity demand
V_{EDTA}	volume of 0,1 mol/l EDTA solution, expressed in millilitres
V_{F}	filtrate volume
$V_{\text{H}_2\text{SO}_4}$	volume of 0,05 mol/l (0,1 N) sulfuric acid, expressed in millilitres
V_{K}	whole-drilling-fluid alkalinity, expressed in millilitres of 0,05 mol/l sulfuric acid
V_{M}	receiver volume at specific mark, expressed in millilitres
V_{NaOH}	volume of 0,1 mol/l (0,1 N) NaOH, expressed in millilitres
V_{O}	volume of oil, expressed in millilitres
V_{PPT}	volume of filtrate from PPT, expressed in millilitres
V_{R}	total volume of condensed liquids (oil and water), expressed in millilitres
V_{RC}	volume of retort cup, expressed in millilitres

V_S	drilling fluid sample volume, expressed in millilitres;
V_W	water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2)
Δv_a	change in annular velocity, expressed in feet per minute
v_a	annular velocity, expressed in feet per minute.
v_f	static filtration rate (velocity of flow), expressed in millilitres per minute ^{1/2}
w_{CaCl_2}	aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{CaCl_2,SAT}$	aqueous-phase mass fraction of calcium chloride of a super-saturated fluid, expressed as a percentage of the total aqueous-phase mass
w_{NaCl}	aqueous-phase mass fraction of sodium chloride, expressed as a percentage of the total aqueous phase mass
$w_{NaCl,MAX}$	maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass
$w_{NaCl,MAX-C}$	recalculated maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass
Y_{PA}	yield point, expressed in pascals
Y_{PB}	yield point, expressed in pounds per one hundred square feet, often expressed as YP
β_{10m}	gel strength at 10 min, expressed in pounds per one hundred square feet
β_{10s}	gel strength at 10 s, expressed in pounds per one hundred square feet
$\Gamma_{DFG,A}$	drilling fluid gradient, expressed in kilopascals per metre
$\Gamma_{DFG,B}$	drilling fluid gradient, expressed in pounds per square inch per foot
γ_A	shear strength of the drilling fluid, expressed in pounds per square inch per foot
γ_B	shear strength of the drilling fluid, expressed in pascals
$\dot{\gamma}$	fluid shear rate, expressed in reciprocal seconds
η	drillpipe rotation, expressed in revolutions per minute
η_{AV}	apparent viscosity, expressed in millipascal seconds (centipoises)
η_{PV}	plastic viscosity, expressed in millipascal seconds (centipoises)
φ_B	volume fraction of brine, expressed as a percentage of the whole drilling fluid

φ_D	corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid
φ_d	volume fraction of dried retort solids, expressed as a percentage of the total sample volume
φ_{LG}	volume fraction of the low-gravity solids, expressed as a percentage of the total suspended solids
φ_O	volume fraction of oil, expressed as a percentage of the whole drilling fluid
φ_W	volume fraction of pure water, expressed as a percentage of the whole drilling fluid
φ_{WM}	volume fraction of the weighting material solids, expressed as a percentage of the total suspended solids
ρ	drilling fluid density, expressed in pounds per gallon
ρ_B	aqueous-phase density, expressed in grams per millilitre
ρ_C	drilling fluid density, expressed in kilograms per cubic metre
ρ_{B1}	drilling fluid density, expressed in pounds per gallon
ρ_{B2}	drilling fluid density, expressed in pounds per cubic foot
$\overline{\rho_d}$	average density (volumic mass) of the suspended solids.
$\rho_{ECD-hyd}$	pressure drop and extra density effects of drilled cuttings
$\rho_{ECD-tot}$	total predicted equivalent circulating density
$\Delta\rho_{ECD-rot}$	change in pressure due to rotation
ρ_{LG}	density of the low-gravity solids, expressed in grams per millilitre
ρ_{max}	maximum recorded drilling fluid density, expressed in pounds per gallon
ρ_{nom}	nominal drilling fluid density, expressed in pounds per gallon
ρ_O	density of the oil being used, expressed in grams per millilitre
ρ_S	drilling fluid density, expressed in grams per millilitre
ρ_w	water density, expressed in grams per millilitre, at the test temperature (see Table J.1)
ρ_{WM}	density of the weighting material solids, expressed in grams per millilitre
τ_W	wall shear stress, expressed in pounds-force per hundred square feet
τ_Y	drilling fluid yield stress, expressed in pounds-force per hundred square feet

4.2 Abbreviated terms

ACS	American Chemical Society
API	American Petroleum Institute
ASTM	American Society of Testing Materials
AV	apparent viscosity
CAS	Chemical Abstracting Services
ECD	equivalent circulating density [expressed in kilograms per cubic metre (pounds per gallon)]
EDTA	sodium salt of ethylenediaminetetraacetic acid dihydrate
ERD	extended reach drilling
ES	electrical stability
ESD	equivalent static density [expressed in kilograms per cubic metre (pounds per gallon)]
HTHP	high temperature, high pressure
OCMA	Oil Company Materials Association
OBR	oil-to-brine ratio
OWR	oil-to-water ratio
PNP	propylene glycol normal-propyl ether
PPA	permeability plugging apparatus
PPT	permeability plugging test
PTFE	polytetrafluoroethylene (e.g. Teflon®)
PV	plastic viscosity
PVT	pressure, volume and temperature relationship
SI	International System of units
TC	to contain
TD	to deliver
TVD	true vertical depth [expressed in metres (feet)]
USC	United States Customary units
VSST	Viscometer Sag Shoe Test
YP	yield point

5 Determination of drilling fluid density (mud weight)

5.1 Principle

A procedure is given for determining the mass of a given volume of liquid (i.e. density). The density of drilling fluid is expressed as grams per millilitre, or kilograms per cubic metre (pounds per gallon or pounds per cubic foot).

5.2 Apparatus

5.2.1 Any density-measuring instrument having an accuracy of $\pm 0,01$ 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, e.g. bi-weekly or weekly. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m³ (8,345 lb/gal or 62,4 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. A calibration of the upper density should be performed as specified by the manufacturer, and done on a less frequent basis, e.g. annually.

5.2.2 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of $\pm 0,1$ °C ($\pm 0,2$ °F).

5.3 Procedure

5.3.1 The mud balance instrument shall be set on a flat, level surface.

5.3.2 Measure the temperature of the drilling fluid and record.

5.3.3 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.

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

5.3.5 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.

5.3.6 Read the drilling fluid density from one of the four calibrated scales on the arrow side of the sliding weight. As it is considered that the water density is 1 g/ml (see definitions 3.2 and 3.5), the density can be read directly in units of grams per millilitre using specific gravity scale, pounds per gallon and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 ft.

5.4 Calculation

5.4.1 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).

5.4.2 To convert the reading, ρ , to other units, use Equations (1) to (7) and Tables 1 and 2.

Equations (1) to (3) are used to convert the density, ρ_S , expressed in grams per millilitre, to other units:

$$\rho_C = 1000 \times \rho_S \quad (1)$$

where ρ_C is the drilling fluid density, expressed in kilograms per cubic metre.

$$\rho_{B1} = 8,345 \times \rho_S \quad (2)$$

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

$$\rho_{B2} = 62,4 \times \rho_S \quad (3)$$

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

Table 1 provides the multiplication factor for conversion from one density unit to another.

Table 1 — Conversion of density units

Measured in	Multiply to get			
	g/ml	kg/m ³	lb/gal	lb/ft ³
g/ml	1	1 000	8,345	62,43
kg/m ³	0,001	1	0,008 3	0,062 43
lb/gal	0,120	120	1	7,480 5
lb/ft ³	0,016 0	16,02	0,133 7	1

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

$$\Gamma_{DFG,A} = 9,81 \times g/ml \quad (4)$$

$$\Gamma_{DFG,A} = 0,022 6 \times psi/ft \quad (5)$$

$$\Gamma_{DFG,B} = 0,052 0 \times lb/gal \quad (6)$$

$$\Gamma_{DFG,B} = 0,006 94 \times lb/ft^3 \quad (7)$$

where

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

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

A list of density conversions is given in Table 2.

Table 2 — Density conversion

Grams per cubic centimetre ^a g/ml	Kilograms per cubic metre kg/m ³	Pounds per gallon (lb/gal)	Pounds per cubic foot (lb/ft ³)
0,70	700	5,8	43,7
0,80	800	6,7	49,9
0,90	900	7,5	56,1
1,00	1 000	8,345 ^b	62,4
1,10	1 100	9,2	68,7
1,20	1 200	10,0	74,9
1,30	1 300	10,9	81,1
1,40	1 400	11,7	87,4
1,50	1 500	12,5	93,6
1,60	1 600	13,4	99,9
1,70	1 700	14,2	106,1
1,80	1 800	15,0	112,4
1,90	1 900	15,9	118,6
2,00	2 000	16,7	124,8
2,10	2 100	17,5	131,1
2,20	2 200	18,4	137,3
2,30	2 300	19,2	143,6
2,40	2 400	20,0	149,8
2,50	2 500	20,9	156,1
2,60	2 600	21,7	162,3
2,70	2 700	22,5	168,5
2,80	2 800	23,4	174,8
2,90	2 900	24,2	181,0
^a Same value as relative density as specific gravity in grams per cubic centimetre or kilogram per litre.			
^b Accurate conversion factor.			

6 Alternative method for determination of drilling fluid density

6.1 Principle

6.1.1 The pressurized mud balance provides a more accurate method for determining the density of a drilling fluid containing entrained air or gas than does the conventional mud balance. The pressurized mud balance is similar in operation to the conventional mud balance, the difference being that the drilling fluid sample is placed in a fixed-volume sample cup under pressure.

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

6.2 Apparatus

6.2.1 Any **density-measuring instrument** having an accuracy of $\pm 0,01$ g/ml or 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

A pressurized mud balance is the instrument generally used for density determinations of pressurized drilling fluids. 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.

The instrument should be calibrated frequently with fresh water, e.g. bi-weekly or weekly. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m³ (8,345 lb/gal or 62,4 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. A calibration of the upper density should be performed as specified by the manufacturer, and done on a less frequent basis, e.g. annually.

6.2.2 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ± 1 °C (± 2 °F).

6.3 Procedure

6.3.1 Measure the temperature of the drilling fluid and record.

6.3.2 Fill the sample cup of the pressurized mud balance to a level approximately 6,5 mm (0,25 in) below the upper edge of the cup.

6.3.3 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 drilling fluid will be 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.

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

6.3.5 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 force the piston rod inside. A force of approximately 225 N (50 lb-force) or greater should be maintained on the piston rod.

6.3.6 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.

6.3.7 The pressurized drilling fluid 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 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. As it is considered that the water density is 1 g/ml (see definitions 3.2 and 3.5), the density can be read directly in units of grams per millilitre using specific gravity scale, pounds per gallon, and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 feet.

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

6.3.9 Clean the cup, lid and pump assembly. Rinse thoroughly with base oil.

6.4 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 formulae given in 5.4.2.

7 Viscosity and gel strength

7.1 Principle

7.1.1 Viscosity and gel strength are measurements that relate to the flow properties (rheology) of drilling fluids.

7.1.2 The following instruments are used to measure viscosity and gel strength of drilling fluids for elevated and lower temperature applications. The lower temperature rheology may be anticipated in the riser annulus of deepwater drilling at elevated pressures. The equipment is as follows:

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

NOTE Information on the rheology of drilling fluids can be found in API RP 13D:2010.

7.2 Determination of viscosity using the Marsh funnel

7.2.1 Apparatus

7.2.1.1 Marsh funnel, calibrated to deliver 946 ml (1 quart) of fresh water at a temperature of 21 °C ± 3 °C (70 °F ± 5 °F) in 26 s ± 0,5 s, with a graduated cup as a receiver.

The Marsh funnel shall have the following characteristics:

- a) funnel cone, 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);
- b) orifice, length 50,8 mm (2,0 in) and inside diameter 4,7 mm (0,185 in = 3/16 in);
- c) screen, with 1,6 mm (0,063 in = 1/16 in) openings (12 mesh); fixed at 19,0 mm (0,75 in) below top of funnel.

7.2.1.2 Graduated cup, with a capacity of at least 946 ml (1 quart).

7.2.1.3 Stopwatch.

7.2.1.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±1 °C (±2 °F).

7.2.2 Procedure

7.2.2.1 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.

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

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

7.2.2.4 Report the time (7.2.2.2), to the nearest second, with the volume, as the Marsh funnel viscosity. Report the temperature (7.2.2.3) of the fluid to the nearest degree Celsius (degree Fahrenheit).

7.3 Determination of viscosity and gel strength using a direct-reading viscometer

7.3.1 Apparatus

7.3.1.1 Direct-indicating viscometer, powered by an electric motor or a hand crank.

Drilling fluid is placed 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 should be 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.

The components shall meet the following specifications:

a) Rotor sleeve – R1:

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 rad) apart, around rotor sleeve just below scribed line
sleeve surface:	surface roughness average 16 to 32 cross-hatch honed

b) Bob – B1, closed, with flat base and tapered top:

diameter:	34,49 mm (1,358 in)
cylinder length:	38,0 mm (1,496 in)
rotor surface:	surface roughness average 16 to 32 cross-hatch honed

c) Torsion spring constant – F1,0:

torsional stiffness:	10,54 N·m/rad (386 dyne-cm/degree deflection)
shear stress constant:	29,3 pascals per radian deflection (0,511 pascals per degree of deflection) (1,065 lb/100 ft ² and degree of deflection)

d) Rotor sleeve speeds:

high speed:	600 r/min
low speed:	300 r/min

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

7.3.1.2 Stopwatch.

7.3.1.3 Thermostatically controlled viscometer cup:

a) greater than room temperature: thermostatically controlled direct-heated viscometer cup;

- b) less than room temperature: double-walled viscometer cup connected to a thermostatically controlled refrigerated circulating bath.

7.3.1.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ± 1 °C (± 2 °F).

7.3.2 Procedure

7.3.2.1 Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 50 ml to 100 ml) in the cup for displacement of fluid due to the viscometer bob and sleeve. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of drilling fluid sampling. Testing should be carried out at either $50\text{ °C} \pm 1\text{ °C}$ ($120\text{ °F} \pm 2\text{ °F}$) or $65\text{ °C} \pm 1\text{ °C}$ ($150\text{ °F} \pm 2\text{ °F}$) for reference comparisons to historical data. Testing at a lower temperature, such as $4\text{ °C} \pm 1\text{ °C}$ ($40\text{ °F} \pm 2\text{ °F}$), is recommended for low temperature effects. The place of sampling should be stated in the report.

CAUTION — The maximum recommended operating temperature is 90 °C (190 °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 high-temperature fluid and cause the bob to explode.

7.3.2.2 Heat (or cool) the sample to the selected temperature. Use intermittent or constant shear at 600 r/min to stir the sample while heating (or cooling) to obtain a uniform sample temperature. After the cup temperature reaches the selected temperature, immerse the thermometer into the sample and continue stirring until the sample reaches the selected temperature. Record the temperature of the sample.

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

7.3.2.4 Reduce the rotor speed to 300 r/min and wait for the dial reading to reach steady value. Record the dial reading R_{300} .

7.3.2.5 Stir the drilling fluid sample for 10 s at 600 r/min.

7.3.2.6 Stop the rotor and allow the 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. Record the maximum reading as the initial gel strength. For instruments having a 3 r/min speed, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength, β_{10s} , at 10 s, in pounds per one hundred square feet.

NOTE Gel strength value is calculated from dial reading. 1 degree deflection is equal to 1,065 Pa in SI units and to 0,511 lbf/100 ft² in USC units.

7.3.2.7 Restir the drilling fluid sample at 600 r/min for 10 s, stop the motor and then allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 7.3.2.6 and report the maximum reading as β_{10m} , the 10 m gel, in pounds per one hundred square feet (lbf/100 ft²).

NOTE Gel strength value is calculated from dial reading. 1 degree deflection is equal to 1,065 Pa in SI units and to 0,511 lbf/100 ft² in USC units.

7.3.3 Calculation

7.3.3.1 The dimensions for the rotor, bob and spring constant, as described in 7.3.1.1, determine the following:

- 1° deflection of the bob equals a shear stress of approximately 1 lbf/100 ft², or more exactly $1,065\text{ }100\text{ lbf}/100\text{ ft}^2 = 0,511\text{ Pa}$;
- 1 r/min of the rotor equals a shear rate of $1,702\text{ }3\text{ s}^{-1}$.

The viscosity, defined as shear stress in millipascal divided by shear rate in reciprocal seconds, will be expressed in millipascal seconds (which is equivalent to centipoises). At 300 r/min (shear rate of 511 s^{-1}), the degree deflection will correspond to millipascal seconds (or centipoises).

7.3.3.2 The calculation for the plastic viscosity, η_{PV} , expressed in millipascal seconds (centipoises), is given in Equation (8):

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

where

η_{PV} is the plastic viscosity, expressed in millipascal seconds (centipoises);

R_{600} is the dial reading at 600 revolutions per minute, expressed in degrees deflection;

R_{300} is the dial reading at 300 revolutions per minute, expressed in degrees deflection.

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

NOTE 2 1 cP = 1 mPa.s.

7.3.3.3 The calculation for yield point, Y_{PA} , expressed in pascals, is given in Equation (9):

$$Y_{PA} = 0,48 \times (R_{300} - \eta_{PV}) \quad (9)$$

where

Y_{PA} is the yield point, expressed in pascals.

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

$$Y_{PB} = R_{300} - \eta_{PV} \quad (10)$$

where

Y_{PB} is the yield point, expressed in pounds per one hundred square feet.

NOTE Yield point, in 100 lb/ft², is commonly known in the industry by the abbreviation YP.

7.3.3.5 The calculation for apparent viscosity, η_{AV} , expressed in millipascal seconds (centipoises), is given in Equation (11):

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

where

R_{600} is the dial reading at 600 r/min, expressed in degrees deflection;

η_{AV} is the apparent viscosity, expressed in millipascal seconds (centipoises).

NOTE Apparent viscosity, in millipascal seconds, is commonly known in the industry by the abbreviation AV.

Report the plastic viscosity, yield point, 10 s gel, 10 min gel, and apparent viscosity.

8 Static filtration

8.1 Principle

8.1.1 Measurement of the filtration behaviour and the filter cake characteristics of an oil-based drilling fluid are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as the oil, aqueous phase or emulsion concentration.

8.1.2 Filtration characteristics of an oil-based drilling fluid are affected by the quantity, type and size of solid particles and emulsified aqueous phase in the drilling fluid, and by properties of the liquid phase. Interactions of these various components can be influenced by temperature and pressure.

8.1.3 Filtration tests are performed at high-temperature conditions and under static conditions. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing from 175 °C (350 °F) to 230 °C (450 °F). Use only the filtration equipment and procedure specified for the temperature required.

NOTE 1 No low-temperature filtration test procedure for oil-based drilling fluids is specified herein, but it can be performed much like the water-based drilling fluid test provided in ISO 10414-1.

NOTE 2 For the purposes of this provision, API RP 13B-1 is equivalent.

8.1.4 The 175 ml, 250 ml, or 500 ml unit may be used for static filtration testing up to and including 175 °C (350 °F).

8.1.5 For testing above 175 °C (350 °F), only the 500 ml unit shall be used. It shall be equipped with a thermocouple in direct contact with the fluid contained in the cell to more accurately measure the temperature, and it shall use a porous filter stainless-steel media.

8.2 High-temperature/high-pressure test up to 175 °C (350 °F)

8.2.1 Apparatus

8.2.1.1 High-temperature/high-pressure filter press, consisting of:

- a) filter cell, to contain working pressures up to 9 000 kPa (1 300 psi) at temperature;
- b) pressurized gas source, such as carbon dioxide or nitrogen, with regulators;
NOTE Nitrogen is preferred.
- c) heating system with temperature controller or thermostat, to heat to 175 °C (350 °F);
- d) high-pressure filtrate collection vessel, maintained at proper back-pressure (see Table 3) to avoid flashing or evaporation of the filtrate;
- e) filter cell containing a thermometer well, fitted with a removable end, a filter-media support and with oil-resistant seals.

NOTE Valve stems on each end of the cell can be opened or closed during the test.

CAUTION — Strict adherence to manufacturer's recommendations as to sample volumes, equipment temperatures and pressures is essential. Failure to do so could result in serious injury.

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, in accordance with ISO 10414-1:2008, Clause A.5.

NOTE For the purposes of this provision, API RP 13B-1:2009, Clause A.5, is equivalent.

Table 3 — Recommended minimum back-pressure

Test temperature		Vapour pressure		Minimum back-pressure	
°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

8.2.1.2 Filter medium:

- Whatman No. 50 or S&S 576⁴⁾, or equivalent filter paper, for temperatures up to 200 °C (400 °F);
- porous disc, Dynalloy X-5⁵⁾ or equivalent, for temperatures above 200 °C (400 °F): a new disc is required for each test.

8.2.1.3 Mechanical or electronic timer, with at least a 30 min interval.

8.2.1.4 Thermometer, with a range up to 260 °C (500 °F), and with a 12,5 cm (5 in) or longer stem, or a thermocouple with a range up to 260 °C (500 °F), preferred.

8.2.1.5 Graduated cylinder (TC), long, slender glass tube, with a capacity of 10 ml or 20 ml.

8.2.1.6 Graduated cylinder (TC), optional, with a capacity of 25 ml.

8.2.1.7 Field mixer, cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min.

8.2.1.8 Ruler, graduated in millimetres (inches), to measure filter cake thickness.

8.2.2 Procedure

8.2.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 6 °C (10 °F) above the desired test temperature. Adjust the thermostat to the desired test temperature.

If the filtration unit is equipped with a thermocouple in direct contact with the fluid to measure drilling fluid temperature inside the cell (test temperature), then that temperature should be monitored and reported during the filtration test. Filtration results reported for temperatures so measured may differ from results based on cell wall temperature. Under the “Comments” section, record if the results were based on fluid temperature measured with a direct contact thermocouple.

4) Whatman® No. 50 and S&S® 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.

5) Dynalloy® X-5 disc 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 this product.

8.2.2.2 Stir the drilling fluid sample for 10 min using the field mixer set at the 1 000 r/min speed. Pour the fluid sample into the filter cell, leaving at least a 2,5 cm (1 in) space in the cell to allow for fluid expansion. Install the porous disk [or the filter paper if the test temperature is below 200 °C (400 °F)] in the cell.

8.2.2.3 Complete the assembly of the filter cell, with both top and bottom valves closed, and place it in the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.

8.2.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place. Ensure that the collection vessel is completely free of water or oil.

8.2.2.5 Connect the regulated pressure source to the upper valve. Connect a similar regulated pressure source to the filtrate collection vessel and lock these connections in place.

8.2.2.6 Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 3. Open the upper valve stem, and readjust the upper pressure regulator, if required, to maintain the pressure 690 kPa (100 psi) higher than the minimum back-pressure shown in Table 3 for the test temperature. Maintain both pressures until the test temperature is reached.

NOTE If the time required to reach the test temperature exceeds 1 h, the heater might be defective and the validity of the test is questionable.

8.2.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, set the lower pressure regulator at the pressure ("minimum back-pressure") shown for the temperature given in Table 3. Open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ± 3 °C (± 5 °F) during the test, as indicated by the thermometer in the filter cell. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

8.2.2.8 Collect the filtrate in the long slender graduated cylinder (or optional graduated cylinder). Record the volume of the 30 min total (water plus oil) filtrate. Also record the volumes of solid and aqueous phases, if present.

NOTE The long slender glass cylinder allows more accurate detection and measurements of volumes of oil, water and solids in the filtrate. Heating of the cylinder near an emulsion interface can improve separation of water, solids and oil in the filtrate.

8.2.2.9 Immediately after collecting the 30 min filtrate, turn off to stop heating and unplug the heating jacket from the electrical outlet. Close the upper, then lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION — Pressure in the filter cell can be 6 200 kPa (950 psi), even after the cell is cooled. To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembling.

8.2.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. Ensure that pressure is fully released before dislodging the cap. Carefully disassemble the cell.

8.2.2.11 Pour the liquid from the cell.

8.2.2.12 Remove the filter cake on the porous disk (or the filter paper). Measure the filter cake thickness, at its centre, to the nearest millimetre (1/32 in).

8.2.2.13 Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics under the "Comments" on the Oil Mud Report Form (see Annex P). To minimize settling, the times for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

8.2.3 Calculation

8.2.3.1 The filtrate volume, V_F , should be corrected to a filter area of 4 516 mm² (7,0 in²). HTHP filter cells usually have half the standard filter area (2 258 mm²) (3,5 in²), thus double the observed volume before reporting.

8.2.3.2 Report the cake thickness to the nearest millimetre (1/32 in), its texture and the presence of any emulsion or water in the filtrate.

8.3 High-temperature/high-pressure test 175 °C (350 °F) up to and including 230 °C (450 °F)

8.3.1 Apparatus

8.3.1.1 High-temperature/high-pressure filter press, consisting of the following components:

a) 500 ml volume cell, only;

NOTE For safety reasons, it is advisable that only the 500 ml cell be used for testing up to and above 230 °C (450 °F).

b) filter cell, to contain working pressures up to 15 500 kPa (2 250 psi) at a temperature of 230 °C (450 °F);

c) pressurized gas source, nitrogen with regulators (preferred);

d) heating system with temperature controller or thermostat, to heat to 260 °C (500 °F);

e) high-pressure filtrate collection vessel, maintained at proper back-pressure (see Table 3), to avoid flashing or evaporation of the filtrate;

f) filter cell, equipped with an internal thermocouple to monitor temperature of a drilling fluid sample near its centre in the cell, with removable end fitted with oil-resistant seals.

NOTE Valve stems on each end of the cell can be opened or closed during a test.

CAUTION — 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. The 175 ml and 250 ml filtration cells are not recommended for use at these higher temperatures and pressures.

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 materials. Nitrous oxide cartridges shall be used only for Garrett gas train carbonate analysis, in accordance with ISO 10414-1:2008, Clause A.5.

NOTE For the purposes of this provision, API RP 13B-1:2009, Clause A.5, is equivalent.

8.3.1.2 Filter medium, Dynalloy® X-5 or equivalent porous disc, for temperatures above 200 °C (400 °F). A new disc is required for each test.

8.3.1.3 Mechanical or electronic timer, with at least a 30 min interval.

8.3.1.4 Thermometer, with a range up to 260 °C (500 °F), and with a 12,5 cm (5 in) or longer stem, or a thermocouple with a range up to 260 °C (500 °F), preferred.

8.3.1.5 Graduated cylinder (TC), long, slender with a volume of 10 ml or 20 ml.

8.3.1.6 Graduated cylinder (TC), optional, with a volume of 25 ml.

8.3.1.7 Field mixer, cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min.

8.3.1.8 Ruler, graduated in millimetres (inches), to measure filter cake thickness.

8.3.2 Procedure

8.3.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 6 °C (10 °F) above the desired test temperature. Adjust the thermostat to the test temperature.

If the filtration unit is equipped with a thermocouple in direct contact with the fluid to measure drilling fluid temperature inside the cell (test temperature), then that temperature should be monitored and reported during the filtration test. Filtration results reported for temperatures so measured may differ from results based on cell wall temperature. Under the "Comments" section, record if the results were based on fluid temperature measured with a direct contact thermocouple.

8.3.2.2 Stir the drilling fluid sample for 10 min using the field mixer set at the 1 000 r/min speed. Pour the fluid sample into the filter cell, leaving at least a 2,5 cm (1 in) space in the cell to allow for fluid expansion. Install the filter paper in the cell.

8.3.2.3 Complete the assembly of the filter cell. Install the thermocouple into the cell to monitor the temperature of the fluid near the centre of the cell. Place the cell, with upper and lower valve stems closed, in the heating jacket. Connect the thermocouple to the temperature-readout instrument and determine that it is reading correctly.

8.3.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place. Ensure that the filtrate collection vessel is free of residual water or oil.

8.3.2.5 Connect the pressurized gas source to the upper valve. Connect a similar pressurized gas source to the lower collection vessel and lock these connections in place.

8.3.2.6 Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 3. Open the upper valve stem, and readjust the upper pressure regulator, if required, to maintain the pressure 690 kPa (100 psi) higher than the minimum back-pressure shown in Table 3 for the test temperature. Maintain both pressures until the test temperature is reached.

NOTE If the time required to reach the test temperature exceeds 1 h, the heater might be defective and the validity of the test is questionable.

8.3.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, set the lower pressure regulator at the pressure ("minimum back-pressure") shown for the temperature given in Table 3. Open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ± 3 °C (± 5 °F) during the test, as indicated by the thermometer in the filter cell. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

8.3.2.8 Collect the filtrate in the long slender graduated cylinder (or optional graduated cylinder). Record the volume of the 30 min total (water plus oil) filtrate. Also record the volumes of solid and water phases, if present.

8.3.2.9 Immediately after collecting the 30 min filtrate, turn off to stop heating and unplug the heating jacket from the electrical outlet. Close the upper, then lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the pressurization system. Remove the cell from the heating jacket and allow the cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION — Pressure in the filter cell can be 6 200 kPa (950 psi), even after the cell is cooled. To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembling.

8.3.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. Ensure that pressure is fully released before dislodging the cap. Carefully disassemble the cell.

8.3.2.11 Pour the liquid from the cell.

8.3.2.12 Remove the filter cake on the filter paper. Measure the filter cake thickness, at its centre, to the nearest millimetre (1/32 in).

8.3.2.13 Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics. To minimize settling, the times for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

8.3.3 Calculation

8.3.3.1 The filtrate volume, V_F , should be corrected to a filter area of 4 580 mm² (7,0 in²). HTHP filter cells usually have half the standard filter area (2 258 mm²) (3,5 in²), thus double the observed volume before reporting.

8.3.3.2 Report the cake thickness to the nearest millimetre (1/32 in), the texture and the presence of any emulsion or water in the filtrate.

9 Retort test for oil, water and solids concentrations

9.1 Principle

9.1.1 A retort test measures water and oil released from an oil-based drilling fluid sample when heated in a calibrated and properly operating retort instrument. Included in this clause are procedures for performing a retort analysis using either a volumetric or gravimetric method.

NOTE The gravimetric procedure will provide more accurate values than the standard volumetric approach.

9.1.2 Knowledge of water, oil and solids concentrations is fundamental to proper control of drilling fluid properties such as oil-to-water ratio, rheology, density, filtration and salinity of the aqueous (water) phase. Given that knowledge of solids in an oil-based drilling fluid is essential to the evaluation of solids control equipment, reference shall be made to ISO 13501.

NOTE For the purposes of this provision, API RP 13C is equivalent.

9.1.3 In a retort test, a known volume or mass of oil-based drilling fluid is heated in a retort instrument to vaporize the liquid components. These vapours are then condensed and collected in a precision-graduated liquid receiver.

9.1.4 For the volumetric method, the volume fractions expressed as percentages of oil, water and solids are calculated from the total starting volume of oil-based drilling fluid and the condensed liquid volumes of water and oil collected in a precision-graduated liquid receiver.

9.1.5 For the gravimetric method, the volume fractions expressed as percentages of oil, water and solids are calculated from the mass of retorted oil-based drilling fluid, mass of dry solids after retorting, density of the water, oil, and oil-based drilling fluid plus the measured volume of condensed water collected in a precision-graduated receiver. If the density of the oil used is not known, optional procedures are given for either calculating the density of the oil from mass measurements or for using handheld density-measuring devices.

NOTE This gravimetric method is based on the mass lost during retorting and differs from the volumetric method which uses the recovered volume. This might result in the volume fraction solids values being lower than in the volumetric method, especially with oil-based drilling fluids or where volatile components are not fully condensed with the volumetric method.

9.2 Apparatus

9.2.1 Retort instrument, as specified below.

- a) **Retort assembly**, including a retort body, cup and lid constructed of 303 stainless steel, or equivalent.

Standard cup sizes are 10 ml (precision $\pm 0,05$ ml), 20 ml (precision $\pm 0,1$ ml), and 50 ml (precision $\pm 0,25$ ml). When using the volumetric procedure, the retort cup volume with lid shall be verified gravimetrically in accordance with the procedure and calculations given in ISO 10414-1:2008, Annex H.

NOTE For the purposes of this provision, API RP 13B-1:2009, Annex H, is equivalent.

- b) **Condenser**, capable of cooling the oil and water vapours below their vaporization temperature.
- c) **Heating jacket**, nominal power 350 W.
- d) **Temperature controller**, capable of limiting the temperature of the retort to $500\text{ }^{\circ}\text{C} \pm 40\text{ }^{\circ}\text{C}$ ($930\text{ }^{\circ}\text{F} \pm 70\text{ }^{\circ}\text{F}$).

9.2.2 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:

- **precision**: see Table 4;
- **calibration (TC)**: at $20\text{ }^{\circ}\text{C}$ ($68\text{ }^{\circ}\text{F}$);
- **scale**: millilitre or volume fraction (as a percentage);
- **material**: transparent and inert to oil, water and salt solutions at temperatures up to $32\text{ }^{\circ}\text{C}$ ($90\text{ }^{\circ}\text{F}$).

Table 4 — Precision of liquid receiver

		Total volume			
		10 ml	20 ml	50 ml	50 ml tapered
Precision	(0 % to 100 %)	$\pm 0,05$ ml	$\pm 0,10$ ml	$\pm 0,25$ ml	—
Frequency of graduation marks	(0 % to 100 %)	0,10 ml	0,10 ml	0,50 ml	—
	(0 % to 5 %)	—	—	—	0,05 ml
	(5 % to 100 %)	—	—	—	0,25 ml

When using the volumetric procedure, the receiver volume shall be verified gravimetrically in accordance with the procedure and calculations in ISO 10414-1:2008, Annex H.

NOTE For the purposes of this provision, API RP 13B-1:2009, Annex H, is equivalent.

9.2.3 Fine steel wool, oil-free.

Liquid steel wool or coated steel wool substitutes should not be used for this application.

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

9.2.5 Pipe cleaners.

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

9.2.7 Corkscrew.

9.2.8 Syringe, of capacity 10 ml, 20 ml, or 50 ml, to fill retort cup.

9.2.9 Marsh funnel (7.2.1.1).

9.2.10 Top-loading balance, capable of weighing 2 000 g with an accuracy of $\pm 0,01$ g.

9.2.11 Mud balance (5.2.1).

9.3 Procedure — Volumetric method

9.3.1 Ensure that the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use. Thoroughly clean the inside of the sample cup and lid 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 passage can decrease condensation efficiency which can cause erroneous liquid readings in the test and, for some types of equipment, may present a safety hazard.

CAUTION — A moist or partially clogged condenser passage may be a safety hazard.

9.3.2 The heating jacket should be cooled to less than 93 °C (200 °F).

9.3.3 Pack the retort body with steel wool.

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

9.3.5 Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Be careful not to entrain any air, and ensure that no solids remain on the bottom of the container.

NOTE Air or gas entrapment in the retort sample will result in erroneously high retort solids, due to a reduced volume of drilling fluid sample.

9.3.6 Fill the retort sample cup slowly to avoid air entrapment. Lightly tap the side of the cup to expel air. Place the lid on the cup. Rotate the lid to obtain a proper fit. Ensure that a small excess of drilling fluid flows out of the hole in the lid. Wipe excess sample from the lid; avoid wicking out the drilling fluid through the hole.

9.3.7 Apply lubricant/sealant sparingly to the threads of the retort sample cup. With lid in place, hand-tighten the retort sample cup onto the body.

9.3.8 Apply lubricant/sealant sparingly to the threads on the condenser passage stem and attach to the condenser body. Place the retort assembly into the heating jacket. Close the insulating lid.

9.3.9 Place the clean, dry liquid receiver below the condenser passage outlet.

NOTE 1 To improve the ability to make accurate readings of the volume of oil and water, it is possible to pre-wet the inside of the glass liquid receiver with propylene glycol normal-propyl ether (PNP). PNP is the product used to break the oil-based mud emulsion during chemical titration of oil-based drilling fluid (see Clause 10). The basic method for wetting the glass liquid receiver is to add approximately 0,5 ml of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

NOTE 2 PNP degrades easily. It is advisable that only fresh chemical, within the expiration period noted, be used, and that proper disposal in accordance with local, state and federal regulations be ensued. It is readily biodegradable.

NOTE 3 Liquid or vapour PNP can cause moderate eye irritation and moderate corneal injury. Prolonged skin contact can cause slight irritation with local redness, but is unlikely to result in absorption of harmful amounts. Repeated exposure can cause drying and flaking of the skin, irritation, or a burn. Brief inhalation of PNP (minutes) is not likely to cause adverse effects. Excessive inhalation can cause irritation to the nose and throat. Excessive exposure can cause lethargy. PNP has low toxicity if swallowed. Swallowing large amounts can cause injury. Repeated exposure can cause liver, kidney and eye effects. Birth defects were not observed in laboratory animals.

NOTE 4 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

9.3.10 Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Collect the condensate into the glass liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.

9.3.11 Remove the liquid receiver and allow it to cool.

CAUTION — The retort body is still extremely hot and will cause severe burns if contacted.

NOTE 1 If an emulsion interface is present between the oil and water phases, heating the interface might break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. Carefully heat the glass liquid receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquid. After the emulsion interface is broken, allow the liquid receiver to cool.

Record the total liquid volume, V_R , and water volume, V_W , collected in the liquid receiver.

NOTE 2 Reading the meniscus correctly is extremely important for accuracy. Firstly, always read the meniscus with the interface at eye level. Secondly, for the air-to-liquid meniscus, read the volume at the "lowest point" of the meniscus which is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for the water-to-oil meniscus, read the water volume at the lowest point.

9.3.12 Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser. Clean the retort assembly and condenser.

9.4 Calculation — Volumetric method

9.4.1 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.

9.4.2 Calculate the volume of oil in the condensed sample.

$$V_O = V_R - V_W \quad (12)$$

where

V_O is the volume of oil, expressed in millilitres;

V_R is the total volume of condensed liquids (oil and water), expressed in millilitres;

V_W is the water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2).

9.4.3 Calculate the volume fraction of oil in the total sample.

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

where

φ_O is the volume fraction of oil, expressed as a percentage of the total sample volume;

V_O is the volume of oil, expressed in millilitres;

V_S is the drilling fluid sample volume, expressed in millilitres.

9.4.4 Calculate the volume fraction of water.

$$\varphi_W = 100 \times \frac{V_W}{V_S} \quad (14)$$

where

φ_W is the volume fraction of water, expressed as a percentage;

V_W is the water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2);

V_S is the drilling fluid sample volume, expressed in millilitres.

9.4.5 Calculate the volume fraction of solids remaining in the retort.

$$\varphi_d = 100 - \varphi_W - \varphi_O \quad (15)$$

where

φ_d is the volume fraction of dried retort solids, expressed as a percentage of the total sample volume;

φ_W is the volume fraction of water, expressed as a percentage;

φ_O is the volume fraction of oil, expressed as a percentage of the total sample volume.

NOTE The calculated solids overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity can be made using known volume factors for salt solutions (see Clause 12). Calculation for determining the oil-to-water ratio and the corrected solids concentration and density are also found in Clause 12.

9.5 Procedure — Gravimetric method

9.5.1 Ensure that the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use. Thoroughly clean the inside of the sample cup and lid 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 which can cause erroneous liquid readings in the test and, for some types of equipment, may present a safety hazard.

CAUTION — A moist or partially clogged condenser passage may be a safety hazard.

9.5.2 The heating jacket should be cooled to less than 93 °C (200 °F).

9.5.3 Pack the retort body with steel wool.

9.5.4 Apply lubricant/sealant sparingly to the threads of the retort cup. With lid in place, hand-tighten the retort cup onto the body.

9.5.5 Apply lubricant/sealant sparingly to the threads on the condenser passage stem and attach to the condenser body.

9.5.6 Weigh and record the total mass of the empty retort sample cup, lid, and retort body packed with steel wool. Record this as m_1 , expressed in grams.

NOTE All weights are recorded to the nearest 0,01 g.

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

9.5.8 Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Be careful not to entrain any air, and ensure that no solids remain on the bottom of the container.

NOTE Air or gas entrapment in the retort sample will result in erroneously high retort solids, due to a reduced volume of drilling fluid sample.

9.5.9 Measure and record the density of the oil-based drilling fluid using a mud balance as described in Clause 5 or Clause 6 (more accurate). Alternative gravimetric methods, such as a volumetric flask or cup, are also acceptable.

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

Alternatively, there are several small-volume portable handheld density-measuring devices that can be used to accurately measure the density of the drilling fluid. Examples of such devices are Anton Paar® DNA 35N and Mettler® Densito.⁶⁾

9.5.10 If the density of the oil being used is not known, measure the density of the base oil using a mud balance, as described in Clause 5 or 6 (more accurate). Alternative gravimetric methods, such as a volumetric flask or cup, are also acceptable.

Record the oil density as ρ_O , to the nearest 0,01 g/ml, 10 kg/m³ (0,1 lb/gal or 0,5 lb/ft³).

Alternatively, there are several small-volume portable handheld density-measuring devices that can be used to accurately measure the density of the condensed oil. Example of such devices are Anton Paar® DNA 35N and Mettler® Densito.

9.5.11 Remove the retort cup from the retort body. Fill the retort sample cup slowly to avoid air entrapment. Lightly tap the side of the cup to expel air. Place the lid on the cup. Rotate the lid to obtain a proper fit. Ensure that a small excess of drilling fluid flows out of the hole in the lid. Wipe excess sample from the lid; avoid wicking out the drilling fluid through the hole.

9.5.12 Screw the retort sample cup with lid onto the retort body. Weigh the retort sample cup filled with drilling fluid, lid, and retort body (packed with steel wool). Record this as m_2 , expressed in grams.

NOTE All weights are recorded to the nearest 0,01 g.

6) Anton Paar® DNA 35N and Mettler® Densito 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.

9.5.13 Attach the condenser. Place the retort assembly into the heating jacket. Close the insulating lid.

9.5.14 Weigh an empty, clean, dry liquid receiver. Record this as m_3 , expressed in grams. Place the receiver below the condenser passage outlet.

NOTE 1 To improve the ability to make accurate readings of the volume of oil and water, it is possible to pre-wet the inside of the glass liquid receiver with PNP. PNP is the product used to break the oil-based mud emulsion during chemical titration of oil-based drilling fluid (see Clause 10). The basic method for wetting the glass liquid receiver is to add approximately 0,5 ml of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

NOTE 2 PNP degrades easily. It is advisable that only fresh chemical, within the expiration period noted, be used.

NOTE 3 Due to the rounded bottom of the liquid receiver, it might be helpful to place the liquid receiver in a 100 ml graduated cylinder to hold it on the top-loading balance while being weighed.

NOTE 4 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

9.5.15 Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Collect the condensate into the glass liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.

9.5.16 Remove the liquid receiver and allow it to cool.

CAUTION — The retort body is still extremely hot and will cause severe burns if contacted.

NOTE 1 If an emulsion interface is present between the oil and water phases, heating the interface might break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. Carefully heat the glass liquid receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquid. After the emulsion interface is broken, allow the liquid receiver to cool.

Record the total condensed liquid volume, V_R , and water volume, V_W , collected in the liquid receiver. These are converted to mass, expressed in grams, as described in 9.6.

NOTE 2 Reading the meniscus correctly is extremely important for accuracy. Firstly, always read the meniscus with the interface at eye level. Secondly, for the air-to-liquid meniscus, read the volume at the "lowest point" of the meniscus which is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for the water-to-oil meniscus, read the water volume at the lowest point.

9.5.17 Weigh the glass liquid receiver and its liquid content (oil and water). Record as m_4 , expressed in grams.

9.5.18 Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser.

9.5.19 Remove the condenser and passage stem. Weigh the cooled retort assembly (retort sample cup, lid, and retort body with steel wool packing) without the condenser. Record this as m_5 , expressed in grams.

9.5.20 Clean the retort assembly and condenser.

9.6 Calculation — Gravimetric method

9.6.1 Calculate the mass balance factor of the retorted oil-based drilling fluid, dry retorted solids, and condensed liquids to be certain that they are within ± 3 % for an accurate test.

9.6.2 Calculate the mass of the liquid drilling fluid sample before retorting.

$$m_S = m_2 - m_1 \quad (16)$$

where

m_S is the mass of the liquid drilling fluid sample, expressed in grams;

m_2 is the mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams;

m_1 is the mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams.

9.6.3 Calculate the mass of the dry solids after retorting.

$$m_d = m_5 - m_1 \quad (17)$$

where

m_d is the mass of the dried retort cuttings, expressed in grams;

m_5 is the mass of the cooled retort assembly (body packed with steel wool), expressed in grams;

m_1 is the mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams.

9.6.4 Calculate the mass of liquids (oil and water) condensed after retorting.

$$m_L = m_4 - m_3 \quad (18)$$

where

m_L is the mass of the liquid condensed (oil and water), expressed in grams;

m_4 is the mass of the cooled liquid receiver with condensed liquids, expressed in grams;

m_3 is the mass of the empty, dry liquid receiver, expressed in grams.

9.6.5 Calculate the mass balance of the retorting method. The sum of the mass of dried retorted solids, m_d , and condensed liquids, m_L , after retorting divided by the mass of the beginning oil-based drilling fluid sample, m_S , shall be greater than 0,97 but less than 1,03.

$$0,97 \leq \frac{m_d + m_L}{m_S} \leq 1,03 \quad (19)$$

Repeat the procedure if this requirement is not met.

9.7 Calculation — Volume fractions of oil, water and solids

9.7.1 Calculate the volume fractions of oil, water and solids from the measured mass of retorted oil-based drilling fluid, mass of dry solids, density of the water, density of the oil and density of the oil-based drilling fluid plus the measured volume of condensed water.

9.7.2 Calculate the mass of the oil.

$$m_O = m_2 - m_5 - V_W \quad (20)$$

where

m_O is the mass of the oil, expressed in grams;

m_2 is the mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams;

m_5 is the mass of the cooled retort assembly (body packed with steel wool), expressed in grams;

V_W is the condensed water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2).

NOTE Assuming the density of water is 1 g/ml, the mass, in grams of the volume of water, is numerically equivalent to the volume of the water measured in millilitres.

9.7.3 Calculate the volume fraction of oil.

$$\varphi_O = 100 \times \frac{V_O}{V_S} = 100 \times \frac{m_O / \rho_O}{m_S / \rho_S} \quad (21)$$

where

φ_O is the volume fraction of oil, expressed as a percentage of the total sample volume;

V_O is the volume of oil, expressed in millilitres;

V_S is the drilling fluid sample volume, expressed in millilitres;

m_O is the mass of oil, expressed in grams;

ρ_O is the density of the oil being used, expressed in grams per millilitre;

m_S is the mass of the liquid drilling fluid sample, expressed in grams;

ρ_S is the drilling fluid density, expressed in grams per millilitre.

NOTE Convert density in pounds per gallon to grams per millilitre by dividing the pounds per gallon by 8,345.

9.7.4 Calculate the volume fraction of water.

$$\varphi_W = 100 \times \frac{V_W}{(m_S / \rho_S)} \quad (22)$$

where

φ_W is the volume fraction of water, expressed as a percentage;

V_W is the water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2);

m_S is the mass of the liquid drilling fluid sample, expressed in grams;

ρ_S is the drilling fluid density, expressed in grams per millilitre.

NOTE Convert density in pounds per gallon to grams per millilitre by dividing the pounds per gallon by 8,345.

9.7.5 Calculate the fraction solids.

$$\varphi_d = 100 - (\varphi_W + \varphi_O) \quad (23)$$

where

φ_d is the volume fraction of dried retort solids, expressed as a percentage of the total sample volume;

φ_W is the volume fraction of water, expressed as a percentage;

φ_O is the volume fraction of oil, expressed as a percentage of the total sample volume.

NOTE The calculated solids overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity can be made using known volume factors for salt solution (see Clause 12). Calculations for determining the oil-to-water ratio and the correct solids concentration and density are also found in Clause 12.

9.7.6 Calculate the density of the oil.

$$\rho_O = \frac{m_4 - m_3 - V_W}{V_R - V_W} \quad (24)$$

where

ρ_O is the density of the oil being used, expressed in grams per millilitre;

m_4 is the mass of the cooled liquid receiver with condensed liquids, expressed in grams;

m_3 is the mass of the empty, dry liquid receiver, expressed in grams;

V_R is the total volume of condensed liquid after retorting, expressed in millilitres;

V_W is the condensed water volume, expressed in millilitres, or water mass, expressed in grams (1 ml = 1 g) (see 3.2).

NOTE Assuming the density of water is 1 g/ml, the mass, in grams, of water is numerically equivalent to the volume of the water measured in millilitres.

10 Chemical analysis of oil-based drilling fluids

10.1 Principle

10.1.1 The whole-drilling-fluid alkalinity test procedure is a titration method which measures the volume of standard acid required to react with the alkaline (basic) materials in an oil-based drilling fluid sample. The alkalinity value is used to calculate the concentration of non-reacted "excess" lime in oil-based drilling fluid. Excess alkaline materials, such as lime, help to stabilize the emulsion and also neutralize acidic carbon dioxide or hydrogen sulfide gases.

10.1.2 The whole-drilling-fluid chloride test procedure is a titration method which measures the volume of standard silver nitrate required to react with the chloride (and other halide) ions to form insoluble silver chloride (or silver halide) salts. The test procedure can utilize the same sample as the alkalinity test, provided the sample is acidic (pH below 7,0). The chloride value reported in the whole oil-based drilling fluid can be

assigned to the water phase up to saturation point. The water-soluble salt concentration is related to the effectiveness of an oil-based drilling fluid in controlling shale through the “aqueous phase activity” concept. The aqueous-phase salinity value is also needed to adjust the retort water value to obtain a corrected solids concentration for the oil-based drilling fluid.

10.1.3 The whole-drilling-fluid calcium test is a titration method which measures the volume of a standard calcium-chelating agent (EDTA) required to react with all the calcium (and other water-soluble alkaline earth ions), released from the oil-based drilling fluid into the aqueous phase, when a mixture of solvents is used to extract the oil-based drilling fluid. The calcium measured in this test can come from the calcium chloride (CaCl_2) and lime [CaO or $\text{Ca}(\text{OH})_2$] used to prepare the oil-based drilling fluid, but some calcium could come from drilled up gypsum or anhydrite (CaSO_4).

10.1.4 In addition, some normally insoluble calcium could be measured from drilled up calcium carbonate or from calcium carbonate being used as a drilling fluid additive. The calcium analysis results can be used, along with the chloride and water concentration test, to calculate the CaCl_2 and sodium chloride (NaCl) salinity of the aqueous phase of the drilling fluid.

10.1.5 This calcium analysis excludes magnesium (Mg^{+2}) ions. If Mg^{+2} is expected, special calcium and magnesium ion titration and calculation procedures should be arranged by the drilling fluid engineer and operator.

10.1.6 Procedures for the calculations based on these chemical analyses are provided in Clause 12.

10.1.7 Included in these procedures is an optional filtration step that can remove interfering colour from the fluid sample. An example is when the drilling fluid contains haematite. Filtration makes it easier to see end points for all titrations, including the calcium test even when haematite is not present. However, be aware that the alkalinity may be lowered by filtration.

10.1.8 Care shall be taken when obtaining sample to ensure homogeneity. Settling or separation in the sample container may lead to erroneous test results, so stirring the container may be required. Other factors to consider include shelf-life of test reagent solution, cross-contamination from pipettes and contamination from water other than from distilled or deionized sources. To avoid cross-contamination, pipettes shall never be used for more than one reagent solution. All reagent solutions should be tightly capped when not in use and properly discarded per vendor instructions when the expiration date occurs.

10.1.9 The titration chemicals utilized for alkalinity determination will consume a small amount of alkalinity. This has been labelled “base alkalinity demand”, V_B . This V_B can be determined by using 0,1 mol/l sodium hydroxide to titrate the phenolphthalein end point of the titrating mixture without drilling fluid. Once determined, the V_B can be added to the alkalinity measurement for the oil-based drilling fluid to obtain a more accurate representation of the alkalinity/lime concentration.

NOTE If V_B values are greater than 1 ml of 0,1 mol/l sodium hydroxide, historical data indicates that the PNP might be contaminated or degraded. A new bottle of PNP should be implemented as soon as possible.

10.1.10 The assumption that lime is 100 % active can lead to confusion when comparing alkalinity results to the expected alkalinity value based on lime additions. Field-grade lime is normally less than 100 % active. To reconcile the differences between the calculated versus the added lime, it will be necessary to determine the activity of the alkalinity source (lime). There is a published procedure in ASTM C25 which can be used to assay field lime.

10.2 Reagents and apparatus

10.2.1 Solvent, consisting of propylene glycol normal-propyl ether (PNP) (CAS No. 1569-01-3).

10.2.2 Titration vessel, 400 ml beaker, with cap.

10.2.3 Phenolphthalein (CAS No. 77-09-8) indicator solution, 1 g/100 ml in a 1:1 isopropanol:water solution.

10.2.4 Sulfuric acid (CAS No. 7664-93-9) solution, 0,05 mol/l.

10.2.5 Potassium chromate (CAS No. 7789-00-6) indicator solution, 5 g/100 ml of water.

CAUTION — This product is known to be carcinogenic and should be handled with care.

10.2.6 Silver nitrate (CAS No. 7761-88-8) reagent, $c_{\text{AgNO}_3} = 47,91 \text{ g/l}$ ($c_{\text{Cl}^-} = 0,01 \text{ g/ml}$ or $0,282 \text{ mol/l}$ – $0,282 \text{ N}$), stored in an amber or opaque bottle.

10.2.7 Distilled or deionized water.

10.2.8 Disposable syringe, 5 ml.

10.2.9 Graduated cylinder (TC), 25 ml.

10.2.10 Graduated pipettes (TD), two each of 1 ml and 10 ml.

NOTE One pair of pipettes is for sulfuric acid and one pair is for silver nitrate.

10.2.11 Pipette balloon.

10.2.12 Magnetic stirrer, with 4 cm (1,5 in) stirring bar (coated).

10.2.13 Buffer solution for calcium, $c_{\text{NaOH}} = 1 \text{ mol/l}$ (CAS No. 1310-73-2; NaOH) prepared from fresh “Certified ACS” grade sodium hydroxide, which contains less than 1 % mass fraction sodium carbonate.

Keep buffer solution for calcium in a closed, sealed bottle to minimize absorption of CO_2 from air.

10.2.14 Calcium indicator, of the type Calver® II⁷⁾ or hydroxynaphthol blue (CAS No. 63451-35-4).

10.2.15 EDTA (CAS No. 60-00-4) solution, $c_{\text{EDTA}} = 0,1 \text{ mol/l}$, standardized disodium ethylenediamine-tetraacetate dihydrate (1 ml = 10 000 mg/l CaCO_3 , 1 ml = 4 000 mg/l Ca^{+2}).

NOTE This EDTA solution is ten times more concentrated than that used in water-based drilling fluid testing.

10.3 Base alkalinity demand, V_B

10.3.1 Procedure

10.3.1.1 Add 100 ml of PNP solvent to a 400 ml beaker or pint jar. See the notes to 9.3.9 for further information about PNP.

10.3.1.2 Add 200 ml of distilled (or deionized) water.

10.3.1.3 Add 15 drops of phenolphthalein indicator solution.

10.3.1.4 While stirring with a magnetic stirrer such that the vortex is one-half of the liquid height, titrate immediately with 0,1 mol/l sodium hydroxide until pink colour just appears. Record as V_{NaOH} .

NOTE The stirring technique is important because more vigorous stirring will produce erroneous test results.

10.3.1.5 Continue stirring for 5 min. If the pink colour remains, the end point has been reached. If the pink colour disappears, repeat 10.3.1.4.

7) Calver® II 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 this product.

10.3.2 Calculation

$$V_B = V_{\text{NaOH}} \quad (25)$$

where

V_B is the base alkalinity demand;

V_{NaOH} is the volume of 0,1 mol/l NaOH, expressed in millilitres.

10.4 Whole-drilling-fluid alkalinity, V_K

10.4.1 Procedure

10.4.1.1 Add 100 ml of PNP solvent to the titration vessel.

10.4.1.2 Fill the 5 ml syringe with whole drilling fluid beyond the 3 ml mark.

10.4.1.3 Displace 2,0 ml of whole drilling fluid into the titration vessel. Record as V_S .

10.4.1.4 Swirl the oil-based drilling fluid and solvent until the mixture is homogeneous.

10.4.1.5 Add 200 ml of distilled (or deionized) water. If no filtration is needed, skip to 10.4.1.7.

10.4.1.6 Filtration option:

- 1) Stir the mixture for 5 min with a magnetic stirrer such that the vortex is one-half of the liquid height.
- 2) Pour the mixture into an API low-temperature/low-pressure filter press fitted with filter paper in accordance with ISO 10414-1.

NOTE For the purposes of this provision, API RP 13B-1 is equivalent.

- 3) Close the cell and apply a pressure of 690 kPa (100 psi). Do not use CO₂ cartridges or other CO₂ bottles as the pressure source. The recommended source for gas pressure is nitrogen or nitrous oxide.

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 materials. Nitrous oxide cartridges shall be used only for Garrett gas train carbonate analysis, in accordance with ISO 10414-1:2008, Clause A.5.

- 4) Collect all the filtrate in a clean beaker until the cell blows dry.
- 5) Use all the filtrate and continue with 10.4.1.7.

10.4.1.7 Add 15 drops of phenolphthalein indicator solution (10.2.3).

10.4.1.8 While stirring with a magnetic stirrer such that a vortex is one-half of the liquid height, titrate immediately with 0,05 mol/l sulfuric acid until the pink colour just disappears.

NOTE 1 The stirring technique is important because too vigorous stirring will produce erroneous test results.

NOTE 2 It might be necessary to stop the stirring of the mixture and allow separation of the two phases in order to see the colour in the aqueous phase more clearly.

10.4.1.9 Continue stirring for 5 min. If no pink colour reappears, the end point has been reached. If a pink colour returns, return to 10.4.1.8. Record the volume of 0,05 mol/l sulfuric acid as $V_{\text{H}_2\text{SO}_4}$, in millilitres.

10.4.1.10 Use the volume of 0,05 mol/l sulfuric acid solution, $V_{\text{H}_2\text{SO}_4}$, expressed in millilitres, required to reach the end point in 10.4.1.9, to calculate the whole-drilling-fluid alkalinity, V_K , as the whole-drilling-fluid total lime concentration, $c_{\text{Ca(OH)}_2, \text{DF}}$.

10.4.2 Calculation

$$V_K = \frac{V_{\text{H}_2\text{SO}_4} + V_B}{V_S} \quad (26)$$

$$c_{\text{Ca(OH)}_2, \text{DF}, B} = 1,295 \times V_K \quad (27)$$

$$c_{\text{Ca(OH)}_2, F} = \frac{c_{\text{Ca(OH)}_2, \text{DF}, B}}{c_{\text{Ca(OH)}_2, \%}} \quad (28)$$

where

- V_K is the whole-drilling-fluid alkalinity, expressed in millilitres of 0,05 mol/l sulfuric acid;
- V_B is the base alkalinity demand, expressed in millilitres;
- $V_{\text{H}_2\text{SO}_4}$ is the volume of 0,05 mol/l sulfuric acid, expressed in millilitres;
- V_S is the drilling fluid sample volume, expressed in millilitres;
- $c_{\text{Ca(OH)}_2, \text{DF}, B}$ is the whole-drilling-fluid total lime concentration, expressed in pounds per barrel;
- $c_{\text{Ca(OH)}_2, F}$ is the lime concentration of the field lime, expressed in kilograms per cubic metre or pounds per barrel;
- $c_{\text{Ca(OH)}_2, \%}$ is the lime assay value, expressed as a weight fraction.

NOTE V_K is the equivalent to P_{OM} used in the previous edition of this part of ISO 10414. V_B is the volume of 0,1 mol/l sodium hydroxide solution required to reach a phenolphthalein end point in the titration fluid mixture without any drilling fluid. Field lime refers to the amount of field-grade lime that will be added to a drilling fluid system to achieve a target lime concentration. Field-grade lime is usually less than 100 % active.

10.4.3 The whole-drilling-fluid lime concentration, $c_{\text{Ca(OH)}_2}$, is calculated as follows:

$$c_{\text{Ca(OH)}_2, \text{DF}, C} = 3,705 \times V_K \quad (29)$$

$$c_{\text{Ca(OH)}_2, \text{DF}, B} = 1,299 \times V_K \quad (27)$$

where

- V_K is the whole-drilling-fluid alkalinity, expressed in millilitres of 0,1 N sulfuric acid;
- $c_{\text{Ca(OH)}_2, \text{DF}, B}$ is the whole-drilling-fluid total lime concentration, expressed in pounds per barrel;
- $c_{\text{Ca(OH)}_2, \text{DF}, C}$ is the whole-drilling-fluid total lime concentration, expressed in kilograms per cubic metre.

10.5 Whole-drilling-fluid chloride concentration

10.5.1 Procedure

10.5.1.1 Perform the alkalinity procedures in 10.3 through 10.4. Use this fluid for the chloride concentration analysis.

Make certain the mixture to be titrated for chloride is acidic (below pH 7,0) by adding 10 drops to 20 drops, or more, of 0,05 mol/l sulfuric acid.

10.5.1.2 Add 3 ml of potassium chromate indicator solution (10.2.5).

10.5.1.3 Filtration option:

- 1) Stir the mixture for 5 min with a magnetic stirrer such that the vortex is one-half of the liquid height.
- 2) Pour the mixture into an API low-temperature/low-pressure filter press fitted with filter paper in accordance with ISO 10414-1.

NOTE For the purposes of this provision, API RP 13B-1 is equivalent.

- 3) Close the cell and apply a pressure of 690 kPa (100 psi). Do not use CO₂ cartridges or other CO₂ bottles as the pressure source. The recommended source for gas pressure is nitrogen or nitrous oxide.

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 materials. Nitrous oxide cartridges shall be used only for Garrett gas train carbonate analysis, in accordance with ISO 10414-1:2008, Clause A.5.

- 4) Collect all the filtrate in a clean beaker until the cell blows dry.

- 5) Use all the filtrate and continue with 10.5.1.4.

10.5.1.4 Titrate slowly with 0,282 mol/l (0,282 N) silver nitrate reagent (10.2.6) while stirring rapidly with a magnetic stirrer, until a salmon-pink colour remains stable for at least 1 min.

NOTE 1 It might be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to see the colour in the aqueous phase more clearly.

NOTE 2 Additional potassium chromate indicator solution might be required during the titration process.

10.5.1.5 Use the volume of 0,282 mol/l (0,282 N) silver nitrate reagent required, V_{AgNO_3} , in millilitres, to reach the end point in 10.5.1.3, to calculate the whole-drilling-fluid chloride concentration, $c_{\text{Cl}^-, \text{DF}}$

10.5.2 Calculation

$$c_{\text{Cl}^-, \text{DF}} = \frac{10\,000 \times V_{\text{AgNO}_3}}{V_{\text{S}}} \quad (30)$$

where

$c_{\text{Cl}^-, \text{DF}}$ is the whole-drilling-fluid chloride concentration, in milligrams per litre;

V_{AgNO_3} is the volume of 0,282 mol/l (0,282 N) silver nitrate reagent, in millilitres;

V_{S} is the drilling fluid sample volume, in millilitres.

10.6 Whole-drilling-fluid calcium concentration

10.6.1 Procedure

10.6.1.1 Add 100 ml of the PNP solvent to a titration vessel that has a cap.

10.6.1.2 Fill the 5 ml syringe beyond the 3 ml mark with whole oil-based drilling fluid.

10.6.1.3 Displace 2,0 ml of whole drilling fluid into the titration vessel. Record as V_S .

10.6.1.4 Swirl the oil-based drilling fluid and solvent until the mixture is homogeneous.

10.6.1.5 Add 200 ml distilled or deionized water to the titration vessel.

10.6.1.6 Filtration option:

- 1) If filtration is needed, stir the mixture for 5 min with a magnetic stirrer such that the vortex is on-half of the liquid height.
- 2) Pour the mixture into an API low-temperature/low-pressure filter press fitted with filter paper in accordance with ISO 10414-1.

NOTE For the purposes of this provision, API RP 13B-1 is equivalent.

- 3) Close the cell and apply a pressure of 690 kPa (100 psi). Do not use CO₂ cartridges or other CO₂ bottles as the pressure source.

- 4) Collect all the filtrate in a clean beaker until the cell blows dry.

- 5) Use all the filtrate and continue with 10.6.1.7

10.6.1.7 Add 6,0 ml (1 mol/l) sodium hydroxide buffer solution (10.2.13).

10.6.1.8 Add 0,7 g to 1,0 g Calver®II calcium indicator powder.

10.6.1.9 Place the titration vessel on the magnetic stirrer and drop in a stirring bar.

10.6.1.10 Titrate very slowly, adding 0,1 mol/l EDTA solution, drop by drop, from the pipette while stirring only fast enough to agitate the aqueous (lower) phase without re-mixing the upper and lower phases. A distinct colour change from the reddish colour to a blue-green colour occurs at the end point. Note the volume of EDTA added (V_{EDTA}), in millilitres.

10.6.1.11 Use the volume of EDTA required, V_{EDTA} , to reach the end point in 10.6.1.10, to calculate the whole-drilling-fluid calcium concentration, $c_{\text{Ca}^{+2},\text{DF}}$.

10.6.2 Calculation

$$c_{\text{Ca}^{+2},\text{DF}} = \frac{4\,000 \times V_{\text{EDTA}}}{V_S} \quad (31)$$

where

$c_{\text{Ca}^{+2},\text{DF}}$ is the whole-drilling-fluid calcium concentration, in milligrams per litre;

V_{EDTA} is the volume of 0,1 mol/l EDTA solution, in millilitres;

V_S is the drilling fluid sample volume, in millilitres.

11 Electrical stability test

11.1 Principle

11.1.1 The electrical stability (ES) of an oil-based drilling fluid is a property related to its emulsion stability and oil-wetting capability. ES is determined by applying a voltage-ramped, sinusoidal electrical signal across a pair of parallel, flat-plate electrodes immersed in the drilling fluid. The resulting current remains low until a threshold voltage is reached, whereupon the current rises very rapidly. This threshold voltage is referred to as the ES of the oil-based drilling fluid and is defined as the voltage (in peak volts) measured when the current reaches 61 μA .

NOTE The sine wave specified for the voltage ramp results in more efficient energizing of the fluid and generates considerably lower ES values (often less than half) than the spiky waveform of the instrument described in previous editions of this part of ISO 10414/API RP 13-B. The symmetry of the sinusoidal signal also inhibits build-up of solids on the electrode faces and enhances reproducibility. To further enhance reproducibility, the instrument specifications include automatic voltage ramping at the fixed ramp rate.

11.1.2 Chemical composition and shear history of a drilling fluid control the absolute magnitude of ES in a complex fashion. Consequently, interpreting the oil-wet state of a drilling fluid from single ES measurements is not appropriate. Only trends shall be used in making treatment decisions based on ES values.

11.2 Apparatus

11.2.1 Electrical stability meter, with the following characteristics:

Waveform	sine, < 5 % total harmonic distortion
A.C. frequency	340 Hz $\pm$ 10 Hz
Output units	peak volts
Ramp rate	150 V/s $\pm$ 10 V/s, automatic operation
Range	3 V to 2 000 V (peak)
Trip current	61 μA $\pm$ 5 μA (peak)

11.2.2 Electrodes, with the following characteristics:

Housing	material resistant to oil-based drilling fluid components up to 105 °C (220 °F)
Material	corrosion-resistant metal
Diameter	3,18 mm $\pm$ 0,03 mm (0,125 in $\pm$ 0,001 in)
Spacing (gap)	1,55 mm $\pm$ 0,03 mm (0,061 in $\pm$ 0,001 in) at 22 °C (72 °F)

11.2.3 Calibration resistors/diodes, with the following characteristics:

Number	two (low and high)
Types	standard resistor or Zener diodes
Range	give voltage (ES) readings of: low: 500 V to 1 000 V high: >1 900 V
Accuracy	2 % of expected voltage, temperature corrected by manufacturer's table (if needed and provided)

NOTE The standard resistors are used to check the ratio of readout voltage to trip current. The Zener diodes check the readout voltage directly. Optimum confidence in the performance of the ES meter is obtained from tests with both resistors and diodes.

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

11.2.5 Marsh funnel.

11.2.6 Thermostatically controlled viscometer cup.

11.2.7 Isopropyl alcohol (CAS No. 67-63-0).

11.3 Equipment calibration/performance test

11.3.1 Inspect the electrode probe and cable for evidence of damage.

11.3.2 Ensure that the entire electrode gap is free of deposits, and that the connector to the instrument is clean and dry.

11.3.3 Disconnect the electrode probe (if possible) and run a voltage ramp test, following instructions in the ES meter operating manual. If the meter is working properly, the ES reading should reach the maximum voltage permitted by the instrument.

11.3.4 Reconnect the electrode probe to the ES meter and repeat the voltage ramp test in air. Again, the ES reading should reach the maximum permitted voltage. If not, the electrode probe and connector may need to be cleaned or replaced.

11.3.5 Repeat the voltage ramp test with the electrode probe in tap water. The ES reading should not exceed 3 V. If ES does exceed 3 V, either clean the electrode probe or replace it.

11.3.6 Check the accuracy of the ES meter with the standard resistors or Zener diodes (see the Note to 11.2.3). The ES readings should fall within 2,5 % (combined uncertainty of meter and resistor/diodes) of the expected values. If any of the ES readings fall outside this range, the instrument should be returned to the supplier for adjustment or repair.

NOTE The procedure described in this subclause (11.3) does not check waveform, a.c. frequency or ramp rate. A periodic check of the ES meter and electrode probe by a qualified technician ensures that both units meet all specifications.

11.4 Procedure

11.4.1 Verify equipment calibration/performance (see 11.3).

11.4.2 Screen the drilling fluid sample through a Marsh funnel (see 7.2).

11.4.3 Place the drilling fluid sample in a viscometer cup maintained at $50\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ($120\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$). Record the drilling fluid temperature.

11.4.4 Clean the electrode probe body thoroughly by wiping with a clean paper towel. Pass the towel through the electrode gap a few times. Swirl the electrode probe in the base oil used to formulate the drilling fluid. If the base oil is not available, a different oil or a mild solvent, such as isopropyl alcohol, is acceptable. Clean and dry the electrode probe as before.

NOTE Do not use detergent solutions or aromatic solvents, such as xylene, to clean the electrode probe or cable.

11.4.5 Hand-stir the $50\text{ }^{\circ}\text{C}$ ($120\text{ }^{\circ}\text{F}$) sample with the electrode probe for approximately 10 s to ensure that the composition and temperature of the drilling fluid are uniform. Position the electrode probe so that it does not touch the bottom or sides of the container, and be certain that the electrode surfaces are completely covered by the sample.

NOTE A stand to secure the probe will assist in maintaining the probe off the bottom and away from the sides.

11.4.6 Begin the voltage ramp test. Follow the procedure described in the ES meter operating manual. Do not move the electrode probe during the voltage ramp test.

11.4.7 At the conclusion of the ramp test, note the ES value displayed on the readout device.

11.4.8 Repeat the procedure with the same drilling fluid sample. The two ES values should not differ by more than 5 %. If they differ by more than 5 %, check the meter or electrode probe for malfunction.

11.4.9 Record the average of the two ES measurements.

12 Lime, salinity and solids calculations

12.1 Principle

12.1.1 Lime, salinity and solids calculations using a direct binary salt calculation method that does not require iterations was developed to assist in easier and less complicated calculations. The calculations require the use of a calculator or computer spreadsheet. Equations in this procedure allow calculations of the concentration of the following components for an oil-based drilling fluid:

- a) lime, expressed as $\text{Ca}(\text{OH})_2$, in the whole drilling fluid;
- b) CaCl_2 and NaCl in the whole drilling fluid;
- c) CaCl_2 and NaCl dissolved in the aqueous phase;
- d) low-gravity solids (NaCl salt and drilled solids);
- e) weighting material.

12.1.2 Data listed below and used in the equations are determined from oil-based drilling fluid tests specified in Clauses 5, 9 and 10:

- a) drilling fluid density (see Clause 5);
- b) whole-drilling-fluid alkalinity, V_K (see 10.4);
- c) whole-drilling-fluid calcium concentration, $c_{\text{Ca}^{+2},\text{DF}}$, (see 10.6);
- d) whole-drilling-fluid chloride concentration, $c_{\text{Cl}^-, \text{DF}}$, (see 10.5);
- e) volume of retort oil, V_O , and volume of retort water, V_W , (see Clause 9).

12.1.3 Data known or assumed are as follows:

- a) density of weighting material (in grams per millilitre), ρ_{WM} ;
- b) density of low-gravity solids (in grams per millilitre), ρ_{LG} ;
- c) density of the base oil (in grams per millilitre), ρ_O .

12.1.4 These calculations do not utilize a “lime correction” for adjusting the whole-drilling-fluid calcium concentration as determined by the EDTA titration.

12.1.5 Be certain to use the units requested in the equations. Units for all input data and results for each equation are indicated; calculations should preferably be in SI units although USC units are permitted.

12.1.6 Examples of calculations can be found in Annex E.

12.2 Apparatus

A calculator or computer spreadsheet should be used to perform the mathematical operations detailed in this procedure.

12.3 Whole-drilling-fluid calculations

12.3.1 Whole-drilling-fluid lime concentration

The whole-drilling-fluid lime concentration, $c_{\text{Ca(OH)}_2}$, is calculated as follows (see 10.4.3):

$$c_{\text{Ca(OH)}_2, \text{DF}, \text{C}} = 3,705 \times V_K \quad (29)$$

$$c_{\text{Ca(OH)}_2, \text{DF}, \text{B}} = 1,299 \times V_K \quad (27)$$

where

V_K is the whole-drilling-fluid alkalinity, expressed in millilitres of 0,5 mol/l (0,1 N) sulfuric acid;

$c_{\text{Ca(OH)}_2, \text{DF}, \text{B}}$ is the whole-drilling-fluid total lime concentration, expressed in pounds per barrel;

$c_{\text{Ca(OH)}_2, \text{DF}, \text{C}}$ is the whole-drilling-fluid total lime concentration, expressed in kilograms per cubic metre.

NOTE $c_{\text{Ca(OH)}_2, \text{DF}}$ is often referred to as “excess lime” and the calculation is found in 10.4.2.

12.3.2 Whole-drilling-fluid calcium chloride and sodium chloride concentrations

12.3.2.1 An oil-based drilling fluid normally contains calcium chloride (CaCl_2), and sodium chloride (NaCl). Use the chemical analysis results and the methods below to determine if NaCl and CaCl_2 are present and the concentration of each. For a fluid with a super-saturated aqueous phase, some salt will be present as an insoluble solid.

12.3.2.2 The whole-drilling-fluid chloride ion concentration expressed as total chloride from CaCl_2 , based on the calcium analysis, calculated in milligrams per litre, kilograms per cubic metre or pounds per barrel, is:

$$c_{\text{Cl}^-(\text{CaCl}_2), \text{DF}} = 1,769 \times c_{\text{Ca}^{+2}, \text{DF}} \quad (32)$$

where

$c_{\text{Ca}^{+2}, \text{DF}}$ is the calcium concentration of the whole drilling fluid, expressed in milligrams per litre;

$c_{\text{Cl}^-(\text{CaCl}_2), \text{DF}}$ is the whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre.

12.3.2.3 To determine if sodium chloride is present, compare the whole-drilling-fluid chloride ion concentration, $c_{\text{Cl}^-, \text{DF}}$ determined from the chloride ion analysis, Equation (30), with the whole-drilling-fluid chloride ion concentration $c_{\text{Cl}^-(\text{CaCl}_2), \text{DF}}$, determined from calcium ion analysis, Equation (32).

If $c_{\text{Cl}^-, \text{DF}}$ is greater than $c_{\text{Cl}^-(\text{CaCl}_2), \text{DF}}$, excess chloride ions are indicated and sodium chloride is present.