INTERNATIONAL STANDARD

ISO 10414-2

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

Part 2:
Oil-based fluids

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

Partie 2: Fluides à base d'huiles

Reference number
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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.

INTERNATIONAL STANDARD **ISO 10414-2:2011(E)**

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

ISO 13501[2], *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)

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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
$c_{\mathrm{Ca}^{+2},\mathrm{DF}}$	whole-drilling-fluid calcium concentration, expressed in milligrams per litre
$c_{\mathrm{Ca}^{+2},\mathrm{H_2O}}$	aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per litre
$c_{\mathrm{CaCl_2},\mathrm{AQ}}$	aqueous-phase calcium chloride concentration, expressed in milligrams per litre
$c_{\mathrm{CaCl_2},\mathrm{DF},\mathrm{A}}$	whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre
$c_{\mathrm{CaCl_2},\mathrm{DF},\mathrm{B}}$	whole-drilling-fluid calcium chloride concentration, expressed in pounds per barrel
$c_{\mathrm{CaCl_2},\mathrm{DF},\mathrm{C}}$	whole-drilling-fluid calcium chloride concentration, expressed in kilograms per cubic metre
$c_{\mathrm{Ca(OH)_2},\%}$	lime assay value, expressed as a weight fraction
$c_{\mathrm{Ca(OH)_2},\mathrm{DF},\mathrm{B}}$	whole-drilling-fluid total lime concentration, expressed in pounds per barrel
$c_{\mathrm{Ca(OH)_2},\mathrm{DF},\mathrm{C}}$	whole-drilling-fluid total lime concentration, expressed in kilograms per cubic metre
$c_{\mathrm{Ca(OH)_2},\mathrm{F}}$	lime concentration of field lime, expressed in kilograms per cubic metre or pounds per barrel

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

$c_{\mathrm{Cl^-,DF}}$	whole-drilling-fluid chloride concentration, expressed in milligrams per litre
$c_{\mathrm{Cl^-(CaCl_2),DF}}$	whole-drilling-fluid chloride concentration as calcium chloride, expressed in milligrams per litre
$c_{\mathrm{Cl^-(NaCl),DF}}$	whole-drilling-fluid chloride concentration from sodium chloride, expressed in milligrams per litre
$c_{\mathrm{NaCl,AQ}}$	aqueous-phase sodium chloride concentration, expressed in milligrams per litre
$c_{\mathrm{LG,B}}$	low-gravity solids concentration, expressed in pounds per barrel
$c_{\mathrm{LG,C}}$	low-gravity solids concentration, expressed in kilograms per cubic metre
$c_{\mathrm{NaCl,DF,A}}$	whole-drilling-fluid sodium chloride concentration, expressed in milligrams per litre
$c_{\mathrm{NaCl,DF,B}}$	whole-drilling-fluid sodium chloride concentration, expressed in pounds per barrel
$c_{\mathrm{NaCl,DF,C}}$	whole-drilling-fluid sodium chloride concentration, expressed in kilograms per cubic metre
$c_{\mathrm{NaCl,DF,INSOL,A}}$	whole-drilling fluid insoluble sodium chloride concentration, expressed in milligrams per litre
$c_{\mathrm{NaCl,DF,INSOL,B}}$	whole-drilling-fluid insoluble sodium chloride concentration, expressed in pounds per barrel
$c_{\mathrm{NaCl,DF,SOL,A}}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in milligrams per litre
$c_{\mathrm{NaCl,DF,SOL,B}}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in pounds per barrel
$c_{\mathrm{NaCl,DF,SOL,C}}$	whole-drilling-fluid soluble sodium chloride concentration, expressed in kilograms per cubic metre
$c_{\mathrm{S-2}}$	concentration of active sulfides, expressed in milligrams per litre
$c_{\mathrm{WM,B}}$	weighting material concentration, expressed in pounds per barrel
$c_{\mathrm{WM,C}}$	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
$\frac{\Delta P}{\Delta L_\mathrm{A}}$	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
$V_{\mathrm{AgNO_3}}$	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
$V_{\mathrm{H_2SO_4}}$	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$^{1/2}$
$w_{\mathrm{CaCl_2}}$	aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{\mathrm{CaCl_2,SAT}}$	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
$w_{\mathrm{NaCl,MAX}}$	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
$w_{\mathrm{NaCl,MAX\text{-}C}}$	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
$\Gamma_\mathrm{DFG,A}$	drilling fluid gradient, expressed in kilopascals per metre
$\Gamma_\mathrm{DFG,B}$	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
γ_i	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
$\overline{\rho_{\text{d}}}$	average density (volumic mass) of the suspended solids.
$\rho_{\text{ECD-hyd}}$	pressure drop and extra density effects of drilled cuttings
$\rho_{\text{ECD-tot}}$	total predicted equivalent circulating density
$\Delta\rho_{\text{ECD-rot}}$	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 ±0,01 g/ml or ±10 kg/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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/m^3 (8,345 lb/gal or 62,4 lb/ft^3) 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 ±0,1 °C (±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/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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:

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

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

$$\rho_{B1} = 8{,}345 \times \rho_S \tag{2}$$

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

$$\rho_{B2} = 62{,}4 \times \rho_S \tag{3}$$

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):

$$\Gamma_{DFG,A} = 9{,}81 \times \text{g/ml} \tag{4}$$

$$\Gamma_{DFG,A} = 0{,}022\ 6 \times \text{psi/ft} \tag{5}$$

$$\Gamma_{DFG,B} = 0{,}052\ 0 \times \text{lb/gal} \tag{6}$$

$$\Gamma_{DFG,B} = 0{,}006\ 94 \times \text{lb/ft}^3 \tag{7}$$

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 ±0,01 g/ml or 10 kg/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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/m^3 (8,345 lb/gal or 62,4 lb/ft^3) 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/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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 °C ± 1 °C (120 °F ± 2 °F) or 65 °C ± 1 °C (150 °F ± 2 °F) for reference comparisons to historical data. Testing at a lower temperature, such as 4 °C ± 1 °C (40 °F ± 2 °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 ft^2 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 (lb$_f$/100 ft^2).

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 ft^2 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 lb$_f$/100 ft^2, or more exactly 1,065 100 lb$_f$/100 ft^2 = 0,511 Pa;
- 1 r/min of the rotor equals a shear rate of 1,702 3 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 degress 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):

$$\eta_{PV} = R_{600} - R_{300} \tag{8}$$

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 = 1mPa·s.

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

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

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:

$$Y_{PB} = R_{300} - \eta_{PV} \tag{10}$$

where

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

NOTE Yield point, in 100 lb/ft^2, 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):

$$\eta_{AV} = \frac{R_{600}}{2} \tag{11}$$

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:

a) Whatman No. 50 or S&S 576[4], or equivalent filter paper, for temperatures up to 200 °C (400 °F);

b) porous disc, Dynalloy X-5[5] 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.

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[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 mm^2 (7,0 in^2). HTHP filter cells usually have half the standard filter area (2 258 mm^2) (3,5 in^2), 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 mm^2 (7,0 in^2). HTHP filter cells usually have half the standard filter area (2 258 mm^2) (3,5 in^2), 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 ±0,05 ml), 20 ml (precision ±0,1 ml), and 50 ml (precision ±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 °C ± 40 °C (930 °F ± 70 °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 °C (68 °F);
- **scale**: millilitre or volume fraction (as a percentage);
- **material**: transparent and inert to oil, water and salt solutions at temperatures up to 32 °C (90 °F).

Table 4 — Precision of liquid receiver

		Total volume			
		10 ml	20 ml	50 ml	50 ml tapered
Precision	(0 % to 100 %)	±0,05 ml	±0,10 ml	±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 ±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 ensuled. 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.

$$V_\mathrm{O} = V_\mathrm{R} - V_\mathrm{W} \qquad (12)$$

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.

$$\varphi_{\mathrm{O}} = 100 \times \frac{V_{\mathrm{O}}}{V_{\mathrm{S}}} \tag{13}$$

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.

$$\varphi_{\mathrm{W}} = 100 \times \frac{V_{\mathrm{W}}}{V_{\mathrm{S}}} \tag{14}$$

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.

$$\varphi_{\mathrm{d}} = 100 - \varphi_{\mathrm{W}} - \varphi_{\mathrm{O}} \tag{15}$$

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/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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.

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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.

$$m_\mathrm{S} = m_2 - m_1 \tag{16}$$

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.

$$m_\mathrm{d} = m_5 - m_1 \tag{17}$$

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.

$$m_\mathrm{L} = m_4 - m_3 \tag{18}$$

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.

$$0{,}97 \leqslant \frac{m_\mathrm{d} + m_\mathrm{L}}{m_\mathrm{S}} \leqslant 1{,}03 \tag{19}$$

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.

$$m_O = m_2 - m_5 - V_W \tag{20}$$

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.

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

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.

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

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.

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

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.

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

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 $Ca(OH)_2$] 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, $c_{\mathrm{AgNO_3}} = 47{,}91$ g/l ($c_{\mathrm{Cl}^-} = 0{,}01$ g/ml or 0,282 mol/l – 0,282 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, $c_{\mathrm{NaOH}} = 1$ 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® II[7] or hydroxynaphthol blue (CAS No. 63451-35-4).

10.2.15 EDTA (CAS No. 60-00-4) solution, $c_{\mathrm{EDTA}} = 0{,}1$ 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.

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[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

$$V_B = V_{NaOH} \tag{25}$$

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 CO_2 cartridges or other CO_2 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_{H2SO4}, in millilitres.

10.4.1.10 Use the volume of 0,05 mol/l sulfuric acid solution, $V_{\mathrm{H_2SO_4}}$, 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, $c_{\mathrm{Ca(OH)_2,DF}}$.

10.4.2 Calculation

$$V_{\mathrm{K}} = \frac{V_{\mathrm{H_2SO_4}} + V_{\mathrm{B}}}{V_{\mathrm{S}}} \tag{26}$$

$$c_{\mathrm{Ca(OH)_2,DF,B}} = 1{,}295 \times V_{\mathrm{K}} \tag{27}$$

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

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;
$V_{\mathrm{H_2SO_4}}$	is the volume of 0,05 mol/l sulfuric acid, expressed in millilitres;
V_{S}	is the drilling fluid sample volume, expressed in millilitres;
$c_{\mathrm{Ca(OH)_2,DF,B}}$	is the whole-drilling-fluid total lime concentration, expressed in pounds per barrel;
$c_{\mathrm{Ca(OH)_2,F}}$	is the lime concentration of the field lime, expressed in kilograms per cubic metre or pounds per barrel;
$c_{\mathrm{Ca(OH)_2,\%}}$	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, $c_{\mathrm{Ca(OH)_2}}$, is calculated as follows:

$$c_{\mathrm{Ca(OH)_2,DF,C}} = 3{,}705 \times V_{\mathrm{K}} \tag{29}$$

$$c_{\mathrm{Ca(OH)_2,DF,B}} = 1{,}299 \times V_{\mathrm{K}} \tag{27}$$

where

V_{K}	is the whole-drilling-fluid alkalinity, expressed in millilitres of 0,1 N sulfuric acid;
$c_{\mathrm{Ca(OH)_2,DF,B}}$	is the whole-drilling-fluid total lime concentration, expressed in pounds per barrel;
$c_{\mathrm{Ca(OH)_2,DF,C}}$	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 CO_2 cartridges or other CO_2 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, $V_{\mathrm{AgNO_3}}$, in millilitres, to reach the end point in 10.5.1.3, to calculate the whole-drilling-fluid chloride concentration, $c_{\mathrm{Cl^-,DF}}$

10.5.2 Calculation

$$c_{\mathrm{Cl^-,\,DF}} = \frac{10\,000 \times V_{\mathrm{AgNO_3}}}{V_{\mathrm{S}}} \qquad (30)$$

where

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

$V_{\mathrm{AgNO_3}}$ 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 CO_2 cartridges or other CO_2 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, $c_{Ca^{+2},DF}$.

10.6.2 Calculation

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

where

$c_{Ca^{+2},DF}$ 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 ± 10 Hz
Output units	peak volts
Ramp rate	150 V/s ± 10 V/s, automatic operation
Range	3 V to 2 000 V (peak)
Trip current	61 µA ± 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 ± 0,03 mm (0,125 in ± 0,001 in)
Spacing (gap)	1,55 mm ± 0,03 mm (0,061 in ± 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 °C ± 2 °C (120 °F ± 5 °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 °C (120 °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 $Ca(OH)_2$, 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, $c_{Ca^{+2},DF}$, (see 10.6);

d) whole-drilling-fluid chloride concentration, $c_{Cl^-,DF}$, (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):

$$c_{\text{Ca(OH)}_2\text{,DF,C}} = 3{,}705 \times V_\text{K} \tag{29}$$

$$c_{\text{Ca(OH)}_2\text{,DF,B}} = 1{,}299 \times V_\text{K} \tag{27}$$

where

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

$c_{\text{Ca(OH)}_2\text{,DF,B}}$ 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.

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:

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

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.

If sodium chloride is present, the whole-drilling-fluid chloride ion concentration from NaCl is calculated as follows:

$$c_{\mathrm{Cl^-(NaCl),DF}} = c_{\mathrm{Cl^-,DF}} - c_{\mathrm{Cl^-(CaCl_2),DF}} \tag{33}$$

where

$c_{\mathrm{Cl^-(NaCl),DF}}$ is the whole-drilling-fluid chloride ion concentration from NaCl, expressed in milligrams per litre;

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

$c_{\mathrm{Cl^-(CaCl_2),DF}}$ is the whole-drilling-fluid chloride concentration based on $CaCl_2$, expressed in milligrams per litre.

If $c_{\mathrm{Cl^-(NaCl),DF}}$ is a positive value, then NaCl is present in the drilling fluid. Proceed to 12.3.2.5, Equation (37).

If $c_{\mathrm{Cl^-(NaCl),DF}}$ is a negative value, then only $CaCl_2$ is present in the drilling fluid and no NaCl is present.

12.3.2.4 When no sodium chloride is present, the whole-drilling-fluid chloride analysis is more reliable. The whole-drilling-fluid $CaCl_2$ concentration [see Equation (30)] is based on the chloride ion analysis, as follows:

$$c_{\mathrm{CaCl_2,DF,A}} = 1{,}565 \times c_{\mathrm{Cl^-,DF}} \tag{34}$$

$$c_{\mathrm{CaCl_2,DF,B}} = 0{,}000\ 548\ 5 \times c_{\mathrm{Cl^-,DF}} \tag{35}$$

$$c_{\mathrm{CaCl_2,DF,C}} = 0{,}001\ 565 \times c_{\mathrm{Cl^-,DF}} \tag{36}$$

where

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

$c_{\mathrm{CaCl_2,DF,A}}$ is the whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre;

$c_{\mathrm{CaCl_2,DF,B}}$ is the whole-drilling-fluid calcium chloride concentration, expressed in pounds per barrel;

$c_{\mathrm{CaCl_2,DF,C}}$ is the whole-drilling-fluid calcium chloride concentration, expressed in kilograms per cubic metre.

For fluids with no sodium chloride, proceed to 12.4.

12.3.2.5 For fluids with both sodium chloride and calcium chloride, the whole-drilling-fluid $CaCl_2$ concentration is calculated from the whole-drilling-fluid calcium ion analysis [see Equation (31)] as follows:

$$c_{\mathrm{CaCl_2,DF,A}} = 2{,}769 \times c_{\mathrm{Ca^{+2},DF}} \tag{37}$$

$$c_{\mathrm{CaCl_2,DF,B}} = 0{,}000\ 970\ 7 \times c_{\mathrm{Ca^{+2},DF}} \tag{38}$$

$$c_{\mathrm{CaCl_2,DF,C}} = 0{,}002\ 769 \times c_{\mathrm{Ca^{+2},DF}} \tag{39}$$

where

$c_{\mathrm{Ca}^{+2},\mathrm{DF}}$	is the whole-drilling-fluid calcium concentration, expressed in milligrams per litre;
$c_{\mathrm{CaCl}_2,\mathrm{DF,A}}$	is the whole-drilling-fluid $CaCl_2$ concentration, expressed in milligrams per litre;
$c_{\mathrm{CaCl}_2,\mathrm{DF,B}}$	is the whole-drilling-fluid $CaCl_2$ concentration, expressed in pounds per barrel;
$c_{\mathrm{CaCl}_2,\mathrm{DF,C}}$	is the whole-drilling-fluid $CaCl_2$ concentration, expressed in kilograms per cubic metre.

12.3.2.6 The whole-drilling-fluid NaCl concentration is calculated as follows:

$$c_{\mathrm{NaCl,DF,A}} = 1{,}648 \times c_{\mathrm{Cl}^-(\mathrm{NaCl}),\mathrm{DF}} \tag{40}$$

$$c_{\mathrm{NaCl,DF,B}} = 0{,}000\,578 \times c_{\mathrm{Cl}^-(\mathrm{NaCl}),\mathrm{DF}} \tag{41}$$

$$c_{\mathrm{NaCl,DF,C}} = 0{,}001\,648 \times c_{\mathrm{Cl}^-(\mathrm{NaCl}),\mathrm{DF}} \tag{42}$$

where

$c_{\mathrm{Cl}^-(\mathrm{NaCl}),\mathrm{DF}}$	is the whole-drilling-fluid chloride concentration from sodium chloride, in milligrams per litre;
$c_{\mathrm{NaCl,DF,A}}$	is the whole-drilling-fluid sodium chloride concentration, in milligrams per litre;
$c_{\mathrm{NaCl,DF,B}}$	is the whole-drilling-fluid sodium chloride concentration, in pounds per barrel;
$c_{\mathrm{NaCl,DF,C}}$	is the whole-drilling-fluid sodium chloride concentration, in kilograms per cubic metre.

12.4 Aqueous phase calculations

12.4.1 The calculations in 12.3 involved the chemical concentrations in the whole drilling fluid. The calculations in this subclause (12.4) determine the salt concentrations in the aqueous phase of the drilling fluid and determine if some of the sodium chloride salt is insoluble.

12.4.2 If all salts are soluble, Equations (45) and (46) calculate the mass fraction of $CaCl_2$ and NaCl, respectively, in the aqueous phase of an oil-based drilling fluid.

Mass fraction of $CaCl_2$ in the aqueous phase is calculated as follows:

$$w_{\mathrm{CaCl}_2} = \frac{100 \times c_{\mathrm{Cl}^-(\mathrm{CaCl}_2),\mathrm{DF}}}{\left[c_{\mathrm{Cl}^-(\mathrm{CaCl}_2),\,\mathrm{DF}}\right] + \left[c_{\mathrm{Cl}^-(\mathrm{NaCl}),\,\mathrm{DF}}\right] + \left(10\,000 \times \varphi_{\mathrm{W}}\right)} \tag{43}$$

where

w_{CaCl_2}	is the aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass;
$c_{\mathrm{Cl}^-(\mathrm{CaCl}_2),\,\mathrm{DF}}$	is the whole-drilling-fluid calcium chloride, expressed in milligrams per litre;

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

φ_{W} is the volume fraction of water, expressed as a percentage.

For fluids with no sodium chloride, proceed to 12.4.5.

12.4.3 The amount of NaCl is:

$$w_{\text{NaCl}} = \frac{100 \times c_{\text{NaCl, DF}}}{c_{\text{CaCl}_2\text{,DF}} + c_{\text{NaCl, DF}} + 10\,000\ \varphi_{\text{W}}} \qquad (44)$$

where

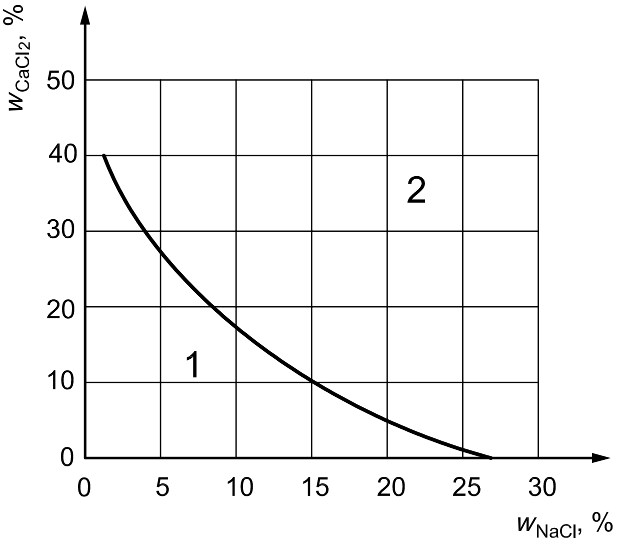
w_{NaCl} is the aqueous-phase mass fraction of sodium chloride, expressed as a percentage of the total aqueous phase;

$c_{\text{CaCl}_2\text{,DF,A}}$ is the whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre;

$c_{\text{NaCl,DF,A}}$ is the whole-drilling-fluid sodium chloride concentration, expressed in milligrams per litre;

φ_{W} is the volume fraction of water, expressed as a percentage.

12.4.4 The maximum mass fraction of soluble NaCl that can exist for the mass fraction of CaCl_2 present in the aqueous phase at 25 °C (77 °F) can be determined from empirical mutual solubility data (see Reference [13]), using Equation (45). If the value from Equation (44) is greater than the maximum soluble value from Equation (45), the aqueous phase is super-saturated and some of the sodium chloride exists as an insoluble solid (see Figure 1, Region 2). If this is the case, the aqueous-phase mass fractions calculated in Equation (43) and Equation (44) are incorrect because all of the NaCl is not in solution.



Key

1 region in which NaCl is soluble

2 region in which NaCl is insoluble

Figure 1 — Maximum concentrations of NaCl in CaCl_2 brine at 25 °C (77 °F)

12.4.4.1 The maximum mass fraction of soluble NaCl that can exist in the aqueous phase is calculated as follows:

$$w_{\text{NaCl,MAX}} = 26{,}432 - \left[1{,}047\,2\left(w_{\text{CaCl}_2}\right)\right] + \left[7{,}981\,91 \times 10^{-3}\left(w_{\text{CaCl}_2}\right)^2\right] + \left[5{,}223\,8 \times 10^{-5}\left(w_{\text{CaCl}_2}\right)^3\right] \quad (45)$$

where

$w_{\text{NaCl,MAX}}$ is the maximum aqueous-phase soluble sodium chloride that can exist for a given mass fraction of calcium chloride (see Reference [13]), expressed as a percentage of the total aqueous phase mass;

w_{CaCl_2} is the aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass.

12.4.4.2 If w_{NaCl} from Equation (44) is less than $w_{\text{NaCl,MAX}}$ from Equation (45), all the NaCl is soluble in the aqueous phase (Figure 1, Region 1). Proceed to 12.4.5 and use w_{CaCl_2} from Equation (43) and w_{NaCl} from Equation (44) for the remaining calculations.

12.4.4.3 If w_{NaCl} is greater than $w_{\text{NaCl,MAX}}$, the aqueous phase is super-saturated and insoluble NaCl is present (Figure 1, Region 2). For these fluids the mass fraction of both salts, w_{CaCl_2} from Equation (43) and w_{NaCl} from Equation (44), shall be recalculated.

12.4.4.4 For super-saturated fluids, the correct mass fraction of CaCl_2, $w_{\text{CaCl}_2\text{,SAT}}$, can be determined from empirical mutual solubility data (see Reference [13]) using the calcium concentration expressed as the mass of calcium per volume of pure water in the aqueous phase, $c_{\text{Ca}^{+2},\text{H}_2\text{O}}$, determined from the whole-drilling-fluid calcium ion concentration and the retort analysis.

The calcium concentration expressed per volume of pure water in the aqueous phase is calculated as follows:

$$c_{\text{Ca}^{+2},\text{H}_2\text{O}} = \frac{100 \times c_{\text{Ca}^{+2},\text{DF}}}{\varphi_{\text{W}}} \quad (46)$$

where

$c_{\text{Ca}^{+2},\text{H}_2\text{O}}$ is the aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per litre;

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

φ_{W} is the volume fraction of water, expressed as a percentage.

12.4.4.5 For super-saturated fluids, the actual mass fraction of calcium chloride, w_{CaCl_2}, in the aqueous phase is calculated as follows:

$$w_{\text{CaCl}_2\text{,SAT}} = \left[\left(c_{\text{Ca}^{+2},\text{H}_2\text{O}}\right)^2 \times (-2{,}470\,8) \times 10^{-10}\right] + \left[\left(c_{\text{Ca}^{+2},\text{H}_2\text{O}}\right) \times 2{,}256\,7 \times 10^{-4}\right] - \left(2{,}134\,8 \times 10^{-1}\right) \quad (47)$$

where

$w_{\text{CaCl}_2\text{,SAT}}$ is the aqueous-phase calcium chloride mass fraction in a super-saturated fluid, expressed as percentage of the total aqueous phase mass;

$c_{\text{Ca}^{+2},\text{H}_2\text{O}}$ is the aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per litre.

Use this actual $w_{\mathrm{CaCl_2,SAT}}$ for $w_{\mathrm{CaCl_2}}$ in the remaining calculations for fluids with super-saturated aqueous phases.

NOTE Undissolved NaCl has a lower specific gravity than most drill solids. If it is present in the oil-based drilling fluid, solids calculations will be in error. No technique is provided in this procedure to compensate for that type of error.

12.4.4.6 Now that the actual mass fraction of calcium chloride is known for a super-saturated aqueous phase, the correct mass fraction of NaCl in the aqueous phase is $w_{\mathrm{NaCl,MAX}}$ and shall be recalculated from Equation (47) using $w_{\mathrm{CaCl_2,SAT}}$ for $w_{\mathrm{CaCl_2}}$. Use this recalculated $w_{\mathrm{NaCl,MAX}}$ for w_{NaCl} in the remaining calculations for fluids with super-saturated aqueous phases.

12.4.5 The density of the brine, ρ_{B}, in grams per millilitre, is calculated as follows:

$$\begin{aligned}\rho_{\mathrm{B}} = 0{,}997\,07 + \left[0{,}006\,504\left(w_{\mathrm{NaCl}}\right)\right] + \left[0{,}007\,923\left(w_{\mathrm{CaCl_2}}\right)\right] + \left[0{,}000\,083\,34\left(w_{\mathrm{NaCl}}\right)\left(w_{\mathrm{CaCl_2}}\right)\right]\\ + \left[0{,}000\,043\,95\left(w_{\mathrm{NaCl}}\right)^2\right] + \left[0{,}000\,049\,64\left(w_{\mathrm{CaCl_2}}\right)^2\right]\end{aligned} \quad (48)$$

where

ρ_{B} is the aqueous-phase density, expressed in grams per millilitre;

$w_{\mathrm{CaCl_2}}$ is the aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass;

w_{NaCl} is the aqueous-phase mass fraction of sodium chloride, expressed in a percentage of the total aqueous phase mass.

12.4.6 The $\mathrm{CaCl_2}$ concentration of the aqueous phase, expressed in milligrams per litre, is calculated as follows:

$$c_{\mathrm{CaCl_2,AQ}} = 10\,000 \times w_{\mathrm{CaCl_2}} \times \rho_{\mathrm{B}} \quad (49)$$

where

$c_{\mathrm{CaCl_2,AQ}}$ is the aqueous-phase calcium chloride concentration, expressed in milligrams per litre;

$w_{\mathrm{CaCl_2}}$ is the aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous phase mass;

ρ_{B} is the aqueous-phase density, expressed in grams per millilitre.

NOTE The mass fractions and density for salt solutions where two salts are present do not correspond to single-salt values found in salt tables.

For fluids with no sodium chloride, proceed to 12.6.

12.4.7 The soluble NaCl concentration of the aqueous phase, expressed in milligrams per litre, is calculated as follows:

$$c_{\mathrm{NaCl,AQ}} = 10\,000 \times w_{\mathrm{NaCl}} \times \rho_{\mathrm{B}} \quad (50)$$

where

$c_{\mathrm{NaCl,AQ}}$ is the aqueous-phase sodium chloride concentration, expressed in milligrams per litre;

w_{NaCl} is the aqueous-phase mass fraction of sodium chloride, expressed as a percentage of the total aqueous phase;

ρ_B is the aqueous-phase density, expressed in grams per millilitre.

For fluids with no insoluble sodium chloride, proceed to 12.6.

12.5 Soluble and insoluble whole-drilling-fluid sodium chloride calculations

12.5.1 For drilling fluids with a super-saturated aqueous phase containing some insoluble NaCl, Equations (51) to (55) can be used to determine the soluble and insoluble quantities in the whole drilling fluid.

12.5.2 The soluble NaCl content of the whole drilling fluid is calculated as follows:

$$c_{\text{NaCl,DF,SOL,A}} = \frac{w_{\text{NaCl,MAX-C}} \times \left[c_{\text{CaCl}_2\text{,DF,A}} + (10\,000 \times \varphi_W) \right]}{100 - w_{\text{NaCl,MAX-C}}} \tag{51}$$

where

$c_{\text{NaCl,DF,SOL,A}}$ is the whole-drilling-fluid soluble sodium chloride concentration, expressed in milligrams per litre;

$w_{\text{NaCl,MAX-C}}$ is the recalculated maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of $CaCl_2$, expressed as a percentage of the total aqueous phase mass;

$c_{\text{CaCl}_2\text{,DF,A}}$ is the whole-drilling-fluid calcium chloride concentration, expressed in milligrams per litre;

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

12.5.3 The concentration of soluble NaCl in the whole drilling fluid, expressed in USC units, is calculated as follows:

$$c_{\text{NaCl,DF,SOL,B}} = 0{,}000\,35 \times c_{\text{NaCl,DF,SOL,A}} \tag{52}$$

$$c_{\text{NaCl,DF,SOL,C}} = 0{,}001 \times c_{\text{NaCl,DF,SOL,A}} \tag{53}$$

where

$c_{\text{NaCl,DF,SOL,B}}$ is the whole-drilling-fluid soluble sodium chloride concentration, expressed in pounds per barrel;

$c_{\text{NaCl,DF,SOL,C}}$ is the whole-drilling-fluid soluble sodium chloride concentration, expressed in kilograms per cubic metre;

$c_{\text{NaCl,DF,SOL,A}}$ is the whole-drilling-fluid soluble sodium chloride concentration, expressed in milligrams per litre.

12.5.4 The concentration of insoluble NaCl in the whole drilling fluid is calculated as follows:

$$c_{\text{NaCl,DF,INSOL,A}} = c_{\text{NaCl,DF,A}} - c_{\text{NaCl,DF,SOL,A}} \tag{54}$$

$$c_{\text{NaCl,DF,INSOL,B}} = c_{\text{NaCl,DF,B}} - c_{\text{NaCl,DF,SOL,B}} \tag{55}$$

where

$c_{\mathrm{NaCl,DF,INSOL,A}}$	is the whole-drilling-fluid insoluble sodium chloride concentration, expressed in milligrams per litre;
$c_{\mathrm{NaCl,DF,A}}$	is the whole-drilling-fluid sodium chloride concentration, expressed in milligrams per litre;
$c_{\mathrm{NaCl,DF,SOL,A}}$	is the whole-drilling-fluid soluble sodium chloride concentration, expressed in milligrams per litre;
$c_{\mathrm{NaCl,DF,INSOL,B}}$	is the whole-drilling-fluid insoluble sodium chloride concentration, expressed in pounds per barrel;
$c_{\mathrm{NaCl,DF,B}}$	is the whole-drilling-fluid sodium chloride concentration, expressed in pounds per barrel;
$c_{\mathrm{NaCl,DF,SOL,B}}$	is the whole-drilling-fluid soluble sodium chloride concentration, expressed in pounds per barrel.

12.6 Calculation — Solids in the whole drilling fluid

12.6.1 Corrected solids concentration

12.6.1.1 The combination of water and dissolved salts is known as brine, and the solids concentration should be corrected to accommodate this brine mass. The volume fraction of brine in the whole drilling fluid is calculated as follows:

$$\varphi_{\mathrm{B}} = \frac{100 \times \varphi_{\mathrm{W}}}{\rho_{\mathrm{B}} \times \left[100 - \left(w_{\mathrm{NaCl}} + w_{\mathrm{CaCl_2}}\right)\right]} \tag{56}$$

where

φ_{B}	is the whole-drilling-fluid volume fraction of brine, expressed as a percentage;
$w_{\mathrm{CaCl_2}}$	is the aqueous-phase mass fraction calcium chloride, expressed as a percentage of the total aqueous phase mass;
w_{NaCl}	is the aqueous-phase mass fraction sodium chloride, expressed in a percentage of the total aqueous phase mass.

12.6.1.2 The corrected volume fraction of solids in the whole drilling fluid is calculated as follows:

$$\varphi_{\mathrm{D}} = 100 - \left(\varphi_{\mathrm{O}} + \varphi_{\mathrm{B}}\right) \tag{57}$$

where

φ_{D}	is the corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid;
φ_{O}	is the volume fraction of oil, expressed as a percentage of the whole drilling fluid;
φ_{B}	is the volume fraction of brine, expressed as a percentage of the whole drilling fluid.

12.6.2 Calculation — Oil-to-water ratio

12.6.2.1 The oil and water ratio values, R_{O} and R_{W}, are calculated from the volume fraction of recovered liquids in the retort analysis and are calculated as follows:

$$R_{\mathrm{O}} = \frac{100 \times \varphi_{\mathrm{O}}}{\varphi_{\mathrm{O}} + \varphi_{\mathrm{W}}} \tag{58}$$

where

φ_{O} is the volume fraction of oil, expressed as a percentage of the whole drilling fluid;

φ_{W} is the volume fraction of pure water, expressed as a percentage of the whole drilling fluid;

R_{O} is the 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.

12.6.2.2 The ratio of water is calculated as follows:

$$R_{\mathrm{W}} = \frac{100 \times \varphi_{\mathrm{W}}}{\varphi_{\mathrm{O}} + \varphi_{\mathrm{W}}} \tag{59}$$

where

φ_{O} is the volume fraction of oil, expressed as a percentage of the whole drilling fluid;

φ_{W} is the volume fraction of pure water, expressed as a percentage of the whole drilling fluid;

R_{W} is the 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.

NOTE The oil-to-water ratio is sometimes expressed as OWR, and the numerical values are often rounded to the nearest whole number.

12.6.3 Calculation — Oil-to-brine ratio

12.6.3.1 The oil and brine ratio values, R_{O} and R_{B}, are calculated from the volume fraction of oil from the retort analysis and the volume fraction of brine calculated in 12.6.1, and Equations (60) and (61):

$$R_{\mathrm{O}} = \frac{100 \times \varphi_{\mathrm{O}}}{\varphi_{\mathrm{O}} + \varphi_{\mathrm{B}}} \tag{60}$$

where

φ_{O} is the volume fraction of oil, expressed as a percentage of the whole drilling fluid;

φ_{B} is the volume fraction of brine, expressed as a percentage of the whole drilling fluid;

R_{O} is the ratio of the volume fraction of oil to the sum of the volume fractions of oil and brine, expressed as a percentage.

12.6.3.2 The ratio of brine is calculated as follows:

$$R_{\mathrm{B}} = \frac{100 \times \varphi_{\mathrm{B}}}{\varphi_{\mathrm{O}} + \varphi_{\mathrm{B}}} \tag{61}$$

where

φ_O is the volume fraction of oil, expressed as a percentage of the whole drilling fluid;

φ_B is the volume fraction of brine, expressed as a percentage of the whole drilling fluid;

R_B is the ratio of the volume fraction of brine to the sum of the volume fractions of oil and brine, expressed as a percentage.

NOTE The oil-to-brine ratio is sometimes expressed as OBR, and the numerical values are often rounded to the nearest whole number.

12.6.4 Calculation — Solids concentration and density

12.6.4.1 The solids in an oil-based drilling fluid consist of low-gravity solids, such as drill solids, and weighting material, such as barite or haematite. Densities for materials commonly occurring in oil-based drilling fluids are found in Table 5.

12.6.4.2 The average density (volumic mass) of the suspended solids is calculated as follows:

$$\overline{\rho_d} = \frac{100\rho_S - (\varphi_O \times \rho_O) - (\varphi_B \times \rho_B)}{\varphi_D} \qquad (62)$$

where

φ_O is the volume fraction of oil, expressed as a percentage of the whole drilling fluid;

φ_B is the volume fraction of brine, expressed as a percentage of the whole drilling fluid;

φ_D is the corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid;

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

ρ_B is the density of the brine, expressed in grams per millilitre;

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

$\overline{\rho_d}$ is the average density (volumic mass) of the suspended solids.

Equation (62) may be calculated in USC units by using density values expressed in pounds per gallon for the drilling fluid density, ρ_S, and the oil and brine densities, ρ_O and ρ_B.

Table 5 — Commonly used densities

Drilling fluid component		Density (volumetric mass)		
		g/ml	kg/m^3	lb/gal
Fresh water (tap)		1,00	1 000	8,345
Diesel oil (typical)		0,84	840	7,00
Low-viscosity oil (typical)		0,80	800	6,70
Low-gravity solids		2,65	2 650	22,10
Weighting materials	Barite	4,20	4 200	35,00
	Haematite	5,05	5 050	42,10

12.6.5 Calculation — Volume fraction and concentration of weighting material and low-gravity solids

12.6.5.1 The volume fraction of weighting material is calculated as follows:

$$\varphi_{\mathrm{WM}} = \frac{\overline{\rho_{\mathrm{d}}} - \rho_{\mathrm{LG}}}{\rho_{\mathrm{WM}} - \rho_{\mathrm{LG}}} \varphi_{\mathrm{D}} \tag{63}$$

where

φ_{D} is the corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid;

φ_{WM} is the volume fraction of the weighting material solids, expressed as a percentage of the total suspended solids;

ρ_{WM} is the density of the weighting material solids, expressed in grams per millilitre;

ρ_{LG} is the density of the low-gravity solids, expressed in grams per millilitre.

NOTE For fluids with super-saturated aqueous phase and insoluble NaCl, the insoluble NaCl has a lower specific gravity than most drill solids such that solids calculations using the specific gravity of drill solids would be in error. No technique is provided in this procedure to compensate for that type of error.

12.6.5.2 The volume fraction of the low-gravity solids is calculated as follows:

$$\varphi_{\mathrm{LG}} = \varphi_{\mathrm{D}} - \varphi_{\mathrm{WM}} \tag{64}$$

where

φ_{WM} is the volume fraction of the weighting material solids, expressed as a percentage of the total suspended solids;

φ_{D} is the corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid;

φ_{LG} is the volume fraction of the low-gravity solids, expressed as a percentage of the total suspended solids.

12.6.5.3 The mass concentration of the weighting material is calculated as follows:

$$c_{\mathrm{WM,C}} = 10 \times \rho_{\mathrm{WM}} \times \varphi_{\mathrm{WM}} \tag{65}$$

$$c_{\mathrm{WM,B}} = 3{,}5 \times \rho_{\mathrm{WM}} \times \varphi_{\mathrm{WM}} \tag{66}$$

where

$c_{\mathrm{WM,C}}$ is the weighting material concentration, expressed in kilograms per cubic metre;

$c_{\mathrm{WM,B}}$ is the weighting material concentration, expressed in pounds per barrel.

12.6.5.4 The mass concentration of low-gravity solids can be calculated in kilograms per cubic metre (pounds per barrel) as:

$$c_{\mathrm{LG,C}} = 10 \times \rho_{\mathrm{LG}} \times V_{\mathrm{LG}} \tag{67}$$

$$c_{\mathrm{LG,B}} = 3{,}5 \times \rho_{\mathrm{LG}} \times V_{\mathrm{LG}} \tag{68}$$

where

$c_{\mathrm{LG,C}}$ is the low-gravity solids concentration, expressed in kilograms per cubic metre;

$c_{\mathrm{LG,B}}$ is the low-gravity solids concentration, expressed in pounds per barrel.

Annex A
(informative)

Measurement of shear strength using shearometer tube

A.1 Principle

A.1.1 Experience has shown that some drilling fluids tend to develop excessive shear strength under static conditions, especially at elevated temperatures. Excessive shear strength results in high pump pressures to “break circulation”, and can result in loss of returns. High shear strength can also cause difficulties in logging, perforating and other “downhole” operations.

A.1.2 The technique described in this annex can be used to determine this tendency and to estimate the extent to which the drilling fluid will develop excessive shear strength. This shear strength measurement is normally made on a static, heat-aged drilling fluid sample. Ageing temperatures are therefore selected to be near the estimated bottom-hole temperature of the well. Ageing cells or vessels meeting the pressure and temperature requirements for the test are required.

A.2 Apparatus

A.2.1 **Stainless steel shearometer tube**, having the following characteristics:

length	89 mm (3,5 in)
outside diameter	36 mm (1,4 in)
wall thickness	0,2 mm (0,008 in)

NOTE A slight outside taper on the bottom of the shear tube has been found to improve reproducibility of the test results.

A.2.2 **Platform for weights**.

A.2.3 **Set of weights**, in gram increments.

A.2.4 **Ruler**, graduated in millimetres (inches).

A.3 Procedure

A.3.1 Place and balance the shear tube carefully on the surface of the aged sample that has cooled to room temperature. It may be necessary to shift the weights on the platform to ensure that the tube's initial penetration into the drilling fluid is vertical. If a crust develops on the heat-aged sample, this crust should be gently broken before setting the shear tube in place for the test.

A.3.2 Carefully place sufficient weights on the platform to start the downward movement of the shear tube. Unless too much mass is added, the tube will stop its downward travel at the point where the shear strength of the aged drilling fluid against the surface of the tube is sufficient to support the applied mass. It is desirable to submerge at least one-half of the length of the tube.

A.3.3 Record the total mass, in grams, of the platform and weights. Measure the portion of the tube submerged in the fluid, in centimetres. The length of the tube submerged can be most accurately determined by measuring the length of the non-submerged portion while the tube is at its maximum penetration depth. A small ruler held at the drilling fluid surface and alongside the tube will facilitate this measurement. The length of the tube minus the exposed length equals the submerged portion.

A.4 Calculation

A.4.1 Shear strength in USC unit is calculated as follows[8]:

$$\gamma_A = \frac{3,61 \times (m_{st} + m_{tot})}{l_A} - 0,256\rho \qquad (A.1)$$

where

γ_A is the shear strength of the drilling fluid, expressed in pounds per square inch per foot;

m_{st} is the mass of the shear tube, expressed in grams;

m_{tot} is the total shear mass (sum of platform and weights), expressed in grams;

l_A is the submerged length of shear tube, expressed in inches;

ρ is the drilling fluid density, expressed in pounds per gallon.

A.4.2 Shear strength in SI units is calculated as follows[8]:

$$\gamma_B = \frac{4,40 \times (m_{st} + m_{tot})}{l} - 1,02\rho_S \qquad (A.2)$$

where

γ_B is the shear strength of the drilling fluid, expressed in pascals;

m_{st} is the mass of shear tube, expressed in grams;

m_{tot} is the total shear mass (sum of platform and weights), expressed in grams;

l is the submerged length of shear tube, expressed in centimetres;

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

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[8] Derived from Reference [12].

Annex B
(informative)

Determination of oil and water retained on cuttings

B.1 Principle

B.1.1 This procedure measures the amount of oil from cuttings generated during a drilling operation. It is a retort test, which measures all oil and water released from a cuttings sample when heated in a calibrated and properly operating retort instrument.

B.1.2 A known mass of cuttings is heated in the retort chamber to vaporize the liquids associated with the sample. The oil and water vapours are then condensed, collected and measured in a precision-graduated receiver.

NOTE Obtaining a representative sample requires special attention to the details of sample handling (location, method, frequency). The sampling procedure in a given area can be specified by local or governmental rules.

B.2 Apparatus

B.2.1 Retort instrument.

B.2.1.1 Retort assembly, which includes a retort body, cup and lid with a volume of 50 ml (precision ±0,25 ml), constructed of 303 stainless steel, or equivalent.

The retort cup volume with lid should be verified gravimetrically. The procedure and calculations are given in Annex J.

B.2.1.2 Condenser, capable of cooling the oil and water vapours below their vaporization temperatures.

B.2.1.3 Heating jacket, nominal power 350 W.

B.2.1.4 Temperature controller, capable of limiting temperature of the retort to 500 °C ± 40 °C (930 °F ± 70 °F).

B.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 B.1;
- **calibration (TC)**: at 20 °C (68 °F);
- **scale**: millilitres, cubic centimetres or volume fraction (as a percentage);
- **material**: transparent and inert to oil, water and salt solutions at temperatures up to 32 °C (90 °F).

Table B.1 — Precision of liquid receiver

		Total volume			
		10 ml	20 ml	50 ml	50 ml tapered
Precision	(0 % to 100 %)	±0,05 ml	±0,10 ml	±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.

B.2.3 Fine steel wool, oil-free.

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

B.2.4 High-temperature silicone grease, to be used for thread sealant and lubricant.

B.2.5 Pipe cleaners.

B.2.6 Putty knife or **spatula**, with blade shaped to fit the inside dimensions of the sample cup of the retort.

B.2.7 Corkscrew.

B.2.8 Top-loading balance, capable of weighing 2 000 g with an accuracy of ±0,1 g.

B.2.9 Brush.

B.3 Procedure

B.3.1 Clean and dry the retort assembly and condenser.

B.3.2 Pack the retort body with steel wool.

B.3.3 Apply lubricant/sealant to threads of retort cup and retort stem.

B.3.4 Weigh and record the total mass of the retort 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.

B.3.5 Collect a representative wet cuttings sample and allow to cool to approximately 27 °C (80 °F).

B.3.6 Partially fill the retort cup with cuttings and place the lid on the cup. Screw the retort cup (with cuttings and lid) onto the retort body (packed with steel wool), weigh and record the total mass. Record this as m_2, expressed in grams.

B.3.7 Attach the condenser. Place the retort assembly into the heating jacket. Close the insulating lid.

B.3.8 Weigh a clean, dry liquid receiver. Record this as m_3, expressed in grams. Place the receiver below the condenser 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 and that proper disposal in accordance with local, state and federal regulations be done. 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 amount can cause injury. Repeated exposure can cause liver, kidney and eye effects. Birth defects were not observed in laboratory animals.

NOTE 4 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 5 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.

B.3.9 Turn on the heating jacket and allow the retort assembly to run for a minimum of 1 h. Collect the condensate into the glass liquid receiver. If solids boil over into the receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.

B.3.10 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 water volume, V_W, collected in the liquid receiver. These are converted to mass, expressed in grams, in Clause B.4.

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 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 water-to-oil meniscus, read the water volume at the lowest point.

B.3.11 Weigh the glass liquid receiver and its liquid content (oil and water). Record this as m_4, expressed in grams.

B.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.

B.3.13 Remove the condenser body and passage stem. Weigh the cooled retort assembly [retort sample cup, lid and retort body (packed with steel wool)]. Record this as m_5, expressed in grams.

B.3.14 Clean the retort assembly and condenser.

B.4 Calculations

B.4.1 Calculate the mass of oil retained on cuttings as follows:

$$m_\mathrm{W} = m_2 - m_1 \tag{B.1}$$

where

m_W is the mass of the wet cuttings, 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.

$$m_d = m_5 - m_1 \tag{B.2}$$

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.

$$m_O = m_4 - (m_3 + V_W) \tag{B.3}$$

where

m_O is the mass of the oil, 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;

V_W is the 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.

Mass balance requirement: the sum of m_d, m_O and V_W, should be within 5 % of the mass of the wet sample.

$$0{,}95 \leqslant \frac{m_d + m_O + V_W}{m_W} \leqslant 1{,}05 \tag{B.4}$$

Repeat the procedure if this requirement is not met.

B.4.2 Report the retained oil on cuttings, ROC, as follows:

— mass of oil (in grams) per kilogram of wet cuttings sample

$$ROC = \frac{m_O}{m_W} \times 1000 \tag{B.5}$$

— mass of oil (in grams) per kilogram of dry retorted cuttings

$$ROC = \frac{m_O}{m_d} \times 1000 \tag{B.6}$$

where

ROC is the retained oil on cuttings, expressed in grams per kilogram of cuttings (either wet or dry).

NOTE 1 Many environmental regulations specifically request oil retained on cuttings, prior to offshore disposal.

NOTE 2 When reporting oil retained on cuttings in terms of the dry (retorted) cuttings, the oil concentration might be higher than that calculated from the wet cuttings sample, due to interstitial water extracted during retorting. Also, a cutting containing a higher percentage of water will appear to give a higher oil-retention mass for the same reasons. The water concentration is reported as the mass of water (in grams) per kilogram of wet cuttings sample (see B.3.10).

Annex C

(informative)

Determination of aqueous-phase activity of emulsified water using an electrohygrometer

C.1 Principle

C.1.1 This annex describes a procedure for the use of an electrohygrometer to measure relative humidity in a closed-air space above an oil-based drilling fluid, and to relate humidity to the activity of the emulsified water. Activity, a_W, is a measure of the chemical potential or reaction availability of a chemical species, diluted in water. Hole conditions in water-sensitive shales can be correlated with changes in the activity of emulsified water in oil-based drilling fluids.

C.1.2 The abundance of charged particles, surfaces and ions in shales reduces the activity of water and increases a shale's tendency to absorb water. Water will migrate through a semi-permeable membrane from a region of lower salinity (higher activity) to one of higher salinity (lower activity). The oil films surrounding emulsified water droplets in an oil-based drilling fluid constitute semi-permeable membranes through which water can pass by osmosis.

C.1.3 Water transfer from an oil-based drilling fluid can be prevented by dissolving salt in the emulsified water. Salt lowers the activity of the water phase, thus reducing the tendency of water to be absorbed by shale. Water transfer to shale is eliminated when the activity of the emulsified water is reduced to a value equal to or lower than that of the exposed shale. Hole condition and cuttings texture can guide proper activity adjustment.

C.1.4 The most efficient, commonly available salt for reducing the activity of water in oil-based drilling fluids is calcium chloride. Brine, having a given activity, will retain that activity when emulsified in oil. The aqueous-phase activity of an oil-based drilling fluid will be changed by water evaporation or water addition, or by incorporation by dissolution of other water-soluble salts.

NOTE A practice for combatting shale hydration based on a partial ion analysis of the water phase of the oil-based drilling fluid can be used. However, this method is prone to error, because it relies on substantial water dilution, several titrations, and a retort analysis for each test. Furthermore, insufficient data exist on the activity of mixed salt systems to allow ion analyses to be related accurately to aqueous-phase activity when multiple salts are present. The use of an electrohygrometer as described here is a way to determine activity, regardless of the composition of the water phase.

C.2 Reagents and apparatus

C.2.1 Standard saturated salt solutions

C.2.1.1 The standard saturated salt solutions are described in Table C.1.

Table C.1 — Standard saturated salt solutions

Salt	CAS No.	a_W at 25 °C (77 °F)	Concentration g/100 ml water
Calcium chloride	10043-52-4	0,295	100
Calcium nitrate	13477-34-4	0,505	200
Sodium chloride	7647-14-5	0,753	200
Potassium nitrate	7757-79-1	0,938	200

C.2.1.2 Each salt solution should be prepared by adding the number of grams of salt indicated in Table C.1 to 100 ml of distilled or deionized water, stirring at temperatures between 65 °C and 90 °C (150 °F and 200 °F) for 30 min, then cooling to a temperature between 25 °C and 27 °C (75 °F and 80 °F). Solid salt shall be visible in the liquid after equilibration.

C.2.1.3 If no crystals are visible, some additional crystals or grains of the same salt should be added to induce precipitation. This is often necessary with calcium chloride. Salts of reagent-grade purity are recommended. However, if necessary, field-grade NaCl or $CaCl_2$ can be substituted.

C.2.2 Desiccant, anhydrous **calcium chloride (CAS No. 10043-52-4)**. Other desiccants may be calcium sulfate (CAS No. 10101-41-4), Drierite® (CAS No. 7778-18-9)[9], or silica gel (CAS No. 112926-00-8).

C.2.3 Electrohygrometer, incorporating a portable relative humidity sensor and indicator, with the specifications below. If this instrument is equipped with a temperature probe, a separate thermometer is unnecessary.

a) The relative humidity sensor probe shall be suitable for monitoring the air space of a sample container with the sample.

b) The linearity response of the instrument shall be such that the correlation coefficient between published standard brine activity and relative humidity, as measured by the instrument above each brine, is greater than 0,95.

c) The instrument should indicate within 10 % of the true activity within 30 min, when measuring the brine activity of the standard salt solutions in C.2.1.

C.2.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F), not required if instrument has an internal thermometer.

C.2.5 Measuring container, as suggested or supplied by the manufacturer, having a volume sufficient to provide a liquid surface area of approximately 30 cm^2 to 50 cm^2 (4,5 in^2 to 7,75 in^2). The container should have a flexible elastomer cap (airtight) which, when bored through, will form an airtight seal around the shaft of the sensor probe and thermometer (if used).

C.2.6 Linear graph paper.

C.3 Procedure

C.3.1 Calibration of hygrometer

C.3.1.1 Prepare sufficient volume of each known relative humidity standard to cover the bottom of the measuring container to a liquid depth of 12 mm (0,5 in), or follow the manufacturer's recommendations. These salt solutions shall be kept firmly capped to prevent spillage, evaporation or contamination.

C.3.1.2 Desiccate the probe with anhydrous calcium chloride in a measuring container by placing other desiccant or the sensor probe (which is in the container cap) in the air space above the salt. Wait 10 min to 15 min. A relative humidity reading of 24 % or lower indicates that the probe is dry. Keep a distance of 12 mm (0,5 in) between the sensing probe and the desiccant, and maintain the same distance for salt solutions and drilling fluid samples. Never allow the probe to directly contact any samples or solutions.

C.3.1.3 Transfer the probe in the measuring container cap to the first (lowest a_W) standard solution and allow 30 min for equilibrium to be reached.

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[9] Drierite® 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.

Temperature of the standard solutions should be maintained between 24 °C and 25 °C (75 °F and 77 °F).

C.3.1.4 Remove the probe from the first solution and place it into the container with the solution of second lowest known activity.

C.3.1.5 Proceed as in C.3.1.3 and measure the relative humidity for each standard solution, proceeding from that with the lowest to that with the highest activity a_W.

C.3.1.6 Allow each standard to equilibrate for the same length of time. Record temperature and percent relative humidity (% RH). Plot the % RH versus the activity of the known solution, a_W.

C.3.2 Measurement of oil-based drilling fluid activity, a_DF

C.3.2.1 Desiccate the probe for 10 min to 15 min, as in C.3.1.2.

C.3.2.2 Transfer sufficient volume of room-temperature drilling fluid (40 ml) to cover the bottom of the sample container to a depth of 12 mm (0,5 in).

C.3.2.3 Insert the probe into the air space 12 mm (0,5 in) above the fluid surface. Sample temperature should be between 24 °C and 25 °C (75 °F and 77 °F), or the same temperature at which the standards were run.

C.3.2.4 Turn on the hygrometer and wait 30 min (the same time period used for standard calibration), and record the relative humidity and temperature indicated.

Examine the fluid to ensure that separation has not occurred. Separation of an oil layer will lead to erroneous readings.

Make certain that the sample container and cap are clean and free of salt before each new sample is poured into the container.

C.4 Calculation — Drilling fluid activity, a_DF

C.4.1 Find the relative humidity obtained in C.3.2.4 on the graph plotted in C.3.1.6. Read and record the corresponding activity that appears on the graph as the drilling fluid activity, a_DF.

C.4.2 If not measured directly, follow these plotting instructions:

a) using data for standards given in C.3.1, plot % RH versus a_W on linear graph paper.

b) plot the data obtained for each sample (C.3.2) on the linear graph paper and read the drilling fluid activity, a_DF.

Annex D
(informative)

Determination of aniline point

D.1 Principle

D.1.1 The aniline point is the lowest temperature at which equal volumes of aniline and hydrocarbon samples are completely miscible. This test method covers the determination of the aniline point of petroleum products and hydrocarbon solvents, such as diesel oils and mineral oils, used in preparation of oil-based drilling fluids. This method is suitable for transparent liquid samples having an initial boiling point above room temperature, and where the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture.

NOTE See ISO 2977 for additional information.

D.1.2 The aniline point is useful in characterizing pure hydrocarbons. For example, aromatic hydrocarbons exhibit low aniline points, and paraffinic hydrocarbons exhibit high values. Cycloparaffins and olefins exhibit values that lie between those of paraffins and aromatics. In a homologous series, the aniline point increases with increasing molecular mass. The aniline point for mixtures of hydrocarbons, such as diesel oils and mineral oils, serves as a guideline for judging the aromatic hydrocarbon concentration of the oil and for comparing oils.

D.1.3 When testing base oils for use in oil-based drilling fluids, low aniline point values (assumed to be related to higher aromatic concentrations) warns of potential damage to the elastomeric (rubber) components which are in contact with the oil-based drilling fluid, such as pump swabs, drillpipe rubbers, BOP rubbers and hoses. High aniline point values (assumed to represent lower aromatic concentrations) indicate lowered risk of elastomer damage, but may indicate less solubility of asphaltic materials and lowered ability to disperse asphaltic materials in the oil-based drilling fluid (see ASTM D3183).

D.2 Reagents and apparatus

D.2.1 Aniline (CAS No. 62-53-3), pure, water-free and not excessively dark in colour. Store in a dark, cool place.

CAUTION — Aniline is extremely toxic. Do not pipette by mouth. Aniline is also toxic by absorption through the skin. Handle carefully while wearing proper plastic gloves. Dispose of aniline and its mixtures in a safe and proper manner in accordance with local environmental and health regulations.

D.2.2 Calcium sulfate (CAS No. 7778-18-9) or sodium sulfate (anhydrous) (CAS No. 7757-82-6), to be used as a drying agent.

D.2.3 Test tube, 25 mm (1 in) in diameter, 150 mm (6 in) in length, and made of heat-resistant glass.

D.2.4 Jacket, approximately 37 mm to 42 mm (1-7/16 in to 1-11/16 in) in diameter and 150 mm (6 in) in length, and made of heat-resistant glass.

D.2.5 Thermometer, with a range of 25 °C to 105 °C (75 °F to 220 °F).

D.2.6 Cork stoppers, one to fit inside the test tube to hold the thermometer, and one to fit inside the jacket to hold the test tube. Do not use rubber stoppers.

D.2.7 Wire stirrer, constructed of 1,5 mm to 1,7 mm (14 gauge) wire, bent with a 19 mm (0,75 in) diameter loop, to be used to stir the mixture in the test tube.

D.2.8 Heating bath, e.g. a hot-air bath, a non-volatile transparent liquid bath (oil or silicone — not water), or infrared heating lamp (250 W to 375 W), to provide a means for heating the test tube and sample.

NOTE Water is not an appropriate heating medium because aniline is hygroscopic. Moist aniline will cause erroneously high results.

D.2.9 Cooling bath, e.g. a cool-air bath or a transparent liquid bath (not water), to provide a means for cooling the test tube and sample below room temperature.

NOTE Water is not an appropriate cooling medium because aniline is hygroscopic. Moist aniline will cause erroneously high results.

D.2.10 Pipette (TD), with a volume of 10 ml.

D.2.11 Plastic pipette suction bulb.

D.2.12 Safety goggles.

D.2.13 Plastic gloves, of a material that is impervious to aniline.

D.3 Procedure

D.3.1 Dry the oil sample by adding approximately 10 % volume fraction drying agent in the oil. Shake vigorously for 3 min to 5 min. Filter or centrifuge to remove the drying agent.

D.3.2 Clean and dry the test tube, jacket, thermometer and wire stirrer.

D.3.3 Pipette 10 ml of aniline (using the suction bulb) into the test tube. Likewise, pipette 10 ml of oil sample into the test tube.

D.3.4 Assemble the thermometer and stirrer. Position the thermometer in the test tube so that the immersion mark is at the top of the liquid. Be certain that the thermometer bulb does not touch the sides of the tube. Centre the test tube in the jacket tube.

D.3.5 Stir the mixture rapidly, at room temperature, using an up-and-down motion of the stirrer. Avoid forming air bubbles. Observe whether mixture is miscible (clear) or immiscible (cloudy). If the mixture is clear, go to D.3.12 and proceed using a cooling bath.

NOTE For most base oils, the aniline-oil mixture will be immiscible at room temperature.

D.3.6 If the mixture is cloudy, apply heat to the jacket tube to increase the temperature of the mixture in the test tube at a rate of 1 °C/min to 3 °C/min (2 °F/min to 5 °F/min).

D.3.7 Continue heating while stirring until the mixture becomes completely miscible (clear). Remove the mixture from the heating source.

CAUTION — Do not heat an aniline-oil mixture beyond 90 °C (200 °F). To do so could be unsafe.

D.3.8 Continue stirring while the clear mixture cools at a rate of 0,5 °C/min to 1,0 °C/min (1 °F/min to 2 °F/min). Notice when the cloudiness first reappears. Record as “aniline point” the temperature at which the mixture becomes cloudy throughout.

NOTE The true aniline point is characterized by a cloudiness that is sufficient to obscure the thermometer bulb in reflected light.

D.3.9 Continue cooling the mixture, while stirring, to a temperature of 1 °C to 2 °C (2 °F to 3,5 °F) below the first appearance of cloudiness, as described in D.3.8.

D.3.10 Heat the mixture again (as described in D.3.7) until it again becomes miscible. Remove the heat and cool the mixture until the cloudiness returns to obtain another observation of the aniline point temperature.

D.3.11 Repeat observations of the aniline point several times until a value has been determined within ±0,5 °C (±1 °F).

D.3.12 If the aniline-oil mixture was completely miscible at ambient temperature (D.3.5), substitute a cooling bath for the heating bath. Allow the mixture to cool slowly while stirring rapidly at the rate specified in D.3.5. Determine the aniline point as described in D.3.8 to D.3.11.

Annex E
(informative)

Lime, salinity and solids calculations

E.1 Principle

The example calculations in this annex are designed to show how the equations in Clauses 10 and 12 are used to determine the amount of salt and solids present in an oil-based drilling fluid containing barite as the weighting material. Four different cases are presented:

- Drilling fluid A: the drilling fluid contains only $CaCl_2$ salt;
- Drilling fluid B: the drilling fluid contains only NaCl salt;
- Drilling fluid C: the drilling fluid contains both $CaCl_2$ and soluble NaCl salts;
- Drilling fluid D: the drilling fluid contains both $CaCl_2$ and NaCl salts, with some NaCl soluble in the aqueous phase and some NaCl as insoluble solids in the whole drilling fluid.

This annex includes examples in which the units are typical for the relevant application. A spreadsheet with equations is included for ease of calculation.

NOTE This annex shows the relevant equation numbers from Clauses 10 and 12 in the normative text of this part of ISO 10414 after the formulae. Final values for mass concentration of weight material and solids are rounded to nearest whole number to reflect accuracy and the effect of significant digits.

E.2 Test data used in examples

Physical and chemical properties are presented in Table E.1.

Table E.1 — Physical and chemical properties of examples of drilling fluids

Property	Drilling fluid A only $CaCl_2$ salt	Drilling fluid B only NaCl salt	Drilling fluid C $CaCl_2$ and NaCl salts	Drilling fluid D $CaCl_2$ and excess NaCl salts
Drilling fluid density, g/ml	1,32	1,32	1,32	1,32
Oil concentration (φ_O), % volume	59,5	59,5	59,5	59,5
Water concentration (φ_W), % volume	25,0	25,0	25,0	25,0
EDTA (V_{EDTA}), ml/ml of drilling fluid	10,0	0,0	8,4	8,4
Silver nitrate (V_{AgNO_3}), ml/ml of drilling fluid	5,8	4,4	7,0	10,0
Sulfuric acid (V_K), ml/ml of drilling fluid	2,6	2,0	2,6	3,0
Oil density, g/ml	0,84	0,84	0,84	0,84
Density of low gravity solids, g/ml	2,65	2,65	2,65	2,65
Density of weighting material (barite), g/ml	4,20	4,20	4,20	4,20

E.3 Drilling fluid containing only $CaCl_2$ salt (drilling fluid A)

E.3.1 Whole-drilling-fluid chloride ion concentration [see 10.5.2, Equation (30)]:

$$c_{\mathrm{Cl}^-,\mathrm{DF}} = 10\ 000 \times 5{,}8 = 58\ 000 \text{ mg/l of drilling fluid}$$

E.3.2 Whole-drilling-fluid calcium concentration [see 10.6.2, Equation (31)]:

$$c_{\mathrm{Ca}^{+2},\mathrm{DF}} = 4\ 000 \times 10{,}0 = 40\ 000 \text{ mg/l of drilling fluid}$$

E.3.3 Whole-drilling-fluid lime concentration [see 10.4.3 or 12.3.1, Equation (29) and Equation (27)]:

$$c_{\mathrm{Ca(OH)}_2,\mathrm{DF,C}} = 3{,}705 \times 2{,}6 = 9{,}6 \text{ kg/m}^3$$

$$c_{\mathrm{Ca(OH)}_2,\mathrm{DF,B}} = 1{,}299 \times 2{,}6 = 3{,}4 \text{ lb/bbl}$$

where

$c_{\mathrm{Ca(OH)}_2,\mathrm{DF,C}}$ is the total lime concentration, expressed in kilograms per cubic metre;

$c_{\mathrm{Ca(OH)}_2,\mathrm{DF,B}}$ is the total lime concentration, expressed in pounds per barrel.

E.3.4 Whole-drilling-fluid chloride ion concentration as $CaCl_2$, from calcium analysis [see 12.3.2.2, Equation (32)]:

$$c_{\mathrm{Cl}^-(\mathrm{CaCl}_2),\mathrm{DF}} = 1{,}769 \times 40\ 000 = 70\ 760 \text{ mg/l of drilling fluid}$$

E.3.5 Whole-drilling-fluid chloride concentration associated as NaCl [see 12.3.2.3, Equation (33)]:

$$c_{\mathrm{Cl}^-(\mathrm{NaCl}),\ \mathrm{DF}} = 58\ 000 - 70\ 760 = -12\ 760 \text{ mg/l of drilling fluid}$$

The negative number indicates that no NaCl is present in this drilling fluid and only $CaCl_2$ is present. Calculate the whole-drilling-fluid $CaCl_2$ concentration from the more reliable chloride ion analysis.

E.3.6 Whole-drilling-fluid calcium chloride concentration [see 12.3.2.4, Equation (34), Equation (35) and Equation (36)]:

$$c_{\mathrm{CaCl}_2,\mathrm{DF,A}} = 1{,}565 \times 58\ 000 = 90\ 770 \text{ mg/l of drilling fluid}$$

$$c_{\mathrm{CaCl}_2,\mathrm{DF,B}} = 0{,}000\ 548\ 5 \times 58\ 000 = 31{,}8 \text{ lb/bbl of drilling fluid}$$

$$c_{\mathrm{CaCl}_2,\mathrm{DF,C}} = 0{,}001\ 565 \times 58\ 000 = 90{,}8 \text{ kg/m}^3 \text{ of drilling fluid}$$

E.3.7 Aqueous phase mass fraction of calcium chloride, expressed as a percentage [see 12.4.2, Equation (43)]:

$$w_{\mathrm{CaCl}_2} = \frac{100 \times 90\ 770}{70\ 760 + 0 + (10\ 000 \times 25{,}0)} = 26{,}7\ \%$$

E.3.8 Density of the aqueous phase brine [see 12.4.5, Equation (48)]:

$$\rho_{\mathrm{B}} = 0{,}997\ 07 + 0 + (0{,}007\ 923 \times 26{,}7) + 0 + \left(0{,}000\ 049\ 64 \times (26{,}7)^2\right) = 1{,}24 \text{ g/ml}$$

E.3.9 Aqueous phase calcium chloride concentration, in milligrams per litre [see 12.4.6, Equation (49)]:

$$c_{\mathrm{CaCl_2,AQ}} = 10\,000 \times 26{,}7 \times 1{,}244 = 332\ 15 \text{ mg/l}$$

E.3.10 Volume fraction (as a percentage) of brine in whole drilling fluid [see 12.6.1.1, Equation (56)]:

$$\varphi_{\mathrm{B}} = \frac{100 \times 25{,}0}{1{,}24 \times \left[100 - \left(0 + 26{,}7\right)\right]} = 27{,}4\% \text{ volume fraction in whole drilling fluid}$$

E.3.11 Volume fraction (as a percentage) of corrected solids in whole drilling fluid [see 12.6.1.2, Equation (57)]:

$$\varphi_{\mathrm{D}} = 100 - (59{,}5 + 27{,}4) = 13{,}1 \text{ \% volume fraction in whole drilling fluid}$$

E.3.12 Oil and water ratio (volume fraction) calculations (see 12.6.2, Equation (58) and Equation (59)]:

$$R_{\mathrm{O}} = \frac{100 \times 59{,}5}{59{,}5 + 25{,}0} = 70{,}4 \text{ \% oil}$$

$$R_{\mathrm{W}} = \frac{100 \times 25{,}0}{59{,}5 + 25{,}0} = 29{,}6 \text{ \% water}$$

$$R_{\mathrm{O}}:R_{\mathrm{W}} = 70{,}4:29{,}6$$

NOTE $R_{\mathrm{O}}:R_{\mathrm{W}}$ is the oil-to-water ratio, often denoted as OWR. In this example, the OWR is approximately 70:30.

E.3.13 Oil and brine ratio (volume fraction) calculations [see 12.6.3, Equation (60) and Equation (61)]:

$$R_{\mathrm{O}} = \frac{100 \times 59{,}5}{59{,}5 + 27{,}4} = 68{,}5 \text{ \% oil}$$

$$R_{\mathrm{B}} = \frac{100 \times 27{,}4}{59{,}5 + 27{,}4} = 31{,}5 \text{ \% brine}$$

NOTE $R_{\mathrm{O}}:R_{\mathrm{B}}$ is the oil-to-brine ratio, often denoted as OBR. In this example, the OBR is approximately 68:32.

E.3.14 Average density (volumic mass) of suspended solids [see 12.6.4, Equation (62)]:

$$\overline{\rho_{\mathrm{d}}} = \frac{\left(100 \times 1{,}32\right) - \left(59{,}5 \times 0{,}84\right) - \left(27{,}4 \times 1{,}244\right)}{13{,}1} = 3{,}66 \text{ g/ml}$$

E.3.15 Volume fraction (as a percentage) of weighting material [see 12.6.5.1, Equation (63)]:

$$\varphi_{\mathrm{WM}} = \frac{3{,}66 - 2{,}65}{4{,}20 - 2{,}65} \times 13{,}1 = 8{,}5 \text{ \% weighting material}$$

E.3.16 Volume fraction (as a percentage) of low-gravity solids [see 12.6.5.2, Equation (64)]:

$$\varphi_{\mathrm{LG}} = 13{,}1 - 8{,}5 = 4{,}6 \text{ \% low-gravity solids}$$

E.3.17 Mass concentration of weighting material [see 12.6.5.3, Equation (65) and Equation (66)]:

$c_{\mathrm{WM,A}} = 10 \times 4{,}20 \times 8{,}5 = 357$ kg/m^3 weighting material

$c_{\mathrm{WM,B}} = 3{,}5 \times 4{,}20 \times 8{,}5 = 125$ lb/bbl weighting material

E.3.18 Mass concentration of low-gravity solids [see 12.6.5.4, Equation (67) and Equation (68)]:

$c_{\mathrm{LG,A}} = 10 \times 2{,}65 \times 4{,}6 = 122$ kg/m^3 low-gravity solids

$c_{\mathrm{LG,B}} = 3{,}5 \times 2{,}65 \times 4{,}6 = 43$ lb/bbl low-gravity solids

E.4 Drilling fluid containing only NaCl salt (drilling fluid B)

E.4.1 Whole-drilling-fluid chloride ion concentration [see 10.5.2, Equation (30)]:

$c_{\mathrm{Cl^-,DF}} = 10\ 000 \times 4{,}4 = 44\ 000$ mg/l of drilling fluid

E.4.2 Whole-drilling-fluid calcium concentration [see 10.6.2, Equation (31)]:

$c_{\mathrm{Ca^{+2},DF}} = 4\ 000 \times 0 = 0$ mg/l of drilling fluid

E.4.3 Whole-drilling-fluid lime concentration [see 10.4.3 or 12.3.1, Equation (29) and Equation (27)]:

$c_{\mathrm{Ca(OH)_2,DF,C}} = 3{,}705 \times 2{,}0 = 7{,}4$ kg/m^3 of drilling fluid

$c_{\mathrm{Ca(OH)_2,DF,B}} = 1{,}299 \times 2{,}0 = 2{,}6$ lb/bbl of drilling fluid

where

$c_{\mathrm{Ca(OH)_2,DF,C}}$ is the total lime concentration, expressed in kilograms per cubic metre;

$c_{\mathrm{Ca(OH)_2,DF,B}}$ is the total lime concentration, expressed in pounds per barrel.

E.4.4 Whole-drilling-fluid chloride ion concentration as $CaCl_2$, from calcium analysis [see 12.3.2.2, Equation (32)]:

$c_{\mathrm{Cl^-(CaCl_2),DF}} = 1{,}769 \times 0 = 0$ mg/l of drilling fluid

The $CaCl_2$ content is 0.

E.4.5 Whole-drilling-fluid chloride ion concentration as NaCl [see 12.3.2.3, Equation (33)]

$c_{\mathrm{Cl^-(NaCl),DF}} = 44\ 000 - 0 = 44\ 000$ mg/l of drilling fluid

The positive number indicates that NaCl is present in this drilling fluid.

E.4.6 Whole-drilling-fluid sodium chloride concentration [see 12.3.2.6, Equation (40), Equation (41) and Equation (42)]:

$c_{\mathrm{NaCl,DF,A}} = 1{,}648 \times 44\ 000 = 72\ 512$ mg/l of drilling fluid

$c_{\mathrm{NaCl,DF,B}} = 0{,}000\ 577 \times 44\ 000 = 25{,}4$ lb/bbl of drilling fluid

$c_{\mathrm{NaCl,DF,C}} = 0{,}001\ 648 \times 44\ 000 = 72{,}5$ kg/m^3 of drilling fluid

E.4.7 Aqueous phase mass fraction of sodium chloride, expressed as a percentage [see 12.4.3, Equation (44)]:

$$w_{\text{NaCl}} = \frac{100 \times 72\ 512}{0 + 72\ 512 + (10\ 000 \times 25{,}0)} = 22{,}5\ \%$$

E.4.8 The maximum mass fraction of soluble NaCl that can exist in the aqueous phase is calculated as follows [see 12.4.4.1, Equation (45)]:

$$w_{\text{NaCl,MAX}} = 26{,}432 - 0 + 0 + 0 = 26{,}4\ \%$$

Since $w_{\text{NaCl}} < w_{\text{NaCl,MAX}}$, all NaCl is soluble in the aqueous phase and no calcium chloride is present.

E.4.9 Density of the aqueous phase brine [see 12.4.5, Equation (48)]:

$$\rho_{\text{B}} = 0{,}997\ 07 + (0{,}006\ 504 \times 22{,}5) + 0 + 0 + \left[0{,}000\ 043\ 95 \times (22{,}5)^2\right] + 0 = 1{,}166\ \text{g/ml}$$

E.4.10 The soluble NaCl concentration of the aqueous phase in brine, expressed in milligrams per litre [see 12.4.7, Equation (50)]:

$$c_{\text{NaCl,AQ}} = 10\ 000 \times 22{,}5 \times 1{,}166 = 262\ 350\ \text{mg/l}$$

For this example all of the sodium chloride is soluble. However, if some insoluble sodium chloride were present, the soluble and insoluble concentrations in the whole drilling fluid could be determined.

E.4.11 Volume fraction (as a percentage) of brine in whole drilling fluid [see 12.6.1, Equation (56)]:

$$\varphi_{\text{B}} = \frac{100 \times 25{,}0}{1{,}166 \times \left[100 - (22{,}5 + 0)\right]} = 27{,}7\ \%$$

E.4.12 Volume fraction (as percent) corrected solids in whole drilling fluid [see 12.6.1, Equation (57)]:

$$\varphi_{\text{D}} = 100 - (59{,}5 + 27{,}7) = 12{,}8\ \%$$

E.4.13 Oil and water ratio (volume fraction) calculations [see 12.6.2, Equation (58) and Equation (59)]:

$$R_{\text{O}} = \frac{100 \times 59{,}5}{59{,}5 + 25{,}0} = 70{,}4\ \%$$

$$R_{\text{W}} = \frac{100 \times 25{,}0}{59{,}5 + 25{,}0} = 29{,}6\ \%$$

$$R_{\text{O}}{:}R_{\text{W}} = 70{,}4{:}29{,}6$$

NOTE $R_{\text{O}}{:}R_{\text{W}}$ is the oil-to-water ratio, often denoted as OWR. In this example, the OWR is approximately 70:30.

E.4.14 Oil and brine ratio (volume fraction) calculations [see 12.6.3, Equation (60) and Equation (61)]:

$$R_{\text{O}} = \frac{100 \times 59{,}5}{59{,}5 + 27{,}7} = 68{,}2\ \%$$

$$R_{\text{B}} = \frac{100 \times 27{,}7}{59{,}5 + 27{,}7} = 31{,}8\ \%$$

$R_O{:}R_B = 68{,}2{:}31{,}8$

NOTE $R_O{:}R_B$ is the oil-to-brine ratio, often denoted as OBR. In this example, the OBR is approximately 68:32.

E.4.15 Average density (volumic mass) of suspended solids [see 12.6.4.2, Equation (62)]:

$$\overline{\rho_d} = \frac{(100 \times 1{,}32) - (59{,}5 \times 0{,}84) - (27{,}7 \times 1{,}166)}{12{,}8} = 3{,}88 \text{ g/ml}$$

E.4.16 Volume fraction (as a percentage) of weighting material [see 12.6.5.1, Equation (63)]:

$$\varphi_{WM} = \frac{3{,}88 - 2{,}65}{4{,}20 - 2{,}65} \times 12{,}8 = 10{,}2\ \%$$

E.4.17 Volume fraction (as percent) low-gravity solids [see 12.6.5.2, Equation (64)]:

$$\varphi_{LG} = 12{,}8 - 10{,}2 = 2{,}6\ \%$$

E.4.18 Mass concentration of weighting material [see 12.6.5.3, Equation (65) and Equation (66)]:

$$c_{WM,C} = 10 \times 4{,}20 \times 10{,}2 = 428 \text{ kg/m}^3$$

$$c_{WM,B} = 3{,}5 \times 4{,}20 \times 10{,}2 = 150 \text{ lb/bbl}$$

E.4.19 Mass concentration of low-gravity solids [see 12.6.5.4, Equation (67) and Equation (68)]:

$$c_{LG,C} = 10 \times 2{,}65 \times 2{,}6 = 69 \text{ kg/m}^3$$

$$c_{LG,B} = 3{,}5 \times 2{,}65 \times 2{,}6 = 24 \text{ lb/bbl}$$

E.5 Drilling fluid containing both $CaCl_2$ and NaCl salts (drilling fluid C)

E.5.1 Whole-drilling-fluid chloride ion concentration [see 10.5.2, Equation (30)]:

$$c_{Cl^-,DF} = 10\,000 \times 7{,}0 = 70\,000 \text{ mg/l of drilling fluid}$$

E.5.2 Whole-drilling-fluid calcium concentration [see 10.6.2, Equation (31)]:

$$c_{Ca^{+2},DF} = 4\,000 \times 8{,}4 = 33\,600 \text{ mg/l of drilling fluid}$$

E.5.3 Whole-drilling-fluid lime concentration [see 10.4.3 or 12.3.1, Equation (29) and Equation (27)]:

$$c_{Ca(OH)_2,DF,C} = 3{,}705 \times 2{,}6 = 9{,}6 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{Ca(OH)_2,DF,B} = 1{,}299 \times 2{,}6 = 3{,}4 \text{ lb/bl of drilling fluid}$$

where

$c_{Ca(OH)_2,DF,C}$ is the total lime concentration, expressed in kilograms per cubic metre;

$c_{Ca(OH)_2,DF,B}$ is the total lime concentration, expressed in pounds per barrel.

E.5.4 Whole-drilling-fluid chloride ion concentration as $CaCl_2$, from calcium analysis [see 12.3.2.2, Equation (32)]:

$$c_{\mathrm{Cl^-(CaCl_2),DF}} = 1{,}769 \times 33\,600 = 59\,438 \text{ mg/l of drilling fluid}$$

E.5.5 Whole-drilling-fluid chloride ion concentration as NaCl [see 12.3.2.3, Equation (33)]:

$$c_{\mathrm{Cl^-(NaCl),DF}} = 70\,000 - 59\,438 = 10\,562 \text{ mg/l of drilling fluid}$$

The positive number indicates that NaCl is present in this drilling fluid.

E.5.6 Whole-drilling-fluid calcium chloride concentration [see 12.3.2.5, Equation (37), Equation (38) and Equation (39)]:

$$c_{\mathrm{CaCl_2,DF,A}} = 2{,}769 \times 33\,600 = 93\,038 \text{ mg/l of drilling fluid}$$

$$c_{\mathrm{CaCl_2,DF,B}} = 0{,}000\,970\,7 \times 33\,600 = 32{,}6 \text{ lb/bbl of drilling fluid}$$

$$c_{\mathrm{CaCl_2,DF,C}} = 0{,}002\,769 \times 33\,600 = 93{,}0 \text{ kg/m}^3 \text{ of drilling fluid}$$

E.5.7 Whole-drilling-fluid sodium chloride concentration [see 12.3.2.6, Equation (40), Equation (41), and Equation (42)]:

$$c_{\mathrm{NaCl,DF,A}} = 1{,}648 \times 10\,562 = 17\,406 \text{ mg/l of drilling fluid}$$

$$c_{\mathrm{NaCl,DF,B}} = 0{,}000\,577 \times 10\,562 = 6{,}1 \text{ lb/bbl of drilling fluid}$$

$$c_{\mathrm{NaCl,DF,C}} = 0{,}001\,648 \times 10\,562 = 17{,}4 \text{ kg/m}^3 \text{ of drilling fluid}$$

E.5.8 Aqueous-phase mass fraction of calcium chloride, expressed as a percentage [see 12.4.2, Equation (43)]:

$$w_{\mathrm{CaCl_2}} = \frac{100 \times 93\,038}{93\,038 + 17\,406 + (10\,000 \times 25{,}0)} = 25{,}8\ \%$$

E.5.9 Aqueous-phase mass fraction of sodium chloride, expressed as a percentage [see 12.4.3, Equation (44)]:

$$w_{\mathrm{NaCl}} = \frac{100 \times 17\,406}{93\,038 + 17\,406 + (10\,000 \times 25{,}0)} = 4{,}8\ \%$$

E.5.10 Since both $CaCl_2$ and NaCl are present, the maximum mass fraction of soluble NaCl that can exist in the aqueous phase is calculated as follows [see 12.4.4.1, Equation (45)]:

$$w_{\mathrm{NaCl,MAX}} = 26{,}432 - (1{,}047\,2 \times 25{,}8) + \left[7{,}981\,91 \times 10^{-3} \times (25{,}8)^2\right] + \left[5{,}223\,8 \times 10^{-5} \times (25{,}8)^3\right] = 5{,}6\ \%$$

Since $w_{\mathrm{NaCl}} < w_{\mathrm{NaCl,MAX}}$, all NaCl is soluble in the aqueous phase.

NOTE Checking Figure 1, Region 1 confirms that all of the sodium chloride is in solution.

E.5.11 Density of the aqueous phase brine [see 12.4.5, Equation (48)]:

$$\rho_\text{B} = 0{,}997\ 07 + (0{,}006\ 504 \times 4{,}8) + (0{,}007\ 923 \times 25{,}8) + (0{,}000\ 083\ 34 \times 4{,}8 \times 25{,}8)$$
$$+ \left[0{,}000\ 043\ 95 \times (4{,}8)^2\right] + \left[0{,}000\ 049\ 65 \times (25{,}8)^2\right] = 1{,}277\ \text{g/ml}$$

E.5.12 Mass concentration calcium chloride, expressed in milligrams per litre [see 12.4.6, Equation (49)]:

$$c_{\text{CaCl}_2,\text{AQ}} = 10\ 000 \times 25{,}8 \times 1{,}277 = 329\ 466\ \text{mg/l}$$

E.5.13 Mass concentration sodium chloride, expressed in milligrams per litre [see 12.4.7, Equation (50)]:

$$c_{\text{NaCl,AQ}} = 10\ 000 \times 4{,}8 \times 1{,}277 = 61\ 296\ \text{mg/l}$$

No insoluble sodium chloride is present.

E.5.14 Volume fraction (as a percentage) of brine in whole drilling fluid [see 12.6.1.1, Equation (56)]:

$$\varphi_\text{B} = \frac{100 \times 25{,}0}{1{,}277 \times \left[100 - (25{,}8 + 4{,}8)\right]} = 28{,}2\ \%$$

E.5.15 Volume fraction (as a percentage) of corrected solids in whole drilling fluid [see 12.6.1.2, Equation (57)]:

$$\varphi_\text{D} = 100 - (59{,}5 + 28{,}2) = 12{,}3\ \%$$

E.5.16 Oil and water rations (volume fraction) calculations [see 12.6.2, Equation (58) and Equation (59)]:

$$R_\text{O} = \frac{100 \times 59{,}5}{59{,}5 + 25{,}0} = 70{,}4\ \%$$

$$R_\text{W} = \frac{100 \times 25{,}0}{59{,}5 + 25{,}0} = 29{,}6\ \%$$

$$R_\text{O}:R_\text{W} = 70{,}4:29{,}6$$

NOTE $R_\text{O}:R_\text{W}$ is the oil-to-water ratio, often denoted as OWR. In this example, the OWR is approximately 70:30.

E.5.17 Oil and brine ratio (volume fraction) calculations [see 12.6.3, Equation (60) and Equation (61)]:

$$R_\text{O} = \frac{100 \times 59{,}5}{59{,}5 + 28{,}2} = 67{,}8\ \%$$

$$R_\text{B} = \frac{100 \times 28{,}2}{59{,}5 + 28{,}2} = 32{,}2\ \%$$

$$R_\text{O}:R_\text{B} = 67{,}8:32{,}2$$

NOTE $R_\text{O}:R_\text{B}$ is the oil-to-brine ratio, often denoted as OBR. In this example, the OBR is approximately 68:32.

E.5.18 Average density (volumic mass) of suspended solids [see 12.6.4.2, Equation (62)]:

$$\overline{\rho_\text{d}} = \frac{(100 \times 1{,}32) - (59{,}5 \times 0{,}84) - (28{,}2 \times 1{,}277)}{12{,}3} = 3{,}74\ \text{g/ml}$$

E.5.19 Volume fraction (as a percentage) of weighting material [see 12.6.5.1, Equation (63)]:

$$\varphi_{\text{WM}} = \frac{3{,}74 - 2{,}65}{4{,}20 - 2{,}65} \times 12{,}3 = 8{,}6\ \%$$

E.5.20 Volume fraction (as a percentage) of low-gravity solids [see 12.6.5.2, Equation (64)]:

$$\varphi_{\text{LG}} = 12{,}3 - 8{,}6 = 3{,}7\ \%$$

E.5.21 Mass concentration of weighting material [see 12.6.5.3, Equation (65) and Equation (66)]:

$$c_{\text{WM,C}} = 10 \times 4{,}20 \times 8{,}6 = 361\ \text{kg/m}^3$$

$$c_{\text{WM,B}} = 3{,}5 \times 4{,}20 \times 8{,}6 = 126\ \text{lb/bbl}$$

E.5.22 Mass concentration of low-gravity solids [see 12.6.5.4, Equation (67) and Equation (68)]:

$$c_{\text{LG,C}} = 10 \times 2{,}65 \times 3{,}7 = 98\ \text{kg/m}^3$$

$$c_{\text{LG,B}} = 3{,}5 \times 2{,}65 \times 3{,}7 = 34\ \text{lb/bbl}$$

E.6 Drilling fluid containing both $CaCl_2$ and excess NaCl salts (drilling fluid D)

E.6.1 Whole-drilling-fluid chloride ion concentration [see 10.5.2, Equation (30)]:

$$c_{\text{Cl}^-\text{,DF}} = 10\ 000 \times 10{,}0 = 100\ 000\ \text{mg/l of drilling fluid}$$

E.6.2 Whole-drilling-fluid calcium concentration [see 10.6.2, Equation (31)]:

$$c_{\text{Ca}^{+2}\text{,DF}} = 4\ 000 \times 8{,}4 = 33\ 600\ \text{mg/l of drilling fluid}$$

E.6.3 Whole-drilling-fluid lime concentration [see 10.4.3 or 12.3.1, Equation (29) and Equation (27)]:

$$c_{\text{Ca(OH)}_2\text{,DF,C}} = 3{,}705 \times 3{,}0 = 11{,}1\ \text{g/m}^3\ \text{of drilling fluid}$$

$$c_{\text{Ca(OH)}_2\text{,DF,B}} = 1{,}299 \times 3{,}0 = 3{,}9\ \text{lb/bbl of drilling fluid}$$

where

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

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

E.6.4 Whole-drilling-fluid chloride ion concentration as $CaCl_2$, from calcium analysis [see 12.3.2.2, Equation (32)]:

$$c_{\text{Cl}^-(\text{CaCl}_2)\text{,DF}} = 1{,}769 \times 33\ 600 = 59\ 438\ \text{mg/l of drilling fluid}$$

E.6.5 Whole-drilling-fluid chloride ion concentration as NaCl [see 12.3.2.3, Equation (33)]:

$$c_{\text{Cl}^-(\text{NaCl})\text{,DF}} = 100\ 000 - 59\ 438 = 40\ 562\ \text{mg/l of drilling fluid}$$

The positive number indicates that NaCl is present in this drilling fluid.

E.6.6 Whole-drilling-fluid calcium chloride concentration [see 12.3.2.5, Equation (37), Equation (38) and Equation (39)]:

$$c_{\mathrm{CaCl_2,DF,A}} = 2{,}769 \times 33\,600 = 93\,038 \text{ mg/l of drilling fluid}$$

$$c_{\mathrm{CaCl_2,DF,B}} = 0{,}000\,970\,7 \times 33\,600 = 32{,}6 \text{ lb/bbl of drilling fluid}$$

$$c_{\mathrm{CaCl_2,DF,C}} = 0{,}002\,769 \times 33\,600 = 93{,}0 \text{ kg/m}^3 \text{ of drilling fluid}$$

E.6.7 Whole-drilling-fluid sodium chloride concentration [see 12.3.2.6, Equation (40), Equation (41) and Equation (42)]:

$$c_{\mathrm{NaCl,DF,A}} = 1{,}648 \times 40\,562 = 66\,846 \text{ mg/l of drilling fluid}$$

$$c_{\mathrm{NaCl,DF,B}} = 0{,}000\,577\,0 \times 40\,562 = 23{,}4 \text{ lb/bbl of drilling fluid}$$

$$c_{\mathrm{NaCl,DF,C}} = 0{,}001\,648 \times 40\,562 = 66{,}8 \text{ kg/m}^3 \text{ of drilling fluid}$$

E.6.8 Aqueous-phase mass fraction of calcium chloride, expressed as a percentage [see 12.4.2, Equation (43)]:

$$w_{\mathrm{CaCl_2}} = \frac{100 \times 93\,038}{93\,038 + 66\,846 + (10\,000 \times 25{,}0)} = 22{,}7\ \%$$

E.6.9 Aqueous-phase mass fraction of sodium chloride, expressed as a percentage [see 12.4.3, Equation (44)]:

$$w_{\mathrm{NaCl}} = \frac{100 \times 66\,846}{93\,038 + 66\,846 + (10\,000 \times 25{,}0)} = 16{,}3\ \%$$

E.6.10 Since both $CaCl_2$ and NaCl are present, the maximum mass fraction of soluble NaCl that can exist in the aqueous phase is calculated as follows [see 12.4.4.1, Equation (45)]:

$$w_{\mathrm{NaCl,MAX}} = 26{,}432 - (1{,}047\,2 \times 22{,}7) + \left[7{,}981\ \ 91 \times 10^{-3} \times (22{,}7)^2\right] + \left[5{,}223\,8 \times 10^{-5} \times (22{,}7)^3\right] = 7{,}4\ \%$$

From the calculations, $w_{\mathrm{NaCl}} > w_{\mathrm{NaCl,MAX}}$, the aqueous phase is super-saturated and insoluble NaCl is present (Figure 1, Region 2). For these fluids the mass fraction of both salts, w_{CaCl2} from Equation (43) and w_{NaCl} from Equation (44), shall be recalculated.

E.6.11 Calcium concentration expressed per volume of pure water in the aqueous phase [see 12.4.4.4, Equation (46)]:

$$c_{\mathrm{Ca^{+2},H_2O}} = \frac{100 \times 33\,600}{25{,}0} = 134\,400 \text{ mg/l}$$

E.6.12 For super-saturated fluids, the actual mass fraction of calcium chloride, $w_{\mathrm{CaCl_2}}$, in the aqueous phase is calculated [see 12.4.4.5, Equation (47)]:

$$w_{\mathrm{CaCl_2,SAT}} = \left[(134\,000)^2\left(-2{,}470\,8 \times 10^{-10}\right)\right] + \left[(134\,000)\left(2{,}256\,7 \times 10^{-4}\right)\right] - \left(2{,}134\,8 \times 10^{-1}\right) = 25{,}7\ \%$$

Use the actual $w_{\mathrm{CaCl_2,SAT}}$ for $w_{\mathrm{CaCl_2}}$ in the remaining calculations or fluids with super-saturated aqueous phases.

E.6.13 Since this fluid is super-saturated, the correct mass fraction of NaCl in the aqueous phase is $w_{\text{NaCl,MAX-C}}$ but shall be recalculated as described in 12.4.4.6 using Equation (45) but using $w_{\text{CaCl}_2\text{,SAT}}$ for w_{CaCl_2}.

$$w_{\text{NaCl,MAX-C}} = 26{,}432 - \left(1{,}047\,2 \times 25{,}7\right) + \left[7{,}981\ \ 91 \times 10^{-3} \times (25{,}7)^2\right] + \left[5{,}223\,8 \times 10^{-5} \times (25{,}7)^3\right] = 5{,}7\ \%$$

Use $w_{\text{CaCl}_2\text{,SAT}}$ for w_{CaCl_2} and the recalculated $w_{\text{NaCl,MAX-C}}$ for w_{NaCl} in the remaining calculations for fluids with super-saturated aqueous phases.

E.6.14 Density of the aqueous phase brine [see 12.4.5, Equation (48)]:

$$\rho_{\text{B}} = 0{,}997\,07 + \left(0{,}006\,504 \times 5{,}7\right) + \left(0{,}007\,923 \times 25{,}7\right) + \left(0{,}000\,083\,34 \times 5{,}7 \times 25{,}7\right) + \left[0{,}000\,043\,95 \times \left(5{,}7\right)^2\right] + \left[0{,}000\,049\,64 \times \left(25{,}7\right)^2\right] = 1{,}284\ \text{g/ml}$$

E.6.15 Mass concentration of calcium chloride in the aqueous phase, expressed in milligrams per litre [see 12.4.6, Equation (49)]:

$$c_{\text{CaCl}_2\text{,AQ}} = 10\,000 \times 25{,}7 \times 1{,}284 = 329\,988\ \text{mg/l}$$

E.6.16 Mass concentration of sodium chloride in the aqueous phase, expressed in milligrams per litre [see 12.4.7, Equation (50)]:

$$c_{\text{NaCl,AQ}} = 10\,000 \times 5{,}7 \times 1{,}284 = 73\,188\ \text{mg/l}$$

E.6.17 The soluble NaCl content of the whole drilling fluid is calculated [see 12.5.2, Equation (51)]:

$$c_{\text{NaCl,DF,SOL,A}} = \frac{5{,}7 \times \left[329\,314 + (10\,000 \times 25{,}0)\right]}{100 - 5{,}7} = 35\,017\ \text{mg/l}$$

E.6.18 The soluble NaCl of the whole drilling fluid in USC units (pounds per barrel) is calculated [see 12.5.3, Equation (52)]:

$$c_{\text{NaCl,DF,SOL,B}} = 0{,}000\,35 \times 35\,017 = 12{,}3\ \text{lb/bbl}$$

E.6.19 The concentration of insoluble NaCl in the whole drilling fluid is calculated [see 12.5.4, Equation (54) and Equation (55)]:

$$c_{\text{NaCl,DF,INSOL,A}} = 66\,836 - 35\,017 = 31\,719\ \text{mg/l}$$

$$c_{\text{NaCl,DF,INSOL,B}} = 23{,}4 - 12{,}3 = 11{,}1\ \text{lb/bbl}$$

E.6.20 Volume fraction of brine in whole drilling fluid, expressed as a percentage [see 12.6.1.1, Equation (56)]:

$$\varphi_{\text{B}} = \frac{100 \times 25{,}0}{1{,}284 \times [100 - (25{,}7 + 5{,}7)]} = 28{,}4\ \%$$

E.6.21 Volume fraction of corrected solids in whole drilling fluid, expressed as a percentage [see 12.6.1.2, Equation (57)]:

$$\varphi_{\text{D}} = 100 - \left(59{,}5 + 28{,}4\right) = 12{,}1\ \%$$

E.6.22 Oil and water ratio (volume fraction) calculations [see 12.6.2, Equation (58) and Equation (59)]:

$$R_{\mathrm{O}} = \frac{100 \times 59{,}5}{59{,}5 + 25{,}0} = 70{,}4\ \%$$

$$R_{\mathrm{W}} = \frac{100 \times 25{,}0}{59{,}5 + 25{,}0} = 29{,}6\ \%$$

$$R_{\mathrm{O}}{:}R_{\mathrm{W}} = 70{,}4{:}29{,}6$$

NOTE $R_{\mathrm{O}}{:}R_{\mathrm{W}}$ is the oil-to-water ratio, often denoted as OWR. In this example, the OWR is approximately 70:30.

E.6.23 Oil and brine ratio (volume fraction) calculations [see 12.6.3, Equation (60) and Equation (61)]:

$$R_{\mathrm{O}} = \frac{100 \times 59{,}5}{59{,}5 + 28{,}4} = 67{,}7\ \%$$

$$R_{\mathrm{B}} = \frac{100 \times 28{,}4}{59{,}5 + 28{,}4} = 32{,}3\ \%$$

$$R_{\mathrm{O}}{:}R_{\mathrm{B}} = 67{,}7{:}32{,}3$$

NOTE $R_{\mathrm{O}}{:}R_{\mathrm{B}}$ is the oil-to-brine ratio, often denoted as OBR. In this example, the OBR is approximately 68:32.

E.6.24 Average density (volumic mass) of suspended solids [see 12.6.4, Equation (62)]:

$$\overline{\rho}_{\mathrm{d}} = \frac{(100 \times 1{,}32) - (59{,}5 \times 0{,}84) - (28{,}4 \times 1{,}284)}{12{,}1} = 3{,}76\ \mathrm{g/ml}$$

E.6.25 Volume fraction of weighting material, expressed as a percentage [see 12.6.5.1, Equation (63)]:

$$\varphi_{\mathrm{WM}} = \frac{3{,}76 - 2{,}65}{4{,}20 - 2{,}65} \times 12{,}1 = 8{,}7\ \%$$

E.6.26 Volume fraction of low-gravity solids, expressed as a percentage [see 12.6.5.2, Equation (64)]:

$$\varphi_{\mathrm{LG}} = 12{,}1 - 8{,}7 = 3{,}4\ \%$$

E.6.27 Mass concentration of weighting material [see 12.6.5.3, Equation (65) and Equation (66)]:

$$c_{\mathrm{WM,C}} = 10 \times 4{,}20 \times 8{,}7 = 365\ \mathrm{kg/m^3}$$

$$c_{\mathrm{WM,B}} = 3{,}5 \times 4{,}20 \times 8{,}7 = 128\ \mathrm{lb/bbl}$$

E.6.28 Mass concentration of low-gravity solids [see 12.6.5.4, Equation (67) and Equation (68)]:

$$c_{\mathrm{LG,C}} = 10 \times 2{,}65 \times 3{,}4 = 90\ \mathrm{kg/m^3}$$

$$c_{\mathrm{LG,B}} = 3{,}5 \times 2{,}65 \times 3{,}4 = 32\ \mathrm{lb/bbl}$$

E.7 Calculation in spreadsheet — Drilling fluid A to drilling fluid D

		DRILLING FLUID A (only $CaCl_2$ salt)		DRILLING FLUID B (only NaCl salt)		DRILLING FLUID C ($CaCl_2$ and soluble NaCl)		DRILLING FLUID D ($CaCl_2$ and soluble/insoluble NaCl)	
	DRILLING FLUID PROPERTIES	SI	USC	SI	USC	SI	USC	SI	USC
	1) Drilling fluid density, g/ml (lb/gal)	1,32	11,018	1,32	11,018	1,32	11,018	1,32	11,018
	2) Oil concentration, φ_c, % volume	59,5		59,5		59,5		59,5	
	3) Water concentration, φ_W, % volume	25		25		25		25	
	4) EDTA, V_{EDTA}, ml/ml of drilling fluid	10		0		8,4		8,4	
	5) Silver nitrate, V_{AgNO3}, ml/ml of drilling fluid	5,8		4,4		7		10	
	6) Sulfuric acid, V_{HOSO4}, ml/ml of drilling fluid	2,6		2		2,6		3	
	7) Oil density, g/ml (lb/gal)	0,84	7,012	0,84	7,012	0,84	7,012	0,84	7,012
	8) Density of low-gravity solids, g/ml	2,65		2,65		2,65		2,65	
	9) Density of weighting material (barite), g/ml	4,2		4,2		4,2		4,2	
EQUATION									
	WHOLE DRILLING FLUID CALCULATIONS								
(29)	Whole-drilling-fluid chloride ion concentration, Cl^+, from chloride, mg chloride/litre of drilling fluid	58,000		44,000		70,000		100,000	
(30)	Whole-drilling-fluid calcium ion concentration, Ca^{++}, mg calcium/litre of drilling fluid	40,000		0		33,600		33,600	
(29)/(27)	Whole-drilling-fluid lime concentration, $Ca(OH)_2$, kg lime/m^3 of drilling fluid	9,6	3,4	7,4	2,6	9,6	3,4	11,1	3,9
(30)	Whole-drilling-fluid chloride ion concentration, Cl^-, as $CaCl_2$ from calcium, mg chloride/litre of drilling fluid	70,760		0		59,438		59,438	
(33)	Whole-drilling-fluid chloride concentration, Cl, from NaCl, mg chloride/litre of drilling fluid	−12,760		44,000		10,562		40,562	
	WHICH SALTS ARE PRESENT?	Only $CaCl_2$		Only NaCl		Both $CaCl_2$ & NaCl		Both $CaCl_2$ & NaCl	
	If Sodium chloride is present, skip to "Sodium chloride is present"								
(34)/(35)/(36)	Whole-drilling-fluid calcium chloride, $CaCl_2$, from chloride, mg $CaCl_2$/litre of drilling fluid (lb/bbl)	90,770	31,8	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	If only calcium chloride is present, skip to "Aqueous-phase calculation"								

Figure E.1 — Spreadsheet of data and calculations (*continued*)

		DRILLING FLUID A (only $CaCl_2$ salt)		DRILLING FLUID B (only NaCl salt)		DRILLING FLUID C ($CaCl_2$ and soluble NaCl)		DRILLING FLUID D ($CaCl_2$ and soluble/insoluble NaCl)	
	Sodium chloride is present								
(37)/(38)/(39)	Whole-drilling-fluid calcium chloride concentration, $CaCl_2$, from calcium, mg $CaCl_2$/litre of drilling fluid (lb/bbl)	110,760	38,8	0	0	93,038	32,6	93,038	32,6
(40)/(41)/(42)	Whole-drilling-fluid sodium chloride concentration, NaCl, mg NaCl/litre of drilling fluid (lb/bbl)	0	0	72,512	25,4	17,406	6,1	66,846	23,4
(34)/(37) & (35)/(38)	Corrected whole-drilling-fluid calcium chloride concentration, mg $CaCl_2$/litre of drilling fluid (lb/bbl)	90,770	31,8	0	0	93,038	32,6	93,038	32,6
	AQUEOUS PHASE CALCULATIONS								
(43)	Percent mass fraction calcium chloride in aq.ph., % mass $CaCl_2$/aqueous phase	26,6		0		25,8		22,7	
	For fluids with no sodium chloride, skip to "Aqueous-phase density"								
(44)	Percent mass fraction sodium chloride in aq.ph., % mass NaCl in aqueous phase	0		22,5		4,8		16,3	
(45)	Maximum percent mass fraction soluble sodium chloride in aq.ph., % mass NaCl/mass aqueous phase	5,2		26,4		5,6		7,4	
	Does aqueous phase contain insoluble NaCl?	No insoluble NaCl		All NaCl soluble		All NaCl soluble		Insoluble NaCl	
	If no insoluble NaCl is present, skip to "Aqueous-phase density"								
	SUPER-SATURATED AQUEOUS PHASE CALCULATIONS								
(46)	Calcium concentration per volume of pure water, mg Ca^{+2}/litre of water	n.a.		0		134,400		134,400	
(47)	Actual percent mass fraction calcium chloride in aq.ph., % mass $CaCl_2$/mass aq.ph.	26,6		0		25,8		25,7	
(45)	Recalculated maximum percent mass fraction soluble sodium chloride in aq.ph., % mass NaCl/mass aq.ph.	n.a.		26,4		5,6		5,7	
	Actual percent mass fraction sodium chloride in aq.ph., % mass NaCl/mass aq.ph.	0		22,5		4,8		5,7	
	AQUEOUS PHASE CALCULATION (continued)								
(43) or (47)	Corrected percent mass fraction calcium chloride in aq.ph., % mass $CaCl_2$/ mass aq.ph.	26,6		0		25,8		25,7	
(49)	Aqueous phase density, g brine/ml brine	1,243		1,166		1,277		1,284	
(50)	**Calcium chloride concentration in aqueous phase, mg $CaCl_2$/litre of aq.ph.**	331,183		0		329,744		329,314	

Figure E.1 — Spreadsheet of data and calculations (*continued*)

		DRILLING FLUID A (only $CaCl_2$ salt)		DRILLING FLUID B (only NaCl salt)		DRILLING FLUID C ($CaCl_2$ and soluble NaCl)		DRILLING FLUID D ($CaCl_2$ and soluble/insoluble NaCl)	
(51)	**Sodium chloride concentration in aqueous phase, mg NaCl/litre of aq.ph.**	0		262,050		61,688		73,203	
	If no insoluble NaCl is present, skip to "Solids concentration"								
	BINARY SALT CALCULATIONS OF SOLUBLE & INSOLUBLE SODIUM CHLORIDE IN WHOLE DRILLING FLUID								
(52), (53)	Whole-drilling-fluid soluble sodium chloride, mg NaCl/litre of drilling fluid (lb NaCl/bbl of drilling fluid)	0	0	72,512	25,4	17,406	6,1	35,033	12,3
(54), (55)	Whole-drilling-fluid insoluble sodium chloride, mg NaCl/litre of drilling fluid (lb NaCl/bbl of drilling fluid)	0	0	0	0	0	0	31,812	11,1
	SOLIDS CALCULATIONS IN WHOLE DRILLING FLUID								
(56)	Percent volume fraction brine in whole drilling fluid, % volume brine/volume of drilling fluid	27,4		27,7		28,2		28,4	
(57)	Corrected percent volume fraction solids in whole drilling fluid, % volume solids/volume of drilling fluid	13,1		12,8		12,3		12,1	
	OIL-TO-WATER RATIO								
(58)	Oil volume ratio oil per volume of oil plus water from retort, % vol oil per vol oil + water	70,4		70,4		70,4		70,4	
(59)	Water volume ratio water per volume of oil plus water from retort, % vol water per vol oil + water	29,6		29,6		29,6		29,6	
	OIL-TO-BRINE RATIO								
(60)	Oil volume ratio oil per volume of oil plus brine, % vol oil per vol oil + water	68,5		68,3		67,8		67,7	
(61)	Brine volume ratio brine per volume of oil plus brine, % vol brine per vol oil + brine	31,5		31,7		32,2		32,3	
	SOLIDS CONCENTRATION AND DENSITY								
(62)	Average density of suspended solids, g average solids/ml of solids	3,662		3,879		3,743		3,760	
	VOLUM FRACTION AND CONCENTRATION OF WEIGHTING MATERIAL AND LOW-GRAVITY SOLIDS								
(63)	Weighting material volume fraction per volume whole drilling fluid, % vol wt.mtl. per vol drilling fluid	8,5		10,2		8,7		8,7	
(64)	Low-gravity solids volume fraction per volume of whole drilling fluid, % vol lgs per vol drilling fluid	4,5		2,7		3,6		3,4	
(65), (66)	Weighting material concentration in whole drilling fluid, kg wt.mtl. per litre drilling fluid (lb wm/bbl df)	359,0	125,6	427,4	149,6	363,7	127,3	364,7	127,6
(67), (68)	Low-gravity solids concentration in whole drilling fluid, kg lgs per litre drilling fluid (lb lgs/bbl df)	120,4	42,2	70,3	24,6	96,1	33,6	91,3	32,0

Figure E.1 — Spreadsheet of data and calculations

Table E.2 — Equations for calculation of binary salt systems

CELL	EQUATION
C15	=C8*10000
C15	=C7*4000
C17	=C9*3.691
C18	=C16*1.769
C19	=C15-C18
C20	=IF(C$7=0,"Only NaCl Present",IF(C$19<=0,"Only $CaCl_2$ Present","Both $CaCl_2$ and NaCl Present"))
C22	=IF(C$19>0, "n.a.", IF(C7=0,0,C15*1.565))
C25	=C16*2.769
C26	=IF(C19<=0,0,1.648*C19)
C28	=IF(C19<=0,C22,C25)
C30	=100*C28/(C28+C26+10000*C6)
C32	=100*C26/(C28+C26+10000*C6)
C33	=26.432-1.0472*C30+0.00798191*C30^2+0.000052238*C30^3
C34	=IF(C32=0,"No Insoluble NaCl is Present", IF(C32>C39, "Insoluble NaCl is Present", "All NaCl is Soluble"))
C37	=IF(C$19<=0, "n.a.", 100*C16/C6)
C38	=IF(C33>C32, C30, IF(C$7=0,0, C37^2*-0.00000000024708+C37*0.00022567-0.21348))
C39	=IF(C$19<=0, "n.a.", 26.432-1.0472*C38+0.00798191*C38^2+0.000052238*C38^3)
C40	=IF(C39>C32,C32,C39)
C42	=IF(C32>C39,C38,C30)
C43	=0.99707+0.006504*C40+0.007923*C42+0.00008334*C42*C40+0.00004395*C40^2+0.00004964*C42^2
C44	=10000*C42*C43
C45	=10000*C40*C43
C48	=IF(C39>C32,C26,C40*(C44+(10000*C6))/(100-C40))
C49	=C26-C48
C51	=100*C6/(C43*(100-(C42+C40)))
C52	=100-C51-C5
C54	=100*C5/(C5+C6)
C55	=100*C6/(C5+C6)
C57	=100*C5/(C5+C51)
C58	=100*C51/(C5+C51)
C60	=(100*C4-C5*C10-C51*C43)/C52
C62	=C52*(C60-C11)/(C12-C11)
C63	=C52-C62
C64	=10*C12*C62
C65	=10*C11*C63

Table E.2 (*continued*)

CELL	EQUATION
D4	=C4*1000/119.8
D10	=C10*1000/119.8
D17	=C9*1.299
D22	=IF(C$19>0, "n.a.", IF(C7=0,0,C15*0.0005478))
D25	=C16*0.0009692
D26	=IF(C19<=0,0,C19*0.000577)
D28	=IF(C19<=0,D22,D25)
D48	=C48*0.00035
D49	=C49*0.00035
D64	=3.5*C12*C62
D65	=3.5*C11*C63
E15	=E8*10000
E15	=E7*4000
E17	=E9*3.691
E18	=E16*1.769
E19	=E15-E18
E20	=IF(E$7=0,"Only NaCl Present",IF(E$19<=0,"Only $CaCl_2$ Present","Both $CaCl_2$ and NaCl Present"))
E22	=IF(E$19>0, "n.a.", IF(E7=0,0,E15*1.565))
E25	=E16*2.769
E26	=IF(E19<=0,0,1.648*E19)
E28	=IF(E19<=0,E22,E25)
E30	=100*E28/(E28+E26+10000*E6)
E32	=100*E26/(E28+E26+10000*E6)
E33	=26.432-1.0472*E30+0.00798191*E30^2+0.000052238*E30^3
E34	=IF(E32=0,"No Insoluble NaCl is Present", IF(E32>E39, "Insoluble NaCl is Present", "All NaCl is Soluble"))
E37	=IF(E$19<=0, "n.a.", 100*E16/E6)
E38	=IF(E33>E32, E30, IF(E$7=0,0,E37^2*-0.00000000024708+E37*0.00022567-0.21348))
E39	=IF(E$19<=0, "n.a.", 26.432-1.0472*E38+0.00798191*E38^2+0.000052238*I38^3)
E40	=IF(E39>E32,E32,E39)
E42	=IF(E32>E39,E38,E30)
E43	=0.99707+0.006504*E40+0.007923*E42+0.00008334*E42*E40+0.00004395*E40^2+0.00004964*E42^2
E44	=10000*E42*E43
E45	=10000*E40*E43
E48	=IF(E39>E32,E26,E40*(E44+(10000*E6))/(100-E40))
E49	=E26-E48
E51	=100*E6/(E43*(100-(E42+E40)))

Table E.2 (*continued*)

CELL	**EQUATION**
E52	=100-E51-E5
E54	=100*E5/(E5+E6)
E55	=100*E6/(E5+E6)
E57	=100*E5/(E5+E51)
E58	=100*E51/(E5+E51)
E60	=(100*E4-E5*E10-E51*E43)/E52
E62	=E52*(E60-E11)/(E12-E11)
E63	=E52-E62
E64	=10*E12*E62
E65	=10*E11*E63
F4	=E4*1000/119.8
F10	=E10*1000/119.8
F17	=E9*1.299
F22	=IF(E$19>0, "n.a.", IF(E7=0,0,E15*0.0005478))
F25	=E16*0.0009692
F26	=IF(E19<=0,0,E19*0.000577)
F28	=IF(E19<=0,F22,F25)
F48	=E48*0.00035
F49	=E49*0.00035
F64	=3.5*E12*E62
F65	=3.5*E11*E63
G15	=G8*10000
G15	=G7*4000
G17	=G9*3.691
G18	=G16*1.769
G19	=G15-G18
G20	=IF(G$7=0,"Only NaCl Present",IF(G$19<=0,"Only $CaCl_2$ Present","Both $CaCl_2$ and NaCl Present"))
G22	=IF(G$19>0, "n.a.", IF(G7=0,0,G15*1.565))
G25	=G16*2.769
G26	=IF(G19<=0,0,1.648*G19)
G28	=IF(G19<=0,G22,G25)
G30	=100*G28/(G28+G26+10000*G6)
G32	=100*G26/(G28+G26+10000*G6)
G33	=26.432-1.0472*G30+0.00798191*G30^2+0.000052238G30^3
G34	=IF(G32=0,"No Insoluble NaCl is Present", IF(G32>G39, "Insoluble NaCl is Present", "All NaCl is Soluble"))

Table E.2 (*continued*)

CELL	**EQUATION**
G37	=IF(G$19<=0, "n.a.", 100*G16/G6)
G38	=IF(G33>G32, G30, IF(G$7=0,0,G37^2*-0.00000000024708+G37*0.00022567-0.21348))
G39	=IF(G$19<=0, "n.a.", 26.432-1.0472*G38+0.00798191*G38^2+0.000052238*G38^3)
G40	=IF(G39>G32,G32,G39)
G42	=IF(G32>G39,G38,G30)
G43	=0.99707+0.006504*G40+0.007923*G42+0.00008334*G42*G40+0.00004395*G40^2+0.00004964*G42^2
G44	=10000*G42*G43
G45	=10000*G40*G43
G48	=IF(G39>G32,G26,G40*(G44+(10000*G6))/(100-G40))
G49	=G26-G48
G51	=100*G6/(G43*(100-(G42+G40)))
G52	=100-G51-G5
G54	=100*G5/(G5+G6)
G55	=100*G6/(G5+G6)
G57	=100*G5/(G5+G51)
G58	=100*G51/(G5+G51)
G60	=(100*G4-G5*G10-G51*G43)/G52
G62	=G52*(G60-G11)/(G12-G11)
G63	=G52-G62
G64	=10*G12*G62
G65	=10*G11*G63
H4	=G4*1000/119.8
H10	=G10*1000/119.8
H17	=G9*1.299
H22	=IF(G$19>0, "n.a.", IF(G7=0,0,G15*0.0005478))
H25	=G16*0.0009692
H26	=IF(G19<=0,0,G19*0.000577)
H28	=IF(G19<=0,H22,H25)
H48	=G48*0.00035
H49	=G49*0.00035
H64	=3.5*G12*G62
H65	=3.5*G11*G63
NOTE	The following are equations for “I” cells. It is important to differentiate clearly between “1” (number) and “I” (letter).
I15	=I8*10000
I15	=I7*4000
I17	=I9*3.691

Table E.2 (*continued*)

CELL	**EQUATION**
I18	=I16*1.769
I19	=I15-I18
I20	=IF(I$7=0,"Only NaCl Present",IF(I$19<=0,"Only $CaCl_2$ Present","Both $CaCl_2$ and NaCl Present"))
I22	=IF(I$19>0, "n.a.", IF(I7=0,0,I15*1.565))
I25	=I16*2.769
I26	=IF(I19<=0,0,1.648*I19)
I28	=IF(I19<=0,I22,I25)
I30	=100*I28/(I28+I26+10000*I6)
I32	=100*I26/(I28+I26+10000*I6)
I33	=26.432-1.0472*I30+0.00798191*I30^2+0.000052238*I30^3
I34	=IF(I32=0,"No Insoluble NaCl is Present", IF(I32>I39, "Insoluble NaCl is Present", "All NaCl is Soluble"))
I37	=IF(I$19<=0, "n.a.", 100*I16/I6)
I38	=IF(I33>I32, I30, IF(I$7=0,0,I37^2*-0.00000000024708+I37*0.00022567-0.21348))
I39	=IF(I$19<=0, "n.a.",26.432-1.0472*I38+0.00798191*I38^2+0.000052238*I38^3))
I40	=IF(I39>I32,I32,I39)
I42	=IF(I32>I39,I38,I30)
I43	=0.99707+0.006504*I40+0.007923*I42+0.00008334*I42*I40+0.00004395*I40^2+0.00004964*I42^2
I44	=10000*I42*I43
I45	=10000*I40*I43
I48	=IF(I39>I32,I26,I40*(I44+(10000*I6))/(100-I40))
I49	=I26-I48
I51	=100*I6/(I43*(100-(I42+I40)))
I52	=100-I51-I5
I54	=100*I5/(I5+I6)
I55	=100*I6/(I5+I6)
I57	=100*I5/(I5+I51)
I58	=100*I51/(I5+I51)
I60	=(100*I4-I5*I10-I51*I43)/I52
I62	=I52*(I60-I11)/(I12-I11)
I63	=I52-I62
I64	=10*I12*I62
I65	=10*I11*I63
NOTE The following equations include “I” cell designations. It is important to differentiate clearly between “1” (number) and “I” (letter).	
J4	=I4*1000/119.8
J10	=I10*1000/119.8
J17	=I9*1.299

Table E.2 (*continued*)

CELL	EQUATION
J22	=IF(I$19>0, "n.a.", IF(I7=0,0,I15*0.0005478))
J25	=I16*0.0009692
J26	=IF(I19<=0,0,I19*0.000577)
J28	=IF(I19<=0,J22,J25)
J48	=I48*0.00035
J49	=I49*0.00035
J64	=3.5*I12*I62
J65	=3.5*I11*I63

Annex F
(informative)

Sampling, inspection and rejection of drilling materials

F.1 Principle

The procedure described in this annex provides sampling, inspection and rejection methods for drilling fluid materials conforming to ISO 13500. It is applicable to barite, haematite, bentonite, non-treated bentonite, OCMA-bentonite, attapulgite, sepiolite, technical-grade low-viscosity carboxymethylcellulose, technical-grade high-viscosity carboxymethylcellulose, starch, low-viscosity polyanionic cellulose, high-viscosity polyanionic cellulose, drilling-grade xanthan polymer and barite 4,10.

F.2 Sampling of powdered material in packages

F.2.1 Take 15 or more samples of 0,5 kg (1 lb) per lot to be combined as the test sample (see F.4).

F.2.2 For each lot of 1 000 packages or less, 15 packages should be sampled.

F.2.3 The sampling should be carried out by either of the following methods as agreed upon by the contracting parties:

a) a sample weighing at least 0,5 kg should be taken from the top of each package, or

b) a sampling tube capable of taking a core not less than 2,5 cm (1 in) in diameter should be used. The tube should be inserted into the package being sampled so that it will take a core of material for essentially the entire length of the package.

F.3 Sampling powdered material in bulk

F.3.1 A sampling tube capable of extracting a core of a diameter no less than 2,5 cm (1 in) should be used to collect samples from storage containers holding 25 000 kg to 100 000 kg (25 metric tonnes to 100 metric tonnes) (25 long tons to 98 long tons). The tube should be of sufficient length to permit taking a substantial sample from the top to the bottom of the container of material being sampled.

F.3.2 Fifteen samples should be taken from each lot (container). If the dimensions of the container are such that the sample cannot be taken in this manner, the sample should be taken by a method agreed upon by the contracting parties.

F.3.3 Take at least one sample from each container holding less than 20 000 kg (20 metric tonnes) (20 long tons), up to lots of 10 containers. The total number of samples taken from each lot of 100 000 kg (100 metric tonnes) (98 long tons) or less should not be fewer than 10.

F.4 Preparation of test sample

F.4.1 The samples from each lot should be combined, mixed, and quartered or riffled to furnish a test sample of 7 kg (15 lb) which should be divided into three equal portions.

F.4.2 Each portion should be sealed in a suitable airtight, moisture-proof container. One sample should be delivered to the purchaser, one to the supplier, and the third sample should be retained for a reference test, if required.

F.5 Test

Each party should make measurements on his test sample in accordance with the specific test procedure(s) outlined in ISO 13500.

NOTE For the purposes of this provision, API Spec 13A is equivalent.

F.6 Inspection

The purchaser's inspector should be afforded all reasonable facilities for careful sampling and inspection. A period of six days should be allowed for sampling and completion of the acceptance test.

F.7 Rejection

Rejection of material, based on failure to pass the test prescribed in the specifications, should be reported to the supplier immediately upon completion of the tests, and the cause for rejection should be stated.

F.8 Retesting

F.8.1 Either of the contracting parties may request a retest within one week of the date of the original test report.

F.8.2 Should the contracting parties be unable to reach an agreement, the third sample of material should be delivered, unopened, to a mutually satisfactory referee laboratory for tests, and the results of this referee test shall be binding on both parties.

Annex G
(informative)

Rig-site sampling

G.1 Principle

This annex describes a recommended procedure for obtaining test samples, at the rig site, from specific loads of drilling fluid materials. Upon agreement by contracting parties, this procedure can be used for testing the properties of material as delivered to the rig site.

G.2 Bottom-flow sampling (preferred)

G.2.1 Apparatus

G.2.1.1 **Side-stream sampler**, as shown in Figure G.1.

G.2.1.2 **Canvas bag**, approximately 14 cm (5,5 in) in diameter and 30 cm (12 in) long.

G.2.1.3 **Suitable container**, e.g. a tie-bag, constructed such that it retains all fines.

G.2.2 Procedure

G.2.2.1 Connect a side-stream sampler to the truck outlet.

G.2.2.2 Take three 1,5 kg to 2,5 kg (3 lb to 5 lb) samples while unloading, spaced so they are taken when approximately 1/4, 1/2 and 3/4 of the load has been transferred.

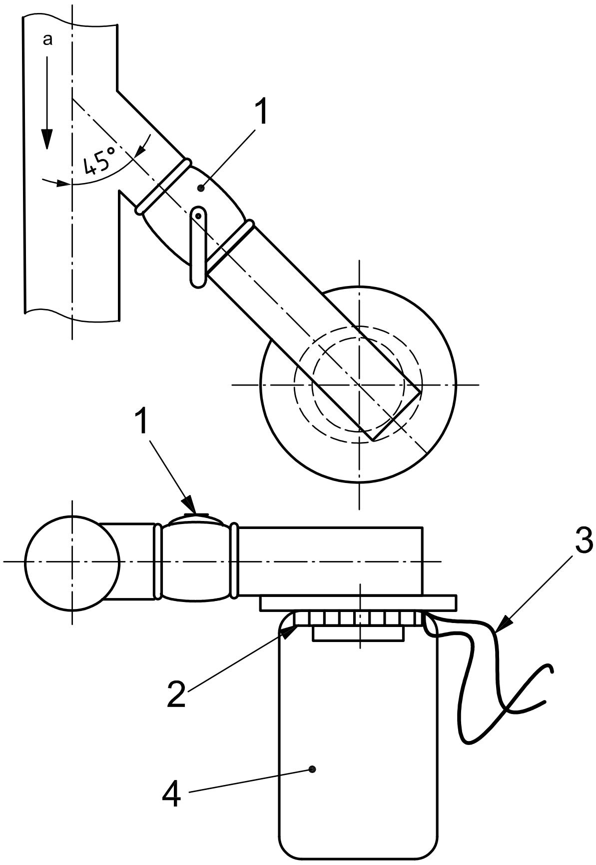
NOTE One 4 kg to 7 kg (9 lb to 15 lb) sample can be taken with the recognition that there is a chance of obtaining a non-representative sample. The greatest potential for error in sampling is contamination from prior shipments of material in the same vessel. This potential error can be minimized by not taking samples from the very top surface or from the very first material out of the bottom. A second potential error in sampling arises on taking material from the side-stream sampler when the transfer hose is only partially full. This can occur when the container is nearly empty or material flow has been interrupted. Segregation has been shown to occur in these instances.

G.2.2.3 Open the valve momentarily to flush out any material trapped in the side pipe before taking each individual sample. Tie a clean canvas bag to the sampler and fill it in one valve-opening operation.

NOTE Partial closing of the air-jet valve on the bottom outlet prior to taking the sample often helps get a full sack with a minimum of dusting.

G.2.2.4 Carefully transfer the sample from the canvas bag to a tie-bag or other suitable container. Label this container with all pertinent information.

G.2.2.5 Empty the canvas bag before taking the next sample. Always use a clean or new canvas bag for each container.



Key

1 5 cm (2 in) ball valve in 5 cm (2 in) side pipe
2 bead or lip to hold bag
3 draw string
4 canvas bag 14 cm × 30 cm (5,5 in × 12 in)

[a] Flow direction.

Figure G.1 — Side-stream sampling device

G.3 Grab sampling

G.3.1 Apparatus

G.3.1.1 **Sample scoop**, as shown in Figure G.2, of dimensions 10 cm to 15 cm (4 in to 6 in) deep and 30 cm to 45 cm (12 in to 18 in) long.

G.3.1.2 **Suitable container**, e.g. a tie-bag, constructed such that it retains all fines.

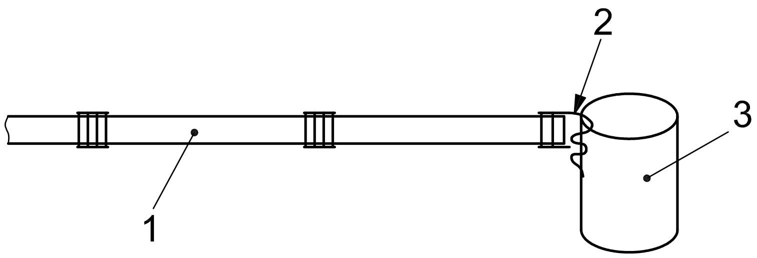
G.3.2 Procedure

G.3.2.1 Open the top hatch carefully. Do not let the cover bump the top of the tank.

G.3.2.2 Remove the top surface material under the hatch to form a trench 1,5 times wider than the sample scoop.

G.3.2.3 Scoop out a 1,5 kg to 2,5 kg (3 lb to 5 lb) sample from the bottom of this trench and transfer to a tie-bag or other suitable container. Label the container.

G.3.2.4 Repeat this operation at two other spots on the top of the material near the midpoint between the middle and the ends of the truck tank.



Key

1 painter's extension handle with end piece flattened for hinge

2 strap hinge

3 metal can

Figure G.2 — Sample scoop

Annex H
(informative)

Determination of cutting activity by the Chenevert method

H.1 Principle

H.1.1 This procedure describes the electrohygrometer measurement of the relative humidity of stagnant air in equilibrium with a freshly collected and cleaned cuttings (shale) sample taken from a wellbore. The measured activity a_W, a value ranging from 0 to 1, is proportional to the tendency of water vapour to escape (fugacity) from the sample, and relates to the water's chemical potential to react. The water being measured in cuttings by this method is primarily water associated with clay and other hydrous minerals in the shales.

H.1.2 This procedure provides specific directions on sample collection, handling and washing. It provides instructions for testing cuttings using the electrohygrometer. It closely parallels the procedure in Annex C for measuring the activity of an oil-based drilling fluid, and allows for a reliable comparison between activity data for an oil-based drilling fluid and activity data for the cuttings from the same well.

H.1.3 Cutting activities measured at the surface by this test probably do not represent the activity of the same shale downhole. A cutting can be altered due to changes in temperature, pressure and stress state as it moves from the wellbore to the surface. In addition, a significant difference between the drilling fluid and the cutting activity causes the cutting's activity to approach that of the oil-based drilling fluid. Measured trends of oil-based drilling fluid and cuttings activities from the same well will, in most cases, allow one to judge which has the greater or lesser activity value — the cutting or the oil-based drilling fluid.

H.2 Reagents and apparatus

H.2.1 Standard saturated salt solutions

H.2.1.1 The standard saturated salt solutions are described in Table H.1.

Table H.1 — Saturated salt solutions

Salt	**CAS No.**	a_W **at** 25 °C (77 °F)	**Concentration** g/100 ml water
Calcium chloride	10043-52-4	0,295	100
Calcium nitrate	13477-34-4	0,505	200
Sodium chloride	7647-14-5	0,753	200
Potassium nitrate	7757-79-1	0,938	200

H.2.1.2 Each salt solution should be prepared by adding the number of grams of salt indicated in Table H.1 to 100 ml of distilled or deionized water, stirring at temperatures between 65 °C and 90 °C (150 °F and 200 °F) for 30 min, then cooling to a temperature between 25 °C and 27 °C (75 °F and 80 °F). Solid salt shall be visible in the liquid after equilibration.

H.2.1.3 If no crystals are visible, some additional crystals or grains of the same salt should be added to induce precipitation. This is often necessary with calcium chloride. Salts of reagent-grade purity are recommended. However, if necessary, field-grade NaCl or $CaCl_2$ can be substituted.

H.2.2 Desiccant, anhydrous **calcium chloride (CAS No. 10043-52-4)**. Other desiccants may be calcium sulfate (CAS No. 10101-41-4), Drierite® (CAS No. 7778-18-9), or silica gel (CAS No. 112926-00-8).

H.2.3 Electrohygrometer, incorporating a portable, relative humidity sensor and indicator, with the specifications below. If this instrument is equipped with a temperature probe, a separate thermometer is unnecessary.

a) The relative humidity sensor probe should be suitable for insertion in the air space of a measuring container with the sample.

b) The linearity response of the instrument should be such that the correlation coefficient between published standard brine activity and relative humidity, as measured by the instrument above each brine, is greater than 0,95.

c) The instrument should give a reading within 10 % of the true activity within 30 min, when measuring the brine activity of the standard salt solutions in H.2.1.

H.2.4 Base-oil supply, 1,0 l to 1,5 l (0,25 gal to 0,4 gal), clean, water-free.

Use the same oil as has been used in the oil-based drilling fluid. Ensure that no free water is present.

H.2.5 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F), not required if instrument has an internal thermometer.

H.2.6 Measuring container, as suggested or supplied by the manufacturer, having a volume sufficient to provide a liquid surface area of approximately 30 cm^2 to 50 cm^2 (4,5 in^2 to 7,75 in^2). The container should have a flexible elastomer cap (airtight) which, when bored through, will form an airtight seal around the shaft of the sensor probe and thermometer (if used).

H.2.7 Insulating jacket or cover, made of synthetic foam, for the measuring container. The cover should fit snugly and should insulate the side and bottom of the container from temperature fluctuations. The instrument may have equipment to maintain a constant sample temperature. Alternatively, the container can be kept in a constant-temperature water bath.

H.2.8 Linear graph paper.

H.2.9 Paper towels, one or more rolls.

H.2.10 Washing bottle, plastic or glass bottle with a volume of 500 ml and with a screw lid.

H.2.11 Sieve, with openings of 0,589 mm (28 mesh).

H.2.12 Graduated beaker, 400 ml.

H.3 Procedure

H.3.1 Calibration of hygrometer

H.3.1.1 Prepare sufficient volume of each known relative humidity standard to cover the bottom of the measuring container to a liquid depth of 12 mm (0,5 in), or follow the manufacturer's recommendations. These salt solutions shall be kept firmly capped to prevent spillage, evaporation or contamination.

H.3.1.2 Desiccate the probe with anhydrous calcium chloride or other desiccant in a measuring container by placing the sensor probe (which is in the measuring container cap) in the air space above the salt. Wait 10 min to 15 min. A relative humidity reading of 24 % or lower indicates that the probe is dry. Keep a distance of 12 mm (0.5 in) between the sensing probe and the desiccant, and maintain the same distance for salt solutions and cuttings samples. Never allow the probe to directly contact any samples or solutions.

H.3.1.3 Transfer the probe in the measuring container cap to the first (lowest a_W) standard solution and allow 30 min for equilibrium to be reached. Temperature of the standards should be maintained between 24 °C and 25 °C (75 °F and 77 °F).

H.3.1.4 Remove the probe from the first solution and place it into the container with the solution of second lowest known activity.

H.3.1.5 Proceed as in H.3.1.3 and measure the relative humidity for each solution, proceeding from that with the lowest to that with the highest activity a_W.

H.3.1.6 Allow each standard to equilibrate for the same length of time. Record the temperature and percent relative humidity (% RH) indicated. Plot the % RH versus the activity of each solution, a_W.

H.3.2 Measurement of oil-contaminated cuttings activity, a_C

H.3.2.1 Desiccate the probe for 10 min to 15 min as in H.3.1.2.

H.3.2.2 Collect a fresh sample of cuttings directly from the final stage solids control equipment, taking care to minimize its exposure to air. Minimize the amount of oil-based drilling fluid adhering to the cuttings.

H.3.2.3 Add 300 ml of room-temperature base oil to the 500 ml washing bottle.

H.3.2.4 Measure approximately 100 ml of loosely packed, fresh cuttings (H.3.2.2) into the 400 ml graduated beaker. Transfer these to the bottle containing the base oil and fasten the lid.

H.3.2.5 Gently shake the bottle by hand for 30 s.

H.3.2.6 Remove the lid and place the sieve over the mouth of the bottle. Pour off and discard the wash oil, retaining the cuttings in the bottle.

H.3.2.7 Again, add 300 ml of base oil to the bottle containing the same cuttings and repeat steps in H.3.2.5 and H.3.2.6. Continue these washing steps until the oil looks clean after shaking with the cuttings (usually two to four washings).

H.3.2.8 Drain off the oil after washing the cuttings. Pour the cuttings onto three to four layers of absorbent paper towel. Fold the towels over the cuttings and gently blot them dry. Minimize exposure of the cuttings to the open air.

H.3.2.9 Quickly transfer 35 ml to 45 ml of the washed cuttings into the measuring container. Place the measuring container into the insulating foam jacket or constant-temperature water bath, if one is used. Install the cap.

H.3.2.10 Replace the container lid with the lid that has the pre-calibrated hygrometer probe inserted. Ensure that the probe is inserted into the air space 12 mm (0,5 in) above the cuttings and does not contact the cuttings. The cuttings sample temperature should be maintained between 24 °C and 25 °C (75 °F and 77 °F).

H.3.2.11 Turn on the hygrometer and wait 30 min (the same time period used for standard calibration), and record the percent relative humidity and temperature.

H.4 Calculation — Cuttings activity, a_C

H.4.1 Find the relative humidity obtained in H.3.2 on the graph plotted in H.3.1.6. Read and record the corresponding activity that appears on the graph as the drilling fluid activity, a_{DF}.

H.4.2 If not measured directly, follow these plotting instructions:

a) using data for standards given in H.3.1, plot % RH versus a_W on linear graph paper;

b) plot the data obtained for each sample (H.3.2) on the linear graph paper and read the cuttings activity, a_C.

Annex I

(informative)

Chemical analysis of active sulfides by the Garrett gas train method

I.1 Principle

I.1.1 This procedure uses the Garrett gas train to measure active sulfides in an oil-based drilling fluid. The gas train separates gas from a liquid sample and passes the gas through a hydrogen sulfide detector, the Dräger[10] tube, for quantitative sulfide analysis. The Dräger tube responds to H_2S by progressively darkening along its length as H_2S reacts with the tube reagent. Two Dräger tubes cover a wide range of sulfide concentrations, from approximately 1 mg/l to 4 000 mg/l. The low-range tube is white until H_2S turns it brownish black. The high-range tube is pale blue until H_2S turns it black. No known common drilling fluid component or contaminant other than H_2S causes this discolouration in Dräger tubes.

I.1.2 Lead-acetate paper disks can be placed in the Garrett gas train to detect qualitatively the presence or absence of sulfide. If the presence of sulfide is indicated by darkening of the lead-acetate paper, a Dräger tube should be used for quantitative analysis.

I.1.3 Whole oil-based drilling fluid, not filtrate, is analysed in order to capture all of the active sulfides. Active sulfides are typically the neutralization products of H_2S and lime, plus any non-reacted H_2S gas. Use of a weak acid in the gas train liberates H_2S gas from active sulfides, but not from inert sulfides such as zinc sulfide, copper sulfide or lead sulfide.

I.2 Reagents and apparatus

I.2.1 Citric acid (**CAS No. 77-929**)**/demulsifier/isopropanol** (**CAS No. 67-63-0**) **solution**. Prepare a 2 mol/l citric acid solution by dissolving 420 g reagent-grade citric acid in deionized water to make 1 000 ml of solution. Mix 200 ml of isopropanol and 25 ml of demulsifier[11] into 1 000 ml of 2 mol/l citric acid solution.

I.2.2 Antifoam, **octanol** (**CAS No. 111-87-5**), in a dropper bottle.

I.2.3 Acetone (CAS No. 67-64-1).

I.2.4 Carrier gas, inert to hydrogen sulfide, sulfide salts, citric acid, demulsifier, isopropanol and Dräger tube reagents. Nitrogen is preferred but carbon dioxide is acceptable. Avoid air or other oxygen-containing gases.

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[10] Dräger® tubes are 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.

[11] Schlumberger Dowell® W35 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.

I.2.5 Dräger H_2S analysis tubes, as specified in Table I.1.

Table I.1 — Dräger H_2S analysis tubes

Sulfide tube range	Tube marking	Tube part number
Low-range tube	H_2S 100/a	No. CH-291-01
High-range tube	H_2S 0,2 %/A	No. CH-281-01

I.2.6 Garrett gas train apparatus, consisting of a gas train of transparent material or glass, an inert gas supply and pressure regulator, a floating-ball flow meter, and a Dräger tube. The components shall have the following characteristics:

I.2.6.1 Body:

- Chamber 1:
  - depth: 90 mm (3,54 in)
  - diameter: 39 mm (1,52 in)
- Chambers 2 and 3:
  - depth: 90 mm (3,54 in)
  - diameter: 30 mm (1,18 in)
- Passages between chambers:
  - diameter: 2,0 mm (0,08 in)
- Material:
  - transparent material or glass which is inert to acid, sulfides and hydrogen sulfide gas.

I.2.6.2 Dispersion tube:

- Stem:
  - diameter: 8,0 mm (0,315 in)
  - length: 150 mm (5,9 in)
- Dispersion frit (bell-shaped, fine):
  - diameter: 30 mm (1,18 in)
- Material:
  - low coefficient of expansion, heat-resistant glass.

I.2.6.3 Flow meter, floating-ball type preferred, capable of measuring 300 ml/min of nitrogen (preferred) or CO_2 gas.

I.2.6.4 Flexible tubing, inert to hydrogen sulfide and carrier gas. Latex rubber or equivalent is preferred.

I.2.6.5 Fittings and rigid tubing, inert to hydrogen sulfide and acid.

I.2.7 Syringe, 10 ml, constructed of glass or plastic that is inert to oil-based drilling fluid and sulfides.

I.2.8 Graduated cylinder, 25 ml.

I.2.9 Magnetic stirrer, electric.

I.2.10 Stirring bar, 2,5 cm (1 in) stirring bar, coated with glass or PTFE.

I.2.11 Sample injection tube, with 15 mm (0,6 in) needle of PTFE or equivalent plastic that is inert to oil-based drilling fluid and sulfides.

I.2.12 Soft brush.

I.2.13 Pipe cleaners.

I.3 Procedure

I.3.1 Follow the manufacturer's instructions for assembly and operation of Garrett gas train equipment.

I.3.2 Pass the injection tube through the rubber septum on top of Chamber 1, making sure it seals in the septum.

I.3.3 Set the gas train body, with top removed, on the magnetic stirrer with the centre of Chamber 1 over the centre of the stirrer. Place the stirring bar in Chamber 1. Remove the rubber feet from the gas train body so it will rest flat on the stirrer.

I.3.4 Add 20 ml of the citric acid/demulsifier/isopropanol solution (I.2.1) into Chamber 1.

I.3.5 Add 10 drops of antifoam into Chamber 1.

I.3.6 See Table I.2 for sample volume and type of Dräger tube required for the expected range of sulfide. Select the proper type of Dräger tube. Break the tip off each end of the tube.

I.3.7 Insert the Dräger tube, with the arrow pointing downward, into the bored receptacle. Likewise, insert the flow meter tube, with “TOP” upward. Ensure the O-rings seal around the body of each tube.

I.3.8 Place the top on the gas train and hand-tighten all screws evenly to seal the O-rings. Position the dispersion tube high enough in Chamber 1 to be above the liquid level.

I.3.9 Connect the regulated gas supply, by a flexible hose, to the top of the dispersion tube of Chamber 1.

I.3.10 Attach the flexible tubing from the Chamber 3 outlet to the Dräger tube.

CAUTION — Do not clamp flexible tubing; unclamped tubing provides pressure relief in the event of over-pressurization.

I.3.11 Use a syringe without a needle to withdraw the drilling fluid sample volume, as determined in I.3.6, plus 0,5 ml of excess drilling fluid.

NOTE The excess drilling fluid compensates for the hold-up volume inside the injection tube, which is approximately 0,5 ml.

I.3.12 Attach the syringe to the injection tube and restrain the plunger, e.g. with a rubber band, to keep it from being blown out under pressure.

I.3.13 Allow the carrier gas to flow for 15 s, while keeping the dispersion tube frit above the liquid level in Chamber 1, to purge air from the gas train. Avoid frothing of the solution in Chamber 1. With the carrier gas flowing, check the gas train for leaks. Stop the gas flow.

I.3.14 Turn on the magnetic stirrer. Adjust its speed until a vortex is formed. Carefully lower the gas dispersion tube into the liquid to a point just above the rotating stirring bar.

I.3.15 Lower the injection tube until the outlet is slightly below the dispersion tube. This will allow the oil-based drilling fluid to enter the vortex.

I.3.16 Inject the sample from the syringe very slowly into Chamber 1. Increase the stirrer speed to improve dispersion and to prevent the oil-based drilling fluid from sticking to the walls of Chamber 1. Continue stirring for about 1 min.

I.3.17 Restrain the syringe plunger again and restart carrier gas flow. Adjust the flow rate to between 200 ml/min and 400 ml/min by keeping the flow-meter ball between the marks.

NOTE One nitrogen (preferred) or CO_2 cartridge generally provides about 15 min to 20 min of flow at this rate.

I.3.18 Observe changes in appearance of the Dräger tube. Note and record the maximum darkened length (in units marked on the tube) before the front starts to smear. Continue the carrier gas flow for a total of 15 min, although the front may attain a diffuse and feathery colouration. In the high-range tube, an orange colour (caused by SO_2) may appear ahead of the black front if sulfites are present in the sample. The orange SO_2 region should be ignored when recording darkened length.

For the best Dräger tube accuracy, the “darkened length” should fill more than half the tube's length; therefore the drilling fluid sample volume shall be carefully selected.

Table I.2 — Dräger tube (or equivalent) identification, sample volume, and tube factors to be used for various sulfide concentration ranges

Sulfide concentration range mg/l	**Sample volume** ml	**Dräger tube identification** (see tube body)	**Tube factor**[a] (used in calculation)
1,2 to 24	10,0	H_2S 100/a	0,133
2,4 to 48	5,0	H_2S 100/a	0,133
4,8 to 96	2,5	H_2S 100/a	0,133
30 to 1 050	10,0	H_2S 0,2 %/A	1 330
60 to 2 100	5,0	H_2S 0,2 %/A	1 330
120 to 4 200	2,5	H_2S 0,2 %/A	1 330

[a] If other tubes are used, the tube factors in column 4 shall be changed according to manufacturer's specifications.

I.3.19 Clean the gas train apparatus immediately after each use to minimize deterioration of the plastic. Clean the chambers and passages with warm water and mild detergent, using a soft brush and pipe cleaner. Wash the dispersion tube frits and the injection tube with an organic solvent, such as acetone, and then with water. Use gas to blow out the frits. Occasionally, the dispersion tube may need to be soaked in strong acid to remove calcium carbonate deposits. Rinse the entire gas train unit with deionized water and allow it to drain dry.

I.4 Calculation

Using the measured drilling fluid sample volume, V_S, in millilitres, the Dräger tube's maximum darkened length, l_D, in units marked on the tube, and the tube factor, f, from Table I.2, calculate the mass concentration of active sulfides in the drilling fluid sample:

$$c_{S^{-2}} = \frac{l_D \times f}{V_S} \quad (I.1)$$

where

$c_{S^{-2}}$ is the concentration of active sulphide, expressed in milligrams per litre;

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

l_D is the Dräger tube darkened length, marked in units on the tube;

f is the tube factor, taken from Table I.2.

Annex J
(informative)

Calibration and verification of glassware, thermometers, viscometers, retort kit cup and drilling fluid balances

J.1 Principle

This annex suggests methods for the calibration of field testing equipment not covered under test procedures in this part of ISO 10414.

J.2 Calibration of apparatus

J.2.1 Volumetric glassware

Calibration of pipettes, graduated cylinders and similar equipment is generally performed by the glassware supplier and may be part of the purchase specification. Where deemed important, glassware users should obtain documented evidence of glassware calibration from the supplier. Calibration may be checked gravimetrically. Periodic recalibration is not required. A procedure for calibration of graduated cylinders is given in J.3.

J.2.2 Thermometers

Calibrate thermometers used in field testing, especially commonly used metal-dial thermometers, against a standard thermometer. Thermometers should be calibrated before being put into service, and then periodically, depending on the importance of the measurement and the stability of the thermometer. A procedure for the calibration of thermometers is given in J.4.

J.2.3 Viscometers

Calibrate viscometers used in field testing against traceable standard viscosity fluids. Viscometers should be calibrated before being put into service, and then periodically, depending on the importance of the measurements and the stability of the viscometer. While in service, viscometers should be checked at least monthly and more frequently if the viscometer indicates instability. A procedure for the calibration of viscometers is given in J.5.

J.2.4 Drilling fluid balances

Calibrate drilling fluid balances using deionized water, as specified in the test procedure given in 5.2.1. The recommended frequency is prior to each set of measurements, or as specified by the operator, drilling fluid company or other interested party.

J.2.5 Retort kit cups

Calibrate retort kit cups using deionized water, as specified in the test procedure given in J.6. The recommended frequency is prior to each set of measurements, or as specified by the operator, drilling fluid company or other interested party.

J.3 Procedure — Calibration of graduated cylinders

J.3.1 Allow the receiver and distilled water to reach ambient temperature. Record the temperature to the nearest 0,5 °C (1 °F).

J.3.2 Place the clean, empty receiver, with its base, on the balance and tare to zero.

J.3.3 Fill the receiver with distilled water, while it is on the balance, to the various graduation marks in increments of 2 ml for the 10 ml receiver, in increments of 4 ml for the 20 ml receiver, and in increments of 10 ml for the 50 ml receiver. Use a pipette or syringe to carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.

J.3.4 Record the masses for each incremental volume of water at the specific graduation mark, m_W, to the nearest 0,01 g.

J.3.5 Calculate the volume of the receiver at each mark, V_M:

$$V_\mathrm{M} = \frac{m_\mathrm{W}}{\rho_\mathrm{W}} \tag{J.1}$$

where

V_M is the receiver volume at a specific mark, expressed in millilitres;

m_W is the mass of water, expressed in grams;

ρ_W is the water density, expressed in grams per millilitre, at the test temperature (see Table J.1).

J.4 Procedure — Calibration of thermometers

J.4.1 Place the thermometer to be calibrated alongside a standard thermometer in a constant temperature bath [or suitable container of 4 l (1 gal) capacity or more, filled with water, in a room where temperature is relatively constant] and allow to equilibrate for 30 min.

J.4.2 Read both thermometers and record the readings.

J.4.3 Repeat the readings at 5 min intervals to obtain at least four sets of readings.

J.4.4 Calculate the average reading for each thermometer.

J.4.5 For adjustable-scale thermometers, adjust the scale to read the same as the standard thermometer. For other thermometers, follow the procedure in J.4.6.

Table J.1 — Density of water as a function of temperature

°C	°F	Density g/ml
15,0	59,0	0,999 1
15,5	59,9	0,999 1
16,0	60,8	0,999 0
16,5	61,7	0,998 9
17,0	62,6	0,998 8
17,5	63,5	0,998 7
18,0	64,4	0,998 6
18,5	65,3	0,998 5
19,0	66,2	0,998 4
19,5	67.1	0,998 3
20,0	68,0	0,998 2
20,5	68,9	0,998 1
21,0	69,8	0,998 0
21,5	70,7	0,997 9
22,0	71,6	0,997 7
22,5	72,5	0,997 6
23,0	73,4	0,997 5
23,5	74,3	0,997 4
24,0	75,2	0,997 3
24,5	76,1	0,997 1
25,0	77,0	0,997 0
25,5	77,9	0,996 9
26,0	78,8	0,996 8
26,5	79,7	0,996 6
27,0	80,6	0,996 5
27,5	81,5	0,996 4
28,0	82,4	0,996 2
28,5	83,3	0,996 1
29,0	84,2	0,995 9
29,5	85,1	0,995 8
30,0	86,0	0,995 6
30,5	86,9	0,995 5
31,0	87,8	0,995 3
31,5	88,7	0,995 2
32,0	89,6	0,995 0
32,5	90,5	0,994 9
33,0	91,4	0,994 7
33,5	92,3	0,994 5
34,0	93,2	0,994 4
34,5	94,1	0,994 2
35,0	95,0	0,994 0

J.4.6 Calculate the thermometer correction as follows:

$$C = R_1 - R_2 \tag{J.2}$$

where

C is the correction value to add to the thermometer reading;

R_1 is the average reading for the standard thermometer, expressed in degrees;

R_2 is the average reading for the working thermometer, expressed in degrees.

EXAMPLE 1 Thermometer correction determination, in degrees Celsius:

$R_1 = 23{,}9\ °\text{C}$

$R_2 = 24{,}2\ °\text{C}$

$C = 23{,}9 - 24{,}2 = -\ 0{,}3\ °\text{C}$

EXAMPLE 2 Thermometer correction application:

$C = -\ 0{,}3\ °\text{C}$

$R = 25{,}0\ °\text{C}$

$R_\text{c}\ \text{(corrected)} = 25{,}0 + (-\ 0{,}3) = 24{,}7\ °\text{C}$

See ISO 386 for a more complete procedure description.

J.4.7 Mark and identify the thermometer with its correction and calibration date.

It is not good practice to use thermometers whose correction exceeds twice the allowable tolerance of the measurement.

J.5 Procedure — Calibration of viscometers

J.5.1 Obtain certified calibration fluids with chart (viscosity versus temperature) to cover the range of interest, such as 50 mPa·s, 100 mPa·s, etc. Make certain that the lot number on the chart matches the lot number on the fluid container. Each lot of standard fluid is individually certified. The viscosity will normally vary slightly from lot to lot.

J.5.2 Clean and dry the viscometer bob, rotor and cup. Place the viscometer and fluid side by side on a countertop in a room with a reasonably constant temperature [<3 °C (<5 °F) variation per hour]. Allow to stand at least 2 h to equilibrate.

NOTE Water will contaminate the standard fluid.

J.5.3 Operate the viscometer in air for 2 min to 4 min to loosen the bearings and gears.

Correct or replace the rotor if it wobbles excessively.

J.5.4 Pour the calibration fluid into the cup, filling it to the inscribed line, and place the cup on the viscometer stage. Move the stage until the fluid level is aligned with the inscribed line on the rotor sleeve.

J.5.5 Place a thermometer [accurate to ±0,1 °C (±0,2 °F)] into the fluid and hold or tape it in place to prevent breakage. Operate the viscometer at low speed until the thermometer reading is stable to within 0,1 °C (0,2 °F) per 30 s. Record the temperature reading.

J.5.6 Operate the viscometer and take readings at 600 r/min and 300 r/min. Estimate readings to the nearest 0,5 dial unit. If needed, use a magnifying glass.

J.5.7 Using the temperature/viscosity chart supplied with the calibration fluid, determine certified viscosity to the nearest 0,5 mPa-s. Compare the 300 r/min dial reading to the standard viscosity and record the deviation (plus or minus). Divide the 600 r/min dial reading by 1,98, compare it to the standard viscosity, and record the deviation.

J.5.8 Deviations exceeding 1,5 units are not acceptable. If the deviation exceeds these tolerances, adjust or calibrate the viscometer.

J.5.9 Record the viscometer serial number, date and deviation. Mark the viscometer with the date of calibration and indication of its calibration status.

J.6 Procedure — Calibration of retort kit cups

J.6.1 Allow the retort cup, lid and distilled water to reach ambient temperature. Record the temperature to the nearest 0,5 °C (1 °F).

J.6.2 Place the clean, empty retort cup and lid on the balance and tare to zero.

J.6.3 Fill the retort cup with distilled water. Place the lid on the cup. Rotate the lid to obtain a proper fit. Be certain a small excess of water flows out of the hole in the lid. Wipe excess water from the lid; avoid wicking out water.

J.6.4 Place the filled retort cup with lid on the previously tared balance. Record the water mass to the nearest 0,01 g.

J.6.5 Calculate the retort cup volume, V_{RC}, using the water density at ambient temperature (see Table J.1)

$$V_{\mathrm{RC}} = m_{\mathrm{w}}/\rho_{\mathrm{w}} \tag{J.3}$$

where

V_{RC} is the volume of the retort cup, expressed in millilitres;

m_{w} is the mass of water, expressed in grams;

ρ_{w} is the water density, expressed in grams per millilitres, at the test temperature (see Table J.1).

J.7 Procedure — Calibration of filter press gasket and O-rings

J.7.1 Gaskets or O-rings for the low-pressure, low-temperature filter press shall have an inside diameter of between 75,8 mm (2,99 in) and 76,9 mm (3,03 in).

J.7.2 Check the gasket or O-ring using a gauge with a diameter of between 75,8 mm (2,99 in) and 76,9 mm (3,03 in).

J.7.3 Do not use gaskets or O-rings with diameters outside this range for filter loss testing.

Annex K
(informative)

High-temperature/high-pressure filtration testing of oil-based drilling fluids using the permeability plugging apparatus and cells equipped with set-screw secured end caps

K.1 Principle

K.1.1 Measurements of the filtration behaviour and wall-building characteristics of drilling fluid are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water or emulsion concentration.

K.1.2 These characteristics are affected by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The permeability plugging apparatus (PPA) is a modified high-temperature, high-pressure filter press used to evaluate these interactions through various types of filter media at pressures up to 13 800 kPa (2 000 psi) and temperatures from ambient to as high as 260 °C (500 °F). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

K.2 Safety considerations

K.2.1 The pressure limitation in the use of the PPA depends upon the cell in use. There are two types of cells available: those with threaded end caps (see Annex L) and those with set-screw-secured end caps (as considered in this annex). Among these cells are a total of five different pressure ratings. For safety, it is imperative that the operator know the maximum operating pressure of the test apparatus with certainty and that this pressure not be exceeded. If in doubt, contact the manufacturer or use the lowest of the possible limits.

K.2.2 Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the apparatus. Improper assembly, incorrect operation or the use of defective parts create the possibility of cell leakage or failure, which could result in serious injury or apparatus damage.

K.2.3 The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching hot parts of the apparatus during normal operation.

K.2.4 These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury and apparatus damage. These instruments should be used only on grounded circuits.

K.2.5 For safe operation of the hydraulic pressurization system, follow the instructions below.

a) Make certain the hydraulic pressure has been released and the pressure gauge on the pump reads zero before:

    1) attempting to disconnect the pressure hose from the cell at quick coupler;

    2) attempting to remove the cell from the heating jacket;

    3) moving the PPA;

    4) refilling the hydraulic pump;

5) performing any maintenance, including the tightening of leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

b) After refilling or repairing the hydraulic system, clean any spilled oil. Oil left on the floor is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

c) Make certain, when assembling the cell, that the end cap set screws are properly aligned and tightened.

K.2.6 For safe pneumatic pressurization of the back-pressure receiver, follow the instructions below.

a) Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it shall be supplied in an approved nitrogen gas cylinder or the nitrogen supply system should be built into the laboratory. Nitrogen cylinders should be secured to meet safety standards. CO_2 is normally supplied in small cartridges pressurized to about 6 200 kPa (900 psi). They are primarily used for field operations.

CAUTION — Do not allow CO_2 cartridges to be heated or exposed to fire. They can explode if overheated.

CAUTION — Do not use nitrous oxide cartridges as pressure sources for the 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 10414-1:2008, Clause A.5.

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

b) Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

c) Repair or replace leaking hydraulic or pneumatic pressurization systems. Gauges, fittings and hoses should be kept in good condition, and leaks should be found and corrected. Periodically, test the pressure-relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

d) Always open the supply pressure first when pressurizing the back-pressure assembly. Then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the apparatus rating or relief-valve settings. When relieving back-pressure, shut the supply pressure, bleed the pressure from the system, and back out the regulator T-screw.

K.2.7 For safe heating, follow the instructions below.

a) Exercise caution to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

b) Avoid the practice of removing the cell and cooling it with water. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

K.2.8 For safe electrical operation, follow the instructions below.

a) Make certain that the electrical source is fused and grounded (earthed). Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

b) Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time, or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

K.2.9 For test cell maintenance, the user should be aware that the filtration cell is a pressure vessel and should be considered a source of potential danger. The safety precautions listed below should be followed to ensure safe operation.

a) Cell material shall be compatible with the test samples.

b) Do not use cells that show signs of severe pitting or stress cracking.

c) Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

d) Use only undamaged, hardened-steel set-screws. Others are unsafe.

K.3 Apparatus — Permeability plugging apparatus (PPA) with set-screw-secured end caps

K.3.1 PPA cell

a) There are three different types of cell available that use set-screw-secured end caps. Those of current and recent manufacture are rated at either 13 800 kPa (2 000 psi) or 12 400 kPa (1 800 psi).

There are still a number of older cells in use which are stamped “2 500 psi”. In 1996, the rating on these cells was reduced to “1 800 psi” (12 420 kPa). Unless the user can absolutely verify that the cell to be used is rated at 13 800 kPa (2 000 psi), the “1 800 psi” (12 420 kPa) pressure limit should be observed.

The operating manual, or this procedure, should be attached to the apparatus and read by anyone who is unfamiliar with the apparatus, before using it.

CAUTION — Follow the manufacturer's recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

b) As received from the manufacturer, the PPA will be equipped with valves that are rated to 260 °C (500 °F). If it becomes necessary to change any valves during the life of the apparatus, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

c) The PPA is designed to provide improved static filtration measurements. It can be operated at pressure differentials and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top, and the filtrate collected from the top. A small hydraulic hand-pump applies the cell pressure. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of hydraulic oil with the sample.

d) The PPA utilizes ceramic disks as filtration media. Ceramic disks are available with mean pore throat sizes ranging from 10 µm to 120 µm.

e) Test pressures are limited by the safety limits of the cell as specified by the manufacturer, usually either 12 400 kPa (1 800 psi) or 13 800 kPa (2 000 psi) at some defined temperature. If back-pressure is used in the test, the test pressure may have to be reduced to avoid exceeding the pressure limit of the cell and thus damaging the cell.

f) Cell damage caused by excessive pressure can be categorized as follows: end-cap bending, end-cap compression, cylinder shear, and cylinder stress. End-cap bending can be detected visually or by measurement. End-cap compression can de detected by the distortion of the set-screw holes or seats, which become oval rather than round. Caps showing signs of damage should not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set-screw holes, should not be used.

g) For temperatures above 90 °C (195 °F), the back-pressure receiver shall be pressurized to prevent boiling of the filtrate. The standard back-pressure receiver uses a CO_2 pressurizing source to provide the back-pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

h) The PPA cell is encased in a thermostatically controlled aluminium heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature has been obtained once, it can be repeated by setting the thermostat knob to the same reference setting or, alternatively, by using a temperature controller association with a thermocouple. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 W.

i) The PPA can be used in the field or in the laboratory. A stainless-steel carrying case with a fold-down workshelf is available for use in the field.

K.3.2 **Filter medium**, ceramic disks

a) Standard disk thickness is 6,5 mm (0,25 in) but, with adapters, thicker disks can be used. A new disk is required for each test. For oil-based drilling fluids, the disk shall be soaked for 5 min to 10 min in a sample of the base oil before use.

Mercury intrusion porosimetry shall be used to characterize ceramic disk media.

CAUTION — There are health considerations when using mercury in its vapour form. Follow all manufacturer's instructions and local, state, and federal guidelines concerning mercury.

The manufacturers run a quality-control test for disk classification and these disks are sold with an average mean pore throat number as determined by mercury injection porosimetry (see Reference [14]). Table K.1 lists the commonly available ceramic disks and the mean pore throat size designation.

Table K.1 — Mean pore throat diameter of ceramic disks

Values in micrometres

API designation	New mercury injection pore throat size	Previous air designation
API designation 10	10	3
API designation 12	12	5
API designation 20	20	10
API designation 40	40	20
API designation 50	50	35
API designation 55	55	60
API designation 120	120	90

NOTE Mercury injection porosimetry is not a viable technique for measuring pore throat sizes exceeding 120 µm. There is no API designation for previously offered 150 µm and 190 µm ceramic disks.

b) Ceramic disks were adopted for use in the PPA as a method to test plugging/bridging materials in drilling fluids. This media was chosen based on pore size. It should be noted that ceramic disks do not exhibit the normal relationship between pore throat size and permeability as naturally occurring sandstone and, therefore, any work conducted to assess bridging characteristics should be conducted based on pore throat size.

c) Ceramic disks differ from reservoir rocks in three important ways. Firstly, the disks have extremely narrow pore size distribution. Secondly, the disks have an extremely high permeability due to this narrow pore size distribution and a less tortuous pore flow path. Thirdly, the disks have an extremely high porosity, explessed as a percentage.

d) Depending on the pore size distribution of the reservoir rock, multiple tests using a range of ceramic disks may be necessary to properly assess the benefit of a particular bridging treatment.

K.3.3 Timer, accurate to 0,1 min over the test interval.

K.3.4 Thermometer, graduated to 260 °C (500 °F), with an accuracy of ±1 °C (±2 °F).

K.3.5 Graduated cylinder (TC), 25 ml or 50 ml.

K.3.6 Multimixer®[12], capable of operating at 11 500 r/min ± 300 r/min under load, with a single corrugated impeller of approximately 25,4 mm (1 in) in diameter.

K.3.7 Container, for mixing, 180 mm deep (7,125 in), $d = 97$ mm (3,75 in) at top and 70 mm (2,75 in) at bottom.

K.4 Procedure for high-temperature, high-pressure (HTHP) filtration

K.4.1 Preheating the heating jacket

K.4.1.1 Connect the power cord to the proper voltage as indicated on the nameplate.

K.4.1.2 Turn the thermostat to mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will illuminate when the heating jacket temperature has reached the thermostat setting.

K.4.1.3 Readjust the thermostat to 6 °C (10 °F) over the desired test temperature.

K.4.2 Loading the filtration cell

K.4.2.1 The filtration cell is a pressure vessel. The following safety precautions should be followed to ensure safe operation:

a) the cell material should be resistant to the test sample;

b) cell bodies that show signs of stress cracking or severe pitting should not be used;

c) use only undamaged, hardened-steel set-screws: the use of damaged, or common, mild-steel set-screws is hazardous.

K.4.2.2 Loosen the set-screws securing the end caps, then remove the caps from the cell by pulling them straight out, using the nipples and connected hardware as handles. The cell cap removal tool is not a standard item supplied with the PPA.

K.4.2.3 Check the O-rings on the nipples, the floating piston, the cell body and the end caps, and replace any that are damaged or brittle [all O-rings should be replaced routinely after tests at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease all around the O-rings, being especially careful to ensure that those on the piston are well lubricated.

K.4.2.4 Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely (the bottom of the cell, the inlet end, has a shorter recess than the top). Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

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12) Multimixer® Model 9B with B29 impeller is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of this product.

K.4.2.5 Fill the space above the piston with hydraulic oil to just above the end face.

K.4.2.6 Install the hydraulic end cap onto the bottom of the cell by pushing in the back-pressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to slide into the cell more easily. Install and tighten the set-screws.

Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

K.4.2.7 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

The steps that follow can be accomplished in the jacket that is being preheated, in an unheated jacket if one is available, or in a specially constructed stand.

CAUTION — If performing these tasks in a jacket that is heating, use extreme caution to avoid burns.

K.4.2.8 Turn the cell upright and fill with approximately 275 ml of drilling fluid. This allows for expansion while heating. Do not exceed this amount.

CAUTION — Filling with more than 275 ml may present a condition where the cell pressure exceeds the recommended maximum pressure rating of the cell. This could lead to personal injury or damage to the equipment.

For improved consistency in test results, stir the drilling fluid for 5 min immediately before loading the cell.

K.4.2.9 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

K.4.2.10 Install the O-ring and set the selected ceramic disk on top of it.

K.4.2.11 Position the top end cap in the cell, tighten the set screws snugly, and close the valve on the top end cap.

K.4.2.12 Install the cell in the heating jacket. Make certain that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

K.4.2.13 Thermal expansion of cell contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid and prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the excess periodically.

K.4.3 Pressurizing the cell

K.4.3.1 Refer to Table K.2 for the pressure corresponding to the test temperature, and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it should always be operated at about one stroke per second.

Filtration at temperatures above the boiling point of the fluid sample requires the use of the back-pressure receiver to prevent vaporization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling.

Nitrogen manifolds having a 4 100 kPa (600 psi) gauge for back-pressure need to be modified to attain 4 850 kPa (700 psi). However, tests are possible within the limits of the manifolds.

K.4.3.2 While the cell is heating, use the procedure described below to prepare the back-pressure receiver.

a) Check to ensure that the regulator T-screw has been rotated counterclockwise enough to release all pressure. When the pressure has been released, the screw will turn freely.

b) Open the pressure-relief valve to relieve any remaining pressure and remove the CO_2 cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one, and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time.

c) Verify that the pressure-relief valve on the CO_2 assembly and the filtrate drain valve are closed.

d) Set the back-pressure assembly aside. It will be installed in K.4.3.4.

K.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as one hour.

K.4.3.4 When the cell is at the desired temperature and cell pressure has stabilized, mount the back-pressure receiver on the upper valve adapter. Secure the receiver with a retaining pin. Install the CO_2 pressurizing unit on top of the receiver. Lock the CO_2 pressurizing unit in place with the other retaining pin.

K.4.3.5 If a drain hose is to be used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space between the filtration medium and the back-pressure receiver outlet, and the receiver valve, should be filled with the base fluid before starting the test. This will ensure that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

K.4.3.6 See Table K.2 to determine the appropriate pressure for the back-pressure receiver and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

Table K.2 — Starting cell pressures and back-pressures for various test temperatures

Temperature range θ		**Heating pressure or back-pressure** P	
°C	°F	kPa	psi
Less than 95	Less than 200	0	0
95 to 150	200 to 300	690	100
151 to 175	301 to 350	1 050	150
178 to 190	351 to 375	1 400	200
191 to 205	376 to 400	1 725	250
206 to 218	401 to 425	2 420	350
219 to 232	426 to 450	3 100	450
233 to 246	451 to 475	3 800	550
247 to 260	476 to 500	4 850	700

K.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the back-pressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the back-pressure receiver.

K.4.4 Conducting the filtration test

K.4.4.1 Verify that the back-pressure, as read on the pressure regulator gauge, is correct. Adjust if required.

K.4.4.2 Set the timer for the desired filtration test times. Filtrate shall be collected at 1 min, 7,5 min and 30 min intervals. Additional data can be collected if desired; however, the first sample should not be taken prior to 1 min. Accurately recorded test times and filtrate measurements are necessary for accurate calculation of the filtration parameters.

For improved definition of spurt loss, collect filtrate at 1 min, 5 min, 7,5 min, 15 min, 25 min and 30 min, and plot cumulative filtrate volumes versus the square root of time.

K.4.4.3 Open the filtration valve to start the test. The cell pressure, as read on the pump gauge, will drop initially. Operate the pump to maintain it as close to the test pressure as possible. If a manually actuated pump is used, it shall be operated at about one stroke per second.

K.4.4.4 At the desired times, use the drain valve to bleed the filtrate from the back-pressure receiver into the graduated cylinder, and record the time and cumulative volume received.

It is recommended that the filtrate be recovered directly from the back-pressure receiver, not from a drain hose attached to it. Should a hose be used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

K.4.4.5 The pressure may slowly decrease as the test continues, due to the volume loss through filtration. Additional pressure shall be applied to the cell in order to maintain a constant pressure. Hold the desired pressure on the cell and on the back-pressure receiver for the duration of the test.

K.4.4.6 After 30 min, close the filtrate valve and drain any remaining filtrate from the back-pressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder shall be recorded.

K.5 Test conclusion and disassembly

K.5.1 Disconnect the heating jacket from the power source. The temperature of the sample in the cell should be reduced to below 38 °C (100 °F) to ensure that the cell can safely be opened.

K.5.2 The pressurized cell assembly shall be allowed to cool in the heating jacket. When these tests are run with sufficient frequency to justify it, a cooling stand, station or bath can be provided to expedite the cooling process. A cell-handling tool is available which should be used any time a hot cell is to be handled.

CAUTION — Extreme care should be exercised in cooling hot cells.

This procedure, as outlined, makes it difficult to perform more than one test in an 8 h work day with a single PPA. In the interest of improving productivity, users may want to design their own cell cooling procedures and apparatus. Safety should be the primary consideration in these designs.

K.5.3 Close the valve between the cell and the back-pressure receiver.

K.5.4 Release the pressure on the pump and cell by opening the pump valve, then disconnect the quick coupler between the pump and the nipple adapter on the bottom of the cell.

K.5.5 Release the back-pressure by turning the T-screw on the regulator counterclockwise until it turns freely.

K.5.6 Bleed the pressure from the back-pressure receiver by opening the pressure relief valve on the CO_2 unit. Open the drain valve on the back-pressure receiver and collect the last few drops of filtrate in the graduated cylinder. After removing the locking pin and securing it, remove the CO_2 assembly from the top nipple adapter. After removing its locking pin, remove the back-pressure receiver.

K.5.7 The cell may be opened after its contents have been allowed to cool. The cell should be opened only when the user is confident that the contents are no longer under pressure.

a) If pressure is suspected to remain in the cell, and the lower end cap does not include a screen, the following procedure can be used to determine the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom. If the piston is not at the bottom, there is no pressure. If the piston is at the bottom, there could be pressure remaining in the cell. Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the number of strokes of the piston required to move it and then the force to be applied.

b) If the indications are that the cell is pressurized, completely remove the filtration valve assembly from the cell and insert a small drill bit or wire into the cell cap to remove the obstruction. The drill or wire will stop when it contacts the filter disk. Make certain that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

K.5.8 The cell may be raised in the heating jacket and placed on the cell support, or laid on a bench, while it is being opened.

K.5.9 Loosen the end cap and, using the valve and nipple adapter as a handle, pull the cap from the cell. If it is stuck, a rocking motion of the valve and nipple adapter may free it. If it cannot be freed in this manner, unscrew the valve and nipple adapter, install the cap removal tool (if available) in its place and use it to remove the cap.

K.5.10 The end cap should be removed with the cell in the vertical position and the filtration end facing upwards.

K.5.11 Remove the filter disk. Use a small knife, small screwdriver or similar thin-bladed tool to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh base oil, then measure and record its thickness and any remarks concerning its composition and texture.

K.5.12 Pour the remaining fluid from the cell. Wash the inside of the cell with a suitable solvent. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 150 °C (300 °F) or higher.

If testing was conducted at temperatures above 150 °C (300 °F), the O-rings should be replaced.

K.5.13 Perform the three steps described below to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the bottom end cap using the procedure outlined in K.5.9 and K.5.10.

b) Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. Note that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all of the O-rings on the piston and the cap.

c) Clean the parts for reuse.

K.6 Calculation — Data reporting

K.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, in millilitres, collected through each of the selected time periods.

K.6.2 Spurt loss

The spurt loss (3.3) can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume [doubled to correct for filtration area when using 22,6 cm^2 (3,5 in^2) filtration media] is plotted along the y-axis. Alternatively, an approximate value can be calculated using Equation (K.2).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with K.4.4.2.

K.6.3 Calculation

Calculate the Permeability Plugging Test volume, spurt loss and static filtration rate using Equations (K.1), (K.2) and (K.3), respectively.

$$V_{\mathrm{PPT}} = 2\,V_{30} \tag{K.1}$$

$$V_1 = 2\left[V_{7,5} - \left(V_{30} - V_{7,5}\right)\right] = 2\left(2V_{7,5} - V_{30}\right) \tag{K.2}$$

$$v_{\mathrm{f}} = \frac{2\left(V_{30} - V_{7,5}\right)}{\sqrt{30} - \sqrt{7,5}} = \frac{2\left(V_{30} - V_{7,5}\right)}{2,739} \tag{K.3}$$

where

$V_{7,5}$ is the filtrate volume at the initial volume reading (after 7,5 min), in millilitres;

V_{30} is the filtrate volume at the final volume reading (after 30 min), in millilitres;

V_{PPT} is the PPT volume, in millilitres;

V_1 is the spurt loss, in millilitres;

v_{f} is the static filtration rate (velocity of flow), in millilitres per minute$^{1/2}$.

Observe that all three of these parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard low-pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant (2 in this case) can be modified as necessary to accommodate tests made utilizing other filtration areas.

K.6.4 Filter cake reporting

Measure and record the filter cake thickness to the nearest 1,0 mm (1/32 in). Include a description such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgments, they can convey important information.

Annex L
(informative)

High-temperature/high-pressure filtration testing of oil-based drilling fluids using the permeability plugging apparatus and cells equipped with threaded end caps

L.1 Principle

L.1.1 Measurements of the filtration behaviour and wall-building characteristics of drilling fluid are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate itself, such as its oil, water or emulsion concentration.

L.1.2 These characteristics are affected by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The PPA is a modified high-temperature, high-pressure filter press used to evaluate these interactions through ceramic-disk filter media at pressures up to 34 500 kPa (5 000 psi) and temperatures from ambient to as high as 260 °C (500 °F). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

L.2 Safety considerations

L.2.1 The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded end caps (considered in this annex) and those with set-screw-secured end caps (see Annex K). Among these cells are a total of five different pressure ratings. For safety, it is imperative that the operator know the maximum operating pressure of the test apparatus with certainty and that this pressure not be exceeded. If in doubt, contact the manufacturer or use the lowest possible limits.

L.2.2 Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the apparatus. Improper assembly, incorrect operation or the use of defective parts create the possibility of cell leakage or failure, which could result in serious injury or apparatus damage.

L.2.3 The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching parts of the apparatus during normal operation.

L.2.4 These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury and apparatus damage. These devices should be used only on grounded circuits.

L.2.5 For safe operation of the hydraulic pressurization system, follow the instructions below.

a) Make certain the hydraulic pressure has been released and the pressure gauge on the pump reads zero before:

    1) attempting to disconnect the pressure hose from the cell at the quick-coupler;

    2) attempting to remove the cell from the heating jacket;

    3) moving the PPA;

    4) refilling the hydraulic pump;

5) performing any maintenance, including tightening of leaking fittings on the hydraulic pump, hydraulic fittings or cell assembly.

b) After refilling or repairing the hydraulic system, clean any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

c) Make certain that the O-rings in the end caps are properly seated when assembling the cell.

L.2.6 For safe pneumatic pressurization of the back-pressure receiver, follow the instructions below.

a) Always use either nitrogen or carbon dioxide to pressurize the back-pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it should be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system should be built into the laboratory. Nitrogen cylinders should be secured to meet safety standards. CO_2 is normally supplied in small cartridges pressurized to about 6 200 kPa (900 psi). They are primarily used for field operations.

CAUTION — Do not allow CO_2 cartridges to be heated or exposed to fire. They can explode if overheated.

CAUTION — Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous 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.

b) Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

c) Repair or replace leaking hydraulic or pneumatic pressurization systems. Gauges, fittings and hoses should be kept in good condition, and leaks should be found and corrected. Periodically, test the pressure-relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

d) Always open the supply pressure first when pressurizing the back-pressure assembly. Then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the apparatus rating or relief-valve settings. When relieving back-pressure, shut the supply pressure, bleed the pressure from the system, and back out the regulator T-screw.

L.2.7 For safe heating, follow the instructions below.

a) Exercise caution to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

b) Avoid the practice of removing the cell and cooling it with water. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

L.2.8 For safe electrical operation, follow the instructions below.

a) Make certain that the electrical source is fused and grounded. Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

b) Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

L.2.9 For safe test cell maintenance, the user should be aware that the filtration cell is a pressure vessel and should be considered to be a source of potential danger. The safety precautions listed below should be followed to ensure safe operation.

a) Cell material shall be compatible with the test samples.

b) Do not use cells that show signs of severe pitting or stress cracking.

c) Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

L.3 Apparatus — Permeability plugging apparatus (PPA) with threaded end caps

L.3.1 PPA cell

a) There are two manufacturers of PPAs. Each supplies threaded end caps for the cells used for tests run at pressures in excess of 13 800 kPa (2 000 psi). There are threaded caps with three different pressure ratings available: 20 700 kPa (3 000 psi), 27 600 kPa (4 000 psi), and 34 500 kPa (5 000 psi), respectively.

The operating manual, or this procedure, should be attached to the apparatus and read by anyone who is unfamiliar with the apparatus before using it. If the user is unable to determine the operating limits with certainty, the lowest pressure limit should be assumed.

CAUTION — Follow the manufacturer's recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

b) As received from the manufacturer, the PPA will be equipped with valves that are rated to 260 °C (500 °F). If it becomes necessary to change any valves during the life of the apparatus, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

c) The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top, and the filtrate collected from the top. A small hydraulic hand-pump applies the cell pressure. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent contamination by hydraulic oil from the sample.

d) The PPA utilizes ceramic disks as filtration media. Ceramic disks are available with mean pore throat sizes ranging from 10 µm to 120 µm.

e) Test pressures are limited by the safety limits of the cell as specified by the manufacturer, usually 20 700 kPa (3 000 psi), 27 600 kPa (4 000 psi), and 34 500 kPa (5 000 psi) at 260 °C (500 °F). The back-pressure receiver may be used at pressures as high as 5 170 kPa (750 psi). If back-pressure is used in the test, the maximum test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

f) Cell caps showing signs of damage should not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting should not be used.

g) For temperatures above 90 °C (195 °F), the back-pressure receiver shall be pressurized to prevent boiling of the filtrate. The standard back-pressure receiver uses a CO_2 pressurizing source to provide the back-pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

h) The PPA cell is encased in a thermostatically controlled aluminium heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting or, alternatively, by using a temperature controller association with a thermocouple. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 W.

i) The PPA can be used in the field or in the laboratory. A stainless-steel carrying case with a fold-down workshelf is available for use in the field.

L.3.2 Filter medium, ceramic disks.

a) Standard disk thickness is 6,5 mm (0,25 in) but, with adapters, thicker disks can be used. A new disk is required for each test. For oil-based drilling fluids, the disk shall be vacuum-saturated at least 5 min to 30 min prior to use.

Mercury intrusion porosimetry shall be used to characterize ceramic disk media.

CAUTION — There are health considerations when using mercury in its vapour form. Follow all the manufacturer's instructions and local, state, and federal guidelines concerning mercury.

The manufacturers run a quality-control test for disk classification and these disks are sold with an average mean pore throat number as determined by mercury injection porosimetry (see Reference [14]). Table L.1 lists the commonly available ceramic disks and the mean pore throat size designation.

Table L.1 — Mean pore throat diameter of ceramic disks

Values in micrometres

API designation	New mercury injection pore throat size	Previous air designation
API designation 10	10	3
API designation 12	12	5
API designation 20	20	10
API designation 40	40	20
API designation 50	50	35
API designation 55	55	60
API designation 120	120	90

NOTE Mercury injection porosimetry is not a viable technique for measuring pore throat sizes exceeding 120 µm. There is no API designation for previously offered 150 µm and 190 µm ceramic disks.

b) Ceramic disks were adopted for use in the PPA as a method to test plugging/bridging materials in drilling fluids. This medium was chosen based on pore size. It should be noted that ceramic disks do not exhibit the normal relationship between pore throat size and permeability as naturally occurring sandstone and, therefore, any work conducted to assess bridging characteristics should be conducted based on pore throat size.

c) Ceramic disks differ from reservoir rocks in three important ways. Firstly, the disks have extremely narrow pore size distribution. Secondly, the disks have an extremely high permeability due to this narrow pore size distribution and a less tortuous pore flow path. Thirdly, the disks have an extremely high porosity (%).

d) Depending on the pore size distribution of the reservoir rock, multiple tests using a range of ceramic disks may be necessary to properly assess the benefit of a particular bridging treatment.

L.3.3 Timer, accurate to 0,1 min over the test interval.

L.3.4 Thermometer, graduated to 260 °C (500 °F), with an accuracy of ±1 C (±2 °F).

L.3.5 Graduated cylinder (TC), 25 ml or 50 ml.

L.3.6 Multimixer, capable of operating at 11 500 r/min ± 300 r/min under load, with a single corrugated impeller of approximately 25,4 mm (1 in) in diameter.

L.3.7 Container, for mixing, 180 mm deep (7,125 in), $d = 97$ mm (3,75 in) at top and 70 mm (2,75 in) at bottom.

L.4 Procedure for high-temperature, high-pressure (HTHP) filtration

L.4.1 Preheating the heating jacket

L.4.1.1 Connect the power cord to the proper voltage as indicated on the nameplate.

L.4.1.2 Turn the thermostat to the mid-scale setting and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will illuminate when the heating-jacket temperature has reached the thermostat setting.

L.4.1.3 Readjust the thermostat to 6 °C (10 °F) above the desired test temperature.

L.4.2 Loading the filtration cell

L.4.2.1 The filtration cell is a pressure vessel. The following procedure should be followed to ensure safe operation:

a) cell material shall be resistant to the test sample;

b) cell bodies that show signs of stress cracking or severe pitting shall not be used.

L.4.2.2 Use the spanner wrench to remove the end caps. Then unscrew the nipples from the caps and remove the piston from the cell.

L.4.2.3 Check the O-rings on the nipples, the floating piston, the cell body and the end caps, and replace any that are damaged or brittle [all O-rings should be replaced routinely after tests at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease all around the O-rings, being especially careful to ensure that those on the piston are well lubricated.

L.4.2.4 Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely (the bottom of the cell, the inlet end, has a shorter recess than the top). Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

L.4.2.5 Fill the space above the piston with hydraulic oil to just above the end face.

L.4.2.6 Lubricate the end face of the cell bore, the horizontal area at the end of the bore, with anti-seizing compound and fill the space above the piston with hydraulic oil to just above the end face.

L.4.2.7 Lubricate the threads with high-temperature-resistant grease and screw the end cap into place, tightening it moderately with the two-pin spanner wrench. Over-tightening will not improve the seal, and will make the cap difficult to remove.

L.4.2.8 Install the hydraulic end cap onto the bottom of the cell by pushing in the back-pressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to be screwed into the cell more easily.

Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

L.4.2.9 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

The steps that follow can be accomplished in the jacket that is being preheated, in an unheated jacket if one is available, or in a specially constructed stand.

CAUTION — If performing these tasks in a jacket that is heating, use extreme caution to avoid burns.

For improved consistency in test results, stir drilling fluid for 5 min immediately before loading the cell.

L.4.2.10 Turn the cell upright and fill with approximately 275 ml of drilling fluid.

CAUTION — Filling with more than 275 ml may present a condition where the cell pressure exceeds the recommended maximum pressure rating of the cell. This could lead to personal injury or damage to the equipment.

For improved consistency in test results, stir the drilling fluid for 5 min immediately before loading the cell.

L.4.2.11 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

L.4.2.12 Install the O-ring and set the selected ceramic disk on top of it.

L.4.2.13 Install the top end cap in the cell.

L.4.2.14 Lubricate the threads and the bottom of the retainer ring, and screw the ring into the top of the cell. Tighten it, using the single-pin spanner wrench if necessary, until the outer knurled flange of the retainer ring is flush against the top of the cell body. Attempting to tighten it further will not improve the seal and will make the cap more difficult to remove.

This step applies only to cells that utilize retainer rings for the top end caps.

L.4.2.15 Install the cell in the heating jacket. Make certain that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

L.4.2.16 Thermal expansion of cell contents and of the hydraulic fluid will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid and prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the excess periodically.

L.4.3 Pressurizing the cell

L.4.3.1 Filtration at temperatures above the boiling point of the fluid sample requires the use of the back-pressure receiver to prevent vaporization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling. Refer to Table K.2 for the pressure corresponding to the test temperature and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it shall always be operated at about one stroke per second.

L.4.3.2 While the cell is heating, use the procedure described below to prepare the back-pressure receiver.

a) Check to ensure that the regulator T-screw has rotated counterclockwise enough to release all pressure. When the pressure has been released, the screw will turn freely.

b) Open the pressure release valve to relieve any remaining pressure and remove the CO_2 cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one, and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time.

c) Verify that the pressure release valve on the CO_2 assembly and the filtrate drain valve are closed.

d) Set the back-pressure assembly aside. It will be installed in L.4.3.4.

L.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as one hour.

L.4.3.4 When the cell is at the desired temperature and cell pressure has stabilized, mount the back-pressure receiver on the upper valve adapter. Secure the receiver with a retaining pin. Install the CO_2 pressurizing unit on top of the receiver. Lock the CO_2 pressurizing unit in place with a retaining pin.

L.4.3.5 If a drain hose is to be used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space between the filtration medium and the back-pressure receiver outlet, and the receiver valve, should be filled with the base fluid before starting the test. This will ensure that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

L.4.3.6 Refer to Table K.2 to determine the appropriate pressure for the back-pressure receiver and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

L.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the back-pressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the back-pressure receiver.

L.4.4 Conducting the filtration test

L.4.4.1 Verify that the back-pressure as read on the pressure regulator gauge is correct. Adjust if required.

L.4.4.2 Set the timer for the desired filtration test times. Filtrate shall be collected at 1 min, 7,5 min and 30 min intervals. Additional data can be collected if desired; however, the first sample should not be taken prior to 1 min. Accurately recorded test times and filtrate measurements are necessary for accurate calculation of the filtration parameters.

For improved definition of spurt loss, collect filtrate at 1 min, 5 min, 7,5 min, 15 min, 25 min and 30 min, and plot cumulative filtrate volumes versus the square root of time.

L.4.4.3 Open the filtration valve to start the test. The cell pressure, as read on the pump gauge, will drop initially. Operate the pump to maintain it as close to test pressure as possible. If a manually actuated pump is used, it shall be operated at about one stroke per second.

L.4.4.4 At the desired times, use the drain valve to bleed the filtrate from the back-pressure receiver into the graduated cylinder, and record the time and cumulative volume received.

It is recommended that the filtrate be recovered directly from the back-pressure receiver, not from a drain hose attached to it. Should a hose be used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

L.4.4.5 The pressure may slowly decrease as the test continues, due to the volume loss through filtration. Additional pressure shall be applied to the cell in order to maintain a constant pressure. Hold the desired pressure on the cell and on the back-pressure receiver for the duration of the test.

L.4.4.6 After 30 min, close the filtrate valve and drain any remaining filtrate from the back-pressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder shall be recorded.

L.5 Test conclusion and disassembly

L.5.1 Disconnect the heating jacket from the power source. The temperature of the sample in the cell should be reduced to below 38 °C (100 °F) before the cell can safely be opened.

L.5.2 The pressurized cell assembly shall be cooled in the heating jacket. When these tests are run with sufficient frequency to justify it, a cooling stand, station or bath can be provided to expedite the cooling process. A cell-handling tool is available which should be used any time a hot cell is to be handled.

CAUTION — Extreme care should be exercised in cooling hot cells.

This procedure, as outlined, makes it difficult to perform more than one test in an 8 h work day with a single PPA. In the interest of improving productivity, users may want to design their own cell-cooling procedures and apparatus. Safety should be the primary consideration in these designs.

L.5.3 Close the valve between the cell and the back-pressure receiver.

L.5.4 Release the pressure on the pump and cell by opening the pump valve, then disconnect the quick coupler between the pump and the nipple adapter on the bottom of the cell.

L.5.5 Release the back-pressure by turning the T-screw on the regulator counterclockwise until it turns freely.

L.5.6 Bleed the pressure from the back-pressure receiver by opening the pressure relief valve on the CO_2 unit. Open the drain valve on the back-pressure receiver and collect the last few drops of filtrate in the graduated cylinder. After removing the locking pin securing it, remove the CO_2 assembly from the top nipple adapter. After removing its locking pin, remove the back-pressure receiver.

L.5.7 The cell may be opened after its contents have been allowed to cool. The cell should be opened only when the user is confident that the contents are no longer under pressure.

L.5.8 Open the filtration valve to relieve any pressure remaining between the cell filter and the back-pressure receiver.

L.5.9 If pressure is suspected to remain in the cell, and the lower end cap does not contain a screen, the following procedure can be used to verify the position of the floating piston:

- remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom;
- if the piston is not at the bottom, there is no pressure; if the piston is at the bottom, there could be pressure remaining in the cell;
- reconnect the hydraulic pump and pump several strokes to move the piston; if the cell is pressurized, it will be obvious from the number of strokes of the piston required to move it and then the force to be applied.

L.5.10 If the indications are that pressure remains in the cell, completely remove the filtration valve assembly from the cell and insert a small drill bit or wire into the cell cap to remove the obstruction. The drill or wire will stop when it contacts the filter disk. Make certain that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

The cell should be opened only when the operator is fully confident that the contents are no longer under pressure.

L.5.11 Raise or remove the cell assembly. If desired, the cell may be raised in the heating jacket either by lifting it by the filter valve assembly or by using the optional cell-handling tool. Attach this tool to the back-pressure inlet nipple just above the filtrate valve where the back-pressure receiver is normally attached. Secure it using the valve stem locking pin. The cell can be supported on the cell support, or lifted out of the heating well and laid on a bench while the cell is being opened.

L.5.12 Remove threaded caps using spanner wrenches. It may be necessary to tap on the wrench to get it started. Opening difficulty is an indication of insufficient lubrication, over-tightening, or insufficient cleaning. It may be necessary to use a suitable holding tool, such as a soft-jaw vice, chain wrench, strap wrench or similar device, to secure the cell while the cap is unscrewed.

L.5.13 Reposition the cell so that the filter end is up and unscrew the top cap.

L.5.14 Remove the filter disk. Use a small knife, small screwdriver or similar thin blade to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh water, or the base oil if the sample is oil-based, then measure and record its thickness and any remarks concerning its composition.

L.5.15 Pour the remaining fluid from the cell and wash the inside of the cell with a suitable solvent. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 150 °C (300 °F) or higher.

If testing was conducted at temperatures above 150 °C (300 °F), the O-rings should be replaced.

L.5.16 Perform the three steps described below to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the bottom end cap using the procedure outlined in L.5.10 and L.5.11, except that the cell position is reversed and the two-pin spanner wrench is used.

b) Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. Note that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all of the O-rings on the piston and the cap.

c) Clean the parts for reuse.

L.6 Calculating — Data reporting

L.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, in millilitres, collected through each of the selected time periods.

L.6.2 Spurt loss

The spurt loss can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume [doubled to correct for filtration area when using 22,6 cm^2 (3,5 in^2) filtration media] is plotted along the y-axis. Alternatively, an approximate value can be calculated using the equations specified in L.6.3.

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with L.4.4.2.

L.6.3 Calculation

Calculate the Permeability Plugging Test volume, spurt loss and static filtration rate using Equations (K.1), (K.2) and (K.3), respectively.

L.6.4 Filter cake reporting

Measure and record the filter cake thickness to the nearest 1,0 mm (1/32 in). Include a description such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgments, they can convey important information.

Annex M
(informative)

Compatibility of elastomeric materials with non-aqueous-based drilling fluids

M.1 Principle

M.1.1 The effects of drilling fluids, particularly non-aqueous-based fluids on elastomeric materials used in a drilling operation, can have a significant impact on the safety, the success and the cost of the operation. Articles prepared with elastomeric materials which may be exposed to such fluids during drilling operations include seals, gaskets, hoses, diaphragms, sleeves, downhole motor stators, etc. Various properties of such articles deteriorate during exposure to the non-aqueous fluids, affecting the performance of the articles.

M.1.2 Measurement/predictability of these effects is fundamental to the selection of appropriate elastomer/drilling fluid combinations for the drilling operation and to the establishment of operational guidelines.

M.1.3 Many test methods exist which evaluate these various effects, and the methods employed vary throughout the drilling industry.

M.1.4 It is the intent of this annex to provide standard methods for testing the effects of non-aqueous-based drilling fluids on elastomers intended for contact with the fluids during drilling operations. Since the relationship between elastomer/fluid screen test conditions and actual service conditions is only approximate, and since test result acceptance criteria vary with the user, no guidelines for test result interpretation are provided.

M.1.5 Non-aqueous-based drilling fluids, as defined in this annex, are fluids prepared with either naturally occurring or refined hydrocarbon oils, synthetic hydrocarbons or other synthetic liquids. These fluids may have aqueous internal phases, however the continuous phase is comprised of the non-aqueous liquid. All test methods described or referenced herein involve measurements of initial properties of a sample of an elastomer, immersion of the elastomer in a fluid at prescribed temperatures for prescribed time periods, and subsequent measurements of the same properties. The methods described yield comparative data on which to base judgment as to expected service quality.

M.1.6 This annex references ISO standards appropriate for reference testing. This annex also references ASTM test methods and standard reference liquids which have been reviewed and found to be appropriate for testing the effects of drilling fluids on elastomeric materials used in drilling operations.

M.1.7 The full step-by-step procedures are not reproduced, but can be obtained from ISO or ASTM.

M.2 Apparatus and reagents

M.2.1 Standard reference elastomers

Elastomer compositions to be used as standard references are described in ISO standards.

a) **SRE NBR 34S**[13] (see ISO 13226:2005, Clause B.2, Table B.1) is an acrylonitrile-butadiene rubber with 34 % (weight) acrylonitrile (see ASTM D3182). It is considered representative of a routinely used, non-extreme service material.

b) **SRE HNBR/1**[13] (see ISO 13226:2005, Clause G.2, Table G.1) is a hydrogenated acrylonitrile-butadiene rubber with 19 % (weight) nitrile groups and less than 0,1 % residual double bonds (see ASTM D3182). It is considered representative of a material required for use in a harsh chemical environment and at high temperature.

M.2.2 Standard reference test liquids

Liquid compositions to be used as standard references are described in ASTM standards.

a) **IRM 903**[14] (see ASTM D471-10, Clause 6) is an industry reference hydrocarbon oil with relatively high aniline point, which produces relatively mild effects on elastomers.

b) **ASTM Service Liquid 101**[15] (see ASTM D471-10, Clause 6) is an industry reference ester oil, di-2-ethyl hexyl sebacate, which is quite aggressive towards elastomers.

M.3 Procedure — Test specimen preparation

M.3.1 Test specimen preparation for all testing is described in ASTM D3182 and ASTM D3183.

M.3.2 Standard test specimen dimensions are specified in ASTM D471-10, 9.1.

M.4 Procedure — Elastomer physical changes after immersion

M.4.1 Principle

M.4.1.1 Test methods are given in ASTM D471-10, Clauses 8, 9, 11 and 15. These measurements are believed to be sufficient for defining the effects of a given fluid on a given elastomer. The immersion temperatures chosen are those routinely used by elastomer manufacturers. Other temperatures may be used as required.

M.4.1.2 Changes in volume, tensile strength, elongation and hardness of elastomeric materials after contact with non-aqueous fluids are common and degrade the performance of elastomeric articles. The changes can be either positive or negative.

M.4.1.3 These methods cover the laboratory determination of such changes in unconfined elastomer specimens after static immersion in a test fluid at specified temperatures, for a specified time period and at ambient pressure. Specified immersion temperatures are 70 °C (158 °F) and 100 °C (212 °F).

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[13] SRE NBR 34S and SRE HNBR/1 are an 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.

[14] IRM 903 is an example of a suitable product available commercially. It may be obtained from Penreco, 4426 East Washington Blvd., Los Angeles, CA 90023-4476, +1-888-227-5448, www.penreco.com. 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.

[15] ASTM Service Liquid 101 is an example of a suitable product available commercially. It may be obtained as Anderol 774 from Robeco International Limited, Rowley House, School Close, Chandlers Ford, Eastleigh, Hampshire, SO53 4BY, England, +44 (0)23 8024 6999, www.robeco.co.uk; or Synthetic Oil A774 from NYE Lubricants, Inc. 10534 Success Lane, Centerville, Ohio 45458, +1-508-996-6721, www.nyelubricants.com. 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.

M.4.1.4 Temperatures are maintained by appropriate means, e.g. water bath, oil bath, hot air oven or ageing block.

M.4.1.5 An immersion time period of 168 h (7 d) is routinely used by elastomer manufacturers and is specified here to reasonably assure equilibrium.

M.4.1.6 Standard reference liquids and standard reference elastomers may be tested along with the operational elastomers and fluids being evaluated to assess relative effects.

M.4.2 Summary of immersion procedure

M.4.2.1 Immersion procedures are stated in ASTM D471-10, Clauses 8 and 9.

M.4.2.2 Pre-immersion measurements of the properties are made on the specimens by the referenced ASTM procedure and recorded.

M.4.2.3 The specimens are then placed in a test tube, or other glass vessel, fitted with a water-cooled reflux condenser, and test fluid is added to completely immerse the specimens. The flow of water through the reflux condenser is started and the temperature of the test fluid is increased to the required test value and maintained at that temperature for the required immersion time.

M.4.2.4 The specimens are then removed from the test fluid, excess fluid is removed from them and post-immersion measurements of the properties are made and recorded. If temperatures and immersion times other than those specified are utilized, record the temperature and time.

M.4.3 Summary of change in volume procedure

M.4.3.1 Change in volume procedure is stated in ASTM D471-10, Clause 11.

M.4.3.2 Three specimens, as identical as possible, are tested. The initial volume of each specimen is measured by water displacement.

M.4.3.3 Water is removed from the specimens and the immersion procedures described in M.4.2 is conducted on each specimen.

M.4.3.4 After immersion, the volume of each specimen is again measured by water displacement.

M.4.3.5 The volume change, expressed as a percentage of the original volume, is calculated for each specimen and an average change is determined and recorded.

NOTE This procedure is only applicable for testing water-insoluble fluids.

M.4.4 Summary of changes in tensile strength, elongation and hardness procedures

M.4.4.1 Changes in tensile strength, elongation and hardness are stated in ASTM D471-10, Clause 15.

M.4.4.2 Original values for tensile strength and elongation are measured in accordance with ASTM D412, using three specimens cut from the same sheet or the article used for the immersion testing, M.4.2. The tensile strength and elongation tests are also run on the samples after the immersion test. The change, expressed as a percentage of the original value, is calculated for each specimen and an average change is determined and recorded.

Since the ultimate elongation measurement is destructive, separate specimens should be used for the original measurement procedure and immersion measurements.

M.4.4.3 The original value of hardness is measured in accordance with ASTM D1415 or ASTM D2240, using three specimens cut from the same sheet or the article used for immersion testing, M.4.2. The hardness is also run on the samples after the immersion test. The change, expressed as a percentage of the original value, is calculated for each specimen and an average change is determined and recorded.

Since the hardness measurement alters the properties of the elastomer, separate specimens shall be used for the original measurement procedure and immersion measurements.

M.5 Procedure — Effect of immersion on a stressed elastomer specimen

M.5.1 In addition to the measurement of changes in volume, tensile strength, elongation and hardness after immersion in a test fluid, a method is specified where the test specimens are stressed (bent) and subjected to the immersion procedures in M.4.

M.5.2 Visual observations of the condition of the specimens are made after immersion and recorded.

M.5.3 Test specimens are bent to 180° and secured in that position with a suitable retainer which shall be inert to the test fluid.

M.5.4 The specimens are subjected to the immersion test procedure in M.4. After immersion, the specimens are removed from the test vessel, excess fluid is removed and visual observations of the specimen's physical condition are made.

M.5.5 Discolouration, excessive swelling, cracking of the stressed area, extent of cracking and other conditions are observed and recorded.

Annex N
(informative)

Sand content procedure for non-aqueous fluids

N.1 Principle

The sand content of drilling fluids is the percentage (volume fraction) of particles of diameter larger than 74 µm. It is measured by a sand-screen test method. In this test, any material retained on the screen is considered sand, as defined by the particle size. It may or may not be chemically sand.

N.2 Apparatus

N.2.1 Sieve, 74 µm (200 mesh) and 63,5 mm (2,5 in) in diameter.

N.2.1 Funnel, to fit screen.

N.2.3 Glass measuring tube, marked for the volume of drilling fluid to be added and graduated from 0 % to 20 % in order to read the percentage of sand directly.

N.3 Procedure

N.3.1 Fill the glass measuring tube with drilling fluid to the “drilling fluid” mark. Add the appropriate non-aqueous base fluid (diesel, mineral or synthetic base fluid) to the next mark. Close the mouth of the tube and shake vigorously.

N.3.2 Pour a small quantity of the base fluid through the screen so that the entire surface is wet by the fluid.

N.3.3 Pour the mixture on the wetted screen. Retain the liquid passing through the screen for proper disposal. Add more base fluid to the tube, shake and again pour through the screen. Repeat until the tube is clean. Wash the solids retained on the screen with the base fluid to free it of any remaining drilling fluid.

NOTE Do not stir or manually force the mixture through the screen. Tapping the side of the screen holder as the drilling fluid is added might help the mixture pass through the screen. If water-wet solids appear on the screen, add several drops of the oil-wetting chemical used in the drilling fluid to smooth out any water-wet solids.

N.3.4 Put the funnel upside down over the top of the screen. Slowly invert the assembly and insert the tip of the funnel into the mouth of the glass measuring tube. Wash the solids into the tube by adding a volume of base fluid or spraying them with base fluid from a wash bottle. Allow the solids to settle. From graduations on the tube, read the volume percentage of sand.

N.4 Calculation

Report the sand size content of the drilling fluid as a percentage (volume fraction). Report the source of the drilling fluid sample, e.g. above the shaker, suction pit, etc. Coarse solids other than sand will be retained on the screen (e.g. weight material, lost circulation material, calcium carbonate, shale particles, etc.) and will be reported as sand.

Annex O
(informative)

Identification and monitoring of weight-material sag

O.1 Principle

O.1.1 The objective of this annex is to provide an understanding of, and guidance on, weight-material sag, its impact on drilling operations, and methods to monitor sag at the well site.

O.1.2 The target audience for this annex covers both office and well-site engineers.

O.1.3 Weight-material sag is recognized by a significant (greater than 0,5 lb/gal) drilling fluid density variation, lighter followed by heavier than the nominal fluid density, measured when circulating bottoms up, usually where a weighted fluid has remained uncirculated for a period of time in a directional well. It is recognized that sag is both a static and a dynamic phenomenon and has the potential to occur when the drilling fluid is in motion.

NOTE Pounds per gallon is the common notation for pounds-mass per gallon and is used throughout this annex.

O.1.4 The definition of weight material extends to any particulate material which is added intentionally to the drilling fluid to increase the fluid density. Common example materials include barium sulphate, haematite and calcium carbonate.

O.1.5 Weight-material sag is applicable to the complex wells that are now commonly drilled. These include: high-temperature, high-pressure (HTHP), extended-reach drilling (ERD), and high-angle wells. Weight-material sag is important in the following circumstances:

a) calculating downhole hydrostatic pressure;

b) controlling downhole pressures to ensure that the well is stable with respect to influxes and losses;

c) induced wellbore instability due to reduction in effective fluid density;

d) stuck drillpipe;

e) difficulty running casing and logs;

f) estimating hole-cleaning efficiency.

O.2 Guidelines on using sag monitoring methods

O.2.1 Experience shows that weight-material sag cannot be totally eliminated. However, it can be managed within levels that do not adversely affect the drilling operation. Weight-material sag is not entirely a drilling fluid-related problem. The occurrence of sag will also be governed by the wellbore geometry, wellbore environment and the operating conditions. Certain conditions in the drilling operation are conducive to creating dynamic sag. The potential for dynamic sag is promoted by an eccentric pipe, such as when sliding in deviated wells, and by low annular velocities, e.g. less than 33 m/min (100 ft/min).

O.2.2 This annex includes example calculations to illustrate how weight-material sag may impact downhole pressures and how such changes can be monitored at the well site. In recognition of the complexity of the problem, it is not possible to offer a single preferred method to monitor weight-material sag. The recommendation is to use a range of techniques in combination to provide a qualitative assessment of sag potential and to assess the benefits of potential remedial actions, e.g. addition of chemicals to the drilling fluid system. This annex provides five different methods to monitor weight-material sag. Four of these methods are based on direct measurement and one method is based on hydraulics calculations. All five methods are suitable for application at the well site.

O.2.3 The methods are as follows:

a) surface monitoring of weight-material sag (Clause O.3);

b) field sag monitoring based on downhole density (Clause O.4);

c) dynamic weight-material sag test — VSST method (Clause O.5);

d) rheological measurements of drilling fluids exhibiting weight-material sag (Clause O.6);

e) field sag monitoring based on critical wall shear stress (Clause O.7).

O.2.4 The choice of monitoring method or methods is an engineering judgment that is linked to the complexity of the well and the probability of weight-material sag occurring in the field. Surface monitoring of mud-weight variation is logged as the *de facto* determination of whether sag is present. Downhole density determination will not always be an option because it relies upon specific downhole tools being run in the drill string. The experimental methods proposed may be used individually or in combination. The modelling method based on critical wall shear stress is relatively simple to employ and only requires simple spreadsheet calculations.

O.2.5 Each method is described in more detail in Clauses O.3 to O.6.

O.2.6 The root causes of weight-material sag are only now being understood. As such, there is still considerable research effort devoted towards developing new methods to monitor and quantify sag. Some of the more recent techniques are referenced in the Bibliography. At a future date, these may become incorporated into an updated annex on weight-material sag monitoring.

O.3 Surface monitoring of weight-material sag

O.3.1 Principle

O.3.1.1 Changes in drilling fluid density measured at the surface are often the first indications that weight-material sag is occurring within the wellbore.

O.3.1.2 All drilling well sites are capable of measuring and monitoring surface drilling fluid density on a regular basis.

O.3.1.3 Fluctuations of surface density have been classified as the *de facto* standard in the definition of weight-material sag. See O.1.3.

O.3.1.4 Variations in drilling fluid density may be identified from the regular surface density determinations which form part of routine operations and are recorded on an ongoing basis.

O.3.1.5 The accepted practice for quantifying weight-material sag from surface density measurement is to capture return fluid samples when tripping-in the hole and stage circulating, or when circulating bottoms-up with the bit at total depth, e.g. prior to tripping-out of the hole.

O.3.2 Apparatus

O.3.2.1 **Any density-measuring instrument** having an accuracy of ±0,01 g/ml or 10 kg/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

A mud balance is the instrument generally used for drilling fluid density measurements. It 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/m^3 (8,33 lb/gal or 62,4 lb/ft^3) 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.

NOTE The preferred method is to use a pressurized mud balance. This is particularly important in situations where the drilling fluid and surface flow system inherently create situations with high air entrainment or where the fluid is gas-cut. See Clause 5 of this part of ISO 10414/API RP 13B-2:2005 for procedures using a pressurized mud balance.

O.3.2.2 **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±0,25 °C (±0,5 °F).

O.3.3 Procedure

O.3.3.1 The procedure uses surface density measurement to infer the density of samples taken from different locations in the annulus corresponding to a series of pre-determined depths or time intervals.

O.3.3.2 Before tripping-in or tripping-out of the hole, identify a series of well depths or time intervals to take fluid measurements. The exact intervals and frequency of sampling should be determined on a well-specific basis and should take account of the well geometry, well conditions and other operating considerations.

O.3.3.3 Measure the density of drilling fluid samples from pre-determined depths or time intervals with a mud balance. Use a pressurized mud balance if appropriate (see O.3.2.1). Samples should be collected immediately after the primary solids control equipment (shakers) based on lagged transit time. The calculation of the lagged depth should take account of non-uniformity in wellbore geometry, e.g. known hole enlargement, and volumetric pump efficiency. These are outlined in O.3.4.

O.3.3.4 If practical, make all surface density determinations at a uniform temperature, e.g. 21 °C (70 °F). Regardless, the density shall be taken together with the recorded temperature. These data can then be used to graphically relate the measured density to a preferred reference temperature. Temperature should be recorded with an accuracy of ±0,5 °C (±1,0 °F).

O.3.4 Calculation

O.3.4.1 Calculate the lagged depth of samples based on annular volume capacity and discharge pump capacity (mechanical efficiency). This calculation should take account of any known changes in annular volume capacity due to geometry changes, including wellbore enlargement.

$$t = \frac{4\,200 V_{\mathrm{A}}}{QE} \tag{O.1}$$

where

E is the pump efficiency, expressed as percentage;

Q is the pump rate, expressed in gallons per minute;

t is the time, expressed in minutes;

V_{A} is the annular volume, expressed in barrels.

O.3.4.2 If appropriate, use a linear plot of density versus temperature to convert all measured data to a single reference temperature. An example of density variation with temperature for an oil-based drilling fluid is given in Figure O.1.

O.3.4.3 Calculate the difference in the maximum fluid density and nominal fluid density by Equation (O.2) which can then be used to calculate the sag register, S, by Equation (O.3).

$$\Delta\rho = \rho_{\text{max}} - \rho_{\text{nom}} \tag{O.2}$$

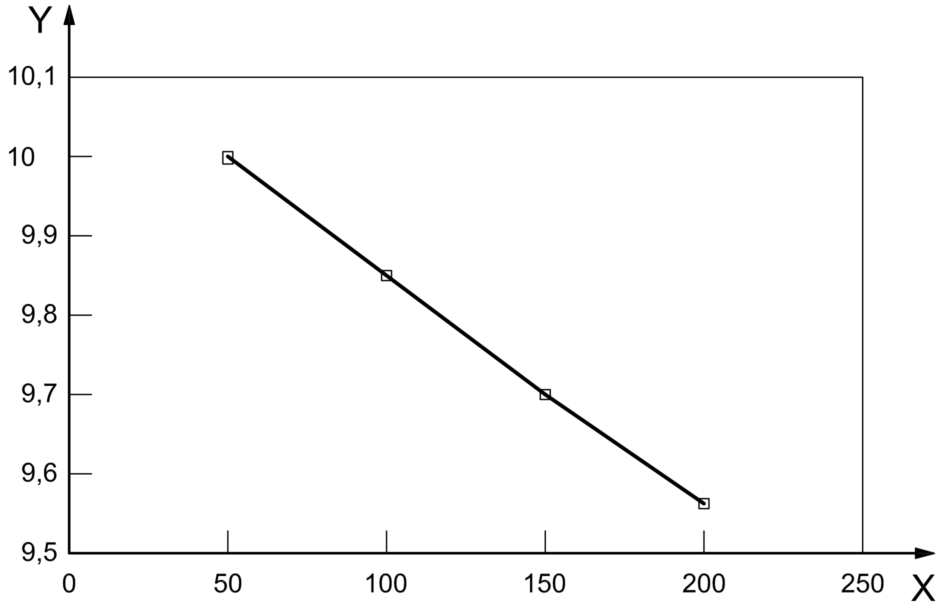
$$S = \exp\left(-10 \times \frac{\Delta\rho}{\rho_{\text{nom}}}\right) \tag{O.3}$$

where

S is the sag register (see Reference [15]);

ρ_{max} is the maximum recorded drilling fluid density, expressed in pounds per gallon;

ρ_{nom} is the nominal drilling fluid density, expressed in pounds per gallon.



Key

X temperature, °F

Y drilling fluid density, lb/gal

Figure O.1 — Example of surface density variation with temperature (oil-based drilling fluid)

O.3.5 Data reporting

O.3.5.1 Record operational conditions of the well on a trip-out sheet similar to the one shown in Table O.1. This should also include the raw data measurements of drilling fluid density versus pump strokes and time.

Table O.1 — Sample trip-out sag reporting sheet

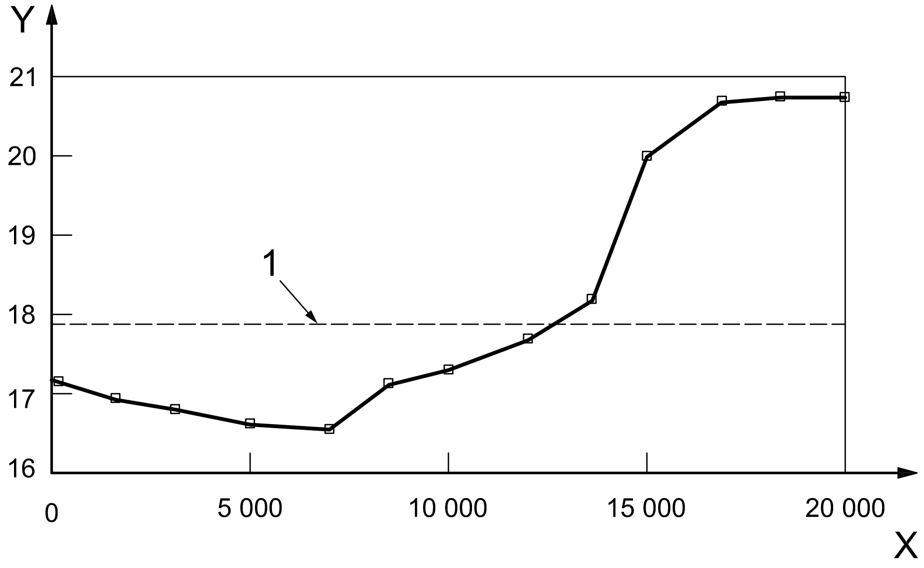
DRILLING FLUID BOTTOMS-UP TRIP REPORT

PAGE ____ OF ____

Operator		Date		
Well name and number		Depth/TVD	/	ft
Field/block number		Hole inclination		°
Location		Hole size		in
State/province/country		Time out of hole		n
Engineer		Flow rate		gal/min
Comments, activities		Nominal fluid density at ______ °F		lb/gal
		Max. fluid density at ________ °F		lb/gal
		Min. fluid density at _________ °F		lb/gal
		Drilling flow rate prior to tripping		gal/min
		VSST sag		lb/gal
		Sag register (R)		
		VSST PBU		%

BOTTOMS-UP TRIP DATA								DRILLING FLUID RHEOLOGY 120 °F			
Time	Pump strokes	Lagged depth ft	Fluid density, lb/gal		Flowline temp. °F	Funnel viscosity s	Gas units				
			Rig bal.	Press-urized							
								R_{602}		PV	
								R_{300}		YP	
								R_{200}		LSYP	
								R_{100}		$R_{3\text{-}10\ s}$	
								R_6		$R_{3\text{-}10\ min}$	
								R_3		$R_{3\text{-}30\ min}$	
								COMMENTS			

O.3.5.2 Plot the surface density profile as a function of logged depth. An example is given in Figure O.2.



Key

X measured depth,ft

Y lagged fluid density, lb/gal

1 nominal drilling fluid density, 17,9 lb/gal

Figure O.2 — Example surface profile based on bottoms-up data

O.4 Field sag monitoring based on downhole density

O.4.1 Principle

O.4.1.1 The occurrence of weight-material sag in a wellbore can be identified by a variety of downhole measurements which include density measurements and pressure measurements converted to equivalent circulating density (ECD). However, interpretation of changes in downhole density is not straightforward, since other things that cause changes in downhole pressure can affect the density without necessarily having an effect on the potential for weight-material sag.

O.4.1.2 These factors can include drillpipe rotation, changes in pump rate, and fluid density changes resulting from changes in temperature and pressure. Correct interpretation of downhole events is important. These areas are discussed in this clause.

O.4.2 Measurements of downhole pressure

O.4.2.1 Use of downhole pressure tools is common in today's drilling arenas. These annular pressure-while-drilling tools, usually placed near the bit, have sensors that read the pressure exerted by the drilling fluid column at that particular point downhole where the tool is placed (see References [16] and [17]). At the surface, the measurements received from downhole are then converted to density using the standard Equation (O.4).

$$\rho = \frac{P}{0{,}052 \times D_{\text{TVD}}} \tag{O.4}$$

where

ρ is the drilling fluid density, expressed in pounds per gallon;

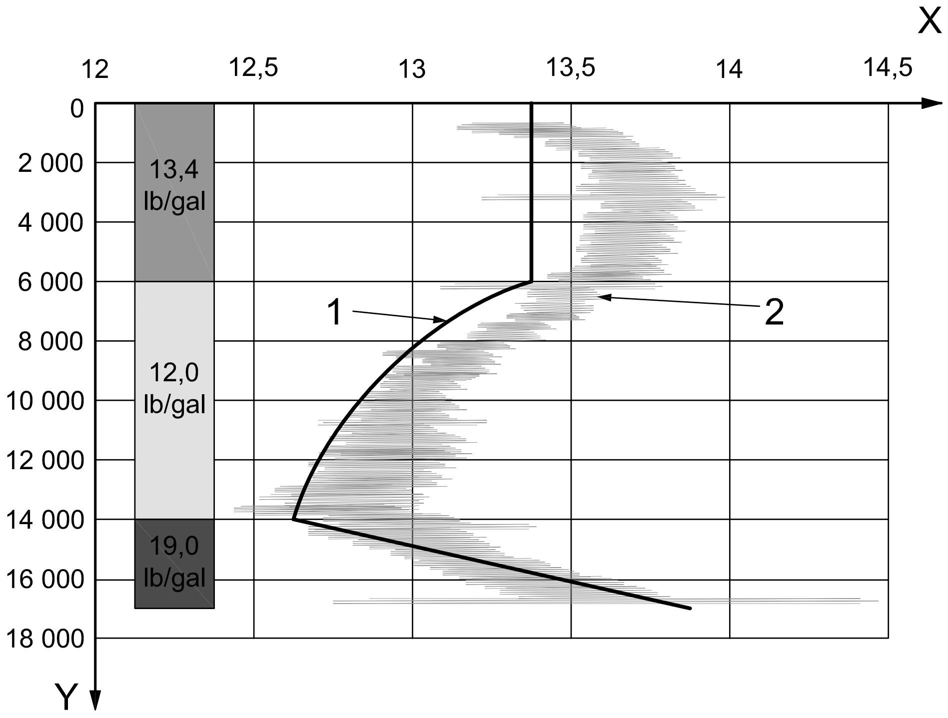
P is the measured pressure, expressed in pounds-gauge per square inch;

D_{TVD} is the true vertical depth, expressed in feet.

O.4.2.2 The density calculated from downhole pressure measurements while the drilling fluid is being circulated is called Equivalent Circulating Density (ECD) while that measured with a static drilling fluid is called Equivalent Static Density (ESD). ECD will always be greater than ESD for a typical drilling case.

O.4.3 Weight-material signatures using downhole pressure tools

O.4.3.1 Weight-material incidents can be seen and measured by downhole pressure tools while the drilling fluid is either static or dynamic. Evidence of weight-material sag while the fluid was static can be seen in Figure O.3, where the annular pressure-while-drilling sensors were being recorded while the drill string was being run in the hole.



Key

X equivalent fluid density, lb/gal

Y depth TVD, ft

1 simulated data

2 pressure data for trip in hole

NOTE See Reference [18].

Figure O.3 — Downhole density changes measured while running the drill string in the hole

O.4.3.2 With an ESD of 13,4 lb/gal, the downhole tool began to measure declining density starting around 6 000 ft TVD. The density continued to decline until around 14 000 ft TVD. The measured density began to increase above the base density of 13,4 lb/gal, eventually reaching a maximum of approximately 19,0 lb/gal. This figure shows the signature of a weight-material sag occurrence below 6 000 ft TVD:

a) declining density followed by increasing density;

b) a density swing between 19,0 lb/gal and 13,4 lb/gal, which gives a theoretical $\Delta\rho$ of 5,6 lb/gal (19,0 lb/gal – 13,4 lb/gal);

c) a magnitude of sag of 5,6 lb/gal, which is greater than 0,5 lb/gal by the definition of weight-material sag.

Therefore, the event qualified as a weight-material sag event.

O.4.3.3 In another case, while running in the hole, the occurrence of weight-material sag was picked up with the drilling bit at several depths, as shown in Figure O.4. While running in the hole, repeated fluctuations in the ESD that could not be explained (characteristic of weight-material sag occurrence) are seen at 1 500 ft, 4 000 ft, and 15 000 ft. At 15 000 ft, the pumps were turned on. The low measured density of nearly 10 lb/gal quickly increased to 12,3 lb/gal, as the drill string rotation picked up weight-material particles lying on the low side of the hole, and incorporated them into the main flow stream. Later, when the bit reached 21 200 ft, the pumps were turned on again and the ECD quickly increased from 11,2 lb/gal to nearly 14,2 lb/gal. As with the previous example, a swing in drilling fluid density of 3 lb/gal qualified this as a weight-material sag event.

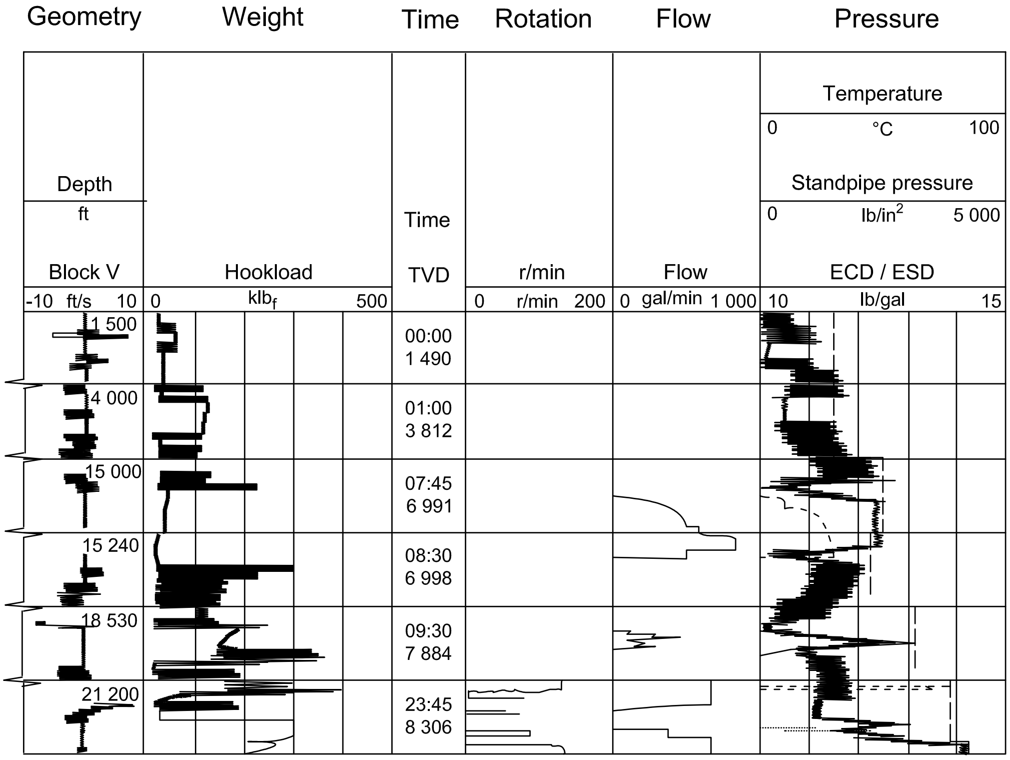


Figure O.4 — Weight-material sag occurrence during dynamic conditions

O.4.3.4 Left untreated, the circulating system can exhibit lighter and heavier densities for many circulations until the system eventually reaches equilibrium. However, these fluctuations can lead to well control difficulties, so it is important that weight-material sag occurrences be quickly corrected in the field.

O.4.4 Downhole pressure changes that are not caused by weight-material sag

O.4.4.1 Field interpretation

In the field, changes in downhole pressure can be caused by other factors which are unrelated to weight-material sag. Changes in pump rate, drillpipe rotational speed and fluid density (especially with invert emulsion drilling fluids) may produce changes in downhole density. It is important to recognize that density changes due to these factors are not events related to weight-material sag.

O.4.4.2 Changes in pump rate

O.4.4.2.1 When pump rates are increased or decreased, the friction at the wellbore wall is increased or decreased accordingly. Downhole pressure tools will measure these changes in annular pressure which may be converted to ECD.

O.4.4.2.2 Changes in ECD caused by changes in the pump rate can be predicted using drilling fluid hydraulic programs, which can verify whether the measured downhole change in pressure is expected or not.

O.4.4.3 Changes in downhole density

O.4.4.3.1 Some drilling fluids, particularly invert emulsions and those formulated with oils, exhibit changes in density as a function of temperature and pressure.

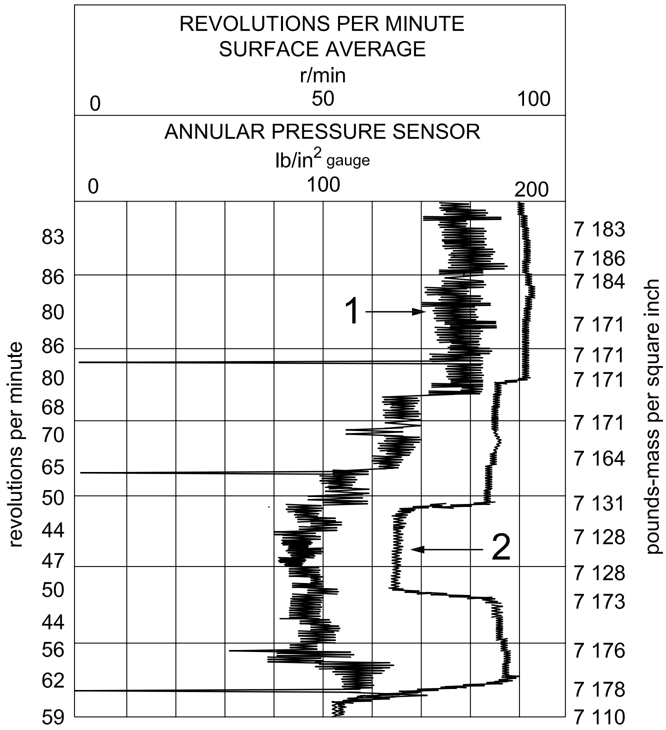
O.4.4.3.2 This phenomenon is purely a function of the fluid's compressibility and thermal expansion. These properties can be characterized using coefficients derived from laboratory PVT data.

O.4.4.3.3 The current API RP 13D contains procedures and coefficients needed to predict the density of drilling fluids as a function of temperature and pressure.

O.4.4.4 Changes in drillpipe rotational speed

O.4.4.4.1 Changes in drillpipe rotational speed can also change downhole pressure measurements.

O.4.4.4.2 Figure O.5 displays the changes in downhole pressure produced by fluctuations in drillpipe rotational speed that were obtained from downhole pressure tools.



Key

1 rotary speed, r/min

2 downhole pressure, lb/in^2

Figure O.5 — Effect of changes in drillpipe rotational speed on downhole pressure

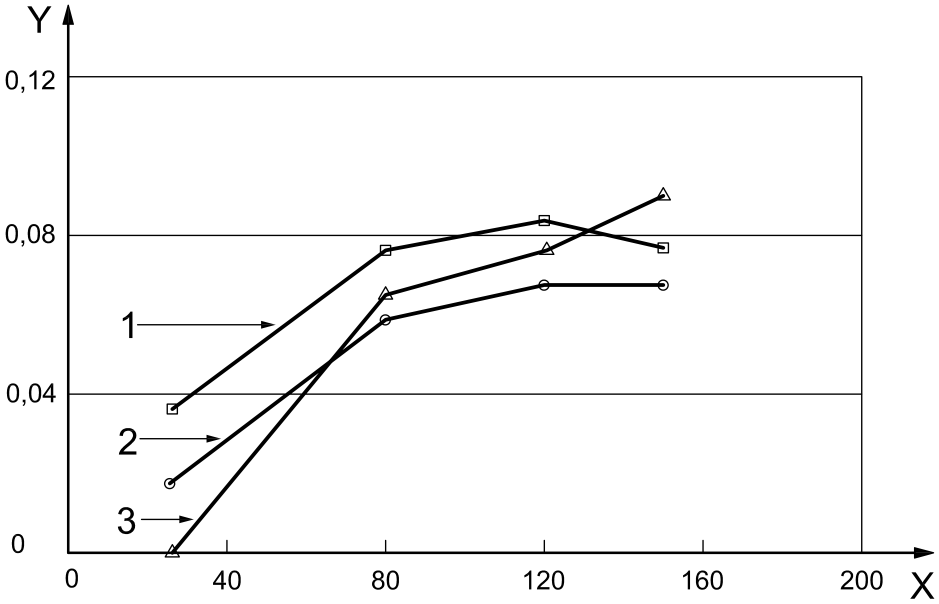
O.4.4.4.3 The coupling of tangential velocities produced by the rotating drill string with the axial velocities produced by the pump rate results in helical flow and higher downhole pressures. These are commonly seen with downhole pressure tools. These increases in downhole pressure have been measured in field experiments (see References [19], [20] and [21]). Published data show these increases commonly range between 0,05 lb/gal and 0,3 lb/gal using drill string rotation speeds of 50 r/min and 200 r/min.

a) For a particular drilling project, changes in downhole ECD produced solely by drill string rotation can be measured in the field. These measurements are often called "fingerprinting." It is generally best to conduct these tests inside newly cemented casing before drilling out. This eliminates the presence of drilled cuttings in the annulus as a contributor to any density changes. A range of pump rates and drill string rotational speeds can be selected; these values should incorporate expected minimum and maximum levels to be used while drilling. An example fingerprinting matrix that could be used is shown in Table O.2.

Table O.2 — Matrix for fingerprinting drill string rotation effects on downhole density

	Minimum pump rate 1	**Intermediate pump rate** 2	**Maximum pump rate** 3
r/min speed 1	X_{11}	X_{21}	X_{31}
r/min speed 2	X_{12}	X_{22}	X_{32}
r/min speed 3	X_{13}	X_{23}	X_{33}

b) Results from a fingerprinting exercise in the field are shown in Figure O.6. These may then be used to interpolate predicted increases in downhole density with varying pump rate and drill string rotation speed while drilling ahead in the open hole interval. It is important to circulate the system at a high annular velocity (225 ft/min to 250 ft/min if possible) before taking measurements to remove gelled fluid from the annulus.



Key

X drilpipe rotation speed, r/min

Y measured increase in ECD, lb/gal

1 fingerprint at 1,000 gal/min

2 fingerprint at 900 gal/min

3 fingerprint at 1,100 gal/min

NOTE See Reference [21].

Figure O.6 — North Sea fingerprinting for three flow rates and four drill string rotation speeds

c) Pressure changes caused by drill string rotation can be mathematically modelled. A complex mathematical model for helical flow has been constructed to predict the increase in downhole pressure produced by rotation speeds commonly used in the field (see Reference [21]). In the referenced work, the tangential velocities produced by drill string rotation are coupled with the axial velocities to generate the fluid helical velocities. The velocities near the conduit walls are then used to determine the predicted shear rates at the wall, which in turn are used to calculate fluid pressure drop.

d) Because these calculations are complex and cannot be worked with simple equations, a different approach has been taken to derive a simple heuristic equation or set of equations that can provide a usable solution (see Reference [22]). The derived equation is comprised of two principal variables: the wellbore geometry diameter ratio and the drill string rotational speed (r/min). These may be used to calculate the anticipated pressure increase in a particular interval. In this work, the general equation is given as Equation (O.5).

$$\Delta P = \left(-1{,}079\,2 \frac{d}{D} \right) + \left[\left(17{,}982 \frac{d}{D} \right)^2 \left(0{,}000\,01 \times L \right)\, \eta \right] \qquad (O.5)$$

where

$\Delta\rho$ is the anticipated pressure increase, expressed in pounds-gauge per square inch;

d is the inner pipe diameter, expressed in inches;

D is the outer pipe diameter or inner diameter of open hole, expressed in inches;

L is the length of the hydraulic section, expressed in feet;

η is the drillpipe rotation, expressed in revolutions per minute.

e) The increase in ECD is calculated as:

$$\Delta\rho_{\text{ECD-rot}} = \frac{\Delta P}{0{,}052 \times D_{\text{TVD}}} \tag{O.6}$$

O.4.5 Theoretical downhole density excluding effects in O.4.4

O.4.5.1 Theoretical downhole densities can be calculated for both static and dynamic cases. In the static case, the downhole density should be equivalent to the ESD. Any densities lighter or heavier than the ESD ± 0,5 lb/gal may be considered a potential weight-material sag event. This excludes parts of the annulus where a weighted pill has been pumped before pulling out of the hole. In this case, a higher density in part of the annulus is planned and deliberate.

O.4.5.2 In dynamic cases, the downhole density while rotating the drill string should include:

a) $\rho_{\text{ECD-hyd}}$ which is comprised of the ESD, the circulating pressure drop and the extra density effects of drilled cuttings in the annulus predicted from hole cleaning modelling;

b) $\Delta\rho_{\text{ECD-rot}}$ determined using Equation (O.6) or interpolated as discussed in O.4.4.4.3.

O.4.5.3 The total predicted ECD of the drilling fluid in the annulus while circulating should be:

$$\rho_{\text{ECD-tot}} = \rho_{\text{ECD-hyd}} + \Delta\rho_{\text{ECD-rot}} \tag{O.7}$$

where

$\rho_{\text{ECD-hyd}}$ is the pressure drop and extra density effects of drilled cuttings;

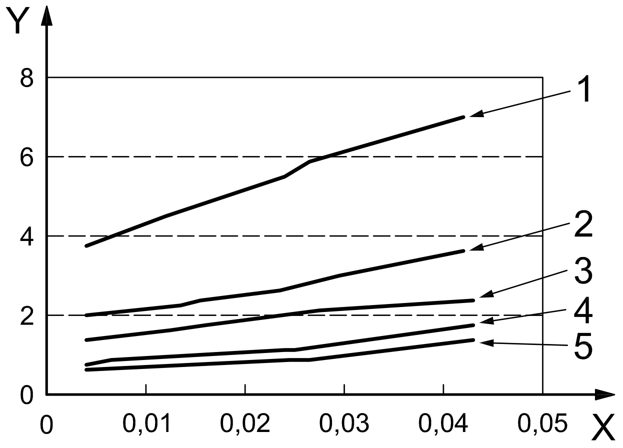
$\rho_{\text{ECD-rot}}$ is the change in pressure due to rotation.

O.4.5.4 If ρ_{ECD} in the wellbore, measured using downhole pressure tools, is greater or less than the $\rho_{\text{ECD-tot}}$ ± 0,5 lb/gal, then there is a potential weight-material sag event and further investigation is needed.

O.4.6 Weight-material sag events caused by drillpipe rotation only

O.4.6.1 Some modelling of weight-material sag potential has been performed on the effects of rotating a drill string slowly in a static fluid (see Reference [22]). An example of the need for this modelling is the field case where, while waiting on weather, the driller rotates the drill string at a slow rate (between 5 r/min and 25 r/min) in order to eliminate a potential case of differential sticking.

O.4.6.2 In the modelling work, it was shown that, at slow drill string rotation speeds in static fluids, there exists a potential weight-material sag occurrence. Fluid viscosities are lowest close to the rotating drill string, as shown in Figure O.7.



Key

X gap, m

Y viscosity, Pa·s

1 5 r/min

2 10 r/min

3 15 r/min

4 20 r/min

5 25 r/min

NOTE See Reference [23].

Figure O.7 — Distribution of fluid viscosity across the annular gap caused by drill string rotation in a static drilling fluid

O.4.6.3 Such reductions in fluid viscosity in a fluid already exhibiting low viscosity could hasten a weight-material sag event. The drop in fluid viscosities near the rotating drill string wall has a greater consequence for eccentric cases than for concentric cases.

O.5 Dynamic weight-material sag test — VSST method

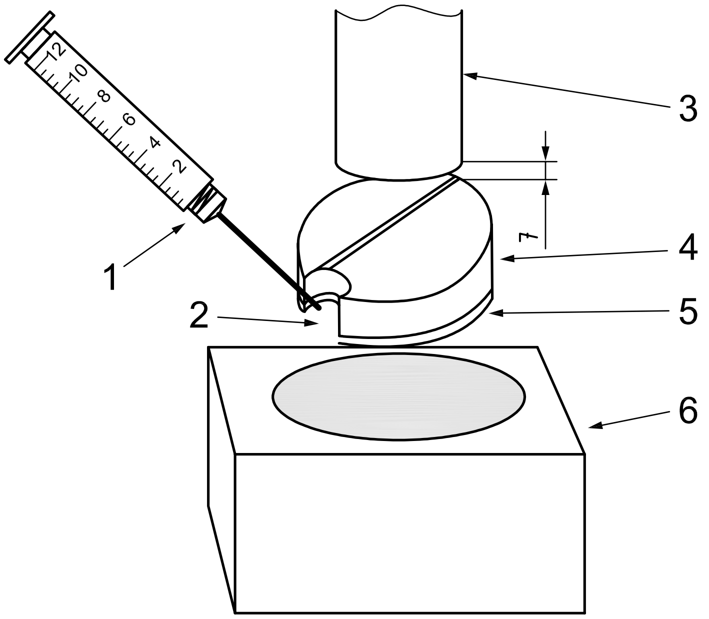
O.5.1 Principle

O.5.1.1 The Viscometer Sag Shoe Test (VSST) is a well site and laboratory test that measures weight-material sag tendencies of field and lab-prepared drilling fluids under dynamic conditions.

O.5.1.2 The VSST provides an intrinsic fluid property without regard to the conditions under which the fluid has been or will be used. As such, results shall be combined with operational factors to correlate with sag experienced in the field. (This is analogous to measuring a yield point, but still having to combine it with operational factors in order to correlate with pressure loss.)

O.5.1.3 The VSST designation is derived from the rotational viscometer used as a mixer and the thermoplastic insert (Sag Shoe) designed to concentrate sagged weight material in the bottom of a viscometer thermocup. Figure O.8 shows the basic equipment needed to determine VSST.

Dimensions in millimetres



Key

1	syringe
2	collection well
3	viscometer rotor
4	VSST Sag Shoe
5	metal-backing plate
6	thermocup

Figure O.8 — Key equipment for VSST method

O.5.1.4 Sag tendency is determined by the density increase of samples extracted from the collection well over a 30 min period at a standard temperature and under a consistent rate of shear.

O.5.1.5 Weight-material bed pickup can be run as an optional measurement to characterize bed removal by higher shear levels. Results can be used to suggest opportunities for bed removal in the field prior to tripping out of the hole.

O.5.2 Apparatus

O.5.2.1 **Direct-indicating viscometer**, powered by an electric motor; see 7.3.1.1 for dimensions.

O.5.2.2 **Stopwatch** or **timer**, accurate to ±1 s.

O.5.2.3 **Thermostatically controlled viscometer cup**.

NOTE The VSST Sag Shoe is intended to fit inside the thermocup without being too tight.

O.5.2.4 **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±1 °C (±2 °F).

O.5.2.5 **Luer syringe**, 10 ml, two-piece, solvent-resistant, Norm-Jet® from HSW GmBH or equivalent.[16]

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[16] Norm-Jet® 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.

O.5.2.6 **Pipetting needle**, blunt-end, 6 in, 14-gauge needle with Luer connection.

O.5.2.7 **Balance**, digital, with 0,01 g resolution. A triple-beam balance may be used in the field where excessive vibration is expected.

O.5.2.8 **Pycnometer** or **retort cup**, 10 ml.

NOTE The pycnometer equipment is not required if the test is run at the well site or if the procedure includes the pickup test described in O.5.3.3.

O.5.2.9 **Spatula**, 6 in or similar.

O.5.2.10 **Distilled water**.

O.5.2.11 **Multimixer**, capable of operating at 11 500 r/min ± 300 r/min under load, with a single corrugated impeller of approximately 25,4 mm (1 in) in diameter.

O.5.2.12 **Container**, for mixing, 180 mm deep (7,125 in), $d = 97$ mm (3,75 in) at top and 70 mm (2,75 in) at bottom.[17)]

O.5.2.13 **VSST Sag Shoe**, shaped, cylindrical disk manufactured from Delrin®[18)] or equivalent thermoplastic material, maximum diameter of 57 mm ± 0,38 mm (2,35 ± 0,015 in) and length of 27,7 mm (1,09 in). The well is a notch cut through the thermoplastic disk and metal-backing plate that connects with the thermocup bottom. The metal plate is used solely to increase the overall mass of the Sag Shoe so that it does not float when used with very dense fluids.

O.5.3 Procedure

O.5.3.1 Equipment setup

O.5.3.1.1 Insert the Sag Shoe into the thermocup and place both on the viscometer plate.

O.5.3.1.2 Raise the plate until the top of the Sag Shoe touches the bottom of the viscometer sleeve, and mark the support leg at the upper edge of the locking mechanism.

O.5.3.1.3 Lower the plate and thermocup to the base, and mark the support leg 7 mm (0,25 in) below the first mark.

O.5.3.1.4 Calibrate the syringe with blunt-end pipetting needle attached, using distilled water and digital balance. Expel as much water as possible from the syringe barrel and wipe exterior dry.

NOTE Calibrate pycnometer or retort cup with distilled water if it will be used for density measurements.

O.5.3.2 Sag measurement

O.5.3.2.1 Insert the Sag Shoe into the thermocup with the collection well positioned for easy access by the syringe, e.g. 60° to 90° either side of the viscometer centreline.

O.5.3.2.2 Pre-heat the thermocup with Sag Shoe to 49 °C (120 °F).

[17)] Hamilton Beach®Mixer Cup No. M110-D is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of this product.

[18)] The VSST Sag Shoe manufactured from Delrin® is an example of a suitable product available commercially. It may be obtained from several oilfield testing equipment suppliers or licensed from M-I SWACO, 5950 N. Course Drive, Houston, TX 77079, USA. 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.

O.5.3.2.3 Collect a 350 ml fluid sample in a container, mix appropriately, and pour approximately 140 ml into the thermocup. Most consistent results are obtained if the starting fluid temperature is close to 49 °C (120 °F) to minimize sag during heating. Heat the drilling fluid to 49 °C (120 °F) while stirring at 600 r/min.

O.5.3.2.4 Position and lock the upper edge of the viscometer locking mechanism to coincide with the lower mark on the support leg. The top of the Sag Shoe should be 7 mm (0,25 in) below the viscometer sleeve.

O.5.3.2.5 Set the viscometer at 100 r/min and start the 30 min timer.

O.5.3.2.6 Using the syringe with blunt-end pipetting needle attached and cleared of air, draw slightly over 10 ml from the drilling fluid remaining in the container. Carefully clear the syringe and pipetting needle of residual air and push the plunger to the 10 ml calibration mark. Wipe the pipetting needle and syringe surfaces until clean and dry.

O.5.3.2.7 Weigh the fluid-filled syringe and record the mass as m_{F1}, expressed in grams.

NOTE When using the pycnometer or retort cup, transfer the fluid from the syringe, weigh and record total mass as m_{F1}, expressed in grams.

O.5.3.2.8 Stop viscometer rotation at the end of the 30 min test period.

O.5.3.2.9 Repeat O.5.3.2.6, this time taking the sample from the collection well of the Sag Shoe. Use the pipetting needle tip to find the collection well.

O.5.3.2.10 Weigh the fluid-filled syringe and record the total mass as m_{F2}, expressed in grams.

NOTE When using the pycnometer or retort cup, transfer the fluid from the syringe, weigh and record total mass as m_{F2}, expressed in grams.

O.5.3.3 Bed pickup measurement (m_{F3}) (optional)

O.5.3.3.1 Gently return the 10 ml test sample from the fluid-filled syringe obtained in O.5.3.2.9 to the Sag Shoe collection well.

O.5.3.3.2 Run the viscometer at 600 r/min for 20 min.

O.5.3.3.3 Collect the sample the from Sag Shoe collection well as in O.5.3.2.9. Weigh the fluid-filled syringe and record total mass as m_{F3}, expressed in grams.

O.5.4 Calculation

O.5.4.1 Calculate the B_{VSST} using Equation (O.8):

$$B_{\mathrm{VSST}} = 0{,}834\ \left(m_{\mathrm{F2}} - m_{\mathrm{F1}}\right) \tag{O.8}$$

where

B_{VSST} is the amount of weight-material sag, expressed in pounds-mass per gallon;

m_{F1} is the mass of drilling fluid following shear at 100 r/min, expressed in grams;

m_{F2} is the mass of drilling fluid taken from the Sag Shoe following shear at 100 r/min, expressed in grams.

Report B_{VSST} in pounds-mass per gallon.

O.5.4.2 Calculate optional R_{BPU} using Equation (O.9):

$$R_{\mathrm{BPU}} = \frac{83{,}4\ \left(m_{\mathrm{F2}} - m_{\mathrm{F3}}\right)}{B_{\mathrm{VSST}}} \tag{O.9}$$

where

R_{BPU} is the calculated bed pickup measurement ratio, expressed as a percentage;

B_{VSST} is the amount of weight-material sag, expressed in pounds-mass per gallon;

m_{F2} is the mass of drilling fluid taken from the Sag Shoe following shear at 100 r/min, expressed in grams;

m_{F3} is the mass of drilling fluid taken from the Sag Shoe following shear at 600 r/min, expressed in grams.

Report R_{BPU} as a percentage (%).

O.6 Rheological measurements of drilling fluids exhibiting weight-material sag

O.6.1 Principle

O.6.1.1 Advanced rheometers are able to measure a wider range of properties than conventional oilfield viscometers and to make these measurements more accurately. This clause relates to the use of such instruments in the measurement and analysis of drilling fluids exhibiting weight-material sag.

O.6.1.2 Drilling fluids that exhibit weight-material sag are, by definition, unstable with respect to time. This makes rheological measurements on them difficult. The magnitude of any measured values can be influenced by sample preparation methods and the shear history of the test fluid.

O.6.1.3 Establishing guidelines for sample preparation and equipment selection will facilitate more meaningful analyses of drilling fluid samples during sag investigations.

O.6.1.4 The method given in this clause will only yield correct information when the annular velocity is 100 ft/min or greater.

O.6.2 Scope

O.6.2.1 Currently, there are no accepted industry methods relating to the equipment or methodology to be used in the measurement of rheological parameters related to weight-material sag in drilling fluids.

O.6.2.2 There is a generally accepted view that viscosity measurements at low (less than 1,0 s^{-1}) shear rates and various rheological parameters derived from oscillatory measurements are useful in quantifying the actual or potential ability of a fluid to exhibit weight-material sag.

O.6.2.3 This clause is applicable to both field drilling fluids and fluids mixed in the laboratory.

O.6.3 Apparatus

O.6.3.1 **Rheometers**: for the purpose of this procedure distinguished from viscometers by their greater degree of accuracy and range of measurement.

a) Typical capabilities found only in rheometers are very low shear rates, oscillatory measurements and the capability to make measurements under elevated temperatures and pressures.

b) Rheometers suitable for detailed investigation of sagging drilling fluids should be capable of the following:

   1) accurate measurement of viscosity at shear rates from approximately 1 000 s^{-1} continuously to 0,01 s^{-1} or below;

   2) oscillatory functionality to allow the calculation of the storage modulus (G') and loss modulus (G");

   3) accurate measurement of stresses below 0,02 $lb_f/100\ ft^2$.

O.6.3.2 **Water bath**, maintained at 60 °C to 70 °C (140 °F to 160 °F).

O.6.3.3 **Mixer**, high speed, e.g. Silverson®LR4 or Waring Blendor®.[19)]

O.6.4 Sample preparation

O.6.4.1 Field samples delivered to a laboratory have been subjected to a wide variety of shear histories. For meaningful laboratory measurements, samples need to be fully reconstituted. If measurements of different fluids are to be compared, it is important to ensure that the fluids are fully reconstituted and re-sheared as close to stable properties as possible. Measurements may then be made at differing times after this condition.

O.6.4.2 Sample mixing should involve the entire content of the storage container. By their nature, fluid samples collected because of sag problems may be expected to have suffered from solids settling during storage. All solids shall be removed from the container prior to mixing.

O.6.4.3 The sample should be mixed at a high shear rate using a suitable high-speed mixer, e.g. Silverson® L4R, Waring® Blendor, or equivalent, for a period of 15 min per 350 ml volume. A volume of 1 400 ml should be sheared for 1 h. Cooling the sample to between 60 °C and 71 °C (140 °F and 160 °F) should include the use of a water bath. Once this temperature is reached, the sample should be covered to prevent vaporization of water. If the sample is too large to mix in a single batch, multiple batches can be mixed as above and then combined. All steps to minimize the time delay between mixing the first and last batches should be taken in such cases.

O.6.4.4 For each rheological measurement, the time between the fluid being sheared as described in O.6.4.3 and the measurement being made should be recorded.

O.6.4.5 Immediately prior to each rheological measurement, the fluid should be sheared in the rheometer at approximately 1 000 s^{-1} for a minimum of 2 min.

O.6.5 Potential rheological tests

O.6.5.1 Through the use of rheometers, a great number and variety of tests may be performed. By exploring several of these tests, a better understanding of the sample fluid may be obtained. Tests should be selected and carried out with this goal in mind.

O.6.5.2 Potential tests of interest for the examination of drilling fluids include:

a) Thixotropy loops (hysteresis) – observing the structure-building tendency of the fluid and how easily that structure is broken by shear;

b) Yield stress measurements – through multiple methods, observing where the fluid actually yields;

c) Controlled rate/stress sweep – producing a flow curve that demonstrates the relation of stress and viscosity to strain rate;

[19)] Silverson® L4R and Waring Blendor® 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.

d) Oscillatory strain/stress sweep – important for determining the linear viscoelastic region (for further oscillatory tests) and for determining the dynamic yield stress;

e) Oscillatory frequency sweep – giving information on structural behaviour of the test fluid over a range of deformation rates, usually performed on a fluid which has been allowed a gel growth period immediately prior to testing;

f) Oscillatory time sweep – observing how the fluid structure grows and is maintained under low-frequency deformations over long periods of time, usually performed on a fluid without allowing gel growth before testing.

O.6.5.3 Unlike the common six-speed field viscometer, which exclusively uses a rotating sleeve about a torsion spring bob (Couette geometry), rheometers have a variety of test geometries from which to choose. These include the Couette geometry, double-gap Couette, multivane spindles, parallel plates, cone and plate, and any of these modified with roughened surfaces for mitigation of wall slip effects which can occur at the very low shear rates. The test geometry should be selected in accordance with the needs of the test to be performed and the fluid being tested.

O.6.5.4 During the preparation of Clause O.6, a series of round-robin tests were performed by several laboratories. These laboratories used different instruments and followed the individual manufacturer's instructions for the instrument. The list of instruments used includes the following but other manufacturers exist:

a) Bohlin® Gemini[20];

b) Brookfield® PVS[20];

c) Grace® M3500a-1[20];

d) OFI® Model 900[20];

e) Physica® MCR101[20];

f) RJF® Viscometer[20].

O.6.6 Data interpretation

O.6.6.1 Various publications (see Reference [24]) have suggested that weight-material sag is closely correlated with the viscosity of the fluid at very low shear rates. The shear rates of interest are typically in the range of 0,1 s^{-1} to 1,0 s^{-1}. Measurement of viscosity at these shear rates is not possible at the rig site using conventional field viscometers.

O.6.6.2 More sophisticated devices have become available since around 2000. Advanced rheometers of the type discussed in this subclause are also fully capable of making these measurements as part of a basic series of tests designed to provide a complete rheological analysis of a given fluid.

O.6.6.3 Viscosity values, which should be adequate to prevent sag of invert emulsion drilling fluids under dynamic field conditions, have been proposed (see Reference [25]), and a typical graph is shown in Figure O.9. In the graph, the solid parallel lines represent the upper and lower bounds of acceptable viscosity, i.e. viscous enough to prevent dynamic weight-material sag under typical drilling conditions, but not so viscous as to cause other drilling-related problems. Note that the viscosity and shear rate are based on the nominal shear rate calculated for Newtonian fluids. This is consistent with previous publications on this technique. However, the variations arising from the non-Newtonian behaviour of typical invert drilling fluids will result in relatively small deviations from these nominal values.

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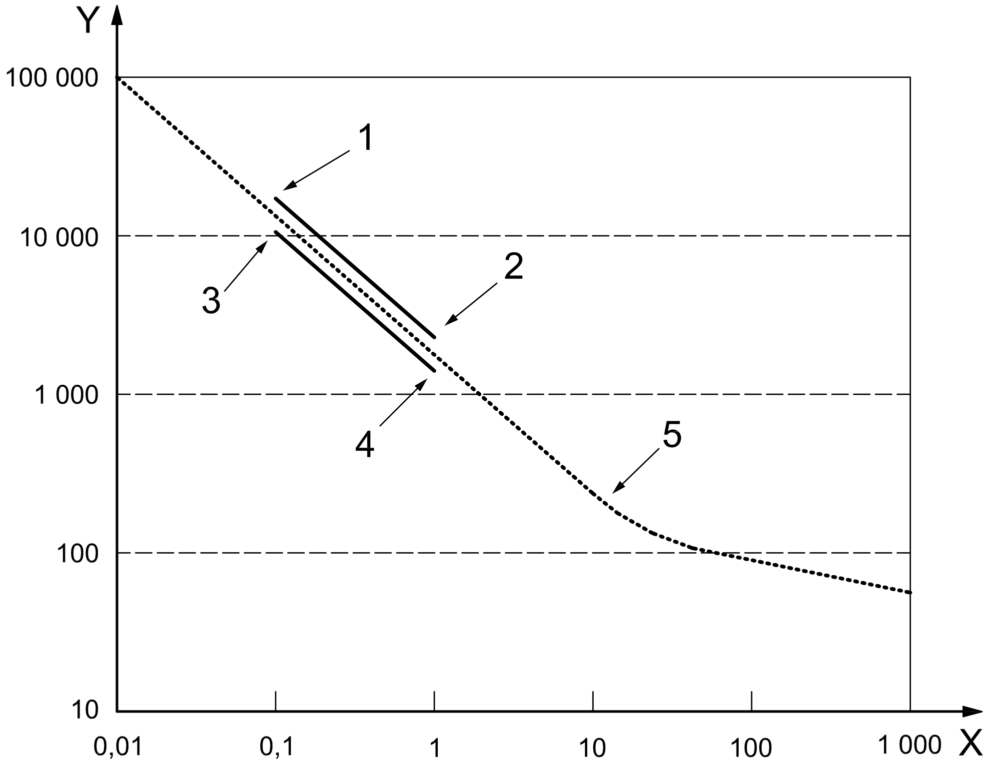
[20] Bohlin® Gemini, Brookfield® PVS, Grace® M3500a-1, OFI Model 900, Physica® MCR101, and RJF® Viscometer are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.



Key

X shear rate, s^{-1}

Y viscosity, cP

1 20 000 cP

2 2 500 cP

3 12 000 cP

4 1 500 cP

5 viscosity curve for a drilling fluid with minimal expected sag tendency

NOTE See Reference [24].

Figure O.9 — Published “Sag Window” for drilling fluids versus shear rate

O.6.6.4 Interpretation of data from rheological testing should be made in the context of the specific fluid being tested. It is easy to generalize from the rheological behaviour of a particular fluid system and attempt application to other systems.

O.6.6.5 If the basic characteristics of the system differ, i.e. different weight materials and oil-to-water ratios, different viscosifier types, significant changes in internal phase composition, and significantly different emulsifier chemistry, the conclusions of one system may not apply to the other. The rheological testing and evaluation of each fluid should be taken with knowledge of the physical characteristics of that fluid. Likewise, all of the rheological testing performed should be considered when drawing conclusions as to a fluid performance.

O.6.6.6 It is often beneficial to observe trends in changes of rheological characteristics of a drilling fluid as small changes (treatments) are made to the system. Under such conditions, the effects of such treatments should be monitored. Specifically, note should be taken of how viscosity shifts with changes in component concentrations. Similarly, note should be made of the changes in G', G" and loss tangent affected by changes in components. One should specifically look for improved/optimal performance in properties, e.g. maximal structure without extreme viscosity or raising ECD issues, based on component changes.

O.7 Field sag monitoring based on critical wall shear stress

O.7.1 Principle

Use of hydraulic modelling to predict the onset of weight-material sag under dynamic conditions has been presented (see Reference [26]). In this prediction method, it is assumed that, given sufficient shear stress in the drilling fluid at the wall on the low side of the deviated wellbore, barite bed formation will not begin to occur. If the moving fluid does not have sufficient shear stress, then accumulation of barite particles will begin to occur (see Reference [25]).

O.7.2 Predictive model

O.7.2.1 Obtain drilling fluid rheological properties from viscometer data, from HTTP viscometers, or from predicted downhole data.

O.7.2.2 Calculate the fluid Herschel-Bulkley rheological parameters through a mathematical regression analysis as outlined in API RP 13D:2010, 4.9.4 and 5.2.3. The use of a computer program or spreadsheet is recommended to perform this complex data analysis.

O.7.2.3 In the hydraulic calculations, use the geometry outer and inner diameters for the particular interval where weight-material sag occurrence is suspected. Set the inner pipe eccentricity to a high value ($\varepsilon = 0{,}7$ is recommended).

O.7.2.4 Calculate the minimum pressure drop in this geometry to shear the annulus across the narrow gap.

$$\frac{\Delta P}{\Delta L_\mathrm{A}} = \frac{2\tau_\mathrm{Y}}{L_\mathrm{A}} \tag{O.10}$$

where

$\frac{\Delta P}{\Delta L_\mathrm{A}}$ is the pressure gradient, expressed in pounds-gauge per square inch per foot;

τ_Y is the drilling fluid yield stress, expressed in pounds-force per hundred square feet;

L_A is the length, expressed in feet.

O.7.2.5 Using iterative techniques, find the circulation rate required to provide this pressure drop in the eccentric annulus. Add a 10 % circulation rate to this value to ensure that the value obtained is above this minimum circulation rate.

O.7.2.6 From the pressure-drop equations given in API RP 13D, obtain the predicted fluid velocity profiles at a slight distance from the wall using different circulation rates (ensure that flow is laminar); see API RP 13D:2010, Clause 4 and 7.4. By definition, the fluid velocity at the wall should be zero. Also calculate the average annular velocity, v_a, for each case.

O.7.2.7 From the velocity values near the wall, calculate the corresponding fluid shear rates, γ_i, and shear stresses, τ_i.

$$\gamma_\mathrm{i} = \frac{\Delta v_\mathrm{a}}{5\, d_1} \tag{O.11}$$

where

γ_i is the fluid shear rate, expressed in reciprocal seconds;

Δv_a is the change in annular velocity, expressed in feet per minute;

d_1 is the distance from the outer wall, expressed in inches.

$$\tau_W = \tau_Y + k_C \gamma_i \tag{O.12}$$

where

τ_W is the 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;

k_C is the consistency factor, expressed in pounds-force per second;

γ_i is the fluid shear rate, expressed in reciprocal seconds.

O.7.2.8 Model increased circulation rates (four cases are usually sufficient) to find the slope b between annular velocity and fluid shear stress at the wall. In laminar flow, the slope relating annular velocity and fluid shear stress should be linear or near-linear.

O.7.2.9 Calculate the critical wall shear stress for an annular velocity of 30 ft/min.

$$\tau_W = \tau_Y + (b \times v_a) \tag{O.13}$$

where

τ_W is the wall shear stress, expressed in pounds-force per hundred square feet;

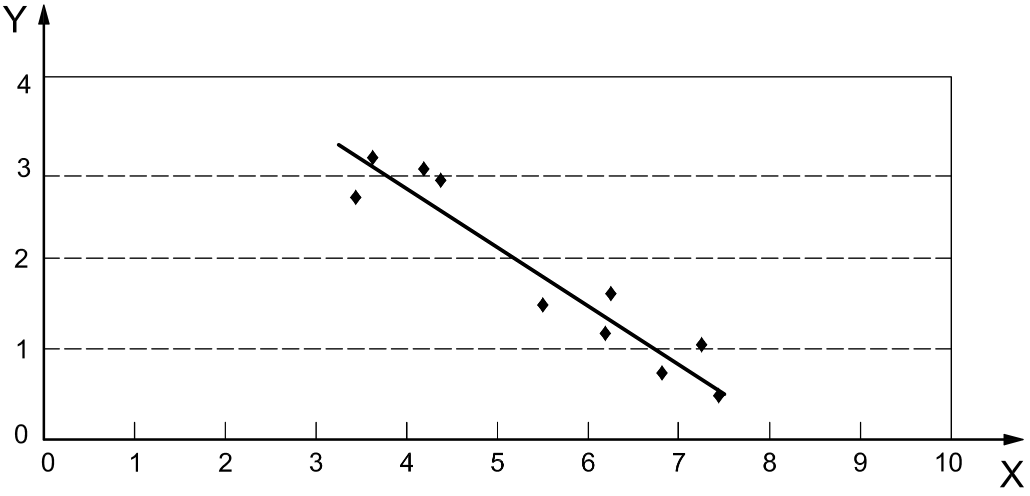
τ_Y is the drilling fluid yield stress, expressed in pounds-force per hundred square feet;

b is the slope of the annular velocity and shear stress at the wall in laminar flow, as defined in O.7.2.8;

v_a is the annular velocity, expressed in feet per minute.

NOTE The selected annular velocity of 30 ft/min is based on published data; it corresponds to the approximate velocity at which the maximum level of sag occurs (see Reference [26]).

O.7.2.10 With the value calculated in Equation (O.13), read the predicted maximum weight-material sag from Figure O.10.



Key

X wall shear stress, $lb_f/100\ ft^2$

Y predicted dynamic sag, lb/gal

Figure O.10 — Predicted dynamic sag as a function of calculated values of tau wall, τ_W

O.7.3 Comparison of laboratory and field data

O.7.3.1 The predicted maximum weight-material sag under dynamic conditions was developed from laboratory data where testing conditions were very favourable for the initiation of weight-material sag.

O.7.3.2 In the field, these conditions are often not as favourable, and the hydraulic method described here usually over-predicts the magnitude of measured sag.

O.8 Additional resource literature

The analysis of weight-material sag remains an active research area. The Bibliography lists additional resource literature (see References [28], [29], [30], [31] and [32]), which expands on the technology given in this annex. More articles will probably be available as new concepts are evaluated and reported.

Annex P
(informative)

Oil-based drilling fluid report form

OIL-BASED DRILLING FLUID REPORT

DATE ....20........	DEPTH .... ☐ MD ☐ TVD
SPUD DATE	PRESENT ACTIVITY ....

API WELL NO.	STATE		COUNTY			WELL						S/T

OPERATOR	CONTRACTOR	RIG NO.
REPORT FOR	REPORT FOR	SECTION, TOWNSHIP, RANGE

WELL NAME AND NO.	FIELD OR BLOCK NO.	COUNTY, PARISH, OFFSHORE AREA	STATE/PROVINCE

BIT DATA	DRILLING STRING	CASING	CIRCULATION DATA

MUD PROPERTIES				
Sample taken from	☐ F.L. ☐ Pit	☐ F.L. ☐ Pit	☐ F.L. ☐ Pit	☐ F.L. ☐ Pit
Time sample taken				
Flowline temperature, °C or °F				
Density ☐ sg ☐ kg/m^3 ☐ lb/ft^3 ☐ lb/gal at .... °C or °F				
Funnel viscosity, s/qt at .... °C or °F				
Plastic viscosity, cP at .... °C or °F				
Yield point, lb/100 ft^2				
Gel strength, lb/100 ft^2 10 s/10 min				
API filtrate, ml/30 min				
HTHP filtrate, ml/30 min at .... °C or °F				
Cake thickness, in/32 or mm				
Electrical stability, v Meter type.........				
Retort solids, % volume				
Retort liquid oil/water, % volume				
Oil/water ratio				
Alkalinity V_{SA}, ml H_2SO_4				
Filtered ☐ yes ☐ no				
Chloride whole mud $c(Cr)_{TOT}$, mg/l				
Calcium whole mud $c(Ca^{++})_{TOT}$, mg/l				

MUD PROPERTY SPECIFICATIONS		
Weight	Viscosity	Filtrate

RECOMMENDED TOUR TREATMENT

REMARKS

PRODUCTS	SOLIDS EQUIPMENT

DRILLING FLUID VOLUME	SOLIDS ANALYSIS	FLUID RHEOLOGY & HYDRAULICS	COST ANALYSIS

REPRESENTATIVE... PHONE... WAREHOUSE PHONE..

Bibliography

[1] ISO 386, *Liquid-in-glass laboratory thermometers — Principles of design, construction and use*

[2] ISO 2977, *Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point*

[3] ISO 13226:2005, *Rubber — Standard reference elastomers (SRE's) for characterizing the effect of liquids on vulcanized rubber*

[4] ISO 13500[21], *Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests*

[5] ASTM C25, *Standard test methods for chemical analysis of limestone, quicklime, and hydrated lime*

[6] ASTM D412, *Standard test methods for vulcanized rubber and thermoplastic elastomers — Tension*

[7] ASTM D471-10, *Standard test method for rubber property — Effect of liquids*

[8] ASTM D1415, *Standard test method for rubber property — International hardness*

[9] ASTM D2240, *Standard test method for rubber property — Durometer hardness*

[10] ASTM D3182, *Standard practice for rubber-materials, equipment, and procedures for mixing standard compounds and preparing standard vulcanized sheets*

[11] ASTM D3183, *Standard practice for rubber — Preparation of pieces for test purposes from products*

[12] WATKINS, T.E. and NELSON, M.D., High temperature gelation of drilling fluids, *AIME Transactions*, **193**, 1953, pp. 213-218

[13] CAMERON, F.K., BELL, J.M. and ROBINSON, W.O., The solubility of certain salts present in alkali soils, *J. Phys. Chem*, January 1907, pp. 396-420

[14] NEASHAM, J.W. API Report Number 1, API-WG ceramic disk pore system analysis utilizing mercury intrusion porosimetry. July 2008

[15] ZAMORA, M. and JEFFERSON, D. Controlling barite sag can reduce drilling problems, *Oil and Gas Journal*, February 1994, pp. 47-52

[16] WARD, C. and ANDREASSEN, E. Pressure while drilling data improve reservoir drilling performance, Society of Petroleum Engineers Drilling and Completion, March 1998, pp. 19-24

[17] WHITE, W., ZAMORA, M. and SVOBODA, C. Downhole measurements of synthetic-based drilling fluid in offshore well quantify dynamic pressure and temperature distributions, IADC/SPE 35057, International Association of Drilling Contractors/Society of Petroleum Engineers Conference, 1998

[18] VAN OORT, E., LEE, J., FRIEDHEIM, J. and TOUPS, J. New flat-rheology synthetic-based mud for improved deepwater drilling, SPE 90987, Society of Petroleum Engineers Annual Conference, 2004

[19] ISAMBOURG, P., BERTIN, D. and BRANGHETTO, M. Field hydraulic tests improve HPHT drilling safety and performance, Society of Petroleum Engineers Drilling Completion, December 1999, pp. 219-227

---

[21] For the purposes of this part of ISO 10414, API Spec 13A, *Specification for drilling fluid materials*, is equivalent.

[20] Charlez, P., Easton, M. and Morrice, G. Validation of advanced hydraulic modelling using PWD data, OTC 8804, Offshore Technology Conference, 4-7 May 1998

[21] Hemphill, T., Bern, P., Rojas, J.C. and Ravi, K. Field validation of drillpipe rotation effects on equivalent circulating density, SPE 110470, Society of Petroleum Engineer Annual Conference, 2007

[22] Hemphill, T., Ravi, K., Bern, P. and Rojas, J.C. A simplified method for prediction of ECD increase with drillpipe rotation, SPE 115378, Society of Petroleum Engineers Annual Conference, 21-24 September 2006

[23] Hemphill, T. and Ravi, K. Turning on barite sag with drillpipe rotation: sometimes surprises are really not surprises, AADE-06-DF-HO-28, American Association of Drilling Engineers FluidsTechnical Conference, 2006

[24] Dye, W., Gusler, W. and Mullen, G. Field-proven technology to manage dynamic barite sag, SPE 98167, International Association of Drilling Contractors/Society of Petroleum Engineers Drilling Conference, 2006

[25] Dye, W., Hemphill, T., Gusler, W. and Mullen, G. Correlation of ultra-low shear rate viscosity and dynamic barite sag, Society of Petroleum Engineers Drilling and Completion, March 2001, pp. 27-34

[26] Hemphill, T. and Rojas, J.C. Improved prediction of barite sag using a fluid dynamics approach, AADE-04-DF-HHE-20, American Association of Drilling Engineers Fluid Conference, 2004

[27] Hemphill, T. Comparisons of barite sag measurements and numerical prediction, AADE-09 NTCE-08-03, American Association of Drilling Engineers Fluid Conference, 2009

[28] Bern, P., van Oort, E., Neusstadt, B., Zurdo, C., Zamora, M. and Slater, K. Barite sag measurement modelling and management, SPE 47784, International Association of Drilling Contractors/Society of Petroleum Engineers Asia Pacific Drilling Conference, 1998

[29] Murphy, R., Jamison, D., Hemphill, T., Bell, S. and Albrecht, C. Measuring and predicting dynamic sag, Society of Petroleum Engineers Drilling and Completion, June 2008, pp. 142-149

[30] Omland, T.H., Sassen, A. and Amundsen, P.A. Detection techniques determining weigh material sag in drilling fluid and relationship to rheology, Nordic Rheology Society Transactions, **18**, 2007

[31] Tehrani, A., Zamora, M. and Power, D. Role of rheology in barite sag in SMB and OBM, AADE-04-DF-HO-22, American Association of Drilling Engineers Fluids Conference, 2004

[32] Zamora, M. and Bell, R. Improved well site test for monitoring barite sag, AADE-04-DF-HO-19, American Association of Drilling Fluids Conference, 2004

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