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Petroleum and natural gas industries — Field testing of drilling fluids

Part 1:
Water-based fluids

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

Partie 1: Fluides aqueux

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.

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ISO 10414-1 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

This second edition cancels and replaces the first edition (ISO 10414-1:2001), to which Annexes I, J and K have been added and other minor changes made to the sentence structure, grammar and other non-technical editing.

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

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

Introduction

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

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

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

INTERNATIONAL STANDARD **ISO 10414-1:2008(E)**

Petroleum and natural gas industries — Field testing of drilling fluids

Part 1:
Water-based fluids

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

1 Scope

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

a) drilling fluid density (mud weight);

b) viscosity and gel strength;

c) filtration;

d) water, oil and solids contents;

e) sand content;

f) methylene blue capacity;

g) pH;

h) alkalinity and lime content;

i) chloride content;

j) total hardness as calcium.

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

— chemical analysis for calcium, magnesium, calcium sulfate, sulfide, carbonate and potassium;

— determination of shear strength;

— determination of resistivity;

— removal of air;

— drill-pipe corrosion monitoring;

— sampling, inspection and rejection;

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

2 Terms and definitions

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

2.1
ACS reagent grade
chemical meeting the purity standards specified by the American Chemical Society (ACS)

2.2
darcy
permeability of a porous medium, where one darcy is the flow of a single-phase fluid of 1 cP viscosity that completely fills the voids of the porous medium, flowing through the medium under conditions of viscous flow at a rate of 1 ml·s^{-1}·cm^{-2} cross-sectional area and under a pressure or equivalent hydraulic gradient of 1 atm·cm^{-1}

NOTE 1 cP = 1 mPa·s.

2.3
quarter
⟨verb⟩ mix and divide into four specimens to ensure homogeneity of specimens

2.4
spurt loss
volume of fluid that passes through the filtration medium before a filter cake is formed

2.5
tube sampling
sampling method consisting of the withdrawal of powdered sample from bag or bulk via a cylindrical device pushed into the sample, locked shut and withdrawn

3 Symbols and abbreviated terms

3.1 Symbols

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

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

$c_{\text{Ca+Mg,A}}$	concentration of calcium and magnesium ion (total hardness), in milligrams per litre
$c_{\text{Ca+Mg,B}}$	concentration of calcium and magnesium ion (total hardness), in parts per million (USC)
$c_{\text{CaSO4,A}}$	concentration of calcium sulfate, in milligrams per litre
$c_{\text{CaSO4,B}}$	concentration of calcium sulfate, in parts per million by mass (USC)
$c_{\text{CO2+CO3+HCO3,A}}$	concentration of total soluble carbonates, in milligrams per litre
$c_{\text{CO2+CO3+HCO3,B}}$	concentration of total soluble carbonates, in parts per million by mass (USC)
$c_{\text{Cl,A}}$	concentration of chloride ion, in milligrams per litre
$c_{\text{Cl,B}}$	concentration of chloride ion, in parts per million by mass (USC)
$c_{\text{ex-CaSO4,A}}$	concentration of excess, undissolved calcium sulfate, in milligrams per litre
$c_{\text{ex-CaSO4,B}}$	concentration of excess, undissolved calcium sulfate, in parts per million by mass (USC)
$c_{\text{f,KCl,A}}$	concentration of potassium chloride in filtrate, in milligrams per litre
$c_{\text{f,KCl,B}}$	concentration of potassium chloride in filtrate, in parts per million by mass (USC)
$c_{\text{K,A}}$	concentration of potassium ion, in milligrams per litre
$c_{\text{K,B}}$	concentration of potassium ion, in parts per million by mass (USC)
$c_{\text{KCl,A}}$	concentration of potassium chloride, in milligrams per litre
$c_{\text{KCl,B}}$	concentration of potassium chloride, in parts per million by mass (USC)
$c_{\text{lg,A}}$	concentration of low-gravity solids, in kilograms per cubic metre
$c_{\text{lg,B}}$	concentration of low-gravity solids, in pounds per barrel
$c_{\text{lime,A}}$	lime content of the drilling fluid, in kilograms per cubic metre
$c_{\text{lime,B}}$	lime content of the drilling fluid, in pounds per barrel
$c_{\text{NaCl,A}}$	concentration of sodium chloride, in milligrams per litre
$c_{\text{NaCl,B}}$	concentration of sodium chloride, in parts per million by mass (USC)
$c_{\text{S,A}}$	concentration of sulphide ion, in milligrams per litre
$c_{\text{S,B}}$	concentration of sulphide ion, in parts per million by mass (USC)
$c_{\text{SS,A}}$	suspended solids concentration, in kilograms per cubic metre
$c_{\text{SS,B}}$	suspended solids concentration in pounds per barrel
c_{MBT}	methylene blue capacity
c_{th}	thermometer correction to be added to the working thermometer reading
D	outer diameter
$E_{\text{BE,A}}$	bentonite equivalent, expressed in kilograms per cubic metre

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

t_{30}	final reading taken at 30 min
V_{df}	volume of drilling fluid sample, in millilitres
V_{EDTA}	volume of EDTA solution, in millilitres
$V_{\mathrm{EDTA,df}}$	EDTA volume of whole drilling fluid
$V_{\mathrm{EDTA,f}}$	EDTA volume of the drilling fluid filtrate
V_{f}	volume of the filtrate, in millilitres
V_{mb}	volume of methylene blue solution, in millilitres
V_{o}	volume of oil, in millilitres
V_{PPT}	PPT volume, in millilitres
V_{RC}	retort cup volume, expressed in millilitres
V_{s}	volume of the sample, in millilitres
V_{sn}	volume of silver nitrate solution, in millilitres
V_{W}	volume of water, in millilitres
V_1	spurt loss, in millilitres
$V_{7,5}$	filtrate volume after 7,5 min, in millilitres
V_{30}	filtrate volume after 30 min, in millilitres
v_{st}	static filtration rate (velocity of flow), millilitres per square root of the minutes, in millilitres per minute
$Y_{\mathrm{P,A}}$	yield point, in pascals
$Y_{\mathrm{P,B}}$	yield point, in pounds per one hundred square feet
γ_{A}	shear strength, expressed in pascals
γ_{B}	shear strength, expressed in pounds per hundred square feet
$\Gamma_{\mathrm{DFG,A}}$	drilling fluid gradient, expressed in kilopascals per metre
$\Gamma_{\mathrm{DFG,B}}$	drilling fluid gradient, in pounds per square inch per foot
η_{a}	apparent viscosity, in millipascal seconds
$\eta_{\mathrm{p,A}}$	plastic viscosity, in millipascal seconds
$\eta_{\mathrm{p,B}}$	plastic viscosity, in pounds per one-hundred square feet
θ	temperature
ρ	density, expressed in g/ml when compared to distilled water
ρ_{A}	density, expressed in kilograms per cubic metre
ρ_{B1}	density, expressed in pounds per gallon

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

3.2 Abbreviations

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

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

4 Drilling fluid density (mud weight)

4.1 Principle

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

4.2 Apparatus

4.2.1 Density-measuring instrument, of accuracy to within 0,01 g/ml or 10 kg/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. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m^3 (8,33 lb/gal or 62,3 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.

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

4.3 Procedure

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

4.3.2 Measure and record the temperature of the drilling fluid.

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

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

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

4.3.6 Read the drilling fluid density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of g/ml, lb/gal, and lb/ft^3, or as a drilling fluid gradient in psi/1 000 ft.

4.4 Calculation

4.4.1 Report the drilling fluid density to the nearest 0,01 g/ml or 10 kg/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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

$$\rho_A = 1\,000 \times \rho \tag{1}$$

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

$$\rho_{B1} = 8{,}33 \times \rho \tag{2}$$

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

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

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

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

Equations (4) to (7) are used to convert the density to the drilling fluid gradient, 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 1.

Table 1 — Density conversion

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

Table 1 (*continued*)

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

[a] Same value as relative density.

[b] Accurate conversion factor.

Table 2 — Conversion of density units

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

5 Alternative drilling fluid density method

5.1 Principle

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

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

5.2 Apparatus

5.2.1 Density-measuring instrument, of accuracy to within 0,01 g/ml or 10 kg/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

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

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 1,00 g/ml or 1 000 kg/m^3 (8,33 lb/gal or 62,3 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.

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

5.3 Procedure

5.3.1 Measure and record the temperature of the drilling fluid.

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

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

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

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

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

5.3.7 The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place the instrument on the knife edge. Move the sliding weight to the right or left until the beam is balanced. The beam is balanced when the attached bubble is centred between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of g/ml, lb/gal and lb/ft^3, or as a drilling fluid gradient in psi/1 000 ft.

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

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

5.4 Calculation

Report the drilling fluid density to the nearest 0,01 g/ml or 10 kg/m^3 (0,1 lb/gal or 0,5 lb/ft^3).

For conversions, use the equations given in 4.4.2.

6 Viscosity and gel strength

6.1 Principle

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

a) Marsh funnel — a simple device for indicating viscosity on a routine basis;

b) direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

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

6.2 Determination of viscosity using the Marsh funnel

6.2.1 Apparatus

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

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

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

6.2.1.1.3 Screen, with 1,6 mm (0,063 in = 1/16 in) openings (12 mesh); fixed at 19,0 mm (0,75 in = ¾ in) below top of funnel.

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

6.2.1.3 Stopwatch.

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

6.2.2 Procedure

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

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

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

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

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

6.3.1 Apparatus

6.3.1.1 Direct-indicating viscometer.

This type of viscometer is a rotational instrument powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity. The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob. Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 r/min and 600 r/min.

A direct-indicating viscometer shall meet the following specifications:

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

b) bob, closed, with flat base and tapered top:
   - diameter 34,49 mm (1,358 in);
   - cylinder length 38,0 mm (1,496 in);

c) torsion spring constant:
   - 386 dyne-cm/degree deflection;

d) rotor sleeve speed:
   - high speed 600 r/min;
   - low speed 300 r/min.

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

6.3.1.2 Stopwatch.

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

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

6.3.2 Procedure

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

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

6.3.2.2 Record the temperature of the sample.

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

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

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

6.3.2.6 Allow drilling fluid sample to stand undisturbed for 10 s. Slowly and steadily turn the hand-wheel in the appropriate direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a speed of 3 r/min, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength (10-second gel) in pounds per 100 ft^2.

NOTE To convert the dial reading to pascals: 1 Pa = 0,511 lb/100 ft^2.

6.3.2.7 Re-stir the drilling fluid sample at 600 r/min for 10 s, stop the rotor and allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 6.3.2.6 and report the maximum reading as the 10-minute gel in pounds per 100 ft^2.

NOTE To convert the dial reading to pascals: 1 Pa = 0,511 lb/100 ft^2.

6.3.3 Calculation

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

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

where

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

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

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

NOTE 2 1 cP = 1 mPa·s.

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

$$Y_\text{P,A} = 0{,}48 \times \left(R_{300} - \eta_\text{P}\right) \tag{9}$$

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

$$Y_\text{P,B} = R_{300} - \eta_\text{P} \tag{10}$$

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

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

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

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

7 Filtration

7.1 Principle

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

7.2 Low-temperature/low-pressure test

7.2.1 Apparatus

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

This cell is made of materials resistant to strongly alkaline solutions and is so fitted that a pressure medium can be conveniently admitted into, and bled from, the top. It shall also be fitted such that a sheet of 90 mm (3,54 in) diameter filter paper can be placed in the bottom of the cell just above a suitable support. The filtration area is (45,8 ± 0,6) cm^2 [(7,1 ± 0,1) in^2]. Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets and the entire assembly supported by a stand. Pressure can be applied with any non-hazardous fluid medium. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midget pressure cartridges or means for utilizing hydraulic pressure. To obtain correlative results, one thickness of the proper 90 mm diameter filter paper (e.g. Whatman No. 50, S&S No. 576[1] or equivalent) shall be used.

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

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

7.2.1.2 Timer, with at least a 30 min interval.

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

7.2.2 Procedure

7.2.2.1 Be sure each part of the cell, particularly the screen, is clean and dry and that the gaskets are not distorted or worn. Pour the drilling fluid sample into the cell to within 1 cm to 1,5 cm (0, 4 in to 0,6 in) of the top (to minimize CO_2 contamination of filtrate), and complete the assembly with the filter paper in place.

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

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

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

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

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

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

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

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

7.3.1 Apparatus

7.3.1.1 **HT/HP filter press**, consisting of a controlled pressure source (CO_2 or nitrogen), regulators, a drilling-fluid cell able to contain working pressures from 4 000 kPa to 8 900 kPa (600 psi to 1 300 psi), a system for heating the cell, a pressurized collection cell able to maintain proper backpressure (see Table 3) in order to prevent flashing or evaporation of the filtrate, and a suitable stand. The drilling-fluid cell has a thermometer well, oil-resistant gaskets, a support for the filter medium and a valve on the filtrate delivery tube to control flow from the cell. It can be necessary to replace the gaskets frequently.

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

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

DANGER — Do not use nitrous oxide cartridges as pressure sources for HT/HP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas-train carbonate analysis.

7.3.1.2 **Filter media**[2].

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

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

7.3.1.3 **Timer**, with at least a 30 min interval.

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

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

7.3.1.6 **High-speed mixer**.

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

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

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

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

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

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

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

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

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

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

7.3.2.8 Correct the filtrate volume to a filter area of 45,8 cm^2 (7,1 in^2). For example, if the filter area is 22,6 cm^2 (3,5 in^2), double the filtrate volume reported.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

7.3.3.10 Correct the filtrate volume to a filter area of 45,8 cm^2 (7,1 in^2). For example, if the filter area is 22,6 cm^2 (3,5 in^2), double the filtrate volume reported.

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

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

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

Table 3 — Recommended minimum backpressure

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

8 Water, oil and solids contents

8.1 Principle

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

8.2 Apparatus

8.2.1 Retort instrument.

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

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

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

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

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

8.2.1.4 **Temperature control** (optional), capable of limiting the temperature of the retort to 500 °C ± 40 °C (930 °F ± 70 °F).

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

Total volume, ml:	10	20	50

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

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

8.2.3 Fine steel wool, oil-free.

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

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

8.2.5 Pipe cleaners.

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

8.2.7 Marsh funnel.

8.2.8 Defoaming agent.

8.2.9 Corkscrew.

8.3 Procedure

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

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

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

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

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

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

NOTE This is determined from experience.

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

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

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

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

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

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

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

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

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

8.4 Calculation

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

a) volume fraction water:

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

$$\varphi_{\mathrm{W}} = 100 \times \frac{V_{\mathrm{W}}}{V_{\mathrm{df}}} \tag{12}$$

where

V_{W} is the volume of water, expressed in millilitres;

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

b) volume fraction oil:

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

$$\varphi_{\mathrm{o}} = 100 \times \frac{V_{\mathrm{o}}}{V_{\mathrm{df}}} \tag{13}$$

where

V_{o} is the volume of oil, expressed in millilitres;

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

c) volume fraction retort solids:

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

$$\varphi_{\mathrm{S}} = 100 - (\varphi_{\mathrm{W}} + \varphi_{\mathrm{O}}) \tag{14}$$

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

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

$$\varphi_{SS} = \varphi_S - \left(\frac{\varphi_W \times c_{Cl}}{1\,680\,000 - 1{,}21\, c_{Cl}} \right) \tag{15}$$

where

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

φ_W is the percentage (volume fraction) of water;

φ_S is the percentage (volume fraction) of solids.

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

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

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

where

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

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

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

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

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

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

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

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

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

$$\varphi_b = \varphi_{SS} - \varphi_{lg} \tag{19}$$

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

$$c_{lg,A} = 10 \rho_{lg} \times \varphi_{lg} \tag{20}$$

$$c_{b,A} = 10 \rho_b \times \varphi_b \tag{21}$$

$$c_{SS,A} = c_{lg,A} + c_{b,A} \tag{22}$$

where

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

φ_{b} is the volume fraction of barite, expressed as a percentage.

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

$$c_{\text{lg,B}} = 3{,}49\rho_{\text{lg}} \times \varphi_{\text{lg}} \tag{23}$$

$$c_{\text{b,B}} = 3{,}49\rho_{\text{b}} \times \varphi_{\text{b}} \tag{24}$$

$$c_{\text{SS,B}} = c_{\text{lg,B}} + c_{\text{b,B}} \tag{25}$$

where

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

φ_{b} is the volume fraction of barite, expressed as a percentage.

9 Sand content

9.1 Principle

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

9.2 Apparatus

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

9.2.2 **Funnel**, to fit sieve.

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

9.3 Procedure

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

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

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

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

10 Methylene blue capacity

10.1 Principle

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

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

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

10.2 Reagents and apparatus

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

The moisture content of reagent grade methylene blue shall be determined each time the solution is prepared. Dry a 1,000 g portion of methylene blue to a constant mass at 93 °C ± 3 °C (200 °F ± 5 °F). Make the appropriate correction in the mass, m_s, of methylene blue, expressed in grams, to be taken to prepare the solution as given in Equation (26):

$$m_\text{s} = \frac{3{,}2}{m_\text{ds}} \tag{26}$$

where m_ds is the mass of the dried sample, expressed in grams.

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

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

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

DANGER — H_2SO_2 is a strong and toxic acid.

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

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

10.2.5 Erlenmeyer flask, capacity 250 ml.

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

10.2.7 Graduated cylinder, 50 ml (TD).

10.2.8 Stirring rod.

10.2.9 Hot plate.

10.2.10 Filter paper, Whatman No. 1[3] or equivalent.

10.3 Procedure

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

a) Remove the air or gas entrained in the drilling fluid (see Annex D for information on air or gas removal). Stir the drilling fluid to break the gel and quickly draw the drilling fluid into the syringe. Then slowly discharge the syringe back into the drilling fluid, keeping the tip submerged.

b) Again draw the drilling fluid into the syringe until the end of the plunger is at the last graduation on the syringe (e.g. at the 3-ml line on a 3-ml syringe).

c) Deliver 2,0 ml of drilling fluid by pushing the plunger until the end of the plunger is exactly 2 ml from the last graduation on the syringe. This, in a 3-ml syringe, is at the 1-ml line.

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

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

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

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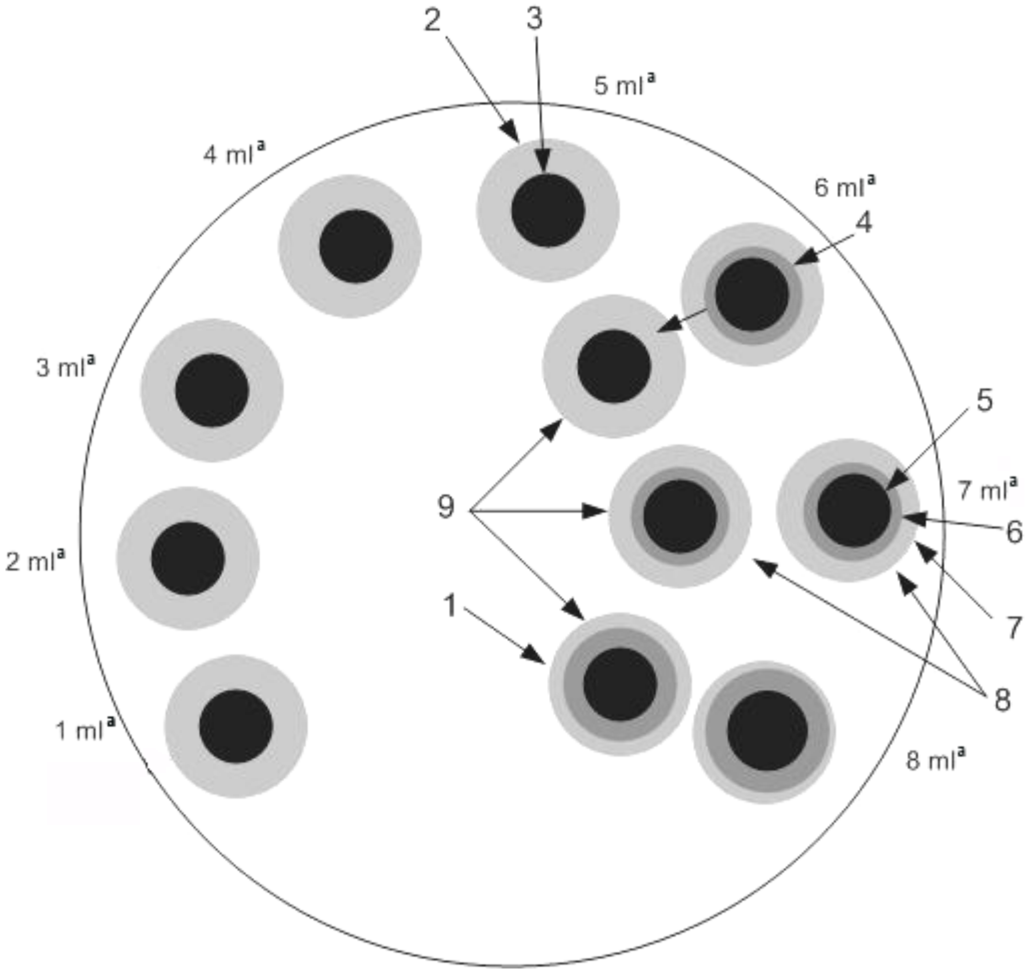
[3] Whatman No. 1 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 10414 and does not constitute an endorsement by ISO of these products.



Key

1 endpoint overshot
2 moisture
3 dyed drilling fluid solids (no free, unadsorbed dye present)
4 free dye, visible immediately after adding the sixth ml, is adsorbed after 2 min and indicates that the endpoint has not quite been reached
5 dyed drilling fluid solids
6 free, unadsorbed dye
7 moisture
8 endpoint
9 appearance of spot after an additional 2 min absorption

[a] Volume of methylene blue solution added, in millilitres.

Figure 1 — Spot tests for endpoint of methylene blue titration

10.4 Calculation

Report the methylene blue capacity, c_{MBT}, of the drilling fluid, calculated as follows:

$$C_{\text{MBT}} = \frac{V_{\text{mb}}}{V_{\text{df}}} \tag{27}$$

where

V_{mb} is the volume of methylene blue solution, expressed in millilitres;

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

Alternatively, the methylene blue capacity can be reported as bentonite equivalent (based on bentonite with a cation exchange capacity of 70 meq/100 g), $E_{\text{BE,A}}$, expressed in kilograms per cubic metre, as given in Equation (28) or $E_{\text{BE,B}}$, expressed in pounds per barrel, as given in Equation (29)]:

$$E_{\text{BE,A}} = \frac{14{,}25 \times V_{\text{mb}}}{V_{\text{df}}} \tag{28}$$

$$E_{\text{BE,B}} = \frac{5 \times V_{\text{mb}}}{V_{\text{df}}} \tag{29}$$

NOTE The kilograms per cubic metre (or pounds per barrel) bentonite equivalent from Equations (28) and (29) is not equal to the amount of commercial bentonite in the drilling fluid. Reactive clays in the drill solids contribute to this quantity as well as commercial bentonite. See ISO 10416[4] or API RP 13I[5] for additional information on estimating the amount of commercial bentonite and drill solids present.

11 pH

11.1 Principle

11.1.1 Field measurement of drilling fluid (or filtrate) pH and adjustments to the pH are fundamental to drilling fluid control. Clay interactions, solubility of various components and contaminants, and effectiveness of additives are all dependent on pH, as is the control of acidic and sulfide-corrosion processes.

11.1.2 The term “pH” denotes the negative logarithm of the hydrogen ion, H^+, activity in aqueous solutions (activity and concentration are equal only in dilute solutions): $\text{pH} = -\log [H^+]$. For pure water at 24 °C (75 °F) the hydrogen ion activity $[H^+]$ is 10^{-7} mol/l and pH = 7. This system is termed “neutral” because the hydroxyl ion activity $[OH^-]$ is also 10^{-7} mol/l. In aqueous systems at 24 °C (75 °F) the ion product, $[H^+] \times [OH^-]$, is 10^{-14} (a constant). Consequently, an increase in H^+ denotes a like decrease in $[OH^-]$. A change in pH of one unit indicates a ten-fold change in both $[H^+]$ and $[OH^-]$. Solutions with pH less than 7 are termed “acidic” and those with pH greater than 7 are termed “basic” or “alkaline”.

11.1.3 The recommended method for measurement of drilling fluid pH is with a glass-electrode pH meter. This method is accurate and gives reliable pH values, being free of interferences if a high-quality electrode system is used with a properly designed instrument. Rugged pH instruments are available that automatically temperature-compensate the slope and are preferred over the manually adjusted instruments.

Colour-matching pH paper and sticks are used for field pH measurements, but are not the methods recommended. These methods are reliable only in very simple water-based drilling fluids. Drilling fluid solids, dissolved salts and chemicals and dark-coloured liquids cause serious errors in pH-paper values. Readability is normally about 0,5 pH unit.

11.2 Reagents and apparatus

11.2.1 Buffer solutions, to calibrate and set the slope of pH meter prior to sample measurement at the following pH values:

a) pH = 4,0: potassium hydrogen phthalate at 0,05 mol/l in water; gives 4,01 pH at 24 °C (75 °F);

b) pH = 7,0: potassium dihydrogen phosphate at 0,020 66 mol/l and disodium hydrogen phosphate at 0,029 34 mol/l in water; gives 7,00 pH at 24 °C (75 °F);

c) pH = 10,0: sodium carbonate at 0,025 mol/l and sodium bicarbonate at 0,025 mol/l in water; gives 10,01 pH at 24 °C (75 °F).

Buffers may be obtained from supply houses as pre-made solution, dry-powder packages, or a given formula. Shelf life of all buffers should not exceed six months before disposal. The date of preparation of the buffer should be shown on bottles used in the field. Bottles should be kept tightly stoppered.

11.2.2 Distilled or **deionized water**, in spray bottle.

11.2.3 Mild liquid detergent.

11.2.4 Sodium hydroxide, (CAS No. 1310-73-2), 0,1 mol/l (approximately), to recondition electrode.

DANGER — NaOH is a strong caustic alkaline chemical. Avoid skin contact.

11.2.5 Hydrochloric acid, (CAS No. 7674-01-0), 0,1 mol/l (approximately), to recondition electrode.

DANGER — HCl is a strong and toxic acid.

11.2.6 Ammonium bifluoride (CAS No. 1341-49-7), 10 % solution (approximately), to recondition electrode.

DANGER — Avoid skin contact.

11.2.7 Millivolt-range potentiometer, calibrated to show pH units for measuring the potential between a glass-membrane electrode and a standard “reference” electrode.

The instrument should preferably be water-, shock- and corrosion-resistant and portable. Specifications are the following:

a) pH range: 0 to 14;

b) electronics type: solid state (preferred);

c) power source: batteries (preferred);

d) operating temperature range: 0 °C to 66 °C (32 °F to 150 °F);

e) readout: digital (preferred);

f) resolution: 0,1 pH unit;

g) accuracy: ± 0,1 pH unit;

h) repeatability: 0,1 pH unit;

i) adjustments:

- “temperature” compensation of electrode system;

- "slope" of electrode system (preferred);
- "calibration" setting of readout. (Instrument with the above internal temperature compensation is preferred.)

11.2.8 Electrode system, a combination of a glass electrode for sensing H^+ ions and a standard voltage reference electrode, preferably constructed as a single electrode.

The body of this probe should be constructed of durable material. A flat-end probe is preferred for better protection and easier cleaning of the electrode. Waterproof connection to the meter is recommended. Specifications are the following:

a) glass pH electrode response range: 0 to 14 pH units;

b) electrodes: glass electrode and a silver/silver chloride electrode in combination, having a ceramic or a plastic single or double junction;

NOTE Use double-junction electrode for measuring liquids containing sulfide or bromide ion to avoid damaging (silver) reference electrode system.

c) electrolyte in reference electrode: KCl gel;

d) glass composition: suitable for low sodium-ion error;

e) sodium-ion error: at pH = 13 or at 0,1 mol Na^+ ion, an error less than 0,1 pH unit.

11.2.9 Tissue, soft, to blot electrodes.

11.2.10 Thermometer, glass, 0 °C to 105 °C (32 °F to 220 °F).

11.2.11 Test-tube brush, soft bristle, to clean electrode.

11.2.12 Electrode-storage vial, to keep electrodes moist.

11.3 Procedure for pH measurement

11.3.1 Obtain a sample of fluid to be tested. Allow it to reach 24 °C ± 3 °C (75 °F ± 5 °F).

11.3.2 Allow buffer solution to reach the same temperature as the fluid to be tested.

For accurate pH measurement, the test fluid, buffer solution and reference electrode should all be at the sample temperature. The pH of the buffer solution indicated on the container label is the correct pH only at 24 °C (75 °F). If attempting to calibrate at another temperature, the actual pH of the buffer at this temperature should be used. Tables of buffer pH values at various temperatures are available from the suppliers and should be used in the calibration procedure.

11.3.3 Clean electrodes by washing with distilled water and blot dry.

11.3.4 Place probe into pH 7,0 buffer.

11.3.5 Turn on meter; wait 60 s for reading to stabilize (see 11.4 if meter reading is not stable).

11.3.6 Measure temperature of pH 7,0 buffer solution.

11.3.7 Set this temperature on "temperature" knob.

11.3.8 Set meter reading to "7,0" using "calibration" knob.

11.3.9 Rinse probe with distilled water and blot dry.

11.3.10 Repeat operations in 11.3.6 through 11.3.9 using either pH 4,0 or pH 10,0 buffer. Use pH 4,0 if “acidic” sample, or pH 10,0 if “alkaline” sample is to be tested. Set meter to number “4,0” or “10,0,” respectively, using “slope” adjustment knob. (If no “slope” knob exists, use the “temperature” knob to set “4,0” or “10,0” on meter.)

11.3.11 Check the meter again with pH 7,0 buffer. If it has changed, reset to “7,0” with “calibration” knob. Repeat 11.3.6 through 11.3.9. If meter does not calibrate properly, recondition or replace electrodes as given in 11.4.

Discard and do not reuse the sample of buffer solutions used in calibration. Meter should be fully calibrated every day, as per 11.3.2 through 11.3.9, using two buffers. Check with pH 7,0 buffer every 3 h when using the meter continuously and prior to use, if more than 3 h had lapsed between measurements.

11.3.12 If meter calibrates properly, rinse electrode with distilled water and blot dry. Place electrode in sample to be tested and stir gently. Allow 60 s to 90 s for reading to stabilize.

11.3.13 Record sample pH to nearest 0,1 pH unit and the temperature of sample.

11.3.14 Carefully clean the electrode in preparation for next usage. Store in vial of pH 4,0 buffer. Never let the probe tip become dry.

11.3.15 Turn meter off and close cover to protect instrument. Avoid storing instrument at extreme temperatures [below 0 °C (32 °F) or above 50 °C (120 °F)].

11.4 Care of electrode

11.4.1 Cleaning the electrode is necessary periodically, especially if oil or clay particles coat the face of the glass electrode or the porous frit of the reference electrode. Clean electrode with a soft-bristle brush and a mild detergent.

11.4.2 Reconditioning the electrode can be necessary if plugging becomes severe, as indicated by a slow response, drifting of readings, or if “slope” and “calibration” cannot be mutually set.

11.4.3 Recondition by soaking electrode for 10 min in 0,1 mol/l HCl, followed by rinsing in water and soaking for 10 min in 0,1 mol/l NaOH and rinsing again.

11.4.4 Check electrode for response by performing calibration in 11.3.1 through 11.3.15.

11.4.5 If electrode continues to perform poorly, soak electrode for 2 min only in 10 % ammonium bifluoride solution. Repeat 11.3.1 through 11.3.15 to check for calibration capability.

11.4.6 Replace electrode system if steps 11.4.3 to 11.4.5 fail to recondition it.

12 Alkalinity and lime content

12.1 Principle

12.1.1 Alkalinity can be considered as the acid-neutralizing power of a substance. In drilling fluid testing, alkalinity measurements can be made on either the whole drilling fluid (designated with a subscript “df”) or on the filtrate (designated with a subscript f). The data collected from the alkalinity test can also be used to estimate the concentrations of hydroxyl $[OH^-]$, carbonate $[CO_3^{-2}]$ and bicarbonate $[HCO_3^-]$ ions in the drilling fluid.

12.1.2 Knowledge of the drilling fluid and filtrate alkalinities is important in many drilling operations to ensure proper control of the drilling fluid chemistry. Drilling fluid additives, particularly some deflocculants, require an alkaline environment to function properly. Alkalinity arising from hydroxyl ions is generally accepted as being beneficial, while alkalinities resulting from carbonates and/or bicarbonates can have adverse effects on the drilling fluid performance.

12.1.3 The ions that are primarily responsible for filtrate alkalinities are the hydroxyl (OH^-), carbonate (CO_3^{-2}), and bicarbonate (HCO_3^-) ions. It is important to realize that the carbonate species can change from one form to another form by changing the solution pH. The interpretation of filtrate alkalinities involves calculating differences between the titration values obtained by the following procedures. It is for this reason that special attention to accurate measurement of the various reagents is important in all steps of the procedure. In addition, it is important to realize that the following calculations are only estimates of the concentrations of the reported ionic species based on theoretical chemical equilibrium reactions.

12.1.4 The composition of drilling fluid filtrates is often so complex that the interpretation of alkalinities in terms of estimated ionic components can be misleading. Any particular alkalinity value represents all of the ions that react with the acid in the pH range over which that particular value was tested. Inorganic ions that can contribute to the alkalinity, in addition to the hydroxyl, carbonate and bicarbonate ions, are borates, silicates, sulfides and phosphates. Perhaps more serious in drilling fluids are anionic organic thinners, filtrate reducers and their degradation products that can contribute to a large portion of the alkalinity value as well as masking the endpoint colour change. These organic materials make a particularly large contribution to the M_f alkalinity and thus render the test highly inaccurate in drilling fluids treated with organic thinners. However, for simple bentonite-base drilling fluid systems containing no organic thinners, the P_f and M_f alkalinities (see 12.3) can be used as guidelines to determine both the presence of carbonate/bicarbonate contamination and the treatment necessary to alleviate the contamination.

12.2 Reagents and apparatus

12.2.1 **Sulfuric acid** (CAS No. 7664-93-9) solution: standardized 0,02 N (N/50).

DANGER — H_2SO_2 is a strong and toxic acid.

12.2.2 **Phenolphthalein** (CAS No. 518-51-4) indicator solution: 1 g/100 ml in 1:1 alcohol:water solution.

12.2.3 **Methyl orange** (CAS No. 547-58-0) indicator solution: 0,1 g/100 ml of water.

12.2.4 **pH meter** (optional).

NOTE A pH meter is more accurate than an indicator solution.

12.2.5 **Titration vessel**, 100 ml or 150 ml, preferably white.

12.2.6 **Graduated pipettes**, 1 ml (TD) and 10 ml (TD).

12.2.7 **Volumetric pipette**, 1 ml (TD).

12.2.8 **Syringe**, 1 ml (TD).

12.2.9 **Stirring rod**.

12.3 Procedure — Phenolphthalein and methyl orange filtrate alkalinities

12.3.1 Measure one or more millilitres of filtrate into the titration vessel. Add two or more drops of the phenolphthalein indicator solution. If the indicator turns pink, add 0,02 N (N/50) sulfuric acid, drop by drop from the graduated pipette, while stirring, until the pink colour just disappears. If the sample is so coloured that the indicator colour change is masked, the endpoint can be taken when the pH drops to 8,3 as measured with a pH meter. (Refer to Clause 11 for proper pH measurement).

12.3.2 Report the phenolphthalein alkalinity, P_f, of the filtrate as the number of millilitres of 0,02 N acid required per millilitre of filtrate.

12.3.3 To the sample that has been titrated to the P_f endpoint, add two or three drops of methyl orange indicator solution. Add the standard acid drop by drop from the pipette, while stirring, until the colour of the indicator changes from yellow to pink. The endpoint can also be taken when the pH of the sample drops to 4,3 as measured by a pH meter. (Refer to Clause 11 for proper pH measurement.)

12.3.4 Report the methyl orange alkalinity, M_f, of the filtrate as the total millilitres of 0,02 N acid per millilitre of filtrate required to reach the methyl orange endpoint (including that amount required for the P_f endpoint).

12.4 Procedure — Phenolphthalein drilling fluid alkalinity

12.4.1 Measure 1,0 ml of drilling fluid into the titration vessel using a syringe or volumetric pipette. Dilute the drilling fluid sample with 25 ml to 50 ml of distilled water. Add 4 drops to 5 drops of phenolphthalein indicator solution and if the indicator turns pink, while stirring, titrate rapidly with 0,02 N (N/50) standard sulfuric acid solution until the pink colour disappears. If the endpoint colour change cannot be seen, it can be taken when the pH drops to 8,3 as measured by a pH meter. (Refer to Clause 11 for proper pH measurement.)

If cement contamination is suspected, the titration shall be performed as rapidly as possible and the endpoint reported as the first disappearance of the pink colour.

12.4.2 Report the phenolphthalein alkalinity, P_{df}, of the drilling fluid as the number of millilitres of 0,02 N (N/50) acid required per millilitre of drilling fluid.

12.5 Calculation of ion concentrations from P_f and M_f

The mass concentrations of hydroxyl, carbonate and bicarbonate ions can be estimated from P_f and M_f as shown in Table 4.

Table 4 — Concentrations of hydroxyl, carbonate and bicarbonate ions

Relative values of P_f and M_f	Concentration mg/l		
	OH^-	CO_3^{-2}	HCO_3^-
$P_f = 0$	0	0	1 220 M_f
2 $P_f < M_f$	0	1 200 P_f	1 220 $(M_f - 2P_f)$
2 $P_f = M_f$	0	1 200 P_f	0
2 $P_f > M_f$	340 (2 $P_f - M_f$)	1 200 $(M_f - P_f)$	0
$P_f = M_f$	340 M_f	0	0

12.6 Estimation of lime content

12.6.1 Determine the P_f and P_{df} of the filtrate and drilling fluid as described in 12.3 and 12.4.

Determine the volume fraction, F_W, of water in the drilling fluid using the value for volume fraction, expressed as a decimal fraction, of water from the liquid and solids determination (Clause 8) in the following equation:

$$F_W = \frac{\varphi_W}{100} \qquad (30)$$

where φ_W is the volume fraction, expressed as a percentage, of water in the drilling fluid (see Clause 8).

12.6.2 Report the lime content of the drilling fluid, $c_{lime,A}$, in kilograms per cubic metre, as given in Equation (31) [or $c_{lime,B}$, in pounds per barrel, as given in Equation (32)]:

$$c_{lime,A} = 0{,}742 \times (P_{df} - F_W P_f) \qquad (31)$$

$$c_{lime,B} = 0{,}26 \times (P_{df} - F_W P_f) \qquad (32)$$

where

F_W is the volume fraction of water in the drilling fluid, expressed as a decimal;

P_{df} is the phenolphthalein alkalinity of the drilling fluid;

P_f is the phenolphthalein alkalinity of the filtrate.

13 Chloride ion content

13.1 Principle

The chloride test measures the chloride ion concentration in drilling fluid filtrate.

13.2 Reagents and apparatus

13.2.1 Silver nitrate (CAS No. 7761-88-8) solution, containing 4,791 g/l (0,0282 N; equivalent to 0,001 g/ml chloride ion) , stored in an amber or opaque bottle.

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

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

13.2.3 Sulfuric acid (CAS No. 7664-93-9) or **nitric acid** (CAS No. 7697-37-2) solution, standardized 0,02 N (N/50).

DANGER — H_2SO_4 and HNO_3 are strong and toxic acids.

13.2.4 Phenolphthalein (CAS No. 518-51-4) indicator solution, 1 g/100 ml of 1:1 alcohol/water solution.

13.2.5 Calcium carbonate (CAS No. 471-34-1), precipitated, chemically pure grade.

13.2.6 Distilled water.

13.2.7 Graduated pipettes, 1 ml (TD) and 10 ml (TD).

13.2.8 Titration vessel, 100 ml or 150 ml, preferably white.

13.2.9 Stirring rod.

13.3 Procedure

13.3.1 Measure 1 ml or more of filtrate into the titration vessel. Add 2 drops to 3 drops phenolphthalein solution. If the indicator turns pink, add acid drop by drop from pipette, while stirring, until the colour has disappeared. If the filtrate was originally deeply coloured, add an additional 2 ml of 0,02 N (N/50) sulfuric acid or nitric acid and stir. Then add 1 g calcium carbonate and stir.

13.3.2 Add 25 ml to 50 ml distilled water and 5 drops to 10 drops potassium chromate solution. Stir continuously, while adding standard silver nitrate solution drop by drop from the pipette, until the colour changes from yellow to orange-red and persists for 30 s. Record the volume of silver nitrate solution required to reach the endpoint. If over 10 ml of silver nitrate solution is used, repeat the test with a smaller sample of filtrate.

NOTE If the chloride ion concentration of the filtrate exceeds 10 000 mg/l, a silver nitrate solution equivalent to 0,01 g/ml (0,282 N) chloride ion can be used. The factor 1 000 in Equation (33) is then changed to 10 000.

13.4 Calculation

13.4.1 Report the chloride ion concentration, c_{Cl}, of the filtrate, expressed in milligrams per litre, calculated as given in Equation (33):

$$c_{\mathrm{Cl}} = 1\,000 \times \frac{V_{\mathrm{sn}}}{V_{\mathrm{f}}} \tag{33}$$

where

V_{sn} is the volume of silver nitrate solution, expressed in millilitres;

V_{f} is the volume of the filtrate sample, expressed in millilitres.

13.4.2 Use Equation (34) to convert $c_{\mathrm{Cl,}}$ to the sodium chloride concentration, $c_{\mathrm{NaCl,A}}$, expressed in milligrams per litre:

$$c_{\mathrm{NaCl,\,A}} = 1{,}65\ c_{\mathrm{Cl}} \tag{34}$$

13.4.3 To convert from milligrams per litre to parts per million by mass (USC unit), use Equation (35) and the density of the filtrate from Equation (18):

$$c_{\mathrm{NaCl,B}} = \frac{c_{\mathrm{NaCl,A}}}{\rho_{\mathrm{f}}} \tag{35}$$

NOTE In very dilute solutions, milligrams per litre is equal to parts per million.

14 Total hardness as calcium

14.1 Principle

The hardness of water or drilling fluid filtrate is due primarily to the presence of calcium and magnesium ions. When EDTA (or its salt) is added to the water or filtrate, it combines with both the calcium and magnesium and the endpoint is determined with a suitable indicator. The total hardness of the water or filtrate is expressed as milligrams calcium per litre. An endpoint obscured by dark components can often be remedied by oxidizing with a reagent such as sodium hypochlorite.

14.2 Reagents and apparatus

14.2.1 EDTA solution (CAS No. 6381-92-6), 0,01 mol/l; standardized disodium ethylenediaminetetraacetate dihydrate (1 ml/ml sample = 1 000 mg $CaCO_3$, 1 ml/ml sample = 400 mg Ca^{2+}).

NOTE EDTA is distributed with various supplier names, Versenate®[4] or “standard Versenate solution” being the most common.

14.2.2 Buffer solution, 67,5 g ammonium chloride (CAS No. 12125-02-9) and 570 ml ammonium hydroxide (CAS No. 1336-21-6) (15 N) diluted to 1 000 ml with distilled water.

14.2.3 Hardness indicator solution, 1 g/l Calmagite®[5] or equivalent; 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid (CAS No. 3147-14-6) in distilled water.

14.2.4 Acetic acid (CAS No. 64-19-7), glacial.

DANGER — Avoid skin contact.

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[4] Versenate® 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.

[5] Calmagite® is an example 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.

14.2.5 Masking agent, 1:1:2 volume mixture of triethanolamine (CAS No. 102-71-6):tetraethylenepentamine (CAS No. 112-57-2):water.

14.2.6 Sodium hypochlorite (CAS No. 7681-52-9) solution, 5,25 % mass fraction in deionized water.

DANGER — Avoid skin contact.

Many brands of commercial laundry bleach contain calcium hypochlorite or oxalic acid and should not be used. Ensure the sodium hypochlorite is fresh, as it will deteriorate with time.

14.2.7 Deionized or distilled water.

The deionized water and sodium hypochlorite solution should be tested for hardness by using 50,0 ml of the deionized water and 10 ml of the sodium hypochlorite solution without the test sample, and continuing with 14.3.7 and 14.3.8. If the procedure is then repeated with the test sample utilizing 50 ml deionized water and 10 ml sodium hypochlorite solution in 14.3.2 through 14.3.6, the hardness of the test sample can be determined by subtracting the hardness of the deionized water and hypochlorite.

14.2.8 Titration vessel, 150 ml beaker.

14.2.9 Graduated pipettes, 5 ml (TD) and 10 ml (TD).

14.2.10 Volumetric pipettes, 1 ml (TD), 2 ml (TD) and 5 ml (TD).

14.2.11 Hot plate (required if filtrate is coloured).

14.2.12 pH paper strip.

14.3 Procedure

14.3.1 Measure one or more millilitres of sample into a 150 ml beaker. (If filtrate is clear, or is only lightly coloured, omit steps 14.3.2 through 14.3.5.)

14.3.2 Add 10 ml sodium hypochlorite solution and mix.

14.3.3 Add 1 ml glacial acetic acid and mix.

14.3.4 Boil the sample for 5 min. Maintain the sample volume by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required.

Work in an adequately ventilated area.

14.3.5 Cool the sample.

14.3.6 Rinse the inside of the beaker with deionized water and dilute the sample to 50 ml with deionized water. Add approximately 2 ml buffer solution and swirl to mix.

NOTE The presence of soluble iron can interfere with the endpoint determination. If this is suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume) has proven to be a suitable masking agent. 1 ml of the mixture is used per titration.

14.3.7 Add sufficient hardness indicator (2 drops to 6 drops) and mix. A wine-red colour develops if calcium and/or magnesium is/are present.

14.3.8 While stirring, titrate with EDTA solution to the proper endpoint. Calcium indicators produce a change from red to blue. The endpoint is best described as the point at which additional EDTA produces no further red to blue colour change. The titration volume of EDTA is used in the calculation in 14.4.

14.4 Calculation

The total hardness, calcium plus magnesium ion concentration, $c_{\mathrm{Ca+Mg}}$, expressed in milligrams per litre as calcium, is calculated as given in Equation (36):

$$c_{\mathrm{Ca+Mg}} = 400 \times \frac{V_{\mathrm{EDTA}}}{V_{\mathrm{s}}} \qquad (36)$$

where

V_{EDTA} is the volume of EDTA solution, expressed in millilitres;

V_{s} is the volume of the sample, expressed in millilitres.

NOTE The concentration of calcium and magnesium is commonly known in the industry as total hardness, and reported as calcium.

Annex A

(informative)

Chemical analysis of water-based drilling fluids

A.1 Calcium

A.1.1 Principle

When EDTA (or its salt) is added to water or drilling fluid filtrate containing both calcium and magnesium, it combines first with calcium. Calcium can be determined with EDTA when the pH of the sample is sufficiently high so that magnesium is precipitated as the hydroxide and an indicator specific for calcium is used. Several indicators give colour changes when all of the calcium has been complexed by EDTA at a pH of 12 to 13. An endpoint obscured by dark organic components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

A.1.2 Reagents and apparatus

A.1.2.1 **EDTA** (CAS No. 6381-92-6): 0,01 mol/l solution, standardized disodium ethylenediaminetetraacetate dihydrate (1 ml/ml sample = 1 000 mg $CaCO_3$, 1 ml/ml sample = 400 mg Ca^{2+}).

NOTE EDTA is distributed with various supplier names, Versentate® or “standard Versenate solution” being the most common.

A.1.2.2 **Calcium buffer solution**: 1 mol/l (1 N) sodium hydroxide (CAS No. 1310-73-2).

DANGER — NaOH is a strong caustic alkaline chemical. Avoid skin contact.

A.1.2.3 **Calcium indicator**, Calver® **II**[6) or hydroxy naphthol blue (CAS No. 63451-35-4).

A.1.2.4 **Acetic acid** (CAS No. 64-19-7), glacial.

DANGER — Avoid skin contact.

A.1.2.5 **Titration vessel**, 150 ml beaker.

A.1.2.6 **Graduated pipettes**, 1 ml (TD) and 10 ml (TD).

A.1.2.7 **Volumetric pipettes**, 1 ml (TD), 2 ml (TD) and 5 ml (TD).

A.1.2.8 **Hot plate** (required if filtrate is coloured).

A.1.2.9 **Masking agent**: 1:1:2 volume mixture of triethanolamine (CAS No. 102-71-6): tetraethylenepentamine (CAS No. 112-57-2):water.

A.1.2.10 **pH paper**.

A.1.2.11 **Graduated cylinder**, 50 ml (TC).

A.1.2.12 **Sodium hypochlorite** (CAS No. 7861-52-9), solution: 5,25 % mass fraction in deionized water.

DANGER — Avoid skin contact.

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6) 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.

Many brands of commercial laundry bleach contain calcium hypochlorite or oxalic acid and should not be used. Ensure the sodium hypochlorite is fresh, as it deteriorates with time.

A.1.2.13 **Deionized** or **distilled water**.

The deionized water and sodium hypochlorite solution should be tested for calcium by using 50,0 ml of the deionized water and 10 ml of the sodium hypochlorite solution without the test sample. If the procedure is then repeated with the test sample, utilizing 50,0 ml of the deionized water and 10 ml of the sodium hypochlorite solution as given in A.1.3, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and sodium hypochlorite solution.

A.1.3 Procedure

A.1.3.1 With a volumetric pipette, add 1 ml or more of sample to a 150-ml beaker. This sample volume is used in the calculation shown in Equation (A.1). If filtrate is colourless or is only slightly coloured, omit A.1.3.2 through A.1.3.5.

A.1.3.2 With graduated pipette, add 10 ml hypochlorite solution and mix.

A.1.3.3 With graduated pipette, add 1 ml glacial acetic acid and mix.

A.1.3.4 Boil the sample for 5 min. Maintain the sample by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required. A sufficiently boiled sample shows a pH of 5,0.

A.1.3.5 Cool the sample.

A.1.3.6 Rinse the inside of the beaker with deionized water and dilute the sample to approximately 50 ml with deionized water. Add 10 ml to 15 ml of calcium buffer solution or sufficient sodium hydroxide to produce a pH of 12 to 13.

NOTE The presence of soluble iron can interfere with the endpoint determination. If this is suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume) is a suitable masking agent. Add 1,0 ml of the mixture after A.1.3.6.

A.1.3.7 Add sufficient calcium indicator (0,1 g to 0,2 g) to produce a pink to wine-red colour if calcium is present. Too much indicator obscures the endpoint.

NOTE The addition of several drops of methyl orange along with the calcium indicator can improve the visibility of the endpoint.

A.1.3.8 While stirring, titrate with standard EDTA to the proper endpoint. Calcium indicators produce a change from red to blue. The endpoint is best described as that point where additional EDTA produces no further red to blue colour change. The EDTA volume is used in the calculation in Equation (A.1).

A.1.4 Calculation

The calcium ion concentration, c_{Ca}, expressed in milligrams per litre, is calculated as given in Equation (A.1):

$$c_{\mathrm{Ca}} = 400 \times \frac{V_{\mathrm{EDTA}}}{V_{\mathrm{f}}} \tag{A.1}$$

where

c_{Ca} is the calcium ion concentration, expressed in milligrams per litre;

V_{EDTA} is the volume of EDTA solution, expressed in millilitres (1 ml = 400 mg Ca^{+2});

V_f is the volume of the filtrate sample, expressed in millilitres.

A.2 Magnesium

A.2.1 Principle

The magnesium content of the drilling fluid filtrate can be calculated by subtracting the calcium ion content from the total hardness. This gives the magnesium content in terms of calcium which is converted to magnesium by multiplying the value by the ratio of atomic weights (24,3/40 = 0,6).

A.2.2 Procedure

A.2.2.1 Determine the total hardness as calcium (14.3 through 14.4).

A.2.2.2 Determine the calcium content as described in Clause A.1.

A.2.3 Calculation

The magnesium concentration, c_{Mg}, expressed in milligrams per litre, is calculated as given in Equation (A.2):

$$c_{\mathrm{Mg}} = 0{,}6 \times \left(c_{\mathrm{Ca+Mg}} - c_{\mathrm{Ca}}\right) \tag{A.2}$$

where

$c_{\mathrm{Ca+Mg}}$ is the total hardness, expressed as calcium in milligrams per litre;

c_{Ca} is the calcium concentration, expressed in milligrams per litre.

A.3 Calcium sulfate

A.3.1 Principle

The calcium sulfate content of drilling fluid is determined by using the EDTA method as described in Clause A.1 to determine the total calcium in a drilling fluid filtrate and the whole drilling fluid. The total and undissolved calcium sulfate contents of the drilling fluid can then be calculated.

A.3.2 Reagents and apparatus

A.3.2.1 **EDTA** (CAS No. 6381-92-6): 0,01 mol solution, standardized disodium ethylenediaminetetraacetate dihydrate (1 ml/ml sample = 1 000 mg $CaCO_3$, 1 ml/ml sample = 400 mg Ca^{2+}).

NOTE EDTA is distributed with various supplier names, Versentate® or “standard Versenate solution” being the most common.

A.3.2.2 **Calcium buffer solution**: 1 mol/l (1 N) sodium hydroxide (CAS No. 1310-73-2).

DANGER — NaOH is a strong caustic alkaline chemical. Avoid skin contact.

A.3.2.3 **Calcium indicator**, Calver® II(7) or hydroxy naphthol blue (CAS No. 63451-35-4).

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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.

A.3.2.4 **Acetic acid** (CAS No. 64-19-7), glacial.

DANGER — Avoid skin contact.

A.3.2.5 **Masking agent**: 1:1:2 volume mixture of triethanolamine (CAS No. 102-71-6): tetraethylenepentamine (CAS No. 112-57-2):water.

A.3.2.6 **Sodium hypochlorite** (CAS No. 7861-52-9) solution, mass fraction of 5,25 % in deionized water.

DANGER — Avoid skin contact.

Many brands of commercial laundry bleach contain calcium hypochlorite or oxalic acid and should not be used. Ensure the sodium hypochlorite is fresh, as it will deteriorate with time.

A.3.2.7 **Deionized or distilled water**.

The deionized water and sodium hypochlorite solution should be tested for calcium sulfate by using 10 ml of the deionized water and 10 ml of the sodium hypochlorite solution without the test sample. If the procedure is then repeated with the test sample utilizing 10 ml of the deionized water and 10 ml of the sodium hypochlorite solution in A.3.3, the calcium sulfate of the test sample can be determined by subtracting the calcium sulfate of the deionized water and sodium hypochlorite solution.

A.3.2.8 **Titration vessel**, 150 ml beaker.

A.3.2.9 **Graduated pipettes**, 1 ml (TD) and 10 ml (TD).

A.3.2.10 **Volumetric pipettes**, 1 ml (TD), 2 ml (TD), 5 ml (TD) and 10 ml (TD).

A.3.2.11 **Hot plate** (required, if filtrate is coloured).

A.3.2.12 **pH paper**.

A.3.2.13 **Graduated cylinder**, 50 ml (TC).

A.3.2.14 **Drilling fluid retort**, as described in Clause 8.

A.3.3 Procedure

A.3.3.1 Add 5 ml of whole drilling fluid to 245 ml deionized water. Stir the mixture for 15 min and filter through a standard filter press in accordance with 7.2.2. Collect only clear filtrate. Into a 150 ml beaker, add 10 ml of clear filtrate with the 10 ml volumetric pipette and titrate to the EDTA endpoint as described in Clause A.1 and designate this volume of EDTA as $V_{\mathrm{EDTA,df.}}$

A.3.3.2 Titrate 1 ml of the original drilling fluid filtrate (obtained as described in 7.2) to the EDTA endpoint. Designate this volume of EDTA as $V_{\mathrm{EDTA,f.}}$

A.3.3.3 Determine the volume fraction of water, F_{W}, expressed as a decimal, in the drilling fluid by using the value for volume fraction, φ_{W}, expressed as a percent, of water from the liquid and solids determination (Clause 8) and Equation (A.3):

$$F_{\mathrm{W}} = \frac{\varphi_{\mathrm{W}}}{100} \tag{A.3}$$

A.3.4 Calculation

A.3.4.1 Calculate the calcium sulfate concentration of the drilling fluid, $c_{\mathrm{CaSO4,A}}$, expressed in kilograms per cubic metre, as given in Equation (A.4) [or $c_{\mathrm{CaSO4,B}}$, expressed in pounds per barrel, as given in Equation (A.5)]:

$$c_{\text{CaSO4,A}} = 6{,}60\ V_{\text{EDTA,df}} \tag{A.4}$$

$$c_{\text{CaSO4,B}} = 2{,}31\ V_{\text{EDTA,df}} \tag{A.5}$$

where $V_{\text{EDTA,df}}$ is the EDTA titration volume of whole drilling fluid; see A.3.3.1.

A.3.4.2 Calculate the (excess) undissolved calcium sulfate concentration of the drilling fluid, $c_{\text{ex-CaSO4,A}}$, expressed in kilograms per cubic metre, as given in Equation (A.6) [or $c_{\text{ex-CaSO4,B}}$, expressed in pounds per barrel, as given in Equation (A.7)]:

$$c_{\text{ex-CaSO4,A}} = 6{,}6\ V_{\text{EDTA,df}} - 1{,}32\ \left(V_{\text{EDTA,f}} \times F_{\text{W}}\right) \tag{A.6}$$

$$c_{\text{ex-CaSO4,B}} = 2{,}31\ V_{\text{EDTA,df}} - 0{,}463\ \left(V_{\text{EDTA,f}} \times F_{\text{W}}\right) \tag{A.7}$$

where

$c_{\text{ex-CaSO4,A}}$	is the excess (undissolved calcium sulfate concentration, expressed in kilograms per cubic metre;
$c_{\text{ex-CaSO4,B}}$	is the excess (undissolved calcium sulfate concentration, expressed in pounds per barrel;
F_{W}	is the volume fraction of water in the drilling fluid, expressed as a decimal;
$V_{\text{EDTA,df}}$	is the EDTA volume of whole drilling fluid; see A.3.3.1;
$V_{\text{EDTA,f}}$	is the EDTA volume of the drilling fluid filtrate; see A.3.3.2.

A.4 Sulfide

A.4.1 Principle

A.4.1.1 The concentration of soluble sulfides in a drilling fluid can be determined by this method. Soluble sulfides include H_2S and the sulfide (S^{2-}) and bisulfide (HS^{-}) ions. Drilling fluid filtrate is acidified in a Garrett gas train, converting all sulfides to H_2S, which is evolved by bubbling an inert carrier gas through the sample. The gas train separates the gas from the liquid. The gas stream is passed through a Dräger8) tube, which responds to H_2S by darkening along its length. The darkened length is proportional to the total sulfide in the drilling-fluid filtrate. The low-range Dräger tube turns from white to brownish-black and the high-range Dräger tube turns from pale blue to jet-black. No common drilling-fluid contaminant causes these colour changes.

A.4.1.2 Lead-acetate paper disks can be accommodated in the Garrett gas train to determine 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.

A.4.2 Reagents and apparatus

A.4.2.1 **Sulfuric acid** (CAS No. 7664-93-9), approximately 2,5 mol/l (5N), ACS reagent grade.

DANGER — H_2SO_4 is a strong and toxic acid.

A.4.2.2 **Defoamer**, in a dropper bottle.

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8) 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 of ISO of this product.

A.4.2.3 **Carrier gas**, inert to hydrogen sulfide, acid and Dräger tube reagents.

Nitrogen is preferred but carbon dioxide is acceptable. (Avoid air or other oxygen-containing gases.)

A.4.2.4 **Dräger H_2S analysis tubes**:

A.4.2.4.1 Low range: marked H_2S 100/a (No. CH 29101, 100 mg/l to 200 mg/l H_2S);

A.4.2.4.2 High range: marked H_2S 0,2 %/A (No. CH 28101, 0,2 volume % to 7 volume % H_2S).

A.4.2.5 **Garrett gas train apparatus**, consisting of a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flow meter and a Dräger tube.

Specifications of the Garrett gas train:

a) Body
- Chamber 1:
  - Depth 90 mm (3,54 in)
  - Diameter 38 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 that is inert to acid, sulfides and hydrogen sulfide gas

b) 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

c) Flow meter, floating-ball type preferred, capable of measuring 300 ml/min of CO_2 gas.
d) Flexible tubing, type inert to hydrogen sulfide and carrier gas. Latex rubber or equivalent is preferred.
e) Fittings and rigid tubing, type inert to hydrogen sulfide and acid.
f) Rubber septum.

A.4.2.6 **Lead-acetate paper disk** (see A.4.3.16).

A.4.2.7 **Hypodermic syringes**, 10 ml and 2,5 ml (for acid), and 5 ml and 10 ml (for sample).

A.4.2.8 **Hypodermic needles**, 38 mm (1,5 in) 21-gauge needles.

A.4.3 Procedure

A.4.3.1 Ensure that the gas train is clean, dry and on a level surface, with the top removed.

NOTE Moisture in the train can cause the ball in the flow meter to float erratically and can affect the accuracy of the Dräger tube reading.

A.4.3.2 Add 20 ml of deionized water to chamber 1.

A.4.3.3 Add 5 drops defoamer to chamber 1.

A.4.3.4 See Table A.1 for sample volume and type of Dräger tube required for the expected sulfide range. Select the proper type Dräger tube. Break the tip from each end of the tube.

A.4.3.5 Install the Dräger tube with the arrow pointing downward into the bored receptacle. Likewise, install the flow-meter tube with the word “TOP” upward. Ensure that the O-rings seal around the body of each tube.

A.4.3.6 Install the top on the gas train and hand-tighten all screws evenly to seal the O-rings.

Table A.1 — Dräger tube (or equivalent) identification, sample volume and tube factors for various sulfide ranges

Sulfide range mg/l	**Sample volume** V_s ml	**Dräger tube identification** [a]	**Tube factor** [b,c] f
1,2 to 24	10,0	H_2S 100/a	0,12 [d]
2,4 to 48	5,0	H_2S 100/a	0,12 [d]
4,8 to 96	2,5	H_2S 100/a	0,12 [d]
30 to 1 050	10,0	H_2S 0,2 %/a	1 450 [e]
60 to 2 100	5,0	H_2S 0,2 %/a	1 450 [e]
120 to 4 200	2,5	H_2S 0,2 %/a	1 450 [e]

[a] See tube body.

[b] Used in calculation.

[c] If other tubes are used, it is necessary to change the tube factors in Table A.1 in accordance with the manufacturer's specification.

[d] Tube factor 0,12 applies to tubes marked H_2S 100/a (Cat. No. CH 29101) with 100 mg/l to 2 000 mg/l scale. For older tubes with the 1 to 20 scale, use a tube factor of 12.

[e] Tube factor of 1 450 applies to tubes marked H_2S 0,2%/a (Cat. No. CH 28101) with 0,2 volume % to 7,0 volume % scale. For older tubes with the 1 to 17 cubic centimetre scale, use a tube factor 600 times the ratio: Batch Factor/0,40.

A.4.3.7 With the regulator backed off, connect the carrier gas to the dispersion tube of chamber 1 using flexible tubing. If a CO_2 cartridge is used, install and puncture cartridge and connect to dispersion tube.

A.4.3.8 Attach the flexible tubing from chamber 3 outlet to the Dräger tube.

Use only latex rubber or inert plastic tubing. Do not clamp flexible tubing; unclamped tubing provides pressure relief in the event of over-pressurization.

A.4.3.9 Adjust the dispersion tube in chamber 1 to approximately 6 mm (0,25 in) above the bottom.

A.4.3.10 Gently flow carrier gas for 30 s to purge air from the system. Check for leaks. Shut off the carrier gas.

A.4.3.11 Collect a sufficient volume of solids-free filtrate for analysis. (If a low concentration of soluble sulfides is to be detected, a large volume of filtrate is required. Use Table A.1 as a guide.)

A.4.3.12 Inject a measured volume of the solids-free filtrate sample into chamber 1 through the rubber septum, using a hypodermic syringe and needle.

A.4.3.13 Slowly inject 10 ml sulfuric acid solution into chamber 1 through the rubber septum using the hypodermic syringe and needle.

A.4.3.14 Immediately restart the carrier gas flow. The flow rate should be maintained between 200 ml/min and 400 ml/min.

NOTE One CO_2 cartridge should provide about 15 min to 20 min of flow at this rate.

A.4.3.15 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 flowing for a total of 15 min although the front can attain a diffuse and feathery colouration. In the high-range tube, an orange colour (caused by SO_2) can 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 best Dräger tube accuracy, the “darkened length” should fill more than half the tube's length, therefore the filtrate “sample volume” shall be carefully selected.

A.4.3.16 A lead acetate paper disk fitted under the O-ring of chamber 3 can be substituted for the Dräger tube in the gas train. The lead-acetate paper qualitatively indicates the presence or absence of sulfides in the sample. A dark discoloration of the paper is a positive indication of sulfides. After a positive indication, the Dräger tube should be used on a separate sample for quantitative analysis.

A.4.3.17 To clean the gas train, remove the flexible tubing and remove the top. Take the Dräger tube and the flowmeter out of the receptacles and plug the holes with stoppers to keep them dry. Wash out the chambers with warm water and mild detergent, using a soft brush. Use a pipe cleaner to clean the passages between the chambers. Wash, rinse and blow out the dispersion tube with a dry gas. Rinse the unit with deionized water and allow to drain dry.

A.4.4 Calculation

The sulfide concentration, c_{S}, expressed in milligrams per litre, in the sample can be calculated as given in Equation (A.8):

$$c_{\mathrm{S}} = \frac{l_{\mathrm{st}} \times f}{V_{\mathrm{s}}} \qquad \text{(A.8)}$$

where

l_{st} is the Dräger tube's maximum darkened length, in units marked on the tube;

V_{s} is the measured sample volume, expressed in millilitres;

f is the tube factor from Table A.1.

A.5 Carbonate

A.5.1 Principle

The concentration of soluble carbonates in a drilling fluid filtrate can be determined by this method. Total soluble carbonates include CO_2 and the carbonate (CO_3^{2-}) and bicarbonate (HCO_3^{-}) ions. Drilling fluid filtrate is acidified in a Garrett gas train, converting all carbonates to CO_2, which is then evolved by bubbling an inert carrier gas through the sample. The gas train separates the gas from the liquid. The gas stream is collected in a 1 l gas bag (to allow CO_2 to mix uniformly) and subsequently drawn through a Dräger[9] tube at a fixed flowrate. The Dräger tube responds to CO_2 by progressively staining purple along its length. A reaction between CO_2 and a hydrazine chemical causes a crystal violet indicator to turn purple. The stain length is proportional to the total carbonate concentration in the filtrate.

A.5.2 Reagents and apparatus

A.5.2.1 **Garrett gas train apparatus**, consisting of a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flow meter and a Dräger tube.

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9) 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 of ISO of this product.

Specifications of the Garrett gas train:

a) Body
    - Chamber 1:
        - Depth 90 mm (3,54 in)
        - Diameter 38 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 that is inert to acid, sulfides, and hydrogen sulphide gas

b) 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

c) Flow meter, floating ball type preferred, capable of measuring 300 ml/min of CO^2 gas.

d) Flexible tubing, type inert to hydrogen sulfide and carrier gas. Latex rubber or equivalent is preferred.

e) Fittings and rigid tubing, type inert to hydrogen sulfide and acid.

f) Rubber septum.

A.5.2.2 **Carrier gas**, high-purity nitrogen (N_2) bottle with low-pressure regulator (preferred), or N_2O gas cartridges.

DANGER — Do not use nitrous oxide cartridges as pressure sources for high-temperature/high pressure (HTHP) filtration. Under high temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Use nitrous oxide cartridges only for Garrett gas train carbonate analysis.

A.5.2.3 **Dräger CO_2 analysis tube**, marked CO_2 100/a (cat. No. 8101811), 100 mg/l to 3 000 mg/l.

A.5.2.4 **Dräger 1 l Alcotest gas bag**, No. 7626425, or equivalent.

A.5.2.5 **Dräger Accuro®** [10] **hand-operated bellows gas-detector vacuum pump**, Model 31, or equivalent.

A.5.2.6 **Stopcock**, 2-way bore, 8 mm (0,315 in) glass with PTFE plug.

A.5.2.7 **Sulfuric acid** (CAS No. 7664-93-9), approximately 2,5 mol/l (5 N), ACS reagent grade.

DANGER — H_2SO_4 is a strong and toxic acid.

A.5.2.8 **Defoamer**, in a dropper bottle.

A.5.2.9 **Hypodermic syringes**, 1,0 ml, 5 ml and 10 ml (for acid) and 10 ml (for sample).

A.5.2.10 **Hypodermic needles**, 38 mm (1,5 in) 21-gauge needles.

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10) Dräger Accuro® 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 of ISO of this product.

NOTE Nitrogen is preferred over N_2O as the carrier gas. Because N_2O cools upon expansion and chills the diaphragm in the regulator, prolonged N_2O flow causes the regulator to perform erratically.

A.5.3 Procedure

A.5.3.1 Ensure that the gas train is clean, dry and on a level surface, with the top removed.

If CO_2 has been used as the carrier gas in the previous test (i.e. sulfide analysis), the regulator, tubing and dispersion tube should be purged with carrier gas at this time.

A.5.3.2 Add 20 ml deionized water to chamber 1.

A.5.3.3 Add 5 drops of defoamer to chamber 1.

A.5.3.4 Install the top on the gas train and hand-tighten evenly to seal all O-rings.

A.5.3.5 Adjust the dispersion tube to approximately 6 mm (0,25 in) off bottom.

A.5.3.6 With regulator backed off, connect carrier gas supply to glass dispersion tube of chamber 1 using flexible tubing.

A.5.3.7 Flow carrier gas through train for 1 min to purge air from the system. Check for leaks in gas train unit.

A.5.3.8 Fully collapse the gas bag and simultaneously check the system for leaks. To do this, connect the gas bag and stopcock to the hand pump. (Use a discarded Dräger tube as connection and start with the bag essentially empty.) Fully depress and release the hand pump. When the bag is completely empty and free of leaks, the pump will remain depressed for several minutes. If leakage is detected, check the pump and all connections. To check the pump alone, insert a sealed Dräger tube into the pump opening and depress the bellows. It will remain depressed if pump does not leak.

A.5.3.9 With the bag fully collapsed, install flexible tubing from the stopcock and bag onto the outlet of chamber 3.

A.5.3.10 Inject a measured sample volume of solids-free filtrate into chamber 1 through the septum with a hypodermic syringe and needle. See Table A.2.

A.5.3.11 Slowly inject 10 ml sulfuric acid solution into chamber 1 through the rubber septum using a clean syringe and needle. Gently shake the gas train to mix acid with sample in chamber 1.

Table A.2 — Dräger tube (or equivalent) identification, sample volumes and tube factors for various carbonate ranges

Carbonate range mg/l	**Sample volume** V_s ml	**Dräger tube identification** [a]	**Tube factor** [b,c] f
25 to 750	10,0	CO_2 100/a	2,5 [d]
50 to 1 500	5,0	CO_2 100/a	2,5 [d]
100 to 3 000	2,5	CO_2 100/a	2,5 [d]
250 to 7 500	1,0	CO_2 100/a	2,5 [d]

[a] See tube body.

[b] Used in calculation.

[c] If other tubes are used, the tube factors in Table A.2 shall be changed according to manufacturer's specification.

[d] Tube factor 2,5 applies to tubes marked CO_2 100/a (Cat. No. 8101811) with 100 mg/l to 3 000 mg/l scale.

A.5.3.12 Open the stopcock on the gas bag. Restart gas flow and allow gas bag to fill steadily during a 10 min interval. When bag is firm to the touch (do not burst it) shut off flow and close the stopcock. Immediately proceed to next step.

A.5.3.13 Break the tip off each end of the Dräger tube.

A.5.3.14 Remove the tubing from chamber 3 outlet and reinstall it onto the upstream end of the Dräger tube. (Observe that an arrow on the tube indicates gas flow direction.) Attach the Dräger hand pump to the downstream end of the Dräger tube.

A.5.3.15 Open the stopcock on the bag. With steady hand-pressure, fully depress the hand pump. Release the pump so that gas flows out of the bag and through the Dräger tube. Operate the pump and count the strokes until the bag is empty. (Ten strokes should empty the bag. More than ten strokes indicates that leakage has occurred and that the test results will not be correct.)

A.5.3.16 Observe the purple stain on the Dräger tube when CO_2 is present in the gas bag and record the stain length in units marked on the Dräger tube. (Include the faint blue tinge in the purple stain length reading.)

For best Dräger tube accuracy, the "stain length" should fill more than half the tube length, therefore, "sample volume" shall be carefully selected.

A.5.3.17 To clean the gas train, remove the flexible tubing and remove the top. Wash out the chambers with warm water and mild detergent, using a brush. Use a pipe cleaner to clean the passages between chambers. Wash, rinse and then blow out the dispersion tube with dry gas. Rinse the unit with deionized water and allow to drain dry. Periodically replace the disposable gas bag to avoid leaks and contamination in the bag. (Bag replacement is suggested after ten analyses).

A.5.4 Calculation

Calculate total soluble carbonate concentration, $c_{\text{CO2+CO3+HCO3}}$, expressed in milligrams per litre, in the filtrate sample using Equation (A.9):

$$c_{\text{CO2+CO3+HCO3}} = \frac{l_{\text{st}} \times f}{V_{\text{s}}} \qquad \text{(A.9)}$$

where

V_{s} is the sample volume, expressed in millilitres;

l_{st} is the Dräger tube stain length;

f is the tube factor from Table A.2.

A.6 Potassium (concentration above 5 000 mg/l)

A.6.1 Principle

Potassium ion is used in drilling fluids to aid in the stabilization of shales and to control swelling clays. The accurate determination of the potassium ion content is necessary to control the properties of the drilling fluid. This procedure is used to measure the potassium ion content in drilling fluid filtrates at levels above 5 000 mg/l, which is 10 kg/l (3,5 lb/bbl) potassium chloride. Potassium is precipitated in a centrifuge tube as the perchlorate salt and the volume of precipitate is measured. The potassium ion content is read from a prepared standard curve.

A.6.2 Reagents and apparatus

A.6.2.1 **Sodium perchlorate** ($NaClO_4$) (CAS No. 7601-89-0), solution: 150,0 g/100 ml distilled water.

DANGER — Sodium and potassium perchlorates are explosive in the dry state if heated or if in contact with organic reducing agents. The perchlorates are not hazardous if kept wet. They decompose harmlessly in water.

A.6.2.2 **Standard potassium chloride** (CAS No. 7447-40-7), solution: 14,0 g made up to 100 ml with deionized or distilled water.

A.6.2.3 **Centrifuge**, horizontal-swing rotor head (manual or electric), capable of producing approximately 1 800 r/min.

NOTE A fairly constant 1 800 r/min can be obtained with a manual centrifuge as follows. Determine the number of revolutions of the rotor per each turn of the crank; i.e. move the crank very slowly and count the number of revolutions of the rotor head during one turn of the crank. For example, 15 revolutions of the rotor per one turn of the crank. Calculate the number of crank turns required to obtain 1 800 revolutions of the rotor head. In the example, to obtain 1 800 revolutions of the head would require 120 turns of the crank (1 800/15). Thus the crank must be turned 120 times in one minute to obtain the rate of 1 800 r/min. At this rate, in 5 s the handle must be turned 10 times [i.e. (120/60) × 5]. By counting the crank turns in 5 s and adjusting the rate to obtain the required number of turns, a constant 1 800 r/min can be obtained in 15 s to 20 s. It is necessary to add the interval used to adjust to the 1 800 r/min to the centrifuge time of the sample.

A.6.2.4 **Clinical centrifuge tube**, 10 ml Kolmer type.

A.6.2.5 **Graduated volumetric pipettes**, 1 ml (TD), 2 ml (TD) and 5 ml (TD).

A.6.2.6 **Hypodermic syringe** or **serological (graduated) pipette**, 10 ml (TD).

A.6.2.7 **Distilled or deionized water**.

A.6.3 Preparation of standard calibration curve

A.6.3.1 A standard calibration curve is required for each type of centrifuge. A minimum of three points [10 kg/m^3 (3,5 lb/bbl), 30 kg/m^3 (10,5 lb/bbl) and 50 kg/m^3 (17,5 lb/bbl) KCl] is required to obtain an accurate graph.

A.6.3.2 Samples can be prepared by using the standard potassium chloride solution [0,5 ml of standard potassium chloride solution is equivalent to 10 kg/m^3 (3,5 lb/bbl) KCl]. To obtain KCl concentrations of 10 kg/m^3 (3,5 lb/bbl), 30 kg/m^3 (10,5 lb/bbl) and 50 kg/m^3 (17,5 lb/bbl) KCl, use 0,5 ml, 1,5 ml and 2,5 ml of the standard potassium chloride solution, respectively.

A.6.3.3 Dilute each sample to the 7,0 ml mark with distilled water and agitate.

A.6.3.4 Add 3,0 ml of standard sodium perchlorate solution (but do not agitate).

A.6.3.5 Centrifuge at a constant speed (approximately 1 800 r/min) for 1 min and read the precipitate volume immediately.

Counterbalance the centrifuge tube with another tube and liquid of the same mass.

A.6.3.6 Clean the centrifuge tube immediately after use to facilitate ease of cleaning.

A.6.3.7 Plot the volume of precipitate (millilitres) versus potassium chloride content in kilograms per cubic metre (or pounds per barrel) on rectangular graph paper as shown in Figure A.1.

A.6.4 Test procedure

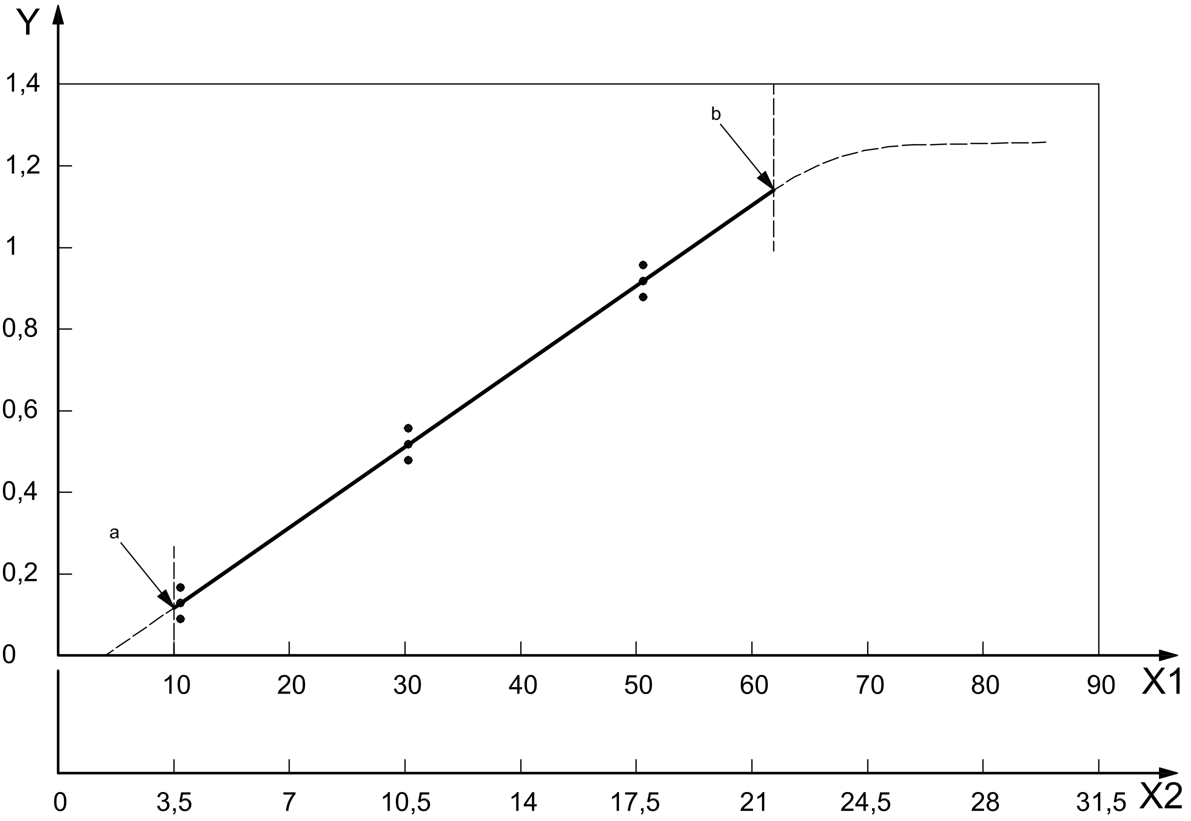
A.6.4.1 Measure the appropriate volume of filtrate into the centrifuge tube (see Table A.3 for range).

A.6.4.2 If less than 7,0 ml filtrate volume is used, dilute to 7,0 ml with distilled water and agitate.

A.6.4.3 Add 3,0 ml of standard sodium perchlorate solution but do not agitate. If potassium is present, precipitation occurs at once.

A.6.4.4 Centrifuge at constant speed (approximately 1 800 r/min) for 1 min. Read the precipitate volume immediately and record.

Counterbalance the centrifuge tube with another tube and liquid of the same mass.



Key

X1 $c_{\text{KCl,A}}$, expressed in kilograms per cubic metre
X2 $c_{\text{KCl,B}}$, expressed in pounds per barrel
Y volume of precipitate, expressed in millilitres

[a] Do not use this procedure for concentrations less than 10 kg/m^3 (3,5 lb/bbl).

[b] Refer to Table A.3 beyond this point.

Figure A.1 — Example of plotted calibration curve for potassium chloride (do not use for calibration)

Table A.3 — Filtrate volumes to be used at various KCl concentrations

KCl concentration range		**K$^+$ in filtrate**	**Filtrate volume to use**
$c_{\text{KCl,A}}$ kg/m^3	$c_{\text{KCl,B}}$ (lb/bbl)	mg/l	V_f Ml
10 to 50	3,5 to 17,5	5 250 to 26 250	7,0
50 to 100	17,5 to 35	26 250 to 52 500	3,5
100 to 200	35 to 70	52 500 to 105 000	2,0
over 200	over 70	over 105 000	1,0

A.6.4.5 Add 2 drops to 3 drops of the sodium perchlorate solution to the tube. If precipitate still forms, the total amount of potassium was not measured. See Table A.3 and use the next smaller filtrate volume. Repeat A.6.4.1 through A.6.4.4.

A.6.4.6 Determine the potassium chloride concentration of the diluted test sample by comparing the precipitate volume measured with the standard calibration curve as prepared in A.6.3. Report the potassium chloride concentration as $c_{\text{KCl,A}}$, expressed in kilograms per cubic metre (or as $c_{\text{KCl,B}}$, expressed in pounds per barrel) KCl. The potassium concentration may also be reported as milligrams per litre potassium ion. If the diluted sample potassium chloride concentration, $c_{\text{KCl,A}}$ ($c_{\text{KCl,B}}$), from the standard calibration curve exceeds a 50 kg/m^3 (or 18 lb/bbl) reading, accuracy of the results is reduced. For more accurate results, use the next smaller filtrate volume as noted in Table A.3, and repeat A.6.4.1 through A.6.4.4.

A.6.5 Calculation

Calculate the filtrate potassium chloride concentration, $c_{\text{,f,KCl,A}}$, expressed in milligrams per litre, as given in Equation (A.10) [or $c_{\text{f,KCl,B}}$, expressed in pounds per barrel, as given in Equation (A.11)]:

$$c_{\text{f,KCl,A}} = \left(\frac{7}{V_{\text{f}}}\right) \times c_{\text{KCl,A}} \qquad \text{(A.10)}$$

$$c_{\text{f,KCl,B}} = \left(\frac{7}{V_{\text{f}}}\right) \times c_{\text{KCl,B}} \qquad \text{(A.11)}$$

where

$c_{\text{KCl,A}}$ is the corresponding concentration on the X1 axis of the standard curve (see Figure A.1), expressed in kilograms per cubic metre;

$c_{\text{KCl,B}}$ is the corresponding concentration on the X2 axis of the standard curve (see Figure A.1), expressed in pounds per barrel;

V_{f} is the volume of filtrate used, expressed in millilitres.

Calculate the filtrate potassium ion concentration, $c_{\text{K,,A}}$, expressed in milligrams per litre, from $c_{\text{KCl,A}}$ expressed in kilograms per cubic meter, as given in Equation (A.12) [or $c_{\text{K,,B}}$, expressed in pounds per barrel, from $c_{\text{KCl,B}}$ as given in Equation (A.13)]:

$$c_{\text{K,A}} = 525 \times c_{\text{KCl,A}} \qquad \text{(A.12)}$$

$$c_{\text{K,B}} = 1\,500 \times c_{\text{KCl,B}} \qquad \text{(A.13)}$$

where $c_{\text{KCl,A}}$ and $c_{\text{KCl,B}}$ are as defined above.

A.7 Potassium (concentration below 5 000 mg/l)

A.7.1 Principle

This procedure is used to measure potassium ion content in drilling fluid filtrates at levels below 5 000 mg/l. Potassium ion is precipitated as the tetraphenylborate salt by adding an excess of standard sodium tetraphenylborate (STPB) solution. The unreacted STPB is then determined by titration with a quaternary ammonium salt (QAS), hexadecyltrimethyl ammonium bromide, using bromophenol blue as an indicator. The endpoint is a colour change from purple-blue to light blue. The potassium ion concentration, c_{K}, in the sample is calculated by subtracting the amount of unreacted STPB from the amount of STPB originally added to the sample.

A.7.2 Reagents and apparatus

A.7.2.1 **Standard sodium tetraphenylborate** (STPB) (CAS No. 143-66-8), solution: 8,754 g in 800 ml deionized water.

Add 10 g to 12 g of aluminium hydroxide, stir 10 min and filter. Add 2 ml of 20 % NaOH solution to the filtrate and dilute to 1 l with deionized water.

A.7.2.2 **Quaternary ammonium salt** (QAS) (CAS No. 57-09-0), solution: 1,165 g hexadecyltrimethyl ammonium bromide per 500 ml deionized water.

A.7.2.3 **Sodium hydroxide** (CAS No. 1310-73-2), solution: 20 % mass fraction in deionized water.

DANGER — NaOH is a strong caustic alkaline chemical. Avoid skin contact.

A.7.2.4 **Bromophenol blue** (CAS No. 115-39-9), indicator: 0,04 g tetrabromophenolsulfonphthalein per 3 ml 0,1 mol/l NaOH. Dilute to 100 ml with deionized water.

A.7.2.5 **Deionized** or **distilled water**.

A.7.2.6 **Graduated pipettes**, 2 ml (TD) graduated in 0,01 ml subdivisions, 5 ml (TD) and 10 ml (TD).

A.7.2.7 **Graduated cylinders**, capacity 25 ml (TD) and 100 ml (TC).

A.7.2.8 **Beakers**, capacity 250 ml.

A.7.2.9 **Funnel**.

A.7.2.10 **Filter paper**.

A.7.3 Procedure

A.7.3.1 Place the proper amount of filtrate into a 100 ml graduated cylinder, using Table A.4 to determine sample size. Be sure to use a pipette to measure the amount of filtrate.

Table A.4 — Filtrate volumes to be used at various KCl concentrations

KCl concentration range		**K$^+$ in filtrate**	**Filtrate volume to use**
kg/m^3	(lb/bbl)	mg/l	ml
0,5 to 3,0	0,18 to 1,05	263 to 1 575	10,0
3,0 to 6,0	1,05 to 2,1	1 575 to 3 150	5,0
6,0 to 20,0	2,1 to 7,0	3 150 to 10 500	2,0

A.7.3.2 Add 4 ml of NaOH solution (20 % mass fraction; measured with a 5 ml pipette), 25 ml of STPB solution (measured with a 25 ml graduated cylinder), and enough deionized water to bring the level of the solution to 100 ml mark.

A.7.3.3 Mix and allow to stand 10 min.

A.7.3.4 Filter into a 100 ml graduated cylinder. If the filtrate is cloudy, re-filter the solution.

A.7.3.5 Transfer 25 ml of the above filtrate (measured with a 25 ml graduated cylinder) into a 250 ml beaker.

A.7.3.6 Add 10 drops to 15 drops of bromophenol blue indicator.

A.7.3.7 Titrate with QAS solution until colour changes from purple-blue to light blue.

It is important to check the concentration of QAS solution versus the STPB solution at monthly intervals. To determine the equivalent QAS, dilute 2 ml of the STPB solution in a titration vessel with 50 ml deionized water. Add 1 ml of 20 % NaOH solution and 10 drops to 20 drops of the bromophenol blue indicator. Titrate with the QAS solution until colour changes from purple-blue to light blue.

The ratio, $R_{\text{QAS/STPB}}$, of the concentration of QAS to that of STPB is calculated as given in Equation (A.14):

$$R_{\text{QAS/STPB}} = \frac{V_{\text{QAS}}}{2} \tag{A.14}$$

where V_{QAS} is the QAS volume, expressed in millilitres.

If the ratio is other than 4,0 ± 0,5, calculate a correction factor, k_{cor}, for use in the calculation of the potassium ion concentration, $c_{\text{K,VQAS}}$, expressed in milligrams per litre, as given in Equation (A.15):

$$k_{\text{cor}} = 8/V_{\text{QAS}} \tag{A.15}$$

A.7.4 Calculation

If the ratio falls in the 4,0 ± 0,5 range, the potassium ion concentration, $c_{\text{K,VQAS}}$, in milligrams per litre, is calculated using V_{QAS} as given in Equation (A.16):

$$c_{\text{K,VQAS}} = \frac{1\,000 \times \left(25 - V_{\text{QAS}}\right)}{V_{\text{f}}} \tag{A.16}$$

where V_{f} is the filtrate volume, expressed in millilitres.

If a correction factor is necessary, calculate the potassium ion concentration in the filtrate, $c_{\text{K,VQAS}}$, expressed in milligrams per litre, as given in Equation (A.17):

$$c_{\text{K,VQAS}} = 1\,000 \left[\frac{25\text{-}\left(k_{\text{cor}} \times V_{\text{QAS}}\right)}{V_{\text{f}}} \right] \tag{A.17}$$

Calculate the potassium chloride concentration in the filtrate, $c_{\text{f,KCl,A}}$, expressed in kilograms per cubic metre, as given in Equation (A.18) [or $c_{\text{f,KCl,B}}$, expressed in pounds per barrel, using Equation (A.19)]:

$$c_{\text{f,KCl,A}} = \frac{c_{\text{K,VQAS}}}{525} \tag{A.18}$$

$$c_{\text{f,KCl,B}} = \frac{c_{\text{K,VQAS}}}{1\,500} \tag{A.19}$$

Annex B
(informative)

Shear strength measurement using shearometer tube

B.1 Principle

B.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, therefore, result in loss of circulation. High shear strength can also cause difficulties in logging, perforating and other “downhole” operations.

B.1.2 The following technique 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.

B.2 Apparatus

B.2.1 **Stainless steel shearometer tube**, with 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.

B.2.2 **Platform**, for weights.

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

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

B.3 Procedure

B.3.1 The shear tube and platform are placed and balanced carefully on the surface of the aged sample cooled to room temperature. It can be necessary to shift the weights on the platform to assure 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 placing the shear tube in place for the test.

B.3.2 Sufficient weights are placed carefully 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 the length of the tube.

B.3.3 Record the total mass in grams, which includes 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 facilitates this measurement. The length of the tube minus the exposed length equals the submerged portion.

B.4 Calculation

B.4.1 Calculate the shear strength, γ_A, expressed in pascals, as given in Equation (B.1) [or γ_B, expressed in pounds per square foot, as given in Equation (B.2)]:

$$\gamma_A = \frac{4,40 \times (m_{st} + m_{tot})}{l_A} - 1,02\rho_{df,A} \qquad \text{(B.1)}$$

where

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 centimetres;

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

B.4.2 Calculate the shear strength, γ_B, expressed in pounds per square foot, as given in Equation (B.2):

$$\gamma_B = \frac{3,61 \times (m_{st} + m_{tot})}{l_B} - 0,256\rho_{df,B} \qquad \text{(B.2)}$$

where

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

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

Annex C
(informative)

Resistivity

C.1 Principle

Control of the resistivity of a drilling fluid and drilling fluid filtrate can be desirable to better evaluate formation characteristics from electric logs.

C.2 Apparatus

C.2.1 Direct-reading resistivity meter, or similar resistivity meter.

Follow manufacturer's instructions for current source, calibration, measurement and calculations.

C.2.2 Calibrated resistivity cell.

C.2.3 Thermometer, reading 0 °C to 105 °C (32 °F to 220 °F).

C.2.4 Bottle brush, suitable for size and type of cell.

C.2.5 Laboratory detergent solution, appropriate for cleaning metal or plastic surfaces.

C.3 Procedure

C.3.1 Fill the clean, dry resistivity cell with freshly stirred drilling fluid or drilling fluid filtrate. No air or gas should be entrained in the sample.

C.3.2 Connect cell to meter.

C.3.3 Measure the resistivity in ohm metres (direct-reading) or resistance in ohms (not direct-reading). Meter or manufacturer's instructions will indicate type of reading.

C.3.4 Measure the sample temperature to the nearest 0,5 °C (1 °F).

C.3.5 Clean the cell. Scrub with brush and detergent if necessary. Rinse with distilled water and allow to dry.

C.4 Calculation

C.4.1 Report the drilling fluid resistivity, r_{df}, or filtrate resistivity, r_{f}, in ohm metres, to the nearest 0,01 $\Omega \cdot \mathrm{m}$.

C.4.2 Report the sample temperature in degrees Celsius (Fahrenheit).

C.4.3 If the reading, R_r, is in ohms, convert to ohm·metres as given in Equations (C.1) and (C.2):

$$r_\mathrm{df} = R_\mathrm{r} \times K \tag{C.1}$$

$$r_\mathrm{f} = R_\mathrm{r} \times K \tag{C.2}$$

where

K is the cell constant, expressed in metres squared per metre;

R_r is the meter reading, expressed in ohms.

Annex D
(informative)

Removal of air or gas prior to testing

D.1 Principle

The majority of drilling fluids require no special equipment to remove entrained air or gas prior to testing. Usually, gentle agitation together with a few drops of an appropriate defoamer are all that is necessary. Stirring with a spatula or pouring back and forth is sufficient in most cases. When a drilling fluid is encountered that retains air or gas after the preceding steps have been taken, the following procedure can be followed to de-aerate the drilling fluid.

NOTE If drilling fluid density is the only property desired, the pressurized fluid density balance described in Clause 5 can be used.

D.2 Apparatus

D.2.1 **Device**, which can be evacuated.

D.2.2 **Defoamer**, commercial liquid drilling fluid.

D.3 Procedure

D.3.1 Fill clean, dry reservoir about one-half full with the air-cut drilling fluid.

D.3.2 Add several drops of defoamer to the drilling fluid surface.

D.3.3 Insert stirrer and cap; cover with gasketed lid.

D.3.4 Affix vacuum line from the pump to the instrument to hold about 83 kPa (620 mm Hg; 24,4 in Hg) vacuum.

D.3.5 Increase the vacuum to 10 kPa to 16 kPa (75 mm Hg to 120 mm Hg; 3,0 in to 4,7 in Hg) and proceed according to the manufacturer's instructions.

D.3.6 When drilling fluid has been de-aerated, partially relieve vacuum to about 50 kPa to 65 kPa (375 mm Hg to 490 mm Hg; 14,8 in to 19,3 in Hg) vacuum and observe drilling fluid for air bubbles.

D.3.7 If de-aeration is not sufficient, repeat D.3.4 to D.3.6 until air is removed.

D.3.8 With cylinder on end, relieve vacuum completely and remove drilling fluid sample for testing.

Annex E
(informative)

Drill pipe corrosion ring coupon

E.1 Principle

E.1.1 The placement of corrosion test rings in the drill string is one of the more common techniques used to evaluate the corrosiveness of drilling fluid environments on the drill string and other steel equipment. Removal and examination of these rings after a period of exposure downhole can be highly informative as to the corrosiveness of the drilling fluid as well as to the type of corrosion encountered. An examination of scales and pits on the exposed rings gives clues as to the cause of the corrosion, thus aiding in choosing proper remedial action.

E.1.2 The ring technique is specifically designed for detection of the type of corrosion characterized by metal loss, whether it be localized pitting or generalized attack. The test ring is not designed to give information relating to hydrogen embrittlement, stress-corrosion cracking or other forms of fracture formation, except in the manner in which pitting may relate to these failures.

E.2 Reagents and apparatus

E.2.1 **Hydrochloric acid** (CAS No. 7647-01-0), inhibited, mass fraction of 15 % in distilled water.

DANGER — HCl is a strong and toxic acid.

E.2.2 **Acetone**, anhydrous (CAS No. 67-64-1).

E.2.3 **Methanol** (CAS No. 67-56-1).

E.2.4 **Petroleum ether** (CAS No. 8002-05-9).

E.2.5 **Deionized** or **distilled water.**

E.2.6 **Detergent solution**.

E.2.7 **Corrosion ring**.

a) Ring construction:

   The ring-type drill-string corrosion coupon, or corrosion ring, should be machined to fit in the tool box recess at the end of the pin and should have a bore the same as that of the tool joint to minimize turbulence.

b) Ring composition:

   Ideally, the ring should be made from steel identical to that of the tool joint in which it is placed to avoid galvanic corrosion. However, such a requirement is impractical and the use of a steel that is similar in chemical composition, such as AISI 4130, is recommended. The grade of steel used should be identified on the report form. The rings are normally cut from tubes that have not been quenched and tempered. The similarity in composition of the 4130 steel and the tool joint should be adequate to minimize galvanic effects and provide useful data.

c) Ring marking:

The rings should be stencilled with a serial number for permanent identification.

d) Ring preparation (by the supplier):

The rings should be scrubbed with a stiff fibre-bristle brush and detergent solution and rinsed with clean water and with anhydrous acetone or methanol. Allow to dry, weigh to nearest milligram and record this mass on the report form. Store the ring in a dry container, such as a desiccator, to prevent corrosion. The corrosion rings should be shipped to the field in sealed envelopes or wrappers to minimize atmospheric corrosion.

E.2.8 **Ultrasonic bath**, (preferred) or **brush**, fibre-bristle, or fine-grade (000) steel wool.

E.2.9 **Gloves,** acid resistant, part of personal protective equipment when handling strong acids.

E.2.10 **Iron sulfide test solution — Acid arsenic test**.

E.3 Procedure

E.3.1 Drill-pipe corrosion rings should be kept in the drill string for a minimum of 40 h (a normal time for exposure is 100 h). Exposure periods of less than 40 h should not be used because initial corrosion rates can be unusually high and can give misleading data. The ring is usually placed in the tool joint at the top of the first stand above the drill collars and can be left in the drill string for more than one bit run. An additional ring can be placed in the kelly-saver sub to monitor corrosion at that point. Care should be taken to ensure that the box recess is clean to prevent interference with proper make-up of the joint and to avoid damage to the ring. In some instances, specially manufactured subs have been used for the ring placement in the string. During installation, the ring should be handled with clean, dry gloves.

E.3.2 The drill-pipe corrosion coupon form should be filled out completely. At a minimum, each form should have a space for ring material, drilling fluid properties, type of corrosion, location of ring in the drill string, initial mass, time, depth in, depth out, ring number, colour of scale and any other information of significance in the specific test. The form may be printed on a mailing envelope for the ring or on a separate form to be enclosed with the ring.

E.3.3 The drilling fluid residue should be removed from the coupon by wiping with a cloth when the ring is pulled from the drill string. The ring should be examined for severity of corrosion or mechanical damage. If severe corrosion is evident, the cause of the corrosion should be determined promptly so remedial action can be taken. Following visual observation, place the coupon in the original envelope or wrapper containing vapour-phase corrosion inhibitor for return to the laboratory.

E.3.4 The recommended cleaning solution is a detergent solution, not acetone or petroleum ether. Prior to cleaning for weighing, a spot test should be made for corrosion by-products and mineral scale. For example, the surface can be examined qualitatively for sulfides by the acid arsenic (iron sulfide test solution) test. The rings should be cleaned with a detergent solution and a stiff, fibre-bristle brush. It can be necessary to dip the ring for 5 s to 10 s in inhibited 10 % to 15 % hydrochloric acid solution one or more times to remove corrosion products. The ring should be scrubbed with detergent solution after each acid dip. Rinse thoroughly with clean water and then with anhydrous acetone or methanol. Allow to dry prior to weighing. Very abrasive materials or strong, uninhibited acids should not be used. An ultrasonic bath can be useful in cleaning the rings.

For the inhibited 10 % to 15 % hydrochloric acid, several inhibitive additives may be used to prevent further corrosion of the ring after cleaning with acid. These include chemicals of the classes propargyls, acetylenics, pyridines and amines. Other appropriate chemicals may be suggested by the manufacturer of the corrosion rings.

E.3.5 After the pre-weighed drill pipe corrosion coupon has been properly cleaned and the corrosion film and type of attack noted, the ring should be re-weighed to the nearest milligram and the mass loss determined. If significant loss of metal due to mechanical damage is evident, it should be noted and taken into consideration in evaluation of the ring. The corrosion rate may be reported as $kg/m^2 \cdot year$ or mm/year (lb/ft^2 or mils/year). Equations for calculating for corrosion rate are given in Clause E.5.

E.4 Comments on visual examination

E.4.1 If visual corrosion is evident, it is normally detectable as pitting corrosion. Uniform attack or general corrosion can best be determined by a mass-loss measurement. Mechanical damage to the ring is most often evidenced by cuts or dents on the outer surfaces of the ring. In some cases, the ring exhibits a series of dents and worn spots, indicating considerable movement of the ring in the box recess.

E.4.2 In assessing the magnitude of the corrosion rates as calculated from mass loss measurements, it should be remembered that the rate is also influenced by the erosive effects of the drilling fluid. Since the bore of the ring is exposed to the drilling fluid pumped down the drill pipe, the loss of metal includes that removed by erosion as well as from corrosion. Loss from erosion can be substantial when the drilling fluid contains a high concentration of sand.

E.4.3 Examination of the ring can reveal a few deep pits with a relatively low mass loss. This condition would indicate a rather severe corrosion problem, even though the calculated corrosion rate is considered low.

E.5 Calculation

E.5.1 The corrosion rate, q_A, expressed in kilograms per square metre per year, is calculated according to Equation (E.1) and as q_B, expressed in kilograms per square foot per year, as given in Equation (E.2):

$$q_\mathrm{A} = \left(\frac{\Delta m}{1\times 10^6}\right)\left(\frac{1\times 10^4}{A_\mathrm{A}}\right)\left(\frac{8\,760}{t}\right) = 87{,}6\times\left(\frac{\Delta m}{A_\mathrm{A}\cdot t}\right) \tag{E.1}$$

where

Δm is the mass loss, expressed in milligrams;

A_A is the area, expressed in square centimetres;

t is the exposure time, expressed in hours.

$$q_\mathrm{B} = \left(\frac{\Delta m}{453\,600}\right)\left(\frac{144}{A_\mathrm{B}}\right)\left(\frac{8\,760}{t}\right) \tag{E.2}$$

where

Δm is the mass loss, expressed in milligrams;

A_B is the area, expressed in square inches;

t is the exposure time, expressed in hours.

NOTE 1 Total surface area of the ring is used in these calculations.

NOTE 2 Time used is based on total time in the drill string.

NOTE 3 Equations (E.3) to (E.7) give the conversion rates between the various units for steel coupons (with a relative density of 7,86):

$$\text{mils/y} = 5{,}01 \times q_\mathrm{A} \tag{E.3}$$

$$\text{mils/y} = 24{,}6 \times q_\mathrm{B} \tag{E.4}$$

$$\text{mm/y} = 0{,}127 \times q_\mathrm{A} \tag{E.5}$$

$$\text{mm/y} = 0{,}621 \times q_\text{B} \tag{E.6}$$

$$\text{lb/ft}^2\text{/y} = 1{,}61 \times (\text{mm/y}) \tag{E.7}$$

NOTE 4 Corrosion rings available from drilling fluid service companies or corrosion test laboratories are generally supplied with a multiplication factor that includes the metal density and surface area of the ring. Thus, it is necessary only to multiply the mass loss divided by the total exposure time in the string by the supplied factor to obtain the corrosion rate.

Annex F
(informative)

Sampling, inspection and rejection

F.1 General

The following procedure provides the sampling, inspection and rejection method for materials conforming to ISO 13500 [1]. It is applicable to barite, hematite, bentonite, nontreated bentonite, attapulgite, sepiolite, technical-grade low-viscosity carboxymethylcellulose, technical-grade high-viscosity carboxymethylcellulose and OCMA-grade bentonite.

F.2 Sampling of powdered material in packages

F.2.1 The number of samples taken to be combined for the test sample (see Clause F.4) should be 15 samples of 0,5 kg (1 lb) or more per lot.

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

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

a) A sample weighing at least 0,5 kg (1 lb) should be taken from the top of each package.

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 takes a core of material for essentially the entire length of the package.

F.3 Sampling of powdered material in bulk

F.3.1 Tube sampling is used in the collection of samples from storage containers holding 25 000 kg to 100 000 kg (25 tonnes to 100 tonnes), with 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 of sufficient length to permit taking a sample essentially from the top to the bottom of the mass being sampled.

F.3.2 Fifteen samples should be taken from each lot (considering each container as one lot). 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 For containers holding less than 20 000 kg (20 tonnes), at least one sample should be taken from each container up to lots of 10 containers, and the total number of samples taken from each lot of 100 000 kg (100 tonnes) 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 one measurement on his test sample. A control or known reference sample should be tested in the same manner, at the same time as part of the test series. If this test result is within the acceptable range, the test results are considered valid.

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 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 make claim for retest within one week of the date of the original test report.

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

Annex G
(informative)

Rig-site sampling

G.1 General

The following is a recommended procedure for obtaining test samples at the rig site from specific loads. 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) diameter and 30 cm (12 in) long.

G.2.1.3 **Suitable container**, e.g. a tie-bag, so constructed as to retain 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 as to be taken when approximately 1/4, 1/2 and 3/4 of the load has been transferred.

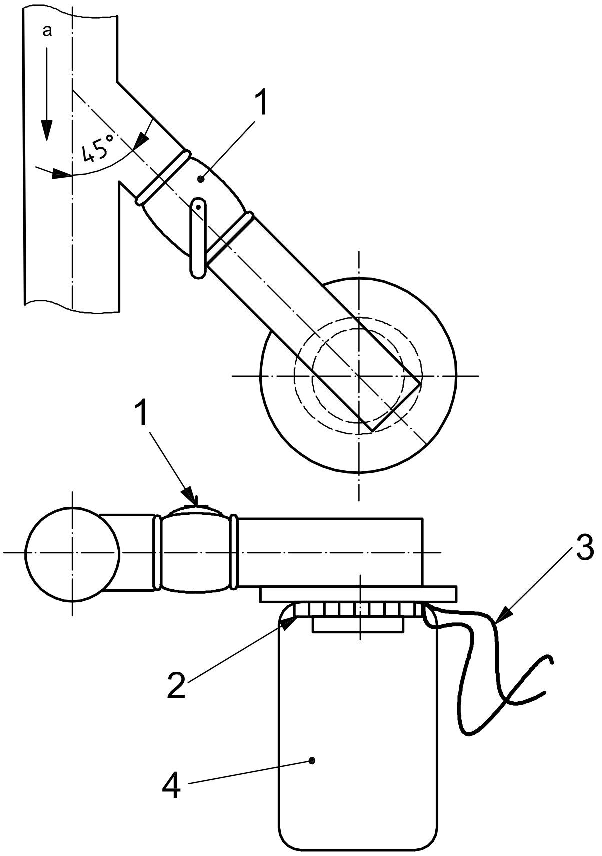
NOTE One 4 kg to 7 kg (9 lb to 15 lb) sample can be taken with the recognition that there is a chance of obtaining a non-representative sample. The greatest potential for error in sampling is contamination from prior shipments of material in the same vessel. This potential error can be minimized by following the instructions closely. Do not take samples from the very top surface or from the very first material out of the bottom. A second potential for error in sampling is 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 Before each individual sample is taken, open the valve momentarily to flush out any material trapped in the side pipe. Tie a clean canvas bag to the sampler and fill it with one valve-opening operation.

NOTE Partial closing of the air-jet valve on the bottom outlet prior to taking the sample often helps to 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 DN50 cm (2 in) ball valve in DN50 cm (2 in) side pipe
2 bead or lip to hold bag
3 drawstring
4 14 cm × 30 cm (5,5 in × 12 in) canvas bag

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, so constructed as to retain all fines.

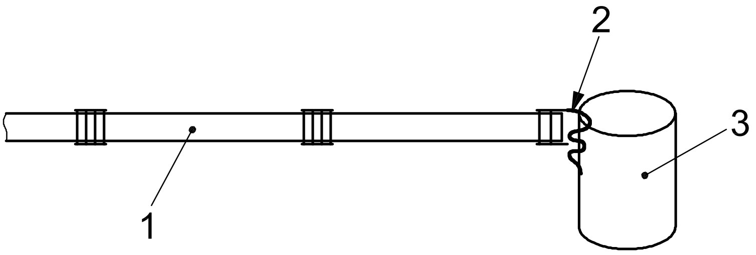
G.3.2 Procedure

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)

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

H.1 General

This annex covers calibration of field testing equipment not covered as part of a test procedure.

H.2 Calibration of apparatus

H.2.1 Volumetric glassware

Calibration of pipettes, graduated cylinders and similar equipment is generally performed by the glassware supplier and can be part of the purchase specification. Where deemed important, glassware users should obtain documented evidence of glassware calibration from the supplier. Calibration can be checked gravimetrically. Periodic re-calibration is not required. See Clause H.3 for the procedure for calibration of graduated cylinders.

H.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 initial service and then periodically, depending on the importance of the measurement and the stability of the thermometer. See H.3.2 for the procedure for calibration of thermometers.

H.2.3 Viscometer

Calibrate viscometers used in field testing against traceable standard viscosity fluids. Viscometers should be calibrated before being put into initial 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. See H.3.3 for the procedure for calibration of viscometers.

H.2.4 Drilling fluid balance

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

H.2.5 Retort-kit cup

Calibrate retort-kit cup as specified in the test procedure using deionized water. The required frequency is set prior to its being put into initial service and then periodically or as specified by the operator, drilling fluid company or other interested party. See H.3.4 for the procedure for calibration of retort-kit cups.

H.3 Procedure for calibration

H.3.1 Liquid receivers

H.3.1.1 Allow the receiver and distilled water to reach ambient temperature. Record the temperature to the nearest 0,5 °C (1 °F).

H.3.1.2 Place the clean, empty receiver with its base on the balance and tare to zero.

H.3.1.3 While the receiver is on the balance, fill it with distilled water to the various graduations marks: 2 ml, 4 ml, 6 ml, 8 ml, 10 ml for the 10 ml receiver; 4 ml, 8 ml, 12 ml, 16 ml, 20 ml for the 20 ml receiver; and 10 ml, 20 ml, 30 ml, 40 ml, and 50 ml for the 50 ml receiver. Using a pipette or syringe, carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.

H.3.1.4 Record the mass, $m_{\mathrm{W},i}$ for each incremental volume of water at graduation mark i, to the nearest 0,01 g.

H.3.1.5 Calculate the volume, V_i, of the receiver at graduation mark i, as given in Equation (H.1):

$$V_i = \frac{m_{\mathrm{W},i}}{\rho_{\mathrm{W}}} \tag{H.1}$$

where

$m_{\mathrm{W},i}$ is the mass of water at graduation mark i, expressed in grams;

ρ_{W} is the water density, expressed in grams per cubic centimetre, at test temperature; see Table H.1.

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

Temperature		Density	Temperature		Density
°C	(°F)	g/ml	°C	(°F)	g/ml
15,0	59,0	0,999 1	25,5	77,9	0,996 9
15,5	59,9	0,999 1	26,0	78,8	0,996 8
16,0	60,8	0,999 0	26,5	79,7	0,996 6
16,5	61,7	0,998 9	27,0	80,6	0,996 5
17,0	62,6	0,998 8	27,5	81,5	0,996 4
17,5	63,5	0,998 7	28,0	82,4	0,996 2
18,0	64,4	0,998 6	28,5	83,3	0,996 1
18,5	65,3	0,998 5	29,0	84,2	0,995 9
19,0	66,2	0,998 4	29,5	85,1	0,995 8
19,5	67,1	0,998 3	30,0	86,0	0,995 6
20,0	68,0	0,998 2	30,5	86,9	0,995 5
20,5	68,9	0,998 1	31,0	87,8	0,995 3
21,0	69,8	0,998 0	31,5	88,7	0,995 2
21,5	70,7	0,997 9	32,0	89,6	0,995 0
22,0	71,6	0,997 7	32,5	90,5	0,994 9
22,5	72,5	0,997 6	33,0	91,4	0,994 7
23,0	73,4	0,997 5	33,5	92,3	0,994 5
23,5	74,3	0,997 4	34,0	93,2	0,994 4
24,0	75,2	0,997 3	34,5	94,1	0,994 2
24,5	76,1	0,997 1	35,0	95,0	0,994 0
25,0	77,0	0,997 0			

H.3.2 Thermometers

H.3.2.1 Place the thermometer to be calibrated side-by-side with a standard thermometer in a constant-temperature bath [or suitable container of 4 l (1 gal) or more] filled with water on a countertop in a room where temperature is relatively constant and allow to equilibrate for 30 min.

H.3.2.2 Read both thermometers and record the readings.

H.3.2.3 Repeat the readings at 5 min intervals to obtain at least four sets of readings.

H.3.2.4 Calculate the average reading for each thermometer.

H.3.2.5 Set adjustable thermometers to read the same as the standard thermometer. For other thermometers, refer to H.3.2.6.

H.3.2.6 Calculate the value, C_{th}, of the thermometer correction to be added to the working thermometer reading as given in Equation (H.2):

$$C_{\text{th}} = R_1 - R_2 \tag{H.2}$$

where

R_1 is the average reading for the standard thermometer;

R_2 is the average reading for the working thermometer.

EXAMPLE 1 Thermometer correction determination:

In SI units:

$R_1 = 23{,}9\ °\text{C}$

$R_2 = 24{,}2\ °\text{C}$

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

In USC units:

$R_1 = 75{,}0\ °\text{F}$

$R_2 = 75{,}5\ °\text{F}$

$C_{\text{th}} = 75{,}0\ °\text{F} - 75{,}5\ °\text{F} = -\ 0{,}5\ °\text{F}$

EXAMPLE 2 Calculation of the corrected reading, $R_{2,\text{cor}}$

$C_{\text{th}} = -\ 0{,}3\ °\text{C}$

$R_2 = 25{,}0\ °\text{C}$

$R_{2,\text{cor}} = 25{,}0\ °\text{C} + (-\ 0{,}3\ °\text{C}) = 24{,}7\ °\text{C}$

H.3.2.7 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.

H.3.3 Viscometers

H.3.3.1 Obtain a certified calibration fluid or fluids with a chart (viscosity versus temperature) or viscosity data table, to cover the range of interest: for example, 50 mPa•s (50 cP), 100 mPa•s (100 cP), and so forth. 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 normally varies slightly from lot to lot.

NOTE Most calibration fluids are labelled in centipoise. 1 mPa•s = 1 cP.

H.3.3.2 Clean and dry the viscometer bob, rotor and cup.

NOTE Water contaminates the standard fluid.

Place the viscometer and the fluid side-by-side on a countertop in a room with reasonably constant temperature [temperature variation, $\Delta\theta < 3$ °C/hr (< 5 °F/hr)]. Allow to stand at least 2 h to equilibrate.

H.3.3.3 Operate the viscometer in air for 2 min to 4 min to loosen bearings and gears.

If rotor wobbles excessively, correct or replace rotor.

H.3.3.4 Fill cup to scribed line with calibration fluid and place on meter stage. Move the stage until fluid level is to inscribed line on rotor sleeve.

H.3.3.5 Place a thermometer [capable of measuring to ± 0,1 °C (± 0,2 °F)] into the fluid and hold or tape it to prevent breakage. Operate the viscometer at a low-speed setting until the thermometer reading is stable to within 0,1 °C (0,2 °F) per 30 s. Record temperature reading.

H.3.3.6 Operate viscometer and take readings at 600 r/min and 300 r/min. Estimate readings to nearest 0,5 dial unit. If needed, use a magnifying glass.

H.3.3.7 Using the temperature-viscosity chart or table supplied with the calibration fluid, determine certified viscosity to the nearest 0,5 mPa•s. Compare the 300 r/min dial reading to standard viscosity and record deviation (plus or minus). Divide the 600 r/min dial reading by 2,00, compare to standard viscosity, and record deviation.

H.3.3.8 Deviations exceeding 1,5 units are not acceptable. If deviation exceeds these tolerances, adjust or repair the viscometer.

H.3.3.9 Record the viscometer serial number, date and deviation. Mark the viscometer with the date of calibration and indication of calibration status.

H.3.4 Retort-kit cups

H.3.4.1 Allow retort cup, lid and distilled water to reach ambient temperature. Record the temperature to the nearest 0,5 °C (1 °F).

H.3.4.2 Place the clean, empty retort cup and lid on the balance and tare to zero.

H.3.4.3 Fill retort cup with distilled water. Place lid on the cup. Rotate lid to obtain proper fit. Be sure a small excess of water flows out of the hole in the lid. Wipe excess water from lid; avoid wicking out water.

H.3.4.4 Place filled retort cup with lid on the previously tared balance. Record mass of water to the nearest 0,01 g.

H.3.4.5 Calculate retort cup volume, V_{RC}, expressed in millilitres, using the density of water at ambient temperature (see Table H.1), as given by Equation (H.3):

$$V_{\mathrm{RC}} = \frac{m_{\mathrm{W}}}{\rho_{\mathrm{W}}} \tag{H.3}$$

where

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

ρ_{W} is the water density, expressed in grams per cubic centimetre, at test temperature (see Table H.1).

H.3.5 Filter press gasket and O-rings

H.3.5.1 Gaskets or O-rings for the low-pressure, low-temperature filter press shall have an inside diameter between 75,8 mm (2,99 in) and 76,9 mm (3,03 in).

H.3.5.2 Check the gasket or O-ring using a gauge having these diameters.

H.3.5.3 Do not use gaskets or O-rings outside these tolerances for filter loss testing.

Annex I
(normative)

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

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

I.1 Principle

I.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.

I.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.

I.2 Safety considerations

I.2.1 The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cell available: those with threaded end caps and those with set-screw-secured end caps. Among these cells 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.

I.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.

I.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.

I.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.

I.2.5 For safe operation of the hydraulic pressurization system, follow the instructions below.

I.2.5.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before

a) attempting to disconnect pressure hose from cell at quick coupler,

b) attempting to remove cell from heating jacket,

c) moving the PPA,

d) refilling the hydraulic pump,

e) performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

I.2.5.2 After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

I.2.5.3 Make sure, when assembling the cell, that the end cap set screws are properly aligned and tightened.

I.2.6 For safe pneumatic pressurization of backpressure receiver, follow the instructions below.

I.2.6.1 Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it shall be supplied in an approved nitrogen gas cylinder or the nitrogen supply system should be built into the laboratory. Nitrogen cylinders should be secured to meet safety standards. CO_2 is normally supplied in small cartridges pressurized to about 6 200 kPa (900 psi). They are primarily used for field operations.

DANGER — Do not allow CO_2 cartridges to be heated or exposed to fire. They can explode if overheated.

DANGER — Do not use nitrous oxide cartridges as pressure sources for HT/HP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas-train carbonate analysis.

I.2.6.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

I.2.6.3 Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings and hoses should be kept in good condition and leaks should be 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.

I.2.6.4 When pressurizing the backpressure assembly, always open the supply pressure first, then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the apparatus rating or relief-valve settings. When relieving backpressure, shut the supply pressure, bleed the pressure from the system, and then back out the regulator T-screw.

I.2.7 For safe heating, follow the instructions below.

I.2.7.1 Caution should be exercised to avoid injury while operating the PPA, which becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

I.2.7.2 The practice of removing the cell and cooling it with water is dangerous and should be avoided. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell or by accidentally dropping the cell.

I.2.8 For safe electrical operation, follow the instructions below.

I.2.8.1 Make sure that the electrical source is fused and grounded (earthed). Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

I.2.8.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

I.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.

I.2.9.1 Cell material shall be compatible with the test samples.

I.2.9.2 Do not use cells that show signs of severe pitting or stress cracking.

I.2.9.3 Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

I.2.9.4 Use only undamaged, hardened steel set screws. Others are unsafe.

I.3 Apparatus — Permeability-plugging apparatus (PPA) with set-screw-secured end caps

I.3.1 PPA cell.

I.3.1.1 There are three different types of cell available that use set-screw-secured end caps. Those of current and recent manufacture are rated at either 13 800 kPa (2 000 psi) or 12 420 kPa (1 800 psi).

There are still a number of older cells in use which are stamped “2 500 psi”. In 1996, the rating on these cells was reduced to “1 800 psi” (12 420 kPa). Unless the user can absolutely verify that the cell 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.

DANGER — Follow the manufacturer’s recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

I.3.1.2 The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top and the filtrate collected from the top. A small hydraulic hand-pump applies the cell pressure. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

I.3.1.3 The PPA can use any one of a number of filtration media, including porous ceramic or sintered metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from 100 millidarcy to 100 darcy. The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provides the user with a greatly improved picture of what is happening downhole.

To improve the uniformity of test conditions, and the repeatability of results, the disks may be classified utilizing the user’s own flow test procedure or that which is outlined in I.3.2.6.

I.3.1.4 Test pressures are limited by the safety limits of the cell as specified by the manufacturer; usually either 12 420 kPa (1 800 psi) or 13 800 kPa (2 000 psi) at some defined temperature. If backpressure is used in the test, it can be necessary to reduce the test pressure to avoid exceeding the pressure limit of the cell and thus damaging the cell.

I.3.1.5 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.

I.3.1.6 For temperatures above 93 °C (200 °F), the backpressure receiver shall be pressurized to prevent boiling of the filtrate. The standard backpressure receiver uses a CO_2 pressurizing source to provide the backpressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

I.3.1.7 The PPA cell is encased in a thermostatically controlled aluminium heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 W.

I.3.2 Filter medium, disks of any porous material such as ceramic, sintered metal, or resin-coated sand, graded sands, or core samples.

I.3.2.1 Standard disk thickness is 6,5 mm (0,25 in) but, with adapters, thicker disks can be used. A new disk is required for each test. For water-based drilling fluid samples, the disks shall be soaked in fresh water or brine until saturated, at least 5 min to 30 min prior to use. Vacuum saturation shall be used for filter media with low porosity and permeability.

There is unavoidable variability in the pore throat sizes of the ceramic disks normally used in these tests. Consequently, when running comparative tests, it is recommended that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality-control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with fresh water to further classify the disk.

I.3.2.2 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores can have some variability in porosity and permeability, and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 6,5 mm (0,25 in*)* thick. With modification of the cylinder, 25,5 mm (1 in) cores can also be used.

I.3.2.3 Resin-coated sand can be made into a solid disk, selecting the sand grain size to provide the desired permeability. The sand is heated at 150 °C (300 °F) for 1 h to 3 h in moulds slightly larger than the normal disk size, and either 6,5 mm (0,25 in) or 25,5 mm (1 in) thick. The moulds shall be coated with silicone grease prior to heating. Resin-coated sand disks can be manufactured to provide a substantial variation in pore throat size and permeability by varying the mesh sizes of the sands. Coarser sands can be used to provide a filter medium for testing lost-circulation material to be used to control seepage losses in severe fluid-loss environments.

I.3.2.4 Sintered metal disks or slotted metal disks can be used to simulate fractures or high permeability formations. In the evaluation of seepage-loss material needed to seal off a specific formation, the disk pore-throat size should be matched with that of the formation.

I.3.2.5 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed, and then weigh the amount of sand necessary to obtain that height. The sand bed shall be soaked with the base fluid prior to the test. If the user desires to run the test in the standard manner, with the filter medium at the top of the cell, resin-coated sand can be placed in the cell, heated for 1 h to 3 h at 150 °C (300 °F), cooled, and then inverted for the test.

I.3.2.6 Procedure for ceramic disk comparison: install disk in a PPA cell and fill the cell with water. Using the air permeability apparatus, with the upper cell valve closed, adjust the pressure on 207 kPa (30 psi) test gauge to 28 kPa to 31 kPa (4,0 psi to 4,5 psi). Open the valve on top of the cell and adjust pressure to 14 kPa ± 0,7 kPa (2,0 psi ± 0,1 psi). After opening the valve at the bottom of the cell, readjust pressure with the upper valve to 14 kPa ± 0,7 kPa (2,0 psi ± 0,1 psi). Measure time for 300 ml to pass through using a 500 ml graduated cylinder, timing the flow from the 100 ml mark to the 400 ml mark. If the PPT is used for comparison purposes, run several disks, classify the disks and use those of similar values.

I.3.3 Timer, accurate to 0,1 min over the test interval.

I.3.4 Thermometer, with scale up to 260 °C (500 °F).

I.3.5 Graduated cylinder, 25 ml (TC) or 50 ml (TC).

I.3.6 High-speed mixer.

I.4 Procedure for high-temperature, high-pressure (HTHP) filtration

I.4.1 Preheating the heating jacket

I.4.1.1 Connect the power cord to the proper voltage as indicated on the nameplate.

I.4.1.2 Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light illuminates when the heating-jacket temperature reaches the thermostat setting.

I.4.1.3 Readjust the thermostat to 6 °C (10 °F) over the desired test temperature.

I.4.2 Loading the filtration cell

I.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.

I.4.2.2 As received from the manufacturer, the PPA is equipped with valves that are rated to 260 °C (500 °F). If it becomes necessary to change any valves during the life of this apparatus, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

I.4.2.3 Loosen the set screws securing the end caps, then remove the caps from the cell by pulling them straight out, using the nipples and connected hardware as handles. If the cap is stuck and cannot be freed by rocking it gently, screw the cell-cap-removal tool into the seat for the nipple and press downward on the handle to remove it. Then unscrew the nipples from the caps and remove the piston from the cell.

I.4.2.4 Check the O-rings on the nipples, the floating piston, the cell body and the end caps, and replace any that are damaged or brittle [all O-rings should be replaced routinely after tests at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease completely around all of the O-rings, being especially careful to ensure that those on the piston are well lubricated.

I.4.2.5 Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely (the bottom of the cell, the inlet end, has a shorter recess than the top). Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

I.4.2.6 Fill the space above the piston with hydraulic oil to just above the end face.

I.4.2.7 Install the hydraulic end cap onto the bottom of the cell by pushing in on the backpressure ball on the nipple of the end cap on the pressure-inlet end of the cell to relieve the pressure, and allow the cap to slide into the cell more easily. Install and tighten the set screws.

Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

I.4.2.8 Connect the bottom nipple assembly to the pump hose and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

Steps 1.4.2.9 to 1.4.2.13 can be accomplished in the jacket that is being preheated, in an unheated jacket if one is available, or in a specially constructed stand.

I.4.2.9 Turn the cell upright and fill with approximately 275 ml of drilling fluid. This allows for expansion while heating. Do not exceed this amount.

For improved consistency in test results, stir drilling fluid for 5 min immediately before loading the cell.

I.4.2.10 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

I.4.2.11 Install the O-ring and set the selected ceramic disk or other filtering medium on top of it.

I.4.2.12 Position the top end cap in the cell, tighten the set screws snugly and close the valve on the top end cap.

Thermal expansion of the cell contents and of the hydraulic fluid causes the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the excess fluid periodically.

I.4.2.13 Install the cell in the heating jacket. Make sure that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

I.4.3 Pressurizing the cell

I.4.3.1 Refer to Table I.1 for the pressure corresponding to the test temperature and use the hydraulic pump to apply this pressure to the cell. If a manually operated pump is used, it should always be operated at about one stroke per second.

Filtration at temperatures above the boiling point of the fluid sample requires the use of the backpressure receiver to prevent vaporization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling.

Nitrogen manifolds having a 4 100 kPa (600 psi) gauge for backpressure need to be modified to attain 4 850 kPa (700 psi). However, tests are possible within the limits of the manifolds.

I.4.3.2 While the cell is heating, use the following procedure to prepare the backpressure receiver.

Check to ensure that the regulator T-screw has been rotated counter-clockwise enough to release all pressure. When the pressure has been released, the screw will turn freely.

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.

Verify that the pressure-relief valve on the CO_2 assembly and the filtrate drain valve are closed.

Set the backpressure assembly aside. It will be installed as described in I.4.3.4.

I.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as 1 h.

Table I.1 — Starting cell pressures and backpressures for various test temperatures

Temperature range θ		**Heating pressure or backpressure** p	
°C	°F	kPa	psi
Less than 95	200	0	0
95 to 150	200-300	690	100
151 to 175	301-350	1 050	150
178 to 190	351-375	1 400	200
191 to 205	376-400	1 725	250
206 to 218	401-425	2 420	350
219 to 232	426-450	3 100	450
233 to 246	451-475	3 800	550
247 to 260	476-500	4 850	700

I.4.3.4 When the cell is at the desired temperature and cell pressure stabilized, mount the backpressure receiver on the upper valve adapter. Secure the receiver with a retaining pin. Install the CO_2 pressurizing unit on top of the receiver. Lock the CO_2 pressurizing unit in place with the other retaining pin.

I.4.3.5 If a drain hose is used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space between the filtration medium and the backpressure receiver outlet, and the receiver valve, should be filled with the base fluid before starting the test. This ensures that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

I.4.3.6 See Table I.1 to determine the appropriate pressure for the backpressure receiver and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

I.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the backpressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the backpressure receiver.

I.4.4 Conducting the filtration test

I.4.4.1 A laboratory timer shall be used and filtrate shall be collected and its volume recorded at 1 min, 7,5 min and 30 min. Plotting these data against the square root of time provides a useful description of the spurt loss. If desired, samples can be taken more frequently, but none should be taken before 1 min. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

For improved definition of spurt loss, filtrate may be collected at 1 min, 5 min, 7,5 min, 15 min, 25 min and 30 min, and the cumulative filtrate volumes plotted versus the square root of the time.

I.4.4.2 Begin filtration by opening the valve between the cell and the backpressure receiver. Verify that both the cell pressure, as indicated on the pump gauge, and the backpressure are at desired levels. Adjust them as necessary throughout the test.

I.4.4.3 Cell pressure can be expected to decrease slightly as filtration and any leakage at the pump reduce the cell contents. The pump should be used as necessary to maintain the pressure at the desired level. If a manually operated pump is in use, actuate it at about one stroke per second.

I.4.4.4 After each interval, the filtrate shall be drained into the graduated cylinder from the backpressure receiver and the time and cumulative volume shall be recorded.

It is recommended to recover the filtrate directly from the backpressure receiver not from a drain hose attached to it. If a hose is used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

I.4.4.5 After 30 min, close the filtrate valve and drain any remaining filtrate from the backpressure receiver into the graduated cylinder. The total volume of the filtrate in the graduated cylinder shall be recorded.

I.5 Test conclusion and disassembly

I.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.

I.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.

DANGER — Extreme care should be exercised in cooling hot cells.

This procedure, as recommended, makes it difficult to perform more than one test in an 8 h work day with a single PPA. In the interest of improving productivity, users may want to design their own cell cooling procedures and apparatus. Safety should be the primary consideration in these designs.

I.5.3 Close the valve between the cell and the backpressure receiver.

I.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.

I.5.5 Release the backpressure by turning the T-screw on the regulator counter-clockwise until it turns freely.

I.5.6 Bleed the pressure from the backpressure receiver by opening the pressure relief valve on the CO2 unit. Open the drain valve on the backpressure receiver and collect the last few drops of filtrate in the graduated cylinder. After removing the locking pin and securing it, remove the CO2 assembly from the top nipple adapter. After removing its locking pin, remove the backpressure receiver.

I.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.

I.5.7.1 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 is obvious from the force required to move the piston.

I.5.7.2 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 sure that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

I.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.

I.5.9 Loosen the set screws securing the end cap and, using the valve and nipple adapter as a handle, pull the cap from the cell. If it is stuck, a rocking motion of the valve and nipple adapter can free it. If it cannot be freed in this manner, unscrew the valve and nipple adapter, install the cap removal tool in its place and use it to remove the cap.

I.5.10 The end cap should be removed with the cell in the vertical position and the filtration end facing upwards.

I.5.11 Remove the filter disk. Use a small knife, small screwdriver or similar thin-bladed tool to pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with fresh water, then measure and record its thickness and remarks concerning its composition and texture.

I.5.12 Pour the remaining fluid from the cell. Wash the inside of the cell with fresh water. 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.

I.5.13 Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the bottom end cap using the procedure outlined in I.5.9 and I.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.

I.6 Test reports

I.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, in millilitres, collected through each of the selected time periods.

I.6.2 Spurt loss

The spurt loss (2.4) 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 (I.2).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with I.4.4.1.

I.6.3 Calculation

Set the permeability-plugging test volume, V_{PPT}, expressed in millilitres, equal to two times the V_{30}, the filtrate volume after 30 min, expressed in millilitres, as given in Equation (I.1):

$$V_{\mathrm{PPT}} = 2\,V_{30} \tag{I.1}$$

Calculate the spurt loss, V_1, expressed in millilitres, as given in Equation (I.2):

$$V_1 = 2\left[V_{7,5} - \left(V_{30} - V_{7,5}\right)\right] = 2\left(2V_{7,5} - V_{30}\right) \tag{I.2}$$

where $V_{7,5}$ is the filtrate volume after 7,5 min, expressed in millilitres.

Calculate the static filtration rate (velocity of flow), v_{sf}, expressed in millilitres per square root of the minutes, as given in Equation (I.3):

$$v_{\mathrm{sf}} = \frac{2\left(V_{30} - V_{7,5}\right)}{\sqrt{t_2} - \sqrt{t_1}} = \frac{2\left(V_{30} - V_{7,5}\right)}{2{,}739} \tag{I.3}$$

where

t_1 is the time at initial reading, expressed in minutes;

t_2 is the time at final reading, expressed in minutes.

Observe that all three of these parameters, V_{PPT}, V_1 and v_{sf}, 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.

I.6.4 Test report for filter cake

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 J
(normative)

High-temperature/high-pressure filtration testing of water-based drilling fluids using the permeability-plugging apparatus and cells equipped with threaded end caps

J.1 Principle

J.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 content.

J.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 (HTHP) filter press used to evaluate these interactions through various types of 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.

J.2 Safety considerations

J.2.1 The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cell available: those with threaded end caps and those with set-screw-secured end caps. Among these cells are a total of five different pressure ratings. For safety, it is imperative that the operator knows 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.

J.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.

J.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.

J.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.

J.2.5 For safe operation of the hydraulic pressurization system, follow the instructions below.

J.2.5.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before

a) attempting to disconnect pressure hose from cell at the quick-coupler;

b) attempting to remove the cell from the heating jacket;

c) moving the PPA;

d) refilling the hydraulic pump;

e) performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

J.2.5.2 After refilling or repairing the hydraulic system, clean up any spilled oil. Oil left on floors is hazardous, and accumulations of spilled oil near the PPA are fire hazards.

J.2.5.3 Make sure that the O-rings in the end caps are properly seated when assembling the cell.

J.2.6 For safe pneumatic pressurization of the backpressure receiver, follow the instructions below.

J.2.6.1 Always use either nitrogen or carbon dioxide to pressurize the backpressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gas. If nitrogen is used, it should be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system should be built into the laboratory. Nitrogen cylinders should be secured to meet safety standards. CO_2 is normally supplied in small cartridges pressurized to about 6 200 kPa (900 psi). They are primarily used for field operations.

DANGER — Do not allow CO_2 cartridges to be heated or exposed to fire. They can explode if overheated.

DANGER — Do not use nitrous oxide cartridges as pressure sources for HT/HP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas-train carbonate analysis.

J.2.6.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

J.2.6.3 Repair or replace leaking hydraulic or pneumatic pressurization systems. Gauges, fittings and hoses should be kept in good condition, and leaks should be found and corrected. Periodically test the pressure-relief valve on the hydraulic pump to verify that it will function properly if excessive pressure develops. Never plug or bypass this safety valve.

J.2.6.4 Always open the supply pressure first when pressurizing the backpressure assembly. Then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the apparatus rating or relief-valve settings. When relieving backpressure, shut the supply pressure, bleed the pressure from the system, and back out the regulator T-screw.

J.2.7 For safe heating, follow the instructions below.

J.2.7.1 Exercise caution to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended without posting a warning.

J.2.7.2 Avoid the practice of removing the cell and cooling it with water. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

J.2.8 For safe electrical operation, follow the instructions below.

J.2.8.1 Make sure that the electrical source is fused and grounded. Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

J.2.8.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time, or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

J.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.

J.2.9.1 Cell material shall be compatible with the test samples.

J.2.9.2 Do not use cells that show signs of severe pitting or stress cracking.

J.2.9.3 Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.

J.3 Apparatus — Permeability plugging apparatus (PPA) with threaded end caps

J.3.1 PPA cell.

J.3.1.1 There are two manufacturers of PPAs. Each supply-threaded end cap for the cells used for tests run at pressures in excess of 13 800 kPa (2 000 psi). There are threaded caps with three different pressure ratings available: 20 700 kPa (3 000 psi), 27 600 kPa (4 000 psi), and 34 500 kPa (5 000 psi). The operating manual or this 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 to be applicable.

DANGER — Follow the manufacturer's recommendations concerning maximum temperature, pressure and sample size. Failure to do so can lead to serious injury.

J.3.1.2 As received from the manufacturer, the PPA is equipped with valves that are rated to 260 °C (500 °F). If it becomes necessary to change any valves during the life of this apparatus, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

J.3.1.3 The PPA is designed to provide improved static filtration measurements. It can be operated at pressures and temperatures approximating those prevailing downhole and it permits the use of filtration media chosen to simulate exposed sands. The fluid cell is inverted with the pressure applied from the bottom of the cell, the filter medium on top and the filtrate collected from the top. A small hydraulic hand-pump applies the cell pressure. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

J.3.1.4 The PPA can use any one of a number of filtration media, including porous ceramic or sintered metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from 100 millidarcies to 100 darcies. The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provide the user with a greatly improved picture of what is happening downhole.

To improve the uniformity of test conditions and the repeatability of results, the disks can be classified utilizing the user's own flow test procedure or that which is outlined in J.3.2.7.

J.3.1.5 Test pressures are limited by the safety limits of the cell as specified by the manufacturer; usually 20 700 kPa (3 000 psi), 27 600 kPa (4 000 psi), or 34 500 kPa (5 000 psi) at 260 °C (500 °F). The backpressure receiver may be used at pressures as high as 5 170 kPa (750 psi). If backpressure is used in the test, it can be necessary to reduce the maximum test pressure to avoid exceeding the pressure limit of the cell.

Cell caps showing signs of damage should not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting should not be used.

J.3.1.6 For temperatures above 93 °C (200 °F), the backpressure receiver shall be pressurized to prevent boiling of the filtrate. The standard backpressure receiver uses a CO_2 pressurizing source to provide the backpressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

J.3.1.7 The PPA cell is encased in a thermostatically controlled aluminium heating chamber during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel. Power consumption for the PPA heating jacket is 800 W.

J.3.1.8 The PPA can be used in the field or in the laboratory. A stainless steel carrying case with a fold-down workshelf is available for use in the field.

J.3.2 **Filter medium**, disks of any porous material such as ceramic, sintered metal, or resin-coated sand, graded sands, or core samples.

J.3.2.1 Standard disk thickness is 6,5 mm (0,25 in) but, with adapters, thicker disks can be used. A new disk is required for each test. For water-based drilling fluid samples, the disks shall be soaked in fresh water or brine until saturated, at least 5 min to 30 min prior to use. Vacuum saturation shall be used for filter media with low porosity and permeability.

There is unavoidable variability in the pore throat sizes of the ceramic disks normally used in these tests. Consequently, when running comparative tests, it is recommended that the disks be tested and classified to achieve as much uniformity as possible. The manufacturers run a quality control test for a disk classification and can, upon request, provide the user the mean pore throat diameter and an average porosity. The user can use a simple flow test with fresh water to further classify the disk.

J.3.2.2 Other disk types are available, including Berea Sand cores of different porosities and permeabilities. The user should note that these cores have some variability in porosity and permeability and that this can affect the repeatability of test results. Cores can be cut to fit the apparatus cylinder and are usually 6,5 mm (0,25 in) thick. With modification of the cylinder, 25,5 mm (1 in) cores can also be used.

J.3.2.3 Resin-coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The sand is heated at 150 °C (300 °F) for 1 h to 3 h in moulds slightly larger than the normal disk size, and either 6,5 mm (0,25 in) or 25,5 mm (1,0 in) thick. The moulds shall be coated with silicone grease prior to heating.

J.3.2.4 Resin-coated sand disks can be manufactured to provide a substantial variation in pore-throat size and permeability by varying the mesh sizes of the sands. Coarser sands can be used to provide a filter medium for testing lost-circulation material to be used to control seepage losses to severe fluid-loss environments.

J.3.2.5 Sintered metal disks or slotted metal disks can be used to simulate fractures or high-permeability formations. In the evaluation of seepage-loss material needed to seal off a specific formation, the disk pore-throat size should be matched with that of the formation.

J.3.2.6 Sand beds can be used as a filtering medium if the PPA cell is oriented with the filter at the bottom of the cell. For greater repeatability in the height of the sand bed, first determine the desired height of the bed and then weigh the amount of sand necessary to obtain that height. The sand bed shall be saturated with the base fluid before the test. If the user desires to run the test in the standard manner, with the filter medium at the top of the cell, resin-coated sand can be placed in the cell, heated for 1 h to 3 h at 150 °C (300 °F), cooled and then inverted for the test.

J.3.2.7 Procedure for ceramic-disk comparison: install disk in a PPA cell and fill the cell with water. Using the air-permeability apparatus, with the upper cell valve closed, adjust the pressure on 200 kPa (30 psi) test gauge to 28 kPa to 31 kPa (4,0 psi ± 4,5 psi). Open the valve on top of the cell and adjust pressure to 14 kPa ± 0,7 kPa (2,0 psi ± 0,1 psi). After opening valve at the bottom of the cell, readjust pressure with the upper valve to 14 kPa ± 0,7 kPa (2,0 psi ± 0,1 psi). Measure the time for 300 ml to pass through using a 500 ml graduated cylinder; timing from the 100 ml mark to the 400 ml mark. If the PPT is used for comparison purposes, run several disks, classify the disks and use those of similar values.

J.3.3 **Timer**, accurate to 0,1 min over the test interval.

J.3.4 **Thermometer**, with scale reading up to 260 °C (500 °F).

J.3.5 **Graduated cylinder**, 25 ml (TC) or 50 ml (TC).

J.3.6 **High-speed mixer**.

J.4 Procedure for high-temperature/high-pressure (HTHP) filtration

J.4.1 Preheating the heating jacket

J.4.1.1 Connect the power cord to the proper voltage as indicated on the nameplate.

J.4.1.2 Turn the thermostat to the mid-scale setting and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light illuminates when the heating-jacket temperature has reached the thermostat setting.

J.4.1.3 Readjust the thermostat to 6 °C above the desired test temperature.

J.4.2 Loading the filtration cell

J.4.2.1 The filtration cell is a pressure vessel. Cell bodies that show signs of stress cracking or severe pitting should not be used. The following procedure should be followed to ensure safe operation.

J.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.

J.4.2.3 Check the O-rings on the nipples, the floating piston, the cell body and the end caps, and replace any that are damaged or brittle [all O-rings should be replaced routinely after tests at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease completely around all of the O-rings, being especially careful to ensure that those on the piston are well lubricated. Screw the floating piston onto the T-bar wrench and install the piston into the bottom of the cell, working it up and down to ensure that it moves freely (the bottom of the cell, the inlet end, has a shorter recess than the top). Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

J.4.2.4 Fill the space above the piston with hydraulic oil to just above the end face.

J.4.2.5 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.

J.4.2.6 Lubricate the threads with high-temperature-resistant grease and then screw the end cap into place, tightening it moderately with the two-pin spanner wrench. Over-tightening does not improve the seal, and makes the cap difficult to remove.

J.4.2.7 Install the hydraulic end cap onto the bottom of the cell: push in on the backpressure ball on the nipple of the end cap on the pressure-inlet end of the cell to relieve the pressure and allow the cap to be screwed into the cell more easily.

Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

J.4.2.8 Connect the bottom nipple assembly to the pump hose, and pump enough hydraulic oil to expel all air from the nipple. Then, being careful not to allow any oil to spill from the nipple, connect the nipple assembly to the bottom cell cap and disconnect the pump hose.

The steps 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.

For improved consistency in test results, stir drilling fluid for 5 min immediately before loading the cell.

J.4.2.9 Turn the cell upright and fill with approximately 275 ml of drilling fluid. This allows for fluid expansion while heating. Do not exceed this amount.

J.4.2.10 Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the O-ring recess.

J.4.2.11 Install the O-ring and set the selected ceramic disk or other filtering medium on top of it.

J.4.2.12 Install the top end cap in the cell.

J.4.2.13 Lubricate the threads and the bottom of the retainer ring and screw the ring into the top of the cell. Tighten it, using the single pin spanner wrench if necessary, until the outer knurled flange of the retainer ring is flush against the top of the cell body. Attempting to tighten it further does not improve the seal and makes the cap more difficult to remove.

This step applies only to cells that utilize retainer rings for the top end caps.

J.4.2.14 Install the cell in the heating jacket. Make sure that the cell support has been pulled outward using the handle, then insert the cell assembly and rotate it so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

J.4.2.15 Thermal expansion of cell contents, and of the hydraulic fluid, cause the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the excess periodically.

J.4.3 Pressurizing the cell

J.4.3.1 Filtration at temperatures above the boiling point of the fluid sample requires the use of the backpressure receiver to prevent vapourization of the filtrate. It also requires that the sample be pressurized to prevent it from boiling. Refer to Table 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.

J.4.3.2 While the cell is heating, use the following procedure to prepare the backpressure receiver.

Check to ensure that the regulator T-screw has rotated counter-clockwise enough to release all pressure. When the pressure has been released, the screw turns freely.

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.

Verify that the pressure-release valve on the CO_2 assembly and the filtrate drain valve are closed.

Set the backpressure assembly aside. It will be installed in J.4.3.4.

J.4.3.3 Monitor the cell temperature with the thermometer in the well in the cell wall, not the well in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature. Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. This can take as long as 1 h.

J.4.3.4 When the cell is at the desired temperature and cell pressure stabilized, mount the backpressure receiver on the upper valve adapter. Secure the receiver with a retaining pin. Install the CO_2 pressurizing unit on top of the receiver. Lock the CO_2 pressurizing unit in place with a retaining pin.

J.4.3.5 If a drain hose is used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space between the filtration medium and the backpressure receiver outlet, and the receiver valve, should be filled with the base fluid before starting the test. This ensures that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

J.4.3.6 Refer to Table I.1 to determine the appropriate pressure for the backpressure receiver and apply it by turning the T-screw on the pressure regulator until the desired pressure is reached.

J.4.3.7 Actuate the pump to raise the cell pressure to the desired level, then open the valve between the cell and the backpressure receiver to start the test.

The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the backpressure receiver.

J.4.4 Conducting the filtration test

J.4.4.1 Verify that the backpressure as read on the pressure regulator gauge is correct. Adjust if required.

J.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. Precisely 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.

J.4.4.3 Open the filtration valve to start the test. The cell pressure, as read on the pump gauge, drops 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.

J.4.4.4 At the desired times, use the drain valve to bleed the filtrate from the backpressure receiver into the graduated cylinder and record the time and cumulative volume received.

J.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 backpressure receiver for the duration of the test.

It is recommended to recover the filtrate directly from the backpressure receiver, not from a drain hose attached to it. Should a hose be used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

J.4.4.6 After 30 min, close the filtrate valve and drain any remaining filtrate from the backpressure receiver into the graduated cylinder. The total volume of filtrate in the graduated cylinder shall be recorded.

J.5 Test conclusion and disassembly

J.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.

J.5.2 Allow the pressurized cell assembly to cool in the heating jacket. When these tests are run with sufficient frequency to justify it, a cooling stand, station or bath can be provided to expedite the cooling process. There is a cell-handling tool available that should be used any time a hot cell is to be handled.

DANGER — Extreme care should be exercised in cooling hot cells.

This procedure, as recommended, makes it difficult to perform more than one test in an 8 h work day with a single PPA. In the interest of improving productivity, users may want to design their own cell-cooling procedures and apparatus. Safety should be the primary consideration in these designs.

J.5.3 Isolate the backpressure assembly from its pressure source by turning the T-screw on the backpressure regulator counter-clockwise until it turns freely.

J.5.4 Open the bleed valve on the CO_2 unit to release the pressure in the backpressure receiver.

J.5.5 After removing the locking pin and securing it, remove the CO_2 assembly from the top nipple adapter.

J.5.6 After removing its locking pin, remove the backpressure receiver.

J.5.7 Open the valve on the hydraulic pump to release cell pressure, then disconnect the hydraulic quick-coupler.

J.5.8 Open the filtration valve to relieve any pressure remaining between the cell filter and the backpressure receiver.

J.5.9 If it is suspected that the cell may be pressurized and the screened end cap is not in the lower position, the following procedure can be used to verify the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell and insert a small drill bit or wire through the end cap to determine whether the floating piston is at the bottom. If the piston is NOT at the bottom, there is no pressure. If the piston is at the bottom, there is pressure remaining in the cell. Reconnect the hydraulic pump and pump several strokes to move the piston. If the cell is pressurized, it will be obvious from the force required to move the piston.

J.5.10 If there are indications 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 sure that gloves are worn and that the opening is pointed away from the operator when inserting the bit or wire.

The cell should be opened only when the operator is fully confident that the contents are no longer under pressure.

J.5.11 Raise or remove the cell assembly. If desired, the cell may be raised in the heating jacket either by lifting it by the filter valve assembly or using the optional cell-handling tool. Attach this tool to the backpressure-inlet nipple just above the filtrate valve where the backpressure receiver is normally attached. Secure it using the valve-stem locking pin. The cell can be supported on the cell support or lifted out of the heating well and laid on a bench while the cell is being opened.

J.5.12 Remove threaded caps using spanner wrenches. It can be necessary to tap on the wrench to get it started. Opening difficulty is an indication of insufficient lubrication, over-tightening or insufficient cleaning. It can be necessary to use a suitable holding tool, such as a soft-jaw vice, chain wrench, strap wrench or similar device, to secure the cell while the cap is unscrewed.

J.5.13 Reposition the cell so that the filter end is up and unscrew the top cap.

J.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, then measure and record its thickness and remarks concerning its composition.

J.5.15 Pour the remaining fluid from the cell and wash the inside of the cell with fresh water. 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.

J.5.16 Perform the following three steps to replace the O-rings on the floating piston and the bottom end cap.

a) Remove the bottom end cap using the procedure outlined in J.5.10 and J.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.

J.6 Test reports

J.6.1 Filtrate reporting

Report the actual cumulative filtrate volume, expressed in millilitres, collected through each of the selected time periods.

J.6.2 Spurt loss

The spurt loss (2.4) 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 (I.2).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with J.4.4.2.

J.6.3 Calculation

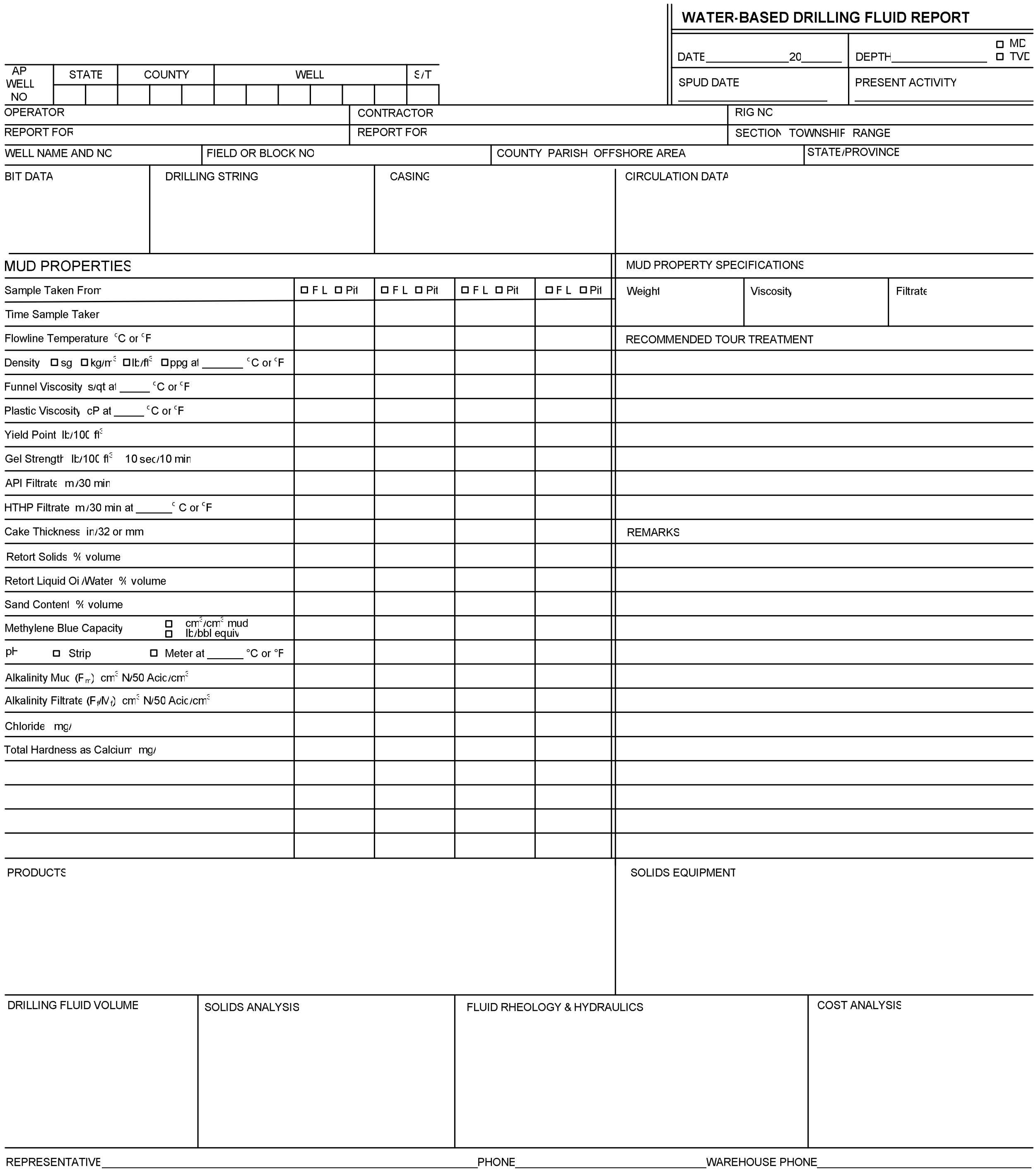
Calculate the permeability plugging test volume, spurt loss and static filtration rate using Equations (I.1), (I.2) and (I.3), respectively.

J.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 K
(informative)

Water-based drilling fluids report form



WATER-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 ☐ ppg 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 sec/10 min				
API Filtrate ml/30 min				
HTHP Filtrate ml/30 min at ______ °C or °F				
Cake Thickness in/32 or mm				
Retort Solids % volume				
Retort Liquid Oil/Water % volume				
Sand Content % volume				
Methylene Blue Capacity ☐ cm^3/cm^3 mud ☐ lb/bbl equiv				
pH ☐ Strip ☐ Meter at ______ °C or °F				
Alkalinity Mud (P$_m$) cm^3 N/50 Acid/cm^3				
Alkalinity Filtrate (P$_f$/M$_f$) cm^3 N/50 Acid/cm^3				
Chloride mg/l				
Total Hardness as Calcium 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______________

Figure K.1 — Sample drilling fluid report form

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