Recommended Practice for Field Testing of Oil-based Drilling Fluids

API RECOMMENDED PRACTICE 13B-2
FOURTH EDITION, MARCH 2005

API
American Petroleum Institute

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Upstream Segment

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Contents

Recommended Practice for Field Testing of Oil-based Drilling Fluids

1 Scope

This Recommended Practice provides standard procedures for determining the following characteristics of oil-based drilling fluids:

a) drilling fluid density (mud weight);

b) viscosity and gel strength;

c) filtration;

d) oil, water and solids contents;

e) alkalinity, chloride content and calcium content;

f) electrical stability;

g) lime and calcium contents, calcium chloride and sodium chloride contents;

h) low-gravity solids and weighting material contents.

Annexes A, B, C, D, H, I, K and L provide additional test methods that may optionally be used for the determination of

i) shear strength,

j) oil and water contents from cuttings,

k) drilling fluid activity,

l) aniline point,

m) cuttings activity,

n) active sulfides.

o) PPA test method for cells with set screws.

p) PPA test method for cells with screw-on caps.

Annexes F, G and J provide procedures that may optionally be used for

q) sampling, inspection and rejection,

r) rig-site sampling,

s) calibration and verification of glassware, thermometers, viscometers, retort kit cups and drilling fluid balances.

Annex E provides examples of calculations for

t) lime, salinity and solids content.

Annex M contains an example of a drilling fluid report form.

2   Terms and definitions

For the purposes of this Standard, the following term and definition applies:

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

2.2
API
American Petroleum Institute, 1220 L Street NW, Washington, DC 20005

2.3
CAS
Chemical Abstracting Service

2.4
USC
United States Customary unit, shown in parentheses following SI unit

3   Abbreviations

ACS   American Chemical Society

BAD   Base alkalinity demand

EDTA   eithylenediaminetetraacetic acid

ES   electrical stability

HT/HP   high temperature, high pressure

OCMA   Oilfield Chemical Manufacturer's Association

PNP   propylene glycol normal-propyl ether

PTFE   polytetrafluoreoethylene, brand name Teflon®

TC   to contain

TD   to deliver

R_{300} viscometer reading at 300 r/min

R_{600} viscometer reading at 600 r/min

R   static filtration rate

m_1	mass of retort cup, lid and body with steel wool, g
m_2	mass of retort cup, lid, body and cuttings, g
m_3	mass of empty liquid receiver, g
m_4	mass of liquid receiver and fluid collected during solids analysis, g
m_5	mass of solids remaining in retort cup following solids analysis, g
R	static filtration rate
V	volume of liquid collected in receiver, ml
V_o	volume of oil, cm^3
V_s	volume of solids, cm^3
V_1	volume of filtrate after 7,5 min, cm^3
V_2	volume of filtrate after 30 min, cm^3
V_w	volume of water, cm^3
η_P	viscosity of plastic viscosity
η_Y	viscosity of yield point
η_A	appararent viscosity
φ_o	volume fraction of oil
φ_s	volume fraction of solids
φ_w	volume fraction of water
ρ	density
$\nabla\rho$	density gradient

4 Determination of drilling fluid density (mud weight)

4.1 Principle

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

4.2 Apparatus

a)   Any **density-measuring instrument** having an accuracy of $\pm$ 0,01 g/cm^3, $\pm$ 10 kg/m^3, $\pm$ 0,1 lb/gal, or $\pm$ 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/cm^3 or 1 000 kg/m^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.

b)   **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 the temperature of the drilling fluid and record.

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.

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 at the edge of the rider toward the drilling-fluid cup. Make appropriate corrections when a range extender is used.

4.4   Calculation

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

4.4.2   To convert the reading to other units, use the following:

$$\rho_s = 1\ 000 \times \text{g/cm}^3 \tag{1}$$

$$\rho_s = 16 \times \text{lb/ft}^3 \tag{2}$$

$$\rho_s = 119,8 \times \text{lb/US gal} \tag{3}$$

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

$$\nabla \rho_s = 9,81 \times \text{g/cm}^3 \tag{4}$$

$$\nabla \rho_s = 0,0226 \times \text{psi/1 000 ft} \tag{5}$$

where $\nabla \rho_s$ is the drilling fluid density gradient, expressed in kilopascals per metre.

A list of density conversions from SI to USC units is given in Table 1.

Table 1 — Density conversions between SI and USC units

Grams per cubic centimetre[a] g/cm^3	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	74,8
1,30	1 300	10,9	81,0
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.

5 Alternative method for determination of drilling fluid density

5.1 Principle

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

5.1.2   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 obtained under downhole conditions.

5.2   Apparatus

a)   Any **density-measuring instrument** having an accuracy of $\pm$ 0,01 g/cm^3, $\pm$ 10 kg/m^3, $\pm$ 0,1 lb/gal, or $\pm$ 0,5 lb/ft^3.

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

Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 1,0 g/cm^3 or 1 000 kg/m^3 at 21 °C (69,8 °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.

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

5.3   Procedure

5.3.1   Measure the temperature of the drilling fluid and record.

5.3.2   Fill the sample cup to a level slightly (approximately 6 mm) below the upper edge of the cup.

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 will be expelled through the check-valve. When the lid has been placed on the cup, pull the check-valve up into the closed position, rinse off the cup and threads with water, and screw the threaded cap on the cup.

5.3.4   The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submersing 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 instrument on the knife edge. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the attached bubble is centred between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of grams per cubic centimetre, pounds per gallon, and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1 000 feet.

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 base oil.

5.4   Calculation

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

For conversions, use the formulas 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 API RP 13D.

6.2 Determination of viscosity using the Marsh funnel

6.2.1 Apparatus

a)   **Marsh funnel**, calibrated to deliver 946 cm^3 (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.

The Marsh funnel shall have the following characteristics:

   1) **funnel cone**, length 305 mm (12,0 in), diameter 152 mm (6,0 in) and a capacity to bottom of screen of 1 500 cm^3 (1,6 quarts);

   2) **orifice**, length 50,8 mm (2,0 in) and inside diameter 4,7 mm (0,185 in);

   3) **screen**, with 1,6 mm (0,063 in) openings (12 mesh); fixed at 19,0 mm (0,748 in) below top of funnel.

b)   **Graduated cup,** with capacity at least 946 cm^3 (1 quart).

c)   **Stopwatch**.

d)   **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 the stopwatch. Measure the time for drilling fluid to fill to the 946 cm^3 (1 quart) mark of the cup.

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

6.2.2.4   Report the time (6.2.2.2), to the nearest second, with the volume, as the Marsh funnel viscosity. Report the temperature (6.2.2.3) of the 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

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

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

The components shall meet the following specifications.

1) **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 rad) apart, around rotor sleeve just below scribed line.

2) **Bob**, closed, with flat base and tapered top

Diameter:	34,49 mm (1,358 in)
Cylinder length:	38,0 mm (1,496 in)

3) **Torsion spring constant:**

386 dyne-cm/degree deflection

4) **Rotor sleeve speeds**

High speed:	600 r/min
Low speed:	300 r/min

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

b)   **Stopwatch.**

c)   **Thermostatically controlled viscometer cup.**

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

6.3.2   Procedure

6.3.2.1   Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 100 cm^3) in the cup for displacement of fluid due to the viscometer bob and sleeve. Immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay from the time of drilling fluid sampling. Testing should be carried out at either (50 ± 1) °C [(120 ± 2) °F] or (65 ± 1) °C [(150 ± 2) °F]. The place of sampling should be stated on the report.

The maximum recommended operating temperature is 90 °C (200 °F). If fluids have to be tested above this temperature, either a solid metal bob, or a hollow metal bob with a completely dry interior should be used.

CAUTION   Liquid trapped inside a hollow bob may vaporize when immersed in high-temperature fluid and cause the bob to explode.

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

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

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

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

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. Record the maximum reading as the initial gel strength. For instruments having a 3 r/min speed, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength (10-second gel) in pounds per 100 square feet.

NOTE   To convert the dial reading to pounds per 100 square feet: 1 Pa = 0,48 lb/100 ft^2.

6.3.2.7   Restir the drilling fluid sample at 600 r/min for 10 s and then 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 pascals (pounds per 100 square feet).

NOTE   To convert the dial reading to pounds per 100 square feet: 1 Pa = 0,48 lb/100 ft^2.

6.3.3   Calculation

$$\eta_P = R_{600} - R_{300} \tag{6}$$

$$\eta_Y = 0{,}48 \times (R_{300} - \eta_P) \tag{7}$$

$$\eta_A = R_{600}/2 \tag{8}$$

where

η_P is the plastic viscosity, in millipascal seconds;

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

η_Y is the yield point, in pascals;

η_A is the apparent viscosity, in millipascal seconds;

R_{600} is the dial reading at 600 r/min, in pascals (pounds per 100 square feet);

R_{300} is the dial reading at 300 r/min, in pascals (pounds per 100 square feet).

NOTE 1   To convert to CGS units of centipoise, 1 mPa·s = 1 cP.

NOTE 2   When calculating values in USC units, the yield point (in pounds per 100 square feet) is calculated as follows:

$$\eta_Y = R_{300} - \eta_P$$

7   Filtration

7.1   Principle

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

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

7.1.3   Filtration tests are performed at ambient (low) temperature and at high-temperature conditions to provide data for comparison purposes. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing from 175 °C (350 °F) to 230 °C (450 °F). Use only the filtration equipment and procedure specified for the temperature required.

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

7.1.4   Either the 175 cm^3, 250 cm^3, or 500 cm^3 unit can be used for testing filtration up to and including 175 °C (350 °F). For testing above 175 °C (350 °F), only the 500 cm^3 unit shall be used. It shall be equipped with a thermocouple to measure the temperature of drilling fluid in the cell, and it shall use a porous filter media.

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

7.2.1   Apparatus

a)   **High-temperature/high-pressure filter press**, consisting of:

  1)   **filter cell**, to contain working pressures up to 9 000 kPa (1 300 psi) at temperature;

  2)   **pressurized gas source**, such as carbon dioxide or nitrogen, with regulators;

NOTE   Nitrogen is preferred.

  3)   **heating system**, to heat to 175 °C (350 °F);

  4)   **high-pressure filtrate collection vessel**, maintained at proper back-pressure (see Table 2) to avoid flashing or evaporation of the filtrate;

  5)   **filter cell**, containing a thermometer well, fitted with a removable end, a filter-media support and with oil-resistant seals.

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

CAUTION   Not all manufacturers' equipment is capable of achieving the same temperatures and pressures. Rigid adherence to manufacturer's recommendations as to sample volumes, temperatures and pressures is essential. Failure to do so could result in serious injury.

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 (see annex I).

Table 2 — Recommended minimum back-pressure

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

b)　　　**Filter medium**[1]), including Whatman No. 50 or S&S 576, or equivalent filter paper, for temperatures to 200 °C (400 °F).

c)　　　**Mechanical or electronic timer**, with at least a 30 min interval.

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

e)　　　**Long, slender graduated cylinder** (TC), with a capacity of 10 cm^3 or 20 cm^3.

f)　　　**Graduated cylinder**, optional, (TC), with a capacity of 25 cm^3.

g)　　　**Field mixer**, cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min

h)　　　**Ruler**, measured in millimetres, to measure filter cake thickness.

7.2.2   Procedure for temperatures up to 175 °C (350 °F)

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

If the filtration unit is equipped with a thermocouple to measure drilling fluid temperature inside the cell (test temperature), then that temperature should be monitored and maintained during the filtration test. Results may differ from this standard procedure, which uses the cell wall temperature as the test temperature. Note if the thermocouple method was used.

7.2.2.2　　　Stir the drilling fluid sample for 5 min using the field mixer. Pour the fluid sample into the filter cell, leaving at least 2,5 cm (1 in) space in the cell to allow for fluid expansion. Install the filter paper in the cell.

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

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

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

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

7.2.2.6   Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 2. Set the lower regulator to the minimum back-pressure value, as shown in Table 2, for the test temperature. Maintain this pressure until the test temperature is reached.

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

7.2.2.7   When the sample reaches the selected test temperature, open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ± 3 °C (± 5 °F) during the test. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

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

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

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

CAUTION   Pressure in the filter cell can be dangerously high, even after the cell is cooled. Opening cell before pressure is released can result in injury.

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

7.2.2.11   Pour the liquid from the cell.

7.2.2.12   Remove the filter cake on the filter paper. Measure the filter cake thickness, at its centre, to the nearest millimetre.

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

7.2.3   Calculation

7.2.3.1   The filtrate volume should be corrected to a filter area of 4 580 mm^2 (7,1 in^2). HT/HP filter cells usually have half the standard filter area or 2 258 mm^2 (3,5 in^2), thus double the observed volume before reporting.

7.2.3.2   Report the cake thickness to the nearest millimetre (32nd inch).

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

7.3.1   Apparatus

a)   **High temperature/high pressure filter press**, consisting of the following components:

1) **filter cell**, to contain working pressures up to 14 500 kPa (2 250 psi) at a temperature of 230 °C (450 °F);

2) **pressurized gas source**, such as carbon dioxide or nitrogen with regulators;

3) **heating system**, to heat up to 230 °C (450 °F);

4) **high-pressure filtrate collection vessel**, maintained at proper back-pressure (see Table 2), to avoid flashing or evaporation of the filtrate;

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

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

CAUTION Not all filtration testing equipment is rated to perform safely at temperatures and pressures required in this procedure. It is essential to follow the manufacturer's recommendations regarding maximum safe operating temperature, pressure and sample volumes. Failure to do so could result in injury. The 175 cm^3 and 250 cm^3 filtration cells are not recommended for use at these higher temperatures and pressures.

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 (see annex I).

b)   **Filter medium**, Dynalloy X-5[2]) or equivalent porous disc, for temperatures above 200 °C (400 °F). A new disc is required for each test.

c)   **Mechanical or electronic timer**, with at least a 30-min interval.

d)   **Thermometer**, able to measure up to 260 °C (500 °F) with a 20 cm (8 in) or longer stem.

e)   **Long, slender graduated cylinder** (TC), with a volume of 10 cm^3 or 20 cm^3.

f)   **Graduated cylinder**, optional (TC), with a volume of 25 cm^3.

g)   **Field mixer**, of a cup type, to operate at 10 r/min, 1 000 r/min and 15 000 r/min.

h)   **Ruler**, graduated in millimetres (or inches), to measure filter cake thickness.

7.3.2   Procedure for temperatures from 175 °C (350 °F) to 230 °C (450 °F)

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

7.3.2.2   Stir the drilling fluid sample for 5 min using the field mixer. Pour the fluid sample into the filter cell, leaving at least 2,5 cm (1 in) space in the cell to allow for drilling fluid expansion. Install the filter medium in the cell.

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

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

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[2])   Dynalloy X-5 discs is an example of a suitable product available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by API of these products.

7.3.2.5 Connect the pressure source to the upper valve. Connect a similar pressure source to the lower collection vessel and lock these connections in place.

7.3.2.6 Keeping the two valve stems closed, adjust the pressure on the upper pressure regulator to 690 kPa (100 psi) higher than the minimum back-pressure value, as shown in Table 2. Set the lower regulator to the minimum back-pressure value, as shown in Table 2, for the test temperature. Maintain this pressure until the test temperature is reached.

7.3.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, open the lower valve stem and immediately increase the pressure on the upper regulator to 3 450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within $\pm 3\,°C$ ($\pm 5\,°F$) during the test. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

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

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

CAUTION   Pressure in the filter cell can be dangerously high, even after the cell is cooled. Opening the cell before pressure is released can result in injury.

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

7.3.2.11 Pour the liquid from the cell.

7.3.2.12 Remove the filter cake on the filter medium. Measure the filter cake thickness, at its centre, to the nearest millimetre.

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

7.3.3   Calculation

7.3.3.1 The filtrate volume should be corrected to a filter area of 4 580 mm^2 (7,1 in^2). HT/HP filter cells usually have half the standard filter area or 2 258 mm^2 (3,5 in^2), thus double the observed volume before reporting.

7.3.3.2 Report the cake thickness to the nearest millimetre (32nd inch).

8   Retort test for oil, water and solids contents

8.1   Principle

8.1.1 A retort test measures water and oil released from an oil-based drilling fluid sample when heated in a calibrated and properly operating retort instrument.

8.1.2 Knowledge of water, oil and solids contents is fundamental to proper control of drilling fluid properties such as oil/water ratio, rheology, density, filtration and salinity of aqueous phase. Knowledge of solids in an oil-based drilling fluid is essential to evaluation of solids control equipment.

8.1.3   In a retort test, a known volume of oil-based drilling fluid is heated in a retort instrument to vaporize the liquid components. These vapours are then condensed and collected in a precision-graduated receiver. The volume fraction, expressed as a percentage, of retort solids is calculated by subtracting the total liquid volume from the starting volume of oil-based drilling fluid and dividing by the starting volume

8.2   Apparatus

a)   **Retort instrument**.

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

Standard cup sizes are 10 cm^3 (precision $\pm\,0.05 \text{ cm}^3$), 20 cm^3 (precision $\pm\,0.1 \text{ cm}^3$), and 50 cm^3 (precision $\pm\,0.25 \text{ cm}^3$). The retort cup volume with lid should be verified gravimetrically. The procedure and calculations are given in annex J.

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

2)   **Condenser**, capable of cooling the oil and water vapours below their vaporization temperature.

3)   **Heating jacket**, nominal power 350 W.

4)   **Temperature controller**, capable of limiting temperature of the retort to $500 \,°C \pm 40 \,°C$ ($930 \,°F \pm 70 \,°F$).

b)   **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:	10 cm^3	20 cm^3	50 cm^3
Precision (0 % to 100 %):	$\pm\,0.05 \text{ cm}^3$	$\pm\,0.05 \text{ cm}^3$	$\pm\,0.05 \text{ cm}^3$
Frequency of graduation marks (0 % to 100 %):	0.10 cm^3	0.10 cm^3	0.50 cm^3

Calibration: To contain "TC" at 20 °C (68 °F)
Scale: millilitre, cubic centimetre 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 given in annex J.

c)   **Fine steel wool, oil-free**.

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

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

e)   **Pipe cleaners**.

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

g)   **Corkscrew**.

h)   **Syringe**, of capacity 10 cm^3, 20 cm^3 or 50 cm^3, to fill retort cup.

i)   **Marsh funnel**.

8.3   Procedure

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

8.3.2   Collect a representative sample of oil-based drilling fluid and allow it to cool to approximately 26 °C (80 °F). Screen the test sample through the 1,68 mm (0,066 in or 12-mesh) screen on the Marsh funnel to remove lost circulation material, large cuttings or debris.

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

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

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

8.3.5   Pack the retort body with steel wool.

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

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

8.3.8   Place the clean, dry liquid receiver below the condenser outlet.

NOTE   The length of the receiver may require that it be angled out from the retort and perhaps supported off the edge of the worktable.

8.3.9   Turn on the heating jacket, and allow the retort assembly to run a minimum of 45 min.

If drilling fluid boils over into the receiver, pack the retort body with a larger amount of steel wool and repeat the test.

8.3.10   Remove the liquid receiver and allow it to cool. Record the total liquid volume, oil volume and water volume in the receiver.

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

8.3.11   Turn off the heating jacket, and allow the retort assembly to cool before cleaning.

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 cm^3, 20 cm^3 or 50 cm^3), calculate, as percentages, the volume fractions of water, oil and total solids in the drilling fluid.

a)   Volume fraction oil:

$$\varphi_o = 100 \times \frac{V_o}{V_s} \qquad (9)$$

where

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

V_o is the volume of oil, in millilitres;

V_s is the volume of the drilling fluid sample, in millilitres.

b) Volume fraction water:

$$\varphi_w = 100 \times \frac{V_w}{V_s} \qquad (10)$$

where

φ_w is the volume fraction of water, expressed as a percentage of the total sample volume;

V_w is the volume of water, in millilitres;

V_s is the volume of the drilling fluid sample, in millilitres.

c) Volume fraction retort solids:

$$\varphi_s = 100 - (\varphi_w + \varphi_o) \qquad (11)$$

where φ_s is the volume fraction of retort solids, expressed as a percentage of the total sample volume.

NOTE   The calculated retort solids overestimates the volume of suspended solids because of dissolved salts. A volumetric correction for salinity can be made using known volume factors for salt solutions (see clause 11).

9   Chemical analysis of oil-based drilling fluids

9.1   Principle

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

9.1.2   The whole-drilling-fluid chloride test procedure is a titration method which measures the volume of standard silver nitrate required to react with the chloride (and other halide) ions to form insoluble silver chloride (or silver halide) salts. The test procedure can utilize the same sample as the alkalinity test, provided the sample is acidic (pH below 7,0). The chloride value reported in the whole oil-based drilling fluid can be assigned to the water phase up to the saturation point. The water-soluble salt concentration is related to the effectiveness of an oil-based drilling fluid in controlling shale through the "aqueous phase activity" concept. The aqueous-phase salinity value is also needed to adjust the retort water value to obtain a corrected solids content for the oil-based drilling fluid.

9.1.3   The whole-drilling-fluid calcium test is a titration method which measures the volume of a standard calcium-chelating agent (EDTA) required to react with all the calcium (and other water-soluble alkaline earth ions), released from the oil-based drilling fluid into the aqueous phase, when a mixture of solvents is used to extract the

oil-based drilling fluid. The calcium measured in this test can come from the calcium chloride ($CaCl_2$) and lime [CaO or $Ca(OH)_2$] used to prepare the oil-based drilling fluid, but some calcium could come from drilled-up gypsum or anhydrite ($CaSO_4$).

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

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

9.1.6   Procedures for the calculations based on these chemical analyses are provided in clause 11.

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

9.1.8   Care must be taken when obtaining sample to assure homogeneity. Settling or separation in the sample container may lead to erroneous results, therefore stirring the container may be required. Other factors to consider include shelf life of test reagent solutions, cross-contamination from pipettes and contamination from water other than distilled or deionised sources. To avoid cross-contamination, pipettes must never be used for more than one (1) reagent solution. All reagent solutions should be tightly capped when not in use and properly discarded per vendor instructions when the expiration date occurs.

9.1.9   The titration media utilised for alkalinity determifnation in oil-based drilling fluid will consume a small amount of alkalinity. This has been labelled as Base Alkalinity Demand (BAD). This BAD can be determined by using 0,1 N sodium hydroxide to titrate the phenolphthalein endpoint of the titrating matrix without any drilling fluid. Once determined, the BAD can be added to the alkalinity measurement for the oil-based drilling fluid to obtain a more accurate representation of the alkalinity/lime content.

NOTE   The BAD correction cannot be applied to fluids which do not exhibit any alkalinity.

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

9.2   Reagents and apparatus

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

b)   **Titration vessel**, 400 cm^3 beaker, with cap, or pint jar.

c)   **Phenolphthalein (CAS No. 77-09-8)** indicator solution, 1 g/100 cm^3 in a 1:1 isopropanol:water solution.

d)   **Sulfuric acid (CAS No. 7664-93-9)** solution, $c(H_2SO_4) = 0,05$ mol/l.

e)   **Potassium chromate (CAS No. 7789-00-6)** indicator solution, 5 g/100 cm^3 of water.

f)   **Silver nitrate (CAS No. 7761-88-8)** reagent, $c(AgNO_3) = 47,91$ g/l [$c(Cl^-) = 0,01$ g/ cm^3 or 0,282 mol/l], stored in an amber or opaque bottle.

g)   **Distilled or deionised water**.

h) **Disposable syringe**, two 5 cm^3.

i) **Graduated cylinder** (TC), one 25 cm^3.

j) **Graduated pipettes** (TD), two 1 cm^3 and two 10 cm^3.

NOTE   One pair (1 cm^3 and 10 cm^3) of pipettes is for sulfuric acid and one pair is for silver nitrate.

k) **Pipette balloon**.

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

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

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

n) **Calcium indicator**, of the type Calver® II[3] or hydroxynaphthol blue (CAS No. 63451-35-4).

o) **EDTA (CAS No. 139-33-3)** solution, $c(\text{EDTA})$ 0,1 mol/l, standardized disodium ethylenediamine-tetraacetate dihydrate (1 cm^3 = 10 000 mg/l $CaCO_3$, 1 cm^3 = 4 000 mg/l Ca^{++}).

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

p) **Sodium hydroxide (CAS No. 1310-73-2)** solution, $c(\text{NaOH}) = 1$ mol/l.

q) **API low-temperature/low-pressure filter press**

r) **Filter paper,** Whatman 50 , S&S 376, or equivalent.

s) **Filter press pressure source**, pressure can be applied with any nonhazardous fluid medium, either gas or liquid, except compressed CO2.

9.3   Base alkalinity demand (BAD)

9.3.1   Procedure

9.3.1.1   Add 100 cm^3 of PNP solvent to a 400-cm^3 beaker or pint jar.

9.3.1.2   Add 200 cm^3 of distilled (or deionised) water.

9.3.1.3   Add 15 drops of phenolphthalein indicator solution.

9.3.1.4   While stirring with a magnetic stirrer such that the vortex is one-half the liquid height, titrate immediately with 0,1 mol/l sodium hydroxide until pink colour just appears.

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

9.3.1.5   Continue stirring for 5 min. If the pink color remains, the end point has been reached. If the pink color disappears, repeat 9.3.1.4.

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[3]   Calver® II is an example of a suitable product available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by API of these products.

9.3.2   Calculation

Base Alkalinity Demand = 0,1 mol/l sodium hydroxide, cm^3 (12)

9.4   Whole-drilling-fluid alkalinity

9.4.1   Procedure

9.4.1.1 Add 100 cm^3 of PNP solvent to a 400-cm^3 beaker or pint jar.

9.4.1.2 Fill the 5- cm^3 syringe with whole drilling fluid beyond the 3- cm^3 mark.

9.4.1.3 Displace 2,0 cm^3 of whole drilling fluid into the beaker or pint jar.

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

9.4.1.5 Add 200 cm^3 of distilled (or deionised) water. If no filtration is needed, skip to 9.4.1.11.

9.4.1.6 (Optional filtration) Stir the mixture for 5 min with a magnetic stirrer such that the vortex is one-half the liquid height.

a)   Pour the mixture into an API Low-temperature/low-pressure filter press fitted with filter paper.

b)   Close the cell and apply 690 kPa (100 psi) pressure. Do not use CO_2 cartridges or other CO_2 bottles as the pressure source.

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

d)   Use all the filtrate and continue with 9.4.1.7.

9.4.1.7 Add 15 drops of phenolphthalein indicator solution [9.2 c)].

9.4.1.8 While stirring with a magnetic stirrer such that the vortex is one-half the liquid height, titrate immediately with 0,1 mol/l sulphuric acid until pink colour just disappears.

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

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

9.4.1.9 Continue stirring for 5 min. If no pink colour reappears, the end point has been reached. If a pink colour returns, return to 9.4.1.8. Record the volume of 0,1 mol/l sulfuric acid (V_{H2SO4}), in millilitres.

9.4.1.10 Use the volume of 0,1 mol/l sulfuric acid solution, expressed in millilitres, required to reach the end point in 9.4.1.12 to calculate the whole-drilling-fluid alkalinity, (Alk_{TOT}).

9.4.2   Calculation

$$V_{SA} = (V_{H2SO4} + \text{BAD})/V_S$$ (13)

$Lime_{OM}$, lb/bbl $= 1{,}295\ V_{SA}$(14)

$Field\ Lime$, lb/bbl $= Lime_{OM}$, lb/bbl / Lime assay, weight fraction   (15)

where

V_{SA} is the whole-drilling-fluid alkalinity;

V_{H2SO4} is the volume of 0,05 mol/l sulfuric acid, in millilitres;

V_s is the volume of the drilling fluid sample, in millilitres.

NOTE V_{SA} is equivalent to P_{OM} used in previous editions of this standard. BAD is the cm^3 of 0,1 mol/.l sodium hydroxide solution required to reach a phenolphthalein endpoint in the titration fluid matrix without any drilling fluid. Field Lime referes to the amount of field-grade lime that must be added to a drilling fluid system to achieve a target lime concentration. Field-grade lime is usually less than 100% active.

9.5   Whole-drilling-fluid chloride content

9.5.1   Procedure

9.5.1.1 Perform the alkalinity procedures in 9.4.1.1 through 9.4.1.10.

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

9.5.1.2 Add 10 to 15 drops of potassium chromate indicator solution [9.2 e)].

9.4.1.3 Titrate slowly with 0,282 mol/l silver nitrate reagent [9.2.f)] while stirring rapidly with a magnetic stirrer, until a salmon-pink colour remains stable for at least 1 min. It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to more clearly see the colour in the aqueous phase.

NOTE   Additional potassium chromate indicator solution may be required during titration process.

9.4.1.4 Use the volume of 0,282 mol/l silver nitrate reagent required (V_{AgNO3}) in millilitres to reach the end point in 9.4.1.3 to calculate the whole-drilling-fluid chloride content [$c(Cl^-)_{TOT}$].

9.5.2   Calculation

$$c(Cl^-)_{TOT} = (10\ 000 \times V_{AgNO3}) / V_s \tag{13}$$

where

$c(Cl^-)_{TOT}$ is the whole-drilling-fluid chloride content, in milligrams per litre;

V_{AgNO3} is the volume of 0,282 mol/l silver nitrate reagent, in millilitres;

V_s is the volume of the drilling fluid sample, in millilitres.

9.6   Whole-drilling-fluid calcium content

9.6.1   Procedure

9.5.1.1 Add 100 cm^3 of the PNP solvent to a titration vessel that has a cap.

9.5.1.2 Fill the 5 cm^3 syringe beyond the 3 cm^3 mark with whole oil-based drilling fluid.

9.5.1.3 Displace 2,0 cm^3 of oil-based drilling fluid into the titration vessel.

9.5.1.4 Swirl the oil-based drilling fluid and solvent until the mixture is homogenous.

9.5.1.5 Add 200 cm^3 distilled or deionized water to the titration vessel.

9.5.1.6 (Optional)   If filtration is needed, stir the mixture for 5 min with a magnetic stirrer such that the vortex is one-half the liquid height.

a)   Pour the mixture into an API Low-temperature/low-pressure filter press fitted with filter paper.

b)   Close the cell and apply 690 kPa (100 psi) pressure. Do not use CO_2 cartridges or other CO_2 bottles as the pressure source.

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

d)   Use all the filtrate and continue with 9.5.1.6.

9.5.1.6 Add 6,0 cm^3 sodium hydroxide buffer solution.

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

9.5.1.8 Place the titration vessel on the magnetic stirrer and drop in a stir bar.

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

9.5.1.10 Use the volume of EDTA required (V_{EDTA}) to reach the end point in 9.5.1.9 to calculate the whole-drilling-fluid calcium content, $c(Ca^{++})_{TOT}$.

9.6.2   Calculation

$$c(Ca^{++})_{TOT} = (4\,000 \times V_{EDTA})/V_s \tag{14}$$

where

$c(Ca^{++})_{TOT}$ is the whole-drilling-fluid calcium content, in milligrams per litre;

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

V_s is the volume of the drilling fluid sample, in millilitres.

10 Electrical stability test

10.1 Principle

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

NOTE   The sine wave specified for the voltage ramp results in more efficient energising of the fluid and generates considerably lower ES values (off less than half) than the spiky waveform of the instrument described in previous editions of 10414-2/RP 13B-2. The symmetry of the sinusoidal signal also inhibits build-up of solids on the electrode faces and enhances

reproducibility. To further enhance reproducibility, the instrument specifications include automatic voltage ramping at a fixed ramp rate.

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

10.2 Apparatus

a) **Electrical stability meter**, with the following characteristics:

1) waveform: sine, < 5 % total harmonic distortion;
2) AC frequency: 340 Hz ± 10 Hz;
3) output units: peak volts;
4) ramp rate: 150 V/s ± 10 V/s, automatic operation;
5) range: 3 V to 2 000 V (peak);
6) trip current 61 µA ± 5 µA (peak).

b) **Electrodes**, with the following characteristics:

1) housing: material resistant to oil-based drilling fluid components up to 105 °C (220 °F);
2) material: corrosion-resistant metal;
3) diameter: 3,18 mm ± 0,03 mm (0,125 in ± 0,001 in);
4) spacing (gap): 1,55 mm ± 0,03 mm (0,061 in ± 0,001 in) at 22 °C (72 °F).

c) **Calibration resistors/diodes**, with the following characteristics:

1) number: two (low and high);
2) types: standard resistor or Zener diodes;
3) range: give voltage (ES) readings of
   (a) low: 500 V to 1 000 V and
   (b) high: > 1 900 V;
4) accuracy: 2% of expected voltage, temperature corrected by manufacturer's table (if needed and provided).

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

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

e) **Marsh funnel**.

f) **Thermostatically controlled viscometer cup**.

g) **Isopropanol (CAS No. 67-63-0)**.

10.3 Equipment calibration/performance test

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

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

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

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

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

10.3.6 Check the accuracy of the ES meter with the standard resistors and/or Zener diodes (see NOTE under 10.2.3). The ES readings should fall within 2,5 % (combined uncertainty of meter and resistor/diodes) of the expected values. If any of the ES readings fall outside this range, the instrument should be returned to the supplier for adjustment or repair.

NOTE   This subclause 10.3, does not check waveform, AC frequency or ramp rate. Have the ES meter and electrode probe periodically checked by a qualified technician to ensure that both units meet all specifications.

10.4 Electrical stability measurements

10.4.1 Verify equipment calibration/performance as described in 10.3.

10.4.2 Screen the drilling fluid sample through a Marsh funnel.

10.4.3 Place the drilling fluid sample in a viscometer cup maintained at 50 °C ± 2 °C (120 °F ± 5 °F). Record the drilling fluid temperature.

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

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

10.4.5 Hand-stir the 50 °C (120 °F) sample with the electrode probe for approximately 10 s to ensure that the composition and temperature of the drilling fluid are uniform. Position the electrode probe so that it does not touch the bottom or sides of the container, and be sure that the electrode surfaces are completely covered by the sample.

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

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

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

10.4.9 Record the average of the two ES measurements.

11 Lime, salinity and solids calculations

11.1 Principle

11.1.1 Equations in this procedure allow calculations of the content of the following components for an oil-based drilling fluid:

a)   lime, expressed as $Ca(OH)_2$, in the whole drilling fluid;

b) $CaCl_2$ and/or NaCl in the whole drilling fluid;

c) $CaCl_2$ and/or NaCl dissolved in the aqueous phase;

d) low-gravity solids (NaCl salt and/or drilled solids);

e) weighting material.

11.1.2 Data listed below and used in the equations are determined from oil-based drilling fluid tests specified in clauses 4, 8 and 9:

a) drilling fluid density (see clause 4), ρ;

b) whole-drilling-fluid alkalinity, Alk_{TOT} (see 9.4);

c) whole-drilling-fluid calcium content, $c(Ca^{++})_{TOT}$, (see 9.6);

d) whole-drilling-fluid chloride content, $c(Cl^-)_{TOT}$, (see 9.5);

e) volumes of retort oil, V_o, and water, V_w, (see clause 8).

11.1.3 Data known or assumed are as follows:

a) density of weighting material (in grams per cubic centimetre), ρ_{wm};

b) density of low-gravity solids (in grams per cubic centimetre), ρ_{lg};

c) density of the base oil (in grams per cubic centimetre), ρ_o.

11.1.4 These calculations do not utilize a "lime correction" for adjusting the whole-drilling-fluid calcium content as determined by the EDTA titration.

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

11.1.6 Examples of calculations can be found in annex E.

11.2 Apparatus

A calculator should be used to quickly and correctly perform the mathematical operations detailed in this procedure.

11.3 Whole-drilling-fluid calculations

11.3.1 Whole-drilling-fluid lime content

The total lime [Ca(OH)$_2$] content, calculated in kilograms per cubic metre (pounds per barrel) is:

$$c[Ca(OH)_2]_{TOT} = 3{,}691 \times Alk_{TOT} \text{ kg/m}^3 \tag{15}$$

$$(c[Ca(OH)_2]_{TOT} = 1{,}295 \times Alk_{TOT} \text{ lb/bbl}) \tag{16}$$

where

$c[Ca(OH)_2]_{TOT}$ is the total lime content of the whole drilling fluid;

Alk_{TOT} is the alkalinity of the whole drilling fluid.

NOTE $c[Ca(OH)_2]_{TOT}$ is often referred to as "excess lime."

11.3.2 Whole-drilling-fluid calcium chloride and sodium chloride contents

11.3.2.1   An oil-based drilling fluid will probably contain at least one and perhaps both of the salts calcium chloride ($CaCl_2$), and sodium chloride (NaCl). Use the titration data and proceed below to determine if both NaCl and $CaCl_2$ are present.

11.3.2.2   The $CaCl_2$ content, based on the total calcium analysis, calculated in milligrams per litre or kilograms per cubic metre (pounds per barrel) is:

$$c(CaCl_2)_{TOT} = 2{,}774 \times c(Ca^{++})_{TOT}, \text{ milligrams per litre} \tag{17}$$

$$c(CaCl_2)_{TOT} = 0{,}002\ 774 \times c(Ca^{++})_{TOT}, \text{ kilograms per cubic metre} \tag{18}$$

$$[c(CaCl_2)_{TOT} = 0{,}000\ 971 \times c(Ca^{++})_{TOT}, \text{ pounds per barrel}] \tag{19}$$

where

$c(CaCl_2)_{TOT}$ is the $CaCl_2$ content of the whole drilling fluid, expressed in milligrams per litre;

$c(Ca^{++})_{TOT}$ is the calcium content of the whole drilling fluid, expressed in milligrams per litre.

11.3.2.3   The chloride ion associated with the $CaCl_2$, based on the total calcium analysis, is:

$$c\left(Cl^-_{CaCl_2}\right)_{TOT} = 1{,}77 \times c(Ca^{++})_{TOT} \tag{20}$$

where

$c\left(Cl^-_{CaCl_2}\right)_{TOT}$ is the chloride content of the whole drilling fluid based on $CaCl_2$, expressed in milligrams per litre;

$c(Ca^{++})_{TOT}$ is the calcium content of the whole drilling fluid, expressed in milligrams per litre.

11.3.2.4   Verify which numerical value is larger.

a) If $c\left(Cl^-_{CaCl_2}\right)_{TOT}$ is larger than or equal to $c(Cl^-)_{TOT}$, then assume that only $CaCl_2$ is present in the drilling fluid and that no NaCl is present. Proceed to 11.3.2.5, equation (21).

b)   If $c(Cl^-)_{TOT}$ is larger than $c\left(Cl^-_{CaCl_2}\right)_{TOT}$ then assume that both $CaCl_2$ and NaCl are present in the drilling fluid. Proceed to 11.3.2.7.

11.3.2.5 The $CaCl_2$ concentration, based on the total chloride ion analysis, is:

$$c(CaCl_2)_{TOT} = 1{,}57 \times c\left(Cl^-\right)_{TOT} \tag{21}$$

where $c(CaCl_2)_{TOT}$ is the $CaCl_2$ concentration in the whole drilling fluid, expressed in milligrams per litre.

Use this result in place of the value calculated in equation (17) when NaCl is present.

11.3.2.6 The amount of $CaCl_2$ present can be converted to kilograms per cubic metre (pounds per barrel) as follows:

$$CaCl_2, kg/m^3 = 0{,}001 \times c(CaCl_2)_{TOT} \tag{22}$$

$$[CaCl_2, lb/bbl = 0{,}000\ 35 \times c(CaCl_2)_{TOT}] \tag{23}$$

Use these results in place of the value calculated in equation (18) or (19) when no NaCl is present.

At this point proceed directly to 11.4.

11.3.2.7 The total $CaCl_2$ concentration has already been determined in equation (17), (18) or (19). The amount of chloride ion associated with NaCl is:

$$c\left(Cl^-_{NaCl}\right) = c\left(Cl^-\right)_{TOT} - c\left(Cl^-_{CaCl_2}\right)_{TOT} \tag{24}$$

where $c\left(Cl^-_{NaCl}\right)$ is the chloride concentration from NaCl in the whole drilling fluid, in milligrams per litre.

11.3.2.8 The NaCl content is:

$$c(NaCl)_{TOT} = 1{,}65 \times c\left(Cl^-_{NaCl}\right)_{TOT} \tag{25}$$

where $c(NaCl)_{TOT}$ is the NaCl concentration in the whole drilling fluid, in milligrams per litre.

11.3.2.9 The amount of NaCl present is converted to kilograms per cubic metre (pounds per barrel) as follows:

$$NaCl, kg/m^3 = 0{,}001 \times c(NaCl)_{TOT} \tag{26}$$

$$[NaCl, lb/bbl = 0{,}000\ 35 \times c(NaCl)_{TOT}] \tag{27}$$

11.4 Aqueous phase calculations

11.4.1 The calculations above have involved only the whole drilling fluid salt content. In order to determine the salt concentrations in the aqueous phase of the drilling fluid, the titration data in the following calculations must be combined with the retort data described in clause 8.

11.4.2 The following equations (28) and (29) calculate the amounts of NaCl and $CaCl_2$ respectively in the aqueous phase of an oil-based drilling fluid. Mass fraction $CaCl_2$ is:

$$w_{CaCl_2} = \frac{100 \times c(CaCl_2)_{TOT}}{c(CaCl_2)_{TOT} + c(NaCl)_{TOT} + 10\ 000\ \varphi_w} \tag{28}$$

where

w_{CaCl_2} is the mass fraction of $CaCl_2$ in the aqueous phase, expressed as a percentage of the total aqueous phase;

$c(CaCl_2)_{TOT}$ is the $CaCl_2$ content in the whole drilling fluid, expressed in milligrams per litre;

$c(NaCl)_{TOT}$ is the NaCl content in the whole drilling fluid, expressed in milligrams per litre;

φ_w is the volume fraction of water, expressed as a percentage of the retort sample volume.

11.4.3 The mass fraction of NaCl is:

$$w_{NaCl} = \frac{100 \times c(NaCl)_{TOT}}{c(CaCl_2)_{TOT} + c(NaCl)_{TOT} + 10\,000\ \varphi_w} \tag{29}$$

where

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

$c(CaCl_2)_{TOT}$ is the $CaCl_2$ content in the whole drilling fluid, expressed in milligrams per litre;

$c(NaCl)_{TOT}$ is the NaCl content in the whole drilling fluid, expressed in milligrams per litre;

φ_w is the volume fraction of water, expressed as a percentage of the retort sample volume.

11.4.4 Check Figure 1 to determine if the mass fraction of NaCl, w_{NaCl}, is outside the limit of mutual solubility for NaCl/CaCl$_2$ brine solutions at 25 °C. If it is, undissolved NaCl is present in the oil-based drilling fluid. The aqueous-phase salinities, as calculated in equations (28) and (29), are therefore incorrect because all the NaCl is not actually in solution.

To determine the correct $c(NaCl)_{TOT}$, systematically lower it by using a ratio method involving equations (28) and (29), until the calculated w_{NaCl} and w_{CaCl_2} fall within the range shown in Figure 1. Following the steps in 11.4.4.1 to 11.4.4.6, provide an iteration solution approximating the correct values of $c(NaCl)_{TOT}$ and w_{NaCl} and w_{CaCl_2}. An example of this calculation is given in annex E.

11.4.4.1 Calculate the value of the ratio below. It will be less than 1,00 even when there is a large amount of insoluble NaCl present in the drilling fluid. No more than three "trial" calculations are required.

$$\frac{w_{nFig1}}{w_{NaCl}} \tag{30}$$

where

w_{nFig1} is the mass fraction of NaCl in the aqueous phase, expressed as a percentage of the total aqueous phase from Figure 1,

w_{NaCl} is the mass fraction of NaCl in the aqueous phase, expressed as a percentage of the total measured aqueous phase.
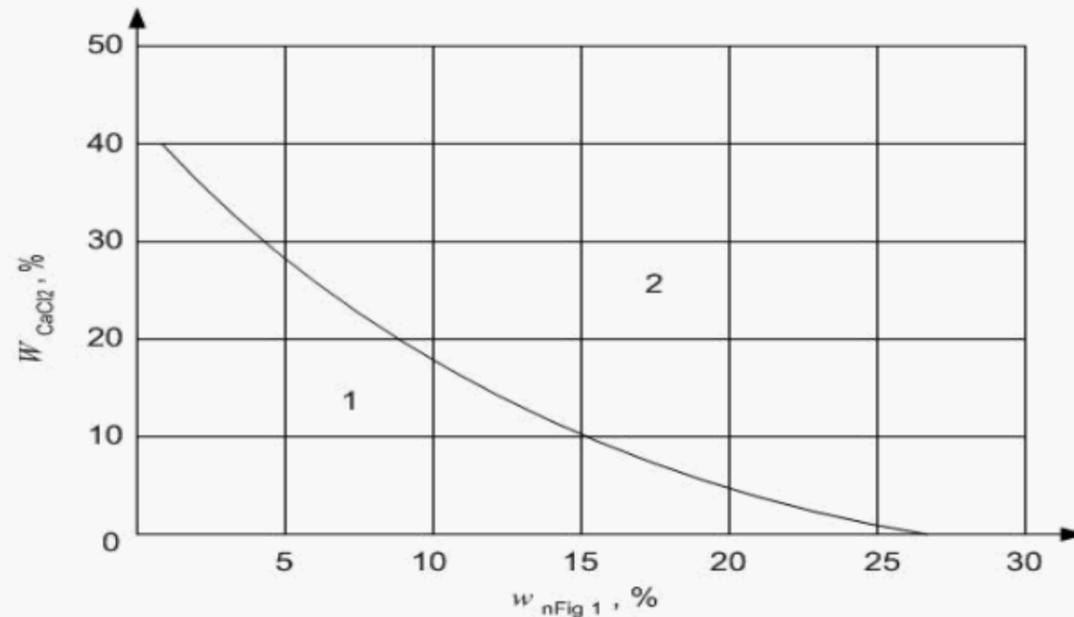
Key

1   NaCl soluble

2   NaCl undissolved

Figure 1 — Maximum concentrations of NaCl in CaCl$_2$ brine at 25 °C

11.4.4.2   Multiply the $c(NaCl)_{TOT}$ values used in equations (28) and (29) by the value of this ratio to obtain a new $c(NaCl)_{TOT}$ trial value:

$$\text{New } c(NaCl)_{TOT} = \text{Ratio} \times w_{NaCl} \tag{31}$$

11.4.4.3   Use the new $c(NaCl)_{TOT}$ in the denominator of equation (28) to calculate a new w_{CaCl_2} and use the new $c(NaCl)_{TOT}$ value in the denominator and numerator of equation (29) to calculate a new w_{NaCl}.

11.4.4.4   Read a new value of $c(NaCl)_{TOT}$ from Figure 1 using the new w_{CaCl_2} from 11.4.4.3 on the graph.

11.4.4.5   Calculate a second new ratio value using $c(NaCl)_{TOT}$ found from Figure 1 from 11.4.4.4 and w_{NaCl} calculated from 11.4.4.3.

11.4.4.6   Calculate a second new w_{CaCl_2} and w_{NaCl}, as in 11.4.4.3. Record these values when the value of the ratio becomes 0,95 or larger.

NOTE 1   When the value of the ratio becomes 0,95 or larger, no more calculations of new values for $c(NaCl)_{TOT}$ and w_{CaCl_2} and w_{NaCl} are needed. This usually requires no more than three cycles of this calculation.

NOTE 2   Undissolved NaCl has a lower specific gravity than most drill solids. If it is present in the oil mud, it will make the solids calculations in error. No technique is provided in this procedure to compensate for that type of error.

11.4.4   The density of the brine, ρ_B, in grams per cubic centimetre, is:

$$\rho_B = 0,997\ 07 + 0,006\ 504\ (w_{NaCl}) + 0,007\ 923\ (w_{CaCl_2}) + 0,000\ 083\ 34\ (w_{NaCl})\ (w_{CaCl_2}) +$$
$$0,000\ 043\ 95\ (w_{NaCl})^2 + 0,000\ 049\ 64\ (w_{CaCl_2})^2 \tag{32}$$

NOTE   Table 3 contains the densities of single salt solutions of CaCl$_2$. Table 4 contains the densities of single salt solutions of NaCl.

11.4.5 The corrected $CaCl_2$ concentration, expressed in milligrams per litre, is:

$$c(CaCl_2) = 10\ 000 \times w_{CaCl_2} \times \rho_B \tag{33}$$

11.4.6 The NaCl concentration, expressed in milligrams per litre, is:

$$c(NaCl) = 10\ 000 \times w_{NaCl} \times \rho_B \tag{34}$$

11.5 Solids calculations

11.5.1 Corrected solids content

The combination of water and dissolved salts is known as brine.

Determine the corrected solids content in the drilling fluid as follows:

The volume fraction of brine is:

$$\varphi_B = \frac{100 \times V_w}{\rho_B \times \left[100 - \left(w_{NaCl} + w_{CaCl_2} \right) \right]} \tag{35}$$

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

The corrected volume fraction of solids is:

$$\varphi_{sol} = 100 - (V_o + \varphi_B) \tag{36}$$

where φ_{sol} is the corrected volume fraction of solids, expressed as a percentage of the whole drilling fluid.

11.5.2 Oil/water ratio calculations

The oil/water ratio, φ_o/φ_w, is calculated from the following components:

$$\varphi_o = \frac{100 \times V_o}{V_o + V_w} \tag{37}$$

where φ_o is the volume fraction of oil, expressed as a percentage.

$$\varphi_w = \frac{100 \times V_w}{V_o + V_w} \tag{38}$$

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

NOTE   The oil to water ratio is sometimes expressed as OWR.

Table 3 — Calcium chloride brine table

Mass fraction (CaCl$_2$)	Calcium chloride content	Chloride content	Final volume	Water activity[a]	Density	
%	mg/l	mg/l	L		g/ cm^3	(lb/gal)
0	0	0	1,000	1,000	0,998 0	8,330
2	20 330	12 990	1,004	0,996	1,016 6	8,484
4	41 340	26 410	1,008	0,989	1,033 4	8,624
6	63 030	40 280	1,013	0,979	1,050 5	8,766
8	85 420	54 590	1,018	0,967	1,067 8	8,911
10	108 540	69 360	1,024	0,951	1,085 4	9,058
12	132 400	84 600	1,030	0,933	1,103 3	9,207
14	157 050	100 360	1,037	0,912	1,121 8	9,361
16	182 510	116 630	1,044	0,888	1,140 7	9,519
18	208 780	133 410	1,051	0,862	1,159 9	9,679
20	235 920	150 750	1,060	0,832	1,179 6	9,844
22	263 930	168 650	1,069	0,800	1,199 7	10,011
24	292 820	187 100	1,078	0,765	1,220 1	10,182
26	322 660	206 170	1,089	0,727	1,241 0	10,356
28	353 420	225 820	1,100	0,686	1,262 2	10,533
30	385 140	246 090	1,113	0,643	1,283 8	10,713
32	417 890	267 020	1,126	0,597	1,305 9	10,898
34	451 620	288 570	1,141	0,548	1,328 3	11,047
36	486 430	310 780	1,156	0,496	1,351 2	11,276
38	522 310	333 740	1,173	0,441	1,374 5	11,470
40	559 280	357 370	1,192	0,384	1,398 2	11,668

NOTE 1   The calcium ion content can be determined by subtracting the chloride ion content from the CaCl$_2$ content.

NOTE 2   Properties at 20 °C, except water activity which is at 25 °C.

[a]   See reference [6].

Table 4 — Sodium chloride brine table

Mass fraction NaCl	Sodium chloride content	Chloride content	Final volume	Water activity[a]	Density	
%	mg/l	mg/l	L		g/ cm^3	(lb/gal)
0	0	0	1,000	1,000	0,998 0	8,330
2	20 250	12 310	1,006	0,989	1,014 3	8,464
4	41 140	24 960	1,013	0,976	1,028 6	8,584
6	62 590	37 980	1,020	0,964	1,043 1	8,705
8	84 620	51 350	1,028	0,950	1,057 8	8,827
10	107 260	60 680	1,036	0,935	1,072 6	8,951
12	130 510	79 200	1,045	0,919	1,087 6	9,076
14	154 390	93 680	1,054	0,901	1,102 8	9,203
16	178 910	108 570	1,065	0,882	1,118 2	9,331
18	204 100	123 840	1,076	0,861	1,133 9	9,462
20	229 960	139 540	1,087	0,839	1,149 8	9,595
22	265 520	155 660	1,099	0,815	1,166 0	9,730
24	283 800	172 210	1,113	0,788	1,182 5	9,868
26	311 820	189 210	1,127	0,759	1,199 3	10,008

NOTE 1   The sodium ion content can be determined by subtracting the chloride ion content from the NaCl content.

NOTE 2   Properties at 20 °C, except water activity which is at 25 °C.

[a]   See reference [7].

11.5.3 Oil/brine ratio calculations

The oil/brine ratio, φ_o/φ_B, is calculated from the following components:

$$\varphi_o = \frac{100 \times V_o}{V_o + V_B} \tag{39}$$

where φ_o is the volume fraction of oil, expressed as a percentage

$$\varphi_B = \frac{100 \times V_B}{V_o + V_B} \tag{40}$$

where φ_B is the volume fraction of brine, expressed as a percentage

NOTE   The oil to brine ratio is sometimes expressed as OBR.

11.5.4 Solids concentration and density

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

The average density (volumic mass) of the suspended solids is:

$$\overline{\rho}_{sol} = \frac{100 \quad \rho_s - (\varphi_o \cdot \rho_o) - (\varphi_B \cdot \rho_B)}{\varphi_{sol}}$$

(41)

where

$\overline{\rho}_{sol}$ is the average density of the suspended solids, expressed in grams per cubic centimetre;

ρ_s is the drilling fluid density, expressed in grams per cubic centimetre;

φ_o is the volume fraction of oil, expressed as a percentage of the retort sample volume;

ρ_o is the oil density, expressed in grams per cubic centimetre;

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

ρ_B is the brine density, expressed in grams per cubic centimetre;

φ_{sol} is the corrected volume fraction of solids.

Equation (41) may also be calculated in US customary units by using density values expressed in pounds per barrel for the drilling fluid density (ρ_s) and the oil and brine densities (ρ_o and ρ_B).

Table 5 — Commonly used densities

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

11.5.5 Volume fraction and concentrations of weighting material and low-gravity solids

11.5.5.1 The volume fraction of weighting material is:

$$\varphi_{wm} = \frac{\overline{\rho}_{sol} - \rho_{lg}}{\rho_{wm} - \rho_{lg}} (\varphi_{sol})$$

(42)

where

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

ρ_{wm} is the density of the weighting material solids, expressed in grams per cubic centimetre;

ρ_{lg} is the density of the low-gravity solids, expressed in grams per cubic centimetre.

11.5.5.2   The volume fraction of the low-gravity solids is:

$$\varphi_{lg} = \varphi_{sol} - \varphi_{wm} \tag{43}$$

where

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

11.5.5.3   The mass concentration of the weighting material, calculated in kilograms per cubic metre (pounds per barrel), can be calculated as:

$$c(wm), \text{kg/m}^3 = 10 \, \rho_{wm} \cdot \varphi_{wm} \tag{44}$$

$$c(wm), \text{lb/bbl} = 3,5 \, \rho_{wm} \cdot \varphi_{wm} \tag{45}$$

where $c(wm)$ is the weighting material concentration.

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

$$c(lg), \text{kg/m}^3 = 10 \, \rho_{lg} \cdot V_{lg} \tag{46}$$

$$[c(lg), \text{lb/bbl} = 3,5 \, \rho_{lg} \cdot V_{lg}] \tag{47}$$

where $c(lg)$ is the low-gravity solids concentration.

Annex A
(informative)

Measurement of shear strength using shearometer tube

A.1 Principle

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

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

A.2 Apparatus

a) **Stainless steel shearometer tube**, having the following characteristics:

   1) Length: 89 mm (3,5 in);

   2) Outside diameter: 36 mm (1,4 in);

   3) 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) **Platform** for weights.

c) **Set of weights**, in gram increments.

d) **Ruler**, graduated in millimetres (inches).

A.3 Procedure

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

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

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

A.4 Calculation

Shear strength is calculated as follows:

$$\gamma = \frac{4,40 \times \left(m_{st} + m_{tot} \right)}{l} - 1,02\rho \tag{A.1}$$

where

γ is the shear strength of the drilling fluid, in pascals;

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

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

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

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

The shear strength may be converted to USC units using the following formula:

$$\gamma, \text{ in lb/100 ft}^2 = 2,083 \times (\gamma, \text{ in Pa}) \tag{A.2}$$

Annex B
(informative)

Determination of oil and water content of cuttings for percentage greater than 10%

B.1 Principle

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

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

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

B.2 Apparatus

a) **Retort instrument**.

1) **Retort assembly**, which includes a retort body, cup and lid with a volume of 50 cm^3 (accuracy $\pm\,0,25$ cm^3), constructed of 303 stainless steel, or equivalent.

2) The **retort cup** volume with lid should be verified gravimetrically. The procedure and calculations are given in annex J.

3) **Condenser,** capable of cooling the oil and water vapours below their vaporization temperatures.

4) **Heating jacket**, nominal power 350 W.

5) **Temperature controller**, capable of limiting temperature of the retort to 500 °C $\pm$ 40 °C (930 °F $\pm$ 70 °F).

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

	10 cm^3	20 cm^3	50 cm^3
Total volume			
Accuracy (0 % to 100 %)	$\pm\,0,05$ cm^3	$\pm\,0,05$ cm^3	$\pm\,0,5$ cm^3
Frequency of graduation marks (0 % to 100 %)	0,10 cm^3	0,10 cm^3	0,50 cm^3
Calibration: To contain "TC" at 20 °C (68 °F)			
Scale: ml, cm^3 or volume fraction (as percent)			

1) Material: Transparent and inert to oil, water and salt solutions at temperatures up to 32 °C (90 °F).

2) The receiver volume should be verified gravimetrically. The procedure and calculations are given in annex J.

c) **Top-loading balance**, capable of weighing 2 000 g with an acccuracy of $\pm$ 0,1 g.

d) **Fine steel wool, oil-free**. "Liquid steel wool" or coated steel wool substitutes shall not be used for this application.

e) **High-temperature silicone grease**, to be used for thread sealant and lubricant.

f) **Pipe cleaners**.

g) **Brush**.

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

i) **Corkscrew**.

B.3 Procedure

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

B.3.2 Pack the retort body with steel wool.

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

B.3.4 Weigh and record the total mass of the retort cup, lid, and retort body with steel wool. This is mass m_1, expressed in grams.

B.3.5 Collect a representative wet cuttings sample.

B.3.6 Partially fill the retort cup with cuttings, and place the lid on the cup. Screw the retort cup (with lid) onto the retort body, weigh and record the total mass. This is mass m_2, expressed in grams.

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

B.3.8 Weigh and record the mass of the clean and dry liquid receiver. This is mass m_3, expressed in grams. Place the receiver below the condenser outlet.

B.3.9 Turn on the heating jacket. Allow the retort assembly to run for a minimum of 1 h.

NOTE   If solids boil over into receiver, pack the retort body with a greater amount of steel wool and repeat the test.

B.3.10 Remove the liquid receiver. Allow it to cool. Record the volume of water recovered. This is volume V, expressed in millilitres, which is converted to grams (see note in B.4)

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

B.3.11 Weigh and record the mass of the receiver and its liquid contents (oil plus water). This is mass m_4, expressed in grams.

B.3.12 Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser.

B.3.13 Weigh and record the mass of the cooled retort assembly without the condenser. This is mass m_5, in grams.

B.3.14 Clean the retort assembly and condenser.

B.4 Calculations

Calculate the mass of oil from the cuttings as follows:

a) $\quad m_w = m_2 - m_1$ (B.1)

where

$\quad m_w$ is the mass of the wet cuttings, expressed in grams;

$\quad m_2$ is the mass of the filled retort assembly, expressed in grams;

$\quad m_1$ is the mass of the empty retort assembly, expressed in grams.

b) $\quad m_d = m_5 - m_1$ (B.2)

where

$\quad m_d$ is the mass of the dry retorted cuttings, expressed in grams;

$\quad m_5$ is the mass of the cooled retort assembly, expressed in grams;

$\quad m_1$ is the mass of the empty retort assembly, expressed in grams.

c) $\quad m_o = m_4 - (m_3 + V_w)$ (B.3)

where

$\quad m_o$ is the mass of the oil, expressed in grams;

$\quad m_4$ is the mass of the liquid receiver with its contents, expressed in grams;

$\quad m_3$ is the mass of the dry receiver, expressed in grams;

$\quad V_w$ is the volume of the water, expressed in millilitres = grams.

NOTE   Assuming the density of water is 1 g/cm^3, the mass, in grams, of the volume of water is numerically equivalent to the volume of the water measured in millilitres.

d) Mass balance requirement: the sum of m_d, m_o and V_w, should be within 5 % of the mass of the wet sample, i.e.:

$$0{,}95 \leq \frac{m_d + m_o + V_w}{m_w} \leq 1{,}05$$ (B.4)

Repeat the procedure if this requirement is not met.

Reporting oil from cuttings:

e) Mass of oil (in grams) per kilogram of wet cuttings sample

$$= (m_o/m_w) \times 1\ 000$$ (B.5)

f)   Mass of oil (in grams) per kilogram of dry retorted cuttings

$$= (m_o/m_d) \times 1\ 000 \qquad\qquad\qquad (B.6)$$

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

$$= (m_o/m_d) \times 1\ 000$$

Annex C
(informative)

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

C.1 Principle

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

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

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

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

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

C.2 Reagents and apparatus

a) **Standard saturated solutions of the salts**

    The standard saturated solutions of the salts are described in Table C.1.

Table C.1 — Standard saturated solutions of salts

Salt	CAS No.	Aw At 25 °C (77 °F)	Concentration g/100 cm^3 water
Calcium chloride	10043-52-4	0,295	100
Calcium nitrate	10124-37-5	0,505	200
Sodium chloride	7647-14-5	0,753	200
Potassium nitrate	7757-79-1	0,938	200

1) Each salt solution should be prepared by adding the number of grams of salt indicated in Table C.1 to 100 cm^3 of distilled or deionized water, stirring at temperatures between 65 °C and 90 °C (150 °F and 200 °F) for 30 min, then cooling to a temperature between 25 °C and 27 °C (75 °F and 80 °F). Solid salt shall be visible in the liquid after equilibration.

2) If no crystals are visible, some additional crystals or grains of the same salt should be added to induce precipitation. This is often necessary with calcium chloride. Salts of reagent-grade purity are recommended. However, if necessary, field-grade NaCl or CaCl2 can be substituted.

b) **Desiccant**, anhydrous calcium chloride (CAS No. 10043-52-4).

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

1) The relative humidity sensor probe shall be suitable for insertion in the air space of a jar containing the sample.

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

3) The instrument should indicate within 10 % of the true activity within 30 min, when measuring the brine activity of the standard salt solutions in C.2 a).

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

e) **Small wide-mouth jar**, having a volume of approximately 150 cm^3 and a liquid surface area of approximately 38 cm^2 (5,9 in^2). The container should have a flexible elastomer cap which, when bored through, will form an airtight seal around the shaft of the sensor probe.

f) **Insulating cover**, made of synthetic foam, for the wide-mouth jar. The cover should fit snugly and should insulate the side and bottom of the jar from temperature fluctuations. Alternatively, the jar can be kept in a constant-temperature water bath.

g) **Linear** graph paper.

h) **Andyrous calcium chloride** (CAS 10043-52-4), or other desiccant, such as Ca$_2$SO$_4$ (CAS 10101-41-4), Drierite® (CAS 7778-18-9), or silica gel (CAS 112926-00-8).

C.3 Procedure

C.3.1 Calibration of hygrometer

C.3.1.1 Prepare each known relative humidity standard in accordance with Table C.1. A total volume of 40 cm^3 is sufficient for each sample container. These salt solutions shall be kept firmly capped to prevent spillage, evaporation or contamination.

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

C.3.1.3 Transfer the probe and the jar cap to the first (lowest a_w) standard solution and allow 30 min for equilibrium to be reached.

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

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

C.3.1.6 Proceed as in C.3.1.3, and measure the relative humidity for each standard solution, proceeding from that with the lowest to the highest activity a_w.

C.3.1.7 Allow each standard to equilibrate the same length of time. Record temperature and percent relative humidity (% RH).

C.3.1.8 When complete data are obtained, plot % RH versus a_w on linear graph paper.

C.3.2 Measurement of oil-based drilling fluid activity, a_{df}

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

C.3.2.2 Place a sample of room-temperature drilling fluid (40 cm^3) into the sample container.

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

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

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

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

C.4 Calculation — Drilling fluid activity, a_{df}

Find the relative humidity obtained in C.3.2.4 on the graph plotted in C.3.1.8. Read and record the corresponding activity that appears on the graph as the drilling fluid activity (a_{df}).

Annex D
(informative)

Determination of aniline point

D.1 Principle

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

NOTE   The reader is referred to ISO 2977 [1] for additional information.

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

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

D.2 Reagents and apparatus

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

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

b) **Drying agent**, calcium sulfate (CAS No. 7778-18-9) or sodium sulfate (anhydrous) (CAS No. 7757-82-6).

c) **Test tube**, 25 mm in diameter, 150 mm in length, and made of heat-resistant glass.

d) **Jacket**, approximately 37 mm to 42 mm in diameter and 150 mm in length, and made of heat-resistant glass.

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

f) **Cork stoppers**, one to fit inside the test tube to hold thermometer, and one to fit inside the jacket to hold the test tube. Do not use rubber stoppers.

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

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

NOTE   Water should not be used as a heating medium, because aniline is hygroscopic. Moist aniline will cause erroneously high results.

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

NOTE   Water should not be used as a cooling medium, because aniline is hygroscopic. Moist aniline will cause erroneously high results.

j) **Pipette** (TD), with a volume of 10 cm^3.

k) **Plastic pipette suction bulb**.

l) **Safety goggles**.

m) **Plastic gloves**, of a material that is impervious to aniline.

D.3 Procedure

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

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

D.3.3   Pipette 10 cm^3 of aniline (using the suction bulb) into the test tube. Likewise, pipette 10 cm^{3}of oil sample into the test tube.

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

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

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

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

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

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

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

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

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

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

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

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

Annex E
(informative)

Lime, salinity and solids calculations

E.1 General

The following example calculations are designed to show how the equations in clause 11 are used to determine the amount of salt and solids present in an oil-based drilling fluid containing barite as the weighting material. Four different cases are presented:

— Drilling fluid A: the drilling fluid contains *only* $CaCl_2$ salt;

— Drilling fluid B: the drilling fluid contains *only* NaCl salt;

— Drilling fluid C: the drilling fluid contains *both* $CaCl_2$ and NaCl salts;

— Drilling fluid D: the drilling fluid contains *both* $CaCl_2$ and *excess* NaCl salts.

This annex includes examples in which the units are typical for the relevant application.

NOTE 1   A significant figure to one decimal place is considered accurate enough for all field calculations. Two decimal places are used in these calculations for the average density of the suspended solids, weighting material, low-gravity solids and the brine density.

NOTE 2   This annex shows the relevant equation numbers from clauses 9 and 11 in the main text of this Standard in brackets after the formulae.

E.2 Test data used in examples

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

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

Property	Drilling fluid A only $CaCl_2$ salt	Drilling fluid B only NaCl salt	Drilling fluid C $CaCl_2$ and NaCl salts	Drilling fluid D $CaCl_2$ and excess NaCl salts
1) Drilling fluid density, g/cm^3	1,32	1,32	1,32	1,32
2) Oil content (φ_o), % volume fraction	59,5	59,5	59,5	59,5
3) Water content (φ_w), volume %	25,0	25,0	25,0	25,0
4) EDTA (V_{EDTA}), cm^3/ cm^3 of drilling fluid	10,0	0,0	8,4	8,4
5) Silver nitrate (V_{SN}), cm^3/ cm^3 of drilling fluid	5,8	4,4	7,0	10,0
6) Sulfuric acid (V_{SA}) cm^3/ cm^3 of drilling fluid	2,6	2,0	2,6	3,0
7) Oil density, g/cm^3	0,84	0,84	0,84	0,84
8) Density of low gravity solids, g/cm^3	2,65	2,65	2,65	2,65
9) Density of weighting material (barite), g/cm^3	4,20	4,20	4,20	4,20

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

E.3.1 Total lime content

$$c[Ca(OH)_2]_{TOT} = 3{,}691 \times 2{,}6 = 9{,}6 \text{ kg/m}^3 \tag{15}$$

$$(c[Ca(OH)_2]_{TOT} = 1{,}295 \times 2{,}6 = 3{,}4 \text{ lb/bbl}) \tag{16}$$

E.3.2 Total calcium content

$$c(Ca^{++})_{TOT} = 4\ 000 \times 10{,}0 = 40\ 000 \text{ mg/l of drilling fluid} \tag{14}$$

E.3.3 Total chloride content

$$c(Cl^-)_{TOT} = 10\ 000 \times 5{,}8 = 58\ 000 \text{ mg/l of drilling fluid} \tag{13}$$

E.3.4 Total calcium chloride content

$$c\left(CaCl_2\right)_{TOT} = 2{,}774 \times 40\ 000 = 110\ 960 \text{ mg/l of drilling fluid} \tag{17}$$

Expressed in other units:

$$c\left(CaCl_2\right)_{TOT} = 0{,}002\ 774 \times 40\ 000 = 110{,}0 \text{ kg/m}^3 \tag{18}$$

$$[c\left(CaCl_2\right)_{TOT} = 0{,}000\ 971 \times 40\ 000 = 38{,}8 \text{ lb/bbl}] \tag{19}$$

E.3.5 Chloride content associated with the $CaCl_2$

$$c\left(Cl^-_{CaCl_2}\right)_{TOT} = 1{,}77 \times 40\ 000 = 70\ 800 \text{ mg/l of drilling fluid} \tag{20}$$

E.3.6 Sodium chloride content check

$$c\left(Cl^-_{NaCl}\right)_{TOT} = 58\ 000 - 70\ 800 = -12\ 800 \text{ mg/l of drilling fluid} \tag{24}$$

The negative number indicates that no NaCl is present in this mud. Therefore, the calcium chloride content must be recalculated.

E.3.7 Total calcium chloride content recalculated

$$c\left(CaCl_2\right)_{TOT} = 1{,}57 \times 58\ 000 = 91\ 060 \text{ mg/l of drilling fluid} \tag{21}$$

Expressed in other units:

$$CaCl_2 = 0{,}001 \times 91\ 060 = 91{,}06 \text{ kg/m}^3 \tag{22}$$

$$(CaCl_2 = 0{,}000\ 35 \times 91\ 060 = 31{,}9 \text{ lb/bbl}) \tag{23}$$

E.3.8 Calcium chloride mass fraction, expressed as a percentage

$$w_{CaCl_2} = \frac{100 \times 91\ 060}{91\ 060 + 0 + (10\ 000 \times 25{,}0)} = 26{,}7 \text{ \% mass fraction} \tag{28}$$

E.3.9 Density of the calcium chloride brine using only the CaCl$_2$ value

$$\rho_B = 0{,}997\,07 + 0{,}007\,923\,(26{,}7) + 0{,}000\,049\,64\,(26{,}7)^2 = 1{,}24 \text{ g/cm}^3 \tag{32}$$

E.3.10 Calcium chloride, in milligrams per litre

$$c(\text{CaCl}_a) = 10\,000 \times 26{,}7 \times 1{,}24 = 331\,808 \text{ mg/l} \tag{33}$$

E.3.11 Volume fraction (as percent) brine

$$\varphi_B = \frac{100 \times 25{,}0}{1{,}24 \times (100 - 26{,}7)} = 27{,}5\,\% \tag{35}$$

E.3.12 Volume fraction (as percent) corrected solids

$$\varphi_{sol} = 100 - (59{,}6 + 27{,}5) = 13{,}0\,\% \tag{36}$$

E.3.13 Oil/water ratio (volume fraction) calculations

$$\varphi_o = \frac{100 \times 59{,}5}{59{,}5 + 25{,}0} = 70{,}4\,\% \text{ oil} \tag{37}$$

$$\varphi_w = \frac{100 \times 25{,}0}{59{,}5 + 25{,}0} = 29{,}6\,\% \text{ water} \tag{38}$$

$$OWR = 70{,}4 / 29{,}6$$

E.3.14 Oil/brine ratio (volume fraction) calculations

$$\varphi_o = \frac{100 \times 59{,}5}{59{,}5 + 27{,}5} = 68{,}4\,\% \text{ oil} \tag{39}$$

$$\varphi_B = \frac{100 \times 27{,}5}{59{,}5 + 27{,}5} = 31{,}6\,\% \text{ brine} \tag{40}$$

$$OBR = 68{,}4 / 31{,}6$$

E.3.15 Average density (volumic mass) of suspended solids

$$\overline{\rho}_{sol} = \frac{(100 \times 1{,}32) - (59{,}5 \times 0{,}84) - (27{,}5 \times 1{,}24)}{13{,}0} = 3{,}68 \text{ g/cm}^3 \tag{41}$$

E.3.16 Volume fraction (as percent) weighting material

$$\varphi_{wm} = \frac{3{,}68 - 2{,}65}{4{,}2 - 2{,}65}(13{,}0) = 8{,}6\,\% \tag{42}$$

E.3.17 Volume fraction (as percent) low-gravity solids

$$\varphi_{lg} = 13{,}0 - 8{,}6 = 4{,}4\,\% \tag{43}$$

E.3.18 Mass concentration weighting material

$$c(\text{wm}) = 10 \times 4,20 \times 8,6 = 361,2 \text{ kg/m}^3 \tag{44}$$

$$[c(\text{wm}) = 3,5 \times 4,20 \times 8,6 = 126,4 \text{ lb/bbl}] \tag{45}$$

E.3.19 Mass concentration low-gravity solids

$$c(\text{lg}) = 10 \times 2,65 \times 4,4 = 116,6 \text{ kg/m}^3 \tag{46}$$

$$[c(\text{lg}) = 3,5 \times 4,20 \times 4,4 = 40,8 \text{ lb/bbl}] \tag{47}$$

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

E.4.1   Total lime content

$$c[\text{Ca(OH)}_2]_{\text{TOT}} = 3,691 \times 2,0 = 7,38 \text{ kg/m}^3 \tag{15}$$

$$(c[\text{Ca(OH)}_2]_{\text{TOT}} = 1,295 \times 2,0 = 2,6 \text{ lb/bbl}) \tag{16}$$

E.4.2   Total calcium content

$$c(\text{Ca}^{++})_{\text{TOT}} = 4\ 000 \times 0,0 = 0,0 \text{ mg/l of drilling fluid} \tag{14}$$

E.4.3   Total chloride content

$$c(\text{Cl}^-)_{\text{TOT}} = 10\ 000 \times 4,4 = 44\ 000 \text{ mg/l of drilling fluid} \tag{13}$$

E.4.4   Total calcium chloride content

$$c(\text{CaCl}_2)_{\text{TOT}} = 2,774 \times 0,0 = 0,0 \text{ mg/l of drilling fluid} \tag{17}$$

$$[c(\text{CaCl}_2)_{\text{TOT}} = 0,000\ 971 \times 0,0 = 0,0 \text{ lb/bbl}] \tag{19}$$

E.4.5   Chloride content associated with CaCl_2

$$c\left(\text{Cl}^-_{\text{CaCl}_2}\right)_{\text{TOT}} = 1,77 \times 0,0 = 0,0 \text{ mg/l of drilling fluid} \tag{20}$$

E.4.6   Sodium chloride content check

$$c\left(\text{Cl}^-_{\text{NaCl}}\right) = 44\ 000 - 0 = 44\ 000 \text{ mg/l of drilling fluid} \tag{24}$$

The positive number indicates that NaCl is present in this mud, therefore the sodium chloride content must be calculated.

E.4.7   Total sodium chloride content recalculated

$$c(\text{NaCl})_{\text{TOT}} = 1,65 \times 44\ 000 = 72\ 600 \text{ mg/l of drilling fluid} \tag{25}$$

Expressed in other units:

$$\text{NaCl} = 0,001 \times 72\ 600 = 72,6 \text{ kg/m}^3 \tag{26}$$

$$\left(\text{NaCl, lb/bbl} = 0,000\ 35 \times 72\ 600 = 25,4\ \text{lb/bbl}\right) \tag{27}$$

E.4.8 Mass fraction (as percent) sodium chloride

$$w_{\text{NaCl}} = \frac{100 \times 72\ 600}{0 + 72\ 600 + \left(10\ 000 \times 25,0\right)} = 22,5\ \% \tag{29}$$

E.4.9 Density of the sodium chloride brine using only the NaCl value

$$\rho_{\text{B}} = 0,997\ 07 + \left(0,006\ 504 \times 22,5\right) + \left(0,000\ 043\ 95 \times 22,5^2\right) = 1,17\ \text{g/cm}^3 \tag{32}$$

E.4.10 Sodium chloride content in brine, in mg/l

$$c(\text{NaCl}) = 10\ 000 \times 22,5 \times 1,17 = 263\ 250\ \text{mg/l} \tag{34}$$

E.4.11 Volume fraction (as percent) of brine

$$\varphi_{\text{B}} = \frac{100 \times 25,0}{1,17\left(100 - 22,5\right)} = 27,6\ \% \tag{35}$$

E.4.12 Volume fraction (as percent) of corrected solids

$$\varphi_{\text{sol}} = 100 - \left(59,5 + 27,6\right) = 13,0\ \% \tag{36}$$

E.4.13 Oil/water ratio (volume fraction) calculations

$$\varphi_{\text{o}} = \frac{100 \times 59,5}{59,5 + 25,0} = 70,4\ \%\ \text{oil} \tag{37}$$

$$\varphi_{\text{w}} = \frac{100 \times 25,0}{59,5 + 25,0} = 29,6\ \%\ \text{water} \tag{38}$$

$$OWR = 70,4 / 29,6$$

E.4.14 Oil/brine ratio (volume fraction) calculations.

$$\varphi_{\text{o}} = \frac{100 \times 59,5}{59,5 + 27,6} = 68,3\ \%\ \text{oil} \tag{39}$$

$$\varphi_{\text{B}} = \frac{100 \times 27,6}{59,5 + 27,6} = 31,7\ \%\ \text{brine} \tag{40}$$

$$OBR = 68,3 / 31,7$$

E.4.15 Average density of suspended solids.

$$\overline{\rho}_{\text{sol}} = \frac{\left(100 \times 1,32\right) - \left(59,5 \times 8,84\right) - \left(27,6 \times 1,17\right)}{12,9} = 3,85\ \text{g/cm}^3 \tag{41}$$

E.4.16 Volume fraction weighting material.

$$\varphi_{wm} = \frac{3,85 - 2,65}{4,2 - 2,65} \times 12,9 = 10,0 \ \% \tag{42}$$

E.4.17 Volume fraction of low-gravity solids.

$$\varphi_{lg} = 12,9 - 10,0 = 2,9 \ \% \tag{43}$$

E.4.18 Mass concentration of the weighting material.

$$c(wm) = 10 \times 4,20 \times 10,0 = 147,0 \ kg/m^3 \tag{44}$$

E.4.19 Mass concentration of low-gravity solids.

$$c(lg) = 10 \times 2,65 \times 2,9 = 76,9 \ kg/m^3 \tag{46}$$

E.5 Drilling fluid containing both CaCl$_2$ and NaCl salts (drilling fluid C)

E.5.1 Total lime content.

$$c\left[Ca(OH)_2\right]_{TOT} = 3,691 \times 2,6 = 9,6 \ kg/m^3 \tag{15}$$

E.5.2 Total calcium content.

$$c\left(Ca^{++}\right)_{TOT} = 4\ 000 \times 8,4 = 33\ 600 \ mg/l \ of \ drilling \ fluid \tag{14}$$

E.5.3 Total chloride content.

$$c\left(Cl^-\right)_{TOT} = 10\ 000 \times 7,0 = 70\ 000 \ mg/l \ of \ drilling \ fluid \tag{13}$$

E.5.4 Total calcium chloride content.

$$c\left(CaCl_2\right)_{TOT} = 2,774 \times 33\ 600 = 93\ 206 \ mg/l \ of \ drilling \ fluid \tag{17}$$

$$[c\left(CaCl_2\right)_{TOT} = 0,000\ 971 \times 33\ 600 = 32,6 \ lb/bbl] \tag{19}$$

E.5.5 Chloride content associated with CaCl$_2$.

$$c\left(Cl^-_{CaCl_2}\right)_{TOT} = 1,77 \times 33\ 600 = 59\ 472 \ mg/l \ of \ drilling \ fluid \tag{20}$$

E.5.6 Sodium chloride content check.

$$c\left(Cl^-_{NaCl}\right) = 70\ 000 - 59\ 472 = 10\ 528 \ mg/l \ of \ drilling \ fluid \tag{24}$$

The positive number indicates that NaCl is present in this mud, therefore the sodium chloride content shall be calculated.

E.5.7 Total calcium chloride content recalculated.

$$c\left(CaCl_2\right)_{TOT} = 1{,}57 \times 59\ 472 = 93\ 371 \quad mg/l \text{ of drilling fluid} \tag{21}$$

$$CaCl_2 = 0{,}001 \times 93\ 371 = 93{,}4\ kg/m^3 \tag{22}$$

$$[\,CaCl_2 = 0{,}000\ 35 \times 93\ 371 = 32{,}7\ lb/bbl\,] \tag{23}$$

E.5.8 Total sodium chloride content.

$$c\left(NaCl\right)_{TOT} = 1{,}65 \times 10\ 528 = 17\ 371\ mg/l \tag{25}$$

$$NaCl = 0{,}001 \times 17\ 371 = 17{,}371\ kg/m^3 \tag{26}$$

$$[\,NaCl = 0{,}000\ 35 \times 17\ 371 = 6{,}1\ lb/bbl\,] \tag{27}$$

E.5.9 Mass fraction calcium chloride, as a percent.

$$w_{CaCl_2} = \frac{100 \times 93\ 371}{93\ 371 + 17\ 371 + \left(10\ 000 \times 25{,}0\right)} = 25{,}9\ \% \tag{28}$$

E.5.10 Mass fraction sodium chloride, as a percent.

$$w_{NaCl} = \frac{100 \times 17\ 371}{93\ 371 + 17\ 371 + \left(10\ 000 \times 25{,}0\right)} = 4{,}8\ \% \tag{29}$$

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

E.5.11 Density of calcium chloride and sodium chloride brine.

$$\rho_B = 0{,}997\ 07 + 0{,}006\ 504\left(4{,}8\right) + 0{,}007\ 923\left(25{,}9\right) + 0{,}000\ 083\ 34\left(4{,}8\right)\left(25{,}9\right) + 0{,}000\ 043\ 95\left(4{,}8\right)^2$$
$$+ 0{,}000\ 049\ 64\left(25{,}9\right)^2 = 1{,}28\ g/cm^3 \tag{32}$$

E.5.12 Mass concentration calcium chloride, expressed in milligrams per litre.

$$c\left(CaCl_2\right) = 10\ 000 \times 25{,}9 \times 1{,}28 = 33\ 150\ mg/l \tag{33}$$

E.5.13 Mass concentration sodium chloride, expressed in milligrams per litre.

$$c\left(NaCl\right) = 10\ 000 \times 4{,}8 \times 1{,}28 = 61\ 440\ mg/l \tag{34}$$

E.5.14 Volume fraction (as percent) of brine.

$$\varphi_B = \frac{100 \times 25{,}0}{1{,}28\ \left[100 - \left(25{,}9 + 4{,}8\right)\right]} = 28{,}2\ \% \tag{35}$$

E.5.15 Volume fraction (as percent) of corrected solids.

$$\varphi_{sol} = 100 - \left(59{,}5 + 28{,}2\right) = 12{,}3\ \% \tag{36}$$

E.5.16 Oil/water ratio (volume fraction) calculations.

$$\varphi_o = \frac{100 \times 59,5}{59,5 + 25,0} = 70,4 \text{ \% oil} \tag{37}$$

$$\varphi_w = \frac{100 \times 25,0}{59,5 + 25,0} = 29,6 \text{ \% water} \tag{38}$$

$$OWR = 70,4 / 29,6$$

E.5.17 Oil/brine ratio (volume fraction) calculations.

$$\varphi_o = \frac{100 \times 59,5}{59,5 + 28,2} = 67,8 \text{ \% oil} \tag{39}$$

$$\varphi_B = \frac{100 \times 28,2}{59,5 + 28,2} = 32,2 \text{ \% brine} \tag{40}$$

$$OBR = 67,8 / 32,2$$

E.5.18 Average density of suspended solids.

$$\overline{\rho}_{sol} = \frac{\left[100 \times 1,32\right] - \left[59,5 \times 0,84\right] - \left[28,2 \times 1,28\right]}{12,3} = 3,75 \text{ g/cm}^3 \tag{41}$$

E.5.19 Volume fraction (as percent) of weighting material.

$$\varphi_{wm} = \frac{3,75 - 2,65}{4,2 - 2,65} \times 12,3 = 8,5 \text{ \%} \tag{42}$$

E.5.20 Volume fraction (as percent) of low-gravity solids.

$$\varphi_{lg} = 12,3 - 8,5 = 3,8 \text{ \%} \tag{43}$$

E.5.21 Mass concentration weighting material.

$$c(wm) = 10 \times 4,20 \times 8,5 = 357,0 \text{ kg/m}^3 \tag{44}$$

$$[\, c(wm) = 3,5 \times 4,20 \times 8,5 = 125,0 \text{ lb/bbl} \,] \tag{45}$$

E.5.22 Mass concentration low-gravity solids.

$$c(lg) = 10 \times 2,65 \times 3,8 = 100,7 \text{ kg/m}^3 \tag{46}$$

$$[\, c(lg) = 3,5 \times 2,65 \times 3,8 = 35,3 \text{ lb/bbl} \,] \tag{47}$$

E.6 Drilling fluid containing both CaCl$_2$ and excess NaCl salts (drilling fluid D)

E.6.1 Total lime content.

$$c\left[Ca(OH)_2\right]_{TOT} = 3{,}691 \times 3{,}0 = 11{,}1 \text{ kg/m}^3 \tag{15}$$

E.6.2 Total calcium content.

$$c\left(Ca^{++}\right)_{TOT} = 4\ 000 \times 8{,}4 = 33\ 600 \text{ mg/l of drilling fluid} \tag{14}$$

E.6.3 Total chloride content.

$$c\left(Cl^-\right)_{TOT} = 10\ 000 \times 10{,}0 = 100\ 000 \text{ mg/l of drilling fluid} \tag{13}$$

E.6.4 Calcium chloride content.

$$c\left(CaCl_2\right)_{TOT} = 2{,}774 \times 33\ 600 = 93\ 206 \text{ mg/l of drilling fluid} \tag{17}$$

$$[\,c\left(CaCl_2\right)_{TOT} = 0{,}000\ 971 \times 33\ 600 = 32{,}6 \text{ lb/bbl}\,] \tag{19}$$

E.6.5 Chloride content associated with CaCl$_2$.

$$c\left(Cl^-_{CaCl_2}\right) = 1{,}77 \times 33\ 600 = 59\ 472 \text{ mg/l of drilling fluid} \tag{20}$$

E.6.6 Sodium chloride content check.

$$c\left(Cl^-_{NaCl}\right) = 100\ 000 - 59\ 472 = 40\ 528 \text{ mg/l of drilling fluid} \tag{24}$$

The positive number indicates that both CaCl$_2$ and NaCl are present in the drilling fluid; therefore, the calcium chloride content must be recalculated with the Cl$_{CaCl2}$ value.

E.6.7 Calcium chloride content

$$c\left(CaCl_2\right)_{TOT} = 1{,}57 \times 59\ 472 = 93\ 371 \text{ mg/l of drilling fluid} \tag{21}$$

$$CaCl_2 = 0{,}001 \times 93\ 371 = 93{,}4 \text{ kg/m}^3 \tag{22}$$

$$(\,CaCl_2 = 0{,}000\ 35 \times 93\ 371 = 32{,}7 \text{ lb/bbl}\,) \tag{23}$$

E.6.8 Sodium chloride content.

$$c\left(NaCl\right)_{TOT} = 1{,}65 \times 40\ 528 = 66\ 871 \text{ mg/l of drilling fluid} \tag{25}$$

$$NaCl = 0{,}001 \times 66\ 871 = 66{,}9 \text{ kg/m}^3 \tag{26}$$

$$\left[NaCl = 0{,}000\ 35 \times 66\ 871 = 23{,}4 \text{ lb/bbl}\right] \tag{27}$$

E.6.9 Mass fraction (as percent) of calcium chloride.

$$w_{CaCl_2} = \frac{100 \times 93\,371}{93\,371 + 66\,871 + (10\,000 \times 25{,}0)} = 22{,}8\,\% \tag{28}$$

E.6.10 Mass fraction (as percent) of sodium chloride.

$$w_{NaCl} = \frac{100 \times 66\,871}{93\,371 + 66\,871 + (1\,000 \times 25{,}0)} = 16{,}3\,\% \tag{29}$$

NOTE 1   For this example, according to Figure 1, the sodium chloride in solution cannot exceed 7,0 % mass fraction for this amount of $CaCl_2$ in this brine. To find the true sodium chloride in solution, w_{NaCl}, one must go through the calculation below to arrive at the w_{CaCl_2} and w_{NaCl} that agree with the Figure 1 mutual solubility restraints. In this example one must calculate 1st, 2nd and 3rd "ratios" and w_{CaCl_2} and w_{NaCl} values to achieve accuracy above 95 %.

$$1^{st}\ \text{ratio} = \frac{7{,}0\,\%\,(\text{Figure 1})}{16{,}3\,\%\,(\text{see}\ \ \text{E.6.10})} = 0{,}429 \tag{30}$$

$$1^{st}\ \text{new}\ c(NaCl)_{TOT} = 0{,}429 \times 66\,871 = 28\,687\ \text{mg/l of drilling fluid} \tag{31}$$

$$1^{st}\ \text{new}\ w_{CaCl_2} = \frac{100 \times 93\,371}{93\,371 + 28\,687 + (10\,000 \times 25{,}0)} = 25{,}1\,\% \text{ mass fraction} \tag{28}$$

$$1^{st}\ \text{new}\ w_{NaCl} = \frac{100 \times 28\,687}{93\,371 + 28\,687 + (10\,000 \times 25{,}0)} = 7{,}7\,\% \text{ mass fraction} \tag{29}$$

Read from Figure 1: NaCl = 6,0 % mass fraction.

$$2^{nd}\ \text{ratio} = \frac{6{,}0\,\%}{7{,}7\,\%} = 0{,}779 \tag{30}$$

$$2^{nd}\ \text{new}\ c(NaCl)_{TOT} = 0{,}779 \times 28\,687 = 22\,347\ \text{mg/l of drilling fluid} \tag{31}$$

$$2^{nd}\ \text{new}\ w_{CaCl_2} = \frac{100 \times 93\,371}{93\,371 + 22\,347 + (10\,000 \times 25{,}0)} = 25{,}5\,\% \text{ mass fraction} \tag{28}$$

$$2^{nd}\ \text{new}\ w_{NaCl} = \frac{100 \times 22\,347}{93\,371 + 22\,347 + (10\,000 \times 25{,}0)} = 6{,}1\,\% \text{ mass fraction} \tag{29}$$

Read from Figure 1: NaCl = 6,0 % mass fraction.

$$3^{rd}\ \text{ratio} = \frac{6{,}0\,\%}{6{,}1\,\%} = 0{,}984\ (\text{see Note 2 below}) \tag{30}$$

$$3^{rd}\ \text{new}\ c(NaCl)_{TOT} = 0{,}984 \times 21\,347 = 21\,981\ \text{mg/l of drilling fluid} \tag{31}$$

$$3^{rd}\ \text{new}\ w_{CaCl_2} = \frac{100 \times 93\,371}{93\,371 + 21\,981 + (10\,000 \times 25{,}0)} = 25{,}5\,\% \text{ mass fraction} \tag{28}$$

$$3^{rd} \text{ new } w_{NaCl} = \frac{100 \times 21\,981}{93\,371 + 21\,981 + (10\,000 \times 25,0)} = 6,0\ \% \text{ mass fraction} \tag{29}$$

NOTE 2 When the "ratio" approaches 1,00 no further trials for new values are needed. (In practice, ratios above 0,95 are satisfactory). In this example the 3rd iteration "ratio" was 0,984 and w_{CaCl_2} and w_{NaCl} were accurate enough after the 2nd trial.

E.6.11 Density of calcium chloride and sodium chloride brine.

$$\rho_B = 0,997\,07 + (0,006\,504 \times 6,0) + (0,007\,923 \times 25,5) + (0,000\,083\,34 \times 6,0 \times 25,5) + 0,000\,043\,95 \times 6,0^2$$
$$+ 0,000\,049\,64 \times 25,5^2 = 1,28\ \text{g/cm}^3 \tag{32}$$

E.6.12 Mass concenration calcium chloride, expressed in milligrams per litre.

$$c(CaCl_2) = 10\,000 \times 25,5 \times 1,28 = 326\,400\ \text{mg/l} \tag{33}$$

E.6.13 Mass concenration sodium chloride, expressed in milligrams per litre.

$$c(NaCl) = 10\,000 \times 6,0 \times 1,28 = 76\,800\ \text{mg/l} \tag{34}$$

E.6.14 Volume fraction (as percent) of brine.

$$\varphi_B = \frac{100 \times 25,0}{1,28 \left[100 - (25,5 + 6,0) \right]} = 28,5\ \% \tag{35}$$

E.6.15 Volume fraction (as percent) of corrected solids.

$$\varphi_{sol} = 100 - (59,5 + 28,5) = 12,0\ \% \tag{36}$$

E.6.16 Oil/water ratio (as volume fraction) calculations.

$$\varphi_o = \frac{100 \times 59,5}{59,5 + 25,0} = 70,4\ \% \text{ oil} \tag{37}$$

$$\varphi_w = \frac{100 \times 25,0}{59,5 + 25,0} = 29,6\ \% \text{ water} \tag{38}$$

$$OWR = 70,4/29,6$$

E.6.17 Oil/brine ratio (as volume fraction) calculations.

$$\varphi_o = \frac{100 \times 59,5}{59,5 + 28,5} = 67,6\ \% \text{ oil} \tag{39}$$

$$\varphi_B = \frac{100 \times 28,5}{59,5 + 28,5} = 32,4\ \% \text{ brine} \tag{40}$$

$$OBR = 67,6 / 32,4$$

E.6.18 Average density of suspended solids.

$$\overline{\rho}_{sol} = \frac{\left[100 \times 1,32\right] - \left[59,5 \times 0,84\right] - \left[28,5 \times 1,28\right]}{12,0} = 3,79\,\text{g/cm}^3 \tag{41}$$

E.6.19 Volume fraction (as percent) of weighting material.

$$\varphi_{wm} = \frac{3,79 - 2,65}{4,2 - 2,65} \times 12,0 = 8,8\,\% \tag{42}$$

E.6.20 Volume fraction (as percent) of low-gravity solids.

$$\varphi_{lg} = 12,0 - 8,8 = 3,2\,\% \tag{43}$$

E.6.21 Mass concentration of weighting material.

$$c\left(wm\right) = 10 \times 4,20 \times 8,8 = 369,6\;\text{kg/m}^3 \tag{44}$$

E.6.22 Mass concentration of low-gravity solids.

$$c\left(lg\right) = 10 \times 2,65 \times 3,2 = 84,8\,\text{kg/m}^3 \tag{46}$$

Annex F
(informative)

Sampling, inspection and rejection of drilling materials

F.1 General

The following procedure provides sampling, inspection and rejection methods for drilling fluid materials conforming to ISO 13 500 [2]. It is applicable to barite, haematite, 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   Take 15 or more samples of 0,5 kg (1,1 lb) per lot to be combined as the test sample (see F.4).

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

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

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

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

F.3 Sampling powdered material in bulk

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

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

F.3.3   Take at least one sample from each container holding less than 20 000 kg (20 metric tonnes), up to lots of 10 containers. The total number of samples taken from each lot of 100 000 kg (100 metric 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 measurement(s) on his test sample in accordance with the specific test procedure(s) outlined in ISO 13500 [2].

F.6 Inspection

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

F.7 Rejection

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

F.8 Retesting

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

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

Annex G
(informative)

Rig-site sampling

G.1 General

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

G.2 Bottom-flow sampling (preferred)

G.2.1 Apparatus

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

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

c) **Suitable c**ontainer, e.g. a tie-bag, constructed such that it retains all fines.

G.2.2 Procedure

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

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

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

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

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

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

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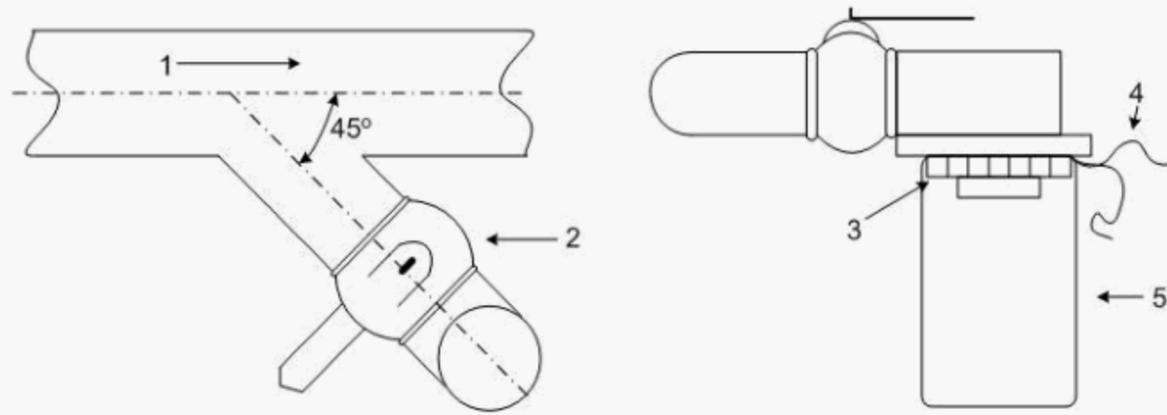
Key

1   Flow direction

2   5 cm (2 in) ball valve in 5 cm (2 in) side pipe

3   Bead or lip to hold bag

4   Draw string

5   Canvas bag 14 cm × 30 cm (5,5 in × 12 in)

Figure G.1 — Side-stream sampling device

G.3 Grab sampling

G.3.1 Apparatus

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

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

G.3.2 Procedure

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

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

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

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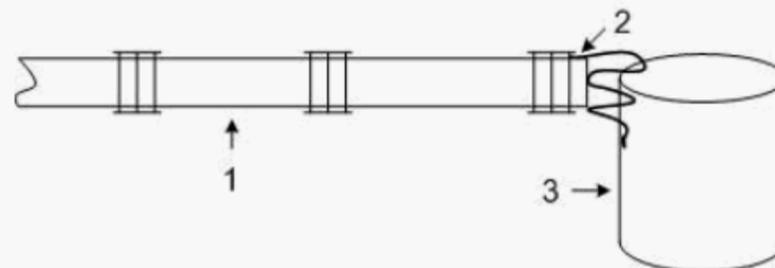
Key

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

2   Strap hinge

3   Metal can

Figure G.2 — Sample scoop

Annex H
(informative)

Determination of cutting activity by the Chenevert method

H.1 Principle

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

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

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

H.2 Reagents and apparatus

a) **Saturated solutions** of the salts listed in Table H.1.

Table H.1 — Saturated salt solutions

Salt	CAS No.	a_w at 25 °C (77 °F)	Concentration g/100 cm^3 water
Calcium chloride	10043-52-4	0,295	100
Calcium nitrate	10124-37-5	0,505	200
Sodium chloride	7647-14-5	0,753	200
Potassium nitrate	7757-79-1	0,938	200

1) Each salt solution should be prepared by adding the number of grams of salt indicated in Table H.1 to 100 cm^3 of distilled or deionized water, stirring at temperatures between 65 °C and 90 °C (150 °F and 200 °F) for 30 min, then cooling to a temperature between 25 °C and 27 °C (75 °F and 80 °F). Solid salt shall be visible in the liquid after equilibration.

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

b) **Desiccant**, anhydrous calcium chloride (CAS No. 10043-52-4).

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

　　1) The relative humidity sensor probe should be suitable for insertion in the air space of a jar containing the sample, as shown in Figure C.1.

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

　　3) The instrument should give a reading within 10 % of the true activity within 30 min, when measuring the brine activity of the standard salt solutions in C.2 a).

d) **"Base oil" supply**, 1,0 l to 1,5 l.

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

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

f) **Small wide-mouth jar**, with a volume of approximately 150 cm^3, which has a liquid surface area of approximately 38 cm2 (5,9 in2). The container should have a flexible elastomer cap which, when bored through, will form an airtight seal around the shaft of the sensor probe.

g) **Insulating cover**, made of synthetic foam, for the wide-mouth jar. The cover should fit snugly and should insulate the side and bottom of the jar from temperature fluctuations. Alternatively, the jar can be kept in a constant-temperature water bath.

h) **Linear** graph paper.

i) **Paper towels**, one or more rolls.

j) **Washing vessel**, plastic bottle with a volume of 500 cm^3 and with a screw lid.

k) **Sieve,** with openings of 0,589 mm (28 mesh).

l) **Graduated beaker**, 400 cm^3.

H.3 Procedure

H.3.1 Calibration of hygrometer

H.3.1.1　　Prepare each known relative humidity standard. A total volume of 40 cm^3 is sufficient for each sample container. These containers shall be kept firmly capped to prevent spillage, evaporation or contamination of the solutions.

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

H.3.1.3　　Transfer the probe and the jar cap to the first (lowest activity a_w) standard solution and allow 30 min for equilibrium to be reached. Temperature of the standards should be maintained at 24 °C to 25 °C (75 °F to 77 °F).

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

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

H.3.1.6 Allow each standard to equilibrate the same length of time. Record the temperature and percent relative humidity (% RH) indicated.

H.3.1.7 When complete data are obtained, plot % RH versus a_w on linear graph paper.

H.3.2 Measurement of oil-contaminated cuttings activity

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

H.3.2.2 Collect a fresh sample of cuttings directly from the shale shaker screen, taking care to minimize its exposure to air. Minimize the amount of oil-based drilling fluid adhering to the cuttings.

H.3.2.3 Add 300 cm^3 of clean, water-free base oil, which is at room temperature, to the 500 cm^3 bottle.

H.3.2.4 Measure approximately 100 cm^3 loosely-packed, fresh cuttings (H.3.2.2) into the 400 cm^3 graduated beaker. Transfer these to the bottle containing the base oil and fasten the lid.

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

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

H.3.2.7 Again, add 300 cm^3 of clean, room-temperature base oil to the bottle containing the same cuttings, and repeat steps in H.3.2.5 and H.3.2.6. Continue these washing steps until the oil looks clean after shaking with the cuttings.

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

H.3.2.9 Quickly transfer 35 cm^3 to 45 cm^3 of the washed cuttings into the 150 cm^3 hygrometer container. Place the sample container into the insulating foam jacket or constant-temperature water bath. Install the cap.

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

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

H.4 Calculation of cuttings activity (a_{ws})

Find the percent relative humidity obtained in H.3.2.11 on the graph plotted in H.3.1.7. Read and record the corresponding cuttings activity (a_{ws}) that appears on the graph.

Annex I
(informative)

Chemical analysis of active sulfides by the Garrett gas train method

I.1 Principle

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

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

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

I.2 Reagents and apparatus

a) **Citric acid (CAS No. 77-929)/demulsifier/isopropanol (CAS No. 67-63-0) solution**. Prepare a 2 mol/l citric acid solution by dissolving 420 g reagent-grade citric acid in deionized water to make 1 000 cm^3 of solution. Mix 200 cm^3 of isopropanol and 25 cm^3 of demulsifier[5], into 1 000 cm^3 of 2 mol/l citric acid solution.

b) **Anti-foam**, octanol (CAS No. 111-87-5) in a dropper bottle.

c) **Acetone** (CAS No. 67-64-1).

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

e) **Dräger H$_2$S analysis tubes**

   1) Low-range tube, marked H_2S 100/a (No. CH-291-01);

   2) High-range tube, marked H_2S 0,2 %/A (No. CH-281-01).

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[4]   Dräger tubes are an example of suitable products available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by API of these products

[5]   Schlumberger Dowell W35 is an example of a suitable product available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by API of these products.

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

1) Body

   (a) Chamber 1:

      (1) Depth   90 mm (3,54 in)

      (2) Diameter 39 mm (1,52 in)

   (b) Chambers 2 and 3:

      (1) Depth   90 mm (3,54 in)

      (2) Diameter 30 mm (1,18 in)

   (c) Passages between chambers:

      Diameter   2,0 mm (0,08 in)

   (d) Material:

      Transparent material or glass which is inert to acid, sulfides and hydrogen sulfide gas.

   (e) Dispersion tube

      (1) Stem:

         1. Diameter 8,0 mm (0,315 in)

         2. Length   150 mm (5,9 in)

      (2) Dispersion frit (bell-shaped, fine):

         Diameter   30 mm (1,18 in)

      (3) Material:

         Low coefficient of expansion, heat-resistant glass.

g) **Flow meter**, floating-ball type preferred, capable of measuring 300 cm^3/min of CO_2 gas.

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

i) **Fittings and rigid tubing**, inert to hydrogen sulfide and acid.

j) **Syringe**, 10 cm^3, constructed of glass or plastic that is inert to oil-based drilling fluid and sulfides.

k) **Graduated cylinder**, 25 cm^3.

l) **Magnetic stirrer**, 2,5 cm (1 in) stirring bar, coated with glass or PTFE.

m) **Stirring bar**, coated with PTFE.

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

o) **Soft brush**.

p) **Pipe cleaners.**

I.3 Procedure

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

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

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

I.3.4   Add 20 cm^3 of the citric acid/demulsifier/isopropanol solution [I.2. a)] into Chamber 1.

I.3.5   Add 10 drops of anti-foam into Chamber 1.

I.3.6   See Table I.1 for sample volume and type of Dräger tube required for the expected range of sulfide. Select the proper type of Dräger tube. Break the tip off each end of the tube.

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

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

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

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

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

I.3.11   Use a syringe without a needle to withdraw the drilling fluid sample volume, as determined in I.3.6, plus 0,5 cm^3 of excess drilling fluid.

NOTE   The excess drilling fluid compensates for the hold-up volume inside the injection tube, which is approximately 0,5 cm^3.

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

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

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

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

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

I.3.17   Restrain the syringe plunger again and restart carrier gas flow. Adjust the flow rate to 200 cm^3/min to 400 cm^3/min by keeping the flow meter ball between the marks.

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

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

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

Table I.1 — Dräger tube (or equivalent) identification, sample volume, and tube factors to be used for various sulfide concentration ranges

Sulfide concentration range	Sample volume	Dräger tube identification	Tube factor[a]
mg/l	cm^3	(see tube body)	(used in calculation)
1,2 to 24	10,0	H_2S 100/a	0,12
2,4 to 48	5,0	H_2S 100/a	0,12
4,8 to 96	2,5	H_2S 100/a	0,12
30 to 1 050	10,0	H_2S 0,2 %/A	1 500
60 to 2 100	5,0	H_2S 0,2 %/A	1 500
120 to 4 200	2,5	H_2S 0,2 %/A	1 500

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

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

I.4 Calculation

Using the measured drilling fluid sample volume (V_s) in millilitres, the Dräger tube's maximum darkened length (l) in units marked on the tube and the tube factor (f) from Table I.1, calculate the mass concentration of active sulfides in the drilling fluid sample:

$$\text{Active sulfides, in milligrams per litre} = \frac{l \cdot f}{V_s} \qquad (I.1)$$

Annex J
(informative)

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

J.1 General

This annex suggests methods for calibration of field testing equipment not covered under test procedures in this Standard.

J.2 Calibration of apparatus

J.2.1 Volumetric glassware

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

J.2.2 Thermometers

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

J.2.3 Viscometers

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

J.2.4 Drilling fluid balance

Calibrate drilling fluid balances using deionized water, as specified in the test procedure given in 5.2 a). The recommended frequency is prior to each set of measurements, or as specified by the operator, drilling fluid company or other interested party.

J.2.5 Retort kit cup

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

J.3 Procedure — Calibration of graduated cylinders

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

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

J.3.3 Fill the receiver with distilled water, while it is on the balance, to the various graduation marks in increments of 2 cm^3: 2 cm^3, 4 cm^3, 6 cm^3, 8 cm^3, 10 cm^3 for the 10 cm^3 receiver and in increments of 4 cm^3: 4 cm^3, 8 cm^3, 12 cm^3, 16 cm^3, 20 cm^3 for the 20 cm^3 receiver. Use a pipette or syringe to carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.

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

J.3.5 Calculate the volume of the receiver at each mark, V_{mark}:

$$V_{mark} = m_{iv} / \rho_w \tag{J.1}$$

where

V_{mark} is the receiver volume at specific mark, expressed in millilitres;

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

ρ_w is the water density, expressed in grams per cubic centimetres, at the test temperature (see Table J.1).

J.4 Procedure — Calibration of thermometers

J.4.1 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) capacity or more, filled with water, in a room where temperature is relatively constant] and allow to equilibrate for 30 min.

J.4.2 Read both thermometers and record the readings.

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

J.4.4 Calculate the average reading for each thermometer.

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

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

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

J.4.6 Calculate the thermometer correction as follows:

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

where

C is the correction value to add to thermometer reading;

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 degrees Celsius:

$R_1 = 23{,}9\ °C$

$R_2 = 24{,}2\ °C$

$C = 23{,}9 - 24{,}2 = -0{,}3\ °C$

EXAMPLE 2   Thermometer correction application:

$C = -0{,}3\ °C$

$R = 25{,}0\ °C$

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

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

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

J.5 Procedure — Calibration of viscometers

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

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

NOTE   Water will contaminate the standard fluid.

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

Correct or replace the rotor if it wobbles excessively.

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

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

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

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

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

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

J.6   Procedure — Calibration of retort kit cups

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

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

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

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

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

$$V_{RC} = m_w / \rho_w \qquad\qquad\qquad (J.3)$$

where

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

    m_w is the mass of water, expressed in grams;

    ρ_w is the water density, expressed in grams per cubic centimetres, at the test temperature (see Table J.1).

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

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

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

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

Annex K
(informative)

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

K.1 Principle

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

K.1.1.2 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.

CAUTION The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded-end caps and those with set-screw-secured end caps. Among them they have a total of five different pressure ratings. For safety, it is imperative that the operator know the maximum operating pressure of the equipment with certainty, and that this pressure not be exceeded. If in doubt, contact the manufacturer, or use the lowest of possible limits.

K.1.1.3 On all PPAs with a threaded-end cap, manufacturers have modified the hydraulic pressurisation system with a means of pressure relief. These might be available on the earlier models using a set-screw-secured end cap. This should relieve the hydraulic pressure should it approach the sample cell maximum pressure rating. The operator should familiarise himself with this portion of the apparatus and ensure that it is working properly.

K.1.1.4 Safe operation of the PPA requires that the operator understand and practice correct assembly and operation of the equipment. Improper assembly, incorrect operation, or the use of defective parts can create the possibility of cell leakage or failure, resulting in serious injury or equipment damage.

K.1.1.5 The sample cell is hot during operation. The operator must be aware of the hot areas and avoid contact with them. Burns can result from touching parts of the equipment during normal operation. These instruments are electrically heated, and, as with any electrical device, if the wiring is damaged or faulty, electrical shorts can occur and create the risk of fire, injury, and equipment damage. Use these devices only on grounded circuits.

K.1.2 Safe operation of the hydraulic pressurisation system

K.1.2.1 Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before doing the following:

a) Attempting to disconnect the pressure hose from cell at quick coupler;

b) Attempting to remove the 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.

K.1.2.2 After refilling or repairing the hydraulic system, clean any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

K.1.2.3 Make sure, when assembling the cell, that the end cap set screws are properly aligned and tightened.

K.1.3   Safe pneumatic pressurisation of back-pressure receiver

K.1.3.1 Always use either nitrogen or carbon dioxide to pressurise the back-pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gas. If nitrogen is used, it must be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system must be built into the laboratory. Nitrogen cylinders must be secured to meet safety standards. CO_2 is normally supplied in small, pressurized cartridges at about 6 200 kPA (900 psi). They are primarily used for field operations. Do not allow these cartridges to be heated or exposed to fire. They can explode if overheated.

K.1.3.2 Maintain pressure regulators and gages in good condition. Never use oil on pressure regulators.

K.1.3.3 Repair or replace leaking hydraulic or pneumatic pressurisation systems. Gages, fittings, and hoses must be kept in good condition, and leaks 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.

K.1.3.4 When pressurising the back-pressure assembly, always open the supply pressure first, then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the equipment rating or relief valve settings. When relieving back pressure, shut off the supply pressure, bleed the pressure from the system, then back out the regulator t-screw.

K.1.4   Safe heating

K.1.4.1 Caution must be exercised 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.

K.1.4.2 The practice of removing the cell and cooling it with water is dangerous and should be avoided. Serious injuries can be cause by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

K.1.5   Safe electrical operation

K.1.5.1 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.

K.1.5.2 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.

K.1.6   Safe test cell maintenance

The filtration cell is a pressure vessel. Consider it to be a source of potential danger. The following safety precautions listed to ensure safe operation:

a)   cell material should be compatible with the test samples;

b)   never use cells that show signs of severe pitting or stress cracking;

c)   never use cells, cell caps, or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage;

d)   use only undamaged, hardened steel set screws. Others are unsafe.

K.2 Apparatus

K.2.1 Permeability plugging apparatus

K.2.1.1 PPA is designed to provide improved static filtration measurements and can be operated at pressures and temperatures approximating those prevailing downhole. It permits the use of filtration media chosen to simulated exposed sands as closely as possible.

K.2.1.2 The fluid cell is inverted and the pressure is applied to the bottom of the cell. The filter medium is at the top end of the cell, through which the filtrate is collected.

K.2.1.3 Pressure is applied to the cell by a small hydraulic hand pump. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Hydraulic oil/sample contamination is prevented by redundant o-ring seals on the piston.

CAUTION 1 There are three different types of cells 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 "2500 psi." In 1996, the rating on these cells was reduced to 12 420 kPa (1 800 psi). Unless the user is absolutely certain that the cell to be used is rated at 13 800 kPa (2 000 psi), the 12 420 kPa (1 800 psi) pressure limit must be observed.

CAUTION 2 It is imperative that the manufacturer's recommendations concerning maximum temperature, pressure and sample size be followed. Failure to do so can lead to serious injury. Attach the operating manual, or this procedure, to the apparatus. Before using the PPA, these instructions must be read by anyone who is unfamiliar with the equipment. If the user is unable to determine the operating limits with certainty, the lowest pressure limit must be assumed to be applicable.

NOTE As received from the manufacturer, the PPA will be equipped with valves that are rated to 260 °C (500 °F). Should is become necessary to change any valves during the life of this equipment it is imperative that the replacements be designed and rated for use at 260 °C (500 °F).

K.2.1.4 Test pressures are limited by the safety limits of the cells as specified by the manufacturer. See K.2.1.3

CAUTION The pressure is usually either 12 420 kPa or 13 800 kPa (1 800 psi or 2 000 psi) at some defined temperature. If back pressure is used in the test, the test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

NOTE Cell damage caused by excessive pressure is categorised as end cap bending, end cap compression, cylinder shear, or cylinder stress. End cap bending can be detected visually or by measurement. End cap compression can be detected by distortion of the set screw holes or seats, which become oval rather than round. Caps showing signs of damage must not be used and must be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set screw holds, must not be used.

K.2.1.5 For temperatures above 95 °C (200 °F), the back pressure receiver must be pressurised to prevent boiling of the filtrate. The standard back pressure receiver used a CO_2 pressurising source to provide the back pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

K.2.1.6 The PPA cell is encased in a thermostatically controlled aluminium well during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 250 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the wall of the cell. 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 know to the same reference setting.

K.2.1.7 The standard cells for the PPA filter press are made of stainless steel. Power consumption for the heating jacket is 800 J (800 w).

K.2.2 Filter medium

K.2.2.1 The PPA can use any one of a number of filtration media, including porous ceramic or sintered metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from 100 mD to 100 D. The use of media that simulate exposed sand faces, together with the use of

relevant test pressures and temperatures, provide the user with a greatly improved picture of what is happening downhole.

K.2.2.2 The filter medium can be any porous materials such as ceramic, sintered metal, or resin-coated sand disks, graded sands, or core samples. Standard disk thickness if 6,4 mm (0,25 in) but with adapters, thicker disks can be used. A new disk is required for each test. For oil-based drilling fluids, soak the disk for 5 min to 10 min in a sample of the base oil prior to use. Vacuum saturation should be used for filter media with low porosity and permeability.

K.2.2.3 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 fo fit the apparatus cylinder and are usually 6,4 mm (0,25 in) thick. With modification of the cylinder, 25,4 mm (1,00 in) cores can also be used.

K.2.2.4 Resin coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The san should be heated at 150 °C (300 °F) for 1 hr to 3 hr in moulds slightly larger than the normal disk size, and either 6,4 mm or 25,4 mm (0,25 in or 1,00 in) thick. The moulds should 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 materials to be used to control seepage losses to severe fluid loss environments.

K.2.2.5 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.

K.2.2.6 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 should be saturated 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 1 hr to 3 hr at 150 °C (300 °F), cooled, and then inverted for the test.

K.2.2.7 There is an 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, up 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.

NOTE   Procedure for ceramic disk comparison – install a disk in a PPA cell and fill the cell with water. Using the air permeability equipment, with the upper cell valve closed, adjust the pressure on 207 kPa (30 psi) test gage to 27,6 kPa to 31,0 kPa (4,0 psi to 4,5 psi). Open the valve on top of the cell and adjust pressure to 13,8 kPa ± 1,4 kPa (2,0 psi °± 0,1 psi). After opening valve at the bottom of the cell, readjust pressure with the upper valve to 13,8 kPa ± 1,4 kPa (2,0 psi °± 0,1 psi). Measure time for 300 cm^3 to pass through using a 500 cm^3 graduated cylinder. Timing from the 100 cm^3 mark to the 400 cm^3 mark. If the PPT (Permeability Plugging Test) is to be used for comparison purposes, run several disks classify the disks, and use those of similar values.

K.2.2.8 Timer, 30 min interval.

K.2.2.9 Thermometer, range up to 260 °C (500 °F).

K.2.2.10 Graduated cylinder (TC), 25 cm^3 or 50 cm^3.

K.2.2.11 High speed mixer.

K.3 Procedure — Preheating the heating jacket

K.3.1 Connect the power cord to the proper voltage as indicated on the nameplate.

K.3.2 Turn the thermostat to the midscale.

K.3.3 Place a metal stem dial thermometer in the thermometer well of the heating jacket.

K.3.4 The pilot lamp will light when the heating jacket temperature has reached the thermostat setting.

K.3.5 Adjust the thermostat to 5,6 °C (10 °F) over the desired test temperature.

K.4 Procedure — Loading the filtration cell

K.4.1 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.

CAUTION   The filtration cell is a pressure vessel. Te following safety precautions must be followed to ensure safe operation: the cell material must be resistant to the test sample. Cell bodies that show signs of stress cracking or severe pitting must not be used. Use only undamaged, hardened steel set screws. The use of damaged, or common, mild steel set screws is hazardous.

NOTE   As received from the manufacturer, the PPA will be equipped with valves that are rated to 260 °C (500 °F). Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F).

K.4.2 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 sorter 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.

K.4.3 Fill the space above the piston with hydraulic oil to just above the end face.

K.4.4 Install the hydraulic end cap onto the bottom of the cell. Push in on the back pressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to slide into the cell more easily. Install and tighten the set screws.

NOTE   Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

K.4.5 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.

NOTE 1   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.

NOTE 2   For improved consistency in test results, stir drilling fluid for five minutes immediately before loading the cell.

K.4.6 Turn the cell upright and fill with approximately 275 cm^3 of drilling fluid. This allows for expansion while heating. Do not exceed this amount.

K.4.7   Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the o-ring recess.

K.4.8   Install the o-ring and set the selected disk, or other filtering medium on top of it.

NOTE   The disk should be soaked in the base oil for at least five minutes before use. Never reuse disks.

K.4.9   Position the top end cap in the cell, tighten the set screws snugly, and close the valve on the top end cap.

CAUTION   Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurisation. During heating, the pressure in the cell must be controlled by periodically bleeding off the excess.

K.4.10   Unless it was installed in K.4.6 above, 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 fo the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

K.5 Procedure — Pressurising the cell

K.5.1   Refer to Table K.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.

NOTE   Filtration at temperatures above the boiling point of the fluid sample requires the use of the back pressure receiver to prevent vapourisation of the filtrate. It also requires that the sample be pressurised to prevent it from boiling.

CAUTION   Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurisation. During heating, the pressure in the cell must be controlled by periodically bleeding off the excess.

Table K.1 – Starting cell pressures and back pressures for test temperatures

Temperature		Heating Pressure or Back Pressure	
°C	°F	kPa	psi
Less than 93	Less than 200	0	0
93-149	200-300	690	100
150-176	301-350	1035	150
177-190	351-375	1380	200
191-204	376-400	1725	250
205-218	401-425	2415	350
219-232	426-450	3100	450
233-246	451-475	3800	550
247-260	476-500	4825	700
NOTE Nitrogen manifolds having a (600 psi) gauge for back pressure need to be modified to attain the (700 psi). However, tests are possible within the limits of the manifolds.			

K.5.2   While the cell is heating, prepare the back pressure receiver.

a)   Check to ensure that the regulator t-screw has been rotated counterclockwise enough to release all pressure. When the pressure has been released, the screw will turn freely.

b)   Open the pressure release valve to relieve any remaining pressure and remove the CO2 cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one, and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time.

c)   Verify that the pressure release valve on the CO2 assembly and the filtrate drain valve are closed.

d) Set the back pressure assembly aside. It will be installed at K.5.4.

K.5.3 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 an hour.

K.5.4 When the cell is at the desired temperature and cell pressure stabilised, mount the back pressure receiver on the upper valve adapter and secure it with a retaining pin. Then install the CO_2 pressurising unit on top of the receiver and lock it in place with the remaining retaining pin.

K.5.5 If a drain hose is to be used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

NOTE To ensure accurate measurements, the space between the filtration medium and the back pressure receiver outlet and the receiver valve must be filled with the base fluid before starting the test. This will ensure that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

K.5.6 Refer to Table K.1 to determine the appropriate pressure for the back pressure receiver, and apply it by turning the t-screw on the pressure regulator until the desire pressure is reached.

K.5.7 Actuate the pump to raise the cell pressure to the desired level. Then open the valve between the cell and the back pressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the back pressure receiver.

K.6 Procedure — Conducting the filtration test

K.6.1 A laboratory timer should be used and filtrate should be collected. Record the volume at 1 min, 7,5 min and 30 min. Plotting this data against the square root of time provides a useful description of the spurt loss. If desired, samples can be taken more frequently, but no should be taken before one minute. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

NOTE For improved definition of spurt loss, collect filtrate at 1 min, 5 min, 7,5 min, 15 min, 25 min, and 30 min. Plot cumulative filtrate volumes versus the square root of time.

K.6.2 Begin filtration by opening the valve between the cell and the back pressure receiver. Verify that both the cell pressure, as indicated on the pump gage, and the back pressure are at desired levels. Adjust them as necessary throughout the test.

K.6.3 Cell pressure can be expected to decrease slightly as filtration reduces the cell contents, and from any leakage at the pump. The pump should be used as necessary to maintain the pressure at the desired level. If a manually operated pump is used, it should be operated at about one stroke per second.

K.6.4 After each interval, the filtrate should be drained into the graduated cylinder from the back pressure receiver, and the time and cumulative volume should be recorded.

NOTE It is recommended that the filtrate be recovered directly from the back pressure receiver, not through a drain hose attached at the discharge valve. If a hose must be used, minimise its length to reduce the error caused by liquid retention within the hose.

K.6.5 After 30 min, close the filtrate valve and drain all of the filtrate from the back pressure receiver into the graduated cylinder. Record the total volume of filtrate in the graduated cylinder.

K.7 Procedure — Finalising test and cell disassembly

K.7.1   Allow the pressurised cell assembly to cool in the heating jacket. When these test 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 availably which should be used any time a hot cell is to be handled.

CAUTION   Do not proceed with disassembly until the temperature of the sample in the cell has been reduced to less than 38 °C (100 °F).

NOTE   Extreme care must be exercised in cooling hot cells. This procedure, as recommended, makes it difficult to perform more than one test in an eight hour work day with a single PPA. In the interest of improving productivity, users may want to design their own cell cooling procedures and equipment. Safety must be the primary consideration in these designs.

K.7.2   Close the valve between the cell and the back pressure receiver.

K.7.3   Release the pressure on the pump and cell by opening the pump valve. Disconnect the quick coupler between the pump and the nipple adapter on the bottom of the cell.

K.7.4   Release the back pressure by turning the t-screw on the regulator counterclockwise until it turns freely.

K.7.5   Bleed the pressure from the back pressure receiver by opening the pressure relief valve on the CO_2 unit.

K.7.6   Open the drain valve on the back pressure receiver and collect the last few drops of filtrate in the graduated cylinder.

K.7.7   After removing the locking pin securing it, remove the CO_2 assembly from the top nipple adapter.

K.7.8   After removing its locking pin, remove the back pressure receiver.

K.7.9   The cell may be opened after its contents have been allowed to cool.

CAUTION   If it is suspected that the cell is still pressured, and the lower end cap does not include a screen, the following procedure can be used to verify the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell. 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 pressurised, it will be obvious from the force required to move the piston.

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 user is confident that the contents are no longer under pressure.

NOTE   The cell may be raised in the heating jacket and placed on the cell support, or laid on a bench, while it is being opened.

K.7.10   Loosen the six set screws securing the cap and using the valve and nipple adapter as a handle, pull the cap from the cell. If it is stuck, a rocking motion of the valve and nipple adapter may free it. If it cannot be freed in this manner, unscrew the valve and nipple adapter, install the cap removal tool in its place, and use it to remove the cap.

K.7.11   The end cap must be removed with the cell in the vertical position and the filtration end of the cell facing upwards.

K.7.12   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 the base oil, then measure and record its thickness and remarks concerning its composition.

K.7.13 Dispose of the drilling fluid and wash the inside of the cell with a suitable solvent. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 150 °C (300 °F) or higher.

NOTE   If testing was conducted at temperatures above 150 °C (300 °F), the o-rings must be replaced. 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 K.7.10.

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. Not that the floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all the o-rings on the piston and the cap.

c) Clean the parts for reuse.

K.8 Calculation — Data reporting

K.8.1   Filtrate reporting

Report the actual cumulative filtrate volume, in cm^3, collected through each selected time period.

K.8.2   Spurt loss reporting

The spurt loss is the volume of fluid that passes through the filtration medium before a filter cake is formed. It can be depicted by the intercept, on the y-axis, of the straight line representing the static filtrate rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume. The filtrate volume should be corrected to a filter area of 4 580 mm^2 (7,1 in^2). PPA filter cells usually have half the standard filter area or 2 258 mm^2 (3,5 in^2), thus double the observed volume before reporting. Alternatively, an approximate value can be calculated using the equation in K.9.

NOTE   To more accurately define the spurt loss, collect and record the filtrate more frequently, and plot the data as described in the NOTE following K.6.1.

K.8.3   Filter cake reporting

Measure and record the filter cake thickness to the nearest 0,8 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.

K.9 Calculation — filtrate volume and filtration rate

$$V_t = 2\,V \tag{K.1}$$

$$V_i = 2\,[V_1 - (V_2 - V_1)] \tag{K.2}$$

$$R = 2\,[(V_2 - V_1)]\,/\,2{,}739 \tag{K.3}$$

where:

V volume of filtrate collected in receiver, cm^3

V_1 volume of filtrate collected after 7,5 min, cm^3

V_2 volume of filtrate collected after 30 min, cm^3

V_t PPT value, cm^3

V_i spurt loss, cm^3

R static filtration rate

NOTE   Observe that all three parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard ISO/API low pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant, 2, can be modified as necessary to accommodate tests made utilizing other filtration area filter media.

Annex L
(informative)

High-temperature/high-pressure filtration testing of oil-based drilling fluid using the permeability plugging apparatus and cells equipped with threaded-end caps

L.1 Principle

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

L.1.1.2   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.

CAUTION The pressure limitation in the use of the PPA depends upon the sample cell in use. There are two types of cells available: those with threaded-end caps and those with set-screw-secured end caps. Among them they have a total of five different pressure ratings. For safety, it is imperative that the operator know the maximum operating pressure of the equipment with certainty, and that this pressure not be exceeded. If in doubt, contact the manufacturer, or use the lowest of possible limits.

L.1.1.3   On all PPAs with a threaded-end cap, manufacturers have modified the hydraulic pressurisation system with a means of pressure relief. These might be available on the earlier models using a set-screw-secured end cap. This should relieve the hydraulic pressure should it approach the sample cell maximum pressure rating. The operator should familiarise himself with this portion of the apparatus and ensure that it is working properly.

L.1.1.4   Safe operation of the PPA requires that the operator understand and practice correct assembly and operation of the equipment. Improper assembly, incorrect operation, or the use of defective parts can create the possibility of cell leakage or failure, resulting in serious injury or equipment damage.

L.1.1.5   The sample cell is hot during operation. The operator must be aware of the hot areas and avoid contact with them. Burns can result from touching parts of the equipment during normal operation. These instruments are electrically heated, and, as with any electrical device, if the wiring is damaged or faulty, electrical shorts can occur and create the risk of fire, injury, and equipment damage. Use these devices only on grounded circuits.

L.1.2   Safe operation of the hydraulic pressurisation system

L.1.2.1   Make sure the hydraulic pressure has been released and that the pressure gauge on the pump reads zero, before doing the following:

a)   Attempting to disconnect the pressure hose from cell at quick coupler;

b)   Attempting to remove the 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.

L.1.2.2 After refilling or repairing the hydraulic system, clean any spilled oil. Oil left on floors is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

L.1.2.3 Make sure, when assembling the cell, that the end cap set screws are properly aligned and tightened.

L.1.3   Safe pneumatic pressurisation of back-pressure receiver

L.1.3.1 Always use either nitrogen or carbon dioxide to pressurise the back-pressure receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gas. If nitrogen is used, it must be supplied in an approved nitrogen gas cylinder, or the nitrogen supply system must be built into the laboratory. Nitrogen cylinders must be secured to meet safety standards. CO_2 is normally supplied in small, pressurized cartridges at about 6 200 kPA (900 psi). They are primarily used for field operations. Do not allow these cartridges to be heated or exposed to fire. They can explode if overheated.

L.1.3.2 Maintain pressure regulators and gages in good condition. Never use oil on pressure regulators.

L.1.3.3 Repair or replace leaking hydraulic or pneumatic pressurisation systems. Gages, fittings, and hoses must be kept in good condition, and leaks 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.

L.1.3.4 When pressurising the back-pressure assembly, always open the supply pressure first, then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the equipment rating or relief valve settings. When relieving back pressure, shut off the supply pressure, bleed the pressure from the system, then back out the regulator t-screw.

L.1.4   Safe heating

L.1.4.1 Caution must be exercised 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.

L.1.4.2 The practice of removing the cell and cooling it with water is dangerous and should be avoided. Serious injuries can be cause by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

L.1.5   Safe electrical operation

L.1.5.1 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.

L.1.5.2 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.

L.1.6   Safe test cell maintenance

The filtration cell is a pressure vessel. Consider it to be a source of potential danger. The following safety precautions listed to ensure safe operation:

a) Cell material should be compatible with the test samples;

b) never use cells that show signs of severe pitting or stress cracking;

c) never use cells, cell caps, or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage;

d) use only undamaged, hardened steel set screws. Others are unsafe.

L.2 Apparatus

L.2.1 Permeability plugging apparatus

L.2.1.1 PPA is designed to provide improved static filtration measurements and can be operated at pressures and temperatures approximating those prevailing downhole. It permits the use of filtration media chosen to simulated exposed sands as closely as possible.

L.2.1.2 The fluid cell is inverted and the pressure is applied to the bottom of the cell. The filter medium is at the top end of the cell, through which the filtrate is collected.

L.2.1.3 Pressure is applied to the cell by a small hydraulic hand pump. Pressure is transferred to the drilling fluid sample through a floating piston within the cell. Hydraulic oil/sample contamination is prevented by redundant o-ring seals on the piston.

CAUTION 1 There are two manufacturers of PPAs. Both use threaded-end caps for the cells used for tests run at pressures in excel of 13 800 kPa (2 000 psi). There are threaded caps with three different pressure ratings – 20 700 kPa, 27 600 kPa, and 34 600 kPa (3 000 psi, 4 000 psi, and 5 000 psi).

CAUTION 2 It is imperative that the manufacturers' recommendations concerning maximum temperature, pressure, and sample size be followed. Failure to do so can lead to serious injury. The operating manual, or this procedure, should be attached to the apparatus and read by anyone who is unfamiliar with the equipment before using it. If the user is unable to determine the operating limits with certainty, the lowest pressure limit must be assumed to be applicable.

NOTE As received from the manufacturer, the PPA will be equipped with valves that are rated to 260 °C (500 °F). Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F) or more.

L.2.1.4 Test pressures are limited by the safety limits of the cell as specified by the manufacturer, usually 20 700 kPa, 27 600 kPa, or 34 500 kPa (3 000 psi, 4 000 psi, or 5 000 psi) at 260 °C (500 °F). The back pressure receiver may be used at pressures as high as 5 170 kPa (750 psi). If back pressure is used in the test, the maximum test pressure may have to be reduced to avoid exceeding the pressure limit of the cell.

L.2.1.5 For temperatures above 95 °C (200 °F), the back pressure receiver must be pressurised to prevent boiling of the filtrate. The standard back pressure receiver used a CO_2 pressurising source to provide the back pressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO_2 when desired.

L.2.1.6 The PPA cell is encased in a thermostatically controlled aluminium well during heating and filtration. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 250 °C (500 °F). The cell temperature can be measured using a metal stem thermometer inserted into the well in the wall of the cell. 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 know to the same reference setting.

L.2.1.7 The standard cells for the PPA filter press are made of stainless steel. Power consumption for the heating jacket is 800 J (800 w). The PPA can be used either in the field or in a laboratory environment. A stainless steel carrying case with fold down working shelf is available for convenience in field operations.

L.2.2 Filter medium

L.2.2.1 The PPA can use any one of a number of filtration media, including porous ceramic or sintered metal disks, core samples, and beds of coated or uncoated sand. Ceramic disks are available with permeabilities ranging from 100 mD to 100 D. The use of media that simulate exposed sand faces, together with the use of relevant test pressures and temperatures, provide the user with a greatly improved picture of what is happening downhole.

L.2.2.2   The filter medium can be any porous materials such as ceramic, sintered metal, or resin-coated sand disks, graded sands, or core samples. Standard disk thickness if 6,4 mm (0,25 in) but with adapters, thicker disks can be used. A new disk is required for each test. For oil-based drilling fluids, soak the disk for 5 min to 10 min in a sample of the base oil prior to use. Vacuum saturation should be used for filter media with low porosity and permeability.

L.2.2.3   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 fo fit the apparatus cylinder and are usually 6,4 mm (0,25 in) thick. With modification of the cylinder, 25,4 mm (1,00 in) cores can also be used.

L.2.2.4   Resin coated sand can be made into a solid disk, selecting the sand size to provide the desired permeability. The san should be heated at 150 °C (300 °F) for 1 hr to 3 hr in moulds slightly larger than the normal disk size, and either 6,4 mm or 25,4 mm (0,25 in or 1,00 in) thick. The moulds should 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 materials to be used to control seepage losses to severe fluid loss environments.

L.2.2.5   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.

L.2.2.6   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 should be saturated 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 1 hr to 3 hr at 150 °C (300 °F), cooled, and then inverted for the test.

L.2.2.7   There is an 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, up 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.

NOTE   Procedure for ceramic disk comparison – install a disk in a PPA cell and fill the cell with water. Using the air permeability equipment, with the upper cell valve closed, adjust the pressure on 207 kPa (30 psi) test gage to 27,6 kPa to 31,0 kPa (4,0 psi to 4,5 psi). Open the valve on top of the cell and adjust pressure to 13,8 kPa ± 1,4 kPa (2,0 psi °± 0,1 psi). After opening valve at the bottom of the cell, readjust pressure with the upper valve to 13,8 kPa ± 1,4 kPa (2,0 psi °± 0,1 psi). Measure time for 300 cm^3 to pass through using a 500 cm^3 graduated cylinder. Timing from the 100 cm^3 mark to the 400 cm^3 mark. If the PPT (Permeability Plugging Test) is to be used for comparison purposes, run several disks classify the disks, and use those of similar values.

L.2.2.8   **Timer,** 30 min interval.

L.2.2.9   **Thermometer**, range up to 260 °C (500 °F).

L.2.2.10 Graduated cylinder (TC), 25 cm^3 or 50 cm^3.

L.2.2.11 High speed mixer.

L.3 Procedure — Preheating the heating jacket

L.3.1   Connect the power cord to the proper voltage as indicated on the nameplate.

L.3.2   Turn the thermostat to the midscale.

L.3.3   Place a metal stem dial thermometer in the thermometer well of the heating jacket.

L.3.4   The pilot lamp will light when the heating jacket temperature has reached the thermostat setting.

L.3.5   Adjust the thermostat to 5,6 °C (10 °F) over the desired test temperature.

L.4 Procedure — Loading the filtration cell

L.4.1   Use the spanner wrench to remove the end caps. Then unscrew the nipples from the caps and remove the piston from the cell.

CAUTION   The filtration cell is a pressure vessel. Te following safety precautions must be followed to ensure safe operation: the cell material must be resistant to the test sample. Cell bodies that show signs of stress cracking or severe pitting must not be used. Use only undamaged, hardened steel set screws. The use of damaged, or common, mild steel set screws is hazardous.

As received from the manufacturer, the PPA will be equipped with valves that are rated to 260 °C (500 °F). Should it become necessary to change any valves during the life of this equipment, it is imperative that the replacements be designed and rated for use at 260 °C (500 °F).

L.4.2   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 sorter recess than the top. Position the piston so that it is at or near the bottom end of the cell, then unscrew the wrench from the piston.

L.4.3   Lubricate the end face of the cell bore, the horizontal area at the end of the bore, with antiseize compound. Fill the space above the piston with hydraulic oil to just above the end face.

L.4.4   Lubricate the thread with high temperature grease. Then while pushing on the back pressure ball on the nipple of the end cap to permit air to escape, screw the end cap into place. Tighten the end cap moderately with the two-pin spanner wrench. Over tightening will not improve the seal, and will make the cap difficult to remove

L.4.5   Install the hydraulic end cap onto the bottom of the cell. Push in on the back pressure ball on the nipple of the end cap on the pressure inlet end of the cell to relieve the pressure and allow the cap to be screwed into the cell more easily.

NOTE   Some oil will flow from the threaded hole in the end cap, indicating that no air is trapped between the piston and the end cap.

L.4.6   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.

NOTE 1   The steps that follow can be accomplished in the jacket that is being preheated, in an unheated jacket (if available) or in a specially constructed stand.

NOTE 2   For improved consistency in test results, stir drilling fluid for five minutes immediately before loading the cell.

L.4.7   Turn the cell upright and fill with approximately 275 cm^3 of drilling fluid. This allows for expansion while heating. Do not exceed this amount.

L.4.8   Reconnect the pump hose to the quick-connect coupling on the nipple at the bottom of the cell and close the pressure valve on the pump. Operate the pump to raise the level of the fluid sample to the o-ring recess.

L.4.9   Install the o-ring and set the selected disk, or other filtering medium, on top of it.

NOTE　　　The disk should be soaked in the base oil for at least five minutes before use. Disks should never be reused.

L.4.10　　Install the top end cap in the cell.

NOTE　　　The following step applies only to cells that utilise retainer rings for the top end caps.

L.4.11　　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. Tighten until the outer knurled flange of the retainer ring is flush against the top of the cell body. Attempting to tighten it further will not improve the seal and will make the cap more difficult to remove.

CAUTION　Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurisation. During heating, the pressure in the cell must be controlled by periodically bleeding off the excess.

L.4.12　Unless it was installed in L.4.7 above, 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 hold in the bottom of the cell body. This prevents rotation of the cell.

L.5　Procedure — Pressurising the cell

L.5.1　　Refer to Table K.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.

NOTE　　　Filtration at temperatures above the boiling point of the fluid sample requires the use of the back pressure receiver to prevent vapourisation of the filtrate. It also requires that the sample be pressurised to prevent it from boiling.

CAUTION　Thermal expansion of its contents, and of the hydraulic fluid, will cause cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump must be connected quickly to permit the release of hydraulic fluid to prevent over-pressurisation. During heating, the pressure in the cell must be controlled by periodically bleeding off the excess.

L.5.2　　While the cell is heating, prepare the back pressure receiver.

a)　Check to ensure that the regulator t-screw has been rotated counterclockwise enough to release all pressure. When the pressure has been released, the screw will turn freely.

b)　Open the pressure release valve to relieve any remaining pressure and remove the CO2 cartridge barrel from the pressure unit. Dispose of the empty cartridge, replace it with a new one, and tighten the barrel enough to puncture the cartridge. Do not adjust the regulator at this time.

c)　Verify that the pressure release valve on the CO2 assembly and the filtrate drain valve are closed.

d)　Set the back pressure assembly aside. It will be installed at L.5.4.

L.5.3　　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 an hour.

L.5.4　　When the cell is at the desired temperature and cell pressure stabilised, mount the back pressure receiver on the upper valve adapter and secure it with a retaining pin. Then install the CO_2 pressurising unit on top of the receiver and lock it in place with the remaining retaining pin.

L.5.5　　If a drain hose is to be used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

NOTE   To ensure accurate measurements, the space between the filtration medium and the back pressure receiver outlet and the receiver valve must be filled with the base fluid before starting the test. This will ensure that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

L.5.6   Refer to Table K.1 to determine the appropriate pressure for the back pressure receiver, and apply it by turning the t-screw on the pressure regulator until the desire pressure is reached.

L.5.7   Actuate the pump to raise the cell pressure to the desired level. Then open the valve between the cell and the back pressure receiver to start the test.

NOTE   The differential filtration pressure is the difference between the pressure applied to the cell and that maintained on the back pressure receiver.

L.6 Procedure — Conducting the filtration test

L.6.1   A laboratory timer should be used and filtrate should be collected. Record the volume at 1 min, 7,5 min and 30 min. Plotting this data against the square root of time provides a useful description of the spurt loss. If desired, samples can be taken more frequently, but no should be taken before one minute. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

NOTE   For improved definition of spurt loss, collect filtrate at 1 min, 5 min, 7,5 min, 15 min, 25 min, and 30 min. Plot cumulative filtrate volumes versus the square root of time.

L.6.2   Begin filtration by opening the valve between the cell and the back pressure receiver. Verify that both the cell pressure, as indicated on the pump gage, and the back pressure are at desired levels. Adjust them as necessary throughout the test.

L.6.3   Cell pressure can be expected to decrease slightly as filtration reduces the cell contents, and from any leakage at the pump. The pump should be used as necessary to maintain the pressure at the desired level. If a manually operated pump is used, it should be operated at about one stroke per second.

L.6.4   After each interval, the filtrate should be drained into the graduated cylinder from the back pressure receiver, and the time and cumulative volume should be recorded.

NOTE   It is recommended that the filtrate be recovered directly from the back pressure receiver, not through a drain hose attached at the discharge valve. If a hose must be used, minimise its length to reduce the error caused by liquid retention within the hose.

L.6.5   After 30 min, close the filtrate valve and drain all of the filtrate from the back pressure receiver into the graduated cylinder. Record the total volume of filtrate in the graduated cylinder.

L.7 Procedure — Finalising test and cell disassembly

L.7.1   Disconnect the heating jacket from the power source.

CAUTION   Do not proceed with disassembly until the temperature of the sample in the cell has been reduced to less than 38 °C (100 °F).

NOTE   Extreme care must be exercised in cooling hot cells. This procedure, as recommended, makes it difficult to perform more than one test in an eight hour work day with a single PPA. In the interest of improving productivity, users may want to design their own cell cooling procedures and equipment. Safety must be the primary consideration in these designs.

L.7.2   Allow the pressurised cell assembly to cool in the heating jacket. When these test 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 availably which should be used any time a hot cell is to be handled.

L.7.3   Isolate the back pressure assembly from its pressure source by turning the t-screw on the back pressure regulator counterclockwise until it turns freely.

L.7.4   Open the bleed valve on the CO_2 unit to release the pressure in the back pressure receiver.

L.7.5   Remove the locking pin and remove the CO_2 pressuring assembly from the top nipple adapter.

L.7.6   Remove the locking pin and remove the back pressure receiver.

L.7.7   Open the valve on the hydraulic pump to release cell press. Disconnect the hydraulic quick coupler.

L.7.8   Open the filtration valve. This will bleed any pressure remaining between the cell filter and the back pressure receiver.

CAUTION   If it is suspected that the cell is still pressured, and the lower end cap does not include a screen, the following procedure can be used to verify the position of the floating piston. Remove the quick-connect assembly from the bottom end cap of the cell. 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 pressurised, it will be obvious from the force required to move the piston.

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 user is confident that the contents are no longer under pressure.

NOTE   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.

L.7.9   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 back pressure inlet nipple just above the filtrate valve where the back pressure receiver is normally attached. Secure the tool using the valve stem locking pin.

L.7.10   Threaded-end caps are removed using spanner wrenches. It may be necessary to tap on the wrench to get it started. Opening difficulty is an indication of insufficient lubrication, over tightening, or insufficient cleaning. It may be necessary to use a suitable holding tool such as a soft jac vice, chain wrench, strap wrench, or other similar device to secure the cell while the cap is unscrewed.

L.7.11   Reposition the cell as required so that the filter end is up, then unscrew the top cap.

L.7.12   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 the base oil. Measure and record its thickness and remarks concerning its composition.

L.7.13   Empty the cell and wash it with a suitable solvent. It is usually not necessary to remove the floating piston and the bottom end cap unless the test was run at 150 °C (300 °F) or higher.

NOTE   If testing was conducted at temperatures above 150 °C (300 °F), the o-rings must be replaced. Perform the following three steps to replace the o-rings on the floating piston and the bottom end cap.

L.7.14   Remove the bottom end cap using the procedure in L.7.11 and L.7.12, except that the cell position is reversed, and the two-pin spanner wrench is used.

L.7.15   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 o-rings on the piston and cap.

L.8 Calculation — Data reporting

L.8.1   Filtrate reporting

Report the actual cumulative filtrate volume, in cm^3, collected through each selected time period.

L.8.2   Spurt loss reporting

The spurt loss is the volume of fluid that passes through the filtration medium before a filter cake is formed. It can be depicted by the intercept, on the y-axis, of the straight line representing the static filtrate rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume. The filtrate volume should be corrected to a filter area of 4 580 mm^2 (7,1 in^2). PPA filter cells usually have half the standard filter area or 2 258 mm^2 (3,5 in^2), thus double the observed volume before reporting. Alternatively, an approximate value can be calculated using the equation in L.9.

NOTE   To more accurately define the spurt loss, collect and record the filtrate more frequently, and plot the data as described in the NOTE following L.6.1.

L.8.3   Filter cake reporting

Measure and record the filter cake thickness to the nearest 0,8 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.

L.9 Calculation — Filtrate volume and filtration rate

$$V_t = 2\ V \tag{K.1}$$

$$V_i\ =\ 2\ [V_1 - (V_2 - V_1)] \tag{K.2}$$

$$R\ =\ 2\ [(V_2 - V_1)] / 2{,}739 \tag{K.3}$$

where:

V volume of filtrate collected in receiver, cm^3

V_1 volume of filtrate collected after 7,5 min, cm^3

V_2 volume of filtrate collected after 30 min, cm^3

V_t PPT value, cm^3

V_i spurt loss, cm^3

R static filtration rate

NOTE   Observe that all three parameters are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard ISO/API low pressure filtration test. Doubling the filtrate volume compensates for this area difference. The constant, 2, can be modified as necessary to accommodate tests made utilizing other filtration area filter media.

Annex M
(informative)

Oil-based drilling fluid report form

DRILLING MUD REPORT

OIL MUD REPORT NO.

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:

	☐F.L. Pit☐	☐F.L. Pit☐	☐F.L. Pit☐	☐F.L. Pit☐
Sample from				
Time Sample Taken				
Flowline Temperature (°C or °F)				
Density ☐(ppg) ☐(lb/cu ft) ☐(sp gr) ____°C or °F				
Funnel Viscosity (sec/qt) API @ ____°C or °F				
Plastic Viscosity cp@ ____°C or °F				
Yield Point (lb/100 ft^2)				
Gel Strength (lb/100 ft^2) 10 sec/10 min	/	/	/	/
Filtrate API (m/30 min)				
HTHP Filtrate (cm^3/30 min) @ ____°C or °F				
Cake Thickness 32nd in. or mm	/	/	/	/
Electrical Stability (v), Meter Type ____				
Retort Solids, % volume				/
Retort Liquid, % volume Oil/Water	/	/	/	
Oil/Water Ratio	/	/	/	
Alkalinity (V_{SA}), (cm^3 H_2SO_4)				
Filtered yes☐ no☐				
Chloride Whole Mud Cl_{OM} (mg/L)				
Calcium Whole Mud Ca_{OM} (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 _____

Annex N
(informative)

Determining compatibility of elastomeric materials with non-aqueous-based drilling fluids

N.1 Principle

N.1.1   The effects of drilling fluids, particularly non-aqueous-based fluids on elastomeric materials used in a drilling operation can have significant impact on the safety, the success and the cost of the operation. Articles prepared with elastomeric materials which may be exposed to such fluids during drilling operations include seals, gaskets, hoses, diaphragms, sleeves, downhole motor stators, etc. Various properties of such articles deteriorate during exposure to the non-aqueous fluids, affecting the performance of these articles. Measurement/predictability of these effects is fundamental to the selection of appropriate elastomer/drilling fluid combinations for the drilling operation and to the establishment of operational guidelines.

N.1.2   Many test methods exist which evaluate these various effects, and the methods employed vary throughout the drilling industry.

N.1.3   It is the intent of this standard to provide standard methods for testing the effects of non-aqueous-based drilling fluids on elastomers intended for contact with the fluids during drilling operations. Since the relationship between elastomer/fluid screening test conditions and actual service conditions is only approximate, and since test result acceptance criteria vary with the user, no guidelines for test result interpretation are provided.

N.1.4   Non-aqueous-based drilling fluids, as defined in this annex, are fluids prepared with either naturally occurring or refined hydrocarbon oil, synthetic hydrocarbons or other synthetic liquids. These fluids may have aqueous internal phases, however the continuous phase is comprised of the non-aqueous liquid.

N.1.5   All test methods described or referenced here involve measurements of initial sample properties of the elastomer, immersion of the elastomer in a fluid at prescribed temperatures for prescribed time periods, and subsequent measurements of the same properties. The methods described here yield comparative data on which to base judgment as to expected service quality.

N.1.6   This annex references ASTM test methods and standard reference liquids which API Subcommittee 13 on Drilling Fluid Materials has reviewed and found appropriate for testing the effects of drilling fluids on elastomeric materials used in drilling operations. This annex also references ISO standard materials appropriate for reference testing.

N.1.7   The full ASTM procedures are not reproduced , but can be obtained from ASTM. Neither is the standardization information for ISO selection of standard reference materials reproduced, but can be obtained from ISO.

N.2 Apparatus

N.2.1   The apparatus required for each test is described in the appropriate ASTM procedure and is only described in general terms. The specific test equipment is normative in order to perform the standard tests correctly and safely and to achieve results that meet requirements.

N.2.2   Standard reference elastomers

N.2.2.1 Elastomer compositions to be used as reference standards are described in ISO 13 226. Test specimens prepared with these compositions can be supplied by a variet of rubber compounding companies. Users may use their discretion in sourcing such samples.

N.2.2.2 From Annex BB, subclause BB2 composition, Table BB1, SRE NBR 34S is to be used. This elsastomer formulation is for an acrylonitrile-butadiene rubber with 34 % (w/w) acrylonitrile. It is considered representative of a rountinely used, non-extreme service material.

N.2.2.3 From Annex Ga, subclause GA2 composition, Table GA1, SRE HNBR/1 is to be used. This elastomer formulation is for a hydrogenated acrylonitrile-butadiene rubber with 19 % (w/w) nitrile groups and less than 0,1% residual double bonds. It is considered representative of a material required for sue in a harsh chemical environment and/or at a high temperature.

N.2.3 Standard reference test liquids

N.2.3.1 Two standard test liquids to be used as test medium are described in ASTM D 471.

N.2.3.2 From Section 6, IRM 903 is an industry reference hydrocarbon oil with a relatively high aniline point, which produces relative mild effects on elastomers.

N.2.3.3 From Section 6, Service Liquid 101 is an industry reference ester oil, di-2-ethyl hexyl sebacate, which is quite aggressive toward elastomers.

N.2.3.4 Suggested suppliers[6] are:

a)   IRM 903 from Penreco, 4426 East Washington Blvd., Los Angeles, CA 90023-4476 USA. Telephone: +1-888-227-5448. Internet: www.penreco.com

b)   Service Liquid 101 sold as Anderol 774 from Robeco International Limited, Rowley House, School Close, Chandlers Ford, Eastleigh, Hampshire SO53 4BY, UK. Telelphone +44 (0)23 8-24 6999. Internet: www.robeco.co.uk/location.htmA774

c)   Service Liquid 101 sold as Synthetic Oil A774 from NYE Lubricants, Inc., 10534 Success Lane, Centerville, OH 45458 USA. Telephone: +1-508-996-6721. Internet: www.nyelubricants.com.

N.3   Procedure — preparation of test specimens

N.3.1   Test specimen preparation for all testing is covered in ASTM D3182-89 and ASTM D3183-84.

N.3.2   Standard test specimen dimensions are specified in ASTM D471-96, Section 9.1.

N.4   Procedure — volume, tensile strength, elongation and hardness of elastomers after immersion

N.4.1   The test methods are described fully in ASTM D471-96, Sections 8, 9, 11 and 15.

N.4.2   These measurements are believed to be sufficient for defining the effects of a given fluid on a given elastomer. The immersion temperatures chosen are those rountinely used by elastomer manufacturers. Other temperatures may be used as required.

N.4.3   Changes in volume, tensile strength, elongation, and hardness of elastomeric materials after contact with non-aqueous fluids are common and degrade the performance of elastomeric articles. The chanes can be either positive or negative. These methods cover the laboratory determination of such changes in unconfined elastomer specimens after static immersion in a test fluid at specific temperatures, for a specific time periods, at ambient pressure. Two immersion temperatures are specified – 70 °C (158 °F) and 100 °C (212 °F). Temperatures are maintained by convenient/appropriate means:

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6)   The suppliers listed in this Annex are only suggested suppliers, and do not constitute endorsement of the supplier or its goods by ISO.

a) water baths;

b) oil baths;

c) hot air ovens or;

d) ageing blocks.

An immersion time period of 168 h (7 d) is routinely used by elastomer manufacturers and is specified here to reasonably assure equilibrium.

N.4.4 Summary of immersion procedure (ASTM D471-96, sections 8 and 9)

Pre-immersion measurements of the properties are made on the specimens by the referenced ASTM procedures, and recorded. The specimens are placed in a test tube, or other glass vessel, fitted with a water-cooled reflux condenser. Test fluid is added to completely immerse the specimens. Flow of water through the reflux condenser is started and the temperature of the test fluid is increased to the required test value. The temperature is maintained for the required immersion time. The specimens are removed from the test fluid and excess fluid is removed from the specimens. Post-immersion measurements of the properties are made and recorded. If temperatures and/or immersion times other than those specified in this standard are used, they should be reported.

N.4.5 Summary of change in volume procedure (ASTM D471-96, section 11)

Three specimens, as identical as possible, are tested. The initial volume of each specimen is measured by water displacement. Water is removed from the specimens and the immersion procedure described in N.4.4 is conducted on each specimen. After immersion, the volume of each specimen is again measured by water displacement. The volume change, expressed as a percentage of the original volume, for each is calculated and an average change is determined and recorded. This procedure should only be used for testing water insoluble fluids.

N.4.6 Summary of change in tensile strength, elongation and hardness procedures (ASTM D471-96, section 15)

N.4.6.1 Change in tensile strength and elongation

The original tensile strength and ultimate elongation are determined by ASTM D412-98a, using three specimens cut from a sheet or article adjacent to those that are to be immersed in the fluid. Since ultimate elongation measurement is destructive, separate specimens shall be used for the immersion procedure and post-immersion measurements. Tensile strength and ultimate elongation measurements are made on the same specimen. The immersion procedure described in N.4.4 is conducted on each specimen and measurements of tensile strength and ultimate elongation are again made on each specimen. The change, expressed as a percentage of the original value, is calculated for each specimen. An average is determined and recorded.

N.4.6.2 Change in hardness

The original hardness is measured by ASTM D1415-88 or ASTM D2240-00, using three specimens cut from the sheet or article adjacent to those that are to be immersed in the fluid. Since the hardness measurement alters the properties of the elastomer, separate specimens shall be used for the immersion procedure and post-immersion measurements. The immersion procedure described in N.4.4 is conducted on each specimen and measurement of hardness again performed on each specimen. The change, expressed as a percentage of the original value is calculated for each specimen. An average change is determined and recorded.

N.4.6.3 Effects of immersion on a stressed elastomer specimen

N.4.6.3.1   In addition to the measurements of changes in volume, tensile strength, elongation and hardness after immersion in a test fluid, a method is specified in which the test specimens are stressed (bent) and subjected

to the immersion procedures as described in N.4.4. Visual observation of the condition of the specimens is made after immersion and recorded.

N.4.6.3.2 Test specimens are bent to 180° and secured in that position with a suitable retainer which is inert to the test fluid. The specimens are then subjected to the immersion test procedure in N.4.4.. After immersion, the specimens are removed from the test vessel, excess fluid is removed and visual observation of the specimen condition is made. Discoloration, excessive swelling, cracking of the stressed area, and extens of any cracking are noted. The observations are recorded.

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[3]   ISO 10416/API RP 13I, *Standard procedure for laboratory testing drilling fluids*

[4]   ISO 13226, *Rubber – Standard reference elastomers (SRE's) for characterizing the effect of liquids on vulcanized rubber*

[5]   ISO 13500/API Spec 13A, *Petroleum and natural gas industries — Drilling fluid materials — Specifications and test*

[6]   API RP 13B-2, February 1998, *Standard procedure for field testing oil-based drilling fluids (third edition)*

[7]   API RP 13D, *Rheology and hydraulics of oil-well drilling fluids*

[8]   ASTM C25, *Standard test methods for chemical analysis of limestone, quicklime, and hydrated Lime*

[9]   ASTM D412, *Standard test methods for vulcanized rubber and thermoplastic elastomers tension*

[10]   ASTM D471, *Standard test method for rubber property – effect of liquids*

[11]   ASTM D1415, *Standard test method for rubber property – international hardness*

[12]   ASTM D2240, *Standard test method for rubber property – durometer hardness*

[13]   ASTM D3182, *Standard practice for rubber – materials, equipment and procedures for mixing standard compounds and preparing standard vulcanized sheets*

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