Testing Water-based Drilling Fluids

API RECOMMENDED PRACTICE 13B-1
SIXTH EDITION, Ballot Draft

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AMERICAN PETROLEUM INSTITUTE

Special Notes , Forword and contents to be inserted by API

Introduction

This standard is based on API Recommended Practice 13B-1, Field Testing Water-based Drilling Fluids, Fifth Edition, May 2019.

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

In this standard, quantities expressed in the international System of Units (SI) are also, where practical, expressed in U.S. customary units (USC) in parentheses for information. The units do not necessarily represent a direct conversion of SI units to USC units, or USC units to SI units. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one-degree increments, thus temperature values have been rounded to the nearest degree.

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

This document uses a format for numbers which follows the examples given in API Document Format and Style Manual. In this document, the decimal mark is a period and separates the whole part from the fractional part of a number. No spaces are used in the numbering format. The thousands separator is a comma and is only used for numbers greater than 10,000 (i.e. 5000 items, 12,500 bags).

Testing Water-based Drilling Fluids

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

1   Scope

This recommended practice provides standard procedures for determining the following characteristics of water-based drilling fluids:

a)   drilling fluid density (mud weight);

b)   viscosity and gel strength;

c)   static filtration;

d)   water, oil, and solids contents;

e)   sand content;

f)   methylene blue capacity;

g)   pH;

h)   alkalinity and lime content;

i)   chloride content;

j)   total hardness as calcium;

k)   low-gravity solids and weighting material concentrations.

l)   static filtration at high-temperature and high-pressure through permeability plugging testing.

Annexes provide additional test methods that may be used for:

—   chemical analysis for calcium, magnesium, calcium sulfate, sulfide, and carbonate, (Annex A)

—   chemical analysis of potassium (Annex B);

—   determination of shear strength (Annex C);

—   determination of resistivity (Annex D);

—   removal of air or gas before testing (Annex E);

—   drill-pipe corrosion monitoring (Annex F);

— sampling, inspection, and rejection (Annex G);

— rig-site sampling (Annex H);

— calibration and verification of glassware, thermometers, timers, viscometers, retort cup, and drilling fluid balances (Annex I);

— weight-material dynamic sag testing (Annex J);

— examples of solids calculations (Annex K).

Annex L is an abbreviated analytical analysis for water-based drilling fluids which are recommended, Section 13 (alkalinity), Section 14 (chloride content) , Section 15 (total hardness), Section 11 (methylene blue capacity)   and Annex A for calcium, magnesium and calcium sulfate.

Annex M provides an example for a water-based drilling fluid daily report.

2   Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

API Recommended Practice 13C, *Drilling Fluid Processing Systems Evaluation*

API Recommended Practice 13D, *Rheology and Hydraulics of Drilling Fluids*

ISO 3696 [1], *Water for analytical laboratory use — Specification and test methods*

3   Terms and Definitions

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

3.1
ACS reagent grade
Chemical meeting the purity standards specified by the American Chemical Society (ACS).

3.2
bentonite equivalent value
A measure of the cation-exchange capacity of a drilling fluid sample volume, reported as a bentonite equivalent expressed in kilograms per cubic meter (kg/m3) [pounds per barrel (lb/bbl)] of drilling fluid.

3.3
deionized water
distilled water
Water for laboratory use of analysis of barite requiring high standard water that has met or exceeded the purity of standards specified by ISO 3696 for water Grade III.

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[1]   International Organization for Standardization, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, www.iso.org.

3.4
density of water
The volume of 1 kg of water is 1 L for the purposes of this standard, and the density of water in grams is numerically equivalent to the volume of the water measured in cubic centimeters or milliliters, i.e. 1 g ≡ 1 mL. See Table I.1 for further details.

NOTE   Deionized or distilled water is used for equipment calibration.

3.5
drill solids
Solid substrate materials incorporated into the drilling fluid as a result of drilling formations.

3.6
low-gravity solids
Any solid substrate material in the drilling fluid having a density less than or equivalent to 2.6 g/cm3 (21.7 lb/gal).

3.7
quarter (quartered)
A verb: mix and divide a sample into four specimens to ensure homogeneity of specimens.

NOTE   API 19C [5] provides apparatus and procedure to quarter a sample.

3.8
spurt loss
The volume of fluid that passes through the filtration medium before a filter cake is formed.

3.9
tube sampling
A sampling method consists of the withdrawal of powdered sample from bag or bulk via a cylindrical device pushed into the sample, locked shut and withdrawn.

4   Symbols and Abbreviations

4.1   Symbols

NOTE 1 Subscript "SI" to symbol denotes metric units. Subscript "USC" to symbol denotes United States customary units.

NOTE 2 Pounds per gallon is the common notation for pounds-mass per gallon and is used throughout this document.

A_{SI} area, expressed in square centimeters

A_{USC} area, expressed in square inches

B_{VSST} weight-material sag, expressed in pounds-mass per gallon

a_{H^+} hydrogen ion activity, expressed in mole per liter

a_{OH^-} hydroxyl ion activity, expressed in mole per liter

C_{MBT}	methylene blue capacity of drilling fluid, expressed in milliequivalents per milliliters of drilling fluid
C_{th}	thermometer correction factor to be added to the working thermometer reading, expressed in degrees Celsius (degrees Fahrenheit)
c_{Ca}	concentration of calcium ion, expressed in milligrams per liter
$c_{CaSO4,SI}$	concentration of soluble calcium sulfate, expressed in kilograms per cubic meter of drilling fluid
$c_{CaSO4,USC}$	concentration of soluble calcium sulfate, expressed in pounds per barrel of drilling fluid
c_{Ca+Mg}	total hardness concentration (concentration of calcium and magnesium ions), expressed as calcium ion concentration in milligrams per liter
c_{Cl}	concentration of chloride ion, expressed in milligrams per liter
$c_{CO_2+CO_3+HCO_3}$	concentration of total soluble carbonates, expressed in milligrams per liter
$c_{K-f,QAS}$	concentration of potassium ion in filtrate (QAS titration), expressed in milligrams per liter
$c_{K,f}$	concentration of potassium ion in filtrate, expressed in milligrams per liter
$c_{KCl,SI}$	concentration of potassium chloride in tested sample of filtrate, expressed in kilograms per cubic meter
$c_{KCl,USC}$	concentration of potassium chloride in tested sample of filtrate, expressed in pounds per barrel
$c_{KCl-f,SI}$	concentration of potassium chloride in filtrate, expressed in kilograms per cubic meter
$c_{KCl-f,USC}$	concentration of potassium chloride in filtrate, expressed in pounds per barrel
$c_{LG,SI}$	concentration of low-gravity solids, expressed in kilograms per cubic meter
$c_{LG,USC}$	concentration of low-gravity solids, expressed in pounds per barrel
c_{Mg}	concentration of magnesium ion, expressed in milligrams per liter
$c_{NaCl,SI}$	concentration of sodium chloride, expressed in milligrams per liter
$c_{NaCl,ppm}$	concentration of sodium chloride, expressed in parts per million by mass
c_S	concentration of sulfide ion, expressed in milligrams per liter
$c_{SS,SI}$	concentration of suspended solids, expressed in kilograms per cubic meter

$c_{SS,USC}$	concentration of suspended solids, expressed in pounds per barrel
$c_{WM,SI}$	concentration of weighting material, expressed in kilograms per cubic meter
$c_{WM,USC}$	concentration of weighting material, expressed in pounds per barrel
$c_{ex-CaSO_4,SI}$	concentration of excess, undissolved calcium sulfate, expressed in kilograms per cubic meter of drilling fluid
$c_{ex-CaSO_4,USC}$	concentration of excess, undissolved calcium sulfate, expressed in pounds per barrel of drilling fluid
$c_{lime,SI}$	lime content of the drilling fluid, expressed in kilograms per cubic meter
$c_{lime,USC}$	lime content of the drilling fluid, expressed in pounds per barrel
$E_{BE,SI}$	bentonite equivalent, expressed in kilograms per cubic meter
$E_{BE,USC}$	bentonite equivalent, expressed in pounds per barrel
F_W	volume fraction of water in the drilling fluid, expressed as a decimal
f_{CO_2}	tube factor for CO_2 gas detection tube, from Table A.2
f_S	tube factor for H_2S gas detection tube, from Table A.1
K	resistivity-cell constant, expressed in square meters per meter
k_C	consistency factor, expressed in pounds-force·second per one hundred square feet
k_{cor}	correction factor for $R_{QAS/STPB}$
l_{SI}	submerged length of shear tube, expressed in centimeters
l_{USC}	submerged length of shear tube, expressed in inches
l_{st}	gas detection Dräger (or equivalent) tube darkened or stain length, expressed in units marked on the tube
M_f	methyl orange alkalinity of the filtrate, expressed in millimeters of 0.01 mol/L sulfuric acid solution per milliliter of filtrate (to reach the methyl orange endpoint)
m_{F1}	initial mass of the 10 mL sample of drilling fluid (plus syringe), expressed in grams
m_{F2}	mass of 10 mL sample of drilling fluid (plus syringe) taken at the sag shoe following 30 min shear at 100 r/min, expressed in grams

m_{F3}	mass of 10 mL sample of drilling fluid (plus syringe) taken at the sag shoe following 20 min shear at 600 r/min, expressed in grams
m_L	mass of the condensed liquids (water and oil), expressed in grams
m_W	mass of water, filling receiver at a specified mark or cup, expressed in grams
m_{df}	mass of the drilling fluid sample, expressed in grams
m_{dmb}	mass of the methylene blue dried sample, expressed in grams
m_{mb}	corrected mass of methylene blue powder to be used per liter water, expressed in grams
m_{ds}	mass of the dried retort solids, expressed in grams
m_{st}	mass of the shear tube, expressed in grams
m_{tot}	total shear mass (sum of platform and weights), expressed in grams
m_1	mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams
m_2	mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams
m_3	mass of the empty, dry liquid receiver, expressed in grams
m_4	mass of the cooled liquid receiver with condensed liquids, expressed in grams;
m_5	mass of the cooled retort assembly (cup, lid and body packed with steel wool), expressed in grams
P_{df}	phenolphthalein alkalinity of the drilling fluid, expressed in millimeters of 0.01 mol/L sulfuric acid solution per milliliter of drilling fluid (to reach the phenolphthalein endpoint)
P_f	phenolphthalein alkalinity of the filtrate, expressed in millimeters of 0.01 mol/L sulfuric acid solution per milliliter of filtrate (to reach the phenolphthalein endpoint)
q_{SI}	corrosion rate, expressed in kilograms per square meter-year
q_{USC}	corrosion rate, expressed in pounds per square foot-year
R	temperature reading for the working thermometer, expressed in degrees Celsius (degrees Fahrenheit)
R_{BPU}	calculated bed pickup measurement ratio, expressed as a percentage

$R_{\text{QAS/STPB}}$ — ratio of QAS to STPB (4.0 ±0.5)

R_{cor} — corrected temperature reading for the working thermometer, expressed in degrees Celsius (degrees Fahrenheit)

$R_{\text{r-df}}$ — resistivity-meter reading with drilling fluid expressed in ohms;

$R_{\text{r-f}}$ — resistivity-meter reading with drilling fluid filtrate, expressed in ohms.

R_1 — average temperature reading for the standard thermometer, expressed in degrees Celsius (degrees Fahrenheit)

R_2 — average temperature reading for the working thermometer, expressed in degrees Celsius (degrees Fahrenheit)

R_3 — viscometer dial reading at 3 r/min, expressed in degrees of deflection

R_6 — viscometer dial reading at 6 r/min, expressed in degrees of deflection

R_{30} — viscometer dial reading at 30 r/min, expressed in degrees of deflection

R_{60} — viscometer dial reading at 60 r/min, expressed in degrees of deflection

R_{100} — viscometer dial reading at 100 r/min, expressed in degrees of deflection

R_{200} — viscometer dial reading at 200 r/min, expressed in degrees of deflection

R_{300} — viscometer dial reading at 300 r/min, expressed in degrees of deflection

R_{600} — viscometer dial reading at 600 r/min, expressed in degrees of deflection

r_{df} — drilling fluid resistivity, expressed in ohm·meters

r_{f} — filtrate resistivity, expressed in ohm·meters

t — exposure time, expressed in hours, or lagged time, expressed in minutes

t_1 — initial reading time (taken at 7.5 min), expressed in minutes

t_2 — final reading time (taken at 30 min), expressed in minutes

V_{EDTA} — total hardness titration volume of EDTA solution, expressed in milliliters

$V_{\text{EDTA-df}}$ — calcium titration volume of EDTA solution of whole drilling fluid, expressed in milliliters

$V_{\text{EDTA-f}}$ — calcium titration volume of EDTA solution of the drilling fluid filtrate, expressed in milliliters

V_L	volume of condensed liquids (oil and water) after retorting, expressed in milliliters
V_M	receiver volume at a specified mark, expressed in milliliters
V_O	volume of oil, expressed in milliliters
V_{PPT}	PPT test volume, expressed in milliliters
V_{QAS}	volume of QAS, expressed in milliliters
V_{RC}	retort cup volume, expressed in milliliters
V_{SN1X}	volume of silver nitrate 0.0282 N solution, expressed in milliliters
V_{SN10X}	volume of silver nitrate 0.282 N solution, expressed in milliliters
V_W	volume of condensed water, expressed in milliliters, or mass of condensed water, expressed in grams (1 mL $\equiv$ 1 g)
V_{df}	volume of whole drilling fluid sample, expressed in milliliters
V_f	volume of the filtrate (or reported HTHP filtrate volume), expressed in milliliters
V_{mb}	volume of methylene blue solution, expressed in milliliters
V_p	volume of precipitate, expressed in milliliters.
V_s	volume of sample (drilling fluid or filtrate), expressed in milliliters
V_{sl}	spurt loss, expressed in milliliters
V_1	filtrate volume after 1 min, expressed in milliliters
$V_{7.5}$	filtrate volume after 7.5 min, expressed in milliliters
V_{30}	filtrate volume after 30 min, expressed in milliliters
$Y_{P,SI}$	yield point, expressed in pascals
$Y_{P,USC}$	yield point, expressed in pounds-force per one hundred square feet
β_{10m}	10 min gel strength, expressed in pounds-force per one hundred square feet
β_{10s}	10 s initial gel strength, expressed in pounds-force per one hundred square feet
$\Gamma_{DFG,SI}$	drilling fluid depth gradient, expressed in kilopascals per meter

$\Gamma_{DFG,USC}$	drilling fluid depth gradient, in pounds per square inch, per foot
γ_{SI}	shear strength of the drilling fluid, expressed in pascals
γ_{USC}	shear strength of the drilling fluid, expressed in pounds-force per hundred square feet
Δm	mass loss, expressed in milligrams
η_{AV}	apparent viscosity, expressed in millipascal-seconds (centipoises)
η_{PV}	plastic viscosity, expressed in millipascal-seconds (centipoises)
ρ	drilling fluid relative density (specific gravity), dimensionless
ρ_{LG}	density of low-gravity solids, expressed in grams per milliliter (use 2.6 g/mL if unknown)
ρ_{O}	density of oil, expressed in grams per milliliter (use 0.8 g/mL if unknown)
ρ_{W}	water density at test temperature, expressed in grams per milliliter
ρ_{WM}	density of weighting material, expressed in grams per milliliter
ρ_{df}	drilling fluid density, expressed grams per milliliter
$\rho_{df,SI}$	drilling fluid density, expressed in kilograms per cubic meter
$\rho_{df,USC}$	drilling fluid density, expressed in pounds per gallon
$\rho_{df,USC2}$	drilling fluid density, expressed in pounds per cubic foot
ρ_{f}	density of filtrate, expressed in grams per milliliter
v_{sf}	static filtration rate, expressed in milliliters per square root of minute
φ_{LG}	volume fraction of low-gravity solids, expressed as a percentage of the drilling fluid volume
φ_{B}	volume fraction of brine, expressed as a percentage of the drilling fluid volume
φ_{O}	volume fraction of oil, expressed as a percentage of the drilling fluid volume
φ_{SS}	volume fraction of suspended solids, expressed as a percentage of the drilling fluid volume
φ_{W}	volume fraction of water, expressed as a percentage of the drilling fluid volume

| φ_{WM} | volume fraction of weighting material, expressed as a percentage of the drilling fluid volume |
| φ_{ds} | volume fraction of dried solids from retort, expressed as a percentage of the drilling fluid volume |

4.2   4.2 Abbreviations

ACS	American Chemical Society
BE	bentonite equivalent
CAS	Chemical Abstracts Service
DFG	drilling fluid gradient
DN	nominal diameter
ECD	equivalent circulating density
EDTA	ethylenediaminetetraacetic acid
ERD	extended-reach drilling
ESD	equivalent static density
HTHP	high-temperature high-pressure
LSYP	low-shear yield point
MBT	methylene blue test (often also used to mean the methylene blue capacity)
meq	milliequivalents
NAF	nonaqueous fluid
NIST	National Institute of Standards and Technology
PPA	permeability plugging apparatus
PPE	personal protective equipment
ppm	part per million
PPT	permeability plugging test
psi	pound-force per square inch
QAS	quaternary ammonium salt
QC	quality control

RMS	root mean square
STPB	sodium tetraphenylborate
TC	to contain
TD	to deliver
TVD	total vertical depth
VSST	viscometer sag shoe test

5   Determination of Drilling Fluid Density (Mud Weight)

5.1   Principle

A field procedure is provided for determining the mass of a given volume of liquid (i.e. density) under ambient pressure conditions. The density of drilling fluid is expressed as grams per milliliter or kilograms per cubic meter (pounds per gallon or pounds per cubic foot).

5.2   Apparatus

5.2.1   Ambient-pressure Mud Balance, any ambient-pressure density-measuring instrument having an accuracy of ±0.01 g/mL or ±10 kg/m^3 (±0.1 lb/gal or ±0.5 lb/f^{t3}).

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

The mud balance shall be calibrated frequently with fresh water. The recommended frequency is prior to each set of measurements (e.g. daily or by each work shift), or as specified by the operator, prevailing regulation, drilling contractor, drilling fluid company, or other controlling party. Fresh water shall give a reading of 1.00 g/mL ±0.005 g/mL or 1000 Kg/m^3 ±5 kg/m^3 (8.345 lb/gal ±0.05 lb/gal or 62.4 lb/ft^3 ±0.25 lb/ft^3) at 21 °C (70 °F) when the bubble level is centered. If it does not, the instrument shall be calibrated by adjusting the balancing screw or the amount of shot (metal beads) in the well at the end of the graduated arm until it meets the accuracy requirement, or the instrument shall be removed from service. The instrument shall be calibrated on a less frequent basis (e.g. annually) according to the procedure as specified by the manufacturer or using a two-point calibration. Calibration according to the procedure as specified by the manufacturer or using a two-point calibration shall always be performed after damage, repair, or replacement of items such as the sample cup lid or the shot well screw.

NOTE: For laboratory applications, gravimetric methods such as volumetric flask, density cup, or water pycnometer methods are acceptable for measuring density.

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

5.3   Ambient-pressure Mud Balance Procedure

In order to determine drilling fluid density, the following procedure shall be used.

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

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

c) Start with a clean and dry mud balance. Fill the balance cup to the lip with the drilling fluid to be tested. Put the lid on the filled sample cup and rotate the lid, expelling any trapped air and excess drilling fluid. Ensure that some of the drilling fluid is expelled through the hole in the lid and that the lid is firmly seated.

d) Holding the lid firmly on the filled sample cup (with the hole in the lid covered by a finger), wash and/or wipe the outside of the cup to be clean and dry.

e) Place the beam on the base support knife edge and level by moving the sliding weight along the graduated scale. The beam is level when the bubble in the liquid-filled vial is centered between the two black lines or under a single centerline.

f) The drilling fluid density is read from one of the four calibrated scales on the balance arm, at the arrow side of the sliding counterweight. The density can be read directly in units of grams per milliliter using relative density (specific gravity) scale, pounds per gallon, pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1000 ft of vertical depth.

5.4 Calculation

5.4.1 Drilling fluid density

The drilling fluid density shall be reported to the nearest 0.01 g/mL or 10 kg/m^3 (0.1 lb/gal or 0.5 lb/ft^3).

To convert the drilling fluid relative density reading, ρ (dimensionless), which is numerically equivalent to the density, ρ_{df}, expressed in grams per milliliter to other units, Equations (1) to (7) and Tables 1 and 2 can be used..

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

Table 1—Conversion of Density Units

Measured In	Multiply to Convert To			
	g/mL	kg/m^3	lb/gal	lb/ft^3
g/mL	1	1000	8.345	62.43
kg/m^3	0.001	1	0.008345	0.06243
lb/gal	0.120	120	1	7.4805
lb/ft^3	0.0160	16.02	0.1337	1

Equations (1) to (3) shall be used to convert the density, ρ_{df}, expressed in grams per milliliter, to other units.

a) to kilograms per cubic meter:

$$\rho_{df,SI} = 1000 \times \rho_{df} \tag{1}$$

where

$\rho_{df,SI}$ is the drilling fluid density, expressed in kilograms per cubic meter;

b)   to pound per gallon:

$$\rho_{df,USC} = 8.345 \times \rho_{df} \tag{2}$$

where

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

c)   to pounds per cubic feet:

$$\rho_{df,USC2} = 62.43 \times \rho_{df} \tag{3}$$

where

$\rho_{df,USC2}$ is the drilling fluid density, expressed in pounds per cubic foot.

A table of density equivalents for commonly used units is shown in Table 2.

5.4.2 Drilling fluid depth gradient

Equations (4) to (7) shall be used to convert density to the drilling fluid depth gradient, $\Gamma_{DFG,SI}$ expressed in kilopascals per meter or, $\Gamma_{DFG,USC}$ expressed in pounds-force per square inch, per foot.

$$\Gamma_{DFG,SI} = 9.81 \times \rho_{df} \tag{4}$$

$$\Gamma_{DFG,SI} = 22.6 \times \Gamma_{DFG,USC} \tag{5}$$

$$\Gamma_{DFG,USC} = 0.0520 \times \rho_{df,USC} \tag{6}$$

$$\Gamma_{DFG,USC} = 0.00694 \times \rho_{df,USC2} \tag{7}$$

where

ρ_{df} is the drilling fluid density, expressed in grams per milliliter;

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

$\rho_{df,USC2}$ is the drilling fluid density, expressed in pounds per cubic foot.

Table 2—Density Equivalents

Grams per milliliter [a]	Kilograms per Cubic Meter	Pounds per Gallon	Pounds per Cubic Foot
g/mL	kg/m^3	lb/gal	lb/ft^3
0.70	700	5.8	43.7
0.80	800	6.7	49.9
0.90	900	7.5	56.1
1.00	1000	8.345 [b]	62.4
1.10	1100	9.2	68.7
1.20	1200	10.0	74.9
1.30	1300	10.9	81.1
1.40	1400	11.7	87.4
1.50	1500	12.5	93.6
1.60	1600	13.4	99.9
1.70	1700	14.2	106.1
1.80	1800	15.0	112.4
1.90	1900	15.9	118.6
2.00	2000	16.7	124.8
2.10	2100	17.5	131.1
2.20	2200	18.4	137.3
2.30	2300	19.2	143.6
2.40	2400	20.0	149.8
2.50	2500	20.9	156.1
2.60	2600	21.7	162.3
2.70	2700	22.5	168.5
2.80	2800	23.4	174.8
2.90	2900	24.2	181.0

[a] Same value as relative density in grams per milliliter or kilogram per liter.
[b] Accurate conversion factor.

6   Determination of Drilling Fluid Density ─ Pressurized Balance Method

6.1   Principle

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

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

6.2   Apparatus

6.2.1 Pressurized Mud Balance, any pressurized density-measuring instrument **,** any instrument having an accuracy of ±0.01 g/mL or 1±0 kg/m^3 (±0.1 lb/gal or ±0.5 lb/ft^3) designed such that the pressurizing mechanism exerts 1723 kilopascals (250 lbf/ in.^{2}) of pressure to fluid sample for a plunger force of 225 N (50 lbf).

NOTE   1723 kilopascals (250 lbf/in.^{2}) of pressure applied to a fluid sample will result in a density measurement within 2 % of the uncut liquid density for a sample with up 20 % volume entrained air or gas.

The pressurized mud balance is designed such that the assembled pressurized sample cup, at one end of the beam, is balanced by a sliding-counterweight that moves along a graduated scale. A bubble-level is mounted on the beam to allow for accurate leveling. Attachments for extending the range of the balance may be used when necessary.

The mud balance shall be calibrated frequently with fresh water. The recommended frequency is prior to each set of measurements (e.g. daily or by each work shift), or as specified by the operator, prevailing regulation, drilling contractor, drilling fluid company, or other controlling party. Fresh water shall give a reading of 1.00 g/mL ±0.005 g/mL or 1000 kg/m^3 ±5 kg/m^3 (8.345 lb/gal ±0.05 lb/gal or 62.4 lb/ft^3 ±0.25 lb/ft^3) at 21 °C (70 °F) when the bubble level is centered. If it does not, the instrument shall be calibrated by adjusting the balancing screw or the amount of shot (metal beads) in the well at the end of the graduated arm until it meets the accuracy requirement, or the instrument shall be removed from service. The instrument shall be calibrated on a less frequent basis (e.g. annually) according to the procedure as specified by the manufacturer or using a two-point calibration. Calibration according to the procedure as specified by the manufacturer or using a two-point calibration shall always be performed after damage, repair, or replacement of items such as the sample cup lid, threaded lid retainer or the shot well screw.

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

6.3   Pressurized Mud Balance Procedure

In order to determine the drilling fluid density under pressure, the following procedure shall be used.

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

a)   Measure and record the temperature of the drilling fluid.

b)   Start with a clean and dry pressurized mud balance. Inspect the check valve for free smooth movement and check the O-rings on the lid body and check valve nipple, replace check valve or O-rings if damaged. Fill the balance cup to a level approximately 6 mm (0.25 in.) below the upper edge of the cup.

    Caution—While assembling and pressurizing the pressurized mud balance, personal protective equipment (PPE) and care should be taken to avoid contact and exposure to fluid that may be expelled from the cup check valve or plunger. While assembling the cup, lid and threaded retainer, it is recommended to cover the check-valve opening with a rag.

c)   Place the lid on the cup with the check-valve in the down (open) position. Carefully 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. Excess drilling fluid shall be expelled through the open check-valve. With

the check valve opening firmly covered with a finger, wash or wipe fluid from the lid and threaded area of the cup. Then place the threaded lid retainer over the lid and screw into place. This secures the lid to the cup and expels excess fluid through the open check valve.

NOTE If excess fluid is not expelled from the open check valve during assembly, the sample cup was not filled sufficiently, and the fill procedure shall be repeated.

d) The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submerging the open end into the drilling fluid with the piston rod knob pushed all the way down, then pull the piston rod knob to the fully upward position to fill. This first fill volume shall be discarded into another container and the plunger shall be refilled with drilling fluid in case the first fill plunger volume was contaminated with a previous sample or a liquid from the last clean-up of the plunger assembly.

e) Place the pressurized mud balance on a stable surface, attach the filled plunger by pushing the nose of the plunger onto the mating O-ring surface of the lid check valve. Pressurize the sample cup by holding a downward force on the plunger housing with one hand in order to hold the check-valve down (open) and at the same time push down and maintain a force of approximately 225 N (50 lbf) or greater on the plunger piston rod knob.

f) The check-valve in the lid is pressure-actuated; when the cup is pressurized, the check valve is pushed upward into the closed position. To close the check valve, gradually decrease the downward force on the plunger housing while maintaining downward pressure on the plunger piston rod knob. After the downward pressure is reduced on the plunger, the check-valve moves up slightly and closes as pressure is released. Release pressure on the piston rod before disconnecting the plunger.

g) The pressurized drilling fluid sample is now ready for weighing. Rinse and/or wipe the outside of the cup to be clean and dry. Place the beam on the base support knife edge and level by moving the sliding weight along the graduated scale. The beam is level when the bubble in the liquid-filled vial is centered between the two black lines.

h) The drilling fluid density is read from one of the four calibrated scales on the balance arm, at the arrow side of the sliding counterweight. The density can be read directly in units of grams per milliliter using relative density scale (specific gravity), pounds per gallon, pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1000 ft of vertical depth.

i) To release the pressure inside the cup, reconnect the empty plunger assembly and push down on the plunger housing until the check valve opens and stays in the down position.

j) Empty and clean the cup, lid (including check-valve), threaded lid retaining ring, and plunger assembly.

6.4 Calculation

The drilling fluid density shall be reported to the nearest 0.01 g/mL or 10 kg/m3 (0.1 lb/gal or 0.5 lb/ft3).

For conversions, use the equations, conversion factors, or tables of values provided in 5.4.

7 Drilling Fluid Viscosity and Gel Strength

7.1 Principle

7.1.1 Viscosity and gel strength are measurements that relate to the flow or rheological properties of drilling fluids under dynamic conditions and thixotropic under static.

7.1.2 The following instruments are used to measure viscosity and gel strength of drilling fluids at temperatures from 4 °C to 65 °C (40 °F to 150 °F). Low-temperature rheology may be anticipated in the riser annulus of deepwater drilling operations at elevated pressures. The equipment is as follows:

a) Marsh funnel: a simple device for indicating viscosity on a routine basis.

b) Direct-indicating viscometer: a mechanical device for measuring viscosity at varying shear rates.

NOTE   Information on the rheology of drilling fluids can be found in API 13D [3].

7.2   Determination of Funnel Viscosity Using the Marsh Funnel

7.2.1 Apparatus

7.2.1.1   Marsh funnel, calibrated to deliver 946 mL (1 qt) of fresh water at a temperature of 21 °C ±3 °C (70 °F ±5 °F) in 26 s ±0.5 s, using a graduated cup as a receiver.

The Marsh funnel shall have the following characteristics:

a) funnel cone, length 305 mm (12.0 in.), diameter 152 mm (6.0 in.), and a capacity to bottom of screen of 1500 mL (1.6 qt);

b) orifice, length 50.8 mm (2.0 in.) and inside diameter 4.7 mm (0.185 in. = 3/16 in.);

c) screen, with 1.68 mm (0.066 in.)openings (US sieve No.12), fixed at 19.0 mm (0.75 in.) below top of funnel.

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

7.2.1.3   Timer or **stopwatch,** accuracy within ±5 seconds per hour.

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

7.2.2   Procedure

In order to determine funnel viscosity using a Marsh funnel, the following procedure shall be used.

a) 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.

b) Remove finger and start the stopwatch simultaneously. Measure the time for drilling fluid to fill to the 946 mL (1 qt) -mark of the cup.

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

Report the time (see item b) to the nearest second as the Marsh funnel viscosity. Report the temperature (see item c) of the fluid to the nearest degree Celsius (degree Fahrenheit).

7.3   Determination of Viscosity and Gel Strengths Using a Direct-indicating Viscometer

7.3.1 Apparatus

7.3.1.1 Direct-indicating viscometer [8], 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, the bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob. Instrument constants shall be adjusted so that the plastic viscosity and yield point are obtained by using shear stress readings from rotor sleeve speeds of 300 r/min and 600 r/min.

NOTE Some of the current generation of electronic digital drilling fluid viscometers use electronic speed control and optical encoders to measure deflection of the bob (or strain gauge measurements of the torque acting on the bob) for more precise measurements. Therefore, these units typically have digital outputs for the equivalent dial readings or degrees.

The components shall meet the following specifications:

a) **Rotor sleeve—R1:**

   inside diameter: 36.83 mm (1.450 in.);

   total length: 87.0 mm (3.425 in.);

   scribed line: 58.4 mm (2.30 in.) above the bottom of sleeve, with two rows of 3.18 mm (0.125 in.) holes, spaced 2.09 rad (120°) apart, around rotor sleeve just below scribed line;

   sleeve surface: surface finish (roughness) average 0.4 μm to 0.8 μm (16 μin. to 32 μin.) RMS cross-hatched honed.

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

   diameter: 34.49 mm (1.358 in.);

   cylinder length: 38.0 mm (1.496 in.);

   rotor surface: surface finish (roughness) average 0.4 μm to 0.8 μm (16 μin. to 32 μin.) RMS cross-hatched honed.

   NOTE   For testing at temperatures higher than 90 °C (190 °F) a solid bob shall be required.

c) **Torsion spring constant—F1.0:**

   torsional stiffness: 10.54 N•m/rad ≡ 0.184 N•m/degree of deflection
                               (386 dyne•cm/degree deflection);

   shear stress constant: 29.3 pascals/radian of deflection (0.511 pascals/degree of deflection) (1.067 lbf/100·ft²/degree of deflection).

d) **Rotor sleeve speeds:**

   high speed: 600 r/min;

   low speed: 300 r/min.

NOTE   Other rotor speeds are available in viscometers from various manufacturers. Additional measurements are typically made at rotor speeds of 200 r/min, 100 r/min, 6 r/min and 3 r/min. Improved accuracy for hydraulics calculations can be achieved by utilizing shear stress values at 60 r/min and 30 r/min.

7.3.1.2 Timer or Stopwatch, accuracy within ±5 seconds per hour.

7.3.1.3 Thermostatically controlled viscometer cup (thermocup), such as temperature control for:

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

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

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

7.3.1.5 Field mixer, cup type, to operate at 10,000 r/min to 15,000 r/min.

7.3.2 Procedure

In order to determine drilling fluid viscosity and gel strengths using a direct-indicating viscometer, the following procedure shall be used.

a)   Stir the drilling fluid sample for 5 min using the field mixer set at the 10,000 r/min speed.

b)   Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 50 mL to 100 mL) in the cup for displacement of fluid due to the viscometer bob and sleeve. Immerse the rotor sleeve exactly to the scribed line.

Measurements in the field shall be made with minimum delay from the time of drilling fluid sampling. Testing shall be carried out at either 50 °C ±1 °C (120 °F ±2 °F) or 65 °C ±1 °C (150 °F ±2 °F) for reference comparisons to historical data. Testing at a lower temperature, such as 4 °C ±1 °C (40 °F ±2 °F), is recommended for low temperature effects. The place of sampling shall be stated in the report.

Caution—The maximum recommended operating temperature is 90 °C (190 °F). If it is necessary to test fluids above this temperature, a solid metal bob shall be used. Liquid trapped inside a hollow bob can vaporize when immersed in high temperature fluid and cause the bob to explode.

c)   Heat (or cool) the sample to the selected temperature. Use constant shear at 300 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.

d)   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 (or digital) shear stress reading, R_{600}.

e) Reduce the rotor speed to 300 r/min and wait for the dial reading to reach steady value. Record the dial (or digital) shear stress reading, R_{300}.

   NOTE Additional dial reading values are often measured and reported, typically R_{200}, R_{100}, R_6, and R_3. Measurement of all dial readings shall be performed stepwise from the highest to the lowest revolutions per minute-i.e. typically, 600 r/min, 300 r/min, 200 r/min, 100 r/min, 6 r/min and 3 r/min. Improved modeling accuracy can be achieved by also measuring the R_{60} and R_{30} shear stress readings at speeds of 60 r/min and 30 r/min.

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

g) Stop the rotor and allow the drilling fluid sample to stand undisturbed for 10 s.

   If using a hand-crank viscometer, slowly and steadily turn the gel-knob in the appropriate direction to produce a positive dial reading. Record the maximum reading as the initial or 10 s gel strength.

   For instruments having a 3 r/min speed, the maximum dial (or digital) reading immediately attained after starting rotation at 3 r/min is the initial or 10 s gel strength.

   Record the initial gel strength, β_{10s}, in pounds-force per one hundred square feet (lbf/100•ft²).

   NOTE The gel strength value reported directly from the dial reading is an approximation value of lbf/100•ft². Dial reading units are degrees of deflection (1/360 of 1 full rotation). Each 1 degree of deflection is equal to 0.511 Pa in SI units and to 1.067 lbf/100•ft² in USC units. These exact unit corrections are often neglected for simplicity in reporting. Common field practice when reporting pascals is to divide dial units by 2 for simplicity.

h) Re-stir the drilling fluid sample at 600 r/min for 10 s, stop the motor and then allow the drilling fluid to stand undisturbed for 10 min ±10s. Repeat the measurements as in item g) and record the maximum dial (or digital) reading as the 10 min gel strength, β_{10m}, in pounds-force per one hundred square feet (lbf/100•ft²).

7.3.3 Calculations

7.3.3.1 Direct-indicating viscometer calculation constants

The dimensions for the rotor R1, bob B1 and spring F1.0 as described in 7.3.1.1, determine the following (or thorough calibration of the electronic instruments) constants:

— 1° deflection of the bob equals a shear stress of approximately 1 lbf/100•ft² (0.5 Pa), or more exactly 1 degree of deflection = 1.067 lbf/100•ft² and = 0.511 Pa;

— 1 r/min of the rotor equals a shear rate of 1.7023 s⁻¹.

For field reporting in USC units, plastic viscosity (η_{PV}) is reported with centipoise units and yield point ($Y_{P,USC}$) is reported with lbf/100 ft² units. In Equations (8) and (10) these parameters actually have direct-indicating viscometer units (degrees/s⁻¹ and degrees, respectively). This practice allows the parameters to be reported as integer values for simplicity.

NOTE Plastic viscosity is commonly known in the industry by the abbreviation PV and yield point by the abbreviation YP expressed in lbf/ft², units are generally omitted.

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

7.3.3.2 Drilling fluid plastic viscosity

The plastic viscosity, η_{PV}, expressed in millipascal-seconds (centipoises), shall be calculated using Equation (8):

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

where

R_{600} is the shear stress reading at 600 r/min, expressed in degrees of deflection, or equivalent;

R_{300} is the shear stress reading at 300 r/min, expressed in degrees of deflection, or equivalent.

7.3.3.3 Drilling fluid yield point

The yield point can be calculated expressed in either SI units ($Y_{P,SI}$) or in USC units ($Y_{P,USC}$).

a) Yield point expressed in SI units

$Y_{P,SI}$, expressed in pascals ($Y_{P,SI}$) , shall be calculated using Equation (9):

$$Y_{P,SI} = 0.511 \times \left(R_{300} - \eta_{PV} \right) \tag{9}$$

where

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

R_{300} is the dial reading at 300 r/min, expressed in degrees of deflection, or equivalent.

NOTE   The conversion from lbf/100•ft^2 to pascals is to multiply by 0.479. However, each 1 degree of deflection is exactly equal to 0.511 Pa in SI units, so Equation (9) converts the values obtained from dial units to pascals. Common field practice when reporting yield point in pascals is to divide degrees deflection [Equation (10)] by 2 for simplicity.

b) Yield point expressed in USC units

$Y_{P,USC}$, expressed in pounds-force per 100 square feet, shall be calculated using Equation (10):

$$Y_{P,USC} = R_{300} - \eta_{PV} \tag{10}$$

where

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

R_{300} is the dial reading at 300 r/min, expressed in degrees of deflection, or equivalent.

NOTE   The yield point value calculated with Equation (10) from the dial readings is an approximation value of lbf/100•ft^2. Dial reading units are degrees of deflection (1/360 of 1 full rotation). Each 1 degree of deflection is equal to 1.067 lbf/100•ft^2 in USC units. In Equation (10), the exact unit correction is neglected for simplicity in reporting.

7.3.3.4 Drilling fluid apparent viscosity

The apparent viscosity, η_{AV}, expressed in millipascal-seconds (centipoises), shall be calculated using Equation (11):

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

where

R_{600} is the dial reading at 600 r/min, expressed in degrees of deflection, or equivalent.

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

7.3.3.5 Reporting

The plastic viscosity, yield point, initial 10-s gel strength, and 10-min gel strength, shall be reported on the daily drilling fluid report.

8   Static Filtration

8.1   Principle

Measurement of the filtration behavior and the filter cake characteristics of a drilling fluid are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as the oil [nonaqueous fluid (NAF)], water, or emulsion concentration.

Filtration characteristics of a drilling fluid are affected by the quantity, type, and size of solid particles in the drilling fluid and by properties of the liquid phase. Interactions of these various components can be influenced by temperature and pressure.

Tests are run under static conditions at both low-temperature, low-pressure and high-temperature, high-pressure, and each requires different equipment and techniques.

8.2   Low-temperature, Low-pressure Filtrate Test

8.2.1 Apparatus

8.2.1.1   Filter press, consisting mainly of a cylindrical drilling fluid cell having a filtration area inside diameter of 76.2 mm ±0.5 mm (3.0 in. ±0.02 in.) and a height of at least 64.0 mm (2.5 in.).

This cell shall be made of materials resistant to strongly alkaline solutions and designed such that the pressure source is conveniently fitted to the top of the cell with a mechanism that allows pressure to be isolated and safely bled from the cell for disassembly. It shall also be designed such that a sheet of 90 mm (3.54 in.) diameter filter paper can be placed in the bottom of the cell just above a suitable support. The filtration area

shall be 45.8 cm^2 ±0.6 cm^2 (7.1 in. 2 ±0.1 in.^{2}). Below the filter paper support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets and the entire assembly supported by a stand. Pressure can be applied with any nonhazardous gas medium. The filter press is equipped with a pressure regulator that can be pressurized using a portable pressure cylinder, pressure cartridge or other means for applying a controllable differential pressure. To obtain correlative results, only one thickness of filter paper shall be used.

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

8.2.1.2   Filter paper, qualitative, hardened low-ash, slow filtration rate, grade 50 [2]. A new paper shall be required for each test.

8.2.1.3   Timer, digital or mechanical, with at least a 30 min interval, accuracy within ±5 s per hour.

8.2.1.4   Graduated cylinder, to contain (TC) with a capacity of 10 mL (0.1 mL graduations), 20 mL (0.2 mL graduations), or 25 mL (0.2 mL graduations).

8.2.1.5   Ruler, graduated in millimeters (1/32 in.), to measure filter cake thickness.

8.2.2 Procedure

In order to conduct a low-temperature, low-pressure test on a sample of drilling fluid, the following procedure shall be used.

a)   Measure and record the initial temperature of the drilling fluid to the nearest degree Celsius (degree Fahrenheit).

b)   Inspect the cell and all components, and especially the screen, to ensure that all parts are clean and dry, and the gaskets are not cracked, distorted, or worn. Assemble the cell by placing a gasket in the base cap, place the screen on top of the gasket, place the filter paper on top of the screen, place the sealing gasket on top of the paper, then assemble the cell. Pour the drilling fluid sample into the cell to within 12 mm (0.5 in.) of the top and complete the assembly.

c)   Place a clean dry graduated cylinder under the drain tube to collect the filtrate. Close the relief valve and adjust the regulator so that a pressure of 700 kPa ±35 kPa (100 lbf/in.^{2} ±5 lbf/in.^{2}) is applied within 30 s or less. Start the 30 min timer immediately at the time of pressure application.

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

NOTE   Reading the meniscus correctly is extremely important for accuracy. First, always read the meniscus with the interface at eye level. Second, for the air-to-liquid meniscus, read the volume at the lowest point of the meniscus

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[2] This grade 50 filter paper is a thin, highly glazed, qualitative, hardened low-ash filter paper made from high-quality cotton linters, designed for slow filtration and the retention of very fine crystalline precipitates. It should have a lint-free surface and be highly resistant to acid and alkaline solutions. The filter paper has the following typical properties that may vary by manufacturer: slow filtration rate: 2685 s/100 mL (Herzbergs); particle retention in liquid: 2 μm to 5 μm (at 98 % efficiency); ash content: <0.015 % by weight; basis weight: 96 g/m^2 (0.02 lb/ft^2); and thickness: 0.115 mm (0.0045 in.).

that is in the middle of the graduated cylinder receiver at the very bottom of the liquid. For opaque liquids, it might be necessary to estimate the top of the liquid in the middle of the cylinder.

e) Report the volume of filtrate, V_f, in milliliters (to the nearest 0.1 mL) and the initial drilling fluid temperature in degrees Celsius (degrees Fahrenheit). Save the filtrate for chemical analysis.

Caution—Before attempting to open or disassemble the cell, verify that all pressure in the cell has been relieved by following the apparatus manufacturer's recommended procedures.

f) Remove the cell assembly from its support, open the cell and discard any fluid remaining in the cell. Disassemble the cell and remove the filter paper and deposited filter cake. During this process, use care to avoid disturbing the filter cake and any associated drilling fluid. Using a gentle, low-pressure stream of water to avoid erosion; wash the excess drilling fluid from the deposited filter cake.

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

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

8.3   High-temperature, High-pressure Filtration Test up to 175 °C (350 °F)

8.3.1   Principle 8.3.1.1 Filtration tests performed at high-temperature, high-pressure (HTHP) static conditions measure the filtration behavior and filter cake characteristics which are fundamental to the treatment and control of drilling fluids. Filtration characteristics are affected by the quantity, type and size of solid particles and the filtration control additives, any emulsified nonaqueous liquids and by properties of the liquid phase

8.3.1.2 Filtration tests are performed at HTHP static conditions using a differential pressure of 3450 kPa (500 lbf/in.²) for 30 min. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing above 175 °C (350 °F) to 230 °C (450 °F) [see Section 8.4]. Use only filtration equipment and procedure rated by the manufacturer for the temperature required.

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

Caution—Always follow the manufacturers' recommendations when using their equipment and media.

8.3.2 Apparatus for Testing up to 175 °C (350 °F)

8.3.2.1   HTHP filter press, consisting of the following components:

a) **filter cell**, rated for working pressure up to 9000 kPa (1300 lbf/in.²) at a temperature of 175 °C (350 °F). Designed with a thermometer well or thermocouple in direct contact with the fluid near the center of the cell, fitted with a removable end and filter-media support, chemical resistant seals, and valves or valve stems on each end of the cell can be opened or closed during the test.

b) **pressurized gas source**, shall be nitrogen or carbon dioxide, with regulators;

NOTE   Nitrogen is the preferred gas for this application.

Warning—Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for water-based drilling fluid Garrett gas train carbonate analysis (Annex A.5).

c) heating system with temperature controller or thermostat, capable of heating to 175 °C (350 °F);

d) high-pressure filtrate collection vessel and regulator capable of controlling pressures to 1380 kPa (200 lbf/in.^{2}) to maintain the minimum backpressure (see Table 3), to avoid flashing or evaporation of the filtrate;

Warning—Not all manufacturers' equipment can be used above 150 °C (300 °F). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions. Strict adherence to manufacturer's recommendations as to sample volumes, equipment temperatures and pressures, O-ring material selection and inspection, and other operating instructions is essential. Using appropriate personal protective equipment and safety barriers is recommended when operating HTHP equipment. Failure to follow these precautions could result in serious injury.

8.3.2.2 Filter paper, qualitative, hardened low-ash, slow filtration rate, grade 50 [2]. A new paper is required for each test.

8.3.2.3 Timer, digital or mechanical, with at least a 30 min interval and accuracy within ±5 seconds per hour.

8.3.2.4 Thermometer, with a range up to 260 °C (500 °F), accuracy of ±3 °C (±5 °F) with a 12.5 cm (5 in.) or longer stem, or a thermocouple and temperature meter with a range up to 260 °C (500 °F), preferred.

8.3.2.5 Graduated cylinder, (TC) with a capacity of 10 mL (0.1 mL graduations), 20 mL (0.2 mL graduations), or 25 mL (0.2 mL graduations).

8.3.2.6 Field mixer, cup type, to operate at 10,000 r/min to 15,000 r/min.

8.3.2.7 Ruler, graduated in millimeters (1/32 in.), to measure filter cake thickness.

Table 3—Recommended Minimum Backpressure

Test Temperature		Vapor Pressure		Minimum Backpressure	
°C	°F	kPa	lbf/in.^{2}	kPa	lbf/in.^{2}
95–149	200–299	84–462	12.1–67	700	100
150–175	300-350	462–932	67–135	1104	160
175–189	350–375	932–1269	135–184	1400	200
190–199	375–399	1269–1704	184–247	1900	275
200–219	400–424	1704–2245	247–326	2500	350
220–230	425–450	2245–2912	326–422	3100	450

8.3.3 Procedure

In order to conduct an HTHP filtration test on a sample of drilling fluid up to 175 °C (350 °F) using a differential pressure of 3450 kPa (500 lbf/in.^{2}), the following procedure shall be used.

a) 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 maintain this temperature.

Warning—The outside of the jacket can get hot enough to cause burns.

If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, then that temperature shall be monitored and reported during the filtration test. Under the "Comments" section, record if the results were based on fluid temperature measured with a direct contact thermocouple or using the 30 min heat-up time (after the cell wall reaches test temperature).

b) Stir the drilling fluid sample for 5 min using the field mixer set at the 10,000 r/min speed.

Install and close the upper valve stem on the test cell. Pour the fluid sample into the filter cell, leaving at least a 2.5 cm (1 in.) space in the cell to allow for fluid expansion. Install the filtration media.

c) Ensuring that all O-rings are in place, install the lower end-cap, with the valve stem open, above the filtration media and secure. Close the lower valve stem.

d) Complete the assembly of the filter cell and place it in the preheated heating jacket with the filter media on the bottom. Rotate the cell to lock it in place with the pin inside the heating jacket. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, insert a thermometer into the well of the filter cell wall.

e) Ensure that the filtrate collection vessel is completely free of water or oil (NAF). Connect the regulated-pressure filtrate collection vessel assembly onto the lower valve stem and lock it in place. Connect the regulated-pressure assembly to the upper valve and lock it in place.

f) Keeping the two valve stems closed, adjust the pressure on the upper and lower pressure regulators to the minimum backpressure listed in Table 3 for the test temperature. Open the upper valve stem and readjust the upper pressure regulator to the minimum backpressure. Maintain these pressures during the heat-up time. If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, proceed to step g) when the thermocouple temperature reaches the test temperature. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, wait 30 min after the filter cell wall reaches the test temperature before proceeding to step g).

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

g) When the sample reaches the selected test temperature, as indicated by the thermocouple or after the 30 min heat-up time, ensure that the lower pressure regulator is at the minimum backpressure.

Open the lower valve stem and immediately increase the pressure on the upper regulator to a value 3450 kPa (500 lbf/in.^{2}) above the minimum backpressure. This will start the filtration process. Start the timer.

Maintain the test temperature to within ±3 °C (±5 °F) during the test, as indicated by the thermocouple or thermometer in the filter cell wall. Maintain these pressures during the test, if the backpressure rises above the minimum backpressure, cautiously draw off and collect a portion of the filtrate to reduce the backpressure.

Warning—Filtrate will be hot, and steam may escape if the valve is left open too long.

h) Collect the filtrate in the graduated cylinder. Record the total volume of filtrate collected during the 30 min test, in milliliters (to the nearest 0.1 mL). Correct the filtrate volume as V_f, for a filter area of 45.8 cm^2 (7.1 in.^{2}). For example, the typical HTHP cell has a filter area of 22.6 cm^2 (3.5 in.^{2}), so double the filtrate volume collected. Also note the presence of, or volume of any solids, oil, or emulsion if present.

   NOTE   Reading the meniscus correctly is extremely important for accuracy. First, always read the meniscus with the interface at eye level. Second, read the graduation that coincides with the bottom of the curved surface of the liquid in the middle of the graduated cylinder.

i) Immediately after collecting the 30 min filtrate, close the lower, then the upper valve stem. Switch off the heating jacket and unplug from the electrical power source. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, and then carefully disconnect the pressurization system.

   Warning—Cell and heating jacket are still hot!

j) Using appropriate personal protective equipment, carefully 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.

   Warning—The filter cell may still be pressurized even after the cell is cooled. Safety cell clamps are available which keep the cell upright and the lid clamped during depressurization. To avoid possible serious injury, keep the cell upright and cool to room temperature, and then bleed pressure from cell before disassembling. The use of a safety apparatus to secure the cell and prevent uncontrolled release of pressure during disassembly is recommended.

k) After cooling the cell, bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. A tight-fitting hose may be used to divert the pressurized gas and any liquid to a safe contained area during depressurization. Ensure that pressure is fully released before removing the cap. Carefully disassemble the cell.

   NOTE If there is resistance when loosening the cap, pressure may still be inside the cell. Ensure that all pressure is released before attempting to remove the end-cap.

l) Remove the filter cake with the filtration medium. Measure the filter cake thickness, at its center, to the nearest millimeter (1/32 in.).

m) Pour the liquid from the cell.

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

8.3.4 Calculation and Reporting

8.3.4.1 The reported filtrate volume, V_f, shall be corrected to a filter area of 45.8 cm^2 (7.1 in.^{2}). HTHP filter cells usually have half the standard filter area, 22.6 cm^2 (3.5 in.^{2}), thus double the measured filtrate volume before reporting.

8.3.4.2 Report the cake thickness to the nearest millimeter (1/32 in.), its texture, character, and the presence of any emulsion or oil in the filtrate.

8.4 HTHP Static Filtration Test above 175 °C (350 °F) up to and Including 230 °C (450 °F)

8.4.1 Principle

8.4.1.1 Filtration tests performed at high-temperature high-pressure (HTHP) static conditions measure the filtration behavior and filter cake characteristics which are fundamental to the treatment and control of drilling fluids. Filtration characteristics are affected by the quantity, type and size of solid particles and the filtration control additives, any emulsified nonaqueous liquids and by properties of the liquid phase.

8.4.1.2 Filtration tests are performed at HTHP static conditions using a differential pressure of 3450 kPa (500 lbf/in.^{2}) for 30 min. Two filtration procedures are given: one for testing up to 175 °C (350 °F) [see 8.3] and here for testing above 175 °C (350 °F). Use only filtration equipment and procedure rated by the manufacturer for the temperature required.

8.4.1.3 It is suggested that the equipment be equipped with a thermocouple in direct contact with the fluid contained in the cell to more accurately measure the temperature. Do not use filter paper (including fiberglass backed filter paper) if the test temperature is above 200 °C (400 °F). A porous stainless-steel filter media up to maximum manufacturer's recommendation for the equipment may be used. Ceramic disks may also be used in equipment modified to utilize these disks.

Caution—Always follow the manufacturers' recommendations when using their equipment and media.

8.4.2 Apparatus

8.4.2.1 HTHP filter press, consisting of the following components:

a) **Filter Cell,** 500 mL volume or larger, with a rated working pressure up to 15,500 kPa (2250 lbf/in.^{2}) at a temperature of 230 °C (450 °F) Only use cells and equipment rated by the manufacturer for the test temperature and pressures.

   Designed with a thermometer well or thermocouple in direct contact with the fluid near the center of the cell, fitted with a removable end and filter-media support, chemical resistant seals, and valves or valve stems on each end of the cell can be opened or closed during the test.

b) **pressure gas source** shall be nitrogen or carbon dioxide, with regulators;

   NOTE Nitrogen is the preferred gas for this application.

c) **heating system with temperature controller** or **thermostat**, capable of heating to 230 °C (450 °F);

d) **high-pressure filtrate collection vessel and regulator,** capable of controlling pressures to 3450 kPa (500 lbf/in.^{2}) to maintain the minimum backpressure (see Table 3), to avoid flashing or evaporation of the filtrate;

Warning—Not all manufacturers' equipment can be used above 150 °C (300 °F). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions. The 175 mL and 250 mL filtration cells are not recommended for use at these higher temperatures and pressures. Strict adherence to manufacturers' recommendations as to sample volumes, equipment temperatures and pressures, O ring material selection and inspection, and other operating instructions is essential. High temperature O-rings and elastomer seals are required for high temperatures.

Equipment manufacturers often recommend replacement or inspection after every test. Inspection should include making sure the O rings and elastomer seals are pliable with no permanent deformations, nicks, or cuts, etc. A loss of pressure during a test may be an indication of an O-ring or elastomer seal failure and should be corrected. Using appropriate personal protective equipment and safety barriers is recommended when operating HTHP equipment. Failure to follow these precautions could result in serious injury.

Warning—Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges shall be used only for Garrett gas train carbonate analysis (Annex A.5).

8.4.2.2 Filter media

Two types of filter media shall be used to conduct HTHP filtration test.

a) Filter paper, qualitative, hardened low-ash, slow filtration rate, grade 50 [2] (see 8.3.2.2), and glass fiber backing disk [3] for temperatures above 175 °C (350 °F) up to temperatures of 200 °C (400 °F). A new filter medium shall be required for each test.

b) Porous sintered stainless-steel disk [4], Dynalloy Grade 5 [5] or equivalent, diameter 6.35 cm (2.5 in.), for temperatures above 200 °C (400 °F). A new disc is required for each test.

8.4.2.3 Timer, digital or **mechanical,** with at least 30 min interval and accuracy within ±5 s per hour.

8.4.2.4 Thermometer, with a range up to 260 °C (500 °F), accuracy of ±3 °C (±5 °F) with a 12.5 cm (5 in.) or longer stem, or a thermocouple and temperature meter with a range up to 260 °C (500 °F), preferred.

8.4.2.5 Graduated cylinder, with a capacity of 10 mL (0.1 mL graduations), 20 mL (0.2 mL graduations), or 25 mL (0.2 mL graduations).

8.4.2.7 Field mixer, cup type, to operate at 10,000 r/min to 15,000 r/min.

8.4.2.8 Ruler, graduated in millimeters (1/32 in.), to measure filter cake thickness.

8.4.3 Procedure

In order to conduct an HTHP static filtration test above 175 °C (350 °F) up to and including 230 °C (450 °F) using a differential pressure of 3450 kPa (500 lbf/in.[2]), the following procedure shall be used.

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[3] Glass fiber backing disk is required for testing between 175 °C (350 °F) up to 200 °C (400 °F) with a particle retention rating in liquid not less than 2 µm (at 98 % efficiency). These backing disks are a separate item and are inserted between the filter paper and the filter media support of the filter cell.

[4] The backing disk shall be comprised of sintered stainless steel metal fibers with a particle retention rating in liquid of not less than 5 µm nominal and 10 µm absolute for temperatures above 200 °C (400 °F).

[5] Dynalloy Grade 5 is an example of a suitable brand porous sintered stainless-steel disc available commercially. This information is given for convenience of users of API 13B-1 and does not constitute and endorsements by API of these products.

a) 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 maintain this temperature.

Warning—The outside of the jacket can get hot enough to cause burns.

If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, then that temperature shall be monitored and reported during the filtration test. Under the "Comments" section, record if the results were based on fluid temperature measured with a direct contact thermocouple or using the 30 min of heat-up time (after the cell wall reaches test temperature).

b) Stir the drilling fluid sample for 5 min using the field mixer set at the 10,000 r/min speed.

c) Install and close the upper valve stem on the test cell. Pour the fluid sample into the filter cell, leaving at least a 5.0 cm (2 in.) space in the cell to allow for fluid expansion. Install the filtration media.

d) Ensuring that all O-rings are in place, install the lower end-cap, with the valve stem open, above the filtration media and secure. Close the lower valve stem.

e) Complete the assembly of the filter cell and place it in the preheated heating jacket with the filter media on the bottom. Rotate the cell to lock it in place with the pin inside the heating jacket. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, insert a thermometer into the well of the filter cell wall.

f) Ensure that the filtrate collection vessel is completely free of water or oil (NAF). Connect the regulated-pressure filtrate collection vessel assembly onto the lower valve stem and lock it in place. Connect the regulated pressurized gas source to the upper valve and lock in place.

g) Keeping the two valve stems closed, adjust the pressure on the upper and lower pressure regulators to the minimum backpressure for the test temperature as shown in Table 3. Open the upper valve stem and readjust the upper pressure regulator to the minimum backpressure temperature.

h) Maintain these pressures during the heat-up time. If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, proceed to item i) when the thermocouple temperature reaches the test temperature. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, wait 30 min after the filter cell wall reaches the test temperature before proceeding to step i).

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

i) When the sample reaches the selected test temperature, as indicated by the thermocouple or after the 30 min heat-up time, ensure that the lower pressure regulator is at the minimum backpressure for the test temperature as shown in Table 3.

Open the lower valve stem and immediately increase the pressure on the upper regulator to a pressure 3450 kPa (500 lbf/in.^{2}) higher than the backpressure. This will start the filtration process. Start the timer.

j) Maintain the test temperature to within ±3 °C (±5 °F) during the test, as indicated by the thermocouple or thermometer in the filter cell wall. Maintain these pressures during the test, if the backpressure rises above the selected backpressure, cautiously draw off and collect a portion of the filtrate to reduce the backpressure.

Warning—Filtrate will be hot and steam may escape if the valve is left open too long.

k) Collect the filtrate in the graduated cylinder. Record the total volume of filtrate collected during the 30 min test, in milliliters (to the nearest graduation). Correct the filtrate volume as V_f, for a filter area of 45.8 cm^2 (7.1 in.^{2}). For example, the typical HTHP cell has a filter area of 22.6 cm^2 (3.5 in.^{2}), so double the filtrate volume reported. Also note the presence of, or volumes of, any solids, oil, or emulsions, if present.

   NOTE   Reading the meniscus correctly is extremely important for accuracy. First, always read the meniscus with the interface at eye level. Second, read the graduation that coincides with the bottom of the curved surface of the liquid in the middle of the graduated cylinder.

l) Immediately after collecting the 30 min filtrate, close the lower, then the upper valve stem. Switch off the heating jacket and unplug from the electrical power source. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, and then carefully disconnect the pressurization system.

Warning—Cell and heating jacket are still hot!

m) Using appropriate personal protective equipment, carefully 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.

   Warning—The filter cell may still be pressurized even after the cell is cooled. Safety cell clamps are available, which keep the cell upright and the lid clamped during depressurization. To avoid possible serious injury, keep the cell upright and cool to room temperature, and then bleed pressure from cell before disassembling. The use of a safety apparatus to secure the cell and prevent uncontrolled release of pressure during disassembly is recommended.

n) After cooling the cell, bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. A tight-fitting hose may be used to divert the pressurized gas and any liquid to a safe contained area during depressurization. Ensure that pressure is fully released before removing the cap. Carefully disassemble the cell.

   NOTE   If there is resistance to loosening the cap, pressure may still be inside the cell. Ensure that all pressure is released before attempting to remove the end-cap.

o) Remove the filter cake with the filtration medium. Measure the filter cake thickness, at its center, to the nearest millimeter (1/32 in.).

p) Pour the liquid from the cell.

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

8.4.4 Calculation and Reporting

8.4.4.1 The reported filtrate volume, V_f, shall be corrected to a filter area of 45.8 cm² (7.1 in.²). HTHP filter cells usually have half the standard filter area 22.6 cm² (3.5 in.²), thus double the observed volume before reporting.

8.4.4.2 Report the cake thickness to the nearest millimeter (1/32 in.), its texture, and the presence of any emulsion or oil in the filtrate.

9 Retort Test for Water, Oil (NAF), and Solids Concentrations

9.1 Principle

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

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

9.1.2 Knowledge of water, oil (NAF), and solids concentrations is fundamental to proper control of drilling fluid properties such as rheology, density, filtration, oil (NAF) content, and salinity of the aqueous (water) phase. Knowledge of solids contents is essential to the evaluation of drilling fluid processing equipment performance, see API 13C

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

9.1.4 For the volumetric method, the volume fractions expressed as percentages of water, oil (NAF), and solids are calculated from the volume of the retort cup and the condensed liquid volumes of water and oil (NAF) collected in a liquid receiver.

 9.1.5 For the gravimetric method, the volume fractions expressed as percentages of water, oil (NAF), and solids are calculated from the starting mass of retorted drilling fluid, mass of dry solids after retorting, mass of recovered liquids, densities of the water, oil (NAF), and drilling fluid plus the measured (observed) volume of oil (NAF) collected in a precision graduated receiver.

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

Warning—Formate-based or nitrate-based drilling fluids should not be retorted. This can lead to an explosion.

9.2 Apparatus

9.2.1 Retort instrument, as specified below.

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

Standard cup sizes are 10 mL (tolerance ±0.05 mL), 20 mL (tolerance ±0.1 mL), and 50 mL (tolerance ±0.25 mL). When using the volumetric procedure, the retort cup volume with lid shall be verified gravimetrically in accordance with the procedure and calculations provided in Annex I (see I.6).

b) **Condenser**, capable of cooling the oil (NAF) and water vapors below their vaporization temperature.

c) **Heating jacket**, of sufficient power to raise the temperature of the sample to 500 °C ±40 °C (930 °F ±70 °F).

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

9.2.2   Graduated cylinder (TC) or **Special graduated liquid receiver** (TC) (JP tube)

Graduated cylinders are used for standard accuracy retort measurements, special graduated liquid receivers are recommended for more accurate retort measurements. The special graduated liquid receivers are specially designed cylindrical glassware with a rounded bottom to facilitate cleaning and a funnel-shaped top to catch falling drops, often referred to in industry as JP-tubes. The two types and three sizes of receivers shall meet the following specifications:

— tolerance (milliliter): see Table 4;

— frequency of graduations: see Table 4;

— calibration**:** TC at 20 °C (68 °F);

— scale: milliliter or volume fraction (as a percentage);

— material: transparent and inert to oil (NAF), water, and salt solutions at temperatures up to 32 °C (90 °F);

When using the volumetric procedure, the receiver volume shall be verified gravimetrically in accordance with the procedure and calculations in Annex I (see I.6).

Table 3—Specifications of Retort Liquid Receivers

Graduated Cylinder	Tolerance	Frequency of Graduations
Graduated cylinder 10 mL (TC)	±0.10 mL *	0.10 mL and/or 1 %
Graduated cylinder 20 mL (TC)	±0.15 mL	0.20 mL and/or 1 %
Graduated cylinder 50 mL (TC)	±0.25 mL *	≤ 1.0 mL and/or ≤ 2 %
Special Graduated Liquid Receiver	**Tolerance**	**Frequency of Graduations**
Special liquid receiver 10 mL (TC)	±0.05 mL	0.10 mL and/or 1 %
Special liquid receiver 20 mL (TC)	±0.05 mL	0.10 mL and/or 0.5 %
Special liquid receiver 50 mL (TC)	±0.15 mL	≤ 0.25 mL and/or ≤ 0.5 %
* Glass graduated cylinder: meets ASTM E1272 [7]. Class A standards.		

9.2.3   Fine steel wool, oil-free.

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

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

9.2.5   Pipe cleaners and/or **T-drill**.

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

9.2.7   Corkscrew.

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

9.2.9   Marsh funnel (see 7.2.1.1).

9.2.10   Top-loading balance, maximum capacity capable of weighing fully assembled and loaded retort assembly with a precision of ±0.01 g.

9.2.11   Mud balance (see 5.2.1 or 6.2.1 preferred).

9.2.12   Defoaming agent.

9.3   Procedure—Volumetric Method

In order to perform a retort test for a sample of drilling fluid using the volumetric method, the following procedure shall be used.

a) 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 shall also be cleaned and dried before each test using pipe cleaners and/or T-drill. A build-up of material in the condenser passage can decrease condensation efficiency that can cause erroneous liquid readings in the test and, for some types of equipment, may present a safety hazard.

   Caution—A partially clogged condenser passage is a safety hazard.

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

c) Pack the retort body with steel wool.

d) Collect a representative sample of the drilling fluid and allow it to cool to approximately 27 °C (80 °F). Screen the test sample through the 1.68 mm (0.066 in.) screen (US Sieve No.12) of the Marsh funnel to remove lost circulation material, large cuttings, or debris.

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

   NOTE   Air or gas entrapment in the retort sample will result in erroneously high retort solids, due to a reduced volume of drilling fluid sample. If drilling fluid sample contains gas or air, add 2 drops to 3 drops of defoaming agent to about 300 mL of drilling fluid and stir slowly for 2 min to 3 min to release gases. Alternatively, the Annex E procedure can be used for severe cases of foaming or air/gas entrapment.

f) Fill the retort sample cup slowly to avoid air entrapment with drilling fluid (cup = sample volume V_{df}) 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 the excess sample fluid from the lid; avoid wicking out the drilling fluid through the hole.

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

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

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

   NOTE   If using the special liquid receiver (JP-tube), due to the rounded bottom and length, it often must be angled out from the retort condenser passage/retort assembly and off the edge of the worktable such that a condenser tube clip or other special support is needed.

j) Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Continue heating for 10 min after the last condensate is collected. Collect the condensate into the glass liquid receiver. If any drilling fluid solids boil over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool in the retort body.

k) Remove the liquid receiver and allow it to cool.

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

l) Record the total liquid volume, V_L, (water and oil), and oil (NAF) volume, V_O, collected in the liquid receiver.

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

m) Turn off the heating jacket. After the retort assembly has cooled carefully remove the retort assembly and condenser from the heating jacket and allow them to cool. After the retort cell has cooled, remove the condenser. Clean and dry the retort assembly and condenser.

9.4   Calculation —Volumetric Method

9.4.1   Drilling Fluid Retort Volume Fractions (Water, Oil, and Retort Solids)

Using the measured volumes of water and oil (NAF) with the volume of the drilling fluid sample volume (10 mL, 20 mL, or 50 mL), the retort volume fractions of water, oil (NAF), and retort solids in the drilling fluid shall be calculated using Equations (12) to (15).

NOTE The retort solids from the retort test includes both dissolved soluble solids (for example, salt) and suspended insoluble solids (weight material, drilled solids, and additives).

9.4.1.1 Water

The volume of water, V_W, in the condensed sample, expressed in milliliters, shall be calculated using Equation (12):

$$V_W = V_L - V_O \tag{12}$$

where

V_L is the total volume of condensed liquids [water and oil (NAF)], expressed in milliliters;

V_O is the volume of condensed oil (NAF), expressed in milliliters.

The volume fraction of water, φ_W, in the drilling fluid sample, expressed as a percentage, shall be calculated using Equation (13):

$$\varphi_W = 100 \times \frac{V_W}{V_{df}} \tag{13}$$

where

V_W is the condensed water volume, expressed in milliliters;

V_{df} is the whole drilling fluid sample volume, expressed in milliliters.

9.4.1.2 Oil (NAF)

The volume fraction of oil (NAF), φ_O, in the drilling fluid sample, expressed as a percentage, shall be calculated using Equation (14):

$$\varphi_O = 100 \times \frac{V_O}{V_{df}} \tag{14}$$

where

V_O is the volume of oil, expressed in milliliters;

V_{df} is the whole drilling fluid sample volume, expressed in milliliters.

NOTE   Some drilling fluid additives decompose in the retort test as nonaqueous components which condense as oil, even though no oil actually exists in the drilling fluid.

9.4.1.3 Dried retort solids

The volume fraction of dried retort solids, φ_{ds}, expressed as a percentage of the drilling fluid volume, shall be calculated using Equation (15).

$$\varphi_{ds} = 100 - \left(\varphi_W + \varphi_O \right) \tag{15}$$

where

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

φ_O is the volume fraction of oil (NAF), expressed as a percentage of the drilling fluid volume.

NOTE The calculated dried retort solids, φ_{ds}, Equation (15), includes both dissolved soluble materials (e.g. salt) and insoluble suspended solids (weighting material, drilling fluid additives, and drilled solids). The quantity of insoluble suspended solids can be determined by correcting for the volume of soluble solids from salts using the chloride concentration, c_{Cl} (see Section 14), using known volume factors for salt solutions.

9.4.2   Drilling Fluid Solids Volume Fractions (Suspended, Low-gravity, Weighting Material)

9.4.2.1 Additional calculations are required to find the percentage (volume fraction) of suspended solids and relate them to the relative volumes of low-gravity solids and weighting material. To make these calculations, an accurate drilling fluid density and chloride concentration are needed to correct for the soluble solids content of the dried retort solids.

9.4.2.2 Drilling Fluid Suspended Solids Volume Fraction

The corrected volume fraction of suspended solids, φ_{SS}, expressed as a percentage of the drilling fluid volume, for sodium chloride containing fluids shall be calculated using Equation (16):

$$\varphi_{SS} = \varphi_{ds} - \left[\frac{\varphi_W \times c_{Cl}}{\left(1.68 \times 10^6\right) - 1.21 \times c_{Cl}} \right] \tag{16}$$

where

φ_{ds} is the volume fraction of dried retort solids, expressed as a percentage of the drilling fluid volume;

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

c_{Cl} is the chloride ion concentration of the filtrate, expressed in milligrams per liter (see 14.4.1).

NOTE   The corrected volume fraction of suspended solids calculation using Equation (16), is for sodium chloride only using the chloride concentration, c_{Cl}, expressed in milligrams per liter. Other salt types require alternative calculations.

9.4.2.3   Drilling Fluid Low-gravity Solids Volume Fraction

The density of the filtrate, ρ_f, expressed in grams per milliliter, for sodium chloride containing fluids, shall be calculated using Equation (17):

$$\rho_f = 1.0 + \left(1.09 \times 10^{-6}\right) \times c_{Cl} \tag{17}$$

where

c_{Cl} is the chloride ion concentration of the filtrate, expressed in milligrams per liter (see 14.4.1).

NOTE   The filtrate density, ρ_f, calculation, using Equation (17), is for sodium chloride only using the chloride concentration, c_{Cl}, expressed in milligrams per liter. Filtrate density is the term used for density of the continuous brine phase. Other salt types require alternative calculations.

The density of the water-based drilling fluid shall be measured using a mud balance as described in Section 5 or Section 6 (more accurate). Alternative gravimetric methods, such as a volumetric flask or cup, are also acceptable.

Record the drilling fluid density as ρ_{df}, to the nearest 0.01 g/mL(0.1 lb/gal).

If the density of the oil being used is not known, the density of the base oil (NAF) shall be measured using a mud balance as described in Section 5. Gravimetric methods such as a volumetric flask (or volumetric cup) or an electronic portable handheld density-measuring instrument may also be used to measure the density of oil (NAF).

Record the density of the oil (NAF) as ρ_O, to the nearest 0.01 g/mL(0.1 lb/gal).

The volume fraction of low-gravity solids, ρ_{LG}, expressed as a percentage of the drilling fluid volume, shall be calculated using Equation (18):

$$\varphi_{LG} = \frac{1}{\rho_{WM} - \rho_{LG}}\left[100\rho_f + \left(\rho_{WM} - \rho_f\right)\times\varphi_{SS} - 100\rho_{df} - \left(\rho_f - \rho_O\right)\times\varphi_O\right] \tag{18}$$

where

φ_{SS} is the volume fraction of suspended solids, expressed as a percentage of the drilling fluid volume;

φ_O is the volume fraction of oil (NAF), expressed as a percentage of the drilling fluid volume;

ρ_{df} is the drilling fluid density, expressed in grams per milliliter;

ρ_f is the density of the filtrate for sodium chloride, expressed in grams per milliliter;

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

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

ρ_O is the density of oil (NAF), expressed in grams per milliliter.

NOTE 1   For calculation in USC units, Equation (18) can be used provided all densities are expressed in pounds per gallon.

NOTE 2   Convert density in pounds per gallon to grams per milliliter by dividing the pounds per gallon by 8.345 or convert density in grams per milliliter to pounds per gallon by multiplying the grams per milliliter by 8.345.

9.4.2.3 Drilling Fluid Weighting-Material Volume Fraction

The volume fraction of weighting material, φ_{WM}, expressed as a percentage of the drilling fluid volume shall be calculated using Equation (19):

$$\varphi_{WM} = \varphi_{SS} - \varphi_{LG} \tag{19}$$

where

   φ_{SS} is the volume fraction of suspended solids, expressed as a percentage of the drilling fluid volume;

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

NOTE The volume fraction of weighting material, φ_{WM}, is often referred to as percent high-gravity solids.

9.4.2.4 Drilling Fluid Brine Volume Fraction

For drilling fluids with high salt contents, it may be useful to monitor the brine volume fraction (corrected water content).

The volume fraction of brine, φ_B, expressed as a percentage of the drilling fluid volume shall be calculated using Equation (20):

$$\varphi_B = 100 - \left(\varphi_{WM} + \varphi_{LG}\right) \tag{20}$$

where

   φ_{WM} is the volume fraction of weighting material, expressed as a percentage of the drilling fluid volume;

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

9.4.3   Drilling Fluid Solids Concentrations (Suspended, Low-gravity, Weighting Material)

9.4.3.1 The concentration of low-gravity solids, $c_{LG,SI}$, weighting material, $c_{WM,SI}$, and suspended solids, $c_{SS,SI}$, expressed in kilograms per cubic meter, shall be calculated using Equations (21), (22), and (23), respectively:

$$c_{LG,SI} = 10 \times \left(\rho_{LG} \times \varphi_{LG}\right) \tag{21}$$

$$c_{WM,SI} = 10 \times \left(\rho_{WM} \times \varphi_{WM}\right) \tag{22}$$

$$c_{SS,SI} = c_{WM,SI} + c_{LG,SI} \tag{23}$$

9.4.3.2 The concentration of low-gravity solids, $c_{LG,USC}$, weighting material, $c_{WM,USC}$, and suspended solids, $c_{SS,USC}$, expressed in pounds per barrel, shall be calculated using Equations (24), (25), and (26), respectively:

$$c_{LG,USC} = 3.50 \times \left(\rho_{LG} \times \varphi_{LG} \right) \tag{24}$$

$$c_{WM,USC} = 3.50 \times \left(\rho_{WM} \times \varphi_{WM} \right) \tag{25}$$

$$c_{SS,USC} = c_{WM,USC} + c_{LG,USC} \tag{26}$$

where

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

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

φ_{WM} is the volume fraction of weighting material, expressed as a percentage of the drilling fluid volume;

ρ_{WM} is the density of weighting material, expressed in grams per milliliter.

NOTE   For water-based drilling fluids with added bentonite, additional calculations can be made to determine the relative concentration of bentonite and drill solids components of the low-gravity solids (see API 13I [4]).

9.5 Procedure—Gravimetric Method

In order to perform a retort analysis using the gravimetric method, the following procedure shall be used:

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

    Caution—A partially clogged condenser passage is a safety hazard.

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

c) Pack the retort body with steel wool.

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

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

NOTE   All weights are to be recorded to the nearest 0.01 g.

f)   Collect a representative sample of the drilling fluid and allow it to cool to approximately 27 °C (80 °F). Pour the test sample through the screen with 1.68 mm (0.066 in.) openings (US sieve No. 12) of the Marsh funnel to remove lost circulation material, large cuttings or debris.

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

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

Record the drilling fluid density as ρ_{df}, to the nearest 0.01 g/mL (0.1 lb/gal).

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

j)   Screw the retort sample cup with lid onto the retort body. Weigh the filled retort assembly (sample cup with sample drilling fluid, lid, and retort body packed with steel wool). Record this as m_2, expressed in grams and for sample volume, V_{df} (retort cup volume).

k)   Apply lubricant/sealant sparingly to the threads of the condenser passage stem and attach to the condenser body.

l)   Place the retort assembly into the heating jacket. Close the insulating lid.

m)   Weigh an empty, clean, dry special graduated liquid receiver (JP Tube). Record this as m_3, expressed in grams.

n)   Place the liquid receiver below the condenser passage outlet.

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

NOTE   Due to the rounded bottom and length of the special liquid receiver, it often must be angled out from the retort condenser passage/retort assembly and off the edge of the worktable such that a condenser tube clip or other special support is needed.

o)   Turn on the heating jacket and allow the retort assembly to run a minimum of 1 hr. Continue heating for 10 min after the last condensate is collected. Collect the condensate into the special liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.

p)   Remove the liquid receiver and allow it to cool.

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

q)   Record the total condensed liquid volume (water and oil), V_L, and oil (NAF) volume, V_O, collected in the liquid receiver.

NOTE 1   Reading the meniscus correctly is extremely important for accuracy. Firstly, always read the meniscus with the interface at eye level. Secondly, for the air-to-liquid meniscus, read the volume at the lowest point of the meniscus which is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for the water-to-oil meniscus, read the water volume at the lowest point.

NOTE 2   The total condensed liquid volume value, V_L, is not used in the gravimetric calculations but can be used to determine the lost mass volume equivalent.

r)   Weigh the cooled glass liquid receiver and its condensed liquid content [water and oil (NAF)]; record as m_4, expressed in grams.

s)   Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser and passage stem.

t)   Weigh the cooled retort assembly (i.e. retort sample cup with dried solids, lid, and retort body packed with steel wool); record this as m_5, expressed in grams.

u)   Clean the retort assembly and condenser.

9.6   Gravimetric Method Calculation-Mass Balance

9.6.1   The mass balance factor of the retorted drilling fluid, dry retorted solids, and condensed liquids shall be within ± 3 % using Equation (29), or if not within the range, the procedure shall be repeated.

9.6.2   The mass of the drilling fluid sample, m_{df}, before retorting shall be calculated using Equation (27).

$$m_{df} = m_2 - m_1 \tag{27}$$

where

    m_{df} is the mass of the drilling fluid sample, expressed in grams;

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

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

9.6.3   The mass of the dried retort solids after retorting shall be calculated using Equation (28).

$$m_{ds} = m_5 - m_1 \tag{28}$$

where

    m_{ds} is the mass of the dried retort solids, expressed in grams;

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

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

9.6.4   The mass of liquids [water and oil (NAF)] condensed after retorting shall be calculated using Equation (29).

$$m_L = m_4 - m_3 \tag{29}$$

where

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

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

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

9.6.5   The mass balance factor of the gravimetric retorting method shall be calculated using Equation (30).

The sum of the mass of dried retorted solids, m_{ds}, plus condensed liquids, m_L, after retorting divided by the mass of the drilling fluid sample, m_{df}, shall be greater than 0.97 but less than 1.03.

$$0.97 \leq \frac{m_{ds} + m_L}{m_{df}} \leq 1.03 \tag{30}$$

The retort procedure shall be repeated if this requirement is not met.

9.7   Gravimetric Method Calculation—Volume Fractions of Water, Oil (NAF), and Solids

9.7.1 Volume Fractions Oil (NAF), Water and Solids

9.7.1.1 Calculate the volume fractions of oil (NAF), water, and solids from the measured mass of retorted drilling fluid, mass of dry solids, density of the water, density of the oil, and density of the drilling fluid plus the measured volume of condensed water.

The volume of drilling fluid retorted, V_{df}, expressed in milliliters, shall be calculated using Equation (31):

$$V_{df} = \frac{m_{df}}{\rho_{df}} \tag{31}$$

where

m_{df} is the mass of the liquid drilling fluid sample, expressed in grams;

ρ_{df} is the drilling fluid density, expressed in grams per milliliter.

The volume fraction of oil (NAF), φ_O, expressed as a percentage of the drilling fluid volume, shall be calculated using Equation (32);

$$\varphi_O = 100 \times \frac{V_O}{V_{df}} \tag{32}$$

where

    V_O is the volume of oil, expressed in milliliters;

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

If the density of the oil (NAF) being used is not known, measure it's density using a mud balance, as described in Section 5. Gravimetric methods such as a volumetric flask (or volumetric cup) or an electronic portable handheld density-measuring instrument may also be used to measure the density of oil (NAF).

The density of the oil (NAF) shall be recorded as ρ_O, to the nearest 0.01 g/mL(0.1 lb/gal) or 10 kg/m^3 (0.5 lb/ft^3).

NOTE Some drilling fluid additives decompose in the retort test as nonaqueous components which condense as oil, even though no oil (NAF) actually exists in the drilling fluid.

9.7.1.2 The volume fraction of water, φ_W, expressed as a percentage shall be calculated using Equation (33):

$$\varphi_W = 100 \times \frac{\left[m_{df} - m_{ds} - \left(V_O \times \rho_O \right) \right]}{V_{df}} \tag{33}$$

where

    m_{df} is the mass of the drilling fluid sample, expressed in grams;

    m_{ds} is the mass of the dried retort solids, expressed in grams;

    V_O is the volume of oil, expressed in milliliters;

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

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

9.7.1.3 The volume fraction of dried retort solids, φ_{ds}, expressed as a percentage of the drilling fluid volume shall be calculated using Equation (34);

$$\varphi_{ds} = 100 - \left(\varphi_W + \varphi_O \right) \tag{34}$$

where

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

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

NOTE   The calculated dried retort solids, φ_{ds}, Equation (34), includes both dissolved soluble materials (e.g. salt) and insoluble suspended solids (weighting material, drilling fluid additives, and drilled solids). The quantity of insoluble suspended solids can be determined by correcting for the volume of soluble solids from salts using the chloride concentration, c_{Cl}, (see 14.4.1), using known volume factors for salt solutions.

9.7.2   Drilling Fluid Solids Volume Fractions (Suspended, Low-gravity, Weighting Material)

9.7.2.1 Additional calculations are required to find the percentage (volume fraction) of suspended solids and relate them to the relative volumes of low-gravity solids and weighting material. To make these calculations, an accurate drilling fluid density and chloride concentration are needed to correct for the soluble solids content of the dried retort solids.

9.7.2.2 Drilling Fluid Solids Volume Fractions (Suspended, Low-gravity, Weighting Material).

The calculations presented in Section 9.4.2 shall be used to determine the drilling fluid solids volume fractions (suspended, solids low-gravity solids, weighting material).

9.7.2.3 Drilling Fluid Brine Volume Fraction

For drilling fluids with high salt contents, it may be useful to monitor the brine volume fraction (corrected water content). The calculation in Section 9.4.3 shall be used to determine the volume fraction of brine.

9.7.3 Drilling Fluid Solids Concentrations (Suspended, Low-gravity, Weighting Material)

The concentration of low-gravity solids, weighting material, and suspended solids shall be calculated using the equations presented in Section 9.4.4.

NOTE   For water-based drilling fluids with added bentonite, additional calculations can be made to determine the relative concentration of bentonite and drill solids components of the low-gravity solids (see API 13I [4]).

10 Sand Content

10.1 Principle

The sand content of drilling fluid is the percentage (volume fraction) of particles of diameter larger than 75 μm (US sieve No. 200). It is measured by a sand-screen test method. In this test, any material retained on the screen is reported as sand content, as defined by the particle size. It may or may not be sand mineralogically. Recent additions of barite and lost circulation materials are often separated and included in this measurement.

10.2 Apparatus

10.2.1 Sieve, with 75 μm (0.0029 in.) openings (US Sieve No. 200), and 63.5 mm (2.5 in.) in diameter.

10.2.2 Funnel, to fit sieve.

10.2.3 Sand content graduated glass tube, marked for the volume of drilling fluid to be added and graduated from 0 % to 20 % in order to read the percentage of sand directly.

10.2.4 Solids-free water or **brine,** compatible with the fluid being measured.

10.3 Procedure

In order to measure sand content in a drilling fluid sample, the following procedure shall be used.

a) Fill the glass measuring tube with drilling fluid to the "drilling fluid" mark. Add solids-free water or brine to the next mark. Close the mouth of the tube and shake vigorously.

b) Wet the screen with solids-free water or brine so that the entire surface of the screen is wetted by the fluid.

c) Pour the well mixed sample from the sand content tube onto the clean, wet sieve. Discard the liquid passing through the sieve. Add more solids-free water or brine to the tube, shake, and again pour onto the sieve. Repeat until the tube is clean. Wash the sand retained on the sieve to free it of any remaining drilling fluid.

   NOTE Do not stir or manually force the mixture through the screen. Tapping the side of the screen holder as the drilling fluid is added can help the mixture pass through the screen.

d) Put the funnel upside down over the top of the sieve. Slowly invert the assembly and insert the tip of the funnel into the mouth of the sand content tube. Wash the sand into the tube by adding a small volume of solids-free water or brine to the sieve or by rinsing the solids with a wash bottle. Allow the sand to settle. From the graduations on the tube, read the volume percent of the sand.

10.4 Reporting

The sand content of the drilling fluid shall be reported as a percentage (volume fraction).

The source of the drilling fluid sample, i.e. possum belly, below shaker, suction pit, etc. shall be reported. All coarse solids >75 microns (0.0029 in.), including particles other than sand will be retained on the screen and shall be reported as sand content in this procedure. It shall be noted as a comment if measurable quantities of barite or lost circulation material are observed in this measurement. There is a significant difference in hardness and erosion potential between barite, quartz, sand and most lost circulation materials.

11 Methylene Blue Capacity of Drilling Fluid

11.1 Principle

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

Methylene blue solution is added to a sample of drilling fluid (which has been treated with hydrogen peroxide and acidified) until saturation is noted by formation of a persistent blue dye ring or halo around a drop of solids suspension placed on filter paper. Variations of the procedure used on the drilling fluid can be

performed on drill solids and commercial bentonite to allow an estimate of the amount of each type of solid present in the fluid as described in API 13I [4].

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

11.2 Reagents and Apparatus

11.2.1 Methylene blue solution, reagent grade methylene blue (CAS No. 61-73-4), 3.20 g ±0.01 g of dry reagent diluted to 1 L [1 mL ≡ 0.01 milliequivalent (meq)].

The moisture content of reagent grade methylene blue shall be determined each time the solution is prepared. Dry a 1.00 g ± 0.01 portion of methylene blue powder to a constant mass at 93 °C ±3 °C (200 °F ±5 °F). Make the appropriate correction in the mass, m_{mb}, of methylene blue powder, expressed in grams, to be taken to prepare the solution as calculated using Equation (26):

$$m_{mb} = \frac{3.2}{m_{dmb}}$$

(26)

where

m_{dmb} is the mass of the dried methylene blue powder sample, expressed in grams;

m_{mb} is corrected mass of methylene blue powder to be used per liter of water, expressed in grams.

11.2.2 Hydrogen peroxide (CAS No. 7722-84-1), 3 % solution.

Warning—H_2O_2 is a strong oxidizer. Skin contact with solutions stronger than 3 % should be avoided.

11.2.3 Sulfuric acid (CAS No. 7664-93-9), dilute, approximately 2.5 mol/L (5N).

Warning—H_2SO_4 is a strong and toxic acid.

11.2.4 Deionized or **distilled water,** shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.

11.2.5 Syringe, 2.5 mL (TD) or 3 mL (TD).

11.2.6 Conical flask (Erlenmeyer), capacity 250 mL.

11.2.7 Burette, 10 mL (TD); micropipette, 0.5 mL (TD); or graduated pipette, 1 mL (TD).

11.2.8 Graduated cylinder, 50 mL (TD).

11.2.9 Stirring rod.

11.2.10 Hot plate.

11.2.11 Filter paper [6], qualitative, standard cellulose, grade 1, medium retention and flow rate, 125 mm (5 in.) circle diameter.

11.3 Procedure

In order to determine the methylene blue capacity, the following procedure shall be used.

a) Add 2.0 mL of drilling fluid as accurately as possible (or suitable volume of drilling fluid to require a minimum of 2 mL methylene blue solution) to 10 mL of deionized water in the Erlenmeyer flask. The syringe used should have a capacity of more than 2 mL, generally 2.5 mL or 3 mL. By using a larger syringe, it is not necessary to remove the air trapped in the syringe. To ensure that exactly 2.0 mL of drilling fluid is being added, use the following procedure:

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

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

   3) Deliver a 2 mL drilling fluid sample volume (V_{df}) by pushing the plunger until the end of the plunger is exactly 2 mL from the last graduation on the syringe (e.g. in a 3 mL syringe, at the 1 mL line).

b) Add 15 mL of 3 % hydrogen peroxide and 0.5 mL of 2.5 mol/L sulfuric acid. Boil gently for 10 min, but do not allow to boil to dryness. Dilute to about 50 mL total volume with deionized water.

c) Add methylene blue solution to the flask in increments of 0.5 mL. If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous testing, larger increments (1 mL to 2 mL) can be used at the beginning of the titration. After each addition of methylene blue solution, swirl the contents of the flask for about 30 s. While the solids are still suspended, using the stirring rod, remove one drop of liquid and place it on the filter paper. The initial endpoint of the titration is reached when unadsorbed dye appears as a blue or turquoise ring or halo surrounding the dyed solids, as shown in Figure 1, Key item 5 (6 mL addition).

d) Whenever a blue tint spreading from the spot is detected, shake/stir the flask for an additional 2 min and place another drop on the filter paper, adjacent to the previous drop. If the blue ring or halo is again evident, the 2 min endpoint has been reached, as shown in Figure 1, Key item 7 (7 mL). If the faint blue ring or halo does not appear, such as in Figure 1, key item 7 for second drop of the 6 mL addition, continue as described in 11.3, item f) until the second drop taken after 2 min still shows the faint blue ring or halo. Free (unadsorbed) dye detected immediately after adding methylene blue solution which is adsorbed after 2 min indicates that the end point has not been reached.

e) After the 2 min end point has been reached, continue stirring an additional 10 min and place another drop on the test paper. If the faint blue ring or halo is again evident, the final end point has been reached. If the faint blue ring or halo or halo does not appear, continue as described in 11.3, item f) and 11.3 , item g) until a drop taken after stirring 2 min and 10 min still shows the faint blue ring or halo, indicating

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[6] This standard cellulose grade 1 filter paper is designed for medium retention and flow. The filter paper has the following typical properties that may vary by manufacturer: medium filtration rate; particle retention in liquid: 11 μm (at 98 % efficiency); ash content: <0.06 % by weight; and thickness: 0.180 mm (0.0071 in.).

unadsorbed excess dye. The 8 mL test is indicative of the end point being exceeded. Volume of methylene blue used to reach the final end point is recorded as V_{mb}.
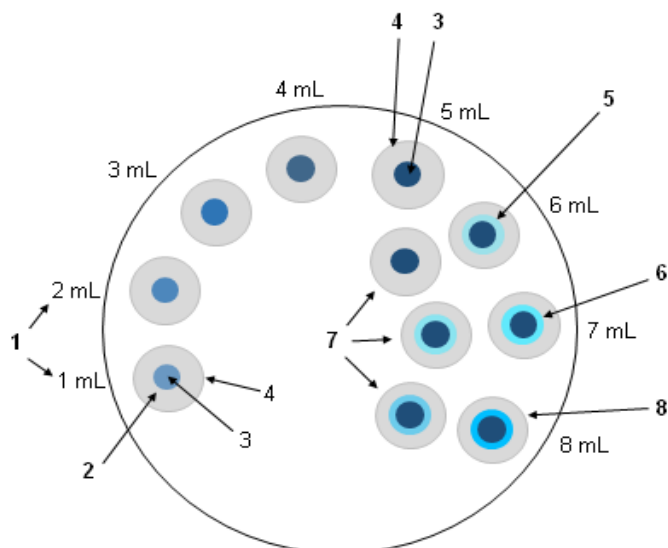
Key

1	volume of methylene blue solution added	5	free unadsorbed dye ring
2	no free unadsorbed dye present	6	endpoint reached initial
3	dyed drilling fluid solids (no free dye present)	7	endpoint retest after additional 2 min
4	moisture, no color	8	endpoint exceeded

NOTE   10 min retest spot not shown.

Figure 1—Spot Tests for Endpoint of Methylene Blue Test

11.4 Calculation

11.4.1 Methylene blue capacity

Report the methylene blue capacity, C_{MBT}, of the drilling fluid, expressed in milliequivalents per milliliter of drilling fluid (meq/mL), which shall be calculated using Equation (27):

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

where

V_{mb} is the volume of methylene blue solution used in titration, expressed in milliliters;

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

11.4.2 Methylene blue capacity expressed as bentonite equivalent

Alternatively, the methylene blue capacity can be reported as bentonite equivalent (based on bentonite with a cation-exchange capacity of 70 meq/100 g), $E_{BE,SI}$, expressed in kilograms per cubic meter, calculated using Equation (28), or $E_{BE,USC}$, expressed in pounds per barrel, calculated using Equation (29):

$$E_{BE,SI} = \frac{14.27 \times V_{mb}}{V_{df}} \tag{28}$$

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

where

V_{mb} is the volume of methylene blue solution used in titration, expressed in milliliters;

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

NOTE   The methylene blue capacity reported as bentonite equivalent, expressed in kilograms per cubic meter from Equation (28) [reported in pounds per barrel from Equation (29)], is not equal to the amount of commercial bentonite in the drilling fluid. Reactive clays in the drill solids contribute to this quantity as well as commercial bentonite. See API 13I [4] for additional information on estimating the amount of commercial bentonite and drill solids present.

12 pH

12.1 Principle

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

The term "pH" denotes the "potential of hydrogen ion" and is the negative logarithm (base 10) of the hydrogen ion activity, a_{H^+}, in aqueous solutions (activity and concentration are equal only in dilute solutions):

$$pH = -\log_{10}\left(a_{H^+}\right)$$

For pure water at 24 °C (75 °F) the hydrogen ion activity, a_{H^+}, is 10^{-7} mol/L and the pH is 7. This system is termed "neutral" because the hydroxyl ion activity a_{OH^-}, is also 10^{-7} mol/L. In aqueous systems at 24 °C (75 °F) the ionic product, [H$^+$] [OH$^-$], is 10^{-14}, a constant. Consequently, an increase in [H$^+$] concentration denotes a like decrease in [OH$^-$] concentration. A change in pH of one unit indicates a 10-fold change in both ions [H$^+$] and [OH$^-$] concentrations. Solutions with pH less than 7 are termed "acidic" and those with pH greater than 7 are termed "basic" or "alkaline".

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

Color-matching pH-paper and sticks are often used for field pH measurements but are not the recommended method. These methods are reliable only in simple water-based drilling fluids. Drilling fluid solids, dissolved salts and chemicals, and dark-colored liquids cause serious errors in pH-paper values. The readability of pH-paper and sticks is normally only about 0.5 pH unit.

Electronic portable and pocket pH-meters are also often used for field pH measurements but are outside the scope of this document. It is recommended to follow the manufacturers' recommendations for maintenance and calibration.

12.2 Reagents and Apparatus

12.2.1 Buffer solutions, to calibrate and set the slope of pH-meter prior to sample measurement at the 4.0, 7.0, and 10.0 pH values.

Buffers may be obtained from supply houses as pre-made solutions, dry-powder packages, or a given formula. Shelf life of buffers should not exceed 6 months before disposal. The date of preparation of the buffer should be shown on bottles used in the field. Bottles should be kept tightly sealed when not in use.

12.2.2 Deionized or **distilled water**, in spray bottle shall be in accordance with ISO 3696 of at least Grade III.**12.2.3 Mild liquid detergent**.

12.2.4 Sodium hydroxide (CAS No. 1310-73-2), 0.1 mol/L (approximately), to recondition the electrode.

Warning—Sodium hydroxide (NaOH) is a strong caustic alkaline chemical. Avoid skin contact.

12.2.5 Hydrochloric acid (CAS No. 7674-01-0), 0.1 mol/L (approximately), to recondition electrode.

Warning—Hydrochloric acid (HCl) is a strong and toxic acid.

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

Warning—Ammonium bifluoride (NH_4HF_2) is toxic and corrosive. Handle accordingly and avoid skin contact.

12.2.7 pH-meter, calibrated to show pH units for measuring pH.

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

— pH range: 0.0 to 14.0;

— electronics type: solid state (preferred);

   NOTE   A double junction probe is preferred.

— power source: batteries (preferred);

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

— readout: digital (preferred);

— resolution: 0.1 pH unit;

— accuracy: ±0.1 pH unit;

— repeatability: 0.1 pH unit.

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

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

— pH electrode response range: 0.0 to 14.0 pH units;

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

 NOTE   To prolong the life of the reference electrode system, use double-junction electrode for measuring liquids avoid liquids containing sulfide or bromide ions.

— electrolyte in reference electrode: KCl gel;

— glass or epoxy composition: suitable for low sodium-ion (Na⁺) error;

— sodium-ion error: at pH 13.0 or at 0.1 mol/L Na⁺ ion, an error less than 0.1 pH unit.

12.2.9   Tissue, soft, to blot electrodes.

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

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

12.2.12   Electrode-storage vial, to keep electrodes moist.

12.3   Procedure for pH Measurement

In order to measure pH for a drilling fluid sample, the following procedure shall be used.

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

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

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

c)   Clean electrodes by washing with deionized water spray or tap water or pH 7.0 buffer solution and blot dry.

d)   Place probe into pH 7.0 buffer solution or tap water.

e) Always perform at least a 2-point calibration within the expected pH range.

f) Most modern pH meters have automatic temperature compensation. If not, measure temperature of pH 7.0 buffer solution. Tables of pH values at various temperatures are available.

g) Most modern pH-meters have automatic pH buffer recognition.

h) Follow the manufacturer's instructions for the calibration procedure. Repeat the procedure 12.3 item c) through item h) until the meter reads both pH buffers accurately.

i) Rinse probe with tap water or pH 7.0 buffer solution and blot dry.

j) Repeat operations in 12.3 item f) to item h) using either pH 4.0 or pH 10.0 buffer. Use pH 4.0 if an "acidic" sample, or pH 10.0 if an "alkaline" sample is to be tested.

k) Check the meter again with pH 7.0 buffer. If it has changed, reset to "7.0." Repeat the calibration procedure. If meter does not calibrate properly, recondition or replace electrodes as per12.4 procedure.

    Discard and do not reuse the sample of buffer solutions used in calibration. Meter should be fully calibrated daily, as per 12.3 b) through 12.3 j), using two buffers. Check with pH 7.0 buffer every 3 h when using the meter continuously and prior to use, if more than 3 h had lapsed between measurements.

l) If meter calibrates properly, rinse electrode with deionized water spray or tap water or pH 7.0 buffer solution and blot dry. Place electrode in sample to be tested and stir gently. Allow the reading to stabilize.

m) Record sample pH to nearest 0.1 pH unit and the temperature of sample.

n) Carefully clean the electrode in preparation for next usage. Store in a vial of pH 4.0 or pH 7.0 buffer solution. Never let the probe tip become dry and never store the electrode in distilled or deionized water.

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

12.4 Care of Electrode

Periodically cleaning the electrode is necessary, especially if oil or clay particles coat the face of the glass electrode or the porous frit of the reference electrode. Procedure to take care of electrode shall be as follows.

a) Clean electrode with a soft-bristle brush and a mild detergent. Rinse clean with deionized water spray or tap water.

b) Reconditioning the electrode can be necessary if plugging becomes severe, as indicated by a slow response, drifting of readings, or if calibration cannot be set.

c) Recondition by soaking electrode for 10 min in 0.1 mol/L HCl, followed by rinsing in deionized water spray or tap water and soaking for 10 min in 0.1 mol/L NaOH and rinsing again. This may temporarily restore the life of an old electrode.

d) Check electrode for response by performing calibration in 12.3.

e) If electrode continues to perform poorly, soak electrode for 2 min only in 10 % ammonium bifluoride solution and immediately rinse with deionized water spray or tap water. Repeat 12.3 item h) to check for calibration capability.

f) Replace electrode system if 12.4 item c) to 12.4 item e) fail to recondition it.

13 Alkalinity and Lime Content

13.1 Principle

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

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

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

13.1.3 The composition of drilling fluid filtrates is often so complex that the interpretation of alkalinities in terms of estimated ionic components can be misleading. Any particular alkalinity value represents the ions that react with the acid in the pH range over which that particular value was tested. Inorganic ions that can contribute to the alkalinity, in addition to the hydroxyl, carbonate and bicarbonate ions, are borates, silicates, sulfides, and phosphates. Perhaps more serious in drilling fluids are anionic organic thinners, filtrate reducers, and their degradation products that can contribute to a large portion of the alkalinity value as well as masking the endpoint color change. These organic materials make a particularly large contribution to the M_f alkalinity and thus render the test highly inaccurate in drilling fluids treated with organic thinners, polyamine shale inhibitors, or any product containing a carboxylic acid functionality. For simple bentonite-base drilling fluid systems, the P_f and M_f alkalinities (see 13.3) can be used as guidelines to determine both the presence of carbonate/bicarbonate contamination and the treatment necessary to alleviate the contamination.

13.2 Reagents and Apparatus

13.2.1 **Sulfuric acid** (CAS No. 7664-93-9) solution, standardized 0.01 mol/L [0.02N (N/50)].

Warning—H$_2$SO$_4$ is a strong and toxic acid.

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

13.2.3   Methyl orange (CAS No. 547-58-0) indicator solution, 0.1 g/100 mL of water.

13.2.4   Deionized or **distilled water,** shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.

13.2.5   pH-meter (optional).

NOTE   A pH-meter is more accurate than an indicator solution and functions equally well in deeply colored filtrate.

13.2.6   Titration vessel, 100 mL or 150 mL, preferably white.

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

13.2.8   Volumetric pipette, 1 mL (TD).

13.2.9   Syringe, 1 mL (TD).

13.2.10 Stirring rod.

13.3   Procedure—Phenolphthalein and Methyl Orange Filtrate Alkalinities

In order to measure alkalinities in a sample of drilling fluid filtrate (from the Section 8.2 Low-temperature Low-pressure Filtrate Test), the following procedure shall be used.

a) Measure 1 mL or more of filtrate into the titration vessel. Add 2 or more drops of the phenolphthalein indicator solution; do not exceed 10 drops. If the indicator turns pink, add 0.01 mol/L [0.02N (N/50)] sulfuric acid, drop by drop from the graduated pipette, while stirring, until the pink color just disappears. If the sample is so colored that the indicator color change is masked, the endpoint can be taken when the pH drops to 8.3 as measured with a pH meter. (Refer to Section 12 for proper pH measurement.)

b) Report the phenolphthalein alkalinity, P_f, of the filtrate as the number of milliliters of 0.01 mol/L [0.02N (N/50)] sulfuric acid required per milliliter of filtrate.

c) To the sample that has been titrated to the P_f endpoint, add 2 drops to 3 drops of methyl orange indicator solution. Add the standard acid drop by drop from the pipette, while stirring, until the color of the indicator changes from yellow to pink. The endpoint can also be taken when the pH of the sample drops to 4.3 as measured by a pH meter.

d) Report the methyl orange alkalinity, M_f, of the filtrate as the total milliliters of 0.01 mol/L [0.02N (N/50)] sulfuric acid per milliliter of filtrate required to reach the methyl orange endpoint (including that amount required for the P_f endpoint).

13.4   Procedure—Phenolphthalein Drilling Fluid (Whole Mud) Alkalinity

In order to determine the whole mud alkalinity of a drilling fluid sample, the following procedure shall be used.

a) Measure 1.0 mL of drilling fluid into the titration vessel using a syringe or volumetric pipette.

b) Dilute the drilling fluid sample with 25 mL to 50 mL of deionized or distilled water.

c) Add 4 drops to 5 drops of phenolphthalein indicator solution and if the indicator turns pink, while stirring, titrate rapidly with 0.01 mol/L [0.02N (N/50)] sulfuric acid solution until the pink color disappears. If the endpoint color change cannot be seen, it can be taken when the pH drops to 8.3 as measured by a pH meter. (Refer to Section 12 for proper pH measurement.)

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

d) Report the phenolphthalein alkalinity, P_{df}, of the drilling fluid as the number of milliliters of 0.01 mol/L [0.02N (N/50)] sulfuric acid required per milliliter of drilling fluid.

NOTE P_{df} Drilling Fluid phenolphthalein alkalinity expressed in milliliters of 0.01 mol/L [0.02N (N/50)] sulfuric acid per milliliter of drilling fluid is commonly known in the industry by the abbreviation Pm.

13.5 Calculation of Ion Concentrations from P_f and M_f

The mass concentrations of hydroxyl, carbonate, and bicarbonate ions can be estimated from P_f and M_f as shown in Table 5.

Table 5—Concentrations of Hydroxyl, Carbonate and Bicarbonate ions

Relative values of P_f and M_f	Concentration mg/L		
	OH⁻	CO₃ ²⁻	HCO₃ ⁻
$P_f = 0$	0	0	$1220\,M_f$
$2\,P_f < M_f$	0	$1200\,P_f$	$1220\,(M_f - 2\,P_f)$
$2\,P_f = M_f$	0	$1200\,P_f$	0
$2\,P_f > M_f$	$340\,(2\,P_f - M_f)$	$1200\,(M_f - P_f)$	0
$P_f = M_f$	$340\,M_f$	0	0

NOTE   This method for estimating the concentrations of hydroxyl, carbonate and bicarbonate ions from P_f and M_f can be highly inaccurate when certain products referenced in 13.1 are used. The Garrett gas train testing method for carbonate and bicarbonate ions described in A.5 should be used.

13.6 Estimation of Lime Content

The P_f and P_{df} of the filtrate and drilling fluid shall be determined as described in 13.3 and 13.4.

The volume fraction, F_W, of water in the drilling fluid using the value for volume fraction, expressed as a decimal fraction, of water from the liquid and solids determination (see Section 9), shall be determined using Equation (30):

$$F_W = \frac{\varphi_W}{100} \tag{30}$$

where

φ_W is the volume fraction, expressed as a percentage, of water in the drilling fluid (see Section 9).

The lime content of the drilling fluid shall be calculated and reported as, $c_{lime,SI}$, expressed in kilograms per cubic meter, using Equation (31), or $c_{lime,USC}$, expressed in pounds per barrel, using Equation (32):

$$c_{lime,SI} = 0.742 \times \left(P_{df} - F_w P_f\right) \tag{31}$$

$$c_{lime,USC} = 0.26 \times \left(P_{df} - F_w P_f\right) \tag{32}$$

where

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

P_{df} is the phenolphthalein alkalinity of the drilling fluid [see 13.4 item d)];

P_f is the phenolphthalein alkalinity of the filtrate [see 13.3 item b)].

14   Chloride Ion Content

14.1   Principle

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

14.2   Reagents and Apparatus

14.2.1   Silver nitrate (CAS No. 7761-88-8) solution, containing 4.791 g/L [0.0282 mol/L (0.0282N), equivalent to 0.001 g/mL chloride ion] or containing 47.910 g/L [0.282 mol/L (0.282N), equivalent to 0.01 g/mL chloride ion], stored in an amber or opaque bottle.

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

Warning—Potassium chromate (K_2CrO_4), the hexavalent chromium is known to be carcinogenic and should be handled with care.

14.2.3   Sulfuric acid (CAS No. 7664-93-9) or **nitric acid** (CAS No. 7697-37-2) solution, 0.01 mol/L [0.02N (N/50)].

Warning—Sulfuric acid (H_2SO_4) and nitric acid (HNO_3) are corrosive and toxic acids.

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

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

14.2.6 Deionized or **distilled water**, shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.

14.2.7 Graduated pipettes, 1 mL (TD) and 10 mL (TD).

14.2.8 Titration vessel, 100 mL or 150 mL, preferably white.

14.2.9 Stirring rod.

14.3 Procedure

In order to measure chloride ions in a sample of drilling fluid filtrate (from the Section 8.2 Low-temperature Low-pressure Filtrate Test), the following procedure shall be used.

a) Measure 1 mL or more of filtrate, V_s into the titration vessel. Add 2 drops to 3 drops of phenolphthalein solution. If the indicator turns pink, add acid drop by drop from pipette, while stirring, until the color has disappeared. Ensure that sufficient acid is added to prevent the pink color from the indicator returning while titrating. If it does, the chloride endpoint will be difficult to detect. If the filtrate was originally deeply colored, add an additional 2 mL of 0.01 mol/L [0.02N (N/50)] sulfuric acid or nitric acid and stir. Then add 1 g calcium carbonate and stir.

b) Add 25 mL to 50 mL of deionized water and 5 drops to 10 drops of potassium chromate solution.

c) Start to titrate with the silver nitrate solution. If the chloride ion concentration is greater than 10,000 mg/L, utilize the 0.282N equivalent to 0.01 g/mL chloride ion solution. If the chloride ion concentration is less than 10,000 mg/L, utilize the 0.0282N equivalent to 0.001 g/mL chloride ion solution.

   Stir continuously, while adding silver nitrate solution drop by drop from the pipette, until the color changes from yellow to orange-red and persists for 30 s. Record the volume of silver nitrate solution, V_{SN1X} (0.0282 N solution) or V_{SN10X} (0.282 N solution), required to reach the endpoint. If over 10 mL of silver nitrate solution is used, repeat the test with a smaller sample of filtrate.

14.4 Calculation

14.4.1 Chloride ion concentration

The chloride ion concentration, c_{Cl}, of the filtrate, expressed in milligrams per liter, shall be calculated using Equation (33) or Equation (34), according to the concentration of silver nitrate solution used for titration:

a) Titration using 0.0282N silver nitrate

$$c_{Cl} = 1000 \times \frac{V_{SN1X}}{V_s} \tag{33}$$

b) Titration using 0.282N silver nitrate

$$c_{Cl} = 10,000 \times \frac{V_{SN10X}}{V_s} \tag{34}$$

where

    V_s is the volume of the filtrate sample, expressed in milliliters;

    V_{SN1X} is the volume of silver nitrate solution 0.0282N, expressed in milliliters;

or,

    V_{SN10X} is the volume of silver nitrate solution 0.282N, expressed in milliliters.

14.4.2 Sodium chloride concentration

To convert c_{Cl} to the sodium chloride concentration Equation (35) (SI units) or, Equation (36) [parts per million (ppm)] shall be used.

a)   Sodium chloride concentration, $c_{NaCl,SI}$, expressed in milligrams per liter

$$c_{NaCl,SI} = 1.65 \times c_{Cl} \tag{35}$$

b)   sodium chloride concentration, $c_{NaCl,ppm}$, expressed in parts per million by mass (ppm);

$$c_{NaCl,ppm} = \frac{c_{NaCl,SI}}{\rho_f} \tag{36}$$

where

    ρ_f is the density of filtrate for sodium chloride, expressed in grams per milliliter, calculated using Equation (18) (see 9.4.2).

NOTE   In dilute solutions, milligrams per liter is approximately equal to parts per million.

15 Total Hardness as Calcium

15.1 Principle

The hardness of water or drilling fluid filtrate is due primarily to the presence of calcium and magnesium ions. When ethylenediaminetetraacetic acid (EDTA) (or its salt) is added to the water or filtrate, it combines with both the calcium and magnesium and the endpoint is determined with a suitable indicator. The total hardness of the water or filtrate is expressed as milligrams calcium per liter. An endpoint obscured by dark components can often be remedied by oxidizing with a reagent such as sodium hypochlorite. The individual concentrations of calcium and magnesium can be determined from titrations in Annex A (see A.1 and A.2).

15.2 Reagents and Apparatus

15.2.1 EDTA solution (CAS No. 6381-92-6), 0.01 mol/L; standardized disodium ethylenediamine tetraacetate dihydrate (1 mL/mL sample equivalent to 1000 mg $CaCO_3$, 1 mL/mL sample equivalent to 400 mg Ca^{+2}).

NOTE   EDTA is distributed with various supplier names, "standard versenate solution" being the most common.

15.2.2 Buffer solution, 67.5 g ammonium chloride (NH_4Cl) (CAS No. 12125-02-9) and 570 mL ammonium hydroxide, 28 % to 30 % $NH_3 \cdot H_2O$ by weight (CAS No. 1336-21-6) diluted to 1000 mL with deionized or distilled water.

15.2.3 Hardness indicator solution, 1 g/L 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid (CAS No. 3147-14-6) (or "calmagite") in deionized or distilled water.

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

Warning—Acetic acid (CH_3COOH) is corrosive. Avoid skin contact.

15.2.5 Masking agent, 1:1:4 volume mixture of triethanolamine (CAS No. 102-71-6): tetraethylenepentamine (CAS No. 112-57-2): deionized or distilled water.

15.2.6 Sodium hypochlorite (CAS No. 7681-52-9) solution, 5.25 % mass fraction in deionized or distilled water.

Warning—Sodium hypochlorite (NaClO) is highly corrosive and can cause burns and damage the eyes.

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

15.2.7 Deionized or **distilled water**, shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.

15.2.8 Titration vessel, 150 mL beaker.

15.2.9 Graduated pipettes, 5 mL (TD) and 10 mL (TD).

15.2.10 Volumetric pipettes, 1 mL (TD), 2 mL (TD),

15.2.11 Graduated cylinder, 50 mL (TC).

15.2.12 Hot plate (required if filtrate is colored).

15.2.13 pH-paper strip.

15.3 Reagent Quality Control (QC)

If distilled water is used this reagent and deionized water QC shall not be required, proceed to titration procedure 15.4.

a) Add 50 mL of deionized water to a 150 mL beaker. Add 2 mL of buffer solution (see 15.2.2) and 2 drops to 6 drops hardness indicator solution (see 15.2.3) until a blue color is achieved. Swirl to mix.

b) A solution with color other than distinct blue indicates contamination of equipment and/or unacceptable deionized water. If contamination is indicated, find and eliminate the source before proceeding to titration procedure 15.4.

15.4   Procedure

In order to measure total hardness as calcium in a sample of drilling fluid filtrate (from the Section 8.2 Low-temperature Low-pressure Filtrate Test), the following procedure shall be used.

a) With a pipette, add 1 mL or more of filtrate into a 150 mL beaker. Record the volume of filtrate sample used in mL as V_s.

   If filtrate is clear, or is only lightly colored, omit next steps 15.4 item b) through 15.4 item e).

b) Add 10 mL sodium hypochlorite solution and mix.

c) Add 1 mL glacial acetic acid and mix.

d) Boil the sample for 5 min. Maintain the sample volume by adding distilled water (or deionized water) as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample (do not use plastic backed pH-indicator strips). If the colored pH-paper is bleached white, continued boiling is required. A sufficiently boiled sample shows a pH of 5.0.

   Warning—Work in an adequately ventilated area as chlorine gas is highly toxic.

e) Cool the sample.

f) Rinse the inside of the beaker with distilled water (or deionized water) and, dilute the sample to 50 mL with distilled or deionized water.

g) With a graduated pipette, add approximately 2 mL buffer solution and swirl to mix.

   NOTE   The presence of soluble iron can interfere with the endpoint determination. If this is suspected, a mixture of 1:1:4 triethanolamine:tetraethylenepentamine:water (see 15.2.5) has proven to be a suitable masking agent. Add 2 mL of the masking agent to each titration.

h) Add sufficient hardness indicator solution, 2 drops to 6 drops, and mix. A wine-red color develops if calcium and/or magnesium is/are present.

i) While stirring, titrate with the EDTA solution to the proper endpoint. Calcium indicators produce a change from red to blue. The endpoint is best described as the point at which additional EDTA produces no further red to blue color change. The titration volume of EDTA, V_{EDTA}, is used in the calculation in 15.5.

15.5 Calculation

The total hardness in the drilling fluid filtrate, calcium plus magnesium ion concentration, c_{Ca+Mg}, expressed in milligrams per liter as calcium, shall be calculated using Equation (37):

$$c_{\mathrm{Ca+Mg}} = 400 \times \frac{V_{\mathrm{EDTA}}}{V_{\mathrm{s}}} \tag{37}$$

where

V_{EDTA} is the total hardness titration volume of EDTA solution, expressed in milliliters;

V_{s} is the original volume of filtrate used [e.g. 1.0 mL from 15.4 a)], expressed in milliliters.

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

16 HTHP Static Filtration Using the Permeability Plugging Apparatus (PPA)

16.1 Principle

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

The permeability plugging apparatus (PPA) shall be operated at pressure differentials and temperatures approximating those prevailing down-hole and permit the use of filtration media chosen to simulate permeable formations. The PPA shall utilize an inverted fluid cell with the pressure applied from the bottom, the filter medium on top and the filtrate collected from the top. A hydraulic pump applies pressure to the cell, which is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

The PPA utilizes ceramic discs as filtration media. Ceramic discs are available with mean pore throat sizes ranging from 10 μm to 120 μm. The PPA uses media more representative of sands which allows evaluation of filtration/bridging agents in drilling fluids.

Filtration characteristics are affected by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The PPA is a modified high-temperature, high-pressure filter press used to evaluate these interactions through ceramic-disc filter media at pressures up to 34,500 kPa (5000 lbf/in.^{2}) and temperatures from ambient to 260°C (500 °F) (refer to manufacturer's specifications for temperature and pressure limits). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

PPA units usually have half the filter area (2258 mm^2) (3.5 in. ^{2}) of the standard API low-temperature low pressure test, thus the PPA filtrate volumes shall be doubled to corrected to a filter area of 4516 mm$_2$ (7.0 in.$_2$) for reporting the results.

The three parameters measured and reported from PPA testing should be:

— the filtrate volume collected in 30 minutes,

— the spurt loss (filtrate volume collected while filter cake is being established, usually during the first minute of testing), and

— the static filtration rate (after the spurt loss).

16.2   Safety Considerations

16.2.1 General

16.2.1.1 The pressure limitation in the use of the PPA depends upon the cell in use. For safety, it is imperative that the operator know the maximum operating pressure of the test apparatus with certainty and that this pressure not be exceeded. If in doubt, contact the manufacturer or use the lowest of the possible limits.

16.2.1.2 Cell damage can be categorized as follows: end-cap bending, end-cap compression, cylinder shear, cylinder shear and cylinder stress, or the use of non-original equipment manufacturer set screws. End-cap bending can be detected visually or by measurement. End-cap bending can be detected visually or by measurement. End-cap compression can be detected by the distortion of the set-screw holes or seats, which become oval rather than round. Caps showing signs of damage shall not be used and should be discarded. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set-screw holes, shall not be used.

In use of the screw end-caps, if any difficulty is experienced with seating the cap into the threads, do not use the cell. Visibly inspect the external and internal threads for contaminants or distortion, and do not use if damaged

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

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

16.2.1.5 These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury, and apparatus damage. These instruments should be used only on ground circuits.

16.2.2 Hydraulic pressurization system

To ensure safe operation, the hydraulic pressurization system shall be checked. Hydraulic pressure shall be released and checked before carrying out the following operations:

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

b)   attempting to remove cell from heating jacket;

c)   moving the PPA;

d)   refilling the hydraulic pump;

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

Warning—After refilling or repairing the hydraulic system, clean up any spilled oil from the equipment, counter, and surrounding area. Oil left on the floor is hazardous. Also, accumulations of spilled oil near the PPA are fire hazards.

16.2.3 Backpressure system

For safe operation of the pneumatic pressurization of the backpressure receiver, the following instructions shall be considered.

a) Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen, or other non-recommended gases. If nitrogen is used, it shall be supplied in an approved nitrogen gas cylinder or the nitrogen supply system. CO_2 is normally supplied in small cartridges pressurized to about 6200 kPa (900 lbf/in.^{2}).

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

Warning—Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for Garrett gas train carbonate analysis for water-based fluids (see Annex A.5).

b) Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

c) Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition, and leaks to be found and corrected. Periodically, test the pressure-relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

d) When pressurizing the backpressure assembly, ensure that the regulator T-screw or knob has been rotated counterclockwise enough to release all pressure. When the pressure has been released, the regulator adjusting T-screw or knob will turn freely. Always open the supply pressure valve first then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the manufacturer's recommended rating or relief valve settings. When relieving backpressure, shut the supply pressure valve, bleed the pressure from the system and loosen the regulator adjusting T-screw or knob fully.

Warning—Leaking pressurization systems, hydraulic or pneumatic, should be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition and leaks should be found and corrected. Periodically test the pressure-relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.

16.2.4 PPA heating system

16.2.4.1 For safe heating, the instructions below shall be considered.

a) Caution should 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.

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

16.2.4.2 For safe electrical operation, instructions below shall be considered.

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

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

16.2.5 PPA cell maintenance

For test-cell maintenance, the user should be aware that the filtration cell is a pressure vessel and should be considered a source of potential danger. The safety precautions listed below shall be followed to ensure safe operation.

a) Cell material should be compatible with the test samples.

b) Do not use cells that show signs of severe pitting or stress cracking.

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

d) For cells that use set screws, use only hardened steel set screws as supplied by the manufacturer. Deformed set screws are unsafe.

e) The operating manual and this procedure should be read by anyone who is unfamiliar with the apparatus, before using it.

16.3   Apparatus—Permeability Plugging Apparatus

16.3.1 PPA cell

16.3.1.1 PPA pressure rating

There are different ratings for cells. Users shall refer to manufacturer's specifications for temperature and pressure limits.

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 "1800 psi" [12,400 kPa (1800 lbf/in.^{2})]. Unless the user can absolutely verify that the cell used is rated at 13,800 kPa (2000 lbf/in.^{2}), the "1800 psi" 12,400 kPa (1800 lbf/in.^{2}) pressure limit should be observed.

Warning—Follow the manufacturer's recommendations concerning maximum temperature, pressure, sample size, maintenance, and operating procedure. Failure to do so can lead to serious injury.

16.3.1.2 The PPA utilizes an inverted fluid cell with the pressure applied from the bottom, the filter medium on top, and the filtrate collected from the top. A hydraulic pump applies pressure to the cell, which is transferred to the drilling fluid sample through a floating piston within the cell. Redundant O-ring seals on the piston prevent the mixing of the hydraulic oil with the sample.

16.3.1.3 The PPA utilizes ceramic discs as filtration media. Ceramic discs are available in various mean pore throat sizes (see Table 6).

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

16.3.1.5 The PPA cell is encased in a thermostatically controlled heating chamber. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F) (check with manufacturer's specifications). The cell temperature can be measured using a thermocouple inserted into the well in the cell wall. The temperature is adjusted by means of a temperature controller. For some apparatus, temperature can be measured using a metal stem thermometer and the temperature is adjusted by means of a knob. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel.

Figure 2 is an example diagram of a permeability plugging apparatus. Different manufacturers have incorporated different design features, so this drawing indicates only the main operational features. For instance, many designs have inlet pressure valves between the pump and the test cell. Refer to the manual provided by the manufacturer for explicit instructions.

16.3.1.6 Rupture discs shall be included in various locations in the pressurization portion for the safety of the operator. The exact location depends on the design consideration of the manufacturer.

16.3.2 Filter Medium

16.3.2.1 Ceramic discs were adopted for use in the PPA as a method to test plugging/bridging materials in drilling fluids. Ceramic discs do not exhibit the normal relationship between pore throat size and permeability as naturally occurring sandstone and have a very narrow pore size distribution. Work conducted to assess bridging characteristics should be conducted based on the larger pore throat sizes and not permeability.

16.3.2.2 For PPA testing, standard ceramic disc thickness should be 6.5 mm (0.25 in.). Thicker discs can be used with properly manufactured cells. A new disc is required for each test. Each ceramic disc shall be soaked in water or brine with a similar composition to the drilling fluid filtrate so that the porous volume of the disc is filled and filtration to the receiver begins immediately. When possible, ceramic discs should be vacuum saturated to -95 kPa [-14 lbf/in.² (-28 in. Hg)] gauge for a minimum of 30 min prior to use.

These API designated ceramic discs are named according to the mean pore throat diameter as determined by mercury intrusion porosimetry [9]. Table 6 lists the commonly available ceramic discs and their mean pore throat diameter. Also shown is the previous industry designation based on misleading air measurements.

NOTE   Mercury porosimetry is not a viable technique for measuring pore throat sizes exceeding 120 μm. Therefore, there is no API designation for filtration media previously sold under the air designated size of 150 μm and 190 μm or larger.

16.3.3 Timer, digital or mechanical, with at least a 30-min interval, accuracy within ±5 seconds per hour

16.3.4 Thermometer, graduated to 260 °C (500 °F), with an accuracy of ±3°C (±5°F).

16.3.5 Graduated cylinder, (TC) with a capacity of 20 mL (0.2 mL graduations), 25 mL (0.2 mL graduations), or 50 mL (0.5 mL graduations).

16.3.6   Field mixer, cup type, to operate at 10,000 r/min to 15,000 r/min.

16.3.7 Stopcock grease.
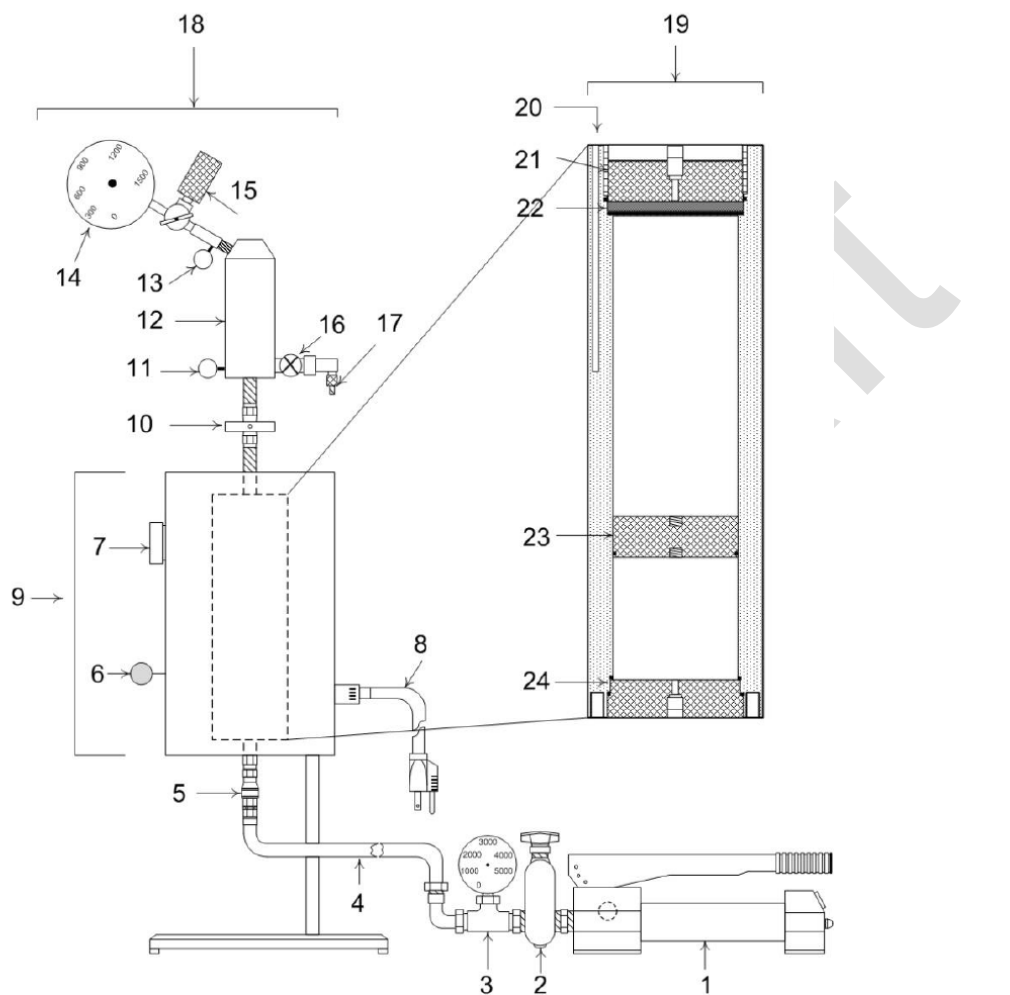
Key

1	Manual hydraulic pump	9	Heating chamber	17	Filtrate collection nozzle
2	Hydraulic fluid overflow (optional)	10	Outlet valve	18	CO_2 pressurizing unit
3	Pressure gauge	11	Locking pin	19	High-pressure cell assembly
4	High pressure hose	12	Backpressure receiver	20	Thermometer well
5	Quick connect coupling	13	Locking pin	21	Top end-cap (outlet end)
6	Cell support-release knob	14	Pressure gauge	22	Ceramic disc
7	Thermostat	15	CO_2 cartridge barrel	23	Floating piston
8	Heater jacket power cord	16	Filtrate collection valve	24	Bottom end-cap (inlet end)

Figure 2—Typical Permeability Plugging Apparatus

Table 6—Ceramic Discs, API Designation and Mean Pore Throat Diameter

API Designation	Mercury Injection Mean Pore Throat Diameter μm	Previous Air Designation μm
API 10	10	3
API 12	12	5
API 20	20	10
API 40	40	20
API 50	50	35
API 55	55	60
API 120	120	90

16.4 Procedure for HTHP Filtration Test using the PPA

16.4.1 Summary

The following procedures shall be used to perform the HTHP filtration test using the PPA. These procedures are described in 16.4.2 through 16.5.11:

— assemble the filtration cell;

— preheat the cell heating jacket;

— load test fluid into the cell;

— pressurize the cell;

— conduct the filtration test; and,

— test conclusion and disassembly.

16.4.2 Assembling the filtration cell

The following procedure shall be used for assembling the filtration cell.

If the cell is completely disassembled and the floating piston has been removed follow 16.4.2 item a) through item j). If the piston is in the cell with hydraulic fluid and with the inlet nipple and quick connect attached, then proceed to 16.4.2 item g to item j).

NOTE   Instructions 16.4.2 item g) to item j) should be performed with the cell placed in an unheated jacket or in a specially constructed stand to keep the cell upright.

Warning—The filtration cell is a pressure vessel.

a) Remove the end-caps from the cell. A cell cap removal tool is available from certain manufacturers for some cells.

b)   Check the O-rings on the valve stems, the piston, the cell body, and the end-caps. Replace any that are damaged or brittle [O-rings should be replaced routinely after each test at temperatures above 150 °C (300 °F)]. Apply a thin coating of stopcock grease completely around the O-rings, being careful to ensure that those on the piston and cell caps are well lubricated.

c)   Screw the floating piston onto the T-bar wrench and install the piston into the inlet end of the cell, working it up and down to ensure that it moves freely (the inlet end, has a shorter recess than the outlet end). Position the piston so that it is at the lip of the O-ring groove in the cell body. Leave the piston T-bar wrench in place.

d)   Fill the inlet space with hydraulic oil just above the O-ring groove or cell shoulder.

e)   Install and tighten the hydraulic-inlet bottom end-cap onto the cell.

Ensure that some oil is expelled from the hole in the end-cap as the cap is inserted, indicating that no air is trapped between the piston and the end-cap.

f)   Install and tighten the inlet nipple and hose quick connect.

g)   Invert the cell so the outlet end is upwards and place in an unheated jacket.

h)   Connect the cell inlet to the pump hose quick connect and follow the manufacturer's recommended procedure to purge all air from the hydraulic system.

i)   Apply pressure to the cell using the hydraulic pump. Using the piston T-handle as a guide, stroke the pump enough so the piston rises 5 cm (2 in.) inside the cell. Remove the piston T-handle.

j)   Disconnect the inlet quick connect and remove cell from heating jacket. Place on a suitable stand.

16.4.3   Preheating the heating jacket

The following procedure shall be used for preheating the heating jacket.

a)   Connect the power cord to the proper voltage as indicated on the nameplate.

b)   Turn the thermostat to mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will illuminate when the heating jacket temperature has reached the thermostat setting. Digital temperature controllers shall be set at the desired test temperature.

c)   After the jacket reaches the desired temperature, readjust the thermostat or temperature controller to obtain a jacket temperature of 6 °C (10 °F) over the desired test temperature.

16.4.4   Loading test fluid into the cell

In order to load the test drilling fluid into the cell, the following procedure shall be used.

Warning—Thermal expansion of the cell contents and of the hydraulic fluid causes the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly to permit the release of hydraulic fluid to prevent over pressurization. During heating, the pressure in the cell should be controlled by bleeding off the fluid periodically.

a)   Drilling fluid sample shall be stirred for 5 min immediately before loading into the PPA cell.

b) With the cell on a suitable stand, fill the cell to the bottom of the outlet O-ring shoulder groove with the test fluid. If the floating piston has been elevated at least 5 cm (2 in.) above the inlet cap face, then any fluid expansion due to heat will force the piston toward the inlet cap, and the ceramic disc will maintain pre-saturation.

c) Install the outlet cell O-ring and set the selected pre-saturated ceramic disc or other filtration media on top of it.

d) To ensure accurate measurements, the space between the filtration medium and the outlet valve should be purged with the base drilling fluid (i.e. water or brine) before starting the test. To fill this space, a small quantity of base fluid (water or brine) is added on top of the ceramic disk immediately before installing the outlet end-cap. This procedure ensures that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

e) With the outlet valve open, position the outlet end-cap in the cell and tighten allowing excess base fluid to be expelled. After tightening, close the valve on the outlet end-cap .

f) Install the cell in the heating jacket. Lower the cell fully into the heating jacket. Rotate the cell so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell.

Warning—Thermal expansion of the cell contents and of the hydraulic fluid causes the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket. When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly, and the inlet valve opened to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell shall be controlled by bleeding off the fluid periodically to maintain the required backpressure from Table 7.

g) Connect the cell inlet to the quick connect.

h) Transfer the thermometer from the heating jacket and place it into the cell body. Immediately pressurize the cell as described below. Monitor the cell temperature with the thermometer in the cell wall, not in the heating jacket.

16.4.5 Pressurization and heating the cell

The following procedure shall be used to pressurize and heat the cell.

a) Apply inlet pressure to the cell using the hydraulic pump immediately after transferring the thermometer to the cell body. This pressure shall equal the pressure that will be applied to the backpressure receiver. (Refer to Table 7 for the pressure corresponding to the test temperature).

Filtration at temperatures above the boiling point of the fluid sample requires the use of pressure to the cell while heating and of the backpressure receiver to prevent vaporization of the filtrate.

NOTE   The differential filtration pressure, is the difference between the inlet pressure applied to the cell and that maintained on the backpressure receiver.

NOTE   For cells with tapered nipple sealing connections, they should be opened during heating and throughout testing.

b) When the cell reaches the desired temperature, adjust the thermostat or temperature controller to maintain the cell at the desired test temperature. Hold the cell at the desired temperature until thermal

expansion is complete, and the cell pressure stops increasing. The test fluid should not be heated more than 1 h prior to filtrate collection. If the test fluid cell temperature has not reached the test temperature after 1 h the heater may be defective, and the validity of the test is questionable.

c) While the cell is heating, use the following procedure to prepare the receiver.

d) Check to ensure that the regulator T-screw or knob has been rotated counterclockwise enough to turn freely. The regulator T-screw will turn freely when no pressure is introduced into the regulator.

e) Verify that the pressure-relief valve on the assembly and the filtrate drain valve are closed.

f) Mount the backpressure receiver on the upper valve adapter. Secure the receiver with a locking pin. Install the pressurizing unit on top of the receiver. Lock the pressurizing unit in place with the other locking pin. Do not adjust the backpressure outlet regulator at this time.

NOTE 1   A single CO_2 cartridge may not reach the maximum pressures in Table 7. If necessary, loosen the regulator T-screw, remove the empty cartridge, and replace with a new one. Tighten the barrel enough to puncture the cartridge.

NOTE 2   If a nitrogen manifold capable of 4900 kPa (700 lbf/in.^{2}) is available, it may be used.

g) If a drain hose is to be used for collecting the filtrate, connect it from the filtrate collection nozzle to the graduated cylinder receiving the filtrate.

h) See Table 7 to determine the appropriate pressure for the backpressure receiver and apply it by turning the pressure adjusting T-screw or knob on the regulator clockwise until the desired pressure is reached.

i) After the test drilling fluid has heated for 1 h, proceed to 16.4.5 item a)

Table 7—Recommended Minimum Cell Heat-up Pressure and Backpressure

Test Temperature		Vapor Pressure Water		Minimum Cell Heat-up Pressure and Backpressure	
°C	°F	kPa	lbf/in.^{2}	kPa	lbf/in.^{2}
<95	<200	<84	<12.1	0	0
95–149	200–299	84–462	12.1–67	700	100
150-175	300-350	462-932	67-135	1104	160
150–189	300–374	462–1269	67–184	1400	200
190–199	375–399	1269–1704	184–247	1900	275
200–219	400–424	1704–2245	247–326	2500	350
220–229	425–449	2245–2912	326–422	3100	450
230–246	450–475	2912–3750	422–545	4000	575
247–260	476–500	3750–4692	545–680	5000	725

16.4.6 Conducting the Filtration Test

The filtration procedure shall be conducted as follows.

a) Begin the filtration by opening the valve between the cell and the backpressure receiver, then apply the desired inlet pressure to the cell using the hydraulic pump. Verify that both the cell pressure, as indicated on the pump gauge, and the backpressure are at desired levels. Adjust them as necessary throughout the test.

b) A laboratory timer should be used, and filtrate is collected after approximately 15 s to ensure that the fluid does not have excessive spurt and exhibits controlled filtration. Collect filtrate and record the volume at 1 min (V_1), 7.5 min ($V_{7.5}$), and 30 min (V_{30}).

   Plotting the data against the square root of time provides a useful method to determine spurt loss, see 16.5.3. If desired, samples can be collected more frequently. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

c) Cell pressure can decrease slightly as filtration and any leakage at the pump reduces the cell contents. The pump is used as necessary to maintain the inlet pressure at the desired level.

d) After each time interval, the filtrate is drained into the graduated cylinder from the backpressure receiver and the time and cumulative volume are recorded.

   It is recommended to recover the filtrate directly from the backpressure receiver, not from a drain hose attached to it. If a hose is used, its length shall be minimized to reduce the error caused by liquid retention on its internal surface.

e) After 30 min, close the valve between the cell and the backpressure receiver and drain any remaining filtrate from the backpressure receiver into the graduated cylinder by opening the receiver outlet valve.

16.4.7 Test Conclusion and Disassembly

For test conclusion and disassembling the filtration cell, the following procedure shall be used.

a) Disconnect the heating jacket from the power source.

The pressurized cell assembly should be allowed to cool in the heating jacket.

Warning—Extreme care should be exercised in cooling hot cells.

b) Turn the T-screw or the knob on the backpressure regulator counterclockwise until it turns freely.

c) Bleed the pressure from the backpressure receiver by opening the pressure relief valve.

d) Open the drain valve and collect the last few drops of filtrate into the graduated cylinder. After removing the locking pin, remove the backpressure assembly from the cell.

e) Because of the design of the backpressure receiver, it may still have some trapped filtrate inside. With the backpressure receiver drain or inlet over the graduated cylinder, gently rock it side-to-side to remove any remaining filtrate.

f) Record the total volume of the filtrate in the graduated cylinder as V_{30}.

g) The temperature of the sample in the cell should be reduced to below 38 °C (100 °F) to ensure that the cell can safely be opened. Once the cell has cooled, release the pressure in the cell by opening the hydraulic pump valve. Disconnect the inlet quick coupler between the pump and the bottom of the cell.

   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.

h) Only open the cell when the user is confident that the contents are no longer under pressure. If pressure is suspected to remain in the cell, consult the manufacturers' procedure for pressure release process. Trapped pressure is usually indicated by difficulty in removing either end-caps.

i) Remove the outlet end-cap with the cell in the vertical position and the outlet or filtration end facing upwards.

j) To remove the filter disc, pry the edge of the disc up, then remove the disc and the filter cake. If required, gently wipe, or rinse the filter cake lightly with water. Then measure and record its thickness and remark on its composition and texture.

k) Pour the remaining fluid from the cell. Wash the inside of the cell. It is usually not necessary to remove the floating piston and the bottom inlet end-cap unless the last test was run at 150 °C (300 °F) or higher.

l) If testing was conducted at temperatures above 150 °C (300 °F), the O-rings should be replaced, which will require removal of the inlet cell cap and floating piston.

m) Perform the following three steps to replace the O-rings on the floating piston and the inlet end-cap.

   1. Remove the inlet end-cap using the manufacturer's procedure.

   2. 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. The floating piston can be removed through the top end without the removal of the bottom end-cap. Remove and dispose of the O-rings on the floating piston and the inlet end-cap.

   3. Clean the parts for reuse.

16.5   PPA Test Report

16.5.1 Filtrate reporting

Report the actual cumulative filtrate volume, in milliliters, collected through each of the selected time periods. The filtrate volumes collected are designated with a subscript indicating the time of the sample, i.e. V_{30} for 30 min volume.

Reported PPA filtrate volumes shall be doubled to corrected to a filter area of 4516 mm^2 (7.0 in.^{2}) for reporting the results since PPA units usually have half the filter area (2258 mm^2) (3.5 in.^{2}) of the standard API low- temperature low-pressure test.

16.5.2 Spurt loss

The spurt loss, V_{sl}, expressed in milliliters, can be determined from the intercept, on the *y*-axis, of the straight line drawn through the collected filtration volumes plotted against the square root of filtration time on the *x*-axis for data collected after one minute. This *y*-axis intercept value is then doubled to correct for filtration area when using 2258 mm^2 (3.5 in.^{2}) filtration media and shall be reported as the spurt loss. The preferred spurt loss value is the calculated value using Equation (38).

The graphical estimated spurt loss method is to collect and record the filtrate volumes frequently as described 16.4.6 item b), plot the data on the *y*-axis versus the square root of time on the *x*-axis, then insert a linear trendline regression or draw a straight line through the data to obtain the y-axis intercept. Then double this value to get the corrected estimated spurt loss (V_{sl}).

16.5.3 Calculation

The permeability-plugging test volume, V_{PPT}, expressed in milliliters, equal to two times the V_{30}, the filtrate volume after 30 min, expressed in milliliters, shall be calculated using Equation (38):

$$V_{PPT} = 2 \times V_{30} \tag{38}$$

The spurt loss, V_{sl}, expressed in milliliters, should be calculated using Equation (39):

$$V_{sl} = 2\left[V_{7.5} - \left(V_{30} - V_{7.5}\right)\right] = 2\left(2V_{7.5} - V_{30}\right) \tag{39}$$

where

$V_{7.5}$ is the filtrate volume after 7.5 min, expressed in milliliters;

V_{30} is the filtrate volume after 30 min, expressed in milliliters.

The static filtration rate (velocity of flow), v_{sf}, expressed in milliliters per square root of minute, shall be calculated using Equation (40).

$$v_{sf} = \frac{2\left(V_{30} - V_{7.5}\right)}{\sqrt{t_2} - \sqrt{t_1}} = \frac{2\left(V_{30} - V_{7.5}\right)}{2.739} \tag{40}$$

where

t_1 is the time at initial reading, expressed in minutes, i.e. 7.5 min;

t_2 is the time at final reading, expressed in minutes, i.e. 30 min;

$V_{7.5}$ is the filtrate volume after 7.5 min, expressed in milliliters;

V_{30} is the filtrate volume after 30 min, expressed in milliliters.

All three parameters, V_{PPT}, V_{sl}, and v_{sf}, shall be calculated and reported on the basis of filtrate volume corrected for filtration area to the 4516 mm^2 (7.0 in^2) area standard. The filter media routinely used in these tests have half the filtration area of that used in the standard low-pressure filtration test. Doubling the filtrate volume compensates for this area difference.

16.5.4 Filter Cake Reporting

Filter cake thickness shall be measured and recorded to the nearest 1.0 mm (1/32 in.). A description of the filter cake should be included, such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgments, the description does convey important information.

Annex A
(normative)

Additional Chemical Analysis of Water-based Drilling Fluids

A.1   Calcium

A.1.1   Principle

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

A.1.2   Reagents and Apparatus

A.1.2.1   EDTA (CAS No. 6381-92-6), 0.01 mol/L solution, standardized disodium ethylenediamine tetraacetate dihydrate (1 mL/mL sample equivalent to 1000 mg $CaCO_3$, 1 mL/mL sample equivalent to 400 mg Ca^{+2}).

NOTE   EDTA is distributed with various supplier names, "standard Versenate solution" being the most common.

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

Warning—NaOH is a strong caustic chemical. Avoid skin contact.

A.1.2.3   Calcium indicator, hydroxynaphthol blue (CAS No. 63451-35-4).

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

Warning—Acetic acid (CH_3COOH) is corrosive. Avoid skin contact.

A.1.2.5   Sodium hypochlorite (CAS No. 7861-52-9) solution, 5.25 % mass fraction in deionized or distilled water.

Warning—Sodium hypochlorite (NaClO) is highly corrosive and can cause burns and damage the eyes. Avoid skin contact.

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

A.1.2.6   Masking agent, 1:1:4 volume mixture of triethanolamine (CAS No. 102-71-6):tetraethylenepentamine (CAS No. 112-57-2):water.

A.1.2.7   Methyl orange (CAS No. 547-58-0) indicator solution, 0.1 g/100 mL of water.

A.1.2.8   Deionized or **distillation water,** shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.

A.1.2.9   Titration vessel, 150 mL beaker.

A.1.2.10   Volumetric pipettes, 1 mL (TD), 2 mL (TD).

A.1.2.11   Graduated pipettes, 1 mL (TD) and 10 mL (TD).

A.1.2.12   Graduated cylinder, 50 mL (TC).

A.1.2.13   Hot plate (required if filtrate is colored).

A.1.2.14   pH-paper strip.

A.1.3   Reagent Quality Control

If distilled water is used, this reagent and deionized water QC shall not be required, proceed to titration procedure A.1.4.

Reagent and deionized water QC shall be proceeded as per 15.4**:**

a)   Reagent QC hardness indicator solution, 1 g/L 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid (CAS No. 3147-14-6) (or "calmagite") in deionized or distilled water (see 15.2.3).

b)   Deionized Water QC Procedure**:**

1)   Add 50 mL of deionized water to a 150 mL beaker. Add 2 mL of buffer solution (see A.1.2.2) and 2 drops to 6 drops hardness indicator solution until a blue color is achieved. Swirl to mix.

2)   A solution with color other than distinct blue indicates contamination of equipment and/or unacceptable deionized water. If contamination is indicated, find and eliminate the source before proceeding to titration procedure A.1.4.

A.1.4   Procedure

In order to conduct a test for calcium in a sample of drilling fluid filtrate where magnesium is also present, the following procedure shall be used. Obtain a drilling fluid filtrate sample using Section 8.2 (Low-temperature Low-pressure Filtration Test).

a)   Using a pipette, add 1 mL or more of filtrate into a 150 mL beaker. Record the volume of filtrate sample used in mL as V_s.

If filtrate is clear, or is only slightly colored, omit A.1.4 item b) through item e).

b)   Add 10 mL sodium hypochlorite solution and mix.

c)   Add 1 mL glacial acetic acid and mix.

d)   Boil the sample for 5 min. Maintain the sample by adding distilled water (or deionized water) as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified

by immersing a strip of pH paper in the sample (do not use plastic backed pH-indicator strips). If the paper is bleached white, continued boiling is required. A sufficiently boiled sample shows a pH of 5.0.

Warning—Work in an adequately ventilated area as chlorine gas is highly toxic.

e) Cool the sample.

f) Rinse the inside of the beaker with distilled water (or deionized water) and dilute the sample to 50 mL with distilled water (or deionized water).

g) With a graduated pipette, add 2 mL of calcium buffer solution or sufficient sodium hydroxide to produce a pH value of 12 to 13.

   NOTE 1   If instructions A.1.4 item b) through item e) were performed it may be necessary to use as much as 10 mL to 15 mL of calcium buffer solution to neutralize the glacial acetic acid and produce a pH value of 12 to 13.

   NOTE 2   The presence of soluble iron can interfere with the endpoint determination. If soluble iron is suspected, add 2 mL of a suitable masking agent after A.1.4 item g). A mixture of triethanolamine: tetraethylenepentamine:water (1:1:4 by volume) is a suitable masking agent.

h) Add sufficient calcium indicator 0.1 g to 0.2 g (see A.1.2.3) and mix. A pink to wine-red color develops if calcium is present. The addition of too much indicator may obscure the endpoint.

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

i) While stirring, titrate with the EDTA solution to the proper endpoint. Calcium indicators produce a change from red to blue. The endpoint is best described as that point where additional EDTA produces no further red to blue color change. The titration volume of EDTA, $V_{\text{EDTA-f}}$, is used in the calculation in A.1.5.

A.1.5   Calculation

The calcium ion concentration in drilling fluid filtrate, c_{Ca}, expressed in milligrams per liter, shall be calculated using Equation (A.1):

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

where

$V_{\text{EDTA-f}}$ is the calcium titration volume of EDTA solution, expressed in milliliters (1 mL ≡ 400 mg Ca^{+2});

V_{s} is the sample volume of the filtrate used [e.g. 1.0 mL from A.1.4 item a)], expressed in milliliters.

A.2   Magnesium

A.2.1   Principle

The magnesium content of the drilling fluid filtrate can be calculated by subtracting the calcium ion content from the total hardness. This gives the magnesium content in terms of calcium that is converted to magnesium by multiplying the value by the ratio of atomic weights (24.3/40.1 = 0.606).

A.2.2   Procedure

To determine the magnesium ion concentration in the drilling fluid filtrate, the procedure shall be as follows.

a)   Determine the total hardness as calcium ion, c_{Ca+Mg}, as described in Section 15.

b)   Determine the calcium ion content, c_{Ca}, as described Section A.1.

A.2.3   Calculation

The magnesium ion concentration in the drilling fluid filtrate, c_{Mg}, expressed in milligrams per liter, shall be calculated using Equation (A.2):

$$c_{Mg} = 0.606 \times \left(c_{Ca+Mg} - c_{Ca} \right) \tag{A.2}$$

where

c_{Ca+Mg} is the total hardness (see Section 15), expressed as calcium in milligrams per liter;

c_{Ca} is the calcium ion concentration (see A.1), expressed in milligrams per liter.

A.3   Calcium Sulfate

A.3.1   Principle

The calcium sulfate content of drilling fluid can be determined by using the EDTA method as described in A.1 to determine the total calcium in the drilling fluid filtrate and in the whole drilling fluid. Total calcium in the whole drilling fluid is determined after a dilution of the drilling fluid with water to solubilize any insoluble (excess) calcium sulfate. Then the total and undissolved calcium sulfate contents of the drilling fluid can be calculated.

A.3.2   Reagents and Apparatus

A.3.2.1   EDTA (CAS No. 6381-92-6), 0.01 mol/L solution, standardized disodium ethylenediamine tetraacetate dihydrate (1 mL/mL sample equivalent to 1000 mg $CaCO_3$, 1 mL/mL sample equivalent to 400 mg Ca^{2+}).

NOTE   EDTA is distributed with various supplier names, "standard versenate solution" being the most common.

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

Warning—NaOH is a strong caustic chemical. Avoid skin contact.

A.3.2.3   Calcium indicator, hydroxynaphthol blue (CAS No. 63451-35-4).

A.3.2.4 Masking agent, 1:1:4 volume mixture of triethanolamine (CAS No. 102-71-6):tetraethylenepentamine (CAS No. 112-57-2):water.

A.3.2.5 Methyl orange (CAS No. 547-58-0) indicator solution, 0.1 g/100 mL of water

A.3.2.6 Deionized or **distilled water,** shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.

A.3.2.7   Titration vessel, 150 mL beaker.

A.3.2.8   Graduated pipettes, 1 mL (TD) and 10 mL (TD).

A.3.2.9 Volumetric pipettes, 5 mL (TD), 10 mL (TD).

A.3.2.10   Beaker , 500 mL (TC).

A.3.2.11 Graduated cylinder, 50 mL (TC).

A.3.2.12 Drilling fluid retort, as described in Section 9.

A.3.3   Reagent Quality Control

If distilled water is used this reagent and deionized water QC shall not be required, proceed to titration procedure A.3.4.

a)   Reagent and deionized water QC shall be proceeded as per 15.4:Reagent QC hardness indicator solution, 1 g/L 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid (CAS No. 3147-14-6) (or "calmagite") in deionized or distilled water (see 15.2.3).

b)   Deionized Water QC procedure:

1)   Add 50 mL of deionized water to a 150 mL beaker. Add 2 mL of buffer solution (see A.3.2.2) and 2 drops to 6 drops hardness indicator until a blue color is achieved. Swirl to mix.

2)   A solution with color other than distinct blue indicates contamination of equipment and/or unacceptable deionized water. If contamination is indicated, find and eliminate the source before proceeding to titration procedure A.3.4.

A.3.4 Procedure

To determine the soluble and insoluble (excess) calcium sulfate content of a drilling fluid sample, the following procedure shall be used.

a)   Add 5 mL of whole drilling fluid to 245 mL distilled water (or deionized water). Stir the mixture for 15 min and filter through a standard filter press in accordance with 8.2. (low-pressure, low-temperature filtration test)

NOTE   Collect only clear filtrate.

b) Add 10 mL of the clear filtrate with the 10 mL volumetric pipette into a 150 mL beaker and titrate to the EDTA endpoint as described in A.1 using A.1.4 item g) through A.1.4 item i) . Record this titration volume of EDTA as $V_{\text{EDTA-df}}$.

c) Determine the volume fraction of water, F_{W}, expressed as a decimal, in the drilling fluid by using the value for volume fraction, φ_{W}, expressed as a percent, of water from the retorting liquid and solids determination (see Section 9), using Equation (A.3):

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

A.3.5 Calculation

A.3.5.1 The total (soluble and insoluble) calcium sulfate concentration of the drilling fluid, $c_{\text{CaSO}_4,\text{SI}}$, expressed in kilograms per cubic meter, or $c_{\text{CaSO}_4,\text{USC}}$, expressed in pounds per barrel, shall be calculated respectively using Equation (A.4) or Equation (A.5)].

$$c_{\text{CaSO}_4,\text{SI}} = 6.80 \times V_{\text{EDTA-df}} \tag{A.4}$$

$$c_{\text{CaSO}_4,\text{USC}} = 2.38 \times V_{\text{EDTA-df}} \tag{A.5}$$

where

   $V_{\text{EDTA-df}}$ is the calcium titration volume of EDTA solution of whole drilling fluid sample [see A.3.3 item b)], expressed in milliliters.

NOTE Titration volume for 10 mL filtrate sample of a whole drilling fluid sample diluted at 2 % [see A.3.3 item a)].

A.3.5.2 The (excess) undissolved calcium sulfate concentration of the drilling fluid, $c_{\text{ex-CaSO4,SI}}$, expressed in kilograms per cubic meter, or $c_{\text{ex-CaSO4,USC}}$, expressed in pounds per barrel, shall be calculated respectively using Equation (A.6) or Equation (A.7)]:

$$c_{\text{ex-CaSO}_4,\text{SI}} = 6.80 \times V_{\text{EDTA-df}} - 1.36 \times V_{\text{EDTA-f}} \times F_{\text{W}} \tag{A.6}$$

$$c_{\text{ex-CaSO}_4,\text{USC}} = 2.38 \times V_{\text{EDTA-df}} - 0.477 \times V_{\text{EDTA-f}} \times F_{\text{W}} \tag{A.7}$$

where

   F_{W} is the volume fraction of water in the drilling fluid, expressed as a decimal;

   $V_{\text{EDTA-df}}$ is the calcium titration volume of EDTA solution of whole drilling fluid sample [see A.3.4 item b)];

         NOTE Titration volume for 10 mL filtrate sample of a whole drilling fluid sample diluted at 2% [see A.3.3 item a) 5/250] .

$V_{\text{EDTA-f}}$ is the calcium titration volume of EDTA solution of the drilling fluid filtrate [filtrate sample 1 mL, see A.3.4 item c)].

A.4   Sulfide

A.4.1   Principle

The concentration of soluble sulfides present in a drilling fluid filtrate can be determined by this method. Soluble sulfides include H_2S and the sulfide (S^{-2}) and bisulfide (HS^-) ions. In this procedure, the drilling fluid filtrate is acidified in a Garrett gas train, converting sulfides to H_2S, which then evolved by bubbling an inert carrier gas through the sample. The gas train thereby separates the gas from the liquid. The separated gas stream is then passed through an H_2S gas indicator tube such as Dräger tube [7] or equivalent, which responds to H_2S by darkening along its length. The darkened length is proportional to the total sulfide in the drilling fluid filtrate. The low-range Dräger tube or its equivalent turns from white to brownish-black and the high-range Dräger tube or its equivalent turns from pale blue to jet-black. No known common drilling fluid contaminants cause these color changes.

Although lead-acetate paper disks can be accommodated in the Garrett gas train to determine the presence or absence of sulfide, (the presence of sulfide is indicated by darkening of the lead-acetate paper), a fresh filtrate sample and a Dräger tube or equivalent shall be used for quantitative analysis.

A.4.2   Reagents and Apparatus

A.4.2.1   **Sulfuric acid** (CAS No. 7664-93-9), approximately 2.5 mol/L (5N), ACS reagent grade.

Warning—H_2SO_4 is a strong and toxic acid.

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

A.4.2.3   **Deionized** or **distilled water**, shall be in accordance with ISO 3696 of at least Grade III.

A.4.2.4   **Carrier gas**, inert to hydrogen sulfide, acid and gas detection Dräger tube reagents.

Nitrogen shall be preferred, but carbon dioxide should be acceptable. (Avoid air or other oxygen-containing gases, such as nitrous oxide.)

A.4.2.5   **H_2S gas detection tubes**, Dräger H_2S analysis tubes or equivalent.

— Low range: marked H_2S 100/a (No. CH 29101, 100 mg/L to 2000 mg/L H_2S).

— High range: marked H_2S 0.2 %/A (No. CH 28101, 0.2 volume % to 7 volume % H_2S).

A.4.2.6   **Garrett gas train apparatus**, consisting of a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flow meter and a Dräger tube.

Specifications of the Garrett gas train:

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[7] Dräger tube is an example of a suitable product available commercially. This information is given for the convenience of users of this part of API 13B-1 and does not constitute an endorsement by API of this product.

a) Body:

— Chamber 1:

Depth   90 mm (3.54 in.);

Diameter   38 mm (1.52 in.).

— Chambers 2 and 3:

Depth   90 mm (3.54 in.);

Diameter   30 mm (1.18 in.).

— Passages between chambers:

Diameter   2.0 mm (0.08 in.).

— Material: Transparent material or glass that is inert to acid, sulfides and hydrogen sulfide gas.

b) Dispersion tube:

— Stem:

Diameter   8.0 mm (0.315 in.);

Length   150 mm (5.9 in.).

— Dispersion frit (bell-shaped, fine):

Diameter   30 mm (1.18 in.).

— Material: Low coefficient of expansion, heat-resistant glass.

c) Flow meter, floating-ball type preferred, capable of measuring a flow rate of approximately 300 mL/min of a carrier gas.

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

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

f) Rubber septum.

A.4.2.7   Lead-acetate paper disk [see A.4.3 item q)].

A.4.2.8   Hypodermic syringes, (TD) 10 mL and 2.5 mL (for acid), and 5 mL and 10 mL (for sample).

A.4.2.9   Hypodermic needles, 40 mm (1.5 in.) 21-gauge needles.

A.4.3 Procedure

In order to determine soluble sulfide levels in a drilling fluid sample with a Garrett gas train, the following procedure shall be used.

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

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

c) Add 20 mL of deionized water to chamber 1.

d) Add 5 drops of defoamer to chamber 1.

e) See Table A.1 for sample volume and type of gas detection Dräger tube required for the expected sulfide range. Select the proper type Dräger tube or equivalent. Break the tip from each end of the tube.

f) Install the gas detection Dräger tube (or equivalent) with the arrow pointing downward into the bored receptacle. Likewise, install the flow-meter tube with the word "TOP" upward. Ensure that the O-rings seal around the body of each tube.

g) Install the top on the gas train and hand-tighten the screws evenly to seal the O-rings.

h) With the regulator T-Screw backed off to prevent gas flow, connect the carrier gas to the dispersion tube of chamber 1 using flexible tubing. If a CO_2 cartridge is used, install and puncture the cartridge and connect it to the dispersion tube.

i) Attach the flexible tubing from the outlet of chamber 3 to the gas detection Dräger tube or equivalent.

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

j) Adjust the dispersion tube in chamber 1 to approximately 6 mm (0.25 in.) above the bottom.

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

l) Collect a sufficient volume of fresh, solids-free API low-pressure/low-temperature filtrate (see 8.2) filtrate for analysis. (If a low concentration of soluble sulfides is to be detected, a large volume of filtrate is required. Use Table A.1 as a guide.)

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

n) Slowly inject 10 mL sulfuric acid solution into chamber 1 through the rubber septum using the hypodermic syringe and needle.

o) Immediately restart the carrier gas flow. The flow rate shall be maintained between 200 mL/min and 400 mL/min.

   NOTE   A typical CO_2 cartridge provides about 15 min to 20 min of flow at this rate.

p) Continuously observe changes in appearance of the gas detection Dräger tube. Record the maximum darkened length, l_{st}, in units marked on the tube, before the front starts to smear. Although the front can attain a diffuse and feathery coloration, continue flowing for a total of 15 min. In the high-range tube, an orange color (caused by SO_2) can appear ahead of the black front if sulfites are present in the sample. The orange SO_2 region shall be ignored when recording the darkened length.

q) For best gas detection tube accuracy, the "darkened length" shall fill more than half the tube's length, but less than 90 % of the total length, therefore the filtrate "sample volume" and gas detection tube combination shall be carefully selected. If the stain length is outside the 50 % to 90 % range, rerun the test using an appropriate sample volume. If stain length is less than 50 % increase the volume of filtrate used. If greater than 90 % use a smaller volume of filtrate.

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

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

Table A.1—Dräger Tube (or Equivalent) Identification, Sample Volume, and Tube Factors for Various Sulfide Ranges

Sulfide Range c_S mg/L	Sample Volume V_s mL	Dräger Tube Identification [a]	Tube Factor [b,c] f_S
1.2 to 24	10.0	H_2S 100/a	0.12 [d]
2.4 to 48	5.0	H_2S 100/a	0.12 [d]
4.8 to 96	2.5	H_2S 100/a	0.12 [d]
30 to 1050	10.0	H_2S 0.2 %/a	1500 [e]
60 to 2100	5.0	H_2S 0.2 %/a	1500 [e]
120 to 4200	2.5	H_2S 0.2 %/a	1500 [e]

[a] See tube body.

[b] Used in calculation.

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

[d] Tube factor 0.12 applies to tubes marked H_2S 100/a (Cat. No. CH 29101) with 100 mg/L to 2000 mg/L scale. For older tubes with the 1 to 20 scale, use a tube factor of 12.

[e] Tube factor of 1450 applies to tubes marked H_2S 0.2 %/a (Cat. No. CH 28101) with 0.2 volume % to 7.0 volume % scale. For older tubes with the 1 mL to 17 mL scale, use a tube factor 600 times the ratio: batch factor/0.40.

A.4.4   Calculation

The sulfide concentration, c_S, expressed in milligrams per liter, in the filtrate sample can be calculated using Equation (A.8):

$$c_S = \frac{l_{st} \times f_S}{V_s} \tag{A.8}$$

where

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

V_s is the sample volume, expressed in milliliters;

f_S is the tube factor for H_2S gas detection tube, from Table A.1.

A.5   Carbonate

A.5.1   Principle

The concentration of soluble carbonates in a drilling fluid filtrate can be determined by this method. Total soluble carbonates include CO_2 and the carbonate (CO_3^{2-}) and bicarbonate (HCO_3^-) ions. Drilling fluid filtrate is acidified in a Garrett gas train, converting all carbonates to CO_2, which then evolved by bubbling an inert carrier gas through the sample. The gas train separates the gas from the liquid. The gas stream is collected in a 1 L gas bag (to allow CO_2 to mix uniformly) and subsequently drawn through a CO_2 gas indicator tube such as Dräger tube [8] or equivalent at a fixed flowrate. The Dräger tube or equivalent responds to CO_2 by progressively staining purple along its length. A reaction between CO_2 and a hydrazine chemical causes a crystal violet indicator to turn purple. The stain length is proportional to the total carbonate concentration in the filtrate.

A.5.2   Reagents and Apparatus

A.5.2.1   Garrett gas train apparatus, consisting of a transparent plastic gas train, an inert gas supply, and pressure regulator, a floating-ball flow meter and a gas detection Dräger tube or equivalent (see A.4.2.6).

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

NOTE 1   Nitrogen is preferred over N_2O as the carrier gas because N_2O cools upon expansion and chills the diaphragm in the regulator; prolonged N_2O flow causes the regulator to perform erratically.

NOTE 2   Use nitrous oxide cartridges only for Garrett gas train carbonate analysis; do not use for sulfide determination.

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[8] Dräger tube is an example of a suitable product available commercially. This information is given for the convenience of users of this part of API 13B-1 and does not constitute an endorsement by API of this product.

Warning—Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under high temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials.

A.5.2.3 CO_2 gas detection tubes, Dräger CO_2 analysis tube, marked CO_2 100/a (Cat. No. 8101811), 100 mg/L to 3000 mg/L, or equivalent.

A.5.2.4   Gas bag 1 L collector, Dräger Alcotest gas bag, No. 7626425, or equivalent.

A.5.2.5   Hand-operated bellows gas-detector pump, Dräger or equivalent.

A.5.2.6   Stopcock, 2-way bore, 8 mm (0.315 in.) glass with polytetrafluoroethylene (PTFE) plug.

A.5.2.7   Sulfuric acid (CAS No. 7664-93-9), approximately 2.5 mol/L (5N), ACS reagent grade.

Warning—H_2SO_4 is a strong and toxic acid.

A.5.2.8   Defoamer, in a dropper bottle.

A.5.2.9   Deionized or **distilled water**, shall be in accordance with ISO 3696 of at least Grade III.

A.5.2.10   Hypodermic syringes, (TD) 1.0 mL, 5 mL, and 10 mL (for acid) and 10 mL (for sample).

A.5.2.11   Hypodermic needles, 40 mm (1.5 in.) 21-gauge needles.

A.5.3   Procedure

To conduct a test for soluble carbonates in a drilling fluid sample using a Garrett gas train, the following procedure shall be used.

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

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

b)   Add 20 mL of deionized water to chamber 1.

c)   Add 5 drops of defoamer to chamber 1.

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

e)   Adjust the dispersion tube to approximately 6 mm (0.25 in.) off bottom.

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

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

h)   Fully collapse the gas bag and simultaneously check the system for leaks. To do this, connect the gas bag and stopcock to the hand pump. Use a discarded gas detection Dräger tube or equivalent as connection and start with the bag essentially empty. Fully depress and release the hand pump. When the bag is completely empty and free of leaks, the pump will remain depressed for several minutes. If leakage is

detected, check the pump and all connections. To check the pump alone, insert a sealed gas detection Dräger tube into the pump opening and depress the bellows. It will remain depressed if pump does not leak.

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

j) Inject and record a measured sample volume, V_s, of solids-free, API low-pressure/low-temperature filtrate (see 8.2) into chamber 1 through the rubber septum with a hypodermic syringe and needle. See Table A.2 for required volume.

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

Warning—Avoid transfer of fluid from chamber 1 to chamber 2 while shaking to avoid injury or death.

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

m) Break the tip off each end of the gas detection Dräger tube, or equivalent.

n) Disconnect the end of the tubing (attached to the stopcock) from chamber 3 outlet and connect it onto the upstream end of the gas detection Dräger tube or equivalent. (Observe that an arrow on the tube indicates gas flow direction.) Attach the vacuum Dräger hand pump or equivalent to the downstream end of the gas detection tube, with the arrow pointing toward the pump.

o) Open the stopcock on the bag. Using steady pressure, fully depress and release the hand pump so that gas flows out of the bag and through the gas detection Dräger tube or equivalent. Operate the pump and count the strokes until the bag is empty. (Ten strokes should empty the bag. More than 10 strokes indicates that leakage has occurred and that the test results will not be correct.)

p) When CO_2 is present in the gas bag, a purple stain will develop along the length of the gas detection Dräger tube or equivalent. Observe and record the stain length, l_{st}, in units marked on the gas detection Dräger tube. (Include the faint blue tint in the purple stain length reading.)

q) For best gas detection CO_2 tube accuracy, the stain length shall fill more than 50 % of the tube length, but not exceed 90 %. If the stain length is outside the 50 % to 90 % range, rerun the test using an appropriate sample volume. If stain length is less than 50 % increase the volume of filtrate used. If greater than 90 % use a smaller volume of filtrate.

r) To clean the gas train, remove the flexible tubing and remove the top. Wash out the chambers with warm water and mild detergent, using a brush. Use a pipe cleaner to clean the passages between chambers. Wash, rinse, and then blow out the dispersion tube with dry gas. Rinse the unit with deionized water and allow it to drain dry. Periodically replace the disposable gas bag to avoid leaks and contamination in the bag. (Bag replacement is suggested after 10 analyses.)

Table A.2—Dräger Tube (or Equivalent) Identification, Sample Volumes, and Tube Factors for Various Carbonate Ranges

Carbonate Range mg/L	Sample Volume V_s mL	Dräger Tube Identification [a]	Tube Factor [b,c] f_{CO_2}
25 to 750	10.0	CO_2 100/a	2.5 [d]
50 to 1500	5.0	CO_2 100/a	2.5 [d]
100 to 3000	2.5	CO_2 100/a	2.5 [d]
250 to 7500	1.0	CO_2 100/a	2.5 [d]

[a] See tube body.

[b] Used in calculation.

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

[d] Tube factor 2.5 applies to tubes marked CO_2 100/a (Cat. No. 8101811) with 100 mg/L to 3000 mg/L scale.

A.5.4 Calculation

The total soluble carbonates concentration, $c_{CO_2+CO_3+HCO_3}$, expressed in milligrams per liter, in the filtrate sample shall be calculated using Equation (A.9):

$$c_{CO_2+CO_3+HCO_3} = \frac{l_{st} \times f_{CO_2}}{V_s}$$ (A.9)

where

V_s is the filtrate sample volume, expressed in milliliters;

l_{st} is the gas detection Dräger tube stain length, expressed in units marked on the tube;

f_{CO_2} is the tube factor for CO_2 gas detection tube, from Table A.2.

Annex B
(informative)

Potassium Testing

B.1 Potassium (Concentration above 5000 mg/L)

B.1.1 Principle

Potassium ion is used in drilling fluids to aid in the stabilization of shales and to control swelling clays. The accurate determination of the potassium ion content is necessary to control the properties of the drilling fluid. This procedure is used to measure the potassium ion content in drilling fluid filtrates at levels above 5000 mg/L, approximately 1% wt. or 10 kg/L (3.5 lb/bbl) potassium chloride. In this procedure, potassium is precipitated in a centrifuge tube and the volume of precipitate is measured after centrifuging. The potassium chloride content is then determined from a standard calibration curve.

NOTE: Alternative potassium test methods such as visual colorimetric test strips and reflectometer evaluated colorimetric test strips are sometimes used in field testing but are outside the scope of this document. When using these methods, care should be taken to determine if interference from other ions is an issue and that volumetric dilutions are performed properly.

B.1.2 Reagents and Apparatus

B.1.2.1   Sodium perchlorate ($NaClO_4$) (CAS No. 7601-89-0) solution, 150.0 g/100 mL deionized or distilled water.

Warning—Perchlorates, $NaClO_4$ and $KClO_4$, are explosive in the dry state if heated or if in contact with organic reducing agents. The perchlorates are not hazardous if kept wet. They decompose harmlessly in water.

B.1.2.2   Standard potassium chloride (CAS No. 7447-40-7) solution, 14.0 g potassium chloride per 100 mL deionized or distilled water.

B.1.2.3   Centrifuge, horizontal-swing rotor head (manual or electric), capable of producing approximately 1800 r/min.

B.1.2.4   Clinical centrifuge tube, 10 mL Kolmer type.

B.1.2.5   Graduated volumetric pipettes, 1 mL (TD), 2 mL (TD), and 5 mL (TD).

B.1.2.6   Hypodermic syringe or **serological (graduated) pipette**, 10 mL (TD).

B.1.2.7   Deionized or **distilled water**, shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water

B.1.3 Preparation of Standard Calibration Curve

A standard calibration curve is required for each type of centrifuge. To construct the calibration curve, the following procedure should be used.

a) A minimum of three measurement points [10 kg/m^3 (3.5 lb/bbl), 30 kg/m^3 (10.5 lb/bbl), and 50 kg/m^3 (17.5 lb/bbl) KCl] are required to prepare an accurate calibration curve graph.

b) Samples are prepared by using the standard potassium chloride solution [0.5 mL of standard potassium chloride solution is equivalent to 10 kg/m^3 (3.5 lb/bbl) KCl]. The KCl calibration curve is constructed from concentrations between 10 kg/m^3 (3.5 lb/bbl) and 50 kg/m^3 (17.5 lb/bbl) KCl, using aliquots of 0.5 mL, 1.5 mL, and 2.5 mL of standard potassium chloride solution into three separate centrifuge tubes.

c) Dilute each sample to the 7.0 mL-mark on the centrifuge tube with deionized or distilled water and agitate.

d) To each centrifuge tube, add 3.0 mL of standard sodium perchlorate solution (but do not agitate).

e) Centrifuge at a constant speed (approximately 1800 r/min) for 1 min and read the precipitate volume immediately.

   A fairly constant 1800 r/min can be obtained with a manual centrifuge as follows. Determine the number of revolutions of the rotor per each turn of the crank, i.e. move the crank slowly and count the number of revolutions of the rotor head during one turn of the crank, then crank at an appropriate rate to achieve 1800 r/min. Add the time required to achieve the 1800 r/min speed to the 1 min centrifuge time for the sample.

   For example, if one turn of the crank results in 15 revolutions of the rotor, to obtain 1800 r/min would require 120 turns of the crank per minute [i.e. (1800 r/min ÷ 15 r/turn) = 120 turns/min]. Then determine the number of turns required every 5 s [i.e. (120 turns/min ÷ 60 s/min) x 5 s = 10 turns per 5 s]. Therefore, 10 turns every 5 s produces 1800 r/min.

   Caution—Prior to operating, counterbalance the centrifuge by placing another centrifuge tube with liquid of the same mass, opposite to the test sample.

f) Clean the centrifuge tube immediately after use to facilitate ease of cleaning.

g) Prepare a calibration curve for potassium chloride by plotting the volume of precipitate, expressed in milliliters, versus potassium chloride content of sample, expressed in kilograms per cubic meter (pounds per barrel), on rectangular graph paper. An example of calibration curve for potassium chloride is shown Figure B.1.

   NOTE   Figure B.1 is an example of calibration curve for potassium chloride and should not be used as the calibration curve.
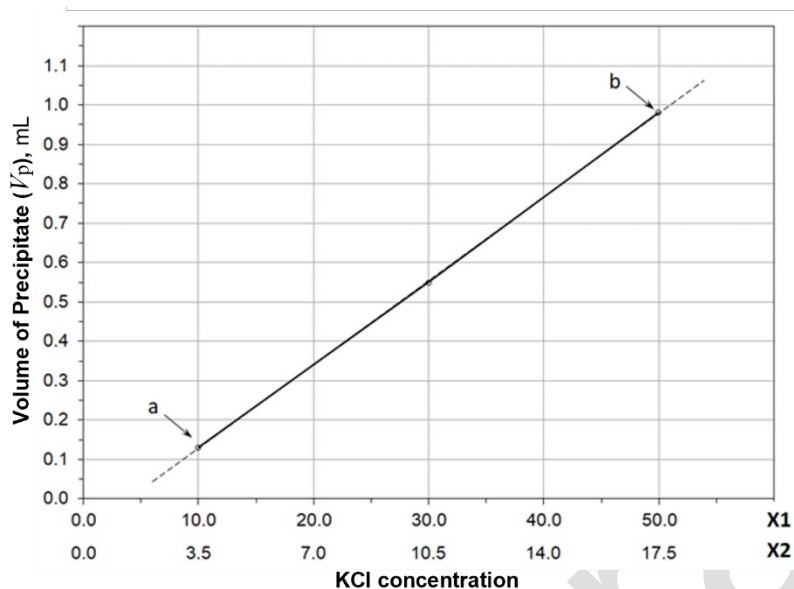
Key

X1	$c_{KCl,SI}$, KCl concentration, expressed in kilograms per cubic meter
X2	$c_{KCl,USC}$, KCl concentration, expressed in pounds per barrel
a	This procedure should not be used for KCl concentrations <10 kg/m^3 (3.5 lb/bbl).
b	For precipitate volumes greater above this value use a reduced filtrate volume using Table B.1.

Figure B.1—Example of Calibration Curve for Potassium Chloride

B.1.4 Test Procedure

To measure potassium ions concentrations above 5000 mg/L, the following procedure should be used.

a) Measure the appropriate volume of filtrate, V_f, into the centrifuge tube (see Table B.1 for range).

b) If less than 7.0 mL filtrate volume is used, dilute to 7.0 mL with deionized water and agitate.

c) Add 3.0 mL of standard sodium perchlorate solution but do not agitate. If potassium is present, precipitation occurs at once.

d) Centrifuge at constant speed (approximately 1800 r/min) for 1 min. Read the precipitate volume, V_p, expressed in milliliters, immediately and record.

Caution—Prior to operating, counterbalance the centrifuge by placing another centrifuge tube with liquid of the same mass, opposite to the test sample.

e) After centrifuging the sample, add 2 drops to 3 drops of additional sodium perchlorate solution to the tube. If no additional precipitate forms proceed to B.1.4 item f). If additional precipitate forms, the total amount of potassium was not measured. See Table B.1 and use the next smaller filtrate volume. Repeat B.1.4 item a) through item e).

f) Determine the potassium chloride concentration of the tested sample of filtrate by comparing the measured precipitate volume, V_p, with the standard calibration curve line prepared in B.1.3.

Record the potassium chloride concentration of the tested sample of filtrate as $c_{KCl,SI}$, expressed in kilograms per cubic meter or as $c_{KCl,USC}$, expressed in pounds per barrel.

If the tested sample of filtrate potassium chloride concentration, $c_{KCl,SI}$ ($c_{KCl,USC}$), from the standard calibration curve exceeds the 50 kg/m^3 (17.5 lb/bbl) reading, use the next smaller filtrate volume from Table B.1, and repeat B.1.4 item a) through B.1.4 item e).

Table B.1—Filtrate Volumes to Be Used at Various KCl Concentrations

KCl Concentration Range		K ion in Filtrate	Filtrate Volume to Use
$c_{KCl,SI}$ kg/m^3	$c_{KCl,USC}$ lb/bbl	$c_{K\text{-}f}$ mg/L	V_f mL
10 to 50	3.5 to 17.5	5250 to 26,250	7.0
50 to 100	17.5 to 35	26,250 to 52,500	3.5
100 to 175	35 to 61	52,500 to 92,000	2.0
Over 175	Over 61	Over 92,000	1.0

B.1.5 Calculations

B.1.5.1 Filtrate potassium chloride concentration

The filtrate potassium chloride concentration, , expressed in milligrams per liter, should be calculated using Equation (B.1):

$$c_{KCl\text{-}f,SI} = \left(\frac{7}{V_f}\right) \times c_{KCl,SI} \tag{B.1}$$

and, to expressed filtrate potassium chloride concentration , $c_{KCl\text{-}f,USC}$, in pounds per barrel, Equation (B.2) should be used:·

$$c_{KCl\text{-}f,USC} = \left(\frac{7}{V_f}\right) \times c_{KCl,USC} \tag{B.2}$$

where

$c_{KCl,SI}$ is the tested sample of filtrate potassium chloride concentration, expressed in kilograms per cubic meter;

$c_{KCl,USC}$ is the tested sample of filtrate potassium chloride, expressed in pounds per barrel;

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

NOTE 1 $c_{KCl,SI}$ is the corresponding potassium chloride concentration on the X1 axis of the standard calibration curve (see Figure B.1), and $c_{KCl,USC}$ is the corresponding potassium chloride concentration on the X2 axis of the standard curve (see Figure B.1).

NOTE 2   If V_f = 7 mL , $c_{KCl-f,SI}$,= c_{KCl-SI} ($c_{KCl-f,USC}$,= $c_{KCl-USC}$).

B.1.5.2 Filtrate potassium ion concentration

The filtrate potassium ion concentration, c_{K-f}, expressed in milligrams per liter, should be calculated using Equation (B.3) from $c_{KCl-f,SI}$, or using Equation (B.4) from $c_{KCl-f,USC}$:

$$c_{K-f} = 525 \times c_{KCl-f,USC} \tag{B.3}$$

$$c_{K-f} = 1500 \times c_{KCl-f,USC} \tag{B.4}$$

where

$c_{KCl-f,SI}$ is the filtrate potassium chloride concentration, expressed in milligrams per liter (see Equation (B.1),

$c_{KCl-f,USC}$ is the filtrate potassium chloride concentration, expressed in pounds per barrel (see Equation B.2)

B.2   Potassium (Concentrations below 5000 mg/L)

B.2.1   Principle

This procedure is used to measure potassium ion content in drilling fluid filtrates at levels below 5000 mg/L. Potassium ion is precipitated as the tetraphenylborate salt by adding an excess of standard sodium tetraphenylborate (STPB) solution. The unreacted STPB is then determined by titration with a quaternary ammonium salt (QAS), hexadecyltrimethyl ammonium bromide, using bromophenol blue as an indicator. The endpoint is a color change from purple blue to light blue. The potassium ion concentration, c_{K-f}, in the sample is calculated by subtracting the amount of unreacted STPB from the amount of STPB originally added to the sample.

NOTE   Alternative potassium test methods such as visual colorimetric-test strips and reflectometer evaluated colorimetric-test strips are sometimes used in field testing but are outside the scope of this document. When using these methods care should be taken to determine if interference from other cations is an issue and that volumetric dilutions are performed properly.

B.2.2   Reagents and Apparatus

B.2.2.1   Standard STPB (CAS No. 143-66-8) solution, 8.754 g sodium tetraphenylborate in 800 mL deionized or distilled water.

Add 10 g to 12 g of aluminum hydroxide to the standard tetraphenylborate solution prepared above, stir 10 min and filter. Add 2 mL of 20 % NaOH solution to the filtered solution and dilute to 1 L with deionized or distilled water.

B.2.2.2   QAS (CAS No. 57-09-0) solution, 1.165 g hexadecyltrimethyl ammonium bromide per 500 mL deionized or distilled water.

B.2.2.3   Sodium hydroxide (CAS No. 1310-73-2) solution, 20 % mass fraction in deionized or distilled water.

Warning—NaOH is a strong alkaline chemical. Avoid skin and eye contact.

B.2.2.4   Bromophenol blue (CAS No. 115-39-9), indicator, prepared by adding 0.04 g tetrabromophenolsulfonphthalein added to 3 mL of 0.1 mol/L NaOH, diluted to 100 mL with deionized or distilled water.

B.2.2.5   Deionized or **distilled water,** shall be in accordance with ISO 3696 of at least Grade III.

Use deionized or distilled water for preparation of all reagents and calibration standards and as dilution water.**B.2.2.6   Graduated pipettes**, 2 mL (TD) graduated in 0.01 mL subdivisions, 5 mL (TD), and 10 mL (TD).

B.2.2.7   Graduated cylinders, capacity 25 mL (TD) and 100 mL (TC).

B.2.2.8   Beakers, capacity 250 mL.

B.2.2.9   Conical Filtration Funnel.

B.2.2.10   Filter paper,[9] grade 1573 ½, prepleated filter paper, 18.5 cm (7.28 in.) diameter, 12 μm to 25 μm retention.

B.2.2.11   Stirring rod, glass or inert to acid or caustic, 25 cm (10 in.) length.

B.2.3   Reagents Quality Control

The equivalent concentration of QAS solution to STPB solution should be tested monthly using the following QC titration procedure to determine the ratio of QAS to STPB, $R_{\text{QAS/STPB}}$.

a)   Dilute 2 mL of the STPB solution in a titration vessel with 50 mL deionized water.

b)   Add 1 mL of 20 % NaOH solution and 10 drops to 20 drops of the bromophenol blue indicator.

c)   Titrate with the QAS solution until the color changes from purple blue to light blue.

d)   The ratio, $R_{\text{QAS/STPB}}$, of QAS to STPB is calculated using Equation (B.5):

$$R_{\text{QAS/STPB}} = \frac{V_{\text{QAS}}}{2} \tag{B.5}$$

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[9] This grade 1573 ½ pre-pleated filter paper is a thin, qualitative, hardened low-ash filter paper made from high-quality cotton wadding designed for qualitative analysis filtration. It should be highly resistant to wetting and to acid solutions and alkaline solutions. The filter paper has the following typical properties that may vary by manufacturer: medium filtration rate: 100 s/100 mL (Herzbergs); particle retention in liquid: up to 25 μm; ash content: <0.05 % by weight; basis weight: 88 g/m^2 (0.02 lb/ft^2); and thickness: 0.170 mm (0.0067 in.).

where

V_{QAS} is the QAS volume used, expressed in milliliters.

e) If $R_{QAS/STPB}$ is outside the range of 4.0 ±0.5, calculate a correction factor, k_{cor}, using Equation (B.6) and use Equation (B.8) to calculate the potassium ion concentration:

$$k_{cor} = \frac{8}{V_{QAS}}$$

(B.6)

B.2.4 Procedure

To determine the potassium ion concentration in the drilling fluid filtrate at concentrations below 5000 mg/L, the following procedure should be used.

a) Determine if the equivalent concentration of QAS solution to STPB solution has been tested in the past 30 days using B.2.3. If not, titrate the ratio of QAS to STPB, $R_{QAS/STPB}$, using the B.2.3 procedure and calculate a correction factor, if needed.

b) Use Table B.2 to determine the appropriate filtrate sample size. Use a pipette to measure the filtrate sample and transfer into the 100 mL graduated cylinder.

c) Using a 5 mL pipette, add 4 mL of NaOH solution (see B.2.2.3, 20 % mass fraction) to the 100 mL graduated cylinder, containing the filtrate sample [see B.2.4 item b)]. Using a 25 mL graduated cylinder, add 25 mL of STPB solution to the 100 mL graduated cylinder, and add deionized water to bring the level of the solution to the 100 mL-mark.

d) Mix thoroughly with a stirring rod and allow solution to stand for 10 min.

e) Using the filtration funnel and pleated filter paper, filter the solution into a clean/dry 100 mL graduated cylinder. If the filtrate is cloudy, re-filter the solution until it is clear.

f) Using a 25 mL graduated cylinder, transfer 25 mL of the filtered solution into a 250 mL beaker.

g) Add 10 drops to 15 drops of bromophenol blue indicator, producing a purple-blue solution.

h) Titrate slowly with QAS solution until color changes from purple blue to light blue. Record the volume of QAS solution used as V_{QAS}.

Table B.2—Filtrate Volumes to Be Used at Various KCl Concentrations

KCl Concentration Range		

$c_{KCl,SI}$ kg/m^3	$c_{KCl,USC}$ lb/bbl	K Ion in Filtrate c_{K-f}	Filtrate Volume to Use V_f
0.5 to 3.0	0.18 to 1.05	263 to 1575	10.0
3.0 to 6.0	1.05 to 2.1	1575 to 3150	5.0
6.0 to 20.0	2.1 to 7.0	3150 to 10,500	2.0

B.2.5 Calculation

B.2.5.1 Potassium Ion Concentration

If the ratio of QAS to STPB, $R_{QAS/STPB}$, from B.2.3 is within the 4.0 ±0.5 range, the potassium ion concentration in the filtrate, $c_{K-f,QAS}$, in milligrams per liter, should be calculated using Equation (B.7):

$$c_{K-f,QAS} = 1000 \times \frac{\left(25 - V_{QAS}\right)}{V_f}$$ (B.7)

If the ratio of QAS to STPB, $R_{QAS/STPB}$, from B.2.3 is outside the 4.0 ±0.5 range, calculate the potassium ion concentration in the filtrate, $c_{K-f,QAS}$, , expressed in milligrams per liter, using Equation (B.8):

$$c_{K-f,QAS} = 1000 \times \frac{\left[25 - \left(k_{cor} \times V_{QAS}\right)\right]}{V_f}$$ (B.8)

where

V_f is the filtrate sample volume, expressed in milliliters;

V_{QAS} is the volume of QAS, expressed in milliliters;

k_{cor} is the correction factor for $R_{QAS/STPB}$.

B.2.5.2 Potassium Chloride Concentration

The potassium chloride concentration in the filtrate, $c_{KCl-f,SI}$, expressed in kilograms per cubic meter,or $c_{KCl-f,USC}$, expressed in pounds per barrel, should be calculated respectively using Equation (B.9) or Equation (B.10):

$$c_{KCl-f,SI} = \frac{c_{K-f,QAS}}{525}$$ (B.9)

$$c_{KCl-f,USC} = \frac{c_{K-f,QAS}}{1500}$$ (B.10)

where

$c_{\text{K-f,QAS}}$ is the potassium ion concentration expressed in milligrams per liter, calculated using Equation (B.7) if no correction factor is needed, or using Equation (B.8) if a correction factor is needed.

Annex C
(normative)

Measurement of Shear Strength Using Shearometer Tube

C.1 Principle

C.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 circulation. High shear strength can also cause difficulties in logging, perforating and other "downhole" operations.

C.1.2   The following 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 made on a static, heat-aged drilling fluid sample. Aging temperatures are therefore selected to be near or above the estimated bottom-hole temperature of the well.

Caution—Always follow the manufacturers' procedures when using heat aging cells.

Standard heat aging cells and the procedure described in API 13I [4] are generally rated for the range of most bottom-hole temperatures, although they are not rated for elevated pressures. Standard heat aging cells are used for fluid evaluations, comparisons, screening, and are normally used for this static shear procedure. Static shear testing is conducted on a heat aged fluid that has cooled and is at atmospheric pressure conditions (standard conditions).

Heat aging or testing at high temperatures and elevated pressures provides a better relationship to downhole conditions for higher temperature wells but requires specialized equipment. To evaluate the gelation of a drilling fluid at high temperature and elevated pressure, a consistometer, HTHP viscometer, or other specialized equipment is required.

C.2 Apparatus

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

—   length   89 mm (3.5 in.)

—   outside diameter   36 mm (1.4 in.)

—   wall thickness   0.2 mm (0.008 in.)

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

C.2.2   Platform for weights, metal square platform suitable to support the weights on top of the shearometer tube, larger than 36 mm (1.4 in.), OD of the tube, but smaller than the bore of the heat aging cell. It is recommended that the mass of the platform be stamped on the surface of the platform.

C.2.3   Set of weights, in gram increments.

C.2.4 **Ruler**, graduated in millimeters (inches).

C.3 Procedure

Shear strength using a shearometer tube shall be measured as per the following procedure.

a) After static heat aging for the predetermined time (see API 13I [4]) and cooled to room temperature while being kept upright and static. Place and balance the shear tube carefully on the surface of the aged sample, it may be necessary to shift the tube and weights on the platform to ensure that the tube's initial penetration into the drilling fluid is vertical. If a crust had developed on the heat-aged sample, the surface of this crust shall be gently broken using the shear tube as it is placed into the fluid for the test.

b) Carefully place the platform and just enough weight on the platform to start the downward movement of the shear tube. Unless too much weight is added or the fluid has no measurable static shear strength, the tube will stop its downward travel at the point where the shear strength resistance of the aged drilling fluid against the surface of the tube is sufficient to support the applied mass. It is desirable to submerge at least one-half of the length of the tube.

c) Record the total mass, in grams, of the platform and weights, m_{tot}. Record the mass, in grams, of the shear tube, m_{st}. Measure the portion of the tube submerged in the fluid. 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.

C.4 Calculation

Shear strength [10] of the drilling fluid shall be calculated using Equation (A.1) expressed in pascals (SI unit) or Equation (A.2) expressed in pounds-force per one hundred square feet (lbf/100ft²) (USC units)

a) Shear strength in SI units (pascals):

$$\gamma_{SI} = \frac{4.40 \times (m_{st} + m_{tot})}{l_{SI}} - (1.02 \times \rho_{df})$$ (A.1)

where

γ_{SI} is the shear strength of the drilling fluid, expressed in pascals;

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

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

l_{SI} is the submerged length of shear tube, expressed in centimeters;

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[10] Shear strength Equations derived from reference [10] in bibliography.

ρ_{df} is the drilling fluid density, expressed in grams per milliliter.

b) Shear strength in USC units (pounds-force per 100 square feet);

$$\gamma_{USC} = \frac{3.61 \times (m_{st} + m_{tot})}{l_{USC}} - \left(0.256 \times \rho_{df,USC}\right) \qquad (C.2)$$

where

γ_{USC} is the shear strength of the drilling fluid, expressed in lbf/100ft²;

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

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

l_{USC} is the submerged length of shear tube, expressed in inches;

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

Annex D
(informative)

Resistivity

D.1 Principle

Control of the resistivity of a drilling fluid, drilling fluid filtrate and filter cake can be desirable to better evaluate formation characteristics from electric logs. This may be accomplished using either a direct-reading or a digital resistivity meter.

D.2 Direct-Reading Resistivity Meter

D.2.1 Apparatus

D.2.1.1 Direct-reading resistivity meter, or similar resistivity meter.

Follow manufacturer's instructions for current source, calibration, measurement, and calculations. Many direct-reading resistivity meters have the ability to measure the resistivity of fluids, slurries and semisolids such as filter cakes with resistivities from 0.01 to 10 ohm-meters. The resistivity can be converted to an equivalent concentration of parts per million sodium chloride concentration that would produce the measured resistivity. Use the tables or nomograms provided in the manufacturer's manual or published log interpretation charts (see Reference [11] in the bibliography).

D.2.1.2 Calibrated resistivity cell.

D.2.1.3 Thermometer, reading 0 °C to 105 °C (32 °F to 220 °F), accurate to ±0.5 °C (±1 °F).

D.2.1.4 Bottle brush, suitable for size and type of cell.

D.2.1.5 Pipe cleaner, suitable for size and type of cell

D.2.1.6 Laboratory detergent solution, appropriate for cleaning metal or plastic surfaces Do not use solvents.

D.2.2 Procedure

In order to conduct a resistivity test on a drilling fluid sample, the following procedure should be used.

a)   Fill the clean, dry resistivity cell with freshly stirred drilling fluid or drilling fluid filtrate. No air or gas should be entrained in the sample. Empty and refill the cell several times to ensure wetting of the cell walls.

b)   Connect cell to resistivity meter.

c)   Allow for the sample to equilibrate to room temperature.

d)   Measure the resistivity in ohm-meters (direct-reading) or resistance in ohms (not direct-reading).

Meter or manufacturer's instructions will indicate the type of reading.

e)   Measure the sample temperature to the nearest 0.5 °C (1 °F).

f)   Clean the cell. Scrub with brush, pipe cleaner and detergent if necessary. Rinse with distilled or deionized water and allow to dry.

D.2.3 Calculations and reporting

Should be reported:

a)   the drilling fluid resistivity, r_{df}, or the filtrate resistivity, r_f, in ohm-meters, to the nearest 0.01 ohm-meters;

b)   the sample temperature in degrees Celsius (Fahrenheit).

If the resistivity-meter reading, R_r, is in ohms, drilling fluid resistivity and filtrate resistivity shall be converted to ohm-meters as calculated respectively using Equations (D.1) and (D.2):

$$r_{df} = R_{r\text{-}df} \times K \tag{D.1}$$

$$r_f = R_{r\text{-}f} \times K \tag{D.2}$$

where

r_{df} is the drilling fluid resistivity, expressed in ohm-meters;

r_f is the filtrate resistivity, expressed in ohm-meters;

K is the resistivity-cell constant, expressed in square meters per meter;

$R_{r\text{-}df}$ is the resistivity-meter reading with drilling fluid expressed in ohms;

$R_{r\text{-}f}$ is the reistivity-meter reading with drilling fluid filtrate, expressed in ohms.

D.3 Digital-Reading Resistivity Meter

D.3.1 Apparatus

D.3.1.1 Digital reading resistivity meter

Follow manufacturer's instructions for current source, calibration, measurement, and calculations. Many digital-reading resistivity meters measure the resistivity of fluids, slurries and semisolids such as filter cakes with resistivities from 0.01 up to 400 ohm-meters, depending on the model. The displays allow for reading of resistivity, temperature and equivalent sodium chloride concentrations in parts per million.

D.3.1.2 Calibrated resistivity cell.

D.3.3.3 Standard resistivity solutions as suggested by manufacturer for calibration.

D.3.1.4 Thermometer, reading 0 °C to 105 °C (32 °F to 220 °F), accurate to ±0.5 °C (±1 °F).

D.3.1.5 Bottle brush, suitable for size and type of cell.

D.3.1.6 Pipe cleaner suitable for size and type of cell.

D.3.1.7 Laboratory detergent solution, appropriate for cleaning metal or plastic surfaces, Do not use solvents.

D.3.2 Procedure

In order to conduct a resistivity test on a drilling fluid sample, the following procedure should be used.

a)   Fill the clean, dry resistivity cell with freshly stirred drilling fluid or drilling fluid filtrate. No air or gas should be entrained in the sample. Empty and refill the cell several times to ensure wetting of the cell walls.

b)   Connect cell to resistivity meter.

c)   Allow for the sample to equilibrate to room temperature.

d)   Measure the resistivity in ohm-meters or sodium chloride in parts per million

Meter or manufacturer's instructions will indicate the type of reading.

e)   Measure the sample temperature to the nearest 0.5 °C (1 °F), if not provided by the instrument.

f)   Clean the cell. Scrub with brush, pipe cleaner and detergent if necessary. Rinse with distilled or deionized water and allow to dry.

D.3.3 Calculations and Reporting

Should be reported drilling fluid resistivity, r_{df}, or drilling fluid filtrate resistivity, r_f, expressed in ohm-meters as fluids temperature in degrees Celsius (degrees Fahrenheit); refer to D.2.3 .

Annex E
(informative)

Removal of Air or Gas prior to Testing

E.1 Principle

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

NOTE   If drilling fluid density is the only property desired, the pressurized fluid density balance described in Section 6 can be used. Solids analysis (retort, Section 9) is sensitive to the presence of entrained air. Careful deaeration of the fluid sample used in the retort, is essential in the measurement of solids content.

E.2 Apparatus

E.2.1 Fluid reservoir, constructed from clear plastic with o-ring or gasketed lid and sealed stirring apparatus or paddle, which can be completely sealed to atmospheric pressure and connected to a vacuum source.

E.2.2 Vacuum pump, typically handheld unit with vacuum gauge

E.2.2 Defoamer, suitable for use with drilling fluid to be tested.

E.3 Procedure

In order to remove entrained air from a drilling fluid sample, the following procedure should be used.

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

b)   Add several drops of defoamer to the drilling fluid surface.

c)   Insert stirrer and gasketed/o-ring sealing lid to the fluid reservoir.

d)   Attach the vacuum hose from the pump to the sealed fluid reservoir and apply approximately -15 kPa (-120 mm Hg) [- 2 lbf/in.² (-5.0 in. Hg)] vacuum. Check the lid for an airtight seal by observing a steady gauge reading.

e)   Increase the vacuum to approximately -90 kPa (-650 mm Hg) [-13 lbf/in.² (-26 in. Hg)] vacuum and turn the paddle crank assembly at a moderate rate (to achieve a slow smooth stirring action) for 3 to 5 minutes.

f)   Slowly relieve the vacuum to about -15 kPa -120 mm Hg) [-2 lbf/in.² (-5 in. Hg)] vacuum and observe drilling fluid for air or gas bubbles.

g)   If adequately deaerated, with fluid reservoir upright, relieve vacuum completely and remove drilling fluid sample for testing.

h)   If not adequately deaerated, repeat the procedure E.3 item e) to item f) until air or gas is removed from the sample.

Annex F
(normative)

Drill Pipe Corrosion Ring Coupon

F.1   Principle

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

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

F.2   Reagents and Apparatus

F.2.1   Hydrochloric acid (CAS No. 7647-01-0), inhibited, concentration 10 % to 15 % in deionized water.

Warning—Hydrochloric acid (HCl) is a strong and toxic acid.

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

Warning—Acetone is highly flammable.

F.2.3   Methanol (CAS No. 67-56-1).

Warning—Methanol is highly flammable.

F.2.4   Deionized or **distilled water**, shall be in accordance with ISO 3696 of at least Grade III.

F.2.5   Detergent solution.

F.2.6   Corrosion ring

Corrosion rings shall meet the following requirements.

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

b)   **Ring composition**: Ideally, the ring should be made from steel identical to that of the tool joint in which it is placed to avoid galvanic corrosion. However, such a requirement is impractical and the use of a grade of steel that is similar in chemical composition, such as AISI 4130, is recommended. The grade of steel used should be identified on the report form. The rings are normally cut from tubes that have

not been quenched and tempered. The similarity in composition of the 4130 steel and the tool joint should be adequate to minimize galvanic effects and provide useful data.

c) **Ring marking:** The rings should be etched or stamped with a serial number for permanent identification.

d) **Ring preparation** (by the supplier): The rings should be scrubbed with a stiff fiber-bristle brush and detergent solution and rinsed with clean water and with anhydrous acetone or methanol. Allowed to dry, weighed to nearest ±0.1 milligram, and the original mass recorded on the coupon form. Store the ring in a dry container, such as a desiccator, to prevent corrosion. The corrosion rings should be shipped to the field in sealed packaging and preferably wrapped in desiccant paper to minimize atmospheric corrosion.

F.2.7   **Ultrasonic bath** (preferred) or **brush**, fiber-bristle, or **steel wood,** fine-grade (000).

F.2.8   **Gloves**, acid resistant, part of PPE when handling strong acids.

F.2.9   **Iron sulfide test solution**—Acid arsenate test.

F.3   Procedure

To use a drill-pipe corrosion ring to measure corrosivity of drilling fluid environments on the drill-string and other steel equipment, the following procedure shall be used.

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

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

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

   NOTE   Photos as received and after cleaning are recommended.

d) Prior to cleaning for weighing testing shall be made for corrosion by-products and mineral scale and the results reported. If by-products are attracted to the magnet, it is an indication of oxides. The surface can be spot tested qualitatively for sulfides by the acid arsenate (iron sulfide test solution) test and 15%

hydrochloric acid. Effervescence from either will indicate mineral scale (fast effervescence) or iron carbonate (slow effervescence).

e) The recommended cleaning solution is an aqueous detergent solution. Simple volatile solvents such as acetone or methanol should not be used. The rings should be cleaned with a detergent solution and a stiff, fiber-bristle brush. It is recommended that mineral scale and corrosion by-products be removed by immersing the ring in inhibited 10 % to 15 % hydrochloric acid solution one or more times to for 5 s to 10 s. The ring should be rinsed with water or lightly brushed using the detergent solution after each acid dip. Rinse thoroughly with clean deionized water followed by anhydrous acetone or methanol. Allow the coupon to dry prior to weighing. Abrasive materials or strong, uninhibited acids should not be used. An ultrasonic bath, with detergent solution, can be useful in cleaning the rings.

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

f) After the preweighed drill pipe corrosion coupon has been properly cleaned and the corrosion film and type of corrosion appearance and by-products noted, the ring should be weighed to the nearest ±0.1 milligram and the mass loss determined. If significant loss of metal due to mechanical damage is evident, it should be noted and taken into consideration in evaluation of the ring. The corrosion rate may be reported as kg/(m^2-year) or mm/year [lb/(ft^2-year) or mils/year].

g) Equations for calculating the corrosion rate are provided in F.5.

F.4   Comments on Visual Examination

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

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

Examination of a ring that reveals a few deep pits with a relatively low mass loss should be noted. This condition would indicate a rather severe corrosion problem, even though the calculated corrosion rate is considered low.

F.5   Calculation

The corrosion rate, q_{SI}, expressed in kilograms per square meter-year, shall be calculated using Equation (F.1).

$$q_{SI} = \left(\frac{\Delta m}{1 \times 10^6} \right) \left(\frac{1 \times 10^4}{A_{SI}} \right) \left(\frac{8760}{t} \right) = 87.6 \times \left(\frac{\Delta m}{A_{SI} \times t} \right) \tag{F.1}$$

where

Δm is the mass loss, expressed in milligrams;

A_{SI} is the area, expressed in square centimeters;

t is the exposure time, expressed in hours;

and as q_{USC}, expressed in pounds per square foot-year, shall be calculated using Equation (F.2):

$$q_{USC} = \left(\frac{\Delta m}{453{,}600}\right)\left(\frac{144}{A_{USC}}\right)\left(\frac{8760}{t}\right) = 2.781 \times \left(\frac{\Delta m}{A_{USC} \times t}\right) \qquad \text{(F.2)}$$

where

Δm is the mass loss, expressed in milligrams;

A_{USC} is the area, expressed in square inches;

t is the exposure time, expressed in hours.

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

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

Equations (F.3) to (F.7) give the conversion rates between the various corrosion rate units for steel coupons (with a relative density of 7.86).

a)   Corrosion rate in SI units (millimeters per year):

      mm/year = 0.127 × q_{SI} (F.3)

b)   mm/year = 0.621 × q_{USC}
     (F.4)Corrosion rate in USC units (mils per year)

      mils/year = 5.01 × q_{SI} (F.5)

      mils/year = 24.6 × q_{USC} (F.6)

      lb/(ft^2-year) = 1.61 × (mm/year)   (F.7)

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

Annex G
(informative)

Sampling, Inspection, and Rejection of Drilling Materials

G.1   Principle

The following procedure provides the sampling, inspection, and rejection method for materials conforming to API 13A[1]. It is applicable to all grades of barite, hematite, bentonite, attapulgite, sepiolite, carboxymethylcellulose, polyanionic cellulose, starch, xanthan gum as well as other powdered products.

G.2   Sampling of Powdered Material in Packages

Procedure to prepare sample of powdered material in packages should be as per the following.

a)   Take 15 or more samples of 0.5 kg (1 lb) per lot to be combined as the test sample (see G.4).

b)   For each lot of 1000 packages or less, 15 packages should be sampled.

c)   The sampling should be carried out by either of the following methods as agreed upon by the contracting parties:

1)   a sample weighing at least 0.5 kg (1 lb) should be taken from the top of each package, or

2)   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.

G.2.1 Sampling of Powdered Material in Bulk

Procedure to prepare sample of powdered material in bulk should be as per the following.

a)   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 tons to 100 metric tons) (25 long tons to 98 long tons). The tube should be of sufficient length to permit taking a substantial sample from the top to the bottom of the container of material being sampled.

b)   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.

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

G.3   Preparation of Test Sample

G.3.1   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.

G.3.2   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.

G.4   Test

Each party should make measurements on the allocated test sample in accordance with the specific test procedure(s) outlined in API 13A [1] and API 13I [4] or other quality assurance procedure.

G.5   Inspection

The purchaser's inspector should be afforded reasonable facilities for careful sampling and inspection. A period of 6 days should be allowed for sampling and completion of the acceptance test.

G.6   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.

G.7   Retesting

G.7.1   Either of the contracting parties may make claim for retest within one week of the date of the original test report.

G.7.2   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 H
(informative)

Rig-site Sampling

H.1   Principle

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

H.2   Bottom-flow Sampling (Preferred)

H.2.1   Apparatus

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

H.2.1.2   Canvas bag, approximately 14 cm (5.5 in.) diameter and 30 cm (12 in.) long.

H.2.1.3   Suitable container, e.g. a tie-bag, so constructed as to retain fines.

H.2.2   Procedure

Rig-sampling using a side-stream sampler should be as per the following procedure.

a) Connect a side-stream sampler to the truck outlet.

b) Take three [2.5 kg to 5 kg (5 lb to 13 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 nonrepresentative 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 for error 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.

c) 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 to get a full sack with a minimum of dusting.

d) Carefully transfer the sample from the canvas bag to a tie-bag or other suitable container. Label this container with pertinent information, such as but not limited to well name and number, date, lot, and chemical name.

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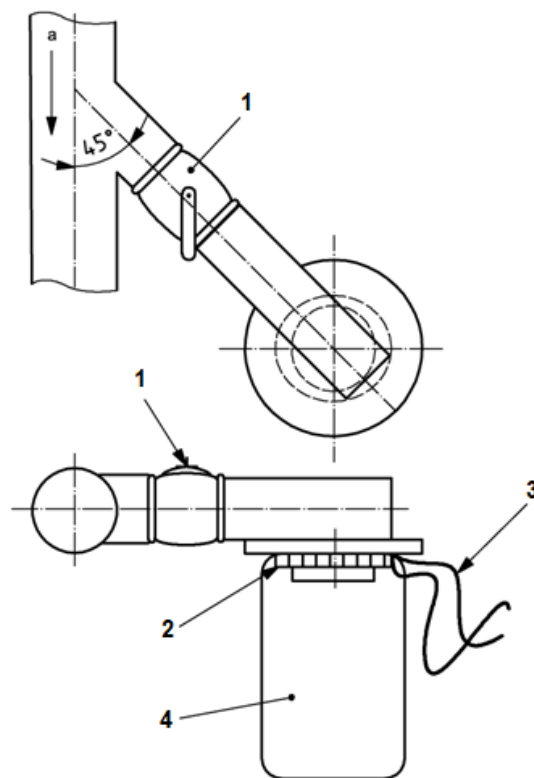
Key

1	5 cm (2 in.) ball valve with 5 cm (2 in.) side pipe
2	bead or lip to hold bag
3	draw string
4	canvas bag 14 cm x 30 cm (5.5 in. x 12 in.)
a	Flow direction.

Figure H.1—Side-stream Sampling Device

H.3   Grab Sampling

H.3.1   Apparatus

H.3.1.1   Sample scoop, as shown in Figure H.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.

H.3.1.2   Suitable container, e.g. a tie-bag, so constructed as to retain fines.

H.3.2   Procedure

Grab sampling using a sample scoop should be done as per the following procedure.

Warning—Be sure all pressure is removed from the tank prior to opening the hatch to avoid injury or death.

a) Open the top hatch carefully. Do not let the cover bump the top of the tank.

b) Remove the top surface material under the hatch to form a trench 1.5 times wider than the sample scoop.

c) 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 with all pertinent information, such as but not limited to well name, well number, date, lot, and chemical name.

d) 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.
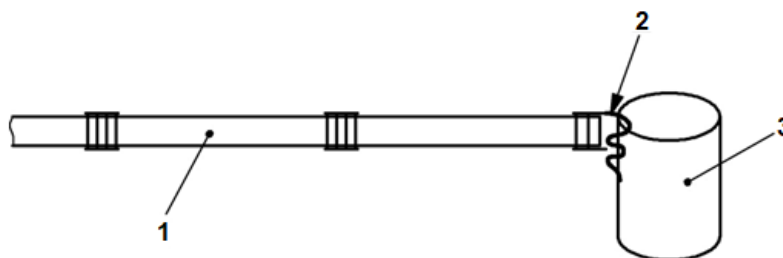
Key

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

2   strap hinge

3   metal can

Figure H.2—Sample Scoop

Annex I
(normative)

Calibration and Verification of Glassware, Thermometers, Timers, Viscometers, Retort Cup, and Drilling Fluid Balances

I.1   Principle

This annex contains methods for the calibration of equipment not covered under test procedures in this standard.

I.2   Calibration of Apparatus

I.2.1   Volumetric Glassware

Calibration of pipettes, graduated cylinders, and similar equipment shall be normally performed by the glassware manufacturer as the compliance with the relevant product specification. Glassware users shall obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically, and the procedure should use the tolerance from the glassware specification. For glassware where a specification cannot be determined the calculated volume at the maximum capacity shall be within 2% of the indicated volume. Periodic recalibration is not required. A procedure for calibration of graduated cylinders is provided in I.3.

I.2.2   Thermometers

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

I.2.3   Timers

Timers shall be accurate to within ±5 s per hour and shall be verified over a period no less than 12 min and shall be checked annually. Calibration may use the time signal from the NIST or similar web sites or radio stations. If not within required accuracy, the units shall be adjusted or replaced.

NOTE   Viscometer sag shoe test (VSST) (see K.5) requires a timer accurate to ±2 s per hour.

I.2.4   Direct-indicating Viscometers

Direct-indicating viscometers used in field testing shall be calibrated against traceable standard viscosity fluids. Viscometers shall 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 shall be checked at least monthly and more frequently if the viscometer indicates instability. A procedure for the calibration of viscometers is provided in I.5.

I.2.5   Drilling Fluid Balances

I.2.5.1   Drilling fluid balances shall be calibrated using deionized or distilled water as specified in the procedure 5.2 and 6.2. The mud balance shall be calibrated frequently. The recommended frequency is

prior to each set of measurements (e.g. daily or by each work shift), or as specified by the operator, prevailing regulation, drilling contractor, drilling fluid company or other controlling party. Fresh water shall give a reading of 1.00 ±0.005 g/mL or 1000 ±5 kg/m^3 (8.345 ±0.05 lb/gal or 62.4 ±0.25 lb/ft^3) at 21 °C (70 °F) when the bubble level is centered. If it does not, the instrument shall be calibrated by adjusting the balancing screw or the amount of shot (metal beads) in the well at the end of the graduated arm until it meets the accuracy requirement, or the instrument shall be removed from service.

I.2.5.2 The instrument shall be calibrated on a less frequent basis (e.g. annually) according to the procedure as specified by the manufacturer or using a two-point calibration. Calibration according to the procedure as specified by the manufacturer or using a two-point calibration shall always be performed after damage, repair, or replacement of items such as the sample cup lid, threaded lid retainer or the shot well screw.

I.2.6 Retort Cups

Retort cups shall be calibrated using deionized water or distilled water (see 3.3), as specified in the test procedure provided in I.6. The recommended frequency is prior to each set of measurements, or as specified by the operator, drilling fluid company or other interested party.

I.3 Procedure—Calibration of Graduated Glassware

In order to calibrate graduated cylinders (receivers), the following procedure shall be used.

a) Allow the receiver and distilled or deionized water (see 3.3) to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).

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

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

d) Record the masses for each incremental volume of water at the specific graduation mark, m_W, to the nearest 0.01 g.

e) Calculate the volume of the receiver at each mark, V_M, as calculated using Equation (I.1):

$$V_M = \frac{m_W}{\rho_W}$$ (I.1)

where

V_M is the receiver (graduated cylinder) volume at a specified mark, expressed in milliliters;

m_W is the mass of water, filling receiver at a specified mark, expressed in grams;

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

Table I.1—Density of Water as a Function of Temperature [11]

Temperature °C	Density g/mL	Temperature °F	Density g/mL
15.0	0.9991	59	0.9991
15.5	0.9990	60	0.9990
16.0	0.9989	61	0.9989
16.5	0.9989	62	0.9988
17.0	0.9988	63	0.9987
17.5	0.9987	64	0.9986
18.0	0.9986	65	0.9985
18.5	0.9985	66	0.9984
19.0	0.9984	67	0.9983
19.5	0.9983	68	0.9982
20.0	0.9982	69	0.9981
20.5	0.9981	70	0.9980
21.0	0.9980	71	0.9978
21.5	0.9979	72	0.9977
22.0	0.9978	73	0.9976
22.5	0.9977	74	0.9975
23.0	0.9975	75	0.9973
23.5	0.9974	76	0.9972
24.0	0.9973	77	0.9970
24.5	0.9972	78	0.9969
25.0	0.9970	79	0.9968
25.5	0.9969	80	0.9966
26.0	0.9968	81	0.9965
26.5	0.9966	82	0.9963
27.0	0.9965	83	0.9961
27.5	0.9964	84	0.9960
28.0	0.9962	85	0.9958
28.5	0.9961	86	0.9956
29.0	0.9959	87	0.9955
29.5	0.9958	88	0.9953
30.0	0.9956		
30.5	0.9955		
31.0	0.9953		

[11] Water density data referenced from *Handbook of Chemistry and Physics,* 66th Edition, CRC Press Inc., Boca Raton FL (1985-86)

I.4 Procedure—Calibration of Thermometers

In order to calibrate a thermometer, the following procedure shall be used.

a) Place the thermometer to be calibrated alongside either a thermometer or thermocouple that is certified and traceable to a national standard or a standard reference thermometer such as a high-quality mercury-in-glass thermometer from the ASTM precision series, ASTM 62C or 63C (ASTM 62F or 63F) [capable of measuring to ±0.1 °C (±0.2 °F)] 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.

b) Read both thermometers and record the readings.

c) Repeat the readings at 5 min intervals to obtain at least four sets of readings.

d) Calculate the average reading, R_1, for the standard reference thermometer and the average reading, R_2, for the working thermometer.

e) For adjustable-scale thermometers, adjust the scale to read the same as the standard reference thermometer. For other thermometers, follow the procedure in I.4 item f).

f) Calculate the thermometer correction, C_{th}, expressed in degrees (°C or °F) to add to the thermometer reading, using Equation (I.2):

$$C_{th} = R_1 - R_2 \qquad\qquad (I.2)$$

where

C_{th} is the correction value to add to the thermometer reading, expressed in degrees (°C or °F);

R_1 is the average reading for the standard thermometer, expressed in degrees (°C or °F);

R_2 is the average reading for the working thermometer, expressed in degrees (°C or °F).

EXAMPLE   Thermometer correction determination, in degrees Celsius:

R_1 = 23.9 °C

R_2 = 24.2 °C

C_{th} = 23.9 – 24.2 = – 0.3 °C

This temperature correction is used by adding the value to the thermometer reading. An example thermometer correction application is shown below for a correction factor of C_{th} = – 0.3 °C

Thermometer correction application:

Temperature reading: R (measured) = 25.0 °C

                                            R_c (corrected) = 25.0 + (– 0.3) = 24.7 °C

NOTE   ISO 386 [6] can be seen for a more complete procedure description.

g) 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.

I.5   Procedure—Calibration of Viscometers

To calibrate direct-indicating viscometers, the following procedure shall be used.

a) Obtain two certified calibration fluids closest to the viscosity range of interest, common viscosities are 20 mPa•s (20 cP), 50 mPa•s (50 cP), 100 mPa•s (100 cP), and 200 mPa•s (200 cP).

   For most R1 B1 F1.0 viscometer configuration and equivalent applications, 50 mPa•s (50 cP) and 100 mPa•s (100 cP) viscosity standards are recommended.

   Since the actual viscosity of the certified calibration fluid will vary slightly from lot to lot, each sample comes with a chart or table of the actual measured viscosity values versus temperature, typically in increments of 0.1 °C. Viscosity standards are normally certified by methods traceable to a standards body such as the United States National Institute of Standards and Technology (NIST) or equivalent. Each lot of certified calibration fluid is individually certified and shall have a stated expiry date. Make certain that the lot number on the chart matches the lot number on the fluid container and that the expiry date is not exceeded.

   Calibration fluid shall not be used if it is discolored, cloudy, has evidence of water or drilling fluid contamination, or appears to contain any suspended or settled solids.

b) Thoroughly clean and dry all parts of the viscometer that come into contact with the calibration fluid, including the fluid cup, bob, bob shaft, and rotor.

c) 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.

d) 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.

e) Pour the calibration fluid into the sample cup, leaving enough empty volume (approximately 50 mL to 100 mL) in the cup for displacement of fluid due to the viscometer bob and sleeve. Place the cup on the viscometer and adjust the viscometer until the fluid level is aligned exactly with the scribed line on the rotor sleeve.

f) Place a thermometer calibrated and accurate to $\pm$0.1 °C ($\pm$0.2 °F), (e.g. ASTM 90C or 91C grade or equivalent) into the fluid and hold or tape it in place to prevent breakage. Operate the viscometer at 100 r/min until the thermometer reading is stable to within 1 °C (0.2 °F) for a 30 s period (approximately 3 min). Record the temperature reading to the nearest 0.1 °C (0.2 °F).

g) Operate the viscometer and take readings at 600 r/min and 300 r/min. For visually read dial viscometers, estimate and record readings to the nearest 0.5 dial unit, using a magnifying glass if needed. For viscometers with digital readings, record readings to the nearest 0.1 equivalent dial unit.

h) Using the temperature/viscosity chart supplied with the calibration fluid, determine certified viscosity at the test temperature. Compare the 300 r/min dial reading [same as mPa•s (cP)] to the certified viscosity in mPa•s (cP) and record the deviation (plus or minus). Divide the 600 r/min dial reading by 1.98 to obtain the measured viscosity in mPa•s (cP) at 600 r/min, compare it to the certified viscosity in mPa•s (cP), and record the deviation (plus or minus).

NOTE   The 1.98 factor comes from the calibration of the original direct-indicating viscometer with open and closed sleeve fluid ports; see Reference [8] in bibliography.

i)   Remove the fluid cup and calibration fluid. Certified calibration fluid may be reused. Used certified calibration fluid shall not be reused if it is discolored, cloudy, has evidence of water contamination, or appears to contain any suspended or settled solids.

j)   Thoroughly clean and dry all parts of the viscometer that come into contact with the calibration fluid, including the fluid cup, bob, bob shaft, and rotor.

k)   Repeat procedure item c) through j) with the second calibration fluid viscosity.

a)   Deviations exceeding 1.5 mPa•s (1.5 cP) shall not be allowed. If any of the measured values exceeds these tolerances, adjust the viscometer (see viscometer supplier maintenance instructions) and repeat this calibration procedure.

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

I.6   Procedure—Calibration of Retort Cups and Heating jackets

I.6.1 Calibration of Retort Cups

In order to calibrate retort cups, the following procedure shall be used.

a)   Allow the retort cup, lid and distilled or deionized water (see 3.3) to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).

b)   Place the clean, empty retort cup and lid on the balance and tare to zero.

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

d)   Place the filled retort cup with lid on the previously tared balance. Record the water mass to the nearest 0.01 g.

e)   Calculate the retort cup volume, V_{RC}, expressed in milliliters, using Equation (I.3) and, the water density at the recorded ambient test temperature (see Table I.1).

$$V_{RC} = \frac{m_W}{\rho_W} \tag{I.3}$$

where

m_W is the mass of water filling retort cup , expressed in grams;

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

I.6.2 Calibration of Retort Heating Jacket

I.6.2.1 Apparatus and equipment

I.6.2.1.1 Calibration thermocouple instrument, NIST or equivalent, traceable using type J or K probe.

I.6.2.1.2 Retort Heating Jacket to be tested.

I.6.2.2 Procedure

In order to calibrate a retort heating jacket, the following procedure shall be used.

a) Insert the J or K thermocouple probe into the retort chamber making sure that it rests on the bottom of the cavity where the retort goes. The retort cell should not be loaded in the jacket, only the thermocouple. Close the lid prior to testing.

b) Start the retort or for digital programmable temperature-controlled retorts, program the retort to ramp to 500 °C (930°F) then start.

c) For thermostat switch-controlled retorts, record the temperature then the indicator light goes out.

d) For digital programmable temperature-controlled retorts, allow the heating jacket to heat for approximately 40 minutes.

e) After 40 minutes compare the traceable calibration thermocouple to the digital display on the retort, record both values.

f) The recorded retort temperature shall be 500 °C ±40 °C (930 °F ±70 °F).

g) If the retort temperature is outside the allowed range, remove the instrument from service and repair or calibrate according to the manufacturer's instructions until the instrument calibrates to the temperature specification.

I.7   Procedure—Calibration of Filter Press Gasket and O-rings

In order to calibrate filter press gasket and O-rings, the following procedure shall be used.

h) Gaskets or O-rings for the low-pressure, low-temperature filter press shall have an inside diameter of between 75.9 mm (2.98 in.) and 76.9 mm (3.02 in.).

i) Check the gasket or O-ring using a gauge with a diameter of between 75.9 mm (2.98 in.) and 76.9 mm (3.02 in.).

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

Annex J
(normative)

Testing of Weight-material Sag

J.1 Weight-material Sag

J.1.1 General

The objective of this annex is to provide the two most common testing methods used to monitor weight-material sag tendencies of field and lab-prepared drilling fluids under dynamic conditions. For additional information see 13B-2[2, 12].

Weight-material sag is recognized by a significant variation in drilling fluid density [greater than 0.06 g/mL or 60 kg/m^3 (0.5 lb/gal)], lighter followed by heavier than the nominal fluid density, measured when circulating bottoms up, usually where a weighted fluid has remained uncirculated for a period of time in a directional well. It is recognized that sag is both a static and a dynamic phenomenon and has the potential to occur even when the drilling fluid is in motion.

The definition of weight-material extends to any particulate material which is added intentionally to the drilling fluid to increase the fluid density. Common example materials include barite, hematite, and calcium carbonate.

Weight-material sag is a potential problem for many of the complex well types that are now commonly drilled. These include HTHP, extended-reach drilling (ERD), and high-angle/horizontal wells. Weight-material sag may be a complicating factor for the following issues: controlling downhole pressures to ensure that the well is stable with respect to influxes and losses, wellbore instability due to reduction in hydrostatic pressure, accurate calculation of downhole hydrostatic pressure, and difficulty running casing or logging.

Experience shows that weight-material sag cannot be totally eliminated. However, it can be managed within levels that do not adversely affect the drilling operation. Weight-material sag is not entirely a drilling fluid-related problem. The occurrence of sag will be also influenced by wellbore geometry, wellbore environment and the operating conditions. Certain conditions in the drilling operation are conducive to creating dynamic sag. The potential for dynamic sag is promoted by an eccentric non-rotating pipe, such as when sliding in deviated wells, and slow circulation rates with annular velocities less than 33 m/min (<100 ft/min).

In recognition of the complexity of the problem, it is not possible to offer a single preferred method to monitor weight-material sag. The recommendation is to use a range of techniques in combination to provide a qualitative assessment of sag potential and to assess the need for remedial actions, e.g. modifying drilling fluid properties and changing operational parameters/practices.

J.1.2 Testing for Monitoring Weight Material Sag

This annex is limited to two dynamic test methods for monitoring weight material sag.

a) Viscometer sag shoe test (VSST) (J.2), a normative section.

b) Dynamic sag window test (J.3), an informative section.

J.2 Viscometer Sag Shoe Test (VSST) Method (normative section)

J.2.1 Principle

VSST is a well site and laboratory test that measures weight material sag tendencies of field and lab-prepared drilling fluids under dynamic conditions. It provides an intrinsic fluid property without regard to the conditions under which the fluid has been or will be used. As such, results should be combined with operational factors to correlate test results with actual measured sag experienced in the field.

The VSST designation is derived from the direct-indicating viscometer used as a mixer and the thermoplastic insert (Sag Shoe) designed to collect fluid with concentrated weight material in a well at the bottom of a viscometer thermocup [13, 14]. Figure J.1 shows the basic equipment used in the VSST method.
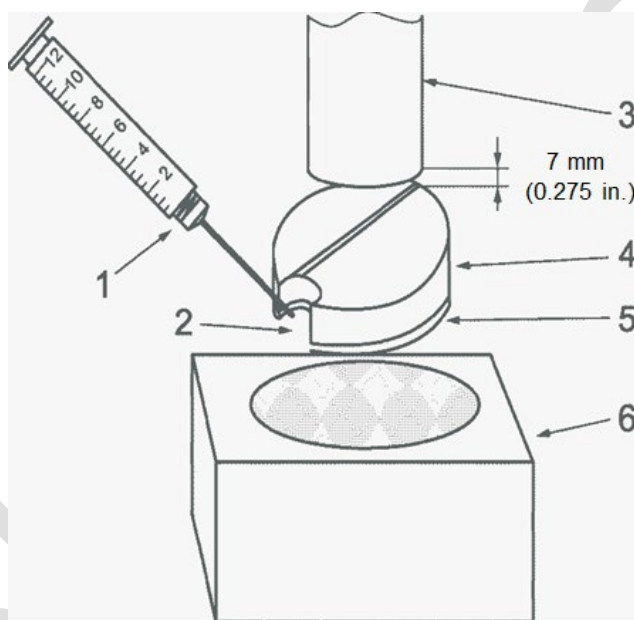
Key

1	Luer syringe		4	VSST Sag Shoe
2	Collection well		5	Metal-backing plate
3	Viscosimeter rotor		6	Thermocup

Figure J.1—Key Equipment for VSST Method

Sag tendency is determined by the density increase of samples extracted from the collection well over a 30 min period at a standard temperature and under a consistent rate of shear.

A weight-material bed pickup test can be performed as an optional measurement to characterize bed removal by higher shear levels. Results can be used to suggest opportunities for bed removal in the field prior to tripping out of the hole.

J.2.2 Apparatus

J.2.2.1 **Direct-indicating viscometer**, powered by an electric motor; see 7.3.1.1 for dimensions.

J.2.2.2 **Stopwatch** or **timer**, accuracy within ±5 seconds per hour.

J.2.2.3 **Thermostatically controlled viscometer cup**.

NOTE   The VSST Sag Shoe is intended to fit snug inside the thermocup but without being wedged.

J.2.2.4 **Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±1 °C (±2 °F).

J.2.2.5 **Luer syringe**, 10 mL, two-piece, solvent resistant.

J.2.2.6 **Syringe needle**, blunt-end, 150 mm (6 in.), 14-gauge needle with Luer connection.

J.2.2.7 **Balance**, digital, with 0.01 g resolution. A triple-beam balance may be used in the field where excessive vibration is expected.

J.2.2.8 **Pycnometer** or **retort cup**, 10 mL.

NOTE   The pycnometer equipment is not required if the test is run at the well site or if the procedure includes the pickup test described in J.2.3.3.

J.2.2.9 **Spatula**, 150 mm (6 in.) or similar.

J.2.2.10 **Deionized** or **Distilled water**. shall be in accordance with ISO 3696 of at least Grade III.

J.2.2.11 **Mixer**, to operate at 10,000 r/min to 15,000 r/min with a single sine-wave impeller of approximately 25.4 mm (1 in.) in diameter.

J.2.2.12 **Container**, for mixing, 180 mm deep (7.125 in.), diameter 97 mm (3.75 in.) at top and 70 mm (2.75 in.) at bottom.

J.2.2.13 **VSST Sag Shoe**, shaped, cylindrical disk manufactured from thermoplastic material, maximum diameter of 59.7 mm ±0.4 mm (2.35 in. ±0.015 in.) and height of 27.7 mm (1.09 in.). The well is a notch cut through the thermoplastic disk and metal-backing plate that connects with the thermocup bottom. The metal plate is used solely to increase the overall mass of the Sag Shoe so that it does not float when used with very dense fluids.

J.2.3   Procedure

In order to conduct a VSST, the following procedure shall be used.

J.2.3.1   Equipment setup

a)   Insert the Sag Shoe into the thermocup and place both on the viscometer plate. Make sure the outside diameter of the sag shoe is just smaller than the inside diameter of the thermocup such that it fits snug.

b)   Raise the plate until the top of the Sag Shoe touches the bottom of the viscometer rotor sleeve and mark the support leg at the upper edge of the locking mechanism.

c)   Lower the plate and thermocup to the base and mark the support leg 7 mm (0.25 in.) below the first mark.

d)   Verify or calibrate the 10 mL-mark on the syringe with blunt-end pipetting needle attached, using deionized water and digital balance. Draw 10 mL water then expel as much water as possible from the syringe barrel, wipe exterior dry and weigh the empty syringe or tare the balance. Draw 10mL water and weigh the syringe. The mass of the water at the 10 mL mark should be 10.0 g ±0.2 g.

NOTE   Calibrate pycnometer or retort cup with distilled water if used for density measurements instead of the syringe.

J.2.3.2   Sag measurement

a)   Insert the Sag Shoe into the thermocup with the collection well positioned for easy access by the syringe, e.g. 60° to 90° either side of the viscometer centerline.

b)   Pre-heat the thermocup with Sag Shoe to 49 °C (120 °F).

c)   Collect a 350 mL drilling fluid sample in the container, mix for 5 min to homogenize the sample then pour approximately 140 mL into the thermocup. Most consistent results are obtained if the starting fluid temperature is close to 49 °C (120 °F) to minimize sag during heating and to be able to measure fluid densities at uniform temperatures.

d)   Position and lock the upper edge of the viscometer locking mechanism at the lower mark on the support leg. The top of the Sag Shoe should be 7 mm (0.25 in.) below the viscometer rotor sleeve. Begin heating the drilling fluid to 49 °C (120 °F) while stirring at 600 r/min.

e)   Using the syringe with blunt-end pipetting needle attached, draw slightly over 10 mL from the drilling fluid remaining in the container, preferably at 49 °C (120 °F). Carefully clear the syringe and pipetting needle of residual air by expelling some fluid and positioning the plunger to the 10 mL calibration mark. Wipe the pipetting needle and syringe surfaces until clean and dry.

f)   Weigh the fluid-filled syringe and record the mass as m_{F1}, expressed in grams.

NOTE   When using the 10 mL pycnometer or retort cup, transfer the fluid from the syringe, weigh and record total mass as m_{F1}, expressed in grams.

g)   Once the viscometer test sample has reached 49 °C (120 °F), set the viscometer speed to 100 r/min and start the 30 min timer.

h)   Stop viscometer rotation at the end of the 30 min test period.

i)   Repeat items e) and f), this time taking the sample from the collection well of the Sag Shoe. Use the pipetting needle tip to find the collection well.

j)   Weigh the fluid-filled syringe and record the total mass as m_{F2}, expressed in grams.

NOTE   When using the 10 mL pycnometer or retort cup, transfer the fluid from the syringe, weigh, and record total mass as m_{F2}, expressed in grams.

J.2.3.3   Bed pickup measurement (optional)

a)   Gently return the 10 mL test sample from the fluid-filled syringe obtained in J.2.3.2 item i) to the Sag Shoe collection well.

b)   Run the viscometer at 600 r/min for 20 min.

c) Collect the sample from the Sag Shoe collection well as in J.2.3.2 item i). Weigh the fluid-filled syringe and record total mass as m_{F3}, expressed in grams.

J.2.4 VSST Calculations and Reporting

J.2.4.1 Weight- material sag

The amount of weight-material sag, B_{VSST}, shall be calculated using Equation (J.4):

$$B_{VSST} = 0.834 \times (m_{F2} - m_{F1})$$ (J.4)

where

B_{VSST} is the amount of weight-material sag, expressed in pounds per gallon;

m_{F1} is the initial mass of 10 mL drilling fluid (plus the syringe), expressed in grams;

m_{F2} is the mass of 10 mL drilling fluid (plus the syringe) taken from the sag shoe following 30 min shear at 100 r/min, expressed in grams [see J.2.3.2 item j)].

Report B_{VSST} in pounds per gallon.

J.2.4.2 Bed pickup measurement ratio

The optional bed pickup measurement ratio, R_{BPU}, as a percent shall be calculated using Equation (J.5):

$$R_{BPU} = \frac{83.4 \times (m_{F2} - m_{F3})}{B_{VSST}}$$ (J.5)

where

R_{BPU} is the calculated bed pickup measurement ratio, expressed as a percentage;

B_{VSST} is the amount of weight-material sag, expressed in pounds per gallon;

m_{F2} is the mass of 10 mL drilling fluid (plus the syringe) taken from the sag shoe following 30 min shear at 100 r/min, expressed in grams [see J.2.3.3 item i)] ;

m_{F3} is the mass of 10 mL drilling fluid (plus the syringe) taken from the sag shoe following 20 min shear at 600 r/min, expressed in grams [see J.2.3.3 item c)].

J.3   Dynamic Sag Window Test (informative section)

J.3.1   Principle

The dynamic sag window test measures low shear rate viscosity values within the range of 0.17 s^{-1} and 1.7 s^1 (0.1 r/min to 3 r/min) to determine if the fluid viscosity falls within a window of values which have been shown to bracket fluids which exhibit minimal sag potential [15]. Dynamic sag studies have shown that barite sag occurs at shear rate values less than 4 s^{-1} in deviated wells. This method is applicable to situations where the nominal annual velocity is 30.5 m/min (100 ft/min) or greater. This section is applicable to both field drilling fluids and fluids mixed in the laboratory; however, it should be noted that for the same formulation, field muds exhibit less dynamic sag due to shear history, the incorporation of drilled solids, and the presence of a higher colloidal content.

J.3.2   Apparatus

J.3.2.1   Multi-speed Viscometer, capable of accurately measuring viscosity from 0.17 s^{-1} (0.1 r/min) to 1022 s^{-1} (600 r/min), calibration to include the 0.17 s^{-1} and 1.7 s^{-1} (0.1 r/min to 3 r/minpm) range.

J.3.2.2   Thermostatically controlled viscometer cup (thermocup)

J.3.2.3   Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±1 °C (±2 °F).

J.3.2.4   Mixer, to operate at 10,000 r/min to 15,000 r/min with a single sine-wave impeller of approximately 25.4 mm (1 in.) in diameter.

J.3.2.5   High Shear Mixer, variable speed, heavy-duty laboratory mixer with a high shear, square-hole impeller screen. Typical impeller blade diameter 31.20 mm (1.228 in.), impeller screen inside diameter 34.85 mm (1.372 in.). The rotor/stator diameter on these mixers is 31.24 mm (1.23 in.). The tolerance between the rotor and stator is between 0.30 mm and 0.38 mm (0.012 in. and 0.015 in.) on the diameter. The hole size on the square-hole screen is 2.4 mm (3/32 in.).

J.3.3   Dynamic sag window procedure

Lab and field samples are subjected to a wide variety of shear histories. For meaningful measurements, samples need to be fully conditioned prior to testing. If measurements of different fluids are to be compared, it is important that the fluids have similar preparation and length of time between high-speed shearing and the viscosity measurements.

To prepare the sample, the following procedure should be used:

a)   Sample mixing involves the entire fluid and any settled solids in the sample container. Lab samples and field samples collected because of sag problems may be expected to have suffered from solids settling during transportation and storage. All fluid and any settled solids should be removed from the sample container and homogenized on the mixer prior to mixing and testing.

b)   Mix the entire sample at a 6000 r/min shear rate using the high shear mixer described in J.3.2.5, for a period of 5 min.

c)   Fill the thermostatically controlled viscometer cup to the appropriate level and immerse the rotor sleeve to the scribed line (be sure the sample temperature is below 50 °C (120 °F) prior to loading the sample).

d) Heat the sample to 49°C (120 °F) while running the viscometer at 300 r/min.

e) For each sample, the length of time between the fluid being sheared as described in J.3.3 item b) and the viscosity measurement should be recorded.

f) Measure and record the viscosity (or shear stress as dial reading) at each of the following shear rates; expressed in s^{-1} 1022, 511, 340, 170, 10.2, 5.1, 1.7, 1.02, 0.51, 0.17, and 0.10 (or expressed in r/min 600, 300, 200, 100, 6, 3, 1, 0.6, 0.3, 0.1, and 0.06) allowing 60 s between each measurement below the 0.51 s^{-1} (3 r/min) shear rate.

g) Based on Table J.1, for each shear rate reading report the viscosity (or shear stress as dial reading) measured by the viscometer. For viscometers which display dial readings (expressed in degrees), these values can be converted to viscosity in cP by multiplying dial unit degrees times the factors shown in column 4 of Table J.1.

h) Plot the viscosity in cP vs shear rate in s^{-1} data on a log-log format as shown in Figure J.2 and determine if the measure data fall between the 0.17 s^{-1} and 1.7 s^{-1} dynamic sag window upper and lower viscosity bounds (see values listed in the key for Figure J.2).

Table J.1—Dynamic Sag Window Data Table

Shear Rate		Shear Stress Dial Reading	Multiplication Factor	Viscosity
s^{-1}	r/min	degrees		cP
1022	600		0.5	
511	300		1	
340	200		1.5	
170	100		3	
10.2	6		50	
5.1	3		100	
1.7	1		300	
1.02	0.6		500	
0.51	0.3		1000	
0.17	0.1		3000	
0.10	0.06		5000	

J.3.4 Data Interpretation

In Figure J.2, the solid parallel lines between the 0.17 s^{-1} and 1.7 s^{-1} represent the upper and lower bounds of preferred viscosity to exhibit minimum sag potential. Values above the lower limit line indicate the fluid is viscous enough to prevent dynamic weight-material sag under typical drilling conditions, while values below indicate a high potential for sag. Values below the upper limit line indicate that the fluid is not so viscous as to cause other drilling-related problems, while values above the upper limit line may be excessive. Note that the viscosity and shear rate are based on the nominal shear rate calculated for Newtonian fluids. This is consistent with previous publications on this technique. However, the variations arising from the non-

Newtonian behavior of typical invert drilling fluids should result in relatively small deviations from these nominal values.

Interpretation of data from rheological testing should be made in the context of the specific fluid being tested [16]. It is easy to generalize from the rheological behavior of a particular fluid system and attempt application to other systems. If the basic characteristics of the system differ, i.e. different weight materials or different formulations & products, the conclusions of one system may not apply to the other.
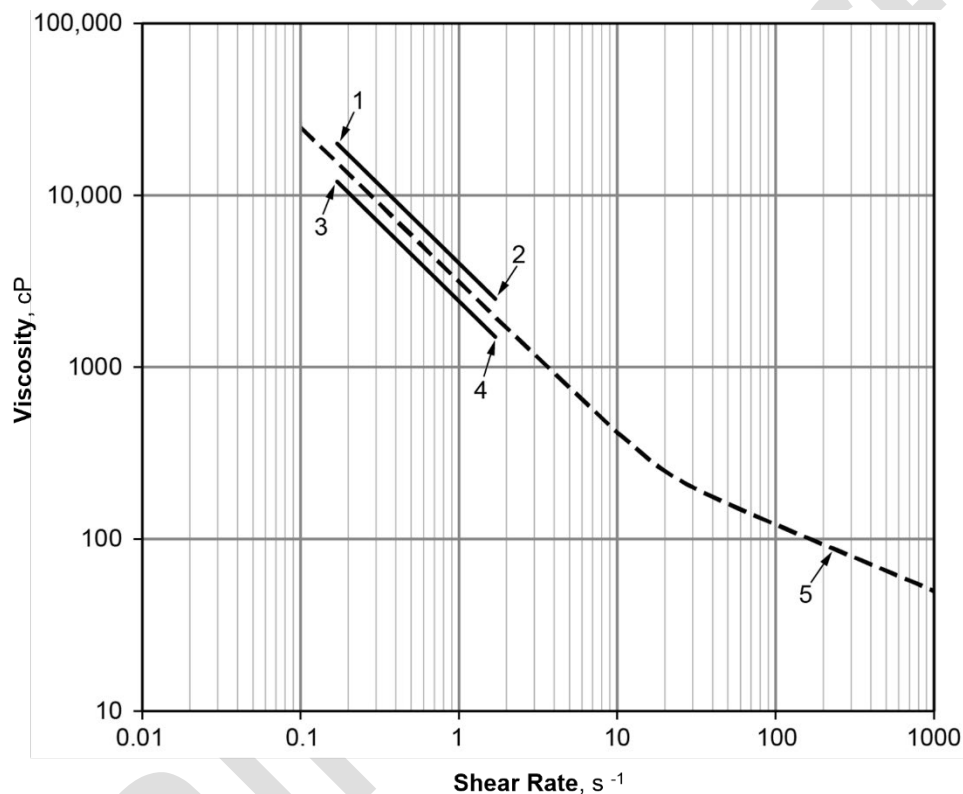
Key

1	20,000 cP at 0.17 s^{-1}	3	12,000 cP at 0.17 s^{-1}
2	2500 cP at 1.7 s^{-1}	4	1500 cP at 1.7 s^{-1}
5	Viscosity curve for a drilling fluid with minimal expected weight-material sag tendency		
NOTE	See Reference [15] in the bibliography		

Figure J.2—Published "Sag Window" for Drilling Fluids Versus Shear Rate

J.3.5 Advanced Testing and Sag Validation—Profiled Rotor and Bob Method

The use of a profiled bob and rotor can be recommended for evaluating fluids for Dynamic Sag. The rotor/bob are identical to the standard but with the addition of shallow grooves cut into the bob and inner wall of the rotor. These grooves minimize wall slip, allowing a more accurate measurement. There is no standard slot size for profiled rotors and bobs. Figure J.3 shows an example of one manufacturer's profiled rotor and bob.

Profiled Rotor   Profiled Bob
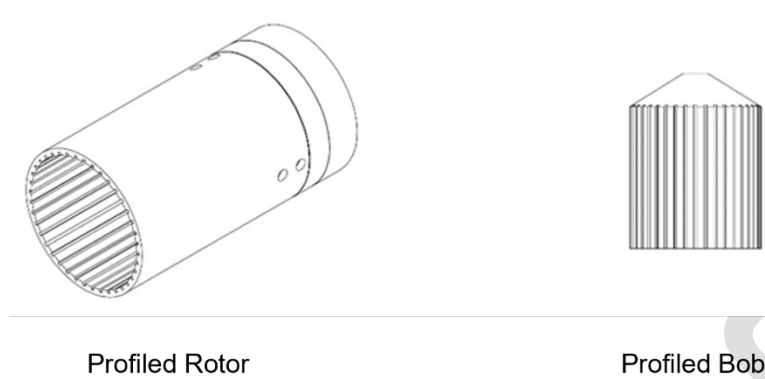
Figure J.3—Example Profiled Rotor and Bob

The data shown in Figure J.4 is from a fluid tested with standard and profiled bob/rotor which exhibits wall slip. The fluid with the standard bob and sleeve experienced wall slip in the test, the viscosity curve fell sharply out of the window and gave the false indication of the potential for sag. When tested using the profiled bob and rotor, the effects of wall slip were minimized and a more accurate comparison to the sag window was made.
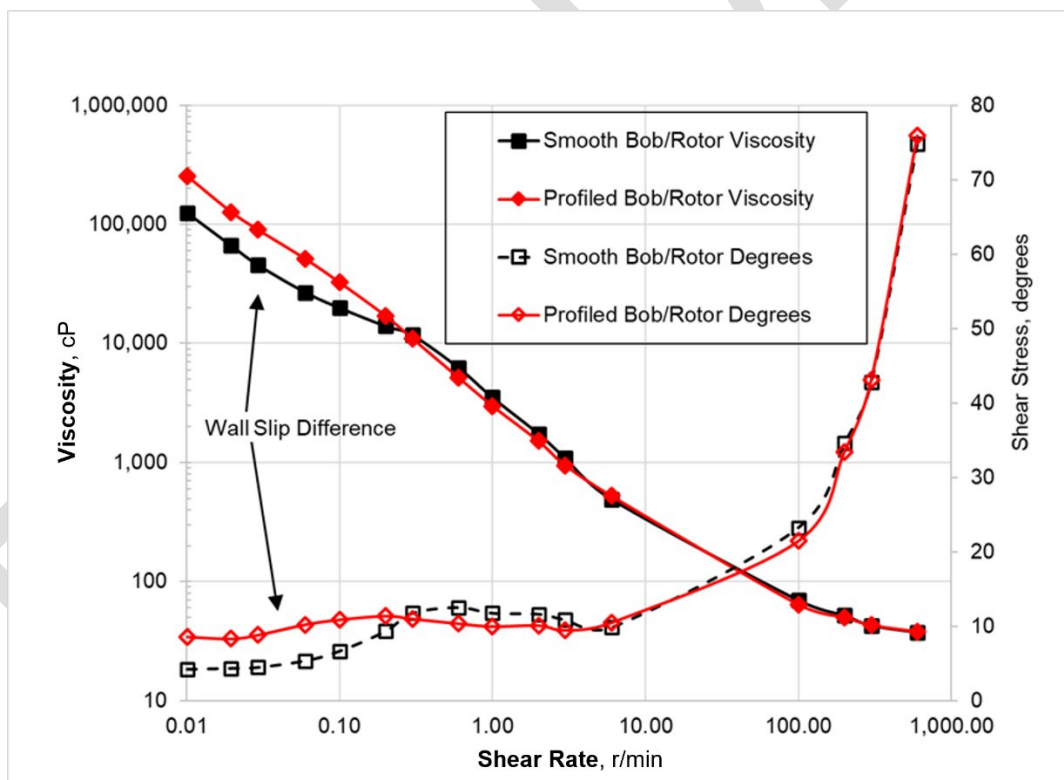
Figure J.4 —Wall slip example from smooth and profiled rotor and bob

J.3.6 Advanced Testing and Sag Validation—Air Bearing Rheometer Method

Rheometers which incorporate air bearings have much lower internal friction compared to ball bearing units and are more accurate for low shear stress measurements. These improved measurements can be used to validate that a fluid's viscosity is within the dynamic sag window more accurately.

J.3.6.1 Apparatus

J.3.6.1.1 Air bearing rheometer capable of measuring at least micro newton-meter torque for the sag window values accurately.

J.3.6.1.2 Measuring geometry fixture appropriate for the sag window shear stress and shear rate ranges.

J.3.6.1.3 Mixer, to operate at 10,000 r/min to 15,000 r/min with a single sine-wave impeller of approximately 25.4 mm (1 in.) in diameter.

J.3.6.1.4 Container, for mixing, 180 mm deep (7.125 in.), diameter 97 mm (3.75 in.) at top and 70 mm (2.75 in.) at bottom.

J.3.6.2 Procedure

This procedure should describe how to perform the measurements for the Dynamic Sag Window using an air bearing rheometer.

For detailed information on operation, motor adjustment, rotational mapping and maintenance of the rheometer, refer to the rheometer manufacturer's instructions. Consult manufacturers' recommendations for equipment and software test preparation procedures.

a)   Inspect air pressure, power, chiller (If available) and instrument level according to the manufacturers criteria.

b)   Inspect the rheometer coupling and measuring geometry fixture to ensure they are clean, dry, and grease free. Clean and dry if necessary.

c)   Check measuring geometry for abrasion or other physical damage and replace if damaged.

d)   Turn on the rheometer and allow the instrument software to boot and initialize

e)   Turn on external accessories, if utilized (external temperature controller and heater/cooler for instance).

f)   Connect desired geometry as per manufacturer's instructions. Ensure that installation and configuration for setup is correct.

   NOTE   Different geometries can be used, each with its' own advantages and disadvantages. For these very low shear measurements, if available, a profiled geometry is recommended to minimize the wall slip phenomena and obtain more accurate measurements.

g)   Perform motor adjustment/rotational mapping as specified by manufacturer.

h)   Pre-Mix the fluid to be tested on the mixer for a minimum of 5 minutes to homogenize.

Note   Two of the most crucial aspects for the accuracy and reproducibility of rheological measurement are the sample amount and how it is handled during loading. Prior to the measurement, it is essential that all samples be treated in a similar manner.

i)   Load the sample. Required sample volume and loading procedure can be found at respective manufacturer's software/guideline/manual.

j)   Bring the measuring geometry to the measuring position as per manual.

k)   Set the desired temperature and allow the sample to reach the desired temperature.

l)   Setup the test method as per manufacture's instruction.

m)   Record the shear stress readings or viscosity displayed from the rheometer software in Table J.2

Table J.2—Air Bearing Rheometer Dynamic sag window data table

Shear Rate sec^{-1}	Shear Stress Pa	Viscosity cP (mPa•s)
1022 (equivalent to 600 r/min)		
511 (equivalent to 300 r/min)		
340 (equivalent to 200 r/min)		
170 (equivalent to 100 r/min)		
10.2 (equivalent to 6 r/min)		
5.11 (equivalent to 3 r/min)		
1.7 (equivalent to 1 r/min)		
1.02 (equivalent to 0.6 r/min)		
0.51 (equivalent to 0.3 r/min)		
0.17 (equivalent to 0.1 r/min)		
0.102 (equivalent to 0.06 r/min)		

n)   After test been completed, cool and clean measuring geometries

i)   Plot the viscosity in cP vs shear rate in s^{-1} data on a log-log format as shown in Figure J.2 and determine if the measure data fall between the 0.17 s^{-1} and 1.7 s^{-1} dynamic sag window upper and lower viscosity bounds

The upper bound is defined by the solid line defined by the points 0.17 s^{-1}/20,000 cP and 1.7 s^{-1}/2500 cP, and the lower bound by the solid line defined by the points 0.17 s^{-1}/12,000 cP and 1.7 s^{-1}/1500 cP (see Figure J.2).

j)   Follow the dynamic sag window data interpretation information in J.3.4

Annex K
(informative)

Solids Calculations

K.1 Principle

The example calculations in this annex are intended to show how the equations in Section 9 are used to determine the amount of solids present in a water-based drilling fluid containing barite as the weighting material. Two different cases are presented:

— Drilling Fluid A: the drilling fluid contains freshwater with minimal chlorides from NaCl salt;

— Drilling Fluid B: the drilling fluid contains NaCl salt.

Physical and chemical properties for both drilling fluids are presented in Table K.1.The gravimetric procedure calculations are shown for each fluid. Since the volumetric calculations use the water volume fraction and oil (NAF) volume fraction from section 9.41 Equations (13) and (14), the calculations are the same starting at K.3.8 for the freshwater example and at K.4.8 for the NaCl salt example.

K.2 Test Data Used in Examples

Table K.1—Physical and Chemical Properties of Examples of Water-based Drilling Fluids

Property	Drilling Fluid A Freshwater	Drilling Fluid B NaCl Salt
Drilling fluid density (ρ_{df}), g/mL	1.70	1.50
Empty retort assembly (m_1), g	878.23	858.65
WBDF fluid and retort assembly (m_2), g	963.18	933.42
Empty glass receiver tube (m_3), g	45.34	45.74
Condensed liquid and receiver tube (m_4), g	80.92	82.07
Solids and retort assembly (m_5), g	926.45	895.35
Total condensed fluid (V_L), mL	36.75	38.5
Volume oil (NAF) (V_O), mL	0.00	2.00
Volume fraction water (φ_W), % volume	73.5	73.0
Volume fraction oil (NAF) (φ_O), % volume	0.0	4.0
Chloride ion concentration (c_{Cl}), mg/L	1200	140,000
Oil density (NAF) (ρ_O), g/mL	0.84	0.84
Low-gravity solids density (ρ_{LG}), g/mL	2.65	2.65
Weighting material density (ρ_{WM}), mg/L	4.10	4.10

K.3 Water-based Fluid Containing Freshwater Example (Drilling Fluid A)

K.3.1 Mass of the drilling fluid sample [see 9.6.2, Equation (27)]

$$m_{df} = 963.18 - 878.23 = 84.95 \text{ g}$$

K.3.2 Mass of the dried retort solids [see 9.6.3, Equation (28)]

$$m_{ds} = 926.45 - 878.23 = 22 \text{ g}$$

K.3.3 Mass of condensed liquids [see 9.6.4, Equation (29)]

$$m_L = 80.92 - 45.34 = 35.58 \text{ g}$$

K.3.4 Mass balance factor (see 9.6.5, Equation (30)]

$$0.97 \leq \frac{48.22 + 35.58}{84.95} = 0.99 \leq 1.03 \quad \text{ACCEPTABLE}$$

k.3.5 Volume of drilling fluid retorted [see 9.7.1, Equation (31)]

$$V_{df} = \frac{84.95}{1.70} = 49.97 \text{ mL}$$

K.3.6 Volume fraction of oil (NAF) [see 9.7.1, Equation (32)]

$$V_O = 0 \text{ mL}$$

$$\varphi_O = 100 \times \frac{0.0}{49.97} = 0 \text{ \%}$$

K.3.7 Volume fraction of water [see 9.7.1, Equation (33)]

$$\varphi_W = 100 \times \frac{\left[84.95 - 48.22 - (0 \times 0.84) \right]}{49.97} = 73.5\%$$

K.3.8 Volume fraction of dried retort solids [see 9.4.1, (Equation (15)]

$$\varphi_{ds} = 100 - (73.5 + 0) = 26.5 \text{ \%}$$

K.3.9 Drilling fluid suspended solids volume fraction [see 9.4.2.1 Equation (16)]

$$\varphi_{SS} = 26.5 - \frac{(73.5 \times 1200)}{\left[\left(1.68 \times 10^6 \right) - (1.21 \times 1200) \right]} = 26.45\%$$

K.3.10 Density of the filtrate [see 9.4.2.2, Equation (17)]

$$\rho_f = 1 + (1.09 \times 10^{-6}) \times 1200 = 1.00 \text{ g/mL}$$

K.3.11   Volume fraction of low-gravity solids [see 9.4.2.2, Equation (18)]

$$\varphi_{LG} = \frac{1}{4.10-2.65}\left[(100 \times 1) + (4.10-1) \times 26.45 - (100 \times 1.70) - (1.00-0.84) \times 0.0\right] = 8.27\,\%$$

K.3.12   Drilling fluid weighting-material volume fraction [see 9.4.2.3, Equation (19)]

φ_{WM} = 26.45 - 8.27 = 18.18 %

K.3.13   Drilling fluid solids concentrations (suspended, low-gravity, weighting material) [see 9.4.4, Equation (21) through Equation (26)]

Concentration expressed in kg/m³:

Low gravity solids: $c_{LG,SI}$ = 10 x 2.65 x 8.27 = 219.1 kg/m³

Weighting material: $c_{WM,SI}$ = 10 x 4.10 x 18.18 = 754.4 kg/m³

Total suspended solids: $c_{SS,SI}$ = 219.1 + 754.4 = 964.5 kg/m³

Concentration expressed in lb/bbl:

Low gravity solids: $c_{LG,USC}$ = 3.50 x 2.65 x 8.27 = 76.7 lb/bbl

Weighting material: $c_{WM,USC}$ = 3.50 x 4.10 x 18.18 = 260.9 lb/bbl

Total suspended solids: $c_{SS,USC}$ = 76.7 + 260.9 = 337.6 lb/bbl

K.4   Water-based Fluid Containing NaCl Salt Example (Drilling Fluid B)

K.4.1   Mass of the drilling fluid sample [see 9.6.2, Equation (27)]

m_{df} = 933.42 – 858.65 = 74.77 g

K.4.2   Mass of the dried retort solids [see 9.6.3, Equation (28)]

m_{ds} = 895.35 – 858.65 = 36.70 g

K.4.3   Mass of condensed liquids [see 9.6.4, Equation (29)]

m_L = 82.07 – 45.74 = 36.33 g

K.4.4   Mass balance factor [see 9.6.5, Equation (30)]

$$0.97 \leq \frac{36.70+36.33}{74.77} = 0.98 \leq 1.03 \quad \text{ACCEPTABLE}$$

K.4.5   Volume of drilling fluid retorted [see 9.7.1, Equation (31)]

$$V_{df} = \frac{74.77}{1.50} = 49.85 \text{ mL}$$

K.4.6   Volume fraction of oil (NAF) [see 9.7.1, Equation (32)]

$$\varphi_O = 100 \times \frac{2.00}{49.85} = 4.01 \text{ \%}$$

K.4.7   Volume fraction of water [see 9.7.1, Equation (33)]

$$\varphi_W = 100 \times \frac{\left[74.77 - 36.70 - (2.00 \times 0.84)\right]}{49.85} = 73.00 \text{ \%}$$

K.4.8   Volume fraction of dried retort solids [see 9.4.1, Equation (15)]

$$\varphi_{ds} = 100 - (73.00 + 4.01) = 22.99 \text{ \%}$$

K.4.9   Drilling fluid suspended solids volume fraction [see 9.4.2.1 Equation (16)]

$$\varphi_{SS} = 22.99 - \frac{(73 \times 140{,}000)}{\left[\left(1.68 \times 10^6\right) - (1.21 \times 140{,}000)\right]} = 16.22 \text{ \%}$$

K.4.10   Density of the filtrate [see 9.4.2.2, Equation (17)]

$$\rho_f = 1 + (1.09 \times 10^{-6}) \times 140{,}000 = 1.15 \text{ g/mL}$$

K.4.11   Volume fraction of low-gravity solids [see 9.4.2.2, Equation (18)]

$$\varphi_{LG} = \frac{1}{4.10 - 2.65}\left[(100 \times 1.15) + (4.10 - 1.15) \times 16.22 - (100 \times 1.50) - (1.15 - 0.84) \times 4.01\right] = 8.00 \text{ \%}$$

K.4.12   Drilling fluid weighting-material volume fraction [see 9.4.2.3, Equation (19)]

$$\varphi_{WM} = 16.22 - 8.00 = 8.22 \text{ \%}$$

Annex L
(informative)

Abbreviated Analytical Analysis for Water-based Drilling Fluids

Table L.1 is an abbreviation of chemical titrations which shall be applicable to determine methylene blue capacity of drilling fluid (see Section 11), alkalinities of filtrate and drilling fluid (see Section 13), drilling fluid chloride ion content (see Section 14), total hardness as calcium ion (see Section 15), in water-based drilling fluids.

Table L.2 is an abbreviation and interpretation of chemical titrations which shall be applicable to determine calcium magnesium and calcium sulfate (total and excess undisolved) in water-based drilling fluids (see Annex A).

Table L.3 is an abbreviation of the determination of sulfide and carbonate contents of drilling fluid using a Garrett gas train.

Table L.1 —Abbreviated Chemical Titrations for Water-based Drilling Fluids

Alkalinity Tests (See Section 13)	Sample Type	Sample Volume	Dilution	Reagents	Indicator	Titrant	Titration Method	Interpretation	Comments
Filtrate Phenolphthalein Alkalinity (P_f)	Filtrate	1 mL	—	—	Phenolphthalein drops: 2 to 10 Forms pink color	N/50 Sulfuric acid (H_2SO_4) 0.01 mol/L (0.02N)	If pink, add titrant drop-by-drop while stirring until color just disappears.	P_f = titrant volume (relative value of N/50 H_2SO_4, mL)	Using a pH-meter: Endpoint when pH drops at 8.3.
Filtrate Methyl Orange Alkalinity (M_f)	Filtrate	1 mL Use P_f endpoint sample	—	N/50 Sulfuric acid (H_2SO_4) volume used for P_f endpoint	Methyl orange drops: 2 to 3. Forms yellow color.	N/50 Sulfuric acid (H_2SO_4) 0.01 mol/L (0.02N)	Add titrant drop-by-drop while stirring until color changes from yellow to pink.	M_f = titrant volume (Including P_f volume relative value of N/50 H_2SO_4, in mL)	Using a pH-meter: Endpoint when pH drops at 4.3.
Drilling Fluid Phenolphthalein Alkalinity (P_{df})	Drilling fluid	1 mL	25 to 50 mL deionized water	—	Phenolphthalein drops : 4 to 5 Forms pink color	N/50 Sulfuric acid (H_2SO_4) 0.01 mol/L (0.02N)	If pink, add titrant drop-by-drop while stirring until color just disappears.	P_{df} = titrant volume (relative value of N/50 H_2SO_4, mL)	Using a pH-meter: Endpoint when pH drops at 8.3.

Chlorides (Cl^-) (See Section 14)	Sample Type	Sample Volume	Dilution	Reagent		Indicator		Titrant	
	Filtrate	1 mL	deionized water	N/50 Sulfuric acid (H_2SO_4) 0.01 mol/L (0.02N)		Potassium chromate : drops: 5 to 10 Forms yellow color.		Silver nitrate ($AgNO_3$) 0.0282 mol/L	
	Titration Method				**Interpretation**			**Comment**	
	a) Add phenolphthalein (2 to 3 drops). If turn pink add N/50 H_2SO_4 acid till colors disappears (volume P_f endpoint requirement) . b) Dilute with deionized water 25 mL to 50 mL c) Add Titrant drop by drop while stirring until endpoint : color changes from yellow to orange red, persists for > 30 s.				Chloride ion concentration (mg/L) : $$c_{Cl} = 1000 \times \frac{V_{SN1X}}{V_s}$$ V_{SN1X} volume titrant (0.0282 mol/L), (mL) V_s filtrate sample volume of filtrate, (mL)			— If the filtrat sample is originally deeply colored, at item a) add additional 2 mL of 0.02N H_2SO_4 or HNO_3 acid and stir; then add 1g of calcium carbonate, and stir. — If chloride are > 10,000 mg/L, titrate with 0.282 mol/L silver nitrate solution.	

Total Hardness as Ca^{2+} See Section 15	Sample Type	Sample Volume	Dilution Water	Reagent	Indicator	Titrant	[a] **Reagent and Deionized Water Quality Control** Not required when using distilled water.	
	Filtrate	1 mL	50 mL distilled or deionized [a]	2 mL Hardness buffer: NH_4Cl+ NH_4OH	Hardness Indicator ("calmagite") Drops: 2 to 6 Forms red-wine color	EDTA Solution (0.01mol/L)	a) With 50 mL deionized water add 2mL buffer solution + hardness indicator until a blue color forms. Swirl to mix b) A solution with color than distinct blue indicates contamination of equipment and/or unacceptable deionized water. Find and eliminate the source before proceeding to titration .	
	Titration Method				**Interpretation**		**Comments**	
	Add titrant drop-by-drop while stirring until endpoint: Red color changes to blue				Total hardness concentration as Ca^{2+} (mg/L): $$c_{Ca+Mg} = 400 \times \frac{V_{EDTA}}{V_s}$$ V_{EDTA} volume titrant (0.01 mol/L), (mL) V_s filtrate sample volume of filtrate, (mL)		EDTA sol. 1mL/mL1 mL/mL<==>400 mg Ca^{2+} If the filtrat is darkly colored , see 15.4 item b) through item e) to bleach the sample before titration.	

Table L.1 —Abbreviated Chemical Titrations for Water-based Drilling Fluids (continued)
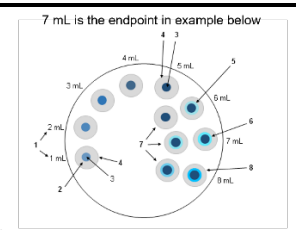
Chemical Titration Calculations: Alkalinity Tests and Chloride Ion Concentration			
Alkalinity Tests calculation See Section 13	From P_f and P_{df} Estimation of concentrations of hydroxyl, carbonate and bicarbonate ions (mg/L) see Table 5.	Estimation of lime content: $c_{lime,SI} = 0.742 \times (P_{df} - F_W P_f)$ (kg/m³) $c_{lime,USC} = 0.26 \times (P_{df} - F_W P_f)$ (lb/bbl)	with F_W volume fraction from retorting test (Section 9) $F_W = 0.01\,\varphi_W$ φ_W water volume fraction in %
Chloride Test Calculation See Section 14	Conversion of chloride ion concentration to sodium chloride (mg/L) $c_{NaCl,SI} = 1.65 \times c_{Cl}$	or, expressed in parts per million by mass (ppm) $c_{NaCl,ppm} = 1.65 \times \left(\dfrac{c_{Cl}}{\rho_f} \right)$	with c_{Cl} chloride ion concentration (mg/L) ρ_f density of filtrate for NaCl (g/mL)

Test	Sample Type	Sample Volume	Dilution	Reagents	Titrant / Indicator	Comments
Methylene Blue Test **(MBT)** Section 11	Drilling fluid	2 mL	10 mL Deionized water	15 mL Hydrogen peroxide (H₂O₂) (3%) + 0.5 mL H₂SO₄ 2.5 mol/L (5N)	Methylene Blue Solution (3.20 g/L)	— Methylene blue dry reagent grade: Check moisture content for each preparation and correct mass to dissolve. — Filter paper Grade 1

Titration Method	Interpretation	Titration example
a) Boil gently 10 min (not to dryness) , then dilute to 50 mL with deionized water. b) Add methylene blue solution in increments of 0.5 mL. c) After each addition, swirl flask 30 sec. d) While the solids are still suspended, using the stirring rod, remove one drop of liquid and place it on filter paper. e) The initial endpoint of the titration is reached when unadsorbed dye appears as a blue or turquoise ring surrounding the dyed solids. f) Whenever a blue tint spreading from the spot is detected, shake/stir the flask for an additional 2 min and place another drop on the filter paper, adjacent to the previous drop. g) If the blue ring is again evident, the final endpoint has been reached, as shown in Figure 1, Key item 7 (7 mL).	Methylene blue capacity (meq/mL): $$C_{MBT} = \frac{V_{mb}}{V_{df}}$$ Bentonite equivalent: $$E_{BE,SI} = \frac{14.27 \times V_{mb}}{V_{df}} \quad (kg/m^3)$$ $$E_{BE,USC} = \frac{5 \times V_{mb}}{V_{df}} \quad (lb/bbl)$$ V_{mb} volume of methylene blue solution (mL) V_{df} sample volume of drilling fluid (mL)	 7 mL is the endpoint in example below **Key** 1 volume methylene blue added 2 no free unadsorbed dye present 3 dyed drilling fluid solids (no free dye present) 4 moisture, no color 5 free unadsorbed dye ring 6 endpoint reached initial 7 endpoint retest after 2min additional 8 Endpoint exceeded

Warning	—Sulfuric acid (H2SO4) And nitic acid (HNO3) are corrosive and toxic acids. —Potassium chromate (K2CRO4), the hexavalent chromium is known to be carcinogenic and should be handled with care. —Hydrogen peroxyde (H2O2) is a strong oxidize, Avoid skin contact for solutions above 3%.

Table L.2 —Abbreviated Chemical Titrations for Calcium, Magnesium and Calcium Sulfate in Water-based Drilling Fluids

Calcium	Sample Type	Sample Volume	Dilution Water	Reagent	Indicator	Titrant	[a] Reagent and Deionized Water Quality Control Not required when using distilled water		
Calcium Ca²⁺ See Annex A.1	Filtrate	1 mL	50 mL distilled or deionized [a]	2 mL Calcium buffer: NaOH 1 mol/L (1N)	Calcium indicator hydroxynaphtanol blue 0.1 g to 0,2 g Forms pink to red color	EDTA Solution (0.01 mol/L) 1 mL/mL<==>400 mg Ca²⁺	a) With 50 mL deionized water add 2mL buffer solution + hardness indicator ("calmagite) until a blue color forms. Swirl to mixb) b) A solution with color than distinct blue indicates contamination or unacceptable deionized water. Find and correct before to proceed to titration .		
	Titration Method					**Interpretation**		**Comments**	
	Add titrant drop-by-drop while stirring until endpoint: Red color changes to blue Endpoint is best described when additional EDTA produces no further red to blue color change.					Calcium concentration (mg/L): $$c_{Ca} = 400 \times \frac{V_{EDTA\text{-}f}}{V_s}$$ $V_{EDTA\text{-}f}$ volume titrant (0.01 mol/L), (mL) V_s filtrate sample volume of filtrate, (mL)		— If the filtrat is darkly colored , see A.1.4 item b) through item e) to bleach the sample before titration. — Visibility of Endpoint can be improve by addition of several drops of methyl orange along with the calcium indicator	

Magnesium (Mg²⁺) See Annex A.2	**Principle**	Magnesium content in filtrate is calculated by substracting the calcium ion content from total hardness. That give magnesium content in terms of calcium to be converted in Magnesium by multtiplying the value by the ratio of atomic weight, i.e. 24.3/40.1 = 0.606.	
	Titration Method	**Interpertation**	
	a) Determine total hardness as calcium ion (section 15) using "Calmagite" as hardness indicator. b) Determine calcium ion (Annex A.1) using hydroxynaphthol blue as calcium indicator.	Magnesium concentration (mg/L): $$c_{Mg} = 0.606 \times \left(c_{Ca+Mg} - c_{Ca} \right)$$	With c_{Ca+Mg} total hardness as Ca²⁺ of filtrate (mg/L) c_{Ca} calcium ion concentration of filtrate (mg/L)

Warning	**—Sodium hydroxyde (NaOH) is a strong caustic chemical. Avoid skin contact.**

	Principle						Calcium sulfate is determined from total calcium concentration in the whole drilling fluid after a dilution into water to solubilize insoluble (excess) calcium sulfate. Calcium concentration in filtrate is related to soluble calcium sulfate (Annex A.1).
Calcium Sulfate CaSO4 See Annex A.3	**Sample Type**	**Sample Volume**	**Dilution Water**	**Reagent**	**Indicator**	**Titrant**	**[a] Reagent and Deionized Water Quality Control** Not required when using distilled water.
	filtrat	1mL	50 mL Distilled or deionized [a]	2 mL Hardness buffer: (NH_4Cl + NH_4OH)	Calcium Indicator hydroxynaphthol blue 0.1g to 0.2 g Forms red-wine color	EDTA Solution (0.01 mol/L)	a) With 50 mL deionized water add 2mL buffer solution + hardness indicator ("calmagite) until a blue color forms. Swirl to mix
	Drilling Fluid	Drilling fluid 5 mL Filtrat 5 mL	245 mL Distilled or deionized [a]				b) A solution with color than distinct blue indicates contamination or unacceptable deionized water. Find and correct before to proceed to titration .
	Titration Method				**Interpretation**		**Comments**

	a) Titrate Ca^{2+} in filtrate (see A.1) to be converted into Soluble $CaSO_4$.
	 b) Titrate Ca^{2+} in diluted whole drilling fluid to be converted into total $CaSO_4$.
	   1) Diluted 5 mL of whole drilling fluid into 245 mL distilled water. Stir 15 min and prepare a filtrat from a Low-pressure, low temperature filtration test (see 8.2).
	   2) Dilute 5 mL of filtrate to 250 mL (add 245 mL distilled water).
	   3) Titrate 10 mL of clear filtrate with EDTA to end point as per procedure used for Ca^{2+} titration (see A.1)
	   4) Obtain/ calculate F_W volume fraction from retorting test (Section 9)
	   $F_W = 0.01\,\varphi_W$ (φ_W water volume fraction in percent)
					Total $CaSO4$ in drilling fluid in kg/m3 (SI) or lb/bbl (USC):
					 $c_{CaSO_4,SI} = 6.80 \times V_{EDTA\text{-}df}$ $c_{CaSO_4,USC} = 2.38 \times V_{EDTA\text{-}df}$
					 Undisolved $CaSO_4$ in drilling fluid in kg/m3 (SI) or lb/bbl (USC):
					 $c_{ex\text{-}CaSO_4,SI} = 6.80 \times V_{EDTA\text{-}df} - 1.36 \times V_{EDTA\text{-}f} \times F_W$
					 $c_{ex\text{-}CaSO_4,USC} = 2.38 \times V_{EDTA\text{-}df} - 0.477 \times V_{EDTA\text{-}f} \times F_W$
					 $V_{EDTA\text{-}df}$ volume of EDTA used on filtrat (0.01 mol/L), (mL)
					 $V_{EDTA\text{-}df}$ volume of EDTA used on diluted whole drilling fluid filtrat
							If the filtrat is darkly colored , see 15.4 item b) through item e) to bleach the sample before titration.

Annex K
(informative)

Water-based Drilling Fluids Daily Report

Example

WATER-BASED DRILLING FLUID DAILY REPORT

Date: Report No.

Operator :	Well Name :
Oper. Rep :	Country:
Contractor:	State/County:
Contr. Rep:	Field/Area :
Rig :	Water Depth :
Location:	API Well Number:

Measured Depth:
TVD:
Present Activity :
Fluid Type :
Spud Date :
Interval:

DRILL STRING

Description	OD (in.)	ID (in.)	Length (ft)

BIT

Size (in.)	
Type	
Bit No.	
TFA (in.²)	
Jet Impact F	
Bit HHSI	
Jets (1/32 in.)	

CIRCULATING VOLUMES & TIMES

	Hole	Active Pits
Total Circulating Volume		
Depth Drilled Last 24 hours		
Volume Drilled Last 24 hours		

	Surf-Bit
Bbls	
Stks	
Min	
Bit-Surf	
Bbls	
Stks	
Min	

CIRCULATION DATA

Pump No.	1	2
Model		
Liner (in.)		
Stroke (in.)		
Efficiency (%)		
gal/stk		
stks/min		
gal/min		

SPP psi
3	4

CASING

Description	OD (in)	ID (in)	Top (ft)	Shoe (ft)

RESERVE VOLUMES

Pit # & Type	Bbls	Density

DRILLING INFO

WOB	klbs	
Surface	RPM	
Motor	RPM	
Total	RPM	
Torque	kft/lbs	

@ MD (ft)			Hours
S/O wt	klbs	ROP	ft/hr
P/U wt	klbs	Sliding	hr
Inclination	°	Sliding	ft
Azimuth	°	Rotating	hr
Survey Depth	ft	Rotating	ft

RHEOLOGY & HYDRAULICS

RISER (Boost Rate: gal/min)

		@ MD (ft)		Hours
n		AV Riser	ft/min	psi
k		AV Pipe/OH	ft/min	Bit Press Loss
Yield Stress		AV DC/OH	ft/min	%
ECD @ Shoe	lb/gal	Critical AV: Turb / Lam		Bit HHP
ECD @ TD	lb/gal	Pipe/OH	ft/min	Jet Vel. ft/min
MPD Pressure	psi	DC/OH	ft/min	

FLUID SPECIFICATIONS

LAST FIT or LOT				
ft	PV		ES	
psi	Density		YP	WPS
EMW	HTHP		Gels	LGS

DRILLING FLUID PROPERTIES

Measured Depth	ft				
Total Vertical Depth	ft				
Sample Source & Time					
Drlg Fld Density IN	lb/gal @ °F	/			
Drlg Fld Density OUT	lb/gal @ °F	/			
Funnel Viscosity	sec/qt				
Rheology Temp	°F				
Plastic Viscosity	cP				
Yield Point	lb/100 ft²				
Gel Strengths 10s/10m/30m	lb/100 ft²	/ /	/ /	/ /	/ /
API Filtrate	mL/30 min				
HTHP Filtrate	mL/30 min @ °F	@	@	@	@
HTHP Pressure	psi				
Filter Cake API / HPHT	1/32 in.	/	/	/	/
Retort Solids	% vol				
Retort Water	% vol				
Retort Oil (NAF)	% vol				
Corrected Solids	% vol				
Sand Content	% vol				
Methylene Blue Capacity	lb/bbl equiv				
pH					
Drilling Fluid Alkalinity (Pm)	mL 0.02 N H₂SO₄				
Filtrate Alkalinity (Pf / Mf)	mL 0.02 N H₂SO₄	/	/	/	/
Chlorides, Filtrate	mg/L				
Total Hardness (as Ca++), Filt	mg/L				
Calcium (Ca++), Filtrate	mg/L				
Magnesium (Mg++), Filtrate	mg/L				
Excess Lime	lb/bbl				
Inverval Weighted LGS	%				
Cummulative Solids Removal Eff	%				
Cummulative Interval Volume	bbl				
Cummulative Dilution Volume	bbl				

VISCOMETER DIAL READINGS @ ____°F	600 / 300	/	/	/	/
	200 / 100	/	/	/	/
	60 / 30	/	/	/	/
	6 / 3	/	/	/	/

SOLIDS CONTROL EQUIPMENT

	Shaker 1	Shaker 2	Shaker 3	Shaker 4	Drying Shakers
Make					
Model					
API No. Screens					
Oper. Hours					
ROC wt %					

	De-Sander	De-Silter	Mud Cleaner	Centrifuge 1	Centrifuge 2
Model					
Size / Speed					
gpm @ lb/gal					
UF / OF lb/gal	/	/	/	/	/
Oper Hours					

HAUL OFF

No of Loads	
Est. Volume	
Bbls	
Est. Weight	
klbs	
Avg. Makeup	
MCR	vol/vol
Base	% vol
Oil	% vol
Slds	% vol

TIME DISTRIBUTION

Drilling	hr
Reaming	hr
Circulating	hr
POOH	hr
Survey	hr
NPT	hr
On Bottom	hr
Cumul. On Bottom	hr

SOLIDS ANALYSIS

Salt		wt %
Salt		lb/bbl
LGS vol %		%
LGS lb/bbl		g/mL
HGS vol %		%
HGS lb/bbl		lb/bbl
Daily SRE		%
		%
% Base Clean		%

FLUID VOLUME ACCOUNTING

INITIAL VOLUME:	
ADDITIONS:	
Base Fluid	
Clean DF Dilution	
Barite	
DF Products	
TOTAL ADDITIONS:	
LOSSES & TRANSFERS:	
Lost on Cuttings	
Transferred (out)	
Dumped volume	
Downhole Losses	
Left behind casing	
TOTAL LOSSES:	
FINAL SYSTEM VOL:	

FLUID COMMENTS & RIG ACTIVITIES

PRODUCTS USED LAST 24 HRS

Product	Unit Size	Used	On Hand	Conc. lb/bbl	Cost

DRILLING FLUID SUPPLIER CONTACT INFORMATION

DF Eng. Days	
Phone	
Email	
DF Eng. Nights	
Phone	
Email	
Rig Phone	
Warehouse	
Office Phone	

NAF Base	Name / Type	Density	Flash Point °F
	Max DF Density with Barite on Hand		lb/gal

TOURLY TREATMENT | COST INFORMATION

Product Cost	
Equip. Cost	
Eng. Cost	
Tax %	
Daily Cost	
Cumul. Cost	

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