Recommended Practice for Field Testing Water-based Drilling Fluids

ANSI/API RECOMMENDED PRACTICE 13B-1
FIFTH EDITION, XXXX 2017
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Suggested revisions are invited and should be submitted to the Standards Department, API, 1220 L Street NW, Washington, DC 20005, standards@api.org.
Introduction

This standard is based on API Recommended Practice 13B-1, Recommended Practice for Field Testing of Water-based Drilling Fluids, Fourth Edition dated March 2009.

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, First Edition, June 2007 (Editorial Revision, November 2017). This numbering format is different than that used in API 13B-1, Fourth Edition. 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).
Field Testing of Water-based Drilling Fluids

Caution—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) 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.

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

- chemical analysis for calcium, magnesium, calcium sulfate, sulfide, carbonate and potassium;
- determination of shear strength;
- determination of resistivity;
- removal of air;
- drill-pipe corrosion monitoring;
- sampling, inspection and rejection;
- rig-site sampling;
- calibration and verification of glassware, thermometers, timers, viscometers, retort-kit cup and drilling fluid balances;
- permeability-plugging testing at high temperature and high pressure for two types of equipment;
- SAG testing;
- example of a report form for water-based drilling fluid.
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 RP 13B-2, Field Testing Oil-Based Drilling Fluids

API RP 13C, Drilling Fluids Processing Systems Evaluation

API RP 13D, Rheology and Hydraulics of Oil-well Drilling Fluids

3 Terms and Definitions

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

3.1 ACS reagent grade
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 measured mass of drilled solids, expressed as an equivalent expressed in pound of bentonite per barrel of drilling fluid (lb/bbl).

3.3 density of water
the density of 0.999 g/ml (8.334 lb/gal) for de-ionized or distilled water and 1 g/ml (8.345 lb/gal) for clean tap water.

NOTE De-ionized or distilled water is used for equipment calibration. 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.

3.4 darcy
the permeability of a porous medium, where one darcy is the flow of a single-phase fluid of 1 cP viscosity that completely fills the voids of the porous medium, flowing through the medium under conditions of viscous flow at a rate of 1 mL/s•cm² cross-sectional area and under a pressure or equivalent hydraulic gradient of 1 atm•cm⁻¹.

NOTE Darcy is not a SI unit. In SI units, permeability is expressed in square meters, 1 Darcy = 9.869 x 10⁻¹³ m².

NOTE 1 cP = 1 mPa•s

NOTE The atmosphere (atm) is neither a SI nor an USC unit of pressure. Standard atmosphere was defined (CGPM 1954) as being equal to 101,325 Pa (14.696 lbf/in²), this value as intended to represent the mean atmospheric pressure at mean sea level at the latitude of Paris, France.

3.5 drill solids
solids 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
a verb: mix and divide a sample into four specimens to ensure homogeneity of specimens.

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 consisting 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 Subscript “SI” to symbol denotes metric units. Subscript “USC” to symbol denotes U.S. customary units.

- **D** outer pipe diameter, expressed in inches
- **E** pump efficiency, expressed as a percentage
- **K** cell constant, expressed in squared meters per meter
- **L** length of the hydraulic section, expressed in feet
- **P** pressure (heating or backpressure), expressed in kilopascals (pounds-force per square inch)
- **Q** pump flow rate, expressed in gallons per minute
- **R** temperature reading for the working thermometer, expressed in degrees Celsius (degrees Fahrenheit)
- **S** SAG register
- **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
- **C_{MBT}** methylene blue capacity of drilling fluid, expressed in milliequivalents per liter
- **C_{MBT-B}** methylene blue capacity of commercial bentonite, expressed in milliequivalents per 100 grams
- **D_{TVD}** true vertical depth, expressed in feet
- **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
- **P_{USC}** measured pressure, expressed in pounds-force per square inch
- **R_{BPU}** calculated bed pickup measurement ratio, expressed as a percentage
$R_{QAS/STPB}$  ratio of the concentration of QAS to that of STPB

$V_{EDTA}$  titration volume of EDTA solution, expressed in milliliters

$V_{EDTA,df}$  titration volume of EDTA solution of whole drilling fluid, expressed in milliliters

$V_{EDTA,f}$  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

$V_O$  volume of oil, expressed in milliliters

$V_{PPT}$  PPT volume, expressed in milliliters

$V_{QAS}$  volume of quaternary ammonium salt (QAS), expressed in milliliters

$V_{RC}$  retort cup volume, expressed in milliliters

$V_{USC}$  annular volume, expressed in barrels

$V_W$  volume of condensed water, expressed in milliliters, or mass of condensed water, expressed in grams ($1 \text{ ml} = 1 \text{ g}$)

$Y_{P,SI}$  yield point, expressed in pascals

$Y_{P,USC}$  yield point, expressed in pounds-force per one hundred square feet

$C_{th}$  thermometer correction factor to be added to the working thermometer reading, expressed in degrees Celsius (degrees Fahrenheit)

$M_f$  methyl orange alkalinity of the filtrate

$P_{df}$  phenolphthalein alkalinity of the drilling fluid

$P_f$  phenolphthalein alkalinity of the filtrate

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

$R_r$  resistivity-meter reading, expressed in ohms

$V_{df}$  volume of whole drilling fluid sample, expressed in milliliters

$V_f$  volume of the filtrate or filtrate test sample, expressed in milliliters

$V_m$  receiver volume at a specified graduation mark, expressed in milliliters

$V_{mb}$  volume of methylene blue solution, expressed in milliliters

$V_{sn}$  volume of silver nitrate solution, expressed in milliliters

$R_1$  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

$V_1$ spurt loss, 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

$G'$ storage modulus, expressed in pascals ($= \text{N/m}^2$)

$G''$ loss modulus, expressed in pascals ($= \text{N/m}^2$)

$b$ slope of the annular velocity and shear stress at the wall in laminar flow (see K.7.2.8)

d inner pipe diameter, expressed in inches

$f$ tube factor from either Table A.1 for sulfide or Table A.2, for carbonate

$t$ lagged time of sample, expressed in hours

$c_{Ca}$ concentration of calcium ion, expressed in milligrams per liter

$c_{Ca+Mg}$ concentration of calcium and magnesium ions (total hardness), expressed 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

$c_{K,I\text{-VQAS}}$ concentration of potassium ion in filtrate (QAS titration), expressed in milligrams per liter

$c_{K,I}$ concentration of potassium ion in filtrate, expressed in milligrams per liter

$c_{KCl,SI}$ concentration of potassium chloride in diluted filtrate test sample, expressed in kilograms per cubic meter

$c_{KCl,USC}$ concentration of potassium chloride in diluted filtrate test sample, expressed in pounds per barrel

$c_{KCl,I\text{-SI}}$ concentration of potassium chloride in filtrate, expressed in kilograms per cubic meter

$c_{KCl,I\text{-USC}}$ 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
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$c_S$</td>
<td>concentration of sulfide ion, expressed in milligrams per liter</td>
</tr>
<tr>
<td>$c_{SS,SI}$</td>
<td>concentration of suspended solids, expressed in kilograms per cubic meter</td>
</tr>
<tr>
<td>$c_{SS,USC}$</td>
<td>concentration of suspended solids, expressed in pounds per barrel</td>
</tr>
<tr>
<td>$c_{WM,SI}$</td>
<td>concentration of weighting material, expressed in kilograms per cubic meter</td>
</tr>
<tr>
<td>$c_{WM,USC}$</td>
<td>concentration of weighting material, expressed in pounds per barrel</td>
</tr>
<tr>
<td>$k_C$</td>
<td>consistency factor, expressed in pounds-force-second per one hundred square feet</td>
</tr>
<tr>
<td>$l_{SI}$</td>
<td>submerged length of shear tube, expressed in centimeters</td>
</tr>
<tr>
<td>$l_{USC}$</td>
<td>submerged length of shear tube, expressed in inches</td>
</tr>
<tr>
<td>$m_B$</td>
<td>mass of commercial bentonite, expressed in grams</td>
</tr>
<tr>
<td>$m_{F1}$</td>
<td>initial mass of the 10 ml-sample of drilling fluid (plus syringe), expressed in grams</td>
</tr>
<tr>
<td>$m_{F2}$</td>
<td>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</td>
</tr>
<tr>
<td>$m_{F3}$</td>
<td>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</td>
</tr>
<tr>
<td>$m_L$</td>
<td>mass of the liquid condensed (oil and water), expressed in grams</td>
</tr>
<tr>
<td>$m_O$</td>
<td>mass of the oil, expressed in grams</td>
</tr>
<tr>
<td>$m_W$</td>
<td>mass of water, expressed in grams</td>
</tr>
<tr>
<td>$q_{SI}$</td>
<td>corrosion rate, expressed in kilograms per square meter-year</td>
</tr>
<tr>
<td>$q_{USC}$</td>
<td>corrosion rate, expressed in pounds per square foot-year</td>
</tr>
<tr>
<td>$c_{ex-CaSO_4,SI}$</td>
<td>concentration of excess, undissolved calcium sulfate, expressed in kilograms per cubic meter</td>
</tr>
<tr>
<td>$c_{ex-CaSO_4,USC}$</td>
<td>concentration of excess, undissolved calcium sulfate, expressed in pounds per barrel</td>
</tr>
<tr>
<td>$c_{lime,SI}$</td>
<td>lime content of the drilling fluid, expressed in kilograms per cubic meter</td>
</tr>
<tr>
<td>$c_{lime,USC}$</td>
<td>lime content of the drilling fluid, expressed in pounds per barrel</td>
</tr>
<tr>
<td>$k_{cor}$</td>
<td>correction factor</td>
</tr>
<tr>
<td>$l_{st}$</td>
<td>gas detection Dräger (or equivalent) tube stain length, expressed in units marked on the tube</td>
</tr>
<tr>
<td>$m_{df}$</td>
<td>mass of the drilling fluid sample, expressed in grams</td>
</tr>
<tr>
<td>$m_{dmb}$</td>
<td>mass of the methylene blue dried sample, expressed in grams</td>
</tr>
<tr>
<td>$m_{ds}$</td>
<td>mass of the dried solids from retort, expressed in grams</td>
</tr>
<tr>
<td>$m_{mb}$</td>
<td>corrected mass of methylene blue powder to be used per liter water, expressed in grams</td>
</tr>
<tr>
<td>$m_{st}$</td>
<td>mass of the shear tube, expressed in grams</td>
</tr>
</tbody>
</table>
$m_{\text{tot}}$  total shear mass (sum of platform and weights), expressed in grams

$r_{df}$  drilling fluid resistivity, expressed in ohm•meters

$r_f$  filtrate resistivity, expressed in ohm•meters

$d_1$  distance from the outer wall, expressed in inches

$m_1$  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

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

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

$\varepsilon$  pipe eccentricity, expressed as a percentage

$\eta$  drill pipe rotation, expressed in revolutions per minute

$\theta$  temperature, expressed in degrees Celsius (degrees Fahrenheit)

$\rho$  drilling fluid relative density (specific gravity)

$\Gamma_{\text{DFG,SI}}$  drilling fluid gradient, expressed in kilopascals per meter

$\Gamma_{\text{DFG,USC}}$  drilling fluid gradient, in pounds per square inch, per foot

$\Delta P_{\text{rot}}$  anticipated pressure increase, expressed in pounds-force per square inch

$\Delta m$  mass loss, expressed in milligrams

$\Delta \rho_{\text{ECD-rot}}$  ECD increase due to drill pipe rotation, expressed in pounds per gallon

$\Delta \rho_{\text{rot}}$  maximum recorded drilling fluid density difference, expressed in pounds per gallon

$\Delta v_{\text{USC}}$  change in annular velocity, expressed in feet per minute

$\gamma_{\text{SI}}$  shear strength, expressed in pascals

$\gamma_{\text{USC}}$  shear strength, expressed in pounds-force per hundred square feet

$\eta_{AV}$  apparent viscosity, expressed in millipascal•seconds (centipoises)

$\eta_{PV}$  plastic viscosity, expressed in millipascal•seconds (centipoises)

$v_{\text{USC}}$  annular velocity, expressed in feet per minute

$\rho_{\text{ECD}}$  measured ECD of the drilling fluid while circulating, expressed in pounds per gallon
\( \rho_{\text{ECD-hyd}} \) ECD of the drilling fluid including ESD, circulating pressure drop and extra density effects of drilled cuttings, expressed in pounds per gallon

\( \rho_{\text{ECD-tot}} \) total predicted ECD of the drilling fluid in the annulus while circulating, expressed in pounds per gallon

\( \rho_{\text{LG}} \) density of low-gravity solids, expressed in grams per milliliter (use 2.6 if unknown)

\( \rho_{\text{O}} \) density of oil, expressed in grams per milliliter (use 0.8 if unknown)

\( \rho_{\text{W}} \) water density at test temperature, expressed in grams per milliliter

\( \rho_{\text{WM}} \) density of weighting material, expressed in grams per milliliter

\( \tau_{y} \) drilling fluid yield stress, expressed in pounds-force per one hundred square feet

\( \tau_{w} \) wall shear stress, expressed in pounds-force per one hundred square feet

\( \tau_{wi} \) wall shear stress at section i, expressed in pounds-force per one hundred square feet

\( \phi_{\text{LG}} \) volume fraction of low-gravity solids, in percent

\( \phi_{\text{ds}} \) volume fraction of dried solids from retort, in percent

\( \phi_{\text{O}} \) volume fraction of oil, expressed as a percentage of the drilling fluid volume

\( \phi_{\text{SS}} \) volume fraction of suspended solids, expressed as a percentage of the drilling fluid volume

\( \phi_{\text{W}} \) volume fraction of water, expressed as a percentage of the drilling fluid volume

\( \phi_{\text{WM}} \) volume fraction of weighting-material solids, expressed as a percentage of the drilling fluid volume

\( \gamma_{i} \) shear rate at section i, expressed in reciprocal seconds

\( v_{\text{sf}} \) static filtration rate (velocity of flow), expressed in milliliters per square root of minute

\( \rho_{\text{df}} \) drilling fluid density, expressed grams per milliliter

\( \rho_{\text{df,SI}} \) drilling fluid density, expressed in kilograms per cubic meter

\( \rho_{\text{df, USC}} \) drilling fluid density, expressed in pounds per gallon

\( \rho_{\text{df, USC2}} \) drilling fluid density, expressed in pounds per cubic foot

\( \rho_{f} \) density of filtrate, expressed in grams per milliliter

\( \rho_{\text{max}} \) maximum recorded density of drilling fluid, expressed in pounds per gallon

\( \rho_{\text{nom}} \) nominal density of drilling fluid, expressed in pounds per gallon

\( \beta_{10s} \) 10-second initial gel strength, expressed in pounds-force per one hundred square feet

\( \beta_{10m} \) 10-minute gel strength, expressed in pounds-force per one hundred square feet
### 4.2 Abbreviations

- **ACS**: American Chemical Society
- **API**: American Petroleum Institute
- **ASTM**: American Society for Testing and Materials
- **BE**: bentonite equivalent
- **CAS**: Chemical Abstracts Service
- **DFG**: drilling fluid gradient
- **DN**: nominal diameter
- **ECD**: equivalent circulating density
- **EDTA**: ethylenediaminetetraacetic acid
- **ERD**: extended reach drilling
- **ESD**: equivalent static density
- **HTHP**: high-temperature, high-pressure
- **LGS**: low-gravity solids
- **MBT**: methylene blue test/capacity
- **meq**: milliequivalents
- **PPA**: permeability plugging apparatus
- **PPE**: Personal Protective Equipment
- **PPT**: permeability plugging test
- **PTFE**: polytetrafluoroethylene
- **PV**: plastic viscosity, in common oilfield terminology
- **QAS**: quaternary ammonium salt
- **RMS**: Root Mean Square
- **RPM**: revolutions per minute
- **STPB**: sodium tetraphenylborate
- **TC**: to contain
- **TD**: to deliver
- **VSST**: viscometer sag shoe test
- **YP**: yield point, in common oilfield terminology

### 5 Drilling Fluid Density (Mud Weight)

#### 5.1 Principle

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

5.2.1 Any ambient pressure density-measuring instrument, having an accuracy of ±0.01 g/ml or ±10 kg/m³ (±0.1 lb/gal or ±0.5 lb/ft³).

The mud balance is the density instrument generally used for drilling fluid density measurements at the rig site. 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 balancing. Attachments for extending the range of the balance may be used when necessary.

The mud balance instrument should be calibrated frequently with fresh water, e.g. bi-weekly or weekly. Fresh water should give a reading of 1.00 g/ml or 1000 kg/m³ (8.345 lb/gal or 62.4 lb/ft³) at 21 °C (70 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm, as required. A calibration of the upper density should be performed as specified by the manufacturer, and done on a less frequent basis, e.g. annually.

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

5.3 Mud Balance Procedure

In order to determine drilling fluid density, the following procedure shall be followed:

a) The mud balance instrument should 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 cap on the filled sample cup and rotate the cap, expelling any trapped air and excess drilling fluid. Ensure the cap is firmly seated.

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

e) Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the 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. As it is considered that the water density is 1 g/ml (see definition 3.3), 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 for each 1000 ft of vertical depth.

5.4 Calculation

5.4.1 Report the drilling fluid density to the nearest 0.01 g/ml or 10 kg/m³ (0.1 lb/gal or 0.5 lb/ft³).

5.4.2 To convert the relative density reading, \( \rho \), which is numerically equivalent to the density, \( \rho_{df} \), expressed in grams per milliliter to other units, use Equations (1) to (7) and Tables 1 and 2.

Equations (1) to (3) are used to convert the density, \( \rho_{df} \), expressed in grams per milliliter, to other units:

\[
\rho_{df,SI} = 1000 \times \rho_{df} \tag{1}
\]
where \( \rho_{df,SI} \) is the drilling fluid density, expressed in kilograms per cubic meter.

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

where \( \rho_{df,USC} \) is the drilling fluid density, expressed in pounds per gallon.

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

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

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

<table>
<thead>
<tr>
<th>Measured in:</th>
<th>Multiply to Convert to:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g/ml</td>
</tr>
<tr>
<td>g/ml</td>
<td>1</td>
</tr>
<tr>
<td>kg/m³</td>
<td>0.001</td>
</tr>
<tr>
<td>lb/gal</td>
<td>0.120</td>
</tr>
<tr>
<td>lb/ft³</td>
<td>0.0160</td>
</tr>
</tbody>
</table>

Equations (4) to (7) are used to convert the density to the drilling fluid depth gradient, \( \Gamma_{DFG} \), expressed in kilopascals per meter (pounds 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 \( \Gamma_{DFG,SI} \) is the drilling fluid depth gradient, expressed in kilopascals per meter; and \( \Gamma_{DFG,USC} \) is the drilling fluid depth gradient, expressed in pounds per square inch, per foot.

A list of density conversions is given in Table 2.
Table 2—Density Conversion

<table>
<thead>
<tr>
<th>Grams per Cubic Centimeter&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Kilograms per Cubic Meter</th>
<th>Pounds per Gallon</th>
<th>Pounds per Cubic Foot</th>
</tr>
</thead>
<tbody>
<tr>
<td>g/ml</td>
<td>kg/m³</td>
<td>lb/gal</td>
<td>lb/ft³</td>
</tr>
<tr>
<td>0.70</td>
<td>700</td>
<td>5.8</td>
<td>43.7</td>
</tr>
<tr>
<td>0.80</td>
<td>800</td>
<td>6.7</td>
<td>49.9</td>
</tr>
<tr>
<td>0.90</td>
<td>900</td>
<td>7.5</td>
<td>56.1</td>
</tr>
<tr>
<td>1.00</td>
<td>1000</td>
<td>8.345&lt;sup&gt;b&lt;/sup&gt;</td>
<td>62.4</td>
</tr>
<tr>
<td>1.10</td>
<td>1100</td>
<td>9.2</td>
<td>68.7</td>
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<td>174.8</td>
</tr>
<tr>
<td>2.90</td>
<td>2900</td>
<td>24.2</td>
<td>181.0</td>
</tr>
</tbody>
</table>

<sup>a</sup> Same value as relative density (specific gravity) in grams per cubic centimeter or kilogram per liter.

<sup>b</sup> Accurate conversion factor.

6  Alternative Method for Determination of Drilling Fluid Density

6.1  Principle

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

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

6.2  Apparatus

6.2.1 Any density-measuring instrument, having an accuracy of ±0.01 g/ml or ±10 kg/m³ (±0.1 lb/gal or ±0.5 lb/ft³).

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

The instrument should be calibrated frequently with fresh water, e.g. bi-weekly or weekly. Fresh water should give a reading of 1.00 g/ml or 1000 kg/m³ (8.345 lb/gal or 62.4 lb/ft³) at 21 °C (70 °F). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm, as required. A calibration of the upper density should be performed as specified by the manufacturer, and done on a less frequent basis, e.g. annually.

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

6.3 Pressurized Mud Balance Procedure

To determine drilling fluid density with a pressurized mud balance, the following procedure shall be used:

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

b) Fill the sample cup of the pressurized mud balance to a level approximately 6.5 mm (0.25 in.) below the upper edge of the cup.

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

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

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

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

g) The pressurized drilling fluid sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place the instrument on the knife edge. Move the sliding weight until the beam is balanced. The beam is balanced when the attached bubble is centered between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. As it is considered that the density of water is 1 g/ml (see definition 3.3), the density can be read directly in units of grams per milliliter using relative density (specific gravity) scale, pounds per gallon, and pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch for each 1000 ft of vertical depth.

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

i) Clean the cup, lid, and plunger assembly.
6.4 Calculation

Report the drilling fluid density to the nearest 0.01 g/ml or 10 kg/m$^3$ (0.1 lb/gal or 0.5 lb/ft$^3$).

For conversions, use the equations given in 5.4.2.

7 Viscosity and Gel Strength

7.1 Principle

7.1.1 Viscosity and gel strength are measurements that relate to the flow or rheological properties of drilling fluids.

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. The equipment is as follows:

a) Marsh funnel and graduated cup: a simple device for indicating viscosity on a routine basis;
b) direct-indicating viscometer: a mechanical device for measuring viscosity at varying shear rates.
c) a chilled rheometer cup is typically required for measuring low temperature rheology.

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

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 quart) 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 1.6 mm (0.063 in. = 1/16 in., 12 mesh) openings, 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 qt).

7.2.1.3 Stopwatch.

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

7.2.2 Procedure

In order to determine funnel viscosity using a Marsh Funnel, the following procedure shall be followed:

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).

d) Report the time (7.2.2.b) to the nearest second as the Marsh funnel viscosity. Report the temperature (7.2.2.c) of the fluid to the nearest degree Celsius (degree Fahrenheit).

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

7.3.1 Apparatus

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

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

The components shall meet the following specifications:

a) **Rotor sleeve—R1**:
   - inside diameter: 36.83 mm (1.450 in.)
   - total length: 87.0 mm (3.425 in.)
   - scribed line: 58.4 mm (2.30 in.) above the bottom of sleeve, with two rows of 3.18 mm (0.125 in.) holes, spaced 2.09 rad (120°) apart, around rotor sleeve just below scribed line
   - sleeve surface: surface finish (roughness) average 16 to 32 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 16 to 32 RMS cross-hatched honed

c) **Torsion spring—F1.0**, constant:
   - 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 per radian of deflection ≡ 0.511 pascals per degree of deflection
     (1.067 lbf/100•ft² per degree of deflection)

d) **Rotor sleeve speeds**:
   - high speed: 600 r/min
   - low speed: 300 r/min

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

7.3.1.2 **Stopwatch**.
7.3.1.3  Thermostatically controlled viscometer cup (thermocup):

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

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

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

7.3.2  Procedure

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

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

b) Heat (or cool) the sample to the selected temperature. Use constant shear at 600 r/min or 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.

NOTE Use 300 r/m in highly weighted mud systems to minimize weighting material settling.

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

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

NOTE Additional dial reading values are often measured and reported, typically $R_{200}, R_{100}, R_{6}, R_{3}$. Improved modeling accuracy can be achieved by also measuring the $R_{60}$ and $R_{90}$ dial readings. Measurement of dial readings should be performed stepwise from the highest to the lowest RPM speed.

e) With the sleeve rotating at 600 r/min, wait for the viscometer dial reading to reach a steady value.

f) 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 hand-wheel in the appropriate direction to produce a positive dial reading. Record the maximum reading as the initial gel strength or 10 second gel strength. For instruments having a 3 r/min speed, the maximum reading attained after starting rotation at 3 r/min is the initial gel strength. Record the initial gel strength, $\beta_{10s}$, at 10 s, in pounds-force per one hundred square feet (lbf/100•ft$^2$).

NOTE The gel strength value reported directly from the dial reading is an approximation 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 0.511 Pa in SI units and to 1.067 lbf/100•ft$^2$ in USC units. These exact units corrections are often neglected for simplicity in reporting. Common field practice when reporting pascals is to divide dial units by 2 for simplicity. 1 r/min of the rotor equals a shear rate of 1.7023 s$^{-1}$.

g) Re-stir the drilling fluid sample at 600 r/min until a steady value is achieved, stop the motor and then allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in 7.3.2.f and
report the maximum reading as $\beta_{10m}$, the 10-min gel strength, in pounds-force per one hundred square feet (lbf/100•ft$^2$).

NOTE The gel strength value reported directly from the dial reading is an approximation 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 0.511 Pa in SI units and to 1.067 lbf/100•ft$^2$ 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. 1 r/min of the rotor equals a shear rate of 1.7023 s$^{-1}$.

7.3.3 Calculation

7.3.3.1 The dimensions for the rotor R1, bob B1 and spring F1.0 constant, as described in 7.3.1.1, determine the following:

- 1° deflection of the bob equals a shear stress of 0.511 Pa and approximately 1 lbf/100•ft$^2$, or more exactly 1 degree of deflection = 1.067 lbf/100•ft$^2$
- 1 revolution per minute (r/min) of the rotor equals a shear rate of 1.7023 reciprocal seconds (s$^{-1}$).

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

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

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

where

- $\eta_{PV}$ is the plastic viscosity, expressed in millipascal-seconds (centipoises);
- $R_{600}$ is the dial reading at 600 revolutions per minute, expressed in degrees of deflection;
- $R_{300}$ is the dial reading at 300 revolutions per minute, expressed in degrees of deflection.

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

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

$$Y_{P, USC} = R_{300} - \eta_{PV} \tag{9}$$

where

- $Y_{P, USC}$ is the yield point, expressed in pounds-force per one hundred square feet;
- $R_{300}$ is the dial reading at 300 revolutions per minute, expressed in degrees of deflection;
- $\eta_{PV}$ is the plastic viscosity, expressed in millipascal-seconds (centipoises).

NOTE Yield point, in lbf/100•ft$^2$, is commonly known in the industry by the abbreviation YP. The yield point value calculated with Equation (10) from the dial readings is an approximation 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 0.511 Pa in SI units and to 1.067 lbf/100•ft$^2$ in USC units. In equation (10) the exact units correction is neglected for simplicity in reporting.
7.3.3.4 The calculation for yield point, \( Y_{P, \text{SI}} \), expressed in pascals, is given in Equation (10):

\[
Y_{P, \text{SI}} = 0.511 \times (R_{300} - \eta_{PV})
\]

(10)

where

\( Y_{P, \text{SI}} \) is the yield point, expressed in pascals;

\( R_{300} \) is the dial reading at 300 revolutions per minute, expressed in degrees of deflection;

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

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 pascals is to divide dial units [Equation (10)] by 2 for simplicity.

7.3.3.5 The calculation for apparent viscosity, \( \eta_{AV} \), expressed in millipascal-seconds (centipoises), is given in 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.

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

7.3.3.6 Report the plastic viscosity, yield point, 10 second gel strength, 10 min gel strength, and apparent viscosity.

8 Static Filtration

8.1.1 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, water, or emulsion concentration.

8.1.2 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.

8.1.3 Tests are run under static conditions at both low pressure/low temperature and high pressure/high temperature, 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 an inside diameter of 76.2 mm (3.0 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 is so fitted that a pressure medium can be conveniently admitted into, and bled from, the top. It shall also be fitted such that a sheet of 90 mm (3.54 in.) diameter filter paper can be placed in the bottom of the cell just above a suitable
support. The filtration area is (45.8 cm² ±0.6 cm² (7.1 in.² ±0.1 in.²). Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets and the entire assembly supported by a stand. Pressure can be applied with any non-hazardous gas medium. The filter press is equipped with a pressure regulator that can be energized using a portable pressure cylinder, pressure cartridge or other means for applying a controllable differential pressure. To obtain correlative results, one thickness of the proper 90 mm diameter filter paper (Hardened, low-ash grade filter paper¹) shall be used.

The low-temperature/low-pressure filter press should have a filter area of 45.2 cm² to 46.4 cm² (7.0 in.² to 7.2 in.²) which corresponds to a diameter of 75.86 mm to 76.86 mm (2.987 in. to 3.026 in.). The inner diameter of the filter press gasket determines the dimensions of the filtration area. It is recommended that the filter press gasket to be used should be tested using a conical gauge that has the maximum 76.86 mm (3.026 in.) and the minimum 75.86 mm (2.987 in.) diameters marked on it. Any filter-press gasket found out of this range (either larger or smaller than the markings) shall not 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 Electronic or Mechanical Timer, with at least a 30 min interval.

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

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

8.2.2 Procedure

In order to conduct a low pressure/low temperature 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 1/2 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 690 kPa ±35 kPa (100 psi ±5 psi) 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. 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.

¹ This filter paper is a calendared, hardened, qualitative, low-ash filter paper made from cotton linters. The filter paper has the following typical properties which may vary by manufacturer: Slow filtration rate: 2685 herzbergs; particle retention in liquid: 2-5 μm; ash Content: 0.015% by weight; basis weight: 92 g/m² (0.019 lb/ft²); diameter: 90 mm (3.54 in.); Thickness: 0.137 mm (0.0054 in).
e) Report the volume of filtrate 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 (HTHP) Test

#### 8.3.1 Principle

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

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

8.3.1.3 For testing above 175 °C (350 °F), use only equipment rated by the manufacturer for these temperatures. 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 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 manufacturer’s recommendations when using their equipment and media.**

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

8.3.2.1 **High-temperature High-pressure Filter Press**, consisting of:

a) filter cell, to rated working pressure up to 9000 kPa (1300 psi) at temperature;

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

NOTE Nitrogen is the preferred gas for this application.

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

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

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

f) valve stems on each end of the cell which can be opened or closed during the test.

**Warning—Strict adherence to manufacturer’s recommendations with respect to sample volumes, equipment temperatures and pressures is essential. Failure to do so could result in serious injury.**
Do not use nitrous oxide cartridges as pressure sources for HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials.

Table 3—Recommended Minimum Back-pressure

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Vapor Pressure</th>
<th>Minimum Back-pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C</td>
<td>°F</td>
<td>kPa</td>
</tr>
<tr>
<td>100</td>
<td>212</td>
<td>101</td>
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<td>120</td>
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<td>207</td>
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<td>150</td>
<td>300</td>
<td>462</td>
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<tr>
<td>175</td>
<td>350</td>
<td>932</td>
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<tr>
<td>200</td>
<td>400</td>
<td>1704</td>
</tr>
<tr>
<td>230</td>
<td>450</td>
<td>2912</td>
</tr>
</tbody>
</table>

8.3.1.2 Filter Medium:

a) Hardened, low-ash grade filter paper\(^2\), for temperatures up to 175 °C (350 °F), a new paper is required for each test.

b) Hardened, low-ash grade filter paper\(^2\) backed with a fiberglass disk for temperatures above 175 °C (350 °F) up to temperatures of 200 °C (400 °F), a new paper is required for each test.

8.3.1.3 Electronic or Mechanical Timer, with at least a 30-min interval.

8.3.1.4 Thermometer, with a range up to 260 °C (500 °F), and with a 12.5 cm (5 in.) or longer stem, or a thermocouple with temperature read-out or controller with a range up to 260 °C (500 °F), preferred.

8.3.1.5 Graduated Cylinder, with a volume of 10 ml (TC), 20 ml (TC) or 25 ml (TC), with markings having a minimum of 2 ml graduations.

8.3.1.6 Field Mixer, cup type, to operate at 10,000 r/min to 15,000 r/min.

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

8.3.2 Procedure

In order to conduct a HTHP filtration test on a sample of drilling fluid up to 175 °C (350 °F), 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 the desired test temperature. **Caution**—the outside of the jacket can get hot enough to cause burns.

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

b) Stir the drilling fluid sample for 10 min using the field mixer set at the 10,000 r/min speed. Install and close the upper valve stem into the test cell. Pour the fluid sample into the filter cell, leaving at least a

---

\(^2\) This filter paper is a calendered, hardened, qualitative, low-ash filter paper made from cotton linters. The filter paper has the following typical properties which may vary by manufacturer: Slow filtration rate: 2685 herzbergs; Particle retention in liquid: 2-5 μm; ash content: 0.015 % by weight; basis weight: 92 g/m\(^2\) (0.019 lb/ft\(^2\)); diameter: 63.5 mm (2.5 in.); thickness: 0.137 mm (0.0054 in).
2.5 cm (1 in.) space in the cell to allow for fluid expansion. Install the appropriate filtration media for the test to be run.

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

d) Complete the assembly of the filter cell and place it in the heating jacket, with the filter media on the bottom, and rotate the cell to lock it in place with the pin inside the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.

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

f) Connect the regulated pressure assembly to the upper valve and lock it in place.

g) Keeping the two valve stems closed, adjust the pressure on the upper and lower pressure regulators to 690 kPa (100 psi), as shown in Table 3. Open the upper valve stem, and readjust the upper pressure regulator to maintain a pressure 690 kPa (100 psi) in the cell. Maintain these pressures until the test temperature is reached.

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

h) When the sample reaches the selected test temperature, as indicated by the thermometer or thermocouple, ensure the lower pressure regulator is at the pressure (“minimum back-pressure”) shown for the temperature given in Table 3. Open the lower valve stem and immediately increase the pressure on the upper regulator to 4140 kPa (600 psi), or 3450 kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ±5 °F (±3 °C) during the test, as indicated by the thermometer in the filter cell. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

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

i) Collect the filtrate in the graduated cylinder. Record the volume of the 30-min total filtrate. Correct the filtrate volume to a filter area of 45.8 cm² (7.1 in²). For example, if the filter area is 22.6 cm² (3.5 in²), double the filtrate volume reported.

j) 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.

Caution—Cell and heating jacket are still hot!

k) Using appropriate PPE, 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.

Caution—The filter cell may still be pressurized even after the cell is cooled. To avoid possible serious injury, keep cell upright and cool to room temperature, and then bleed pressure from cell before disassembling.

l) 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. 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 all pressure is released before attempting to remove the end cap.

m) Pour the liquid from the cell.

n) Remove the filter cake with the filtration medium. Measure the filter cake thickness, at its center, to the nearest millimeter (1/32 in.).
o) 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 should be minimized and the cake should be recovered and examined promptly.

8.3.3 Calculation

8.3.3.1 The filtrate volume, \( V_f \), should be corrected to a filter area of 4516 mm\(^2\) (7.0 in.\(^2\)). HTHP filter cells usually have half the standard filter area 2258 mm\(^2\) (3.5 in.\(^2\)), thus double the observed volume before reporting.

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

8.4 High-temperature/High-pressure Test at 175 °C (350 °F) up to and Including 230°C (450 °F)

8.4.1 Apparatus

8.4.1.1 High-temperature/High-pressure Filter Press, consisting of the following components:

a) only use cells and equipment rated to safely operate at the intended test temperature and pressures. See manufacturers’ recommendations.

b) filter cell, with a rated working pressures up to 13,800 kPa (2000 psi) at a temperature of 230 °C (230 °C); filter cell fitted with a removable end, a filter-media support, with oil-resistant seals, and with a thermometer well.

c) pressure regulated gas source, nitrogen (preferred);

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

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

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

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

Caution—Not all manufacturers’ equipment can be used above 150°C (300 °F). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions. The 175 ml and 250 ml filtration cells are generally not recommended for use at these higher temperatures and pressures.

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

8.4.1.2 Filter Media,

a) Hardened, low-ash grade filter paper\(^2\) backed with a fiberglass disk for temperatures above 175 °C (350 °F) up to temperatures of 200 °C (400 °F), a new paper is required for each test.

b) Sintered metal-fiber porous disc should be used for temperatures above 200 °C (400 °F), a new disc is required for each test. Disk is comprised of sintered stainless steel metal-fibers with a particle retention rating in liquid of 5 µm nominal and 10 µm absolute) for temperatures above 200 °C (400 °F). A new disc is required for each test. (Example: Dynalloy X-5 sintered metal fiber media)

8.4.1.3 Electronic or Mechanical Timer, with at least a 30-min interval.

8.4.1.4 Thermometer, with a range up to 260°C (500 °F), and with a 12.5 cm (5 in.) or longer stem, or a thermocouple with a range up to 260°C 500 °F), preferred.
8.4.1.5 **Graduated Cylinder (TC)**, long, slender with a volume of 10 ml or 20 ml.

8.4.1.6 **Graduated Cylinder (TC)**, optional, with a volume of 25 ml.

8.4.1.7 **Field Mixer**, cup type, to operate at 10,000 r/min to 15,000 r/min.

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

8.4.2 **Procedure**

In order to conduct a HTHP test 175 °C (350 °F) up to and including 230°C (450 °F) test, 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 the test temperature.

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

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

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

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

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

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

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

If the sample reaches the selected test temperature, as indicated by the thermocouple, set the lower pressure regulator at the pressure (“minimum back-pressure”) shown for the temperature given in Table 3. Open the lower valve stem and immediately increase the pressure on the upper regulator to 4140 kPa (600 psi), or 3450kPa (500 psi) higher than the back-pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ±3 °C (±5 °F) during the test, as indicated by the thermometer in the filter cell. If the back-pressure rises above the selected back-pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back-pressure.

Collect the filtrate in the graduated cylinder. Record the volume of the 30-min total filtrate. Correct the filtrate volume to a filter area of 45.8 cm² (7.1 in²). For example, if the filter area is 22.6 cm² (3.5 in²), double the filtrate volume reported. Also note the presence of, or volumes of, any solids, oil or emulsions, if present.
Immediately after collecting the 30-min filtrate, turn off to stop heating and unplug the heating jacket from the electrical outlet. Close the upper, then lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the pressurization system. Remove the cell from the heating jacket and allow the cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization and disassembly.

**Warning**—Pressure in the filter cell can be 6550 kPa (950 psi), even after the cell is cooled. To avoid possible serious injury, keep cell upright and cool to room temperature, then bleed pressure from cell before disassembling.

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

Pour the liquid from the cell.

Remove the filter cake on the filter disc or paper. Measure the filter cake thickness at its center, to the nearest millimeter (1/32 in.).

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

8.4.3 Calculation

8.4.3.1 Double the measured filtrate volume, \( V_f \), to correct for to a filter area of 4156 mm² (7.0 in.²). HTHP filter cells usually have half the standard filter area 2258 mm² (3.5 in.²), thus the observed volume shall be doubled before reporting.

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

9 Retort Test for Water, Oil, and Solids Concentrations

9.1 Principle

The retort instrument provides a means for separating and measuring the volumes of water, oil and solids contained in a sample of drilling fluid. In the retort, a known volume of a whole drilling fluid sample is heated to vaporize the liquid components that are subsequently condensed and collected in a graduated cylinder for standard accuracy measurements or in a special liquid receiver for more precise measurements. Liquid volumes are determined directly from reading the water and oil phases in the receiver. The total volume of solids (suspended and dissolved) is obtained by difference (retort sample volume minus all condensed liquid volumes). Calculations are necessary to determine the volume of dissolved and suspended solids, since both soluble (salts) and insoluble solids are retained in the retort. The relative volumes of low-gravity solids and weighting material can also be calculated. Knowledge of water, oil, and solids concentrations is fundamental to proper control of drilling fluid properties such as rheology, density, filtration and salinity of the aqueous (water) phase. Given that knowledge of solids in a drilling fluid is essential to the evaluation of solids control equipment, reference shall be made to API 13C.

9.2 Apparatus

9.2.1 Retort Instrument

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

Standard cup sizes are 10 ml (precision ±0.05 ml), 20 ml (precision ±0.1 ml), and 50 ml (precision ±0.25 ml). The retort cup volume with lid shall be verified gravimetrically in accordance with the procedure and calculations given in Annex I.

b) **Condenser**, capable of cooling the oil and water vapors below their vaporization temperature.
c) **Heating jacket**, nominal power 350 W and of sufficient power to raise the temperature of the sample above the vaporization point of the liquid components within 15 min without causing solids boil-over.

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 Liquid Receiver (TC)**, Graduated cylinders are used for standard accuracy retort measurements or a special liquid receiver may be used for more precise retort measurements. The special liquid receivers are a specially designed cylindrical glassware with a rounded bottom, to facilitate cleaning, and a funnel-shaped top to catch falling drops and are often referred to in industry as JP-tubes. The two types and three sizes of receivers shall meet the following specifications:

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

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

— **frequency of graduations**: milliliter or volume fraction (as a percentage), see Table 4;

— **tolerance**: see Table 4;

— The receiver volume shall be verified gravimetrically in accordance with the procedure and calculations in Annex I (I.6).

<table>
<thead>
<tr>
<th>Table 4—Precision of Retort Liquid Receivers</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tolerance</strong></td>
</tr>
<tr>
<td>Graduated Cylinder 10 mL (TC)</td>
</tr>
<tr>
<td>Graduated Cylinder 20 mL (TC)</td>
</tr>
<tr>
<td>Graduated Cylinder 50 mL (TC)</td>
</tr>
</tbody>
</table>

* Meets ASTM E1272 Class A standards

<table>
<thead>
<tr>
<th><strong>Tolerance</strong></th>
<th><strong>Frequency of graduations</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Special Liquid Receiver 10 mL (TC)</td>
<td>± 0.05</td>
</tr>
<tr>
<td>Special Liquid Receiver 20 mL (TC)</td>
<td>± 0.05</td>
</tr>
<tr>
<td>Special Liquid Receiver 50 mL (TC)</td>
<td>± 0.15</td>
</tr>
</tbody>
</table>

9.2.3 **Fine Steel Wool**, oil-free.

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

9.2.4 **High-temperature-resistant Silicone Grease**, to be used as a thread seal and lubricant.

9.2.5 **Pipe Cleaners 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** (7.2.1.1).

9.2.10 **Defoaming agent**
9.2.11 Mud Balance (5.2.1).

9.3 Procedure

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 should 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 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 should 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 screen of the Marsh funnel to remove lost circulation material, large cuttings or debris.

e) Ensure the drilling fluid sample is homogeneous. If mixing is required, 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 two to three drops of defoaming agent to about 300 ml of drilling fluid and stir slowly for 2 min to 3 min to release gases. Alternatively the procedure described in Annex E can be used for sever cases of foaming or air/gas entrapment.

f) 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, ensuring that a small excess of drilling fluid flows out of the hole in the lid. Wipe the excess drilling 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 normally 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 hr or longer. Continue heating for 10 min after the last condensate is collected. Collect the condensate into the glass liquid receiver. If the drilling fluid boils 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) After the completed retort test, remove the liquid receiver and allow the retort assembly to cool.

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

NOTE If an emulsion interface is present in the receiver tube, between the oil and water phases, heating the interface might break the emulsion. As a suggestion, remove the retort assembly from the heating jacket by grasping the condenser. Carefully heat the glass liquid receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort assembly. Avoid boiling the liquid. After the emulsion interface is broken, allow the liquid receiver to cool. Do not use this procedure when using a plastic or polycarbonate receiving tube.
l) Record the total liquid volume, \( V_L \) (oil and water) and water volume, \( V_W \), collected in the liquid receiver.

NOTE 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 retort assembly to cool 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.

m) 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 the retort assembly and condenser.

9.4 Calculation

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

Using the measured volumes of oil and water, and the volume of the original whole drilling fluid sample (10 mL, 20 mL, or 50 mL), calculate, as percentages, the volume fractions of water, oil, and dried solids in the drilling fluid (dried or total solids from the retort includes both soluble and insoluble solids).

9.4.1.1 Calculate the volume of oil, \( V_O \), in the condensed sample expressed in milliliters.

\[
V_O = V_L - V_W
\]

(12)

where

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

\( V_W \) is the water volume expressed in milliliters or water mass expressed in grams (1 ml = 1 g).

9.4.1.2 Calculate the volume fraction of oil, \( \phi_O \), in the total drilling fluid sample, expressed as a percentage.

\[
\phi_O = 100 \times \frac{V_O}{V_{df}}
\]

(13)

where

\( V_O \) is the volume of oil, expressed in milliliters;

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

9.4.1.3 Calculate the volume fraction of water, \( \phi_W \), expressed as a percentage of the drilling fluid sample volume;

\[
\phi_W = 100 \times \frac{V_W}{V_{df}}
\]

(14)

where

\( V_W \) is the water volume expressed in milliliters or water mass expressed in grams (1 ml = 1 g);

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

9.4.1.4 Calculate the volume fraction of dried solids from retort, \( \phi_{ds} \), includes both soluble and insoluble solids, expressed as a percentage of the drilling fluid volume;

\[
\phi_{ds} = 100 - (\phi_W + \phi_O)
\]

(15)
where

\[ \phi_W \] is the volume fraction of water, expressed as a percentage of the drilling fluid volume;

\[ \phi_O \] is the volume fraction of oil, expressed as a percentage of the drilling fluid volume.

NOTE The calculated dried solids fraction from the retort, \( \phi_{ds} \), equation (15) includes both dissolved soluble materials (e.g. salt) and insoluble suspended solids (weighting material, drilling fluid additives and drill 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)

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

9.4.2.1 The volume fraction of suspended solids, \( \phi_{SS} \), expressed as a percentage of the drilling fluid volume is given in Equation (16):

\[
\phi_{SS} = \phi_{ds} - \left[ \frac{\phi_W \times c_{Cl}}{1,680,000 - 1.21 \times c_{Cl}} \right]
\]

(16)

where

\[ \phi_{ds} \] is the volume fraction of dried solids from retort, expressed as a percentage of the drilling fluid volume;

\[ \phi_W \] is the volume fraction of water, expressed as a percentage of the drilling fluid volume;

\[ c_{Cl} \] is the chloride concentration, expressed in milligrams per liter (see equation 33).

9.4.2.2 The volume fraction of low-gravity-solids, \( \phi_{LG} \), expressed as a percentage of the drilling fluid volume, is given in Equation (17):

\[
\phi_{LG} = \frac{1}{\rho_{WM} - \rho_{LG}} \left[ 100 \rho_f + \left( \rho_{WB} - \rho_f \right) \times \phi_{SS} - 100 \rho_{df} - \left( \rho_f - \rho_O \right) \times \phi_O \right]
\]

(17)

where

\[ \phi_{SS} \] is the volume fraction of suspended solids, expressed as a percentage of the drilling fluid volume;

\[ \phi_O \] is the volume fraction of oil, expressed as a percentage of the drilling fluid volume;

\[ \rho_{df} \] is the drilling fluid density, expressed in grams per milliliter;

\[ \rho_f \] is the density of the filtrate for sodium chloride, expressed in grams per milliliter, as given by Equation (18):

\[
\rho_f = 1 + \left( 1.09 \times 10^{-6} \right) \times c_{Cl}
\]

(18)
\( \rho_{\text{LG}} \) is the density of low-gravity solids, expressed in grams per milliliter;
\( \rho_{\text{WM}} \) is the density of weighting material, expressed in grams per milliliter;
\( \rho_{\text{O}} \) is the density of oil, expressed in grams per milliliter.

NOTE 1 For calculation in USC units, Equation (17) can be used providing 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.

NOTE 3 The filtrate density, \( \rho_f \), calculation as given by Equation (18) is for sodium chloride only using the chloride concentration, \( c_{\text{Cl}} \), expressed in milligrams per liter.

9.4.2.3 The volume fraction of weighting material, \( \varphi_{\text{WM}} \), expressed as a percentage of the drilling fluid volume is given in Equation (19):

\[
\varphi_{\text{WM}} = \varphi_{\text{SS}} - \varphi_{\text{LG}}
\]  

(19)

where

\( \varphi_{\text{SS}} \) is the volume fraction of suspended solids, expressed as a percentage of the drilling fluid volume;

\( \varphi_{\text{LG}} \) is the volume fraction of low gravity solids, expressed as a percentage of the drilling fluid volume.

9.4.2.3 Concentration of low gravity solids, \( c_{\text{LG,SI}} \), weighting material, \( c_{\text{WM,SI}} \), and suspended solids, \( c_{\text{SS,SI}} \), expressed in kilograms per cubic meter can be calculated as given by Equations (20), (21) and (22) respectively:

\[
c_{\text{LG,SI}} = 10\rho_{\text{LG}} \times \varphi_{\text{LG}}
\]

(20)

\[
c_{\text{WM,SI}} = 10\rho_{\text{WM}} \times \varphi_{\text{WM}}
\]

(21)

\[
c_{\text{SS,SI}} = c_{\text{WM,SI}} + c_{\text{LG,SI}}
\]

(22)

or concentration of low gravity solids, \( c_{\text{LG,USC}} \), weighting material, \( c_{\text{WM,USC}} \), and suspended solids, \( c_{\text{SS,USC}} \), expressed in pounds per barrel can be calculated as given by Equations (23), (24) and (25) respectively:

\[
c_{\text{LG,USC}} = 3.50\rho_{\text{LG}} \times \varphi_{\text{LG}}
\]

(23)

\[
c_{\text{WM,USC}} = 3.50\rho_{\text{WM}} \times \varphi_{\text{WM}}
\]

(24)

\[
c_{\text{SS,USC}} = c_{\text{WM,USC}} + c_{\text{LG,USC}}
\]

(25)

where

\( \varphi_{\text{LG}} \) is the volume fraction of low gravity solids, expressed as a percentage of the drilling fluid volume;

\( \rho_{\text{LG}} \) is the density of low-gravity solids, expressed in grams per milliliter;

\( \varphi_{\text{WM}} \) is the volume fraction of weighting material, expressed as a percentage of the drilling fluid volume;
\( \rho_{WM} \) is the density of weighting, expressed in grams per milliliter.

NOTE For water-based drilling fluids with added bentonite, additional calculations can be made to determine the concentration of bentonite and formation drill solids, see RP13I Section 13.

10 Sand Content

10.1 Principle

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

10.2 Apparatus

10.2.1 Sieve, 74 \( \mu \)m (200 mesh) and 63.5 mm (2.5 in.) in diameter.

10.2.2 Funnel, to fit sieve.

10.2.3 Glass Measuring Tube, marked for the volume of drilling fluid to be added and graduated from 0 % to 20 % in order to read the percentage of sand directly. The bottom portion of the tube is graduated in convenient volume percentages to indicate the solids (sand sized) content.

10.3 Procedure

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

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

b) Pour the mixture onto the clean, wet sieve. Discard the liquid passing through the sieve. Add more water 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.

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

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

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 “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 RP13I, 8th Edition, Section 13.

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 lignosulphonates, 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/l (1 ml = 0.01 meq).

The moisture content of reagent grade methylene blue shall be determined each time the solution is prepared. Dry a 1.00 g 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 given in 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.

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

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

Warning—\( \text{H}_2\text{O}_2 \) 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 5 N (2.5 mol/L).

Warning—\( \text{H}_2\text{SO}_4 \) is a strong and toxic acid.

11.2.4 De-ionized or Distilled 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, water absorbent cellulose filter paper circles. Typical properties: alpha cellulose minimum content 98%, particle retention in liquid 11 µm @ 98% efficiency, air flow rate 10.5 s/100 ml/in².
approximate filtration speed 150 s (Herzberg), nominal ash content 0.06% determined by ignition of the cellulose filter at 900°C in air, nominal thickness 180 µm wet burst 1720 Pa (0.25 psi).

11.3 Procedure

In order to conduct the Methylene Blue Titration (MBT), the following procedure shall be used:

11.3.1 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 de-ionized water in the Erlenmeyer flask. The syringe used should have a capacity of more than 2 ml, generally 2.5 ml or 3 ml. By using a larger syringe, it is not necessary to remove the air trapped in the syringe. To assure that exactly 2.0 ml of drilling fluid is being added, use the following procedure.

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

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

c) Deliver 2.0 ml of drilling fluid by pushing the plunger until the end of the plunger is exactly 2 ml from the last graduation on the syringe. This, in a 3-ml syringe, is at the 1-ml line.

11.3.2 Add 15 ml of 3 % hydrogen peroxide and 0.5 ml of 5N sulfuric acid. Boil gently for 10 min, but do not allow to boil to dryness. Dilute to about 50 ml total volume with de-ionized water.

11.3.3 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 dye appears as a blue or turquoise ring surrounding the dyed solids, as shown in Figure 1, key item 5 (6 ml addition).

11.3.4 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 is again evident, the final endpoint has been reached; as shown in Figure 1, key item 7 (7 ml).

In Figure 1, free dye is detected immediately after adding the sixth milliliter, but after two minutes additional agitation, that dye has been adsorbed. This indicates that the endpoint has not been attained. Free dye is again detected after adding the seventh milliliter, and this is not adsorbed after two minutes. This is the endpoint.
Figure 1—Spot tests for Endpoint of Methylene Blue Titration

11.4 Calculation

Report the methylene blue capacity, $C_{MBT}$, of the drilling fluid, expressed in milliequivalents per liter (meq/L), calculated as follows:

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

(27)

where

- $V_{mb}$ is the volume of methylene blue solution, expressed in milliliters;
- $V_{df}$ is the volume of drilling fluid sample, expressed in milliliters.

Key
1. volume of methylene blue solution added
2. no free, unabsorbed dye present
3. drilling fluid solids, dyed blue
4. moisture, no color
5. free dye in water
6. endpoint reached
7. endpoint retest after 2 min reaction
8. endpoint exceeded
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, as given in Equation (28) or $E_{BE,USC}$, expressed in pounds per barrel, as given in Equation (29):

$$E_{BE,SI} = \frac{14.25 \times V_{mb}}{V_{df}}$$ (28)

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

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

12 pH

12.1 Principle

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

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

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

Color-matching pH paper and sticks are used for field pH measurements, but are not the methods recommended. 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. Readability of pH paper and sticks is normally about 0.5 pH unit.

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 solution, dry-powder packages, or a given formula. Shelf life of buffers should not exceed six months before disposal. The date of preparation of the buffer should be shown on bottles used in the field. Bottles should be kept tightly sealed when not in use.

12.2.2 De-ionized or Distilled Water, in spray bottle.

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 (NH₄HF₂) 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:

a) pH range: 0.0 to 14.0; 
b) electronics type: solid state (preferred); 
NOTE A double junction probe is preferred. 
c) power source: batteries (preferred); 
d) operating temperature range: (0 °C to 66 °C (32 °F to 150 °F); 
e) readout: digital (preferred); 
f) resolution: 0.1 pH unit; 
g) accuracy: ±0.1 pH unit; 
h) repeatability: 0.1 pH unit;

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:

a) pH electrode response range: 0.0 to 14.0 pH units; 
b) 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. 
c) electrolyte in reference electrode: KCl gel; 
d) glass or epoxy composition: suitable for low sodium-ion error; 
e) sodium-ion error: at pH 13.0 or at 0.1 molar 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 DI 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.

f) Most modern pH-meters have Automatic Temperature Compensation (ATC). 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 steps 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.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 given in 12.4.

   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 hr when using the meter continuously and prior to use, if more than 3 hr had lapsed between measurements.

l) If meter calibrates properly, rinse electrode with DI 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 de-ionized water.

o) Turn 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

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

12.4.2 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.
12.4.3 Recondition by soaking electrode for 10 min in 0.1 mol/L HCl, followed by rinsing in DI 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.

12.4.4 Check electrode for response by performing calibration in 12.3.

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

12.4.6 Replace electrode system if steps 12.4.3 to 12.4.5 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.

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

13.1.3 The ions that are primarily responsible for filtrate alkalinites 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 alkalinites 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.

13.1.4 The composition of drilling fluid filtrates is often so complex that the interpretation of alkalinities in terms of estimated ionic components can be misleading. Any particular alkalinity value represents 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. However, for simple bentonite-base drilling fluid systems containing no organic thinners, 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 0.02 N (N/50).

Warning—H₂SO₄ 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 pH-meter (optional).

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

13.2.5 Titration Vessel, 100 ml or 150 ml, preferably white.

13.2.6 Graduated Pipettes, 1 ml (TD) and 10 ml (TD).

13.2.7 Volumetric Pipette, 1 ml (TD).

13.2.8 Syringe, 1 ml (TD).

13.2.9 Stirring Rod.

13.3 Procedure—Phenolphthalein and Methyl Orange Filtrate Alkalinities

In order to measure alkalinities in a sample of drilling fluid filtrate, the following procedure shall be used:

a) Measure one or more milliliters of filtrate into the titration vessel. Add two or more drops of the phenolphthalein indicator solution, do not exceed ten drops. If the indicator turns pink, add 0.02 N (N/50) sulfuric acid, drop by drop from the graduated pipette, while stirring, until the pink 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, P₁, of the filtrate as the number of milliliters of 0.02 N sulfuric acid required per milliliter of filtrate.

c) To the sample that has been titrated to the P₁ endpoint, add two or three drops of methyl orange indicator solution. Add the standard acid drop by drop from the pipette, while stirring, until the 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, M₁, of the filtrate as the total milliliters of 0.02 N sulfuric acid per milliliter of filtrate required to reach the methyl orange endpoint (including that amount required for the P₁ 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 de-ionized or distilled water.
c) Add 4 to 5 drops of phenolphthalein indicator solution and if the indicator turns pink, while stirring, titrate rapidly with 0.02 N (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 0.02 N (N/50) sulfuric acid required per milliliter of drilling fluid.

NOTE Phenolphthalein alkalinity expressed in milliliters of 0.2N sulfuric acid per milliliter of drilling fluid is commonly known in the industry by the abbreviation $P_m$.

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 4.

<table>
<thead>
<tr>
<th>Relative values of $P_f$ and $M_f$</th>
<th>Concentration mg/l</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\text{OH}^-$</td>
</tr>
<tr>
<td>$P_f = 0$</td>
<td>0</td>
</tr>
<tr>
<td>$2 P_f &lt; M_f$</td>
<td>0</td>
</tr>
<tr>
<td>$2 P_f = M_f$</td>
<td>0</td>
</tr>
<tr>
<td>$2 P_f &gt; M_f$</td>
<td>$340 (2 P_f - M_f)$</td>
</tr>
<tr>
<td>$P_f = M_f$</td>
<td>$340 M_f$</td>
</tr>
</tbody>
</table>

13.6 Estimation of Lime Content

13.6.1 Determine the $P_f$ and $P_{df}$ of the filtrate and drilling fluid as described in 13.3 and 13.4.

Determine the volume fraction, $F_w$, of water in the drilling fluid using the value for volume fraction, expressed as a decimal fraction, of water from the liquid and solids determination (Section 9) in Equation (30):

$$F_w = \frac{\phi_w}{100}$$

(30)

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

13.6.2 Report the lime content of the drilling fluid, $c_{\text{lime,SI}}$, in kilograms per cubic meter, as given in Equation (31) [or $c_{\text{lime,USC}}$, in pounds per barrel, as given in Equation (32)]:

$$c_{\text{lime,SI}} = 0.742 \times (P_{df} - F_w P_f)$$

(31)
\[ c_{\text{lime,USC}} = 0.26 \times (P_{df} - F_W P_f) \]  
(32)

where

- \( F_W \) is the volume fraction of water in the drilling fluid, expressed as a decimal;
- \( P_{df} \) is the phenolphthalein alkalinity of the drilling fluid;
- \( P_f \) is the phenolphthalein alkalinity of the filtrate.

### 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 N; equivalent to 0.001 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 water.

**Warning**— Potassium Chromate (K\(_2\)CrO\(_4\) (CR\(^{VI}\)) 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, 0.02 N (N/50).

**Warning**— Sulfuric Acid (H\(_2\)SO\(_4\)) 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/water solution.

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

14.2.6 De-ionized or Distilled 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, the following procedure shall be used:

a) Measure 1 ml or more of filtrate into the titration vessel. Add 2 drops to 3 drops phenolphthalein solution. If the indicator turns pink, add acid drop by drop from pipette, while stirring, until the color has disappeared. If the filtrate was originally deeply colored, add an additional 2 ml of 0.02 N (N/50) sulfuric acid or nitric acid and stir. Then add 1 g calcium carbonate and stir.

b) Add 25 ml to 50 ml de-ionized water and 5 drops to 10 drops potassium chromate solution. Stir continuously, while adding standard silver nitrate solution equivalent to 0.001 g/ml (0.0282 N) 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, \( V_{sn} \), required to reach the endpoint. If over 10 ml of silver nitrate solution is used, repeat the test with a smaller sample of filtrate.

**NOTE**  If the chloride ion concentration of the filtrate exceeds 10,000 mg/l, a silver nitrate solution equivalent to 0.01 g/ml (0.282 N) chloride ion can be used. The factor 1000 in Equation (33) is then changed to 10,000.

#### 14.4 Calculation

14.4.1 Report the chloride ion concentration, \( c_{Cl^-} \) of the filtrate, expressed in milligrams per liter, calculated as given in Equation (33):
\[ c_{\text{Cl}} = 1000 \times \frac{V_{\text{sn}}}{V_f} \]  

(33)

where

- \( V_{\text{sn}} \) is the volume of silver nitrate solution, expressed in milliliters;
- \( V_f \) is the volume of the filtrate sample, expressed in milliliters.

14.4.2 Use Equation (34) to convert \( c_{\text{Cl}} \) to the sodium chloride concentration, \( c_{\text{NaCl,SI}} \), expressed in milligrams per liter:

\[ c_{\text{NaCl,SI}} = 1.65 \times c_{\text{Cl}} \]  

(34)

14.4.3 To convert sodium chloride concentration from milligrams per liter to parts per million by mass (ppm), use Equation (35):

\[ c_{\text{NaCl,PPM}} = \frac{c_{\text{NaCl,SI}}}{\rho_f} \]  

(35)

where

- \( c_{\text{NaCl,PPM}} \) is the sodium chloride concentration, expressed in parts per million by mass;
- \( c_{\text{NaCl,SI}} \) is the sodium chloride concentration, expressed in milligrams per liter;
- \( \rho_f \) is the density of the filtrate for sodium chloride, expressed in grams per milliliter, as given by Equation (25) and the chloride ion concentration \( c_{\text{Cl}} \), in milligrams per liter.

\[ \rho_f = 1 + (1.09 \times 10^{-6}) \times c_{\text{Cl}} \]  

(18)

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 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.

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 (CAS No. 12125-02-9) and 570 mL ammonium hydroxide (CAS No. 1336-21-6) (15 N) diluted to 1000 ml with de-ionized 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 distilled or de-ionized water.

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

Caution—Acetic acid (CH₃COOH) is corrosive. Avoid skin contact.

15.2.5 Masking Agent. 1:1:2 volume mixture of triethanolamine (CAS No. 102-71-6): tetraethylenepentamine (CAS No. 112-57-2): de-ionized or distilled water.

15.2.6 Sodium Hypochlorite (CAS No. 7681-52-9) solution, 5.25 % mass fraction in de-ionized 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 the sodium hypochlorite is fresh, as it will deteriorate with time.

15.2.7 De-ionized or Distilled Water.

The de-ionized water and sodium hypochlorite solution should be tested for hardness by using 50.0 ml of the de-ionized water and 10 ml of the sodium hypochlorite solution without the test sample, and continuing with 15.3.7 and 15.3.8. If the procedure is then repeated with the test sample utilizing 50 ml de-ionized water and 10 mL sodium hypochlorite solution in 15.3.2 through 15.3.6, the hardness of the test sample can be determined by subtracting the hardness of the de-ionized water and sodium hypochlorite solution.

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) and 5 ml (TD).

15.2.11 Hot plate (required if filtrate is colored).

15.2.12 pH-paper Strip.

15.3 Procedure

In order to conduct a total hardness as calcium check on a drilling fluid sample, the following procedure shall be used:

a) Measure one or more milliliters of sample into a 150 ml beaker. (If filtrate is clear, or is only lightly colored, omit steps 15.3.b through 15.3.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 de-ionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by
immersing a strip of pH-paper in the sample. If the paper is bleached white, continued boiling is required.

**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 de-ionized water and dilute the sample to 50 ml with de-ionized water. Add approximately 2 ml buffer solution and swirl to mix.

**NOTE** The presence of soluble iron can interfere with the endpoint determination. If this is suspected, a mixture of tri-ethanolamine:tetra-ethylene-pentamine:water (1:1:2 by volume) has proven to be a suitable masking agent. Add 1 ml of the Masking Agent to each titration.

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

h) 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 15.4.

### 15.4 Calculation

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

$$c_{Ca+Mg} = 400 \times \frac{V_{EDTA}}{V_s}$$  \hspace{1cm} (36)

where

- $V_{EDTA}$ is the titration volume of EDTA solution, expressed in milliliters;
- $V_s$ is the volume of the sample, expressed in milliliters.

**NOTE** The concentration of calcium and magnesium is commonly known in the industry as total hardness, and reported as calcium.
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 ethylenediaminetetraacetate dihydrate (1 ml/ml sample equivalent to 1000 mg CaCO₃, 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 (1 N) 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 (CH₃COOH) is corrosive. Avoid skin contact.

A.1.2.5 Titration Vessel, 150 ml beaker.

A.1.2.6 Graduated Pipettes, 1 mL (TD) and 10 mL (TD).

A.1.2.7 Hot Plate (required if filtrate is colored).


A.1.2.9 pH-paper.

A.1.2.10 Graduated Cylinder, 50 ml (TC).

A.1.2.11 Sodium Hypochlorite (CAS No. 7861-52-9), solution: 5.25 % mass fraction in de-ionized 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 should not be used. Ensure the sodium hypochlorite is fresh, as it deteriorates with time.

A.1.2.12 De-ionized or Distilled Water.

The de-ionized or distilled water and sodium hypochlorite solution should be tested for calcium by using 50.0 ml of the water and 10 ml of the sodium hypochlorite solution without the test sample. If the procedure is then repeated with the test sample, utilizing 50.0 ml of the water and 10 ml of the sodium hypochlorite solution as given in A.1.3, the calcium of the test sample can be determined by subtracting the calcium of the water and sodium hypochlorite solution.

A.1.3 Procedure

In order to conduct a test for calcium in a drilling fluid sample where magnesium is also present, the following procedure shall be used:

a) With a pipette, add 1 ml or more of filtrate sample (Section 8.2) to a 150 ml-beaker. This sample volume ($V_f$) is used in the calculation shown in Equation (A.1). If filtrate is colorless or is only slightly colored, omit A.1.3.2 through A.1.3.5.

b) With graduated pipette, add 10 ml sodium hypochlorite solution and mix.

c) With graduated pipette, add 1 ml glacial acetic acid and mix.

d) Boil the sample for 5 min. Maintain the sample by adding de-ionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH-paper in the sample. If the paper is bleached white, continued boiling is required. A sufficiently boiled sample shows a pH of 5.0.

e) Cool the sample.

f) Dilute the sample to approximately 50 ml with de-ionized or distilled water. Add 1ml to 2 ml of calcium buffer solution or sufficient sodium hydroxide to produce a pH of 12 to 13.

NOTE 1 If steps A.1.3.b – A.1.3.e were performed it may be necessary to use as much as 10-15 ml of calcium buffer solution to neutralize the glacial acetic acid.

NOTE 2 The presence of soluble iron can interfere with the endpoint determination. If soluble iron is suspected, add 1 ml of a suitable masking agent after step A.1.3.6. A mixture of tri-ethanolamine:tetra-ethylene-pentamine:water (1:1:2 by volume) is a suitable masking agent.

g) Add sufficient calcium indicator (0.1 g to 0.2 g) to produce a pink to wine-red color 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.

h) While stirring, titrate with standard EDTA (0.01 mol/L) 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 volume of the standard EDTA ($V_{EDTA}$) added is used in the calculation in Equation (A.1).

A.1.4 Calculation

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

$$c_{Ca} = 400 \times \frac{V_{EDTA}}{V_f}$$

(A.1)

where
$c_{Ca}$ is the calcium ion concentration, expressed in milligrams per liter; 

$V_{EDTA}$ is the titration volume of EDTA solution, expressed in milliliters ($1 \text{ ml} \equiv 400 \text{ mg Ca}^{2+}$);

$V_f$ is the volume of the filtrate sample, 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 which is converted to magnesium by multiplying the value by the ratio of atomic weights ($24.3/40 = 0.6$).

A.2.2 Procedure

a) Determine the total hardness as calcium, $c_{Ca+Mg}$, as described in Section 15.

b) Determine the calcium content, $c_{Ca}$, as described in A.1, above.

A.2.3 Calculation

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

$$c_{Mg} = 0.6 \times c_{Ca+Mg} \left[ -c_{Ca} \right]$$

(A.2)

where

- $c_{Ca+Mg}$ is the total hardness (Section 15), expressed as calcium in milligrams per liter;
- $c_{Ca}$ is the calcium concentration (A.1), expressed in milligrams per liter.

A.3 Calcium Sulfate

A.3.1 Principle

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

A.3.2 Reagents and Apparatus

A.3.2.1 EDTA (CAS No. 6381-92-6): 0.01 mol solution, standardized disodium 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 (1 N) 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 Acetic acid (CAS No. 64-19-7), glacial.

Warning—CH₃COOH is corrosive. Avoid skin contact.

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

A.3.2.6 Sodium Hypochlorite (CAS No. 7861-52-9) solution, mass fraction of 5.25 % in de-ionized or distilled water.

Warning—NaClO is highly corrosive and can cause burns and damage the eyes.

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

A.3.2.7 De-ionized or Distilled Water.

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

A.3.2.8 Titration vessel, 150 ml beaker.

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

A.3.2.10 Hot Plate (required, if filtrate is colored).

A.3.2.11 pH-paper.

A.3.2.12 Graduated Cylinder, 50 ml (TC).

A.3.2.13 Drilling Fluid Retort, as described in Section 9.

A.3.3 Procedure

In order to conduct a calcium sulfate test on a sample of drilling fluid, the following procedure shall be followed:

a) Add 5 ml of whole drilling fluid to 245 ml de-ionized water. Stir the mixture for 15 min and filter through a standard filter press in accordance with Section 8.2.

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 Section A.1 and designate this titration volume of EDTA as \( V_{EDTA,df} \).

c) Titrate 1 ml of the original drilling fluid filtrate (obtained as described in Section 8.2) to the EDTA endpoint. Designate this titration volume of EDTA as \( V_{EDTA,f} \).
d) Determine the volume fraction of water, \( F_W \), expressed as a decimal, in the drilling fluid by using the value for volume fraction, \( \phi_W \), expressed as a percent, of water from the liquid and solids determination (Section 9) and Equation (A.3):

\[
F_W = \frac{\phi_W}{100}
\]

(A.3)

**A.3.4 Calculation**

**A.3.4.1** Calculate the calcium sulfate concentration of the drilling fluid, \( c_{CaSO_4,SI} \), expressed kilograms per cubic meter, as given in Equation (A.4), [or \( c_{CaSO_4,USC} \) expressed in pounds per barrel, as given in Equation (A.5)].

\[
c_{CaSO_4,SI} = 6.60 \times V_{EDTA,df}
\]

(A.4)

\[
c_{CaSO_4,USC} = 2.31 \times V_{EDTA,df}
\]

(A.5)

where

\( V_{EDTA,df} \) is the titration volume of EDTA solution of whole drilling fluid sample (see A.3.3.b), expressed in milliliters.

**A.3.4.2** Calculate the (excess) undissolved calcium sulfate concentration of the drilling fluid, \( c_{ex-CaSO_4,SI} \), expressed in kilograms per cubic meter, as given in Equation (A.6) [or \( c_{ex-CaSO_4,USC} \), expressed in pounds per barrel, as given in Equation (A.7)].

\[
c_{ex-CaSO_4,SI} = 6.6 \times V_{EDTA,df} - 1.32 \times V_{EDTA,f} \times F_W
\]

(A.6)

\[
c_{ex-CaSO_4,USC} = 2.31 \times V_{EDTA,df} - 0.463 \times V_{EDTA,f} \times F_W
\]

(A.7)

where

\( c_{ex-CaSO_4,SI} \) is the excess undissolved calcium sulfate concentration, expressed in kilograms per cubic meter;

\( c_{ex-CaSO_4,USC} \) is the excess undissolved calcium sulfate concentration, expressed in pounds per barrel;

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

\( V_{EDTA,df} \) is the titration volume of EDTA solution of whole drilling fluid sample; see A.3.3.b;

\( V_{EDTA,f} \) is the titration volume of EDTA solution of the drilling fluid filtrate; see A.3.3.c.

**A.4 Sulfide**

**A.4.1 Principle**

**A.4.1.1** The concentration of soluble sulfides present in a drilling fluid can be determined by this method. Soluble sulfides include \( H_2S \) and the sulfide (\( S^- \)) and bisulfide (\( HS^- \)) ions. In this procedure, the drilling
fluid filtrate is acidified in a Garrett gas train, converting sulfides to H₂S, which is 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 a Dräger indicator tube, which responds to H₂S by darkening along its' length. The darkened length is proportional to the total sulfide in the drilling fluid filtrate. The low-range Dräger tube turns from white to brownish-black and the high-range Dräger tube turns from pale blue to jet-black. No known common drilling fluid contaminants cause these color changes.

A.4.1.2 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 should always 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—H₂SO₄ is a strong and toxic acid.

A.4.2.2 Defoamer, in a dropper bottle.

A.4.2.3 De-ionized or Distilled Water.

A.4.2.4 Carrier gas, inert to hydrogen sulfide, acid and gas detection Dräger tube reagents.

Nitrogen is preferred but carbon dioxide is acceptable. (Avoid air or other oxygen-containing gases, such as nitrous oxide.)

A.4.2.5 H₂S Gas detection tubes: Dräger H₂S analysis tubes or equivalent:

— Low range: marked H₂S 100/a (No. CH 29101, 100 mg/l to 2000 mg/l H₂S);
— High range: marked H₂S 0.2%/A (No. CH 28101, 0.2 volume % to 7 volume % H₂S).

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:

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:
Dispersed frit (bell-shaped, fine):

- **Diameter**: 30 mm (1.18 in.)
- **Material**: Low coefficient of expansion, heat-resistant glass

A.4.2.7 **Lead-acetate paper disk** (see A.4.3.q).

A.4.2.8 **Hypodermic syringes**, 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 followed:

- a) 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.

- b) Add 20 ml of de-ionized water to chamber 1.

- c) Add 5 drops defoamer to chamber 1.

- d) 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.

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

- f) Install the top on the gas train and hand-tighten the screws evenly to seal the O-rings.
Table A.1—Dräger Tube (or Equivalent) Identification, Sample Volume and Tube Factors for Various Sulfide Ranges

<table>
<thead>
<tr>
<th>Sulfide range mg/l</th>
<th>Sample volume $V_s$ ml</th>
<th>Dräger Tube Identification$^a$</th>
<th>Tube factor$^{b,c,f}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.2 to 24</td>
<td>10.0</td>
<td>$H_2S$ 100/a</td>
<td>0.12$^d$</td>
</tr>
<tr>
<td>2.4 to 48</td>
<td>5.0</td>
<td>$H_2S$ 100/a</td>
<td>0.12$^d$</td>
</tr>
<tr>
<td>4.8 to 96</td>
<td>2.5</td>
<td>$H_2S$ 100/a</td>
<td>0.12$^d$</td>
</tr>
<tr>
<td>30 to 1050</td>
<td>10.0</td>
<td>$H_2S$ 0.2 %/a</td>
<td>1500$^e$</td>
</tr>
<tr>
<td>60 to 2100</td>
<td>5.0</td>
<td>$H_2S$ 0.2 %/a</td>
<td>1500$^e$</td>
</tr>
<tr>
<td>120 to 4200</td>
<td>2.5</td>
<td>$H_2S$ 0.2 %/a</td>
<td>1500$^e$</td>
</tr>
</tbody>
</table>

$^a$ 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 to 17 milliliter scale, use a tube factor 600 times the ratio: Batch Factor/0.40.

---

g) 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 to the dispersion tube.

h) Attach the flexible tubing from the outlet of chamber 3 to the gas detection Dräger tube.

i) 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 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 should 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.

Continuously observe changes in appearance of the gas detection Dräger tube. Record the maximum darkened length (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 $\text{SO}_2$) can appear ahead of the black front if sulfites are present in the sample. The orange $\text{SO}_2$ region should be ignored when recording the darkened length.

p) For best gas detection Dräger tube accuracy, the “darkened length” should fill more than half the tube’s length, but less than 90% of the total length, therefore the filtrate “sample volume” and gas detection Dräger tube combination shall be carefully selected.

q) 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.

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

A.4.4 Calculation

The sulfide concentration, $c_s$, expressed in milligrams per liter, in the filtrate sample can be calculated as given in Equation (A.8):

$$c_s = \frac{l_{st} \times f}{V_s}$$

(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;
- $f$ is the tube factor from Table A.1.

A.5 Carbonate

A.5.1 Principle

The concentration of soluble carbonates in a drilling fluid filtrate can be determined by this method. Total soluble carbonates include $\text{CO}_2$ and the carbonate ($\text{CO}_3^{2-}$) and bicarbonate ($\text{HCO}_3^-$) ions. Drilling fluid filtrate is acidified in a Garrett gas train, converting all carbonates to $\text{CO}_2$, which is then evolved by bubbling an inert carrier gas through the sample. The gas train separates the gas from the liquid. The gas stream is collected in a 1 L gas bag (to allow $\text{CO}_2$ to mix uniformly) and subsequently drawn through a Dräger tube at a fixed flowrate. The Dräger tube responds to $\text{CO}_2$ by progressively staining purple along its length. A reaction between $\text{CO}_2$ 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.4.2.5.

A.5.2.2 Carrier gas, high-purity nitrogen (N$_2$) bottle with low-pressure regulator (preferred), or N$_2$O gas cartridges.

Warning—Do not use nitrous oxide cartridges as pressure sources for high-temperature/high pressure (HTHP) filtration. Under high temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials.

NOTE Use nitrous oxide cartridges only for Garrett gas train carbonate analysis; do not use for sulfide determination.

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 Dräger Hand-operated bellows gas-detector vacuum pump, or equivalent.

A.5.2.6 Stopcock, 2-way bore, 8 mm (0.315 in.) glass with PTFE plug.

A.5.2.7 Sulfuric acid (CAS No. 7664-93-9), approximately 2.5 mol/l (5 N), ACS reagent grade.

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

A.5.2.8 Defoamer, in a dropper bottle.

A.5.2.9 De-ionized or Distilled Water

A.5.2.10 Hypodermic syringes, 1.0 ml, 5 ml and 10 ml (for acid) and 10 ml (for sample).

A.5.2.11 Hypodermic needles, 40mm (1.5 in.) 21-gauge needles.

NOTE Nitrogen is preferred over N$_2$O as the carrier gas because N$_2$O cools upon expansion and chills the diaphragm in the regulator, prolonged N$_2$O flow causes the regulator to perform erratically.

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 followed:

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 should be purged with carrier gas at this time.

b) Add 20 mL de-ionized 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 a measured sample volume, \( V_s \), of solids-free, API low pressure/low temperature filtrate into chamber 1 through the septum with a hypodermic syringe and needle. See Table A.2.

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.

Caution—Avoid transfer of fluid from chamber 1 to chamber 2 while shaking.

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

<table>
<thead>
<tr>
<th>Carbonate range (mg/l)</th>
<th>Sample volume ( V_s ) (ml)</th>
<th>Dräger Tube Identification</th>
<th>Tube factor (^{b,c})</th>
</tr>
</thead>
<tbody>
<tr>
<td>25 to 750</td>
<td>10.0</td>
<td>CO(_2) 100/a</td>
<td>2.5 d</td>
</tr>
<tr>
<td>50 to 1500</td>
<td>5.0</td>
<td>CO(_2) 100/a</td>
<td>2.5 d</td>
</tr>
<tr>
<td>100 to 3000</td>
<td>2.5</td>
<td>CO(_2) 100/a</td>
<td>2.5 d</td>
</tr>
<tr>
<td>250 to 7500</td>
<td>1.0</td>
<td>CO(_2) 100/a</td>
<td>2.5 d</td>
</tr>
</tbody>
</table>

\(^a\) 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.

l) Open the stopcock on the gas bag. Restart gas flow and allow gas bag to fill steadily during a 10 min interval. When bag is firm to the touch (do not burst it) shut off flow and close the stopcock. Immediately proceed to 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 to the downstream end of the gas detection Dräger 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 ten strokes indicates that leakage has occurred and that the test results will not be correct.)
p) When CO₂ is present in the gas bag, a purple stain will develop along the length of the gas detection Dräger tube. Observe and record the stain length 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 Dräger tube accuracy, the “stain length” should fill more than 50 % of the tube length, but not exceed 90 %. If the stain length is outside the 50-90 % 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 de-ionized 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 ten analyses).

A.5.4 Calculation

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

\[
c_{CO2+CO3+HCO3} = \frac{L_{st} \times f}{V_s}
\]

(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;
- \( f \) is the tube factor from Table A.2.
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, which is equivalent to or greater than 10 kg/l (3.5 lb/bbl) potassium chloride. Potassium is precipitated in a centrifuge tube as the perchlorate salt and the volume of precipitate is measured. The potassium ion content is read from a prepared standard curve.

B.1.2 Reagents and Apparatus

B.1.2.1 Sodium perchlorate (NaClO₄) (CAS No. 7601-89-0), solution: 150.0 g/100 ml de-ionized or distilled water.

Warning—Perchlorates, NaClO₄ and KClO₄ 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 made up to 100 ml with de-ionized or distilled water.

B.1.2.3 Centrifuge, horizontal-swing rotor head (manual or electric), capable of producing approximately 1800 r/min.

NOTE 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. (For example, 15 revolutions of the rotor per one turn of the crank.) Calculate the number of crank turns required to obtain 1800 revolutions of the rotor head. In the example, to obtain 1800 revolutions of the head would require 120 turns of the crank. Thus the crank must be turned 120 times in one minute to obtain the rate of 1800 r/min. At this rate, in 5 s the handle must be turned 10 times [i.e. (120/60)x5 =10]. By counting the crank turns in 5 s and adjusting the rate to obtain the required number of turns, a constant 1800 r/min can be obtained in 15 s to 20 s. It is necessary to add the interval used to adjust to the 1800 r/min to the centrifuge time of the sample.

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 De-ionized or distilled water.

B.1.3 Preparation of standard calibration curve
In order to prepare a standard calibration curve to determine potassium ion content in a sample of drilling fluid, a standard calibration curve is required for each type of centrifuge. To calculate the curve, the following procedure shall be followed:

a) A standard calibration curve is required for each type of centrifuge. A minimum of three points [10 kg/m³ (3.5 lb/bbl), 30 kg/m³ (10.5 lb/bbl) and 50 kg/m³ (17.5 lb/bbl) KCl] is required to obtain an accurate graph.

b) Samples can be prepared by using the standard potassium chloride solution [0.5 ml of standard potassium chloride solution is equivalent to 10 kg/m³ (3.5 lb/bbl) KCl]. To obtain a KCl calibration curve for concentrations between 10 kg/m³ (3.5 lb/bbl) and to 50 kg/m³ (17.5 lb/bbl) KCl; use aliquots of 0.5 mL, 1.5 mL, and 2.5 mL of standard potassium chloride solution into 3 separate centrifuge tubes.

c) Dilute each sample to the 7.0 ml mark on the centrifuge tube with de-ionized or distilled water and agitate.

d) Add 3.0 ml of standard sodium perchlorate solution (but do not agitate) to each tube.

e) Centrifuge at a constant speed (approximately 1800 r/min) for 1 min and read the precipitate volume immediately.

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) Plot the volume of precipitate (milliliters) versus potassium chloride content in kilograms per cubic meter (or pounds per barrel) on rectangular graph paper as shown in Figure B.1.

Key

- X1 \( c_{KCl,SI} \), expressed in kilograms per cubic meter
- X2 \( c_{KCl,USC} \), expressed in pounds per barrel
Y \quad \text{volume of precipitate, expressed in milliliters}

a \quad \text{Do not use this procedure for concentrations less than 10 kg/m}^3\ (3.5 \text{ lb/bbl}).

b \quad \text{Refer to Table B.1 beyond this point.}

Figure B.1—Example of Plotted Calibration Curve for Potassium Chloride (do not use for calibration)

B.1.4 Test Procedure

In order to conduct a test for potassium ions in a concentration above 5000 mg/l in a drilling fluid, the following procedure shall 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 de-ionized 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 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 to 3 drops of additional sodium perchlorate solution to the tube. If no precipitate forms proceed to B.1.4.f. If 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.a through B.1.4.e.

d) Determine the potassium chloride concentration of the diluted filtrate test sample by comparing the precipitate volume measured with the standard calibration curve as prepared in B.1.3. Record the potassium chloride concentration in diluted filtrate test sample as $c_{KCl,SI}$, expressed in kilograms per cubic meter or as $c_{KCl,USC}$, expressed in pounds per barrel.

The potassium concentration may also be reported as milligrams per liter potassium ion.

If the diluted filtrate test sample potassium chloride concentration, $c_{KCl,SI}$ ($c_{KCl,USC}$), from the standard calibration curve exceeds a 50 kg/m$^3$ (18 lb/bbl) reading, accuracy of the results is reduced. For more accurate results, use the next smaller filtrate volume as noted in Table B.1, and repeat B.1.4.a through B.1.4.e.

B.1.5 Calculation
B.1.5.1 Calculate the filtrate potassium chloride concentration, $c_{\text{KCl,SI}}$, expressed in milligrams per liter, as given in Equation (B.1) [or $c_{\text{KCl,USC}}$, expressed in pounds per barrel, as given in Equation (B.2)]:

$$c_{\text{KCl,SI}} = \left( \frac{7}{V_f} \right) \times c_{\text{KCl,SI}}$$  \hspace{1cm} (B.1)

$$c_{\text{KCl,USC}} = \left( \frac{7}{V_f} \right) \times c_{\text{KCl,USC}}$$  \hspace{1cm} (B.2)

where

- $c_{\text{KCl,SI}}$ is the diluted filtrate test sample potassium chloride concentration expressed in kilograms per cubic meter;
- $c_{\text{KCl,USC}}$ is the diluted filtrate test sample potassium chloride concentration expressed in pounds per barrel;
- $V_f$ is the volume of filtrate sample, expressed in milliliters.

NOTE $c_{\text{KCl,SI}}$ is the corresponding potassium chloride concentration on the X1 axis of the standard calibration curve (see Figure B.1), and $c_{\text{KCl,USC}}$ is the corresponding potassium chloride concentration on the X2 axis of the standard curve (see Figure B.1).

B.1.5.2 Calculate the filtrate potassium ion concentration, $c_{\text{K,f}}$, expressed in milligrams per liter, from $c_{\text{KCl,SI}}$ expressed in kilograms per cubic meter, as given in Equation (B.3) [or from $c_{\text{KCl,USC}}$, expressed in pounds per barrel, as given in Equation (B.4)]:

$$c_{\text{K,f}} = 525 \times c_{\text{KCl,SI}}$$  \hspace{1cm} (B.3)

$$c_{\text{K,f}} = 1500 \times c_{\text{KCl,USC}}$$  \hspace{1cm} (B.4)

where $c_{\text{KCl,SI}}$ and $c_{\text{KCl,USC}}$ are as defined above.

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, $c_{\text{K,f}}$, in the sample is calculated by subtracting the amount of unreacted STPB from the amount of STPB originally added to the sample.

B.2.2 Reagents and Apparatus
B.2.2.1 **Standard sodium tetraphenylborate (STPB)** (CAS No. 143-66-8), solution: 8.754 g in 800 ml de-ionized 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 liter with de-ionized or distilled water.

B.2.2.2 **Quaternary ammonium salt (QAS)** (CAS No. 57-09-0), solution: 1.165 g hexadecyltrimethyl ammonium bromide per 500 ml de-ionized or distilled water.

B.2.2.3 **Sodium hydroxide** (CAS No. 1310-73-2), solution: 20 % mass fraction in de-ionized 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 de-ionized or distilled water.

B.2.2.5 **De-ionized** or **distilled** 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 **Funnel**.

B.2.2.10 **Filter paper**. Grade 1573 1/2 pleated filter paper, 18.5 cm (7.28 in.) diameter, 12-25 µm retention.

B.2.2.11 **Stirring Rod**, glass or inert to acid or caustic, 25 cm (10 in.) length.

B.2.3 **Procedure**

In order to conduct a test on potassium ions in a drilling fluid sample at concentrations below 5000 mg/l, the following procedure shall be followed:

a) Place the proper amount of filtrate sample, from the low temperature/low pressure filtration test (Section 8.2), into a 100 ml graduated cylinder. Use Table B.2 to determine the appropriate filtrate sample size. Be sure to use a pipette to accurately measure the amount of filtrate.
Table B.2—Filtrate Volumes to be used at Various KCl Concentrations

<table>
<thead>
<tr>
<th>KCl concentration range</th>
<th>$c_{KCl,SI}$ (kg/m³)</th>
<th>$c_{KCl,USC}$ (lb/bbl)</th>
<th>K in filtrate (mg/l)</th>
<th>Filtrate volume to use ($V_f$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5 to 3.0</td>
<td>0.18 to 1.05</td>
<td>263 to 1575</td>
<td>10.0</td>
<td></td>
</tr>
<tr>
<td>3.0 to 6.0</td>
<td>1.05 to 2.1</td>
<td>1575 to 3150</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>6.0 to 20.0</td>
<td>2.1 to 7.0</td>
<td>3150 to 10,500</td>
<td>2.0</td>
<td></td>
</tr>
</tbody>
</table>

b) Using a 5 ml pipette, add 4 ml of NaOH solution (20 % mass fraction) to the 100 ml graduated cylinder, containing the filtrate sample (B.2.3.a). Using a 25 ml graduated cylinder, add 25 ml of STPB solution to the 100 ml graduated cylinder, and add de-ionized water to bring the level of the solution to the 100 ml mark.

c) Mix thoroughly with a stirring rod and allow solution to stand for 10 min.

d) Using the funnel and fluted filter paper, filter the solution above into a clean/dry 100 ml graduated cylinder. If the filtrate is cloudy, re-filter the solution until it is clear.

e) Transfer 25 ml of the filtered solution above, (measured with a 25 ml graduated cylinder) into a 250 ml beaker.

f) Add 10 to 15 drops of bromophenol blue indicator, producing a purple-blue solution.

g) Titrate slowly with QAS solution until color changes from purple-blue to light blue.

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

The ratio, $R_{QAS/STPB}$, of QAS to STPB is calculated as given in Equation (B.5):

$$R_{QAS/STPB} = \frac{V_{QAS}}{2}$$  \hspace{1cm} (B.5)

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

If the ratio is other than 4.0 ±0.5, calculate a correction factor, $k_{cor}$, expressed in milligrams per liter, as given in Equation (B.6) for use in the calculation of the potassium ion concentration in Equation (B.8):

$$k_{cor} = \frac{8}{V_{QAS}}$$  \hspace{1cm} (B.6)

B.2.4 Calculation

B.2.4.1 If the ratio falls in the 4.0 ±0.5 range, the potassium ion concentration in the filtrate, $c_{K,f-VQAS}$, in milligrams per liter, is calculated using $V_{QAS}$ as given in Equation (B.7):

$$c_{K,f-VQAS} = \frac{1000 \times (25 - V_{QAS})}{V_f}$$  \hspace{1cm} (B.7)
where \( V_f \) is the filtrate sample volume, expressed in milliliters.

**B.2.4.2** If a correction factor is necessary, calculate the potassium ion concentration in the filtrate, \( c_{K,f-VQAS} \), expressed in milligrams per liter, as given in Equation (B.8):

\[
c_{K,f-VQAS} = 1000 \left( \frac{25 - (k_{cor} \times V_{QAS})}{V_f} \right)
\]

(B.8)

**B.2.4.3** Calculate the potassium chloride concentration in the filtrate, \( c_{KCl,f,SI} \), expressed in kilograms per cubic meter, as given in Equation (B.9) [or \( c_{KCl,f,USC} \), expressed in pounds per barrel, using Equation (B.10)]:

\[
c_{KCl,f,SI} = \frac{c_{K,f-VQAS}}{525}
\]

(B.9)

\[
c_{KCl,f,USC} = \frac{c_{K,f-VQAS}}{1500}
\]

(B.10)

where \( c_{K,f-VQAS} \) is defined above, Equation (B.8).
Annex C
(normative)

Shear Strength Measurement 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, therefore, 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 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 the estimated bottom-hole temperature of the well. Aging cells or vessels meeting the pressure and temperature requirements for the test are required.

C.2 Apparatus

C.2.1 Stainless Steel or Aluminum Shearometer Tube, with the following characteristics:
- length 89 mm (3.5 in.);
- outside diameter 36 mm (1.4 in.);
- wall thickness 0.2 mm (0.008 in.).

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

C.2.2 Platform, suitable to support weights on top of the tube. The mass of the platform shall 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

In order to conduct a shear strength measurement using a shearometer in a heat-aged drilling fluid sample, the following procedure shall be followed:

a) The shear tube and platform are placed and balanced carefully on the surface of the aged sample cooled to room temperature. It can be necessary to shift the weights on the platform to assure that the tube’s initial penetration into the drilling fluid is vertical. If a crust develops on the heat-aged sample, this crust should be gently broken before placing the shear tube in place for the test.

b) Sufficient weights are placed carefully on the platform to start the downward movement of the shear tube. Unless too much mass is added, the tube will stop its downward travel at the point where the shear strength of the aged drilling fluid against the surface of the tube is sufficient to support the applied mass. It is desirable to submerge at least one-half the length of the tube.
c) Record the total mass in grams, which includes the platform and weights. Measure the portion of the tube submerged in the fluid, in centimeters (inches). The length of the tube submerged can be most accurately determined by measuring the length of the non-submerged portion while the tube is at its maximum penetration depth. A small ruler held at the drilling fluid surface and alongside the tube facilitates this measurement. The length of the tube minus the exposed length equals the submerged portion.

C.4 Calculation

C.4.1 Calculate the shear strength, $\gamma_{\text{USC}}$, expressed in pounds per 100 square feet, as given in Equation (C.1), [or $\gamma_{\text{SI}}$, expressed in pascals, as given in Equation (C.2)]:

$$\gamma_{\text{USC}} = \frac{3.61 \times m_{\text{st}} + m_{\text{tot}}}{l_{\text{USC}}} - 0.256 \rho_{df,\text{USC}} \quad (C.1)$$

where

- $m_{\text{st}}$ is the mass of the shear tube, expressed in grams;
- $m_{\text{tot}}$ is the total shear mass (sum of platform and weights), expressed in grams;
- $l_{\text{USC}}$ is the submerged length of shear tube, expressed in inches;
- $\rho_{df,\text{USC}}$ is the drilling fluid density, expressed in pound per gallon.

C.4.2 Calculate the shear strength, $\gamma_{\text{SI}}$, expressed in pascals, as given in Equation (C.2):

$$\gamma_{\text{SI}} = \frac{4.40 \times m_{\text{st}} + m_{\text{tot}}}{l_{\text{SI}}} - 1.02 \rho_{df} \quad (C.2)$$

where

- $m_{\text{st}}$ is the mass of the shear tube, expressed in grams;
- $m_{\text{tot}}$ is the total shear mass (sum of platform and weights), expressed in grams;
- $l_{\text{SI}}$ is the submerged length of shear tube, expressed in centimeters;
- $\rho_{df}$ is the drilling fluid density, expressed in grams per milliliters.
Annex D
(informative)

Resistivity

D.1 Principle
Control of the resistivity of a drilling fluid and drilling fluid filtrate can be desirable to better evaluate formation characteristics from electric logs.

D.2 Apparatus
D.2.1 Direct-reading Resistivity Meter, or similar resistivity meter. Follow manufacturer's instructions for current source, calibration, measurement and calculations.
D.2.2 Calibrated Resistivity Cell.
D.2.3 Thermometer, reading 0 °C to 105 °C (32 °F to 220 °F), accurate to 0.5 °C (1 °F).
D.2.4 Bottle Brush, suitable for size and type of cell.
D.2.5 Laboratory Detergent Solution, appropriate for cleaning metal or plastic surfaces.

D.3 Procedure
In order to conduct a resistivity test on a drilling fluid sample, the following procedure shall be followed:

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.
b) Connect cell to resistivity meter.
c) 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.
d) Measure the sample temperature to the nearest 0.5 °C (1 °F).
e) Clean the cell. Scrub with brush and detergent if necessary. Rinse with distilled or de-ionized water and allow to dry.

D.4 Calculation
D.4.1 Report the drilling fluid resistivity, \( r_{df} \), or filtrate resistivity, \( r_f \), in ohm-meters, to the nearest 0.01 ohm-meters.
D.4.2 Report the sample temperature in degrees Celsius (Fahrenheit).
D.4.3 If the reading, \( R_r \), is in ohms, convert to ohm-meters as given in Equations (D.1) and (D.2):

\[
r_{df} = R_r \times K \quad \text{(D.1)}
\]
\[ r_1 = R_t \times K \]  \hspace{1cm} (D.2)

where

- \( r_{df} \) is the drilling fluid resistivity, expressed in ohms
- \( r_f \) is the filtrate resistivity, expressed in ohm\( \cdot \)meters
- \( K \) is the cell constant, expressed in squared meters per meter;
- \( R_t \) is the meter reading, expressed in ohms.
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 procedure can be followed to de-aerate the drilling fluid.

NOTE If drilling fluid density is the only property desired, the pressurized fluid density balance described in Section 6 can be used. Solids analysis (retort, Section 9) is sensitive to the presence of entrained air. Careful de-aeration of the fluid sample used in the retort, is essential in the measurement of solids content.

E.2 Apparatus

E.2.1 Fluid Reservoir, with gasketed cap and stirring apparatus, which can be evacuated.

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 shall 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 seal reservoir with gasketed cap.

d) Affix vacuum line from the pump to the reservoir in order to hold about 83 kPa [620 mm Hg (24.4 in. Hg)] vacuum.

e) Increase the vacuum to 10 kPa to 16 kPa [75 mm Hg to 120 mm Hg (3.0 in. Hg to 4.7 in. Hg)] and proceed according to the manufacturer’s instructions.

f) When drilling fluid has been de-aerated, partially relieve vacuum to about 50 kPa to 65 kPa [375 mm Hg to 490 mm Hg (14.8 in. Hg to 19.3 in. Hg)] vacuum and observe drilling fluid for air bubbles.

g) With fluid reservoir upright, relieve vacuum completely and remove drilling fluid sample for testing.

h) If de-aeration is not sufficient to reach expected fluid density, repeat E.3.d to E.3.g until air is removed.
Annex F
(normative)

Drill Pipe Corrosion Ring Coupon

F.1 Principle

F.1.1 The placement of corrosion test rings in the drill string is one of the more common techniques used to evaluate the corrosiveness of drilling fluid environments on the drill string and other steel equipment. Removal and examination of these rings after a period of exposure downhole can be highly informative as to the 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.

F.1.2 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, mass fraction of 15 % in de-ionized or distilled water.

Warning—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 De-ionized or Distilled Water.

F.2.5 Detergent Solution.

F.2.6 Corrosion Ring.

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 stenciled 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. Allow to dry, weigh to nearest milligram and record this mass on the report form. Store the ring in a dry container, such as a desiccator, to prevent corrosion. The corrosion rings should be shipped to the field in sealed envelopes or wrappers to minimize atmospheric corrosion.

F.2.7 Ultrasonic Bath, (preferred) or Brush, fiber-bristle, or fine-grade (000) steel wool.

F.2.8 Gloves, acid resistant, part of personal protective equipment when handling strong acids.

F.2.9 Iron Sulfide Test Solution—Acid arsenate test.

F.3 Procedure

In order 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 followed:

a) Drill-pipe corrosion rings should be kept in the drill string for a minimum of 40 hr (a normal time for exposure is 100 hr). Exposure periods of less than 40 hr should not be used because initial corrosion rates can be unusually high and can give misleading data. The ring is usually placed in the tool joint at the top of the first stand above the drill collars and can be left in the drill string for more than one bit run. An additional ring can be placed in the kelly-saver sub to monitor corrosion at that point. 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 scale and any other information of significance in the specific test. The form may be printed on a mailing envelope for the ring or on a separate form to be enclosed with the ring.

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 wrapper containing vapor-phase corrosion inhibitor to return the coupon to the laboratory.

d) The recommended cleaning solution is an aqueous detergent solution, not a strong solvent like acetone or methanol. Prior to cleaning for weighing, a spot test should be made for corrosion by-products and mineral scale. For example, the surface can be examined qualitatively for sulfides by the acid arsenate (iron sulfide test solution) test. The rings should be cleaned with a detergent solution and a stiff, fiber-bristle brush. It may be necessary to dip the ring in inhibited 10 % to 15 % hydrochloric acid solution one or more times to remove corrosion products for 5 s to 10 s. The ring should be scrubbed with detergent solution after each acid dip. Rinse thoroughly with clean de-ionized water and then with 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.

e) After the pre-weighed drill pipe corrosion coupon has been properly cleaned and the corrosion film and type of attack noted, the ring should be weighed to the nearest milligram and the mass loss
determined. If significant loss of metal due to mechanical damage is evident, it should be noted and taken into consideration in evaluation of the ring. The corrosion rate may be reported as kg/(m² • year) or mm/year (lb/(ft² • year) or mils/year).

f) Equations for calculating for corrosion rate are given in Section F.5.

F.4 Comments on Visual Examination

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

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

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

F.5 Calculation

F.5.1 The corrosion rate, \( q_{\text{SI}} \), expressed in kilograms per (square meter • year), is calculated according to Equation (F.1)

\[
q_{\text{SI}} = \left( \frac{\Delta m}{1 \times 10^6} \right) \left( \frac{1 \times 10^4}{A_{\text{SI}}} \right) \left( \frac{8760}{t} \right) = 87.6 \times \left( \frac{\Delta m}{A_{\text{SI}} \times t} \right)
\]  

(F.1)

where

\( \Delta m \) is the mass loss, expressed in milligrams;

\( A_{\text{SI}} \) is the area, expressed in square centimeters;

\( t \) is the exposure time, expressed in hours.

and as \( q_{\text{USC}} \), expressed in kilograms per (square foot • year), as given in Equation (F.2):

\[
q_{\text{USC}} = \left( \frac{\Delta m}{453,600} \right) \left( \frac{144}{A_{\text{USC}}} \right) \left( \frac{8760}{t} \right) = 2.781 \times \left( \frac{\Delta m}{A_{\text{USC}} \times t} \right)
\]  

(F.2)

where

\( \Delta m \) is the mass loss, expressed in milligrams;

\( A_{\text{USC}} \) is the area, expressed in square inches;

\( t \) is the exposure time, expressed in hours.
NOTE  Total surface area of the ring is used in these calculations.

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

NOTE  Equations (F.3) to (F.7) give the conversion rates between the various units for steel coupons (with a relative density of 7.86):

\[
\begin{align*}
\text{mils/year} & = 5.01 \times q_{\text{SI}} \quad \text{(F.3)} \\
\text{mils/year} & = 24.6 \times q_{\text{USC}} \quad \text{(F.4)} \\
\text{mm/year} & = 0.127 \times q_{\text{SI}} \quad \text{(F.5)} \\
\text{mm/year} & = 0.621 \times q_{\text{USC}} \quad \text{(F.6)} \\
\text{lb/(ft}^2\text{•year)} & = 1.61 \times \text{(mm/year)} \quad \text{(F.7)}
\end{align*}
\]

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

Sampling, inspection and rejection

G.1 General

The following procedure provides the sampling, inspection and rejection method for materials conforming to API 13A. It is applicable to barite, hematite, bentonite, nontreated bentonite, attapulgite, sepiolite, technical-grade low-viscosity carboxymethylcellulose, technical-grade high-viscosity carboxymethylcellulose and xanthan gum or any dry drilling fluid additives.

G.2 Sampling of Powdered Material in Packages

G.2.1 The number of samples taken to be combined for the test sample (see G.4) should be 15 samples of 0.5 kg (1 lb) or more per lot.

G.2.2 For each lot of 1000 packages or less, 15 packages should be sampled.

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

a) A sample weighing at least 0.5 kg (1 lb) should be taken from the top of each package.

b) A sampling tube capable of taking a core not less than 2.5 cm (1 in.) in diameter should be used. The tube should be inserted into the package being sampled so that it takes a core of material for essentially the entire length of the package.

G.3 Sampling of Powdered Material in Bulk

G.3.1 Tube sampling is used in the collection of samples from storage containers holding 25,000 kg to 100,000 kg (25 metric tons to 100 metric tons), with a sampling tube capable of taking a core not less than 2.5 cm (1 in.) in diameter should be used. The tube should be of sufficient length to permit taking a sample essentially from the top to the bottom of the mass being sampled.

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

G.3.3 For containers holding less than 20,000 kg (20 metric tons), at least one sample should be taken from each container up to lots of 10 containers, and the total number of samples taken from each lot of 100,000 kg (100 metric tons) or less should not be fewer than 10.

G.4 Preparation of Test Sample

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

G.4.2 Each portion should be sealed in a suitable airtight, moisture-proof container. One sample should be delivered to the purchaser, one to the supplier and the third sample should be retained for a reference test, if required.
G.5 Test

Each party should make one measurement on his test sample. A control or known reference sample should be tested in the same manner, at the same time as part of the test series. If this test result is within the acceptable range, the test results are considered valid.

G.6 Inspection

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

G.7 Rejection

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

G.8 Retesting

G.8.1 Either of the contracting parties may make claim for retest within one week of the date of the original test report.

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

Rig-site Sampling

H.1 General

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

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

In order to conduct rig-site sampling of a drilling fluid using a side-stream sampler, the following procedure shall be followed:

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 as to be taken when approximately 1/4, 1/2 and 3/4 of the load has been transferred.

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

c) Before each individual sample is taken, open the valve momentarily to flush out any material trapped in the side pipe. Tie a clean canvas bag to the sampler and fill it with one valve-opening operation.

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

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.
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

In order to conduct rig-site sampling of a drilling fluid using a sample scoop, the following procedure shall be followed:

Caution—Be sure all pressure is removed from the tank prior to opening the hatch.
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 Kit Cup, and Drilling Fluid Balances

I.1 General

This annex suggests methods for the calibration of field testing 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 is generally performed by the glassware supplier and may be part of the purchase specification. Glassware users should obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically. Periodic recalibration is not required. A procedure for calibration of graduated cylinders is given in I.3.

I.2.2 Thermometers

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

I.2.3 Timers

Timers shall be accurate to within ±5 seconds per hour and shall be verified over a period no less than 12 minutes 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, annex K.5) requires a timer accurate to ±2 seconds per hour.

I.2.4 Viscometers

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

I.2.5 Drilling Fluid Balances

Calibrate drilling fluid balances using de-ionized or distilled water, as specified in the test procedure given in Section 5.2.1. The recommended frequency is prior to each set of measurements, or as specified by the operator, drilling fluid company or other interested party.
I.2.6  Retort Kit Cups

Calibrate retort kit cups using de-ionized or distilled water, as specified in the test procedure given 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 Cylinders

In order to conduct a field calibration of a graduated cylinder, the following procedure should be used:

I.3.1  Allow the receiver and distilled or de-ionized water to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).

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

I.3.3  Fill the receiver with distilled or de-ionized 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.

I.3.4  Record the masses for each incremental volume of water at the specific graduation mark, \( m_w \), to the nearest 0.01 g.

I.3.5  Calculate the volume of the receiver at each mark, \( V_M \):

\[
V_M = \frac{m_w}{\rho_w}
\]

where

\( V_M \) is the receiver volume at a specific mark, expressed in milliliters;

\( m_w \) is the mass of water, filling receiver at a specified mark, expressed in grams;

\( \rho_w \) is the water density, expressed in grams per milliliter, at the test temperature (see Table I.1).

I.4  Procedure—Calibration of Thermometers

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

I.4.2  Read both thermometers and record the readings.

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

I.4.4  Calculate the average reading for each thermometer.

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

I.4.6  Calculate the thermometer correction as follows:
\[ C_{th} = R_1 - R_2 \]  \hspace{1cm} (I.2)

where

- \( C_{th} \) is the correction value to add to the thermometer reading;
- \( R_1 \) is the average reading for the standard thermometer, expressed in degrees;
- \( R_2 \) is the average reading for the working thermometer, expressed in degrees.

**EXAMPLE 1**
Thermometer correction determination, in degrees Celsius:

\[
R_1 = 23.9 \, ^\circ C \\
R_2 = 24.2 \, ^\circ C \\
C_{th} = 23.9 - 24.2 = -0.3 \, ^\circ C
\]

**EXAMPLE 2**
Thermometer correction application:

\[
C_{th} = -0.3 \, ^\circ C
\]

Temperature reading: \( R = 25.0 \, ^\circ C \)

Corrected temperature reading: \( R_{\text{corr}} = 25.0 + (-0.3) = 24.7 \, ^\circ C \)

**NOTE**  See ISO 386 for a more complete procedure description

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

It is not good practice to use thermometers whose correction exceeds twice the allowable tolerance of the measurement.
### Table I.1—Density of Water as a Function of Temperature

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<th>Temperature °C</th>
<th>Density g/ml</th>
<th>Temperature °F</th>
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</tr>
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<td>0.9956</td>
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</tr>
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<td>30.5</td>
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<td></td>
</tr>
<tr>
<td>31</td>
<td>0.9953</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### I.5 Procedure—Calibration of Viscometers

I.5.1 Obtain certified calibration fluids with chart (viscosity versus temperature) to cover the range of interest, such as 50 mPa•s, 100 mPa•s, (50 cP, 100 cP), etc. Make certain that the lot number on the chart matches the lot number on the fluid container. Each lot of standard fluid is individually certified. The viscosity will normally vary slightly from lot to lot.
I.5.2 Clean and dry the viscometer bob, rotor and cup. Place the viscometer and fluid side by side on a countertop in a room with a reasonably constant temperature [\(<3 \, ^\circ\text{C} \,<5 \, ^\circ\text{F}\) variation per hour]. Allow to stand at least 2 hr to equilibrate.

NOTE Water or any other fluid, including another calibration fluid, will contaminate the standard fluid.

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

Correct or replace the rotor if it wobbles excessively.

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

I.5.5 Place a thermometer [accurate to \(\pm 1 \, ^\circ\text{C} \,(\pm 2 \, ^\circ\text{F})]\) 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 (2 °F) for a 30 s period. Record the temperature reading.

I.5.6 Operate the viscometer and take readings at 600 r/min and 300 r/min. Estimate readings to the nearest 0.5 dial unit. If needed, use a magnifying glass.

I.5.7 Using the temperature/viscosity chart supplied with the calibration fluid, determine certified viscosity to the nearest 0.5 mPa\(\cdot\)s (0.5 cP). Compare the 300 r/min dial reading to the standard viscosity and record the deviation (plus or minus). Divide the 600 r/min dial reading by 1.98, compare it to the standard viscosity, and record the deviation.

I.5.8 Deviations exceeding 1.5 units are not acceptable. If the deviation exceeds these tolerances, recalibrate the viscometer.

I.5.9 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 Kit Cups

I.6.1 Allow the retort cup, lid and distilled or de-ionized water to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).

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

I.6.3 Fill the retort cup with distilled or de-ionized 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.

I.6.4 Place the filled retort cup with lid on the previously tared balance. Record the water mass to the nearest 0.01 g.

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

\[
V_{RC} = \frac{m_w}{\rho_w}
\]  

(I.3)

where

- \(V_{RC}\) is the volume of the retort cup, expressed in milliliters;
- \(m_w\) is the mass of water, expressed in grams;
- \(\rho_w\) is the water density, expressed in grams per milliliters, at the test temperature (see Table I.1).

I.7 Procedure—Calibration of Filter Press Gasket and O-rings

I.7.1 Gaskets or O-rings for the low-pressure, low-temperature filter press shall have an inside diameter of between 75.8 mm (2.99 in.) and 76.9 mm (3.03 in.).
1.7.2 Check the gasket or O-ring using a gauge with a diameter of between 75.8 mm (2.99 in.) and 76.9 mm (3.03 in.).

1.7.3 Do not use gaskets or O-rings with diameters outside this range for filter loss testing.
Annex J
(normative)

High-temperature/High-pressure Filtration Testing of Drilling Fluids Using the Permeability-plugging Apparatus (PPA)

J.1 Principle

J.1.1 The PPA can be operated at pressure differentials and temperatures approximating those prevailing down-hole and it permits the use of filtration media chosen to simulate exposed sands. 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.

The PPA utilizes ceramic disks as filtration media. Ceramic disks 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.

J.1.2 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 oil, water or emulsion concentration.

J.1.3 These characteristics are affected by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The permeability plugging apparatus (PPA) is a modified high-temperature, high-pressure filter press used to evaluate these interactions through ceramic-disk filter media at pressures up to 34,500 kPa (5000 psi) and temperatures from ambient to as high as 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.

J.2 Safety Considerations

J.2.1 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.

J.2.2 Cell damage can be categorized as follows: end-cap bending, end-cap compression, cylinder shear, cylinder stress, and the use of non-hardened set screws. 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 and/or manufacturer’s recommended hardened set screws showing signs of damage shall not be used. Cell bodies that show signs of stress cracking or serious pitting, or have damaged set-screw holes, shall not be used.

J.2.3 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 using defective parts, creates the possibility of cell leakage or failure, which could result in serious injury or apparatus damage.

J.2.4 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.

J.2.5 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 grounded circuits.

J.2.6 For safe operation ensure that the hydraulic pressure has been released before:
a) attempting to disconnect pressure hose from cell at quick coupler
b) attempting to remove cell from heating jacket
c) moving the PPA
d) refilling the hydraulic pump
e) performing any maintenance, including tightening leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly

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

J.2.7 For safe pneumatic pressurization of backpressure receiver, consider the following.

J.2.7.1 Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, use only nitrogen. Never use compressed air, oxygen or other non-recommended gas. If nitrogen is used, it shall be supplied in an approved nitrogen gas cylinder or the nitrogen supply system. CO₂ is normally supplied in small cartridges pressurized to about 6200 kPa (900 psi).

Warning—Do not allow CO₂ 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 shall be used only for Garrett gas-train carbonate analysis.

J.2.7.2 Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.

J.2.7.3 When pressurizing the backpressure assembly, ensure that the regulator T-screw has been rotated counter-clockwise and turns freely. This ensures that no pressure is going into the regulator. Always open the supply pressure 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, loosen the T-screw on the regulator and then bleed the pressure from the system.

Caution—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.

J.2.8 For safe heating, consider the following.

J.2.8.1 Caution should be exercised to avoid injury while operating the PPA, which becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended.

J.2.8.2 The practice of removing the cell and cooling it with water is hazardous and shall 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.

J.2.9 For safe electrical operation, consider the following.

J.2.9.1 Make sure that the electrical source is fused and grounded (earthed). Verify that the power cord on the heating jacket is in good condition and that it is properly grounded.

J.2.9.2 Electrical problems in the wiring or heaters cannot always be detected by visual inspection. The first sign of trouble is often blown fuses, tripped breakers, lengthened heating time or erratic thermostat performance. Never begin any electrical repairs without first disconnecting the unit from the power source.

J.2.10 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 hazard. The safety precautions listed below shall be followed to ensure safe operation.
J.2.10.1 Cell material shall be compatible with the test samples.

J.2.10.2 Do not use cells that show signs of severe pitting or stress cracking.

J.2.10.3 Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect threads carefully for signs of damage.

J.2.10.4 For cells that use set screws, use only hardened steel set screws as supplied by the manufacturer. Deformed set screws are unsafe.

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

J.3 Apparatus—Permeability-plugging Apparatus (PPA)

J.3.1 PPA Cell.

J.3.1.1 There are different ratings for cells. Refer to manufacturer’s specifications for temperature and pressure limits.

Warning—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). Unless the user can absolutely verify that the cell used is rated at 13,800 kPa (2000 psi), the “1800 psi” (12,400 kPa) pressure limit should be observed.

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

J.3.1.2 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.

J.3.1.3 The PPA utilizes ceramic disks as filtration media. Ceramic disks are available in various mean pore throat sizes (see Table J.1).

J.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 CO₂ pressurizing source to provide the backpressure. A nitrogen pressure source and a nitrogen manifold may be substituted for the CO₂ when desired.

J.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 metal stem thermometer inserted into the well in the cell wall. The temperature is adjusted by means of a knob on the thermostat. The dial has a reference scale of 1 to 10. After the desired temperature is obtained once, it can be repeated by setting the thermostat knob to the same reference setting. The standard cells for the PPA filter press are made of stainless steel.

J.3.2 Filtration Media.

J.3.2.1 Standard ceramic disk thickness is 6.5 mm (0.25 in.). Thicker disks can be used with proper manufacturer’s cells. A new disk is required for each test. For water-based drilling fluid samples, the disk shall be soaked in base fluid (water, brine, etc.) for at least 30 min; for non-aqueous drilling fluid the disk shall be soaked in base oil. When possible, vacuum saturate for at least 5 min to 30 min prior to use is preferred.

These disks are sold with a mean pore throat diameter as determined by mercury injection porosimetry. Table J.1 lists the commonly available ceramic disks and the mean pore throat diameter.
Table J.1—API Designation and Mean Pore Throat Diameter

<table>
<thead>
<tr>
<th>API Designation</th>
<th>Hg Injection Mean Pore Throat Diameter μm</th>
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</thead>
<tbody>
<tr>
<td>API 10</td>
<td>10</td>
</tr>
<tr>
<td>API 12</td>
<td>12</td>
</tr>
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<td>API 20</td>
<td>20</td>
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<td>API 40</td>
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<td>50</td>
</tr>
<tr>
<td>API 55</td>
<td>55</td>
</tr>
<tr>
<td>API 120</td>
<td>120</td>
</tr>
</tbody>
</table>

NOTE: Mercury injection porosimetry is not a viable technique for measuring pore throat sizes exceeding 120 μm. Therefore, there is no API designation for previously offered 150 μm and 190 μm ceramic disks.

J.3.2.2 Ceramic disks were adopted for use in the Particle Plugging Apparatus (PPA) as a method to test plugging/bridging materials in drilling fluids. Ceramic disks do not exhibit the normal relationship between pore throat size and permeability as naturally occurring sandstone. Work conducted to assess bridging characteristics should be based on pore throat size.

J.3.3  Timer.

J.3.4  Thermometer, with scale up to 260 °C (500 °F).

J.3.5  Graduated Cylinder, 25 ml (TC) or 50 ml (TC).

J.3.6  High-speed Mixer.

J.4  Procedure for the Permeability Plugging Apparatus

J.4.1  Assembling the Filtration Cell

If the cell is completely disassembled and the floating piston has been removed follow J.4.1.a through J.4.1.j. If the piston is in the cell with hydraulic fluid and with the inlet nipple and quick connect attached, then proceed to J.4.1.g

Caution—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, and 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.

e) Install the hydraulic inlet cap onto the cell.
Some oil should flow from the hole in the end cap, indicating that no air is trapped between the piston and the end cap.

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

NOTE Steps J.4.1.g to J.4.1.j should be performed with the cell placed in an unheated jacket.

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

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

J.4.2 Preheating the Heating Jacket

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

b) Turn the thermostat to the mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light illuminates when the heating-jacket temperature reaches the thermostat setting.

c) Readjust the thermostat to 6 °C (10 °F) over the desired test temperature.

J.4.3 Loading Test Fluid into the Cell.

NOTE For improved consistency in test results, test fluid must be stirred for 5 min immediately before loading the cell.

Caution—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) With the cell on a suitable stand, fill the cell to the bottom of the outlet O-ring 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 disk will maintain pre-saturation.

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

c) Position the outlet end cap in the cell, tighten the cell cap and then install and close the ball valve on the outlet end cap.

d) 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.

e) Connect the cell inlet to the quick connect.

f) Transfer the thermometer from the heating jacket and place into the cell body. Monitor the cell temperature with the thermometer in the cell wall, not in the heating jacket. When the cell reaches the desired temperature, lower the thermostat to reduce the jacket temperature to the test temperature.

J.4.4 Pressurization

a) Apply pressure to the cell using the hydraulic pump immediately after transferring the thermometer to the cell body. This pressure should equal the pressure that will be applied to the Back Pressure Receiver. (Refer to Table J.2 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.

b) Hold the cell at the desired temperature until thermal expansion is complete and the cell pressure stops increasing. The fluid should not be heated more than one hour prior to filtrate collection. If the test fluid cell temperature has not reached the test temperature after one hour 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 has been rotated counter-clockwise. The regulator T-screw will turn freely when no pressure is introduced into the regulator.

e) Verify that the pressure-relief valve on the pressure assembly and the filtrate drain valve on the receiver are closed.

f) Mount the backpressure receiver on the outlet valve assembly. Secure the receiver with a retaining pin. Install the pressurizing unit on top of the receiver. Lock the pressurizing unit in place with the other retaining pin. Do not adjust the regulator at this time.

NOTE A single CO₂ cartridge may not reach the maximum pressures in the table below. 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 If a Nitrogen manifold capable of 4900 kPa (700 psi) is available it may be used.

<table>
<thead>
<tr>
<th>Temperature range</th>
<th>Heating pressure or backpressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C</td>
<td>°F</td>
</tr>
<tr>
<td>Less than 95</td>
<td>200</td>
</tr>
<tr>
<td>95 to 150</td>
<td>200 to 300</td>
</tr>
<tr>
<td>151 to 175</td>
<td>301 to 350</td>
</tr>
<tr>
<td>178 to 190</td>
<td>351 to 375</td>
</tr>
<tr>
<td>191 to 205</td>
<td>376 to 400</td>
</tr>
<tr>
<td>206 to 218</td>
<td>401 to 425</td>
</tr>
<tr>
<td>219 to 232</td>
<td>426 to 450</td>
</tr>
<tr>
<td>233 to 246</td>
<td>451 to 475</td>
</tr>
<tr>
<td>247 to 260</td>
<td>476 to 500</td>
</tr>
</tbody>
</table>

g) If a drain hose is used for the filtrate, connect it from the drain valve to the graduated cylinder receiving the filtrate.

h) To ensure accurate measurements, the space between the filtration medium and the outlet ball valve should be filled with the base fluid before starting the test. This ensures that the fluid passing through the filter displaces an equal volume of fluid to the receiver. Failure to follow this corrective procedure can introduce significant error.

i) See Table J.2 to determine the appropriate pressure for the backpressure receiver and apply it by turning the T-screw on the pressure regulator clockwise until the desired pressure is reached.

j) After the test fluid has heated for one hour, actuate the pump to raise the cell pressure to the desired level and then open the valve between the cell and the backpressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the inlet pressure applied to the cell and that maintained on the backpressure receiver.
J.4.5 Conducting the Filtration Test

a) A laboratory timer shall be used and filtrate shall be collected after approximately 15 s to ensure the fluid has good filtration properties (excessive filtrate). Collect filtrate and record the volume at 1 min, 7.5 min, and 30 min. Plot the data against the square root of time to provide a useful description of the spurt loss. If desired, samples can be collected more frequently. Precisely recorded test times and volumes are necessary for accurate calculation of the filtration parameters.

b) Begin the filtration by opening the valve between the cell and the backpressure receiver. Verify that both the cell pressure, as indicated on the pump gauge, and the backpressure are at desired levels. Adjust them as necessary throughout the test.

c) Cell pressure can decrease slightly as filtration and any leakage at the pump reduce the cell contents. The pump should be used as necessary to maintain the pressure at the desired level.

d) After each interval, the filtrate shall be drained into the graduated cylinder from the backpressure receiver and the time and cumulative volume shall be recorded.

It is recommended to recover the filtrate directly from the backpressure receiver and not from a drain hose attached to it. However, if a hose is used, its’ length should 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.

J.5 Test Conclusion and Disassembly

a) Disconnect the heating jacket from the power source.

The pressurized cell assembly shall be allowed to cool in the heating jacket.

Warning—Extreme care should be exercised when cooling hot cells.

b) Turn the T-screw on the back pressure receiver regulator counter-clockwise until it turns freely.

c) Bleed the pressure from the backpressure receiver by opening the pressure relief valve.

d) Open the drain valve on the backpressure receiver and collect the last few drops of filtrate in the graduated cylinder. After removing the locking pin, remove the backpressure assembly from the cell.

e) Because the back pressure receiver is used with the flat end downward, some filtrate may be trapped inside the receiver body. Place the receiver with the flat end downward and the inlet hole over the graduate cylinder. Gently rock the receiver side to side to remove any remaining liquid.

f) The total volume of the filtrate in the graduated cylinder shall be recorded.

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 on the cell by opening the hydraulic pump valve. Disconnect the 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) The cell should be opened only when the user is confident that the contents are no longer under pressure. If pressure is suspected to remain in the cell consult manufacturers’ procedure for pressure release process. Trapped pressure is usually indicated by difficulty in removing end cap.

i) The end cap should be removed with the cell in the vertical position and the filtration end facing upwards.

j) To remove the filter disk pry the edge of the disk up, then remove the disk and the filter cake. If required, wash the filter cake lightly with base fluid (water, brine, oil, etc.), then measure and record its thickness and remark on its composition and texture.
Pour the remaining fluid from the cell. Wash the inside of the cell with base fluid. It is usually not necessary to remove the floating piston and the bottom end cap unless the last test was run at 150 °C (300 °F) or higher.

If testing was conducted at temperatures above 150 °C (300 °F), the O-rings should be replaced which will require removal of the inlet cell cap and floating piston.

Perform the following three steps to replace the O-rings on the floating piston and the bottom 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 piston and the cap.
3. Clean the parts for reuse.

J.6 Test Reports

J.6.1 Filtrate Reporting

Report the actual cumulative filtrate volume, in milliliters, collected through each of the selected time periods.

J.6.2 Spurt Loss

The spurt loss can be depicted by the intercept, on the y-axis, of the straight line representing the static filtration rate, when the square root of filtration time is plotted along the x-axis, and the filtrate volume [doubled to correct for filtration area when using 22.6 cm² (3.5 in.²) filtration media] is plotted along the y-axis. Alternatively, an approximate value can be calculated using Equation (J.2).

To define the spurt loss more accurately, collect and record the filtrate more frequently and plot the data in accordance with J.4.5.a.

J.6.3 Calculation

Set the permeability-plugging test volume, \( V_{\text{PPT}} \), expressed in milliliters, equal to two times the \( V_{30} \), the filtrate volume after 30 min, expressed in milliliters, as given in Equation (J.1):

\[
V_{\text{PPT}} = 2 \times V_{30} \tag{J.1}
\]

Calculate the spurt loss, \( V_1 \), expressed in milliliters, as given in Equation (J.2):

\[
V_1 = 2 \left[ V_{7.5} - (V_{30} - V_{7.5}) \right] = 2 \left( 2V_{7.5} - V_{30} \right) \tag{J.2}
\]

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.

Calculate the static filtration rate (velocity of flow), \( v_{sf} \), expressed in milliliters per square root of minute, as given in Equation (J.3):

\[
v_{sf} = \frac{2(V_{30} - V_{7.5})}{\sqrt{r_2} - \sqrt{r_1}} = \frac{2(V_{30} - V_{7.5})}{2.739} \tag{J.3}
\]

where
\( t_1 \) is the time at initial reading (i.e. 7.5 min), expressed in minutes;

\( t_2 \) is the time at final reading (i.e. 30 min), expressed in minutes.

Observe that these parameters, \( V_{PPT}, V_1 \) and \( v_{sf} \), are calculated on the basis of filtrate volume corrected for filtration area. The filter media routinely used in these tests have half the filtration area of that used in the standard low-pressure filtration test. Doubling the filtrate volume compensates for this area difference.

**J.6.4 Test Report for Filter Cake**

Measure and record the filter cake thickness to the nearest millimeter (1/32 in.). Include a description, such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are necessarily subjective judgments, they can convey important information.
Annex K
(informative)

Identification and Monitoring of Weight-material Sag

K.1 Principle

K.1.1 The objective of this annex is to provide an understanding of, and guidance on, weight-material sag, its impact on drilling operations, and methods to monitor sag at the well site.

K.1.2 The target audience for this annex covers both office and well-site engineers.

K.1.3 Weight-material sag is recognized by a significant [greater than 0.06 g/ml (0.5 lb/gal)] drilling fluid density variation, lighter followed by heavier than the nominal fluid density, measured when circulating, usually where a weighted fluid has remained uncirculated for a period of time in a directional well. It is recognized that sag is both a static and a dynamic phenomenon and has the potential to occur when the drilling fluid is in motion.

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

K.1.4 The definition of weight material extends to any particulate material which is added intentionally to the drilling fluid to increase the fluid density. Common example materials include barium sulfate, hematite and calcium carbonate.

K.1.5 Weight-material sag is applicable to the complex wells that are now commonly drilled. These include: high-temperature, high-pressure (HTHP), extended-reach drilling (ERD), and high-angle wells. Weight-material sag is important in the following circumstances:

- controlling downhole pressures to ensure that the well is stable with respect to influxes and losses;
- induced wellbore instability due to reduction in effective fluid density;
- calculating downhole hydrostatic pressure;
- stuck drill pipe;
- difficulty running casing and logs;
- estimation of hole-cleaning efficiency.

K.2 Guidelines on Using Sag Monitoring Methods

K.2.1 Experience shows that weight-material sag cannot be totally eliminated. However, it can be managed within levels that do not adversely affect the drilling operation. Weight-material sag is not entirely a drilling fluid-related problem. The occurrence of sag will also be governed by the wellbore geometry, wellbore environment and the operating conditions. Certain conditions in the drilling operation are conducive to creating dynamic sag. The potential for dynamic sag is promoted by an eccentric pipe, such as when sliding in deviated wells, and by low annular velocities, e.g. less than 100 ft/min (33 m/min).

K.2.2 This annex includes example calculations to illustrate how weight-material sag may impact downhole pressures and how such changes can be monitored at the well site. In recognition of the complexity of the problem, it is not possible to offer a single preferred method to monitor weight-material sag. The recommendation is to use a range of techniques in combination to provide a qualitative assessment of sag potential and to assess the benefits of potential remedial actions, e.g. addition of chemicals to the drilling fluid system. This annex provides five different methods to monitor weight-material sag. Four of these methods are based on direct measurement and one method is based on hydraulics calculations. All five methods are suitable for application at the well site.
K.2.3 The methods are as follows:

a) surface monitoring of weight-material sag (K.3);
b) field sag monitoring based on downhole density (K.4);
c) dynamic weight-material sag test—VSST method (K.5);
d) rheological measurements of drilling fluids exhibiting weight-material sag (K.6);
e) field sag monitoring based on critical wall shear stress (K.7).

K.2.4 The choice of monitoring method or methods is an engineering judgment that is linked to the complexity of the well and the probability of weight-material sag occurring in the field. Surface monitoring of returning mud-weight variation is logged as the de facto determination of whether sag is present. Downhole density determination will not always be an option because it relies upon specific downhole tools being run in the drill string. The methods proposed may be used individually or in combination. The modeling method based on critical wall shear stress is relatively simple to employ and only requires simple spreadsheet calculations.

K.2.5 Each method is described in more detail in K.3 to K.7.

K.2.6 The root causes of weight-material sag are only now being understood. As such, there is still considerable research effort devoted towards developing new methods to monitor and quantify sag. Some of the more recent techniques are referenced in the Bibliography. At a future date, these may become incorporated into an updated annex on weight-material sag monitoring.

K.3 Surface Monitoring of Weight-material Sag

K.3.1 Principle

K.3.1.1 Changes in returning drilling fluid density measured at the surface are often the first indications that weight material sag is occurring within the wellbore. A decline in returning mud weight followed by an increase in returning mud weight above the expected value is the classic signature that sag may be occurring.

K.3.1.2 All drilling well sites are capable of accurately measuring and monitoring surface drilling fluid density on a regular basis.

K.3.1.3 Variations in returning drilling fluid density may be identified during regular surface density determinations which form part of routine operations and are recorded on an ongoing basis.

K.3.1.4 The accepted practice for quantifying weight-material sag using surface density measurement is to capture and measure returning fluid samples when tripping-in the hole while stage circulating, or when circulating bottoms-up with the bit at total depth. Such as when circulating prior to tripping-out of the hole.

K.3.2 Apparatus

K.3.2.1 Any density-measuring instrument having an accuracy of ±0.01 g/ml or ±10 kg/m³ (±0.1 lb/gal or 0.5 lb/ft³).

A mud balance is the instrument generally used for drilling fluid density measurements. It is designed such that the drilling fluid holding cup, at one end of the beam, is balanced by a fixed counterweight, at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. Attachments for extending the range of the balance may be used when necessary.
The instrument should be calibrated frequently with fresh water, e.g. bi-weekly or weekly. Fresh water should give a reading of \((1.00 \text{ g/ml or } 1000 \text{ kg/m}^3)\) \((8.34 \text{ lb/gal or } 62.4 \text{ lb/ft}^3)\) at \(21 \text{ °C (70 °F)}\). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm, as required. A calibration of the upper density should be performed as specified by the manufacturer and done on a less frequent basis, e.g. annually.

The preferred method is to use a pressurized mud balance. This is particularly important in situations where the drilling fluid and surface flow system inherently create situations with high air entrainment or where the fluid is gas-cut. See Section 6 of this standard for procedures using a pressurized mud balance.

**K.3.2.2 Thermometer**, with a range of \(0 \text{ °C to } 105 \text{ °C (32 °F to 220 °F)}\) and an accuracy of \(±1 \text{ °C (±2 °F)}\).

**K.3.3 Procedure**

a) The procedure uses surface density measurement to infer the density of samples taken from different locations in the annulus corresponding to a series of pre-determined depths or time intervals.

b) Before tripping-in or tripping-out of the hole, identify a series of well depths or time intervals to take fluid measurements. The exact intervals and frequency of sampling should be determined on a well specific basis and should take account of the well geometry, well conditions and other operating considerations.

c) Measure the density of drilling fluid samples from pre-determined depths or time intervals with a mud balance, recording the density to the nearest \(0.01 \text{ g/ml (0.1 lb/gal)}\). Use a pressurized mud balance if appropriate (see K.3.2.1). Samples should be collected immediately after the primary solids control equipment (shakers) based on lagged transit time. The calculation of the lagged depth should take account of non-uniformity in wellbore geometry, e.g. known hole enlargement, and volumetric pump efficiency. These are outlined in K.3.4.

d) If practical, make all surface density determinations at a uniform temperature, e.g. \(21 \text{ °C (70 °F)}\). Regardless, the temperature shall be measured and recorded together with the recorded density. These data can then be used to graphically relate the measured density to a preferred reference temperature. Temperature should be recorded with an accuracy of \(±1 \text{ °C (±2 °F)}\).

**K.3.4 Calculation**

**K.3.4.1** Calculate the lagged time, \(t\), of samples based on annular volume capacity and discharge pump capacity (mechanical efficiency). This calculation should take account of any known changes in annular volume capacity due to geometry changes, including wellbore enlargement.

\[
t = \frac{4200 \times V_{USC}}{Q \times E}
\]

(K.1)

where

- \(E\) is the pump efficiency, expressed as a percentage;
- \(Q\) is the pump rate, expressed in gallons per minute;
- \(t\) is the lagged time of sample, expressed in minutes;
- \(V_{USC}\) is the annular volume, expressed in barrels.

**K.3.4.2** If appropriate, use a linear plot of density versus temperature to convert all measured data to a single reference temperature. An example of density variation with temperature for an oil-based drilling fluid is given in Figure K.1.
Figure K.1—Example of Surface Density Variation with Temperature (Oil-based Drilling Fluid)

K.3.4.3 Calculate the difference in the maximum fluid density and nominal fluid density by Equation (K.2) which can then be used to calculate the Sag Register, $S$, by Equation (K.3).

$$
\Delta \rho = \rho_{\text{max}} - \rho_{\text{nom}}
$$  \hspace{1cm} \text{(K.2)}

$$
S = \exp \left( -10 \times \frac{\Delta \rho}{\rho_{\text{nom}}} \right)
$$  \hspace{1cm} \text{(K.3)}

where

- $S$ is the Sag Register;
- $\rho_{\text{max}}$ is the maximum recorded drilling fluid density, expressed in pounds per gallon;
- $\rho_{\text{nom}}$ is the nominal drilling fluid density, expressed in pounds per gallon.

K.3.5 Data Reporting

K.3.5.1 Record operational conditions of the well on a trip-out sheet similar to the one shown in Table K.1. This should also include the raw data measurements of drilling fluid density versus pump strokes and time.
### DRILLING FLUID BOTTOMS-UP TRIP REPORT

<table>
<thead>
<tr>
<th>Operator</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Well name and number</td>
<td>Depth/TVD</td>
</tr>
<tr>
<td>Field/block number</td>
<td>Hole inclination</td>
</tr>
<tr>
<td>Location</td>
<td>Hole size</td>
</tr>
<tr>
<td>State/province/country</td>
<td>Time out of hole</td>
</tr>
<tr>
<td>Engineer</td>
<td>Flow rate</td>
</tr>
<tr>
<td>Comments, activities</td>
<td>Nominal fluid density at ______ °F</td>
</tr>
<tr>
<td></td>
<td>Max. fluid density at ______ °F</td>
</tr>
<tr>
<td></td>
<td>Min. fluid density at ______ °F</td>
</tr>
<tr>
<td></td>
<td>Drilling flow rate prior to tripping</td>
</tr>
<tr>
<td></td>
<td>B vsst sag</td>
</tr>
<tr>
<td></td>
<td>Sag register (S)</td>
</tr>
</tbody>
</table>

### BOTTOMS-UP TRIP DATA

<table>
<thead>
<tr>
<th>Time</th>
<th>Pump Strokes</th>
<th>Lagged Depth (ft)</th>
<th>Fluid Density (lb/gal)</th>
<th>Flowline Temp (°F)</th>
<th>Funnel Viscosity (s)</th>
<th>Gas Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rig Balance</td>
<td>Pressurized R600 PV</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>R300 YP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R200 LSYP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R100 Gel 10s</td>
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</tr>
<tr>
<td>R60 Gel 10min</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>R30 Gel 30min</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>R3</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### DRILLING FLUID RHEOLOGY

(120 °F)

| R600 | PV |
| R300 | YP |
| R200 | LSYP |
| R100 | Gel 10s |
| R60 | Gel 10min |
| R30 | Gel 30min |
| R6 |
| R3 |

Comments
K.3.5.2 Plot the surface density profile as a function of logged depth. An example is given in Figure K.2.

![Graph showing surface density profile](image)

**Key**

- **X**: measured depth, ft
- **Y**: lagged drilling fluid density, lb/gal
- **1**: nominal drilling fluid density, 17.9 lb/gal

**Figure K.2—Example Surface Profile Based on Bottoms-up Data**

### K.4 Field Sag Monitoring Based on Downhole Density

#### K.4.1 Principle

**K.4.1.1** The occurrence of weight-material sag in a wellbore can be identified by a variety of downhole measurements which include density measurements and pressure measurements converted to equivalent circulating density (ECD). However, interpretation of changes in downhole density is not straightforward, since other things that cause changes in downhole pressure can affect the density without necessarily having an effect on the potential for weight-material sag.

**K.4.1.2** These factors can include drill pipe rotation, changes in pump rate, and fluid density changes resulting from changes in temperature and pressure. Correct interpretation of downhole events is important. These areas are discussed in this section.
K.4.2 Measurements of Downhole Pressure

K.4.2.1 Use of downhole pressure tools is common in today's drilling arenas. These annular pressure-while-drilling tools, usually placed near the bit, have sensors that read the pressure exerted by the drilling fluid column at that particular point downhole where the tool is placed (see References #12 and #13). At the surface, the measurements received from downhole are then converted to density using the standard Equation (K.4).

\[
\rho_{df,USC} = \frac{P_{USC}}{0.052 \times D_{TVD}} \quad (K.4)
\]

where

- \( \rho_{df,USC} \) is the drilling fluid density, expressed in pounds per gallon;
- \( P_{USC} \) is the measured pressure, expressed in pounds-force per square inch;
- \( D_{TVD} \) is the true vertical depth, expressed in feet.

K.4.2.2 The density calculated from downhole pressure measurements while the drilling fluid is being circulated is called Equivalent Circulating Density (ECD) while that measured with a static drilling fluid is called Equivalent Static Density (ESD). ECD will always be greater than ESD for a typical drilling case.

K.4.3 Weight-material Signatures Using Downhole Pressure Tools

K.4.3.1 Weight-material incidents can be seen and measured by downhole pressure tools while the drilling fluid is either static or dynamic. Evidence of weight-material sag while the fluid was static can be seen in Figure K.3, where the annular pressure-while-drilling sensors were being recorded while the drill string was being run in the hole.

Key

| X | equivalent fluid density, lb/gal |
| Y | depth TVD, ft |
| 1 | simulated data |
| 2 | pressure data for trip in hole |
**NOTE** See Reference #14.

**Figure K.3—Downhole Density Changes Measured While Running the Drill String in the Hole**

**K.4.3.2** With an ESD of 13.4 lb/gal, the downhole tool began to measure declining density starting around 6000 ft TVD. The density continued to decline until around 14,000 ft TVD. The measured density began to increase above the base density of 13.4 lb/gal, eventually reaching a maximum of approximately 19.0 lb/gal. This figure shows the signature of a weight-material sag occurrence below 6000 ft TVD:

1) declining density followed by increasing density;
2) a density swing between 19.0 lb/gal and 13.4 lb/gal, which gives a theoretical $\Delta \rho$ of 5.6 lb/gal (19.0 lb/gal – 13.4 lb/gal);
3) a magnitude of sag of 5.6 lb/gal, which is greater than 0.5 lb/gal by the definition of weight-material sag.

Therefore, the event qualified as a weight-material sag event.

**K.4.3.3** In another case, while running in the hole, the occurrence of weight-material sag was picked up with the drilling bit at several depths, as shown in Figure K.4. While running in the hole, repeated fluctuations in the ESD that could not be explained (characteristic of weight-material sag occurrence) are seen at 1500 ft, 4000 ft, and 15,000 ft. At 15,000 ft, the pumps were turned on. The low measured density of nearly 10 lb/gal quickly increased to 12.3 lb/gal, as the drill string rotation picked up weight-material particles lying on the low side of the hole, and incorporated them into the main flow stream. Later, when the bit reached 21,200 ft, the pumps were turned on again and the ECD quickly increased from 11.2 lb/gal to nearly 14.2 lb/gal. As with the previous example, a swing in drilling fluid density of 3 lb/gal qualified this as a weight-material sag event.
Figure K.4—Weight-material Sag Occurrence during Dynamic Conditions

K.4.3.4 Left untreated, the circulating system can exhibit lighter and heavier densities for many circulations until the system eventually reaches equilibrium. However, these fluctuations can lead to well control difficulties, so it is important that weight-material sag occurrences be quickly corrected in the field.

K.4.4 Downhole Pressure Changes that are not caused by Weight-material Sag

K.4.4.1 Field Interpretation

In the field, changes in downhole pressure can be caused by other factors which are unrelated to weight material sag. Changes in pump rate, drill pipe rotational speed and fluid density (especially with invert emulsion drilling fluids) may produce changes in downhole density. It is important to recognize that density changes due to these factors are not events related to weight-material sag.

K.4.4.2 Changes in Pump Rate

K.4.4.2.1 When pump rates are increased or decreased, the friction at the wellbore wall is increased or decreased accordingly. Downhole pressure tools will measure these changes in annular pressure which may be converted to ECD.

K.4.4.2.2 Changes in ECD caused by changes in the pump rate can be predicted using drilling fluid hydraulic programs, which can verify whether the measured downhole change in pressure is expected or not.

K.4.4.3 Changes in Downhole Density

K.4.4.3.1 Some drilling fluids, particularly invert emulsions and those formulated with oils, exhibit changes in density as a function of temperature and pressure.

K.4.4.3.2 This phenomenon is purely a function of the fluid’s compressibility and thermal expansion. These properties can be characterized using coefficients derived from laboratory PVT data.

NOTE API 13D contains procedures and coefficients needed to predict the density of drilling fluids as a function of temperature and pressure.

K.4.4.4 Changes in Drill-string Rotational Speed

K.4.4.4.1 Changes in drill-string rotational speed can also change downhole pressures.

K.4.4.4.2 Figure K.5 displays the changes in downhole pressure produced by fluctuations in drill-string rotational speed that were obtained from downhole pressure tools.
The coupling of tangential velocities produced by the rotating drill string with the axial velocities produced by the pump rate results in helical flow and higher downhole pressures. These are commonly seen with downhole pressure tools. These increases in downhole pressure have been measured in field experiments (see References #15, #16, and #17. Published data show these increases commonly range between 0.05 lb/gal and 0.3 lb/gal using drill string rotation speeds of 50 r/min and 200 r/min.

For a particular drilling project, changes in downhole ECD produced solely by drill-string rotation can be measured in the field. These measurements are often called “fingerprinting.” It is generally best to conduct these tests inside newly cemented casing before drilling out. This eliminates the presence of drilled cuttings in the annulus as a contributor to any density changes. A range of pump rates and drill-string rotational speeds can be selected; these values should incorporate expected minimum and maximum levels to be used while drilling. An example fingerprinting matrix that could be used is shown in Table K.2.
Results from a fingerprinting exercise in the field are shown in Figure K.6. These may then be used to interpolate predicted increases in downhole density with varying pump rate and drill-string rotation speed while drilling ahead in the open hole interval. It is important to circulate the system at a high annular velocity (225 ft/min to 250 ft/min if possible) before taking measurements to minimize gelled fluid in the annulus.

![Figure K.6—North Sea Fingerprinting for Three Flow Rates and Four Drill-string Rotation Speeds](image)

**Key**
- X: drill-string rotation speed, r/min
- Y: measured increase in ECD, lb/gal
- 1: fingerprint at 1000 gal/min
- 2: fingerprint at 900 gal/min
- 3: fingerprint at 1100 gal/min

**NOTE** See Reference #18.

**Figure K.6—North Sea Fingerprinting for Three Flow Rates and Four Drill-string Rotation Speeds**

Pressure changes caused by drill string rotation can be mathematically modeled. A complex mathematical model for helical flow has been constructed to predict the increase in downhole pressure produced by rotation speeds commonly used in the field (see Reference #17. In the referenced work, the tangential velocities produced by drill string rotation are coupled with the axial velocities to generate the fluid helical velocities. The velocities near the conduit walls are then used to determine the predicted shear rates at the wall, which in turn are used to calculate fluid pressure drop.

Because these calculations are complex and cannot be worked with simple equations, a different approach has been taken to derive a simple heuristic equation or set of equations that can provide a usable solution (see Reference #18). The derived equation is comprised of two principal variables: the wellbore geometry diameter ratio and the drill-string rotational speed (r/min). These may be used to...
calculate the anticipated pressure increase in a particular interval. In this work, the general equation is given as Equation (K.5).

\[
\Delta P_{\text{rot}} = \left( -1.0792 \frac{d}{D} \right) + \left[ \left( 17.982 \frac{d}{D} \right)^2 \times (0.00001 \times L) \times \eta \right]
\]  

(K.5)

where

- \( \Delta P_{\text{rot}} \) is the anticipated pressure increase, expressed in pounds-force per square inch;
- \( d \) is the diameter of inner pipe, expressed in inches;
- \( D \) is the diameter of outer pipe, expressed in inches;
- \( L \) is the length of the hydraulic section, expressed in feet;
- \( \eta \) is the drill-string rotation, expressed in revolutions per minute.

The increase in ECD resulting from drill pipe rotation, \( \Delta \rho_{\text{ECD-rot}} \), expressed in pounds per gallon, at true vertical depth, \( D_{\text{TVD}} \), is calculated as:

\[
\Delta \rho_{\text{ECD-rot}} = \frac{\Delta P_{\text{rot}}}{0.052 \times D_{\text{TVD}}}
\]  

(K.6)

where symbols are defined above (Equation K.5).

**K.4.5 Theoretical Downhole Density Excluding Effects in K.4.4**

**K.4.5.1** Theoretical downhole densities can be calculated for both static and dynamic cases. In the static case, the downhole density should be equivalent to the ESD. Any densities lighter or heavier than the ESD \( \pm 0.5 \text{ lb/gal} \) may be considered a potential weight-material sag event. This excludes parts of the annulus where a weighted pill has been pumped before pulling out of the hole. In this case, a higher density in part of the annulus is planned and deliberate.

**K.4.5.2** In dynamic cases, the downhole density while rotating the drill-string should include:

1) \( \rho_{\text{ECD-hyd}} \) which is comprised of the ESD, the circulating pressure drop and the extra density effects of drilled cuttings in the annulus predicted from hole cleaning modeling;
2) \( \Delta \rho_{\text{ECD-rot}} \) determined using Equation (K.6) or interpolated as discussed in K.4.4.4.3.

**K.4.5.3** The total predicted ECD of the drilling fluid in the annulus while circulating, \( \rho_{\text{ECD-tot}} \), expressed in pounds per gallon, should be:

\[
\rho_{\text{ECD-tot}} = \rho_{\text{ECD-hyd}} + \Delta \rho_{\text{ECD-rot}}
\]  

(K.7)

where

- \( \rho_{\text{ECD-hyd}} \) is the ESD, the circulating pressure drop and extra density effects of drilled cuttings in the annulus, expressed in pounds per gallon;
- \( \Delta \rho_{\text{ECD-rot}} \) is the change in the ECD due to drill-string rotation, expressed in pounds per gallon.
K.4.5.4 If $\rho_{ECD}$ in the wellbore, measured using downhole pressure tools, is greater or less than the $\rho_{ECD\text{-tot}} \pm 0.5$ lb/gal range, then there is a potential weight-material sag event and further investigation is needed.

**K.4.6 Weight-material Sag Events Caused by Drill Pipe Rotation only**

K.4.6.1 Some modeling of weight-material sag potential has been performed on the effects of rotating a drill string slowly in a static fluid. An example of the need for this modeling is the field case where, while waiting on weather, the driller rotates the drill-string at a slow rate (between 5 r/min and 25 r/min) in order to eliminate a potential case of differential sticking.

K.4.6.2 In the modeling work, it was shown that, at slow drill-string rotation in static fluids, there exists a potential for the occurrence of weight-material sag. Fluid viscosities are lowest close to the rotating drill string, as shown in Figure K.7.

![Figure K.7—Distribution of Fluid Viscosity Across the Annular Gap Caused by Drill-String Rotation in a Non-circulating Drilling Fluid](image)

**Key**

<table>
<thead>
<tr>
<th>Key</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>gap, m</td>
<td>3</td>
</tr>
<tr>
<td>Y</td>
<td>viscosity, cP (Pa•s)</td>
<td>4</td>
</tr>
<tr>
<td>1</td>
<td>drill-string rotation speed: 5 r/min</td>
<td>5</td>
</tr>
<tr>
<td>2</td>
<td>drill-string rotation speed: 10 r/min</td>
<td></td>
</tr>
</tbody>
</table>

**NOTE** See Reference #17 and #18.

K.4.6.3 Such reductions in fluid viscosity in a fluid already exhibiting low viscosity could hasten a weight material sag event. The drop in fluid viscosities near the rotating drill string wall has a greater consequence for eccentric cases than for concentric cases.

**K.5 Dynamic Weight-material Sag Test—VSST Method**

**K.5.1 Principle**

K.5.1.1 The Viscometer Sag Shoe Test (VSST) is a well-site and laboratory test that measures weight material sag tendencies of field and lab-prepared drilling fluids under dynamic conditions (see Reference # 11).
K.5.1.2 The VSST provides an intrinsic fluid property without regard to the conditions under which the fluid has been or will be used. As such, results should be combined with operational factors to correlate with sag experienced in the field. (This is analogous to measuring a yield point, but still having to combine it with operational factors in order to correlate with pressure loss.)

K.5.1.3 The VSST designation is derived from the rotational viscometer used as a mixer and the thermoplastic insert (Sag Shoe) designed to concentrate sagged weight material in the bottom of a viscometer thermocup. Figure K.8 shows the basic equipment needed to determine VSST.

![Diagram of VSST equipment](image)

**Key**

1. syringe
2. collection well
3. viscometer rotor
4. VSST Sag Shoe
5. metal-backing plate
6. thermocup

**Figure K.8—Key Equipment for VSST Method**

K.5.1.4 Sag tendency is determined by the density increase of samples extracted from the collection well over a 30 min-period at a standard temperature and under a consistent rate of shear.

K.5.1.5 Weight-material bed pickup can be run as an optional measurement to characterize bed removal by higher shear levels. Results can be used to suggest opportunities for bed removal in the field prior to tripping out of the hole.

K.5.2 Apparatus

K.5.2.1 Direct-indicating viscometer, powered by an electric motor; see K.5.3.1 for setup.

K.5.2.2 Stopwatch or timer, accurate to ±1 second over a period of 30 min (2 s per hour).
K.5.2.3 Thermostatically controlled viscometer cup.

NOTE The VSST Sag Shoe is intended to fit inside the thermocup without being too tight.

K.5.2.4 Thermometer, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of ±1 °C (±2 °F).

K.5.2.5 Luer syringe, 10 ml, two-piece, solvent-resistant.

K.5.2.6 Pipetting needle, blunt-end, 150 mm (6 in.) 14-gauge needle with Luer connection.

K.5.2.7 Balance, digital, with 0.01 g resolution. A triple-beam balance may be used in the field where excessive vibration is expected.

K.5.2.8 Pycnometer or retort cup, 10 ml.

NOTE The pycnometer equipment is not required if the test is run at the well site or if the procedure includes the pickup test described in K.5.3.3.

K.5.2.9 Spatula, 150mm (6 in.) or similar.

K.5.2.10 De-ionized or Distilled water.

K.5.2.11 Multimixer, capable of operating at 11,500 r/min ±300 r/min under load, with a single corrugated impeller of approximately 25.5 mm (1 in.) in diameter.

K.5.2.12 Container, for mixing, 180 mm (7.125 in.) deep, diameter 97 mm (3.75 in.) at top and 70 mm (2.75 in.) at bottom.

K.5.2.13 VSST Sag Shoe, shaped, cylindrical disk manufactured from thermoplastic material, maximum diameter of 57 mm ±0.38 mm (2.35in. ±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 dense fluids.

K.5.3 Procedure

In order to conduct a VSST, the following procedure shall be used:

K.5.3.1 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 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 6.5 mm (0.25 in.) below the first mark.

d) Calibrate the syringe with blunt-end pipetting needle attached, using distilled or de-ionized water and digital balance. Expel as much water as possible from the syringe barrel and wipe exterior dry.

NOTE Calibrate pycnometer or retort cup with de-ionized or distilled water if it will be used for density measurements.
K.5.3.2 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 fluid sample in a container, mix appropriately, and pour approximately 140 ml into the thermocup. Most consistent results are obtained if the starting fluid temperature is close to 49 °C (120 °F) to minimize sag during heating. Heat the drilling fluid to 49 °C (120 °F) while stirring at 600 r/min.

d) Position and lock the upper edge of the viscometer locking mechanism to coincide with the lower mark on the support leg. The top of the Sag Shoe should be 7 mm (0.275 in.) below the viscometer sleeve.

e) Set the viscometer at 100 r/min and start the 30 min timer.

f) Using the syringe with blunt-end pipetting needle attached and cleared of air, draw slightly over 10 ml from the drilling fluid remaining in the container. Carefully clear the syringe and pipetting needle of residual air and push the plunger to the 10 ml-calibration mark. Wipe the pipetting needle and syringe surfaces until clean and dry.

g) Weigh the fluid-filled syringe and record the mass as \( m_{F1} \), expressed in grams.

NOTE When using the pycnometer or retort cup, transfer the fluid from the syringe, weigh and record total mass as \( m_{F1} \), expressed in grams.

h) Stop viscometer rotation at the end of the 30 min test period.

i) Repeat K.5.3.2.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 pycnometer or retort cup, transfer the fluid from the syringe, weigh and record total mass as \( m_{F2} \), expressed in grams.

K.5.3.3 Bed Pickup Measurement (\( m_{F3} \)) (Optional)

K.5.3.3.1 Gently return the 10 ml test sample from the fluid-filled syringe obtained in K.5.3.2.i to the Sag Shoe collection well.

K.5.3.3.2 Run the viscometer at 600 r/min for 20 min.

K.5.3.3.3 Collect the sample the from Sag Shoe collection well as in K.5.3.2.i. Weigh the fluid-filled syringe and record total mass as \( m_{F3} \), expressed in grams.

K.5.4 Calculation

K.5.4.1 Calculate the \( B_{VSST} \) using Equation (K.8):

\[
B_{VSST} = 0.834 \times (m_{F2} - m_{F1})
\]  

(K.8)

where

\( B_{VSST} \) is the amount of weight-material sag, expressed in pounds-mass 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.

Report \(B_{VSST}\) in pounds-mass per gallon.

**K.5.4.2** Calculate optional bed-pickup ratio, \(R_{BPU}\), using Equation (K.9):

\[
R_{BPU} = \frac{83.4 \times (m_{F2} - m_{F3})}{B_{VSST}} \tag{K.9}
\]

where

- \(R_{BPU}\) is the calculated bed pickup measurement ratio, expressed as a percentage;
- \(B_{VSST}\) is the amount of weight-material sag, expressed in pounds-mass per gallon;
- \(m_{F2}\) is the mass of 10 mL drilling fluid (plus the syringe) taken from the Sag Shoe following 30 min shear at 100 r/min, expressed in grams;
- \(m_{F3}\) 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.

Report \(R_{BPU}\) as a percentage (%).

**K.6** Rheological Measurements of Drilling Fluids Exhibiting Weight-material Sag

**K.6.1** Principle

**K.6.1.1** Advanced rheometers are able to measure a wider range of properties than conventional oilfield viscometers and to make these measurements more accurately. This section relates to the use of such instruments in the measurement and analysis of drilling fluids exhibiting weight-material sag.

**K.6.1.2** Drilling fluids that exhibit weight-material sag are, by definition, unstable with respect to time. This makes rheological measurements on them difficult. The magnitude of any measured values can be influenced by sample preparation methods and the shear history of the test fluid.

**K.6.1.3** Establishing guidelines for sample preparation and equipment selection will facilitate more meaningful analyses of drilling fluid samples during sag investigations.

**K.6.1.4** The method given in this section will only yield correct information when the annular velocity is 100 ft/min or greater.

**K.6.2** Scope

**K.6.2.1** Currently, there are no accepted industry methods relating to the equipment or methodology to be used in the measurement of rheological parameters related to weight-material sag in drilling fluids.

**K.6.2.2** There is a generally accepted view that viscosity measurements at low (less than 1.0 s\(^{-1}\)) shear rates and various rheological parameters derived from oscillatory measurements are useful in quantifying the actual or potential ability of a fluid to exhibit weight-material sag.
K.6.2.3  This section is applicable to both field drilling fluids and fluids mixed in the laboratory.

K.6.3  Apparatus

K.6.3.1  Rheometers: for the purpose of this procedure distinguished from viscometers by their greater degree of accuracy and range of measurement.

Typical capabilities found only in rheometers are low shear rates, oscillatory measurements and the capability to make measurements under elevated temperatures and pressures.

Rheometers suitable for detailed investigation of sagging drilling fluids should be capable of the following:

a) accurate measurement of viscosity at shear rates from approximately 1000 s\(^{-1}\) continuously to 0.01 s\(^{-1}\) or below;

b) oscillatory functionality to allow the calculation of the storage modulus (G') and loss modulus (G'');

c) accurate measurement of stresses below 0.02 lbf/100•ft\(^2\).

K.6.3.2  Water bath, maintained at 60 °C to 70 °C (140 °F to 160 °F).

K.6.3.3  Mixer, a high shear, variable speed, heavy-duty laboratory mixer with a high shear, square-hole impeller screen. A typical impeller blade diameter is 31.20 mm (1.228 in.), and a typical impeller screen inside diameter is 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 high shear screen is 2.4 mm (3/32 in.).

K.6.4  Sample Preparation

K.6.4.1  Field samples delivered to a laboratory have been subjected to a wide variety of shear histories. For meaningful laboratory measurements, samples need to be fully reconstituted. If measurements of different fluids are to be compared, it is important to ensure that the fluids are fully reconstituted and re-sheared as close to stable properties as possible. Measurements may then be made at differing times after this condition.

K.6.4.2  Sample mixing should involve the entire content of the storage container. By their nature, fluid samples collected because of sag problems may be expected to have suffered from solids settling during storage. All settled solids shall be re-incorporated into the fluid during mixing.

K.6.4.3  The sample should be mixed at a 6000 r/min shear rate using the high shear mixer described in K.6.3.3, for a period of 15 min per 350 ml volume. A volume of 1400 ml should be sheared for 1 hr. Cooling the sample to between 60 °C and 71 °C (140 °F and 160 °F) should include the use of a water bath. Once this temperature is reached, the sample should be covered to prevent vaporization of water. If the sample is too large to mix in a single batch, multiple batches can be mixed as above and then combined. Steps to minimize the time delay between mixing the first and last batches should be taken in such cases.

K.6.4.4  For each rheological measurement, the time between the fluid being sheared as described in K.6.4.3 and the measurement being made should be recorded.

K.6.4.5  Immediately prior to each rheological measurement, the fluid should be sheared in the rheometer at approximately 1000 s\(^{-1}\) for a minimum of 2 min.
K.6.5 Potential Rheological Tests

K.6.5.1 Through the use of rheometers, a great number and variety of tests may be performed. By exploring several of these tests, a better understanding of the sample fluid may be obtained. Tests should be selected and carried out with this goal in mind.

K.6.5.2 Potential tests of interest for the examination of drilling fluids include:

a) Thixotropy loops (hysteresis)—observing the structure-building tendency of the fluid and how easily that structure is broken by shear.

b) Yield stress measurements—through multiple methods, observing where the fluid actually yields.

c) Controlled rate/stress sweep—producing a flow curve that demonstrates the relation of stress and viscosity to strain rate.

d) Oscillatory strain/stress sweep—important for determining the linear viscoelastic region (for further oscillatory tests) and for determining the dynamic yield stress.

e) Oscillatory frequency sweep—giving information on structural behavior of the test fluid over a range of deformation rates, usually performed on a fluid which has been allowed a gel growth period immediately prior to testing.

f) Oscillatory time sweep—observing how the fluid structure grows and is maintained under low-frequency deformations over long periods of time, usually performed on a fluid without allowing gel growth before testing.

K.6.5.3 Unlike the common six-speed field viscometer, which exclusively uses a rotating sleeve about a torsion spring bob (Couette geometry), rheometers have a variety of test geometries from which to choose. These include the Couette geometry, double-gap Couette, multivane spindles, parallel plates, cone and plate, and any of these modified with roughened surfaces for mitigation of wall slip effects which can occur at low shear rates. The test geometry should be selected in accordance with the needs of the test to be performed and the fluid being tested.

K.6.5.4 During the preparation of K.6, a series of round-robin tests was performed by several laboratories. These laboratories used different instruments and followed the individual manufacturer's instructions for the instrument. The list of instruments used includes:

—Bohlin® Gemini;
—Brookfield® PVS;
—Grace® M3500a-1;
—OFI® Model 900;
—Physica® MCR101.

K.6.6 Data Interpretation

K.6.6.1 Various publications (see Reference #20 have suggested that weight-material sag is closely correlated with the viscosity of the fluid at low shear rates. The shear rates of interest are typically in the range of 0.1 s\(^{-1}\) to 1.0 s\(^{-1}\). Measurement of viscosity at these shear rates is not possible at the rig site using conventional field viscometers.

K.6.6.2 More sophisticated devices have become available since around 2000. Advanced rheometers of the type discussed in this subsection are also fully capable of making these measurements as part of a basic series of tests designed to provide a complete rheological analysis of a given fluid.
**K.6.6.3** Viscosity values, which should be adequate to prevent sag of invert emulsion drilling fluids under dynamic field conditions, have been proposed (see Reference #21), and a typical graph is shown in Figure K.9. In the graph, the solid parallel lines represent the upper and lower bounds of acceptable viscosity, i.e. viscous enough to prevent dynamic weight-material sag under typical drilling conditions, but not so viscous as to cause other drilling-related problems. The viscosity and shear rates 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 will result in relatively small deviations from these nominal values (Reference #2).

**K.6.6.4** Interpretation of data from rheological testing should be made in the context of the specific fluid being tested. It is easy to generalize from the rheological behavior of a particular fluid system and attempt application to other systems.

**K.6.6.5** If the basic characteristics of the system differ, i.e. different weight materials and oil-to-water ratios, different viscosifier types, significant changes in internal phase composition, and significantly different emulsifier chemistry, the conclusions of one system may not apply to the other. The rheological testing and evaluation of each fluid should be taken with knowledge of the physical characteristics of that fluid. Likewise, the rheological testing performed should be considered when drawing conclusions as to a fluid performance.

**K.6.6.6** It is often beneficial to observe trends in changes of rheological characteristics of a drilling fluid as small changes (treatments) are made to the system. Under such conditions, the effects of such treatments should be monitored. Specifically, note should be taken of how viscosity shifts with changes in component concentrations. Similarly, note should be made of the changes in $G'$, $G''$ and loss tangent affected by changes in components. One should specifically look for improved/optimal performance in properties, e.g. maximal structure without extreme viscosity or raising ECD issues, based on component changes.

**K.7 Field Sag Monitoring Based on Critical Wall Shear Stress**

**K.7.1 Principle**

Use of hydraulic modeling to predict the onset of weight-material sag under dynamic conditions has been presented (see Reference #24). In this prediction method, it is assumed that, given sufficient shear stress in the drilling fluid at the wall on the low side of the deviated wellbore, barite bed formation will not begin to occur. If the moving fluid does not have sufficient shear stress, then accumulation of barite particles will begin to occur (see Reference #23).

**K.7.2 Predictive Model**

**K.7.2.1** Obtain drilling fluid rheological properties from viscometer data, from HTHP viscometers, or from predicted downhole data.

**K.7.2.2** Calculate the fluid Herschel-Bulkley rheological parameters through a mathematical regression analysis as outlined in API 13D. The use of a computer program or spreadsheet is recommended to perform this complex data analysis. The yield stress value, $\tau_Y$, will be used in the calculations below.

**K.7.2.3** In the hydraulic calculations, use the geometry outer and inner diameters for the particular interval where weight-material sag occurrence is suspected. Set the inner pipe eccentricity to a high value ($c = 0.7$ is recommended).
Key
1 20,000 cP at 0.17 s⁻¹
2 2500 cP at 1.7 s⁻¹
3 12,000 cP at 0.17 s⁻¹
4 1500 cP at 1.7 s⁻¹
5 viscosity curve for a drilling fluid with minimal expected sag tendency

**Figure K.9**—Published “Sag Window” for Drilling Fluids Versus Shear Rate

**K.7.2.4** Calculate the minimum pressure drop in this geometry to shear the annulus across the narrow gap.

\[
\frac{\Delta P}{\Delta L_{USC}} = \frac{2\tau_Y}{L_{USC}}
\]  
(K.10)

where

- \(\frac{\Delta P}{\Delta L_{USC}}\) is the pressure gradient, expressed in pounds-force per square inch per foot;
- \(\tau_Y\) is the drilling fluid yield stress, expressed in pounds-force per hundred square feet;
- \(L_{USC}\) is the hydraulic length, expressed in feet.

**K.7.2.5** Using iterative techniques, find the circulation rate required to provide this pressure drop in the eccentric annulus. Add a 10% circulation rate to this value to ensure that the value obtained is above this minimum circulation rate.

**K.7.2.6** From the pressure-drop equations given in API 13D, obtain the predicted fluid velocity profiles at a slight distance from the wall using different circulation rates (ensure that flow is laminar); see API 13D.
By definition, the fluid velocity at the wall should be zero. Also calculate the average annular velocity, \( v_a \), for each case.

**K.7.2.7** From the velocity values near the wall, calculate the corresponding fluid shear rates, \( \gamma_i \), and wall shear stresses, \( \tau_{Wi} \):

\[
\gamma_i = \frac{\Delta v_{USC}}{5 \times d_1}
\]

(K.11)

where

- \( \gamma_i \) is the fluid shear rate, expressed in reciprocal seconds;
- \( \Delta v_{USC} \) is the change in annular velocity, expressed in feet per minute;
- \( d_1 \) is the distance from the outer wall, expressed in inches.

and,

\[
\tau_{Wi} = \tau_Y + \left( k_C \times \gamma_i \right)
\]

(K.12)

where

- \( \tau_{Wi} \) is the wall shear stress, expressed in pounds-force per one hundred square feet;
- \( \tau_Y \) is the drilling fluid yield stress, expressed in pounds-force per one hundred square feet;
- \( k_C \) is the consistency factor, expressed in pounds-force second per one hundred square feet;
- \( \gamma_i \) is the fluid shear rate, expressed in reciprocal seconds.

**K.7.2.8** Model increased circulation rates (four cases are usually sufficient) to find the slope \( b \) between annular velocity and fluid shear stress at the wall. In laminar flow, the slope relating annular velocity and fluid shear stress should be linear or near-linear.

**K.7.2.9** Calculate the critical wall shear stress for an annular velocity of 30 ft/min using general Equation (K13).

\[
\tau_W = \tau_Y + \left( b \times v_{USC} \right)
\]

(K.13)

where

- \( \tau_W \) is the wall shear stress, expressed in pounds-force per one hundred square feet;
- \( \tau_Y \) is the drilling fluid yield stress, expressed in pounds-force per one hundred square feet;
- \( b \) is the slope of the annular velocity and shear stress at the wall in laminar flow, as defined in K.7.2.8;
- \( v_{USC} \) is the annular velocity, expressed in feet per minute.

**NOTE** The selected annular velocity of 30 ft/min is based on published data; it corresponds to the approximate velocity at which the maximum level of sag occurs (see Reference #22)
K.7.2.10 With the value calculated in Equation (K.13), read the predicted maximum weight-material sag from Figure K.10.

![Graph showing predicted dynamic sag as a function of calculated values of wall shear stress, $\tau_W$.]

**Key**
- $X$: wall shear stress, lbf/100-ft$^2$
- $Y$: predicted dynamic sag, lb/gal

**Figure K.10—Predicted Dynamic Sag as a Function of Calculated Values of Wall Shear Stress, $\tau_W$**

K.7.3 Comparison of Laboratory and Field Data

K.7.3.1 The predicted maximum weight-material sag under dynamic conditions was developed from laboratory data where testing conditions were favorable for the initiation of weight-material sag.

K.7.3.2 In the field, these conditions are often not as favorable, and the hydraulic method described here usually over-predicts the magnitude of measured sag.

K.7.4 Additional Resource Literature

The analysis of weight-material sag remains an active research area. The Bibliography lists additional resource literature (see References # 24, # 25, # 26 and #27), which expands on the technology given in this annex. More articles will probably be available as new concepts are evaluated and reported.
Bibliography

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