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## Field Testing Non-Aqueous Based Drilling Fluids

### 1 Scope

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

- a) drilling fluid density (mud weight);
- b) viscosity and gel strength;
- c) filtration;
- d) non-aqueous fluid (NAF), water, and solids concentrations;
- e) alkalinity, chloride concentration, and calcium concentration;
- f) electrical stability;
- g) lime and calcium concentrations, calcium chloride, and sodium chloride concentrations;
- h) low-gravity solids and weighting material concentrations.

The annexes provide additional test methods or examples that can optionally be used for the determination of:

- shear strength (Annex A);
- NAF and water concentrations from cuttings (Annex B);
- drilling fluid activity (Annex C);
- aniline point (Annex D);
- lime, salinity, and solids concentration (Annex E);
- sampling, inspection and rejection (Annex F);
- rig-site sampling (Annex G);
- cuttings activity (Annex H);
- active sulfide (Annex I);
- calibration and verification of glassware, thermometers, viscometers, retort kit cups, and drilling fluid balances (Annex J);
- high-temperature/high-pressure filtration using the permeability-plugging apparatus (PPA) (Annex K);
- elastomer compatibility (Annex L);
- sand content of non-aqueous based drilling fluid (Annex M);

- identification and monitoring of weight-material sag (Annex N);
- non-aqueous based drilling fluid test report form (Annex O).

## 2 Normative References

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

API Recommended Practice 13B-1, *Field Testing Water-Based Drilling Fluids*

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

API Recommended Practice 13I, *Laboratory Testing of Drilling Fluids*

## 3 Terms and Definitions

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

### 3.1

#### **ACS reagent grade**

Grade of chemical meeting the purity standards specified by the American Chemical Society<sup>1</sup> (ACS) and listed in the Chemical Abstracting Service (CAS).

### 3.2

#### **Calibration**

Calibration refers to the verification of an instrument or to the adjustments made to the instrument if it is out of calibration.

NOTE In this document, the word "calibrate" refers to instruments that, if not calibrated, as shown by "verification" of instruments that can be adjusted to bring them back into calibration. The word "calibrate" also refers to instruments that cannot be adjusted if they are inaccurate and to determine their correction factors.

### 3.3

#### **Density of water**

Density of deionized or distilled water is 0.999 g/mL (8.334 lb/gal) at 20 °C (60 °F).

NOTE1: Deionized or distilled water is used for all equipment gravimetric calibrations for volume with the density being adjusted for temperature.

NOTE 2: For the purposes of this standard, mud balance calibration using fresh water and retort calculations use the density of water to be 1 g/mL (8.345 lb/gal<sup>2</sup>) for simplicity. Using this value, the volume of the water measured in cubic centimeters or milliliters is numerically equal to the mass in grams, i.e. 1 g = 1 mL.

<sup>1</sup> American Chemical Society, 1155 Sixteenth Street, NW Washington DC 20036, [www.acs.org](http://www.acs.org)

<sup>2</sup> Gallon as used throughout this standard refers to the U.S. gallon of 3.7854 liters.

### 3.4

#### **Non-aqueous drilling fluid (NADF)**

Fluids prepared from non-aqueous base fluids (NAF), such as synthetic oils, mineral oils, linear paraffins, olefins, esters, and diesel.

### 3.5

#### **Pound (lb)**

U.S. customary unit (USC) used to indicate pound-mass (weight), as opposed to pound-force [(lbf), shear stress].

### 3.6

#### **Relative density**

Dimensionless ratio of the mass of a volume of a substance to the mass of the same volume of a reference substance, i.e. the ratio of their respective mass densities.

NOTE 1 Generally, the reference substance is pure water.

NOTE 2 Relative density is commonly known as specific gravity.

### 3.7

#### **Spurt loss**

Volume of fluid that passes through the filtration medium before a filter cake is formed.

## 4 Symbols and Abbreviations

### 4.1 Symbols

For the purposes of this document, the following symbols apply:

$a_C$	measure of the chemical potential (water activity) or reaction availability of drilled cuttings
$a_W$	measure of the chemical potential (water activity) or reaction availability of salt water solutions
$a_{df}$	measure of the chemical potential (water activity) or reaction availability of NADF
$B_{VSST}$	amount of weight-material sag, expressed in pounds per gallon
$b$	slope of the annular velocity and shear stress at the wall in laminar flow, as defined in N.7.2 item h)
$c_{Ca,Aq}$	aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per liter
$c_{Ca,df}$	whole-NADF calcium concentration, expressed in milligrams per liter
$c_{CaCl_2,Aq}$	aqueous-phase calcium chloride concentration, expressed in milligrams per liter
$c_{CaCl_2,df}$	whole-NADF calcium chloride concentration, expressed in milligrams per liter

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$c_{\text{CaCl}_2, \text{df}, \text{SI}}$	whole-NADF calcium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{CaCl}_2, \text{df}, \text{USC}}$	whole-NADF calcium chloride concentration, expressed in pounds per barrel
$c_{\text{Ca}(\text{OH})_2, \text{Act}, \text{SI}}$	active concentration of field lime in the whole-NADF, expressed in kilograms per cubic meter
$c_{\text{Ca}(\text{OH})_2, \text{Act}, \text{USC}}$	active concentration of field lime in the whole-NADF, expressed in pounds per barrel
$c_{\text{Ca}(\text{OH})_2, \text{df}, \text{SI}}$	whole-NADF total lime concentration, expressed in kilograms per cubic meter
$c_{\text{Ca}(\text{OH})_2, \text{df}, \text{USC}}$	whole-NADF total lime concentration, expressed in pounds per barrel
$c_{\text{Cl}, \text{df}}$	whole-NADF chloride concentration, expressed in milligrams per liter
$c_{\text{Cl}-(\text{CaCl}_2), \text{df}}$	whole-NADF chloride concentration as calcium chloride, expressed in milligrams per liter
$c_{\text{Cl}-(\text{NaCl}), \text{df}}$	whole-NADF chloride concentration from sodium chloride, expressed in milligrams per liter
$c_{\text{NaCl}, \text{Aq}}$	aqueous-phase sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl}, \text{df}}$	whole-NADF sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl}, \text{df}, \text{SI}}$	whole-NADF sodium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{NaCl}, \text{df}, \text{USC}}$	whole-NADF sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl}, \text{df}, \text{InSol}}$	whole-NADF insoluble sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl}, \text{df}, \text{InSol}, \text{SI}}$	whole-NADF insoluble sodium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{NaCl}, \text{df}, \text{InSol}, \text{USC}}$	whole-NADF insoluble sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl}, \text{df}, \text{Sol}}$	whole-NADF soluble sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl}, \text{df}, \text{Sol}, \text{SI}}$	whole-NADF soluble sodium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{NaCl}, \text{df}, \text{Sol}, \text{USC}}$	whole-NADF soluble sodium chloride concentration, expressed in pounds per barrel
$c_{\text{LG}, \text{SI}}$	low-gravity solids concentration, expressed in kilograms per cubic meter
$c_{\text{LG}, \text{USC}}$	low-gravity solids concentration, expressed in pounds per barrel
$c_{\text{S}}$	concentration of active sulfides, expressed in milligrams per liter
$c_{\text{WM}, \text{SI}}$	weighting material concentration, expressed in kilograms per cubic meter

$c_{WM,USC}$	weighting material concentration, expressed in pounds per barrel
$C_{th}$	correction value to add to thermometer reading, expressed in degrees Celsius or Fahrenheit
$D$	diameter of outer pipe, expressed in inches
$D_{TVD}$	true vertical depth, expressed in feet
$d$	diameter of inner pipe, expressed in inches
$d_i$	distance from the outer wall, expressed in inches
$E$	pump efficiency, expressed as percentage
$f_{st}$	Dräger tube factor, taken from Table I.2
$G'$	storage modulus, expressed in newtons per square meter
$G''$	loss modulus, expressed in newtons per square meter
$k$	drilling fluid consistency factor (Herschel-Buckley model) , expressed in $lbf \cdot s^n / 100ft^2$ NOTE $n$ , drilling fluid flow behavior index (Herschel-Bulkley, see API 13D).
$L$	length of the hydraulic section, expressed in feet
$l_{SI}$	submerged length of shear tube, expressed in centimeters
$l_{USC}$	submerged length of shear tube, expressed in inches
$l_{st}$	darkened length of the Dräger tube (or equivalent), marked in units on the tube
$m_{DC}$	mass of the dried retort cuttings, expressed in grams
$m_{F1}$	initial mass of 10 mL drilling fluid (plus the syringe), expressed in grams
$m_{F2}$	mass of 10 mL drilling fluid (plus the syringe) taken from sag shoe following 30 min shear at 100 r/min, expressed in grams
$m_{F3}$	mass of 10 mL drilling fluid (plus the syringe) taken from sag shoe following 20 min shear at 600 r/min, expressed in grams
$m_L$	mass of the liquid condensed (NAF and water), expressed in grams
$m_{NAF}$	mass of the NAF, expressed in grams
$m_{NAF-C}$	mass of NAF retained on cuttings, expressed in grams
$m_W$	mass of water, expressed in grams
$m_{WC}$	mass of the wet cuttings, expressed in grams
$m_{df}$	mass of the whole-NAFD sample, expressed in grams

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$m_{ds}$	mass of the dried retort solids, expressed in grams
$m_{st}$	mass of shear tube, expressed in grams
$m_{tot}$	total shear mass (sum of platform and weights), expressed in grams
$m_1$	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, dried solids, body packed with steel wool), expressed in grams;
$P$	measured pressure, expressed in pounds-force per square inch
$Q$	pump rate, expressed in gallons per minute
$RH_{\%}$	relative humidity, expressed as a percentage
$R_B$	ratio of the volume fraction of aqueous-phase (brine) to the sum of the volume fractions of NAF and aqueous-phase (brine), expressed as a percentage
$R_{BPU}$	calculated bed pickup measurement ratio, expressed as a percentage
$R_{NAF-W}$	ratio of the volume fraction of NAF to the sum of the volume fractions of NAF and pure water from the retort analysis, expressed as a percentage
$R_{NAF-B}$	ratio of the volume fraction of NAF to the sum of the volume fractions of NAF and aqueous phase (brine), expressed as a percentage
$ROC_D$	retained NAF mass on retorted dry cuttings, expressed in grams per kilogram of dry cuttings or as a weight percentage of NAF per 100 g of dry cuttings (% $ROC_D$ )
$ROC_W$	retained NAF mass on wet cuttings, expressed in grams per kilogram of wet cuttings or as a weight percentage of NAF per 100 g of wet cuttings (% $ROC_W$ )
$R_W$	ratio of the volume fraction of water to the sum of the volume fractions of NAF and pure water from the retort analysis, expressed as a percentage
$R_1$	average reading for the standard reference thermometer, expressed in degrees
$R_2$	average reading for the working thermometer, expressed in degrees
$R_3$	shear stress reading at 3 revolutions per minute, expressed in degrees deflection, or equivalent

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$R_6$	shear stress reading at 6 revolutions per minute, expressed in degrees deflection, or equivalent
$R_{30}$	shear stress reading at 30 revolutions per minute, expressed in degrees deflection, or equivalent
$R_{60}$	shear stress reading at 60 revolutions per minute, expressed in degrees deflection, or equivalent
$R_{100}$	shear stress reading at 100 revolutions per minute, expressed in degrees deflection, or equivalent
$R_{200}$	shear stress reading at 200 revolutions per minute, expressed in degrees deflection, or equivalent
$R_{300}$	shear stress reading at 300 revolutions per minute, expressed in degrees deflection, or equivalent
$R_{600}$	shear stress reading at 600 revolutions per minute, expressed in degrees deflection, or equivalent
$S$	sag register, dimensionless
$t$	lagged time, expressed in minutes
$t_1$	time at initial reading, expressed in minutes (i.e. 7.5 min)
$t_2$	time at final reading, expressed in minutes (i.e. 30 min)
$V_{\text{AgNO}_3}$	volume of 0.282 mol/L (0.282 N) silver nitrate reagent, expressed in milliliters
$V_{\text{B}}$	base alkalinity demand (volume of 0.1 mol/L (0.1 N) NaOH), expressed in milliliters
$V_{\text{EDTA}}$	volume of 0.1 mol/L EDTA solution, expressed in milliliters
$V_{\text{H}_2\text{SO}_4}$	volume of 0.05 mol/L (0.1 N) sulfuric acid, expressed in milliliters
$V_{\text{K}}$	if positive, whole-NADF alkalinity, expressed in mL of 0.05 mol/L (0.1 N) sulfuric acid; if negative, whole-NADF acidity, expressed in mL of 0.1 mol/L (0.1 N) sodium hydroxide
$V_{\text{L}}$	total volume of condensed liquids (NAF and water), expressed in milliliters
$V_{\text{M}}$	receiver (graduated cylinder) volume at specific mark, expressed in milliliters
$V_{\text{NAF}}$	volume of NAF, expressed in milliliters
$V_{\text{NaOH}}$	volume of 0.1 mol/L (0.1 N) NaOH, expressed in milliliters
$V_{\text{NaOH,B}}$	volume of 0.1 mol/L (0.1 N) NaOH, expressed in milliliters

$V_{PPT}$	volume of filtrate after 30 min from PPT, expressed in milliliters
$V_{RC}$	volume of retort cup, expressed in milliliters
$V_W$	volume of condensed water, expressed in milliliters or water mass expressed in grams (1 mL = 1 g) (see 3.3)
$V_{an}$	annular volume, expressed in barrels
$V_{df}$	volume of the whole-NADF sample, expressed in milliliters;
$V_f$	volume of filtrate collected during the 30 min test, expressed in milliliters
$V_1$	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
$v_{an}$	annular velocity, expressed in feet per minute
$WPS$	total soluble water phase salinity, expressed in mg/kg (same as ppm);
$w_{CaCl_2}$	NADF aqueous-phase mass fraction of calcium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{CaCl_2, Sat}$	aqueous-phase mass fraction of calcium chloride of a super-saturated fluid, expressed as a percentage of the total aqueous-phase mass
$w_{NaCl}$	NADF aqueous-phase mass fraction of sodium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{NaCl, Max}$	maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of calcium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{NaCl, Max-Cal}$	recalculated maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of calcium chloride, expressed as a percentage of the total aqueous-phase mass
$w_{Ca(OH)_2, \%}$	lime assay value, expressed as a weight-percent
$Y_{P, SI}$	yield point, expressed in pascals
$Y_{P, USC}$	yield point, expressed in pounds-force per one hundred square feet
$\beta_{10m}$	gel strength at 10 min, expressed in pounds-force per one hundred square feet
$\beta_{10s}$	initial gel strength at 10 seconds, expressed in pounds-force per one hundred square feet

$\Gamma_{\text{DFG,SI}}$	drilling fluid depth gradient, expressed in kilopascals per meter
$\Gamma_{\text{DFG,USC}}$	drilling fluid depth gradient, expressed in pounds per square inch per foot
$\gamma_{\text{SI}}$	shear strength of the drilling fluid, expressed in pascals
$\gamma_{\text{USC}}$	shear strength of the drilling fluid, expressed in pounds-force per one hundred square feet
$\dot{\gamma}_i$	fluid shear rate, expressed in reciprocal seconds
$\Delta P$	anticipated pressure increase, expressed in pounds-force per square inch
$\Delta P_{\text{Rot}}$	anticipated pressure increase due to drill pipe rotation, expressed in pounds-force per square inch
$\Delta\left(\frac{P}{L}\right)$	minimum pressure gradient, expressed in pounds-force per square inch per foot
$\Delta\rho$	difference between the maximum fluid and nominal fluid density, expressed in pounds per gallon
$\Delta\rho_{\text{ECD-Rot}}$	anticipated ECD increase due to drill pipe rotation, expressed in pounds per gallon
$\Delta v_{\text{an}}$	change in annular velocity, expressed in feet per minute
$\varepsilon$	inner pipe eccentricity, dimensionless
$\eta$	drill-pipe rotation, expressed in revolutions per minute
$\eta_{\text{AV}}$	apparent viscosity, expressed in millipascal•seconds (centipoises)
$\eta_{\text{PV}}$	plastic viscosity, expressed in millipascal•seconds (centipoises)
$v_{\text{sf}}$	static filtration rate (volume rate of flow), expressed in milliliters per minute
$\phi_{\text{B}}$	volume fraction of aqueous-phase (brine), expressed as a percentage of the whole-NADF
$\phi_{\text{DS}}$	corrected volume fraction of solids, expressed as a percentage of the whole-NADF
$\phi_{\text{LG}}$	volume fraction of the low-gravity solids, expressed as a percentage of the whole-NADF
$\phi_{\text{NAF}}$	volume fraction of NAF, expressed as a percentage of the whole-NADF
$\phi_{\text{W}}$	volume fraction of water, expressed as a percentage of the whole-NADF
$\phi_{\text{WM}}$	volume fraction of the weighting-material solids, expressed as a percentage of the whole-NADF

$\rho_{ds}$	volume fraction of dried retort solids, expressed as a percentage of the whole-NADF
$\rho$	drilling fluid relative density (dimensionless),
$\rho_B$	NADF aqueous-phase (brine) density, expressed in grams per milliliter
$\rho_{ECD-hyd}$	hydraulic ECD, includes ESD, circulating pressure drop and added density effects of drilled cuttings in the annulus, expressed in pounds per gallon
$\rho_{ECD-tot}$	total predicted drilling fluid ECD in the annulus while circulating, expressed in pounds per gallon
$\rho_{LG}$	density of the low-gravity solids, expressed in grams per milliliter
$\rho_{NAF}$	density of the NAF being used, expressed in grams per milliliter
$\rho_{SS}$	average density of the suspended solids, expressed in grams per milliliter
$\rho_W$	water density, expressed in grams per milliliter, at the test temperature (see Table J.1)
$\rho_{WM}$	density of the weighting-material solids, expressed in grams per milliliter
$\rho_{df}$	drilling fluid density, expressed in grams per milliliter
$\rho_{df,SI}$	drilling fluid density, expressed in kilograms per cubic meter
$\rho_{df,USC}$	drilling fluid density, expressed in pounds per gallon
$\rho_{df,USC2}$	drilling fluid density, expressed in pounds per cubic foot
$\rho_{max}$	maximum recorded drilling fluid density, expressed in pounds per gallon
$\rho_{nom}$	nominal drilling fluid density, expressed in pounds per gallon
$\tau_{W,Cr}$	critical wall shear-stress, expressed in pounds-force per one hundred square feet
$\tau_{Wi}$	wall shear-stress, expressed in pounds-force per one hundred square feet
$\tau_Y$	drilling fluid yield-stress, calculated from the Herschel-Bulkley model, expressed in pounds-force per one hundred square feet

## 4.2 Abbreviations

For the purposes of this document, the following abbreviations apply.

ACS	American Chemical Society
ASTM	American Society of Testing Materials
BAD	base alkalinity demand

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CAS	Chemical Abstracting Services
ECD	equivalent circulating density
EDTA	sodium salt of ethylenediaminetetraacetic acid dihydrate
ERD	extended reach drilling
ES	electrical stability
ESD	equivalent static density
HTHP	high temperature, high pressure
NAF	non-aqueous fluid
NADF	non-aqueous drilling fluid
OBR	oil-to-brine ratio
OWR	oil-to-water ratio
psi	pound-force per square inch
PNP	propylene glycol normal-propyl ether
PPA	permeability plugging apparatus
PPE	personal protective equipment
PPT	permeability plugging test
PTFE	polytetrafluoroethylene
PV	plastic viscosity
PVT	pressure, volume, and temperature relationship
ROC	retained NAF (oil) on cuttings
SRE	standard reference elastomer
TC	to contain
TD	to deliver
TVD	true vertical depth
VSST	viscometer sag shoe test
YP	yield point

## **5 Determination of Drilling Fluid Density (Mud Weight)**

### **5.1 Principle**

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

### **5.2 Apparatus**

**5.2.1 Any ambient pressure density-measuring instrument** (mud balance) having an accuracy of  $\pm 0.01$  g/mL or  $\pm 10$  kg/m<sup>3</sup> ( $\pm 0.1$  lb/gal or  $\pm 0.5$  lb/ft<sup>3</sup>).

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

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

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

### **5.3 Mud Balance Procedure**

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

- a) The mud balance instrument shall be set on a flat, level surface.
- b) Measure and record the temperature of the drilling fluid.
- c) Start with a clean and dry mud balance. Fill the balance cup to the lip with the drilling fluid to be tested. Put the lid on the filled sample cup and rotate the lid, expelling any trapped air and excess drilling fluid. Ensure that some of the drilling fluid is expelled through the hole in the lid and that the lid is firmly seated.
- d) Holding the lid firmly on the filled sample cup (with the hole in the lid covered by a finger), wash and/or wipe the outside of the cup to be clean and dry.
- e) Place the beam on the base support knife edge and level by moving the sliding weight along the graduated scale. The beam is level when the bubble in the liquid-filled vial is centered between the two black lines or with a single centerline.

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

NOTE NADF density is affected by temperature. A common practice is to convert NADF density measurements to a standard temperature. This can be done graphically with manual measurements at several temperatures or with calculations based on compositional models as described in API 13D.

## 5.4 Calculation

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

To convert the drilling fluid relative density reading,  $\rho$  (dimensionless), 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) shall be used to convert the density,  $\rho_{df}$ , expressed in grams per milliliter, to other units:

$$\rho_{df,SI} = 1000 \times \rho_{df} \quad (1)$$

where

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

$$\rho_{df,USC} = 8.345 \times \rho_{df} \quad (2)$$

where

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

and

$$\rho_{df,USC2} = 62.43 \times \rho_{df} \quad (3)$$

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 1—Conversion of Density Units**

Measured in	Multiply to Convert To			
	g/mL	kg/m <sup>3</sup>	lb/gal	lb/ft <sup>3</sup>
g/mL	1	1000	8.345	62.43
kg/m <sup>3</sup>	0.001	1	0.008345	0.06243

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lb/gal	0.120	120	1	7.4805
lb/ft <sup>3</sup>	0.0160	16.02	0.1337	1

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

$$\Gamma_{\text{DFG,SI}} = 9.81 \times \rho_{\text{df}} \quad (4)$$

$$\Gamma_{\text{DFG,SI}} = 22.6 \times \Gamma_{\text{DFG,USC}} \quad (5)$$

$$\Gamma_{\text{DFG,USC}} = 0.0520 \times \rho_{\text{df,USC}} \quad (6)$$

$$\Gamma_{\text{DFG,USC}} = 0.00694 \times \rho_{\text{df,USC}2} \quad (7)$$

where

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

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

$\rho_{\text{df,USC}2}$  is the drilling fluid density, expressed in pounds per cubic foot.

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

**Table 2—Density Equivalents**

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

<sup>a</sup> Same value as relative density in grams per milliliter or kilogram per liter.  
<sup>b</sup> Accurate conversion factor.

## 6 Determination of Drilling Fluid Density—Pressurized Balance Method

### 6.1 Principle

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

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

## 6.2 Apparatus

**6.2.1 Any pressurized density-measuring instrument** (pressurized mud balance) having an accuracy of  $\pm 0.01$  g/mL or  $\pm 10$  kg/m<sup>3</sup> ( $\pm 0.1$  lb/gal or  $\pm 0.5$  lb/ft<sup>3</sup>).

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

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

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

## 6.3 Pressurized Mud Balance Procedure

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

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

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

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

- c) Place the lid on the cup with the check-valve in the down (open) position. Carefully push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Excess drilling fluid shall be expelled through the open check-valve. With the check valve opening firmly covered with a finger, wash or wipe fluid from the lid and threaded area of the cup. Then place the threaded lid retainer over the lid and screw into place. This secures the lid to the cup and expels excess fluid through the open check valve.

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

- d) The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submerging the open end into the drilling fluid with the piston rod knob pushed all the way down, then pull the piston rod knob to the fully upward position to fill. This first fill volume shall be discarded into another container and the plunger shall be refilled with drilling fluid in case the first fill plunger volume was contaminated with a previous sample or a liquid from the last clean-up of the plunger assembly.
- e) Place the pressurized mud balance on a stable surface, attach the filled plunger by pushing the nose of the plunger onto the mating O-ring surface of the lid check valve. Pressurize the sample cup by holding a downward force on the plunger housing with one hand in order to hold the check-valve down (open) and at the same time push down and maintain a force of approximately 225 N (50 lbf) or greater on the plunger piston rod knob.
- f) The check-valve in the lid is pressure-actuated; when the cup is pressurized, the check valve is pushed upward into the closed position. To close the check valve, gradually decrease the downward force on the plunger housing while maintaining downward pressure on the plunger piston rod knob. After the downward pressure is reduced on the plunger, the check-valve moves up slightly and closes as pressure is released. Release pressure on the piston rod before disconnecting the plunger.
- g) The pressurized drilling fluid sample is now ready for weighing. Rinse and/or wipe the outside of the cup to be clean and dry. Place the beam on the base support knife edge and level by moving the sliding weight along the graduated scale. The beam is level when the bubble in the liquid-filled vial is centered between the two black lines.
- h) The drilling fluid density is read from one of the four calibrated scales on the balance arm, at the arrow side of the sliding counterweight. The density can be read directly in units of grams per milliliter using relative density scale (specific gravity), pounds per gallon, pounds per cubic foot, or as a drilling fluid gradient in pounds per square inch per 1000 ft of vertical depth.

NOTE NADF density is affected by temperature. A common practice is to convert NADF density measurements to a standard temperature. This can be done graphically with manual measurements at several temperatures or with calculations based on compositional models as described in API 13D.
- i) To release the pressure inside the cup, reconnect the empty plunger assembly and push down on the plunger housing until the check valve opens and stays in the down position.
- j) Empty and clean the cup, lid (including check-valve), threaded lid retaining ring, and plunger assembly.

## 6.4 Calculation

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

For conversions, use the equations, conversion factors, or table of values given in 5.4.

## 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 under dynamic conditions and the thixotropic properties under static.

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

- a) Marsh funnel: a simple device for indicating viscosity on a routine basis.
- b) Direct-indicating viscometer: a mechanical device for measuring viscosity at varying shear rates.

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

## 7.2 Determination of Viscosity Using the Marsh Funnel

### 7.2.1 Apparatus

**7.2.1.1 Marsh funnel**, calibrated to deliver (TD) 946 mL (1 quart) of fresh water at a temperature of  $21\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$  ( $70\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ) in 26 seconds  $\pm 0.5$  seconds, with a graduated cup as a receiver.

The Marsh funnel shall have the following characteristics:

- a) funnel cone, length 305 mm (12.0 in.), diameter 152 mm (6.0 in.) and a capacity to bottom of screen of 1500 mL (1.6 quarts);
- b) orifice, length 50.8 mm (2.0 in.) and inside diameter 4.7 mm ( $0.185\text{ in.} = \frac{3}{16}\text{ in.}$ );
- c) screen, with 1.68 mm [0.066 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 quart).

**7.2.1.3 Stopwatch** or **timer**, accuracy within  $\pm 5$  seconds per hour.

**7.2.1.4 Thermometer**, with a range of  $0\text{ }^{\circ}\text{C}$  to  $105\text{ }^{\circ}\text{C}$  ( $32\text{ }^{\circ}\text{F}$  to  $220\text{ }^{\circ}\text{F}$ ) and an accuracy of  $\pm 1\text{ }^{\circ}\text{C}$  ( $\pm 2\text{ }^{\circ}\text{F}$ ).

### 7.2.2 Procedure

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

- a) Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.
- b) Remove finger and start the stopwatch or timer simultaneously. Measure the time for drilling fluid to fill to the 946 mL (1 quart) mark of the cup.
- c) Measure the temperature of the fluid, in degrees Celsius (degrees Fahrenheit).
- d) Report the time (see item b) to the nearest second as the Marsh funnel viscosity. Report the temperature (see item c) of the fluid to the nearest degree Celsius (degree Fahrenheit).

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

### 7.3.1 Apparatus

**7.3.1.1 Direct-indicating viscometer** <sup>[19]</sup>, powered by an electric motor or a hand crank.

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

NOTE Some of the current generation of electronic digital drilling fluid viscometers use electronic speed control and strain gauge measurements of the torque acting on the bob instead of the rotation of a dial restrained by a torsional spring and therefore have digital outputs for the equivalent dial readings or degrees.

The components shall meet the following specifications:

a) **Rotor sleeve—R1:**

inside diameter	36.83 mm (1.450 in.)
total length	87.0 mm (3.425 in.)
scribed line	58.4 mm (2.30 in.) above the bottom of sleeve, with two rows of 3.18 mm (0.125 in.) holes, spaced 120° (2.09 rad) apart, around rotor sleeve just below scribed line
sleeve surface	surface roughness: RMS average 0.4 μm to 0.8 μm (16 μin. to 32 μin.) cross-hatch honed

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

diameter	34.49 mm (1.358 in.)
cylinder length	38.0 mm (1.496 in.)
rotor surface	surface roughness: RMS average 0.4 μm to 0.8 μm (16 μin. to 32 μin.) cross-hatch honed

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

c) **Torsion spring constant—F1.0:**

torsional stiffness	10.54 N•m/rad (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•ft <sup>2</sup> 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 direct-indicating viscometers from various manufacturers. Additional measurements are typically made at rotor speeds of 200 r/min, 100 r/min, 6 r/min and 3 r/min.

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

**7.3.1.3 Thermostatically controlled viscometer cup** (thermocup):

- greater than room temperature: thermostatically controlled direct-heated viscometer cup;
- 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  $\pm 1$  °C ( $\pm 2$  °F).

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

### 7.3.2 Procedure

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

- a) Stir the drilling fluid sample for 5 min using the field mixer set at the 10,000 r/min speed.
- b) Place a sample of the drilling fluid in a thermostatically controlled viscometer cup. Leave enough empty volume (approximately 50 mL to 100 mL) in the cup for displacement of fluid due to the viscometer bob and sleeve. Immerse the rotor sleeve exactly to the scribed line.

Measurements in the field should be made with minimum delay from the time of drilling fluid sampling. Testing shall be carried out at either 50 °C  $\pm 1$  °C (120 °F  $\pm 2$  °F) or 65 °C  $\pm 1$  °C (150 °F  $\pm 2$  °F) for reference comparisons to historical data. Testing at a lower temperature, such as 4 °C  $\pm 1$  °C (40 °F  $\pm 2$  °F), should be recommended for low temperature effects. The place of sampling should be stated on the daily drilling fluid 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 is used. Liquid trapped inside a hollow bob can vaporize when immersed in high-temperature fluid and cause the bob to explode.**

- c) Heat (or cool) the sample to the selected temperature. Use intermittent or constant shear at 600 r/min to stir the sample while heating (or cooling) to obtain a uniform sample temperature.

After the heat cup temperature reaches the selected temperature, immerse the thermometer into the sample and continue stirring until the sample reaches the selected temperature. Record the temperature of the sample.

- d) With the sleeve rotating at 600 r/min, wait for the viscometer dial (or digital) reading to reach a steady value (the time required is dependent on the drilling fluid characteristics). Record the dial (or digital) shear stress reading,  $R_{600}$ .
- e) Reduce the rotor speed to 300 r/min and wait for the dial (or digital) reading to reach steady value. Record the dial (or digital) shear stress reading,  $R_{300}$ .

NOTE Additional shear stress readings  $R_{200}$ ,  $R_{100}$ ,  $R_6$  and  $R_3$ , are often measured and reported, typically at speeds of 200 r/min, 100 r/min, 6 r/min and 3 r/min Improved modeling accuracy can be achieved by also measuring the  $R_{60}$  and  $R_{30}$  shear stress readings at speeds of 60 r/min and 30 r/min.

- f) Stir the drilling fluid sample for 10 seconds at 600 r/min.
- g) Stop the rotor and allow the drilling fluid sample to stand undisturbed for 10 seconds.

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

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

Record the initial gel strength,  $\beta_{10s}$ , at 10 seconds, in pounds-force per one hundred square feet (lbf/100•ft<sup>2</sup>).

NOTE The gel strength value reported directly from the dial reading is an approximation of lbf/100•ft<sup>2</sup>. 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<sup>2</sup> in USC units. These exact unit corrections are often neglected for simplicity in reporting. Common field practice when reporting pascals is to divide dial units by 2 for simplicity.

- h) Re-stir the drilling fluid sample at 600 r/min for 10 seconds, stop the motor and then allow the drilling fluid to stand undisturbed for 10 min. Repeat the measurements as in item g) and report the maximum dial (or digital) reading as  $\beta_{10m}$ , the 10 min gel strength, in pounds-force per one hundred square feet (lbf/100•ft<sup>2</sup>).

### 7.3.3 Calculations

#### 7.3.3.1 Direct-indicating viscometer calculation constants

The dimensions for the rotor, bob and spring constant, as described in 7.3.1.1, determine the following (or through calibration of electronic instruments):

- 1° deflection of the bob equals a shear stress of approximately 1 lbf/100•ft<sup>2</sup> (0.5 Pa), or more exactly 1 degree of deflection = 1.067 lbf/100•ft<sup>2</sup> and = 0.511 Pa;
- 1 r/min of the rotor equals a shear rate of 1.7023 s<sup>-1</sup>.

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

#### 7.3.3.2 Drilling fluid plastic viscosity

The plastic viscosity,  $\eta_{PV}$ , expressed in millipascal•seconds (centipoises), shall be calculated using Equation (8):

$$\eta_{PV} = R_{600} - R_{300} \quad (8)$$

where

$R_{600}$  is the shear stress reading at 600 revolutions per minute, expressed in degrees deflection, or equivalent;

$R_{300}$  is the shear stress reading at 300 revolutions per minute, expressed in degrees deflection, or equivalent.

NOTE 1 Plastic viscosity is commonly known in the industry by the abbreviation PV, (see Annex 0 Example of Non-Aqueous Drilling Fluid Report).

NOTE 2 1 cP = 1mPa•s.

### 7.3.3.3 Drilling fluid yield point

#### 7.3.3.3.1 Yield point expressed in SI units

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

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

Where

$\eta_{PV}$  is the plastic viscosity, expressed in pascals;

$R_{300}$  is the shear stress reading at 300 revolutions per minute, expressed in degrees deflection, or equivalent.

NOTE The conversion from lbf/100•ft<sup>2</sup> 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.

#### 7.3.3.3.2 Yield point expressed in USC units

The yield point,  $Y_{P,USC}$ , expressed in approximate pounds-force per one hundred square feet, shall be calculated using Equation (10)

$$Y_{P,USC} = R_{300} - \eta_{PV} \quad (10)$$

where

$\eta_{PV}$  is the plastic viscosity, expressed in centipoises;

$R_{300}$  is the shear stress reading at 300 revolutions per minute, expressed in degrees deflection, or equivalent.

NOTE Yield point, in lbf/100•ft<sup>2</sup>, is commonly known in the industry by the abbreviation YP, (see Annex 0 Example of Non-Aqueous Drilling Fluid Report).

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

### 7.3.3.4 Drilling fluid apparent viscosity

The apparent viscosity,  $\eta_{AV}$ , expressed in millipascal•seconds (centipoises), shall be calculated using Equation (11):

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

where

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

### **7.3.3.5 Reporting**

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

## **8 Static Filtration**

### **8.1 Principle**

**8.1.1** Measurement of the filtration behavior and the filter cake characteristics of a non-aqueous drilling fluid (NADF) are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as the presence of water (brine), solids, or emulsion.

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

**8.1.3** Filtration tests are performed at high-temperature/high-pressure (HTHP) static conditions. Field static HTHP tests are conducted using a differential pressure of 3450 kPa (500 lbf/in.<sup>2</sup>) for 30 minutes. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing from 175 °C (350 °F) to 230 °C (450 °F). Use only the filtration equipment and procedure specified for the temperature required.

NOTE No low-temperature filtration test procedure for NADFs is specified herein, but it can be performed much like the water-based drilling fluid test provided in API 13B-1.

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

**8.1.5** For testing above 175 °C (350 °F), only the 500 mL unit rated by the manufacturer for the test temperature shall be used. It is recommended that the cell be equipped with a thermocouple in direct contact with the fluid contained in the cell to measure the temperature more accurately. Filter paper (including fiberglass backed filter paper) shall not be used 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 shall be used. Ceramic disks may also be used in equipment modified to utilize these disks.

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

### **8.2 HTHP Static Filtration Test up to 175 °C (350 °F)**

#### **8.2.1 Apparatus**

**8.2.1.1** HTHP filter press, consisting of:

- a) Filter cell, rated for working pressures up to 9000 kPa (1300 lbf/in.<sup>2</sup>) at a temperature of 175 °C (350 °F);
- b) pressurized gas source shall be nitrogen or carbon dioxide, with regulators;

NOTE Nitrogen is the preferred gas for this application.

- c) heating system with temperature controller or thermostat, 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 or thermocouple in direct contact with the fluid near the center of the cell, fitted with a removable end, a filter-media support, and with NAF-resistant seals.

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

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

**Warning—Do not use nitrous oxide cartridges as pressure sources for NADF HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, non-aqueous fluids, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for water-based drilling fluid Garrett gas train carbonate analysis, (see API 13B-1).**

**Table 3—Recommended Minimum Back-pressure**

Test Temperature		Vapor Pressure Water		Minimum Back-pressure	
°C	°F	kPa	lbf/in. <sup>2</sup>	kPa	lbf/in. <sup>2</sup>
95–149	200–299	84–462	12.1–67	700	100
150–189	300–374	462–1269	67–184	1400	200
190–199	375–399	1269–1704	184–247	1900	275
200–219	400–424	1704–2245	247–326	2500	350
220–230	425–450	2245–2912	326–422	3100	450

- 8.2.1.2 **Filter medium:** filter paper <sup>3</sup> qualitative, hardened, low-ash, slow filtration rate, grade 50. A new paper shall be required for each test.
- 8.2.1.3 **Timer, digital or mechanical,** with at least a 30 min interval, accuracy within ±5 seconds per hour.
- 8.2.1.4 **Thermometer,** with a range up to 260 °C (500 °F), accuracy of ±3 °C (±5 °F) with a 12.5 cm (5 in.) or longer stem, or a thermocouple with a range up to 260 °C (500 °F), preferred.
- 8.2.1.5 **Graduated cylinder,** to contain (TC) with a capacity of 10 mL, 20 mL, or 25 mL.
- 8.2.1.6 **Field mixer,** cup type, to operate at 10,000 r/min to 15,000 r/min.
- 8.2.1.7 **Ruler,** graduated in millimeters (<sup>1</sup>/<sub>32</sub> in.), to measure filter cake thickness.

<sup>3</sup> 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 slightly by manufacturer: slow filtration rate: 2685 herzbergs; particle retention in liquid: 2 µm to 5 µm; ash content: 0.015% by weight; basis weight: 92 g/m<sup>2</sup>; diameter: 63.5 mm (2.5 in.); thickness: 0.137 mm.

## 8.2.2 Procedure

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

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

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

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

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

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

- c) Ensuring that all O-rings are in place, install the lower end cap, with the valve stem open, above the filtration media and secure. Close the lower valve stem.
- d) Complete the assembly of the filter cell and place it in the preheated heating jacket with the filter media on the bottom. Rotate the cell to lock it in place with the pin inside the heating jacket. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, insert a thermometer into the well of the filter cell wall.
- e) Ensure that the filtrate collection vessel is completely free of water or NAF. Connect the regulated-pressure filtrate collection vessel assembly onto the lower valve stem and lock it in place. Connect the regulated-pressure assembly to the upper valve and lock it in place.
- f) Keeping the two valve stems closed, adjust the pressure on the upper and lower pressure regulators to 700 kPa (100 lbf/in.<sup>2</sup>) for temperatures below 150 °C (300 °F) or 1400 kPa (200 lbf/in.<sup>2</sup>) for temperatures between 150 °C (300 °F) and 175 °C (350 °F). Open the upper valve stem and readjust the upper pressure regulator to maintain this pressure. Maintain these pressures with the upper valve open during the heat-up time. If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, proceed to next item g) when the thermocouple temperature reaches the test temperature. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, wait 30 min after the filter cell wall reaches the test temperature before proceeding to item g).

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

- g) When the sample reaches the selected test temperature, as indicated by the thermocouple or after the 30 min heat-up time, ensure that the lower pressure regulator is at 700 kPa (100 lbf/in.<sup>2</sup>) for temperatures below 150 °C (300 °F) or 1400 kPa (200 lbf/in.<sup>2</sup>) for temperatures between 150 °C (300 °F) and 175 °C (350 °F).

Open the lower valve stem and immediately increase the pressure on the upper regulator to 4150 kPa (600 lbf/in.<sup>2</sup>) for temperatures below 150 °C (300 °F) or 4850 kPa (700 lbf/in.<sup>2</sup>) for temperatures between 150 °C (300 °F) and 175 °C (350 °F). This will start the filtration process. Start the timer.

Maintain the test temperature to within  $\pm 3$  °C ( $\pm 5$  °F) during the test, as indicated by the thermocouple or thermometer in the filter cell wall. Maintain these pressures with both valves open during the test, if the

back pressure rises above the selected back pressure, cautiously draw off and collect a portion of the filtrate to reduce the back pressure.

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

- h) Collect the filtrate in the graduated cylinder. Record the total volume of filtrate collected during the 30 min test, in milliliters. Correct the filtrate volume,  $V_f$ , to a filter area of 45.8 cm<sup>2</sup> (7.1 in.<sup>2</sup>). For example, if the filter area is 22.6 cm<sup>2</sup> (3.5 in.<sup>2</sup>), double the filtrate volume reported. Also note the presence or volume of any water (brine), solids, or emulsion if present.
- i) Immediately after collecting the 30 min filtrate, close the lower, then the upper valve stem. Switch off the heating jacket and unplug from the electrical power source. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, and then carefully disconnect the pressurization system.

**Warning—Cell and heating jacket are still hot !**

- j) Using appropriate personal protective equipment, carefully remove the cell from the heating jacket and allow cell to cool to below 50 °C (125 °F). Keep the cell upright during cooling, depressurization, and disassembly.

**Warning—The filter cell may still be pressurized even after the cell is cooled. Safety Cell Clamps are available which keep the cell upright and the lid clamped during depressurization. To avoid possible serious injury, keep cell upright and cool to room temperature, and then bleed pressure from cell before disassembling.**

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

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

- l) Remove the filter cake with the filtration medium. Measure the filter cake thickness, at its center, to the nearest millimeter ( $1/32$  in.).
- m) Pour the liquid from the cell.
- n) Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics under the "Comments" on the Drilling Fluid 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.

### 8.2.3 Calculation and reporting

**8.2.3.1** The filtrate volume,  $V_f$ , shall be corrected to a filter area of 45.8 cm<sup>2</sup> (7.1 in.<sup>2</sup>). HTHP filter cells usually have half the standard filter area 22.6 cm<sup>2</sup> (3.5 in.<sup>2</sup>), thus double the measured filtrate volume before reporting.

**8.2.3.2** Report the cake thickness to the nearest millimeter ( $1/32$  in.), its texture, character, and the presence of any water (brine), solids, or emulsion in the filtrate.

### 8.3 HTHP Static Filtration Test from 175 °C (350 °F) up to and Including 230 °C (450 °F)

#### 8.3.1 Apparatus

##### 8.3.1.1 HTHP filter press, consisting of the following components:

- a) 500 mL volume cell, only;

NOTE For safety reasons, only the 500 mL cell shall be used for testing at and above 230 °C (450 °F).

- b) filter cell, rated for working pressures up to 15,500 kPa (2250 lbf/in.<sup>2</sup>) at a temperature of 230 °C (450 °F);
- c) pressurized gas source shall be nitrogen or carbon dioxide, with regulators;

NOTE Nitrogen is the preferred gas for this application.

- d) heating system with temperature controller or thermostat, to heat to 260 °C (500 °F);
- e) high-pressure filtrate collection vessel, maintained at proper back pressure (see Table 3), to avoid flashing or evaporation of the filtrate;

NOTE For temperatures >190 °C (>375 °F) the back-pressure gauge of the high-pressure filtrate collection vessel will need to be capable of measuring pressures above 1400 kPa (200 lbf/in.<sup>2</sup>).

- f) filter cell containing a thermometer well or thermocouple in direct contact with the fluid near the center of the cell, fitted with a removable end, a filter-media support and with NAF-resistant seals.

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

**Warning—Not all manufacturers' equipment can be used above 150 °C (300 °F). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions. The 175 mL and 250 mL filtration cells are not recommended for use at these higher temperatures and pressures. Strict adherence to manufacturer's recommendations as to sample volumes, equipment temperatures and pressures, O-ring material selection and inspection, and other operating instructions is essential. High temperature O-rings and elastomer seals are required for high temperatures. Equipment manufacturers often recommend replacement or inspection after every test. Inspection should include making sure the O-rings and elastomer seals are pliable with no permanent deformations, nicks, or cuts, etc. A loss of pressure during a test may be an indication of an O-ring or elastomer seal failure and should be corrected. Using appropriate PPE and safety barriers is recommended when operating HTHP equipment. Failure to follow these precautions could result in serious injury.**

**Warning—Do not use nitrous oxide cartridges as pressure sources for NADF HTHP filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for water-based drilling fluid Garrett gas train carbonate analysis (see API 13B-1).**

##### 8.3.1.2 Filter medium;

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

- a) Filter paper<sup>6</sup>, qualitative, hardened, low-ash, slow filtration rate, grade 50, (see 8.2.1.2) and glass fiber

backing disk <sup>4</sup> for temperatures up to 200 °C (400 °F). A new filter medium shall be required for each test;

- b) Porous sintered stainless-steel disc, Dynalloy X-5 <sup>5</sup> or equivalent, diameter 6.35 cm (2.5 in.), for temperatures above 200 °C (400 °F). A new disc shall be required for each test.

**8.3.1.3 Mechanical or electronic timer**, with at least a 30 min interval, accuracy within  $\pm 5$  seconds per hour.

**8.3.1.4 Thermometer**, with a range up to 260 °C (500 °F), accuracy of  $\pm 3$  °C ( $\pm 5$  °F) with a 12.5 cm (5 in.) or longer stem, or a thermocouple with a range up to 260 °C (500 °F), preferred.

**8.3.1.5 Graduated cylinder** (TC), with a volume of 10 mL, 20 mL, or 25 mL.

**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 static filtration test at 175 °C (350 °F) up to and including 230 °C (450 °F) using a differential pressure of 3450 kPa (500 lbf/in.<sup>2</sup>), the following procedure shall be used.

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

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

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

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

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

- c) Ensuring that all O-rings are in place, install the lower end cap, with the valve stem open, above the filtration media and secure. Close the lower valve stem.
- d) Complete the assembly of the filter cell and place it in the preheated heating jacket with the filter media on the bottom. Rotate the cell to lock it in place with the pin inside the heating jacket. If the filtration unit is

<sup>4</sup> Glass fiber backing disks shall be required for testing between 175 °C (350 °F) up to 200 °C (400 °F). These Backing disks are a separate item and are inserted between the filter paper and the filter-media support of the filter cell.

<sup>5</sup> Dynalloy X-5 is an example of a suitable brand of porous sintered stainless-steel disc available commercially. This information is given for the convenience of users of API 13-B2 and does not constitute an endorsement by API of these products.

not equipped with a thermocouple in direct contact with the drilling fluid, insert a thermometer into the well of the filter cell wall.

- e) Ensure that the filtrate collection vessel is completely free of water or NAF. Connect the regulated-pressure filtrate collection vessel assembly onto the lower valve stem and lock it in place. Connect the regulated pressurized gas source to the upper valve and lock in place.
- f) Keeping the two valve stems closed, adjust the pressure on the upper and lower pressure regulators to the minimum back-pressure for the test temperature as shown in Table 3. Open the upper valve stem and readjust the upper pressure regulator to the minimum back-pressure temperature. Maintain these pressures with the upper valve open during the heat-up time. If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, proceed to next item g) when the thermocouple temperature reaches the test temperature. If the filtration unit is not equipped with a thermocouple in direct contact with the drilling fluid, wait 30 min after the filter cell wall reaches the test temperature before proceeding to item g).

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

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

Open the lower valve stem and immediately increase the pressure on the upper regulator to a value 3450 kPa (500 lbf/in.<sup>2</sup>) higher than the back-pressure. This will start the filtration process. Start the timer.

Maintain the test temperature to within  $\pm 3$  °C ( $\pm 5$  °F) during the test, as indicated by the thermocouple or thermometer in the filter cell wall. Maintain these pressures with both valves open during the test, if the back pressure rises above the selected back pressure, cautiously draw off and collect a portion of the filtrate to reduce the back pressure.

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

- h) Collect the filtrate in the graduated cylinder. Record the total volume of filtrate collected during the 30 min test, in milliliters. Correct the filtrate volume,  $V_f$ , to a filter area of 45.8 cm<sup>2</sup> (7.1 in.<sup>2</sup>). For example, if the filter area is 22.6 cm<sup>2</sup> (3.5 in.<sup>2</sup>), double the filtrate volume reported. Also note the presence or volume of any water (brine), solids, or emulsion if present.
- i) Immediately after collecting the 30 min filtrate, close the lower, then the upper valve stem. Switch off the heating jacket and unplug from the electrical power source. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, and then carefully disconnect the pressurization system.

**Warning—Cell and heating jacket are still hot!**

- j) Using appropriate 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.

**Warning—The filter cell may still be pressurized even after the cell is cooled. Safety Cell Clamps are available which keep the cell upright and the lid clamped during depressurization. To avoid possible serious injury, keep cell upright and cool to room temperature, and then bleed pressure from cell before disassembling.**

- k) After cooling the cell, bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits the stem. A tight-fitting hose may be used to divert the pressurized gas

and any liquid to a safe contained area during depressurization. Ensure that pressure is fully released before removing the cap. Carefully disassemble the cell.

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

- l) Remove the filter cake with the filtration medium. Measure the filter cake thickness, at its center, to the nearest millimeter ( $1/32$  in.).
- m) Pour the liquid from the cell.
- n) Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics under the "Comments" on the Drilling Fluid 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 and reporting

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

**8.3.3.2** Report the cake thickness to the nearest millimeter ( $1/32$  in.), its texture, character, and the presence of any water (brine), solids, or emulsion in the filtrate.

## 9 Retort Test for Non-aqueous Fluid, Water, and Solids Concentrations

### 9.1 Principle

**9.1.1** A retort test measures water and non-aqueous fluid fraction released from a NADF sample when heated in a calibrated and properly operating retort instrument. Included in this section are procedures for performing a retort analysis using either a volumetric or gravimetric method.

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

**9.1.2** Knowledge of water, NAF, and solids concentrations is fundamental to proper control of drilling fluid properties such as NAF-to-water ratio, rheology, density, filtration, and salinity of the aqueous (water) phase. Knowledge of solids in a NADF is essential to the evaluation of drilling fluid processing equipment performance, see API 13C Section 5.

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

**9.1.4** For the volumetric method, the volume fractions expressed as percentages of NAF, water, and solids are calculated from the total starting volume of NADF and the condensed liquid volumes of water and NAF collected in a precision-graduated liquid receiver.

**9.1.5** For the gravimetric method, the volume fractions expressed as percentages of NAF, water, and solids are calculated from the mass of retorted NADF, mass of dry solids after retorting, densities of the water, NAF, and NADF plus the measured volume of condensed water collected in a precision graduated receiver. If the density of the NAF used is not known, procedures are given for either measuring the density (see 9.5 item h) or calculating it from the gravimetric retort mass measurements (see 9.7.5).

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

## 9.2 Apparatus

### 9.2.1 Retort instrument, as specified below.

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

Standard cup sizes are 10 mL (precision  $\pm 0.05$  mL), 20 mL (precision  $\pm 0.1$  mL), and 50 mL (precision  $\pm 0.25$  mL). When using the volumetric procedure, the retort cup volume with lid shall be verified gravimetrically in accordance with the procedure and calculations given in Annex J (see J.6).

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

- c) **Heating jacket**, nominal power 350 W.

- d) **Temperature controller**, capable of limiting the temperature of the retort to  $500\text{ }^{\circ}\text{C} \pm 40\text{ }^{\circ}\text{C}$  ( $930\text{ }^{\circ}\text{F} \pm 70\text{ }^{\circ}\text{F}$ ).

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

- **precision**: see Table 4;
- **calibration (TC)**: at  $20\text{ }^{\circ}\text{C}$  ( $68\text{ }^{\circ}\text{F}$ );
- **scale**: milliliter or volume fraction (as a percentage);
- **material**: transparent and inert to NAF, water, and salt solutions at temperatures up to  $32\text{ }^{\circ}\text{C}$  ( $90\text{ }^{\circ}\text{F}$ ).

When using the volumetric procedure, the receiver volume shall be verified gravimetrically and shall be in accordance with the procedure and calculations in Annex J (see J.3).

**Table 4—Precision of Liquid Receiver**

		Total Volume			
		10 mL	20 mL	50 mL	50 mL tapered
<b>Precision</b>	(0 % to 100 %)	$\pm 0.05$ mL	$\pm 0.10$ mL	$\pm 0.25$ mL	—
<b>Frequency of graduation marks</b>	(0 % to 100 %)	0.10 mL	0.10 mL	0.50 mL	—
	(0 % to 5 %)	—	—	—	0.05 mL
	(5 % to 100 %)	—	—	—	0.25 mL

### 9.2.3 Fine steel wool, oil-free.

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

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

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

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

**9.2.7 Corkscrew.**

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

**9.2.9 Marsh funnel** (7.2.1.1).

**9.2.10 Top-loading balance**, capable of weighing 2000 g with an accuracy of  $\pm 0.01$  g.

**9.2.11 Mud balance** (5.2.1 or 6.2.1 preferred).

### **9.3 Procedure—Volumetric Method**

In order to conduct a retort analysis using the volumetric method, the following procedure shall be used.

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

**Caution—A moist or partially clogged condenser passage may be a safety hazard.**

- b) The heating jacket shall be cooled to less than 93 °C (200 °F).
- c) Pack the retort body with steel wool.
- d) Collect a representative sample of NADF and allow it to cool to approximately 27 °C (80 °F). Screen the test sample through the 1.68 mm [0.066 in. (12-mesh)] screen of the Marsh funnel to remove lost circulation material, large cuttings, or debris.
- e) Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Be careful not to entrain any air and ensure that no solids remain on the bottom of the container.

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

- 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. Ensure that a small excess of drilling fluid flows out of the hole in the lid. Wipe excess sample from the lid; avoid wicking out the drilling fluid through the hole.
- 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 1 To improve the ability to make accurate readings of the volume of NAF and water, it is possible to pre-wet the inside of the glass liquid receiver with propylene glycol normal-propyl ether (PNP). PNP is the product used to break the non-aqueous fluid (NAF) emulsion during chemical titration of NADF (see Section 10). The basic method for wetting the glass liquid receiver is to add approximately 0.5 mL of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

**Caution—PNP should be used only when the precautions, appropriate PPE, and exposure controls are followed as described on the safety data sheet, as is recommended for all laboratory chemicals. This includes eye/face protection, skin protection, and respiratory protection for both liquid and/or vapor exposure.**

NOTE 2 Due to the rounded bottom of the liquid receiver, it might be helpful to place the liquid receiver in a 100 mL graduated cylinder to hold it on the top-loading balance while being weighed.

NOTE 3 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

- j) Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Collect the condensate into the glass liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.
- k) Remove the liquid receiver and allow it to cool.

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

NOTE If an emulsion interface is present between the NAF 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.

- l) Record the total liquid volume,  $V_L$ , 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 liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for the water-to-NAF meniscus, read the water volume at the lowest point.

- m) Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser. Clean the retort assembly and condenser.

## 9.4 Calculation—Volumetric Method

### 9.4.1 General

Using the measured volumes of condensed base NAF and water, plus the volume of the original whole-NADF sample (10 mL, 20 mL, or 50 mL), the volume fractions of water, NAF, and total solids in the drilling fluid shall be calculated using Equations (13) to (15).

#### 9.4.2 Volume fraction of NAF

The volume of NAF in the condensed sample shall be calculated using Equation (12)

$$V_{\text{NAF}} = V_{\text{L}} - V_{\text{W}} \quad (12)$$

where

$V_{\text{NAF}}$  is the volume of NAF, expressed in milliliters;

$V_{\text{L}}$  is the total volume of condensed liquids (NAF and water), expressed in milliliters;

$V_{\text{W}}$  is the condensed water volume expressed in milliliters or water mass expressed in grams (1 mL = 1 g) (see 3.3).

The volume fraction of NAF in the whole-NADF sample shall be calculated using Equation (13).

$$\phi_{\text{NAF}} = 100 \times \frac{V_{\text{NAF}}}{V_{\text{df}}} \quad (13)$$

where

$\phi_{\text{NAF}}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF ;

$V_{\text{NAF}}$  is the volume of NAF expressed in milliliters;

$V_{\text{df}}$  is the whole-NADF sample volume, expressed in milliliters.

#### 9.4.3 Volume fraction of water

The volume fraction of water in the whole NADF sample shall be calculated using Equation (14)

$$\phi_{\text{W}} = 100 \times \frac{V_{\text{W}}}{V_{\text{df}}} \quad (14)$$

where

$\phi_{\text{W}}$  is the volume fraction of water, expressed as a percentage of the whole-NADF;

$V_{\text{W}}$  is the condensed water volume, expressed in milliliters or water mass expressed in grams (1 mL = 1 g) (see 3.3);

$V_{\text{df}}$  is the whole-NADF sample volume, expressed in milliliters.

#### 9.4.4 Volume fraction of dried solids

The volume fraction of dried solids remaining in the retort shall be calculated using Equation (15).

$$\phi_{\text{ds}} = 100 - (\phi_{\text{W}} + \phi_{\text{NAF}}) \quad (15)$$

where

- $\phi_{ds}$  is the volume fraction of dried retort solids, expressed as a percentage of the whole NADF;
- $\phi_W$  is the volume fraction of water, expressed as a percentage of the whole-NADF;
- $\phi_{NAF}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF.

NOTE The calculated solids will overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity shall be made using known volume factors for salt solutions (see Section 12). Calculations for determining the NAF-to-water ratio and the corrected solids concentration and density are in Section 12.

### 9.5 Procedure—Gravimetric Method

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

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

**Caution—A moist or partially clogged condenser passage may be a safety hazard.**

- b) The heating jacket shall be cooled to less than 93 °C (200 °F).
- c) Pack the retort body with steel wool.
- d) Apply lubricant/sealant sparingly to the threads of the retort cup. With lid in place, hand-tighten the retort cup onto the body.
- e) Weigh and record the total mass of the empty retort assembly (i.e. sample cup, lid, and retort body packed with steel wool). Record this as  $m_1$ , expressed in grams.

NOTE All weights are recorded to the nearest 0.01 g.

- f) Collect a representative sample of NADF and allow it to cool to approximately 27 °C (80 °F). Screen the test sample through the 1.68 mm [0.066 in. (12-mesh)] screen of the Marsh funnel to remove lost circulation material, large cuttings, or debris.
- g) Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Be careful not to entrain any air and ensure that no solids remain on the bottom of the container.

Measure the density of the NADF using a mud balance as described in Section 5 or Section 6 (more accurate). Alternative gravimetric methods, such as a volumetric flask or cup, are also acceptable. Optionally, there are several small-volume portable handheld density-measuring devices that can be used to accurately measure the density of the drilling fluid.

Record the NADF sample density as  $\rho_{df}$  to the nearest 0.01 g/mL (0.1 lb/gal).

- h) If the density of the base NAF being used is not known, measure the density of the base NAF using a mud balance, as described in Section 5 or Section 6 (more accurate). Alternative gravimetric methods,

such as a volumetric flask or cup, are also acceptable. Optionally, there are several small-volume portable handheld density-measuring devices that can be used to accurately measure the density of the condensed NAF. The density of the NAF may also be determined from the gravimetric retort mass measurements as described in 9.7.5.

Record the NAF density as  $\rho_{\text{NAF}}$ , to the nearest 0.01 g/mL, 10 kg/m<sup>3</sup> (0.1 lb/gal or 0.5 lb/ft<sup>3</sup>).

- i) Remove the retort cup from the retort body. Fill the retort sample cup slowly to avoid air entrapment. Lightly tap the side of the cup to expel air. Place the lid on the cup. Rotate the lid to obtain a proper fit. Ensure that a small excess of drilling fluid flows out of the hole in the lid. Wipe excess sample from the lid; avoid wicking out the drilling fluid through the hole.
- j) Screw the retort sample cup with lid onto the retort body. Weigh the retort sample cup filled assembly (i.e. cup with NAFD sample, lid, and retort body packed with steel wool). Record this as  $m_2$ , expressed in grams.

NOTE All weights are recorded to the nearest 0.01 g.

- k) Apply lubricant/sealant sparingly to the threads on the condenser passage stem and attach to the condenser body.
- l) Place the retort assembly into the heating jacket. Close the insulating lid.
- m) Weigh an empty, clean, dry liquid receiver. Record this as  $m_3$ , expressed in grams. Place the receiver below the condenser passage outlet.

NOTE 1 All weights are recorded to the nearest 0.01 g.

NOTE 2 To improve the ability to make accurate readings of the volume of NAF and water, it is possible to pre-wet the inside of the glass liquid receiver with PNP. PNP is the product used to break the emulsion during chemical titration of NADF (see Section 10). The basic method for wetting the glass liquid receiver is to add approximately 0.5 mL of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

**Caution—PNP should be used only when the precautions, appropriate PPE, and exposure controls are followed as described on the safety data sheet, as is recommended for all laboratory chemicals. This includes eye/face protection, skin protection, and respiratory protection for both liquid and/or vapor exposure.**

NOTE 3 Due to the rounded bottom of the liquid receiver, it might be helpful to place the liquid receiver in a 100 mL graduated cylinder to hold it on the top-loading balance while being weighed.

NOTE 4 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

- n) Turn on the heating jacket and allow the retort assembly to run a minimum of 1 h. Collect the condensate into the glass liquid receiver. If drilling fluid boils over into the liquid receiver, cool and clean the equipment and rerun the test using a larger amount of steel wool packed into the retort body.
- o) Remove the liquid receiver and allow it to cool.

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

NOTE If an emulsion interface is present between the NAF 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.

- p) Record the total condensed liquid volume (NAF and water),  $V_L$ , and water volume,  $V_W$ , collected in the liquid receiver. These are converted to mass, expressed in grams, as described in 9.6.

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-NAF meniscus, read the water volume at the lowest point.

- q) Weigh the cooled liquid receiver and its condensed liquid content (NAF and water). Record as  $m_4$ , expressed in grams.
- r) Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool. Remove the condenser and passage stem.
- s) Weigh the cooled retort assembly (i.e. retort sample cup with dried solids, lid, and retort body packed with steel wool). Record this as  $m_5$ , expressed in grams.
- t) Clean the retort assembly and condenser.

## 9.6 Gravimetric Method Calculation—Mass Balance

9.6.1 The mass balance factor of the retorted NADF, dry retorted solids, and condensed liquids shall be within  $\pm 3\%$  using Equation 19, or the procedure shall be repeated.

9.6.2 The mass of the whole-NADF sample before retorting shall be calculated using Equation (16).

$$m_{df} = m_2 - m_1 \quad (16)$$

where

$m_{df}$  is the mass of the whole-NADF sample, expressed in grams;

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

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

9.6.3 The mass of the dried solids after retorting shall be calculated using Equation (17)

$$m_{ds} = m_5 - m_1 \quad (17)$$

where

- $m_{ds}$  is the mass of the dried retort solids, expressed in grams;
- $m_5$  is the mass of the cooled retort assembly (cup, lid, dried solids, body packed with steel wool), expressed in grams;
- $m_1$  is the mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams.

**9.6.4** The mass of liquids [NAF and water] condensed after retorting shall be calculated using Equation (18).

$$m_L = m_4 - m_3 \quad (18)$$

where

- $m_L$  is the mass of the condensed liquids (NAF and water), expressed in grams;
- $m_4$  is the mass of the cooled liquid receiver with condensed liquids, expressed in grams;
- $m_3$  is the mass of the empty, dry liquid receiver, expressed in grams.

**9.6.5** The mass balance of the gravimetric retorting method shall be calculated using Equation (19).

The sum of the mass of dried retorted solids,  $m_{ds}$ , and condensed liquids,  $m_L$ , after retorting divided by the mass of the whole-NADF sample,  $m_{df}$ , shall be greater than 0.97 but less than 1.03.

$$0.97 \leq \frac{m_{ds} + m_L}{m_{df}} \leq 1.03 \quad (19)$$

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

**Note** A common cause of lost mass during retorting is the presence of organic additives, such as plant based lost circulation materials, in the sample. These organic products undergo an oxidation – reduction reaction at high temperature where mass is lost to the formation of carbon dioxide gas that is not condensed.

## 9.7 Gravimetric Method Calculation—Volume Fractions of NAF, Water, and Solids

### 9.7.1 General

The volume fractions of base NAF, water, and solids shall be calculated as per Equations (20) through (23), from the measured mass of retorted whole-NADF sample, mass of dried solids, density of the water, density of the NAF, and density of the NADF plus the measured volume of condensed water.

### 9.7.2 Volume fraction of base NAF

The mass of the NAF shall be calculated using Equation (20):

$$m_{NAF} = m_2 - (m_5 + V_W) \quad (20)$$

where

$m_{\text{NAF}}$	is the mass of the NAF, expressed in grams;
$m_2$	is the mass of the filled retort assembly (cup with sample, lid and body packed with steel wool), expressed in grams;
$m_5$	is the mass of the cooled retort assembly (cup, lid, dried solids, body packed with steel wool), expressed in grams;
$V_{\text{W}}$	is the condensed water volume expressed in milliliters or water mass expressed in grams (1 mL = 1 g) (see 3.3).

NOTE Assuming the density of water is 1 g/mL, the mass, in grams of the volume of water, is numerically equivalent to the volume of the water measured in milliliters.

The volume fraction of NAF in the whole-NADF sample, shall be calculated using Equation (21).

$$\phi_{\text{NAF}} = 100 \times \frac{V_{\text{NAF}}}{V_{\text{df}}} = 100 \times \frac{m_{\text{NAF}}}{\rho_{\text{NAF}}} \times \frac{\rho_{\text{df}}}{m_{\text{df}}} \quad (21)$$

where

$\phi_{\text{NAF}}$	is the volume fraction of NAF, expressed as a percentage of the whole-NADF;
$V_{\text{NAF}}$	is the volume of NAF, expressed in milliliters;
$V_{\text{df}}$	is the whole-NADF sample volume, expressed in milliliters;
$m_{\text{NAF}}$	is the mass of NAF, expressed in grams;
$\rho_{\text{NAF}}$	is the density of the NAF being used, expressed in grams per milliliter;
$m_{\text{df}}$	is the mass of the whole-NADF sample, expressed in grams;
$\rho_{\text{df}}$	is the drilling fluid (NADF) sample density, expressed in grams per milliliter.

NOTE Convert density in pounds per gallon to grams per milliliter by dividing the pounds per gallon by 8.345.

### 9.7.3 Volume fraction of water

The volume fraction of water in the whole-NADF sample, shall be calculated using Equation (22).

$$\phi_{\text{W}} = 100 \times \frac{V_{\text{W}}}{V_{\text{df}}} = 100 \times \frac{V_{\text{W}}}{(m_{\text{df}} / \rho_{\text{df}})} = 100 \times V_{\text{W}} \times \left( \frac{\rho_{\text{df}}}{m_{\text{df}}} \right) \quad (22)$$

where

$\phi_{\text{W}}$	is the volume fraction of water, expressed as a percentage of the whole-NADF;
$V_{\text{W}}$	is the condensed water volume expressed in milliliters or water mass expressed in grams (1 mL = 1 g) (see 3.3);

- $V_{df}$  is the whole-NADF sample volume, expressed in milliliters;
- $m_{df}$  is the mass of the whole-NADF sample, expressed in grams;
- $\rho_{df}$  is the drilling fluid (NAFD) density, expressed in grams per milliliter.

NOTE Convert density in pounds per gallon to grams per milliliter by dividing the pounds per gallon by 8.345.

#### 9.7.4 Volume fraction of dried solids

The volume fraction of dried solids remaining in the retort, in the whole-NADF sample, shall be calculated using Equation (23).

$$\varphi_{ds} = 100 - (\varphi_W + \varphi_{NAF}) \quad (23)$$

where

- $\varphi_{ds}$  is the volume fraction of dried retort solids, expressed as a percentage of the whole-NADF;
- $\varphi_W$  is the volume fraction of water, expressed as a percentage; of the whole-NADF;
- $\varphi_{NAF}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF.

NOTE The calculated solids will overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity shall be made using known volume factors for salt solution (see Section 12). Calculations for determining the NAF-to-water ratio and the correct solids concentration and density are in Section 12.

#### 9.7.5 Density of NAF

The density of the NAF may be calculated from the gravimetric retort mass measurements and the observed water volume using Equation (24).

$$\rho_{NAF} = \frac{(m_4 - m_3 - V_W)}{(V_L - V_W)} \quad (24)$$

where

- $\rho_{NAF}$  is the density of the NAF being used, expressed in grams per milliliter;
- $m_4$  is the mass of the cooled liquid receiver with condensed liquids, expressed in grams;
- $m_3$  is the mass of the empty, dry liquid receiver, expressed in grams;
- $V_L$  is the total volume of condensed liquid (NAF and water), expressed in milliliters;
- $V_W$  is the condensed water volume expressed in milliliters or water mass expressed in grams (1 mL = 1 g) (see 3.3).

NOTE Assuming the density of water is 1 g/mL, the mass, in grams, of water is numerically equivalent to the volume of the water measured in milliliters.

## 10 Chemical Analysis of NADF

### 10.1 Principle

**10.1.1** The NADF alkalinity test procedure is a titration method which measures the volume of standard acid required to react with the alkaline (basic) materials in a NADF sample. The alkalinity value is used to calculate the concentration of non-reacted “excess” lime in NADF. Excess alkaline materials, such as lime, help to stabilize the emulsion and neutralize acidic carbon dioxide or hydrogen sulfide gases. This test procedure can also determine if no “excess” lime is present, if an acidic condition exists in the drilling fluid, and how much lime would be required to bring the drilling fluid to a neutralized state.

**10.1.2** The whole-NADF chloride test procedure is a titration method which measures the volume of standard silver nitrate required to react with the chloride (and other halide) ions to form insoluble silver chloride (or silver halide) salts. The test procedure can utilize the same sample as the alkalinity test, provided the sample is acidic (pH below 7.0). The chloride value reported in the whole-NADF can be assigned to the water phase up to saturation point. The water-soluble salt concentration is related to the effectiveness of a NADF in controlling shale through the “aqueous-phase activity” concept. The aqueous-phase salinity value is also needed to adjust the retort water value to obtain a corrected solids concentration for the NADF.

**10.1.3** The whole-NADF calcium test is a titration method which measures the volume of a standard calcium-chelating agent [EDTA (ethylenediamine-tetraacetic acid) salt, see 10.2.9] required to react with all the calcium released from the NADF into the aqueous-phase, when a mixture of solvents is used to extract the NADF. The calcium measured in this test can come from the calcium chloride ( $\text{CaCl}_2$ ) and lime [ $\text{CaO}$  or  $\text{Ca}(\text{OH})_2$ ] used to prepare the NADF, as well as calcium from drilled gypsum or anhydrite ( $\text{CaSO}_4$ ).

**10.1.4** In addition, some normally insoluble calcium could be measured from drilled calcium carbonate or from calcium carbonate being used as a drilling fluid additive. The calcium analysis results can be used, along with the chloride and water concentration test, to calculate the  $\text{CaCl}_2$  and sodium chloride ( $\text{NaCl}$ ) salinity of the aqueous-phase of NADF.

**10.1.5** This calcium analysis excludes magnesium ( $\text{Mg}^{+2}$ ) ions. If  $\text{Mg}^{+2}$  is expected, special calcium and magnesium ion titration and calculation procedures should be arranged by the drilling fluid engineer and operator.

**10.1.6** Procedures for the calculations based on these chemical analyses are provided in Section 12.

**10.1.7** Included in these procedures is an optional filtration step to remove interfering color from the fluid sample. Filtration makes it easier to see endpoints for all titrations, including the calcium test even when specific insoluble colorants or screening solids are not present. However, be aware that the alkalinity may be lowered by filtration. Filtration is recommended in applications with dark filtrate or in drilling fluids using hematite.

**10.1.8 Sample Preparation and Analysis:** Care shall be taken when obtaining samples to ensure homogeneity. Settling or separation in the sample container may lead to erroneous test results, so stirring the container may be required. Other factors to consider include ensuring that test reagent solutions are within their shelf-life, elimination of cross-contamination from pipettes and contamination from not using distilled or deionized water. To avoid cross-contamination, pipettes shall never be used for more than one reagent solution. All reagent solutions should be tightly capped when not in use and properly discarded per vendor instructions when expired.

**10.1.9** The titration chemicals utilized for alkalinity determination will consume a small amount of alkalinity. This has been labeled “base alkalinity demand” (BAD),  $V_B$  (see 10.3). Once determined, the  $V_B$  shall be added

to the alkalinity measurement for the NADF to obtain a more accurate representation of the alkalinity/lime concentration.

## 10.2 Reagents and Apparatus

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

NOTE PNP degrades easily. It is advisable that only fresh chemical, within the expiration period noted, be used, and that proper disposal in accordance with local, state and federal regulations be followed. It is readily biodegradable.

**Caution— PNP should be used only when the precautions, appropriate PPE, and exposure controls are followed as described on the safety data sheet, as is recommended for all laboratory chemicals. This includes eye/face protection, skin protection, and respiratory protection for both liquid and/or vapor exposure.**

**10.2.2 Titration vessel**, 400 mL beaker, with cap.

**10.2.3 Phenolphthalein** (CAS No. 77-09-8) indicator solution, 1 g/100 mL in a 1:1 (v/v) isopropanol:water solution.

**10.2.4 Sulfuric acid** solution, 0.05 mol/L (0.1 N) (CAS No. 7664-93-9).

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

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

**10.2.6 Silver nitrate** reagent (CAS No. 7761-88-8),  $c_{\text{AgNO}_3} = 47.91 \text{ g/l}$  ( $c_{\text{Cl}^-} = 0.01 \text{ g/mL}$  or 0.282 mol/L—0.282 N), stored in an amber or opaque bottle.

**10.2.7 Buffer solution for calcium**,  $c_{\text{NaOH}} = 1 \text{ mol/L}$  (1 N) (CAS No. 1310-73-2) NaOH prepared from fresh “Certified ACS” grade sodium hydroxide, which contains less than 1 % mass fraction sodium carbonate.

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

**10.2.8 Calcium indicator**, hydroxynaphthol blue (CAS No. 63451-35-4).

**10.2.9 EDTA** solution (CAS No. 60-00-4),  $c_{\text{EDTA}} = 0.1 \text{ mol/L}$  (200 EPM), standardized disodium ethylenediamine-tetraacetate dihydrate (1 mL = 10,000 mg/L  $\text{CaCO}_3$ , 1 mL = 4000 mg/L  $\text{Ca}^{+2}$ ).

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

**10.2.10 Sodium hydroxide** solution (CAS No. 1310-73-2),  $c_{\text{NaOH}} = 0.1 \text{ mol/L}$  (0.1 N)

**10.2.11 Deionized or distilled water.**

**10.2.12 Disposable syringe**, 5 mL.

**10.2.13 Graduated cylinder** (TC), 25 mL.

**10.2.14 Graduated pipettes** TD, two each of 1 mL and 10 mL.

NOTE One pair of pipettes is for sulfuric acid and one pair is for silver nitrate.

#### 10.2.15 Pipette bulb or pump.

#### 10.2.16 Magnetic stirrer, with 4 cm (1.5 in.) stirring bar (coated).

### 10.3 Base Alkalinity Demand (BAD, $V_B$ )

#### 10.3.1 Procedure

The following procedure shall be used to determine the BAD,  $V_B$ , for the whole-NADF alkalinity test.

- a) Add 100 mL of PNP solvent to a 400 mL beaker or pint jar. See 10.2.1 for further information about PNP.
- b) Add 200 mL of deionized (or distilled) water.
- c) Add 15 drops of phenolphthalein indicator solution.
- d) While stirring with a magnetic stirrer such that the vortex is one-half of the liquid height, titrate immediately with 0.1 mol/L (0.1 N) sodium hydroxide until pink color just appears.

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

- e) Continue stirring for 5 min. If the pink color remains, the end point has been reached. If the pink color disappears, repeat item d).

Record the volume of 0.1 mol/L (0.1 N) NaOH, in milliliters, as  $V_{\text{NaOH,B}}$ , once an endpoint has been reached.

#### 10.3.2 Calculation

The BAD of the whole-NADF,  $V_B$ , expressed in milliliters, shall be calculated using Equation (25).

$$V_B = V_{\text{NaOH,B}} \quad (25)$$

where

$V_{\text{NaOH,B}}$  is the volume of 0.1 mol/L (0.1 N) NaOH, expressed in milliliters.

NOTE If  $V_B$  values are greater than 1 mL of 0.1 mol/l (0.1 N) sodium hydroxide, historical data indicates that the PNP might be contaminated or degraded. A new bottle of PNP should be implemented as soon as possible.

### 10.4 Whole-NADF Alkalinity ( $V_K$ )

#### 10.4.1 Procedure

In order to determine the whole-NADF alkalinity,  $V_K$ , the following procedure shall be used.

- a) Add 100 mL of PNP solvent to the titration vessel. See 10.2.1 for further information about PNP.

- b) Fill the 5 mL syringe with whole-NADF beyond the 3 mL-mark.
- c) Displace 2.0 mL of whole-NADF into the titration vessel. Record as  $V_{df}$ .
- d) Stir or swirl the NADF and solvent until the mixture is homogeneous.
- e) Add 200 mL of distilled (or deionized) water. If the filtration option is not used, skip to 10.4.1 item g).
- f) Filtration option:

The filtration option is recommended to be used for all fluids which have a dark filtrate or where the end-point color change of the indicator during titration is difficult to see.

- 1) Stir the mixture for 5 min with a magnetic stirrer such that the vortex is one-half of the liquid height.
- 2) Pour the mixture into an API low-temperature/low-pressure filter press fitted with filter paper (see API 13B-1).
- 3) Nitrogen shall be used as the source for gas pressure for this filtration step. Close the cell and apply a pressure of 690 kPa (100 lbf/in.<sup>2</sup>). Do not use carbon dioxide cartridges or bottles as the pressure source.

**Warning—Do not use nitrous oxide cartridges as pressure sources for NADF filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for water-based drilling fluid Garrett gas train carbonate analysis (see API 13B-1).**

- 4) Collect all the filtrate in a clean beaker until the cell blows dry.
  - 5) Use all the filtrate and continue with 10.4.1 item g)
- g) Add 15 drops of phenolphthalein indicator solution. If there is no color change to pink, skip to step 10.4.1 item k) and use  $V_{H_2SO_4} = 0$  in Equation (26).
  - h) While stirring with a magnetic stirrer such that a vortex is one-half of the liquid height, titrate immediately with 0.05 mol/L (0.1 N) sulfuric acid until the pink color just disappears.

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

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

- i) Continue stirring for 5 min. If no pink color reappears, the end point has been reached. If a pink color returns, return to item h). Record the volume of 0.05 mol/L (0.1 N) sulfuric acid as  $V_{H_2SO_4}$ , in milliliters, once an endpoint has been reached.
- j) The volume of 0.05 mol/L (0.1 N) sulfuric acid solution,  $V_{H_2SO_4}$ , expressed in milliliters, required to reach the end point in item i) shall be used to calculate the whole-NADF alkalinity,  $V_K$ , using Equation (26) and the whole-NADF lime concentration using Equation (29) or (30), see 12.3.1.

- k) For the case of no color change when phenolphthalein is added in item g) while stirring with a magnetic stirrer such that a vortex is one-half the liquid height, titrate immediately with 0.1 mol/L (0.1 N) sodium hydroxide (used in the BAD titration, see 10.2.10) until a pink color just appears.

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

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

- l) Continue stirring for 5 min. If the pink color remains, the end point has been reached. If a pink color disappears, return to item k). Record the volume of 0.1 mol/L (0.1 N) sodium hydroxide ( $V_{\text{NaOH}}$ ), in milliliters once an endpoint has been reached.
- m) Use the volume of 0.1 mol/L (0.1 N) sodium hydroxide ( $V_{\text{NaOH}}$ ), expressed in milliliters, required to reach the end point in 10.4.1 item l) to calculate the whole-NADF alkalinity, ( $V_{\text{K}}$ ). If  $V_{\text{K}}$  is a negative value, this indicates that all of the lime in the drilling fluid has been neutralized by the PNP's acidity and/or by acidic components in the drilling fluid originating from products added and/or acid gases from the formations being drilled, and excess acidity has been being detected. If negative,  $V_{\text{K}}$  is a measurement of the whole-NADF acidity. Negative,  $V_{\text{K}}$  can be used to calculate the amount of lime required to neutralize the excess acidity using Equations (29) to (32) (12.3.1).

Add if color difficult for back and filter xxx

#### 10.4.2 Calculation

The whole-NADF alkalinity,  $V_{\text{K}}$ , shall be calculated using Equation (26).

$$V_{\text{K}} = \frac{V_{\text{H}_2\text{SO}_4} - V_{\text{NaOH}} + V_{\text{B}}}{V_{\text{df}}} \quad (26)$$

where

$V_{\text{K}}$  if positive, is the whole-NADF alkalinity, expressed in milliliters of 0.05 mol/L (0.1 N) sulfuric acid;

if negative, is the whole-NADF acidity, expressed in milliliters of 0.1 mol/L (0.1 N) sodium hydroxide;

$V_{\text{B}}$  is the base alkalinity demand (BAD) volume of 0.1 mol/L (0.1 N) NaOH, expressed in milliliters;

$V_{\text{H}_2\text{SO}_4}$  is the volume of 0.05 mol/L (0.1 N) sulfuric acid, expressed in milliliters;

$V_{\text{NaOH}}$  is the volume of 0.1 mol/L (0.1 N) sodium hydroxide, expressed in milliliters;

$V_{\text{df}}$  is the whole-NADF sample volume, expressed in milliliters;

NOTE  $V_K$ , if positive, is the equivalent to  $P_{OM}$  used in the previous editions of this standard.  $V_B$  is the volume of 0.1 mol/L (0.1 N) sodium hydroxide solution required to reach a phenolphthalein endpoint in the titration fluid mixture without any drilling fluid.

## 10.5 Whole-NADF Chloride Concentration

### 10.5.1 Procedure

In order to determine the whole-NADF chloride concentration, the following procedure shall be used.

- a) Perform the alkalinity procedures in 10.3 through 10.4. Use this NADF for the chloride concentration analysis.

Make certain the mixture to be titrated for chloride is acidic (below pH 7.0) by adding 10 drops to 20 drops, or more, of 0.05 mol/L (0.1 N) sulfuric acid.

- b) Add 3 mL of potassium chromate indicator solution (10.2.5). If the filtration option is not used, skip to 10.5.1 item d).
- c) Filtration option:

The filtration option is recommended to be used for all fluids which have a dark filtrate or where the end-point color change of the indicator during titration is difficult to see.

- 1) Stir the mixture for 5 min with a magnetic stirrer such that the vortex is one-half of the liquid height.
- 2) Pour the mixture into an API low-temperature/low-pressure filter press fitted with filter paper (see API 13B-1).
- 3) Nitrogen shall be used as the source for gas pressure for this filtration step. Close the cell and apply a pressure of 690 kPa (100 lbf/in.<sup>2</sup>). Do not use carbon dioxide cartridges or bottles as the pressure source.

**WARNING—Do not use nitrous oxide cartridges as pressure sources for NADF filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for water-based drilling fluid Garrett gas train carbonate analysis (see API 13B-1).**

- 4) Collect all the filtrate in a clean beaker until the cell blows dry.
- 5) Use all the filtrate and continue with item d).

NOTE Additional potassium chromate indicator solution may be required after the titration option and prior to item d).

- d) Titrate slowly with 0.282 mol/L (0.282 N) silver nitrate reagent (10.2.6) while stirring rapidly with a magnetic stirrer, until a salmon-pink color remains stable for at least 1 min. It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to see the color in the aqueous-phase more clearly. Record the volume of silver nitrate reagent,  $V_{AgNO_3}$ , to reach the end-point of the titration.

- e) Use the volume of 0.282 mol/L (0.282 N) silver nitrate reagent required,  $V_{\text{AgNO}_3}$ , in milliliters, to reach the end point item d), to calculate the whole-NADF chloride concentration,  $c_{\text{Cl,df}}$ .

### 10.5.2 Calculation

The volume of silver nitrate reagent required,  $V_{\text{AgNO}_3}$ , to reach the end point in 10.5.1 item d) , shall be used to calculate the whole-NADF chloride concentration,  $c_{\text{Cl,df}}$  using Equation (27).

$$c_{\text{Cl,df}} = \frac{10,000 \times V_{\text{AgNO}_3}}{V_{\text{df}}} \quad (27)$$

where

$c_{\text{Cl,df}}$  is the whole-NADF chloride concentration, in milligrams per liter;

$V_{\text{AgNO}_3}$  is the volume of 0.282 mol/L (0.282 N) silver nitrate reagent, in milliliters;

$V_{\text{df}}$  is the whole-NADF sample volume, in milliliters.

## 10.6 Whole-NADF Calcium Concentration

### 10.6.1 Procedure

In order to determine the whole-NADF calcium concentration, the following procedure shall be used.

- a) Add 100 mL of the PNP solvent to a titration vessel that has a cap.
- b) Fill the 5 mL syringe beyond the 3 mL mark with whole-NADF.
- c) Displace 2.0 mL of whole-NADF into the titration vessel. Record as  $V_{\text{df}}$ .
- d) Stir or swirl the NADF and solvent until the mixture is homogeneous.
- e) Add 200 mL deionized or distilled water to the titration vessel. If the filtration option is not used, skip to 10.6.1 item g).
- f) Filtration option:

The filtration option is recommended to be used for all fluids which have a dark filtrate or where the end-point color change of the indicator during titration is difficult to see.

- 1) Stir the mixture for 5 min with a magnetic stirrer such that the vortex is on-half of the liquid height.
- 2) Pour the mixture into an API low-temperature/low-pressure filter press fitted with filter paper (see API 13B-1).
- 3) Nitrogen shall be used as the source for gas pressure for this filtration step. Close the cell and apply a pressure of 690 kPa (100 lbf/in<sup>2</sup>). Do not use carbon dioxide cartridges or bottles as the pressure source.

**WARNING—Do not use nitrous oxide cartridges as pressure sources for NADF filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for water-based drilling fluid Garrett gas train carbonate analysis (see API 13B-1).**

- 4) Collect all the filtrate in a clean beaker until the cell blows dry.
- 5) Use all the filtrate and continue with item g).
- g) Add 6.0 mL (1 mol/L (1N) sodium hydroxide) buffer solution for calcium (10.2.13).
- h) Add 0.7 g to 1.0 g hydroxynaphthol blue calcium indicator powder (10.2.8).
- i) Place the titration vessel on the magnetic stirrer and drop in a stirring bar.
- j) Titrate very slowly, adding 0.1 mol/L (200 EPM) EDTA solution, drop by drop, from the pipette while stirring only fast enough to agitate the aqueous (lower) phase without re-mixing the upper and lower phases. A distinct color change from the reddish color to a blue-green color occurs at the end point. Record the volume of EDTA added,  $V_{\text{EDTA}}$ , in milliliters.

### 10.6.2 Calculation

The volume of EDTA required,  $V_{\text{EDTA}}$ , to reach the end point in 10.6.1 item j) , shall be used to calculate the whole-NADF calcium concentration,  $c_{\text{Ca,df}}$ , using Equation (28),

$$c_{\text{Ca,df}} = \frac{4000 \times V_{\text{EDTA}}}{V_{\text{df}}} \quad (28)$$

where

$c_{\text{Ca,df}}$  is the whole-NADF calcium concentration, in milligrams per liter;

$V_{\text{EDTA}}$  is the volume of 0.1 mol/L (200 EPM) EDTA solution, in milliliters;

$V_{\text{df}}$  is the whole-NADF sample volume, in milliliters.

## 11 Electrical Stability Test

### 11.1 Principle

**11.1.1** The electrical stability (ES) of a NADF is a property related to its emulsion stability and NAF-wetting capability. ES is determined by applying a voltage-ramped, sinusoidal electrical signal across a pair of parallel, flat-plate electrodes immersed in the drilling fluid. The resulting current remains low until a threshold voltage is reached, whereupon the current rises very rapidly. This threshold voltage is referred to as the ES of the NADF and is defined as the voltage (in peak volts) measured when the current reaches 61  $\mu\text{A}$ .

**NOTE** The sine wave specified for the voltage ramp results in more efficient energizing of the fluid and generates considerably lower ES values (often less than half) than the spiky waveform of the instrument described in previous editions of API 13B and API 13B-2. The symmetry of the sinusoidal signal also inhibits build-up of solids on the electrode faces and enhances reproducibility. To further enhance reproducibility, the instrument specifications include automatic voltage ramping at the fixed ramp rate.

**11.1.2** Chemical composition and shear history (as well as temperature) of a drilling fluid control the absolute magnitude of ES in a complex fashion. Consequently, interpreting the condition of a NADF from single ES measurements may not be appropriate. Trends in ES values are recommended to be used in making condition and treatment decisions.

## **11.2 Apparatus**

**11.2.1 Electrical stability meter**, with the following characteristics:

Waveform	sine, < 5 % total harmonic distortion
A.C. frequency	340 Hz $\pm$ 10 Hz
Output unit	peak volt
Ramp rate	150 V/s $\pm$ 10 V/s, automatic operation
Range	3 V to 2000 V (peak)
Trip current	61 $\mu$ A $\pm$ 5 $\mu$ A (peak)

**11.2.2 Electrodes**, with the following characteristics:

Housing	material resistant to NAF components up to 105 °C (220 °F)
Material	corrosion-resistant metal
Diameter	3.18 mm $\pm$ 0.03 mm (0.125 in. $\pm$ 0.001 in.)
Spacing (gap)	1.55 mm $\pm$ 0.03 mm (0.061 in. $\pm$ 0.001 in.) at 22 °C (72 °F)

**11.2.3 Calibration resistors/diodes**, with the following characteristics:

Number	two (low and high)
Types	standard resistor or Zener diodes
Range	voltage (ES) readings of: low: 500 V to 1000 V high: >1900 V
Accuracy	2 % of expected voltage, temperature corrected by manufacturer's table (if needed and provided)

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

**11.2.4 Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) accurate to  $\pm$ 1 °C ( $\pm$ 2 °F).

**11.2.5 Marsh funnel.**

**11.2.6 Thermostatically controlled viscometer cup.**

**11.2.7 Isopropyl alcohol** (CAS No. 67-63-0).

### 11.3 Equipment Calibration/Performance Test

Electrical stability instrument shall be calibrated, and performance tested as per the following.

- a) Inspect the electrode probe and cable for evidence of damage.
- b) Ensure that the entire electrode gap is free of deposits, and that the connector to the instrument is clean and dry.
- c) Disconnect the electrode probe (if possible) and run a voltage ramp test, following instructions in the ES meter operating manual. If the meter is working properly, the ES reading shall reach the maximum voltage permitted by the instrument.
- d) Reconnect the electrode probe to the ES meter and repeat the voltage ramp test in air. Again, the ES reading shall reach the maximum permitted voltage. If not, the electrode probe and connector may need to be cleaned or replaced.
- e) Repeat the voltage ramp test with the electrode probe in tap water. The ES reading shall not exceed 3 V. If ES does exceed 3 V, either clean the electrode probe or replace it.
- f) Check the accuracy of the ES meter with the standard resistors or Zener diodes (see the NOTE to 11.2.3). The ES readings shall fall within 2.5 % (combined uncertainty of meter and resistor/diodes) of the expected values. If any of the ES readings fall outside this range, the instrument should be returned to the supplier for adjustment or repair.

NOTE The procedure described in this subsection (11.3) does not check waveform, A.C. frequency or ramp rate. A periodic check of the ES meter and electrode probe by a qualified technician ensures that both units meet all specifications.

### 11.4 Procedure

In order to determine the ES of a NADF, the following procedure shall be used.

- a) Verify equipment calibration/performance (see 11.3).
- b) Screen the drilling fluid sample through a Marsh funnel prior to testing (see 7.2).

NOTE ES values are affected by shear. Test fluids shall not have been recently subjected to high shear in a mixer.

- c) ES shall be tested on a NADF sample after measuring the viscosity and gel strengths using a direct-indicating viscometer at either a temperature of  $50\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  ( $120\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ) or  $65\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  ( $150\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ) as described in 7.3. After the last viscometer gel strength measurement, operate the viscometer for 30 s at 600 rpm, then remove the heat cup from the viscometer. Measure and record the drilling fluid temperature.

NOTE ES values are affected by temperature. For high temperature wells where rheology is measured at higher temperatures, such as  $65\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  ( $150\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ), ES is often measured at the higher temperature as well. ES measurements made at higher temperatures cannot be directly compared to ES measurements made at  $50\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  ( $120\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ) or lower temperatures.

- d) Clean the electrode probe body thoroughly by wiping with a clean paper towel. Pass the towel through the electrode gap a few times. Swirl the electrode probe in the NAF used to formulate the drilling fluid. If the base NAF is not available, a different NAF or a mild solvent, such as isopropyl alcohol, is acceptable. Clean and dry the electrode probe as before.

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

- e) Hand-stir the 50 °C (120 °F) or 65 °C (150 °F) sample with the electrode probe for approximately 10 seconds to ensure that the composition and temperature of the NADF are uniform. Position the electrode probe so that it does not touch the bottom or sides of the container and be certain that the electrode surfaces are completely covered by the sample.

NOTE A stand to secure the probe will assist in maintaining the probe off the bottom and away from the sides.

- f) Begin the voltage ramp test. Follow the procedure described in the ES meter operating manual. Do not move the electrode probe during the voltage ramp test.
- g) At the conclusion of the ramp test, note the ES value displayed on the readout device.
- h) Repeat the procedure with the same drilling fluid sample. The two ES values shall not differ by more than 5 %. If they differ by more than 5 %, check the meter or electrode probe for malfunction.
- i) Record the average of the two ES measurements, expressed in volts.

## 12 Lime, Salinity, and Solids Calculations

### 12.1 Principle

**12.1.1** Lime, salinity, and solids calculations, including a direct binary salt calculation method that does not require iterations. The calculations require the use of a calculator or computer spreadsheet. Equations in this procedure allow calculations of the concentration of the following components for a NADF :

- a) lime, expressed as  $\text{Ca}(\text{OH})_2$ , in the whole-NADF
- b)  $\text{CaCl}_2$  and  $\text{NaCl}$  in the whole-NADF;
- c)  $\text{CaCl}_2$  and  $\text{NaCl}$  soluble in the aqueous-phase and insoluble in the whole-NADF;
- d) low-gravity solids (insoluble salts and drilled solids);
- e) weighting material.

**12.1.2** Data listed below and used in the equations are determined from NADF tests specified in Section 5, Section 9, and Section 10:

- a) drilling fluid density [see Section 5 or Section 6 (preferred)];
- b) whole-NADF alkalinity,  $V_K$  (see 10.4);
- c) whole-NADF calcium concentration,  $c_{\text{Ca,df}}$  (see 10.6);
- d) whole-NADF chloride concentration,  $c_{\text{Cl,df}}$  (see 10.5);
- e) volume of retort NAF,  $V_{\text{NAF}}$ , and volume of retort water,  $V_W$  (see Section 9).

**12.1.3** Data known or assumed are as follows:

- a) density of weighting material (in grams per milliliter),  $\rho_{WM}$ ;
- b) density of low-gravity solids (in grams per milliliter),  $\rho_{LG}$ ;
- c) density of the base NAF (in grams per milliliter),  $\rho_{NAF}$ .

NOTE Base NAF density should be measured or calculated, see Section 9.

**12.1.4** These calculations do not utilize a “lime correction” for adjusting the whole-NADF calcium concentration as determined by the EDTA titration.

**12.1.5** Be certain to use the units requested in the equations. Units for all input data and results for each equation are indicated.

**12.1.6** Examples of calculations can be found in Annex E.

## 12.2 Apparatus

A calculator or computer spreadsheet should be used to perform the mathematical operations detailed in this procedure.

## 12.3 Whole-NADF Calculations

### 12.3.1 Whole-NADF lime concentration

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

The whole-NADF lime concentration,  $c_{Ca(OH)_2,df}$  shall be calculated using Equation (29) in SI units or Equation (30) in USC units

$$c_{Ca(OH)_2,df,SI} = 3.705 \times V_K \quad (29)$$

$$c_{Ca(OH)_2,df,USC} = 1.299 \times V_K \quad (30)$$

and whole-NADF lime active concentration,  $c_{Ca(OH)_2,Act}$ , shall be calculated using Equation (31) in SI units or Equation (32) in USC units.

$$c_{Ca(OH)_2,Act,SI} = \frac{100 \times c_{Ca(OH)_2,df,SI}}{w_{Ca(OH)_2,\%}} \quad (31)$$

$$c_{\text{Ca(OH)}_2, \text{Act, USC}} = \frac{100 \times c_{\text{Ca(OH)}_2, \text{df, USC}}}{w_{\text{Ca(OH)}_2, \%}} \quad (32)$$

where

- $c_{\text{Ca(OH)}_2, \text{df, SI}}$  is the whole-NADF total lime concentration, expressed in kilograms per cubic meter.
- $c_{\text{Ca(OH)}_2, \text{df, USC}}$  is the whole-NADF total lime concentration, expressed in pounds per barrel;
- $V_K$  is the whole-NADF alkalinity, expressed in milliliters of 0.05 mol/L (0.1 N) sulfuric acid;
- $c_{\text{Ca(OH)}_2, \text{Act, SI}}$  is the lime active concentration of the field lime in the whole-NADF, expressed in kilograms per cubic meter;
- $c_{\text{Ca(OH)}_2, \text{Act, USC}}$  is the lime active concentration of the field lime in the whole-NADF, expressed in pounds per barrel;
- $w_{\text{Ca(OH)}_2, \%}$  is the lime assay value (activity or purity), expressed as a weight-percent.

NOTE  $c_{\text{Ca(OH)}_2, \text{df}}$  is normally referred to as “excess lime”.

### 12.3.2 Whole-NADF calcium chloride and sodium chloride concentrations

**12.3.2.1** A NADF normally contains calcium chloride ( $\text{CaCl}_2$ ), or sodium chloride ( $\text{NaCl}$ ) or both calcium and sodium chloride. Use the chemical analysis results and the methods below to determine if  $\text{NaCl}$  and  $\text{CaCl}_2$  are present and the concentration of each. For a fluid with a super-saturated aqueous-phase, some salt will be present as an insoluble solid.

**12.3.2.2** The whole-NADF chloride ion concentration expressed as total chloride from  $\text{CaCl}_2$ , based on the calcium analysis (see 10.6), calculated in milligrams per liter, kilograms per cubic meter or pounds per barrel, shall be calculated using Equation (33):

$$c_{\text{Cl-(CaCl}_2), \text{df}} = 1.769 \times c_{\text{Ca, df}} \quad (33)$$

where

- $c_{\text{Cl-(CaCl}_2), \text{df}}$  is the whole-NADF total chloride concentration from  $\text{CaCl}_2$ , expressed in milligrams per liter;
- $c_{\text{Ca, df}}$  is the calcium concentration of the whole-NADF, expressed in milligrams per liter;

NOTE Equations shall be expressed in consistent units.

**12.3.2.3** To determine if sodium chloride is present, the whole-NADF chloride ion concentration,  $c_{Cl,df}$  determined from the chloride ion analysis Equation (27), shall be compared to the whole-NADF chloride ion concentration,  $c_{Cl-(CaCl_2),df}$  determined from calcium ion analysis, Equation (33).

If  $c_{Cl,df}$  is greater than  $c_{Cl-(CaCl_2),df}$  excess chloride ions are indicated, and sodium chloride is present. When sodium chloride is present, the whole-NADF chloride ion concentration from NaCl shall be calculated using Equation (34).

$$c_{Cl-(NaCl),df} = (c_{Cl,df}) - (c_{Cl-(CaCl_2),df}) \quad (34)$$

where

$c_{Cl-(NaCl),df}$  is the whole-NADF chloride ion concentration from NaCl, expressed in milligrams per liter;

$c_{Cl,df}$  is the whole-NADF chloride concentration, expressed in milligrams per liter;

$c_{Cl-(CaCl_2),df}$  is the whole-NADF chloride concentration from calcium chloride, expressed in milligrams per liter.

**Instructions:**

If  $c_{Cl-(NaCl),df}$  is a positive value, then NaCl is present in the drilling fluid, and skip to 12.3.2.5 Equation (38).

If  $c_{Cl-(NaCl),df}$  is a negative value, then only CaCl<sub>2</sub> is present in the drilling fluid and no NaCl is present.

For fluids with no sodium chloride, skip to 12.4.

**12.3.2.4** When no sodium chloride is present, calculating the calcium chloride content using the whole-NADF chloride value [see 10.5.2, Equation (27)] analysis is more reliable.

The whole-NADF CaCl<sub>2</sub> concentration based on the chloride ion value shall be calculated using Equations (35), (36), or (37):

$$c_{CaCl_2,df} = 1.565 \times c_{Cl,df} \quad (35)$$

$$c_{CaCl_2,df,SI} = 1.565 \times 10^{-3} \times c_{Cl,df} \quad (36)$$

$$c_{CaCl_2,df,USC} = 0.5485 \times 10^{-3} \times c_{Cl,df} \quad (37)$$

where

$c_{Cl,df}$  is the whole-NADF chloride concentration, expressed in milligrams per liter;

$c_{CaCl_2,df}$  is the whole-NADF calcium chloride concentration, expressed in milligrams per liter;

$c_{CaCl_2,df,SI}$  is the whole-NADF calcium chloride concentration, expressed in kilograms per cubic meter.

$c_{\text{CaCl}_2,\text{df,USC}}$  is the whole-NADF calcium chloride concentration, expressed in pounds per barrel;

**12.3.2.5** For fluids with both sodium chloride and calcium chloride, the whole-NADF  $\text{CaCl}_2$  concentration shall be calculated from the whole-NADF calcium ion analysis [see 10.6.2, Equation (28)] using Equations (38), (39), or (40).

$$c_{\text{CaCl}_2,\text{df}} = 2.769 \times c_{\text{Ca,df}} \quad (38)$$

$$c_{\text{CaCl}_2,\text{df,SI}} = 2.769 \times 10^{-3} \times c_{\text{Ca,df}} \quad (39)$$

$$c_{\text{CaCl}_2,\text{df,USC}} = 0.9707 \times 10^{-3} \times c_{\text{Ca,df}} \quad (40)$$

where

$c_{\text{CaCl}_2,\text{df}}$  is the whole-NADF  $\text{CaCl}_2$  concentration, expressed in milligrams per liter;

$c_{\text{Ca,df}}$  is the whole-NADF calcium concentration, expressed in milligrams per liter;

$c_{\text{CaCl}_2,\text{df,SI}}$  is the whole-NADF  $\text{CaCl}_2$  concentration, expressed in kilograms per cubic meter;

$c_{\text{CaCl}_2,\text{df,USC}}$  is the whole-NADF  $\text{CaCl}_2$  concentration, expressed in pounds per barrel.

**12.3.2.6** For fluids with both sodium chloride and calcium chloride, the whole-NADF  $\text{NaCl}$  concentration shall be calculated from the chloride ion content from  $\text{NaCl}$  [12.3.2.3, Equation (34)] using Equations (41), (42), or (43).

$$c_{\text{NaCl,df}} = 1.648 \times c_{\text{Cl-(NaCl),df}} \quad (41)$$

$$c_{\text{NaCl,df,SI}} = 1.648 \times 10^{-3} \times c_{\text{Cl-(NaCl),df}} \quad (42)$$

$$c_{\text{NaCl,df,USC}} = 0.578 \times 10^{-3} \times c_{\text{Cl-(NaCl),df}} \quad (43)$$

where

$c_{\text{NaCl,df}}$  is the whole-NADF sodium chloride concentration, in milligrams per liter;

$c_{\text{Cl-(NaCl),df}}$  is the whole-NADF chloride concentration from sodium chloride, in milligrams per liter;

$c_{\text{NaCl,df,SI}}$  is the whole-NADF sodium chloride concentration, in kilograms per cubic meter;

$c_{\text{NaCl,df,USC}}$  is the whole-NADF sodium chloride concentration, in pounds per barrel.

## 12.4 Aqueous-phase Calculations

### 12.4.1 General

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

Aqueous-phase calculations consider 3 cases:

- a) both sodium and calcium chloride present and soluble;
- b) both sodium and calcium chloride present, some sodium chloride insoluble (super-saturated);
- c) only calcium chloride present, no sodium chloride.

#### 12.4.2 Both sodium and calcium chloride present and soluble

If all salts are soluble, mass fractions of  $\text{CaCl}_2$  and  $\text{NaCl}$  in the aqueous-phase of a NADF shall be calculated respectively using Equations (44) and (45).

- a) Mass fraction of  $\text{CaCl}_2$  in the aqueous-phase:

$$w_{\text{CaCl}_2} = \frac{100 \times c_{\text{CaCl}_2, \text{df}}}{\left[ c_{\text{CaCl}_2, \text{df}} \right] + \left[ c_{\text{NaCl}, \text{df}} \right] + (10,000 \times \varphi_{\text{W}})} \quad (44)$$

where

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

$c_{\text{CaCl}_2, \text{df}}$  is the whole-NADF calcium chloride, expressed in milligrams per liter;

$c_{\text{NaCl}, \text{df}}$  is the whole-NADF sodium chloride concentration, expressed in milligrams per liter;

$\varphi_{\text{W}}$  is the volume fraction of water, expressed as a percentage.

**Instruction:** For fluids with no sodium chloride, skip to 12.4.5.

- b) Mass fraction of  $\text{NaCl}$  in the aqueous-phase:

$$w_{\text{NaCl}} = \frac{100 \times c_{\text{NaCl}, \text{df}}}{\left[ c_{\text{CaCl}_2, \text{df}} \right] + \left[ c_{\text{NaCl}, \text{df}} \right] + (10,000 \varphi_{\text{W}})} \quad (45)$$

where

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

$c_{\text{CaCl}_2, \text{df}}$  is the whole-NADF calcium chloride concentration, expressed in milligrams per liter;

$c_{\text{NaCl}, \text{df}}$  is the whole-NADF sodium chloride concentration, expressed in milligrams per liter;

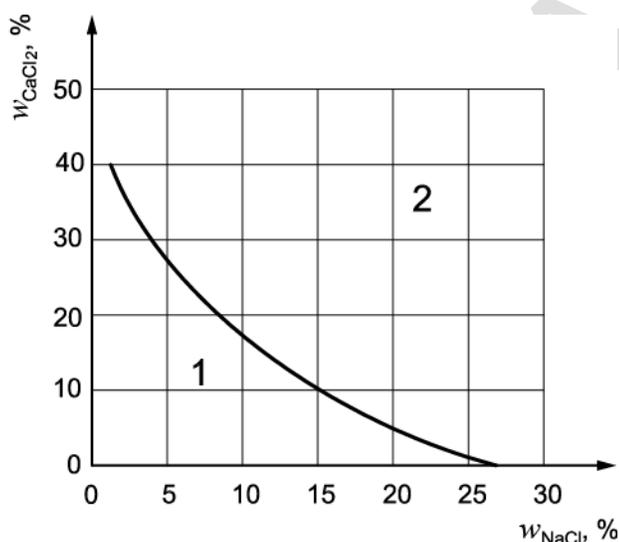
$\phi_W$  is the volume fraction of water, expressed as a percentage.

### 12.4.3 Super-saturated aqueous phase – Both sodium and calcium chloride present with some sodium chloride insoluble

#### 12.4.3.1 Maximum sodium chloride soluble mass fraction with calcium chloride present

The maximum mass fraction of soluble NaCl that can exist for the mass fraction of CaCl<sub>2</sub> present in the aqueous-phase at 25 °C (77 °F) can be determined from empirical mutual solubility data using Equation (46) (see Reference [20]).

If the value from Equation (45) is greater than the maximum soluble value from Equation (46), the aqueous-phase is super-saturated and some of the sodium chloride exists as an insoluble solid (see Figure 1, Region 2). If this is the case, the aqueous-phase mass fractions calculated in Equation (44) and Equation (45) are incorrect because all the NaCl is not in solution.



#### Key

- 1 region in which all NaCl is soluble
- 2 region in which some NaCl is insoluble

**Figure 1—Maximum Concentrations of NaCl in CaCl<sub>2</sub> Brine at 25 °C (77 °F)**

**12.4.3.2** The maximum mass fraction of soluble NaCl that can exist in the aqueous-phase shall be calculated using Equation (46).

$$w_{\text{NaCl,Max}} = 26.432 - \left[ 1.0472 \left( w_{\text{CaCl}_2} \right) \right] + \left[ 7.98191 \times 10^{-3} \left( w_{\text{CaCl}_2} \right)^2 \right] + \left[ 5.2238 \times 10^{-5} \left( w_{\text{CaCl}_2} \right)^3 \right] \quad (46)$$

where

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

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

**12.4.3.3** If  $w_{NaCl}$  from Equation (45) is less than  $w_{NaCl,Max}$  from Equation (46), all the NaCl is soluble in the aqueous-phase (Figure 1, Region 1).

**Instruction:**

If  $w_{NaCl}$  is less than  $w_{NaCl,Max}$ , skip to 12.4.5 and use  $w_{CaCl_2}$  from Equation (44) and  $w_{NaCl}$  from Equation (45) for the remaining calculations.

**12.4.3.4** If  $w_{NaCl}$  is greater than  $w_{NaCl,Max}$ , the aqueous-phase is super-saturated and insoluble NaCl is present (Figure 1, Region 2).

**Instruction:**

If  $w_{NaCl}$  is greater than  $w_{NaCl,Max}$ , the mass fraction of both salts,  $w_{CaCl_2}$  from Equation (44) and  $w_{NaCl}$  from Equation (45) is not correct and shall be recalculated, continue with 12.4.3.5.

**12.4.3.5** For super-saturated fluids, the correct mass fraction of CaCl<sub>2</sub>,  $w_{CaCl_2,Sat}$ , can be determined from empirical mutual solubility data (see Reference [20]) using the calcium concentration expressed as the mass of calcium per volume of pure water in the aqueous-phase,  $c_{Ca,Aq}$ , determined from the whole-NADF calcium ion concentration and the retort analysis.

Calcium concentration expressed per volume of pure water in the aqueous-phase shall be calculated using Equation (47).

$$c_{Ca,Aq} = \frac{100 \times c_{Ca,df}}{\phi_W} \quad (47)$$

where

$c_{Ca,Aq}$  is the aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per liter;

$c_{Ca,df}$  is the whole-NADF calcium concentration, expressed in milligrams per liter;

$\phi_W$  is the volume fraction of water, expressed as a percentage.

**12.4.3.6** For super-saturated fluids, the actual mass fraction of calcium chloride,  $w_{CaCl_2}$ , in the aqueous-phase shall be calculated using Equation (48).

$$w_{CaCl_2,Sat} = \left[ (c_{Ca,Aq})^2 \times (-2.4708 \times 10^{-10}) \right] + \left[ (c_{Ca,Aq}) \times (2.2567 \times 10^{-4}) \right] - [0.21348] \quad (48)$$

where

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

$c_{\text{Ca, Aq}}$  is the aqueous-phase calcium concentration per volume of pure water, expressed in milligrams per liter.

Use this actual  $w_{\text{CaCl}_2, \text{Sat}}$  for  $w_{\text{CaCl}_2}$  in the remaining calculations for fluids with super-saturated aqueous-phases.

NOTE Insoluble (undissolved) NaCl has a lower specific gravity than most drill solids. If it is present in the NADF, solids calculations will be in error. No technique is provided in this procedure to compensate for that type of error.

**12.4.3.7** Now that the actual mass fraction of calcium chloride is known for a super-saturated aqueous-phase, the correct mass fraction of soluble NaCl in the aqueous-phase is  $w_{\text{NaCl, Max-CaI}}$  and shall be recalculated from Equation (46) using  $w_{\text{CaCl}_2, \text{Sat}}$  for  $w_{\text{CaCl}_2}$ .

Use this recalculated  $w_{\text{NaCl, Max-CaI}}$  for  $w_{\text{NaCl}}$  in the remaining calculations for fluids with a super-saturated aqueous-phase.

**12.4.3.8** Now that the correct soluble mass fractions of calcium chloride,  $w_{\text{CaCl}_2}$ , and sodium chloride,  $w_{\text{NaCl}}$ , are known, the total soluble water phase salinity ( $WPS$ ), expressed in milligrams per kilogram (identical to ppm) shall be calculated using Equation (49).

$$WPS = (w_{\text{NaCl}} + w_{\text{CaCl}_2}) \times 10,000 \quad (49)$$

where

$WPS$  is the total soluble water phase salinity, expressed in mg/kg (same as ppm);

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

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

#### 12.4.5 Aqueous-phase NADF density

The density of the NADF aqueous-phase (brine),  $\rho_B$ , in grams per milliliter, shall be calculated using Equation (50).

$$\begin{aligned} \rho_B = & 0.99707 + \left[ 6.504 \times 10^{-3} (w_{\text{NaCl}}) \right] + \left[ 7.923 \times 10^{-3} (w_{\text{CaCl}_2}) \right] \\ & + \left[ 8.334 \times 10^{-5} (w_{\text{NaCl}})(w_{\text{CaCl}_2}) \right] + \left[ 4.395 \times 10^{-5} (w_{\text{NaCl}})^2 \right] \\ & + \left[ 4.964 \times 10^{-5} (w_{\text{CaCl}_2})^2 \right] \end{aligned} \quad (50)$$

where

$\rho_B$  is the NADF aqueous-phase density, expressed in grams per milliliter;

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

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

#### 12.4.6 NADF aqueous-phase calcium chloride concentration

The  $\text{CaCl}_2$  concentration of the NADF aqueous-phase, expressed in milligrams per liter, shall be calculated using Equation (51).

$$c_{\text{CaCl}_2, \text{Aq}} = 10,000 \times w_{\text{CaCl}_2} \times \rho_B \quad (51)$$

where

$c_{\text{CaCl}_2, \text{Aq}}$  is the NADF aqueous-phase calcium chloride concentration, expressed in milligrams per liter;

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

$\rho_B$  is the NADF aqueous-phase density, expressed in grams per milliliter.

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

#### **Instruction:**

For fluids with no sodium chloride, skip to 12.6.

#### 12.4.7 NADF aqueous-phase soluble sodium chloride concentration

The soluble NaCl concentration of the NADF aqueous-phase, expressed in milligrams per liter, shall be calculated using Equation (52).

$$c_{\text{NaCl,Aq}} = 10,000 \times w_{\text{NaCl}} \times \rho_{\text{B}} \quad (52)$$

where

$c_{\text{NaCl,Aq}}$  is the aqueous-phase sodium chloride concentration, expressed in milligrams per liter;

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

$\rho_{\text{B}}$  is the NADF aqueous-phase density, expressed in grams per milliliter.

**Instruction:**

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

**12.5 Whole-NADF Soluble and Insoluble Sodium Chloride Calculations**

**12.5.1** For drilling fluids with a super-saturated aqueous-phase containing some insoluble NaCl, Equations (53) to (57) can be used to determine the soluble and insoluble quantities in the whole-NADF

**12.5.2** The soluble NaCl content of the whole NADF shall be calculated using Equation (53).

$$c_{\text{NaCl,df,Sol}} = \frac{w_{\text{NaCl,Max-Cal}} \times [c_{\text{CaCl}_2,\text{df}} + (10,000 \times \varphi_{\text{W}})]}{100 - w_{\text{NaCl,Max-Cal}}} \quad (53)$$

where

$c_{\text{NaCl,df,Sol}}$  is the whole-NADF soluble sodium chloride concentration, expressed in milligrams per liter;

$w_{\text{NaCl,Max-Cal}}$  is the recalculated maximum aqueous-phase mass fraction of soluble sodium chloride that can exist for a given mass fraction of CaCl<sub>2</sub>, expressed as a percentage of the total aqueous-phase mass;

$c_{\text{CaCl}_2,\text{df}}$  is the whole-NADF calcium chloride concentration, expressed in milligrams per liter;

$\varphi_{\text{W}}$  is the volume fraction of water, expressed as a percentage.

**12.5.3** The concentration of soluble NaCl in the NADF, expressed in SI units, shall be calculated using Equation (54) or expressed in USC units, using Equation (55).

$$c_{\text{NaCl,df,Sol,SI}} = 0.001 \times c_{\text{NaCl,df,Sol}} \quad (54)$$

$$c_{\text{NaCl,df,Sol,USC}} = 0.00035 \times c_{\text{NaCl,df,Sol}} \quad (55)$$

where

$c_{\text{NaCl,df,Sol,SI}}$  is the whole-NADF soluble sodium chloride concentration, expressed in kilograms per cubic meter;

$c_{\text{NaCl,df,Sol,USC}}$  is the whole-NADF soluble sodium chloride concentration, expressed in pounds per barrel;

$c_{\text{NaCl,df,Sol}}$  is the whole-NADF soluble sodium chloride concentration, expressed in milligrams per liter.

**12.5.4** The concentration of insoluble NaCl in the whole-NADF shall be calculated using Equations (56), (57) or (58).

$$c_{\text{NaCl,df,InSol}} = c_{\text{NaCl,df}} - c_{\text{NaCl,df,Sol}} \quad (56)$$

$$c_{\text{NaCl,df,InSol,SI}} = c_{\text{NaCl,df,SI}} - c_{\text{NaCl,df,Sol,SI}} = 0.001 \times c_{\text{NaCl,df,InSol}} \quad (57)$$

$$c_{\text{NaCl,df,InSol,USC}} = c_{\text{NaCl,df,USC}} - c_{\text{NaCl,df,Sol,USC}} = 0.00035 \times c_{\text{NaCl,df,InSol}} \quad (58)$$

where

$c_{\text{NaCl,df,InSol}}$  is the whole-NADF insoluble sodium chloride concentration, expressed in milligrams per liter;

$c_{\text{NaCl,df}}$  is the whole-NADF sodium chloride concentration, expressed in milligrams per liter;

$c_{\text{NaCl,df,Sol}}$  is the whole-NADF soluble sodium chloride concentration, expressed in milligrams per liter;

$c_{\text{NaCl,df,InSol,SI}}$  is the whole-NADF insoluble sodium chloride concentration, expressed in kilograms per cubic meters;

$c_{\text{NaCl,df,SI}}$  is the whole-NADF sodium chloride concentration, expressed in kilograms per cubic meters;

$c_{\text{NaCl,df,Sol,SI}}$  is the whole-NADF soluble sodium chloride concentration, expressed in kilograms per cubic meter,

$c_{\text{NaCl,df,InSol,USC}}$  is the whole-NADF insoluble sodium chloride concentration, expressed in pounds per barrel;

$c_{\text{NaCl,df,USC}}$  is the whole-NADF sodium chloride concentration, expressed in pounds per barrel;

$c_{\text{NaCl,df,Sol,USC}}$  is the whole-NADF soluble sodium chloride concentration, expressed in pounds per barrel.

## 12.6 Calculation—Solids in the Whole-NADF

### 12.6.1 Corrected Solids Concentration

**12.6.1.1** The retort solids volume does not represent the true solids content because it includes soluble salts in the NADF aqueous-phase. The dry retort solids volume shall be corrected to account for the true brine volume. The volume fraction of aqueous-phase (brine) in the whole-NADF shall be calculated using Equation (59).

$$\varphi_B = \frac{100 \times \varphi_W}{\rho_B \times [100 - (w_{\text{NaCl}} + w_{\text{CaCl}_2})]} \quad (59)$$

where

$\varphi_B$  is the volume fraction of aqueous-phase (brine), expressed as a percentage of the whole-NADF;

$w_{\text{CaCl}_2}$  is the aqueous-phase mass fraction calcium chloride, expressed as a percentage of the whole-NADF aqueous-phase mass;

$w_{\text{NaCl}}$  is the aqueous-phase mass fraction sodium chloride, expressed in a percentage of the whole-NADF aqueous-phase mass;

$\varphi_W$  is the volume fraction of water, expressed as a percentage of the whole-NADF;

$\rho_B$  is the NADF aqueous-phase (brine) density, expressed in grams per milliliter.

**12.6.1.2** The corrected volume fraction of solids in the whole-NADF shall be calculated using Equation (60).

$$\varphi_{\text{DS}} = 100 - (\varphi_{\text{NAF}} + \varphi_B) \quad (60)$$

where

$\varphi_{\text{DS}}$  is the corrected volume fraction of solids, expressed as a percentage of the whole-NADF;

$\varphi_{\text{NAF}}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF;

$\varphi_B$  is the volume fraction of aqueous-phase (brine), expressed as a percentage of the whole-NADF.

### 12.6.2 Calculation—NAF-to-Water Ratio

The NAF and water ratio values,  $R_{\text{NAF-W}}$  and  $R_W$ , shall be calculated from the volume fraction of recovered liquids in the retort analysis.

The ratio of NAF shall be calculated using Equation (61):

$$R_{\text{NAF-W}} = \frac{100 \times \varphi_{\text{NAF}}}{\varphi_{\text{NAF}} + \varphi_W} \quad (61)$$

where

$R_{\text{NAF-W}}$  is the ratio of the volume fraction of NAF to the sum of the volume fractions of NAF and pure water from the retort analysis, expressed as a percentage;

$\varphi_{\text{NAF}}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF;

$\varphi_{\text{W}}$  is the volume fraction of pure water, expressed as a percentage of the whole-NADF;

and the ratio of water shall be calculated using Equation (62):

$$R_{\text{W}} = \frac{100 \times \varphi_{\text{W}}}{\varphi_{\text{NAF}} + \varphi_{\text{W}}} \quad (62)$$

where

$R_{\text{W}}$  is the ratio of the volume fraction of water to the sum of the volume fractions of NAF and pure water from the retort analysis, expressed as a percentage.

$\varphi_{\text{NAF}}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF;

$\varphi_{\text{W}}$  is the volume fraction of pure water, expressed as a percentage of the whole-NADF;

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

### 12.6.3 Calculation—NAF-to-Brine Ratio

The NAF and aqueous-phase (brine) ratio values,  $R_{\text{NAF-B}}$  and  $R_{\text{B}}$  shall be calculated from the volume fraction of NAF from the retort analysis and the volume fraction of aqueous-phase (brine) calculated in 12.6.1 as per the following.

The ratio of NAF shall be calculated using Equation (63):

$$R_{\text{NAF-B}} = \frac{100 \times \varphi_{\text{NAF}}}{\varphi_{\text{NAF}} + \varphi_{\text{B}}} \quad (63)$$

where

$R_{\text{NAF-B}}$  is the ratio of the volume fraction of NAF to the sum of the volume fractions of NAF and aqueous-phase (brine), expressed as a percentage;

$\varphi_{\text{NAF}}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF;

$\varphi_{\text{B}}$  is the volume fraction of aqueous-phase (brine), expressed as a percentage of the whole-NADF.

and the ratio of aqueous-phase shall be calculated using Equation (64):

$$R_B = \frac{100 \times \varphi_B}{\varphi_{NAF} + \varphi_B} \quad (64)$$

where

$R_B$  is the ratio of the volume fraction of aqueous-phase (brine) to the sum of the volume fractions of NAF and brine, expressed as a percentage.

$\varphi_{NAF}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF;

$\varphi_B$  is the volume fraction of aqueous-phase (brine), expressed as a percentage of the whole-NADF;

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

#### 12.6.4 Calculation—Solids Concentration and Density

**12.6.4.1** The solids in a NADF consist of low-gravity solids, such as drill solids, and weighting material, such as barite or hematite. Densities for materials commonly occurring in NADF are found in Table 5.

**12.6.4.2** The average density of the suspended solids shall be calculated using Equation (65).

$$\overline{\rho_{SS}} = \frac{(100 \times \rho_{df}) - [(\varphi_{NAF} \times \rho_{NAF}) + (\varphi_B \times \rho_B)]}{\varphi_{DS}} \quad (65)$$

where

$\overline{\rho_{SS}}$  is the average density of the suspended solids;

$\varphi_{NAF}$  is the volume fraction of NAF, expressed as a percentage of the whole-NADF;

$\varphi_B$  is the volume fraction of brine, expressed as a percentage of the whole-NADF;

$\varphi_{DS}$  is the corrected volume fraction of solids, expressed as a percentage of the whole-NADF

$\rho_{df}$  is the density of the NADF;

$\rho_B$  is the density of the aqueous-phase (brine);

$\rho_{NAF}$  is the density of the NAF.

Equation (65) may be calculated using any consistent units for the drilling fluid density,  $\rho_{df}$ , NAF density,  $\rho_{NAF}$ , and brine density,  $\rho_B$ , values.

**Table 5—Commonly Used Densities**

Drilling Fluid Component	Density (volumetric mass)
--------------------------	---------------------------

		g/mL	kg/m <sup>3</sup>	lb/gal
Fresh water (tap)		1.00	1000	8.345
Diesel (typical)		0.84	840	7.00
Low-viscosity oil (typical)		0.80	800	6.70
Low-gravity solids		2.65	2650	22.10
Weighting materials	Barite	4.20	4200	35.00
	Hematite	5.05	5050	42.10

### 12.6.5 Calculation—Volume Fraction and Concentration of Weighting Material and Low-gravity Solids

12.6.5.1 The volume fraction of weighting-material solids shall be calculated using Equation (66).

$$\varphi_{WM} = \frac{\overline{\rho_{SS}} - \rho_{LG}}{\rho_{WM} - \rho_{LG}} \times \varphi_{DS} \quad (66)$$

where

$\varphi_{WM}$  is the volume fraction of the weighting-material solids, expressed as a percentage of the whole-NADF;

$\varphi_{DS}$  is the corrected volume fraction of solids, expressed as a percentage of the whole-NADF;

$\overline{\rho_{SS}}$  is the average density of the suspended solids, expressed in grams per milliliter;

$\rho_{WM}$  is the density of the weighting-material solids, expressed in grams per milliliter;

$\rho_{LG}$  is the density of the low-gravity solids, expressed in grams per milliliter.

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

12.6.5.2 The volume fraction of the low-gravity solids shall be calculated using Equation (67).

$$\varphi_{LG} = \varphi_{DS} - \varphi_{WM} \quad (67)$$

where

$\varphi_{LG}$  is the volume fraction of the low-gravity solids, expressed as a percentage of the whole-NADF;

$\varphi_{WM}$  is the volume fraction of the weighting-material solids, expressed as a percentage of the whole-NADF;

$\phi_{DS}$  is the corrected volume fraction of solids, expressed as a percentage of the whole-NADF.

**12.6.5.3** The mass concentration of the weighting material shall be calculated using Equation (68) for SI units or using Equation (69) for USC units.

$$c_{WM,SI} = 10 \times \rho_{WM} \times \phi_{WM} \quad (68)$$

$$c_{WM,USC} = 3.5 \times \rho_{WM} \times \phi_{WM} \quad (69)$$

where

$c_{WM,SI}$  is the weighting material concentration, expressed in kilograms per cubic meter;

$c_{WM,USC}$  is the weighting material concentration, expressed in pounds per barrel;

$\rho_{WM}$  is the density of the weighting-material solids, expressed in grams per milliliter;

$\phi_{WM}$  is the volume fraction of the weighting-material solids, expressed as a percentage of the whole-NADF.

**12.6.5.4** The mass concentration of low-gravity solids shall be calculated using Equation (70) for SI units or Equation (71) for USC units.

$$c_{LG,SI} = 10 \times \rho_{LG} \times \phi_{LG} \quad (70)$$

$$c_{LG,USC} = 3.5 \times \rho_{LG} \times \phi_{LG} \quad (71)$$

where

$c_{LG,SI}$  is the low-gravity solids concentration, expressed in kilograms per cubic meter;

$c_{LG,USC}$  is the low-gravity solids concentration, expressed in pounds per barrel;

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

$\phi_{LG}$  is the volume fraction of the low-gravity solids, expressed as a percentage of the whole NADF.

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## **Annex A** **(informative)**

### **Measurement of Shear Strength Using Shearometer Tube**

#### **A.1 Principle**

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

**A.1.2** The following technique described in this annex can be used to determine this tendency and to estimate the extent to which the drilling fluid will develop excessive shear strength. This shear strength measurement is made on a static, heat-aged drilling fluid sample. Aging temperatures are therefore selected to be near or above the estimated bottom-hole temperature of the well.

**Caution—Always follow the manufacturers’ procedures when using heat aging cells.**

Standard heat aging cells and the procedure described in API 13I are generally rated for the range of most bottom-hole temperatures, although they are not rated for elevated pressures. Standard heat aging cells are used for fluid evaluations, comparisons, screening, and are normally used for this static shear procedure. Static shear testing is conducted on a heat aged fluid that has cooled and is at atmospheric pressure conditions (standard conditions).

Heat aging or testing at high temperatures and elevated pressures provides a better relationship to down hole conditions for higher temperature wells but requires specialized equipment. To evaluate the gelation of a drilling fluid at high temperature and elevated pressure, a consistometer, HTHP viscometer, or other specialized equipment is required.

#### **A.2 Apparatus**

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

length	89 mm (3.5 in.)
outside diameter	36 mm (1.4 in.)
wall thickness	0.2 mm (0.008 in.)

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

**A.2.2 Platform for weights**, metal square platform suitable to support the weights on top of the shearometer tube, larger than the 36 mm (1.4 in.) OD of the tube but smaller than the bore of the heat aging cell. The mass of the platform shall be stamped on the surface of the platform.

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

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

### A.3 Procedure

Shear strength using a shearometer tube shall be measured as per the following procedure.

- a) After static heat aging for the predetermined time (see API 13I) and cooled to room temperature while being kept upright and static. Place and balance the shear tube carefully on the surface of the aged sample, it may be necessary to shift the tube and weights on the platform to ensure that the tube's initial penetration into the drilling fluid is vertical. If a crust had developed on the heat-aged sample, the surface of this crust shall be gently broken using the shear tube as it is placed into the fluid for the test.
- b) Carefully place the platform and just enough weight on the platform to start the downward movement of the shear tube. Unless too much weight is added or the fluid has no measurable static shear strength, the tube will stop its downward travel at the point where the shear strength resistance of the aged drilling fluid against the surface of the tube is sufficient to support the applied mass. It is desirable to submerge at least one-half of the length of the tube.
- c) Record the total mass, in grams, of the platform and weights,  $m_{tot}$ . Record the mass, in grams, of the shear tube,  $m_{st}$ . Measure the portion of the tube submerged in the fluid. The length of the tube submerged can be most accurately determined by measuring the length of the non-submerged portion while the tube is at its maximum penetration depth. A small ruler held at the drilling fluid surface and alongside the tube will facilitate this measurement. The length of the tube minus the exposed length equals the submerged portion.

### A.4 Calculation

Shear strength <sup>6</sup>,  $\gamma$ , shall be calculated using Equation (A.1) expressed in pascals (SI unit) or Equation (A.2) expressed in pounds-force per one hundred square feet (lbf/100ft<sup>2</sup>) ( USC units)

- a) Shear strength in SI units (Pa):

$$\gamma_{SI} = \frac{4.40 \times (m_{st} + m_{tot})}{l_{SI}} - (1.02 \times \rho_{df}) \quad (A.1)$$

where

$\gamma_{SI}$  is the shear strength of the drilling fluid, expressed in pascals;

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<sup>6</sup> Shear strength Equations derived from reference [21].

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

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

$l_{SI}$  is the submerged length of shear tube, expressed in centimeters;

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

NOTE For SI/metric calculations, drilling fluid density expressed in grams per milliliter is equivalent to the relative density or density expressed in kilograms per liter.

b) Shear strength in USC units (lbf/100ft<sup>2</sup>):

$$\gamma_{USC} = \frac{3.61 \times (m_{st} + m_{tot})}{l_{USC}} - (0.256 \times \rho_{df,USC}) \quad (A.2)$$

where

$\gamma_{USC}$  is the shear strength of the drilling fluid, expressed in pounds-force per one hundred square feet;

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

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

$l_{USC}$  is the submerged length of shear tube, expressed in inches;

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

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## **Annex B** (informative)

### **Determination of Non-Aqueous Fluid Retained on Cuttings**

#### **B.1 Principle**

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

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

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

#### **B.2 Apparatus**

##### **B.2.1 Retort Instrument.**

**B.2.1.1 Retort assembly**, which includes a retort body, cup, and lid with a volume of 50 mL (precision  $\pm 0.25$  mL), constructed of 303 stainless steel, or equivalent.

The retort cup volume with lid shall be verified gravimetrically. The procedure and calculations are given in Annex J (J.6).

**B.2.1.2 Condenser**, capable of cooling the NAF and water vapors below their vaporization temperatures.

**B.2.1.3 Heating jacket**, nominal power 350 W.

**B.2.1.4 Temperature controller**, capable of limiting temperature of the retort to  $500\text{ }^{\circ}\text{C} \pm 40\text{ }^{\circ}\text{C}$  ( $930\text{ }^{\circ}\text{F} \pm 70\text{ }^{\circ}\text{F}$ ).

**B.2.2 Liquid receiver** (TC), especially designed cylindrical glassware with a rounded bottom to facilitate cleaning and a funnel-shaped top to catch falling drops, meeting the following specifications:

- **precision**: see Table B.1;
- **calibration**: TC at  $20\text{ }^{\circ}\text{C}$  ( $68\text{ }^{\circ}\text{F}$ );
- **scale**: milliliters, cubic centimeters or volume fraction (as a percentage);
- **material**: transparent and inert to NAF, water, and salt solutions at temperatures up to  $32\text{ }^{\circ}\text{C}$  ( $90\text{ }^{\circ}\text{F}$ ).

**Table B.1—Precision of Liquid Receiver**

		Total Volume		
		10 ml	20 ml	50 ml
Precision	(0 % to 100 %)	±0.05 ml	±0.10 ml	±0.25 ml
Frequency of graduation marks	(0 % to 100 %)	0.10 ml	0.10 ml	0.50ml

NOTE: It is recommended to use the smallest liquid receiver whose capacity is sufficient for the quantity of condensed liquid. For regulatory compliance monitoring, the 10 mL liquid receiver with 0.10 ml graduations may be required to achieve greater accuracy.

**B.2.3 Fine steel wool**, oil-free.

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

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

**B.2.5 Pipe cleaners**.

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

**B.2.7 Corkscrew**.

**B.2.8 Top-loading balance**, capable of weighing 2000 g with an accuracy of ±0.01 g.

**B.2.9 Brush**.

**B.3 Procedure**

In order to determine NAF and water retained on cuttings, the following procedure should be used.

- a) Clean and dry the retort assembly and condenser.
- b) Pack the retort body with steel wool.
- c) Apply lubricant/sealant to threads of retort cup and retort stem.
- d) Weigh and record the total mass of the empty retort cup, lid, and retort body (packed with steel wool). Record this as  $m_1$ , expressed in grams.

NOTE All weights are recorded to the nearest 0.01 g.

- e) Collect a representative wet cuttings sample and allow to cool to approximately 27 °C (80 °F).
- f) Partially fill the retort cup with NADF wet cuttings to about 6 mm (0.25 in.) below the top of the retort cup and place the lid on the cup. Screw the retort cup (with cuttings and lid) onto the retort body (packed with steel wool), weigh and record the total mass. Record this as  $m_2$ , expressed in grams.

- g) Attach the condenser. Place the retort assembly into the heating jacket. Close the insulating lid.
- h) Weigh a clean, dry liquid receiver. Record this as  $m_3$ , expressed in grams. Place the receiver below the condenser outlet.

NOTE 1 To improve the ability to make accurate readings of the volume of NAF and water, it is possible to pre-wet the inside of the glass liquid receiver with PNP. PNP is the product used to break the emulsion during chemical titration of NADF (see Section 10). The basic method for wetting the glass liquid receiver is to add approximately 0.5 mL of PNP to the liquid receiver, then tilt and roll the liquid receiver to allow the solvent to coat the inside. After coating the inside of the liquid receiver with solvent, turn the receiver upside down long enough to empty all of the excess solvent.

**Caution—PNP should be used only when the precautions, appropriate PPE, and exposure controls are followed as described on the safety data sheet, as is recommended for all laboratory chemicals. This includes eye/face protection, skin protection, and respiratory protection for both liquid and/or vapor exposure.**

NOTE 2 Due to the rounded bottom of the liquid receiver, it might be helpful to place the liquid receiver in a 100 mL graduated cylinder to hold it on the top-loading balance while being weighed.

NOTE 3 The length of the liquid receiver might require that it be angled out from the retort condenser passage and perhaps supported off the edge of the worktable.

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

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

NOTE If an emulsion interface is present between the NAF 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.

- k) Record the total water volume,  $V_W$ , collected in the liquid receiver. This volume of water in milliliters is converted to mass, expressed in grams, in B.4.

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 the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Thirdly, for water-to-NAF meniscus, read the water volume at the lowest point.

- l) Weigh the glass liquid receiver and its liquid content (NAF and water). Record this as  $m_4$ , expressed in grams.
- m) Turn off the heating jacket. Remove the retort assembly and condenser from the heating jacket and allow them to cool.

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

## B.4 Calculations

### B.4.1 Mass of NAF Retained on Cuttings

The mass of NAF retained on cuttings should be calculated using Equations (B.1), (B.2) and (B.3) and must satisfy the mass balance requirement of Equation (B.4).

- a) calculate the mass of wet cuttings

$$m_{WC} = m_2 - m_1 \quad (B.1)$$

where

- $m_{WC}$  is the mass of the wet cuttings, expressed in grams;
- $m_2$  is the mass of the filled retort assembly (cup with sample, lid, and body packed with steel wool), expressed in grams;
- $m_1$  is the mass of the empty retort assembly (cup, lid, and body packed with steel wool), expressed in grams.

- b) Calculate the mass of dried retort cuttings

$$m_{DC} = m_5 - m_1 \quad (B.2)$$

where

- $m_{DC}$  is the mass of the dried retort cuttings, expressed in grams;
- $m_5$  is the mass of the cooled retort assembly (cup with dried retorted cuttings, lid, body packed with steel wool), expressed in grams;
- $m_1$  is the mass of the empty retort assembly (cup, lid and body packed with steel wool), expressed in grams.

- c) Calculate the mass of NAF retained on cuttings (NAF recovered)

$$m_{NAF-C} = m_4 - (m_3 + V_W) \quad (B.3)$$

where

- $m_{NAF-C}$  is the mass of NAF retained on cuttings, expressed in grams;

$m_4$	is the mass of the cooled liquid receiver with condensed liquids, expressed in grams;
$m_3$	is the mass of the empty, dry liquid receiver, expressed in grams;
$V_W$	is the water volume, expressed in milliliters or, water mass expressed in grams (1 mL = 1 g) (see 3.3).

NOTE Assuming the density of water is 1 g/mL, the mass, in grams, of the volume of water is numerically equivalent to the volume of the water measured in milliliters.

d) Mass balance requirement:

The sum of the mass of dried retorted cuttings,  $m_{DC}$ , the mass of NAF retained on cuttings,  $m_{NAF-C}$ , and the mass of water,  $V_W$ , shall be within 5 % of the mass of the wet cutting sample,  $m_{WC}$ .

$$0.95 \leq \frac{m_{DC} + m_{NAF-C} + V_W}{m_{WC}} \leq 1.05 \quad (B.4)$$

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

#### **B.4.2 Retained NAF on Cuttings (ROC)**

Retained NAF on cuttings, ROC, may be reported in various units depending on the local requirements or prevailing regulation:

Reporting ROC in grams of NAF per kilogram of cuttings should be calculated using Equation (B.5) or Equation (B.6)

—  $ROC_W$ , retained NAF mass in grams per kilogram of wet cuttings sample:

$$ROC_W = 1000 \times \frac{m_{NAF-C}}{m_{WC}} \quad (B.5)$$

—  $ROC_D$ , retained NAF mass in grams per kilogram of dry retorted cuttings:

$$ROC_D = 1000 \times \frac{m_{NAF}}{m_{DC}} \quad (B.6)$$

where

$m_{NAF-C}$  is the mass of NAF retained on cuttings, expressed in grams;

$m_{WC}$  is the mass of the wet cuttings, expressed in grams;

$m_{DC}$  is the mass of the dried retort cuttings, expressed in grams;

For reporting ROC as a weight percent of NAF on cuttings (same as grams of NAF per 100 grams of cuttings) should be calculated using Equation (B.7) or Equation (B.8):

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- the weight percent of NAF on wet cuttings sample (grams of NAF per 100 grams of wet cuttings sample),  $\%ROC_W$  (same units as “mass percent NAF base fluid retained on the cuttings” Effluent Limitation Guidelines) United States 40 CFR Parts 9 and 435, Federal Register Vol. 66 No. 14, Part IV, EPA, January 22, 2001, Appendix 7):

$$\%ROC_W = 100 \times \frac{m_{NAF-C}}{m_{WC}} \quad (B.7)$$

- weight percent of NAF from dry retorted cuttings (grams of NAF per 100 grams of dry retorted cuttings),  $\%ROC_D$

$$\%ROC_D = 100 \times \frac{m_{NAF-C}}{m_{DC}} \quad (B.8)$$

where

$m_{NAF-C}$  is the mass of the NAF, expressed in grams;

$m_{WC}$  is the mass of the wet cuttings, expressed in grams;

$m_{DC}$  is the mass of the dried retort cuttings, expressed in grams;

NOTE When reporting NAF retained on cuttings in terms of the dry (retorted) cuttings, the NAF concentration might be higher than that calculated from the wet cuttings sample, due to interstitial water extracted during retorting. Also, a cutting containing a higher percentage of water will appear to give a higher NAF retention mass for the same reasons. The water concentration is reported as the mass of water (in grams) per kilogram of wet cuttings sample [see B.3 item k)].

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## **Annex C** (informative)

### **Determination of Aqueous-phase Activity of Emulsified Water Using an Electro-Hygrometer**

#### **C.1 Principle**

**C.1.1** This annex describes a procedure for the use of an electro-hygrometer to measure relative humidity of a NADF in a closed-air space in order to determine the activity of the emulsified water. Water activity,  $a_w$ , is a measure of the chemical potential or reaction availability of a chemical species, diluted in water. Water activity is expressed as the ratio of the vapor pressure of a solution ( $p$ ) to the vapor pressure of pure water ( $p_0$ ) at the same temperature. Relative humidity of air is defined as the ratio of the vapor pressure of air to its saturation vapor pressure. When vapor and temperature equilibrium are obtained, the water activity of the sample is equal to the relative humidity of air surrounding the sample in a sealed measurement chamber. This property can assist in evaluating the fluid compatibility while drilling in water-sensitive shale formations. The relative rate at which drilling fluids chemically transport water to the formation can be quantified by measurement of water activity. Water activity instruments measure the amount of free (sometimes referred to as unbound or active) water present in the drilling fluid.

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

**C.1.3** Water transfer from an NADF can be controlled by utilizing salts dissolving in emulsified water phase to reduce activity. Salt lowers the activity of the water phase, thus reducing the tendency of water to be absorbed by shale. Water transfer to shale is eliminated when the activity of the emulsified water is reduced to a value equal to or lower than that of the exposed shale formation. The transport of water/ion movement in and out of shale can impact shale stability by considerably changing the pore pressure and swelling pressure in the pore fabric, sometimes changing the chemical and mechanical properties of the shale in the near wellbore. Hole condition and cuttings texture can guide proper activity adjustment.

**C.1.4** The most efficient, commonly available salt for reducing the activity of water in NADFs is calcium chloride. The water activity of the emulsified brine controls the activity of the NADF regardless of it being emulsified. The aqueous-phase activity of a NADF will be changed by water evaporation or water addition and by adding or drilling other water-soluble salts.

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

## C.2 Reagents and Apparatus

### C.2.1 Standard saturated salt solutions

C.2.1.1 The activity of standard saturated salt solutions is described in Table C.1.

**Table C.1—Standard Saturated Salt Solutions**

Salt	CAS No.	Water Activity of Saturated Salt Solution $a_w$ at 25 °C (77 °F)	Salt Concentration g/100 mL water
Calcium chloride	10043-52-4	0.295	100
Calcium nitrate	13477-34-4	0.505	200
Sodium chloride	7647-14-5	0.753	200
Potassium nitrate	7757-79-1	0.938	200

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

C.2.1.3 If no crystals are visible, some additional crystals or grains of the same salt should be added to induce precipitation. This is often necessary with calcium chloride. Salts of reagent-grade purity should be recommended. However, if necessary, field-grade NaCl or CaCl<sub>2</sub> can be substituted.

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

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

- The relative humidity sensor probe should be suitable for monitoring the air space of a sample container with the sample.
- The linearity response of the instrument should be such that the correlation coefficient between published standard brine activity and relative humidity, as measured by the instrument above each brine, is greater than 0.95.
- The instrument should indicate within 10 % of the true activity within 30 min, when measuring the brine activity of the standard salt solutions in C.2.1.

**NOTE** This procedure is for using an electro-hygrometer only. Chilled mirror and capacitive sensor instruments are available which can also measure water activity. Capacitive sensor instruments use capacitance sensors to measure water activity. Chilled mirror instruments use a chilled mirror to measure the dew point temperature (a measure of relative humidity) of air equilibrated with a sample and then convert the dew point temperature to vapor pressure. Chilled mirror devices are common in other industries and increasing used in drilling fluid applications.

**C.2.4 Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) accurate to  $\pm 1$  °C ( $\pm 2$  °F), not required if instrument has an internal thermometer.

**C.2.5 Measuring container**, as suggested or supplied by the manufacturer, having a volume sufficient to provide a liquid surface area of approximately 30 cm<sup>2</sup> to 50 cm<sup>2</sup> (4.5 in.<sup>2</sup> to 7.75 in.<sup>2</sup>). The container shall have a flexible elastomer cap (airtight) which, when bored through, will form an airtight seal around the shaft of the sensor probe and thermometer (if used).

**C.2.6 Linear graph paper.**

### **C.3 Procedure**

#### **C.3.1 Calibration of Hygrometer**

Hygrometer should be calibrated as per the following procedure.

- a) Prepare sufficient volume of each known relative humidity standard to cover the bottom of the measuring container to a liquid depth of 12 mm (0.5 in.) or follow the manufacturer's recommendations. These salt solutions shall be kept firmly capped to prevent spillage, evaporation, or contamination.
- b) Desiccate the probe with anhydrous calcium chloride in a closed sample container (other desiccants may be used) by placing the sensor probe (fitted in the sample container cap) in the air space above the salt. Wait 10 min to 15 min. A relative humidity reading of 24 % or lower indicates that the probe is dry. Keep a distance of 12 mm (0.5 in.) between the sensing probe and the desiccant and maintain the same distance for salt solutions and drilling fluid samples. Never allow the probe to directly contact any samples or solutions.
- c) Transfer the probe in the measuring container cap to the first (lowest  $a_w$ ) standard solution and allow 30 min for equilibrium to be reached. Temperature of the standard solutions shall be maintained between 24 °C and 25 °C (75 °F and 77 °F).
- d) Remove the probe from the first solution and place it into the container with the solution of second lowest known activity.
- e) Proceed as in item c) and measure the relative humidity for each standard solution, proceeding from that with the lowest to that with the highest activity  $a_w$ .
- f) Allow each standard to equilibrate for the same length of time. Record temperature and percent relative humidity ( $RH_{\%}$ ). Plot the  $RH_{\%}$  versus the activity of the known solution,  $a_w$ .

#### **C.3.2 Measurement of NADF Activity, $a_{df}$**

In order to determine the activity of a NADF, the following procedure should be applied.

- a) Desiccate the probe for 10 min to 15 min, as in C.3.1 item b).
- b) Transfer sufficient volume of room-temperature drilling fluid (40 mL) to cover the bottom of the sample container to a depth of 12 mm (0.5 in.).

- c) Insert the probe into the air space 12 mm (0.5 in.) above the fluid surface. Sample temperature shall be between 24 °C and 25 °C (75 °F and 77 °F), or the same temperature at which the standards were tested.
- d) Turn on the hygrometer and wait 30 min (the same time period used for standard calibration) and record the relative humidity ( $RH\%$ ) and temperature indicated.
- e) Examine the fluid to ensure that separation has not occurred. Separation of an NAF layer will lead to erroneous readings.

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

#### **C.4 Calculation—NADF Activity, $a_{df}$**

**C.4.1** The relative humidity,  $RH\%$ , obtained in C.3.2 item d) should be reported on the graph plotted in C.3.1 item f). The corresponding activity that appears on the graph should be read and recorded as the NADF activity,  $a_{df}$ .

**C.4.2** If not measured directly, these plotting instructions should be followed:

- a) using data for standards given in C.3.1, plot  $RH\%$  versus  $a_w$  on linear graph paper.
- b) plot the data obtained for each sample (C.3.2) on the linear graph paper and read the NADF activity,  $a_{df}$ .

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## Annex D (informative)

### Determination of Aniline-point Temperature of Base Non-aqueous Fluids

#### D.1 Principle

**D.1.1** The aniline-point temperature is the lowest temperature at which equal volumes of aniline and hydrocarbon samples are completely miscible. This test method covers the determination of the aniline-point temperature of petroleum products and hydrocarbon solvents, such as diesel oils and mineral oils, used in preparation of NADFs. This method is suitable for transparent liquid samples having an initial boiling point above room temperature, and where the aniline-point temperature is below the bubble point and above the solidification point of the aniline-sample mixture. This test method is not suitable for drilling fluids.

NOTE See ISO 2977 for additional information.

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

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

#### D.2 Reagents and Apparatus

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

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

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

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

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

**D.2.5 Thermometer**, with a range of 25 °C to 105 °C (75 °F to 220 °F) accurate to ± 0.5 °C (± 1 °F).

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

**D.2.7 Wire stirrer**, constructed of 1.5 mm to 1.7 mm (14 gauge) wire, bent with a 19 mm (0.75 in.) diameter loop, to be used to stir the mixture in the test tube.

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

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

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

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

**D.2.10 Pipette**, TD, with a volume of 10 mL.

**D.2.11 Plastic pipette suction bulb** (do not use rubber bulb).

**D.2.12 Safety goggles**.

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

### **D.3 Procedure**

In order to determine aniline-point temperature of a NAF, the following procedure should be used.

- a) Desiccate the NAF sample by adding approximately 10 % volume fraction drying agent in the NAF. Shake vigorously for 3 min to 5 min. Filter or centrifuge to remove the desiccant.
- b) Clean and dry the test tube, jacket, thermometer, and wire stirrer.
- c) Pipette 10 mL of aniline (using the suction bulb) into the test tube. Likewise, pipette 10 mL of NAF sample into the test tube.
- d) Assemble the thermometer and stirrer. Position the thermometer in the test tube so that the immersion mark is at the top of the liquid. Be certain that the thermometer bulb does not touch the sides of the tube. Center the test tube in the jacket tube.
- e) Stir the mixture rapidly, at room temperature, using an up-and-down motion of the stirrer. Avoid forming air bubbles. Observe whether mixture is miscible (clear) or immiscible (cloudy). If the mixture is clear, go to D.3.12 and proceed using a cooling bath.

NOTE For most NAF base fluids, the aniline-NAF mixture will be immiscible (cloudy) at room temperature.

- f) If the mixture is cloudy, apply heat to the jacket tube to increase the temperature of the mixture in the test tube at a rate of 1 °C/min to 3 °C/min (2 °F/min to 5 °F/min).
- g) Continue heating while stirring until the mixture becomes completely miscible (clear). Remove the mixture from the heating source.

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

- h) Continue stirring while the clear mixture cools at a rate of 0.5 °C/min to 1.0 °C/min (1 °F/min to 2 °F/min). Notice when the cloudiness first reappears. Record as “aniline-point temperature” the temperature at which the mixture becomes cloudy throughout.

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

- i) Continue cooling the mixture, while stirring to a temperature of 1 °C to 2 °C (2 °F to 3.5 °F) below the first appearance of cloudiness, as described in D.3 item h)
- j) Heat the mixture again (as described in D.3 item g)) until it again becomes miscible. Remove the heat and cool the mixture until the cloudiness returns to obtain another observation of the aniline point temperature.
- k) Repeat observations of the aniline-point temperature several times until a value has been determined within  $\pm 0.5$  °C ( $\pm 1$  °F).
- l) If the aniline-NAF mixture was completely miscible at ambient temperature (D.3 item e)), substitute a cooling bath for the heating bath. Allow the mixture to cool slowly while stirring rapidly at the rate specified in D.3 item e). Determine the aniline-point temperature as described in D.3 item h) to item k).

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## **Annex E** (informative)

### **Lime, Salinity, and Solids Calculations**

#### **E.1 Principle**

The example calculations in this annex are designed to show how the equations in Section 10 and Section 12 are used to determine the amount of salt and solids present in a NADF containing barite as the weighting material. Four different cases are presented:

- Drilling fluid A: the drilling fluid contains only  $\text{CaCl}_2$  salt;
- Drilling fluid B: the drilling fluid contains only NaCl salt;
- Drilling fluid C: the drilling fluid contains both  $\text{CaCl}_2$  and soluble NaCl salts;
- Drilling fluid D: the drilling fluid contains both  $\text{CaCl}_2$  and NaCl salts, with some NaCl soluble in the aqueous-phase and some NaCl as insoluble solids in the whole drilling fluid.

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

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

#### **E.2 Test Data Used in Examples**

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

**Table E.1—Physical and Chemical Properties of Examples of Non-Aqueous Drilling Fluids**

Property	Drilling Fluid A Only CaCl <sub>2</sub> Salt	Drilling Fluid B Only NaCl Salt	Drilling Fluid C CaCl <sub>2</sub> and NaCl Salts	Drilling Fluid D CaCl <sub>2</sub> and Excess NaCl Salts
Drilling fluid density ( $\rho_{df}$ ), g/mL	1.32	1.32	1.32	1.32
Retort NAF concentration ( $\varphi_{NAF}$ ), % volume	59.5	59.5	59.5	59.5
Retort water concentration ( $\varphi_W$ ), % volume	25.0	25.0	25.0	25.0
EDTA ( $V_{EDTA}$ ), mL/mL of drilling fluid	10.0	0.0	8.4	8.4
Silver nitrate ( $V_{AgNO_3}$ ), mL/mL of drilling fluid	5.8	4.4	7.0	10.0
Sulfuric acid ( $V_K$ ), mL/mL of drilling fluid	2.6	2.0	2.6	3.0
NAF density ( $\rho_{NAF}$ ), g/mL	0.84	0.84	0.84	0.84
Density of low gravity solids ( $\rho_{LG}$ ), g/mL	2.65	2.65	2.65	2.65
Density of weighting material (barite) ( $\rho_{WM}$ ), g/mL	4.20	4.20	4.20	4.20

### E.3 NADF Containing Only CaCl<sub>2</sub> Salt (Drilling Fluid A)

**E.3.1** Whole-NADF chloride ion concentration [see 10.5.2, Equation (27)]:

$$c_{Cl, df} = 10,000 \times 5.8 = 58,000 \text{ mg/L of drilling fluid}$$

**E.3.2** Whole-NADF calcium concentration [see 10.6.2, Equation (28)]:

$$c_{Ca, df} = 4000 \times 10.0 = 40,000 \text{ mg/L of drilling fluid}$$

**E.3.3** Whole-NADF lime concentration [see 12.3.1, Equation (29) and Equation (30)]:

$$c_{Ca(OH)_2, df, SI} = 3.705 \times 2.6 = 9.6 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{Ca(OH)_2, df, USC} = 1.299 \times 2.6 = 3.4 \text{ lb/bbl of drilling fluid}$$

**E.3.4** Whole-NADF chloride ion concentration as CaCl<sub>2</sub>, from calcium analysis [see 12.3.2.2, Equation (33)]:

$$c_{Cl-(CaCl_2), df} = 1.769 \times 40,000 = 70,760 \text{ mg/L of drilling fluid}$$

**E.3.5** Whole-NADF chloride concentration associated as NaCl [see 12.3.2.3, Equation (34)]:

$$c_{Cl-(NaCl), df} = 58,000 - 70,760 = -12,760 \text{ mg/L of drilling fluid}$$

The negative number indicates that no NaCl is present in this drilling fluid and only CaCl<sub>2</sub> is present. For this case, calculate the whole fluid CaCl<sub>2</sub> concentration from the chloride ion analysis.

**E.3.6** Whole-NADF calcium chloride concentration [see 12.3.2.4, Equation (35), Equation (36) and Equation (37)]:

$$c_{\text{CaCl}_2, \text{df}} = 1.565 \times 58,000 = 90,770 \text{ mg/L of drilling fluid}$$

$$c_{\text{CaCl}_2, \text{df, SI}} = 0.001565 \times 58,000 = 90.8 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{CaCl}_2, \text{df, USC}} = 0.0005485 \times 58,000 = 31.8 \text{ lb/bbl of drilling fluid}$$

**E.3.7** Aqueous-phase mass fraction of calcium chloride, expressed as a percentage [see 12.4.2, Equation (44)]:

$$w_{\text{CaCl}_2} = \frac{100 \times 90,770}{90,770 + 0 + (10,000 \times 25.0)} = 26.7 \%$$

**E.3.8** Density of the aqueous-phase brine [see 12.4.5, Equation (50)]:

$$\rho_B = 0.997070 + 0 + (0.007923 \times 26.7) + 0 + 0 + [0.00004964 \times (26.7)^2] = 1.24 \text{ g/mL}$$

**E.3.9** Aqueous-phase calcium chloride concentration, in milligrams per liter [see 12.4.6, Equation (51)]:

$$c_{\text{CaCl}_2, \text{Aq}} = 10,000 \times 26.7 \times 1.244 = 332,148 \text{ mg/mL} \approx 332,150 \text{ mg/mL}$$

**E.3.10** Volume fraction (as a percentage) of aqueous-phase in whole-NADF [see 12.6.1.1, Equation (59)]:

$$\varphi_B = \frac{100 \times 25.0}{1.24 \times [100 - (0 + 26.7)]} = 27.4 \%$$
 volume fraction in whole - NADF

**E.3.11** Volume fraction (as a percentage) of corrected solids in whole-NADF [see 12.6.1.2, Equation (60)]:

$$\varphi_{\text{DS}} = 100 - (59.5 + 27.4) = 13.1 \%$$
 volume fraction in whole-NADF

**E.3.12** NAF and water ratio (volume fraction) calculations (see 12.6.2, Equation (61) and Equation (62)):

$$R_{\text{NAF-W}} = \frac{100 \times 59.5}{59.5 + 25.0} = 70.4 \%$$
 NAF

$$R_W = \frac{100 \times 25.0}{59.5 + 25.0} = 29.6 \%$$
 water

then,

$$R_{\text{NAF-W}} : R_W = 70.4 : 29.6$$

NOTE  $R_{\text{NAF-W}} : R_W$  is the NAF-to-water ratio, often denoted as OWR. For this example, the OWR is normally rounded to 70:30.

**E.3.13** NAF and aqueous-phase (brine) ratio (volume fraction) calculations [see 12.6.3, Equation (63) and Equation (64)]:

$$R_{\text{NAF-B}} = \frac{100 \times 59.5}{59.5 + 27.4} = 68.5 \% \text{ NAF}$$

$$R_{\text{B}} = \frac{100 \times 27.4}{59.5 + 27.4} = 31.5 \% \text{ brine}$$

then,

$$R_{\text{NAF-B}} : R_{\text{B}} = 68.5 : 31.5$$

NOTE  $R_{\text{NAF-B}} : R_{\text{B}}$  is the NAF-to-brine ratio, often denoted as OBR. For this example, the OBR is normally rounded to 68:32.

**E.3.14** Average density of suspended solids [see 12.6.4, Equation (65)]:

$$\rho_{\text{SS}} = \frac{(100 \times 1.32) - [(59.5 \times 0.84) + (27.4 \times 1.24)]}{13.1} = 3.66 \text{ g/mL}$$

**E.3.15** Volume fraction (as a percentage) of weighting-material [see 12.6.5.1, Equation (66)]:

$$\phi_{\text{WM}} = \frac{3.66 - 2.65}{4.20 - 2.65} \times 13.1 = 8.5 \% \text{ weighting-material}$$

**E.3.16** Volume fraction (as a percentage) of low-gravity solids [see 12.6.5.2, Equation (67)]:

$$\phi_{\text{LG}} = 13.1 - 8.5 = 4.6 \% \text{ low-gravity solids}$$

**E.3.17** Mass concentration of weighting-material [see 12.6.5.3, Equation (68) and Equation (69)]:

$$c_{\text{WM,SI}} = 10 \times 4.20 \times 8.5 = 357 \text{ kg/m}^3 \text{ weighting-material}$$

$$c_{\text{WM,USC}} = 3.5 \times 4.20 \times 8.5 = 125 \text{ lb/bbl weighting-material}$$

**E.3.18** Mass concentration of low-gravity solids [see 12.6.5.4, Equation (70) and Equation (71)]:

$$c_{\text{LG,SI}} = 10 \times 2.65 \times 4.6 = 122 \text{ kg/m}^3 \text{ low-gravity solids}$$

$$c_{\text{LG,USC}} = 3.5 \times 2.65 \times 4.6 = 43 \text{ lb/bbl low-gravity solids}$$

## **E.4 NADF Containing Only NaCl Salt (Drilling Fluid B)**

**E.4.1** Whole-NADF chloride ion concentration [see 10.5.2, Equation (27)]:

$$c_{\text{Cl,df}} = 10,000 \times 4.4 = 44,000 \text{ mg/L of drilling fluid}$$

**E.4.2** Whole-NADF calcium concentration [see 10.6.2, Equation (28)]:

$$c_{\text{Ca,df}} = 4000 \times 0 = 0 \text{ mg/L of drilling fluid}$$

**E.4.3** Whole-NADF lime concentration [see 12.3.1, Equation (29) and Equation (30)]:

$$c_{\text{Ca(OH)}_2, \text{df, SI}} = 3.705 \times 2.0 = 7.4 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{Ca(OH)}_2, \text{df, USC}} = 1.299 \times 2.0 = 2.6 \text{ lb/bbl of drilling fluid}$$

**E.4.4** Whole-NADF chloride ion concentration as  $\text{CaCl}_2$ , from calcium analysis [see 12.3.2.2, Equation (33)]:

$$c_{\text{Cl}-(\text{CaCl}_2), \text{df}} = 1.769 \times 0 = 0 \text{ mg/L of drilling fluid}$$

The  $\text{CaCl}_2$  content is 0.

**E.4.5** Whole-NADF chloride ion concentration as  $\text{NaCl}$  [see 12.3.2.3, Equation (34)]:

$$c_{\text{Cl}-(\text{NaCl}), \text{df}} = 44,000 - 0 = 44,000 \text{ mg/L of drilling fluid}$$

The positive number indicates that  $\text{NaCl}$  is present in this drilling fluid. Since no  $\text{CaCl}_2$  is present skip Equations (35-40), 44, 47, 48, and 51.

**E.4.6** Whole-NADF sodium chloride concentration [see 12.3.2.6, Equation (41), Equation (42) and Equation (43)]:

$$c_{\text{NaCl}, \text{df}} = 1.648 \times 44,000 = 72,512 \text{ mg/L} \approx 72,510 \text{ mg/L of drilling fluid}$$

$$c_{\text{NaCl}, \text{df, SI}} = 0.001648 \times 44,000 = 72.5 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{NaCl}, \text{df, USC}} = 0.000578 \times 44,000 = 25.4 \text{ lb/bbl of drilling fluid}$$

**E.4.7** Aqueous-phase mass fraction of sodium chloride, expressed as a percentage [see 12.4.2, Equation (45)]:

$$w_{\text{NaCl}} = \frac{100 \times 72,512}{0 + 72,512 + (10,000 \times 25.0)} = 22.5 \%$$

**E.4.8** The maximum mass fraction of soluble  $\text{NaCl}$  that can exist in the aqueous-phase is calculated as follows [see 12.4.3.2, Equation (46)]:

$$w_{\text{NaCl}, \text{Max}} = 26.432 - 0 + 0 + 0 = 26.4 \%$$

Since  $w_{\text{NaCl}} < w_{\text{NaCl}, \text{Max}}$ , all  $\text{NaCl}$  is soluble in the aqueous-phase.

**E.4.9** Density of the aqueous-phase brine [see 12.4.5, Equation (50)]:

$$\rho_B = 0.997070 + (0.006504 \times 22.5) + 0 + 0 + [0.00004395 \times (22.5)^2] + 0 = 1.166 \text{ g/mL} \approx 1.17 \text{ g/mL}$$

**E.4.10** The soluble  $\text{NaCl}$  concentration of the aqueous-phase in brine, expressed in milligrams per liter [see 12.4.7, Equation (52)]:

$$c_{\text{NaCl}, \text{Aq}} = 10,000 \times 22.5 \times 1.166 = 262,350 \text{ mg/L}$$

For this example, all of the NaCl is soluble. However, if some insoluble sodium chloride was present, the soluble and insoluble concentrations in the whole-NADF could be determined using the calculations shown in 12.5.

**E.4.11** Volume fraction (as a percentage) of brine in whole-NADF [see 12.6.1, Equation (59)]:

$$\varphi_B = \frac{100 \times 25.0}{1.166 \times [100 - (22.5 + 0)]} = 27.7 \%$$

**E.4.12** Volume fraction (as percent) corrected solids in whole-NADF [see 12.6.1, Equation (60)]:

$$\varphi_{DS} = 100 - (59.5 + 27.7) = 12.8 \%$$

**E.4.13** NAF and water ratio (volume fraction) calculations [see 12.6.2, Equation (61) and Equation (62)]:

$$R_{NAF-W} = \frac{100 \times 59.5}{59.5 + 25.0} = 70.4 \% \text{ NAF}$$

$$R_W = \frac{100 \times 25.0}{59.5 + 25.0} = 29.6 \%$$

then,

$$R_{NAF-W} : R_W = 70.4 : 29.6$$

NOTE  $R_{NAF-W} : R_W$  is the NAF-to-water ratio, often denoted as OWR. For this example, the OWR is normally rounded to 70:30.

**E.4.14** NAF and aqueous-phase (brine) ratio (volume fraction) calculations [see 12.6.3, Equation (63) and Equation (64)]:

$$R_{NAF-B} = \frac{100 \times 59.5}{59.5 + 27.7} = 68.2 \% \text{ NAF}$$

$$R_B = \frac{100 \times 27.7}{59.5 + 27.7} = 31.8 \% \text{ brine}$$

then,

$$R_{NAF-B} : R_B = 68.2 : 31.8$$

NOTE  $R_{NAF-B} : R_B$  is the NAF-to-brine ratio, often denoted as OBR. For this example, the OBR is normally rounded to 68:32.

**E.4.15** Average density of suspended solids [see 12.6.4.2, Equation (65)]:

$$\rho_{SS} = \frac{(100 \times 1.32) - (59.5 \times 0.84) - (27.7 \times 1.166)}{12.8} = 3.88 \text{ g/mL}$$

**E.4.16** Volume fraction (as a percentage) of weighting-material [see 12.6.5.1, Equation (66)]:

$$\phi_{WM} = \frac{3.88 - 2.65}{4.20 - 2.65} \times 12.8 = 10.2 \%$$

**E.4.17** Volume fraction (as percent) low-gravity solids [see 12.6.5.2, Equation (67)]:

$$\phi_{LG} = 12.8 - 10.2 = 2.6 \% \text{ low-gravity solids}$$

**E.4.18** Mass concentration of weighting-material [see 12.6.5.3, Equation (68) and Equation (66)]:

$$c_{WM,SI} = 10 \times 4.20 \times 10.2 = 428 \text{ kg/m}^3$$

$$c_{WM,USC} = 3.5 \times 4.20 \times 10.2 = 150 \text{ lb/bbl}$$

**E.4.19** Mass concentration of low-gravity solids [see 12.6.5.4, Equation (69) and Equation (70)]:

$$c_{LG,SI} = 10 \times 2.65 \times 2.6 = 69 \text{ kg/m}^3$$

$$c_{LG,USC} = 3.5 \times 2.65 \times 2.6 = 24 \text{ lb/bbl}$$

## **E.5 NADF Containing Both CaCl<sub>2</sub> and NaCl Salts (Drilling Fluid C)**

**E.5.1** Whole-NADF chloride ion concentration [see 10.5.2, Equation (27)]:

$$c_{Cl,df} = 10,000 \times 7.0 = 70,000 \text{ mg/L of drilling fluid}$$

**E.5.2** Whole-NADF calcium concentration [see 10.6.2, Equation (28)]:

$$c_{Ca,df} = 4000 \times 8.4 = 33,600 \text{ mg/L of drilling fluid}$$

**E.5.3** Whole-NADF lime concentration [see 12.3.1, Equation (29) and Equation (30)]:

$$c_{Ca(OH)_2,df,SI} = 3.705 \times 2.6 = 9.6 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{Ca(OH)_2,df,USC} = 1.299 \times 2.6 = 3.4 \text{ lb/bbl of drilling fluid}$$

**E.5.4** Whole-NADF chloride ion concentration as CaCl<sub>2</sub>, from calcium analysis [see 12.3.2.2, Equation (33)]:

$$c_{Cl-(CaCl_2),df} = 1.769 \times 33,600 = 59,438 \text{ mg/L} \approx 59,440 \text{ mg/L of drilling fluid}$$

**E.5.5** Whole-NADF chloride ion concentration as NaCl [see 12.3.2.3, Equation (34)]:

$$c_{Cl-(NaCl),df} = 70,000 - 59,438 = 10,562 \text{ mg/L} \approx 10,560 \text{ mg/L of drilling fluid}$$

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

**E.5.6** Whole calcium chloride concentration [see 12.3.2.5, Equation (38), Equation (39), and Equation (40)]:

$$c_{\text{CaCl}_2, \text{df}} = 2.769 \times 33,600 = 93,038 \text{ mg/L} \approx 93,040 \text{ mg/L of drilling fluid}$$

$$c_{\text{CaCl}_2, \text{df}, \text{SI}} = 0.002769 \times 33,600 = 93.0 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{CaCl}_2, \text{df}, \text{USC}} = 0.0009707 \times 33,600 = 32.6 \text{ lb/bbl of drilling fluid}$$

**E.5.7** Whole-NADF sodium chloride concentration [see 12.3.2.6, Equation (41), Equation (42), and Equation (43)]:

$$c_{\text{NaCl}, \text{df}} = 1.648 \times 10,562 = 17,406 \text{ mg/L} \approx 17,410 \text{ mg/L of drilling fluid}$$

$$c_{\text{NaCl}, \text{df}, \text{SI}} = 0.001648 \times 10,562 = 17.4 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{NaCl}, \text{df}, \text{USC}} = 0.000578 \times 10,562 = 6.1 \text{ lb/bbl of drilling fluid}$$

**E.5.8** Aqueous-phase mass fraction of calcium chloride, expressed as a percentage [see 12.4.2, Equation (44)]:

$$w_{\text{CaCl}_2} = \frac{100 \times 93,038}{93,038 + 17,406 + (10,000 \times 25.0)} = 25.8 \%$$

**E.5.9** Aqueous-phase mass fraction of sodium chloride, expressed as a percentage [see 12.4.2, Equation (45)]:

$$w_{\text{NaCl}} = \frac{100 \times 17,406}{93,038 + 17,406 + (10,000 \times 25.0)} = 4.8 \%$$

**E.5.10** Since both  $\text{CaCl}_2$  and  $\text{NaCl}$  are present, the maximum mass fraction of soluble  $\text{NaCl}$  that can exist in the aqueous-phase is calculated as follows [see 12.4.3.2, Equation (46)]:

$$w_{\text{NaCl}, \text{Max}} = 26.432 - (1.0472 \times 25.8) + \left[ 7.98191 \times 10^{-3} \times (25.8)^2 \right] + \left[ 5.2238 \times 10^{-5} \times (25.8)^3 \right] = 5.6 \%$$

Since  $w_{\text{NaCl}} < w_{\text{NaCl}, \text{Max}}$ , all  $\text{NaCl}$  is soluble in the aqueous-phase.

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

**E.5.11** Density of the aqueous-phase [see 12.4.5, Equation (50)]:

$$\begin{aligned} \rho_B &= 0.99707 + (0.006504 \times 4.8) + (0.007923 \times 25.8) + (0.00008334 \times 4.8 \times 25.8) \\ &\quad + \left[ 0.00004395 \times (4.8)^2 \right] + \left[ 0.00004965 \times (25.8)^2 \right] = 1.277 \text{ g/mL} \approx 1.28 \text{ g/mL} \end{aligned}$$

**E.5.12** Mass concentration calcium chloride, expressed in milligrams per liter [see 12.4.6, Equation (51)]:

$$c_{\text{CaCl}_2, \text{Aq}} = 10,000 \times 25.8 \times 1.277 = 329,466 \text{ mg/L} \approx 329,470 \text{ mg/L}$$

**E.5.13** Mass concentration sodium chloride, expressed in milligrams per liter [see 12.4.7, Equation (52)]:

$$c_{\text{NaCl,Aq}} = 10,000 \times 4.8 \times 1.277 = 61,296 \text{ mg/L} \approx 61,300 \text{ mg/L}$$

**E.5.14** Volume fraction (as a percentage) of brine (aqueous-phase) in whole-NADF [see 12.6.1.1, Equation (59)]:

$$\varphi_B = \frac{100 \times 25.0}{1.277 \times [100 - (25.8 + 4.8)]} = 28.2 \%$$

**E.5.15** Corrected volume fraction (as a percentage) of solids in whole-NADF [see 12.6.1.2, Equation (60)]:

$$\varphi_{DS} = 100 - (59.5 + 28.2) = 12.3 \%$$

**E.5.16** NAF and water ratio (volume fraction) calculations [see 12.6.2, Equation (61) and Equation (62)]:

$$R_{\text{NAF-W}} = \frac{100 \times 59.5}{59.5 + 25.0} = 70.4 \% \text{ NAF}$$

$$R_W = \frac{100 \times 25.0}{59.5 + 25.0} = 29.6 \%$$

$$R_{\text{NAF-W}} : R_W = 70.4 : 29.6$$

NOTE  $R_{\text{NAF-W}} : R_W$  is the NAF-to-water ratio, often denoted as OWR. For this example, the OWR is normally rounded to 70:30.

**E.5.17** NAF and aqueous-phase (brine) ratio (volume fraction) calculations [see 12.6.3, Equation (63) and Equation (64)]:

$$R_{\text{NAF-B}} = \frac{100 \times 59.5}{59.5 + 28.2} = 67.8 \% \text{ NAF}$$

$$R_B = \frac{100 \times 28.2}{59.5 + 28.2} = 32.2 \%$$

$$R_{\text{NAF-B}} : R_B = 67.8 : 32.2$$

NOTE  $R_{\text{NAF-B}} : R_B$  is the NAF-to-brine ratio, often denoted as OBR. For this example, the OBR is normally rounded to 68:32.

**E.5.18** Average density of suspended solids [see 12.6.4.2, Equation (65)]:

$$\rho_{SS} = \frac{(100 \times 1.32) - [(59.5 \times 0.84) + (28.2 \times 1.277)]}{12.3} = 3.74 \text{ g/mL}$$

**E.5.19** Volume fraction (as a percentage) of weighting-material [see 12.6.5.1, Equation (66)]:

$$\varphi_{WM} = \frac{3.74 - 2.65}{4.20 - 2.65} \times 12.3 = 8.6 \%$$

**E.5.20** Volume fraction (as a percentage) of low-gravity solids [see 12.6.5.2, Equation (67)]:

$$\phi_{LG} = 12.3 - 8.6 = 3.7 \% \text{ low-gravity solids}$$

**E.5.21** Mass concentration of weighting-material [see 12.6.5.3, Equation (68) and Equation (69)]:

$$c_{WM,SI} = 10 \times 4.20 \times 8.6 = 361 \text{ kg/m}^3$$

$$c_{WM,USC} = 3.5 \times 4.20 \times 8.6 = 126 \text{ lb/bbl}$$

**E.5.22** Mass concentration of low-gravity solids [see 12.6.5.4, Equation (70) and Equation (71)]:

$$c_{LG,SI} = 10 \times 2.65 \times 3.7 = 98 \text{ kg/m}^3$$

$$c_{LG,USC} = 3.5 \times 2.65 \times 3.7 = 34 \text{ lb/bbl}$$

## **E.6 NAFD Containing Both $\text{CaCl}_2$ and Excess $\text{NaCl}$ Salts (Drilling Fluid D)**

**E.6.1** Whole-NADF chloride ion concentration [see 10.5.2, Equation (27)]:

$$c_{Cl,df} = 10,000 \times 10.0 = 100,000 \text{ mg/L of drilling fluid}$$

**E.6.2** Whole-NADF calcium concentration [see 10.6.2, Equation (28)]:

$$c_{Ca,df} = 4000 \times 8.4 = 33,600 \text{ mg/L of drilling fluid}$$

**E.6.3** Whole-NADF lime concentration [see 12.3.1, Equation (29) and Equation (30)]:

$$c_{Ca(OH)_2,df,SI} = 3.705 \times 3.0 = 11.1 \text{ g/m}^3 \text{ of drilling fluid}$$

$$c_{Ca(OH)_2,df,USC} = 1.299 \times 3.0 = 3.9 \text{ lb/bbl of drilling fluid}$$

**E.6.4** Whole-NADF chloride ion concentration as  $\text{CaCl}_2$ , from calcium analysis [see 12.3.2.2, Equation (33)]:

$$c_{Cl-(CaCl_2),df} = 1.769 \times 33,600 = 59,438 \text{ mg/L} \approx 59,440 \text{ mg/L of drilling fluid}$$

**E.6.5** Whole- chloride ion concentration as  $\text{NaCl}$  [see 12.3.2.3, Equation (34)]:

$$c_{Cl-(NaCl),df} = 100,000 - 59,438 = 40,562 \text{ mg/L} \approx 40,560 \text{ mg/L of drilling fluid}$$

The positive number indicates that  $\text{NaCl}$  is present in this drilling fluid.

**E.6.6** Whole-NADF calcium chloride concentration [see 12.3.2.5, Equation (38), Equation (39), and Equation (40)]:

$$c_{CaCl_2,df} = 2.769 \times 33,600 = 93,038 \text{ mg/L} \approx 93,040 \text{ mg/L of drilling fluid}$$

$$c_{CaCl_2,df,SI} = 0.002769 \times 33,600 = 93.0 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{CaCl_2,df,USC} = 0.0009707 \times 33,600 = 32.6 \text{ lb/bbl of drilling fluid}$$

**E.6.7** Whole-NADF sodium chloride concentration [see 12.3.2.6, Equation (41), Equation (42), and Equation (43)]:

$$c_{\text{NaCl,df}} = 1.648 \times 40,562 = 66,846 \text{ mg/L} \approx 66,850 \text{ mg/L of drilling fluid}$$

$$c_{\text{NaCl,df,SI}} = 0.001648 \times 40,562 = 66.8 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{NaCl,df,USC}} = 0.000577 \times 40,562 = 23.4 \text{ lb/bbl of drilling fluid}$$

**E.6.8** Aqueous-phase mass fraction of calcium chloride, expressed as a percentage [see 12.4.2, Equation (44)]:

$$w_{\text{CaCl}_2} = \frac{100 \times 93,038}{93,038 + 66,846 + (10,000 \times 25.0)} = 22.7 \%$$

**E.6.9** Aqueous-phase mass fraction of sodium chloride, expressed as a percentage [see 12.4.2, Equation (45)]:

$$w_{\text{NaCl}} = \frac{100 \times 66,846}{93,038 + 66,846 + (10,000 \times 25.0)} = 16.3 \%$$

**E.6.10** Since both  $\text{CaCl}_2$  and  $\text{NaCl}$  are present, the maximum mass fraction of soluble  $\text{NaCl}$  that can exist in the aqueous-phase is calculated as follows [see 12.4.3.2, Equation (46)]:

$$w_{\text{NaCl,Max}} = 26.432 - (1.0472 \times 22.7) + \left[ 7.98191 \times 10^{-3} \times (22.7)^2 \right] + \left[ 5.2238 \times 10^{-5} \times (22.7)^3 \right] = 7.4 \%$$

From the calculations,  $w_{\text{NaCl}} > w_{\text{NaCl,Max}}$ , the aqueous-phase is super-saturated and insoluble  $\text{NaCl}$  is present (Figure 1, Region 2). For this situation the mass fraction of both salts,  $w_{\text{CaCl}_2}$  from Equation (43) and  $w_{\text{NaCl}}$  from Equation (44), must be recalculated using Equation (47) and (48) then the  $w_{\text{NaCl,Max}}$  must be recalculated with Equation 46 as follows.

**E.6.11** Calcium concentration expressed per volume of pure water in the aqueous-phase [see 12.4.3.5, Equation (47)]:

$$c_{\text{Ca,Aq}} = \frac{100 \times 33,600}{25.0} = 134,400 \text{ mg / L}$$

**E.6.12** For super-saturated fluids, the actual mass fraction of calcium chloride,  $w_{\text{CaCl}_2}$ , in the aqueous-phase is calculated [see 12.4.3.6, Equation (48)]:

$$w_{\text{CaCl}_2,\text{Sat}} = \left[ 134,000^2 \times (-2.4708 \times 10^{-10}) \right] + \left[ 134,000 \times (2.2567 \times 10^{-4}) \right] - (2.1348 \times 10^{-1}) = 25.7 \%$$

Use the actual  $w_{\text{CaCl}_2,\text{Sat}}$  for  $w_{\text{CaCl}_2}$  in the remaining calculations or fluids with super-saturated aqueous-phases.

**E.6.13** Since this fluid is super-saturated, the correct mass fraction of NaCl in the aqueous-phase is  $w_{\text{NaCl,Max-Cal}}$  which must be recalculated as described in 12.4.3.5 using Equation (46) but using  $w_{\text{CaCl}_2,\text{Sat}}$  for  $w_{\text{CaCl}_2}$ .

$$w_{\text{NaCl,Max-Cal}} = 26.432 - (1.0472 \times 25.7) + \left[ 7.98191 \times 10^{-3} \times (25.7)^2 \right] + \left[ 5.2238 \times 10^{-5} \times (25.7)^3 \right] = 5.7 \%$$

Use  $w_{\text{CaCl}_2,\text{Sat}}$  for  $w_{\text{CaCl}_2}$  and the recalculated  $w_{\text{NaCl,Max-Cal}}$  for  $w_{\text{NaCl}}$  in the remaining calculations for fluids with super-saturated aqueous-phases.

**E.6.14** Density of the aqueous-phase brine [see 12.4.5, Equation (50)]:

$$\rho_B = 0.99707 + (0.006504 \times 5.7) + (0.007923 \times 25.7) + (0.00008334 \times 5.7 \times 25.7) + \left[ 0.00004395 \times (5.7)^2 \right] + \left[ 0.00004964 \times (25.7)^2 \right] = 1.284 \text{ g / mL} \approx 1.28 \text{ g / mL}$$

**E.6.15** Mass concentration of calcium chloride in the aqueous-phase, expressed in milligrams per liter [see 12.4.6, Equation (51)]:

$$c_{\text{CaCl}_2,\text{Aq}} = 10,000 \times 25.7 \times 1.284 = 329,988 \text{ mg/L} \approx 329,990 \text{ mg/L}$$

**E.6.16** Mass concentration of sodium chloride in the aqueous-phase, expressed in milligrams per liter [see 12.4.7, Equation (52)]:

$$c_{\text{NaCl,Aq}} = 10,000 \times 5.7 \times 1.284 = 73,188 \text{ mg/L} \approx 73,190 \text{ mg/L}$$

**E.6.17** The soluble NaCl content of the whole-NADF is calculated [see 12.5.2, Equation (53)]:

$$c_{\text{NaCl,df,Sol}} = \frac{5.7 \times [93,038 + (10,000 \times 25.0)]}{100 - 5.7} = 20,735 \text{ mg / L} \approx 20,740 \text{ mg / L of drilling fluid}$$

**E.6.18** The soluble NaCl of the whole-NADF in SI and USC units [see 12.5.3, Equation (54) and Equation (55)]:

$$c_{\text{NaCl,df,Sol,SI}} = 0.001 \times 20,735 = 20.735 \text{ kg / m}^3 \approx 21 \text{ kg / m}^3 \text{ of drilling fluid}$$

$$c_{\text{NaCl,df,Sol,USC}} = 0.00035 \times 20,735 = 7.26 \text{ lb / bbl} \approx 7.3 \text{ lb / bbl of drilling fluid}$$

**E.6.19** The concentration of insoluble NaCl in the whole-NADF is calculated [see 12.5.4, Equation (56), Equation (57) and Equation (58)]:

$$c_{\text{NaCl,df,InSol}} = 66,836 - 20,735 = 46,101 \text{ mg/L} \approx 46,100 \text{ mg/L of drilling fluid}$$

$$c_{\text{NaCl,df,InSol,SI}} = 0.001 \times 46,101 \approx 46.1 \text{ kg/m}^3 \text{ of drilling fluid}$$

$$c_{\text{NaCl,df,InSol,USC}} = 23.4 - 7.3 = 16.1 \text{ lb/bbl of drilling fluid}$$

**E.6.20** Volume fraction of aqueous-phase (brine) in whole-NADF, expressed as a percentage [see 12.6.1.1, Equation (59)]:

$$\varphi_B = \frac{100 \times 25.0}{1.284 \times [100 - (25.7 + 5.7)]} = 28.4 \%$$

**E.6.21** Volume fraction of corrected solids in whole-NADF, expressed as a percentage [see 12.6.1.2, Equation (60)]:

$$\varphi_{DS} = 100 - (59.5 + 28.4) = 12.1 \%$$

**E.6.22** NAF and water ratio (volume fraction) calculations [see 12.6.2, Equation (61) and Equation (62)]:

$$R_{NAF-W} = \frac{100 \times 59.5}{59.5 + 25.0} = 70.4 \% \text{ NAF}$$

$$R_W = \frac{100 \times 25.0}{59.5 + 25.0} = 29.6 \%$$

then

$$R_{NAF-W} : R_W = 70.4 : 29.6$$

NOTE  $R_{NAF-W} : R_W$  is the NAF-to-water ratio, often denoted as OWR. For this example, the OWR is normally rounded to 70:30.

**E.6.23** NAF and aqueous-phase (brine) ratio (volume fraction) calculations [see 12.6.3, Equation (63) and Equation (64)]:

$$R_{NAF-B} = \frac{100 \times 59.5}{59.5 + 28.4} = 67.7 \% \text{ NAF}$$

$$R_B = \frac{100 \times 28.4}{59.5 + 28.4} = 32.3 \%$$

then

$$R_{NAF-B} : R_B = 67.7 : 32.3$$

NOTE  $R_{NAF-B} : R_B$  is the NAF-to-brine ratio, often denoted as OBR. For this example, the OBR is normally rounded to 68:32.

**E.6.24** Average density of suspended solids [see 12.6.4.2, Equation (65)]:

$$\rho_{SS} = \frac{(100 \times 1.32) - (59.5 \times 0.84) - (28.4 \times 1.284)}{12.1} = 3.76 \text{ g/mL}$$

**E.6.25** Volume fraction of weighting-material, expressed as a percentage [see 12.6.5.1, Equation (66)]:

$$\varphi_{WM} = \frac{3.76 - 2.65}{4.20 - 2.65} \times 12.1 = 8.7 \%$$

**E.6.26** Volume fraction of low-gravity solids, expressed as a percentage [see 12.6.5.2, Equation (67)]:

---

$$\phi_{LG} = 12.1 - 8.7 = 3.4 \% \text{ low-gravity solids}$$

**E.6.27** Mass concentration of weighting-material [see 12.6.5.3, Equation (68) and Equation (69)]:

$$c_{WM,SI} = 10 \times 4.20 \times 8.7 = 365 \text{ kg/m}^3$$

$$c_{WM,USC} = 3.5 \times 4.20 \times 8.7 = 128 \text{ lb/bbl}$$

**E.6.28** Mass concentration of low-gravity solids [see 12.6.5.4, Equation (70) and Equation (71)]:

$$c_{LG,SI} = 10 \times 2.65 \times 3.4 = 90 \text{ kg/m}^3$$

$$c_{LG,USC} = 3.5 \times 2.65 \times 3.4 = 32 \text{ lb/bbl}$$

BALLOT DRAFT

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## **Annex F** **(informative)**

### **Sampling, Inspection, and Rejection of Drilling Materials**

#### **F.1 Principle**

The procedure described in this annex provides sampling, inspection, and rejection methods for drilling fluid materials conforming to API 13A. It is applicable to barite (both 4.1 and 4.2 grades), hematite, bentonite, non-treated bentonite, OCMA-bentonite <sup>7</sup>, attapulgite, sepiolite, technical-grade low-viscosity carboxymethylcellulose, technical-grade high-viscosity carboxymethylcellulose, starch, low-viscosity polyanionic cellulose, high-viscosity polyanionic cellulose, and drilling-grade xanthan gum as well as other powdered products.

#### **F.2 Sampling of Powdered Material in Packages**

Procedure to prepare sample of powdered material in packages should be as per the following.

- a) Take 15 or more samples of 0.5 kg (1 lb) per lot to be combined as the test sample (see F.4).
- b) For each lot of 1,000 packages or less, 15 packages should be sampled.
- c) The sampling should be carried out by either of the following methods as agreed upon by the contracting parties:
  1. a sample weighing at least 0.5 kg (1 lb) should be taken from the top of each package, or
  2. a sampling tube capable of taking a core not less than 2.5 cm (1 in.) in diameter should be used. The tube should be inserted into the package being sampled so that it will take a core of material for essentially the entire length of the package.

##### **F.2.1 Sampling Powdered Material in Bulk**

Procedure to prepare sample of powdered material in bulk should be as per the following.

- a) A sampling tube capable of extracting a core of a diameter no less than 2.5 cm (1 in.) should be used to collect samples from storage containers holding 25,000 kg to 100,000 kg (25 metric tons to 100 metric tons) (25 long tons to 98 long tons). The tube should be of sufficient length to permit taking a substantial sample from the top to the bottom of the container of material being sampled.

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<sup>7</sup> Oil Company Materials Association (now-defunct organization but an OCMA grade bentonite specification is included in API 13A)

- b) Fifteen samples should be taken from each lot (container). If the dimensions of the container are such that the sample cannot be taken in this manner, the sample should be taken by a method agreed upon by the contracting parties.
- c) Take at least one sample from each container holding less than 20,000 kg (20 metric tons) (20 long tons), up to lots of 10 containers. The total number of samples taken from each lot of 100,000 kg (100 metric tons) (98 long tons) or less should not be fewer than 10.

### **F.3 Preparation of Test Sample**

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

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

### **F.4 Test**

Each party should make measurements on the allocated test sample in accordance with the specific test procedure(s) outlined in API 13A and 13I or other quality assurance procedure.

### **F.5 Inspection**

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

### **F.6 Rejection**

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

### **F.7 Retesting**

**F.7.1** Either of the contracting parties may request a retest within one week of the date of the original test report.

**F.7.2** Should the contracting parties be unable to reach an agreement, the third sample of material should be delivered, unopened, to a mutually satisfactory referee laboratory for tests, and the results of this referee test shall be binding on both parties.

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## **Annex G** **(informative)**

### **Rig-site Sampling**

#### **G.1 Principle**

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

#### **G.2 Bottom-flow Sampling (preferred)**

##### **G.2.1 Apparatus**

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

**G.2.1.2 Canvas bag**, approximately 14 cm (5.5 in.) in diameter and 30 cm (12 in.) long.

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

##### **G.2.2 Procedure**

Rig-sampling using a side-stream sampler should be as per the following procedure.

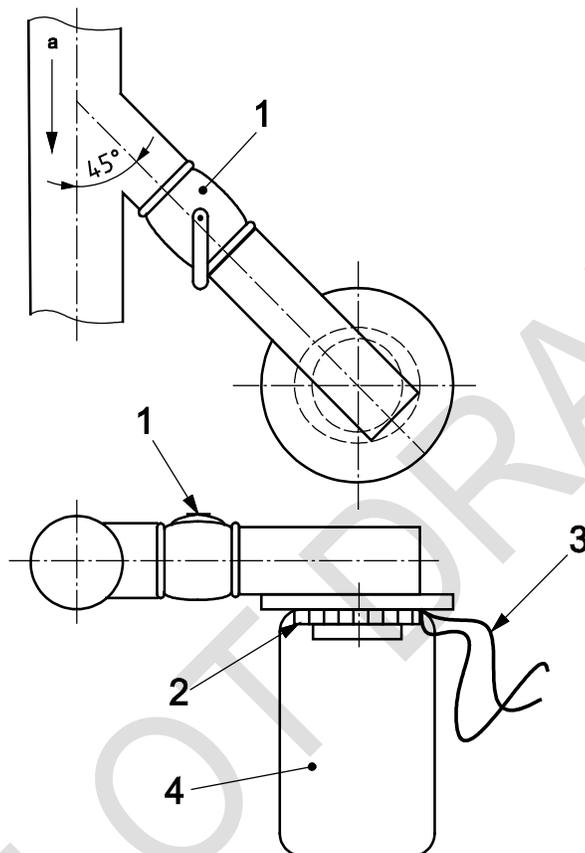
- a) Connect a side-stream sampler to the truck outlet.
- b) Take three 1.5 kg to 2.5 kg (3 lb to 5 lb) samples while unloading, spaced so they are taken when approximately  $\frac{1}{4}$ ,  $\frac{1}{2}$ , and  $\frac{3}{4}$  of the load has been transferred.

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

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

**NOTE** Partial closing of the air-jet valve on the bottom outlet prior to taking the sample often helps 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 all pertinent information.
- e) Empty the canvas bag before taking the next sample. Always use a clean or new canvas bag for each container.



**Key**

- 1 5 cm (2 in.) ball valve in 5 cm (2 in.) side pipe
- 2 bead or lip to hold bag
- 3 draw string
- 4 canvas bag 14 cm × 30 cm (5.5 in. × 12 in.)
- a Flow direction.

**Figure G.1—Side-stream Sampling Device**

### **G.3 Grab Sampling**

#### **G.3.1 Apparatus**

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

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

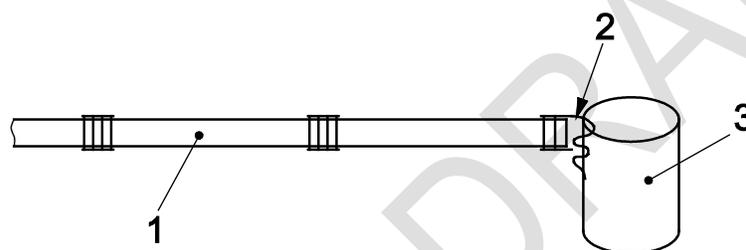
### G.3.2 Procedure

Grab sampling using a sample scoop should be done as per the following procedure.

- a) Open the top hatch carefully. Do not let the cover bump the top of the tank.

**Warning—Prior to opening the hatch to avoid injury or death, be sure all pressure is removed from 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.
- 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 G.2—Sample Scoop**

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## Annex H (informative)

### Determination of Cuttings Activity by the Chenevert Method

#### H.1 Principle

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

**H.1.2** This procedure provides specific directions on sample collection, handling and washing. It provides instructions for testing cuttings using the electro-hygrometer. It closely parallels the procedure in Annex C for measuring the activity of a NADF and allows for a reliable comparison between activity for a NADF and activity for the cuttings from the same well.

**H.1.3** Activities of cuttings measured at the surface by this method is not representative to the activity of the cuttings from downhole formation due to many factors. The activity of a cutting sample is altered due to changes in temperature, pressure, and stress state as it moves from the wellbore to the surface. In addition, a significant difference between the drilling fluid and the cuttings activity causes the cuttings activity to approach that of the NADF. Measured trends of NADF and cuttings activities from the same well will, in most cases, allow one to evaluate which has the greater or lesser activity value—the cuttings or the NADF.

#### H.2 Reagents and Apparatus

##### H.2.1 Standard Saturated Salt Solutions

**H.2.1.1** The activity of standard saturated salt solutions is described in Table H.1.

**Table H.1—Saturated Salt Solutions**

Salt	CAS No.	Water Activity of Saturated Salt Solution $a_w$ at 25 °C (77 °F)	Salt Concentration g/100 mL water
Calcium chloride	10043-52-4	0.295	100
Calcium nitrate	13477-34-4	0.505	200
Sodium chloride	7647-14-5	0.753	200
Potassium nitrate	7757-79-1	0.938	200

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

**H.2.1.3** If no crystals are visible, some additional crystals or grains of the same salt should be added to induce precipitation. This is often necessary with calcium chloride. Salts of reagent-grade purity are recommended. However, if necessary, field-grade NaCl or CaCl<sub>2</sub> can be substituted.

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

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

- a) The relative humidity sensor probe should be suitable for insertion in the air space of a measuring container with the sample.
- b) The linearity response of the instrument should be such that the correlation coefficient between published standard brine activity and relative humidity, as measured by the instrument above each brine, is greater than 0.95.
- c) The instrument should give a reading within 10 % of the true activity within 30 min, when measuring the brine activity of the standard salt solutions in H.2.1.

**NOTE** This procedure is for using an electro-hygrometer only. Chilled mirror and capacitive sensor instruments are available which can also measure water activity. Capacitive sensor instruments use capacitance sensors to measure water activity. Chilled mirror instruments use a chilled mirror to measure the dew point temperature (a measure of relative humidity) of air equilibrated with a sample and then convert the dew point temperature to vapor pressure. Chilled mirror devices are common in other industries and increasing used in drilling fluid applications.

**H.2.4 Non-aqueous fluid supply**, 1.0 L to 1.5 L (0.25 gal to 0.4 gal), clean, water-free.

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

**H.2.5 Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) accurate to ± 1 °C (± 2 °F), not required if instrument has an internal thermometer.

**H.2.6 Measuring container**, as suggested or supplied by the manufacturer, having a volume sufficient to provide a liquid surface area of approximately 30 cm<sup>2</sup> to 50 cm<sup>2</sup> (4.5 in.<sup>2</sup> to 7.75 in.<sup>2</sup>). The container should have a flexible elastomer cap (airtight) which, when bored through, will form an airtight seal around the shaft of the sensor probe and thermometer (if used).

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

**H.2.8 Linear graph paper.**

**H.2.9 Paper towels**, one or more rolls.

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

**H.2.11 Sieve**, with openings of 0.589 mm (28 mesh).

**H.2.12 Graduated beaker**, 400 mL.

### **H.3 Procedure**

#### **H.3.1 Calibration of Hygrometer**

Hygrometer should be calibrated as per the following procedure.

- a) Prepare sufficient volume of each known relative humidity standard to cover the bottom of the measuring container to a liquid depth of 12 mm (0.5 in.) or follow the manufacturer's recommendations. These salt solutions shall be kept firmly capped to prevent spillage, evaporation, or contamination.
- b) Desiccate the probe with anhydrous calcium chloride in a closed sample container (other desiccants may be used) by placing the sensor probe (fitted in the sample container cap) in the air space above the salt. Wait 10 min to 15 min. A relative humidity reading of 24 % or lower indicates that the probe is dry. Keep a distance of 12 mm (0.5 in.) between the sensing probe and the desiccant and maintain the same distance for salt solutions and cuttings samples. Never allow the probe to directly contact any samples or solutions.
- c) Transfer the probe in the measuring container cap to the first (lowest  $a_W$ ) standard solution and allow 30 min for equilibrium to be reached. Temperature of the standards shall be maintained between 24 °C and 25 °C (75 °F and 77 °F).
- d) Remove the probe from the first solution and place it into the container with the solution of second lowest known activity.
- e) Proceed as in H.3.1 item c) and measure the relative humidity for each solution, proceeding from that with the lowest to that with the highest activity  $a_W$ .
- f) Allow each standard to equilibrate for the same length of time. Record the temperature and percent relative humidity ( $RH_{\%}$ ) indicated. Plot the  $RH_{\%}$  versus the activity of each solution,  $a_W$ .

#### **H.3.2 Measurement of NAF-contaminated Cuttings Activity, $a_C$**

In order to determine the activity of NAF-contaminated cuttings, the following procedure should be applied.

- a) Desiccate the probe for 10 min to 15 min as in H.3.1 item b).
- b) Collect a fresh sample of cuttings directly from the final stage solids control equipment, taking care to minimize its exposure to air. Minimize the amount of NADF adhering to the cuttings.
- c) Add 300 mL of room-temperature base NAF to the 500 mL washing bottle.

- d) Measure approximately 100 mL of loosely packed, fresh cuttings [H.3.2. item b)] into the 400 mL graduated beaker. Transfer these to the bottle containing the base NAF and fasten the lid.
- e) Gently shake the bottle by hand for 30 seconds.
- f) Remove the lid and place the sieve over the mouth of the bottle. Pour off and discard the wash NAF, retaining the cuttings in the bottle.
- g) Again, add 300 mL of base NAF to the bottle containing the same cuttings and repeat procedure H.3.2. item e) and item f). Continue these washing steps until the NAF looks clean after shaking with the cuttings (usually two to four washings).
- h) Drain off the NAF after washing the cuttings. Pour the cuttings onto three to four layers of absorbent paper towel. Fold the towels over the cuttings and gently blot them dry. Minimize exposure of the cuttings to the open air.
- i) Quickly transfer 35 to 45 ml of the washed cuttings into the measuring container. Place the measuring container into the insulating foam jacket or constant-temperature water bath if one is used. Install the cap.
- j) Replace the container lid with the lid that has the pre-calibrated hygrometer probe inserted. Ensure that the probe is inserted into the air space 12 mm (0.5 in.) above the cuttings and does not contact the cuttings. The cuttings sample temperature should be maintained between 24 °C and 25 °C (75 °F and 77 °F).
- k) Turn on the hygrometer and wait 30 min (the same time period used for standard calibration), and record the percent relative humidity and temperature.

Note: For some samples, longer periods of time are needed for the hygrometer reading to stabilize. For situations where the hygrometer reading has not converged to a consistent reading at 30 min, continue monitoring the reading, recording the water activity reading every 15 min until it is reasonable constant.

#### **H.4 Calculation—Cuttings Activity, $a_C$**

**H.4.1** Find the relative humidity obtained in H.3.2 on the graph plotted in H.3.1.item f). Read and record the corresponding activity that appears on the graph as the cuttings activity,  $a_C$ .

**H.4.2** If not measured directly, these plotting instructions should be followed:

- a) using data for standards given in H.3.1, plot  $RH_{\%}$  versus  $a_W$  on linear graph paper;
- b) plot the data obtained for each sample (H.3.2) on the linear graph paper and read the cuttings activity,  $a_C$ .

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## **Annex I** (normative)

### **Chemical Analysis of Active Sulfides by the Garrett Gas Train Method**

#### **I.1 Principle**

**I.1.1** This procedure uses the Garrett gas train to measure active sulfides in a NADF. The gas train separates gas from a liquid sample and passes the gas through a hydrogen sulfide detector, such as lead acetate paper for qualitative sulfide analysis or a Dräger tube or equivalent, for quantitative sulfide analysis. Lead acetate paper turns black when the lead acetate reacts with sulfides. If sulfides are detected using the lead acetate paper, a Dräger tube<sup>8</sup> or equivalent shall be used for quantitative analysis. The Dräger tube or its equivalent responds to H<sub>2</sub>S by progressively darkening along its length as H<sub>2</sub>S reacts with the tube reagent. Two Dräger tubes cover a wide range of sulfide concentrations, from approximately 1 mg/L to 3700 mg/L. The low-range tube is white until H<sub>2</sub>S turns it brownish black. The high-range tube is pale blue until H<sub>2</sub>S turns it black. No known common drilling fluid component or contaminant other than H<sub>2</sub>S causes this discoloration in Dräger tubes.

**I.1.2** Whole-NADF, not filtrate, is analyzed in order to capture all of the active sulfides. Active sulfides are typically the neutralization products of H<sub>2</sub>S and lime, plus any non-reacted H<sub>2</sub>S gas. Use of a weak acid in the gas train liberates H<sub>2</sub>S gas from active sulfides, but not from inert sulfides such as zinc sulfide, copper sulfide or lead sulfide.

#### **I.2 Reagents and Apparatus**

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

NOTE Suitable solutions are sold premixed by several oilfield testing chemical suppliers. One suitable demulsifier product is based on ±50% vol. dodecylbenzenesulphonic acid (CAS No. 27176-87-0), ±25% ethoxylated nonylphenol (CAS No. 9016-45-9), and ±25% methyl alcohol (CAS No. 67-56-1).

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

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

**I.2.4 Carrier gas**, inert to hydrogen sulfide, sulfide salts, citric acid, demulsifier, isopropanol and Dräger tube reagents.

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<sup>8</sup> Dräger tube is an example of a suitable product available commercially. This information is given for the convenience of users of this part of API 13B-2 and does not constitute an endorsement by API of this product.

Nitrogen is preferred but carbon dioxide is acceptable. Avoid air, other oxygen-containing gases, or nitrous oxide cartridges; they shall be only used for Garret gas train carbonate analysis.

**I.2.5 H<sub>2</sub>S gas detection tubes**, Dräger analysis tubes as specified in Table I.1 or equivalent .

**Table I.1—Dräger H<sub>2</sub>S Analysis Tubes**

Sulfide Tube Range	Tube Marking	Tube Part Number
Low-range tube	H <sub>2</sub> S 100/a	No. CH29101
High-range tube	H <sub>2</sub> S 0.2 %/A	No. CH28101

**I.2.6 Garrett gas train apparatus**, consisting of a gas train of transparent material or glass, an inert gas supply and pressure regulator, a floating-ball flow meter, and a H<sub>2</sub>S gas analysis tube.

The components shall have the following characteristics:

a) Body:

— Chamber 1:

depth 90 mm (3.54 in.)

diameter 39 mm (1.52 in.)

— Chambers 2 and 3:

depth 90 mm (3.54 in.)

diameter 30 mm (1.18 in.)

— Passages between chambers:

diameter 2.0 mm (0.08 in.)

— Material:

transparent material or glass which is inert to acid, sulfides and hydrogen sulfide gas.

b) Dispersion tube:

— Stem:

diameter 8.0 mm (0.315 in.)

length 150 mm (5.9 in.)

— Dispersion frit (bell-shaped, fine):

diameter 30 mm (1.18 in.)

— Material:

low coefficient of expansion, heat-resistant glass.

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

**I.2.7 Syringe**, 10 mL, constructed of glass or plastic that is inert to non-aqueous based drilling fluid and sulfides.

**I.2.8 Graduated cylinder**, 25 mL.

**I.2.9 Magnetic stirrer**, electric.

**I.2.10 Stirring bar**, 2.5 cm (1 in.) stirring bar, coated with glass or polytetrafluoroethylene (PTFE).

**I.2.11 Sample injection tube**, with 15 mm (0.6 in.) needle of PTFE or equivalent plastic that is inert to NAF and sulfides.

**I.2.12 Soft brush**.

**I.2.13 Pipe cleaners**.

### **I.3 Procedure**

In order to determine active sulfides using the Garrett gas train, the following procedure shall be applied.

- a) Follow the manufacturer's instructions for assembly and operation of Garrett gas train equipment.
- b) Pass the injection tube through the rubber septum on top of Chamber 1, making sure it seals in the septum.
- c) Set the gas train body, with top removed, on the magnetic stirrer with the center of Chamber 1 over the center of the stirrer. Place the stirring bar in Chamber 1. Remove the rubber feet from the gas train body so it will rest flat on the stirrer.
- d) Add 20 mL of the citric acid/demulsifier—isopropanol solution (I.2.1) into Chamber 1.
- e) Add 10 drops of antifoam into Chamber 1.
- f) See Table I.2 for sample volume and type of Dräger tube or equivalent required for the expected range of sulfide. Select the proper type of analysis tube. Break the tip off each end of the tube.
- g) Insert the Dräger or equivalent tube, with the arrow pointing downward, into the bored receptacle. Likewise, insert the flow meter tube, with “TOP” upward. Ensure the O-rings seal around the body of each tube.
- h) Place the top on the gas train and hand-tighten all screws evenly to seal the O-rings. Position the dispersion tube high enough in Chamber 1 to be above the liquid level.

- i) Connect the regulated gas supply, by a flexible hose, to the top of the dispersion tube of Chamber 1.
- j) Attach the flexible tubing from the Chamber 3 outlet to the Dräger tube or equivalent.

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

- k) Use a syringe without a needle to withdraw the whole-NADF sample volume, as determined in I.3. item f), plus 0.5 mL of excess drilling fluid.

NOTE The excess drilling fluid compensates for the hold-up volume inside the injection tube, which is approximately 0.5 mL.

- l) Attach the syringe to the injection tube and restrain the plunger, e.g. with a rubber band, to keep it from being blown out under pressure.
- m) Allow the carrier gas to flow for 15 seconds, while keeping the dispersion tube frit above the liquid level in Chamber 1, to purge air from the gas train. Avoid frothing of the solution in Chamber 1. With the carrier gas flowing, check the gas train for leaks. Stop the gas flow.
- n) Turn on the magnetic stirrer. Adjust its speed until a vortex is formed. Carefully lower the gas dispersion tube into the liquid to a point just above the rotating stirring bar.
- o) Lower the injection tube until the outlet is slightly below the dispersion tube. This will allow the NADF to enter the vortex.
- p) Inject the sample from the syringe very slowly into Chamber 1. Increase the stirrer speed to improve dispersion and to prevent the non-aqueous based drilling fluid from sticking to the walls of Chamber 1. Continue stirring for about 1 min.
- q) Restrain the syringe plunger again and restart carrier gas flow. Adjust the flow rate to between 200 mL/min and 400 mL/min by keeping the flow-meter ball between the marks.

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

- r) Observe changes in appearance of the Dräger or equivalent tube. Note and record the maximum darkened length (in units marked on the tube) before the front starts to smear. Continue the carrier gas flow for a total of 15 min, although the front may attain a diffuse and feathery coloration. In the high-range tube, an orange color, caused by sulfur dioxide (SO<sub>2</sub>), may appear ahead of the black front if sulfites are present in the sample. The orange SO<sub>2</sub> region shall be ignored when recording darkened length.

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

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

**Table I.2—Dräger Tube (or Equivalent) Identification, Sample Volume, and Tube Factors to be Used for Various Sulfide Concentration Ranges**

Sulfide Concentration Range mg/L	NADF Sample Volume $V_{df}$ mL	Dräger Tube Identification (see tube body)	Tube Factor <sup>a</sup> $f_{st}$ (used in calculation)
1.2 to 24	10.0	H <sub>2</sub> S 100/a	0.12
2.4 to 48	5.0	H <sub>2</sub> S 100/a	0.12
4.8 to 96	2.5	H <sub>2</sub> S 100/a	0.12
30 to 1050	10.0	H <sub>2</sub> S 0.2 %/A	1500
60 to 2100	5.0	H <sub>2</sub> S 0.2 %/A	1500
120 to 4200	2.5	H <sub>2</sub> S 0.2 %/A	1500

<sup>a</sup> If other tubes are used, the tube factors in column 4 shall be changed according to manufacturer's specifications.

## I.4 Calculation

Using the measured drilling fluid sample volume,  $V_{df}$ , in milliliters, the Dräger tube's maximum darkened length,  $l_{st}$ , in units marked on the tube, and the tube factor,  $f_{st}$ , from Table I.2, the mass concentration of active sulfides in the drilling fluid sample shall be calculated using Equation (I.1).

$$c_S = \frac{l_{st} \times f_{st}}{V_{df}} \quad (I.1)$$

where

$c_S$  concentration of active sulfides, expressed in milligrams per liter;

$V_{df}$  volume of the whole-NADF sample, expressed in milliliters;

$l_{st}$  darkened length of the Dräger tube (or equivalent), marked in units on the tube;

$f_{st}$  Dräger tube factor, taken from Table I.2.

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## **Annex J** (normative)

### **Calibration and Verification of Glassware, Thermometers, Viscometers, Retort Kit Cup, and Drilling Fluid Balances**

#### **J.1 Principle**

This annex provides methods for the calibration of field testing equipment not covered under test procedures in this standard.

#### **J.2 Calibration of Apparatus**

##### **J.2.1 Volumetric Glassware**

Calibration of pipettes, graduated cylinders, and similar equipment shall be normally performed by the glassware manufacturer and is part product specifications. Glassware users shall obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically. Periodic recalibration should be not required. A procedure for calibration of graduated cylinders is given in J.3.

##### **J.2.2 Thermometers**

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

##### **J.2.3 Direct- indicating Viscometers**

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

##### **J.2.4 Drilling Fluid Balances**

Drilling fluid balances shall be calibrated using fresh or deionized water as specified in the procedure given in 5.3 and 6.3. The mud balance shall be calibrated frequently. The recommended frequency is prior to each set of measurements (e.g. daily or by each work shift) or as specified by the operator, prevailing regulation, drilling

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

### J.2.5 Retort Cups

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

### J.3 Procedure—Calibration of Graduated Cylinders

In order to calibrate graduated cylinders (receivers), the following procedure shall be used.

- a) Allow the receiver and distilled water to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).
- b) Place the clean, empty receiver, with its base, on the balance and tare to zero.
- c) Fill the receiver with deionized or distilled water, while it is on the balance, to the various graduation marks in increments of 2 mL for the 10 mL receiver, in increments of 4 mL for the 20 mL receiver, and in increments of 10 mL for the 50 mL receiver. Use a pipette or syringe to carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.
- d) Record the masses for each incremental volume of water at the specific graduation mark,  $m_W$ , to the nearest 0.01 g.
- e) Calculate the volume of the receiver at each mark,  $V_M$  using Equation (J.1).

$$V_M = \frac{m_W}{\rho_W} \tag{J.1}$$

where

$V_M$  is the receiver (graduated cylinder) volume at specific mark, expressed in milliliters;

$m_W$  is the mass of water, expressed in grams;

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

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**Table J.1—Density of Water,  $\rho_w$ , as a Function of Temperature**

Temperature °C	Density g/mL	Temperature °F	Density g/mL
15.0	0.9991	59	0.9991
15.5	0.9990	60	0.9990
16.0	0.9989	61	0.9989
16.5	0.9989	62	0.9988
17.0	0.9988	63	0.9987
17.5	0.9987	64	0.9986
18.0	0.9986	65	0.9985
18.5	0.9985	66	0.9984
19.0	0.9984	67	0.9983
19.5	0.9983	68	0.9982
20.0	0.9982	69	0.9981
20.5	0.9981	70	0.9980
21.0	0.9980	71	0.9978
21.5	0.9979	72	0.9977
22.0	0.9978	73	0.9976
22.5	0.9977	74	0.9975
23.0	0.9975	75	0.9973
23.5	0.9974	76	0.9972
24.0	0.9973	77	0.9970
24.5	0.9972	78	0.9969
25.0	0.9970	79	0.9968
25.5	0.9969	80	0.9966
26.0	0.9968	81	0.9965
26.5	0.9966	82	0.9963
27.0	0.9965	83	0.9961
27.5	0.9964	84	0.9960
28.0	0.9962	85	0.9958
28.5	0.9961	86	0.9956
29.0	0.9959	87	0.9955
29.5	0.9958	88	0.9953
30.0	0.9956		
30.5	0.9955		
31	0.9953		

NOTE As per Handbook of Chemistry and Physics, 53<sup>rd</sup> Edition (1972-1973),

## **J.4 Procedure—Calibration of Thermometers**

In order to calibrate a thermometer, the following procedure shall be used.

- a) Place the thermometer to be calibrated alongside either a thermometer or thermocouple that is certified and traceable to a national standard or a standard reference thermometer such as a high-quality mercury-in-glass thermometer from the ASTM precision series, ASTM 62C or 63C (ASTM 62F or 63F) [capable of measuring to  $\pm 0.1$  °C ( $\pm 0.2$  °F)] in a constant temperature bath [or suitable container of 4 L (1 gal) capacity or more, filled with water, in a room where temperature is relatively constant] and allow to equilibrate for 30 min.
- b) Read both thermometers and record the readings.
- c) Repeat the readings at 5 min intervals to obtain at least four sets of readings.
- d) Calculate the average reading,  $R_1$ , for the standard reference thermometer and the average reading,  $R_2$ , for the working thermometer.
- e) For adjustable-scale thermometers, adjust the scale to read the same as the standard reference thermometer. For other thermometers, follow the procedure in J.4 item f).
- f) Calculate the thermometer correction,  $C_{th}$ , expressed in degrees Celsius or Fahrenheit, using Equation (J.2):

$$C_{th} = R_1 - R_2 \quad (J.2)$$

where

$C_{th}$  is the correction value to add to the thermometer reading, expressed in degrees;

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

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

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

$$R_1 = 23.9 \text{ °C}$$

$$R_2 = 24.2 \text{ °C}$$

$$C_{th} = 23.9 - 24.2 = -0.3 \text{ °C}$$

This temperature correction is used by added the value to the thermometer reading. An example thermometer correction application is shown below for a correction factor of  $C_{th} = -0.3$  °C

Temperature reading:  $R$  (measured) = 25.0 °C

$$R_c(\text{corrected}) = 25.0 + (-0.3) = 24.7 \text{ °C}$$

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

- g) Mark and identify the thermometer with its correction and calibration date.

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

## **J.5 Procedure—Calibration of Direct-indicating Viscometers**

In order to calibrate direct-indicating viscometers, the following procedure shall be used.

- a) Obtain two certified calibration fluids closest to the viscosity range of interest, common viscosities are 20 mPa•s (20 cP), 50 mPa•s (50 cP), 100 mPa•s (100 cP), and 200 mPa•s (200 cP).

For most applications, 50 mPa•s (50 cP) and 100 mPa•s (100 cP) viscosity standards are recommended.

Since the actual viscosity of the certified calibration fluid will vary slightly from lot to lot, each sample comes with a chart or table of the actual measured viscosity values versus temperature, typically in increments of 0.1 °C. Viscosity standards are normally certified by methods traceable to a standards body such as the United States National Institute of Standards and Technology (NIST). Each lot of certified calibration fluid is individually certified. Make certain that the lot number on the chart matches the lot number on the fluid container.

- b) 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 [ $<3\text{ °C}$  ( $<5\text{ °F}$ ) variation per hour]. Allow the viscometer and the calibration fluids to achieve temperature equilibrium before testing (approximately 2 h).

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

- c) 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.

- d) Pour the calibration fluid into the sample cup, leaving enough empty volume (approximately 50 mL to 100 mL) in the cup for displacement of fluid due to the viscometer bob and sleeve. Place the cup on the viscometer and adjust the viscometer until the fluid level is aligned exactly with the scribed line on the rotor sleeve.

- e) Place a thermometer [calibrated and accurate to  $\pm 0.1\text{ °C}$  ( $\pm 0.2\text{ °F}$ ), e.g. ASTM 90C or 91C grade or equivalent] into the fluid and hold or tape it in place to prevent breakage. Operate the viscometer at 100 r/min until the thermometer reading is stable for at least a 30 second period (approximately 3 min). Record the temperature reading to the nearest 0.1 °C (0.2 °F).

- f) Operate the viscometer and take readings at 600 r/min and 300 r/min. Estimate readings to the nearest 0.5 dial degree. If needed, use a magnifying glass.

- g) Using the temperature/viscosity table supplied with the standard calibration fluid, determine certified fluid viscosity at the test temperature. Compare the 300 r/min dial reading [same as mPa•s (cP)] to the standard fluid viscosity in mPa•s (cP) and record the deviation (plus or minus). Divide the 600 r/min dial reading by 1.98 to obtain the measured viscosity in mPa•s (cP) at 600 r/min, compare it to the standard calibration fluid viscosity in mPa•s (cP), and record the deviation (plus or minus).

NOTE The 1.98 factor comes from the calibration of the original direct-indicating viscometer with open and closed sleeve fluid ports, see Reference [10].

- h) Repeat procedure item b) through g) with the second calibration fluid viscosity.
- i) Deviations exceeding 1.5 mPa•s (1.5 cP) shall not be allowed. If any of the measured values exceeds these tolerances, adjust the viscometer (see viscometer supplier maintenance instructions) and repeat this calibration procedure.
- j) Record the viscometer serial number, date, and deviation. Mark the viscometer with the date of calibration and indication of its calibration status.

## **J.6 Procedure—Calibration of Retort Kit Cups and Heating Jackets**

### **J.6.1 Calibration of Retort Cups**

In order to calibrate retort cups, the following procedure shall be used.

- a) Allow the retort cup, lid and distilled or deionized water to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).
- b) Place the clean, empty retort cup and lid on the balance and tare to zero.
- c) Fill the retort cup with deionized or distilled water. Place the lid on the cup. Rotate the lid to obtain a proper fit. Be certain a small excess of water flows out of the hole in the lid. Wipe excess water from the lid; avoid wicking out water.
- d) Place the filled retort cup with lid on the previously tared balance. Record the water mass to the nearest 0.01 g.
- e) Calculate the retort cup volume,  $V_{RC}$ , using the water density at ambient temperature (see Table J.1)

$$V_{RC} = \frac{m_w}{\rho_w} \quad (J.3)$$

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 J.1).

### **J.6.2 Calibration of Retort Heating Jacket**

#### **J.6.2.1 Equipment**

**J.6.2.1.1 Calibration thermocouple instrument**, NIST or equivalent, traceable using type J or K probe

**J.6.2.1.2 Retort heating jacket** to be tested

### **J.6.2.2 Procedure**

In order to calibrate a retort heating jacket, the following procedure shall be used.

- a) Insert the J or K thermocouple probe into the retort chamber making sure that it rests on the bottom of the cavity where the retort goes. The retort cell should not be loaded in the jacket, only the thermocouple. Close the lid prior to testing.
- b) Start the retort or for digital programmable temperature-controlled retorts, program the retort to ramp to 500 °C (930°F) then start.
- c) For thermostat switch-controlled retorts, record the temperature then the indicator light goes out.
- d) For digital programmable temperature-controlled retorts, allow the heating jacket to heat for approximately 40 minutes.
- e) After 40 minutes compare the traceable calibration thermocouple to the digital display on the retort, record both values.
- f) The recorded retort temperature shall be 500 °C  $\pm$ 40 °C (930 °F  $\pm$ 70 °F).
- g) If the retort temperature is outside the allowed range, remove the instrument from service and repair or calibrate according to the manufacturer's instructions until the instrument calibrates to the temperature specification.

## **Annex K** **(normative)**

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

#### **K.1 Principle**

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

The permeability plugging apparatus (PPA) may be operated at pressure differentials and temperatures approximating those prevailing down-hole and permit 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 discs as filtration media. Ceramic discs are available with mean pore throat sizes ranging from 10  $\mu\text{m}$  to 120  $\mu\text{m}$ . The PPA uses media more representative of sands which allows evaluation of filtration / bridging agents in drilling fluids.

Filtration characteristics are affected by the types and quantities of solids in the fluid, and by their physical and chemical interactions. The PPA is a modified high-temperature, high-pressure filter press used to evaluate these interactions through ceramic-disc filter media at pressures up to 34,500 kPa (5,000 lbf/in.<sup>2</sup>) and temperatures from ambient to 260 °C (500 °F) (refer to manufacturer's specifications for temperature and pressure limits). Like the standard HTHP filter press, the PPA is suitable for use in either the field or the laboratory.

PPA units usually have half the filter area (2,258 mm<sup>2</sup>) (3.5 in.<sup>2</sup>) of the standard API low-temperature low-pressure test, thus the PPA filtrate volumes shall be doubled to corrected to a filter area of 4,516 mm<sup>2</sup> (7.0 in.<sup>2</sup>) for reporting the results.

#### **K.2 Safety Considerations**

##### **K.2.1 General**

**K.2.1.1** The pressure limitation in the use of the PPA depends upon the cell in use. For safety, it is imperative that the operator know the maximum operating temperature and pressure of the test apparatus with certainty and that these not be exceeded. If in doubt, contact the manufacturer or use the lowest of the possible limits.

**K.2.1.2** Cell damage caused by excessive pressure can be categorized as follows: end-cap bending, end-cap compression, cylinder shear and cylinder stress. End-cap bending can be detected visually or by measurement. End-cap compression can be detected by the distortion of the set-screw holes or seats, which become oval rather than round. Caps showing signs of damage shall not be used and should be discarded.

Cell bodies that show signs of stress cracking or serious pitting, or have damaged set-screw holes, shall not be used.

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

**K.2.1.3** Safe operation of the PPA requires that the operator understand and practice the correct assembly and operation of the apparatus. Improper assembly, incorrect operation or the use of defective parts create the possibility of cell leakage or failure, which could result in serious injury or apparatus damage.

**K.2.1.4** The sample cell is hot during operation. The operator should be aware of the hot areas and avoid contact with them. Burns can result from touching hot parts of the apparatus during normal operation.

**K.2.1.5** These instruments are electrically heated and, as with any electrical device, if the wiring is damaged or faulty, electrical short circuits can occur and create the risk of fire, injury and apparatus damage. These instruments should be used only on grounded circuits.

## **K.2.2 Hydraulic Pressurization System**

For a safe operation of the hydraulic pressurization system, the hydraulic pressure shall be released and ensured before to do followings operations.

- a) attempting to disconnect the pressure hose from the cell at quick coupler;
- b) attempting to remove the cell from the heating jacket;
- c) moving the PPA;
- d) refilling the hydraulic pump;
- e) performing any maintenance, including the tightening of leaking fittings on the hydraulic pump, hydraulic fittings, or cell assembly.

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

## **K.2.3 Back-pressure System**

For a safe operation of the pneumatic pressurization of the back-pressure receiver, following instructions shall be considered.

- a) Always use either nitrogen or carbon dioxide to pressurize the receiver. With silicate fluids, nitrogen shall be used. 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 nitrogen supply system. Carbon dioxide is normally supplied in small cartridges pressurized to about 6,200 kPa (900 lbf/in.<sup>2</sup>).

**Warning—Do not allow carbon dioxide cartridges to be heated or exposed to fire. A cartridge may explode if overheated.**

**Warning—Do not use nitrous oxide cartridges as pressure sources for the NADF filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are used only for Garrett gas train carbonate analysis for water-based fluids (see API 13B-1).**

- b) Maintain pressure regulators and gauges in good condition. Never use oil on pressure regulators.
- c) Leaking pressurization systems, hydraulic or pneumatic, must be repaired or replaced. Gauges, fittings, and hoses should be kept in good condition, and leaks must be found and corrected. Periodically, test the pressure-relief valve on the hydraulic pump to verify that it will function properly should excessive pressure develop. Never plug or bypass this safety valve.
- d) When pressurizing the back-pressure assembly, ensure that the regulator adjusting T-screw or knob has been rotated counter-clockwise enough to release all pressure. When the pressure has been released, the regulator adjusting T-screw or knob will turn freely. Always open the supply pressure valve first then adjust the regulator. Do not attempt to operate the apparatus at pressures in excess of the manufacturer's recommended rating or relief valve settings. When relieving back-pressure, shut the supply pressure valve, bleed the pressure from the system, and loosen the regulator adjusting T-screw or knob fully.

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

### **K.2.8 PPA Heating System**

For safe heating, the instructions below shall be considered.

- a) Caution must be exercised to avoid injury while operating the PPA. It becomes hot enough to cause serious burns. Never leave a hot or heating PPA unattended.
- b) The practice of removing the cell and cooling it with water is dangerous and must be avoided. Serious injuries can be caused by the steam generated when a hot cell contacts water, by direct contact with the cell, or by accidentally dropping the cell.

**K.2.9** For safe electrical operation, follow the instructions below.

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

- a) **K.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.

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

- a) Cell material shall be compatible with the test samples.

- b) Do not use cells that show signs of severe pitting or stress cracking.
- c) Do not use cells, cell caps or retainer rings that show any sign of deformation or damage. Inspect all threads carefully for signs of damage.
- d) For cells that use set screws, use only hardened steel set screws as supplied by the manufacturer. Deformed set screws are unsafe.
- e) The equipment manufacturers' operating manual and this procedure should be read by anyone who is unfamiliar with the apparatus, before using it.

### **K.3 Apparatus—Permeability Plugging Apparatus (PPA)**

#### **K.3.1 PPA Cell**

##### **K.3.1.1 PPA pressure rating**

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

**There are still a number of older cells in use which are stamped "2500 psi". In 1996, the rating on these cells was reduced to "1800 psi" [12,400 kPa (1800 lbf/in.<sup>2</sup>)]. Unless the user can absolutely verify that the cell to be used is rated at 13,800 kPa (2000 lbf/in.<sup>2</sup>) or more, the "1800 psi" 12,400 kPa (1800 lbf/in.<sup>2</sup>) pressure limit shall be observed.**

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

**K.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 hydraulic oil with the sample.

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

**K.3.1.4** For temperatures above 93 °C (200 °F), the back-pressure receiver shall be pressurized to prevent boiling of the filtrate. The standard back-pressure receiver uses carbon dioxide (CO<sub>2</sub>) cartridges as the pressurizing source to provide the back-pressure. A nitrogen pressure source and a nitrogen manifold may be substituted as the pressurizing source.

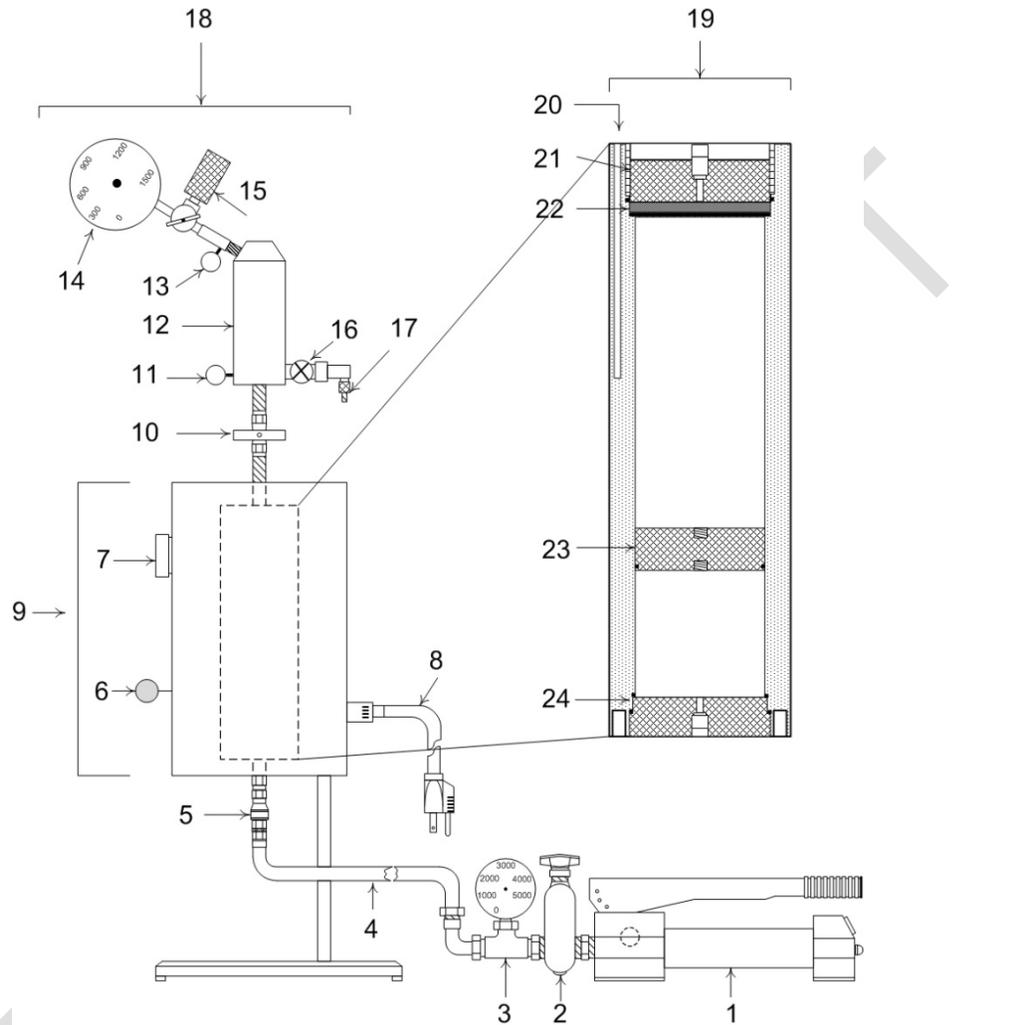
**K.3.1.5** The PPA cell is encased in a thermostatically-controlled heating chamber. This chamber completely encloses the filtering area, permitting filtration at any desired temperature from ambient to 260 °C (500 °F) (check with manufacturer's specifications). The cell temperature can be measured using a thermocouple inserted into the well in the cell wall. The temperature is adjusted by means of a temperature controller. For some apparatus, temperature can be measured using a metal stem thermometer and the temperature is adjusted by means of a knob. After the desired temperature has been 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.

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Figure K.1 is an example diagram a permeability plugging apparatus. Different manufacturers have incorporated different design features, so this drawing indicates only the main operational features. For instance, many designs have inlet pressure valves between the pump and the test cell. Refer to the manual provided by the manufacturer for explicit instructions.

**K.3.1.6** Rupture discs are included in various locations in the pressurization portion for the safety of the operator. The exact location depends on the design consideration of the manufacturer.

BALLOT DRAFT



**Key**

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

**Figure K.1—Typical Permeability Plugging Apparatus**

### K.3.2 Filter Medium

**K.3.2.1** Standard ceramic disc thickness is 6.5 mm (0.25 in.). Thicker discs can be used with properly manufactured cells. A new disc shall be required for each test. For NADF, the disc shall be soaked in base NAF until saturated, so that the porous volume of the disk is filled with NAF and filtration to the receiver begins immediately. When possible, ceramic disks should be vacuum saturated to -95 kPa (-28 in. Hg) gauge in base NAF for a minimum of 30 min prior to use.

These API designated ceramic discs are named according to the mean pore throat diameter as determined by mercury intrusion porosimetry [22]. Table K.1 lists the commonly available API designated ceramic disc sizes and their mean pore throat diameter. Also shown is the previous industry designation based on misleading air measurements.

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

**Table K.1—Ceramic Discs, API Designation and Mean Pore Throat Diameter**

API Designation	Mercury Injection Mean Pore Throat Diameter $\mu\text{m}$	Previous Air Designation $\mu\text{m}$
API 10	10	3
API 12	12	5
API 20	20	10
API 40	40	20
API 50	50	35
API 55	55	60
API 120	120	90

**K.3.2.2 Ceramic discs** were adopted for use in the PPA as a method to test plugging/bridging materials in drilling fluids. Ceramic discs do not exhibit the normal relationship between pore throat size and permeability as naturally occurring sandstone and have a very narrow pore size distribution. Work conducted to assess bridging characteristics should be conducted based on the larger pore throat sizes and not permeability.

**K.3.3 Timer**, digital or mechanical, with at least a 30-min interval, accuracy within  $\pm 5$  seconds per hour.

**K.3.4 Thermometer**, graduated to 260  $^{\circ}\text{C}$  (500  $^{\circ}\text{F}$ ), with an accuracy of  $\pm 3$   $^{\circ}\text{C}$  ( $\pm 5$   $^{\circ}\text{F}$ ).

**K.3.5 Graduated cylinder** (TC), 20mL, 25 mL, or 50 mL.

**K.3.6 High-speed mixer**.

**K.3.7 Stopcock grease**.

## **K.4 Procedure for HTHP Filtration Test using the PPA**

### **K.4.1 Summary**

The following procedures shall be used to perform the HTHP filtration test using the PPA. These procedures are described in K.4.1 through K.5.11.

- Assemble the filtration cell
- Preheat the cell heating jacket
- Load test fluid into the cell
- Pressurize the cell
- Conduct the filtration test
- Test conclusion and disassembly

### **K.4.2 Assembling the Filtration Cell Procedure**

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

If the cell is completely disassembled and the floating piston has been removed, follow K.4.1 a) through K.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 K.4.1 g).

#### **Warning—The filtration cell is a pressure vessel.**

- a) Remove the end caps from the cell. A cell cap removal tool is available from certain manufacturers for some cells.
- b) Check the O-rings on the valve stems, the piston, the cell body, and the end caps. Replace any that are damaged or brittle [O-rings should be replaced 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 to just above the O-ring groove or cell shoulder.
- e) Install and tighten the hydraulic-inlet bottom-end cap onto the cell.

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

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

NOTE Steps K.4.1 g) to K.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 hose quick connect and follow the manufacturer's recommended procedure to purge all air from the hydraulic system.
- i) Apply pressure to the cell using the hydraulic pump. Using the piston T-handle as a guide, stroke the pump enough so the piston rises 5 cm (2 in.) inside the cell. Remove the piston T-handle.
- j) Disconnect the inlet quick connect and remove cell from heating jacket. Place on a suitable stand.

### **K.4.3 Preheating the Heating Jacket Procedure**

The following procedure shall be used for preheating the heating jacket

- a) Connect the power cord to the proper voltage as indicated on the nameplate.
- b) Turn the thermostat to mid-scale and place a metal stem dial thermometer in the thermometer well of the heating jacket. The pilot light will illuminate when the heating jacket temperature has reached the thermostat setting. Digital temperature controllers should be set at the desired test temperature.
- c) After the jacket reaches the desired test temperature, readjust the thermostat or temperature controller to obtain a jacket temperature of 6 °C (10 °F) over the desired test temperature.

NOTE K.4.3 a) to K.4.3 g) should be performed using an unheated jacket or in a specially constructed stand to keep the cell upright.

### **K.4.4 Loading Test Fluid into the Cell Procedure**

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

- a) NAFD sample shall be stirred for 5 min immediately before loading into the PPA cell.
- b) With the cell on a suitable stand, fill the cell to the outlet O-ring groove shoulder 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.
- c) Install the outlet end cell O-ring and seat the selected pre-saturated ceramic disk or other filtration media on top of it.
- d) Position the outlet end cap in the cell, tighten the cell cap, and then install and close the valve on the outlet end cap.

- e) Install the cell in the heating jacket. Lower the cell fully into the heating jacket. Rotate the cell so that the pin in the bottom of the heating jacket seats into the hole in the bottom of the cell body. This prevents rotation of the cell

**Warning—Thermal expansion of the cell contents and of the hydraulic fluid causes the cell pressure to increase rapidly when a closed cell is placed in a hot heating jacket.**

**When a cell at room temperature is placed in a hot jacket, the pump should be connected quickly, and the inlet valve opened to permit the release of hydraulic fluid to prevent over-pressurization. During heating, the pressure in the cell should be controlled by bleeding off the fluid periodically to maintain the required back-pressure from Table K.2**

- f) Connect the cell inlet to the quick connect.
- g) Transfer the thermometer from the heating jacket and place into the cell body. Immediately pressurize the cell as described below. Monitor the cell temperature with the thermometer in the cell wall, not in the heating jacket.

#### **K.4.5 Pressurizing and Heating the Cell Procedure**

The following procedure shall be used to pressurize and heat the cell.

- a) Apply inlet pressure to the cell using the hydraulic pump immediately after transferring the thermometer to the cell body. This pressure should equal the pressure that will be applied to the back-pressure receiver. (Refer to Table K.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 back-pressure receiver to prevent vaporization of the filtrate.

**NOTE For cells with tapered nipple sealing connections, they should be opened during heating and throughout testing.**

- b) When the cell reaches the desired temperature, adjust the thermostat or temperature controller to maintain the cell at the desired test temperature. Hold the cell at the desired temperature until thermal expansion is complete, and the cell pressure stops increasing. The test fluid should not be heated more than 1 h prior to filtrate collection. If the test fluid cell temperature has not reached the test temperature after 1 h the heater may be defective, and the validity of the test is questionable.
- c) While the cell is heating, use the following procedure to prepare the receiver.
- d) Check to ensure that the regulator adjusting T-screw or knob has been rotated counter-clockwise enough to turn freely. The regulator T-Screw will turn freely when no pressure is introduced into the regulator.
- e) Verify that the pressure-relief valve on the assembly and the filtrate drain valve are closed.
- f) Mount the back-pressure receiver on the upper valve adapter. Secure the receiver with a locking pin. Install the pressurizing unit on top of the receiver. Lock the pressurizing unit in place with the other locking pin. Do not adjust the back-pressure outlet regulator at this time.

NOTE 1 A single CO<sub>2</sub> cartridge may not reach the maximum pressures in the table below. If necessary, loosen the regulator adjusting T-screw or knob, remove the empty cartridge, and replace with a new one. Tighten the barrel enough to puncture the cartridge.

NOTE 2 If a nitrogen manifold capable of 4,900 kPa (700 lbf/in.<sup>2</sup>) is available, it may be used.

- g) If a drain hose is to be used for collecting the filtrate, connect it from the filtrate collection nozzle to the graduated cylinder receiving the filtrate.

To ensure accurate measurements, the space between the filtration medium and the outlet valve shall be completely 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.

- h) See Table K.2 to determine the appropriate pressure for the back-pressure receiver and apply it by turning the pressure adjusting T-screw or knob on the regulator clockwise until the desired pressure is reached.
- i) After the test NAFD has heated for 1 h, actuate the pump to raise the inlet cell pressure to the desired differential pressure and then open the valve between the cell and the back-pressure receiver to start the test.

NOTE The differential filtration pressure is the difference between the inlet pressure applied to the cell and that maintained on the back-pressure receiver.

**Table K.2 – Recommended Minimum Cell Heat-up Pressure and Back-pressure**

Test Temperature		Vapor Pressure Water		Minimum Cell Heat-up Pressure and Back-pressure	
°C	°F	kPa	lbf/in. <sup>2</sup>	kPa	lbf/in. <sup>2</sup>
< 95	<200	<84	<12.1	0	0
95–149	200–299	84–462	12.1–67	700	100
150–189	300–374	462–1269	67–184	1400	200
190–199	375–399	1269–1704	184–247	1900	275
200–219	400–424	1704–2245	247–326	2500	350
220–229	425–449	2245–2912	326–422	3100	450
230–246	450–475	2912–3750	422–545	4000	575
247–260	476–500	3750–4692	545–680	5000	725

#### **K.4.6 Conducting the Filtration Test Procedure**

The filtration procedure shall be conducted as follows.

- a) A laboratory timer shall be used, and filtrate shall be collected initially after approximately 15 s to ensure the fluid does not have excessive spurt and exhibits controlled filtration.

Collect filtrate and record the volume at 1 min, 7.5 min and 30 min. Plotting the data against the square root of time provides a useful method to determine spurt loss, see K.6.2. 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 back-pressure receiver, then apply the desired inlet pressure to the cell using the hydraulic pump. Verify that both the cell pressure, as indicated on the pump gauge and the back-pressure are at the 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 shall be used as necessary to maintain the inlet pressure at the desired level.
- d) After each interval, the filtrate shall be drained into the graduated cylinder from the back-pressure receiver and the time and cumulative volume shall be recorded.

It is recommended to recover the filtrate directly from the back-pressure receiver, not from a drain hose attached to it. If a hose is used, its length should be minimized to reduce the error caused by liquid retention on its internal surface.

- e) After 30 min, close the valve between the cell and back-pressure receiver and drain any remaining filtrate from the back-pressure receiver into the graduated cylinder by opening the receiver outlet valve.

#### **K.4.7 Test Conclusion and Disassembly Procedure**

For test conclusion and disassembling the filtration cell, the following procedure shall be used.

- a) Disconnect the heating jacket from the power source.

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

**Warning—Extreme care should be exercised in cooling hot cells.**

- b) Turn the T-screw or knob on the back-pressure regulator counter-clockwise until it turns freely.
- c) Bleed the pressure from the back-pressure receiver by opening the pressure relief valve.
- d) Open the drain valve and collect the last few drops of filtrate into the graduated cylinder. After removing the locking pin, remove the back-pressure assembly from the cell.
- e) Because the design of the back-pressure receiver, it may still have some trapped filtrate inside. With the back-pressure receiver drain or inlet over the graduated cylinder, gently rock it side-to-side to remove any remaining filtrate.
- f) The total volume of 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 in the cell by opening the hydraulic pump valve. Disconnect the inlet quick coupler between the pump and the bottom of the cell.

The cell may be raised in the heating jacket and placed on the cell support or laid on a bench while it is being opened.

- h) 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 the manufacturers' procedure for pressure release process. Trapped pressure is usually indicated by difficulty in removing end cap.
- i) The outlet end cap should be removed with the cell in the vertical position and the outlet or filtration end facing upwards.
- j) To remove the filter disk, pry the edge of the disk up, then remove the filter disk and the filter cake. If required, gently wipe, or rinse the filter cake lightly with base NAF (NADF testing). Then measure and record its thickness and remark on its composition and texture.
- k) Pour the remaining fluid from the cell. Wash the inside of the cell with base NAF. 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.
- l) If testing was conducted at temperatures above 150 °C (300 °F), the O-rings shall be replaced, which will require removal of the inlet end cap and floating piston.
- m) Perform the following three steps to replace the O-rings on the floating piston and inlet end cap.
1. Remove the inlet end cap using the manufacturer's procedure.
  2. Remove the floating piston. Screw the T-bar wrench into the floating piston and push or pull to slide the piston out of either end of the cell. The floating piston can be removed through the top end without removal of the bottom end cap. Remove and dispose of all the O-rings on the floating piston and the inlet end cap.
  3. Clean the parts for reuse.

## **K.5 PPA Test Reports**

### **K.5.1 Filtrate Reporting**

Record the actual cumulative filtrate volume, in milliliters, collected through each of the selected time periods. The filtrate volumes collected are designated with a subscript indicating the time of the sample, i.e.  $V_{30}$  for 30 min volume.

Reported PPA filtrate volumes shall be doubled to corrected to a filter area of 4,516 mm<sup>2</sup> (7.0 in.<sup>2</sup>) for reporting the results since PPA units usually have half the filter area (2,258 mm<sup>2</sup>) (3.5 in.<sup>2</sup>) of the standard API low-temperature low-pressure test.

### K.5.2 Spurt Loss

The spurt loss can be determined from the intercept, on the  $y$ -axis, of the straight line drawn through the collected filtration volumes plotted against the square root of filtration time on the  $x$ -axis. This  $y$ -axis intercept value is then doubled to correct for filtration area when using 2,258 mm<sup>2</sup> (3.5 in.<sup>2</sup>) filtration media and shall be reported as the spurt loss. Alternatively, an approximate value can be calculated using Equation (K.2).

To determine the spurt loss more accurately, collect and record the filtrate more frequently and plot the data as described J.4.6 a),  $f(y$ -axis) versus the square root of time ( $x$ -axis). Take the  $y$ -intercept and double it to get the estimated spurt loss.

### K.5.3 Calculation

The permeability-plugging test volume,  $V_{PPT}$ , expressed in milliliters, is equal to two times the  $V_{30}$ , the filtrate volume after 30 min, expressed in milliliters, as shown in Equation (K.1):

$$V_{PPT} = 2V_{30} \quad (K.1)$$

Calculate the spurt loss,  $V_1$ , expressed in milliliters, using Equation (K.2):

$$V_1 = 2 \times [V_{7.5} - (V_{30} - V_{7.5})] = 2 \times (2V_{7.5} - V_{30}) \quad (K.2)$$

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 the minute, using Equation (K.3):

$$v_{sf} = \frac{2 \times (V_{30} - V_{7.5})}{\sqrt{t_2} - \sqrt{t_1}} = \frac{2 \times (V_{30} - V_{7.5})}{2.739} \quad (K.3)$$

where

$t_1$  is the time at initial reading, expressed in minutes, i.e. 7.5 minutes;

$t_2$  is the time at final reading, expressed in minutes, i.e. 30 minutes.

$V_{7.5}$  is the filtrate volume after 7.5 min, expressed in milliliters.

$V_{30}$  is the filtrate volume after 30 min, expressed in milliliters.

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

#### **K.5.4 Filter Cake Reporting**

Measure and record the filter cake thickness to the nearest 1.0 mm (1/32 in.). Include a description, such as hard, soft, tough, flexible, rubbery, firm, etc. Although these are subjective judgments, the description does convey important information.

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## **Annex L** **(informative)**

### **Compatibility of Elastomeric Materials with Non-aqueous Drilling Fluids**

#### **L.1 Principle**

**L.1.1** It is the intent of this annex to provide guidance for testing the effects of non-aqueous-based drilling fluids on elastomers intended for contact with the fluids during drilling operations. Since the relationship between elastomer/fluid screen test conditions and actual service conditions is only approximate, and since test result acceptance criteria vary with the user, no guidelines for test result interpretation are provided.

**L.1.2** The effects of drilling fluids, particularly NADFs on elastomeric materials used in a drilling operation, can have a significant impact on the safety, the success, and the cost of the operation. Articles prepared with elastomeric materials which may be exposed to such fluids during drilling operations include seals, gaskets, hoses, diaphragms, sleeves, downhole motor stators, etc. Various properties of such articles deteriorate during exposure to the non-aqueous fluids, affecting the performance of the articles.

**L.1.3** Many test methods exist which evaluate these various effects, and the methods employed vary throughout the drilling industry.

**L.1.4** Measurement/predictability of these effects is fundamental to the selection of appropriate elastomer/drilling fluid combinations for the drilling operation and to the establishment of operational guidelines.

**L.1.5** NADF, as defined in this annex, are fluids prepared with either naturally occurring or refined hydrocarbon oils, synthetic hydrocarbons, or other synthetic liquids. These fluids may have aqueous internal phases; however the continuous phase is comprised of the non-aqueous liquid. All test methods described or referenced herein involve measurements of initial properties of a sample of an elastomer, immersion of the elastomer in a fluid at prescribed temperatures for prescribed time periods, and subsequent measurements of the same properties. The methods described yield comparative data on which to base judgment as to expected service quality.

**L.1.6** This annex references ISO standards appropriate for reference testing. This annex also references ASTM test methods and standard reference liquids which have been reviewed and found to be appropriate for testing the effects of drilling fluids on elastomeric materials used in drilling operations.

**L.1.7** The full step-by-step procedures are not reproduced but can be obtained from ISO or ASTM documents listed in Section 2 or Bibliography

**L.1.8** Testing programs should include allowances for environmental extremes in the expected elastomer usage (e.g. temperature, salinity, NAF content). The fluids service provider should be requested to provide a NAF sample that is representative of what would be required for these environmental considerations.

## **L.2 Apparatus and Reagents**

### **L.2.1 Standard Reference Elastomers (SRE)**

Elastomer compositions to be used as standard references are described in ISO 13226:2018. Listed below is a list of common oilfield SREs.

- a) SRE NBR 34S (see ISO 13226:2018, Clause C.2, Table C.1) is an acrylonitrile-butadiene rubber with 34 % (weight) acrylonitrile. It is considered representative of a routinely used, non-extreme service material.
- b) SRE HNBR/1 (see ISO 13226:2018, Clause K.2, Table K.1) is a hydrogenated acrylonitrile-butadiene rubber with 19 % (weight) nitrile groups and less than 0.1 % residual double bonds. It is considered representative of a material required for use in a harsh chemical environment and at high temperature.
- c) SRE FKM1/2X (see ISO 13226:2018, Clause I.2, Table I.1) is fluoro-elastomers such as are used, for instance, for parts in contact with petroleum products in the mechanical-engineering and automobile sectors. It is considered representative of a material required for use in a harsh chemical environment and temperatures up to 200°C (392°F).
- d) FFKM is a perfluoro-elastomer that contains higher amounts of fluorine than standard FKM, and features higher temperature ratings, up to approximately 325°C (617°F). FFKM also has improved chemical resistance, with nearly universal chemical compatibility. Note: This is not included in ISO 13226:2018.
- e) Other representative materials such as YBPO (polythene polyester elastomer) and PTFE may also be representative, depending upon the system exposure being tested.

### **L.2.2 Standard Reference Test Liquids**

Liquid compositions to be used as standard references and the specifications for the IRM (industry reference material) fluids are covered in ASTM D5964-16 and ASTM D471-16a. For testing related to drilling fluids it is common to use also use neat base NAF and/or mixed drilling fluid to assess the impact to elastomers against the standard reference liquids.

- a) IRM 903 is an industry reference hydrocarbon oil with relatively high aniline point, which produces relatively mild effects on elastomers.
- b) ASTM Service Liquid 101 is an industry reference ester oil, di-2-ethyl hexyl sebacate, which is quite aggressive towards elastomers.

## **L.3 Procedure—Test Specimen Preparation**

**L.3.1** Test specimen preparation for all testing is described in ASTM D3182 and ASTM D3183.

**L.3.2** Standard test specimen dimensions are specified in ASTM D471-16a, section 10.1.

## **L.4 Procedure—Elastomer Physical Changes After Immersion**

### **L.4.1 Principle**

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**L.4.1.1** Test methods are given in ASTM D471-16a, Sections 8, 9, 11, and 15. These measurements quantify the effects of a given fluid on a given elastomer. The immersion temperatures chosen are those routinely used by elastomer manufacturers. Other temperatures may be used as required.

**L.4.1.2** Changes in volume, tensile strength, elongation, and hardness of elastomeric materials after contact with non-aqueous fluids are common and degrade the performance of elastomeric articles. The changes can be either positive or negative.

**L.4.1.3** These methods cover the laboratory determination of such changes in unconfined elastomer specimens after static immersion in a test fluid at specified temperatures, for a specified time period and at ambient pressure. Specified immersion temperatures should be 70 °C (158 °F) and 100 °C (212 °F).

**L.4.1.4** Temperatures should be maintained by appropriate means, e.g. water bath, oil bath, hot air oven or ageing block.

**L.4.1.5** An immersion time period of 168 h (7 d) is routinely used by elastomer manufacturers and is specified here to reasonably assure equilibrium. ASTM D471-16a, Table 3 B, lists the standard immersion times but allows that times can be varied according to a material's testing requirements or anticipated exposure in service.

**L.4.1.6** Standard reference liquids and SREs may be tested along with the operational elastomers and fluids being evaluated to assess relative effects.

## **L.4.2 Summary of Immersion Procedure**

**L.4.2.1** Immersion procedures are stated in ASTM D471-16a, Sections 8 and 9.

**L.4.2.2** Pre-immersion measurements of the properties should be made on the specimens by the referenced ASTM procedure and recorded.

**L.4.2.3** The specimens should be then placed in a test tube, or other glass vessel, fitted with a water-cooled reflux condenser, and test fluid is added to completely immerse the specimens. The flow of water through the reflux condenser should be started and the temperature of the test fluid should be increased to the required test value and maintained at that temperature for the required immersion time.

**L.4.2.4** The specimens should be then removed from the test fluid, excess fluid should be removed from them and post immersion measurements of the properties should be made and recorded. If temperatures and immersion times other than those specified are utilized, record the temperature and time.

## **L.4.3 Summary of Change in Volume Procedure**

**L.4.3.1** Change in volume procedure is stated in ASTM D471-16a, Section 12.

**L.4.3.2** Three specimens, as identical as possible, should be tested. The initial volume of each specimen should be measured by water displacement.

**L.4.3.3** Water is removed from the specimens and the immersion procedures described in L.4.2 should be conducted on each specimen.

**L.4.3.4** After immersion, the volume of each specimen should be again measured by water displacement.

**L.4.3.5** The volume change, expressed as a percentage of the original volume, should be calculated for each specimen and an average change determined and recorded.

#### **L.4.4 Summary of Changes in Tensile Strength, Elongation, and Hardness Procedures**

**L.4.4.1** Changes in tensile strength, elongation and hardness are stated in ASTM D471-16a, Section 16 and ASTM 412-16, Section 17.

**L.4.4.2** Original values for tensile strength and elongation should be measured in accordance with ASTM D412-16, using three specimens cut from the same sheet or the article used for the immersion testing, L.4.2. The tensile strength and elongation tests should be also run on the samples after the immersion test. The change, expressed as a percentage of the original value, should be calculated for each specimen and an average change determined and recorded.

Since the ultimate elongation measurement is destructive, separate specimens should be used for the original measurement procedure and immersion measurements.

**L.4.4.3** The original value of hardness should be measured in accordance with ASTM D1415-18 or ASTM D2240-16, using three specimens cut from the same sheet or the article used for immersion testing, L.4.2. The hardness is also run on the samples after the immersion test. The change, expressed as a percentage of the original value, should be calculated for each specimen and an average change determined and recorded.

Since the hardness measurement alters the properties of the elastomer, separate specimens shall be used for the original measurement procedure and immersion measurements.

#### **L.5 Procedure—Effect of Immersion on a Stressed Elastomer Specimen**

**L.5.1** In addition to the measurement of changes in volume, tensile strength, elongation, and hardness after immersion in a test fluid, a method is specified where the test specimens should be stressed (bent) and subjected to the immersion procedures in L.4.

**L.5.2** Visual observations of the condition of the specimens should be made after immersion and recorded.

**L.5.3** Test specimens should be bent to 180° and secured in that position with a suitable retainer which should be inert to the test fluid.

**L.5.4** The specimens should be subjected to the immersion test procedure in L.4. After immersion, the specimens are removed from the test vessel, excess fluid is removed, and visual observations of the specimen's physical condition are made.

**L.5.5** Discoloration, excessive swelling, cracking of the stressed area, extent of cracking and other conditions should be observed and recorded.

## **Annex M** **(informative)**

### **Sand Content Procedure for Non-aqueous Drilling Fluids**

#### **M.1 Principle**

The sand sized content of drilling fluids is the percentage (volume fraction) of all particles of diameter larger than 75  $\mu\text{m}$  (200 mesh). Sand content is measured by a sand-screen test method. In this test, any material retained on the screen is reported as sand content, as defined by the particle size. It may or may not be sand mineralogically.

#### **M.2 Apparatus**

**M.2.1 Sieve**, 75  $\mu\text{m}$  (0.0029 in. - 200 mesh) and 63.5 mm (2.5 in.) in diameter.

**M.2.1 Funnel**, to fit screen.

**M.2.3 Sand content graduated glass tube**, marked for the volume of drilling fluid to be added and graduated from 0 % to 20 % in order to read the percentage of sand directly.

**M.2.4 NAF Base Fluid**

#### **M.3 Procedure**

In order to determine sand content in a NADF, the following procedure should be used.

- a) Fill the glass measuring tube with NADF to the "drilling fluid" mark. Add NAF base fluid to the next mark. Close the mouth of the tube and shake vigorously.
- b) Pour a small quantity of the base NAF through the screen so that the entire surface is wet by the fluid.
- c) Pour the mixture on the wetted screen. Retain the liquid passing through the screen for proper disposal. Add more base NAF to the tube, shake and again pour through the screen. Repeat until the tube is clean. Wash the solids retained on the screen with the base NAF to free it of any remaining drilling fluid.

**NOTE** Do not stir or manually force the mixture through the screen. Tapping the side of the screen holder as the drilling fluid is added can help the mixture pass through the screen. If water-wet solids appear on the screen, measure and report, then conduct a second sand content test with the addition of several drops of the oil-wetting chemical to confirm the solids are water wet, and report.

- d) Put the funnel upside down over the top of the screen. Slowly invert the assembly and insert the tip of the funnel into the mouth of the glass measuring tube. Wash the solids into the tube by adding a small volume

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of base NAF or by rinsing the solids with base NAF from a wash bottle. Allow the solids to settle. From graduations on the tube, read the volume percentage of sand.

#### **M.4 Calculation**

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

BALLOT DRAFT

## **Annex N** **(informative)**

### **Identification and Monitoring of Weight-material Sag**

#### **N.1 Weight Material Sag—Principle**

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.

The target audience for this annex covers both office and well-site personnel.

Weight-material sag is recognized by a significant [greater than 0.06 g/mL or 60 kg/m<sup>3</sup> (0.5 lb/gal)] drilling fluid density variation, lighter followed by heavier than the nominal fluid density, measured when circulating bottoms up, usually where a weighted fluid has remained uncirculated for a period of time in a directional well. It is recognized that sag is both a static and a dynamic phenomenon and has the potential to occur when the drilling fluid is in motion.

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

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.

Weight-material sag is applicable to the complex wells that are now commonly drilled. These include 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;
- estimating hole-cleaning efficiency.

#### **N.2 Guidelines on Using Sag Monitoring Methods**

##### **N.2.1 General**

Experience shows that weight-material sag cannot be totally eliminated. However, it can be managed within levels that do not adversely affect the drilling operation. Weight-material sag is not entirely a drilling fluid-related problem. The occurrence of sag will also be governed by the wellbore geometry, wellbore environment and the operating conditions. Certain conditions in the drilling operation are conducive to creating dynamic sag.

The potential for dynamic sag is promoted by an eccentric pipe, such as when sliding in deviated wells, and by low annular velocities, e.g. less than 33 m/min (<100 ft/min).

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.

## N.2.2 Monitoring Weight-material Sag Methods

The methods to monitor weight-material sag are as follows:

- a) surface monitoring of weight-material sag (N.3);
- b) field sag monitoring based on downhole density (N.4);
- c) dynamic weight-material sag test—viscometer sag shoe test (VSST) method (N.5);
- d) rheological measurements of drilling fluids exhibiting weight-material sag (N.6);
- e) field sag monitoring based on critical wall shear stress (N.7).

The choice of monitoring method or methods is an engineering judgment that is linked to the complexity of the well and the probability of weight-material sag occurring in the field. Surface monitoring of mud-weight variation is logged as the *de facto* determination of whether sag is present. Downhole density determination will not always be an option because it relies upon specific downhole tools being run in the drill string. The experimental methods proposed may be used individually or in combination. The modeling method based on critical wall shear stress is relatively simple to employ and only requires simple spreadsheet calculations.

Each method is described in more detail in N.3 to N.7.

The root causes of weight-material sag are continuing to be investigated. As such, there are still considerable research efforts towards developing new methods to monitor and quantify sag. Some techniques are referenced in the Bibliography, also see Reference [23].

## N.3 Surface Monitoring of Weight-material Sag

### N.3.1 Principle

Changes in drilling fluid density measured at the surface are often the first indications that weight material sag is occurring within the wellbore. All drilling operations measure and monitor surface drilling fluid density on a regular basis. Fluctuations of surface density have been classified as the *de facto* standard in the definition of weight material sag. See N.1.

Variations in drilling fluid density may be identified from the regular surface density determinations which form part of routine operations and are recorded on an ongoing basis.

The accepted practice for quantifying weight-material sag from surface density measurement is to monitor the density of return fluid samples when tripping-in the hole and stage circulating, or when circulating bottoms-up with the bit at total depth, prior to and after tripping-out of the hole.

### N.3.2 Apparatus

**N.3.2.1 Any density-measuring instrument** having an accuracy of  $\pm 0.01$  g/mL or  $10$  kg/m<sup>3</sup> (0.1 lb/gal or 0.5 lb/ft<sup>3</sup>), including mud balances as described in 5.2 and 6.2.

NOTE The preferred method is to use a pressurized mud balance as described in 6.2. 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.

**N.3.2.2 Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of  $\pm 1$  °C ( $\pm 2$  °F).

### N.3.3 Procedure

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.

- a) 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.
- b) Measure the density of drilling fluid samples from pre-determined depths or time intervals with a mud balance. Use a pressurized mud balance if appropriate (see N.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 N.3.4.
- c) If practical, make all surface density determinations at a uniform temperature, e.g. 21 °C (70 °F). Regardless, the density shall be taken together with the recorded temperature. These data can then be used to graphically relate the measured density to a preferred reference temperature. Temperature should be recorded with an accuracy of  $\pm 1$  °C ( $\pm 2$  °F).

### N.3.4 Calculation

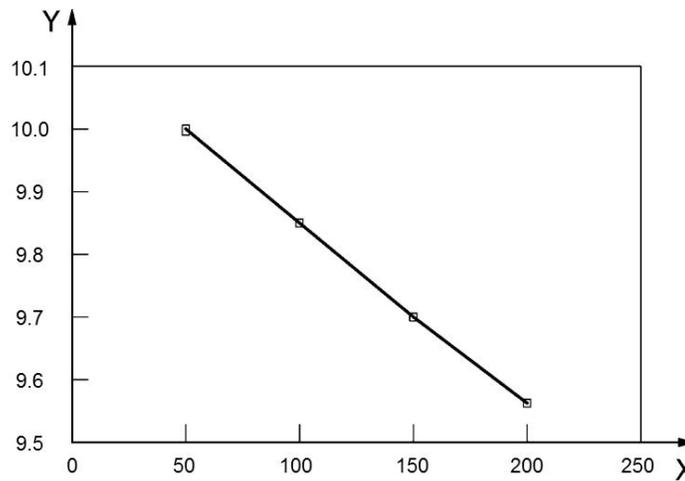
**N.3.4.1** Calculate the lagged time,  $t$ , of samples, using Equation (N.1) based on annular volume capacity and discharge pump capacity (mechanical efficiency). This calculation should take account of any known changes in annular volume capacity due to geometry changes, including wellbore enlargement.

$$t = \frac{4200 \times V_{\text{an}}}{Q \times E} \quad (\text{N.1})$$

where

- |                 |  |
|-----------------|--|
| $t$             | is the lagged time, expressed in minutes;          |
| $E$             | is the pump efficiency, expressed as percentage;   |
| $Q$             | is the pump rate, expressed in gallons per minute; |
| $V_{\text{an}}$ | is the annular volume, expressed in barrels.       |

**N.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 a NADF is given in Figure N.1.



**Key**

**X** temperature, °F

**Y** drilling fluid density, lb/gal

**Figure N.1—Example of Surface Density Variation with Temperature (NADF)**

**N.3.4.3** Calculate the difference,  $\Delta\rho$ , in the maximum fluid density and nominal fluid density (normal circulating density), expressed in pounds per gallon, using Equation (N.2), which can then be used to calculate the sag register,  $S$ , using Equation (N.3).

$$\Delta\rho = \rho_{\max} - \rho_{\text{nom}} \tag{N.2}$$

$$S = \exp\left[10 \times \frac{\Delta\rho}{\rho_{\text{nom}}}\right] \tag{N.3}$$

where

$S$  is the sag register, dimensionless (see Reference [24]);

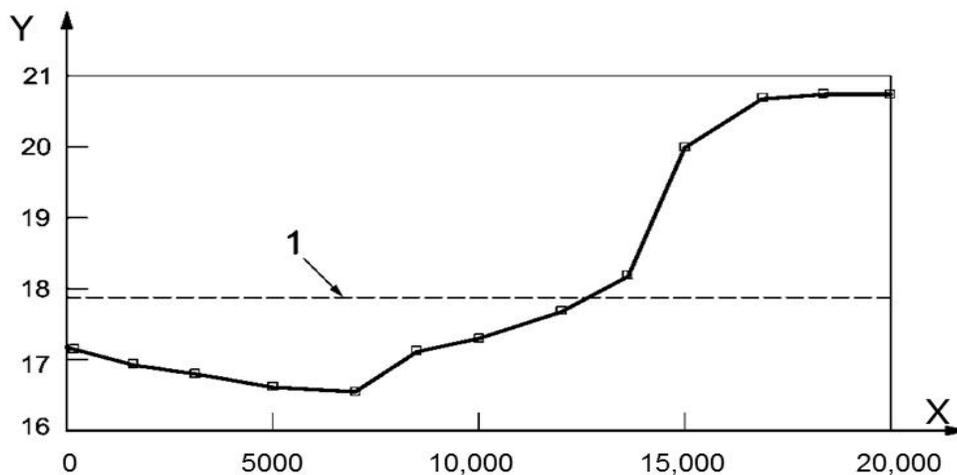
$\rho_{\max}$  is the maximum recorded drilling fluid density, expressed in pounds per gallon;

$\rho_{\text{nom}}$  is the nominal drilling fluid density, expressed in pounds per gallon.

### N.3.5 Data Reporting

**N.3.5.1** Record operational conditions of the well on a trip-out sheet similar to the one shown in Table N.1. This should also include the raw data measurements of drilling fluid density versus pump strokes and time.

**N.3.5.2** Plot the surface lagged density profile as a function of logged depth. An example is given in Figure N.2.



**Key**

**X** measured depth, ft

**Y** lagged fluid density, lb/gal

**1** nominal drilling fluid density, 17.9 lb/gal

**Figure N.2—Example Surface Profile Based on Bottoms-up Data**



## **N.4 Field Sag Monitoring Based on Downhole Density**

### **N.4.1 Principle**

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.

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.

### **N.4.2 Measurements of Downhole Pressure**

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 [25] and [26]). At the surface, the measurements received from downhole are then converted to drilling fluid density,  $\rho_{df,USC}$ , using the standard Equation (N.4).

$$\rho_{df,USC} = \frac{P}{0.052 \times D_{TVD}} \quad (N.4)$$

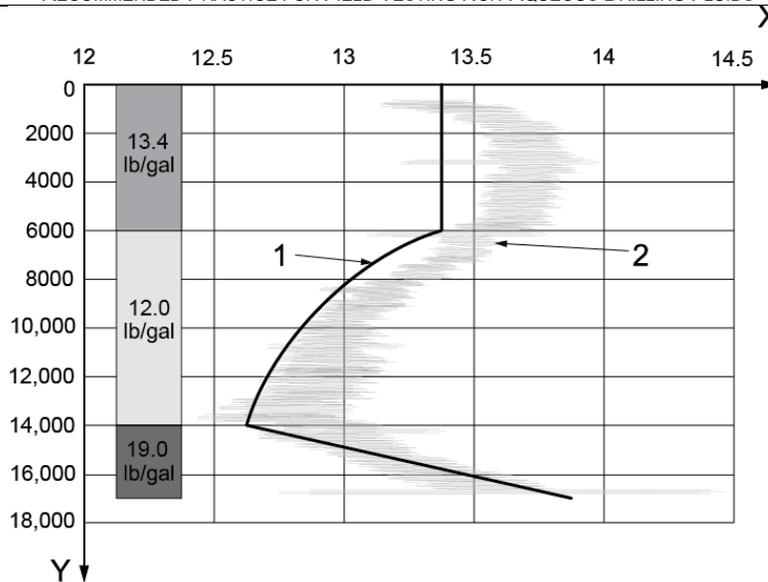
where

- $\rho_{df,USC}$  is the drilling fluid density, expressed in pounds per gallon;25
- $P$  is the measured pressure, expressed in pounds-force per square inch;
- $D_{TVD}$  is the true vertical depth, expressed in feet.

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.

### **N.4.3 Weight-material Signatures Using Downhole Pressure Tools**

**N.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 N.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 true vertical depth (TVD), ft
- 1 simulated data
- 2 pressure data for trip in hole

NOTE See Reference [27].

**Figure N.3—Downhole Density Changes Measured While Running the Drill String in the Hole**

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:

- declining density followed by increasing density;
- a density swing between 19.0 lb/gal and 13.4 lb/gal, which gives a theoretical density difference  $\Delta\rho$  of 5.6 lb/gal (19.0 lb/gal – 13.4 lb/gal);
- 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.

**N.4.3.2** 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 N.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.

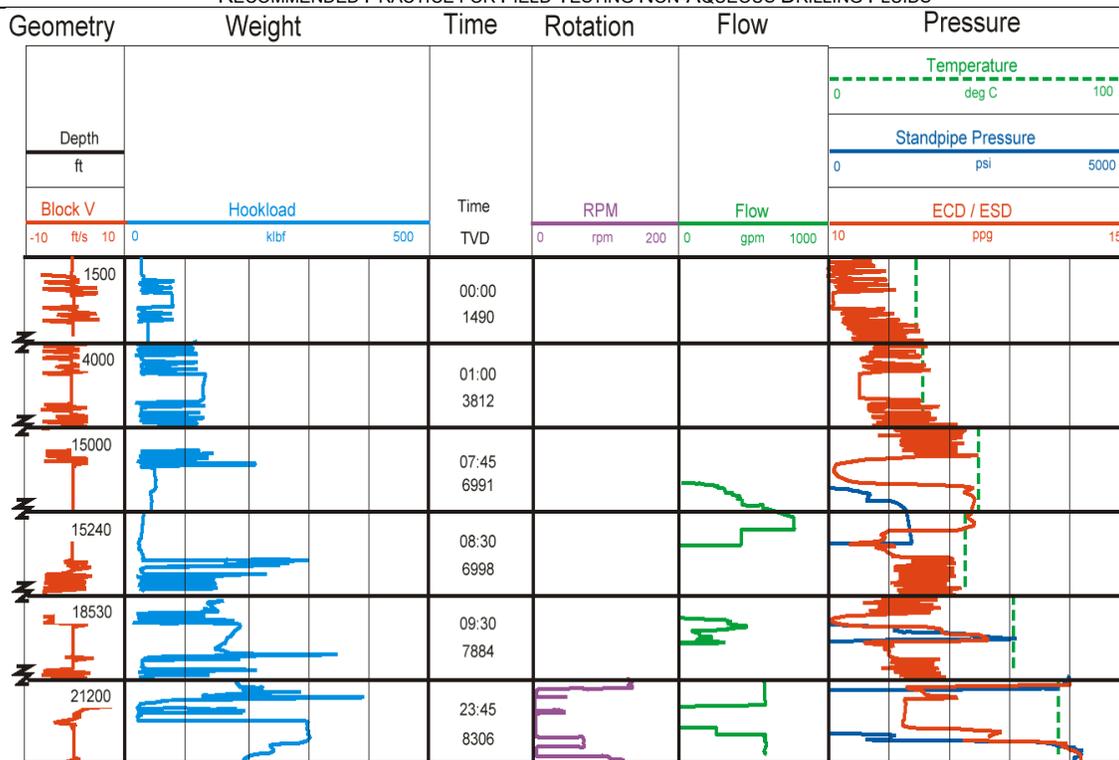


Figure N.4—Weight-material Sag Occurrence During Dynamic Conditions

**N.4.3.3** 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.

## N.4.4 Downhole Pressure Changes that Are Not Caused by Weight-material Sag

### N.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.

### N.4.4.2 Changes in Pump Rate

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.

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.

### N.4.4.3 Changes in Downhole Density

Some drilling fluids, particularly invert emulsions and those formulated with NAFs, exhibit changes in density as a function of temperature and pressure.

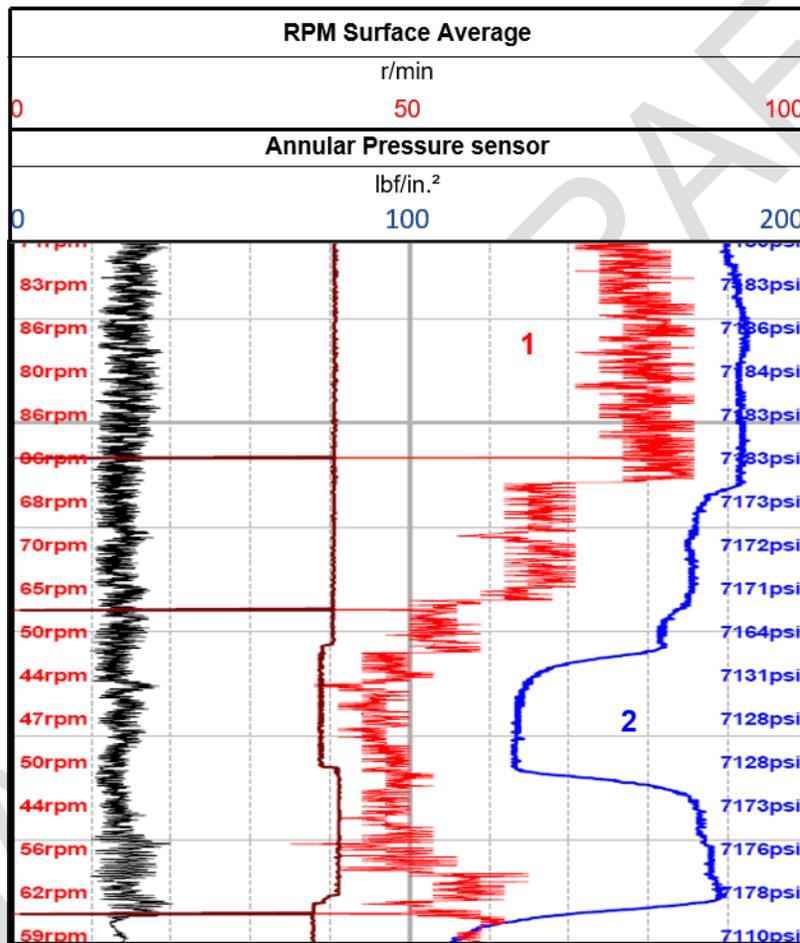
This phenomenon is purely a function of the fluid's compressibility and thermal expansion. These properties can be characterized using coefficients derived from laboratory (pressure, volume, temperature) (PVT) data.

NOTE API 13D contains procedures and coefficients to predict the density of drilling fluids as a function of temperature and pressure.

#### N.4.4.4 Changes in Drill Pipe Rotational Speed

Changes in drill pipe rotational speed can also change downhole pressures.

Figure N.5 displays the changes in downhole pressure produced by fluctuations in drill pipe rotational speed that were obtained from downhole pressure tools.



**Key**

- 1 rotary speed, r/min
- 2 downhole pressure, pounds-force per square inch

**Figure N.5—Effect of Changes in Drill Pipe Rotational Speed on Downhole Pressure**

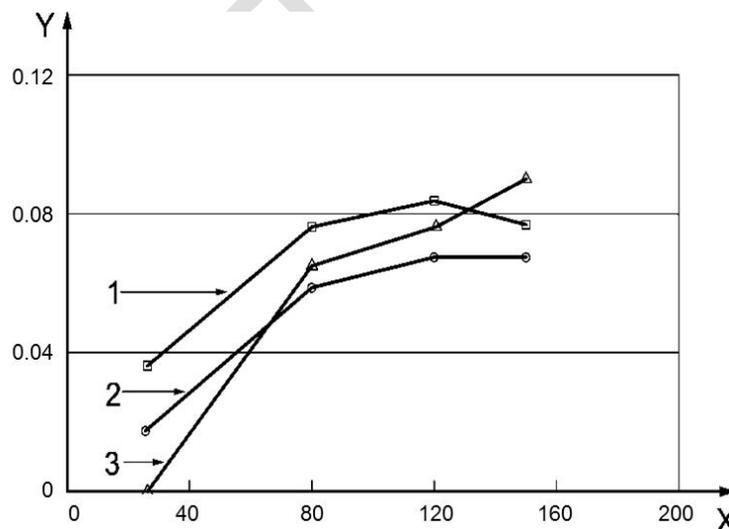
The coupling of tangential velocities produced by the rotating drill string with the axial velocities produced by the pump rate results in helical flow and higher downhole pressures. These are commonly seen with downhole pressure tools. These increases in downhole pressure have been measured in field experiments (see References [28], [29], and [30]). Published data show these ECD 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 N.2.

**Table N.2—Matrix for Fingerprinting Drill String Rotation Effects on Downhole Density**

	Minimum Pump Rate 1	Intermediate Pump Rate 2	Maximum Pump Rate 3
r/min speed 1	X <sub>11</sub>	X <sub>21</sub>	X <sub>31</sub>
r/min speed 2	X <sub>12</sub>	X <sub>22</sub>	X <sub>32</sub>
r/min speed 3	X <sub>13</sub>	X <sub>23</sub>	X <sub>33</sub>

Results from an ECD fingerprinting exercise in the field are shown in Figure N.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.



**Key**

- X drill pipe rotation speed, r/min
- Y measured increase in ECD, lb/gal
- 1 ECD increase at 1000 gal/min
- 2 ECD increase at 900 gal/min
- 3 ECD increase at 1100 gal/min

NOTE See Reference [30].

**Figure N.6—ECD 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 [30]). 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 [31]). 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 (N.5).

$$\Delta R_{\text{Rot}} = \left( -1.0792 \times \frac{d}{D} \right) + \left[ \left( 17.982 \times \frac{d}{D} \right)^2 \times (10^{-5} \times L) \times \eta \right] \quad (\text{N.5})$$

where

$\Delta P_{\text{Rot}}$  is the anticipated pressure increase due to drill pipe rotation, expressed in pounds-force per square inch;

$d$  is the diameter of inner pipe (drill 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 pipe rotation, expressed in revolutions per minute.

f) Increase in ECD is calculated using Equation (N.6):

$$\Delta \rho_{\text{ECD-Rot}} = \frac{\Delta P_{\text{Rot}}}{0.052 \times D_{\text{TVD}}} \quad (\text{N.6})$$

where

$\Delta \rho_{\text{ECD-Rot}}$  is the anticipated ECD increase due to drill pipe rotation, expressed in pounds per gallon;

$\Delta P_{\text{Rot}}$  is the anticipated pressure increase due to drill pipe rotation, expressed in pounds-force per square inch;

$D_{\text{TVD}}$  is the true vertical depth, expressed in feet.

**N.4.5 Theoretical Downhole Density Excluding Effects in N.4.4**

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  $\pm 0.5$  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.

While  $\rho_{\text{ECD-tot}}$  increases in depth,  $\rho_{\text{ESD}}$  may increase, decrease, or remain constant with depth depending on the thermo-physical properties used in its estimation (see Reference [31]).

In dynamic cases, the downhole density while rotating the drill string should include:

- a)  $\rho_{\text{ECD-hyd}}$  which is comprised of the ESD, the circulating pressure drop and the extra density effects of drilled cuttings in the annulus predicted from hole cleaning modeling;
- b)  $\Delta\rho_{\text{ECD-rot}}$  determined using Equation (N.6) or interpolated as discussed in N.4.4.4

The total predicted ECD of the drilling fluid in the annulus while circulating,  $\rho_{\text{ECD-tot}}$ , is calculated using Equation (N.7):

$$\rho_{\text{ECD-tot}} = \rho_{\text{ECD-hyd}} + \Delta\rho_{\text{ECD-rot}} \quad (\text{N.7})$$

where

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

$\rho_{\text{ECD-hyd}}$  is the hydraulic ECD, includes ESD, circulating pressure drop and added density effects of drilled cuttings in the annulus, expressed in pounds per gallon;

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

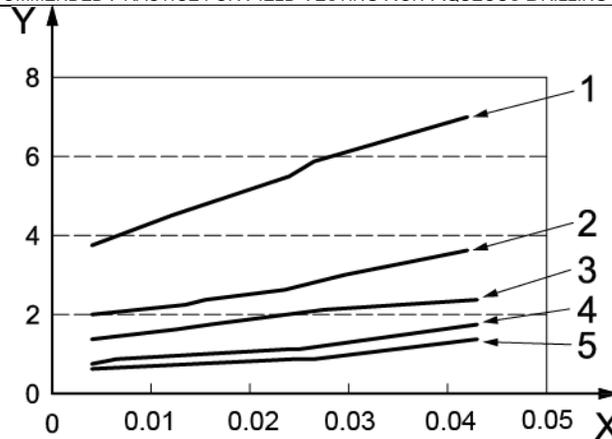
If  $\rho_{\text{ECD}}$  in the wellbore, measured using downhole pressure tools, is greater or less than the  $\rho_{\text{ECD-tot}} \pm 0.5$  lb/gal range, then there is a potential weight-material sag event and further investigation is needed.

#### **N.4.6 Weight-material Sag Events Caused by Drill Pipe Rotation Only**

Some modeling of weight-material sag potential has been performed on the effects of rotating a drill string slowly in a static fluid (see Reference [31]). 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.

In the modeling work, it was shown that, at slow drill string rotation speeds in static fluids, there exists a potential weight-material sag occurrence. Fluid viscosities are lowest close to the rotating drill string, as shown in Figure N.7.

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.



**Key**

- |          |                                       |          |                                       |
|----------|---------------------------------------|----------|---------------------------------------|
| <b>X</b> | gap, m                                | <b>3</b> | drill-string rotation speed: 15 r/min |
| <b>Y</b> | viscosity, Pa*s (cP)                  | <b>4</b> | drill-string rotation speed: 20 r/min |
| <b>1</b> | drill-string rotation speed: 5 r/min  | <b>5</b> | drill-string rotation speed: 25 r/min |
| <b>2</b> | drill-string rotation speed: 10 r/min |          |                                       |

NOTE See Reference [32].

**Figure N.7—Distribution of Fluid Viscosity Across the Annular Gap Caused by Drill String Rotation in a Non-circulating Drilling Fluid**

## N.5 Dynamic Weight-material Sag Test—VSST Method

### N.5.1 Principle

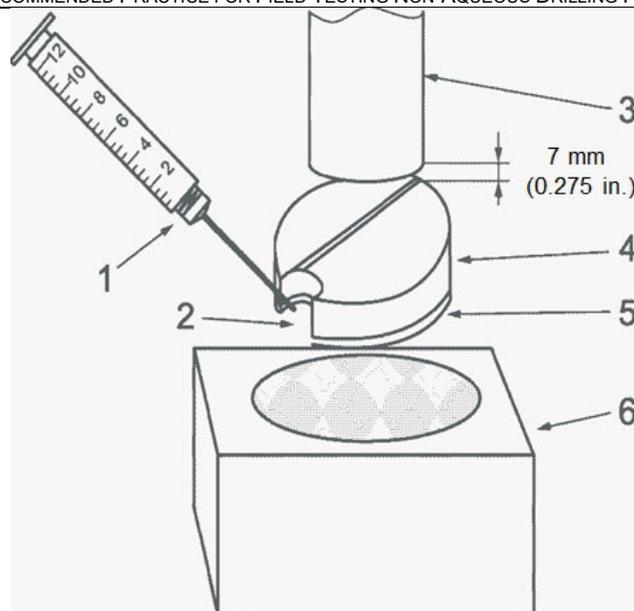
The Viscometer Sag Shoe Test (VSST) is a well site and laboratory test that measures weight material sag tendencies of field and lab-prepared drilling fluids under dynamic conditions.

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 test results with actual measured sag experienced in the field.

The VSST designation is derived from the direct-indicating viscometer used as a mixer and the thermoplastic insert (Sag Shoe) designed to concentrate sagged weight material in the bottom of a viscometer thermocup, see Reference [33]. Figure N.8 shows the basic equipment needed to determine VSST.

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.

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.



**Key**

- |   |                    |   |                     |
|---|--------------------|---|---------------------|
| 1 | syringe            | 4 | VSST Sage Shoe      |
| 2 | Collection well    | 5 | Metal-backing plate |
| 3 | Viscosimeter rotor | 6 | thermocup           |

**Figure N.8—Key Equipment for VSST Method**

## N.5.2 Apparatus

**N.5.2.1 Direct-indicating viscometer**, powered by an electric motor; see 7.3.1.1 for dimensions.

**N.5.2.2 Stopwatch or timer**, accuracy within  $\pm 5$  seconds per hour.

**N.5.2.3 Thermostatically controlled viscometer cup.**

NOTE The VSST Sag Shoe is intended to fit inside the thermocup without being too tight.

**N.5.2.4 Thermometer**, with a range of 0 °C to 105 °C (32 °F to 220 °F) and an accuracy of  $\pm 1$  °C ( $\pm 2$  °F).

**N.5.2.5 Luer syringe**, 10 mL, two-piece, solvent resistant.

**N.5.2.6 Pipetting needle**, blunt-end, 150 mm (6 in.), 14-gauge needle with Luer connection.

**N.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.

**N.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 N.5.3.3.

**N.5.2.9 Spatula**, 150 mm (6 in.) or similar.

**N.5.2.10 Deionized or Distilled water**.

**N.5.2.11 Multimixer**, capable of operating at 11,500 r/min  $\pm$ 300 r/min under load, with a single corrugated impeller of approximately 25.4 mm (1 in.) in diameter.

**N.5.2.12 Container**, for mixing, 180 mm deep (7.125 in.), diameter 97 mm (3.75 in.) at top and 70 mm (2.75 in.) at bottom.

**N.5.2.13 VSST Sag Shoe**, shaped, cylindrical disk manufactured from thermoplastic material, maximum diameter of 59.7 mm  $\pm$ 0.4 mm (2.35 in.  $\pm$ 0.015 in.) and height of 27.7 mm (1.09 in.). The well is a notch cut through the thermoplastic disk and metal-backing plate that connects with the thermocup bottom. The metal plate is used solely to increase the overall mass of the Sag Shoe so that it does not float when used with very dense fluids.

### **N.5.3 Procedure**

In order to conduct a VSST, the following procedure should be used.

#### **N.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 rotor sleeve, and mark the support leg at the upper edge of the locking mechanism.
- c) Lower the plate and thermocup to the base and mark the support leg 7 mm (0.25 in.) below the first mark.
- d) Calibrate the syringe with blunt-end pipetting needle attached, using deionized water and digital balance. Expel as much water as possible from the syringe barrel and wipe exterior dry.

NOTE Calibrate pycnometer or retort cup with distilled water if it will be used for density measurements.

#### **N.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 drilling 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 about 7 mm (0.25 in.) below the viscometer rotor 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 10 mL 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 item f), this time taking the sample from the collection well of the Sag Shoe. Use the pipetting needle tip to find the collection well.
- j) Weigh the fluid-filled syringe and record the total mass as  $m_{F2}$ , expressed in grams.

NOTE When using the 10 mL pycnometer or retort cup, transfer the fluid from the syringe, weigh, and record total mass as  $m_{F2}$ , expressed in grams.

#### **N.5.3.3 Bed pickup measurement (optional)**

- a) Gently return the 10 mL test sample from the fluid-filled syringe obtained in N.5.3.2 item i) to the Sag Shoe collection well.
- b) Run the viscometer at 600 r/min for 20 min.
- c) Collect the sample the from Sag Shoe collection well as in N.5.3.2 item i). Weigh the fluid-filled syringe and record total mass as  $m_{F3}$ , expressed in grams.

#### **N.5.4 Calculation**

**N.5.4.1** The amount of weight-material sag,  $B_{VSST}$ , should be calculated using Equation (N.8):

$$B_{VSST} = 0.834 \times (m_{F2} - m_{F1}) \quad (\text{N.8})$$

where

$B_{VSST}$  is the amount of weight-material sag, expressed in pounds per gallon;

$m_{F1}$  is the initial mass of 10 mL drilling fluid (plus the syringe), expressed in grams;

$m_{F2}$  is the mass of 10 mL drilling fluid (plus the syringe) taken from the sag shoe following 30 min shear at 100 r/min, expressed in grams.

Report  $B_{VSST}$  in pounds per gallon.

**N.5.4.2** The calculated bed pickup measurement ratio,  $R_{BPU}$ , should be calculated as an option using Equation (N.9):

$$R_{\text{BPU}} = \frac{83.4 \times (m_{\text{F2}} - m_{\text{F3}})}{B_{\text{VSST}}} \quad (\text{N.9})$$

where

$R_{\text{BPU}}$  is the calculated bed pickup measurement ratio, expressed as a percentage;

$B_{\text{VSST}}$  is the amount of weight-material sag, expressed in pounds per gallon;

$m_{\text{F2}}$  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;

$m_{\text{F3}}$  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  $R_{\text{BPU}}$  as a percentage.

## **N.6 Rheological Measurements of Drilling Fluids Exhibiting Weight-material Sag**

### **N.6.1 Principle**

Advanced rheometers can 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.

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.

Establishing guidelines for sample preparation and equipment selection will facilitate more meaningful analyses of drilling fluid samples during sag investigations.

The method given in this section will only yield correct information when the annular velocity is 100 ft/min or greater.

### **N.6.2 Scope**

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.

There is a generally accepted view that viscosity measurements at low (less than  $1.0 \text{ s}^{-1}$ ) 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.

This section is applicable to both field drilling fluids and fluids mixed in the laboratory.

### **N.6.3 Apparatus**

**N.6.3.1 Rheometers:** for the purpose of this procedure distinguished from viscometers by their greater degree of accuracy and range of measurement.

- a) Typical capabilities found only in rheometers are very low shear rates, oscillatory measurements and the capability to make measurements under elevated temperatures and pressures.
- b) Rheometers suitable for detailed investigation of sagging drilling fluids should be capable of the following:
  - 1) accurate measurement of viscosity at shear rates from approximately  $1000 \text{ s}^{-1}$  continuously to  $0.01 \text{ s}^{-1}$  or below;
  - 2) oscillatory functionality to allow the calculation of the storage modulus ( $G'$ ) and loss modulus ( $G''$ );
  - 3) accurate measurement of stresses below  $0.02 \text{ lbf}/100\text{-ft}^2$ .

**N.6.3.2 Water bath,** maintained at  $60 \text{ }^\circ\text{C}$  to  $70 \text{ }^\circ\text{C}$  ( $140 \text{ }^\circ\text{F}$  to  $160 \text{ }^\circ\text{F}$ ).

**N.6.3.3 Mixer,** a high shear, variable speed, heavy-duty laboratory mixer with a high shear, square-hole impeller screen. Typical impeller blade diameter  $31.20 \text{ mm}$  ( $1.228 \text{ in.}$ ), impeller screen inside diameter  $34.85 \text{ mm}$  ( $1.372 \text{ in.}$ ). The rotor/stator diameter on these mixers is  $31.24 \text{ mm}$  ( $1.23 \text{ in.}$ ). The tolerance between the rotor and stator is between  $0.30 \text{ mm}$  and  $0.38 \text{ mm}$  ( $0.012 \text{ in.}$  and  $0.015 \text{ in.}$ ) on the diameter. The hole size on the square hole high shear screen is  $2.4 \text{ mm}$  ( $3/32 \text{ in.}$ ).

### **N.6.4 Sample Preparation**

To prepare the sample, the following procedure should be used

- a) 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.
- b) Sample mixing should involve the entire content of the storage container. By their nature, fluid samples collected because of sag problems may be expected to have suffered from solids settling during storage. All solids shall be removed from the container prior to mixing.
- c) The sample should be mixed at a  $6000 \text{ r/min}$  shear rate using the high shear mixer described in N.6.3.3, for a period of  $15 \text{ min}$  per  $350 \text{ mL}$  volume. A volume of  $1400 \text{ mL}$  should be sheared for  $1 \text{ h}$ . Cooling the sample to between  $60 \text{ }^\circ\text{C}$  and  $71 \text{ }^\circ\text{C}$  ( $140 \text{ }^\circ\text{F}$  and  $160 \text{ }^\circ\text{F}$ ) should include the use of a water bath. Once this temperature is reached, the sample should be covered to prevent vaporization of water. If the sample is too large to mix in a single batch, multiple batches can be mixed as above and then combined. All steps to minimize the time delay between mixing the first and last batches should be taken in such cases.
- d) For each rheological measurement, the time between the fluid being sheared as described in N.6.4 item c) and the measurement being made should be recorded.
- e) Immediately prior to each rheological measurement, the fluid should be sheared in the rheometer at approximately  $1000 \text{ s}^{-1}$  for a minimum of  $2 \text{ min}$ .

## **N.6.5 Potential Rheological Tests**

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.

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.

Unlike the common six-speed field viscometer, which exclusively uses a rotating sleeve about a torsion spring bob (Couette geometry), rheometers have a variety of test geometries from which to choose. These include the Couette geometry, double-gap Couette, multivane spindles, parallel plates, cone and plate, and any of these modified with roughened surfaces for mitigation of wall slip effects which can occur at the very low shear rates. The test geometry should be selected in accordance with the needs of the test to be performed and the fluid being tested.

During the preparation of N.6, a series of round-robin tests were performed by several laboratories. These laboratories used different instruments and followed the individual manufacturer's instructions for the instrument. The list of instruments used includes:

- a) Bohlin® Gemini;
- b) Brookfield® PVS;
- c) Grace® M3500a-1;
- d) OFI® Model 900;
- e) Physica® MCR101;
- f) RJF® Viscometer.

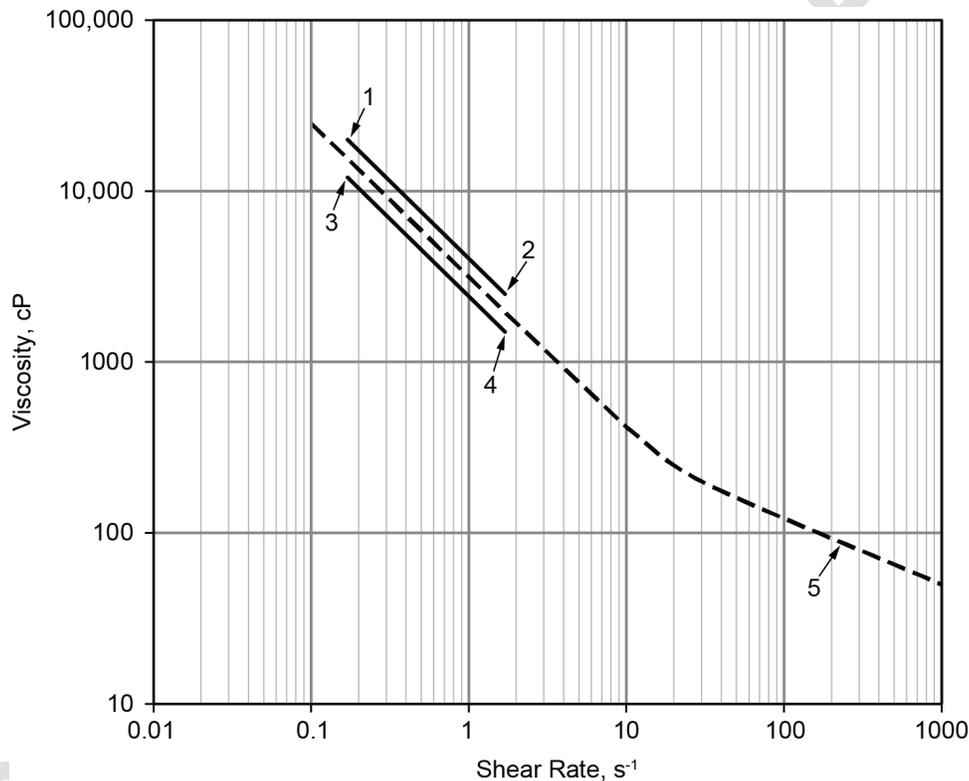
## **N.6.6 Data Interpretation**

Various publications (see Reference [33]) have suggested that weight-material sag is closely correlated with the viscosity of the fluid at very low shear rates. The shear rates of interest are typically in the range of  $0.1 \text{ s}^{-1}$

to  $1.0 \text{ s}^{-1}$ . Measurement of viscosity at these shear rates is not possible at the rig site using conventional field viscometers.

More sophisticated devices have become available since around 2,000. 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.

Viscosity values, which should be adequate to prevent sag of invert emulsion drilling fluids under dynamic field conditions, have been proposed (see Reference [35]), and a typical graph is shown in Figure N.9. In the graph, the solid parallel lines represent the upper and lower bounds of acceptable viscosity, i.e. viscous enough to prevent dynamic weight-material sag under typical drilling conditions, but not so viscous as to cause other drilling-related problems. Note that the viscosity and shear rate are based on the nominal shear rate calculated for Newtonian fluids. This is consistent with previous publications on this technique. However, the variations arising from the non-Newtonian behavior of typical invert drilling fluids will result in relatively small deviations from these nominal values.



**Key**

- 1 20,000 cP at  $0.17 \text{ s}^{-1}$
- 2 2500 cP at  $1.7 \text{ s}^{-1}$
- 3 12,000 cP at  $0.17 \text{ s}^{-1}$
- 4 1500 cP at  $1.7 \text{ s}^{-1}$
- 5 viscosity curve for a drilling fluid with minimal expected sag tendency

NOTE See Reference [33].

**Figure N.9—Published “Sag Window” for Drilling Fluids Versus Shear Rate**

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.

If the basic characteristics of the system differ, i.e. different weight materials and NAF-to-water ratios, different viscosifier types, significant changes in internal phase composition, and significantly different emulsifier chemistry, the conclusions of one system may not apply to the other. The rheological testing and evaluation of each fluid should be taken with knowledge of the physical characteristics of that fluid. Likewise, all of the rheological testing performed should be considered when drawing conclusions as to a fluid performance.

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.

## **N.7 Field Sag Monitoring Based on Critical Wall Shear Stress**

### **N.7.1 Principle**

Use of hydraulic modeling to predict the onset of weight-material sag under dynamic conditions has been presented (see Reference [35]). 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 [26]).

### **N.7.2 Predictive Model**

Maximum weight-material sag should be predicted using the following procedure.

- a) Obtain drilling fluid rheological properties from viscometer data, from HTHP viscometers, or from predicted downhole data.
- b) Calculate the fluid Herschel-Bulkley rheological parameters through a mathematical regression analysis (see API 13D). The use of a computer program or spreadsheet is recommended to perform this

complex data analysis. The drilling fluid yield stress value,  $\tau_Y$ , will be used in the calculations below.

- c) In the hydraulic calculations, use the geometry outer and inner diameters for the particular interval (hydraulic section) where weight-material sag occurrence is suspected. Set the inner pipe eccentricity to a high value ( $\varepsilon = 0.7$  is recommended) in order to determine  $d_i$ , the distance from the outer wall using Equation (N.10).

$$d_i = \frac{\varepsilon \times (D - d)}{2} \quad (\text{N.10})$$

where

$\varepsilon$  is the inner pipe eccentricity, dimensionless (0.7 recommended value);

$D$  is the diameter of outer pipe diameter, expressed in inches ;

$d$  is the diameter of inner pipe, expressed in inches;

- d) Calculate using Equation (N.11) the minimum pressure drop in this geometry to shear the annulus across the narrow gap.

$$\Delta\left(\frac{P}{L}\right) = \frac{2 \times \tau_Y}{L} \quad (\text{N.11})$$

where

$\Delta\left(\frac{P}{L}\right)$  is the pressure gradient in the hydraulic section, expressed in pounds-force per square inch, per foot;

$\tau_Y$  is the drilling fluid yield stress calculated from the Herschel-Bulkley model, expressed in pounds-force per hundred square feet;

$L$  is the length of the hydraulic section, expressed in feet.

- e) 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.
- f) 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,  $v_{an}$ , for each case.
- g) From the velocity values near the wall, the corresponding fluid shear rates,  $\gamma_i$ , and wall shear stresses,  $\tau_{wi}$  should be calculated using Equation (N.12) and Equation (N.13).

$$\gamma_i = \frac{\Delta v_{an}}{5 \times d_i} \quad (\text{N.12})$$

where

$\gamma_i$  is the fluid shear rate, expressed in reciprocal seconds;

$\Delta v_{an}$  is the change in annular velocity, expressed in feet per minute;

$d_i$  is the distance from the outer wall, expressed in inches

and

$$\tau_{Wi} = \tau_Y + (k \times \gamma_i) \quad (N.13)$$

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 calculated from the Herschel-Bulkley model, expressed in pounds-force per one hundred square feet;

$k$  is the drilling fluid consistency factor (Herschel-Bulkley model), expressed in  $\text{lb}\cdot\text{s}^n/100\text{ft}^2$ ;

NOTE  $n$ , is the drilling fluid flow behavior index (Herschel-Bulkley model, see API 13D).

$\gamma_i$  is the fluid shear rate, expressed in reciprocal seconds.

h) 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.

i) The critical wall shear stress for an annular velocity of 30 ft/min is calculated using Equation (N.14).

$$\tau_{W,Cr} = \tau_Y + (b \times v_{an}) \quad (N.14)$$

where

$\tau_{W,Cr}$  is the critical wall shear stress, expressed in pounds-force per one hundred square feet;

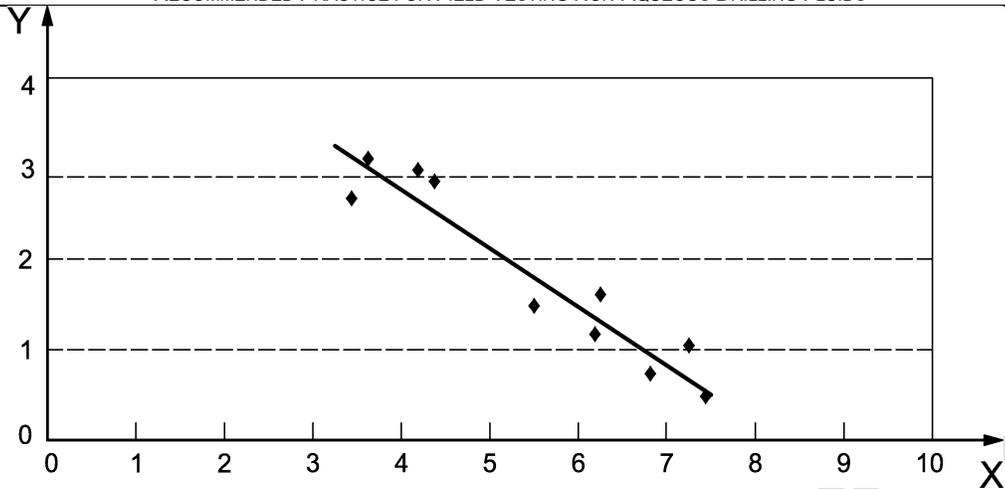
$\tau_Y$  is the drilling fluid yield stress, expressed in pounds-force per one hundred square feet;

$b$  is the slope of the annular velocity and shear stress at the wall in laminar flow, as defined in N.7.2 item h);

$v_{an}$  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 [35]).

j) With the value calculated in Equation (N.14), the predicted maximum weight-material sag, expressed in pounds per gallon, can read from Figure N.10.



**Key**

**X** Wall shear stress, lbf/100-ft<sup>2</sup>

**Y** Predicted dynamic sag, lb/gal

**Figure N.10—Predicted Dynamic Sag as a Function of Calculated Values of Wall Shear Stress,  $\tau_{wi}$**

### N.7.3 Comparison of Laboratory and Field Data

The predicted maximum weight-material sag under dynamic conditions was developed from laboratory data where testing conditions were very favorable for the initiation of weight-material sag.

In the field, these conditions are often not as favorable, and the hydraulic method described here usually over-predicts the magnitude of measured sag<sup>[37]</sup>.

### N.8 Additional Resource Literature

The analysis of weight-material sag remains an active research area. The Bibliography lists additional resource literature (see References [38], [39], [40], [41], and [42]), which expands on the technology given in this annex. A literature search is recommended to find additional references and recent publications.

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**Annex O**  
(informative)  
**Example Non-aqueous Based Drilling Fluid Report**

BALLOT DRAFT



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<sup>9</sup> ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, www.astm.org.

<sup>10</sup> International Organization for Standardization, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, www.iso.org.

<sup>11</sup> Standards Norway, Lilleakerveien 2a, Oslo, P.O. Box 252, NO-1326 Lysaker, Norway; salg@standard.no

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