

Field Testing Nonaqueous-based Drilling Fluids

API RECOMMENDED PRACTICE 13B-2
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Field Testing Nonaqueous-based Drilling Fluids

1 Scope

This recommended practice provides standard procedures for determining the following characteristics of nonaqueous drilling fluids (NADFs):

- a) drilling fluid density (mud weight);
- b) viscosity and gel strength;
- c) filtration;
- d) nonaqueous fluid (NAF), water, and solids concentrations;
- e) alkalinity, chloride concentration, and calcium concentration;
- f) electrical stability (ES);
- g) lime and calcium concentrations, calcium chloride, and sodium chloride concentrations;
- h) low-gravity solids and weighting material concentrations;
- i) sand content;
- j) high-temperature, high-pressure (HTHP) filtration using the permeability plugging apparatus (PPA).

Calibration and verification methods for glassware, thermometers, viscometers, retort kit cups, and drilling fluid balances shall be required for the application of recommended test methods; procedures are provided in Annex J.

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

- shear strength (see Annex A);
- NAF retained on cuttings (see Annex B);
- drilling fluid activity (see Annex C);
- aniline point (see Annex D);
- example of lime, salinity, and solids concentration calculations (see Annex E);
- sampling, inspection, and rejection of drilling fluids materials (see Annex F);
- rig-site sampling (see Annex G);
- cuttings activity (see Annex H);
- active sulfides (see Annex I);
- elastomer compatibility with NADF (see Annex K);
- identification and monitoring of weight-material sag (see Annex L);
- NADF daily report form (see Annex M).

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 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 (ACS) and listed in the Chemical Abstracts Service (CAS).

3.2

calibration

Calibration refers to the verification of the accuracy of an instrument or a piece of equipment and to the adjustments made so that it meets a specification or tolerance.

NOTE In this document, the word “calibrate” also refers to determining correction factors for measurements with instruments and equipment that cannot be adjusted if they are found to be inaccurate.

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).

NOTE 1 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/gal¹) 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.

3.4

nonaqueous drilling fluid

NADF

Drilling fluids prepared using a nonaqueous base fluid (NAF), such as synthetic oils, mineral oils, linear paraffins, olefins, esters, and diesel.

3.5

pound (lb)

US customary (USC) unit 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.

¹ Gallon as used throughout this standard refers to the US gallon of 3.7854 liters.

3.7

spurt loss

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

4 Symbols, Acronyms, 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 reactivity of drilled cuttings
a_W	measure of the chemical potential (water activity) or inhibition potential of salt water solutions
a_{df}	measure of the chemical potential (water activity) or inhibition potential 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 L.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
$c_{CaCl_2,df,SI}$	whole-NADF calcium chloride concentration, expressed in kilograms per cubic meter
$c_{CaCl_2,df,USC}$	whole-NADF calcium chloride concentration, expressed in pounds per barrel
$c_{Ca(OH)_2,Act,SI}$	active concentration of field lime in the whole-NADF, expressed in kilograms per cubic meter
$c_{Ca(OH)_2,Act,USC}$	active concentration of field lime in the whole-NADF, expressed in pounds per barrel
$c_{Ca(OH)_2,df,SI}$	whole-NADF total lime concentration, expressed in kilograms per cubic meter
$c_{Ca(OH)_2,df,USC}$	whole-NADF total lime concentration, expressed in pounds per barrel
$c_{Cl,df}$	whole-NADF chloride concentration, expressed in milligrams per liter
$c_{Cl-(CaCl_2),df}$	whole-NADF chloride concentration as calcium chloride, expressed in milligrams per liter

$c_{\text{Cl-(NaCl),df}}$	whole-NADF chloride concentration from sodium chloride, expressed in milligrams per liter
$c_{\text{NaCl,Aq}}$	aqueous-phase sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl,df}}$	whole-NADF sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl,df,SI}}$	whole-NADF sodium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{NaCl,df,USC}}$	whole-NADF sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl,df,InSol}}$	whole-NADF insoluble sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl,df,InSol,SI}}$	whole-NADF insoluble sodium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{NaCl,df,InSol,USC}}$	whole-NADF insoluble sodium chloride concentration, expressed in pounds per barrel
$c_{\text{NaCl,df,Sol}}$	whole-NADF soluble sodium chloride concentration, expressed in milligrams per liter
$c_{\text{NaCl,df,Sol,SI}}$	whole-NADF soluble sodium chloride concentration, expressed in kilograms per cubic meter
$c_{\text{NaCl,df,Sol,USC}}$	whole-NADF soluble sodium chloride concentration, expressed in pounds per barrel
$c_{\text{LG,SI}}$	low-gravity solids concentration, expressed in kilograms per cubic meter
$c_{\text{LG,USC}}$	low-gravity solids concentration, expressed in pounds per barrel
c_{S}	concentration of active sulfides, expressed in milligrams per liter
$c_{\text{WM,SI}}$	weighting material concentration, expressed in kilograms per cubic meter
$c_{\text{WM,USC}}$	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 $\text{lbf}\cdot\text{s}^n/100\text{ ft}^2$ 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
$m_{\text{NAF-C}}$	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-NADF sample, expressed in grams
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
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_{AgNO_3}	volume of 0.282 mol/L (0.282 N) silver nitrate reagent, expressed in milliliters
V_{B}	base alkalinity demand (volume of 0.1 mol/L (0.1 N) NaOH), expressed in milliliters
V_{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_{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_{L}	total volume of condensed liquids (NAF and water), expressed in milliliters
V_{M}	receiver (graduated cylinder) volume at specific mark, expressed in milliliters
V_{NAF}	volume of NAF, expressed in milliliters
V_{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
β_{10m}	gel strength at 10 min, expressed in pounds-force per one hundred square feet
β_{10s}	initial gel strength at 10 s, expressed in pounds-force per one hundred square feet
$\Gamma_{DFG,SI}$	drilling fluid depth gradient, expressed in kilopascals per meter
$\Gamma_{DFG,USC}$	drilling fluid depth gradient, expressed in pounds-force per square inch per foot
γ_{SI}	shear strength of the drilling fluid, expressed in pascals
γ_{USC}	shear strength of the drilling fluid, expressed in pounds-force per one hundred square feet
$\dot{\gamma}_i$	fluid shear rate, expressed in reciprocal seconds
ΔP	anticipated pressure increase, expressed in pounds-force per square inch
ΔP_{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
Δv_{an}	change in annular velocity, expressed in feet per minute
ε	inner pipe eccentricity, dimensionless
η	drill-pipe rotation, expressed in revolutions per minute
η_{AV}	apparent viscosity, expressed in millipascal•seconds (centipoises)
η_{PV}	plastic viscosity, expressed in millipascal•seconds (centipoises)
v_{sf}	static filtration rate (volume rate of flow), expressed in milliliters per minute
ϕ_{B}	volume fraction of aqueous-phase (brine), expressed as a percentage of the whole-NADF
ϕ_{DS}	corrected volume fraction of solids, expressed as a percentage of the whole-NADF
ϕ_{LG}	volume fraction of the low-gravity solids, expressed as a percentage of the whole-NADF
ϕ_{NAF}	volume fraction of NAF, expressed as a percentage of the whole-NADF
ϕ_{W}	volume fraction of water, expressed as a percentage of the whole-NADF
ϕ_{WM}	volume fraction of the weighting material solids, expressed as a percentage of the whole-NADF
ϕ_{ds}	volume fraction of dried retort solids, expressed as a percentage of the whole-NADF
ρ	drilling fluid relative density (dimensionless)
ρ_{B}	NADF aqueous-phase (brine) density, expressed in grams per milliliter
$\rho_{\text{ECD-Hyd}}$	hydraulic ECD, includes ESD, circulating pressure drop, and added density effects of drilled cuttings in the annulus, expressed in pounds per gallon
$\rho_{\text{ECD-Tot}}$	total predicted drilling fluid ECD in the annulus while circulating, expressed in pounds per gallon
ρ_{LG}	density of the low-gravity solids, expressed in grams per milliliter
ρ_{NAF}	density of the NAF being used, expressed in grams per milliliter
ρ_{SS}	average density of the suspended solids, expressed in grams per milliliter
ρ_{W}	water density, expressed in grams per milliliter, at the test temperature (see Table J.1)

ρ_{WM}	density of the weighting material solids, expressed in grams per milliliter
ρ_{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
ρ_{max}	maximum recorded drilling fluid density, expressed in pounds per gallon
ρ_{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
τ_{Wi}	wall shear-stress, expressed in pounds-force per one hundred square feet
τ_Y	drilling fluid yield stress, calculated from the Herschel-Bulkley model, expressed in pounds-force per one hundred square feet

4.2 Acronyms and Abbreviations

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

ACS	American Chemical Society
BAD	base alkalinity demand
CAS	Chemical Abstracts Service
ECD	equivalent circulating density
EDTA	sodium salt of ethylenediaminetetraacetic acid dihydrate
ES	electrical stability
ESD	equivalent static density
HTHP	high temperature, high pressure
NAF	nonaqueous fluid
NADF	nonaqueous drilling fluid
NIST	National Institute of Standards and Technology
OBR	oil-to-brine ratio
OWR	oil-to-water ratio
PNP	propylene glycol normal-propyl ether
PPA	permeability plugging apparatus

PPE	personal protective equipment
PPT	permeability plugging test
PTFE	polytetrafluoroethylene
PV	plastic viscosity
ROC	retained NAF (oil) on cuttings
SI	International System of Units
SRE	standard reference elastomer
TC	to contain
TD	to deliver
TVD	true vertical depth
USC	US customary
VSST	viscometer sag shoe test

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 ± 0.01 g/mL or ± 10 kg/m³ (± 0.1 lb/gal or ± 0.5 lb/ft³).

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

The mud balance shall be calibrated frequently with fresh water. The recommended frequency is prior to each set of measurements (e.g. daily or by each work shift), or as specified by the operator, prevailing regulation, drilling contractor, drilling fluid company, or other controlling party. Fresh water shall give a reading of 1.00 g/mL ± 0.005 g/mL or 1000 kg/m³ ± 5 kg/m³ (8.345 lb/gal ± 0.05 lb/gal or 62.4 lb/ft³ ± 0.25 lb/ft³) 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 ± 1 °C (± 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 Calculations

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

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

Equations (1) to (3) shall be used to convert the density, ρ_{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.

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

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 ³	lb/gal	lb/ft ³
g/mL	1	1000	8.345	62.43
kg/m ³	0.001	1	0.008345	0.06243
lb/gal	0.120	120	1	7.4805
lb/ft ³	0.0160	16.02	0.1337	1

Equations (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-force per square inch, per foot.

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

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

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

$$\Gamma_{\text{DFG,USC}} = 0.00694 \times \rho_{\text{df,USC2}} \quad (7)$$

where

ρ_{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;

$\rho_{\text{df,USC2}}$ 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

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

6 Determination of Drilling Fluid Density—Pressurized Balance Method

6.1 Principle

6.1.1 The pressurized mud balance provides a more accurate 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 ± 0.01 g/mL or ± 10 kg/m³ (± 0.1 lb/gal or ± 0.5 lb/ft³).

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

The mud balance shall be calibrated frequently with fresh water. The recommended frequency is prior to each set of measurements (e.g. daily or by each work shift), or as specified by the operator, prevailing regulation, drilling contractor, drilling fluid company, or other controlling party. Fresh water shall give a reading of 1.00 g/mL ± 0.005 g/mL or 1000 kg/m³ ± 5 kg/m³ (8.345 lb/gal ± 0.05 lb/gal or 62.4 lb/ft³ ± 0.25 lb/ft³) at 21 °C (70 °F) when the bubble level is centered. If it does not, the instrument shall be calibrated by adjusting the balancing screw or the amount of shot (metal beads) in the well at the end of the graduated arm until it meets the accuracy requirement, or the instrument shall be removed from service. The instrument shall be calibrated on a less frequent basis (e.g. annually) according to the procedure as specified by the manufacturer or using a two-point calibration. Calibration according to the procedure as specified by the manufacturer or using a two-point calibration shall always be performed after damage, repair, or replacement of items such as the sample cup lid, threaded lid retainer, or the shot well screw.

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

6.3 Pressurized Mud Balance Procedure

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

The pressurized mud balance instrument shall be set on a 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.

If excess fluid is not expelled from the open check valve during assembly, the sample cup was not filled sufficiently, and the fill procedure is 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 cleanup 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-force 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/m³ (0.1 lb/gal or 0.5 lb/ft³).

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. The equipment is as follows:

- a) Marsh funnel: a simple device for measuring relative 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 $26\text{ s} \pm 0.5\text{ s}$, 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 $\pm 5\text{ s}$ 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 ^[20], powered by an electric motor or a hand crank.

Drilling fluid is placed in the annular space between two concentric cylinders. The outer cylinder (rotor sleeve) is driven at a constant rotational velocity. The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder (the bob). A torsion spring restrains the movement of the bob and a dial attached to the bob indicates displacement of the bob. Instrument constants shall be adjusted so that 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

For testing at temperature higher than 90 °C (190 °F), a solid bob is 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 ² 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 s per hour.

7.3.1.3 Thermostatically controlled viscometer cup (thermocup):

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

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

7.3.1.5 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 $50\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($120\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$) or $65\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($150\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$) for reference comparisons to historical data. Testing at a lower temperature, such as $4\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($40\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$), is 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 $90\text{ }^{\circ}\text{C}$ ($190\text{ }^{\circ}\text{F}$). 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. Measurements of dial (or digital) readings are performed stepwise from the highest to the lowest revolutions per minute—i.e. typically, at 200 r/min, 100 r/min, 6 r/min and 3 r/min). Improved modeling accuracy can be achieved by also measuring the R_{60} and R_{30} shear stress readings at speeds of 60 r/min and 30 r/min.

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

If using a hand-crank 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, β_{10s} , at 10 s, in pounds-force per one hundred square feet (lbf/100 ft²).

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

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

7.3.3 Calculations

7.3.3.1 Direct-indicating Viscometer Calculation Constants

The dimensions for the rotor, 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 ft² (0.5 Pa), or more exactly 1 degree of deflection = 1.067 lbf/100 ft² and = 0.511 Pa;
- 1 r/min of the rotor equals a shear rate of 1.7023 s⁻¹.

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⁻¹), 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, η_{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 M, Example of Nonaqueous Drilling Fluid Daily Report).

NOTE 2 1 cP = 1 mPa•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

η_{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 ft² 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.

7.3.3.2 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

η_{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 ft², is commonly known in the industry by the abbreviation YP (see Annex M, Example of Nonaqueous Drilling Fluid Daily Report).

The yield point value calculated with Equation (10) from the dial readings is an approximation of lbf/100 ft². Dial reading units are degrees of deflection (1/360 of 1 full rotation). Each 1 degree of deflection is equal to 1.067 lbf/100 ft² in USC units. In Equation (10), the exact unit correction is neglected for simplicity in reporting.

7.3.3.4 Drilling Fluid Apparent Viscosity

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

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

where

R_{600} is the shear stress reading at 600 revolutions per minute, 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 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 HTHP static conditions. Field static HTHP tests are conducted using a differential pressure of 3450 kPa (500 lbf/in.²) for 30 min. Two filtration procedures are given: one for testing up to 175 °C (350 °F) and one for testing 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 for equipment and testing 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 the following:

- a) filter cell, rated for working pressures up to 9000 kPa (1300 lbf/in.²) 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 manufacturers' 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, NAFs, 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. ²	kPa	lbf/in. ²
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 ² 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 s 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 ($1/32$ in.), to measure filter cake thickness.

8.2.2 Procedure

In order to conduct 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.²), the following procedure shall be used.

- 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).

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

² This filter paper is a calendered, hardened, qualitative, low-ash filter paper made from cotton linters. The filter paper has the following typical properties that may vary slightly by manufacturer: slow filtration rate: 2685 herzbergs; particle retention in liquid: 2 μ m to 5 μ m; ash content: 0.015 % by weight; basis weight: 92 g/m²; diameter: 63.5 mm (2.5 in.); thickness: 0.137 mm.

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.²) for temperatures below 150 °C (300 °F) or 1400 kPa (200 lbf/in.²) 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.²) for temperatures below 150 °C (300 °F) or 1400 kPa (200 lbf/in.²) 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.²) for temperatures below 150 °C (300 °F) or 4850 kPa (700 lbf/in.²) 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 ± 3 °C (± 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 cm² (7.1 in.²). For example, if the filter area is 22.6 cm² (3.5 in.²), 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 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 Daily Report. 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 cm² (7.1 in.²). HTHP filter cells usually have half the standard filter area 22.6 cm² (3.5 in.²), thus double the 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.²) 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 $>190\text{ }^{\circ}\text{C}$ ($>375\text{ }^{\circ}\text{F}$) 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.²).

- 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 $150\text{ }^{\circ}\text{C}$ ($300\text{ }^{\circ}\text{F}$). Failure to know the pressure/temperature rating of equipment in use can result in serious injury. Testing at high temperature and high pressure calls for added safety precautions. The 175 mL and 250 mL filtration cells are not recommended for use at these higher temperatures and pressures. Strict adherence to manufacturers' recommendations as to sample volumes, equipment temperatures and pressures, O-ring material selection and inspection, and other operating instructions is essential. High temperature O-rings and elastomer seals are required for high temperatures. Equipment manufacturers often recommend replacement or inspection after every test. Inspection should include making sure the O-rings and elastomer seals are pliable with no permanent deformations, nicks, or cuts, etc. A loss of pressure during a test may be an indication of an O-ring or elastomer seal failure and should be corrected. Using appropriate 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 ², qualitative, hardened, low-ash, slow filtration rate, grade 50 (see 8.2.1.2), and glass fiber backing disk ³ for temperatures up to $200\text{ }^{\circ}\text{C}$ ($400\text{ }^{\circ}\text{F}$). A new filter medium shall be required for each test.
- b) Porous sintered stainless-steel disc, Dynalloy X-5 ⁴ or equivalent, diameter 6.35 cm (2.5 in.), for temperatures above $200\text{ }^{\circ}\text{C}$ ($400\text{ }^{\circ}\text{F}$). 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 ± 5 s per hour.

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

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

³ Glass fiber backing disks is required for testing between $175\text{ }^{\circ}\text{C}$ ($350\text{ }^{\circ}\text{F}$) up to $200\text{ }^{\circ}\text{C}$ ($400\text{ }^{\circ}\text{F}$). These backing disks are a separate item and are inserted between the filter paper and the filter media support of the filter cell.

⁴ 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 13B-2 and does not constitute an endorsement by API of these products.

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.²), the following procedure shall be used.

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

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

If the filtration unit is equipped with a thermocouple in direct contact with the drilling fluid, then that temperature shall be monitored and reported during the filtration test. Under the “Comments” section, record if the results were based on fluid temperature measured with a direct contact thermocouple or using the 30 min 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 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.²) higher than the back-pressure. This will start the filtration process. Start the timer.

Maintain the test temperature to within $\pm 3\text{ }^{\circ}\text{C}$ ($\pm 5\text{ }^{\circ}\text{F}$) 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^2 (7.1 in.^2). For example, if the filter area is 22.6 cm^2 (3.5 in.^2), double the filtrate volume reported. 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 $50\text{ }^{\circ}\text{C}$ ($125\text{ }^{\circ}\text{F}$). 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 ($1/32\text{ in.}$).
- 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 Daily Report. 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 cm^2 (7.1 in.^2). HTHP filter cells usually have half the standard filter area 22.6 cm^2 (3.5 in.^2), thus double the measured filtrate volume before reporting.

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

9 Retort Test for NAF, Water, and Solids Concentrations

9.1 Principle

9.1.1 A retort test measures water and NAF 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.

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 that 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 ± 0.05 mL), 20 mL (precision ± 0.1 mL), and 50 mL (precision ± 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**, sufficient heating capacity to achieve $500\text{ }^{\circ}\text{C} \pm 40\text{ }^{\circ}\text{C}$ ($930\text{ }^{\circ}\text{F} \pm 70\text{ }^{\circ}\text{F}$).

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
Precision	0 % to 100 %	±0.05 mL	±0.10 mL	±0.25 mL
Frequency of graduations marks	0 % to 100 %	0.10 mL	0.10 mL	0.50 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 (see 7.2.1.1).

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

9.2.11 Mud balance (see 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 that 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 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. First, always read the meniscus with the interface at eye level. Second, for the air-to-liquid meniscus, read the volume at the “lowest point” of the meniscus that is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids, it might be necessary to estimate the top of the liquid in the middle of the cylinder. Third, for the water-to-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_{NAF} is the volume of NAF, expressed in milliliters;

V_{L} is the total volume of condensed liquids (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).

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

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

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

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

NOTE V_{df} is the gravimetrically verified volume of the receiver (see 9.2.2).

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

ϕ_{W} is the volume fraction of water, expressed as a percentage of the whole-NADF;

V_{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.

NOTE V_{df} is the gravimetrically verified volume of the receiver (see 9.2.2).

9.4.4 Volume Fraction of Dried Solids

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

$$\varphi_{ds} = 100 - (\varphi_W + \varphi_{NAF}) \quad (15)$$

where

φ_{ds} is the volume fraction of dried retort solids, expressed as a percentage of the whole-NADF;

φ_W is the volume fraction of water, expressed as a percentage of the whole-NADF;

φ_{NAF} is the volume fraction of NAF, expressed as a percentage of the whole-NADF.

NOTE The calculated volume fraction dried retort solids will overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity should 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 that 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 ρ_{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 ρ_{NAF} , to the nearest 0.01 g/mL, 10 kg/m³ (0.1 lb/gal or 0.5 lb/ft³).

- 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 NADF 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. First, always read the meniscus with the interface at eye level. Second, for the air-to-liquid meniscus, read the volume at the “lowest point” of the meniscus that is in the middle of the liquid receiver at the very bottom of the liquid. For opaque liquids it might be necessary to estimate the top of the liquid in the middle of the cylinder. Third, for the water-to-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_{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_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):

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

where

- ϕ_{NAF} is the volume fraction of NAF, expressed as a percentage of the whole-NADF;
- V_{NAF} is the volume of NAF, expressed in milliliters;
- V_{df} is the whole-NADF sample volume, expressed in milliliters;
- m_{NAF} is the mass of NAF, expressed in grams;
- ρ_{NAF} is the density of the NAF being used, expressed in grams per milliliter;
- m_{df} is the mass of the whole-NADF sample, expressed in grams;
- ρ_{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

- ϕ_{W} is the volume fraction of water, expressed as a percentage of the whole-NADF;
- V_{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;
- ρ_{df} is the drilling fluid (NADF) 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):

$$\phi_{\text{ds}} = 100 - (\phi_{\text{W}} + \phi_{\text{NAF}}) \quad (23)$$

where

- ϕ_{ds} is the volume fraction of dried retort solids, expressed as a percentage of the whole-NADF;
- ϕ_{W} is the volume fraction of water, expressed as a percentage of the whole-NADF;
- ϕ_{NAF} is the volume fraction of NAF, expressed as a percentage of the whole-NADF.

NOTE The calculated volume fraction dried retort solids will overestimate the volume of suspended solids because of dissolved salts. A volumetric correction for salinity should 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):

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

where

- ρ_{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 that 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 nonreacted “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 that 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 that measures the volume of a standard calcium-chelating agent [EDTA (ethylenediaminetetraacetic 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 (CaCl_2) and lime [CaO or $\text{Ca}(\text{OH})_2$] used to prepare the NADF, as well as calcium from drilled gypsum or anhydrite (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 CaCl_2 and sodium chloride (NaCl) concentrations in the aqueous phase of NADF.

10.1.5 This calcium analysis excludes magnesium (Mg^{+2}) ions. If 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 and 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 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), $c_{\text{AgNO}_3} = 47.91 \text{ g/L}$ ($c_{\text{Cl}^-} \equiv 0.01 \text{ g/mL}$ 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 CaCO_3 , 1 mL = 4000 mg/L 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.

- Add 100 mL of PNP solvent to a 400 mL beaker or pint jar. See 10.2.1 for further information about PNP.
- Add 200 mL of deionized (or distilled) water.
- Add 15 drops of phenolphthalein indicator solution.
- 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.

- Continue stirring for 5 min. If the pink color remains, the endpoint 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 indicate 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 that have a dark filtrate or where the endpoint 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.²). 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 endpoint 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, $V_{\text{H}_2\text{SO}_4}$, expressed in milliliters, required to reach the endpoint 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 Equation (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 endpoint 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_{NaOH}), in milliliters once an endpoint has been reached.
- m) Use the volume of 0.1 mol/L (0.1 N) sodium hydroxide (V_{NaOH}), expressed in milliliters, required to reach the endpoint in 10.4.1 item l) to calculate the whole-NADF alkalinity (V_K). If V_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_K is a measurement of the whole-NADF acidity. Negative V_K can be used to calculate the amount of lime required to neutralize the excess acidity using Equations (29) to (32) (12.3.1).

10.4.2 Calculation

The whole-NADF alkalinity, V_K , shall be calculated using Equation (26):

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

where

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

V_{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 at least 10 drops to 20 drops of 0.05 mol/L (0.1 N) sulfuric acid.

- b) Add 3 mL of potassium chromate indicator solution (see 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 that have a dark filtrate or where the endpoint 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.²). 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 (see 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 endpoint of the titration.
- e) Use the volume of 0.282 mol/L (0.282 N) silver nitrate reagent required, V_{AgNO_3} , in milliliters, to reach the endpoint 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_{AgNO_3} , to reach the endpoint 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_{AgNO_3} is the volume of 0.282 mol/L (0.282 N) silver nitrate reagent, in milliliters;
- V_{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.

- Add 100 mL of the PNP solvent to a titration vessel that has a cap.
- Fill the 5 mL syringe beyond the 3 mL mark with whole-NADF.
- Displace 2.0 mL of whole-NADF into the titration vessel. Record as V_{df} .
- Stir or swirl the NADF and solvent until the mixture is homogeneous.
- 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).
- Filtration option:

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

- Stir the mixture for 5 min with a magnetic stirrer such that the vortex is on-half of the liquid height.
- Pour the mixture into an API low-temperature, low-pressure filter press fitted with filter paper (see API 13B-1).
- 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.²). 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).

- Collect all the filtrate in a clean beaker until the cell blows dry.
- Use all the filtrate and continue with item g).
- Add 6.0 mL [1 mol/L (1N) sodium hydroxide] buffer solution for calcium (see 10.2.7).
- Add 0.7 g to 1.0 g hydroxynaphthol blue calcium indicator powder (see 10.2.8).
- Place the titration vessel on the magnetic stirrer and drop in a stirring bar.
- 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 endpoint. Record the volume of EDTA added, V_{EDTA} , in milliliters.